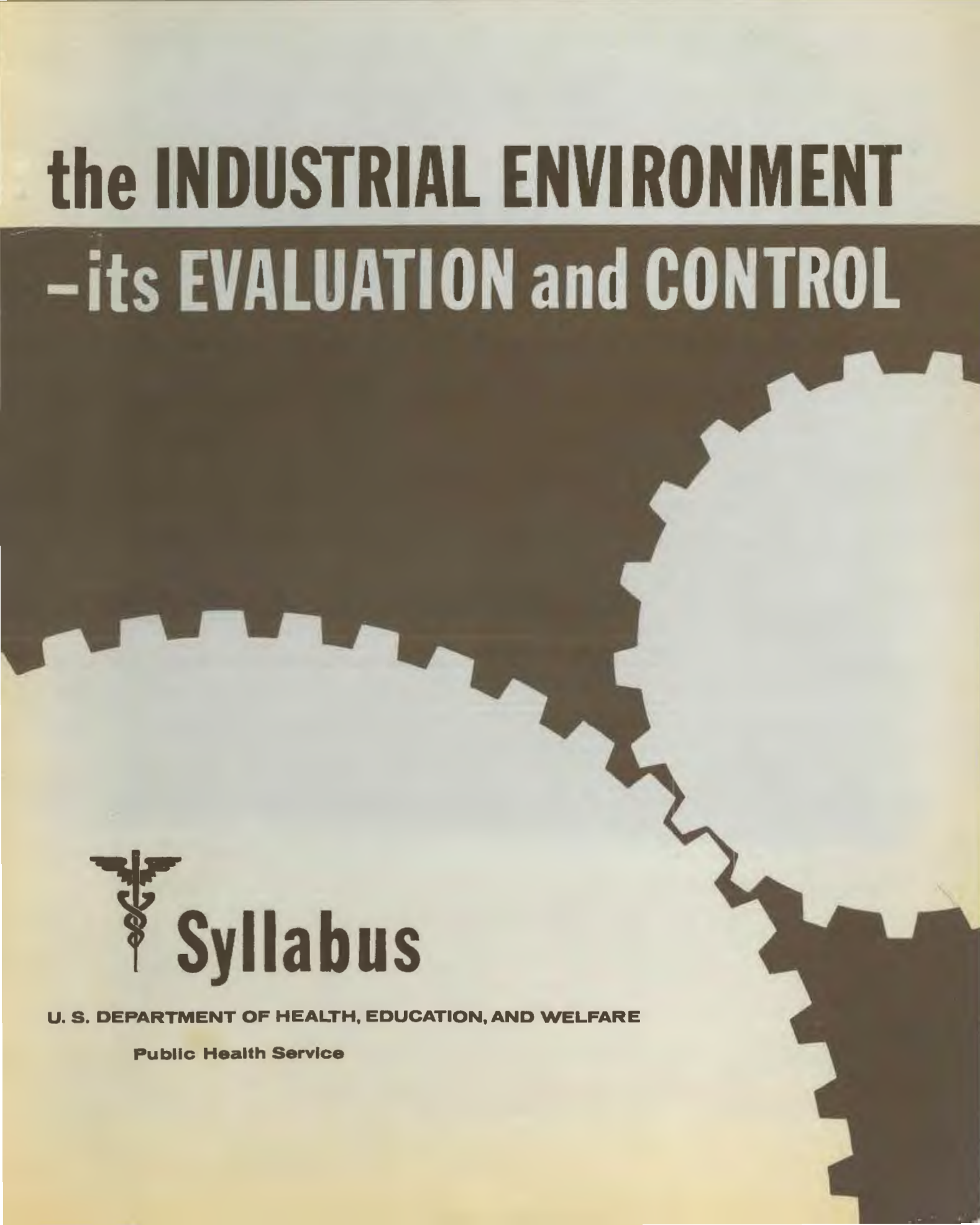
the INDUSTRIAL ENVIRONMENT
–its EVALUATION and CONTROL



Syllabus

U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

Public Health Service

the Industrial Environment .... its Evaluation and Control

Syllabus

Edited by:

Charles H. Powell, Sc.D.
Andrew D. Hosey

U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Environmental Health Service
Bureau of Occupational Safety and Health

First Printing, 1958
Reprinted, 1961
Revised, 1965

Reprinted 1970

PUBLIC HEALTH SERVICE PUBLICATION No. 614

UNITED STATES GOVERNMENT PRINTING OFFICE, WASHINGTON: 1965

PREFACE

This Syllabus presents in summary form the material covered in a two-week training course for industrial hygiene engineers and chemists, offered by the Division of Occupational Health of the Public Health Service. It includes lecture outlines, problems, and reference materials. Laboratory exercises will be published separately. First published in 1958, and reprinted in 1961 with minor corrections, the Syllabus has been extensively revised for this second edition.

Originally designed to supplement instruction offered in the training course, the Syllabus has also been made available to industrial hygienists not attending the course and has been widely used as a guide and reference in the work situation.

The course upon which the Syllabus is based does not attempt to cover the entire field of industrial hygiene. It is designed to instruct engineers and chemists new to the field in the basic procedures and techniques of industrial hygiene, and to give more experienced professionals an opportunity to review these fundamentals and to exchange experiences and information.

Industrial hygiene has been defined as "the science and art of recognizing, evaluating, and controlling occupational health hazards." This volume is devoted primarily, therefore, to the fundamental principles and methods employed in this recognition, evaluation, and control. In addition, however, it includes brief discussions of clinical, toxicological, physiological, and dermatological aspects of occupational health. Many excellent reference sources, including those in the "Selected List of General Reference Sources," are provided on these and other facets of occupational health.

The planning and editing of this edition were done by Dr. Charles H. Powell and Andrew D. Hosey.

Cincinnati, Ohio
May 1965

TABLE OF CONTENTS

Preface	iii
	Section
Introduction	A-1
Selected List of General Reference Sources	A-2
Review of Mathematics	A-3
Review of Analytical Chemistry	A-4
General Principles in Evaluating the Occupational Environment	B-1
Sampling for Particulates	B-2
Air Flow Calibration	B-3
Sampling for Dust	B-4
Optical and Electron Microscopy	B-5
Dust Counting Techniques	B-6
Determination of Particle Size	B-7
Preparation of Known Concentrations of Air Contaminants	B-8
Direct Reading Instruments for Determining Concentrations of Aerosols, Gases, and Vapors	B-9
Sampling and Analysis of Gases and Vapors	B-10
Industrial Toxicology	B-11
The Physician in Industry	B-12
Spectrophotometry	B-13
Emission Spectroscopy	B-14
Polarography	B-15
Principles and Applications of Gas Chromotography	B-16
Ion Exchange Techniques	B-17
Solvent Analysis	B-18
Organic Extraction Methods in Trace Analyses	B-19
Free Silica - Its Forms, Occurrence, and Determination	B-20
Determination of Nitrogen Oxides in Air	B-21
Fluorides - Their Occurrence and Determination	B-22
Sound and Noise	B-23
Physiology of Hearing	B-24
Noise Measurement	B-25
Electromagnetic Spectrum I	B-26
Electromagnetic Spectrum II (Microwave and Coherent Light)	B-27
Heat Stress	B-28
Measurement of the Thermal Environment	B-29
Occupational Dermatoses - Their Recognition, Control and Prevention	B-30
Control of the Occupational Environment	C-1
Control of Noise Exposure	C-2
Comfort Ventilation and Control of the Thermal Environment	C-3
Medical Monitoring of the Occupational Environment	C-4
Dilution Ventilation	C-5
Principles of Ventilation	C-6
Air Flow Measurements	C-7
Local Exhaust Systems	C-8

Section

Fans and Blowers C-9
Design of Ventilation Systems C-10
Methods of Air Cleaning C-11

Conversion Factors and Equivalents D-1
Problems D-2
Solutions of Problems D-3

INTRODUCTION

Development and Practice of Industrial Hygiene

The health hazards associated with certain occupations were probably recognized even before the dawn of history. Throughout antiquity, however, little interest was shown in protecting worker health, perhaps because the dangerous trades were usually restricted to slave labor. Hippocrates, in the fourth century B.C., recognized the toxic properties of lead and recorded a series of attacks of lead colic in a miner. Pliny the Elder, a Roman scholar of the first century A.D., referred to the hazards of handling sulfur and zinc and also described a protective mask, made from a bladder, which was used by workers in dusty trades. About 100 years later, Galen accurately described the pathology of lead poisoning.

In 1473, Ulrich Ellenbog produced the first publication on the subject -- a pamphlet on occupational diseases and injuries among gold miners. He also wrote about the toxic action of carbon monoxide, mercury, lead, and nitric acid. What is more, he offered instruction in hygiene and other preventive measures. In 1556, the German scholar, Agricola, described the diseases of miners and prescribed preventive measures.

The first comprehensive book on occupational medicine was published in 1700 in Italy by the "father of industrial medicine," Bernardino Ramazzini. His "De Morbis Artificum Diatriba" (The Diseases of Workmen) contained accurate descriptions of the occupational diseases of most of the workers of his time. He was perhaps the first person to describe accurately the pathology of silicosis. Unfortunately, his suggested "cautions" were to be largely ignored for several centuries.

In England, Charles Thackrah (1795-1833) devoted his life to the study and prevention of occupational hazards accompanying industrialization, and his influence was felt in both medical and political spheres. The first article on occupational disease to appear in America (in 1837) relied heavily upon Thackrah as an authority.

In early 20th century America, Dr. Alice Hamilton observed industrial conditions at first hand and startled mine owners, factory managers, and State officials with evidence of illness correlated with toxic exposures. She presented concrete proposals for elimination of unhealthful conditions and exercised tremendous influence on both industrial leaders and lawmakers.

Legislation and Compensation

The mechanization which accompanied the Industrial Revolution brought new hazards and intensified old ones. More importantly, the number of industrial workers increased markedly in this period. The resultant increase in injuries eventually brought legislation, such as the Factory Acts of Great Britain. The Health and Morals of Apprentices Act was passed in 1802, and the Child Labor Laws in 1833. In 1901, a law protecting the health of workers was passed.

Prior to the adoption of the compensation principle workers injured on the job had to sue their employers to collect damages, and could not collect if it could be proved that the injury was due to the ordinary risks of the job, or to the negligence of a fellow worker, or if the worker by his own negligence contributed to the injuty.

Switzerland in 1881, and Germany in 1884, led the way in the passage of compensation laws. Within 25 years every major European country had such legislation. The United States lagged in

Prepared by: Charles D. Yaffe
Revised by: Andrew D. Hosey

this respect. In 1908 the Federal government passed a compensation act for certain civil employees and in 1911 the first State compensation laws were passed. Others quickly followed, 42 having such legislation by 1920, and all States by 1948.

Workmen's compensation laws were an important factor in the development of industrial hygiene in the United States. The original compensation laws covered only compensation for accidental injury, occupational diseases being included only insofar as they could be classified as accidents. Today, in most States, there is also some coverage for occupational diseases.

State and Local Programs

Industrial hygiene activities by official agencies, insurance companies and industrial plants were stimulated considerably as occupational diseases became compensable. Investigations into health conditions in industry were begun by State and Federal agencies around 1913 and 1914. During the next twenty years, however, scarcely half-dozen industrial hygiene divisions were put into operation in State agencies. The passage of the Social Security Act in 1935 provided funds with which to establish State programs, and twenty-three programs were started between 1936 and 1939. World War II served as a further stimulus, 31 State, local and territorial units being formed during the 1940-1946 period. The importance of maintaining the health of industrial manpower received recognition and resulted in acceptance of industrial hygiene as a public health function. Today there are occupational health programs in 41 States, Puerto Rico, the District of Columbia, and at least 38 local health departments. In New York, Massachusetts, Illinois, and Washington, the programs are located in labor departments. The other State programs are in the health departments.

Federal Programs

The U. S. Public Health Service and the U. S. Bureau of Mines were the first Federal agencies to investigate the diseases of workmen. Exploratory studies in the mining and steel industries were conducted by the Public Health Service in 1910. The Bureau of Mines, created by an Act of Congress the same year, has maintained an active interest in health problems associated with mining operations.

In 1912, an Act signed by President Taft gave the Public Health Service its first statutory authority to study occupational diseases, and a study was made of health problems in the garment industry. Prompted by the high incidence of tuberculosis among garment workers, this study was one of the earliest attempts to correlate medical findings with the industrial environment.

In 1914, the Public Health Service established its Office of Industrial Hygiene and Sanitation and began its first investigation of silicosis in the mining industry. This was a cooperative study by the Public Health Service and the Bureau of Mines, and it continued until 1923. It was the first of a series of investigations in the dusty trades lasting through 1939. A distinctive feature of the 1914-1923 investigation was its introduction of the epidemiologic approach to the study of occupational diseases. In the years that followed, the Public Health Service studied health hazards in many trades and industries and proposed control measures which were gradually applied to the work environment.

Today, the Division of Occupational Health, with headquarters in Washington, D.C., conducts extensive research and training at its facility in Cincinnati and continues epidemiological investigations of occupational disease on a nation-wide scale. Its field office in Salt Lake City provides consultant service to the western part of the United States and oversees the Division's

long-term study of uranium miners. The Appalachian Coal Miners' Research Unit is located in Beckley, West Virginia.

In 1933, the Tennessee Valley Authority established what is believed to be the first employee health service in a Federal agency.

The three armed services and the Atomic Energy Commission began occupational health programs about 1940. Sixty Naval officers were sent to Harvard and Columbia in 1941 for accelerated training in industrial hygiene, initiating the preventive phase of the Navy's program. Public Law 658, enacted in 1946, authorized health service programs for Federal civilian employees. As a result, in 1946, programs were inaugurated at naval shore installations, the Army established its Environmental Health Laboratory at the Army Chemical Center, Maryland, and the Air Force expanded its activities. In the early 1950's, an occupational health program was established for the Strategic Air Command at Omaha, Nebraska. Today, a majority of Federal employees have access to health services.

Magnitude of the Problem

Despite the rapid growth of industrial hygiene in the last thirty years, it is unfortunately true that, even today, fewer than one-third of the total work force in the United States receives any industrial hygiene services except those provided by official agencies. Private programs are found primarily in large manufacturing establishments and are estimated at fewer than 500. Very few plants with less than 500 employees have their own industrial hygiene personnel. And yet, most American workers are employed in these smaller establishments.

In manufacturing, for example, there were 16.5 million workers in 1960, of whom 9.9 million, or over 60 percent, were in factories having fewer than 500 employees. Only 2,232 manufacturing establishments employ more than 500 workers each, whereas nearly 130,000 plants are in the "under 500 employees" group.

About 50 million Americans are engaged in non-manufacturing employment, such as transportation, trade, and agriculture. Many of these are exposed to occupational hazards as serious as those in manufacturing but only a small percentage are covered by on-the-job medical or hygiene services. There are 9 million agricultural workers in the United States, for example, and many of these may be exposed to potential health damage from pesticides.

Official agencies attempt to provide the most necessary occupational health services to the millions of workers who are not otherwise covered. However, there are only about 738 State and local occupational health personnel (Directory of Governmental Occupational Health Personnel, U. S. Department of Health, Education, and Welfare, Public Health Service, Division of Occupational Health, 1965). The need for more industrial physicians, hygienists, chemists, nurses, and other specialists is evident.

In 1962, it was estimated that, to provide adequate services for all workers, industry would need 1,970 full-time and 3,500 part-time physicians; 5,600 full-time and 590 part-time nurses; and 1,220 industrial hygienists. State and local agencies would need 335 in the three categories, and Federal agencies, 235. By 1975, these needs will have increased in proportion to the growth of our employed population. (An Analysis of the Professional Manpower Need and Demand in the United States for the Various Disciplines of Occupational Health - 1962 and Projected to 1975. Department of Health, Education, and Welfare, Public Health Service, Division of Occupational Health, 1962, unpublished).

Nature of Programs

Virtually all industrial hygiene programs in private industry appraise the

working environment for air contaminants. Evaluations of heat and humidity, noise, radiant heat, illumination, ionizing radiation and ultraviolet radiation are also regular functions of most of these units. More than half of them deal with air-borne waste, analyze biological specimens, make clinical tests, and test the efficiency of air cleaners. They also make detailed recommendations for control of unsatisfactory conditions, including process ventilation, general ventilation and make-up air. Approximately 60% of the units were under the administrative supervision of the medical director and about 10% under directors of personnel or industrial relations, while the others reported directly to various other officials including the company president.

Occupational Health programs of State and local agencies embrace a wide range of functions. A typical State agency offers the following services:

1. Engineering, laboratory, medical, and nursing consultation on industrial health problems.

2. Investigation of occupational disease cases reported by physicians.

3. Assistance in planning or expanding plant medical and nursing services.

4. Surveys and detailed studies of work places for conditions or processes harmful to health.

5. Laboratory analysis of environmental and biological samples and development of methods.

6. Examination of plans prepared by industry for the control of hazards.

7. Provision of educational and informational material on industrial health subjects.

The Division of Occupational Health, U. S. Public Health, performs the following functions:

1. Study of health hazards on the site and in the laboratory.

2. Provision of technical and consultative assistance to State and local health departments.

3. Encouragement of preventive health services for employees.

4. Provision of educational and informational material on industrial health subjects.

5. Specialized training of State, local and industrial health personnel.

SELECTED LIST OF GENERAL REFERENCE SOURCES

Introduction

The following is a list of the scientific periodicals and reference texts used by the industrial hygienist. The list of literature sources presented is necessarily selective but is intended to include those journals and texts of greatest usefulness to the workers in this field.

It is essential that the value of the several abstracting journals be recognized. The Industrial Hygiene Digest is an excellent source of current information on toxicology, pharmacology, dermatology, engineering, and analytical chemistry. Chemical Abstracts and Analytical Abstracts are indispensable to the chemist who is in need of new methods or special modifications of existing procedures. Chemical Abstracts, Vol. 56 beginning, January 1962 has been revised and a new section added on "Toxicology, Air Pollution and Industrial Hygiene" which make this journal even more valuable to the industrial hygienist. Public Health Engineering Abstracts has sections devoted to occupational health and air pollution.

In the group of journals which publish full technical papers, Analytical Chemistry, the Analyst, and the American Industrial Hygiene Association Journal are indispensable to the chemist. Noise Control, Journal of the Air Pollution Control Association, Illuminating Engineering, and Air Conditioning, Heating and Ventilating will be of considerable use in addition to those journals mentioned above.

The following reference sources are classified, for convenience, into two general groups, namely: (1) journals and other periodicals; and (2) textbooks and other references. The latter group is further subdivided into sections of particular interest to the different professional disciplines, i.e., chemistry, engineering, medicine, and toxicology. All references are listed alphabetically according to title to provide a more useful bibliography.

Journals and Other Periodicals

Air Conditioning, Heating and Ventilating. (Vol. 1, 1904), The Industrial Press, 93 Worth Street, New York 3, New York.

Archives of Dermatology (Vol. 73, 1956 to present); formerly Archives of Dermatology and Syphilology (Vol. 1, 1918 - Vol. 72, 1955). American Medical Association, Chicago, Illinois.

Archives of Environmental Health (Vol. 1, July 1960), formerly A.M.A. Archives of Industrial Health (Vol. 11, 1955 to 1960); formerly Archives of Industrial Hygiene and Occupational Medicine (Vol. 1, 1950-Vol. 10, 1954); formerly Journal of Industrial Hygiene and Toxicology (Vol. 20, 1938-Vol. 31, 1949); and formerly Journal of Industrial Hygiene Vol. 1, 1919-Vol. 19, 1937). American Medical Association, Chicago, Illinois.

American Industrial Hygiene Association Journal (Vol. 19, 1958 to date); formerly A.I.H.A. Quarterly (Vol. 1, 1940 to Vol. 18, 1957). American Industrial Hygiene Association, 14125 Prevost, Detroit, Michigan.

American Journal of Public Health and the Nation's Health (Vol. 1, 1911). American Public Health Association, Inc., 883 Broadway, Albany 1, New York.

Compiled by: Lial Brewer
Updated by: Andrew D. Hosey

Analyst (Vol. 1, 1876). W. Heffer and Sons, Ltd., Cambridge, England.

Analytical Abstracts. W. Heffer and Sons, Ltd., Cambridge, England.

Analytical Chemistry (Vol. 20, 1948 to present); formerly Industrial and Engineering Chemistry, Analytical Edition (Vol. 1, 1929 - Vol. 19, 1947). American Chemical Society, Washington, D.C.

Analytica Chimica Acta. Elsevier Publishing Company, Amsterdam.

Applied Science and Technology Index (1958 to present); formerly Industrial Arts Index (until 1957). H. W. Wilson Company, New York, published monthly - Cumulative Indexes - monthly, quarterly, and annually.

Applied Spectroscopy (Vol. 1, 1946); formerly Society for Applied Spectroscopy Bulletin. Society for Applied Spectroscopy, New York.

Bulletin of Hygiene (Vol. 1, 1926). Bureau of Hygiene and Tropical Diseases, Keppel Street, Gower Street, London W.C.I.

Chemical Abstracts (Vol. 1, 1907). American Chemical Society, Washington, D. C.

Chemical Engineering (Vol. 1, 1902 to present); formerly Chemical and Metallurgical Engineering (July 1918 - July 1946). Published by McGraw-Hill Publishing Company, Inc., Albany, New York.

Clinical Chemistry (Vol. 1, 1955). The American Association of Clinical Chemists, Inc., 31 0 Elm Avenue, Baltimore 4, Maryland.

Current List of Medical Literature (Vol. 1, 1941). National Library of Medicine; formerly Armed Forces Medical Library. U. S. Department of Health, Education, and Welfare, Public Health Service, Washington 25, D. C.

Heating, Piping and Air Conditioning (Vol. 1, 1929). Keeney Publishing Company, 6 North Michigan Avenue, Chicago, Illinois.

Illuminating Engineering (Vol. 1,1906). Journal of Illuminating Engineering Society, 346 East 47th Street, New York 17, New York.

Industrial and Engineering Chemistry (Vol. 41, 1949 to present); formerly Industrial and Engineering Chemistry, Industrial Edition (Vol. 1, 1909 - Vol. 40, 1948). American Chemical Society, Washington 6, D. C.

Industrial Hygiene Digest (Vol. 1, 1937). Industrial Hygiene Foundation, Mellon Institute, 4400 Fifth Avenue, Pittsburgh 13, Pennsylvania.

Industrial Hygiene Review (Vol. 1, Dec. 1958). Issued May and December; formerly Monthly Review (Vol. 1, 1922 - Vol. 36, 1957). Division of Industrial Hygiene, New York State Department of Labor, New York.

Journal of the Air Pollution Control Association (Vol. 1, 1952); formerly Air Repair. Air Pollution Control Association, Mellon Institute, 4400 Fifth Avenue, Pittsburgh 13, Pennsylvania.

Journal of the Association of Official Agricultural Chemists (Vol. 1, 1915). The Association of Official Agricultural Chemists, Inc., 414 Water Street, Baltimore 7, Maryland.

Journal of Investigative Dermatology (Vol. 1, 1938 - Vol. 5, 1942 - Vol. 6, 1945 to present). The Society for Investigative Dermatology, Inc., Williams and Wilkins Company, Baltimore, Maryland.

Journal of Laboratory and Clinical Medicine (Vol. 1, 1915). Central Society for Clinical Research. Published by the C. V. Mosby Company, 3207 Washington Boulevard, St. Louis, Missouri.

Noise Control (Vol. 1, No. 1, Jan. 1955). Acoustical Society of America, Lancaster, Pennsylvania. (Noise Control, Subscription Department, 57 East 55th Street, New York 22, New York).

Nuclear Science Abstracts (Vol. 1, 1948). U. S. Atomic Energy Commission, Oak Ridge, Tennessee. (Superintendent of Documents, Washington, D. C.).

Occupational Health Review (Vol. 1, 1949); formerly Industrial Health Review. Occupational Health Division, Department of National Health and Welfare, Canada, Ottawa, Ontario.

Occupational Safety and Health (Vol. 1, 1950). International Labour Office, Geneva.

Public Health Reports (Vol. 1, 1878). Public Health Service, Washington, D.C.

Review of Scientific Instruments (Vol. 1, 1930). American Institute of Physics, published Prince and Lemon Streets, Lancaster, Pennsylvania.

Science (Vol. 1, 1894). American Association for the Advancement of Science, Lancaster, Pennsylvania, published 1515 Massachusetts Avenue, Washington 5, D.C.

Textbooks and Other References

Air Pollution Handbook. Edited by Magill, P.L., F.R. Holden, and C. Ackley. McGraw-Hill Book Company, New York, 1956.

Air Sampling Instruments Manual. American Conference of Governmental Industrial Hygienists, 1014 Broadway, Cincinnati, Ohio, 2nd Ed., 1962.

American Standard Safety Code for Ventilation and Operation of Open Surface Tanks. Z9.1 - 1951, American Standards Association, 10 East 40th Street, New York 16, New York.

American Standard Safety Code for Design, Construction and Ventilation of Spray Finishing Operations. Z9.3 - 1963, American Standards Association, 10 East 40th Street, New York 16, New York.

American Standard Safety Code on Fundamentals Governing the Design and Operation of Local Exhaust Systems. Z9.2 - 1960, American Standards Association, 10 East 40th Street, New York 16, New York.

Chemical Engineers' Handbook. Perry, J. H., Editor-in-Chief. McGraw-Hill Book Company, 3rd Edition, New York, 1950.

Encyclopedia of Chemical Technology (15 Volumes, 1947 - 1956, Supplement No. 1, 1957, Supplement No. 2, 1960). Edited by Kirk, R. E. and D. F. Othmer. Interscience Publishers, Inc., New York, New York.

Encyclopedia of Instrumentation for Industrial Hygiene. Edited by Yaffe, C. D., D. H. Byers, and A. D. Hosey, University of Michigan, Institute of Industrial Health, Ann Arbor, 1956.

Engineering Manual for Control of In-Plant Environment in Foundries. American Foundrymen's Society, Inc., Des Plaines, Illinois, 1956.

Foundry Air Pollution Control Manual. American Foundrymen's Society, Inc., Des Plaines, Illinois, 1956.

Handbook of Acoustic Noise Control, Volumes I and II. Technical Report 52-204, Wright Air Development Center, Air Research and Development Command, U.S.A.F., Wright-Patterson Air Force Base, Ohio, 1953. (Report available as PB 111, 274) Office of Technical Services, U. S. Department of Commerce, Washington 25, D.C.

Handbook on Air Cleaning Particulate Removal. Friedlander, S. K., L. Silverman, P. Drinker, and M. W. First, U. S. Atomic Energy Commission, Washington, D.C., September 1952.

Handbook of Air Conditioning. Edited by Strock, C., and W. B. Foxhall, Industrial Press, 93 Worth Street, New York, 1959.

Handbook of Chemical Microscopy. Chamot, E. M. and C. W. Mason, Vols. I, 3rd Edition 1958, and II, 2nd Edition 1940. John Wiley and Sons, Inc., New York.

Handbook of Chemistry. Compiled and edited by Lange, N. A., McGraw-Hill Book Company, Inc., 10th Edition, New York, 1961.

Handbook of Chemistry and Physics. Hodgman, M. S., Editor-in-Chief. Chemical Rubber Publishing Company, 44th Edition, 1963.

Handbook of Dangerous Materials. Sax, N. I. Reinhold Publishing Corp., New York, 2nd Edition, 1963.

Handbook of Noise Control. Edited by Harris, C. M., McGraw-Hill Book Company, Inc., New York, 1962.

Handbook of Noise Measurement. Peterson, A. P. G. and E. E. Gross, Jr., General Radio Company, West Concord, Massachusetts, 5th Edition, 1963.

Handbook of Solvents. Scheflan, L. and M. B. Jacobs, D. Van Nostrand Company, New York, 1953

Heating, Ventilating, and Air Conditioning Guide. American Society of Heating and Air Conditioning Engineers, Inc., New York, published annually.

Industrial Ventilation - A Manual of Recommended Practice. American Conference of Governmental Industrial Hygienists, Committee on Industrial Ventilation, P. O. Box 453, 8th Edition, Lansing, Michigan, 1964.

Lighting Handbook. The Standard Lighting Guide. Illuminating Engineering Society, 3rd Edition, 1860 Broadway, New York, New York, 1959.

Respiratory Protective Devices Manual. Prepared by Joint AIHA-ACGIH Committee, Braun-Brumfield, Inc., Ann Arbor, Michigan, 1963.

Standards, Definitions, Terms, and Test Codes for Centrifugal, Axial, and Propeller Fans, N.A.F.M. Air Moving and Conditioning Association, Inc., 2nd Edition, Bulletin 110, 2159 Guardian Building, Detroit 26, Michigan, 1952.

Trade Names Index. American Conference of Governmental Industrial Hygienists (A. D. Hosey, Secretary-Treasurer, 1014 Broadway, Cincinnati 2, Ohio, 1955, and Supplement 1959, revision in progress. Available only to official governmental agencies and members of ACGIH).

Chemical

Analytical Absorption Spectroscopy. Edited by Mellon, M. G., John Wiley and Sons, Inc., New York, 1950.

Analytical Chemistry. Treadwell, F. P. and W. T. Hall, Vol. I, Qualitative Analysis; Vol. II, Quantitative Analysis, John Wiley and Sons, Inc., 9th Edition, New York, 1937.

Analytical Chemistry of Industrial Poisons, Hazards and Solvents. Jacobs, M. B., Interscience Publishers, Inc., (John Wiley and Sons) 2nd Edition, New York, 1949.

Applied Inorganic Analysis, Hillebrand, W. F., G. E. F. Lundell, H. A. Bright, and J. I. Hoffman, John Wiley and Sons, Inc., 2nd Edition, New York, 1953.

Chemical Analysis of Industrial Solvents. Jacobs, M. B., and L. Scheflan, Interscience Publishers, Inc., (John Wiley and Sons) New York, 1953.

Chemical Methods in Industrial Hygiene. Goldman, F. H., and M. B. Jacobs, Interscience Publishers, Inc., (John Wiley and Sons) New York, 1953.

Chemistry of Industrial Toxicology. Elkins, H. B., John Wiley and Sons, Inc., 2nd Edition, New York, 1959.

Colorimetric Determination of Traces of Metals. Sandell, E. B., Interscience Publishers, Inc., (John Wiley and Sons) 3rd Edition, revised and enlarged, New York, 1959.

Colorimetric Methods of Analysis. Snell, F. D., and C. T. Snell, D. Van Nostrand Company, Inc., 3rd Edition, New York, Vol. I, 1948; Vol. II, 1949; Vol. IIA, 1959; Vol. III, 1953; Vol. IIIA, 1961; Vol. IV, 1954.

Identification of Pure Organic Compounds. Huntress, E. H., and S. P. Mulliken, John Wiley and Sons, Inc., Vols. I - IV, New York, 1941.

Infrared Spectra of Complex Molecules. Bellamy, L. J., John Wiley and Sons, Inc., New York, 1958.

Instrumental Analysis. Harley, J. H., and S. E. Wiberley, John Wiley and Sons, Inc., New York, 1958.

Instrumental Methods of Analysis. Williard, H. H., and L. L. Merritt, 2nd Edition, D. Van Nostrand Company, Inc., New York, 1951.

New Instrumental Methods in Electrochemistry. Delahay, P., Interscience Publishers, Inc., (John Wiley and Sons) New York, 1954.

Optical Properties of Organic Compounds. Winchell, A.M., Academic Press, New York, New York, 2nd Edition, 1954.

Organic Analysis. Edited by Mitchell, Jr., J., I.M. Kolthoff, E.S. Proskauer, and A. Weissberger, Interscience Press, Inc., New York, Vol. 1, 1953; Vol. II, 1954; Vol. III, 1956; Vol. IV, 1960.

Organic Analytical Reagents. Welcher, F. J., D. Van Nostrand Company, Inc., Vols. I - IV, New York, 1948.

Organic Solvents: Physical Constants and Methods of Purification. Weissberger, A., and E. Proskauer, J. A. Riddick and E. E. Toops. John Wiley and Sons, Inc., 2nd Edition, 1955.

Outlines of Methods of Chemical Analysis. Lundell, G. E. F., and J. I. Hoffman, John Wiley and Sons, Inc., New York, 1938.

Polarography. Kolthoff, I. M., and J. J. Lingane, Interscience Publishers, Inc., (John Wiley and Sons) 2nd Edition, New York, 1952.

Polarographic Techniques. Meites, L., Interscience Publishers, Inc., (John Wiley and Sons) New York, 1955.

Qualitative Analysis by Spot Tests. Feigl, F. (translated by Oesper, R.E.), Elsevier Publishing Company, Inc., 3rd Edition, New York, 1946.

Scott's Standard Methods of Chemical Analysis. Furman, N. H., Editor, D. Van Nostrand Company, Inc., 6th Edition, Vols. I and II, New York, 1963.

Source Book of Industrial Solvents. Mellan, I., Reinhold Publishing Corp., New York, 3 Vols., 1957.

Spot Tests in Inorganic Analysis. Feigl, F. (translated by Oesper, R. E.), Elsevier Publishing Company, New York, 6th Edition, 1960.

Spot Tests in Organic Analysis. Feigl, F. (translated by Oesper, R. E.), Elsevier Publishing Company, Inc., New York, 1956.

Standard Methods for the Examination of Water and Waste Water. American Public Health Association, 11th Edition, New York, 1960.

Statistical Methods for Chemists. Youden, W. J., John Wiley and Sons, New York, 1951.

Systematic Identification of Organic Compounds: A Laboratory Manual. Shriner, R. L., R. C. Fuson, and D. Y. Curtin, John Wiley and Sons, Inc., 4th Edition, New York, 1956.

Vapor Pressure of Organic Compounds. Jordan, T. E., Interscience Publishers, Inc., (John Wiley and Sons) New York, 1954.

Engineering

Absorption and Extraction. Sherwood, T. K., and R. L. Pigford, McGraw-Hill Book Company, Inc., 2nd Edition, New York, 1952.

Acoustic Measurements. Beranek, L. L., John Wiley and Sons, New York, 1949.

Acoustics. Beranek, L. L., McGraw-Hill Book Company, Inc., New York, 1954.

Design of Industrial Exhaust Systems. Alden, J. L., The Industrial Press, 3rd Edition, New York, 1963.

Design of Injectors for Low Pressure Air Flow. McElroy, G. D., Technical Paper 678, U. S. Bureau of Mines, 1945.

Exhaust Hoods. Dalla Valle, J. M., The Industrial Press, New York, 1946.

Fan Engineering. Madison, R. P., Editor, Buffalo Forge Company, 5th Edition, Buffalo, New York, 1948.

Industrial Deafness. Sataloff, J., McGraw-Hill Book Company, Inc., New York, 1957.

Industrial Dust. Drinker, P., and T. F. Hatch, McGraw-Hill Book Company, Inc., 2nd Edition, New York, 1954.

Industrial Environment and Its Control. Dalla Valle, J. M., Pitman Publishing Corp., New York, 1948.

Industrial Health Engineering. Brandt, A. D., John Wiley and Sons, Inc., New York, 1947.

Inhaled Particles and Vapors. Davies, C. N., Pergamon Press, New York, 1961.

Measurement of Air Flow. Ower, E., Chapman and Hall, 3rd Edition, London, 1949.

Noise Reduction. Beranek, L. L., McGraw-Hill Book Company, Inc., New York, 1960.

Noise Symposium. University of Michigan Press, Ann Arbor, Michigan, 1952.

Particle Size: Measurement, Interpretation, and Application. Irani, R. R., and C. F. Callis, John Wiley and Sons, New York, 1963.

Plant and Process Ventilation. Hameon, W. C. L., The Industrial Press, New York, 2nd Edition, 1963.

Principles of Chemical Engineering. Walker, W. H., W. K. Lewis, W. H. McAdams, and E. R. Gilliland, McGraw-Hill Book Company, Inc., 3rd Edition, New York, 1937.

Review of Literature on Dusts. U. S. Department of Interior, Bureau of Mines Bulletin No. 478, 1950.

The Science of Seeing. Luckiesh, M., D. Van Nostrand Company, Inc., New York, 1937.

Thermal Standards in Industry. American Public Health Association Yearbook, 1949-1950.

Medical

Legal Medicine: Pathology and Toxicology. Gonzales, T. A., M. Vance, M. Halpern, and C. J. Umberger, Appleton-Century-Crofts, 2nd Edition, New York, 1954.

Modern Occupational Medicine. Fleming, A. J., C. A. D'Alonzo, and J. A. Zapp, Lea, Philadelphia, Pennsylvania, 2nd Edition, 1960.

Occupational and Related Dermatoses. Birmingham, D. J., and P. C. Campbell, Jr., Public Health Service Publication No. 364, Public Health Bibliography Series No. 12, U. S. Department of Health, Education, and Welfare, 1954.

Occupational Diseases of the Skin. Schwartz, L., L. Tulipan, and D. J. Birmingham, Lea and Febiger, 3rd Edition, Philadelphia, Pennsylvania, 1957.

Occupational Diseases and Industrial Medicine. Johnstone, R. T., and S. E. Miller, W. B. Saunders Company, Philadekogia, Pennsylvania, 1960.

Pathologic Physiology: Mechanism of Disease, Sodeman, W. A., W. B. Saunders Company, Philadelphia, Pennsylvania, 1954.

Pharmacology in Medicine. Edited by Drill, V. A., McGraw-Hill Book Company, Inc., 2nd Edition, New York, 1958.

The Diseases of Occupations. Hunter, D., Little, Boston, Massachusetts, 3rd Edition, 1963.

Toxicology

Clinical Toxicology of Commercial Products. Gleason, M. N., R. E. Gosselin, and H. C. Hodge, Williams and Wilkins Company, Baltimore, 1957, Supplement, 1960.

Halogenated Hydrocarbons: Toxicity and Potential Dangers. Von Oettingen, W.F., Public Health Service Publication No. 414, Washington, 1955.

Industrial Hygiene and Toxicology. Patty, F. A., Editor, Interscience Publishers, Inc., (John Wiley and Sons) New York, Vol. 1, 2nd Edition, 1958, Vol. II, 2nd Edition, 1963.

Industrial Toxicology. Fairhall, L. T., Williams and Wilkins Company, 2nd Edition, Baltimore, 1957.

Industrial Toxicology. Hamilton, A., and H. L. Hardy, Paul B. Hoeber, Inc., New York, 1949.

Manual of Pharmacology and Its Applications to Therapeutics and Toxicology. Sollmann, T., W. B. Saunders Company, 8th Edition, Philadelphia and London, 1957.

Noxious Gases and the Principles of Respiration Influencing Their Action. Henderson, Y., and H. W. Haggard, Reinhold Publishing Corp., 2nd Edition, New York, 1943.

Poisoning. Guide to Clinical Diagnosis and Treatment. Von Oettingen, W. F., W. B. Saunders Company, 2nd Edition, Philadelphia and London, 1958.

Toxicity of Industrial Metals. Browning, E., Butterworths, London, 1961.

Toxicity of Industrial Organic Solvents. Browning, E., Chemical Publishing Company, New York, 1953.

Toxicology Mechanisms and Analytical Methods, Vols. I and II, Academic Press, New York, 1960.

REVIEW OF MATHEMATICS

I. Exponents

A. General Laws of Exponents

A number raised to a power is an exponential number. In the exponential x^n, x is the base, n is the power. The exponent indicates the number of times the base is used as a factor. For example,

$$x^4 = x \cdot x \cdot x \cdot x; \quad x^2 = x \cdot x;$$
$$x^1 = x; \quad x^{1/2} = \sqrt{x}$$

1. Addition and Subtraction:

Direct combination by addition and subtraction is limited to the coefficients of terms having the same base and power.

$$ax^n + bx^n = (a + b)x^n$$
$$ax^n - bx^n = (a - b)x^n$$

2. Multiplication:

The product of two or more exponential numbers of the same base is that base raised to the power which is the sum of the powers of the individual factors.

$$(ax^n)(bx^m)(cx^p) = abcx^{n+m+p}$$
$$x^2 \cdot x^3 = (x \cdot x) + (x \cdot x \cdot x) = x^5$$
$$10^2 \cdot 10^3 = 10^{2+3} = 10^5$$

3. Division:

The quotient of two exponential numbers of the same base is that base raised to the power which is the power of the dividend minus the power of the divisor.

$$\frac{ax^n}{bx^m} = \frac{a}{b} x^{n-m}; \quad \frac{x^5}{x^3} = x^{5-3} = x^2$$

Note: A negative exponent denotes division. Since:

$$x^{-m} = \frac{1}{x^m}$$

4. Powers:

An exponential number raised to a power is the base raised to the power which is the product of the first and second exponents.

$$(ax^n)^m = a^m x^{nm}$$

5. Zero Power:

Any base except zero raised to the zero power equals 1.

$$x^0 = 1(x \pm 0); \quad ax^0 = a(x \pm 0);$$
$$5^0 = 1; \quad 10^0 = 1.$$

B. Powers of Ten

Extremely large or small numbers are conveniently expressed as exponential numbers using the base 10. The general laws of exponents apply.

$1{,}000{,}000 = 10^6$; $0.000{,}001 = 10^{-6}$
Since $10^3 = 1{,}000$ Then $6{,}000 = 6 \times 10^3$
Since $10^2 = 100$ Then $600 = 6 \times 10^2$
Since $10^1 = 10$ Then $60 = 6 \times 10^1$
Since $10^{-1} = 0.1$ Then $0.6 = 6 \times 10^{-1}$

II. Significant Figures

Measurements often result in what are called approximate numbers in contrast to discrete counts.
For example: The dimensions of a table are reported as 29.6″ by 50.2″. This implies that the measurement is to the nearest tenth of an inch and that the table is less than 50.25″ and more than 50.15″ in length. One can show the same thing for the width, using the symbolic notation:

$$29.55'' < \text{width} < 29.65''$$

If, on the other hand, one knows the degree of precision of the measurement (say ± 0.03″ or ± 0.08″) we may write

$$50.2 \pm .03 \text{ or } 50.2 \pm .08$$

to indicate the degree of accuracy of the measurement of the length.

Prepared by: Howard L. Kusnetz and David Quong

In reporting results, the number of significant digits that can be recorded is determined by the precision of the instruments used.

Rules:

1. In any approximate number, the significant digits include the digit that determines the degree of precision of the number and all digits to the left of it, except for zeros used to place the decimal.
2. All digits from 1 to 9 are significant.
3. All zeros that are between significant digits are significant.
4. Final zeros of decimal numbers are significant. For example:

Number	No. of Significant Digits
0.0702	3
0.07020	4
70.20	4
7,002	4

One case where it is difficult to determine the number of significant digits: Example: 7000 - in general, it is considered to have only one significant digit. It is better to use scientific notation.

Standard of scientific notation: Write the number as a number between 1 and 10, in which only the significant digits are shown, multiplied by an exponential number to the base 10. For example:

Number	No. of Significant Digits
$5,320,000 = 5.32 \times 10^6$	3
$= 5.320 \times 10^6$	4
$= 5.3200 \times 10^6$	5
$0.00000532 = 5.32 \times 10^{-6}$	3

A. Addition and Subtraction

The result must not have more decimal places than the number with the fewest decimal places. For example:

21.262	should be	21.3
23.74	should be	23.7
139.6	should be	139.6
184.602	should be	184.6

This can also be written as:

$$\begin{array}{r} 21.262 \pm 0.0005 \\ 23.75 \pm 0.005 \\ 139.6 \pm 0.05 \\ \hline 184.612 \pm 0.0555 \end{array}$$

B. Multiplication and Division

The result must not have more significant places than are possessed by the number with the fewest significant digits. For example:

$$\begin{aligned} (50.20)(29.6) &= 1485.92 \\ &= 1490 \\ &= 1.49 \times 10^3 \end{aligned}$$

III. Logarithms

Logarithms are exponents. The logarithm of any number is the power to which a selected base must be raised to produce the number. The laws of exponents apply to logarithms.

A. Common Logarithms (Briggsian)

Common logarithms use the base 10 and are identified by the notation "log". The common logarithm of a number consists of a characteristic, which locates the decimal point in the number, and a mantissa, which defines the numerical arrangement of the number.

Number (Anti-log)	Exponential Form	Common Logarithmic Form: Characteristic	Mantissa	Complete Log
0.0005	5×10^{-4}	-4	0.7	$\bar{4}.7$
0.05	5×10^{-2}	-2	0.7	$\bar{2}.7$
5.0	5×10^{0}	0	0.7	0.7
500.0	5×10^{2}	2	0.7	2.7
50000.0	5×10^{4}	4	0.7	4.7

A bar over a characteristic indicates a negative characteristic and a positive mantissa. The log may be written $\bar{4}.7$ or 6.7-10 or -3.3. The form -3.3 does not contain a characteristic and mantissa.

B. Natural Logarithms (Naperian)

Natural logarithms use the base e (approximate numerical value 2.718) and are identified by the notation ln or $\log_e$. The characteristics of natural logs are not necessarily integers; therefore, the number to the left of the decimal point in a ln does not indicate the location of the decimal point in the anti-logarithm.

Number (Anti-ln)	Exponential Form	Natural Logarithmic Form
0.0005	5×10^{-4}	$\ln 5 + \ln 10^{-4} = 1.6 - 9.2 = -7.6$
5.0	5×10^{0}	$\ln 5 + \ln 1 = 1.6 - 0 = 1.6$
50,000.0	5×10^{4}	$\ln 5 + \ln 10^{4} = 1.6 + 9.2 = 10.8$

C. Logarithmic Conversions

Conversions between the common and natural systems may be accomplished.

$$\log N = 0.4343 \ln N$$
$$\ln N = 2.3026 \log N$$

IV. Exponential Equations

An exponential equation contains a variable in the exponent. A number of ventilation and dilution equations are of this type.

A. Development

An exponential equation of the type which defines simple dilution ventilation in a space in which contaminant is not being generated is developed as follows: The rate of change of y with respect to x is directly related to the magnitude of the variable y.

$$\frac{dy}{dx} = -ay$$

$$\int_{y_o}^{y} \frac{dy}{y} = \int_{0}^{x} -a\,dx \quad (\text{when } x = 0,\ y = y_o)$$

$$\ln y \Big]_{y_o}^{y} = -ax \Big]_{0}^{x}$$

$$\ln y - \ln y_o = -ax$$

$$\ln \frac{y}{y_o} = -ax$$

$$\frac{y}{y_o} = e^{-ax}$$ An exponential equation

$$y = y_o e^{-ax}$$

B. Solution

Exponential equations may be solved directly with a log-log slide rule or through the use of logarithms and the laws of exponents.

V. Graphical Presentation

An equation is a statement of equality; a graph is its pictorial representation.

A. Arithmetic (Cartesian) Coordinates

Arithmetic graphs have linear scales. An equation of two variables may be depicted in a graph having a vertical (y) axis and a horizontal (x) axis. The vertical (y) value for any point on a graphical curve is called the ordinate, the corresponding horizontal (x) value, the abscissa.

A linear, or first degree, equation may be expressed as: $y = mx + b$

y = dependent variable (ordinate)
x = independent variable (abscissa)
m = slope of the plotted equation
b = y intercept; the value of y when x = 0

B. Logarithmic Coordinates

An exponential equation may be plotted on arithmetic coordinates as a curved line. Transposed to logarithmic form, however, it will plot as a straight line:

$$y = y_o e^{-ax}$$

Transposed:

$$\log y = -ax \log e + \log y_o$$

or $$\ln y = -ax + \ln y_o$$

which have the form $y = mx + b$

Thus, logarithmic coordinates on the y axis and linear coordinates on the x axis (semi-log paper) will permit the original equation, $y = y_o e^{-ax}$ to be plotted directly as a straight line. An equation of the form $yx^n = k$, when transposed to logarithmic form, shows both variables y and x as logarithms. It may be plotted directly as a straight line when both the y and the x axes have logarithmic scales.

VI. Dimensional Analysis

An analysis of dimensions can be useful in:
(1) converting units
(2) checking equations for missing terms
(3) deriving equations and solving for unknown quantities.

A. Basic Dimensions

All physical units can be reduced to three basic dimensions: mass; length; and time. Reducing all units to these basic dimensions simplifies the solution of problems of dimensional analysis.

B. Terms Peculiar to Industrial Hygiene

1. The concentration of gases and vapors is usually expressed as parts per million parts of air = ppm.

$$= \frac{\text{parts}}{\text{million parts}} \text{ (of air)}$$

$$= \frac{\text{liter}}{10^6 \text{ liters}} = \frac{\text{centimeters}^3}{10^6 \text{ centimeters}^3}$$

$$= \frac{\text{feet}^3}{10^6 \text{ feet}^3} = \frac{\text{meters}^3}{10^6 \text{ meters}^3}$$

This is similar to

$$\% = \frac{\text{parts}}{100 \text{ parts}}$$

2. The concentration of fumes, mists, and some dusts, and of gases and vapors on occasion, is expressed as

milligrams per cubic meter(mg/M^3)

$$= \frac{\text{mg of material}}{M^3 \text{ of air}}$$

3. The concentration of mineral dusts is usually expressed as millions of particles per cubic foot of air

$$= \frac{\text{millions of particles}}{\text{cu. ft. of air}}$$

C. Examples:

1. Reduce pressure (force/area) to the basic units of mass, length, and time (M, L, and T)

$$P = \frac{F}{A} = \frac{Ma}{L^2} = \frac{M}{L^2}\frac{V}{T} = \frac{M}{L^2}\frac{L}{T^2} = \frac{M}{LT^2}$$

2. Convert 212 particles/cc. to mppcf concentration,

$$C = \left[\frac{212 \text{ particles}}{\text{cc}}\right]\left[\frac{1000\text{cc}}{\text{liter}}\right]\left[\frac{28.3 \text{ liters}}{\text{cu. feet}}\right]$$

$$= \frac{6{,}000{,}000 \text{ particles}}{\text{cu. feet}} \text{ (ppcf)}$$

$$= \frac{6.0 \text{ million particles}}{\text{cu. feet}} = 6.0 \text{ mppcf}$$

3. Given: the concentration of Hg in ppm, convert to mg/M^3

$$\text{ppm} = \frac{\text{parts}}{\text{million}} = \frac{1 \text{ liter}}{10^6 \text{ liters air}}$$

$$\text{conc.} = \frac{1 \text{ liter}}{10^6 \text{ liters air}} \quad x$$

$$\left[\frac{\text{gram-mole}}{22.4 \text{ liters}}\right]\left[\frac{\text{grams}}{\text{gram-mole}}\right]\left[\frac{10^3 \text{ liters}}{M^3}\right] =$$

$$\frac{\text{grams}}{10^3 M^3} = \frac{\text{mg}}{M^3}$$

4. Derive an equation for the preparation of a known concentration of a liquid given the following:

V_T = Chamber volume in liters

MW = Molecular weight of a substance

T = Absolute temperature

P = Pressure in mm Hg

ρ = Density (grams per milliliter, ml)

v = Volume of material to be used (in ml)

C = Concentration (ppm)

Note: At standard temperature and pressure, one gram-mole of any compound in the gaseous state occupies 22.4 liters. One gram-mole is the amount of material in grams equal to the molecular weight of the material.

$$C(\text{ppm}) = \frac{(v \text{ ml})(\rho \frac{\text{gm}}{\text{ml}}) \left[\frac{22.4 \text{ liters/gram-mole}}{\text{MW grams/ gram-mole}}\right]\left[\frac{T}{273}\right]\left[\frac{760}{P}\right]}{V_T \text{ (Liters)}} \times 10^6 \frac{\text{parts}}{\text{million parts}}$$

$$= \frac{(v)(\rho)\left[\frac{22.4}{MW}\right]\left[\frac{T}{273}\right]\left[\frac{760}{P}\right]}{V_T} \times 10^6$$

REVIEW OF ANALYTICAL CHEMISTRY

Introduction and Background

In every branch of analytical chemistry, the methods to be used for a given element or compound are at least partially dependent upon the nature of the sample and its mode of collection. This is perhaps even more true in industrial hygiene since the difficulties in obtaining an ideal sample from the chemist's point of view may be enormous. To begin with the toxic materials of interest are quite dilute in the air being sampled. A concentration of 0.1% by volume in air would be very high. Added to this difficulty is the lack of efficient collecting systems for sampling large volumes of air for gases and vapors in a short period of time. Most of the absorption devices of convenient size operate at a flow rate of only a few liters per minute and if the operation being sampled is of short duration and the material being sampled is at a very low concentration, the engineer will not be able to collect very much material for an analysis.

A second complication is that the industrial hygienist is not always sure what type of an analysis he wants from the chemist. It may be that a particular industrial operation is using one chemical while the toxic hazard is being caused by some other chemical from another location. Or, a number of poorly identified mixtures of compounds may be in use and only an educated guess can be made as to the hazard involved. In cases such as these it is important for the chemist to understand industrial processes so that he can answer the industrial hygienist's basic question "What toxic materials may be present and in what concentration?"

Several other factors relating to the purpose of an industrial hygiene survey also bear on the method used by the industrial hygiene chemist. Frequently, samples are taken by plant industrial hygiene personnel as part of a continuing monitoring program or for the purpose of evaluating an engineering control method. Here it is desired to detect a relative trend or change from previous conditions. On the other hand, some samples are taken by regulatory agencies for the purpose of determining compliance with a code or standard. The analysis of these samples must be accomplished by recognized methods of demonstrated accuracy and freedom from artifacts. Thus, it is important that the industrial hygiene chemist be familiar with the sampling problems of the industrial hygienist, and with the use to which he intends to put the results of the analyses.

The function of the chemistry laboratory in occupational health is to exercise the needed analytical control over the chemical determinations performed on the samples of environmental and clinical studies. It can be shown that the application of critically tested laboratory methods of analysis to field samples will provide the sensitivity, specificity, and the numerous other cited advantages essential for an accurate evaluation of the environment. As stated previously, one of the ultimate objectives of the environmental study is the recommendation of effective control measures. Once established, the effectiveness of the equipment installed or of the modifications introduced into the processing operations to minimize exposure of the worker to hazardous substances is evaluated periodically. The plant physician usually conducts regularly scheduled physical examinations of the worker. Part of the examination may include the procurement of a blood or urine specimen for chemical analysis to obtain a quantitative estimation of the workman's current level of absorption of a toxic substance. The engineer continues

Prepared by: Richard E. Kupel and
Jeremiah R. Lynch

to conduct evaluations of the performance of control equipment by making air flow measurements, collecting samples of the atmosphere for chemical analysis, and by observing operations, old and new, in the working area involved. The repetitive services by the separate disciplines constitute the practice of a sound occupational health program. If this teamwork approach is to function constructively in the preservation of the health of the workers, then it is fundamental that each discipline must provide the most reliable and efficient instruments and procedures for its segment of these professional activities.

Analytical methods used by the chemistry laboratory to analyze industrial hygiene samples can be divided into two main classes - Chemical and Physical Methods.

Chemical Methods

Colorimetric. Those reactions where a color is developed and is indicative of the presence of the element and its concentration are classified as colorimetric methods. The Dithizone extraction method is one of the most widely used today. Specific elements can be extracted depending on the pH of the solution. Examples include lead in blood and urine, and copper and zinc in tissue samples(1).

Ion Exchange. This technique concentrates or separates elements one from another depending on the strength and type of eluting agent. A column of the appropriate type and length is loaded with the mixture of elements and the element of interest is eluted using a solution of the correct type and strength. The eluting solution can be evaporated to further concentrate the element if necessary. Mercury in urine, fluorine in urine, and fluorine in impinger samples are examples of this technique(2).

Gravimetric Methods. These methods depend upon the formation of a precipitate or a residue which can be weighed. One example is the analysis of dust samples for free silica.

Volumetric Methods. Quantitative analyses are performed by the use of definite volumes of standard solutions of reagents such as the determination of acid gases in air, which are titrated with a basic reagent.

Physical Methods

Emission Spectroscopy. This is an instrumental method utilizing the formation of spectra by exciting the element, under study, by various means causing characteristic radiation to be formed which can be dispersed by a grating or a prism and photographed. Each metallic or metal-like element has its own characteristic spectra that can be identified under most any conditions. The sensitivity of this method for most elements is in the part per million range. Examples of its use include elemental analysis of body tissues, dust and basic materials for contaminants(3).

Infrared Spectroscopy. This method is used to identify organic and inorganic materials by the characteristic spectra obtained by exposing the sample to infrared radiation and measuring the amount of radiation absorbed. Each functional group of an organic molecule will absorb in a specific region of the infrared spectrum allowing the operator to identify the compounds by the bands thus formed. The identification of rubbers, plastics, organic solvents, oils, organic vapors, and beryllium oxalate in beryllium oxide are typical applications(4).

Ultraviolet Spectroscopy. This technique is used to analyze aromatic hydrocarbons. Each aromatic hydrocarbon has its own characteristic absorption pattern in the ultraviolet region of the electromagnetic spectrum that will identify it from other similar hydrocarbons. Materials that absorb in the same wavelength region will have an additive effect to the overall spectrum. This can

be used in the quantitative analysis of mixtures of aromatic hydrocarbons. Benzene and toluene vapors in air can be separated and identified with such an instrument.

Polarographic Analysis. A polarograph produces a continuous visible record of the current-voltage curve which is characteristic of a solution undergoing electrolysis between a dropping mercury electrode and a reference electrode. A polarographic analysis may be made of a substance if that substance is electroreducible or electro-oxidizable within the range of the electrode, if it is in true solution and is stable for the duration of the measurement. For example, determinations can be made for lead, cadmium, zinc, vanadium, iron, thallium, halides, sulfur dioxide, cystine and many other substances.

Mass Spectroscopy. The mass spectrometer is an instrument used to analyze gases, liquids and solids. Analysis is accomplished by ionizing the materials by passing them through an electron beam. Positive ions thus formed are projected down the analyzer tube by means of an electron gun. These ions are separated by means of an electromagnetic or electrostatic field or by the time necessary for them to travel from the ion gun to the collector. The output of the ion multiplier is fed into a recorder or an oscilloscope. Each compound has its own individual ionization pattern which is used to identify the compound and is used also to quantitate this procedure. Examples of use include carbon monoxide in air, gaseous impurities in high purity gases, identification of hydrocarbons, and identification of the effluent of a gas chromatograph.

Gas Chromatography. This method involves the separation of organic mixtures by passing them over a column of specific qualities that will enable the components to be held up for varying periods of time before they are detected and recorded. The instrument consists of a column, containing a solid and a liquid support, a carrier gas, a detector and a recorder. This technique is useful primarily in separating materials with similar chemical properties that heretofore could be separated only by fractional distillation. Examples include the analysis of chlorinated hydrocarbons, and the identification and analysis of solvents, plastics, gases and waxes(5).

X-ray Diffraction. Characteristic spectra are formed when a monochromatic X-ray beam impinges upon a sample of crystalline form. These diffracted X-rays are monitored using an appropriate detector and the spectra recorded. The spectra thus formed can be used to identify the material being analyzed. The combined form of the material is readily discernible from this spectra. This instrument can be used to determine the percentage of free silica in dust samples; for the analysis of human lung for free silica; and for identification of the combined form of a material(6).

X-ray Fluorescence. This technique involves bombarding the sample with an X-ray beam and allowing the resulting X-ray to be refracted from a crystal thus separating the radiation into its component parts. All elements above atomic number 11 can be analyzed by this technique. X-ray fluorescence techniques can be used for solution analysis for impurities; identification of metals; and analysis of tissue samples for metallic impurities(7).

Electron Microprobe. This technique involves focusing an electron beam to as small a diameter as possible - about one micron in diameter. The sample is placed under the beam and primary X-rays are formed. X-rays are refracted with a proper crystal and detected on an appropriate detector. X-rays thus formed are characteristic of the element present under the electron beam. This technique allows one to analyze small inclusions, small areas, grain boundaries, spots on lung specimens and it can be used for other biological applications. Examples include the determination of diffusion

boundaries in metals; calcium deposits across a repaired fracture in bone; and metallic impurities in tissues.

Activation Analysis. This technique involves irradiating a sample with a thermal neutron source and monitoring the induced radiation with an instrument capable of detecting and analyzing the specific activity. Chemical separation may be necessary to eliminate interfering elements. For example, normal body tissue can be analyzed in order to establish base line data for elements not detectable by other means. Furthermore, this method extends the lower limit of detection of some of the more important elements[8].

References

1. Sandell, E. B. Colorimetric Determinations of Trace Metals. Interscience Publishers, Inc., New York, 1944.
2. Dowex Ion Exchange. The Dow Chemical, Company, Midland, Michigan, 1964.
3. Ahrens, L. H. and Taylor, S. R. Spectrochemical Analyses, 2nd Edition. Addison Wesley Publishing Company, Reading, Massachusetts, 1961.
4. Cross, A. D. Introduction to Practical Infrared Spectroscopy. Butterworths Scientific Publications, 1960.
5. Heftmann, Erich. Chromatography. Reinhold Publishing Corporation, New York, 1961.
6. Buerger, M.J. X-Ray Crystallography, John Wiley & Sons, Inc. New York, 1942.
7. Liebhafsky, H. A., Pfeiffer, H. G., Winslow, E. H., Zemany, P. D. X-Ray Absorption and Emission in Analytical Chemistry. John Wiley & Sons, Inc., New York, 1960.
8. Leddicotte, W. G. Radioactivation Analysis - Specific for Trace Element Determinations. Symposium on Extension of Sensitivity for Determining Various Constituents in Metals. ASTM Special Technical Publication No. 308, 1962.

GENERAL PRINCIPLES IN EVALUATING THE OCCUPATIONAL ENVIRONMENT

Introduction

Information in the literature on this subject is scant; consequently, most industrial hygienists have to acquire knowledge in this field by practical experience. It is hoped that the following discussion will materially assist those engineers and chemists who are relatively new in this field. Further, these general principles will serve as a review for those individuals who may not have been active in the field of occupational health for several years.

Purposes for Sampling

There are at least four reasons for industrial hygienists employed by industry to conduct environmental surveys and studies: (1) to determine levels of exposure among workmen to various atmospheric contaminants and physical agents; (2) to determine the effectiveness of control measures, such as local exhaust ventilation; (3) to investigate complaints; and (4) for research purposes, for example, to determine the chemical composition and physical characteristics of dusts, fumes, vapors, gases and mists.

In addition to the reasons cited above, industrial hygienists employed by some official agencies may conduct surveys for the following reasons: (1) to determine compliance with regulations; (2) to check control measures installed as a result of recommendations; (3) to investigate health problems on an industry-wide basis, such as uranium, phosphate and diatomaceous earth operations; and (4) for consultation with plant management or labor unions concerning specific health problems.

Prepared by: Andrew D. Hosey and
Howard L. Kusnetz

Recognition of Hazards

Before any attempt is made to evaluate an occupational environment the industrial hygienist must be aware that certain health hazards may exist and he must be able to recognize these hazardous conditions. Subsequent discussions in this volume and some of the topics included in this section will cover the subject of recognition. How then, does one become aware that potentially hazardous situations exist in the work environment? There are several sources of such information. Among these are: (1) complaints from workers, safety or labor inspectors, and unions; (2) companies may request assistance because certain illnesses or skin diseases have occurred; (3) technical journals are an important source of information since many articles will discuss problems encountered by other investigators especially with some of the newer chemicals or physical agents; (4) popular magazines and newspapers often contain references to persons injured by toxic substances; and (5) reports of unpublished investigations made by private or governmental agencies.

The first step in evaluating the occupational environment is to become familiar with the particular operations in the plant or other establishment to be surveyed. Investigations should be made regarding what hazardous materials may be in use, how they are used, how many workers are exposed, and what control measures are used to protect them. Process flow sheets and technical books that describe the particular operations involved should be obtained and studied. This gives not only a good description of the general operations involved but also serves as an excellent source for the terminology used in that particular industry. Another source of such infor-

mation can be found usually in reports of previous surveys made by a governmental agency or private industry.

Preliminary Survey

The next step in evaluating the occupational environment is to conduct a preliminary survey. Ordinarily much time and effort can be saved by following this procedure because an experienced person, familiar with many types of industrial operations and the use of chemical and physical agents, can by visual inspection and the use of his perceptive senses, eliminate those operations which are clearly non-hazardous.

The preliminary survey should include areas such as: (1) general sanitation, (2) raw materials, products and by-products, (3) sources of air contaminants, (4) physical agents, and (5) control measures in use. The importance of taking adequate notes during a survey or study cannot be over-emphasized.

General Sanitation. One of the best guides on this subject is the ASA Standard Minimum Requirements for Sanitation in Places of Employment.(1) It contains definitions, general requirements on housekeeping, waste disposal, rodent, insect and vermin control, and specific topics on ventilation, water supply, toilet and washing facilities and many others. All of the items should be checked to determine if the requirements are met. Most State health departments have adopted this ASA standard, while some States have adopted more rigid requirements. ASA standards are revised periodically.

Raw Materials, Products and By-Products. The variety of substances capable of producing occupational diseases increases steadily. New products are constantly being introduced which require the use of new raw materials or new combinations of older substances. The introduction of epoxy resins just a few years ago and their increasing use is only one of many examples that could be cited. Therefore, it is of utmost importance to obtain a list of all chemicals used, and to determine the nature of the products and by-products. To accomplish this often requires a bit of detective work on the surveyor's part. In most instances the desired information can be obtained from the purchasing agent or laboratory, but in others it means reading labels on drums of solvent or containers of raw material. Ofttimes, labels do not give complete information, thus requiring further investigation.

After the list is obtained it is then necessary to determine which of the materials are toxic and to what degree. Information of this nature can be found in the latest texts on toxicology, in scientific journals, and by correspondence with toxicologists, technical information centers, and manufacturers. Many companies publish lists of toxic materials used in their plants. This information is usually passed along to supervisors and foremen.

Before any new substance is used in a manufacturing process, or before a new product is placed on the market, its toxicologic properties should be investigated so that adequate safeguards can be inaugurated. Many companies do this voluntarily and some (manufacturers of food additives and certain cosmetics) are required to do so by law.

In addition to the above, the industrial hygienist should be aware and learn about the potential health hazards involved in new processes. Recent examples include the introduction of the plasma jet torch for high temperature deposition of materials, like ceramics, onto metal or other surfaces; a new process whereby pure nickel patterns are produced from wood, metal, or plastic models based on the thermal decomposition of nickel carbonyl - an extremely toxic substance; and the use of CCl_4 in an inert carrier of nitrogen for refining the grain of aluminum-magnesium alloys. In addition to the toxic properties of CCl_4, phosgene is formed when this mixture is introduced into the molten metal.

Source of Air Contaminants. During the preliminary survey many potentially hazardous operations can be detected by visual observations. The most dusty operations can be spotted at this time although this does not necessarily mean they are the most hazardous. It must be remembered that the dust particles which cannot be seen by the unaided eye are the most hazardous since they are of respirable size. Dust concentrations must reach extremely high levels before they are readily visible in the air. The absence of a visible dust cloud does not mean that a dust-free atmosphere exists. Those operations which generate fume, such as occurs in welding, can be spotted visually.

The presence of many vapors and gases can be detected by the sense of smell. Trained observers are able to estimate rather closely the concentration of various gases and solvent vapors present in the workroom air. For many substances the odor threshold concentration is greater than the generally permissible safe exposure levels. For example, if the odor of carbon tetrachloride vapor is present even in barely perceptible amounts it is generally too much for continuous exposure. A word of caution should be interjected here. Certain gases and vapors, such as hydrogen sulfide and carbon tetrachloride produce olifactory fatigue especially if these are present in high concentrations. Thus, concentrations of some gases and vapors may be present in concentrations considerably in excess of the TLV but may not be detected by their odor.

Physical Agents. Sources of radiant heat, abnormal temperatures and humidity, excessive noise, improper or inadequate illumination, ultraviolet radiations, microwaves, lasers, x- and gamma rays, and other ionizing and non-ionizing radiations, should be noted. Without the aid of special instruments it is possible only to note the presence of these potential hazards. However, one can obtain much valuable information during the preliminary survey by observing the manner in which these are generated, the number of people involved, and control measures in use.

Control Measures in Use. The preliminary survey would not be complete unless the types of control measures in use and their effectiveness are noted. The controls include local exhaust and general ventilation, respiratory protective devices, other personnel protective measures, shielding from radiant or ultraviolet energy, and other items which are discussed in Section C-1 on controls. General guides to effectiveness could include the presence or absence of dust on floors and ledges; holes in ductwork, fans not operating; or the manner in which personal protective measures are treated by the worker.

Evaluation of Environmental Exposures

General Considerations in Sampling

The evaluation of environmental exposures requires a teamwork approach by personnel who represent several professional disciplines. No single professional group can solve the various facets of the many types of problems presented by occupational disease entities. The longer his association in the field, the more convinced the industrial hygienist becomes of this fact.

In conducting field studies there is an overlapping area for engineers, chemists, physicians, and other specialists in this field. While the engineer ordinarily collects atmospheric samples in the field for subsequent chemical analysis; in many cases, however, the chemist must go into the field to assist with the collection of samples and to advise on the chemical aspects of the particular problem under study.

The nature of the substance or condition

involved will usually have been determined during the preliminary survey. Thus, the next problem is to determine the intensity of exposure and to do this one must collect samples of the air or use direct reading instruments. Every effort should be made to obtain samples that represent the worker's exposure or the condition to be evaluated. It should be pointed out also that an industrial hygienist can obtain any result he wants depending upon the choice of sampling locations and techniques. To decide what samples are representative the industrial hygienist must be able to answer these five basic questions:

(1) Where to sample
(2) Whom to sample
(3) How long to sample - duration
(4) How many samples are needed
(5) Over what period of time - day, night, summer, or winter.

Considering each of these questions separately:

(1) Samples are usually collected or instrument readings are obtained in these general areas:
    (a) at the worker's breathing zone;
    (b) in the general room air; and
    (c) at the operation itself.

The choice of sampling location is dictated by the type of information or evaluation desired and may include one or more of the above three locations. In general, the purposes for sampling are to determine a worker's or group of worker's exposure so that an average weighted exposure can be calculated. This will be covered in more detail later. In order to make this calculation, it is necessary to collect samples at the worker's breathing zone and also in the area adjacent to the particular operation, or general room air. On the other hand, if the purpose is to define a potential hazard, check compliance with regulations, or obtain data for control purposes, samples would normally be collected in the vicinity of the operation itself. In some instances it is necessary to collect samples of the general room air to define certain exposures.

(2) Whom to sample is closely related to the discussion above on where to sample. In general, samples are collected in the vicinity of:
    (a) workers directly exposed;
    (b) breathing zones of nearby workers; or
    (c) workers remote from the exposure but who complain. Again, this depends on the type of information desired.

(3) No matter who collects atmospheric samples for analyses, whether engineer, chemist, or technician, it is essential that the sample contain sufficient material for an accurate analysis. The volume of air to be sampled or the duration of sampling is based on the following considerations:
    (a) sensitivity of the analytical procedure;
    (b) the Threshold Limit Value (TLV) of the particular contaminant; and
    (c) the estimated air concentration.

Thus, the volume of sample needed may vary from a few liters, where the estimated concentration is high, to several cubic meters where low concentrations are expected. Knowing the sensitivity of the analytical procedure, the TLV, and the sampling rate of the particular instrument in use, the chemist or engineer can determine the minimum time necessary for an adequate sample.

There is at least one other consideration in determining the duration of sampling. It should represent some identifiable period of time - usually a complete cycle of an operation in determining an operator's exposure. Another technique is to sample on a regular schedule, for example, so many minutes out of each hour. This latter procedure usually requires more samples than cyclic-type sampling and is more or less substituting a statistical approach for observation and judgment.

(4) The number of samples collected depends to a great extent upon the

purpose of sampling. For example, two samples may determine the efficacy of control methods - one while the control method is in operation and the other while it is off. On the other hand, several dozen samples may be necessary to accurately define an average daily exposure for a worker who performs a number of tasks. For any given task the number of samples depends to some degree upon the concentration. If the concentration is high, a single sample may suffice; if it is near the TLV, a minimum of 3 to 5 samples may be necessary to indicate exposures for a specific task or cycle of operation.

In a field study of silicosis among metal miners the practice was to collect a 10 minute sample for dust counts out of each 30 minutes of work time for each type of job. Furthermore, if there were 50 different working places for the same type of operation, 20 percent, or 10 of these, would be sampled. Dust count data were tabulated from about 300 impinger samples collected in a single mine representing some 15 jobs. Concentrations from the first 150 samples were essentially the same as the average of all 300 samples. This type of approach was used at the beginning of the study for research purposes. It soon became evident, however, that this technique resulted in the collection of too many samples and it caused unnecessary work on the part of persons counting these samples.

The above situation brings up a point that should be emphasized. There are no set rules regarding the duration of sampling or the number of samples to be collected. These decisions can be reached only after much experience in this field.

(5) The last question - over what period of time should samples be collected, again depends on the type of information desired and the particular operations under study. If, for example, an operation continues for more than one shift, samples should be collected during each shift. Air-borne concentrations of toxic materials or exposure to physical agents may be different for each shift. Furthermore, and this applies especially in plants located in areas where large temperature variations occur during different seasons of the year, samples should be collected during summer and winter months. Under these conditions there is generally more natural ventilation in summer than in winter, which tends to dilute air-borne contaminants.

Types of Field Sampling Instruments

Sampling instruments used in the field of industrial hygiene are generally classified according to type as follows:
(1) direct reading;
(2) those which remove the contaminant from a measured quantity of air; and
(3) those which collect a known volume of air.

Most of the sampling equipment used by industrial hygienists is found under type 2 but in recent years more direct reading instruments have been made commercially available. Detailed discussions on direct reading instruments appear elsewhere in this volume. Apparatus in type 3 includes the use of various types of evacuated flasks for collecting known volumes of contaminated air for subsequent analysis. This technique is not used as often now as in past years.

The choice of a particular sampling instrument depends upon a number of factors. Among these are:
(1) portability and ease of use;
(2) efficiency of the instrument or method;
(3) reliability of the equipment under various conditions of field use;
(4) type of analysis or information desired;
(5) availability; and

(6) personal choice based upon past experience and the other factors.

No single, universal air sampling instrument is available today and it is doubtful if such an instrument will ever be developed. In fact, the present trend is the development of a greater number of specialized instruments, particularly the newer "squeeze-bulb" types.

Grab or Continuous Samples. A discussion on air sampling techniques would not be complete unless this topic is mentioned. It has been the subject of much concern for many years. A grab sample is collected in a relatively short period of time, usually from a fraction of a minute to 3 or 4 minutes. Ordinarily, grab samples indicate either minimum or maximum exposures. Continuous samples, collected from about 5 minutes to hours or days, give information regarding average exposures only. Thus, there is a definite need for both types of samples. Much useful information can be obtained during a preliminary survey, for example, by the use of a colorimetric "squeeze-bulb" detector where a number of samples can be obtained to indicate the range of atmospheric carbon monoxide concentrations. It must be emphasized that these are grab sampling devices and, further, they must be calibrated.

Since industrial hygienists are normally interested in determining a worker's daily average exposure to atmospheric contaminants, continuous sampling techniques are necessary. Many industrial plants use continuous sampling and recording instruments for gases and vapors such as carbon monoxide, sulfur dioxide, chlorinated hydrocarbons and methyl alcohol.

Comparison of Results with Standards

The next step in evaluating the occupational environment is to compare results of air sample analyses or data from direct reading instruments with standards. The American Conference of Governmental Industrial Hygienists' committee on threshold limit values publishes annually a list of TLV's. This list includes values for the maximum average atmospheric concentration of vapors, gases, mists, fumes and dusts to which workers may be exposed for an 8-hour working day without injury to health. These values should be used as guides only in the control of health hazards because they represent only conditions under which it is felt that workers may be repeatedly exposed, day after day, without adverse effect on their health. These values are reviewed annually for changes, revisions or additions as further information becomes available.

Standards for temperature and humidity are found in the ASHRAE Guide, Belding and Hatch's heat stress index, and chapter 3 of the Navy Manual of Preventive Medicine. Those for noise, illumination, and sanitation are found in standard references. Those for ionizing radiation may be found in various publications of the Atomic Energy Commission and National Bureau of Standards. Many States and local governmental agencies have adopted rules, regulations or codes patterned after the above standards. These should be consulted before reaching a decision regarding the recommendation of protective or control measures.

Interpretation of Results

This is the final step in evaluating the environment. A great deal of common sense and sound judgment must be used in interpreting the results of an environmental study. Before an investigator claims that an individual or group of workers is exposed to a hazard injurious to health, he must have the following facts:

(1) nature of the substance or physical condition involved,
(2) intensity of exposure,
(3) duration of exposure, and
(4) the susceptibility of the person exposed.

These considerations cannot be overemphasized!

Items (1) and (2) above will have been obtained during the preliminary and/or environmental study. In order to determine the duration of exposure for an individual a detailed job description must be obtained. This information is usually available from the plant personnel office or foremen, but it should be checked by the Investigator during the study. From this information and results of the environmental survey, a weighted daily 8-hour exposure can be calculated. This assumes that a sufficient number of air samples have been collected or readings obtained with direct reading instruments under various plant operating conditions to give a true picture of the exposure.

Calculation of Weighted Exposure. The following formula is used for this calculation: (hours x conc. task A) + (hours x conc. task B) + etc. ÷ 8 hours

(Note: In general, the hours spent by a worker at various tasks should be determined to the nearest 30 minute intervals. There are, of course, exceptions if a worker has an exposure to a high concentration for short - 10 to 20 minutes - periods of time).

Assume that a study was completed in a plant where triple superphosphate fertilizer was made. Assume further that it is necessary to calculate the average daily weighted exposure of the "cone mixer" operator. His duties were essentially as follows:

(1) spend 4 hours per day regulating the flow of phosphate rock dust and phosphoric acid, which are mixed together in the "cone mixer" (task A);
(2) spend 2 hours per day on the floor below the cone mixer to insure the proper flow of the mixture on the "curing belt" (task B); and
(3) spend 2 hours per day in a control room located some distance from the "cone mixer" (task C).

This individual was exposed to fluorides in the form of phosphate rock dust and hydrofluoric acid, which is released when phosphoric acid reacts with phosphate rock dust. In order to evaluate his exposure 3 samples were collected at different times of the day and on 3 different days. Thus, 9 samples were analyzed for fluorides and HF and were used to evaluate his exposure. The results were as follows:

Task	mg/m^3 of Dust as F	ppm of HF
A	10	1
B	2	6
C	0.5	1

Weighted exposures were calculated for both types of fluoride as follows:

$$\frac{(4x10)+(2x2)+(2x0.5)}{8} = 5.63 \text{ mg/m}^3\text{F}$$

$$\frac{(4x1)+(2x6)+(2x1)}{8} = 2.25 \text{ ppm HF}$$

The TLV (1964) for F in the form of dust is 2.5 mg/m^3 and for HF it is 3.0 ppm. Thus, this worker's exposure to fluoride in phosphate rock dust is above the TLV but his exposure to HF is well below the 3.0 ppm.

A word of caution is indicated at this point regarding interpretation of these results. TLV's as indicated earlier are to be used as guides only in the control of health hazards and should not be regarded as fine lines between safe and dangerous concentrations. Some TLV's are based on various criteria of toxic effects while others are based on marked discomfort. Therefore, before an industrial hygienist recommends the installation of costly control measures, based on the results obtained during a survey, he should first recommend that the worker be given clinical tests to determine if exposure to fluorides and HF, for example, are injurious to his health. He should also ascertain the degree of toxicity of these compounds and their reactions within the body. If it is found that exposures to toxic substances are producing early symptoms of bodily injury then, and only then, should

control measures be instituted. Naturally, this statement applies to border-line situations when the toxicity and the TLV of the particular compound or compounds is considered. When these factors are evaluated and where exposures are considerably above the TLV, even for short duration, some type of protection must be provided for the worker. As indicated earlier, this is where sound judgment on the part of the investigator is necessary in the final evaluation and control of the environment.

Summary

The conduct of environmental surveys and studies is but one phase in the over-all picture in determining occupational health hazards. They are valuable only if all environmental factors relating to workers' potential exposures are included. In evaluating workers' exposures to toxic dusts, fumes, gases, vapors and mists, a sufficient number of samples should be collected for the proper duration to enable the calculation of weighted exposures.

Choice of sampling instruments will probably be a compromise between several desirable characteristics. Among these are high efficiency, portability, reliability and availability. Personal preferences, as a result of experience, will also determine the choice of instruments.

Adequate notes taken during environmental studies are a must - one cannot rely upon one's memory after a study is completed to provide the detailed information necessary for the preparation of a report.

Finally, sound judgment should be exercised both during the actual survey and in preparing the report. Experience, conversation with others in the field, and a thorough knowledge of the particular operations studied will aid in developing these qualities so important in the field of occupational health.

Reference

1. American Standard Minimum Requirements for Sanitation in Places of Employment. ASA Z4.1 Code. American Standards Association, Inc., New York 17, New York, 1955.

SAMPLING FOR PARTICULATES

Introduction

The sampling and analysis of air for particulates in work places is not a new problem. Probably the oldest published statement regarding the harmfulness of exposure to dust is one by Pliny[1] as far back as 75 A.D. An early review of literature on the subject was published by Cunningham in 1873.[2] Since that time there have been several other reviews[3-11]; in most cases the instruments described have become obsolete because of the higher efficiency, greater facility of operation, newer collection techniques, or other advantages of modern apparatus. The intensity and frequency of analyzing air in workplaces has progressively increased in recent years and as industry has developed new processes, introduced new chemicals, and utilized new or different materials the need for new and different sampling methods has also developed.

Classification of Particulates

The term aerosol, comparable to hydrosol, refers to any system of liquid droplets or solid particles dispersed in air, of fine enough particle size, and consequent low settling velocity, to possess considerable stability as an aerial suspension. While all particulate matter does not have to be suspended to be harmful, those of pulmonary concern do exist as aerosols. Harmful materials may enter the body by ingestion with food or by absorption through the skin, but in many industries their most important mode of entry is by inhalation. As a substance may enter the respiratory tract in the form of solid particles or liquid droplets, particulate materials are quite often classified according to their nature or origin as follows:

1. *Liquids.* Liquid particles are usually classified into a single group such as mists but they may be further subdivided into two groups depending on their particle size. Then the larger particles are mists while the smaller particles are fogs. Liquid particles are normally produced by such processes as condensation, atomization or by entrainment of liquid by gases.

2. *Solids.* In the solid group are three categories known as dusts, fumes and smokes. They are all particulate matter and the distinction between them is based on their particle size and method of evolution.

   a. *Dusts.* Dusts are formed from solid organic or inorganic materials by reducing their size through some mechanical process such as crushing, drilling or grinding. Particles thus created vary in size from the visible to the submicroscopic but with their composition being the same as the material from which they were formed.

   b. *Fumes.* Fumes are formed by processes such as combustion, sublimation or condensation and the term is generally applied to the metal oxides of zinc, magnesium, iron, lead and others. Solid organic materials such as waxes may form fumes by the same methods.

   c. *Smokes.* Smokes result from the combustion of materials that are organic in origin.

Since particulate matter, whether liquid or solid, exhibits remarkably similar behavior when air-borne, the physical behavior of particles in inhaled air,

Prepared by: Glen W. Sutton

and in the respiratory system should be of much greater concern than the nature or origin of the particulate material. Hatch and Gross[12] state that it is of greater interest to describe different particulate materials in terms of their characteristic shape, density and size which in turn determine their settling velocity and consequent relative stability as dispersed clouds.

Pulmonary Deposition and Retention

The physical forces operating to bring about aerosol deposition within the respiratory system vary with the size of the aerosol particle and with the velocity and time of transit of air in the system. Experimental studies of the extent of particle deposition in the various parts of the respiratory system as a function of particle size have been performed by Brown, et al.[13], Landahl, et al.[14], Altshuler, et al.[15], Dautrebande, et al.[16], Van Wijk and Patterson[17], Wilson and LaMer[18], and others. Most of their data are reasonably consistent, especially for particle sizes above one micron.

The deposition of dusts are best understood in the light of some knowledge of the components and the functioning of the human respiratory system. The nasal passages and oral opening through which we breathe open into the trachea in the throat which in turn leads to the bronchi. Beginning with the trachea the respiratory airways are composed of progressively branching tubes of decreasing size and cross-sectional area. The total cross-sectional area increases with depth due to the increasing number of these tubes, resulting in a decrease in the velocity of the inflowing air with depth. The branching of the tubes distributes the air uniformly to the many millions of minute alveoli, which consistute the final gas exchange units. Von Hayck[19] estimates the total respiratory surface of the lungs at 30 square meters during expiration, and at the most, at 100 square meters during the deepest inspiration. He further estimates that this area is distributed between 300 and 400 million alveoli.

In order to evaluate potential hazards with any degree of reliability the deposition sites and the retention of the material should be known. The initial deposition of particles is a physical process and can be studied in terms of the laws governing the behavior of aerosols in moving air streams and still air.

The three different forces which are of major significance in affecting the removal of particles from the inhaled air and depositing them in the respiratory tract are inertial, sedimentation and diffusion. As a result of the action of these forces, coarse particles are trapped in the nose and a fraction of the particles reaching the pulmonary air spaces are deposited and the remainder are exhaled. Hatch and Gross[12] have summarized the differing characteristics of dust trapping at various depths in the system as follows:

1. Particles larger than 10 microns are essentially all removed in the nasal chamber and therefore have little probability of penetrating to the lungs. Upper respiratory efficiency drops off as size decreases and becomes essentially zero at about 1 micron.

2. The efficiency of particle removal is high in the pulmonary air spaces, being essentially 100% down to a minimum at about ½ micron. It then increases again as the force of precipitation by diffusion increases with further reduction in size.

3. The percentage penetration of particles into the pulmonary air spaces rises from essentially zero at 10 microns to a maximum at and below about 1 micron, where it equals the fraction of tidal air which reaches the lungs.

4. The percentage of inhaled particles which penetrate to and are deposited in the pulmonary air spaces has a maximum value between 1 and 2 microns. Larger particles are deposited in the lungs in lesser degree because they are trapped higher up in the respiratory tract. Lung deposition of finer particles falls off because the local efficiency of removal decreases as size goes down below 2 microns.

5. Below ½ micron the probability of deposition in the pulmonary air spaces rises in proportion to the increase of the force of precipitation by diffusion with decreasing size.

6. The relative amount deposited and the distribution of the collected particles in the respiratory system changes with breathing frequency and tidal volume. Upper respiratory trapping increases as the rate of inspired air flow goes up with faster breathing frequency. The magnitude of deep lung deposition increases with slow deep breathing because of the larger fraction of tidal air which reaches the pulmonary spaces and the longer transit time of air into and out of the lungs.

In addition to differing collection characteristics which determine the initial site of deposition and the fraction deposited out of the inhaled air, special clearance mechanisms act to remove foreign materials from the upper respiratory tract and from the pulmonary air spaces. These mechanisms operate with varying degrees of efficiency and speed against different materials and at different sites. Particles are generally removed quickly from the upper respiratory tract while the lung clearance process may require days, weeks or even months for substantial removal of particles. The effectiveness of such a clearance system is demonstrated by the fact that large quantities of particulate matter do not accumulate in the lungs over a lifetime of exposure. McCrae[20] working with South African gold miners discovered that the amount of mineral dusts extracted from lungs represented less than one percent of a reasonable estimate of how much dust had been inhaled.

Sampling

The reduction of exposures to dust requires a means of assessing exposures. Since the etiologies of the pneumoconioses are not fully known it is not possible to define all of the characteristics of mineral dusts which contribute to their toxicities or biologic effects. All of the sampling methods used in different parts of the world for estimating exposures to dust are, therefore, empirical. None is an absolute method yielding data from which the hygienic significance of exposures can be judged precisely.

Beadle[21] suggested that the objectives of sampling are: (a) to provide a basis whereby unsatisfactory or unsafe conditions can be detected and their sources identified, (b) to assist in designing controls, (c) to provide a chronicle of changes in operational conditions, (d) to provide a basis for correlating disease or injury with dust exposure, (e) to verify and assess the suppression of dust by equipment and methods designed to do so, and finally, (f) to document compliance with health and safety regulations.

These could probably be summarized into two broad objectives. One is that of sampling for engineering surveillance, testing, or control with the other being for health research or epidemiologic purposes. Engineering applications of sampling generally are for the purpose of determining the sources of dustiness and follow-ups to check the effectiveness of control systems. A sampling program for engineering purposes should

be designed to yield the specific information desired. For example, one might need only a few samples before and after a change to determine whether the change had the desired effect.

Hygienic applications of sampling on the other hand, may be directed at predicting the health effects of an exposure by comparing sampling results with hygienic guides; determining compliance with health codes or regulations; or defining as precisely as possible environmental factors for comparison with observed medical effects. Many samples over a long period of time may be necessary to adequately define a person's exposure. This latter type of sampling may serve as the basis for the development or refinement of the hygienic guides.

It is obvious that before collecting particulate samples one should be quite clear as to the purpose for taking them and also have some knowledge of the physical characteristics of the material or materials that are to be sampled. The way a sample should be taken, the way it should be assessed and the accuracy which should be achieved will all be dependent upon the reason for taking the sample. Also, the instrument or method that should be utilized may be dictated by the character of the particulate matter. Is the material a solid or liquid? Is it soluble in or in some way affected by the collecting medium? Are the particles changed in any way in either the collection or the evaluation of the sample? Is gas, vapor, or an organic dust present in an explosive quantity? In brief, the instrument or method should not change or alter the particle in any way in the collection or assessment process. Thus sampling instruments and methods are individually inadequate or inappropriate for all situations. Conversely, each kind of instrument may be preferred in a given situation.

Among the variables which should be considered in the sampling procedure are the rate of generation of respirable dust, the direction of air motion with respect to the dust source, the person exposed, and the sampling location. The choice of sampling location is largely dependent upon these factors. As air currents change, as workmen move from place to place, individual exposures may be quite different from the concentration measured at a fixed location. Therefore, in sampling for hygienic purposes, it is often necessary to collect the sample from the breathing zone of the worker. This requires that the sampling device be kept as close to the workman as possible so that the air which it captures is representative of the air which he actually breathes.

In assessments of fluctuating dust exposures the number, duration and scheduling of samples is highly important. In assessment of daily exposures, the relative contribution of the dust concentration determined for any particular portion or operation of a work cycle is proportional to the fraction of the total work cycle represented by the operation. Each part of a work cycle should be sampled. The "time weighted average exposure" is the mathematical summation of the products of dust concentration and fractions of time for each operation or activity.

Many samples over several days may be necessary to make a time weighted exposure for hygienic purposes. Frequently the significance of exposure is judged on the basis of too few samples of too short duration. Ashford[22], Hodkinson[23], and Tomlinson[24], and others have dealth with various aspects of this problem. Roach[25] proposed the use of sampling times no more than 1/24 the biologic half time of a substance. For mineral dusts with long half times he suggests that samples be a full working shift in duration. With many instruments, samples of such duration are not now practical.

Sampling Instruments

Every conceivable method for collecting particles from the air has been suggested including washing, settling, scrubbing, filtration, impingement, Tyndall effect, electrostatic precipitation, thermal precipitation and recently, photometer methods. Indeed, there are few principles of modern physics that have not been applied to the problem of air sampling in a never-ending search to find an easier solution to the problem.

In this review only those instruments which are used to collect samples or air-borne particulates for analysis or identification will be discussed. In such a discussion it is better to deal with instrument types rather than specific instruments. In discussing the performance characteristics of a particular type it must be assumed that it is operated at its maximum efficiency and in accordance with the best operating practices. The instruments have been grouped into nine general categories according to their physical basis of operation. It is obvious that there may be some overlapping of these categories.

Sedimentation

The amount of dust which settles from a dusty atmosphere is not a good measure of the concentration of the dust in the air, since the air is constantly in motion and the volume of air associated with the settled dust is not known. A sedimentation cell[4] that has been used is a modification of an instrument described by Owen.[5] A known volume of dusty air trapped in a container is permitted to remain undisturbed for a period of time sufficiently long for particles to settle on a slide. This technique has many disadvantages. The settling time is long, limiting the number of samples. It is a spot sampling device and the air volume is limited. Since dust particles are not perfect spheres, deviations from Stokes' law is expected.

Filtration

The collection of samples of air-borne dust by filtration is one of the most common procedures used. Many different filtering media have been used from cloth to Gooch crucibles. The mass efficiency of filters are generally between 90-100%.[26] Size efficiencies vary widely with many filters showing appreciable penetration loss for certain sizes.[27,28]

The use of Whatman extraction thimbles, sometimes filled with loose packing to reduce clogging, has been described many times.[29,30] Discs of chemical grade filter paper mounted in suitable holders[31,32,33] and special pleated filters[34] for high volume samples have also been described

Tubes of volatile or soluble crystals have been used as methods of dust collection.[5,35] The efficiency of the filter depends on factors such as crystal size, depth of bed, etc. Dust collected in this manner is adaptable to chemical weight or particle size analysis.

For high temperature sampling Alundum and sintered glass filters are available.[35] There is less variation in weight due to water absorption with these than with filter paper. The collected sample may be recovered by brushing the inside of the thimble or it may be treated chemically and dissolved in the filter.

Filters which show uniformly high efficiencies, greater than 99%, for mass and particles of all sizes are limited to two types: (a) those thick deep-bed types from which it is almost impossible to recover the dust for analytical purposes, or (b) those of a high resistance membrane type, such as the molecular filters.[36,37]

In addition to the high collection efficiencies of the molecular filters they have a high surface retention and

can be subjected to a variety of procedures which facilitate the recovery and analysis of the dust including particle size analysis. Methods utilizing the light microscope are quite well established[38] and electron microscope methods have been developed.[37,39] (See also Section B-5.)

Impactors

When a high velocity, dust-laden stream of air strikes a flat surface at right angles, the sudden change in direction of air flow and momentum of the dust particles results in the separation of the dust from the air. The air passes on, leaving the particles adhering to the surface. Instrument performances, based on this principle, have been studied both experimentally and theoretically.[40,41,42]

Single and multiple stage impactors are members of this group. Generally speaking, these instruments are efficient only for larger particles since the momentum of the smaller particles is not sufficient to cause them to impact on the collecting plate.

Owens[43], May[44], and others have described impaction instruments.

Impingers

The impinger[45,46] is an apparatus through which air is sampled at high velocity and impinged from a jet onto a plate which is immersed in a liquid medium. The dust particles are impinged on the plate, thus losing their high velocity, are wetted by the liquid, and thus trapped. Any gases soluble in the absorbent are collected also. The impinger has an advantage over other sampling instruments. It has been used as the standard sampling instrument in the United States and therefore comparisons can be made with a vast amount of past experience and studies.

The midget impinger is merely a smaller version of the standard instrument. Davies[40] compares the efficiency of the impinger with that of other instruments now in use.

A rather arbitrary distinction has been drawn between impingement and impaction. In impaction the particles are directed against a dry surface (or coated with an adhesive-like material) while in impingement a liquid collecting medium is used.

Centrifugal Separators

The centrifugal separator, as is the impinger and impactor, is an inertial separator. Small scale, high velocity cyclones, have been proposed as air sampling instruments.[47,48,49] The Spiral Sampler[50] and the Conifuge[51] are other applications of the basic theory of centrifugal separations.

The dust-laden air stream enters a cone tangentially at the widest point and spirals down to the vortex where it is removed. The dust particles are collected against the side by centrifugal force plus the fact that the radical acceleration is greater than the terminal acceleration due to gravity.

In the spiral sampler the dust-laden air stream is drawn through a channel of decreasing width which is curved in an Archimedes spiral. The particles are deposited on the side in order of their decreasing size.

Spray Techniques

The Venturi Scrubber[52] is a device based on the introduction of a water spray by a nozzle into a Venturi through which the dust-laden air passes at high velocity. The dust and fume particles are wetted by the water spray and the droplets of water containing the dust particles are collected by means of a cyclone or other device. This effectively extends the efficiency of the cyclone separator to much smaller particles. Samples collected by this means are satisfactory for particle size, chemical, or weight analysis.

Electrostatic Precipitation

The electrostatic precipitator[53,54] used for air sampling is a modification of the Cottrell Precipitator.[55] Electrostatic dust samplers have three general electrode configurations - concentric, parallel and point to plane. Basically, they consist of an electrode which is maintained at either a positive or negative potential of about 12,000 - 16,000 volts and a collecting electrode. A very intense electric field, causing a corona discharge between the electrodes, is produced. Dust particles subjected to this discharge become highly charged and are attracted to the collecting electrode where they are deposited.

The effect of this electric field is such that even at relatively high air flows an efficiency of nearly 100 percent for all sizes of particles can be expected. Many investigators have suggested modifications and new designs for both the high voltage power pack and the precipitator head.[56,57] Due to its high efficiency[58] and the large sample which can be taken, this instrument is widely used.

Factors which should not be overlooked when using an electrostatic precipitator are the possibility of producing explosions when certain gases and vapors are present, and its production of ozone.

Thermal Precipitators

The thermal precipitator, developed in England, is based on the principle that a dust-free area exists around a hot wire.[59,60] Certain instruments of this general type are considered the standard for particle size sampling. For particles below 5 microns aerodynamic size there is evidence of uniformly high efficiency.[61,62] Particles can be deposited either directly onto cover slips or directly onto electron microscope grids. A very low flow rate is maintained resulting in deposition of particles in their air-borne state of agglomeration. An instrument with an oscillating stage or heating element[63] has been developed to counteract the tendency toward particle size delineation. Analyzing across the deposit in the direction of air flow will obviate this problem.

Thermal precipitation instruments, including their power supply and air movers, have been relatively large units. There are now available compact self-contained units, completely portable and capable of operating for eight hours.[64]

Elutriators

Elutriators are quite similar to inertial separators in theory but they operate at normal gravitational conditions.[65] Their design is characterized as either "vertical" or "horizontal." If air moves with laminar flow along a horizontal channel, the trajectory of air-borne particles depends upon a combination of forces. The particles of greatest mass tend to cross streamlines and settle quite promptly due to gravity while the smaller particles are kept air-borne by the resistance forces of the air for longer times and distances.

To minimize disturbance at the entrance, insure laminar flow along the elutriator channel, and avoid risk of redispersion of deposited dust, certain rules of elutriator design should be followed. Hamilton and Walter[66] discussed the important requirements in the construction of elutriators.

Although important requirements in the construction of elutriators must be met the design and operation of elutriators is ordinarily simple. As the distinction between that which is kept air-borne and that which settles can be altered above approximately one micron the elutriator can serve as a selective dust sampler.

Respirable Dust Sampling

Sampling for atmospheric particulates

presents problems which are quite different from those encountered in sampling for gases. Gases, when inhaled, can penetrate all portions of the pulmonary system while the size and density of particles influences the site in the pulmonary system at which they will be deposited. In the air passages the particles settle out as they would if passing through an elutriator. All particles which settle rapidly are removed together with a large proportion of those with a slightly slower settling rate, and so on. The proportion reaching the lungs increasing as the settling rate decreases.

The effects produced by particles trapped in the upper respiratory tract may be quite different than those deposited deeper in the pulmonary system. Therefore size selective sampling is generating increased interest. The general approach of sampling for particulates that can penetrate to, and be deposited in the lungs has become known as sampling for the "respirable fraction" of the air-borne particulate. The term "respirable fraction" has been applied to the fraction of the dust respired that reaches the lung, not the fraction of the air-borne dust that is literally respirable. Selective sampling, a term that is also used, is less descriptive as far as evaluation of a hazard is concerned. Morrow[67] points out that when using the term "selective sampling" to simulate deposition in the respiratory tract, it should be clearly stated whether the total respiratory tract (TRT), upper respiratory tract (URT), lower respiratory tract (LRT) or some other subdivision is being simulated.

The appropriate size limits to define the respirable fraction have been the subject of a number of investigations.[13,83] In all cases the size of a dust particle is defined by means of its terminal falling velocity in air, which is related to that of a spherical particle of unit density.

The result of the various investigations into the size of respirable dust were considered at an international conference held at Johannesburg[21] in 1959. At that conference it was concluded that in the light of the present available evidence, the best single parameter to measure in case of coal dust is the mass concentration of respirable dust, and in the case of quartz dust, the surface area of the respirable dust. The respirable fraction was then defined by a sampling efficiency curve which depends on the falling velocity of the particles and which passes through the following points:

100% efficiency at 1 micron and below
50% efficiency at 5 microns
0% efficiency at 7 microns and above

All sizes refer to "equivalent diameters" which is defined as the diameter of a spherical particle of unit density having the same falling velocity in air as the particle in question.

There is no difficulty in adopting a sampling program which follows these recommendations. Long term sampling instruments which sample only the respirable fraction as defined by the Johannesburg curve are available and have been used for some years in Britain.

Work in developing such instruments started as far back as 1942. Morrow[67] reports that Professor J. H. Goddum at Porton first used a selective sampler. In the same year, Bedford and Warner[68] recommended that the standard of permissible dustiness be based on mass concentrations of coal dust due to particles equal to, or less than, 5 microns in diameter. About 1948, Watson was experimenting with vertical elutriation in combination with a thermal precipitator and a gravimetric sampler.[69] In South Africa, Kitto and Beadle[70] were modifying their thermal precipitator with a horizontal elutriator to discriminate against larger particles. The Medical Research Council panels in Great Britain in the early 1950's established the following principles:

1. Selective sampling should be defined in terms of the terminal velocity of particles, not on the basis of their approximate size.

2. A sharp size selective cutoff was not technically feasible nor desirable.

3. A selection curve based on the horizontal elutriator designed by Hamilton and Walton was adopted as a working standard.[71]

May and Druett[72] described their pre-impinger in 1953. The same year Watson[69] discussed the design of a two stage sampling instrument which would separate the larger from the smaller particles. The following year Wright[73] described the design of the Hexhlet elutriator as a selective sampler. The Hexhlet selective sampler collected all particles above 7 microns equivalent diameter, 50% of the 5 microns, and permitted the passage of all particles below 1 micron. All of the escaping particles were collected by a filter thimble.

Dawes and co-workers[74] discussed in 1954 the general philosophy of selective sampling and the design and performance of a long-term selective sampler based on a vertical elutriator as a single stage sampler. Dusty air was pulled through a horizontal mounted filter such that the upward linear velocity of air entering the filter was equivalent to the settling velocity of a 6.25-micron equivalent sphere. Particles exceeding this equivalent size would fall at a greater velocity and be eliminated.

Two other selective sampling instruments were reported in 1956 by Burdekin and Dawes[75] and by Hamilton[76] using a gravity settling stage followed by a thermal precipitator.

In the United States selective sampling efforts began in the early '50's. Burnet and Hatch[77] described some of the criteria for a respiratory sampler designed to estimate radioactive particle hazards. Tests with the Aerotec Tube Design 2 Cyclone[78] indicated that it could collect a sample corresponding to upper respiratory tract deposition of Brown et al.

Selective samplers were investigated by the Mine Safety Appliances Company in 1954[79] in which they reported on the feasibility of collecting and classifying radioactive dusts by this technique.

As a result of this early work, sampling devices designed to reject particles too large to penetrate to the deeper portions of the lung, or to collect these larger particles in a presampler, with the respirable portion collected separately for weighing or analysis have been developed.

Samplers capable of operating continuously during a shift, collecting a sample of sufficient size for weighing and analysis, and collecting a sample based on the equivalent diameter of a particle, are represented by three groups of instruments as follows:

1. Cyclones. A family of size selective samplers[84] has been developed by the Health and Safety Laboratory, U. S. Atomic Energy Commission, New York. These samplers are used for measuring the respirable and non-respirable air-borne dust in the uranium and beryllium industries. They range in capacity from less than a liter per minute to 40 cubic feet per minute. The non-respirable fraction is first separated by means of a cyclone and the respirable fraction is collected on a filter. The cut-off differs from that recommended by the Johannesburg Conference and is as follows:

   100% respirable at 2 microns
   75% respirable at 2.5 microns
   50% respirable at 3.5 microns
   25% respirable at 5 microns
   0% respirable at 10 microns

The size of the particles are given in equivalent diameters.

2. Hexhlet. This sampler was designed in accordance with the recommendations of the Medical Research Council of Great Britain and was first described by Wright.(73) The early instruments sampled at 100 liters per minute but this was later reduced to 50 liters per minute. The non-respirable fraction is first separated by means of a horizontal elutriator and the respirable fraction collected on a filter. The cut-off is the same as that which was later recommended at Johannesburg.

3. Conicycle. The Conicycle(81), also developed by the MRC, is entirely self-contained; power for driving its rotating head being supplied by batteries, which can be recharged. The sampling rate is 10 liters per minute with some having a lower rate of 8 liters per minute.

   The instrument is fitted with a sampling head which performs four functions: (a) it pumps air through itself, (b) it rejects particles above a predetermined equivalent diameter, (c) it retains particles entering the sampler, and (d) it allows the finer particles to pass through to an extent inversely proportional to their equivalent diameters.

   In the current model of this instrument, the upper cut-off is identical to that of the Hexhlet.

Aerosol Concentration

The concentration of air-borne particulate matter can be expressed in any number of ways. It has been customary in the United States to report mineral dust concentrations in terms of numbers of particles per unit of air volume. Other parameters used are the mass, or surface area per unit of air volume. By fractioning the total sample into a series of narrow size bands the material in each fraction can be reported in terms of any parameter.

The composition of the fine dust which can reach the lungs is not necessarily the same as that of the total air-borne dust. The samples yielded by size selective samplers, which simulate the deposition characteristics of the lower pulmonary system, will be of great value in studies of dust composition.

Hatch and Gross(12) summarized the problem very well when they stated "there is no firm and final answer to the question: how best to collect aerosol samples and how to express aerosol concentration and composition. Indeed, there is no one best procedure since the nature of the relationship between the inhaled aerosols and the disease risk, which is mediated in large measure by the behavior of the particles in the respiratory system, differs so much from one disease to another. It seems clear that the relatively simple sampling procedures and analytical methods which were employed successfully in dealing with the relatively gross hazards of the past cannot be depended on to fully meet the needs of the future."

Summary

The control of exposures to particulates requires a means of assessing concentrations. Methods which are used are empirical and results yielded by one method cannot be compared directly with those yielded by another. Each kind of instrument has the possibility of being preferred in a given situation, but no one instrument has all the features that might be desired in an ideal instrument.

Particulate matter should be described in terms of its characteristic ranges in particle shape, density and size which determine its consequent relative stability in the respiratory system

rather than on the nature or origin of this material.

Sampling may be undertaken for two distinct reasons; for control purposes or for health studies. The objective should be kept in mind while planning and carrying out the study.

The respiratory system is selective in the acceptance and deposition of particles, generally only the smaller particles move with the air stream and settle very slowly. As concentrations vary with time and location time weighted average exposures should be calculated with the samples being representative of the breathing zone.

Because of the size selective characteristics of the respiratory system, size selective sampling devices intended to simulate the deposition characteristics of the respiratory system have been developed.

The concentration of air-borne particulate matter can be expressed several different ways; by number, mass, surface area and others. There is no firm and final answer on how to express concentrations. It has been recommended that the best single parameter to measure in the case of coal dust is the mass concentration of respirable dust; and, in the case of quartz dust, the surface area of the respirable dust.

References

1. Plinius Secundus, Caius: Naturalis Historiae. II. Trans. by Kenneth C. Bailey under the title, "The Elder Pliny's Chapters on Chemical Subjects," pt. I, p. 125, 1929.

2. Cunningham, D. D.: Microscopic Examination of the Air. Superintendent of Govt. Printing, Calcutta, 1873.

3. Heymann, D.: Die Verfahren zur quantitative Bestimmung in der Atenluft, Zentr. ges. Hyg., 24: 1 (1931).

4. Silverman, L.: Sampling of Industrial Stacks and Effluents for Atmospheric Pollution Control. Proc. First Nat. Air Pollution Symposium, pp. 55-60, Los Angeles, Calif., March 1950.

5. Determination and Control of Industrial Dust. Public Health Bulletin #217, April 1935.

6. Jacobs, M. B.: Analytical Chemistry of Poisons, Hazards and Solvents. Interscience Publishers, Inc., New York, 2nd Ed., Rev., 1949.

7. Brown, C. E. and H. H. Schrenk: Standard Methods for Measuring the Extent of Air Pollution. U. S. Bureau of Mines, I.C. 7210, May 1942.

8. Drinker, P. and T. Hatch: Industrial Dust. McGraw-Hill Book Co., New York, 2nd Ed., 1954.

9. Green, H. L.: Some Accurate Methods for Determining the Number and Size Frequency of Particles in Dust. J. Ind. Hyg., 16: 29 (1934).

10. Owens, J. S.: Suspended Impurities in Air. Proc. Royal Soc. (London) a., 101: 18 (1922).

11. Inhaled Particles and Vapors, C. N. Davies, Editor, Pergamon Press, London, 1961.

12. Hatch, T. F., and P. Gross: Pulmonary Deposition and Retention of Inhaled Aerosols. Academic Press, New York, 1964.

13. Brown, J. H., K. M. Cook, F. G. Ney and T. Hatch: Influence of Particle Size upon the Retention of Particulate Matter in the Human Lung. Am. J. Public Health, 40: 450 (1950).

14. Landahl, H. D., T.N. Tracewell, and

W. H. Lassen: Retention of Airborne Particulates in the Human Lung. III, A.M.A. Arch. Ind. Hyg. Occupational Med., 6: 508 (1952).

15. Altshuler, B., L. Yarmus, E. D. Palmes, and N. Nelson: Aerosol Deposition in the Human Respiratory Tract. A.M.A. Arch. Ind. Health, 15: 293 (1957).

16. Dautrebande, L., H. Beckmann, and W. Walkenhorst: Studies on Deposition of Submicronic Dust Particles in the Respiratory Tract. A.M.A. Arch. Ind. Health, 19: 383 (1959).

17. Van Wijk, A.M., and H.S. Patterson: The Percentage of Particles of Different Sizes Removed from Dust-Laden Air by Breathing. J. Ind. Hyg. Toxicol., 22: 31 (1940).

18. Wilson, I. B., and V. K. LaMer: The Retention of Aerosol Particles in the Human Respiratory Tract as a Function of Particle Radius. J. Ind. Hyg. Toxicol., 30: 265 (1948).

19. Von Hayek, H.: The Human Lung (translated by V. E. Krahl), Hafner Publishing Company, Inc., New York, 1960.

20. McCrae, J.: The Ash of Silicotic Lungs, Publs. S. African Inst. Med. Research, 1939.

21. Proceedings of the Pneumoconiosis Conference. Johannesburg, 1959, J. & A. Churchill, Ltd., London (Little, Brown, & Co., N. Y.), 1960.

22. Ashford, J.R.: The Design of a Long Term Sampling Programme to Measure the Hazard Associated with an Industrial Environment. J. Royal Statis. Soc., 121, Part 3, 333 (1958).

23. Hodkinson, J. R.: Reproducibility and Correlation of Long-Period and Short-Period Thermal Precipitation Samples of Air-borne Dust in Mines. Ann. Occup. Hyg., 2: 235 (1960).

24. Tomilson, R.C.: A Simple Sequential Procedure to Test Whether Average Conditions Achieve A Certain Standard. Applied Statistics, VI, 3: 198 (1957).

25. Roach, S.A.: The Importance of Body Burden in Establishing Hygienic Standards; presented at 2nd International Symposium on Permissible Limits for Air, Paris, April 1-6, 1963.

26. Handbook on Air Cleaning. Atomic Energy Commission, 1952.

27. Lindeken, C. L., R. L. Morgin, and K.F. Petrock: Collection Efficiency of Whatman 41 Filter Paper for Submicron Aerosols. Report UCRL-6691, 1962.

28. Stanley, N.: The Penetration of Filter Paper by Coal Dust Particles in the Respirable Size Range. Ann. Occup. Hyg., 4: 295 (1962).

29. Trostel, L. S., and H. W. Frevert: Collection and Examination of Explosive Dust in Air. J. Ind. Eng. Chem., 15: 232 (1923).

30. Brady, W., and L. A. Touzalin: The Determination of Dust in Blast Furnace Gas. J. Ind. Eng. Chem., 3: 662 (1911).

31. Silverman, L., and C. Valenzuela: Filter Paper Collection of Cadmium and Iron Fumes in Air. J. Ind. Hyg. Toxicol., 3: 107 (1946).

32. Bourne, H. G., and L.P. Street: The Use of Filter Papers for Collecting Aerosols. Paper Trade Jour., May 11, 1950.

33. Silverman, L., and F. J. Viles: A High Volume Air Sampling and Filter Weighing Method for Certain Aerosols. J.Ind. Hyg. Toxicol., 30:124(1948).

34. Brown, C. E.: Filter Paper Method for Obtaining Dust Concentration

Results Comparable to Impinger Results. U. S. Bureau of Mines, RI 3788, 1948.

35. Arbogast, A. H.: The Quantitative Determination of Dust in Gas. Iron & Steel Eng., 25: 82 (1948).

36. Literature and Bibliography available from: Millipore Filter Corporation, Watertown 72, Massachusetts.

37. Fraser, D. A.: Absolute Method of Sampling and Measurement of Solid Air-Borne Particulates. Combined Use of the Molecular Filter Membrane and Electron Microscopy. A.M.A. Arch. Ind. Hyg. and Occup. Med., 8: 412 (1953).

38. Various Publications of the American Society for Testing and Materials, Philadelphia, Pennsylvania, and ASTM Special Technical Publications, No. 25 - Instrumentation in Atmospheric Analysis.

39. Kalmus, E. H.: Preparation of Aerosols for Electron Microscopy. Z. Wissenshaft Mikroskopie U. Mikro Technik, 64: 414 (1960).

40. Davies, C. N., M. Aylward, and D. Leacey: Impingement of Dust from Air Jets. Arch. Ind. Hyg. & Occup. Med., 4: 354 (1951).

41. Davies, C. N., and M. Aylward: The Trajectories of Heavy Solid Particles in a Two Dimensional Jet of Ideal Fluid Impinging Normally upon a Plate. Proc. Phys. Soc. (London), B., 64: 889 (1951).

42. Ranz, W. E., and J. B. Wong: Jet Impactors for Determining the Particle-size Distribution of Aerosols. Arch. Ind. Hyg. & Occup. Med., 5: 462 (1952).

43. Owens, J. S.: Suspended Impurities in Air. Proc. Royal Soc. (London), a., 101: 18 (1922).

44. May, K. A.: The Cascade Impactor, An Instrument for Sampling Coarse Aerosols. J. Sci. Instru., 22: 187 (1945).

45. Greenburg, L., and G. W. Smith: A New Instrument for Sampling Aerial Dust. U. S. Bureau of Mines, RI 2392, 1922.

46. Dallavalle, J. M.: U. S. Public Health Service, Reprint #1848, 1937.

47. Stern, A. C.: The Measurement and Properties of Cinders and Flyash. Combustion No. 4, 35 (June-July 1933).

48. Hardie, P. H.: Cinder and Flyash Measurement. Combustion No. 4, 10 (March 1933).

49. _____: Engineering Data. The Thermix Engineering Co., Greenwich, Conn., September 20, 1946.

50. Yaffe, C. D., A. D. Hosey, and J.T. Chambers: The Spiral Sampler - A New Tool for Studying Particulate Matter. Arch. Ind. Hyg. & Occup. Med., 5: 62 (1952).

51. Sawyer, K. F., and W. H. Walton: The "Conifuge" - A Size Separating Sampling Device for Air-Borne Particles. J. Sci. Instr. (London) 27: 272 (1950).

52. McGill, P. L.: Report of the Stanford Research Institute, 1949.

53. Drinker, P., R. M. Thomson, and S. J. Fitchet: Atmospheric Particulate Matter: II. The Use of Electric Precipitation for Quantitative Determination and Microscopy. J. Ind. Hyg., 5: 162 (1923).

54. Barnes, E. C., and G. W. Penny: An Electrostatic Dust Count Sampler. J. Ind. Hyg. Toxicol., 18: 167 (1936).

55. Cottrell, F. G.: Problems in Smoke, Fume, and Dust Control. Smithsonian Report for 1913, Pub. 2307, p. 653.

56. Clayton, G. D.: Improvement of the M.S.A. Electrostatic Precipitator. J. Ind. Hyg. Toxicol., 29: 400 (1947).

57. Hosey, A. D., and H. H. Jones: Portable Electrostatic Precipitator Operating from 110 Volts AC or 6 Volts DC. Arch. Ind. Hyg. Occup. Med., 7: 49 (1953).

58. Fraser, D. A.: The Collection of Submicron Air-borne Particulate by Electrostatic Precipitation. AIHA Quart. 17: 75 (1956).

59. Green, H. L., and H. H. Watson: Physical Methods for the Estimation of Dust in Industry. Med. Res. Council, His Majesty's Stationery Office, Code 45, 1934, pp. 5-99.

60. _____: "Konisampler." Jos. B. Ficklen, 3rd., 1848 E. Mountain Street, Pasadena 7, California.

61. Watson, W. H.: The Sampling Efficiency of the Thermal Precipitator. Brit. J. Appl. Phys., 9: 78 (1958).

62. Zebel, G.: Comparative Measurement on Aerosols of Fine Particles With A Thermal Precipitator and a Slit-Ultramicroscope. Staub, 39: 21 (1955).

63. Lauterbach, K.R., S. Laskin Wilson, and D. Meier: Design of Oscillating Thermal Precipitator. USAEC Report No. UR-199, 1952.

64. Orr, C., and R. A. Martin: Thermal Precipitator for Continuous Aerosol Sampling. Rev. Sci. Instr., 29: 129 (1958).

65. Walton, W. H.: The Theory of Size Classification of Air-Borne Dust Clouds by Elutriation. Brit. J. Appl. Phys. Suppl. 3: (1954).

66. Hamilton, R. J., and W. H. Walton: The Selective Sampling of Respirable Dust. Section VII., p. 465-475, Inhaled Particles and Vapors, Pergamon Press, Ltd., London, 1961.

67. Morrow, Paul E.: Evaluation of Inhalation Hazards Based Upon the Respirable Dust Concept and the Philosophy and Application of Selective Sampling. Am. Ind. Hyg. Assoc. J. 25: 213 (1964).

68. Bedford, T., and C. G. Warner: Measurement of Concentration of Air-Borne Dusts. J. Ind. Hyg. Toxicol., 24: 315 (1942).

69. Watson, W. H.: Dust Sampling to Simulate the Human Lung. Brit. J. Ind. Med., 10: 93 (1953).

70. Kitto, P. H., and D. G. Beadle: A Modified Form of Thermal Precipitator. J. Chem. Met. Mining Soc., S. Africa, p. 284 (June 1952).

71. Hamilton, R. J., and W. H. Walton: The Selective Sampling of Respirable Dust. Proceedings of the International Symposium on Inhaled Particles and Vapors. Oxford, p. 465, 1960.

72. May, K. R., and H. A. Druett: The Pre-Impinger - A Selective Aerosol Sampler. Brit. J. Ind. Med., 10: 142 (1953).

73. Wright, B. M.: A Size Selecting Sampler for Air-Borne Dust. Brit. J. Ind. Med., 11: 284 (1954).

74. Dawes, J. G., S. R. Howarth, and A. Slack: Trials with an Experimental Long-Period Sampler for Air-Borne Dust. Safety in Mines Research Establishment. Research Report No. 87, 1954.

75. Burdekin, J. T., and J. G. Dawes: The Use of a Size Selector for Dust Sampling with the Thermal Precipitator. Brit. J. Ind. Med., 13: 196 (1956).

76. Hamilton, R. J.: A Portable Instrument for Respirable Dust Sampling. J. Sci. Instr., 33: 395 (1956).

77. Burnett, T. J., and T. Hatch: Estimating Air-Borne Radioactive Particulate Hazards. A Review of Sampling Criteria. Am. Ind. Hyg. Assoc. Quart. 17: 80 (1956).

78. Harris, W. B., and M. Eisenbud: Dust Sampler which Simulates Upper and Lower Lung Deposition. A.M.A. Arch. Ind. Hyg. & Occup. Med. 8: 446 (1953).

79. Mine Safety Appliance Company: An Investigation of Sampling for the Collection and Classification of Radioactive Air-Borne Particulate Materials. Report NP-5423, 1954.

80. Bruer, H.: A Filter Device for the Selective Sampling, in Coal Mines, of Respirable Fine Dusts: Sizing of the Dusts by Vortex Classifier, Gravimetric Determination of the Concentration, the Ash Content and the Quartz Content of Fine Dusts. Proceedings of the International Symposium on Inhaled Particles and Vapors. Oxford, p. 453, 1960.

81. Wolff, H. S., and S. A. Roach: The Conicycle Selective Sampling System, Inhaled Particles and Vapors, C. N. Davis, Editor, Pergamon Press, London, 1961.

82. Hyatt, E. C., et al.: A Study of Two Stage Air Samplers Designed to Simulate the Upper and Lower Respiratory Tract. Proceedings of the Thirteenth International Congress on Occupational Health, New York, 1960.

83. Davies, C. N.: Inhalation Risk and Particle Size in Dusts and Mists. Brit. J. Ind. Med., 6: 245 (1949).

84. Lippman, M., and W. B. Harris: Size-Selective Sampler for Estimating "Respirable" Dust Concentrations. Health Physics, 8: 155 (1962).

AIR FLOW CALIBRATION

Introduction

In sampling an atmosphere for contaminants the objective is to determine as accurately as possible the concentration of various substances. Usually this requires that the air containing the contaminants be captured and analyzed simultaneously, or that the contaminants be removed from the air and retained for subsequent analysis. The results of such determinations are usually expressed in terms of weight, number of particles, activity, or parts per unit volume of the atmosphere sampled. Therefore, the sample volume is equally as important as the quantitative determination. Since it is possible to determine gas volumes or flow rates to a high degree of accuracy, it is prudent to reduce to a minimum this potential source of error for any sampling system.

Air volume or flow calibration may be defined as the determination, within given limits, of the true value of the scale reading, volume or indications of an instrument. The calibration of air sampling instruments or systems can be divided into two basic groups:

Grab, instantaneous, fixed or captured volume samples. These usually are calibrated by direct volumetric means or by a technique such as the bubble meter. Primary standards usually are available for this type of instrument.

Continuous or integrated samples. These usually are calibrated against a secondary standard such as a gas meter, orifice meter, or in special cases the air-flow velocity through a fixed area or gas dilution technique may be employed.

The frequency of calibration is dependent on the use, care, and handling of the instrument and probably most important - to what purpose the data are to be applied. Ordinarily, air sampling devices are calibrated in the laboratory before they are used in the field. The instruments should be calibrated if they have been subjected to misuse, cannot be properly adjusted or zeroed, or if they have just been repaired or received from a manufacturer. It is also wise to check the calibration of flow measuring devices after they have been used in the field to collect a large number of samples.

Before initiating an air-flow calibration of a given instrument it is necessary to:

(1) Understand the operation of the instrument and how it is to be used,

(2) determine the degree of accuracy necessary,

(3) know what calibration facilities are available, and

(4) have a familiarity with the gas laws and calculations.

Calibration Equipment

Primary Standards

These are usually volume measuring devices. The volume under consideration is measured by some direct physical method independent of the air or gas involved, such as physical dimensions of the containing vessel.

Spirometer. The spirometer[1] (see Figure 1) is simply a bell or cylinder with one end closed floating in water or oil. The weight of the bell is counter-balanced by weights so there is negligible resistance to movement in and out of the fluid as air is removed or added to the cylinder cavity. The volume ($V = AxH$) of the cavity is determined by the cross sectional area (A) of the cylinder and

Prepared by: Julius H. Fanney, Jr.

the height (H) of the cylinder above the fluid. Spirometers are commercially available with capacities ranging from a few hundred centimeters to 10 or more cubic feet.

Other standards. An even simpler, and by far cheaper, apparatus which can be fabricated in most laboratories consists of a carboy of large volume fitted with a drain (Marriotti's bottle) or siphon carboy[2,3] from which water is displaced and measured volumetrically. For smaller volumes a burrette can be used to make a bubble meter[4] for the calibration purposes. (See Figure 2).

Limitations. All of the primary standards available are subject to the following limitations in their application:

(1) Limited to atmospheric pressure applications

(2) limited volume

(3) low flow rates

(4) usually requires some skill in operation.

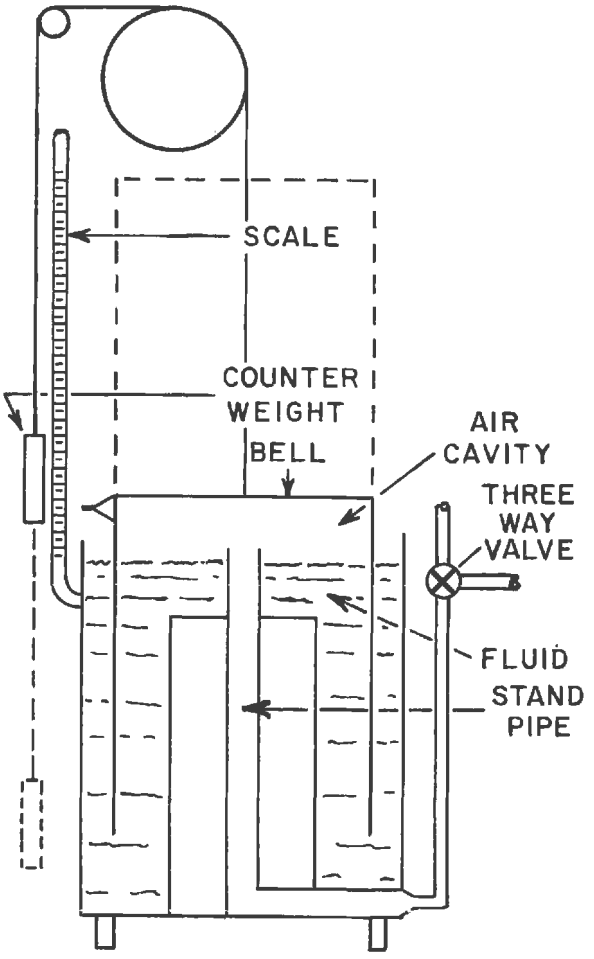


Figure 1. - Schematic drawing of a spirometer.

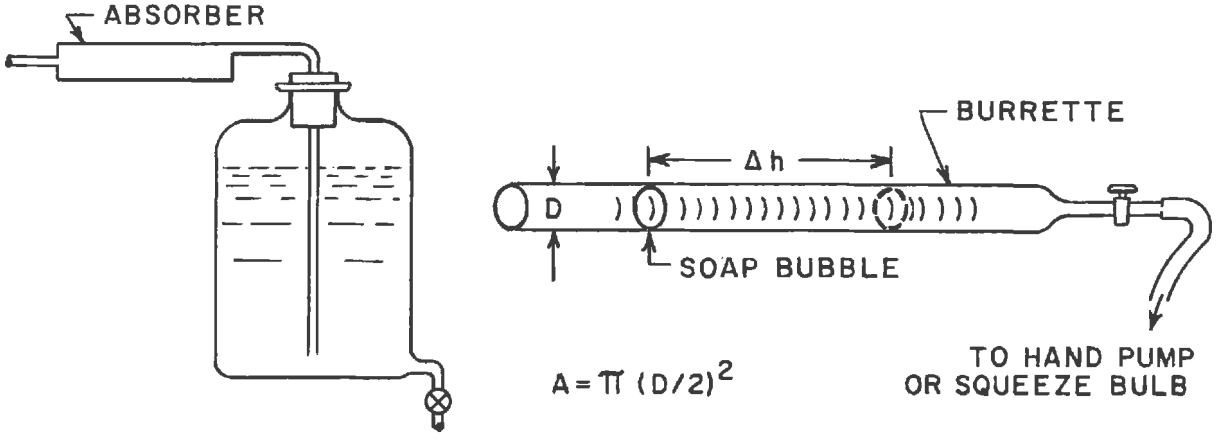


Figure 2. - Marriotti bottle (left) and bubble meter (right).

Manometer. In almost all instances of calibration, it is necessary to have some means of measuring pressure or vacuum in one or more branches of the system. The primary standard device for measuring this variable is the manometer. The simplest form is a glass tube bent in the form of the

letter "U" and partially filled with a liquid. There are many special forms of manometers designed tomeet the specific conditions or measurements required.

In use, if one leg of a manometer is connected to a calibration system in which the pressure is slightly greater than atmosphere, then the column of liquid will go down on the connected side and up on the open leg. The difference in the levels of the liquid in the two legs is a measure of the pressure difference. The amount that the levels will move, or the difference in level depends not only on the pressure differential, but also on the unit volume weight (density) of the liquid being used.

Care must be exercised in the use of these instruments to keep the fluid and tube clean. It is also important to use the correct specific gravity fluid and not to "blow" the meter by applying too much pressure or vacuum.

Intermediate Standards

There are several pieces of equipment which are not primary standards by the strict definition; however, they are accurate and maintain their accuracy if they are handled and used properly. Examples are: wet test meter, dry gas meter, Pitot tube[5], and orifice plates[6].

Wet test meter. A wet test meter consists of a partitioned drum half submerged in a liquid (usually water) with openings at the center and periphery of each radial chamber. Air or gas enters at the center and flows into an individual compartment causing it to rise thereby producing rotation. This rotation is indicated by a dial on the face of the instrument. The volume measured will be dependent on the fluid level in the meter since the liquid is displaced by air. A sight gauge for determining fluid height is provided and the meter may be leveled by screws and a sight bubble which are provided for this purpose.

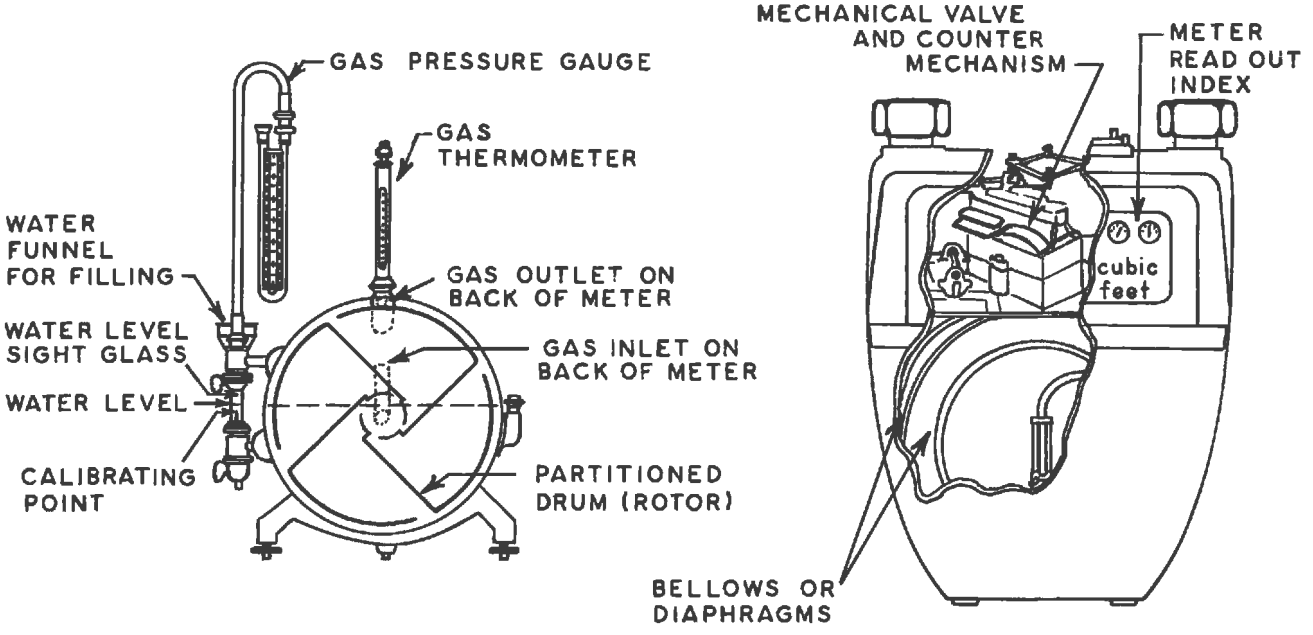


Figure 3. - Wet test meter (left) and dry gas meter (right).

There are several errors inherent in the use of a wet test meter. The drum and moving parts are subject to corrosion and damage from misuse, there is friction in the bearings and the mechanical counter, inertia must be overcome at low flow (1 RPM), and at high flow (3 RPM), the liquid might surge and break the water seal at the inlet or outlet. In spite of these factors, the accuracy of the meter usually is within one percent when used as directed by the manufacturer.

Dry gas meter. The dry gas meter, very similar to the domestic gas meter, consists of two bags interconnected by mechanical valves and a counting device. The air or gas as it fills one bag mechanically empties itself. Any such device has the disadvantage of mechanical drag, pressure drop, and leakage; however, the advantage of being able to use the meter under rather high pressures and volumes often outweighs these errors, which can be determined for a specific set of conditions.

Other meters. The orifice meter[6,7], Pitot tube[5,8] and thermal anemometer[9] also fall into the category of intermediate standards. If carefully designed and constructed these devices can be used as intermediate, secondary standards[10] without calibration against a primary standard. The use of the latter two as calibration instruments is secondary since they were originally designed and intended for air velocity measurements. A discussion of these instruments and their theoretical calculation are covered in Section C-7, Measurement of Air Flow.

The major difficulties with the above instruments are:

1. Setting up a calibration procedure which uses the instrument in the proper velocity range and application.

2. Determining if changes have occurred since they were last checked, or constructed.

3. In the case of the orifice meter there is a high pressure drop across it.

Secondary Standards

A secondary standard is any type of apparatus, either flow or volume meter, which has been accurately calibrated by comparison under a given set of conditions with a primary calibrating device. Secondary standards must be used in other systems under conditions similar to the original calibration for the results to be significant[11]. This is particularly true if the conditions of use would affect the physical properties such as pressure, temperature, density, etc., of the gas passing through the unit.

Examples of flow mass or resistance meters of this type are: rotameters[12], venturi, critical[13] and variable orifice plates[7], and packed plug meters[14]. These instruments are the most commonly used devices to measure air flow in sampling instruments.

Quantity meters. The general formula for volume rate of air flow in a quantity meter is given by the following relationship:

$$(1)\quad Q = 0.0997\left(\frac{CD^2}{\sqrt{1-\beta^4}}\right)\sqrt{\frac{hw}{\rho}}$$

where Q = volume rate of air flow in cubic feet per second
C = coefficient of discharge
D = diameter of throat or orifice in inches
β = ratio of throat or orifice diameter to pipe or tube diameter
h_w = pressure differential in inches of water
ρ = density of fluid (air, gas or liquid) lb/cu. ft.

Variable orifice meter. Flow conditions through these metering devices are affected by a number of factors; however, for orifice meters two of these factors

(C and β) have been determined experimentally[15] for a selected set of conditions related to the Reynolds number. (See Section C-6, Principles of Ventilation, for discussion of Reynolds number.) By letting K represent the function $C/\sqrt{1-\beta^4}$, inserting 0.0749 for ρ (the density of air at 70°F and 760 mm Hg) and converting to more convenient units, the generalized equation (1) becomes:

$$(2) \qquad Q = 21.8\ K\ d_m^{\ 2}\ \sqrt{\Delta h_w}$$

where Q = volume rate of air flow in cu. ft/min.

K = factor representing coefficient of air flow conditions related to Reynolds number[15,16].

d_m = orifice diameter in inches

Δh_w = pressure drop across orifice in inches of water

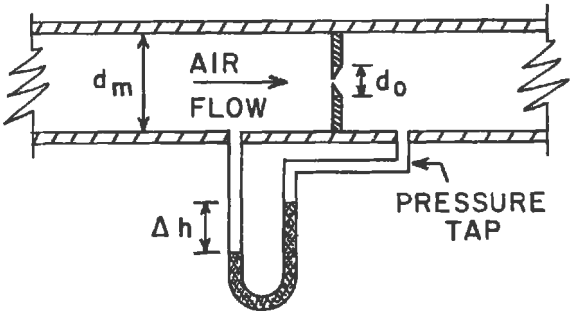


Figure 4. - Drawing of a sharp-edge orifice.

From this equation and the appropriate tables the volume rate of air flow at standard conditions through a thin-plate or sharp-edge orifice with flange taps can be calculated[17]. These calculations are usually sufficiently accurate (± 10%) for secondary calibrations; however, if more precision is desired the unit should be compared against a primary standard.

Critical orifice meter. A specialized, but common application of the orifice meter, for both calibration and sampling, is in the form of a limiting or critical orifice[13]. (A more detailed discussion of this subject appears in Section C-6.)

Venturi meter. The venturi meter, another form of resistance meter, has the advantage, if properly constructed, of low pressure drop over a wide range of flow. This makes its use for calibration purposes extremely advantageous. They are usually calibrated by comparison with a secondary standard or their flow can be calculated within ± 10%. (See Section C-6 for a more detailed discussion.)

Limitations. The difficulties in using orifice and venturi meters as secondary standards as mentioned previously are:

1. Initial calibration of the units are often difficult in the range desired;

2. the device must be used under the same conditions, i.e., pressure, temperature, gas, etc., as originally calibrated;

3. the possibility of the calibration changing because of damage or dirt on the constriction; and

4. high pressure drop across meter.

Rotameters. The rotameter or variable area meter probably is the most used and misused tool for the measurement of air flow. It consists of a precision-bored, tapered, transparent tube with a solid float inside. The float is carried upward by the flow of air until it reaches a level where the force of the air is offset by the weight of the float. The height of the float above the bottom of the tube is a measure of the air flow rate. It operates on the same principle as the orifice meter, except the area of the orifice varies while the differential pressure remains constant. The motion of the float up or down the tapered tube provides the variable area of the orifice.

Rotameters should be calibrated under the same conditions of pressure, temperature, and density (gas) as they are to be used[11]. This is particularly

true if the conditions of use would affect the density of the gas passing through the unit. It is always a good policy to have one end of a rotameter open to the atmosphere in a calibration setup unless the instrument has been specially calibrated for operation other than at standard conditions. Extreme care should be exercised in the use of a calibrated standard rotameter to see that particulate matter or vapors of any type do not enter the meter and that the float is not handled or damaged.

The question as to where to read the float always exists. As a general rule, manufacturers calibrate their units using the highest-widest portion of the float. Figure 5 indicates the correct marker point for representative floats.

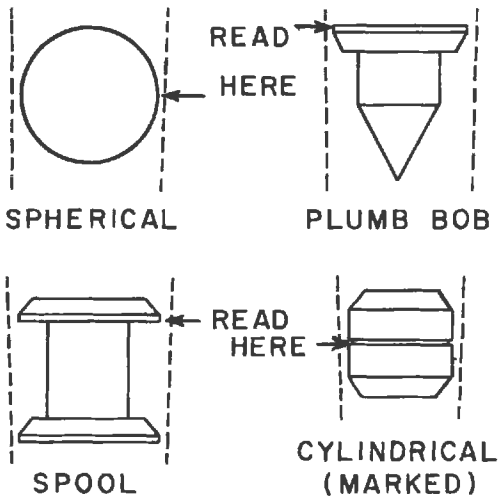


Figure 5. - Typical Rotameter Floats.

Packed plug flow meter. The packed plug flow meter[14,18] is another form of resistance meter. It is a device for both regulating and measuring very low flows, down to 0.01 ml/min. The device consists of a "T" connection, pipet or glass tubing, cylinder and packing material. The outlet arm of the "T" is packed with material, such as asbestos, and the leg is attached to a tube or pipet projecting down into the cylinder filled with water or oil. A calibration curve of the depth of the tube outlet below the water level versus the rate of flow should produce a straight line curve.

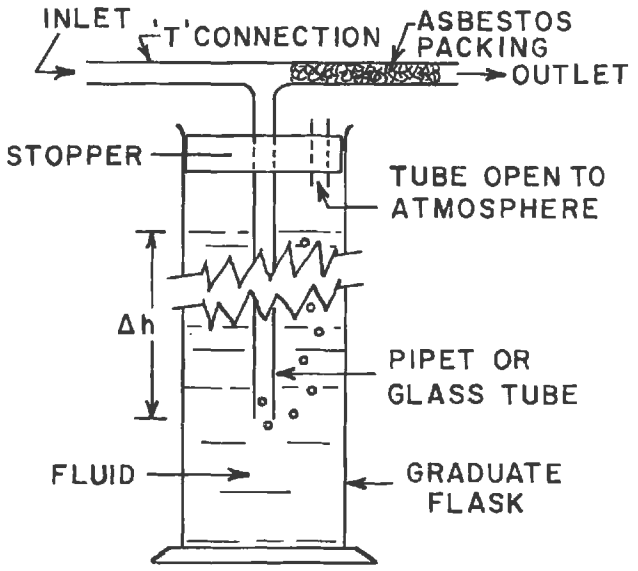


Figure 6. - Drawing of a Packed Plug Flow Meter.

Pressure vacuum gages. Unlike the manometer these devices usually are used on sampling instruments; however, occasionally they may be useful in calibration procedures. They normally consist of a flexible diaphragm connected mechanically or otherwise to a pointer assembly. Naturally, any such device is subject to mechanical damage, deterioration, pressure changes, dirt, moisture, and orientation.

Calibration Procedures

No attempt will be made to describe all of the possible methods or the combinations thereof which have been used for calibration of air sampling instruments. A few classic examples and general procedures will be discussed. For specific methods the reader should consult the numerous references and other sources of literature on this subject.

Precalibration Check

The calibration of a secondary standard

such as a wet test meter, orifice, rotameter, etc., with a gas prover or spirometer is a basic procedure. Before initiating the calibration a few checks on the operation of the spirometer are necessary.

1. Determine that the instrument is level, the fluid is adequate, the mechanical linkage is aligned and that it is free running.

2. With the valves open, move the bell (by means of the counter-weight) to a full down position and then release it. The bell should rise to the center of its traverse. Repeat the procedure by raising the bell to its maximum capacity. Again the bell should drift slowly to the center. Should the bell not come to rest at the mid point of its traverse the counter-weight should be adjusted. On models equipped with eccentric pulleys the bell should remain stationary rather than drift.

3. Check the valve closure by applying air pressure and vacuum to the closed valves and noting any changes in the bell position. The bells of spirometers are usually constructed of very light weight material and are easily damaged. Any dent or deformity in the bell changes its capacity and, therefore, its calibration factor. Do not apply high pressure or vacuum to the instrument.

Having established that the primary standard is in operating condition the calibration setup could be as shown in Figure 7.

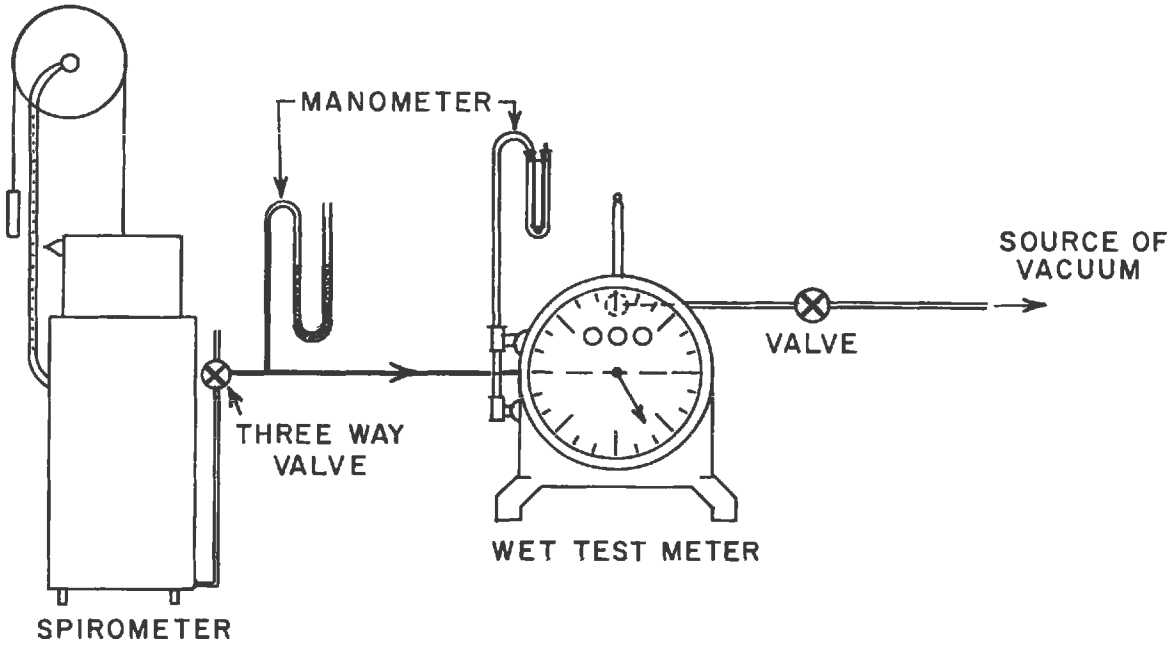


Figure 7. - Calibration Setup for Calibrating a Wet Test Meter.

Calibration of Secondary Standard

The primary standard (spirometer or siphon bottle) is connected to the device to be calibrated by as short a connection as possible. The diameter of the connection should be large enough so that resistance is insignificant. A good rule of thumb is to use the largest diameter connection appearing on the primary standard as the connecting tube diameter. A quick action by-pass valve should be installed at the connection to the standard if one is not present.

This will allow the device being calibrated to be adjusted without being limited by the standard's capacity, and also to facilitate calibration. Temperature and pressure sensing devices should also be available for the standard's air cavity and pressure measurements at the proper location(s) on devices to be calibrated. After all connections have been made, it is a good policy to recheck the level of all instruments and determine that all connections are clear and have minimum resistance. If compressed air is used in a calibration procedure it should be cleaned and dried.

Actual calibration of the wet test meter shown in Figure 7 is accomplished by opening the by-pass valve and adjusting the vacuum source to obtain the desired flow rate. The optimum range of operation is between one and three revolutions per minute. Before actual calibration is initiated the wet test meter should be operated for several hours in this setup to stabilize the meter fluid as to temperature, absorbed gas, and to work in the bearings and mechanical linkage. After all elements of the system have been adjusted, zeroed and stabilized several trial runs should be made. During these runs, should any difference in pressure be indicated, the cause should be determined and corrected. The actual procedure would be to instantaneously divert the air to the spirometer for a predetermined volume indicated by the wet test meter (minimum of one revolution), or to near capacity of the spirometer, then return to the by-pass arrangement. Readings, both quantity and pressure of the wet test meter, must be taken and recorded while it is in motion, unless a more elaborate system is set up. In the case of a rate meter, the interval of time that the air is leaving the spirometer must be accurately timed. The bell should then be allowed to come to equilibrium before making displacement readings. A sufficient number of different flow rates are taken to establish the shape or slope of the calibration curve with the procedure being repeated three or more times for each point. For an even more accurate calibration the setup should be reversed so that air is supplied to the spirometer. In this way any unbalance due to pressure differences would be cancelled.

A permanent record should be made of a sketch of the setup, data, conditions, equipment, results, and personnel associated with the calibration. All readings (volume, temperatures, pressures, displacements, etc.) should be legibly recorded, including trial runs or known faulty data, with appropriate comments. The identifications of equipment, connections and conditions should be so complete that the exact setup with the same equipment and connections could be reproduced by another person solely by use of the records.

After all of the data have been recorded, the calculations such as correction for variations in temperature, pressure and water vapor are made using the gas laws equation:

$$Vs = V_1 \times \frac{P_1}{760} \times \frac{273}{T_1}$$

where Vs = volume at standard conditions (760 mm & 0°C)
V_1 = volume measured at conditions P_1 and T_1
T_1 = temperature of V_1 in °A
P_1 = pressure of V_1 in mm Hg

In most cases the water vapor portion of the measurement is disregarded and the temperature of the gas is adjusted to 22° - 23°C rather than 0°C. The manipulation of the instruments, data reading and recording, calculations and resulting factors or curves should be done with extreme care. Should a calibration disagree with previous calibrations or the suppliers' calibration, the entire procedure should be repeated, and examined carefully to assure its validity. Upon completion of any calibration the instrument should be tagged or marked in a semi-permanent manner to

indicate the calibration factor, where appropriate, date and who performed the calibration.

Reciprocal Calibration by Balanced Flow System

In many commercial instruments it is impractical to remove the flow indicating device for calibration. This may be because of physical limitations, characteristics of the pump, unknown resistance in the system[19] or other limiting factors. In such situations it may be necessary to set up a reciprocal calibration procedure, that is, where a controlled flow of air or gas is compared first with the instrument flow, then with a calibration source. Often a further complication is introduced by the static pressure characteristics of the air mover in the instrument[20]. In such instances supplemental pressure or vacuum must be applied to the system to offset the resistance of the calibrating device. An example of such a system is illustrated in Figure 8.

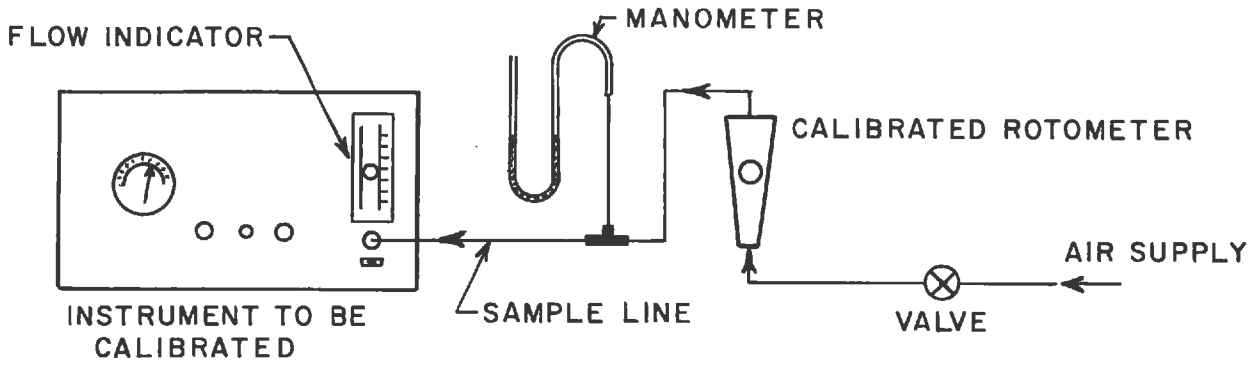


Figure 8. - Setup for Balanced Flow Calibration.

The instrument is connected to a calibrated rotameter and source of compressed air. Between the rotameter and the instrument an open end manometer is installed. The connections, as in any other calibration system, should be as short and resistance free as possible.

In the calibration procedure the flow through the instrument and rotameter is adjusted by means of a valve or restriction at the pump until the manometer indicates "0" pressure difference to the atmosphere. When this condition is achieved the instrument and rotameter are both operating at atmospheric pressure. The indicated and calibrated rates of flow are then recorded and the procedure repeated for other rates of flow.

Dilution Calibration

Normally gas dilution techniques are employed for instrument response calibrations; however, several procedures[20,21 & 22] have been developed whereby sampling rates of flow could be determined. The principle is essentially the same except that different unknowns are involved. In air flow calibration a known concentration of the gas (i.e., carbon dioxide) is contained in a vessel. Uncontaminated air is introduced and mixed thoroughly in the chamber to replace that removed by the instrument to be calibrated. The resulting depletion of the agent in the vessel follows the theoretical dilution formula:

$$C_t = C_o e^{bt}$$

where C_t = concentration of agent in vessel at time, t
C_o = initial concentration at t = 0
e = base of natural logarithms
b = air changes in the vessel per unit time
t = time

The concentration of the gas in the vessel is determined periodically by an independent method. A straight line

plot should result from plotting concentration of agent against elapsed time on semi-log paper. The slope of the line indicates the air changes per minute (b) which can be converted to the rate (Q) of air withdrawn by the instrument from the following relationship: $Q = bV$; where V = volume of the vessel.

This technique offers the advantage that virtually no resistance or obstruction is offered to the air flow through the instrument; however, it is limited by the accuracy of determining the concentration of the agents in the air mixture.

Samplers Without Flow Indicators

A few sampling units are not equipped with flow indicators, or at the most they may have vacuum or pressure gauges. In most cases these devices are considered to have fixed rates of flow. An example of this is the "Denver" or "Roberts" hand cranked pump which is calibrated by turning the crank at a fixed rate (counting revolutions) and collecting and measuring the pumped air volumetrically. The calibration is then denoted as volume per revolution. This volume will vary somewhat (± 15%), depending on the condition of the pump.

Where vacuum and pressure gauges are involved these units should be checked against an appropriate manometer connected into the system at the same location.

Summary

Because the accuracy of all sampling instruments is dependent on the precision of measurement of the air volume involved, extreme care should be exercised in performing all calibration procedures. The following comments summarize the philosophy of air flow calibration:

1. Protect and handle standard devices with "loving care."
2. All standards should be checked periodically to determine their operating condition.
3. Calibrations should be performed whenever a device has been changed, repaired, received from a manufacturer, subjected to use, mishandled or damaged, and at any time when there is a question as to its accuracy.
4. Understand the operation of an instrument before attempting to calibrate it and use a procedure or setup which will not change the characteristics of the instrument or standard within the operating range required.
5. When in doubt about procedures or data, assure their validity before proceeding to the next operation.
6. All connections should be as short and free of constrictions and resistance as possible.
7. Extreme care should be exercised in reading scales, timing, adjusting, and leveling, and in all other operations involved.
8. Allow sufficient time for equilibrium to be established, inertia to be overcome, and conditions to stabilize.
9. Enough points or different rates of flow should be obtained on a calibration curve to give confidence in the plot obtained. Each point should be made up of more than one reading whenever practical.
10. A complete permanent record of all procedures, data and results should be maintained. This should include trial runs, known faulty data with appropriate comments, instrument identification, connection sizes, barometric pressure, temperature, etc.
11. When a calibration differs from

previous records, the cause of change should be determined before accepting the new data or repeating the procedure.

12. Calibration curves and factors should be properly identified as to conditions of calibration, device calibrated and what it was calibrated against, units involved, range and precision of calibration, date and who performed the actual procedure. Often it is convenient to indicate where the original data is filed and to attach a tag to the instrument indicating the above information.

References

1. Hama, George: Calibration of Air Flow Measuring Instruments. Encyclopedia of Instrumentation for Industrial Hygiene. University of Michigan, Institute of Industrial Health, Ann Arbor, Michigan, pp 703-708, 1956.

2. Beckett, E. G.: A Laboratory Gas Meter. J. Soc. Chem. Ind., 36:52 (1917).

3. Kuznetz, Howard L.: Air Flow Calibration of Direct Reading Colorimetric Gas Detecting Devices. Am. Industr. Hyg. Assoc. J., 21: 340-341 (August 1960).

4. Gage, J. C.: The Calibration of Hand Pumps for Air Sampling. Imperial Chem. Ind., Ltd., Analyst. 84: 519-520 (August 1959).

5. Bloomfield, J. J. and J. M. Dalla Valle. The Determination and Control of Industrial Dust. Public Health Bulletin, No. 217, pp 140-144, 1958.

6. American Society of Mechanical Engineers, Power Test Codes Commission, Flow Measurement by Means of Thin Plate Orifices, Flow Nozzles, and Venturi Tubes. A.S.M.E. Power Test Codes. Supplement on Instruments and Apparatus, Part 5, Measurement of Quantity of Materials, Chap. 4, 2nd Ed., p 91, New York, February 1959.

7. Bruum, J. R.: Capillary Flow with Variable Orifice. Ind. Eng. Chem., Anal. Ed., 11: 655 (1939).

8. National Association of Fan Manufacturers, Standard Code for Testing Centrifugal and Axial Fans. Bulletin 110, Detroit, Michigan, 1950.

9. Brandt, Allen D.: Industrial Health Engineering. John Wiley & Sons, New York, pp 192-193, 1947.

10. Madison, Richard D., Ed., Fan Engineering. Buffalo Forge Company, 5th Ed., New York, pp 125-131, 1948.

11. _____: Cavitation May Cause Erroneous Calibration of Air Samplers. HC Inf. Exchange Bulletin, 1: 1, (September 1961).

12. Polentz, L. M.: Theory and Operation of Rotameters. Instruments and Control Systems. 34: 1048 (June 1961).

13. Hatch, T., Warren, H., and Drinker, P.: Modified Form of the Greenburg-Smith Impinger for Field Use, With a Study of Its Operating Characteristics. J. Ind. Hyg., 14: 301-311 (1932).

14. Avera, C. B., Jr.: Simple Flow Regulator for Extremely Low Gas Flows. Rev. of Sci. Instr., 32: 985-986 (August 1961).

15. American Society of Mechanical Engineers. Fluid Meters, Their Theory and Application. A.S.M.E. Research Publication, 4th Ed., New York, 1937.

16. Perry, John H., Ed., Chemical Engineers Handbook. 3rd Ed., McGraw-Hill, New York, 1950.

17. Brief, R. S.: Chart Speeds Orifice Sizing. Air Cond. Heat. & Vent., 57: 57-60 (August 1960).

18. Saltzman, Bernard E.: Preparation and Analysis of Calibrated Low Concentrations of Sixteen Toxic Gases. Anal. Chem., 33: 1100-12 (1961).

19. Tebbens, Bernard D., and Donald M. Keagy: Flow Calibration of High Volume Samplers. Am. Industr. Hyg. Assoc. Quar., 17:327-329 (September 1956).

20. Morley, Morgan J., and Bernard D. Tebbens: The Electrostatic Precipitator Dilution Method of Flow Measurement. Am. Industr. Hyg. Assoc. Quar., 14: 303-306 (December 1953).

21. Setterlind, Alfred N.: Preparation of Known Concentrations of Gases to Vapors in Air. Am. Industr. Hyg. Assoc. Quar., 14: 113-120 (June 1953).

22. Brief, Richard S., and Franklin W. Church: Multi-Operational Chamber for Calibration Purposes. Am. Industr. Hyg. Assoc. J., 21: 239-244 (June 1960).

Additional References

1. Binder, R. C.: Fluid Mechanics. 4th Ed., Prentice Hall Publishing Company, New York, 1962.

2. Jacobs, Morris B.: The Analytical Chemistry of Industrial Poisons Hazards and Solvents. 2nd Ed., Inter-Science Publishers, Inc., New York, 1949.

3. Silverman, Leslie: Industrial Air Sampling and Analysis. Industrial Hygiene Foundation, Chemistry and Toxicology Series, Bulletin No. 1, Pittsburgh, Pennsylvania, 1947.

4. Yaffe, Byers and Hosey, Editors, Encyclopedia of Instrumentation for Industrial Hygiene. University of Michigan, Ann Arbor, Michigan, 1956.

5. Air Sampling Instruments Committee, ACGIH, Air Sampling Instruments for Evaluating Atmospheric Contaminants. 2nd Ed., Amer. Conf. Gov. Ind. Hyg., Cincinnati, Ohio, 1962.

SAMPLING FOR DUST

Introduction

Fifty years ago, in many dusty industries, workmen were exposed to massive concentrations of air-borne mineral dust. It was not uncommon to find pneumoconiosis in thirty to eighty percent of the men examined in studies in the dusty trades,[1] and when the dust was both high in concentration and contained a high proportion of free silica severe disease might develop with a very few years of exposure. During the 25 or 30 years preceding World War II a number of studies of silicosis prevalence were conducted, methods were developed for measuring levels of exposure, and substantial efforts toward dust control were made in many industries.[2-12] Changes in prevalence of pneumoconioses, with the trend toward reduced rates, which have been seen in successive studies during the past five decades are probably the result of a variety of factors including differences in the definitions of populations studied, improvement in technical quality of chest X-rays, changes in diagnostic practices, and legislative or regulatory changes which effect eligibility for compensation. The major reason for lower prevalence of disease, however, is undoubtedly improvement in working conditions, specifically reduction in the quantity of dust to which workmen are exposed.

The Standard Method

The standard method for impinger sampling and counting was presented at an early meeting of the National Conference of Governmental Industrial Hygienists[13] and is reproduced in Section B-6 of this Syllabus. Because the method is empirical, it is essential that it be followed closely by all its users if data obtained by one person are to be comparable to that obtained by another. Even then, variables in sampling and counting within the method, as it is described, may permit two investigators, both applying the method skillfully and conscientiously, to obtain somewhat different results when sampling the same environment. Some of these variables are mentioned in this Section and in Section B-6.

Equipment and Materials

Impingers. The standard method for sampling mineral dusts provides for use of either the Standard or "Greenburg-Smith" impingers or midget impingers. The standard impinger operates at a nominal sample flow rate of one cubic foot of air per minute (cfm) when a suction of 3 inches of mercury is applied to the suction side of the device. The midget impinger has a nominal sampling rate of 0.1 cfm when 12 inches of water suction is applied. The velocity of the dust-laden air through the impinger nozzle of the standard impinger, when operated at 1 cfm, is about 100 meters per second; that through the nozzle of the midget impinger operating at 0.1 cfm is about 65 meters per second. Some impingers are made entirely of glass with a ground glass joint between the flask and the impinger tube-suction arm assembly. Others consist of a glass flask with a rubber stopper through which a glass impinger tube and suction arm are inserted. Upon occasion difficulty may be experienced in obtaining a low blank count in sampling medium which has been carried in flasks with black rubber stoppers. Well scrubbed neoprene stoppers substituted for rubber stoppers seem to eliminate this difficulty.

Pumps. A 110-220 volt electrically driven Willson pump is commonly used as a suction source for sampling with standard impingers. Any suction source capable of drawing 1 cfm against 3 inches of mercury may be used, however. Mine Safety Appliances Company hand-cranked piston pumps, designed specifically for

Prepared by: Robert L. Harris, Jr.

the purpose, are probably the most common suction source for sampling with midget impingers. Line operated or battery powered electric pumps of appropriate capacity also find favor as suction sources for these samplers.

Calibration. For precise work, impingers must be calibrated for air flow. Impingers are best calibrated at a standard suction value, i.e., the amount of air which flows through each standard impinger at 3 inches of mercury suction or through each midget impinger at 12 inches of water is measured. This value is recorded for each impinger and is its air-flow calibration value. The tubing to be used between the impinger and its pump in sampling, or another piece of the same length and bore, should be included in the calibration setup if the usual means of gauging air flow with a suction gauge on the pump is used.

Collecting Medium. In practice, water, alcohol, or a mixture of the two, may be used as the sampling medium. Some kinds of dust seem to be poorly wetted, and possibly therefore poorly captured in water. Alcohol has advantage over water as a sampling medium in temperatures near the freezing point of water. The use of alcohols as sampling media may have other effects upon counting worthy of consideration. These will be discussed in Section B-6.

Cleaning Equipment. Before samples are taken, impingers must be thoroughly cleaned. A final step in cleaning should be a thorough rinsing with the sampling medium. Sampling medium is then placed in the impinger flasks and the inlet tubes and suction arms are closed with clean stoppers or caps. Ordinarily, about 75 milliliters of sampling medium are used in standard impingers and 10 ml are used in the midget impingers. It may be worth-while to tape the joint between the flask and its impinger assembly stopper to prevent accumulation in the joint of dust which may contaminate the sample when the impinger tube is removed from the flask at the time of counting.

Field Technique in Sampling

Sampling may have different purposes which merit different techniques. These generally fall into two major categories:

(1) sampling for industrial health engineering surveillance, testing, or control, and
(2) sampling for health research or epidemiologic purposes.

A sampling program for engineering purposes should be designed to yield the specific information desired. For example, one might need only single samples before and after a change in ventilation to determine whether the change has had the desired effect. On the other hand, many samples over an extended period of operation may be necessary to make a time weighted assessment of a variable exposure for hygienic purposes.

Among the variables which should be considered in the sampling procedure are variations in the rate of generation of respirable dust, and the direction of air motion with respect to the dust source, the person exposed, and the sampling location. The choice of sampling location is largely dependent upon judgment regarding these factors. In the assessment of dust exposures which fluctuate with time the number, duration, and time scheduling of individual samples is highly important. All too frequently the results of too few samples of too short duration form the basis for judging the significance of exposure. Tomlinson,[14] Ashford,[15] and Hodkinson,[16] among others, have dealt with various aspects of this problem. Roach[17] has proposed use of sampling times no more than 1/24 the biologic half time of a substance when results of greatest hygienic significance are being sought. For mineral dusts with long half-times he suggests that samples be a full working shift in duration.

Single impinger samples of such duration are not practical and more work is needed in the development of sampling strategy to accommodate this facet of the problem.

Another type of variation with its source in sampling technique, and one through which criticism has been leveled at the impinger method, is disagglomeration or fracturing of compound particles. Compound particles too large to penetrate the defenses of the upper pulmonary system and reach the lungs may disagglomerate in the liquid medium of an impinger and appear in the count as a larger number of respirable sized particles. An environment containing a substantial proportion of nonrespirable agglomerates might thus be judged by impinger sampling to be much more hazardous than another with the same number of respirable particles but with few agglomerates. Pre-samplers to prevent entrance of nonrespirable particles into the impinger,[18,19] would seem an appropriate addition to the impinger method in some instances. Although limited experience with such devices to date by the Division of Occupational Health has shown very little difference between counts of companion impinger samples with and without pre-samplers, the possibility of significant differences occurring in other instances cannot be discounted. The impinger method should not be criticized too severely at this time for disagglomeration of compound particles of such size that they will penetrate to the lungs. Such disagglomeration may well occur in the lungs and the count related datum for judging the significance of exposures is applied in the Threshold Limit Value. In any sampling situation it is wise to use a guard over the inlet of the impinger to prevent large agglomerates, which could never be inhaled, from falling into the sample.

The importance of adequate field notes must be emphasized. Field notes describing the time, duration, location, operations underway, and all other factors pertinent to the sample and the exposure or condition which it is intended to define, should be made while the sample taker is at the sampling site. Forms with labeled spaces for essential data help to avoid the common failure to record needed information.

Respirable Dust Sampling

Sampling for atmospheric particulates presents problems quite different from those encountered in sampling for gases and vapors. Gases and vapors, when inhaled can penetrate all portions of the pulmonary system and follow the laws of distribution by diffusion and absorption. The size, shape, and density of particles, on the other hand, strongly influence the site at which they will be deposited in the pulmonary system. Since the effect which may be produced by particles trapped in the nasal passages or which are deposited in the cilliated portions of the pulmonary system may be quite different from that of particles which penetrate and are deposited deeper in the pulmonary system, size selected sampling is generating increasing interest among industrial hygienists. At the Pneumoconiosis Conference held in Johannesburg, South Africa, in 1959, in which experts in pneumoconiosis from all over the world participated, it was concluded that in the light of present available evidence the best single parameter to measure in the case of coal dust is the mass concentration of respirable dust, and in the case of quartz dust is the surface area of the respirable dust.[20] The respirable fraction of a dust cloud was then defined as all particles less than 1 micron, half those of 5 microns, and none of those 7 microns or more in equivalent diameter. The equivalent diameter of a particle is that of a unit density sphere having the same falling velocity in air as the particle. Sampling devices designed to reject particles too large to penetrate to the deeper portions of the lung, or to collect these larger particles in a pre-sampler

with the respirable portion collected separately for weighing or analysis have been developed. The Conicycle[21] developed by the Medical Research Council of Great Britain, is representative of the rejection type sampler while the Hexhlet[22] gravimetric dust sampler developed by the same council and the two stage cyclone-filter type samplers as developed by the New York Operations Office of the Atomic Energy Commission, the Los Alamos Scientific Laboratory,[23] and in Germany[24] are examples of pre-sampler types of devices. Although the results of sampling for pneumoconiosis producing mineral dusts, either for gravimetric determinations or for determination of composition, cannot be interpreted by use of the current Threshold Limit Values, such sampling may represent a sensitive method for predicting the disease producing potential of mineral dust exposures and is worthy of further study. The accumulation of data using such devices, with design and operation based on an agreed lung deposition criterion, sampling in parallel with our standard dust sampling devices, will permit eventual judgment regarding their usefulness.

References

1. _____: National Silicosis Conference: Final Report of the Committee on the Prevention of Silicosis Through Medical Control. U. S. Dept. of Labor Bull. No. 21, Part I., Washington, 1938.

2. Higgins, E., Lanza, A. J., Laney, F. B., and Rice, G. S.: Siliceous Dust in Relation to Pulmonary Disease Among Miners in the Joplin District, Missouri. Bureau of Mines Bull. 132, 116 pp., 1917.

3. Harrington, Daniel and Lanza, A. J.: Miners' Consumption in the Mines of Butte, Montana. Bureau of Mines Tech. Paper 260, 19 pp., 1921.

4. Hoffman, F. L.: The Problem of Dust Phthisis in the Granite-stone Industry. U. S. Dept. of Labor, Bureau of Labor Statistics, Bull. 293, Washington, 1922.

5. Sayers, R. R., Meriwether, F. V., Lanza, A. J., and Adams, W. W.: Silicosis and Tuberculosis Among Miners of the Tri-State District of Oklahoma, Kansas and Missouri. I. For the Year Ended June 30, 1928. Bureau of Mines Tech. Paper 545, 30 pp., 1933.

6. Meriwether, F. V., Sayers, R. R., and Lanza, A. J.: Silicosis and Tuberculosis Among Miners of the Tri-State District of Oklahoma, Kansas and Missouri. II. For the Year Ended June 30, 1929. Bureau of Mines Tech. Paper 552, 28 pp., 1933.

7. Sayers, R. R., et al.: Anthracosilicosis Among Hard Coal Miners. Public Health Bull. No. 221, U. S. Public Health Service, 1935.

8. Flinn, R. H., et al.: Silicosis and Lead Poisoning Among Pottery Workers. Public Health Bull. No. 244, U. S. Public Health Service, 1939.

9. Flinn, R. H., et al.: Soft Coal Miners' Health and Working Environment. Public Health Bull. No. 270, U. S. Public Health Service, 1941.

10. Russell, A. E.: Health of Workers in Dusty Trades. Restudy of Granite Workers. Public Health Bull. No. 269, U. S. Public Health Service, 1941.

11. Ellis, P.M., Smith, M.T., Bonebrake, H.E., and Hunter, L. B.: Silicosis. General Considerations and Survey in the Coeur D'Alene Mining District of Idaho, North West Medicine, Vol. 41, 406 pp., December 1942.

12. Dreessen, W.C., Page, R. T., Hough, J. W., Trasko, V. M., Jones, J. L., and Franks, R. W.: Health and Working Environment of Non-ferrous Metal Mine Workers. Public Health Bull. No. 277, Washington, 1942.

13. _____: Transactions of the Fifth Annual Meeting of the National Conference of Governmental Industrial Hygienists. Subcommittee on Standard Methods, Washington, D. C., 1942.

14. Tomlinson, R. C.: A Simple Sequential Procedure to Test Whether Average Conditions Achieve a Certain Standard. Applied Statistics, VI: No. 3, 198 (1957).

15. Ashford, J. R.: The Design of a Long-Term Sampling Programme to Measure the Hazard Associated with an Industrial Environment. Jour. Royal Statis. Soc., 121: Part 3, 333 (1958).

16. Hodkinson, J. R.: Reproducibility and Correlation of Long-Period and Short-Period Thermal Precipitator Samples of Air-borne Dust in Mines. Ann. Occup. Hyg., 2:235 (1960).

17. Roach, S.A.: The Importance of Body Burden in Establishing Hygienic Standards. Presented at 2nd International Symposium on Permissible Limits for Air, Paris, April 1-6, 1963.

18. May, K. R. and Druett, H. A.: The Pre-Impinger: A Selective Aerosol Sampler, Brit. Jour. of Indust. Med., 10:142 (1953).

19. Hatch, T. F.: Developments in the Sampling of Air-borne Dust, AMA Arch. of Indust. Health, 11:212 (1955).

20. _____: Proceedings of the Pneumoconiosis Conference, Johannesburg, 1959. J & A Churchill, Ltd., London (Little, Brown & Co., N.Y.), p. 620, 1960.

21. Wolff, H. S., and Roach, S. A.: The Conicycle Selective Sampling System, Inhaled Particles and Vapors. C. N. Davies, Editor, Pergamon Press, London, p. 460, 1961.

22. Wright, B.M.: A Size Selecting Sampler for Air-borne Dust, Brit. Jour. of Industr. Med., 11: 284 (1954).

23. Hyatt, E.C., et al.: A Study of Two-Stage Air Samplers Designed to Simulate the Upper and Lower Respiratory Tract. Proceedings of the 13th International Congress on Occupational Health, New York, p. 486, 1960.

24. Breuer, H.: A Filter Device for Selective Sampling in Coal Mines of Respirable Fine Dusts, Inhaled Particles and Vapors. C. N. Davies, Editor, Pergamon Press, London, p. 453, 1961.

OPTICAL AND ELECTRON MICROSCOPY

The information in this Section is presented in two parts: I - Optical Microscopy and II - Electron microscopy. Since optical microscopes are used more frequently for evaluating dust exposures than the electron microscope, considerably more detail is included on the former subject.

I - OPTICAL MICROSCOPY

Introduction

The light mocroscope has become the most basic and important single tool in the evaluation of air-borne particulates. Properly used it is capable of a high degree of sophistication and precision; yet it remains quite simple to operate. But this fundamental simplicity has led to two important misconceptions, which in turn have led to widespread misuse or distrust of the microscope.

The first misconception is that the microscope is an elementary tool on the technological level of the ordinary screwdriver, and that it may be operated fruitfully without concern for the principles governing the performance. This attitude assumes that "optical theory is alright, but it doesn't work." Persons approaching microscopy within this frame of reference may expect considerable difficulty in operating their instruments and will possibly obtain great inconsistencies in their results.

The second misconception is exemplified in the attitude that microscopy is more art than science, and may be adequately learned only by long and painful experience. This fear leads to avoidance, and causes many investigators to employ lengthy and indirect methods when the rapid and direct method of the microscope lies easily at hand.

In the occupational environment, the microscope is the most important tool for determining the workers' exposure to mineral dusts. Because a knowledge of the dust concentration is fundamental to protecting the health of the worker, it is imperative that industrial hygienists have a sufficient understanding of the workings of the microscope to assess accurately the samples submitted for analysis. And from a selfish standpoint, an increased knowledge of microscopy will make the microscope a far more comfortable and easy instrument to use. To these ends the following discussion is directed.

Review of Elementary Optics

In a review of the elements of optics, certain characteristics of the nature of visible light must be kept in mind:

1. It is an electromagnetic radiation, and therefore is a wave phenomenon, being characterized by wavelength, frequency, and velocity.

2. In a homogenous medium, light will travel in a straight line.

When light, traveling in a vacuum at the velocity of 3×10^{18}A per second with a given wavelength and frequency, enters another medium having a greater density the velocity and the wavelength will be reduced, but the frequency will remain the same.

The index of refraction (η) of a transparent medium is defined by the following relationship:

$$(1)\ \eta = \frac{c}{c_m}$$

where c and c_m represent the velocity

Prepared by: George H. Edwards and
David A. Fraser, Sc.D.

of light in a vacuum and in the medium, respectively. Light travels in air nearly as fast as in a vacuum, so for practical purposes the index of refraction of air may be taken as 1.0 (actual value is approximately 1.0003). As density increases, so, in general, does the index of refraction.

When a ray of light passes through an interface between a medium of low density (air or vacuum) to one of higher density (glass, water) at an angle greater than 0^{o} to the "normal" (i.e., perpendicular) it will be deviated from its original direction by an amount equal to the difference in its velocity in the first and second media.

This difference in direction of travel is a function of the indices of refraction of the two media. The relationship can be expressed as:

$$n_i \, \mathrm{Sin}_i = n_r \, \mathrm{Sin}_r$$

where n_i and n_r represent the indices of refraction in which the incident and refracted rays travel. The angles are measured from the rays to a normal to the interface (Figure 1).

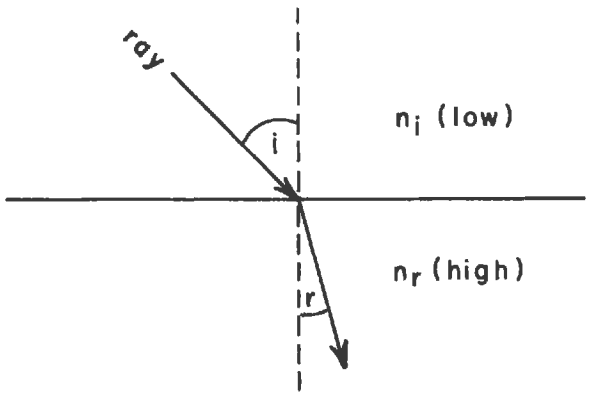


Figure 1. - Refraction of a Ray Passing from a Medium of Low Index to One of High Index.

Knowledge of any three of these values permits the calculation of the fourth. This method is used to determine the index of refraction of unknown materials.

It is important to note that this ray path is reversible. That is, when the ray travels from a medium of higher density to one of lower density, the ray path is bent away from the normal. There is, however, a limiting factor for the angle of incidence when a ray passes from a medium of higher index to one of lower index. This is known as the critical angle, and is defined as the angle of incidence for which the angle of refraction is 90^{o}. Rays having angles of incidence greater than the critical angle will be totally reflected back into the medium of higher index of refraction.

Thus, if a ray of light is passed through a prism of glass (n = 1.515) in air the direction of the ray may be changed as shown in Figure 2.

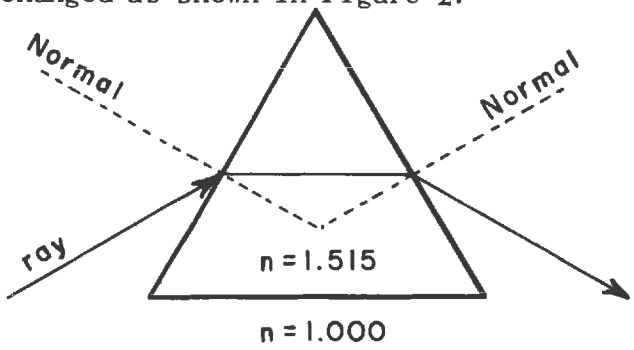


Figure 2. - Refraction of a Ray by a Glass Prism.

If another prism is added to the system a crude means is provided to bring rays from a source back together at a point, or focus (Figure 3).

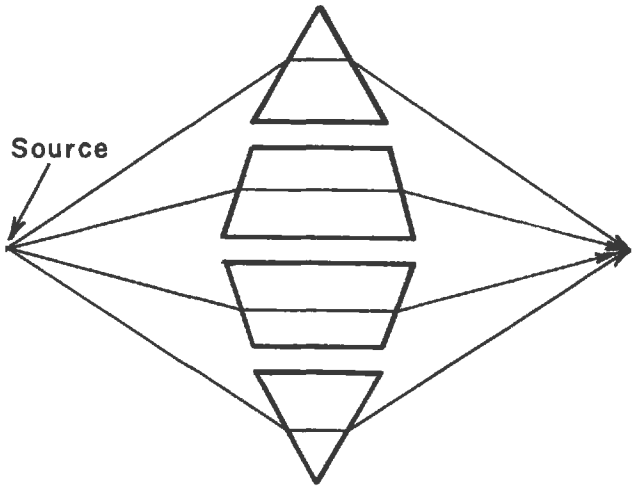


Figure 3. - Focusing of Rays by a Series of Prisms.

It now only remains to smooth the surfaces of the prism into curves (of spherical section) to create the cross-section of a simple biconvex lens, which is capable of creating an image (Figure 4).

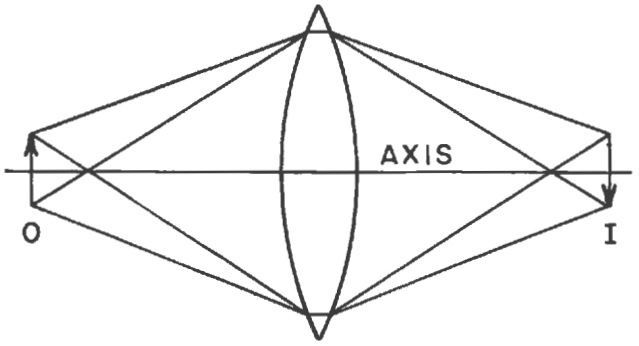


Figure 4. - Formation of an Image by a Lens.

Magnification of the image, I, over the size of the object, O, occurs when the distance from the object to the center of the lens is less than the distance from the center of the lens to the image, and is expressed:

$$(2) \quad M = \frac{d_o}{d_i}$$

where d_o and d_i represent the object distance and the image distance. (It should be noted that this is an approximate expression, ideally applying only to thin lenses. The precise expression for thick lenses can be found in texts on microscopy).

The convex lenses discussed above converge light to a focus. Concave lenses cause the rays to diverge and act as negative, or demagnifying elements.

In Figure 5, rays from the object appear to an observer on the right of the lens, to come from the small virtual image. (This image is called virtual because it is only apparent. It cannot be projected upon a screen as can the real image produced by a converging lens).

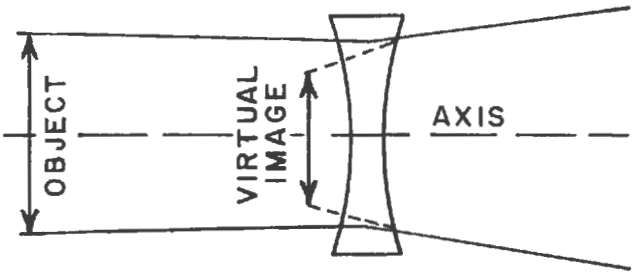


Figure 5. - Virtual Image Formed by Concave Lens.

Many transparent materials exhibit one index of refraction for red light and a somewhat higher index for violet light, at the other end of the visible spectrum. This difference is called dispersion. The net effect of this phenomenon is to spread a beam of polychromatic light into a display of its spectral components.

Defects of Lenses

There are certain characteristics of simple lenses which prohibit them from forming perfect images, no matter how perfect the structure of the lens.

Chromatic aberration. This is due to the dispersion of the glass in the lens, and arises from the fact that, since the glass has a lower index for red light than for violet, it will be more strongly converging for violet than for red. Therefore the violet image will be in focus closer to the lens than the red image (Figure 6). The image formed will not be sharp, but will be surrounded by fringes of color.

This aberration is corrected in compound lenses by inserting a strongly dispersive, but weakly negative (diverging) lens of a different type of glass behind the strongly positive (converging) front lens. This in effect cancels the dispersive action of the front lens, but counteracts only a small part of the magnification.

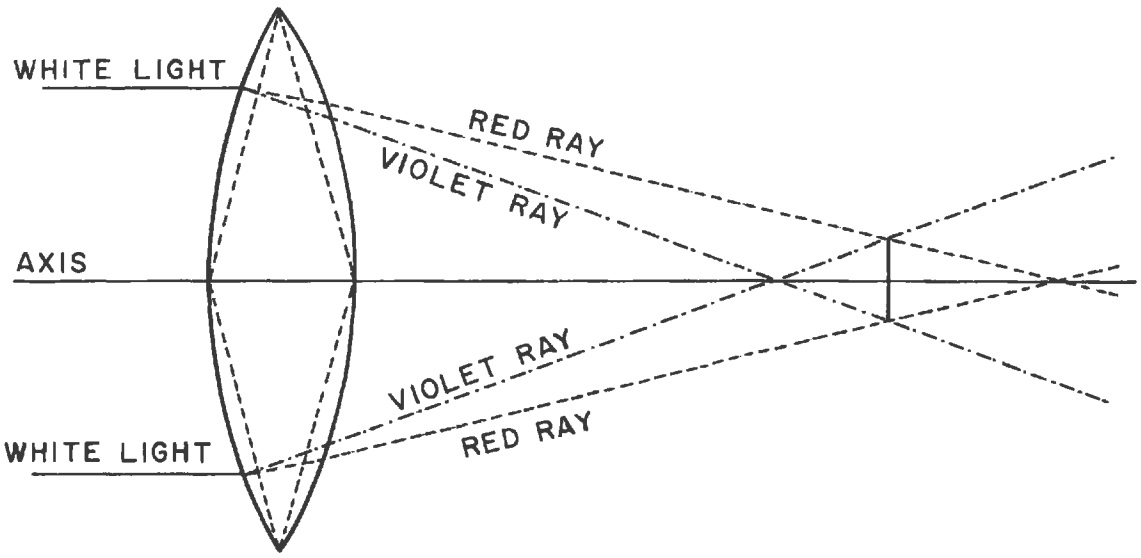


Figure 6. - Chromatic Aberration.

Spherical aberration arises from the fact that outer portions of a spherical lens have more converging power than the central area. Therefore, rays near the periphery will converge closer to the optical center (Figure 7). Spherical

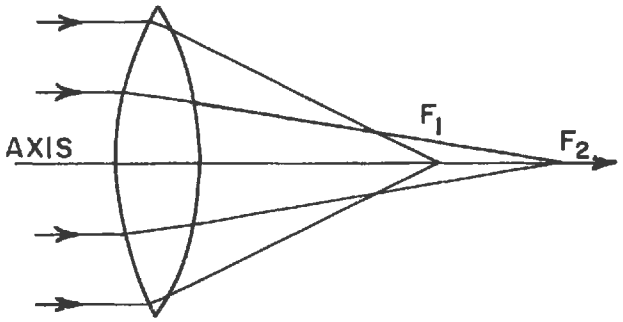


Figure 7. - Spherical Aberration.

aberration causes a lack of distinctness in focusing and a loss of contrast in the image. It may be reduced by inserting an aperture to limit the field of the lens to the central position, by combining several lens elements, or by varying the distance between lenses of a compound system.

Curvature of field. This occurs when the image plane is not flat. That is, the center of the image will be in focus farther from the eye lens than the periphery. This is more apparent in photomicrography than in direct observation, because the eye is able to compensate for differences in focus. Curvature of field should not be a problem in ordinary dust work, but helps explain why only the central portion of the microscope image should be used.

Diffraction and Resolution

It has been noted that light will continue in a straight line while traveling in a homogeneous medium. When a ray of light encounters an inhomogeneity such as a change in index of refraction, it is deviated by refraction. An inhomogeneity in the form of an opaque edge will cause the light to spread out to some extent into the region not directly exposed to the oncoming wave. This diversion of the rays into the area of the geometric shadow is diffraction.

In this manner, light passing through a small aperture (having a diameter of only a few wavelengths of light) located at the focus of a lens, will be diffracted and will not be imaged as a sharp edged disc. Due to interference of the waves passing through the small aperture, it will appear as a central

disc surrounded by rings of light (Figure 8). Thus there is a central maximum surrounded by minima and maxima which

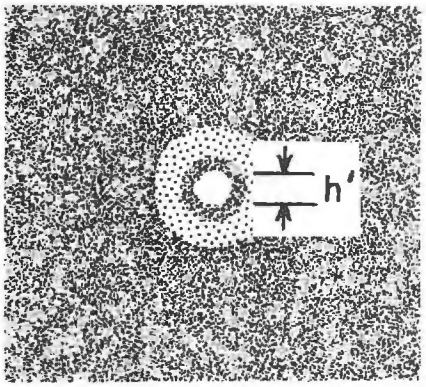


Figure 8. - The Airy Disc.

rapidly decrease in intensity away from the center. Airy showed that the distribution of energy in this image (now known as the Airy disc) is such that the radius of the first minimum is a measure of the ability of the optical system to resolve detail in the object.

The radius (h) of this disc for any lens may be determined by the following relationship:

$$(3) \quad h = \frac{.61\lambda}{NA}$$

where λ is the wavelength of light being used and NA is the numerical aperture of the optical system. This distance h is an expression of the resolving power of the lens.

Numerical aperture is a means of describing the light-gathering power of the lens. It is expressed as half the included angle from the object to the lens times the index of refraction of the medium between the object and the lens:

$$(4) \quad NA = n \sin \alpha$$

Thus to improve the resolution of an optical system (that is to reduce h), one or both of two things can be done: Light of a shorter wavelength can be used; the index of refraction of the medium between the lens and the object can be increased.

The light used is generally polychromatic, but it is advisable to use a "Daylite" or Corning blue filter to remove most of the long red wavelengths. With a special light source and quartz optics it is possible to use ultraviolet illumination having very short wavelength and thus reduce h.

The index of refraction of the intervening medium may be increased by immersing the front of the lens in a suitable material. Lenses have been computed for immersion in water for the study of living materials but the homogeneous oil immersion is by far the most common type. The oil immersion objectives use an oil of index 1.515, approximately that of the glass of the lens and the coverslip.

The greatest numerical aperture which can be achieved using a dry lens must obviously be less than 1.0, since the angle α cannot exceed 90^{o}. In practice, the highest NA ever manufactured is 0.90. By using an extremely wide angle and homogeneous immersion, lenses are manufactured with NA's as high as 1.40.

Using light near the middle of the visible spectrum at about 5500A (yellow-green), equation (3) shows that the common 10X, 16mm focal length objective lens has a resolving power of about 1.3 μ. The resolving power of an oil immersion objective of NA 1.30 or 1.40 under the same conditions of illumination is about 0.2 μ.

It should be noted at this point that the terms "resolving power" and "limit of visibility" are not synonymous. Although structures with spacings smaller than h, as determined by the resolving power equation cannot be resolved, it is possible to detect the presence of diffraction discs of particles having a diameter smaller than this size. The diameter of the diffraction disc is fixed by the optical system. For particles smaller than this size the intensity of the disc is dependent on the particle diameter.

Hopkins (1951) suggested that the minimum particle diameter (d) which will produce a diffraction disc of sufficient intensity for reasonably sure recognition "as a mere darker spot, not significant as to relative size" may be given by:

$$(5) \quad d = \frac{0.21\lambda}{NA}$$

Compare equations (3) and (5). As a general rule of thumb, useful magnification of any image extends up to about 1000 times the NA of the system. Magnification greater than this is referred to as "empty magnification" and results in mere enlargement of the indistinctness of the image.

Depth of Focus

Another functional limitation of the microscope optical system is depth of focus. This is the distance along the optic axis between the upper and lower limits of sharpness. Structures or particles above or below these limits will either not be imaged at all, or be seen as indistinct, out of focus images. With high power lenses, the depth of focus is so small that particles disappear rapidly as they exceed these limits.

Depth of focus (d_f) may be expressed absolutely as:

$$(6) \quad d_f = \frac{\lambda\sqrt{n^2 - (NA)^2}}{(NA)^2}$$

where again, λ is the wavelength of the illumination and n is the index of refraction of the surrounding medium. This relationship applies well to photomicrography, but it fails to take into account the ability of the human eye to accommodate, or to focus both on objects near the eye and at infinity. Most texts assume that the eye can accommodate from 250 mm to infinity. Then the displacement (d_f') of the object plane required to form an image at 250 mm instead of affinity is:

$$(7) \quad d_f' = \frac{250 \text{ mm}}{M^2}$$

This value may be called visual depth of focus, and is to be added to the absolute depth of focus d_f. Hence the total depth of focus (D_f) of an optical system for visual observation is:

$$(8) \quad D_f = \frac{\lambda\sqrt{n^2 - (NA)^2}}{(NA)^2} + \frac{250 \text{ mm}}{M^2}$$

Inserting the appropriate values in these relationships, the following depths of focus for three common lenses using 10X eyepieces are:

Objective	Total depth of focus (D_f)
.25 NA	34.0 μ
.65 NA	2.3 μ
1.30 NA	0.5 μ

Thus, as NA increases, depth of focus becomes extremely small.

All common lenses are designed (or "computed") to examine an object through a definite thickness of optically active material. That is, the correction for spherical aberration in a given lens depends on there being a specific thickness of glass or other material of similar refractive index between the object and the lens. This thickness is provided by the coverslip, which serves to protect the specimen. Because the lens is computed for a given coverslip thickness, the coverslip is an essential, active element of the optical system.

For the vast majority of objectives, this thickness is 0.17 to 0.18 mm, which corresponds to the standard # 1½ coverslip.

Deviation from this thickness will result in the objective being effectively over or under corrected for spherical aberration. The net effect of this improper correction will be a loss of contrast in the image. Since contrast is one of the greatest problems in dust microscopy, deviations in coverslip thickness should be avoided. It has been noted (Leitz Technical Information

Bulletin, Vol. I, No. 4) that for the 43X NA 0.65 objective, a deviation of 10 μ (0.010 mm) from the standard thickness will cause a noticeable deterioration of the image. However, the 10X, 0.25 NA objective is much less sensitive to such variations. For critical work, the thickness of the #1½ coverslip should be checked with a micrometer, and those coverslips which are more than 10 μ out of tolerance should be rejected.

Components of the Modern Compound Microscope

Substage Assembly

Three highly important and often neglected elements of the microscope optical system are located below the stage of the microscope. These are the illuminator, the mirror, and the condenser lens with its adjustments. Together these components serve to provide a carefully controlled cone of light to the specimen and the objective lens.

The various types of illuminators and their adjustments will be discussed later.

The mirror simply reflects the light from the illuminator upwards into the condenser lens. In microscopes having built-in illuminators, the mirror and illuminator are fixed in position, and require no adjustment.

Most microscopes not having built-in illuminators have adjustable mirrors with a plane side and a concave side. The concave mirror is intended to act as a low-power condensing lens for inexpensive low-power systems which lack any other condenser. The curved side of the mirror is not to be used in dust microscopy.

The condenser lens collimates and converges the light from the illuminator and focuses it on the specimen. The proper function of the condenser is vital for accurate results, and its adjustment must not be ignored.

The minimum acceptable type of condenser for dust work is the Abbe condenser, which consists of two lenses and an iris diaphragm. There is no correction for spherical or chromatic aberration, but this will introduce no problem at low powers. For high power work, a more refined type of condenser such as the achromatic or aplanatic condenser, should be used.

Most condensers have a working distance of about 1.1 to 1.2 mm. Since the thickness of the standard microscope slide is approximately 1 mm, it is obvious that the upper lens of the condenser must be nearly in contact with the bottom of the slide if the condenser is to be in focus on the specimen. The condenser assembly is carried in a dovetail slide and is focused by a rack and pinion gear.

When the condenser is at its proper setting the iris diaphragm acts as an aperture which controls the angular aperture and therefore the NA of the condenser. Closing the iris reduces the NA, the resolving power, the amount of light reaching the objective, and hence the intensity of the image. Thus it is very important to remember that the intensity of the image should never be controlled by the substage iris, but by reducing the intensity of the light source. Most inexpensive Abbe condensers have a maximum NA of 1.25 and cannot provide adequate illumination for objectives of higher NA.

Stage

The stage serves as a rigid support for the specimen. It must be oriented so that the optical axis of the lens system is exactly perpendicular to the stage. Older microscopes have stages fixed to the stand, and the objective is focused by moving the body tube. In more modern instruments the body tube and lenses are fixed, while focusing is achieved by moving the stage.

For nearly all dust microscopy some sort of mechanical stage is essential. A mechanical stage incorporates a clip or other means to hold the specimen

slide, and micrometric screws to drive the slide in two directions. Many types are available.

Objective Lens

The simplest of microscope objectives is a low power system of three lenses. Objectives increase in complexity with increasing magnification and degree of correction until the finest high power objectives are combinations of ten or more lenses. Nearly all the magnification is done by the highly curved front lens, the back lenses serving to compensate for the spherical and chromatic aberrations introduced by the extreme curvature of the front lens.

There are basically three grades of objective lenses - achromat, fluorite, and apochromat, plus various modifications of these for special applications.

The achromat. This is the simplest and least expensive of the three types of lenses. Because it is intended solely for visual observation, it is corrected spherically for the part of the spectrum to which the eye is most sensitive (around 5500A, in the yellow-green). Therefore, the sharpest image will be obtained using a filter of this wavelength.

Achromats are available in a wide range of powers and NA's from about 4X, NA 0.10 up to approximately 100X oil immersion with NA of 1.25 or greater. The high-dry, 40X to 60X, achromats with NA's from 0.65 to 0.85 are very common and are frequently used for dust sizing. The high-dry NA 0.85 lens can tolerate a coverslip variation of only 5μ before deterioration of the image takes place. Thus, a coverslip of the correct thickness must be used with these lenses.

Because of this effect of coverslip thickness, most good objectives of NA above 0.65 are fitted with correction collars. These change the spacing between certain lens elements and therefore the correction of the lens for spherical aberration. In this way, it is possible to compensate for variations in coverslip thickness. Compensation may also be made by changing the tube length of instruments having adjustable body tubes if it is possible to determine how much to change the length. The reader is cautioned that this is more trouble than using the correct coverslip.

The NA, magnification, and usually the focal length is engraved on the barrel of all objectives of most manufacturers.

Fluorite objectives. These are so named because one of the lens elements is made of the mineral fluorite. They are intermediate in cost and performance between the achromats and the apochromats. Fluorites are corrected spherically for two colors and chromatically for two. They are available only in the high dry and oil immersion objectives; the low power (10X) provides no better performance than the less costly achromats. Fluorite objectives are sometimes designated as "semi-apochromats."

Apochromats. The mineral fluorite is also used in these objectives but they are more highly corrected than fluorite objectives. Correction is provided for two colors spherically and three colors chromatically, but they contain a certain degree of chromatic difference of magnification. This means that while the images of both the red and blue ends of the spectrum will be focused in the same plane, they will be of slightly different size. For this reason, apochromats must always be used with compensating eyepieces which are so designed as to exactly counteract the chromatic difference of magnification. When so used, an apochromat will provide an image which is very clear and sharp, and nearly perfectly free from color fringes. Therefore, they are particularly useful for photomicrography.

Oil Immersion

To achieve the maximum resolution possible from a lens, the NA must be as large as possible. Referring to Equation 4, if the index of refraction of

the medium between the lens and the object is increased the NA is also increased. The immersion lens depends on this relationship, and nearly all immersion lenses are of the homogeneous oil immersion type. In this lens the immersion medium has the same index of refraction as the glass of the slide and front lenses of the objective and condenser. Ideally, a ray of light will not be deviated from its path between the condenser and the objective except by the specimen. This enables the objective to accept a large cone of light, and therefore resolve smaller objects. Immersion objectives should never be used dry, not only because they will fail to deliver their rated NA, but also because spherical aberration will not be corrected

It should be emphasized at this point that if an oil immersion objective is used the oil must be in contact with the top of the condenser lens and the bottom of the slide, as well as the top of the coverslip and the objective lens. If the oil does not touch both the condenser and the slide, the NA of the cone of illumination provided to the objective will be less than 1.0. The futility of using an objective of high NA but illuminating it with a narrow cone of light is self-evident.

The correct procedure for focusing immersion lenses is:

1. Determine that the coverslip thickness is not greater than the working distance of the lens. If the sample mounting material adds appreciably to the thickness, as in the case of membrane filters, the total thickness of mounting material plus #1½ coverslip may exceed the working distance of the lens. A thinner coverslip should be used to make the total thickness 0.17 or 0.18 mm.

2. Scan the sample with a low power lens to locate a suitable field. Roughly align the illumination system.

3. Rack up with the coarse adjustment and put the oil immersion lens in place. Most manufacturers identify the oil immersion lens by a black band around the barrel. Be sure it is not the high dry, because cleaning oil off the high dry lens is a difficult task.

4. Avoid forming air bubbles - put a small quantity of immersion oil on the lens and a similar amount on the coverslip.

5. Rack the fine adjustment to the bottom of its travel. Using great caution, rack down with the coarse adjustment until the two drops merge. Observe this from the side of the microscope. Never focus downwards while looking through the eyepieces. The mechanical advantage of the focusing system is very great, and only light finger pressure will grind the lens through the coverslip, sample, and perhaps the slide. This will almost certainly ruin the objective.

6. When the objective nearly touches the slide, look through the eyepieces and focus upwards with the fine focus until the image appears. Remember that the depth of focus of the oil immersion lens is very shallow, and the image will pass through focus quite rapidly. If the image is missed the first time, repeat steps 5 and 6 until it is found, never looking through the eyepieces while focusing downwards.

7. When finished, gently wipe the objective and condenser lenses with a piece of high quality lens tissue which has been moistened with a little xylol. Do not soak the lens in xylol, as this will dissolve the cement holding the lens elements in place. Perform this operation with delicacy as optical glass is soft and easily

scratched. Modern non-hardening immersion oils, because they do not dry, have a tendency to seep in between the elements of the objective. For this reason, oil should never be left on a lens for longer than needed - never more than three or four hours at a time.

The Body Tube

Separating the objective lenses and the eyepiece is the body tube, of which there are basically two types available - the monocular and the binocular.

The monocular body tubes are often provided with a means of adjusting the tube length. This adjustment will enable the skilled microscopist to correct for variations in coverslip thickness by changing the effective correction for spherical aberration. This is a somewhat difficult procedure and it should be pointed out that it is far easier to set the body tube at the correct tube length (160 mm) and purchase #1½ coverslips. Changing the tube length will also change the magnification (see Equation 2).

When using a monocular body tube the eye which is not looking down the eyepiece should be kept open. With a little practice, the image from the open eye will be ignored and only the image from the microscope will be seen. It is poor practice to try to keep the unused eye closed. For persons who have difficulty ignoring the image of the table top, notebook, impingers, etc., there is available a flat black eyeshield, supported by an arm, which clamps around the top of the body tube.

Binocular body tubes are preferred by many because they are felt to be more comfortable to use. Image splitting prisms are used to divide the beam from the objective into equal portions and direct these into paired eyepieces. This additional optical equipment makes binocular body tubes more expensive than monoculars.

The distance between the eyepieces of binocular tubes is adjustable to the individual. This adjustment will, in most mocroscopes, change the distance of the eyepieces from the objective, hence the tube length and magnification. This will of course change the calibration of the eyepiece reticle slightly, and should be taken into consideration in laboratories where more than one person counts dust with the same microscope.

Eyepieces

The eyepiece or ocular serves to further enlarge the magnified image produced by the objective, and the total magnification of the microscope system is the product of the power of the objective times the power of the eyepiece. Also, the diaphragm of the eyepiece is in focus to the eye and is the limit of the field seen by the eye.

Huygenian. The most common type of eyepiece is the Huygenian, originally designed by Christian Huygens for the telescope. This is termed a negative eyepiece in that the focus lies between the lenses. The image from the objective is focused by the field lens (Figure 9A), at the plane of the diaphragm, and this plane in turn is in focus to the eye lens.

Huygenian eyepieces are available in many powers from 4X to 15X, but the 8X to 10X are the most useful. Huygenian eyepieces of lower power are usually of too low magnification for general use and have excessive curvature of field. Powers higher than 10X tend to be uncomfortable to use and, because of the lack of correction, provide a rather indistinct image. When a high power eyepiece is needed, one of the more refined types should be used. Special types of eyepieces will be specially marked. Eyepieces having no marking other than the power are almost certainly Huygenians.

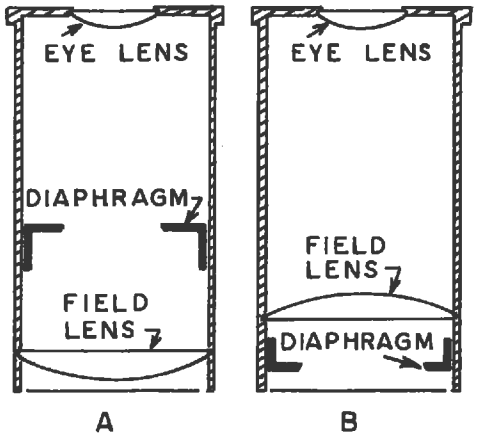


Figure 9. - Two Common Types of Eyepieces A. - Huygenian and B. - Ramsden

Ramsden. The Ramsden eyepiece is as simple as the Huygenian but not nearly so common in microscopy. It also employs two plano-convex lenses, but the convex side of the lower or field lens is away from the sample. Because the focus, and therefore the diaphragm, is outside the eyepiece, the Ramsden is called a positive eyepiece (Figure 9B).

Kellner (Orthoscopic). The Ramsden in its simple form tends to produce chromatic effects in the image, so it is modified by replacing the single eye lens with an achromatic doublet, and in this form is called a Kellner eyepiece.

Hyperplane. The hyperplane eyepiece is a similar modification of the Huygenian; that is, the single eye lens is replaced by an achromatic doublet.

Wide Field. Wide field eyepieces are specially designed to provide a large and flat field of view for scanning and searching the sample. They have the disadvantage of quite low eyepoints for powers of 10X and above.

High Point (also: Hi Point). High point eyepieces are specially designed to have the eye point as far as possible from the eye lens. This enables the observer to wear eyeglasses while using the microscope.

Compensating. Compensating eyepieces are fine quality lenses corrected for chromatic aberration. They are called "compensating" because they are specially corrected to compensate for the slight chromatic difference of magnification of the apochromat objectives. Compensating eyepieces, while intended especially for apochromat objectives, serve excellently for the higher powers of other types of objectives.

Illumination

It is difficult to overemphasize the need for critical adjustment and control of the illumination system when attempting to achieve accuracy and ease of use of the microscope. Adjustment of the illumination system, including the substage condenser, is the easiest and most effective means by which the microscopist may control the performance of the instrument.

It is therefore surprising and unfortunate that so many who use the microscope choose to remain uninformed about the practice of correct illumination. As a matter of record, laboratories which have instituted correct illumination methods have experienced important improvements in the results of routine examinations. Because the procedures for correct illumination are relatively simple, and because their adoption will introduce a standard which will help increase the reliability of routine determinations, there is no reason for any user of the microscope to fail to become familiar with them.

There are basically two types of correct or "critical" illumination which are most often used in transmitted light microscopy - Nelson and Kohler. They differ, as shown in Figure 10, in that the source (filament) is imaged in the plane of the sample in Nelson illumination while in Kohler illumination the source is imaged at the plane of the substage iris and the lamp (field) diaphragm is imaged in the plane of the sample. The Nelson method is sometimes

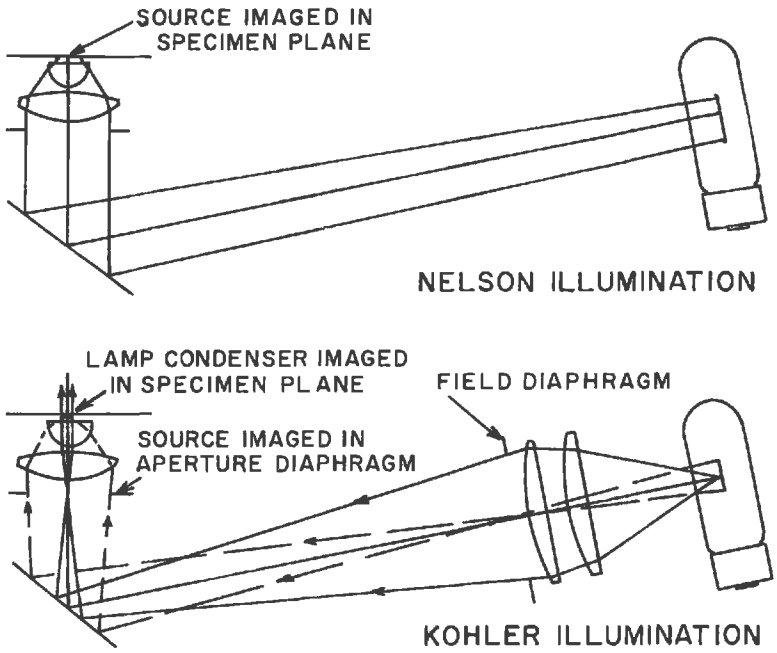


Figure 10. - Nelson (critical) illumination (top) and Kohler Illumination (bottom).

referred to as "Critical Illumination." This might seem to imply that Kohler illumination is noncritical, which is not the case. Because the two methods have been shown to produce identical results, and because the Kohler method is much more easily aligned and controlled, it has become almost universal in application. Almost all microscopes having built-in illuminators are designed around the Kohler method. This is a desirable situation, as little alignment is needed once the instrument is adjusted according to the manufacturer's instruction manual. Kohler illumination is then achieved "automatically" by the mechanical construction of the instrument.

For those who must use external illuminators, the following notes will be of help in achieving Kohler illumination:

Requirements for Kohler-type Critical Illumination

1. A small and relatively compact, but not necessarily perfectly homogeneous light source. Ribbon filament or tight coil filament lamps are best. Illuminators designed to use ribbon filament lamps (such as Bausch and Lomb PR-27) are generally much easier to use than those designed for coil filament lamps.

2. The illuminator must incorporate a condensing lens so that an enlarged image of the lamp filament may be focused in the plane of the substage condenser iris diaphragm.

3. The illuminator must also have an iris diaphragm located as near as possible to its condensing lens. This iris serves as a field diaphragm and is focused in the plane of the specimen.

4. The microscope used must have a substage condenser fitted with an iris diaphragm. This iris serves as an aperture diaphragm.

5. The use of a first surface mirror or prism is recommended, but not required, for Kohler illumination.

Using an ordinary second surface mirror will cause multiple images of the illuminator field iris, which will slightly detract from the crispness of the final stage.

Procedure for Achieving Kohler Illumination

1. Place the microscope on a flat, level, and steady surface at a height such that the eyepieces can be observed without straining.

2. Place a properly mounted sample of a moderate to heavy dust concentration on the stage.

3. Place the illuminator in front of the microscope. For coil filament bulbs the illuminator iris should be about 10 inches from the microscope mirror; for ribbon filament illuminators (i.e., B&L PR-27) the front of the filter holder should be 7 inches from the center of the plane side of the mirror - never use the curved side.

4. Remove all diffusing filters from the system. Insert clear (unfrosted) Corning blue and one or more neutral density filters.

5. By means of the focusing knob on the side of the substage condenser assembly, raise it until the upper lens of the condenser nearly touches the bottom of the slide. Remember that the free working distance of the substage condenser is in the order of 1.2 mm, and that the slide is about 1 mm thick. Therefore the upper lens of the condenser must nearly touch the lower surface of the slide to be in focus on the sample lying on the upper surface of the slide. For "vari-focus" condensers which have the upper lens fixed to the stage and only the lower element and diaphragm are movable, the lower element should be near the top of its adjustment.

6. Turn on the illuminator. Using the tilt controls of the illuminator direct its beam toward the center of the mirror - a piece of ground glass placed over the mirror may be helpful in this. By tilting the mirror, direct the beam upward into the condenser.

7. For coil filament bulbs, close the substage (aperture) iris all the way, and open the illuminator (field) iris fully.

8. By means of the focusing controls on the illuminator focus the image of the filament on the bottom surface of the substage (aperture) iris, while observing this image in the mirror. Steps 7 and 8 are not needed when using ribbon filament illuminators.

9. Open the substage iris about halfway.

10. Put the 10X objective in place and focus it sharply on the sample.

11. Close the illuminator (field) iris fully. If the condenser is in focus, the image of the illuminator iris should appear as a sharply imaged bright disc on a dark field in the eyepieces. If it is not in sharp focus, bring it to focus with the condenser focusing knob. If an ordinary second surface substage mirror is used, there will be faint ghost images about the main image of the field iris opening. These are caused by secondary reflections from the first surface of the glass mirror.

12. If the image of the field iris is not centered, center it with the mirror. Readjust the tilt of the illuminator, if necessary.

13. If the 10X objective that has been used for alignment is used to

examine the sample, open the field iris until it just includes the field of interest. The microscope is now ready for use.

14. If it is desired to work at a high power, leave the field iris at its fully closed position and change to the high dry objective.

15. If the objective lenses are parfocal, the high dry should be nearly in focus on the sample. Very carefully, using the fine focus controls, focus the objective on the sample.

16. The image of the field iris, produced by the condenser, also should be nearly in focus. Trim the adjustment with the condenser focusing knob. The image of the field iris will probably be slightly off center - center it by tilting the mirror.

17. For the B&L PR-27, the front lens must be all the way forward for the 10X objective and all the way back for all higher power objectives.

18. If the above steps have been completed, Kohler illumination has been achieved and the instrument is ready for use.

19. If it is desired to use an oil immersion objective, the procedure for focusing an oil immersion lens should be followed as outlined in the section on objective lenses. Keep in mind that immersion oil must be in contact with the upper lens of the condenser and the bottom of the slide if the maximum resolution of the objective is to be realized.

The ordinary coil filament lamp, having a rather open structure in the form of a "W" will not be adequate for use with a 10X objective because only one wire of the filament will be focused in the center of the substage iris, and the width of the single coil is not sufficient to fill the aperture evenly. In this case, the lamp should be replaced with one having a more compact filament structure.

A simple modification of the less expensive illuminators may be made to achieve the geometry of Kohler illumination, even if a lamp with a sufficiently compact filament structure cannot be substituted. This entails the substitution of a 75 W frosted lamp. The frosting will be far enough from the plane of the field iris so as not to be in focus in the image plane when Kohler illumination is used.

It is important to remember that Kohler illumination cannot be achieved if any sort of frosted filter is placed in the system between the field iris and the sample, or within the illuminator at a place close enough to the field iris for the grain of the ground glass to be focused in the specimen plane along with the field iris.

The Microscope as a Micrometer

One of the most useful applications of the microscope is to measure the dimensions of small objects. In dust determinations this function is twofold: to measure an area for the quantification of dust particles; and to measure the size of individual dust particles.

The simplest method of measurement with the microscope is to use a micrometer eyepiece containing a fixed scale, in arbitrary units, placed at the focus of the eye lens of the eyepiece. This scale will be in focus to the eye, and, when the objective is focused on a sample, will appear to be superimposed on the sample. When the eyepiece scale is calibrated in absolute units for the individual objective used, any object in the sample may be measured in terms of these units by comparison with the scale.

Reticles

The scale is engraved on a glass disc known as a reticle or graticule. Because Huygenian eyepieces contain a field-limiting diaphragm, which is in focus to the eye lens, anything placed on the diaphragm also will be in focus. Most other types, except the wide-field lenses, also contain a field-limiting diaphragm on which a reticle may be placed. When wide-field eyepieces are used the reticle must be held in place by a circular spring clip.

Nearly all reticles are now made by a photographic process and the grid lines are in the center of a glass sandwich having some appreciable thickness. For this reason the grid will not be in precisely the same plane as the diaphragm, and will be slightly out of focus to the eye lens when it is in its normal position. This may be remedied by unscrewing the eye lens a few turns from its fully seated position. In this way it will be possible to bring the reticle lines into sharp focus. This practice, however, tends to chip small fragments of paint from the threads of the eye lens, which then fall on the reticle and appear superimposed over the image of the dust sample. Such a situation leads to some confusion.

A better alternative is to purchase from the manufacturer focusing eyepieces which contain a focusing mechanism to bring the reticle lines into sharp focus. Binocular body tubes have one eyepiece holder equipped with a focusing mechanism which moves the entire eyepiece, relative to the objective, in order to compensate for differences between the individuals' eyes. The focusing eyepiece containing the reticle should be put in the eyepiece holder which is not focusable, but fixed.

A great many types of reticles are available for various special purposes. Three types are in common use in dust work; the Whipple, Page (or the similar American Optical Company Net reticle), and Porton reticle.

The Whipple reticle (or disc) is shown in Figure 11. This reticle is divided into 100 squares. One of these squares having its corner at the center of the

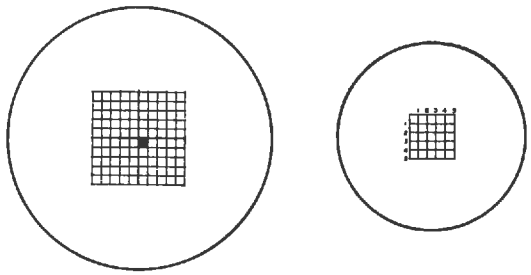


Figure 11. - Whipple Disc (left) and Page Disc (right).

reticle is further sub-divided into 25 smaller squares. The method of sampling and counting dust adopted at the Fifth Annual Meeting of the National Conference of Governmental Industrial Hygienists in 1942 recommends the Whipple disc for dust counting. It should be noted that there are two major drawbacks to the system therein described. First, if the Whipple reticle is used, the 1942 procedure calls for counting one-quarter of the reticle. Because much of any quadrant will be near the periphery of the field of view, part of the dust observed will be in the zone of considerable spherical aberration (remember that only the central portion of any lens field should be used). For the same reason one should avoid any reticle which extends out of the central portion of the objective field.

The Page reticle (Figure 11) was designed to overcome this fault, being one quarter of a Whipple reticle located in the center of the field. However, one of the most easily available versions of the Page reticle, the A-O Net reticle, is so large that again it projects too far toward the periphery of the lens.

The second major drawback to the microscope system as proposed in 1942 by the NCGIH is the recommendation to use an

adjustable body tube and to manipulate it until the Whipple reticle field subtends exactly one square mm of the sample and field. That this practice could probably introduce excessive spherical aberration, thereby reducing contrast, will be understood from the previous discussion of objective lenses and body tubes.

The Porton (or May) reticle is fully described in the discussion on Dust Sizing (Section B-6).

Reticles are usually manufactured with a diameter of 21 mm. This size will fit on the field diaphragm of nearly all makes of eyepieces. However, there are a few manufacturers who build eyepieces with a slightly smaller inside diameter, in which the standard reticle will not fit. To make a reticle fit an undersize eyepiece, lightly grind or sand the edges of the disc until it fits the eyepiece tube.

The units of any reticle are arbitrary, and will be of a different value for each objective used. Therefore, they must be calibrated for all the objectives on the microscope and used only with those objectives, unless another calibration is done.

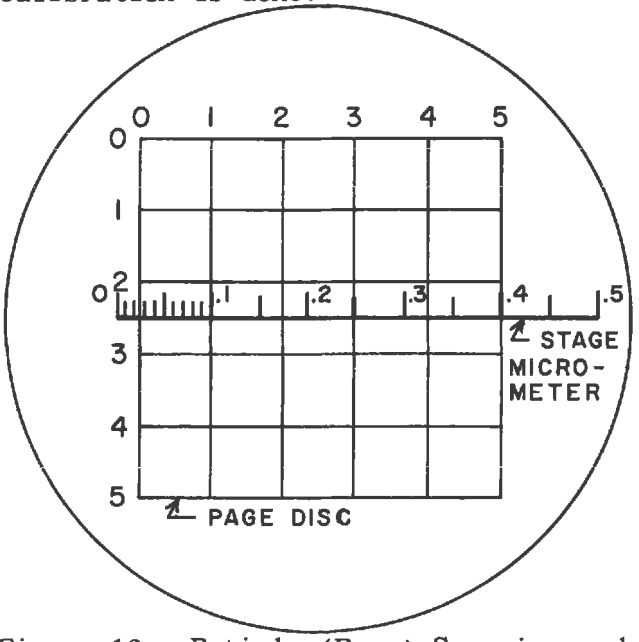


Figure 12. - Reticle (Page) Superimposed Over Stage Micrometer at Low Magnification

Calibration is carried out by comparing the reticle scale with a known scale called a stage micrometer. This is simply a very finely ruled scale (usually in 0.1 mm and 0.01 mm divisions) which fits the stage of the microscope. When the objective is focused on the stage micrometer the reticle scale or field appears in focus superimposed on the stage micrometer scale as in Figure 12.

Using the mechanical stage screws the stage micrometer is aligned with the eyepiece reticle and the size of the reticle measured. Since the 0.01 mm divisions are not continuous across most micrometers but located only at one end, one edge of the reticle should be aligned with one of the even 0.1 mm lines so that the other edge of the reticle field to be measured falls among the fine 0.01 mm divisions.

If an arbitrary scale is to be calibrated rather than the side of a square, essentially the same procedure is followed. If 0.1 mm of the stage micrometer covers 36.5 divisions of the reticle scale, this reticle scale unit will be:

$$\frac{0.1 \text{ mm}}{36.5} = .00274 \text{ mm}$$

or 2.74 μ. Then any measurement made with this objective-eyepiece combination will entail estimating the length (for example) of an object in reticle units; then multiplying this value by 2.74 to arrive at the value in microns.

It should be kept in mind that at higher magnifications the lines of the stage micrometer appear quite enlarged and have appreciable width. For this reason a convention should be established to start and end measurements on the same relative side of the stage micrometer lines.

Once a calibration has been made the optical system of a particular microscope must not be altered in any way; otherwise, it will require recalibration. Eyepieces should not be changed from one microscope to another, nor

should objectives. Objectives with correction collars should be calibrated with the correction collar at a standard setting to which it can be returned for measuring. Adjustable monocular body tubes should be set and locked in some manner at the correct mechanical tube length of 160 mm (or 170 for Leitz). The same convention holds true for adjustable ("zoom") eyepieces.

Phase Contrast Microscopy

A transparent object depends, for its ability to be imaged by the microscope, on the difference between its index of refraction and the index of refraction of the surrounding medium. Where the indices of the particle and the medium surrounding it are identical or nearly so, refraction will not take place, the illuminating rays will pass directly through the particle without being deviated from their paths, and the particle will be invisible.

Optical path distance is the product of the index of refraction and the thickness of the particle through which it passes. In cases where the optical path distance of the sample particle and its surrounding medium is less than one-twentieth of a wave length, the particle will be invisible.

The phase microscope is a modification of the normal bright field optical system which causes very small differences in optical path to become visible in the image.

The formation of the phase contrast image is a topic beyond the scope of this discussion. It is important to note, however, that the substage iris is kept fully open, so the full resolution of the objective is realized. For additional information on phase microscopy, consult standard texts and manufacturer's literature on this subject.

As has been noted in the discussion of optics, the bright field microscope sacrifices some resolution in favor of contrast by partly closing down the substage iris. The phase contrast microscope is highly useful in dust work, as it permits the use of a large aperture, with an enhancement rather than a loss of contrast. Not only are particles with indices near those of the medium surrounding them clearly imaged, but also very small opaque particles, nearly or completely invisible in the bright field microscope, will be sharply imaged. Generally, the phase microscope will enable the observer to see about twice as many dust particles as can be seen using the bright field microscope.

Glossary of Terms Used in Microscopy

Aberration, Chromatic - The defect of a lens occurring when rays of polychromatic light are not brought to the same focus.

Aberration, Spherical - The defect of a lens occurring when monochromatic rays passing through the periphery of the lens do not come to the same focus as rays of the same wavelength which pass through the lens near its center.

Achromat - An objective lens corrected for two colors chromatically and one color spherically.

Annulus, Phase - A plate with a ring-shaped transparent area, located in the front focal plane of the substage condenser, and serving to limit the illuminating beam to a hollow cone.

Aperture, Angular - The angle of the included cone from a point at the focus to the aperture of a lens.

Aperture, Numerical - The dimensionless expression of the relative light gathering power of a lens. Expressed as half the angular aperture times the index of refraction of the medium between the object and the lens.

Aplanatic - Corrected for spherical aberration.

Apochromat - An objective lens corrected for three colors chromatically and two colors spherically.

Aspheric - A lens having one surface ground as a parabolic curve rather than a spherical section.

Compensating Eyepiece - An eyepiece having slight over correction to compensate for the chromatic difference of magnification provided by an apochromat objective.

Condenser - Any lens or system of lenses designed to gather rays of light and cause them to converge at a focus.

Condenser, Substage - The system of lenses, normally including an iris diaphragm, which is fitted to the microscope below the sample stage, and which serves to collect the light from the illuminator and focus it on the specimen.

Correction Collar - An adjustment ring fitted to the barrel of apochromat and fluorite high-dry objectives, which may be rotated to adjust the lens interspacing so as to compensate for deviations in coverslip thickness from the standard of 0.17 to 0.18 mm.

Critical Angle - The angle of incidence, for rays passing from a medium of higher index to one of lower index, for which the angle of refraction is 90°. For angles of incidence greater than the critical angle the rays are totally reflected back into the medium of higher density.

Dark Field - A system of illumination in which the sample is illuminated with a hollow cone of light whose minimum aperture is greater than the maximum aperture of the objective, so that no direct rays enter the objective, but only rays deviated by objects in the sample.

Depth of Focus - The distance along its axis within which objects may lie, to appear in sharp focus in the image.

Dispersion - The difference in index of refraction for violet light and for red light for a given material.

Eyepiece - = Ocular. The uppermost lens system of the microscope, which provides the final enlargement of the image provided by the objective.

Field Iris - = Illuminator iris.

Filter - A transparent or diffusing plate to be inserted in the illuminating beam, designed to transmit only a specific portion of the spectrum, or to reduce the intensity of the beam.

Fluorite (objective) - An objective system, intermediate in quality between an achromat and an apochromat, which has one or more lens elements made of the calcium fluoride mineral fluorite.

Focal Length - The distance from the optical center of a lens to its focus.

Free Working Distance - The distance between the front of an objective mount and the surface of a 0.17-0.18 mm coverslip when the lens is focused on the sample.

Graticule - = Reticle.

High-dry (objective) - Any objective, not computed for immersion, with magnification from about 40X to about 60X and NA from about 0.50 to about 0.85.

Huygenian Eyepiece - An eyepiece having two plano-convex lenses separated by a diaphragm, the convex sides of the lens facing the objective.

Illumination, Critical - Either Nelson or Kohler illumination. In popular usage, sometimes implies only Nelson illumination.

Illumination, Kohler - The system of

critical or precise illumination in which the image of the light source is in focus at the front focal plane of the condenser, and the field iris is in focus at the sample plane.

Illumination, Nelson - The system of critical or precise illumination in which the image of the light source is in focus at the sample plane.

Illuminator Iris = Field Iris - The iris diaphragm contained in the illumination system. In Kohler illumination it is imaged in the plane of the specimen and serves as a field-limiting iris.

Immersion Objective - An objective lens system computed to operate with a thin film of some liquid between its front lens and the coverslip or sample, for the purpose of increasing the numerical aperture, hence the resolving power.

Mechanical Stage - A device, fitted to the stage of the microscope, having a means of holding the sample slide and micrometer screws for traversing the slide in two directions.

Micrometer Eyepiece - An eyepiece containing a scale, in arbitrary units, or a grid pattern, which is in focus to the eye lens, hence appears superimposed over the image from the objective.

Micron (μ) - As a unit of length = 0.001 mm; rarely, a particle between 0.01 and 0.0001 mm in diameter.

Millimicron (mμ) - A unit of length equal to 0.001 μ; one thousandth of a micron.

Monochromatic Light - Literally, light of one single wavelength. Practically, light of one color of the spectrum.

Objective (lens) - The lens system located near the specimen or object which forms the primary image.

Ocular - = Eyepiece.

Paraboloid (condenser) - A type of substage condenser for dark field, utilizing a parabolic mirror surface.

Parfocal - The condition of a set or group of objectives and/or eyepieces if there is little or no change of focus when one lens is exchanged for another.

Peniplan Eyepiece - A type of compensating eyepiece manufactured by E. Leitz with a rather large and flat field. Intended mainly for photomicrography.

Phase Contrast - An optical system in which one part of the illuminating beam is treated differently from the rest, and the two parts caused to interfere in the fine image, allowing the imaging of transparent objects very similar to their surrounding medium in index of refraction.

Polychromatic Light - = White light. Light composed of many wavelengths.

Ramsden Circle - The point above the eye lens where the image forming rays cross.

Ramsden Eyepiece - An eyepiece having two plano-convex lenses, the convex side of the lower (field) lens facing away from the objective. The diaphragm is located outside the two lenses, below the field lens.

Refraction - The change in direction of a ray of light when passing across an interface separating two media or differing indices of refraction.

Refraction, Index of - = n. The ratio of the velocity of light in a vacuum to the velocity of light in a given medium. Also expressed as the ratio of the sines of the angles of incidence and refraction relative to a normal to the interface for a ray passing obliquely from a vacuum to the medium.

Resolving Power - The smallest distance by which two points may be separated and still be imaged as two points by a given lens.

Reticle - = Graticule. A scale or grid or other pattern located in the focus of the eyepiece of the microscope.

Spectrum - Any series of radiant energies arranged in order of their wave length. The visible spectrum consists of a continuous series from 760 mμ (red) to 385 mμ (violet).

Stage Micrometer - A microscope slide bearing a scale of one or two millimeters divided in 0.01 mm units.

Substage Iris - = aperture iris. The iris diaphragm located in the substage condenser assembly. In Kohler illumination it is imaged in the back focal plane of the objective and controls the aperture of the illumination.

Tube Length, Mechanical - The distance from the objective socket to the eyepiece socket.

Tube Length, Optical - The distance from the back focal plane of the objective to the plane of the image formed when the eyepiece is removed.

Ultraviolet Light - Light of wave length between the shortest visible (violet, 385 mμ) down to about 200 mμ.

Wave Length - The distance along the path of a ray from any point to the next point having the same phase.

White Light - = polychromatic light.

II. - ELECTRON MICROSCOPY

Introduction

The electron microscope has been defined as an aid to vision in the study of solid structures in the 0.001 to 10 μ range. This definition completely and simply describes the value of the instrument and indicates the procedure for its use. However, as a relatively young instrument the procedures used are not often standard and each new material observed raises new problems of specimen preparation and interpretation of the micrographs produced.

The first electron microscope was built in 1931 based on the de Broglie theory associating the wave length of an electron beam with the accelerating voltage and published in 1924. The first one on the North American continent was constructed at the University of Toronto in 1938. Since that time the development has been very rapid and there are now a great many commercial instruments for use in this country.

Description of the Instrument

In theory the electron microscope is nearly an exact analogue of the optical microscope. Its physical form, however, is completely different because of the necessity of using a high accelerating potential to produce the electron beam and of handling this beam in a vacuum which assures a mean free path of the electrons greater than the distance from the source to the final image. In essence the instrument is a large vacuum chamber which contains an electron source, a substage condenser, a mechanical specimen stage permitting motion in two directions, a short-focal-length objective lens, a projector lens, a fluorescent viewing screen, and a camera. Each component performs the same function as the corresponding one in the optical microscope.

The electron source is usually a V shaped filament of tungsten wire which is heated to incandescence by an electric current and thus emits electrons.

Directly below the filament is the anode, a metal ring which is maintained at a high potential (50 - 100 KV) with respect to the filament and thus accelerates the electrons down the column. The condenser lens, as well as all other lenses, may be either electromagnetic or electrostatic fields which focus an image of the source directly on the specimen for maximum illumination or slightly above or below the specimen for reduced illumination. Unlike the optical microscope, the lenses need not be moved to change focus but can be made either stronger or weaker by controlling the current through them. The specimen holder is an inverted cone shaped piece which projects downward into the center of the objective lens with the specimen screen held securely at the apex. The objective lens is the heart of the instrument. It is a strong lens which minimizes spherical and chromatic aberrations that would otherwise severely limit the resolving power of the instrument and has a highly compensated pole piece to correct astigmatism. The projection lens controls the final magnification of the image and projects this image on the viewing screen or film. The electronic image is, of course, invisible to the human eye and therefore a fluorescent screen is used to convert it to visible light for preliminary scanning and focusing. The camera consists of a photographic plate placed below the viewing screen so that when this screen is removed or tilted the electronic image falls on the photographic emulsion which is quite sensitive to electron bombardment. This plate is developed using standard photographic techniques.

Preparation of Specimens

As in optical microscopy, the main research problem connected with the use of the electron microscope is in the preparation of specimens which are accurately representative of the substance being studied. The most difficult aspect of this preparation is that no criteria exist for checking morphology in the 0.001 - 1.0 μ range.

Satisfactory specimens for the electron microscope must meet certain conditions. (1) They must be sufficiently thin to allow the electron beam to penetrate the specimen without appreciable loss of energy. Electrons with a reduced energy exhibit a longer wave length and the chromatic aberration present in the lenses produces a loss in resolution. Loss of energy by the electrons implies that energy is gained by the specimen with consequent increase in temperature which may cause a change in structure. The degree or nature of such a change depends on the chemical composition and physical form of the specimen. If large particles are involved the charge adsorbed and the repulsive forces produced may be sufficient to rupture the thin supporting membrane on which the particles rest. (2) The specimen must exhibit sufficient contrast. Since contrast is produced by differences in scattering power or density of various portions of the specimen this is not normally much of a problem when inorganic particles are being studied. (3) The high vacuum of the electron microscope column precludes the observation of wet or live tissue or any other volatile material. In preparation of specimens any artifacts which could be caused by drying the sample, such as surface tension effects, changing pH, etc. should be recognized.

The specimen is usually mounted on an extremely thin film of Formvar(R) or collodion. The Formvar or collodion films being of the order of 100 A thick are quite transparent to the electron beam. Recently some other substrate media such as evaporated films of silicon dioxide, silicon monoxide, and carbon have come into use and appear to hold much promise. All of these films are supported on 1/8 inch circles of 200 mesh copper grid and observations are made through the open areas between the wires.

The problem of transferring the specimen to the previously prepared film on its supporting grid may vary with each sample. If a sample of air-borne dust has been collected in water by an impingement technique a drop of this dust suspension may be placed on the grid and the water allowed to evaporate. In this process, however, the dust will be found to deposit in rings as the evaporation proceeds and therefore the fields observed may not be truly representative of the original suspension.

The filmed grids may be placed in a thermal precipitator or an electrostatic precipitator and the air-borne particles deposited directly on the film. In each of these techniques, however, the deposition of the particles may be affected by their size and the sample obtained may not be representative.(1) It is apparent that a knowledge of the efficiency and operating principles of the sampling instrument is essential as well as an understanding of the electron microscope. Sampling slides used with the oscillating thermal precipitator or the long running thermal precipitator may be coated with Formvar and the sample taken directly on the Formvar. The Formvar is then floated off on water, and picked up with grids from beneath.

A technique which has been used successfully is that of collecting the sample on a membrane filter and then dissolving this filter with a diffusion method using ethyl acetate and allowing the particles to settle gently onto a grid prepared with a Formvar film.(2) The Formvar film is not soluble in this solvent. Many other techniques are described in the current literature(3,4) and older methods of specimen preparation are discussed in standard texts on electron microscopy.(5,6,7,8)

References

1. Fraser, D. A.: The Collection of Submicron Particles by Electrostatic Precipitation, Am. Ind. Hyg. Quart., 17:75 (1956).

2. Fraser, D. A.: Absolute Method of Sampling and Measurement of Solid Air-Borne Particulates, Arch. Ind. Hyg. and Occ. Med., 8:412 (1953).

3. Breese, S. S., Jr., Ed.: Electron Microscopy Vol. I (Non-Biology). Fifth International Congress for Electron Microscopy, Academic Press, New York, 1962.

4. _____: Survey of Preparation Techniques for Electron Microscopy, n.v. Philips, Gloeilampon fabriekon, Scientific Equipment Department, Eindhoven, The Netherlands, (undated).

5. Drummond, D. G.: The Practice of Electron Microscopy, J. Royal Microscopical Soc. LXX: (March 1950).

6. Hall, G.E.: Introduction to Electron Microscopy, McGraw-Hill, New York, 1953.

7. Haine, M.E. The Electron Microscope, E & F.N. Spon, London, 1961.

8. Kay, Desmond: Techniques for Electron Microscopy, Blackwell, Oxford, 1961

General References on Microscopy

1. Benford, J. R.: The Theory of the Microscope, Bausch and Lomb, Inc., Rochester, N. Y., 1960.

2. Clark, G. L., Ed.: The Encyclopedia of Microscopy, Reinhold, New York, 1961.

3. Hopkins, H. H., Proc. Roy. Soc. (London), A 208, 263 (1951).

4. Johnson, B. K.: Optics and Optical Instruments, Dover, New York, 1960.

5. Martin, L. C.: The Light Microscope; in: Physical Techniques in Biological Research, Vol. I - Optical Techniques, G. Oster, Ed., Academic Press, New York, 1955.

6. Needham, G. H.: The Practical Use of the Microscope, Charles C. Thomas, Springfield, Ill., 1958.

7. Wren, L. A.: Understanding and Using the Phase Microscope, Unitron Instrument Co., Newton Highlands, Mass., 1963.

DUST COUNTING TECHNIQUES

Introduction

The method of impinger sampling and light field counting commonly used in the United States is empirical and has remained essentially unchanged for the past two or three decades. In practice, water, alcohol, or a mixture of the two is employed as a sampling medium in standard or midget impingers. After sampling, a portion of the liquid medium containing the collected dust is removed from the impinger flask and is placed in a counting cell. Particles of dust settle to the floor of the cell and the number of particles from a specific volume of liquid, and thus from a measured volume of sampled air, are counted. All particles which have reached the floor of the cell by the time of counting and are visible by light field optical microscopy with a 10 power objective lens are included in the count.

The Standard Method

The number of dust particles which may be detected in a defined volume of air from a dusty working place depends upon the method of detection used. Many methods for making such determinations have been proposed and many are used. All are empirical in some degree; there is yet no practical absolute method for assessing the number concentration of atmospheric dust. The Threshold Limit Values published by the American Conference of Governmental Industrial Hygienists, are guides by which we can judge the hygienic significance of exposures to mineral dusts. The method of assessing exposures for interpretation by these guides is specified, and since the method is empirical departures from the method can greatly influence the results. If there are departures from the standard method in measuring exposures, the results cannot be interpreted by use of the Threshold Limit Values.

Prepared by: Robert L. Harris, Jr.

The standard method presented at the fifth meeting of the National Conference of Governmental Industrial Hygienists in 1942[1] is as follows:

"Standard Impinger Sampling and Counting Technique

I. Application. For the determination of the atmospheric concentration of an insoluble inorganic dust such as that associated with the production of lung fibrosis, the particle size of which is generally below 10 μ and, from the hygienic standpoint, is not of interest below 0.5 μ. This method is not recommended for the determination of organic dusts, metallic fumes, vapors and gases.

II. Sampling instrument. The sampling instrument shall be the standard impinger, operating at a rate of 1.0 cfm at 3" Hg negative pressure (U. S. Public Health Service or modified form), or the micro-impinger operating at 0.1 cfm at 12" H_2O negative pressure (U. S. Bureau of Mines).

III. Flow-producing apparatus and flow meter. Any type of suction apparatus may be employed, provided it is capable of maintaining the rate of flow with ± 3 percent of the rated capacity, i.e., 1.0 cfm for the standard and 0.1 cfm for the micro-impinger, and is provided with a calibrated flow meter and indicating gauge. The use of a constant-flow orifice is recommended with the compressed air ejector and an automatic negative pressure regulator with the motor or hand-driven pump. If an indirect indicating gauge is used, it shall be checked against a U tube or gas meter at regular intervals.

IV. Counting cell. The standard counting cell shall have a liquid depth

of 1.0 mm. Counting cells less than 1.0 mm deep down to 0.1 mm may be employed, provided the liquid depth is reported with dust concentration data thus obtained.

V. Microscope and accessories

A. The microscope shall be a standard biological instrument or its equivalent, preferably with an adjustable tube length. It shall be provided with an Abbe condenser with iris diaphragm, 16 mm objective 7.5 or 10x ocular.

B. The counting area shall be defined by an ocular grid or by rulings on the counting cell. It shall have an area equivalent to ¼ sq. mm in the counting cell and shall be divided into 25 sub-squares.

C. The microscope tube length shall be adjusted so that the counting area corresponds to ¼ sq. mm in the counting cell, or, in the case of a fixed tube length, the ruled area shall be equivalent to ¼ sq. mm ± 3 percent. The counting area shall be accurately measured by means of a stage micrometer and recorded with the dust concentration data.

D. Illumination shall be provided by means of a 15 W substage lamp, placed directly under the condenser or a 75 W microscope lamp, placed approximately 10" from the plain mirror. A 'Daylight' filter shall be used.

E. A microprojector of the type described by the Bureau of Mines[1,2] may be used instead of the preceding system.

The microscope of the microprojector shall be the same as the preceding microscope, except that a 20 or 25 ocular shall be used.

The counting area shall be defined by rulings on the translucent screen. It shall have an area equivalent to ¼ sq. mm in the counting cell and shall be divided into 25 subsquares, each of which may be further subdivided into not more than 10 similar (equal area) rectangles.

The distance between the microscope, with tube length adjusted to 160 mm, and the screen shall be such that the magnification of images upon the screen is 1,000 diameters. The microscope tube length shall be adjusted so that the counting area, or 50 cm square ruling on the screen, corresponds to ¼ sq. mm in the counting cell, or, in the case of a fixed tube length, the ruled area on the screen shall be equivalent to ¼ sq. mm ± 3 percent. The counting area shall be accurately measured by means of a stage micrometer and recorded with the dust concentration data.

Illumination shall be provided by means of an automatic-feed (preferably 4.5-6 ampere direct current) carbon arc lamp so arranged to supply adequate illumination on the screen. A suitable heat filter (water cell or heat-absorbing glass) shall be used between the lamp and the microscope.

VI. Collecting liquid. Distilled water with or without alcohol (ethyl or isopropyl) shall be used as the collecting liquid. The collecting and subsequent washing and diluting liquids shall show not more than six (6) countable particles in ¼ sq. mm counting area in the cell.

The 'blank' count shall be determined under the microscope for every series of samples.

VII. Sampling technic.

A. All glassware shall be clean and the equipment protected against dust contamination in the field. One impinger flask shall be treated exactly like the others except that no air shall be drawn through it. The liquid in it shall be used to determine the blank count which must be subtracted from the sample counts, as described below.

B. The number and spacing of the samples at a given operation, the locating of the sampling point, and the length of each sampling period shall be so chosen as to yield a representative measure of the exposure throughout the cycle of operation and also to indicate the peak concentrations. The minimum number of samples at each sampling point shall be two.

C. In addition to samples collected in the breathing zone of the exposed workers, samples should be taken to determine the dust concentration in the general workroom atmosphere and in special studies, to determine the sources of the dust dispersion or distribution.

VIII. Treatment of collected sample.

A. The sample shall be counted within 36 hours after collection except that in the case of flocculating dusts, the permitted holding time shall not exceed 12 hours. Special collecting liquids may be employed to mimimize the degree of flocculation for certain dusts.

B. The sample shall be made up to a known volume, after washing down and removing the impinged nozzle. The concentration of the suspension shall be such as to give a dust count at least 4 times the 'blank' count and not greater than 150 particles in the ¼ sq. mm counting area. When further dilution from the initial volume is required to reduce the field count to the limits specified above, the dilution shall be made in steps not exceeding 1:10. Complete mixing of the suspension by vigorous agitation is required before removing a portion for dilution and volume removed shall not be less than 10 percent of the total volume of the suspension to be diluted, except that the minimum volume to be removed from the original sample for dilution shall not be less than 5 cc. Excessive dilutions are to be avoided by proper gauging of the sampling time in relation to the dust concentration.

IX. Preparation of the sample for counting.

A. Cells shall be cleaned before use and the effectiveness of cleaning shall be checked by microscopic examination of each cell before being filled.

B. The suspension to be counted shall be shaken vigorously and a portion transferred immediately to the cell by means of a clean pipette, taking care to prevent the inclusion of air bubbles.

C. One or more cells shall be filled from each sample.

D. Cell contents shall settle before counting, at least 30 minutes for the 1.0 mm cell and proportionately less for the cells of less depth.

E. One cell shall be made up from the 'blank' flask for every series of samples.

F. Cells shall not be filled in advance of the time required for settling before counting.

X. Counting

A. Counting shall be done in a darkened room, using artificial illumination in the microscope, as described above.

B. The light and mirror shall be centered so that no shift of position of the particles is seen when focusing up and down. The condenser and diaphragm shall be adjusted to give an even moderate illumination. Each observer should determine and employ the optimum lighting for his eye.

C. Before counting, the field of the microscope shall be examined and, if necessary, the ocular grid cleaned to remove dust particles.

D. The count shall be made in a plane just above the surface of the cell rather than throughout the depth of the liquid. The microscope may be focused up and down by means of the slow-motion adjustment in order to bring individual particles into focus. Before making the count, the cell contents shall be carefully examined throughout the entire depth to make certain that all countable particles have settled.

E. Five fields of ¼ sq. mm in area shall be counted, one in the central part and the other four towards the edges of the cell.

F. Except in the case of abnormal samples, no distinction shall be made between the number of particles above and below 10 μ.

XI. Computation of results. The concentration of dust shall be expressed in millions of particles per cubic foot of air (m.p.p.c.f.), reported to two significant figures only.

Concentration is determined as follows:

$$\text{Conc. (m.p.p.c.f.)} = \frac{(N_s - N_c) \times C.F. \times V_w}{V_a \times 10^6}$$

Where:

N_s = Mean count per field for the sample (5 or more fields)

N_c = Mean count per field for the blank (5 or more fields)

C.F. = Cell factor = 1000/(area of counting field in sq. mm) x (depth of cell in mm)

V_w = Total water volume (equivalent total, when portion diluted, i.e., initial sample volume x dilution factor)

V_a = Air volume sampled in cu. ft. "

EXAMPLE:

Duration of sample time: 5 minutes

Rate of sampling: 0.1 cubic foot per minute

Vol. in flask after washing: 30 ml.

Ratio of added dilution (5 ml in 25 ml flask): 5:1

Cell depth: 1 mm.

Dust count, light field (2 cells) particles less than 10 microns in size

Cell #1	Cell #2
51	48
36	54
44	47
34	40
55	49
220	238

Total #1 220

Total #2 238

458 = 45.8 Av. Total/Field

−5 blank count

40.8 Av. Net count

CALCULATIONS:

$$\frac{\text{Av. Net Count per } \frac{1}{4} \text{ mm} \times 4 \times \text{cu. mm per cc} \times \text{flask vol.} \times \text{extra dil. ratio}}{\text{Cu. ft. per minute sampled} \times \text{sampling time}}$$

$$\frac{40.8 \times 4 \times 1000 \times 30 \times 5}{0.1 \times 5 \times 10^6} = 4.8 \text{ mppcf}$$

particles per cubic foot.

1. Brown, C.E. and Schrenk, H.H.: A Technique for Use of the Impinger Method. Bureau of Mines Information Circular 7026, 20 pp., 1938.

2. Brown, C. E.; Baum, L. A. H.; Yant, W.P.; and Schrenk, H.H.; Microprojection Method for Counting Impinger Dust Samples. Bureau of Mines Report of Investigations 3373, 9pp., 1938.

Even though the method is thus specified, it is possible to adhere to the method yet introduce variables which can affect the results it yields. Some of these will be mentioned in this section. In cases of known variables, it is suggested that until they are accommodated by modification or amendment of the method, the procedure be such that the count yielded be the same as that which would be obtained if an atmosphere of quartz dust, having the same number concentration and size distribution as the atmosphere actually sampled, were sampled and counted in water. In other words, if the method using water as a medium should permit detection of 60 percent of the microscopically visible quartz particles present in the air in a given situation, one should strive to detect also 60 percent of the particles if they should have densities other than that of quartz or be collected in media other than water.

Equipment

The microscope and its use are discussed in Section B-5. Suffice it to say here regarding the microscope that for standard method dust counting a laboratory grade optical microscope with a 16 millimeter, 10 power objective lens is used and that samples are counted by the light field technique.

Counting Cells

Since dust from the air is captured in liquid medium it is necessary to relate the number of particles from a measured volume of liquid to the number in a measured volume of air. The volume of air which passed through the impinger sampler is known from the sampling procedure. The number of particles captured from this air is then determined by counting those present in a measured fraction of the sampling liquid. This is done with a counting cell. A portion of the sampling medium containing dust is placed in a counting cell of known depth, particles are allowed to settle from the liquid to the floor of the cell for a certain period of time, then the particles on a measured area of the cell floor are counted. The area of the cell floor counted and the depth of the cell define the volume from which the counted particles settled. The characteristics of cells ordinarily used in counting are summarized in Table 1.

The Dunn cell is made up of three parts: a base plate, a cell plate, and a cover slip. The cell plate is a 1 mm thick glass plate ground on both sides with a round hole through the center. This hole forms the counting cell when the cell plate is sandwiched between the base plate and cover slip. The Dunn cell is relatively easy to clean because of its three piece construction and because there are no concave corners to trap dust.

Table 1. - Characteristics of Dust Counting Cells

Cell	Graticule Used	Cell Depth, mm	Area Usually Counted Per Field mm^2	Volume Usually Counted Per Field mm^3
Dunn	Whipple, Wardlow, or Page	1	0.25	0.25
Sedgewick-Rafter	"	1	0.25	0.25
Hatch	"	0.25	0.25	0.0625
Spencer Brightline Hemocytometer	Ruled in Cell	0.1	0.04	0.004

The Sedgewick-Rafter cell has 1 mm high glass or metal walls cemented to a base plate. This assembly, with a cover glass makes a rectangular cell. Cleaning is a bit more difficult than with Dunn cells because of the concave corners and the cemented joints.

Spencer Brightline hemocytometer cells were developed as blood counting cells. They have no chamber walls, as such; the liquid is held in place by its surface tension. The cell depth is 0.1 mm and the cell floor is lightly coated with rhodium through which a grid pattern is ruled. With the grid ruled on the floor of the cell, an eyepiece reticle to define the area counted is unnecessary. The cells must be handled with care to avoid damage to the rhodium surface; chemical cleaning solution should never be used.

Hatch cells are similar in design to Spencer hemocytometer cells except they have a depth of 0.25 mm and have no metal coating or ruled grid.

For precise work it is wise to measure counting cell depths with a micrometer rather than accept the nominal depth. Variations up to 20 percent of the nominal depth may be found.

Eyepiece Reticles

Reticles placed at the focal plane of the eyepiece are superimposed on the field of view of the microscope. Such reticles with grid patterns ruled upon them are used to define the areas of counting cells in which the dust particles are counted. These grids must be calibrated for each microscope and each lens with which they are used. To measure the size of the grid image, it is compared with a stage micrometer placed on the object stage. The stage micrometer is a plate on which are ruled, with considerable precision, fine lines spaced at 0.1 and 0.01 mm.

There are three common types of reticles used in dust counting. The original is the Whipple reticle (or disc), which consists of a large square subdivided into 100 squares. One square, adjacent

to the center is further divided into 25 squares. The 100 squares, when used with a 10x objective, subtended about 1 mm^2 on the microscope stage. The standard technique prescribes counting one quarter of this area, or 25 squares. On microscopes which have adjustable tube lengths, the body tube may be adjusted, using a stage micrometer, so that the reticle image will cover exactly 1 mm^2 of the cell floor. This serves only to somewhat simplify the arithmetic.

The grid of a Page reticle is simply one quarter, or twenty-five unit squares, of a Whipple reticle.

One serious drawback of the Whipple and Page reticles is that any dust which settles on them from the air will be in focus and appear in the image of the sample. They can be mistaken for part of the sample. However, these particles on the reticle will stay in focus when the objective is racked up and down, while the particles of the sample will go in and out of focus.

Nevertheless, dust on the reticle is undesirable, and the glass surfaces should be scrupulously cleaned when necessary. However, complete cleaning is nearly impossible, and the microscope manufacturers advise against too frequent opening of the eyepieces. Also, modern lenses bear anti-reflection coatings which are delicate and subject to abrasion. Hence, frequent cleaning and handling may reduce the quality of the optics of a microscope.

A third type of reticle, the Wardlow reticle, was developed to circumvent the problem of dust on the reticle surface. It is a ring with fine filaments stretched across to form the grid pattern. It is extremely difficult to fabricate, and is very, very fragile. At present, there is no commercial source for these reticles.

Counting Technique

The sampling medium should be free of particulate material - "free of particulate material" means that a blank sample, treated in the same way as were regular samples except that it was not used for sampling, shows less than about 6 particles per field count in a counting cell. This usually requires distillation and filtration through membrane filters. For best control two impinger flasks should be reserved as blanks, and carried with the sample flasks in the field.

Sample Ageing

There is an important time factor involved in making the counts. Dusts may agglomerate or flocculate in the liquid suspension, and some small particles may slowly go into solution. For this reason the standard method calls for counting impinger samples within 36 hours after sampling. It is preferable to count them within 24 hours, or even 12 hours, after sampling. This can be troublesome when in the field.

As a check on this ageing process, in one project, some 100 samples were counted first in the field within 24 hours of being taken, then counted again after returning to the laboratory about 1 to 2 weeks later. Calculations were made to determine the limits within which a data pair should fall if the counts were statistically the same. Only 25% of the counts were within the 50% limits, and only 55% were within the 90% limits. Also, there was a weighting toward lower counts in the aged samples; 65% of the samples showed lower counts in the laboratory than they did in the field.

Dilution of Sample

The liquid in the impinger sampling flask must be made up to a known volume while washing down the impinger nozzle. The result desired is a concentration in the liquid that will give a count of 50 to 200 particles per counting field. If, after making up to 15 ml for a light midget impinger sample, or as much as 25 ml for a heavy sample, further dilution is necessary to bring the count

below the desired maximum, dilute the liquid further in steps of not less than one volume of sample to ten volumes of dilution medium. Use at least 10% of the total sample to be diluted in making the dilutions, and in any case not less than 5 ml. Shake the sample well before making the dilution. Use dust-free sampling medium (actually the medium for which a blank count is known) as the diluting medium.

Preparation of Cells

The cells must be clean. First wash them with a good grade of laboratory glassware detergent and rinse well with tap water. Rinse several times with distilled water then rinse well with dust-free water. Finally, rinse well with dust-free alcohol, or, if the cell is to be used immediately, rinse with a portion of the sample. Cells to be stored may be wiped after the alcohol rinse, but should not be wiped after the sample rinse when ready for use. Use an exceptionally lint-free cloth such as clean linen, or a high grade of lens tissue, for wiping the cells. Ordinary lens tissue is not satisfactory. Cells may be stored also in dust-free alcohol.

To fill a cell, assemble it with the cover slip pushed to one side to leave an opening for the tip of a pipette. Shake the sample flask thoroughly to disperse the dust evenly in the liquid, and transfer a portion to the cell with a clean pipette. Getting the cover slip pushed into place without including air bubbles in the cell takes practice.

Settling Time

Common practice is to allow particles to settle 25 to 30 minutes per millimeter of cell depth before counting. For the count to include all particles of one micron equivalent diameter, for example, a sufficiently long settling time must be allowed for all particles of this size to settle through the depth of the cell. Calculation by the Stokes' law equation of the settling time for a 1 micron diameter regularly shaped quartz particle in water at 25°C yields a value of about 25 minutes per millimeter. The same calculation for ethyl alcohol as a medium yields a value of about 33 minutes per millimeter. It is reasonable to assume that these calculated values are the basis for the commonly used 25 to 30 minutes settling time for cells 1 millimeter in depth.

Not only alcohol and water, but their mixtures as well are used as the collecting medium in impinger sampling. These two materials when mixed form a complex and their mixtures do not follow the simple linear mole fraction-viscosity relationship, but have viscosities substantially greater than either of the components. The viscosity of a 50 percent ethyl alcohol-water mixture, for example, is 2 to 2½ times greater than that of either alcohol or water. One would calculate for such a mixture that fewer than half of the 1 micron quartz particles in the cell will settle 1 millimeter in the commonly employed 25 to 30 minute settling time.

The density of the material sampled has a very marked effect on the required settling time and seems to be ignored in many instances. Required settling time varies inversely with the difference between density of the settling particle and that of the liquid medium. One can calculate, for example, that a coal particle with a density of 1.4 grams per cubic centimeter (g/cc) will settle in water only about one-fourth as rapidly as a quartz particle of the same size with a density of 2.6 g/cc, and that only about one-tenth of the 1 micron particles of density 1.4 g/cc, in a 50 percent ethyl alcohol-water mixture, will settle to the floor of a 1 mm depth counting cell in 25 minutes. The use of shallow counting cells is indicated in such situations so reasonable settling time may be used.

If alcohol or mixtures of alcohol and water, are used as impinger sampling

media, the problem of settling time is compounded by the fact that under some psychrometric conditions the composition, and therefore the viscosity, of the sampling medium will change substantially during the course of sampling. One cannot, then, use the value for viscosity of the medium selected for sampling in the calculation of settling time. In studies of midget impinger sampling under controlled psychrometric conditions by Division of Occupational Health personnel it was found that the composition of water-ethyl alcohol mixtures changed relatively slowly with increasing sampling time in a cool environment, and was not greatly affected by humidity. At high ambient temperature the change in composition with increasing sampling time was marked, however, and was strongly influenced by humidity. The higher the temperature, the higher the humidity, and the higher the initial concentration of ethyl alcohol, the greater was the rate of change of composition during sampling. In a warm, humid environment (90°F, 85 percent relative humidity) a sampling medium which initially was commercial 95.5 percent ethyl alcohol was reduced by evaporation of alcohol and consensation and absorption of water to about 60 percent alcohol after 20 minutes of sampling.

Studies of change in composition of other alcohol-water mixtures during sampling under various psychrometric conditions have not been made. Examination of vapor pressure data for alcohols indicates that normal propyl alcohol-water mixtures are unlikely to change as markedly in composition as are mixtures of ethyl alcohol and water under the same psychrometric and sampling conditions, while isopropyl alcohol-water mixtures are expected to change in a manner intermediate between the two. Mixtures of normal or isopropyl alcohol in water, like those of ethyl alcohol and water, show positive deviation from the mole fraction-viscosity relationship of ideal mixtures. The viscosities of these alcohols and their mixtures with water are substantially higher than those of ethyl alcohol and its water mixtures. This results in proportionately longer calculated settling times for dust particles than are obtained for corresponding ethyl alcohol-water mixtures at the same temperatures.

Thus, the counting of samples with a fixed settling time without regard to medium viscosity or particle and medium densities, can, under certain circumstances lead to substantially different results from a pair of samples collected in different media even though the samples may contain the same numbers of particles. This variation will be of little practical significance if the sampling medium is water or ethyl alcohol and not a mixture of the two, if the samples are from cool or moderate environments, if the samples are of short duration, if the densities of the dust particles are about the same or greater than that of quartz, and if the particle size distribution of the sample is such that there is not a preponderance of particles very near in size to the lower limit of detection by the optical system used in counting. On the other hand, great variation in counts from different media may occur if several of these conditions are not met and arbitrary settling times of 20 to 30 minutes per millimeter are used. In the absence of other standard procedures it is suggested that settling times be so chosen that the counts yielded will be the same as would have been obtained had the particles been quartz settling in water for 30 minutes.

The problem of counting variation is further compounded by the fact that some observers with high visual acuity, and by careful adjustment of the optical system specified for the method, can detect some particles as small as about 0.5 micron in size; others may seldom detect particles smaller than 1 micron. Because the impinger sample counting technique includes all of the smallest particles which can be detected, visual acuity of the observer is a factor and

it appears that inter-observer variance cannot be eliminated except by the selection of counting personnel.

Using a hand talley, count all the particles in the 25 squares of the field selected. The smaller particles are difficult to see, look carefully to make sure that all of them are counted. Count in a systematic fashion, somewhat like reading a printed page - jumping around the field will cause the observer to lose track of which particles have been counted, and lead to spurious counts.

Count 5 fields, selected at random, in each cell. Random selection means that the fields are not chosen, since conscious or subconscious biases in choosing may alter the count. A good practice is to remove the eye from the eyepiece while moving the stage from one field to the next.

Calculation of Concentration

With the average particle count per field known the concentration of dust in terms of millions of particles captured per cubic foot of air sampled (mppcf) can be calculated. Although the calculation is shown in the description of the standard method, it is repeated here in slightly different form for emphasis. Known are:

1. Average particle count per field (field count)
2. Average blank count per field (blank count)
3. Cell depth in mm
4. Field area in mm^2
5. Final volume of liquid in impinger flask, ml
6. Volume of air sampled (rate in cfm multiplied by duration of sample = ft^3)

One may calculate then:

$$\frac{\text{Total number of particles in sample}}{\text{Sample air volume, } ft^3} =$$

$$\text{particles}/ft^3$$

This quotient, particles per cubic foot, divided by the factor 10^6 yields the number of millions of particles per cubic foot of air, mppcf. The known factors listed above may be used for this calculation:

$$\frac{(\text{field count-blank count, particles})}{(\text{field area, } mm^2)\,(\text{cell depth, mm})} \times$$

$$\frac{(1000\ mm^3)}{(\text{one ml dilute sample})} \times$$

$$\frac{(\text{total ml sample in impinger flask})}{(\text{vol. of air sampled, } ft^3)\ 10^6} =$$

$$\text{mppcf}$$

In these equations one can see that units may be alternately cancelled from the numerator and the denominator to yield a number in the units of mppcf.

$$\frac{(\text{Field count - blank})}{(\text{area in } mm^2)(\text{depth in mm})} = \text{parts}/mm^3$$

$$\frac{(1000\ mm^3)}{(1\ ml)} = \frac{mm^3}{ml}$$

$$(\text{parts}/mm^3)(mm^3/ml)(\text{dil factor}) \times$$

$$(\text{tot ml in imp}) = (\text{tot parts in samp})$$

$$\frac{\text{total parts in sample}}{(\text{vol air, } ft^3)\ 10^6} = \text{mppcf}$$

Statistics of Counting

Another variable in the method of counting dust is inherent in the distribution of particles in the counting cell. The statistics defining this distribution have been reported by Chapman and Ruhf[2]. Ordinarily, two cells from each impinger sample are counted; marked disagreement in counts is an indication of counting or other error. By use of the statistical methods reported by Chapman and Ruhf, two series of curves have been calculated by which the precision and reliability of two cell counts of the same impinger sample can be judged. They are based only upon the statistical distribution of particles in the cell and do not include other counting variables. These curves are shown in Figure 1.

The family of curves with positive slope permits one to judge the likehood of a blunder in the counting of one of the pair of cells. They represent the expected differences, within selected limits, of counts from two cells of the same sample and are calculated by the equation:

$$t = \frac{X_1 - X_2}{\sigma\Delta x}$$

Where:

$$\sigma\Delta x = \sqrt{\frac{\sigma x_1^{\,2}}{N_1} + \frac{\sigma x_2^{\,2}}{N_2}}$$

$$= \sqrt{X_1 + X_2} \quad \text{When } (N_1 = N_2 = 1)$$

t = The ratio x/σ for defining normal distribution areas (e.g. for the limit for agreement ½ of the time the fraction of the total normal curve area is 0.50 and a table of normal areas yields the value t = 0.675)

X = Number of particles counted per cell

$\sigma\Delta x$ = Standard deviation of difference in cell counts, Poisson distribution

σx = Standard deviation of particle count per cell

N = Number of counting trials per cell (1 in this case)

To illustrate, the point in Figure 1 defined by a high cell count of 60, paired with a low cell count of 40, falls to the left of the locus which limits the range within which pairs of counts are expected to fall nine-tenths of the time. Disagreement of this magnitude resulting only from distribution of particles in the counting cell is expected less than one-tenth of the time in correctly counted pairs of cells. In the absence of other criteria it is suggested that a third cell be counted when counts of the first two fall outside the nine-tenths limit.

The reliability of the count for a sample depends upon the total number of particles counted in all cells from the sample. When judgment has been made that the counts of a pair of cells are acceptable, the reliability of their mean can be judged by use of the loci having negative slopes in Figure 1. The values shown are reported by Chapman and Ruhf and may be calculated by the equation:

$$t = \frac{M_x - \bar{X}}{\sigma m}$$

$$\sigma m = \frac{\sigma x}{\sqrt{N}} = \sqrt{\frac{\bar{X}}{N}}$$

t = The ratio x/σ for defining normal distribution areas. For 95% level the value t = 2 is used. (t = 1.96 is a more precise value).

M_x = True mean for number of particles counted per cell.

$\bar{X}$ = Observed average number of particles counted per cell.

σm = Standard deviation of the mean particle count per cell, Poisson distribution.

N = Number of cells counted (2 in this case).

In the illustration cited in the preceding paragraph the mean cell count is 50; the curve in Figure 1 shows that the true mean of these cell counts is expected to be within about ±20 percent of this observed mean 95 percent of the time. A mean cell count of 200, or a total count of 400, in a pair of cells with acceptable agreement will have a true mean within ± 10 percent of the observed mean 95 percent of the time. About 400 particles, the actual number depending on the needed reliability, seems to be a reasonable minimum number to be counted in an impinger sample under ordinary circumstances. Use of curves such as those of Figure 1 permits definition and limitation of the distribution variable in counting.

References:

1. Transactions of the Fifth Annual Meeting of the National Conference of Governmental Industrial Hygienists. Subcommittee on Standard Methods, Washington, D.C., 1942.

2. Chapman, H. M. and R. C. Ruhf. Dust Counting Reliability, Amer. Ind. Hyg. Assn. Quarterly, 16: 201 (1955).

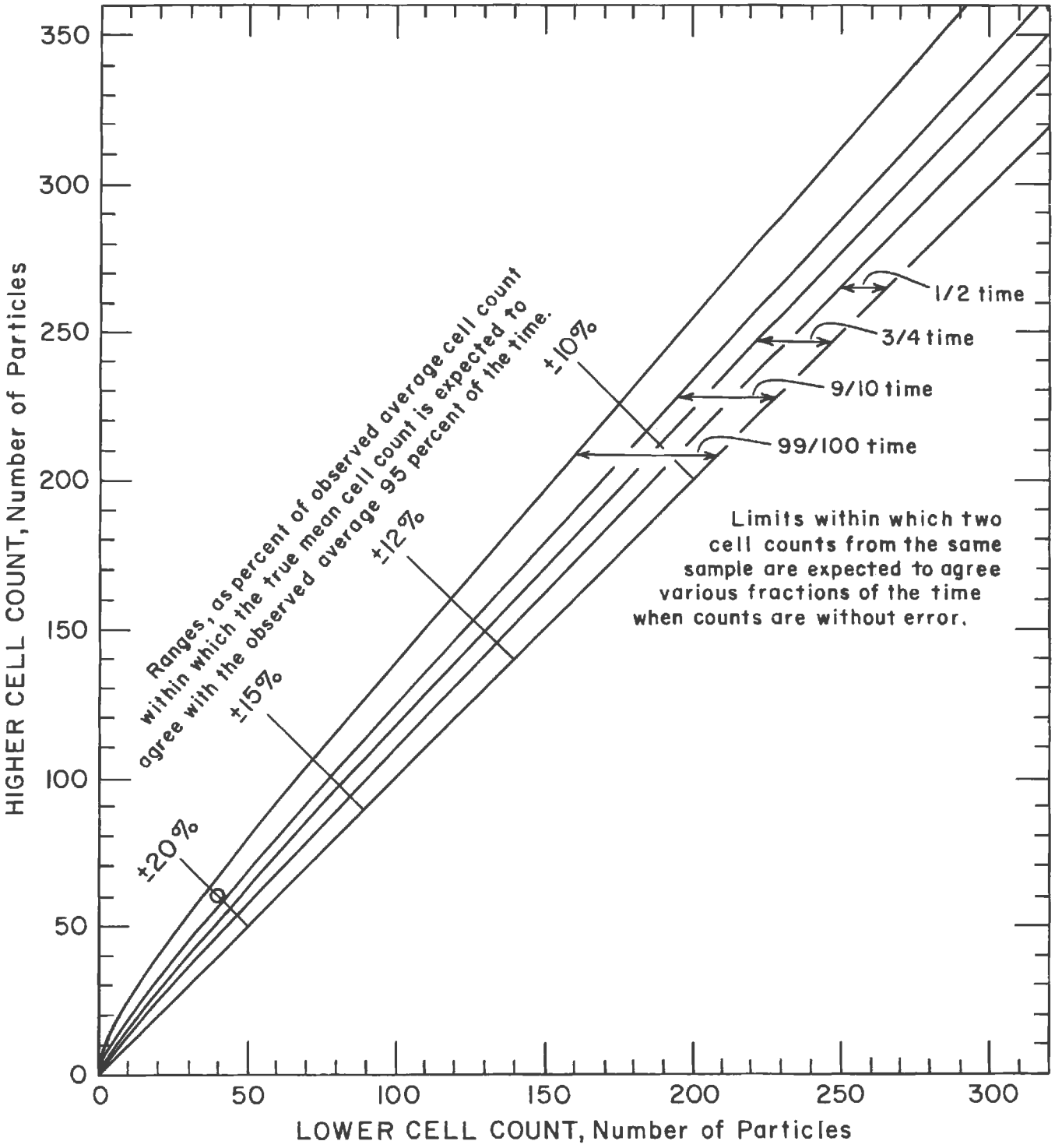


Figure 1. - Precision and Reliability of Independent Counts of Two Cells from the Same Impinger Sample.

DETERMINATION OF PARTICLE SIZE

Introduction

Very early in recorded history the high incidence of respiratory disease among workers in occupations such as mining and stone cutting was noted. The cause of such disease was variously described as pestilential air, the vapors of metals, and metal emanations. Later it was recognized that dust was often the causative agent and that all dusts did not exhibit the same effect upon the exposed workers. Further, it was observed that, in certain instances where visible dust was practically absent from the air, workers still contracted pulmonary disease from dust exposure[1]. Extensive research has indicated that the dusts causing pneumoconiosis, a condition of chronic inflammation of the lungs, are limited in their size. These size limitations indicate that the particles causing lung disease are below the range of visibility which explains why work atmospheres appearing to be only "slightly dusty"[2] have often contained extremely hazardous concentrations of dust. So that the potential hazard of a work environment might be evaluated more comprehensively, methods of particle sizing have been developed. Particle sizing techniques are necessary to arrive at a size distribution of the air-borne dust, which supplements the results of dust count data in evaluating an exposure to potentially hazardous concentrations of respirable dust.

Representation of Size

The size of a dust particle is expressed in terms of microns or a fraction thereof. One micron (μ) is 10^{-3} millimeter (0.001 mm) or 10^{-6} meter (0.000001 m). Figure 1 indicates the range of sizes of many of the contaminants encountered by the industrial hygienist. It should be noted that the limit of visibility for people with normal vision is about 100 microns, a range which excludes contaminants of hygienic significance. This fact, along with the observations and investigations mentioned previously, points out the necessity of sizing if a quantitative picture of the dust exposure is to be obtained.

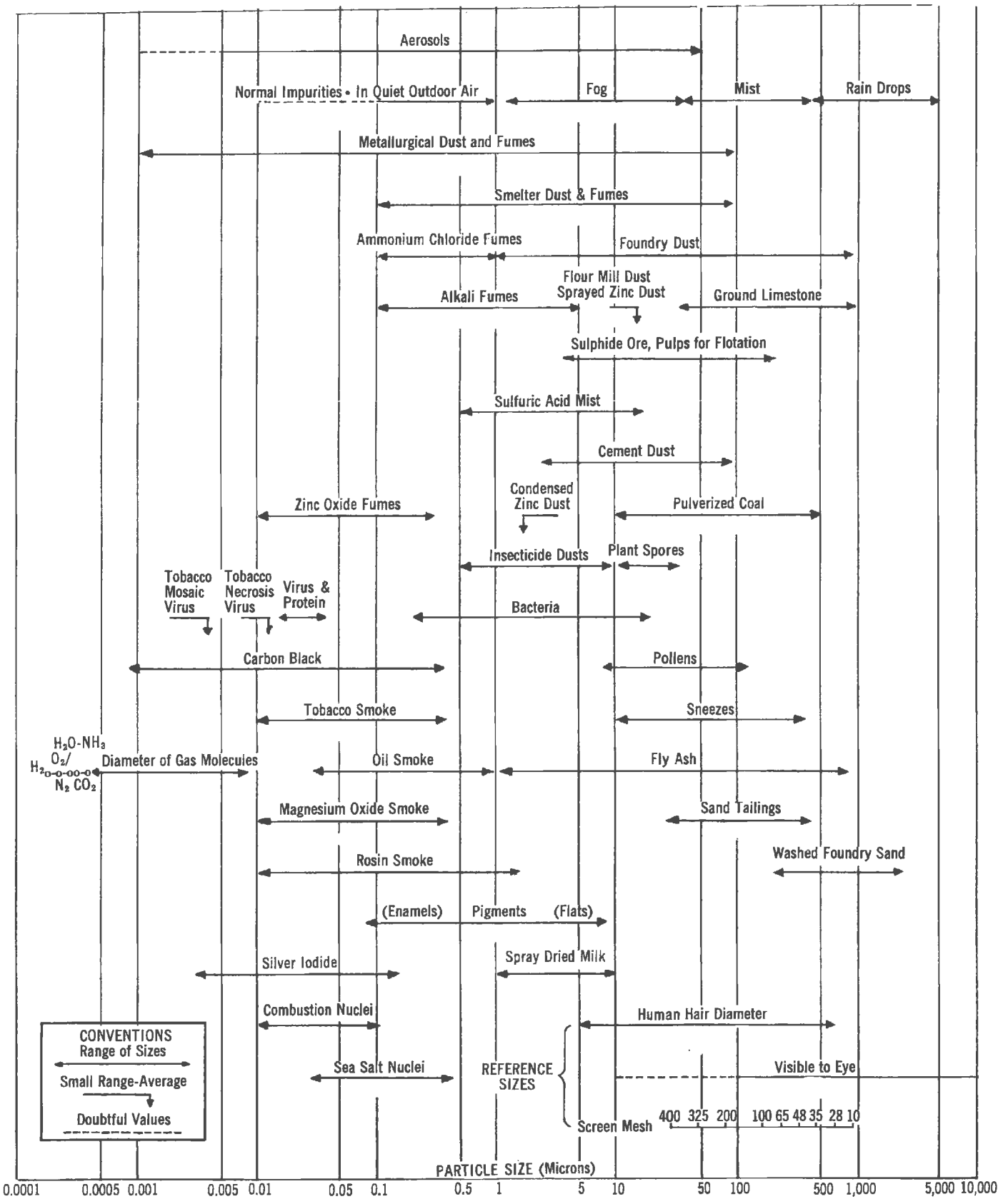
Deposition of Particles in the Lung

There are three mechanisms which affect the removal of particles from the inhaled air and deposit them in the respiratory system. The first of these being the *impaction* of particles along the walls of the upper respiratory system. As inspiration progresses the total cross-sectional area of the air passages increases. Thus, with penetration deeper into the respiratory system during inspiration, the velocity of the particles decreases and some particles are deposited by *sedimentation*. The final physical force affecting deposition is *diffusion*, which occurs in the pulmonary air spaces as a result of bombardment of very small particles by vibrating gas molecules.

Many investigations have been conducted in attempts to determine particle sizes, or particle size ranges of dusts which are of greatest significance in the cause of pulmonary diseases resulting from dust exposures[1]. It is not too presumptious to state that the dust in question must be deposited in the respiratory tract if it is to cause lung disease. Hatch and Gross[3] offer the following compendium of the characteristics of size in the deposition of dust at various depths within the respiratory system:

"1. Particles larger than 10 μ are essentially all removed in the nasal chamber and therefore have little probability of penetrating to the lungs. Upper respiratory efficiency drops off as size

Prepared by: Samuel B. McKee and
Richard D. Fulwiler



(Chart reproduced by courtesy of Mine Safety Appliances Company).
Figure 1. - The Size of Air-Borne Contaminants

decreases and becomes essentially zero at about 1 μ.

2. The efficiency of particle removal is high in the pulmonary air spaces, being essentially 100% down to around 2 μ. Below this size it falls off to a minimum at about ½ μ. It then increases again as the force of precipitation by diffusion increases with further reduction in size.

3. The percentage of penetration of particles into the pulmonary air spaces rises from essentially zero at 10 μ to a maximum at and below about 1 μ where it equals the fraction of tidal air which reaches the lungs.

4. The percentage of inhaled particles which penetrate to and are deposited in the pulmonary air spaces has a maximum value between 1 and 2 μ. Larger particles are deposited in the lungs in lesser degree because they are trapped higher up in the respiratory tract. Lung deposition of finer particles falls off because the local efficiency of removal decreases as size goes down below 2 μ.

5. Below ½ μ, the probability of deposition in the pulmonary air spaces rises in proportion to the increase in the force of precipitation by diffusion with decreasing size.

6. The relative amount deposited and the distribution of the collected particles in the respiratory system changes with breathing frequency and tidal volume. Upper respiratory trapping increases as the rate of inspired air flow goes up with faster breathing frequency. Magnitude of deep lung deposition increases with slow, deep breathing because of the larger fraction of tidal air which reaches the pulmonary spaces and the longer transit time of air into and out of the lungs."

These conclusions, based partially on theory and partially on experimental data, indicate a very definite relationship between particle size and degree of deposition in the lung. A logical assumption can be made that lung damage is related either directly or indirectly to deposition of dust in the lung, therefore particle size determinations are essential in the evaluation of dust hazards(1).

Properties of Small Particles

In addition to indicating the respirability of a dust, particle sizes and particle size distributions are important because of the effects of size on the properties of the particles. The occupational health scientist should be aware of the effects of size on: (1) Total surface activity; (2) free falling speed; (3) solubility; and (4) optical effects.

Total surface activity. This is the cumulative effect of several properties on the surface of a particle. These properties include surface area, crystalline or non-crystalline structure, adsorption, and chemical reactivity. No single component of total surface activity explains the toxicity of a dust. It is well known that there is a tremendous increase of surface area per unit volume with a decrease in diameter. However, there is no conclusive proof that surface area per unit volume is a significant property of a dust causing lung disease. Beryllium has a high surface area per unit volume and causes lung disease, whereas charcoal also has a high surface area per unit volume and is not nearly physiologically as reactive as beryllium.

The significance of crystallinity can be illustrated by the fact that crystalline silica is known to cause silicosis,

whereas amorphous silica is not considered to be a causative agent of this disease. Adsorptive properties also are strongly influenced by the crystallinity or noncrystallinity of a substance.

Chemical reactivity also must be considered as part of the total surface activity since chemical reactions between liquids and solids normally take place at the surface. The rate of reaction increases with a decrease in particle size.

Free falling speed. Stokes' equation,

$$V = \frac{gd^2(\sigma p - \sigma a)}{18\mu}$$

Where: V = terminal settling velocity or free falling speed
g = gravitational acceleration
d = particle diameter
σp = particle specific gravity
σa = specific gravity of air
μ = viscosity of air or material through which particle is passing.

indicates that the free falling speed (terminal settling velocity) of a particle decreases as the square of the particle size. Therefore, small particles tend to remain in a breathing zone for a longer period of time than do large particles. The relationships between free falling speed and the particle size of air-borne dusts indicate that particles with a given falling speed cover a wide range of sizes for the same dusts[4]. This aerodynamic property is somewhat significant in defining the respirability of dust in that it compensates for aggregates and irregularly shaped particles. However, there is not sufficient information available to the industrial hygienist at this time to allow him to evaluate the potential hazard of a dust exposure on the basis of this characteristic. Sedimentation methods of sizing, where the time for particles to settle a given distance is determined, utilize Stokes' equation to arrive at an equivalent particle diameter. Some of the limitations in sedimentation analysis are:

1. There is interaction of particles in a fluid. It is noted that the concentration of the particles affect the falling speed.[5]
2. It is difficult to adequately disperse the particles in a suspension.
3. The sizes are small, less than 0.5 μ, thus making it necessary to apply correction factors to Stokes' equation.

The diameters determined from sedimentation data and Stokes' law will agree with the diameter determined by optical microscopy only if the particles are spheres. Most industrial dust is not spherical but is composed of irregularly shaped particles. A shape factor is used as a factor of proportionality between the two methods for determining particle size.

Solubility. The rate of solution and the solubility both increase with decreasing particle size.

Optical effects. This property is not of physiological significance but is used in some optical analyzers for determining particle size distributions. The amount of light scattered by particles (greater than about 0.5 μ) shows a marked increase as the particle size increases.

Sampling for Size Analysis

Various properties of small particles, which have been discussed in previous paragraphs, are utilized when sampling for size analysis. Methods for obtaining samples which are not entirely dependent on optical microscopic evaluation will be discussed first, followed by methods of sampling which lend themselves to microscopic sizing.

Automatic Sampling and Sizing

Automatic sampling and sizing is a relatively new innovation in the evaluation of the dust environment. Automatic instruments, which operate either as conductrimetric or photoelectric devices[6] were designed initially to count the number of air-borne particles but they have been modified to deliver data concerning particle size distribution as well. One commercially available instrument using the conductrimetric principle permits the operator to adjust the discrimination threshold to yield count data at any number of different particle size ranges.[7] The discrimination of particles of various sizes is more of a problem in the photoelectric devices since these instruments cannot indicate whether pulses arise from one particle, or two overlapping particles, or from only a portion of a particle which is passing through the light beam.[8]

At the present time, automatic sizing instruments are still in the developmental stages. However, it will be extremely advantageous to those evaluating dust environments when the tedious practice of counting and sizing by optical microscopic technique can be facilitated by a valid method utilizing some principle of automation.

Size Classification

Size classification, utilizing multistage sampling procedures, has been developed for two reasons. The first being an attempt to simulate upper and lower lung deposition[9,10] and the second being an attempt to obtain information on the size distribution of the dust. The clearing mechanism of the upper respiratory tract is believed to function at a rate greater than the clearing mechanism of the lower respiratory tract. This is one reason why more significance has been placed on that fraction of dust deposited in the pulmonary air spaces of the lower respiratory tract. The final stage of multistage sampling devices, that which simulates the pulmonary air spaces, usually requires some direct method of size evaluation if a size distribution is desired. Methods of this type will be discussed in a later portion of this Section. Examples of such devices requiring direct methods of sizing in the final evaluation of the sample collected include: (1) A horizontal elutriator developed by Davies[11] followed by a filter; (2) a cyclone[12] followed by a filter; and (3) a pre-impinger[13] followed by an impinger as the second stage.

The topic of size selective samplers is covered in more detail in Section B-2. Suffice it to say that size classification is an approach to the quantitation of size fractions of dust which are of hygienic interest.

Sampling for Optical Sizing

Samples to be sized microscopically are usually collected in the form of a dry powder or from the air-borne dust. When the samples come from a dry powder a representative portion of a well mixed sample is prepared for microscopic examination. Methods of dispersing and mounting a sample of a powder are described by Herdon.[8] Collection of a sample from a dust cloud is accomplished with a suitable sampling instrument, which should not alter the size of the particles as they exist in the dust cloud. Impactor type instruments (cascade impactors) break up agglomerates and may shatter some particles. Thermal precipitators and membrane filters are useful for collecting samples of air-borne dust for particle size measurements.

Microscopic Methods

In preparing slides for particle size determination, a random distribution of particles with respect to size is assumed. It is further assumed that the size distribution determined from a

series of random fields will be representative of the sample. If the deposition of particles with respect to size is not of a random nature, care must be exercised to measure all particles in a mathematically representative portion of the deposit. This particular problem will be encountered in the evaluation of slides from instruments such as the Owens jet dust counter or thermal precipitator. Such instruments are somewhat selective in that particles of various sizes are deposited at different locations on the slide. On a thermal precipitator slide, for example, it will be found that the mean particle size varies across the width of the deposit. A representative size distribution on such a slide may be obtained by measuring all particles in a strip of uniform width extending across the entire dust deposit and perpendicular to the major axis of the deposit.

Every visible particle in the selected field should be measured. An observer must overcome the unconscious tendency to focus the microscope on and measure only the larger, more easily seen particles. On a membrane filter sample, for example, where all particles are not in the same plane, the observer should manipulate the focus so as to measure all particles that appear in the selected field throughout the depth of the deposit. This is critical for an accurate determination of size distribution.

Most optical particle size determinations are made with microscopes using an oil immersion objective lens. For maximum definition and to achieve the limit of resolution, it is necessary in most cases, to apply immersion oil between the top of the condenser and the lower surface of the sample slide and on the objective lens of the microscope. In other words, oil immersion requirements will not be satisfied if a cover slip is placed over a dry mount and immersion oil is used only between the cover slip and the objective lens. As a general rule, there should be no air-glass interfaces between the condenser and the microscope objective. Discussion of this matter can be found in any standard text on microscopy and in manufacturers' literature supplied with the microscopes.

Particle Diameter

Particles are "sized" by comparing a characteristic length of the particle with some known distance observed in the eyepiece. Since irregularly shaped particles have no characteristic length to determine their size as does the diameter of a sphere, an arbitrary length is chosen. Various methods are used for measuring the diameters of particles as shown in Figures 2 and 3. Martin's diameter is the length of a horizontal line which divides the area of the particle in half (Figure 2). Feret's diameter is the length of the longest horizontal dimension (Figure 2) and is generally used in particle size measurements. When a sufficient number of particles are measured and the particles have random orientation on the slide, Feret's diameter is used as shown in Figure 3.

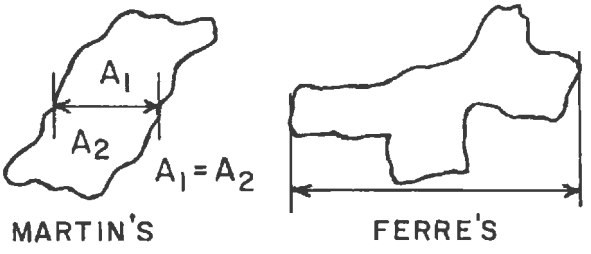


Figure 2.- Martin's and Feret's Methods for Measuring Diameters of Particles.

The known distance observed in the eyepiece is provided by either a Filar micrometer or eyepiece reticle. Both of these devices can be calibrated with a stage micrometer, which is ruled in tenths and hundredths of a millimeter. A calibration must be made for each eyepiece-objective lens combination.

Filar micrometer. The Filar micrometer consists of an eyepiece with a movable hair activated by a calibrated micrometer screw. When measuring a particle with a Filar micrometer, it is good

practice to approach the particle in the same direction as the particle is to be measured. This reduces or eliminates the inaccuracies which result from play in the mechanical linkage between the cross hair and the micrometer scale. Since the cross hair has a significant width, the observer also should use the same side of the cross hair as an index, especially when measuring small particles.

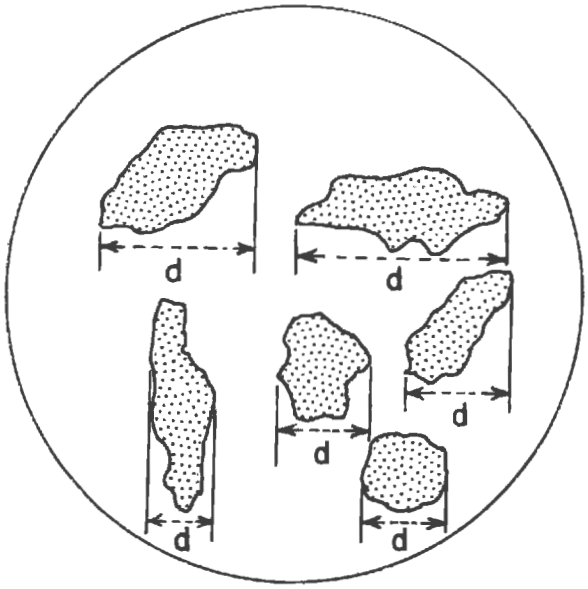


Figure 3. - Statistical Distribution of Particles Showing Feret's Diameter.

Eyepiece reticles. The eyepiece reticle consists of a glass plate on which various geometric figures are inscribed and it is placed in the focal plane of the eyepiece. One such reticle (see Figure 4) has as indices a series of circles and a logarithmic scale for comparing the size of particles. The circles are numbered 1 through 9 and the diameter of each circle is defined by the equation:

$$D = L\sqrt{2^N}$$

Where: D = diameter of circle
N = number of circle
L = unit of length

The logarithmic scales show values for N for the measurement of particles longer than the number 9 circle. The dimensional value of L, however, is dependent upon the optical system used. The large rectangle in this reticle is 200 L units in length and 100 L units in width. Thus, calibration of the eyepiece is easily accomplished by determining the number of microns on a stage micrometer included in either of these dimensions.

Microprojector

A microprojector consists of a microscope equipped with a projection prism accessory, a projection light source, and a viewing screen. The projected images of particles may be measured directly on the viewing screen by use of a calibrated scale. While some loss of definition is suffered in projection, the method has the advantage of being less tiring for most observers than direct microscopic observation. A detailed discussion on the design and use of microprojectors can be found in the literature.(14)

Photographic Technique

A microscope may be fitted with a camera and photographs of the microscope field may be obtained. Such photographs, or photomicrographs, of dust samples may be utilized for particle size determination. A means of calibration must be provided to permit measurements of the

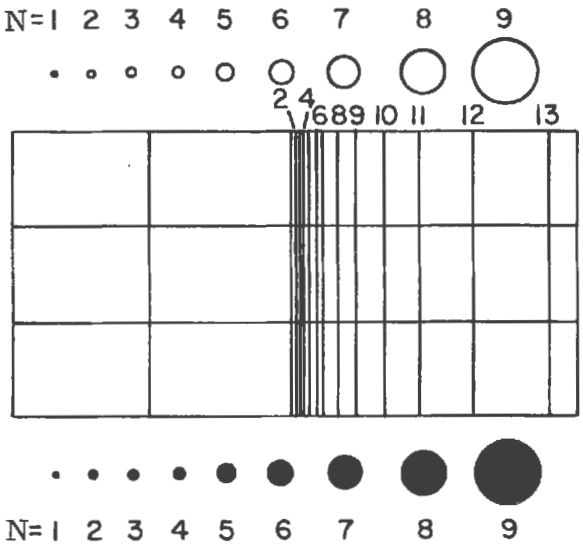


Figure 4. - Eyepiece Reticle.

SAMPLE NO. DATE EXAMINED:

DESCRIPTION: BY:

CIRCLE	PARTICLE TALLY	TOTAL
1	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓✓	15
2	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓	19
3	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓	24
4	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓	44
5	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓	42
6	✓✓✓✓✓ ✓✓✓✓✓ ✓✓✓✓✓ ✓✓	27
7	✓✓✓✓✓ ✓✓✓✓✓ ✓	16
8	✓✓✓✓✓ ✓✓✓	8
9	✓✓✓	3
10	✓	1
11	✓	1
12		
13		
>13		

CIRCLE NO.	1	2	3	4	5	6	7	8	9	10	11	12	13	>13
CIRCLE DIAM.	1.13	1.60	2.26	3.20	4.52	6.40	9.04	12.80	18.08	25.60	36.16	51.20	72.32	
NO. OF PARTICLES	15	19	24	44	42	27	16	8	3	1	1	0	0	0
CUMULATIVE TOTAL	15	34	58	102	144	171	187	195	198	199	200			
CUMULATIVE %	7.5	17.0	29	51	72	85.5	93.5	97.5	99.0	99.5	100			

Figure 5. - Particle Size Data Sheet.

photographed particles. An advantage of the method is that a number of photomicrographs can be taken and evaluated at leisure; a disadvantage is that irregularities in the photographic process, possibly undetected, may preclude or invalidate evaluation of the sample.

Electron Microscope

The electron microscope is a better research instrument for detailed determination of small particle sizes than the optical microscope. The use of this instrument is highly technical and requires an individual with special training for its successful operation. The electron microscope does not have the dimensional limitations of optical systems, and can resolve dust particles several orders of magnitude smaller than can an optical microscope. Since this instrument is not generally available for routine industrial hygiene work, it appears that its greatest value in particle size determination is in the area of basic research.

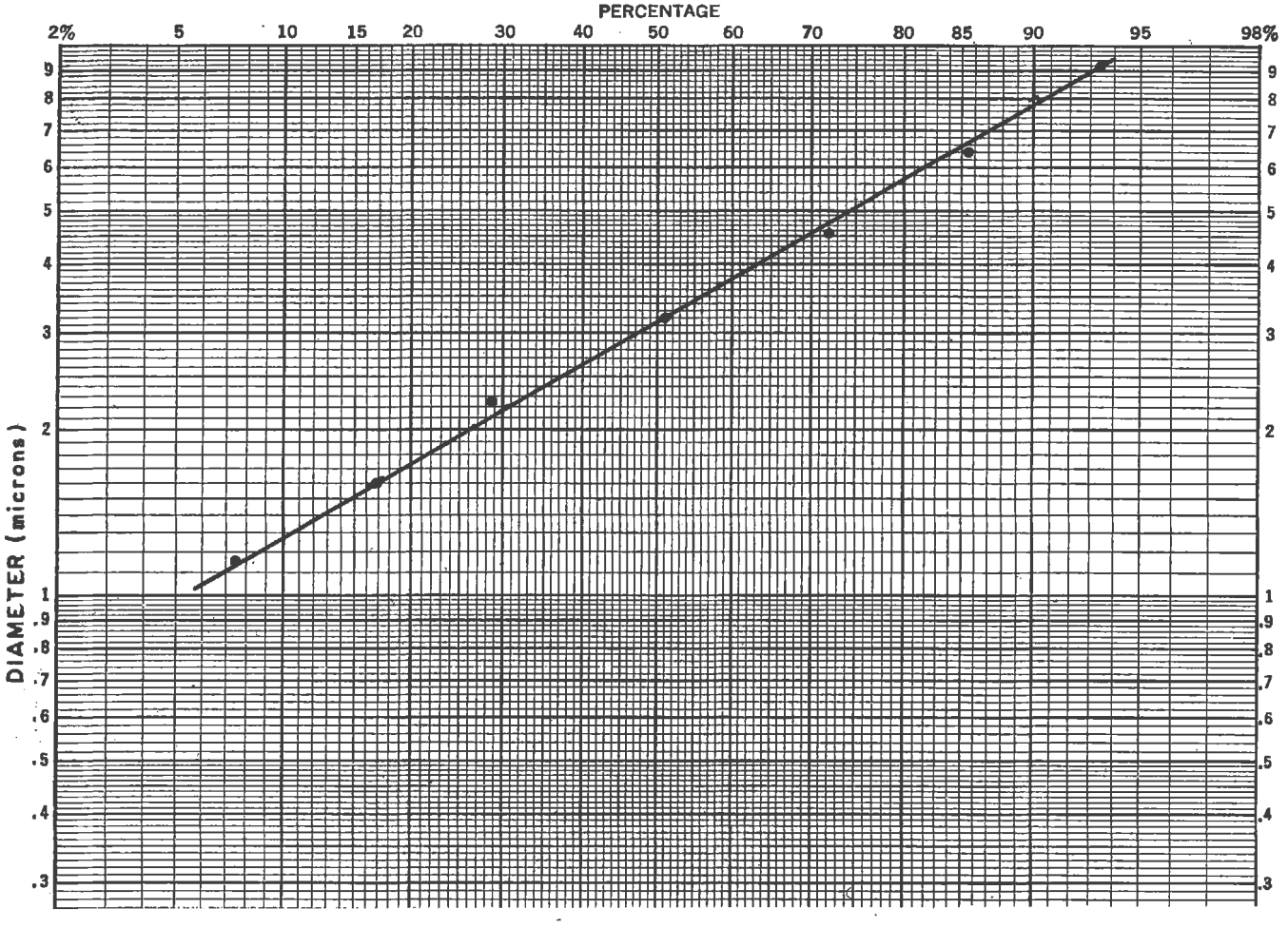


Figure 6. - Data from Figure 5 Plotted on Log Probability Paper.

Presentation of Data

Data obtained from sizing a dust sample with a Porton reticle is shown on the data sheet (Figure 5). First the diameter of the circles is calculated using the formula given above and the calibration of the eyepiece reticle.

The height of the reticle (100L) is

0.080 mm. Then one L unit is 0.00080 mm. Since 1 mm is 1000μ, then L is 0.80μ. The diameter of the number one circle is given by:

$$D_1 = L\sqrt{2^N} \text{ where } D_1 = 0.80\sqrt{2^1} =$$

$$0.80\sqrt{2} = (0.80)(1.41) \therefore D_1 = 1.13\mu$$

Similarly for circle number 2

$$D_2 = 0.80\sqrt{2^2} = (0.80)(2) = 1.60\mu$$

and for circle number 3

$$D_3 = 0.80\sqrt{2^3} = (0.80)(2)\sqrt{2} =$$

$$(0.80)(2)(1.41) = 2.26\mu$$

and for circle number 4

$$D_4 = 0.80\sqrt{2^4} = (0.80)(2)(2) =$$

$$(2)(1.60) = 3.20\mu$$

It will be noted that the diameter of the #3 circle is twice that of the #1 circle and that the diameter of the #4 circle is twice that of the #2 circle. The diameter of the next higher even or odd numbered circle is twice the diameter of the preceding even or odd numbered circle.

The number of particles found to match each circle size is entered in the total column (Figure 5) and also in the appropriate space at the bottom of the page. Next, the cumulative total is determined by adding the number of particles matching circle #2 to those matching #1, those matching #3 to the cumulative total of #2, etc. The cumulative percent is then calculated for each size by dividing the cumulative number of particles found in each size range by the total number of particles counted. The cumulative percent is then plotted versus circle size on log-probability paper as shown in Figure 6.

The logarithm of size is used because most industrial dust is produced by breaking larger particles into smaller ones. It can be predicted that dust particles generated in this manner will have a size distribution which follow the log-normal distribution law.[15] If this law is obeyed a plot of log of size versus cumulative fraction will result in a straight line.

Two important pieces of information may be obtained from the straight line: the geometric mean diameter, and the standard geometric deviation. The geometric mean diameter is the size of 50 cumulative percent. Half of the particles are smaller than the geometric mean diameter and half are larger. The geometric mean diameter determined from the log-probability plot for the data listed in Figure 5 is 3.15 microns. The geometric standard deviation is a measure of how much the dust particles differ in size from the geometric mean diameter. The standard geometric deviation (σg) is determined by the ratio:

$$\sigma g = \frac{84.13 \text{ percent size}}{50 \text{ percent size}} =$$

$$\frac{50 \text{ percent size}}{15.87 \text{ percent size}}$$

For the example cited the standard geometric deviation is:

$$\frac{6.40\mu}{3.15\mu} = \frac{3.15\mu}{1.55\mu} = 2.0$$

A convenient table, showing the mathematical relationships between the geometric mean average diameter and the arithmetic mean, specific surface, surface area, volume, and surface area per unit volume average diameters appears in the text INDUSTRIAL DUST, by Drinker and Hatch.[16] Calculation of these values requires data in terms of frequency by count as would be obtained by direct microscopic measurement. In sedimentation or elutriation procedures, however, particle size data are usually determined as percentage by weight. It has been shown[17] that the standard geometric deviation by weight is the

same as the standard geometric deviation by count. The following relationship exists between the geometric mean diameter by weight and the geometric mean diameter by count:

$$\text{Log } M_g = \log M'_g - 6.9078 \log^2 \sigma g$$

Where: M_g = geometric mean by count

M'_g = geometric mean by weight

σg = standard geometric deviation by count or weight

References

1. Harris, R. L.: The Determination of Particle Size, Distribution in Industrial Dust. Public Health Service Publication No. 614, Section B-7A.

2. Johnstone, R. T. and Miller, S. E.: Ch. 12, The Pneumoconioses in Occupational Disease and Industrial Medicine, W. B. Saunders Co., pp 199-251, 1960.

3. Hatch, T. F. and Gross, P.: Ch. 4, Experimental Studies on Deposition of Inhaled Aerosols, in Pulmonary Deposition and Retention of Inhaled Aerosols. Academic Press, pp 45-68, 1965.

4. Hamilton, R. J.: The Relation Between Free Falling Speed and Particle Size of Air-borne Dust. Brit. J. Applied Physics, Supplement No. 3, pp 590-593 (1954).

5. Kaye, B.: Sizing Solid Particles. International Science and Technology, March, p. 40, 1964.

6. Channell, J. K. and Hanna, R. J.: Experience with Light Scattering Particle Counters. Arch. of Environmental Health, 6:386 (1963).

7. Kinsman, S.: Particle Size Distribution Measurement, Ceramic Age, pp 53-54, August, 1962.

8. Herdan, G.: Small Particle Statistics, Academic Press, Inc., Butterworths, London, 1960.

9. Harris, W.B. and Eisenbud, M.: Dust Sampler Which Simulates Upper and Lower Lung Deposition. AMA Arch. Ind. Hyg. Occup. Med. 8: 446 (1953).

10. Morrow, P. E.: Evaluation of Inhalation Hazards Based Upon the Respirable Dust Concept and the Philosophy and Application of Selective Sampling. Am. Ind. Hyg. Assn. J., 25: 213 (1964).

11. Davies, C. N.: Dust Sampling and Lung Disease, Brit. J. Ind. Med., 9: 120 (1952).

12. Watson, N. H.: Dust Sampling to Simulate the Human Lung, Brit. J. Ind. Med., 10: 93 (1953).

13. May, K. R. and Druett, H. A.: The Pre-Impinger, A Selective Aerosol Sampler, Brit. J. Ind. Med., 10: 142 (1953).

14. Anderson, F. G.: A Technique for Counting and Sizing Dust Samples with a Microprojector, Amer. Ind. Hyg. Assn. J. 23: 330-336 (1962).

15. Epstein, B.: The Mathematical Description of Certain Breakage Mechanisms Leading to Logarithmics - Normal Distributions, J. Franklin Inst. 244: 471 (1947).

16. Drinker, P. and Hatch, T. F.: Industrial Dust, McGraw-Hill Book Co., New York, New York, 1954.

17. Hatch, T. F.: Determination of "Average Particle Size" from the Screen Analysis of Non-Uniform Particulate Substances, J. Franklin Inst. 215: 27 (1933).

PREPARATION OF KNOWN CONCENTRATIONS OF AIR CONTAMINANTS

Introduction

In the field of industrial hygiene, known low concentrations of air contaminants must be prepared frequently for instrument calibration, determination of collection efficiency, and toxicological studies. For gases and vapors, ACGIH threshold limits range from 0.001 ppm to 1,000 ppm. For dusts and fumes, threshold limits of 1 milligram per cubic meter or less are common.

Such concentrations of gases and vapors can be prepared by a number of methods. For a few gases and vapors and for most dusts and fumes the actual concentration must be determined by analysis, using a method of known efficiency. This is the case where the gas or vapor must be generated by a reaction of unknown efficiency. Dusts, fumes, and mists are difficult to prepare in known concentrations because particle settling, precipitation, agglomeration and flocculation cause continuous removal of the generated aerosol. Having determined the concentration produced by a given setting on the generating apparatus, the same setting can be used to approximate the concentration with sufficient accuracy for animal experimentation and the like. Such a concentration must be sampled periodically, however, and preferably monitored continuously. Silverman[1] has discussed in great detail methods of generating gas and vapor concentrations in air. Dynamic concentrations are particularly well covered for gases and vapors, and methods which have been used to prepare them.

Prepared by: Howard E. Ayer
Revised by: Jeremiah R. Lynch

Methods for Preparing Known Concentrations

Static System

Batch Method. For instrument calibration, the static system is generally used. A known amount of gas or vapor is dispersed into an enclosure of known volume. As air samples are withdrawn from the enclosure, the usual practice is to allow air to enter and replace the sample. The effect of dilution is thus dependent on the volume of sample and the volume of the enclosure. If one assumes instantaneous, perfect mixing of the incoming air with the entire sample volume, then the difference in concentration as a small volume is withdrawn is equal to the concentration times the fraction of the volume withdrawn, or

$$dC = C\,\frac{dV}{V_o};$$

this integrates to

$$C = C_o e^{-\left(\frac{V}{V_o}\right)} \text{ or } 2.3 \log_{10} \frac{C_o}{C} = \frac{V}{V_o}$$

where: C = the concentration at any time,
V = the total volume of sample withdrawn,
C_o = the original concentration
V_o = the chamber volume.

For any sampling then, the concentration in the chamber is gradually reduced. If the average concentration of the sample can be permitted to vary 5% from the original, then the concentration can be reduced 10% during sampling. Using the above equation,

$$2.3 \log \frac{100}{90} = 2.3(0.04575) = 0.1053 = \frac{V}{V_o}$$

so that 10.5% of the volume can be sampled and still maintain an average

concentration 95% of the original. The assumption of perfect, instantaneous mixing can be rather poor if no means are provided for circulating the air in a chamber. It can be readily demonstrated with smoke tubes, for example, if the incoming air is introduced near the bottom of a large bottle and perpendicular to it as shown below, that mixing is not immediate, the fresh air mixes only with the bottom layers, and a concentration gradient from top to bottom will thus be established. If such an arrangement is used, the assumption of instantaneous mixing will be on the safe side and the concentration sampled will actually be closer to the original. If the volume of one bottle is not

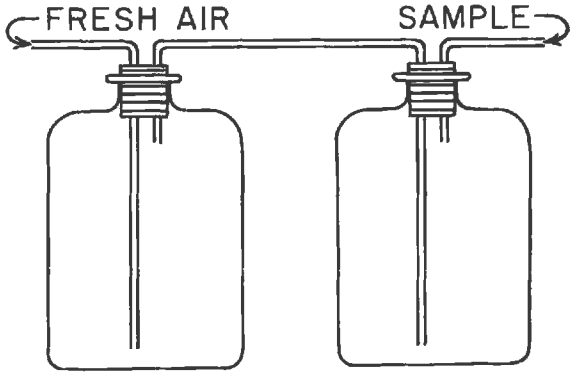


Figure 1. - Bottle Arrangement for Minimum Dilution

sufficient to maintain the desired concentration then two or more bottles in series may be used. This requires that identical concentrations be prepared for each bottle (see Figure 1). Detailed instructions for using 5 or 13 gallon bottles are given in reference (3).

Using this "compound dilution" method the concentration remaining in each of the bottles may be calculated using the formula:

$$C_n = \frac{C_o}{e^{\frac{V}{V_o}}} \left[1 + \frac{V}{V_o} + \frac{1}{2!}\left(\frac{V}{V_o}\right)^2 + \frac{1}{3!}\left(\frac{V}{V_c}\right)^3 + \frac{1}{(n-1)!}\left(\frac{V}{V_o}\right)^{n-1} \right]$$

Where: C_o = the original concentration in each bottle
C_n = the residual concentration in the n^{th} bottle (from which the sample is withdrawn)
V_o = the volume of each bottle
V = the total volume withdrawn in the same units as V_o
e = the base of natural logarithm

Table 1, calculated from this equation, gives the dilution which may be expected for 1, 2, 3, 4 and 5 containers in series. The same table may be used to determine the volume of air which will be necessary to purge the chamber. For a residual of 1% of the original concentration in a single enclosure, for example, 4.6 times the chamber volume would be necessary, assuming perfect mixing. Since, as previously pointed out, perfect mixing may not occur, at least three and preferably ten times the theoretical volume of air should be used.

Larger enclosures have the advantage that most of the common samplers may be used without appreciable dilution. In a 1000 liter chamber, for example, a 15 minute sample at 3 liters per minute will reduce the concentration only 4.4%, and the average concentration only 2.2%. Large chambers also have the advantage of reducing wall effects because of the increased volume-area ratio. Where wall effects may exert an effect, the walls may be pre-conditioned by setting up a concentration in the enclosure, purging the system well, and then setting up the desired concentration. By preparing the lower concentrations of a series first, the whole series may be run without purging the chamber.

One variation of the batch method does not require the calculation of the reduction in concentration of the gas or vapor in the calibration chamber caused by the dilution of the known concentration with outside air. A suitable balloon is placed over the end of the outside air inlet tube, which is enclosed in the container (see Figure 2).

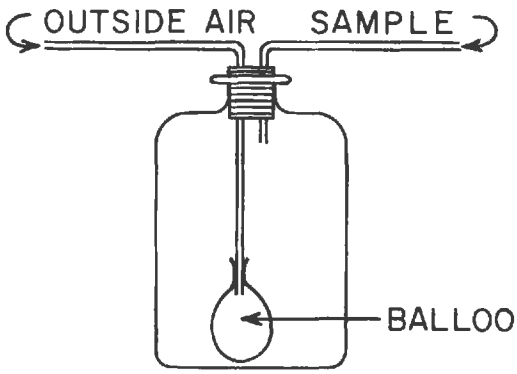


Figure 2. - Single Bottle and Balloon Method

As the sample of contaminated air is removed the air pressure inside the container is proportionately reduced. To balance this difference in air pressure between the atmospheric conditions and the reduced pressure in the container, incoming air is drawn in through the tube that is open to the outside air and expands the rubber balloon until the air pressure inside the container and the atmospheric pressure are approximately equal. The volume of air in the chamber, reduced by the removal of the known concentration mixture, is replaced by the expanding balloon. As a result, no dilution occurs and the concentration remains essentially constant.

Flexible Containers. Plastic films have been developed in the last few years which are suitable not only for the collection of air samples[4,5] but also they can be utilized as flexible chambers for the preparation of known concentrations of air contaminants. This type of chamber is inexpensive; but of greater importance, correction for the error caused by dilution is avoided. The container is normally closed with only one opening through which a metered volume of clean air is introduced, which is less than the total volume required to fill the container. The remainder of the air is introduced after the addition of the gas or vapor at a comparatively high velocity to expedite mixing. If the material of concern is in the form of a liquid it can be injected directly into the flexible chamber with a Hamilton or similar syringe. The additional volume of uncontaminated air required is then metered into the chamber. The sample can then be withdrawn using the same sampling port through which the uncontaminated air was introduced. If the material to be sampled is a gas, a second step in the transfer of the gas to the flexible chamber is usually necessary. A surge tank is fitted to the lecture or other pressurized bottle containing the gas and this surge tank is purged and then filled. The surge tank could be a rubber balloon or rubber hose capped with a self sealing rubber serum stopper. The gas can then be removed from the surge tank and injected directly through the wall of the flexible container with an air tight Hamilton syringe.

Dilution Method. This method uses the theoretical dilution formula for a single container to predict the concentration at any time, with steady removal of the air mixture from the container. The assumption must be made that mixing is instantaneous and complete. By introducing the air through a small diameter tube tangentially to produce a high velocity and produce turbulence, and by installing a small fan within the container to create further mixing, this condition may be approached.

If the air is withdrawn at a constant rate, then

$$C = C_o e^{-(\frac{v}{V}t)} \quad \text{or}$$

$$2.3 \log_{10} \frac{C_o}{C} = (\frac{v}{V}t)$$

Where: C = residual concentration at any time, t
C_o = original concentration
v = rate of air flow out of container (liters per min.)
V = container volume (liters)
t = time in minutes

If this is plotted on semi-log paper, the equation will be a straight line;

C will be 10% of C_o when $(\frac{v}{V}t)$ is 2.3; 1% of C_o when $(\frac{v}{V}t)$ is 4.6; 0.1% at 6.9, etc.

Dynamic Systems

All "dynamic methods" of generating known concentrations of air contaminants have in common the continuous generation of the desired mixture. Consequently, there is hardly any limitation on sampling volume or rate and it is usually quite easy to vary the concentration over a wide range. Silverman[1] gives a list of some 61 gases or vapors for which dynamic methods are appropriate. Only a few more important methods will be discussed here.

Gas Dilution. Probably the simplest system is the dilution of a metered flow of a contaminant gas with a known flow of air. Using a rotameter in the range of one liter per minute and an orifice meter with a range of 100 cfm in the dilution air flow, a range of dilutions up to 1:100,000 is possible.

Vapor Pressure. If a saturated vapor of a material at a known, constant temperature can be obtained conveniently, it can be used as a source of contaminant of known concentration. Such equilibrium mixtures can be used as generated if the concentration is in the desired range, or if diluted, to obtain lower concentrations.

Liquid Feed. Various methods have been developed for feeding liquids at a known rate and vaporizing them into the air system. Syringes can be used to feed the liquid slowly when the plunger is pushed by a screw mechanism. In another syringe method, the weight on the plunger of a vertical syringe feeds the fluid at a constant rate and at a constant head. To prevent the plunger from sticking, it is spun continuously by a stream of air directed at blades mounted on top of the plunger or by use of a magnetic stirrer. Other methods of slow liquid feed include displacement by a plunger lowered into a cylinder by clockwork, or the displacement of the liquid by a steady drip of mercury. All of these methods of liquid feed require that the liquid be vaporized instantaneously into the air stream, using heat if necessary.

Controlled Reaction. The mixing of two liquids may cause a reaction which produces the desired contaminant, which then can be diluted. If the rate of addition of one of the liquids can be controlled, the concentration of the product will be determined. An example of this method is the dropping of formic acid into hot sulfuric acid to produce carbon monoxide.

Electrolysis. A known current of electricity can be utilized to liberate its electrochemical equivalent of a gas in the electrolysis of an ionic solution. The varieties of gas which can be produced in this manner, however, are limited.

Generation of Aerosols

Various methods have been devised for the generation of aerosols. However, none of these produce a "known concentration" in the sense of having a known number of particles per unit volume. It is possible, however, to produce concentrations of known weight under certain circumstances and it is possible to produce homogeneous aerosols of known particle size, and dust clouds of known particle size distribution. Many of these methods are discussed by Silverman[1].

References

1. Silverman, Leslie: Experimental Test Methods. 178 references. Air Pollution Handbook edited by Magill, Holden, and Ackley, Chapter 12, McGraw Hill Book Co., NewYork, 1956.

2. Setterlind, A. N.: Preparation of Known Concentrations of Gases and Vapors in Air. AIHA Quarterly 14: 113 (June 1953).

3. Stead, F. M., and Taylor, G. J.: Calibration of Field Equipment from Air-Vapor Mixtures in a Five Gallon Bottle. Jour. Ind. Hyg. and Tox. 29:408 (Nov. 1947).

4. Conner, W. D., and Nader, J. S.: Air Sampling with Plastic Bags. Am. Ind. Hyg. Assoc. J. 25:291 (1964).

5. Stewart, R. D., Gay, H. H., Erley, D. S., Hake, C. L., and Peterson, J. E.: Observations on Concentration of Trichloroethylene in Blood and Expired Air Following Exposure of Humans. Am. Ind. Hyg. Assoc. J. 23:167-170 (1962).

(See Table 1 on next page).

Table 1. Average concentration of portion withdrawn and residual concentration of each of 5 bottles connected in series.

Amt. withdrawn; % of single bottle vol.	1st Bottle		2nd Bottle		3rd Bottle		4th Bottle		5th Bottle	
	Residual conc.	Avg. conc. of volume withdrawn	Residual conc.	Avg. conc. of volume withdrawn	Residual conc.	Avg. conc. of volume withdrawn	Residual conc.	Avg. conc. of volume withdrawn	Residual conc.	Avg. conc. of volume withdrawn
0	100.00	100.00	100.00	100.00	100.00	100.00	100.000	100.000	100.000	100.0000
10	90.48	95.24	99.53	99.76	99.98	99.99	99.997	99.998	99.997	99.9985
20	81.87	90.78	98.25	99.26	99.88	99.95	99.995	99.997	99.993	99.997
40	67.03	82.69	93.85	97.59	99.21	99.74	99.922	99.977	99.98	99.991
60	54.88	75.57	87.81	95.24	97.69	99.28	99.66	99.912	99.96	99.984
80	44.93	69.29	80.88	92.45	95.26	98.20	99.09	99.76	99.85	99.961
100	36.79	63.73	73.58	89.35	91.97	97.22	98.11	99.51	99.62	99.91
120	30.12	58.80	66.26	86.06	87.95	95.95	96.62	99.13	99.23	99.81
140	24.66	54.42	59.18	82.72	83.35	94.42	94.62	98.59	98.57	99.68
160	20.19	50.52	52.49	79.36	78.34	92.68	92.13	97.91	97.64	99.41
180	16.53	47.03	46.28	76.04	73.06	90.76	89.13	97.07	96.36	99.18
200	13.53	43.91	40.59	72.79	67.65	88.83	85.64	96.07	94.71	98.79
220	11.08	41.11	35.46	69.66	62.27	86.51	81.85	94.92	92.66	98.31
240	9.07	38.58	30.84	66.64	56.96	84.25	77.86	93.60	90.40	97.71
260	7.43	36.30	26.75	63.76	51.86	81.95	73.62	92.24	87.77	97.04
280	6.08	34.24	23.10	61.02	46.94	79.61	69.18	90.72	84.76	96.24
300	4.98	32.36	19.92	58.41	42.33	77.38	64.74	89.12	81.55	95.35
320	4.08	30.67	17.14	55.95	38.03	75.06	60.30	87.44	78.13	94.36
340	3.34	29.11	14.70	53.63	34.00	72.80	55.86	85.70	74.48	93.27
360	2.73	27.69	12.56	51.44	30.25	70.52	51.48	83.91	70.58	92.10
380	2.24	26.39	10.72	49.37	26.92	68.32	47.41	82.09	66.87	90.85
400	1.83	25.20	9.15	47.43	23.82	66.19	43.35	80.24	62.82	89.53
420	1.50	24.13	7.80	45.63	21.03	64.12	39.55	78.38	59.00	88.15
440	1.23	23.09	6.64	43.91	18.55	62.12	36.01	76.54	55.22	86.73
460	1.005	22.15	5.63	42.29	16.26	60.19	32.56	74.70	51.31	85.31
480	0.82	21.28	4.76	40.76	14.20	58.34	29.32	72.88	47.45	83.76
500	0.67	20.48	4.02	39.33	12.40	56.55	26.35	71.09	43.80	82.22

Note: All bottles have equal volume and contain equal concentrations at the start. Residual and average concentrations are given in percent of original. First bottle is the one in which outside air enters. This data is from Setterlind "Preparation of Known Concentrations of Gases and Vapors in Air."(2)

DIRECT READING INSTRUMENTS FOR DETERMINING CONCENTRATIONS OF AEROSOLS, GASES AND VAPORS

Introduction

For purposes of this discussion, direct reading instruments are classified as those which give an immediate indication of the concentration of aerosols, gases or vapors by some means such as, reading a dial or noting the color change of a chemical. These instruments have fulfilled one of the greatest needs of industrial hygienists because these devices give immediate results and they eliminate time-consuming laboratory analyses. This is very helpful in preventing further injury to the worker and in advising management of environmental conditions. Direct reading instruments are dependent on some chemical (colorimetric) or physical reaction, which may be simple or complex.

Direct Physical Methods for Air Analysis

At some time every known physical characteristic of gas-air mixtures has been used for gas analysis. However, in the industrial hygiene instrument field, the fact that such low concentrations are usually encountered means that some of the changes in physical characteristics are too small to be measured easily or accurately. This need for high sensitivity, plus the need for relatively simple, light-weight instruments for field use, has limited the development of field instruments utilizing only certain physical principles. These are summarized in Table 1 which gives the method, principle of operation, type of sample, range of concentration detectable, and applications.

Direct Reading Colorimetric Indicators

This group of direct reading devices is dependent on some chemical reaction to produce a color change. Three types of colorimetric indicators are being used for the determination of contaminant concentrations in air:

(1) glass indicator tubes containing solid chemicals;
(2) chemically treated filter papers; and
(3) liquid reagents.

A comprehensive bibliography in this area has been prepared by Cambell and Miller.[1]

Indicator Tubes

These usually consist of granules of either absorbent gels such as silica gel or activated alumina or simply inert granules such as pumice or sand coated with the detecting chemical. The efficiency of absorption of most gels is dependent upon the rate of air flow through the particular gel, therefore, the air should be drawn through the tube at the same rate as used in the calibration.

Either one of two methods may be used to prepare the indicator tubes. The chemical reagent can be mixed or reacted with the absorbent and sealed in glass tubes at the factory, or the chemicals and absorbent can be packed separately and mixed just prior to use. Air is aspirated through the glass tube, following closely the manufacturer's instructions. The observer then determines the concentration in the air by examining the exposed tube. This is done by matching the color of the indicating gel with color standards or comparing the length of stain with a calibrated scale supplied by the manufacturer. This visual judgment depends on the color vision of the observer and the lighting conditions. Accuracy is dependent on air-flow calibrations, calibration of the indicator tube with

Prepared by: Herbert H. Jones

known concentrations and visual interpretation of color change by the observer. It should be emphasized here that these results indicate only the concentration of the contaminant at the time the sample was collected. Furthermore, several tests should be made at the same location in order to determine if a potential hazard to health exists. (See Section B-1 for additional details on evaluation of health hazards in the work environment).

Chemically Treated Papers

Methods have been developed for using chemically treated papers for detecting and determining concentrations of gases and vapors. Such papers may be prepared fresh and used wet, or prepared in advance, stored, and used in the dry state. Semi-quantitative determinations may be made by hanging the papers in contaminated air. The accuracy of such procedures is limited by the fact that the volume of the air sampled is rather indefinite and the degree of color change in the paper may be influenced by temperature. More quantitative results may be obtained by using a sampling device capable of passing a measured volume of air over or through a definite area of paper at a controlled rate. Some determinations, such as those for chromic acid and lead, are made by the addition of liquid reagents to the sample on a filter paper. Evaluation of the stains on the paper are made visually by comparison with color standards or by photometric instruments. Accuracy of these methods is related to uniform sensitivity of the paper, stability of all chemicals used, and careful calibration.

Liquid Reagents

Some of the simple laboratory procedures using liquid reagents have been adapted for field use. Reagents are supplied in sealed ampoules or tubes, frequently in concentrated or even solid form which are dissolved or diluted before use. Unstable mixtures may be prepared as needed by breaking an ampoule containing one ingredient inside a plastic tube or bottle containing the other. Liquids containing indicators have been used for determining acid or alkaline gases by measuring the volume of air required to produce a color change. Methods employing liquids in the field usually are not considered by the field personnel to be too highly desirable as they require care in handling, are bulkier to transport, and can create disposal problems of used chemicals.

Selection of Instruments

Several factors should be taken into consideration when an instrument is being selected to evaluate a particular environment. Most of these factors are related to the design and performance characteristics of the device. In general, the performance characteristics of an instrument are inherently a result of the design but often performance and results can be enhanced or diminished by superb or inadequate attention, respectively, to the capabilities, limitations, and the appropriate operating procedures of the instrument.

Simplicity

The instrument should be as simple as possible with respect to construction, operation, maintenance and data presentation. Often simplicity is sacrificed for more flexibility and versatility in the application of an instrument. This often results in additional controls and more possibility for error in the operation.

Specificity

This is often a characteristic of the detection method, but is not limited to it alone. In some instruments both physical and chemical methods are used, here the specificity must be determined for the whole system. Instruments which are not specific can be used in the evaluation of an environment if it is known that the interfering agents are

not present or represent only a small percentage of the particular contaminant being measured and do not appreciably interfere with the primary reaction.

Stability and Reliability

The instrument should retain its performance characteristics over a long period of time. It should be possible to reproduce the same measurements at different times for the same sample concentrations and same operating conditions.

Accuracy

Accuracy of calibration of the instrument output in terms of the variable being measured determines the initial accuracy of the measurement. Subsequently, the accuracy of measurement will depend upon the stability and reproducibility of the device. Calibration checks are required periodically. The frequency of such checks is influenced by the stability of the instrument. Thus, a relatively unstable instrument with temperature and zero drift and poor reproducibility, will require frequent calibration checks.

Sensitivity

This is the minimum amount of the contaminant which can be detected repeatedly and is greatly affected by the stability of the system.

Response Time

The response time is the time interval from the instant the instrument samples a contaminant to the time it takes the instrument to show a reading which is a specified percentage of the final value. Most often the specified percent is from 90% to 95% of the final value. Response time may vary from a few seconds for a physical system to several minutes for a chemophysical system. A short response time is necessary to resolve short-term variations in concentrations.

References

1. Cambell, E. E. and Miller, H. E.: Chemical Detectors. A Bibliography for the Industrial Hygienist with Abstracts and Annotations. Los Alamos Scientific Laboratory, LAMS-2378, Vol. 1, Oct. 1961.

2. Air Sampling Instruments, American Conference of Governmental Industrial Hygienists, 1014 Broadway, Cincinnati 2, Ohio, 2nd Ed. 1962. The following sections: Air Sampling and Analysis for Contaminants in Work Places - Leslie Silverman; Direct Reading Colorimetric Indicators - B.E. Saltaman; Direct Reading Physical Instruments - J. S. Nader.

3. Yaffe, C. D., Byers, D. H., Hosey, A. D., editors. Encyclopedia of Instrumentation. Univ. of Michigan, Inst. of Ind. Health, Ann Arbor, Mich., 1956.

Table 1. - Direct Reading Physical Instruments

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
A.	Absorption	Measured volume of air-gas mixture is absorbed by reagent and volume or pressure reduction is determined.	Grab	0.1%	60%	Selective. High Concentrations of gases. Commercial instruments available for CO, CO_2, and O_2.
B.	Electrical 1. Conductivity	Gas-air mixture is drawn through a solution where the gas is absorbed and the electrical conductivity is measured before and after absorption.	Continuous	1 pphm	1000 ppm	Any gas or its pyrolytic products which form electrolytes in any aqueous solution. In many cases not selective. Commercial instruments available for SO_2, NO_2, Cl_2, C_2H_4, H_2S.
	2. Potentiometry	Gases which react with reagents in solution to introduce a change in pH of the solution produce a potentiometric change which reflects the concentration of the reacting gas.	Continuous	200 ppm	1000 ppm	May be made specific by the choice of reagents. No commercial instruments. One described for CO_2 in literature.
	3. Coulometry	Gas-air mixture is drawn through a solution and a measurement of the number of electrons in terms of coulombs that are transferred across an electrode-solution interface to carry to completion the reaction of a particular substance in the sample.	Continuous	1 ppm	25%	Some instruments are specific, others are for chemical groups. Sensitivity may vary widely from one instrument to another. Commercial instruments available for SO_2, NO_2, O_2, H_2S, Olfins, Merceptans, fluorides.
	4. Ionization a. Flame	A gas-air mixture is introduced into a hydrogen flame and the increase in ions is proportional to the volatile carbon content of the gas.	Continuous	ppb	ppm	It is insensitive to the presence of such contaminants as air and water vapor, but responds to most organic compounds. Response is depressed with compounds having electro-negative atoms such as oxygen, sulfur and chlorine.

Table 1. - continued

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
	b. Aerosol	The sampled gas is reacted with a specific reagent to produce an aerosol which, in the process of becoming charged by passing thru a stream of ions produced by a radioactive source, reduces the flow of current.	Continuous	ppb	ppm	Specificity of this method is limited by the specificity of the chemical reaction which is used to generate the aerosol. Precautions must be taken to eliminate any aerosol normally present in the sampled gas stream, which would otherwise act as a contaminant.
C.	Electromagnetic 1. Optical Absorption a. Ultraviolet	Gas-air or vapor-air mixture is passed thru a tube with a UV light source at one end and a photocell at the other. Gases or vapors which have absorption bands in the UV or near UV region absorb light and diminish the cell response which is registered by a calibrated meter.	Continuous	.005 mg/M^3	4 mg/M^3	Maximum sensitivity for mercury vapor. Sensitive to several gases and vapors. Water vapor and CO_2 do not interfere.
	b. Infrared	Gas-air or vapor-air mixture is passed thru a tube with a source of infrared (heated filament) at one end. Detection may be by a thermopile and calibrated meter which measures infrared absorption or may be by a pressure sensitive device which measures change in pressure produced by the heating of the mixture by the absorption of infrared.	Grab or continuous	0.1 ppm	100%	Sensitive to all heteroatomic gases and vapors. Bands for absorption in infrared vary with gases just as in ultraviolet bands. Thus the instrument may be made selective by the use of screening filters. Manufacturers can supply instruments which are selective for a number of gases.

Table 1. - continued

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
	c. Visible	Gas-air or vapor-air mixture is passed through a reagent either in solution or impregnated on test paper. This is then analyzed colorimetrically. The measurement system usually consists of a radiant energy source (electric bulb with an incandescent tungsten filament), the object to be measured, filters, and a detector for the unabsorbed or transmitted radiation.	Grab	0.1 ppm	5000 ppm	By proper selection of reagents and filters can be made selective in many cases. Sensitivity depends on reagents, volume or length of sample. Response time may be very long. Commercial instruments available for Oxidants, NO, NO_2, SO_2.
	2. Optical a. Light scatter	Basically, the principle of operation is the generation of an electrical pulse by a photocell which detects the light scattered by a particle. Electronic counting techniques give a measure of the particle count and electronic pulse height analyzer gives a measure of the effective particle diameter.	Continuous	10 ppcf	10^8 ppcf	In principle the light-scatter method limits the minimum size particle detectable to the wavelength of the incident light which is about 0.3 microns. The upper range is in the order of 10 microns. Must be calibrated for the particular aerosol being measured.
	b. Spectral Emission	The sample is collected on a filter or drawn into a chamber where it is exposed to a source of energy for atomic excitation by an electrical arc or spark. The excitation raises the valence electrons above the stable level and they emit light. The emitted light isdetected by a photocell. The intensity of the light emission lines are characteristic of the element and is related to concentration.	Continuous	0.3 $\mu g/M^3$	80 $\mu g/M^3$	Commercial instruments are available for beryllium and lead. Calibration is by internal standards and the method in principle is specific and lends itself well to quantitative and qualitative analysis.

Table 1. - continued

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
	c. Attenuation and reflection.	The sample is drawn thru a filter paper then the light attenuation or reflectivity change is measured by comparing the sample with clean filter paper. The change in attenuation or reflectivity is recorded on a strip chart.	Continuous	---	---	Range depends on length of sample and can be set for low or high concentrations. Must be calibrated for the particular aerosol which is being sampled.
D.	Radioactivity 1.Electrical a. Ionization chambers	Ionization chambers are essentially confined volumes in which an electric field potential is arranged to collect ions generated by alpha and beta particles colliding with gas molecules and particles.	Continuous	1 $\mu\mu$c/cc	1500 $\mu\mu$c/cc	The method lends itself more readily to alpha detection since this type of activity is significantly greater (orders of magnitude) in its ionizing power. The ion current measure is calibrated in terms of the radioactivity generating it under controlled conditions.
	b. Geiger-Muller	The G-M detector is basically a modification of the ionization chamber in which the potential field is raised to a high enough level such as to cause electrons, initially produced by a radioactive particle, to accelerate and produce other electrons in turn in a select gas medium resulting in an avalanche of electrons and a high voltage discharge. Sample is collected on a filter and the radioactivity is measured.	Continuous	5×10^{-7} μc/cc	---	The avalanche can be triggered by a single electron so the G-M detector is nondiscriminating with respect to the type of activity initiating the discharge. Therefore, it serves as a good detector for gross activity and quantitative measurements. The counter has a "window" through which the radiation passes into the sensitive volume for detection, This window can be of very thin material permitting penetration by the less energetic particles.

Table 1. - continued

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
	2. Electromagnetic a. Scintillation	The scintillation detector consists of a scintillation phosphor in close proximity to a photomultiplier tube. Passage of individual particles through the phosphor results in corresponding light flashes which are detected by the photomultiplier. Appropriate electronic circuitry is used to count the pulse output from the photomultiplier to give a quantitative measure of radioactivity. Sample is collected on a filter and the activity is measured.	Continuous	$2x10^2$ cpm	$2x10^6$ cpm	Phosphors vary in composition and in physical form to include liquid as well as solid substances. These detectors offer an advantage over the ionization and G-M devices with respect to analysis of gamma and x-rays. It is possible to obtain total absorption of the rays energy which is necessary for energy spectrum absorption and related work of isotope identification. Can be used for low-level radioactivity detection of less penetrating radiation such as alpha activity
E.	Radioactive Tracers	A new approach to gas analysis using a radioactive tracer in a clathrate reaction. It is being used for SO_2 which reacts with a clathrate in which Kr-85 is released. The released Kr-85 is detected by a G-M counter and the resulting count rate is related to the SO_2 concentration in initiating the reaction.	Continuous	1 ppm	---	Is specific only to the extent that the initial reaction is limited to the gas being studied.
F.	Thermal 1. Conductivity	Gas or vapor mixture is passed over a heated wire (below ignition temperature). Change in heat loss of wire to surrounding gas mixture is registered by change in wire resistance measured on a balanced Wheatstone bridge.	Grab or continuous	---	1000 ppm	Non-selective. Water vapor and carbon dioxide interfere and must be removed. Can be used with chromographic column in which gas mixtures are resolved into their components.

Table 1. - Continued

No.	Method	Principle of Operation	Type of Sample	Range Lower	Range Upper	Application and Remarks
	2. Combustion	Gas or vapor mixture is passed over a heated wire (above ignition temperature), catalytic heated filaments, or oxidation catalysts and the detection is by the change in a balanced bridge or by thermacouples.	Continuous	5 ppm	100%	Has some limitations as thermal conductivity but can be made more or less specific by operating at specific filament temperatures, so as to ignite gas of interest or by selection of an oxidation catalyst favoring a desirable reaction such as "Hopcolite" for carbon monoxide.
G.	Gas Chromotography	Gas or vapor is absorbed in a porous medium in a column. There is a differential of migration of the components through the porous medium such that by proper selection of media, gas flow and temperature the individual components of the mixture can be separated and detected by thermal conductivity or flame-ionization or other suitable detection method.	Grab	ppb	100%	Selective. Can be used to identify solvents and separate gas mixtures by selective elution.

Table 2 - Contaminants Determined by Direct Reading Colorimetric Indicators +

Contaminant	TLV**	Mfgr.	Range
Acetic acid	10	b	-
Acetone	1000	b,e, f,i	100-10,000
Acetonitrile	40	h	10-200
Acetylene	-	h,i	3-1000
Acrylonitrile	20	f,h,i	5-500
Ammonia	50	b,c,e, h,i	10-1500
Aniline	5	b	-
Arsine	0.05	b,e,f, h,i	0.01-160
Arsenic	0.5*	c	-
Benzene	25	b,e,f, h,i	10-400
Beryllium	0.002*	c	-
Bromine	0.1	b,h,i	5-300
Butadiene	1000	e	500-2000
Butadiene Dioxide	-	b	-
Butanone (MEK)	200	e,i	100-1400
Butyl Acetate	200	e	100-400
Butyl Alcohol	100	e	50-200
n-Butylamine	5	h	2-100
Butyl Cellosolve	50	e	25-100
Carbon Dioxide	5000	e,f,h,i	50-5000
Carbon Disulfide	20	e,f,i	10-3200
Carbon Monoxide	100	a,b,e,f, g,h,i	10-75,000
Carbon Tetrachloride	10	b,e,f, h,i	5-300
Cellosolve	200	e	100-400
Cellosolve Acetate	100	e	50-200
Chlorine	1	b,c,e, f,h,i	0.2-100
Chlorine Dioxide	0.1	i	10-500
Chlorobenzene	75	e,h	10-200
Chlorobromo-methane	200	h	25-500
1-Chloro-1, 1-difluoroethane	-	h	100-4000
Chlorodifluoro-methane (Freon 22)	-	h	200-2000
Chloroform	50	b,e,h	25-100
1-Chloro-1-nitropropane	20	h	2-75
Chloropenta-fluorethane (Freon 115)	-	h	200-2000
Chlorotrifluoro-methane (Freon 13)	-	h	200-3500
Chromic Acid	0.1*	c,h	.05-1.4*
Copper	1.0*	c	-
Cyanogen Chloride	-	b	-
Cyclohexane	400	e,i	100-6000
Cyclohexanol	50	e	25-200
Cyclohexanone	50	e	25-200
Decaborane	0.05	h	.01-1.0
Diborane	0.1	h	.01-3.0
1-2-Dibromo-ethane	25	h	10-200
Dichloro-benzene(ortho)	50	e,h	10-200
Dichlorodi-fluoromethane (Freon 12)	1000	e,h	200-2000
1,1-Dichloro-ethane	100	h	25-500
1,2-Dichloro-ethane (ethylene dichloride)	50	b,e,h	25-500
1,2-Dichloro-ethylene	200	e,h	10-500
Dichloroethyl Ether	15	h	5-75
1,1-Dichloro-1-nitroethane	10	h	2-75
Dichlorotetra-fluoroethane (Freon 114)	1000	h	200-2000
Diethylamine	25	h	5-200
Diethyl Ether	400	b,i	400-1400

Table 2. - Continued

Contaminant	TLV**	Mfgr.	Range
Dimethylamine	10	h	5-200
Dimethyl Ether	-	i	100-1200
Dimethyl-formamide	10	h	5-100
Dimethylhydrazine(VDMH)	0.5	h	0.5-10
Dimethylsulfate	1	b	-
Dipropylamine	-	h	2-70
Eipchlorhydrin	5	e	-
Ethyl Acetate	400	e,i	100-5000
Ethyl Alcohol	1000	e,f,i	100-5000
Ethylamine	25	h	10-500
Ethyl Bromide	200	h	25-400
Ethyl Chloride	1000	e,h	100-2000
Ethyl Ether	400	b,i	400-1400
Ethylene	-	h,i	0.5-100
Ethylene Dichloride (1,2-dichloroethane)	50	b,e,h	50-200
Ethylene Oxide	50	e,i	100-3500
Fluorine	0.1	b	-
Fluorotri-chloromethane (Freon 11)	1000	h	250-2000
Formaldehyde	5	b,c,f	2-40
Heptane	500	e	250-1000
Hexane	500	b,e,i	100-6000
Hydrazine	1	b,h	0.5-20
Hydrocarbons (Gasoline)	500	f,g	100-10,000
Hydrogen Chloride	5	b,f,h	2-500
Hydrogen Cyanide	10	b,d,f, h,i	2-150
Hydrogen Fluoride	3	c,f,h	0.5-15
Hydrogen Sulfide	10	a,b,c,e, f,h,i	1-1700
Iodine	0.1	b	-
Isopropyl Alcohol	400	b,e	200-800
Isopropylamine	5	h	2-100
Mercury	0.1*	b,c,f, h,i	0.05-2*
Methyl Acetate	200	e	100-400
Methyl Alcohol	200	b,e, f,i	100-6000
Methylamine	25	h	10-500
Methyl Bromide	20	e,f, h,i	5-500
Methyl Chloride	100	e,h	25-1000
Methyl Chloro-form (1,1,1-trichloroethane)	350	e,h	100-700
Methyl Ethyl Ketone (MEK)	200	e,i	100-400
Methyl Isobutyl Ketone	100	e	100-400
Methylene Chloride (Dichloromethane)	500	e,h	50-2000
Monochloro-benzene	75	e,h	10-200
Nickel Carbonyl	0.001	i	1-1000
Nitrobenzene	1	b	-
Nitroethane	100	h	10-300
Nitrogen Dioxide	5	c,e,f, h,i	0.1-1000
Nitrogen Oxide	-	b,f	0.5-1200
Nitromethane	100	h	5-300
1-Nitropropane	25	h	2-100
2-Nitropropane	25	h	5-150
Oxidants	-	c	-
Ozone	0.1	f	0.1-100
Pentaborane	0.005	h	0.1-1
Perchloro-ethylene	100	e,g,h	10-400
Phosgene	0.1	b,e,f, h,i	0.1-100
Phosphate	-	c	-
Phosphine	0.3	i	5-800
Propiolactone (beta)	-	b	-

Table 2. - Continued

Contaminant	TLV**	Mfgr.	Range
n-Propyl Nitrate	25	h	10-100
Propylene	-	h	1-400
Propylene Dichloride	75	h	25-500
Propylene Oxide	100	e	50-200
Pryidine	5	h	2-50
Selenium	0.2*	c	-
Stibine	0.1	e	0.05-0.2
Styrene Monomer	100	f	50-400
Sulfate	-	c	-
Sulfur Dioxide	5	b,c,e f,h,i	1-4000
Systox	-	f	1 μg
1,1,2,2-Tetra-chloroethane	5	h	2-50
Tetrachloro-ethylene (Perchloroethylene	100	e,g,h	100-400
Toluene	200	b,e,f, h,i	1-1000
Trichloroethane (methyl chloroform)	350	e,h	25-700
Trichloroethy-lene	100	b,e,f, g,h,i	10-600
1,2,3-Trichloro-propane	50	h	10-500
Triethylamine	25	h	2-100
Trifluoromono-bromomethane (Freon 13B1)	1000	e,h	100-4000
Trimethylamine	-	h	5-250
Vinyl Chloride	500	e,h,i	100-2000
Xylene	200	b,e,h	10-400
Zinc Oxide	5*	c	-

+As listed by manufacturers (frequently, because of non-specificity), detectors listed for chemically related substances may also be applicable, with suitable calibration.

* Milligrams per cubic meter.

**Threshold limit values, as established by the American Conference of Governmental Industrial Hygienists in 1964.

List of Manufacturers Coded in Table 2.

Bacharach Industrial Instrument Co. a
200 N. Braddock Ave.
Pittsburgh, Pennsylvania 15208

Brothers Chemical Company b
575 Forest St.
Orange, New Jersey

Central Scientific Co. c
1700 W. Irving Park Road
Chicago, Illinois 60613

Compact Air Samplers d
825 Belmont Park, North
Dayton, Ohio 45405

Davis Emergency Equipment Co., Inc. e
45 Halleck St.
Newark, New Jersey 07104

Drager Corporation f
432 Park Avenue
New York, New York 10016

LKB Instruments, Inc. g
4840 Rugby Avenue
Washington, D.C. 20014

Mine Safety Appliances Co. h
201 N. Braddock Ave.
Pittsburgh, Pennsylvania 15208

Union Industrial Equipment Corp. i
40 Beech St.
Port Chester, New York

SAMPLING AND ANALYSIS OF GASES AND VAPORS

Introduction

This lecture deals principally with the methods and instrumentation used for the sampling and analysis of gases and vapors present as atmospheric contaminants in the industrial environment. In the sections dealing with sampling procedures, direct reading instruments are not discussed as there is a separate lecture on this topic. In both the sampling and analysis sections, the emphasis is placed on fundamental techniques rather than on specific methods for individual substances. This approach eliminates unnecessary repetition.

Properties of Gases and Vapors

The distinction between gases and vapors is somewhat vague. An "elastic fluid" is generally called a gas if its temperature is very far removed from that required for liquefaction; conversely, it is termed a vapor if it is near its temperature of liquefaction. In the field of industrial hygiene, a substance is considered as a gas if this is its normal physical state at room temperature and atmospheric pressure. It is called a vapor if, under the environmental conditions, conversion of the liquid or solid form to the gaseous state results from its vapor pressure effecting its volatilization or sublimation into the atmosphere of the container, the process equipment, or the workroom. Our chief interest in distinguishing between gases and vapors lies in our need to assess the potential occupational hazards associated with the use of specific chemical agents, an assessment which requires a knowledge of the physical and chemical properties of these agents.

Prepared by: Robert G. Keenan

Sampling Methods

The primary objective of this phase of an environmental survey is to obtain representative samples of the atmospheric contaminant in forms suitable for subsequent analysis. A general discussion of the fundamental aspects of plant surveys to provide adequate evaluations of the environment is given in Section B-1.

Basic Techniques

Two basic methods for the collection of gaseous samples are employed. The first involves the use of a gas collector, such as an evacuated flask or bottle, to obtain a definite volume of air at a known temperature and pressure. The collector is re-sealed immediately to prevent any loss before the sample can be analyzed. The second method involves the passage of a known volume of air through an absorbing or adsorbing medium to remove the desired contaminants from the sampled atmosphere. The absorbing medium, which may be a solvent such as ethyl alcohol or amyl acetate, can be transferred with the collected sample to a reagent type, glass stoppered bottle for transfer to the laboratory.

The selection of one or the other of these techniques depends upon the type and purpose of a particular sample. When the major components of a gas or of air are to be determined, a gas collector is used. When a minute quantity (parts per million level) of a contaminant must be estimated, the method of concentrating the substance in an absorber or on an adsorbent must be employed. The collection of instantaneous samples, those taken within a time period extending from a few seconds to one or two minutes, is made to determine the composition of an atmosphere at a specific time and location. Such samples may best be taken with evacuated

flasks or with gas or liquid displacement collectors. However, an absorption or adsorption device must usually be employed for collecting continuous samples of a non-uniform atmosphere, for the estimation of the average composition during a given time period.

Method Requirements

Sampling methods for gases or vapors should meet the following requirements:

1. Provide an acceptable efficiency of collection for the substance or substances involved.
2. Maintain this efficiency at a rate of air flow which can provide sufficient sample for the analytical procedure in a reasonable period of time.
3. Retain the collected gas or vapor in a form which is transportable to the analytical site.
4. Yield the sample in a form suitable for the analytical method.
5. Require minimal manipulation in the field.
6. Avoid use of corrosive or otherwise hazardous sampling media whenever possible.

These requirements are considered as practical guides in planning a plant survey and, by their wording, indicate the need of consultation between the engineer and the chemist for optimal results from each survey.

Sampling Instruments

In the application of the two basic sampling methods, numerous sampling devices have been employed. For more complete information on the construction, recommended uses, and efficiencies of specific instruments, the appropriate reference works should be consulted.[1,2] For outline purposes, the following tables contain a limited listing of the devices commonly used for gas and vapor sampling, along with selected typical applications of each.

The sampling instruments given in Tables 1 and 2 include those most commonly used in gas and vapor sampling. Other devices, such as glass-bead packed columns containing absorbing solutions, freeze-out traps, the Harrold mist and gas collector, are invaluable in certain situations. The collection efficiencies reported in Table 2 are absolute values based upon sampling of known atmospheric concentrations of the indicated gas or vapor at the specified rate of air flow.

In planning an environmental survey it must be remembered that gases and vapors, unlike dusts, fumes or mists, form a true solution with air and, hence, drastic physical or chemical action is required to collect these substances efficiently from the atmosphere. The industrial hygienist must be familiar with the advantages, limitations, and performance characteristics of each type of sampling device to provide samples of utmost value to the laboratory for chemical analysis.

In addition to the sampling instruments, a suction device, such as an electrically operated pump, which will provide a reasonably constant rate of air flow through the sampler and an air flow measuring instrument, such as an orifice flowmeter, are required to collect the sample. The flowmeter, attached to the assembled equipment, must be calibrated in the laboratory with a wet or dry gas meter or with a mercury manometer. A separate lecture entitled "Air-flow Calibrations" is presented in Section B-3 and, therefore, the details of this calibration will not be discussed in the present lecture.

Instructions on the preparation of known concentrations of gases and vapors are given in Section B-8. The industrial hygienist should be familiar with this technique so as to establish the collection efficiency of his sampling apparatus.

Table 1. - Gas and vapor collectors with typical applications

Collector	Size in ml	Gas or Vapor
Evacuated flask	250 - 300 ml	Gross components of air or of sewer or mine gases
Evacuated flask	1000	Carbon monoxide in air
Vacuum bottle	500 - 2500	Substances not easily trapped by absorbents or adsorbents: total oxides of nitrogen; benzene
Gas or liquid displacement collector, glass	250 - 4000	Gross components of air or gas mixture
Metallic collectors	Ditto, or larger	Non-corrosive gases

Table 2. - Gas and vapor samplers, with partial listing of applications, sorption media and collection efficiencies

Sampler	Sorption Medium	Air Flow l/m	Gas or Vapor	Efficiency %
U-tube	Silica gel or activated carbon	5	Organic solvent vapors	*
Fritted glass bubbler	Sodium hydroxide	5	Hydrochloric Acid	95
	Sodium bisulfite	1-3	Formaldehyde	ca 100
	Barium hydroxide	1	Carbon dioxide	60-80
	Iodine	5	Hydrogen sulfide	95
Midget fritted glass bubbler (60-70 micron max. pore diam.)	Saltzman reagent‡	0.4	Nitrogen dioxide	94-99
Petri bubbler	Nitrating acid	0.25	Benzene	95
Impinger	Sulfuric acid	28.3	Ammonia	95
	2% Glycerol in 0.05 N sodium hydroxide	28.3	Sulfur dioxide	95
Midget impinger	1% KI, 0.1 M KH_2PO_4, and 0.1 M Na_2HPO_4	1-3	Ozone	95+

* Dependent upon nature of vapor, adsorbent, and number of tubes
‡ Dissolve 5 grams sulfanilic acid in almost a liter of nitrite free water containing 140 ml of glacial acetic acid; add 20 ml of 0.1% aqueous solution of N-(1-naphthyl)-ethylenediamine dihydrochloride and dilute to 1 liter.

Fundamental Gas Laws

The actual measurements of the volumes of gases or gas-air mixtures must be corrected to standard conditions of temperature and pressure in order for the measurements to have meaning. The conversion of actually measured volumes to the standard conditions of 0°C (273°K) and 760 mm of mercury pressure requires a knowledge of the fundamental gas laws, given in any text on general or physical chemistry.

The combined laws of Boyle and Charles state that the volume of a gas is inversely proportional to its pressure and directly proportional to its absolute temperature (°C + 273):

$$(1)\quad v \propto \frac{T}{p}$$

By introducing a proportionality factor, "k", expression (1) becomes

$$(2)\quad pv = kT$$

If this equation of state is applied to 1 mole of gas, "R", the molar gas constant, may be substituted for "k", and "V", the molar gas volume, may be used in place of "v". The equation can be made more general by introducing "n", which represents the number of moles in any given system. Equation (2) then is converted to the familiar ideal gas equation

$$(3)\quad pV = nRT$$

where "R" must be expressed in the same terms as those used for the pressure-volume relationship. For example, if the gas volume is expressed in liters and the pressure in atmospheres, "R" has a value of 0.08206 liter-atmospheres per degree. If the volume is expressed in cubic centimeters and the pressure in terms of millimeters of mercury, the value of "R" is 62,370 cc-mm per degree Kelvin.

In dealing with the gas mixtures encountered in industrial hygiene, Dalton's law must be applied first to correct the pressure of the total mixture by that portion of the pressure due to water vapor in the atmosphere. Dalton's law states that the total pressure of a mixture of gases is the sum of the pressures of the individual gaseous components. Therefore, the barometric pressure at the time the gas is collected minus the pressure of the water vapor (often termed the aqueous tension) is the actual pressure of the gas or gas-air mixture. The pressure due to water vapor at the temperature of gas collection, assuming gas saturation with water vapor, can be found in any handbook of physical constants.

With these laws the following equation can be used to convert the measured gas volume to standard conditions of temperature and pressure:

$$(4)\quad V_{S.T.P.} = V_{meas.} \times \frac{P_{bar.} - P_w}{760} \times \frac{273}{273 + t^{\circ}C}$$

where the volume is expressed in any convenient unit, such as liters, and the pressure is expressed in terms of millimeters of mercury.

Special Calculations and Conversion Formulas

The equation for the calculation of the actual volume of a gas collected in a partially evacuated flask is

$$(5)\quad V_S = V_F \left(1 - \frac{T_S P_1}{T_1 P_S}\right)$$

where V_S = volume of gas collected at absolute (°K) temperature T_S and pressure p_S

V_F = volume of the flask

p_1 = residual pressure of flask after partial evacuation for sampling

and T_1 = temperature of flask (°K) when p_1 was measured.

In applying a gas displacement method, the volume of the atmosphere being sampled or flushed through the flask should be 8 to 10 times that of the flask.[3] The time required to raise the concentration of the contaminant in the flask to 99 percent of its concentration in the entering air stream is given in the following equation:

(6) $t_{99\%} = 4.605\ a/b$

where t = time in minutes
a = volume of flask in liters
and b = air flow in liters per minute

The following conversion formulas are useful for gas or vapor calculations:

(7) mg per liter x 1000
= mg per cubic meter

(8) mg per liter x 28.32
= mg per cubic foot

(9) mg per cubic foot x 35.314
= mg per cubic meter

(10) mg per cubic meter x 0.02832
= mg per cubic foot

(11) parts per million =

$$\frac{24{,}450 \times \text{mg per liter}}{\text{molecular weight}}$$

One gram molecular weight of a gas occupies 24.45 liters at 25°C and 760 mm of mercury pressure.

Analysis

A complete review of the analytical methods which have been used successfully for the analysis of gas and vapor samples is beyond the scope of this lecture. These methods are given in detail in the reference texts[1,2,4] as well as an appended summary of satisfactory chemical methods for specific applications.[4] In addition to the methods suggested in these works, new techniques aimed at greater sensitivity and specificity are being developed which will be extremely helpful to the chemist. Of particular assistance will be the applications of vapor phase chromatography[5,6] and infrared spectrophotometry. These techniques provide powerful tools to assist the chemist faced with the problems of identifying and determining part per million concentrations of mixtures of substances which may interfere with each other in the application of certain conventional chemical methods. Such substances as the halogenated hydrocarbons have generally had no specific analytical methods and their estimation on an individual basis has been difficult. With vapor phase chromatography, the physical separation and determination of such mixtures is now becoming possible. Infrared spectroscopy is an important technique in the analysis of such materials and, along with vapor phase chromatography which effects the separations and provides increased sensitivity, can be expected to play an important role in this field.

References

1. Jacobs, Morris B.: The Analytical Chemistry of Industrial Poisons, Hazards and Solvents. Interscience Publishers, Inc., 2nd Ed., New York, N.Y., 1949.

2. Elkins, Hervey B.: The Chemistry of Industrial Toxicology. John Wiley and Sons, Inc., New York, N.Y., 1950.

3. Silver, S. D.: Constant flow gassing chambers: Principles influencing design and operation. J. Lab. & Clin. Med. 31:1153, (1946).

4. Goldman, Frederick H., and Jacobs, Morris B.: Chemical Methods in Industrial Hygiene. Interscience Publishers, Inc., New York, N.Y., 1953.

5. Scott, R.P.W., Editor,: Gas Chromatography 1960. Butterworth Inc., Washington, D.C., 1960.

6. Szymanski, H. A.: Progress in Industrial Gas Chromatography. Plenum Press, New York, N.Y., 1961.

INDUSTRIAL TOXICOLOGY

Introduction

The object of this section is to discuss the relationship of present-day industrial toxicology to industrial hygiene engineering and chemistry. Because a scientific discipline is no better than the technical texts that support it, the student should consult the book and journal references at the end of this section. The most recent and comprehensive text is the first listed, Patty's Industrial Hygiene and Toxicology, Vols. I and II, 2nd Revised Edition. "Detoxication Mechanisms" by Williams is a compendium of the metabolic products of approximately 2000 chemicals of industrial interest, and Elkins' text "Chemistry of Industrial Toxicology" presents practical analytic methods for determining the amounts of industrial chemicals in the environment and in the body tissues and fluids. Hunter's textbook on "Diseases of Occupation," presents interesting case histories of industrial poisonings and is very useful in identifying the cause of industrial diseases; similarly, von Oettingen's book on "Poisoning." The Handbook of Toxicology is a compendium of acute toxicity data for approximately 2000 compounds for various exposure routes. The remainder of the listings deal with special phases of industrial toxicology. Included also is a list of the more important journals dealing with industrial hygiene and toxicology, as well as reference to Hygienic Guides and Documentation of the Threshold Limit Values.

The Concept of Toxicity

Descriptive Definition

Toxicity is an inherent property of all matter that is manifested in a living physiologic environment to produce a derangement in that environment which, if sufficiently intense, is called injury. Injury is produced in response to some dose of a substance. A dose is the amount of the substance experienced over a given time interval. Expressed algebraically, C x T = K = some end point, usually death, where C = concentration, T = time. This is Haber's rule in its simplest form. Its importance lies in permitting predictions as to one of the variables in the equation if the other variable is altered. The conclusion holds generally for most substances, provided that neither variable varies greatly from values used in determining the constant K. K is usually synonymous with LD_{50} or LC_{50}. LD_{50} is the dose of a toxic agent that will kill 50% of a group of test animals. LC_{50} is the concentration of a given substance that when inhaled over a period of time will kill 50% of the animals under test. These values are listed for 2000 substances in the Handbook of Toxicology so that if either the concentration or the time is known, the other variable can be estimated for the particular circumstance in question.

Operational Definition

Toxicity may be regarded as the net effect of two opposing reactions: (1) Toxic substances act on the body; and (2) the body acts on the toxic substances. The net effect is a reduction in the toxic potential. This is diagrammatically represented in Figure 1. Recognition of these two opposing reactions permits several predictions regarding toxicity: (a) The observed toxicity will always be less than the true, potential toxicity; (b) the *in vivo* toxicity will always be less than that *in vitro*; and (c) other predictions from Figure 1 regarding synergism and antagonism will be given subsequently.

Figure 1 shows that several reactions occur in combination to reduce the concentration of the toxic agent by any of a number of routes of excretion and elimination. Of the amount of toxic agents that still remain in the body

Prepared by: Herbert E. Stokinger, Ph.D.

after these processes have acted, metabolic processes still further reduce the effective concentration of the toxic agent in the body. Detoxication, however, is a two-edged sword, as indicated by the reversed arrows, which suggests that not all "detoxication" products are less toxic than the parent substance; moreover, conjugating substances involved in detoxication may be of sufficient magnitude to act as a drain on needed reserves of essential body metabolites, thus acting to increase toxicity. As a net result, the observed toxicity is usually at a small fraction of the maximal toxic potential of any substance.

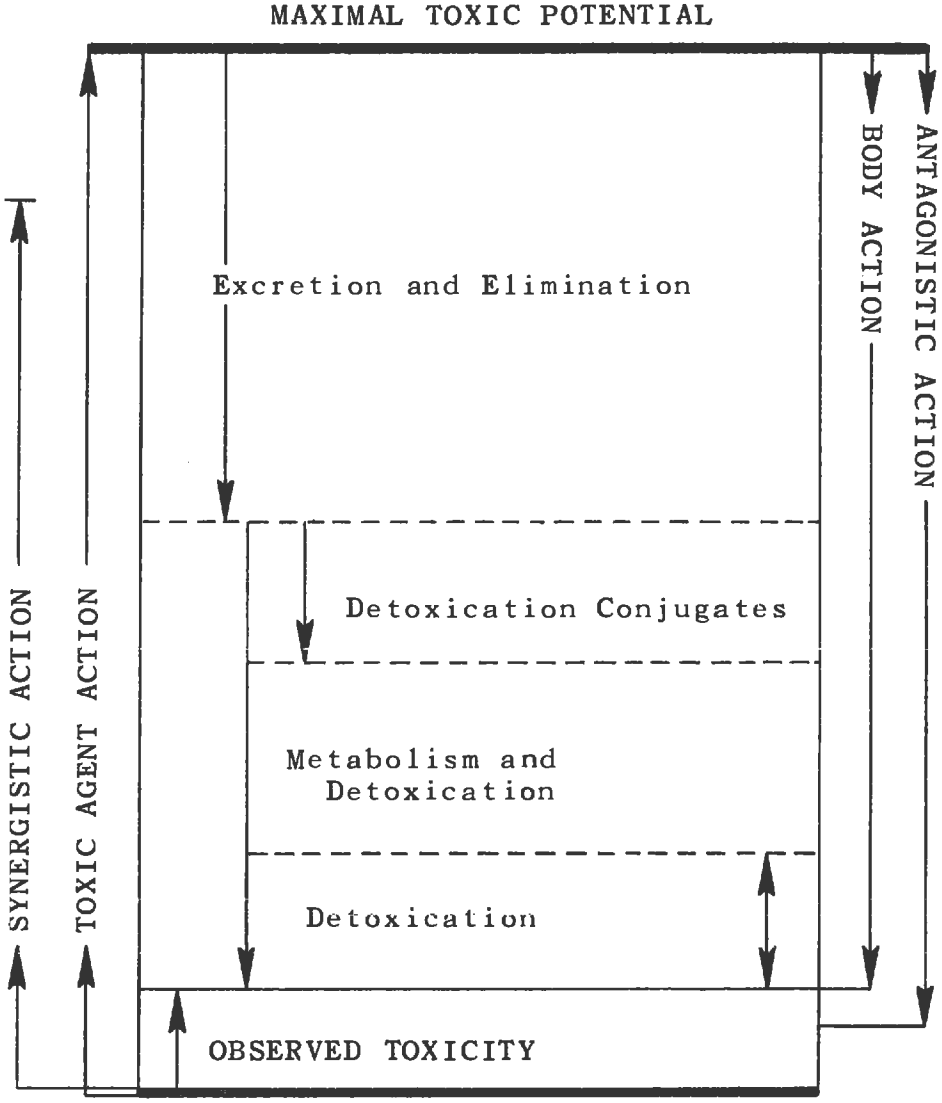
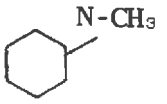
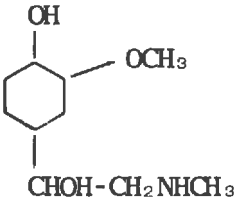
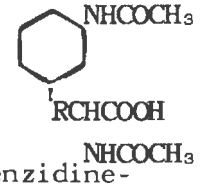
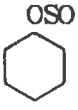
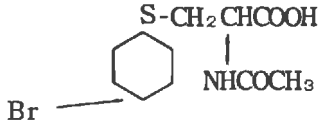
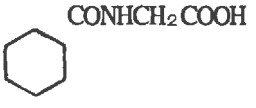


Figure 1. - Diagrammatic Concept of Toxicity.

Metabolism

Most industrial substances taken into the body are metabolized. Because they are foreign in chemical structure compared to ordinary foodstuffs to which the body and its enzymes are accustomed, the metabolism of industrial chemicals is commonly very incomplete. Accordingly, if its elimination and excretion is not rapid, the substance may remain in the body for considerable periods of time to exert its toxic action. Because there is frequently sufficient similarity between industrial chemical structures and natural foodstuffs to compete with sites of enzyme action, the normal metabolizing functions of enzyme are interfered with.

Table 1. - Major Types of Detoxication

TYPE	FOREIGN SUBSTANCE	DETOXICATION PRODUCT EXAMPLES
METHYLATION $-CH_3$	Inorg. Compounds of As, Te	$(CH_3)_2Se$
	Ring N compounds	
	Certain complex aromatic phenols	
ACETYLATION CH_3CO-	Aromatic Amines	
	Amino Acids (Known exceptions: aromatic amine carcinogens, also aliphatic amines).	e.g. Benzidine-hydroxylated aliphatic amines - aldehydes.
ETHEREAL SULFATE $-OSO_3H$	Phenols (Cyclohexanol glucuronide)	
Acetyl MERCAPTURIC ACID $-SCH_2CHCOOH$ $NHCOCH_3$	Aromatic Hydrocarbons Halogenated Aromatic HC's Polycyclic HC's Sulfonated esters $C_2H_5SO_3-CH_3$ Nitroparaffins ($C_4H_9NO_2$)	 C_2H_5 acetyl cysteyl- C_4H_9-acetyl cysteyl-
THIOCYANATE	Cyanide, inorganic Organic Cyanides (Nitriles)	RCNS
GLYCINE $-NHCH_2COOH$	Aromatic Acids Aromatic-aliphatic acids Furane carboxylic acids Thiophene carboxylic acids Polycyclic carboxylic acids (Bile acids)	

(Table 1 - cont.)

TYPE	FOREIGN SUBSTANCE	DETOXICATION PRODUCT EXAMPLES
GLUCURONOSIDE	Aliphatic (1^o, 2^o, 3^o) and Aromatic Hydroxyl	$OC_6H_9O_6$ (Ether)
	Aromatic Carboxyl	C=P $OC_6H_9O_6$ (Ester)
GLUCOSE HYDRAZONE	Hydrazine Hydrazine derivatives?	$NH_2N{=}CHC_5H_8O_5$

Detoxication

Detoxication, as indicated in Figure 1, is a form of metabolism wherein the body contributes some of its essential substances to conjugate with the substances being metabolized. There are eight recognized types of detoxication (see Table 1). Each type is more or less specific for a particular type of chemical configuration. For example, methylation takes place principally with nitrogen ring compounds, such as pyridine, acetyl with aromatic amines, etc. Williams' book on detoxication mechanisms shows not only the detoxication products of most of the important industrial chemicals, but shows the amounts in which these substances are excreted.

The significance of detoxication to industrial hygiene is:

1. That it offers a biologic means of identifying the agent to which a worker is exposed. For example, quantitative urinalysis permits an estimate of the intensity of the exposure.

2. A means of controlling exposures on an individual basis.

3. Permits development of biologic threshold limits relative to air threshold limits.

4. Safety evaluation of new products.

Biologic Threshold Limit Values

As the threshold limit values are a means of controlling the workers' air environment, a corresponding set of biologic threshold limit values may be developed for urinary metabolites. The biologic TLV's use the individual as his own screening agent and thus provide more individual control than commonly obtained from the threshold limit values for air. Far fewer biologic TLVs exist than air TLVs. The reason is that urinary or blood metabolites are commonly more difficult to obtain, more difficult to analyze, and in some instances, are too heavily dependent upon individual idiosyncrasies of metabolism or effects of the toxic substance on metabolism. More investigation on this subject is needed, however.

Synergism and Antagonism

Synergism may be defined as the enhancement of toxicity above that commonly expressed when the toxic agent is used in combination with other substances.

The enhancement may be several-fold. Antagonism is the opposite expression of toxicity when two or more substances are taken into the body in combination; antagonism may result in complete abolition of toxic effects, or toxicity may be partially reduced. Reference to Figure 1 provides a simple straightforward explanation of these two types of combined toxic action. It can readily be seen that if some substance can act to interfere in some way with either the processes of metabolism, detoxication, excretion and elimination, then the observed toxicity will be increased (synergistic action). On the other hand, if the presence of other toxic agents tends to enhance any of these processes, antagonistic action will result. It has been shown for a number of substances that the way these actions occur is through alteration in the normal processes of metabolism through competition of the two or more agents for the same enzyme site. This results in blocking the normal metabolism of the toxic agent, thus tending to maintain its concentration at its original peak, and consequently its toxicity. Altered toxic responses may also be brought about by simultaneous action at different sites, the result of which may either act to reinforce or counteract the toxic effects of each agent.

Development of Threshold Limits For Air

Reference to Figure 2 shows the various types of threshold limits for air, water, biologic tissues and fluids, the special medical procedures for determining these limits, and the various toxicologic components that are involved in the development of threshold limits.

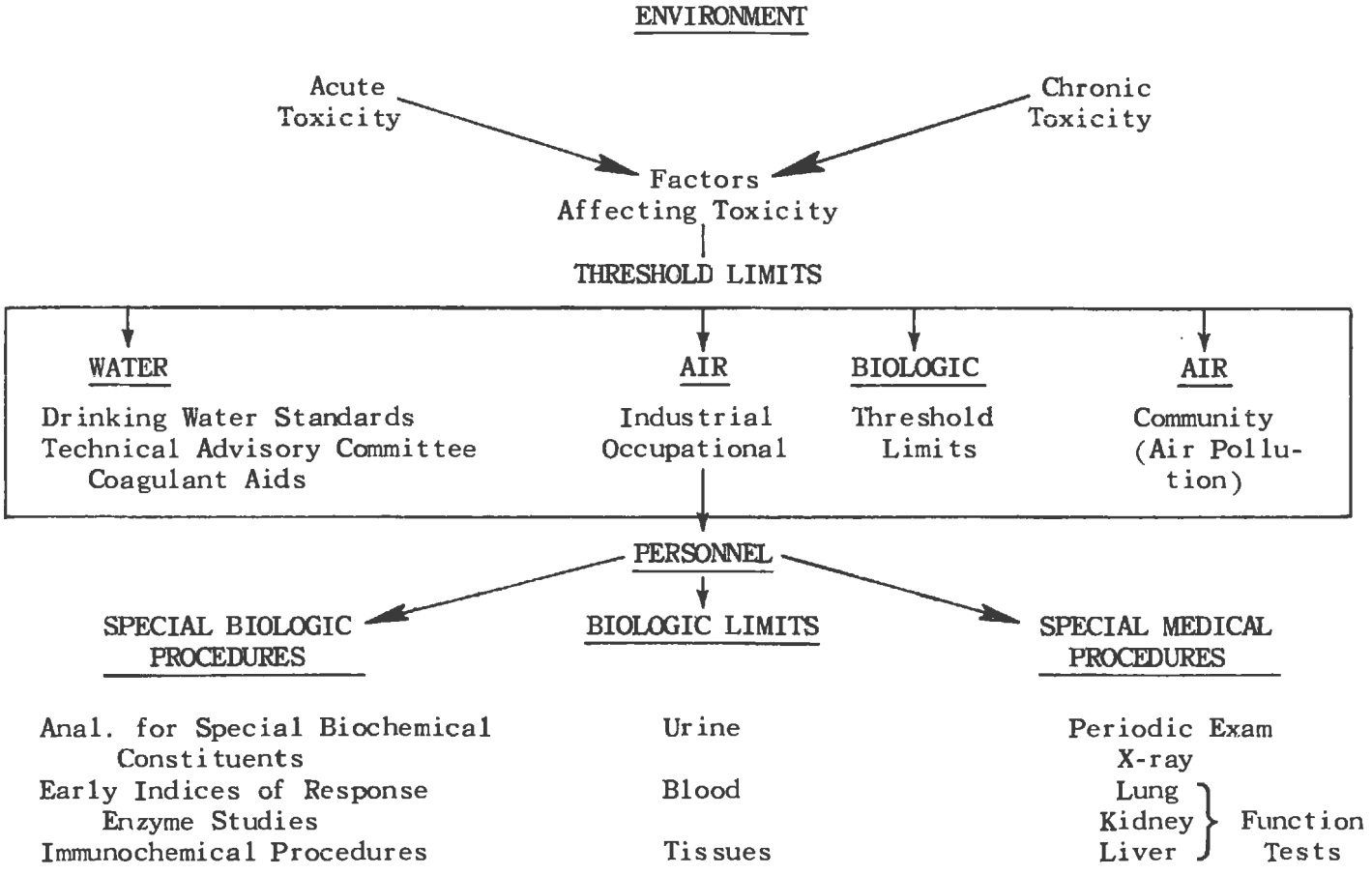


Figure 2. - Objectives and Procedures in Preventive Industrial Toxicology.

All of the four threshold limits are of concern to the present-day industrial hygienist. Whereas in the past the working environment inside the plant was the industrial hygienist's main concern, today not only must the in-plant environment be safe but also his efforts are required to control community air pollution and the effluents in the streams. It is seen also that both acute and chronic toxicity are basic essentials for the development of threshold limits, but these alone, extensive as they are, are not sufficient for the purpose. Factors affecting toxicity may in certain instances so modify the toxicity of a compound even under usual conditions of exposure as to be a major determinant in the setting of a threshold limit.

The three basic aspects, acute toxicity, chronic toxicity, and factors affecting toxicity, will now be discussed in relation to their value in recommending threshold limits.

Acute Toxicity

Before chronic inhalation toxicity studies are planned, it is usually advantageous to perform a certain number of acute "pilot" tests. The purpose of such tests is two-fold: (1) to learn as quickly and inexpensively as possible the approximate range of toxicity of the test substance by various routes of administration as a basis for emergency exposure limits; and (2) to determine the nature of the toxic effect and something of the manner in which the substance acts, its fate in the body, and any unusual or other toxicologic actions that may be appropriate to the circumstance for determining appropriate procedures for the chronic studies. Such information permits a better "zeroing-in" of the different disciplines of toxicology (biochemistry, pharmacology, physiology, hematology) to make the subsequent chronic inhalation studies as revealing and meaningful as possible.

Determination of Acute LC_{50}

First in importance naturally, is the determination of the acute inhalation LC_{50}, usually made on rats or mice. This determination not only establishes the inhalation toxicity of the test substance relative to that of other well-known substances, but provides an opportunity to observe the gross signs of toxicity, the type of response, and most important, establishes the range of exposure levels for the chronic inhalation studies. In general, a factor of from 10 to 100 represents the spread between the acute inhalation LC_{50} level and the level productive of definitive response in the chronically exposed animal.

Determination of Acute LD_{50}

This oral test should also be made (usually on rats) on substances under study because it not only establishes an important toxicologic guidepost, but also because it shows the toxicity of the substance by the oral route relative to that by inhalation, and hence shows the relative contribution by the oral route to the over-all toxicity from inhalation. This is particularly true of test substances that are solid, insoluble particulates, the greater proportion of which find their way into the gastrointestinal tract following inhalation. Should the oral toxicity of a particulate substance under test be relatively high, a significant contribution to the over-all (observed) toxicity would be made by that portion of the inhaled dose reaching the intestinal tract. Conversely, if the oral toxicity is found to be relatively low (one-100th or less) compared with inhalation toxicity, the factor of oral toxicity may be disregarded.

Dermal and Percutaneous Toxicity

The capacity of the test substance to react with the skin, both as a contact irritation and sensitizer, should be established. Dermal reactions are a common type of response to industrial

chemicals (e.g. chlorinated hydrocarbon solvents) and if the substance is percutaneously absorbed to a significant degree (as in the case of nitroglycerine, benzidene, aniline, and parathion, for example) consideration must be taken of such a contribution to the toxicity in recommending a threshold limit.

Ocular Toxicity

Because effects on the eye, and even systemic absorption by this route, can be an important part of the over-all exposure problem, the effect of the test substance in the eye should be determined. For some substances, such as certain substituted aliphatic imines, amines and diamines, ocular toxicity may be one of the basic determinants in recommending a threshold limit.

Supplemental Studies

There is an increasing number of industrial chemicals that, by virtue of their chemical structure and configuration, might be expected to exhibit special or unusual toxicologic actions. Among such compounds are chelating agents, non-metabolizable substances, free radical or free-radical-yielding compounds, substances with mutagenic or carcinogenic potential, substances with specific effects on endocrine or blood-forming organs, and on reproduction, and finally hemolytic agents. Tests of such structures should include the appropriate procedures, in addition to the basic procedures discussed above. The type of structures producing these specific responses are now recognized in many instances.

Tailoring Research to Needs

The general principle that may be derived from the foregoing is that test procedures will delineate the special toxicologic characteristics of the substance under test. The day is past when routine, unimaginative and short cut, procedures are acceptable as evidence of toxic potential. A study of the fate or metabolism of the test substance may reveal how rapidly and completely the substance is eliminated from the body. If it is a volatile solvent, a relatively simple determination of the amount exhaled in the breath may show the rapidity of elimination, thus indicating its relative hazard. Similarly, determination of urinary excretion will show the rate at which the systemically-absorbed material is eliminated. More detailed study of urinary metabolites may give important clues as to the ease with which the body can metabolize, convert, or detoxify the test substance and at the same time may provide a simple test for gauging degree of exposure, and thus subsequently aid in control of worker exposure.

Studies of Subacute Inhalation Toxicity

The purpose of these studies is to develop information to bridge the gap between acute and chronic toxicity studies to permit: (a) More detailed delineation of sign of response to exposure; (b) to determine the nature and rate of accumulation of a substance in the body or accumulation of its effects and manner; and (c) to allow a better estimate of appropriate levels for chronic exposures. The duration of such studies is usually from 30 to 60 days of 6-hour daily exposures, 5 days per week. Usually, three species of animals are exposed at a minimum of two exposure levels, e.g., those producing frank injury in at least one species to permit delineation of the toxic response and another at a level below the LC_{50} to provide an indication of the appropriate level for a "no effect" response during the chronic study.

Studies of Chronic Toxicity

The purpose of these studies is to provide basic data central to the entire objective of affording long-term information for the threshold limit. Here the duration of the study is a minimum of one year, with two years preferred, of six hour daily exposures repeated

five consecutive days per week. The number of animals is greatly increased to permit periodic sacrifice for biologic assay to record the progress of the study. The number of animal species, however, may be reduced to a minimum of two, of which one is a nonrodent. A minimum of two exposure levels is used guided by the same principles as obtained for the subacute inhalation toxicity studies.

Factors Affecting Toxicity

Having developed the basic toxicologic data in animals, it is a long step to recommending a threshold limit value. One of the factors is the numerous conditions that may modify the toxic response. The other factors are the correlation of all the various experimental data and their interpretation and interpolation for converting the animal data to man. (Space permits a consideration of only a few of the many factors affecting toxicity).

Structure. First and foremost of these is the structure of the compound. For example, the removal of but two hydrogen atoms may completely modify the toxicity of the compound; the positioning of a double bond can alter the toxicity of a chemical; and the positioning of a particular atom will also modify toxicity.

Particle size. The effect of particle size on the deposition and retention of particulates in the lung is also an important consideration. Decrease in particle size usually increases toxicity in three ways: (1) By remaining airborne, and thus permitting a greater exposure for longer periods of time; (2) by actually depositing greater amounts of material in the lung; and (3) by being retained longer. Thus it is possible by decreasing particle size to modify the toxicity of particulates ten-fold. Based on this knowledge, present-day sampling procedures in the plant utilize instruments that collect only those fractions of the suspended particulate of inhalable size, 5μ and below.

Surface area. In addition to size, surface area also greatly modifies toxicity. The toxicity of beryllium oxide dust may be altered considerably depending upon its surface area.

Solubility. This factor determines the site of action of toxic agents. If the site is altered, so is the toxicity. SO_2 for example, because of its solubility and at normal exposure levels, never proceeds to the deeper portions of the lung. Thus it is less toxic than those substances that do penetrate deep into the lung.

The effect of nutrition may be equally great on toxicity. For some substances the nutritional effects may predominate. Experiments in this laboratory have shown that the effect of suboptimal diets greatly increase vanadium and carbon disulfide toxicity.

Although animal studies form the basis for the majority of the TLVs, long-term observation of the worker related to his environment may also serve as a basis. Greater difficulty, however, is associated with the determination of the exposure of the worker and the degree of his response, because in general it is difficult to confine a worker's exposure to a single substance or at a uniform or precise concentration. However, unless the predominant effect of exposure is irritation or narcosis, it is not usually possible to determine the onset of injury.

Bases and Definition of Threshold Limit Values for Air

Threshold limits for industrial air are based on the premise that although all chemical substances are toxic at some concentration, experienced for a period of time, a concentration exists for all substances from which no injurious effect will result, no matter how often the exposure is repeated. A similar premise applies to substances whose effects are limited to irritation, narcosis, nuisance or other forms of

stress. Thus the philosophy for limits for chemical substances differs from that of ionizing radiation for which there is theoretically no threshold. A few exceptions exist, mainly those with radiomimetic potential. The philosophy expounded in the U.S.S.R. inclines to viewing all toxic substances in a manner similar to ionizing radiation on the premise that workers should not be subjected to additional stress of chemical toxicity and irritation producing demonstrable response beyond the normal stress of everyday living.

Ceiling and Time-Weighted Average Values

The time-weighted average concentration provides the most satisfactory and practical way of monitoring air-borne agents for compliance with the limit. Certain substances, however, whose action is chiefly irritation, narcosis, or productive of serious long-term effects from a single or few peak exposures, require a ceiling value. In contrast to the time-weighted average, the ceiling value represents one that should not be exceeded. The basis for assigning C values, as well as the permitted fluctuation for the time-weighted average values are explained in the preface to the Threshold Limit Values published annually by the American Conference of Governmental Industrial Hygienists.

In addition to the importance of understanding the definition of the threshold limit value, there is another most important consideration, namely that threshold limit values are guides for the reasonable control of the working environment. They are not fine lines between safe and dangerous concentrations.

Documentation

Recognizing the shortcomings of a single number for use as a guide in controlling the workers' environment, all threshold limit values have been documented with complete pertinent literature references. Revised editions of the Documentation of Threshold Limit Values appear from time to time.

Emergency Exposure Limits

As a result of man's changing activities, the need has become acute, particularly among the Armed Forces, for another type of air exposure limit, namely short-term or emergency exposure limits, (EEL). EEL's are designed to give guidance in the control of single, brief exposures to air-borne contaminants in the working environment. They are designed for unusual conditions of exposure for concentrations that can be tolerated without adversely affecting health permanently, but not necessarily without acute discomfort or other evidence of irritation or intoxication.

References

BOOKS

1. Patty, F. A., Industrial Hygiene and Toxicology, John Wiley & Sons, New York, New York, Vol. I, 2nd Rev. Ed., 1958, Vol. II, 2nd Ed., 1963.

2. Williams, R. T., Detoxication Mechanisms, Chapman & Hall, London, 1959.

3. Elkins, H. B., Chemistry of Industrial Toxicology, John Wiley & Sons, New York, New York, 2nd Ed., 1959.

4. Hunter, D., Diseases of Occupation, Little Brown, Boston, Massachusetts, 1962.

5. von Oettingen, Wm. F., Poisoning; Hoever, N. Y., 2nd Ed., 1958.

6. Handbook of Toxicology, WADC Tech. Rept. No. 55-16, Spector, Ed., 1955.

7. Browning, Ethel, Toxicity of Industrial Metals, Butterworth, London, 1961.

8. The Halogenated Aliphatic, Olefinic, Cyclic, Aromatic, and Aliphatic Aromatic Hydrocarbons, Including the Halogenated Insecticides, their Toxicity and Potential Dangers, von Oettingen, Wm. F., PHS Pub. 414, Washington, D.C., 1955.

9. Gerarde, H. W., Toxicology and Biochemistry of Aromatic Hydrocarbons, Elsevier, New York, 1960.

10. Eckardt, R. E., Industrial Carcinogens, Grune & Stratton, N.Y.C., 1959.

11. Henderson & Haggard, Noxious Gases; Rheinhold, N.Y., 1943.

12. Stokinger, H. E., Toxicology of Air Pollutants in "Air Pollution" Editor, Stern, Academic Press, 1962.

JOURNALS

American Industrial Hygiene Journal
AMA Archives of Environmental Health
British Journal of Industrial Medicine
Industrial Hygiene Digest, Abstracts
Industrial Medicine and Surgery
Journal of Occupational Medicine
Toxicology & Applied Pharmacology

SUPPLEMENTAL SOURCES

Hygienic Guides, American Industrial Hygiene Association

Documentation of Threshold Limit Values, Am. Conf. Govt'l. Ind. Hyg.

THE PHYSICIAN IN INDUSTRY

The physician in industry enjoys a unique position in the industrial complex. He is an employee with special privileges and responsibilities as he plays a double role. On one hand, he acts in the company's behalf in outlining protection for the company in its future responsibility to its employees and helps define the company's position in individual cases of injury or disease. When he puts on his other hat, so to speak, the company physician becomes a family doctor ministering to the needs of the man or woman not, perhaps, primarily as an employee but as a sick or injured human being in need of medical care. This dual role cannot be defined very specifically in that the two roles are intertwined, sometimes indistinguishable and often confused. In this presentation, the emphasis will be placed on the role of the company physician in determining whether injury or illness has occurred or is likely to occur among workers under his jurisdiction.

The industrial physician's relations to the individual worker might be divided into several categories:

1. Pre-placement examinations
2. Periodic examinations during employment
   a. early diagnosis (pre-clinical)
   b. clinical diagnosis
   c. treatment
   d. prevention
3. Separation examinations

Before outlining the kinds of tests used by the physician to evaluate an employee's reaction to his industrial environment, it must be pointed out that the basis for denoting any change lies in having base-line, pre-placement or comparable periodic data on workers. Any evaluation of the function of the body must be as thorough in the base-line tests as in any subsequent tests contemplated. It is obvious that if the lungs were not X-rayed before employment, an X-ray at some future time would have less meaning than if there were a comparison film before an industrial exposure, which could have contributed to a change in the X-ray picture.

The industrial physician must know a good deal about the kinds of processes and products which are used and manufactured in the plant(s) he serves. Although the physician does not receive in his medical training anything more than the barest fundamentals of occupational medicine he will have to learn just what goes on in the plant in order to recognize and treat the industrial diseases which come into his office. During this training the industrial hygienist, engineer or chemist will contribute to the physician's knowledge by filling in the details of the industrial processes involved in the business which the physician serves. The hygienist may also be able to provide some knowledge of the toxicity of particular products and a good deal of data regarding the type and variations in environmental hazards in a specific job situation. The introduction of a new process or product, or the use of new materials, must certainly be brought to the attention of the medical department so that any unusual manifestations of illness or injury can be recognized and identified. The doctor may also call on outside sources of information such as consulting firms or university organizations or Local, State and Federal occupational health facilities.

A pre-placement examination, general in nature but specific in areas pertinent to the job under consideration, provides a valuable baseline against which

Prepared by: William S. Lainhart, M.D.
and Lester D. Scheel, Ph.D.

future health measures can be assessed. Because of the variability in what might be considered normal in the laboratory determinations which a doctor may order, trends in periodic test results can be more accurately evaluated using baseline data.

In addition, the pre-placement examination can be used to protect both the employee and the employer. Thus a pre-existing disease entity might be reactivated or aggravated by a particular occupational environment, producing difficulties for both parties. A lung, already damaged by silicosis, may well be very susceptible to further damage due to the inhalation of other dusts, for example, coal dust. The employee becomes more disabled and the company has an added burden of compensation. There are certain physical handicaps which would preclude a man's performing the functions of his proposed job so that a man should be matched to his job. Even if no overt disease were present, a tendency toward abnormality might be ascertained. An individual with borderline liver function tests should not be placed in a job where exposure to halogenated hydrocarbons might take place.

When the physician has acquired a working knowledge of the health status of the workers in his plant, he then should watch carefully the health of those workers throughout their employment. Depending on the toxic hazards inherent in the job or the processes under use, periodic examinations may take many forms and the periodicity must be tailored to the individual situation. The possibility of lead poisoning in a processing or manufacturing plant immediately suggests a screening test for lead exposures. Such tests may include the following:

(a) basophilic stippling of the red blood cells; and
(b) urinary and blood lead analyses.

These tests are suggestive of an increased absorption of lead into the body. The basophilic stippling test for lead absorption might well be considered a form of pre-clinical diagnostic test because the stippling of the cells can precede any signs or symptoms noticeable to the patient or his physician.

Studies are underway here at the Occupational Health Research and Training Facility of more sophisticated tests of enzyme changes following exposure to toxic materials which might be used to exclude susceptible or reactive prospective employees from work areas where those materials would be used. Although these tests are presently still in the early stages of development it is anticipated that they will provide the physician with a valuable tool in the monitoring of the occupational environment.

During the course of the employee's tenure at the plant or office, he should receive medical surveillance tailored to preventing disease or injury. This involves using many of the tests previously administered at the time of employment. However, when such disease or injury occurs, the employee has the right to expect treatment and/or compensation if his health has been adversely affected by this work. This treatment may take one of many forms: first aid, hospitalization, surgery, etc. The industrial physician is responsible for arranging for such services and following through on the employee's convalescence. This includes close liaison with local private and public health authorities.

At any of the levels of the physician-employee relation outlined earlier, the worker may present himself with one or more of a multitude of complaints. He may even have no complaints but has been found to have abnormal results on some type of screening or periodic test. The physician needs then to categorize the physical findings into an outline such as the following, which might be characterized as a systems review:

1. Respiratory tract
2. Digestive tract
3. Genitourinary tract
4. Nervous and psychiatric system
5. Neuro-muscular system
6. Cardiovascular system
7. Skin
8. Special senses - ear, eye, and nose
9. Infectious diseases
10. Immunological diseases
11. Miscellaneous disorders including alcoholism
12. Undiagnosed disorders

Although it is impossible to outline all the tests available to investigate all the conditions which could be listed in a system review, a sampling of the tests and a notion of some of their uses might provide the non-medical industrial worker or consultant with clues as to the conduct of the medical department and to ways that employees might contribute to and utilize the facilities of the medical department.

The most obvious way to "look at" the respiratory system is the chest X-ray or roentgenogram. In addition to this valuable tool, however, the physical examination and history can help the doctor in his diagnosis. Examination of the sputum can give him valuable clues. More complicated procedures such as bronchoscopy and lung biopsy are needed only in cases of difficult diagnoses in seriously ill patients.

Again, the X-ray is very valuable in ascertaining disorders of the gastrointestinal tract but the history of complaints can help the physician reach a decision regarding the diagnosis. Also, more esoteric procedures can be used to investigate the physiology of the digestive tract. Gastric washings (removal of the stomach contents by tube) may provide clues to certain industrial poisonings.

Urinalysis can provide information about a host of disorders (both urinary and other) so is considered nearly routine for most examinations. Lead excretion through the kidneys is well-known and the urinalysis is a valuable tool in diagnosis of lead poisoning.

In the case of the nervous, psychiatric and muscular disorders, the history and physical examination are of primary concern. Psychological tests and tests for muscle tone, electrical conductivity, and the like are used occasionally.

The cardiovascular system may be investigated by the use of the stethescope, the sphygmomanometer (blood pressure cuff), and the electrocardiogram in addition to the history and physical examination. The blood itself may be subjected to a host of tests.

Although space does not permit elaborating all the tests which might be used, these examples indicate some of the myriad ways that the environment can alter the physiological functioning of the human organism. The non-industrial environment including the psychological one can adversely affect the employee's performance on the job. The industrial physician, therefore, must think of the total man or woman, must consider his working environment as well as his home and recreational environment and his reaction to the various environments in which he finds himself.

It is by judicious use of laboratory diagnostic tests combined with a knowledge of the environmental working conditions that the plant physician can evaluate the problems presented by his patient. Changes found by laboratory diagnostic tests correlated with environmental data make it possible to use the employed population as a bioassay group for the industrial process under surveillance. The presentation of his findings based upon the factual analysis of test data thus allows the physician to carry out his doctor-patient responsibilities and to discharge his responsibility to the company by providing management with early indications of clinical changes which may be related to the production process.

Only by utilizing all the resources potentially available to him, can the industrial physician satisfy his role as a caretaker of the health of the workers under his purview. His first source of information is his medical background but a close second source is the industrial hygienist, engineer, or others who can provide the technical background so that the physician can more intelligently and completely minister to the needs of the workers in his plant.

SPECTROPHOTOMETRY

Introduction

Spectrophotometry involves the measurement of the relative amount of radiant energy as a function of wavelength. The application of spectrophotometry that perhaps has greatest significance in industrial hygiene chemistry is that which deals with the absorption of radiant energy by colored solutions (visible range). Absorption in the ultraviolet and infrared regions is also significant but will not be covered to any great extent in this Section. Analytical methods based on the formation of a colored component or end product are very important for the determination of the concentration of trace constituents. Colorimetric methods are often the most sensitive if sufficient sample is available. While spectrographic methods have the highest absolute sensitivity, their requirements of relatively small sample portions sometimes limits their usable concentration ranges.

Colorimetric procedures for various determinations are presented elsewhere in this Syllabus. These involve the preliminary solution or ashing of samples, followed by various concentration steps such as extraction or distillation to remove interfering substances. A wide range of reagents may then be used to produce colors with various constituents. This discussion will deal with the measurement of absorbance and its conversion to quantity of constituent. The theory of spectral absorption, types of errors to be expected, and handling of data and calculations will be outlined.

General Considerations

The ideal color forming reagent should have the following qualities:

(a) stability
(b) stoichiometric reaction
(c) rapid color development
(d) transparency in spectral region involved
(e) selective or specific reaction
(f) freedom from interference
(g) high capacity
(h) solubility in satisfactory, available solvents.

Methods of color development include:

(a) redox methods
(b) complex formation
(c) diazo and coupling reaction
(d) condensation and addition
(e) salt formation
(f) chromophoric changes in valence
(g) substitution

The ideal color system has the following properties:

(a) stability
(b) high intensity
(c) minor variation with pH, temperature, and other factors
(d) absorbance in the appropriate spectral region
(e) solubility of the colored product
(f) conformity to Beer's law

Absorption of Radiant Energy

The Electromagnetic Spectrum

Visible light occupies a relatively small region in the known range of radiation. Cosmic rays have a wavelength of less than 0.001 A (the angstrom unit A = 10^{-8} centimeters). Nuclear gamma radiation extends from 0.001 to 0.1 A and X-rays from 0.1 to 100 A. The ultraviolet region extends from 100 to 4000 A, followed by the visible region from 4000 A (violet) to 7500 A (red). The near infrared region extends from 7500 A to 250,000 A (0.75 to 25 microns), while the far infrared region extends from 25 to 1000 microns. Longer wavelengths occur in the microwave and radio

Prepared by: Bernard E. Saltzman, Ph.D.
Revised by: Richard E. Kinser

wave regions which extend to several hundred meters in wavelengths. From this it is evident that the visible region represents only a very small portion of the known range of radiation of the electromagnetic spectrum.

Absorption of High-Energy Radiation

In the interaction of radiation and matter some or all of the energy is absorbed. A fundamental principle of such interaction is that the radiation behaves like packets of energy (photons) rather than like waves. The energy of each photon is inversely proportional to the wavelength of the radiation. Many types of interaction between photons and matter occur simultaneously. However, certain interactions predominate for each energy or wavelength range. The most important process for absorbing the energy of very high energy photons (cosmic rays and high energy gamma rays) is by collision with atomic nuclei resulting in disintegration or ejection of particles, and transmutation to another element. In the next lower energy range photons are absorbed in the vicinity of atomic nuclei with the creation of electron-positron pairs, each particle traveling in opposite directions. In the low energy gamma range and the X-ray range most of the energy may be absorbed by collision of the photons with electrons. Energy and momentum are conserved in such a collision which produces a lower energy photon (longer wavelength) and a high speed electron. This process is known as the Compton effect. In the lower X-ray and ultraviolet regions the most important energy absorbing process is the photoelectric effect in which the photons are absorbed in the vicinity of atomic nuclei, and their energy is transmitted to electrons which are ejected.

Quantum Absorption of Low-Energy Radiation

Another process in the ultraviolet and visible region, which is the basis of spectroscopy, is the absorption of photons with energies insufficient to eject electrons from their atoms. The quantum theory is a fundamental principle which states that electrons can exist only in certain orbits which have fixed energy levels for each element. If a photon has the exact energy required to raise the electron to one of its higher energy orbits the photon will be strongly absorbed. Photons of other energies are not absorbed. Thus the absorption spectrum of a gas exhibits sharp bands of absorption in certain narrow wavelength ranges, corresponding to differences in electronic energy levels of the atoms present.

The important energy absorbing processes for spectrophotometry occur in the ultraviolet, visible and infrared regions. All relate to molecular energy levels rather than atomic energy levels. According to the quantum theory molecules also have certain fixed possible energy levels. If a photon has the exact energy required to raise the molecule from its normal low energy state to one of the higher energy levels it will be strongly absorbed. The aromatic benzene ring has a characteristic absorption pattern in the ultraviolet region. This pattern is altered somewhat as various substituent groups are added to the ring. In the visible light region certain chromophoric groups such as N = N absorb strongly, producing colored solutions. The infrared region is particularly rich in absorption bands corresponding to the energies of various stretching and vibrating elemental bonds. In the far infrared and microwave region the energy levels corresponding to molecular rotations and vibrations become the most important ones. Such absorption patterns are invaluable for determining molecular structure.

Quantitative Relations in Spectrophotometry

The basic law of spectrophotometry is that for light of a single wavelength (monochromatic) each molecule of colored solute absorbs the same fraction of the

radiant energy that falls upon it. This may be expressed in differential form by equation (1):

$$(1) \quad \frac{-dI}{I} = KCdl$$

and in integrated form by equation (2):

$$(2) \quad \ln\frac{Io}{I} = 2.303 \log_{10}\frac{Io}{I} = KCL$$

In equation (1) the left side represents the fraction of light absorbed, the -dI being the light intensity absorbed and I being the total light intensity. On the right side, the product of the concentration C and the differential length of solution dl is proportional to the total number of absorbing molecules. The proportionality constant K represents the fractional absorption per unit length of absorbing path. In the integrated form, equation (2), I_o represents the incident light intensity and I represents the emerging light intensity.

Absorbance is a convenient term defined as $\log_{10} (I_o/I)$. Many instruments have dial calibrations giving this value (synonymous with optical density) as well as percent transmittance. Absorbance and concentration are related linearly.

The specific extinction coefficient k is defined as the absorbance per unit concentration and unit cell length:

$$(3) \quad A = kCL$$

Where A is absorbance, C is concentration and L is the cell length.

The molecular extinction coefficient ϵ is defined as the absorbance per mole per liter, per centimeter light path:

$$(4) \quad A = \epsilon C'L$$

Where C' is concentration in moles per liter.

The relationships between the various coefficients are:

$$k = \epsilon/\text{molecular weight}$$
and $k = K/2.303$

Transmittance, T, is a commonly used term denoting the value of the fraction I/I_o. If the photometer gives readings in terms of transmittance, they may be converted to absorbance as follows:

$$(5) \quad A = 2 - \log_{10}\%T$$

Percent transmittance values may be used directly if plotted on semi-log paper against concentration of absorbing species, giving a straight line of slope - kL:

$$(6) \quad \log_{10}T = -kCL$$

It can be seen from the definition that 100% transmittance corresponds to zero absorbance, while 10% transmittance corresponds to 1.0 absorbance.

The above equations (3) (6) present a more complete statement combining the earlier Bouguer's (Lambert's) Law, stating that each unit thickness of a light absorbing medium absorbs an equal fraction of radiant energy traversing it, and Beer's Law, which states that the fractional absorption of light by a thin layer of solution is directly proportional to the concentration of the absorbing solute.

Analytical Application of Beer's Law

In order to secure close compliance with Beer's Law, dilute solutions should be used with monochromatic light. If a broad band of light of many wavelengths is used, it can be readily shown mathematically that although each particular wavelength is absorbed according to Beer's Law, the sum of all the light energy in the band will not follow this relationship. Deviations from Beer's Law may occur also in more concentrated solutions due to association of the absorbing molecules into a different

molecular species. If the ratio of the two species is not a constant in the concentration range being measured, then the total absorption, which would be the sum of the two different species, will not conform to Beer's Law. Other deviations occur if the colorimetric reaction is not stoichiometric because of insufficient excess of colorimetric reagent or the presence of interfering substances.

In actual practice, a series of standard colors is prepared by applying the procedure to known amounts of the substance being determined. A plot is made of the determined absorbances versus the concentration or quantity of substance. If this plot exhibits a linear relationship, Beer's Law is followed. If such is the case it is more convenient in analyzing unknown samples to use a mathematical calculation to relate quantity of substance to absorbance values rather than a graph. The standardization factor is obtained from the graph of the standard sample series as the number of micrograms of substance required to produce an absorbance of exactly 1, when the plot is made with all values corrected by deducting the reagent blank. The corrected absorbance of the unknown multiplied by this standardization factor gives the micrograms of material being determined in the unknown sample. If Beer's Law is not followed the curved plot must be used for the conversion. This colorimetric determination is still perfectly valid, although not as convenient.

Color Comparators, Absorptimeters and Spectrophotometers

Visual Comparators

Nessler tubes. The simplest type of color measurement is by visual comparison in matched test tubes or Nessler tubes. The visual spectral response curve is a bell-shaped pattern with a peak of 555 millimicrons. Intensity differences of 2% may be detected in the most favorable wavelength range. Differences of 15% to 20% are detected at the extreme ends of the wavelength range. This method has the disadvantage of requiring an extensive set of standard colors, which is doubly inconvenient if the colors are not stable.

Duboscq colorimeter. Another method of measurement is by means of a Duboscq colorimeter. This requires only a single standard color. The unknown is matched against the standard color by varying the path lengths. This is done by viewing the two colors downward through glass plungers which may be moved vertically into the two cups containing the colored solutions to give variable path lengths between the bottoms of the plungers and the bottoms of the cups. From equation (2) when the colors are matched $C_1L_1 = C_2L_2$. From the known standard C_1 and the known values of L_1 and L_2 the unknown C_2 may be calculated.

Wedge color comparator. In another type of visual comparator the balance is obtained by means of a glass wedge of a neutral gray tint. Colored filters are used to select a narrow wavelength band of light. When the balance is obtained the values of transmittance or absorbance may be obtained from the wedge calibration.

Photoelectric Absorptimeters

Klett-Summerson. In a more advanced type of absorptimeter the comparison is made with photoelectric cells rather than with the eye. The Klett-Summerson instrument utilizes a 100 watt projection lamp, condenser and colored glass filter as a light source. Half of the beam passes through a mechanical balancing diaphragm to a reference photocell. The other half passes through the sample cell to the sample photocell. Barrier layer type photocells are used so that no power supply is necessary for this portion of the circuit. The output of the sample photocell is balanced on a calibrated potentiometer against the output of the reference photocell using a sensitive galvanometer to give the null

point. The potentiometer is calibrated directly in units proportional to absorbance. Variations in line voltage produce light intensity fluctuations but these are cancelled out by the electrical balancing system. The mechanical balancing diaphragm is adjusted to give a zero reading with the reference material in the sample cell.

Monochromatic sources. Filters are inexpensive and are generally made more selective by combining two or more colored glass plates or dyes. A newer type is based upon interference effects and is much sharper in wavelength band characteristics but also considerably more expensive. Interference filters are used in the Bausch & Lomb monochromatic colorimeter. Readings are obtained using a mirror galvanometer giving a cross hair spotlight beam on a glass scale.

It has been mentioned that conformance to Beer's Law requires truly monochromatic light. More elaborate instruments provide a monochromatic beam by the use of various prism or grating systems in conjunction with mirrors and slits. Diffraction grating instruments have a uniform dispersion of light at various wavelengths. Prism type instruments condense the colors in the red region.

Light detectors. The best light detecting devices are photomultiplier tubes and blue sensitive photocells in the ultraviolet and blue end of the scale and red sensitive photocells at the red end. A lead sulfide detector is most sensitive in the lower portion of the near infrared region. Infrared instruments may use a red hot rod of carborundum (Globar) or a Nernst glower as a light source and a thermocouple or bolometer (resistance thermometer) device to measure the heat liberated in the detector by the absorption of the radiation.

Spectrophotometers

There are several different spectrophotometers on the instrument market today. These instruments are offered by many reliable manufacturers. Each supplier has designed and built equipment which meets the general need and also specific applications by use of various accessories.

The price range of these instruments may go from as low as $500 to as high as $15,000. The price will depend on the spectral range covered, recording or non-recording, null balance or direct reading, single or double beam, standard or special optics, and many other important considerations. Buyers of instrumentation should be well informed before making individual purchases.

No attempt will be made to discuss individual instruments due to the changing nature of the field. All manufacturers or suppliers have descriptive literature available for interested persons.

Errors in Spectrophotometry

The error of measurement of the amount of material in a sample in percent of the amount present is equal to $\Delta C/C$ where ΔC is the error in concentration. The relationship between the error ΔC and the error in absorbance may be computed by dividing the differentiated by the original form of equation (3) giving:

$$(7) \quad \frac{dC}{C} = \frac{dA}{A}$$

Equation (7) shows that for small errors the percentage error in concentration is equal to the percentage error in absorbance.

In general, the error in absorbance, dA, may vary with the value of A. The actual relationship may be obtained experimentally by running a series of replicate analyses at different concentrations. The deviation for each group of replicate samples may then be computed and plotted against the value of absorbance or concentration of the group. Such a

relationship depends upon the type of analysis being conducted and the instruments used for the colorimetric measurements. If the relationship is known, the value of dA in terms of A is substituted in the expression dA/A, which gives the percentage error in the analysis. This fraction may be differentiated to obtain the optimal value of A for minimal percentage error. In the absence of experimental data certain assumptions may be made for the relationship between error and absorbance, as given below.

1. It has been shown experimentally that the error in absorbance dA is a constant in the range of absorbance 0.0 to 1.0 using spectrophotometers such as the Beckman DU and the Cary Model 11. Important errors for which such a relationship would hold include
   (a) instrument zero point errors
   (b) cell blank errors and
   (c) sample blank errors.

In such a case the error dA/A is a minimum for the highest possible value of A. Consequently most accurate work may be carried out at the higher end of the absorbance scale. The Beckman DU has an expanded scale covering the range of absorbance from 1.0 to 2.0. Use of this scale should further increase the accuracy.

2. A second type of relationship between error and concentration may be assumed in terms of transmittance rather than absorbance. It is commonly assumed that a constant error in transmittance is obtained.

Such errors include
   (a) scale reading errors on linear T scales and
   (b) dark current drift.

Equation (6) may be differentiated to give:

$$(8)\quad -kLdC = \frac{dT}{2.303T}$$

which when divided by equation (6) gives the relationship:

$$(9)\quad \frac{dC}{C} = \frac{dT}{2.303\ T\ \log_{10}T}$$

The left side dC/C times 100 is the percentage error in the analysis. On the right side the numerator dT may be assumed to be a constant. Thus, the minimal error will occur when the denominator $T \log_{10} T$ reaches a maximum. In this expression it must be remembered that T is less than 1. By differentiating the expression $T \log_{10} T$ it may be readily shown that the maximal value occurs when T = 1/e or 36.8% (A = 0.434).

3. A third kind of assumption is sometimes made that dT, the error in transmittance, is proportional to the square root of T. This case assumes that the only error is due to electronic noise all of which is due to statistical variations in photon arrival at the cathode. By similar treatment it may be shown that the optimal transmittance is $1/e^2$ or an absorbance of about 1.

The above assumptions are of course purely arbitrary. The exact relationships are obtained experimentally as described above. For the better instruments it may be considered generally that the major errors occur in the chemical portions of the procedure rather than the instrumental. The use of the highest concentration within the range of the instrument will minimize blank and contamination errors. Such a practice is in agreement with the first assumption that a constant error in absorbance occurs, and differs somewhat from the commonly held second assumption that a constant error in transmittance occurs.

Standardization Data and Calculations

Colorimetric procedures are standardized by preparing a series of colors with known quantities of material. It is preferable not to use the zero standard as the photometer reference but rather to use some constant solution which reads close to it, such as distilled

water in many instances. In this way a reagent blank is obtained which gives the analyst an important indication of the levels of contamination and blank errors. In the usual case Beer's Law is followed and a zero and three or four known standards will suffice for the standardization. Four ways of treating standardization data will be described below. In all cases the quantity sought is the standardization factor representing the number of micrograms of material required to produce an absorbance of exactly 1. This factor is used to multiply the absorbances of unknown samples to convert to micrograms of material. If the photometer is calibrated with a transmittance scale these figures may be converted to absorbance by equation (5). As an illustration, actual data will be used for a cobalt analysis.

Treatment of Data

The simplest method of treating the data is to prepare a graph. The actual absorbance of each standard is plotted against the micrograms of cobalt and a straight line is drawn giving the best fit to the data. The intercept of this line with the abcissa (micrograms of cobalt) at zero ordinate (absorbance) gives the best value for the reagent blank. This may differ from that computed from the absorbance for the zero standard. If the relationship is seen to be a straight line, it is not necessary to use this graph for any further purposes as the conversion of unknown samples is done more accurately and conveniently by mathematical rather than by graphical methods. While the graphic method has the advantage of simplicity, three mathematical methods are presented which give more accurate results with little or no extra effort.

Greatest weight to highest values. A simple method of treating the data mathematically is illustrated in Table 1, in which the greatest weight is given to the highest values:

Table 1. - Greatest Weight to Highest Values

	γCo	Absorbance (A)	Corrected Abs.	γ Co Calc From A		Deviation
	0	0.006	0	---		---
	5	0.174	0.168	4.86		-0.14
	10	0.344	0.338	9.77		-0.23
	25	0.883	0.877	25.36		+0.36
sum	40		1.383		sum	0.73

Total γ Co/Total A - 40/1.383 = 28.92

The first two columns give the experimental data for micrograms of cobalt and absorbance. The corrected absorbance in the third column is obtained by deducting the blank absorbance of 0.006 from all the values. The sums of the first and third columns are recorded. Their ratio gives the weighted average standardization of 28.92 micrograms of cobalt per absorbance unit. The fourth column gives the micrograms of cobalt calculated by multiplying corrected absorbances by 28.92. The deviations from the actual micrograms of cobalt are given in the fifth column. A measure of the error in this procedure is given by the sum of the deviations (disregarding algebraic signs), which is 0.73 micrograms of cobalt.

Equal weight to each value. Another method, based upon mathematically calculating the slope for each point, is illustrated in Table 2:

Table 2. - Equal Weight to Each Point

γ Co	Absorbance	Corrected Abs.	γ/A	γ Co Calc From A	Deviation
0	0.006	0	---	---	
5	0.174	0.168	29.76	4.92	-0.08
10	0.344	0.338	29.59	9.90	-0.10
25	0.883	0.877	28.51	25.69	+0.69
		mean	29.29		sum 0.87

The first three columns are identical with those given in Table 1. In the fourth column, the micrograms of cobalt are divided by the corrected absorbances. The working value is taken as the mean of the three figures, or 29.29 micrograms of cobalt per absorbance unit. The fifth column gives the micrograms of cobalt calculated by multiplying corrected absorbance by 29.29. The deviations from the actual micrograms of cobalt are given in the sixth column, the sum being 0.87 micrograms of cobalt. In other cases where blank errors are more important, this method may show higher deviations than the previous ones.

Method of least squares. The most accurate method of handling the data is by using the method of least squares. This is not very laborious for a few values. In this method each value is given equal weight, whereas in the methods illustrated in Tables 1 and 2 the blank value is more heavily weighted, being used in calculating the corrected absorbances for all the other values. The method of least squares is illustrated in Table 3.

Table 3. - Method of Least Squares

	γ Co Y	Absorbance X	X^2	XY	γ Co Calc From A	Deviation
	0	0.006	0.000036		0.165	+0.165
	5	0.174	0.030276	0.870	4.938	-0.062
	10	0.344	0.118336	3.440	9.768	-0.232
	25	0.883	0.779689	22.075	25.081	+0.081
sum	40	1.407	0.928337	26.385		sum 0.540
mean	10	0.3518				
correction			-0.494983	-14.072		
deviation			0.433354	12.313		

$$Y = aX - b$$

$$a = \frac{12.313}{0.433354} = 28.41$$

$$b = a\bar{X} - \bar{Y} = 28.41 \times 0.3518 - 10 = -0.005$$

The first two columns give the experimental data as before. Column 1 is designated as Y and column 2 is designated as X. In the third column, X^2 is tabulated, and in the fourth column XY is tabulated. These calculations must be done on a calculating machine as a slide rule is not sufficiently accurate. The sums of all the columns are recorded and the means of the first two columns (the sums divided by 4) are calculated. The correction term in the third column is equal to the mean of X values multiplied by the sum of X(0.3518 x 1.407). The correction term for the fourth column is the mean X multiplied by the sum of Y (0.3518 x 40). These corrections are deducted from the corresponding X^2 and XY sums to give the deviations listed in the third and fourth columns. The relationships sought are the values of a and b in the equation $Y = aX - b$. The term b represents the best value for the reagent blank, while a is the standardization factor. The ratio of the two deviations, 12.313 ÷ 0.433354 gives the best value of a, which is 28.41 micrograms of cobalt per absorbance unit. The value of b is then calculated from the equation, substituting the mean X and Y and the above value of a. This is found to be -0.005 micrograms of cobalt. This may be regarded as zero for all practical purposes. Generally a positive value is to be expected. The fifth and sixth columns give the values of cobalt calculated from this relationship, and the deviations. The sum of the deviations without regard to signs is 0.54 micrograms of cobalt. It is evident that this is the smallest deviation sum of the three mathematical methods illustrated. For accurate work, when a calculating machine is available, the method of least squares is the most desirable one to use. It is not too tedious for only a few values. The theory behind this method of calculation may be found in the standard statistical texts.

References

1. Cahn, L.: Some observations regarding photometric reproducibility between ultraviolet spectrophotometers. Jour. Optical. Soc. Am. 45:953, (Nov. 1955).

2. Sandell, E. B.: Colorimetric Determination of Traces of Metals. Interscience Publishers, Inc., New York, 1944.

3. Mellon, M. G.: Analytical Absorption Spectroscopy. John Wiley & Sons, Inc., New York, 1950.

4. Bauman, R. P.: Absorption Spectroscopy. John Wiley & Sons, Inc., New York, 1962.

5. Berl, W. G.: Physical Methods in Chemical Analysis. Academic Press, New York, 1960.

6. Willard, H. H., Merritt, L. L., Dean, J. A.: Instrumental Methods of Analysis. D. Van Nostrand Company, Inc., New York, 1958.

7. Ewing, G. W.: Instrumental Methods of Chemical Analysis. McGraw-Hill Book Company, Inc., New York, 1954.

EMISSION SPECTROSCOPY

Introduction

Emission spectroscopy is the study of the radiations emitted by atoms, ions, and a few molecular substances after suitable excitation by means of a thermal or electrical source of energy. These radiations lie in the near infrared, the visible, and the ultraviolet regions of the electromagnetic spectrum. Spectrochemical methods of analysis (spectrographic analysis) make practical use of these radiations for qualitative and quantitative analytical purposes.

Many occupational health problems arise from the industrial exposure of workers to certain toxic materials including inorganic substances. In evaluating the extent of specific exposures, the elemental constituents of these inorganic substances are analyzed in dust or fume samples collected in the working environment. Also, biologic specimens are analyzed to determine the extent of absorption and excretion of the substance by the workers. Frequently the concentrations of the toxic agents are relatively low and analysis becomes difficult for the chemist without a spectrograph. For the analysis of more than 70 metallic or metal-like elements, the emission spectrograph is uniquely suited. This instrument is used to characterize and measure the radiation emitted by specific elements, present as a mixture in the sample material.

Theory

"A spectrum has been defined as the ordered arrangement of radiation according to wave length. Electromagnetic radiations have been discovered that have wave lengths of every value in the range from thousands of kilometers to trillionths of a millimeter... Since no single instrument exists that will separate radiation containing all these wave lengths into a spectrum the electromagnetic spectrum has been divided into various 'regions' in accordance with the types of instruments available to produce and detect the waves of various lengths."(1)

That portion of the electromagnetic spectrum of direct interest to emission spectroscopists extends from 10×10^{-4} mm. (10,000 Angstroms) in the near infrared through the visible, the near and the far ultraviolet regions to about 2×10^{-4} mm. (2,000 Angstroms). Most work in the application of emission spectroscopy to analytical problems is confined to the 2100 - 7500 Angstrom region.

There are three types of emission spectra:

(a) line spectra which are produced by highly excited atoms or atomic ions,
(b) band spectra which originate from highly excited molecules (which must be sufficiently cool so that all the molecules are not dissociated into atoms or ions), and
(c) continuous spectra which result when light is emitted by incandescent solids.

Spectra of the first two types are produced when atoms (or atomic ions) or molecules (ionized or unionized) respectively, are separated sufficiently from their neighbors that they may radiate as individuals between collisions. This condition is realized in the excitation region of an arc, spark or Geissler tube discharge. Continuous spectra originate from incandescent particles in the arc stream and thereby contribute to the background or

Prepared by: Robert G. Keenan

"continuum" observed in spectrograms (photographic recordings of line and band spectra). This continuum tends to mask the weak line spectra of trace elements. Hence, spectrographic methods for specific determinations are based upon employment of optimal exposure conditions and spectroscopic buffer salts, which will minimize background production and, at the same time, increase the intensity of atomic spectra.

Instrumentation

Excitation Equipment

All elements emit characteristic radiation after sufficient excitation by a source of energy. Excitation is accomplished with a suitable "source unit," such as a flame, a 220 volt direct current arc, a high voltage alternating current arc, or a high voltage condensed spark. Each type of source has its own advantages and limitations and the choice of source equipment depends upon the needs of the individual laboratory. The most useful source in an occupational health laboratory is the arc between graphite electrodes, the tips of which are maintained at incandescence by 220 volts of direct current. This arc is indispensable in the spectrographic analysis of refractory materials. Also, it provides the extremely high sensitivity needed for submicrogram quantities of toxic elements. Therefore, this equipment is a basic requirement in all spectrographic laboratories where industrial hygiene samples are analyzed. The other types of source equipment offer definite advantages in special applications, such as the exclusive production of spectra of the more easily excitable elements with an air-acetylene flame; the direct analysis of solutions with a spark technique when the ultimate in sensitivity is not required and an adequate amount of a soluble sample is available; or the analysis of difficultly excitable elements, such as selenium, using a high voltage alternating current arc or spark source. Modern excitation equipment is available commercially in any desired combination of sources as a package unit. Combination sources of this type offer the additional advantage of providing a wide range of discharge characteristics, giving greater flexibility in the selection of excitation conditions.(7)

Spectrograph

There are four main parts to any spectrograph:

1. The *slit* of a spectrograph permits only a narrow beam of light of mixed wave lengths to enter the instrument. Slits of 10, 20, or 30 micron widths are used in prism instruments.

2. The *collimating lens*, mounted between the slit and the dispersing device, brings the individual rays of light into parallel, prior to refraction or diffraction. (Light coming from the slit is divergent).

3. The *dispersing device*, a prism or a grating, "sorts out" the radiation and arranges it according to its component wave lengths.

4. The *recording device*, a photographic plate or film, provides the means of obtaining a permanent record, a spectrogram, of the orderly arranged, discrete line spectra of the elements volatilized from the sample and excited by the energy provided by the spectroscopic source equipment.

For a complete description of the many types of prism and grating spectrographs commercially available, the reader should consult the standard texts on emission spectroscopy.(1,2,3) Instrument requirements vary with the nature of anticipated research and survey projects. With the problems encountered ordinarily in an industrial hygiene laboratory, the most fundamental instrument characteristics should be resolving power, adequate dispersion in the

ultraviolet and lower end of the visible region (where most toxic elements produce their most persistent spectral lines) and speed (line to background) of the instrument. These properties are provided by several types of instruments, including the large Littrow type quartz prism spectrograph. If extensive work in the visible region is anticipated, then grating spectrographs with the desired resolving power, dispersion and speed should be considered.

Photographic Processing Equipment

A completely light tight darkroom is essential to prevent fogging of the spectroscopic plates or films.

Temperature control at 68°F is mandatory for plates and films being processed for quantitative spectrographic analysis. Commercially available developing and fixing equipment, complete with temperature control, may be obtained from the manufacturers of spectroscopic instruments.

Photographic developers and fixers, suitable for spectroscopic emulsions, are commercially available.

Densitometer

Quantitative measurements of the densities of the silver deposits in the developed and fixed spectroscopic emulsion are made with a densitometer. This instrument is a photometric device which is designed to measure the decrease in the intensity of a beam of light passed through a limited area of the emulsion, viz., through a photographically recorded spectral line. Commercially available densitometers vary considerably in optical design and in the method of converting light intensity into measurable electric current. Most instruments use a photovoltaic cell, a vacuum photoemissive tube, or an electron multiplier phototube to effect this conversion. The current is measured with a sensitive galvanometer or with a microammeter after amplification. Most instruments are now constructed to perform the dual role of a densitometer and a comparator, which functions by projecting into juxtaposition on a viewing screen enlarged images of spectrograms from two different spectroscopic plates or films. The comparator is extremely useful for qualitative identifications of unknown sample spectra and, for timesaving purposes, it is a necessity when much qualitative spectrography is performed. For a complete description of modern densitometric instrumentation, the reader should consult the standard texts[1,2,3] and the manufacturers' bulletins.

Other Equipment

Other equipment items required to facilitate the application of spectrographic procedures include a *calculating board*, equipped with movable ordinate and abscissa logarithmic scales and used for converting densitometric transmittance data to analysis line/internal standard intensity ratio values; preformed electrodes of various shapes and sizes (available from commercial sources); and a *step sector disc* and motor mount *or a series of filters* with known light transmittances for the calibration of photographic emulsions.

Applications

The emission spectrograph is used to detect qualitatively and to determine quantitatively the metallic or metal-like elements in any sample which exists as, or can be converted to, a mixture of inorganic substances. Very few chemical methods possess the sensitivity and none can claim the specificity of spectrographic procedures. Also, emission spectroscopy possesses the added and very practical advantage of providing multimetal analyses, a feature which makes feasible the detection and determination of both suspected and

unsuspected elements in sample materials. In addition to these advantages, emission spectroscopic techniques reduce greatly the cost per sample, provided the volume of work warrants the expenditures for the installation and operation of the equipment.

Quantitative Methods

General Considerations. Quantitative spectrographic methods require the development and rigid application of a set of standardized conditions to ensure an acceptable level of analytical accuracy. Adherence to a particular method's conditions of sample preparation and spectroscopic exposure is so strict that, ideally, the only permitted variable is the concentration of the analysis element or elements. Although the relative intensities of the spectral lines of an element are constants of nature, the absolute intensity of any particular line will vary not only with the concentration of the element in the samples subjected to spectroscopic exposure, but also with changes in the concentrations of the major elements contained in the sample matrix. Other factors which cause intensity deviations are variations in spectroscopic exposure conditions, some of which can be controlled directly while others require indirect control. Such conditions as applied voltage, current, the analytical gap, and uniformity of electrode craters can be controlled with a satisfactory degree of precision. However, excitation conditions, resulting from the use of a direct current arc on nonbuffered samples as an example, can vary markedly due to the inherent tendency of the arc to wander and to produce "spot" burning. This situation, if uncontrolled, causes temperature fluctuations which promote erratic volatilization and excitation of the sample elements. For this reason, a spectroscopic buffer salt is mixed with the samples (standards and unknowns) to provide uniform burning of the electrode charge and to prevent erratic volatilization of the elements. Some inorganic substances, used effectively for these purposes, are graphite, aluminum oxide, lithium chloride, lithium carbonate, potassium sulfate and calcium carbonate. In this laboratory, a 2.5 to 1.0 mixture of finely ground graphite and lithium chloride has been adopted as an extremely effective spectroscopic buffer. This mixture not only promotes uniform burning of the sample matrix but it also suppresses background and cyanogen band formation.(5,6) Furthermore, it enhances the production of trace element spectra with the result that unusually high degrees of absolute sensitivity can be realized.(6)

Experimental Conditions. Quantitative spectrographic methods, to be applicable by different analysts, should specify clearly the following conditions and information:

1. Type or types of sample material
2. Exact description of sample preparation
3. Type of excitation for specified analysis element(s)
4. Electrode material and crater description with dimensions
5. Crater waterproofing reagent - composition and how it should be used
6. Name and amount of spectroscopic buffer - commercial source of this material, laboratory preparation and storage, and exact description of addition to sample
7. Name, amount, and commercial source of internal standard element added to sample or buffer
8. Sample electrode drying conditions, if solution analyses with an arc are involved
9. Type of spectrograph and focusing of source employed in development of method
10. Spectrographic slit width
11. Excitation conditions
    (a) Voltage, current, capacitance, inductance, polarity, etc., as applicable to excitation employed
    (b) Exposure period
    (c) Use of a sector or filter -

whether used during all sample exposures or confined to emulsion calibration.

12. Spectroscopic emulsion type
13. Emulsion calibration technique
14. Wave lengths of analysis and internal standard element spectral lines
15. Calculation of intensity ratio data - with or without background correction applied to analysis and internal standard line densitometric values.

Standard Samples

A series of increasing amounts of an element in a series of electrode crater charges, subjected to the proper spectroscopic exposure conditions for the volatilization and excitation of this particular element, produces a series of spectrograms in which the detectable spectral lines of this element are recorded with increasing density relative to increasing concentration. Such a series is actually prepared by the initial and periodic standardization of a quantitative spectrographic method. In addition to the definite and varied quantities of the analysis element (for example, 0.005 to 1.000 microgram) in the series of craters, the electrode charges contain the specified amount of the element-free sample base material (blood, liver, urine, etc., ash from which the element has been removed chemically, if necessary), the prescribed quantity of the spectroscopic buffer, and the specified amount of the internal standard element. All steps in the spectrographic exposure, photographic processing, densitometry, and treatment of densitometric data must be exactly as that to be used for unknown samples.

Densitometry

The densitometer, installed in an area removed from direct room illumination, should be stable at its zero and 100 percent transmittance settings before spectrograms are read. A non-recording type is preferred by emission spectrographers, as only single line transmittance values are needed. The instructions given in the instrument manual should be followed during installation and use.

The galvanometer scale is adjusted to read "0" when a black cloth is inserted in the light path and then adjusted to read 100 percent transmittance for the clear plate or film area just above (or below) the analysis line in the first or last spectrogram. Repeated adjustments of the scale and sensitivity settings are made until the two desired readings are realized. Information about the height and width of the slit of the instrument should be available in the instruction manual.

The plate carriage is adjusted so that the image of the spectral line is parallel to the slit. The maximal deflection of the galvanometer scale is then obtained by slowly moving the spectral line past the light beam, either by manual or motor drive operation of the scanning control (whichever is provided or preferred by the operator). This value is recorded as "% T" in the record book. The plate is then shifted to the internal standard line and the "% T" value of this line is recorded. If background transmittance values are desired, the area adjacent to each spectral line is scanned and the value provided by the least deflection of the galvanometer is taken and recorded. When the series of spectrograms has been read, the densitometric data are converted to Intensity of Analysis Line/Intensity of Internal Standard Line values using the emulsion calibration curve (H and D; Hurter and Driffield curve) and a calculating board to effect this conversion.

Emulsion Calibration

The spectroscopic plate or film emulsion must be calibrated in the wave length region containing the analysis and internal standard lines, each time a new lot of plates is purchased and every few months while a given lot is in storage, because the photographic response decreases with age. This decrease

in response can be minimized by storage in a refrigerator.

The purpose of this calibration is to relate the variation of optical density with the logarithm of the intensity of the source. An excellent fundamental treatment of this subject has been presented by Nachtrieb.(3)

Procedure. Repeated requests have been made for a detailed description of the emulsion calibration procedure, using a rotating logarithmic step sector. The sector may have a step ratio of 1:2, 1:1.5, or some other value stamped on the disc. In this laboratory, a sector having a step ratio of 1:1.5 is used to provide shorter exposure ranges than possible with a sector constructed with 1:2 step ratio, as the exposure times of a sectored light beam from a uniform source are proportional to the angular openings of the disc.

The sector disc is supported in a motor driven assembly and is mounted firmly on the optical bench of the spectrograph as close to the slit as possible, without striking the slit assembly if vibration should occur during adjustment of its rotating speed. The motor is allowed to warm up for 5 to 10 minutes and its rheostat control is adjusted so that the velocity of the disc is the maximum that can be tolerated just short of vibration (which must not occur during the exposure). The full slit of the spectrograph is exposed by removing the Hartmann diaphragm. An iron arc is operated as the source for the preparation of three successive spectrograms for periods of 10, 15, and 20 seconds. Make sure that there is sufficient clearance between the spectrograms on the plate or film whose emulsion is being calibrated.

Develop and fix the plate or film at 68°F. After drying, measure the percent transmittance values of each step of several selected iron lines in the regions where the analysis line occurs. Make selections so that this collection of transmittance values cover the range at least from 10 to 95 percent, the probable working range of standard and sample transmittance data. The values thus obtained are used in preparing the emulsion calibration (H and D) curve, plotting log % T as ordinates and log I as abscissae. A calculating board, provided with movable logarithmic scales, eliminates the calculation of logarithms and antilogarithms each time the emulsion curve is plotted or used. The following table of calibration data will be used to explain how plotting is conducted.

Table 1. - Transmittance Data Obtained from Iron Arc Source Using Logarithmic Step Sector, Ratio 1:1.5.

Step	Intensity	Int. Plot Value	Fe Line No. 1 % T	Int. Plot Value	Fe Line No.2 % T
1	1.0	1.0	91.5	---	---
2	1.5	1.5	76.4	1.0	82.0
3	2.25	2.25	52.0	1.5	67.5
4	3.375	3.38	28.9	2.25	45.3
5	5.063	5.06	12.6	3.38	25.6
6	7.594	7.59	6.2	5.06	10.6
7	11.391	11.39	----	7.59	4.7
8	17.086	17.09	----	11.39	2.0

The logarithms of the transmittance values in Column 4 are plotted versus the logarithms of the intensity values given in Column 3. Then the abscissa scale is shifted to the right to permit plotting of the values given in Columns 5 and 6 in such a manner that a smooth, average curve may be drawn. If the spectrographer is dissatisfied with the resulting curve, then he should obtain additional densitometric data from the iron spectrograms and continue this method of plotting until he is assured that a satisfactory, average curve has been obtained.

This emulsion calibration curve is used for all calculations of the intensity ratio of the analysis line to the internal standard line in the region thus calibrated.

Analysis or Working Curve

The densitometric data obtained from the standard sample spectrograms are converted to intensity ratio (Anal. Line/Int. Std. Line) values, using the emulsion calibration curve and the calculating board to effect the conversion. The concentrations of the analysis element versus the calculated intensity ratios are then plotted logarithmically to obtain the analysis or working curve.

Transmittance data obtained from unknown sample spectrograms are converted to intensity ratio (Anal. Line/Int. Std. Line) values, using the emulsion calibration curve, and the concentrations are then determined graphically from the analysis curve.

References

1. Harrison, G. R.; Lord, R. C.; and Loofbourow, J. R.: Practical Spectroscopy. Prentice-Hall, Inc., New York, 1948.

2. Brode, W. R.: Chemical Spectroscopy, 2nd. Ed. John Wiley and Sons, Inc., New York, 1943.

3. Nachtrieb, N. H.: Principles and Practice of Spectrochemical Analysis, 1st. Ed. McGraw-Hill Book Company, Inc., 1950.

4. Keenan, R. G.: The role of the emission spectrograph in occupational health investigations. Occupational Health, 13: No. 1, 2-4 (1953).

5. Keenan, R. G.; and White, C. E.: Suppression of cyanogen bands in the direct current graphite arc by lithium chloride. Anal. Chem. 25: 887-892 (1953).

6. Keenan, R. G.; and Kopp, J. F.: Spectrochemical determination of trace quantities of cobalt in animal tissues. Anal. Chem. 28: 185-189 (1956).

7. Keenan, R. G.: Excitation sources for spectrographic analysis. A.M.A. Arch. of Ind. Health, 15:253-256 (1957).

POLAROGRAPHY

Introduction

Polarographic analysis is based on the electrolysis of a solution between two electrodes, one electrode which is very small, and easily polarizable, called the indicator electrode; and one large and non-polarizable or reference electrode. The voltage necessary for the electrolysis indicates the nature of the reacting substances, while the current observed is a function of its concentration.

Theory

The indicator, or polarizable, electrode generally used in polarographic analysis is a dropping mercury electrode for a reduction reaction, and a platinum or other noble metal electrode for an oxidation reaction. In either type of reaction, oxidation or reduction, the reference electrode is either a pool of mercury or a saturated calomel electrode.

Polarographic waves that involve reduction reactions are called cathodic waves, and those that involve oxidation reactions are called anodic waves. Similarly, by convention, the diffusion currents for reduction reactions are given a positive sign and for oxidation reactions a negative sign. A polarographic analysis may be made of any substance if it is

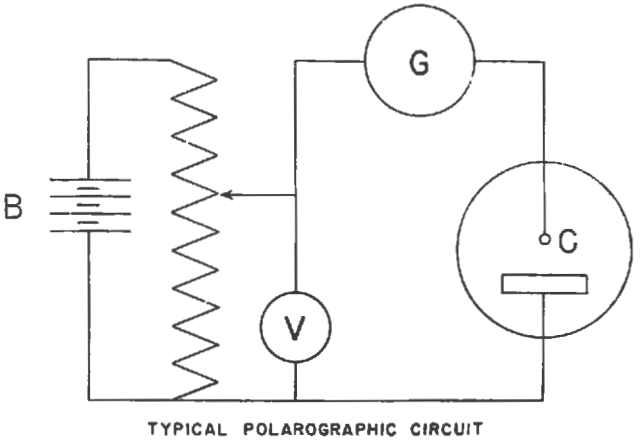
(1) electro-reducible or oxidizable,
(2) within the range of the electrodes used,
(3) in true solution, and
(4) stable for the duration of the measurement.

When using the dropping mercury electrode assembly the quantitative method of polarographic analysis is dependent on the current-voltage relationship obtained when an increasing potential is applied to the electrode. When the decomposition potential of an electro-reducible ion is reached, the current increases rapidly with the increasing voltage and then levels off to a "limiting current", the resulting current-voltage curve being "S" shaped. This limiting current is caused by an extreme case of concentration polarization at the cathode, and in the presence of a large excess of a non-reducible salt is produced solely by the diffusion of the electro-reducible ion to the cathode. This "diffusion current" then is proportional to the concentration of electro-reducible ions.

Polarographic circuits must perform two functions:

(1) apply a direct current voltage across the electrolysis cell and
(2) measure the current thus caused to flow through the cell.

A diagramatic sketch of a typical polarographic circuit is shown in Figure 1.



TYPICAL POLAROGRAPHIC CIRCUIT

Figure 1

Revised by: Lial W. Brewer

The dropping mercury electrode assembly as used is shown schematically in Figure 2.

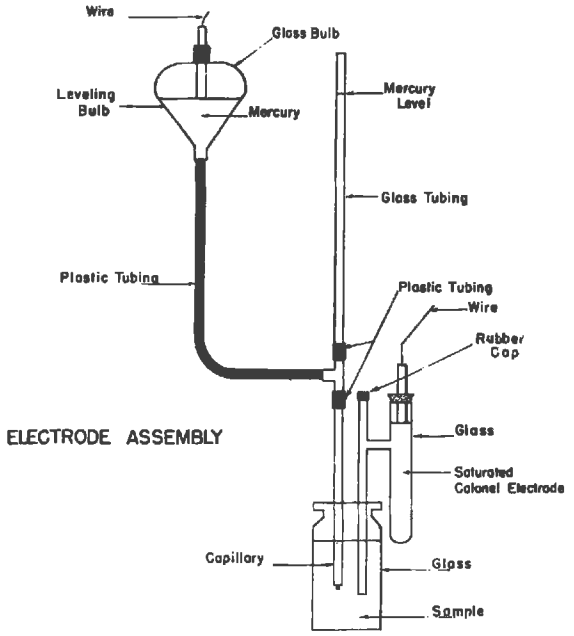


Figure 2

The polarographic cell contains the solution under investigation in an electrolyte, such as KCl. The cell must also have provisions for bubbling an inert gas such as nitrogen through the solution to remove any dissolved oxygen and over the solution to keep the oxygen from redissolving during the electrolysis. The electrolysis cells used are many and of varied types.

Reference electrodes are of two types, internal and external. An internal electrode is in direct contact with the solution being studied, while an external electrode is separated from the solution by a salt bridge or porous membrane. The most commonly used reference electrode is the pool of mercury.

The most commonly used indicator electrode is the dropping mercury electrode, which is used due to its steady and reproducible diffusion currents which are made possible by the continual renewing of the smooth and reproducible size drop of mercury. The dropping mercury electrode is also valuable because it can be used in acid solution.

The diffusion current is based on the Ilkovic equation:

$$i_d = 607nD^{1/2}Cm^{2/3}t^{1/6}$$

i_d = average current during life of a drop, in amperes
n = number of electrons involved in reduction of one molecule of reducible substance
D = diffusion coefficient of reducible substance at a given temperature, in cm^2 per second
C = concentration of reducible ions, in moles per ml
m = weight in grams of mercury flowing from capillary per second
t = time in seconds for each drop of mercury to form

In this equation there are three variables, the concentration, the rate of flow of mercury, and the temperature. The temperature influences the diffusion current, concentration of oxidizable or reducible substances, and the rate of flow of mercury. Therefore, if the temperature is kept constant and the rate of flow of mercury is kept constant, the average current becomes a function of the concentration of the oxidizable or reducible ions.

The polarographic method for qualitative analysis is dependent upon the different decomposition potentials of the reducible substances, expressed as half-wave potentials, $E_{1/2}$. Half-wave potentials, like standard electrode potentials, are a function of the molecular form of the reducible or oxidizable material. Half-wave potentials can therefore be shifted by the addition of complexing agents or by varying the pH of the solution. In cases where it is necessary to determine two or more substances simultaneously, and the half-wave potentials overlap, it is sometimes possible to separate the waves by adding

complexing agents or by varying the pH of the solution. When the half-wave potentials of several ions in the test solution differs sufficiently (in general, substances will not interfere if their half-wave potentials are separated by 0.2 volts or more) they may be determined simultaneously, both quantitatively and qualitatively. The half-wave potential is shown graphically in Figure 3.

GRAPHICAL DETERMINATION
OF HALF—WAVE POTENTIAL

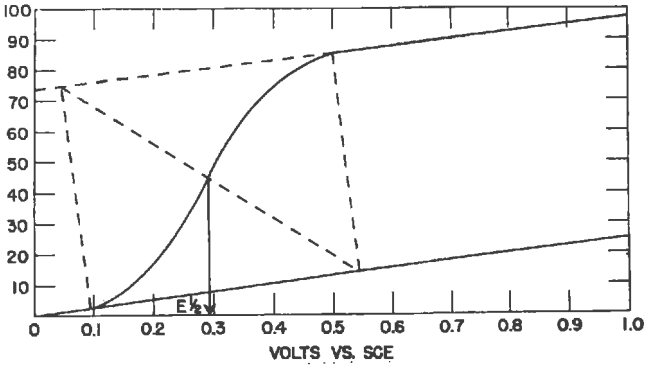


Figure 3

Application

In the field of occupational health, determinations can be made of lead, cadmium, zinc, vanadium, iron, thallium, halides, sulfur dioxide, cystine, and many other substances after the conditions for the determination are worked out. Early analytical applications of polarographic analysis were mostly in the inorganic field, but at the present time utilization of these techniques in the field of organic analyses is increasing. In general, all the principles and techniques described for polarographic analysis of cations apply equally well to qualitative and quantitative analysis dealing with organic molecules containing electroreducible groups. The curves are similar to those recording cation discharge, the half-wave potentials are specific constants of the corresponding reducible groups, electrode and solution characteristics are subject to similar requirements of standardization, and the relation of the diffusion current to concentrations is equally exact.

The limit for qualitative detection and identification in polarographic analysis is in the order of magnitude of 0.01 gamma (100 millionth of a gram). For quantitative measurements, the maximum convenient dilution is about 10^{-5} or 10^{-6}, Normal. The most accurate polarograms are those conducted in the more dilute solutions, since high concentrations entail large diffusion currents and low sensitivity ratios of the galvanometer.

Summary

The polarograph is an instrument employing generally a dropping mercury electrode, whose surface is continually renewed, to measure the current-voltage relationship of the reducible solution. Voltage is increased at a constant rate and the current passing through the test solution is measured and recorded. When the reduction potential of a given ion is reached, greater current flows, which is proportional to the concentration of the ion. The height of the recorded curve (step height) is directly proportional to the concentration of the ions present, other factors being constant. In polarographic analysis concentrations of unknowns are determined by reference to standard solutions. The step height of diffusion current is plotted for the standard against its concentration.

Several makes of instruments for polarographic analysis are commercially available. The potentials needed for analysis can be obtained from any battery that will supply up to 3 volts and current flows up to approximately 16 microamperes. Little time is required for analyses. The sample does not change significantly and can be used for other purposes. Several constituents in the same solution can be determined.

In addition to usual methods of polarographic analysis, the normal instruments with accessories can be used for
(1) amperometric titrations,
(2) derivative polarography,
(3) oscillographic polarography, and
(4) continuous recording at fixed potentials.

References

1. Bibliography of Polarographic Literature, 1903-1949, Leeds & Northrup Company, Philadelphia, 1950.

2. Bibliography of Polarographic Literature, 1922-1955, E. H. Sargent and Company, Chicago, 1956.

3. Delahay, P.: New Instrumental Methods in Electrochemistry: Theory, Instrumentation and Applications to Analytical and Physical Chemistry, Interscience Publishers, Inc., New York, 1954.

4. Harley, J. H.; and Wiberley, S. E.: Instrumental Analysis, pp. 268-91. John Wiley and Sons, Inc., New York, 1954.

5. Kolthoff, I. M.; and Lingane, J. J.: Polarography (in 2 volumes), 2nd Ed. Interscience Publishers, Inc., New York, 1952.

6. Laitinen, H. A.; and Kolthoff, I. M.: A study of stationary and rotating micro-electrodes. J. Phys. Chem. 45: 1061-79, (1941).

7. Meites, L.: Polarographic Techniques. Interscience Publishers, Inc., New York, 1955.

8. Miller, O. H.: The Polarographic Method of Analysis, 2nd Ed. Chem. Education Publishing Company, Eaton. Pa., 1951.

9. Parks, T. D.: Applications of ampermetric titrations to microanalysis. Analytica Chimica Acta. 6:553-66, (June 1952).

10. Willard, H. H.; and Merritt, L. L., Jr.: Instrumental Methods of Analysis. 3rd Ed. D. Van Nostrand Company, Inc., New York, 1958.

11. Zuman, Peter and Kalthoff, I. M.; Progress in Polarography Vols. I and II, Interscience Publishers, Inc., New York, 1962.

12. Schmidt, Helmut and Von Stackelberg, Mark: Modern Polarographic Methods. Academic Press, New York, 1963.

13. Purdy, Wm. C.: The Role of the Electroanalytical Chemist in Biochemical Research. Anal. Chem. 36:29A-39A (April 1964) (25 references).

PRINCIPLES AND APPLICATIONS OF GAS CHROMATOGRAPHY

The analytical chemist has always dreamed of a device, a magic tube, in which he places a sample of a complex mixture, and without further attention, a complete analysis appears along with the separated components. Insofar as the analysis of organic materials is concerned, this has very nearly materialized in the technique of gas chromatography. The organic analyst now has at his disposal a method with excellent potentialities for the rapid analysis of small samples containing mixtures of homologous compounds, isomers, and functional types.

Principles of Operation

Gas chromatography is a process by which a mixture is separated into its constituents by a moving gas phase passing over a sorbent. Gas chromatography is divided into two major categories; gas-solid chromatography (G-S-C) where the sorbent is a solid of large surface area and gas-liquid chromatography (G-L-C) where the sorbent is a non-volatile liquid coated on an inert solid support. The moving gas phase or carrier gas is an inert gas which is made to flow at a constant rate through a packed column, in a small diameter tube, containing the sorbent. (See Figure 1).

When a sample containing a mixture of components is injected into the gas stream, the components move through the column at rates dependent on their respective volatilities and/or interaction with the nonvolatile liquid phase. The various molecules tend to dissolve and revaporize as they pass down the column. The molecules of the component having the greater solubility or greater interaction with the liquid phase are retarded in their passage. With a column

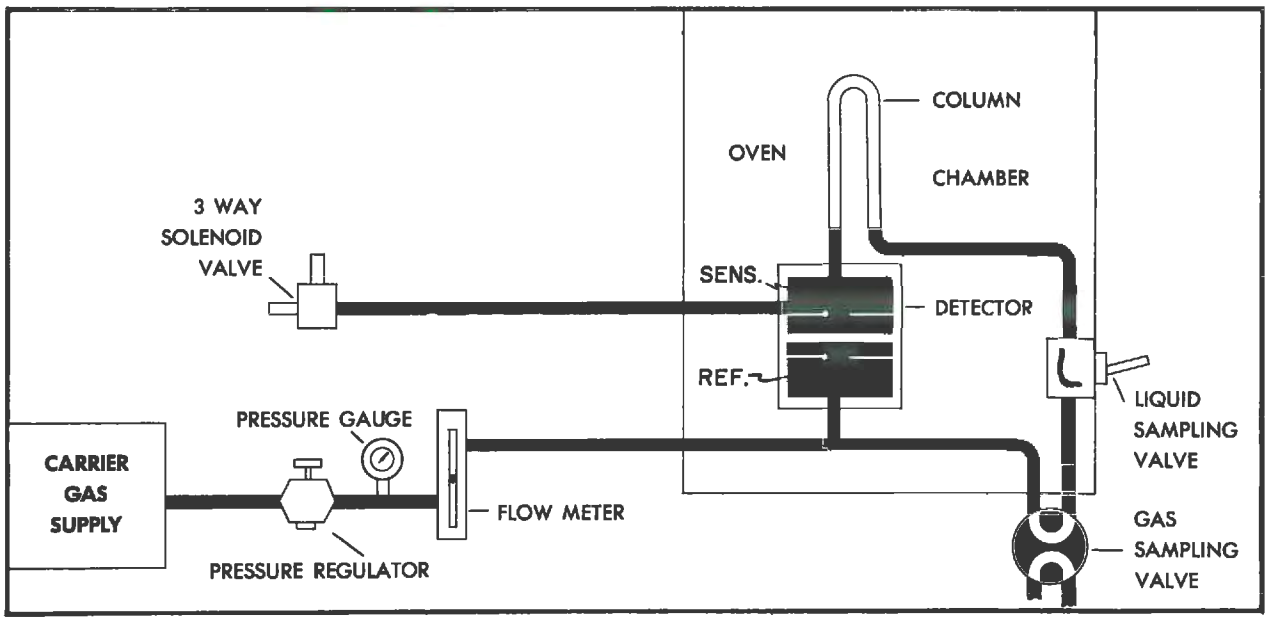


Figure 1. - Schematic Diagram of Gas Chromatograph.

Prepared by: Richard E. Kupel

packing of suitable properties and of sufficient length, the partition process between the gas and liquid phases results in a situation in which each component of the sample emerges in time, from the end of the column, as a binary mixture with the carrier gas. In G-S-C, absorptive processes on the solid phase perform an analogous function in retarding the various components. Activated alumina, molecular sieve, silica gel, and activated charcoal are examples of materials used in G-S-C.

The experimental procedure for obtaining a gas chromatogram is relatively simple. With a hypodermic syringe, a few microliters of the organic liquid are injected rapidly through a rubber diaphragm in a heated sample port into the carrier gas stream. With a suitable differential detector at the exit of the column, the signal obtained is proportional to the instantaneous concentration of the dilute component in the binary gas mixture. The result on the chart of an electronic recorder is a chromatogram with a series of peaks for various components.

One effect which becomes important in gas-solid chromatography is the absorptivity of the carrier gas on the absorbent. It is apparent that the carriage gas may act as a displacer, i.e., it can occupy active adsorption sites, thus reducing the heat of adsorption of the solute and consequently its retention time. The effect is analogous to liquid-liquid chromatography where different solvents are used to elute solutes.

Columns and Packing

The heart of a chromatograph is the column containing the packing on which separations are effected. A wide variety of suitable liquid phases and adsorbents provides a large degree of selectivity.

Column packings are either a finely divided adsorbent G-S-C or a finely divided solid support on which a thin layer of liquid is adsorbed G-L-C. Adsorbents most frequently used are alumina, silica-gel or activated carbon in particle size ranging from 20 to 120 mesh. These are used for gas mixtures and mixtures of low-boiling, relatively non-polar compounds, such as hydrocarbons. Solid-liquid packings are most effective for higher boiling liquids.

The most common supports for the liquid phase are "Celite" or crushed firebrick. The liquid phase is placed on the solid support by simply mixing in the desired proportions, or by adding the liquid as a solution in a volatile solvent which is subsequently removed by vacuum filtration.

Two basic requirements of the liquid phase are that it have a low vapor pressure at the temperature used and that it provide an adequate separation of the components of the mixture to be analyzed. The first qualification can be met if the liquid has a boiling point approximately 200°C above the temperature at which it is to be used.

At high temperatures the liquid phase expansion reduces the free volume of the column and the viscosity of the carrier gas increases. Thus, the pressure drop increases and the maintenance of a given carrier gas flow requires a higher inlet pressure. At higher temperatures the retention time or volume for a solute peak is reduced.

Detectors

Thermal conductivity cells are currently the most widely used detectors. They use a heated filament as the element for sensing changes in thermal conductivity. When the heated detector element is cooled by the pure carrier gas stream, a definite response is obtained. As the binary mixture of the separated component and the carrier gas passes over the element, the binary mixture which usually has lower thermal conductivity, causes less heat to be lost, and the

resistance of the element changes due to its temperature change.

The resistance change is a function of the instantaneous concentration of the component in the gas stream. Since the direct measurement of thermal conductivity is difficult, a differential technique is used. Thermal conductivity detector elements are used in pairs, one is exposed only to the carrier gas while the other is exposed to the column effluent. Reference and sensing elements are incorporated into a Wheatstone bridge circuit and the out-of-balance signal is fed to a recorder.

Many types of detectors are used in present day gas chromatography. The flame ionization detector is one of the sensitive detectors that depends on ionization of the column effluent in a hydrogen flame. The carrier gas and each separated component, in turn, are mixed with hydrogen and burned at the tip of a glass jet. A potential is applied to two electrodes, one the jet itself and the other a probe above the flame. The ion current generated in the flame is amplified and recorded.

For additional sensitivity, argon detectors and electron capture detectors are available.

A number of important points in the operation of a gas chromatograph are:

(1) Selection of the optimal column material for the analysis of gas or liquid samples.

(2) Operating temperature. It must be low enough to separate the low boiling components but high enough to cause the high boiling fractions to come off the column in a reasonable time. Temperature programing is the only answer to some of these problems. With a mixture of high and low boiling components it is imperative that the temperature be programed.

(3) Proper detector for the analysis at hand.

Both qualitative and quantitative analysis can be accomplished on this instrument. In order to be able to duplicate a run at some later date it has been suggested that a definite amount of some known foreign material be added to the sample. The elution time of this material is established and all the components in the mixture are recorded relative to the known material. This allows one at some later date to reproduce the elution time for this added known material which will then allow the analytical parameters to be duplicated.

Quantitative analysis can be accomplished by a number of methods, using peak areas, peak height, peak height X½ peak width, integration by electronic means or any other method that can provide a linear relationship with the concentrations.

Summary

Gas chromatography is an important analytical tool for the occupational health chemist. However, one must be very cautious in expressing one's results since, in some cases, more than one material may have the same retention time on a specific column and it does not necessarily follow that this is the only material present. It may be possible, using different column materials or different temperature settings, to cause other components to be eluted from this second column that might have been retained on the first column. It is imperative that as much as possible be known about the sample before any attempt is made to analyze it so that the best column material is selected and the proper temperature mode is chosen to assure the elution and separation of all of the components in the sample.

References

1. Organic Analysis Vol. IV, Interscience Publishers Inc., New York, 1960.

2. Jour. of Gas Chromatography Vol. 1, 1963 to present.

3. Cassidy, H. G.: Fundamentals of Chromatography, Interscience Publishers Inc., New York, 1957.

4. Keulemans, A.I.M.: Gas Chromatography. Reinhold Publishing Corp., New York, 2nd Ed., 1962.

5. Dal Nogare, Stephen and Juvet, R.S.: Gas-Liquid Chromatography: Theory and Practice. Interscience Publishers, New York, 1962.

ION EXCHANGE TECHNIQUES

Introduction

Ion exchange has been defined "as a reversible exchange of ions between a solid and a liquid in which there is no substantial change in the structure of the solid"[1]. The process was first described in 1850 by an agricultural chemist who reported that a clay-like soil would remove potassium ions from a solution with a concurrent release to the solution of an equivalent amount of other cations[2]. Since that time zeolite clays and synthetic aluminum silicates have been used extensively in water softening processes based upon the exchange of sodium ions from the silicate for the calcium and magnesium ions present in hard waters. In 1935 the first synthetic organic exchangers were prepared. Chemists found these original phenolic-type resins to be suitable for numerous applications in analytical chemistry. In the ensuing years we have witnessed the progressive development of a variety of resins whose improved stabilities, exchange capacities, and ranges of applications have now reached the stage where ion exchange is unmatched for its rapid, clean-cut method of isolating and concentrating trace quantities of an analytically desired elemental constituent of a sample.

The synthetic organic ion exchange resin particle may be visualized as an elastic, three-dimensional hydrocarbon network tied together at random positions by a cross-linking compound. A common type of resin used in analytical chemistry is that produced by the copolymerization of styrene and divinylbenzene. In this type, polystyrene chains make up the hydrocarbon network while the divinylbenzene polymer contributes the cross-linkage.

Prepared by: Robert G. Keenan

The proportion of the latter compound, usually 4 to 12 percent, which is added prior to polymerization determines the length of chain between cross-links. This type of structure possesses a maximum resistance to oxidation, reduction, mechanical wear and breakage, and is insoluble in common solvents[1]. It is usually available in the form of small spheres or beads.

A large number of ionizable, functional groups, is attached to the benzene rings of the hydrocarbon network. The nature of the functional group determines the chemical behavior of a given resin. Thus, strongly acidic cationic exchangers contain sulfonic acid groups, remain highly ionized in both their acid and salt forms, and enter into ion exchange behavior over the entire pH range. As cationic exchangers, these resins are finding numerous analytical applications for the separation of metallic ions. Conversely, strongly basic anionic exchange resins contain a quaternary ammonium type of structure, with a CH_2NMe_3Cl arrangement as a typical functional group. These resins are being used analytically for the separation of nonmetallic elements or radicals from sample solutions as well as certain metals which, by the formation of chloro or other anionic complexes in the sample solution, are retained by the resin. Between these two extreme types of resins, there are weak base resins which operate in the 0-7 pH range and weak acid resins operable above pH 7. Among other applications, the former are useful for the separation of weak and strong acids while the latter are employed to remove basic materials.

Advantages of Method

When the conditions for a desired analytical separation have been worked out in the laboratory, the chemist finds

the ion exchange method to be simple, rapid, inexpensive, and amenable to the processing of a large number of samples. In these separations, a column of the resin, supported in a chromatographic tube, constitutes the solid phase of the 2-phase system alluded to previously, while a solution of the sample functions as the liquid phase. The sample may be a dilute acid solution of a biological tissue ash, a specimen of a body fluid which has been diluted with distilled water or digested with acid reagents prior to dissolution, or it may be an aqueous, acidic, or basic solution of the inorganic constituents from any number of source materials. The essential prerequisite for the application of this method is that the analytically desired substance must be in an ionically available form.

General Application

The actual application of an ion exchange procedure is very straight forward. The solution of a sample is permitted to flow through the column of the resin at an experimentally established rate. During this operation certain sample constituents are retained by the resin upon exchange with the terminal members of the resin's functional groups. If the removed constituents are unwanted substances which interfere with the analytical determination, the column is washed with a suitable liquid (often distilled water) and the combined sample solution and washings, now free from the interfering substances, are treated by the next step of the method. If, however, one or more of the substances retained by the resin is analytically desired, the column is first washed to remove the remainder of the sample solution, and the desired constituent is removed from the column by the admission of an eluting reagent. This reagent is the one selected during the development of the method as that which is most selective in its elution of the desired constituent from the resin column and, at the same time, provides reproducibly quantitative recoveries. The eluted constituent, thus free from interfering substances which are now either in the original sample solution or retained by the resin column, is now ready for the next step of the analytical procedure.

Lead and Mercury

The practical advantages gained by the use of ion exchange procedures are evident in several methods which have been developed and used by occupational health chemists. One of these methods, which provides the separation of fluoride from unashed urine[3], eliminates both the ashing of samples (which must be done in platinum dishes) and the steam distillation of the hydrofluosilicic acid prior to the colorimetric determination. This direct exchange of fluoride from preserved urine for the acetate groups of a strongly basic anionic resin is feasible because fluoride is excreted in an ionically available form in urine.

A separate exchange method which requires previous digestion of urine, has proved to be valuable in distinguishing between the urinary excretion levels of persons with minimal exposure to inorganic mercury or to organic mercurials and those of unexposed subjects[4]. This method takes advantage of the separation of the chloro complex of mercury from other metallic constituents using an anionic exchange resin. Elution of the mercury from the washed column with a thiourea reagent provides complete separation of submicrogram quantities of mercury from 0.5 milligram amounts of the other divalent metals which react with dithizone. The estimation of the eluted mercury is then performed directly by shaking the eluate with standard dithizone reagent and reading the color in a spectrophotometer.

Other Applications

Another example of an ion exchange

separation which has been devised to circumvent the difficulties imposed by substances which interfere with the chemical reagent used in the conventional procedure is a method for thorium[5]. Using a cationic exchanger, this method provides for the complete separation of thorium from such urine constituents as iron, uranium, calcium, barium and other ions which, when present, interfere with the otherwise nonspecific thorin, morin, and chrome Azurol S methods.

A method employing an anionic exchange resin has been developed to extend the sensitivity of the fluorometric determination of uranium in urine down to 0.005 microgram U per liter[6]. This method has been applied to samples from non-exposed subjects.

In addition to the removal of interfering substances, ion exchange methods also serve to remove other undesired sample constituents which provide bulk. In this way, they function as concentration as well as isolation procedures and thus extend the working sensitivity of a method.

There are numerous publications in the literature of physical and analytical chemistry dealing with the fundamental aspects of ion exchange. Some of these should be acquired as reference materials by those interested in becoming better acquainted with this technique.[7,8,9]

References

1. Dowex: Ion Exchange, The Dow Chemical Company, Midland, Michigan, 1958, p. 3.

2. Way, J. T., J. Roy. Agr. Soc. Engl. 11, 313 (1850).

3. Talvitie, N. A., and Brewer, L. W.: Separation of Fluoride by Ion Exchange: Application to Urine Analysis. Amer. Ind. Hyg. Assoc. J. 21, 287-295 (August, 1960).

4. Kopp, J. F., and Keenan, R. G.: Determination of Submicrogram Quantities of Mercury in Urine by Ion Exchange Separation. Amer. Ind. Hyg. Assoc. J. 24, 1-10 (Jan.-Feb. 1963).

5. Welford, G. A., Sutton, D. C., Morse, R. S., and Tarras, T.: Determination of Thorium in Urine. Amer. Ind. Hyg. Assoc. J. 19, 464-468 (Dec. 1958).

6. Welford, G. A., Morse, R. S., and Alercio, J. S.: Urinary Uranium Levels in Non-Exposed Individuals. Amer. Ind. Hyg. Assoc. J. 21, 68-70 (1960).

7. Rieman, W. III, and Sargent, R.: Ion Exchange. Physical Methods in Chemical Analysis, Vol. IV, Edited by Walter G. Berl, Academic Press, New York, 1961.

8. Samuelson, O.: Ion Exchangers in Analytical Chemistry. John Wiley and Sons, Inc., New York, 1953.

9. Analytical Reviews published annually in April by Analytical Chemistry.

SOLVENT ANALYSIS

Introduction

Solvent analysis is a very complex and difficult operation because an extremely wide variety of solvents is in industrial use. No systematic procedure is available for the exact identification and determination of these substances. All the techniques of organic analysis are required. Because of the many types of required tests a sample of at least 500 ml is highly desirable. In the following discussion certain general principles will be given which are applicable to some common solvents including laboratory analysis of bulk solvent samples.

Solvents Used in Various Industries

Preliminary information about the sample is of the utmost importance. No effort should be spared in consulting the chemical and industrial literature in order to determine what classes of solvents may be expected. One of the difficulties in this work is the fact that many plants do not know the chemical composition of the organic solvents they use. Indeed, even the manufacturer may not know the exact composition because of the possible variations in his raw materials. It is not unusual for the composition of solvent mixtures to vary from batch to batch. However, an effort must be made to get an approximate idea of what substances may be expected in the solvent. Table 1 lists the uses of some solvents found in industry.

Vapor Hazard of Various Solvents

Solvents vary widely in toxicity. The exact identification of the less toxic members of a solvent mixture is not as important as the identification and determination of the more toxic components. Table 2 lists several organic gases and vapors in order of decreasing vapor hazard. From the general information available regarding the sample, the analyst may determine which of the substances likely to be present may be important from the toxicity viewpoint. Volatility is a factor which effects the toxic vapor hazard. It may be seen that a relatively high-boiling, non-volatile solvent would be less dangerous than a volatile one of equal toxicity. Substances boiling above 150°C are ordinarily not considered to be a serious hazard for this reason.

Preliminary Operations and Identification Tests

The methods of analysis used by manufacturers and consumers should be consulted. This gives one a rough outline of what is to be expected in spite of the fact that many of these tests are based on physical properties and performance, such as density, flash point, boiling range, solvent properties, viscosity, etc. Methods of analysis for paint thinners and lacquer solvents are well covered by Gardner[1] and Watts[2]. Much information on industrial solvents is given by Mellan[3] and Scheflan[4]. The industrial hygiene viewpoint is presented in the texts by Jacobs[5] and Goldman[6].

Solvents containing dissolved or suspended solids, such as lacquer thinners or paints, may be subjected to a preliminary steam or vacuum distillation to separate the solvents. This operation may introduce additional complications such as the separation of the sample into a water soluble and insoluble portion. However, it is essential for such materials.

Several miscibility tests may be rapidly and conveniently performed. These are carried out by pipetting portions of solvent into stoppered graduates. To these are added equal volumes of test reagent and the liquids are mixed gently

Prepared by: Bernard E. Saltzman, Ph.D.
Revised by: Robert L. Larkin

Table 1. - Solvents Used in Various Industries

Antifreeze

- Ethanol
- Isopropyl alcohol
- Methanol
- Ethylene glycol
- Glycerol

Automotive (exclusive of antifreeze)

- Chlorinated hydrocarbons
- Ether-alcohols
- Plasticizers
- Polyether alcohols

Dry Cleaning

- Chlorinated hydrocarbons
- Naphtha

Paint, Varnish, Lacquer, Ink

- Alcohols
- Coal tar hydrocarbons
- Esters
- Ether alcohols
- Ketones
- Plasticizers
- Turpentine

Petroleum

Selective Solvents

- Benzene-sulfur dioxide
- Cresylic acid
- Dichloroethyl ether
- Furfural

Petroleum (continued)

Selective Solvents

- Nitrobenzene
- Phenol
- Propane

Solvent Dewaxing

- Acetone
- Benzene
- Ethylene dichloride
- Methyl ethyl ketone
- Propane

Pharmaceutical

- Alcohols
- Chlorinated hydrocarbons
- Ethers
- Ketones

Rayon

- Acetone
- Carbon disulfide
- Esters
- Ethers

Textiles (other than rayon)

- Glycol derivatives
- Glycols
- Hydrocarbons
- Lacquer solvents
- Pine tar derivatives

by inversion several times. In order to avoid emulsification, these tests are carried out without violent shaking. The volumes of the two layers should be carefully noted so that the percentage of miscibility may be calculated.

If the first graduate is dried and weighed, the specific gravity may be quickly determined from the gain in weight. Add an equal volume of water. Complete miscibility excludes hydrocarbons, halogenated hydrocarbons, and many of the long chain acids, alcohols, phenols, secondary nitro compounds, neutral compounds, etc.

In the second stoppered graduate the miscibility with an equal volume of concentrated hydrochloric acid (or 4:1 H_2SO_4) may be determined. In addition to the solvents which dissolve in water, others, such as alcohols, esters, aldehydes, ketones, some unsaturated compounds, anhydrides, ethers, and quinones may be expected to dissolve in the acid. Only inert compounds such as hydrocarbons and halogenated hydrocarbons do not dissolve.

Table 2. - Organic Liquids Arranged in Order of Vapor Hazard

Substance	Vapor (a) Hazard	Threshold (b) Limit PPM by Vol.	B. P.(c) °C
Butylamine	34,000	5	77.8
Carbon disulfide	23,000	20	46.3
Allyl alcohol	16,450	2	96.6
Tetranitromethane	15,800	1	126
Carbon tetrachloride	14,170	10	76.8
Diethylamine	12,600	25	55.5
Acrylonitrile	11,200	20	78-9
Methyl formate	7,600	100	32
Pyridine	5,260	5	115-6
Benzene	5,000	25	80.1
Chloroform	4,970	50	61.2
Ethyl formate	3,160	100	54
Ethyl bromide	3,030	200	38.4
1,1-Dichloroethane	2,900	100	57.3
Ethylene dichloride (1,2 dichloroethane)	2,320	50	83.7
Acetic acid	1,970	10	118.1
Ethylene chlorohydrin	1,900	5	128.8
Ethylene diamine	1,710	10	55.5
1,1,2,2-Tetrachloro-ethane	1,684	5	146.3
Benzyl chloride	1,580	1	179.4
Ethyl ether	1,380	400	34.6
Methyl acetate	1,380	200	57.1
Acetic anhydride	1,340	5	139.6
Methylene chloride	1,080	500	40-1
Trichloroethylene	1,000	100	87.2
Propylene dichloride (1,2-dichloropropane)	910	75	96.8
2-Nitropropane	896	25	120.3
Dimethylaniline (N-dimethylaniline)	870	5	193
Methyl alcohol	820	200	64.7
Ethylene dibromide (1,2-dibromoethane)	740	25	131.5
Methyl cellosolve (2-methoxyethanol)	630	25	124-5
2-Butanone	625	200	79.6
Mesityl oxide	536	25	130.0
Methylal (dimethoxy-methane)	526	1000	42-3
Dioxane (diethylene dioxide)	490	100	101
1,1,1, Trichloroethane (Methyl Chloroform)	489	350	74.1
Nitrobenzene	474	1	210.9
Pentane	450	1000	36.3

Table 2. - Organic Liquids Arranged in Order of Vapor Hazard (continued)

Substance	Vapor (a) Hazard	Threshold (b) Limit PPM by Vol.	B. P.(c) °C
Nitromethane	434	100	101
Propyl ether (isopropyl ether)	420	500	69
Hexane (n-hexane)	410	500	69
Propyl acetate	390	200	88.4
Cyclohexane	320	400	80-1
Ethyl acetate	303	400	77.1
Acetone	290	1000	56.5
Methyl isobutyl carbinol (methyl amyl alcohol)	263	25	131.8
Nitroethane	263	100	114.8
Perchloroethylene (Tetrachloroethylene)	237	100	120.8
Amyl alcohol (isoamyl alcohol)	220	100	132
Chlorobenzene (monochlorobenzene)	210	75	132.1
Cresol (all isomers)	184	5	191-203
Toluene	184	200	110.8
Gasoline	176	500	60-120
Methyl cellosolve acetate (ethylene glycol mono-methyl ether acetate	163	25	144.5
Propyl alcohol (isopropyl alcohol)	140	400	82.5
Aniline	132	5	184.4
Phenol	132	5	181.4
Heptane (n-heptane)	121	500	98.4
Methylcyclohexane	121	500	101
Cyclohexanone	113	50	155-6
Pentanone (methyl propyl ketone)	112	200	95
Dichloroethyl ether	96	15	178.5
Styrene monomer (phenyl ethylene)	97	100	145-6
Naphtha (petroleum)	95	500	80-130
Hexone (methyl isobutyl ketone)	93	100	117-9
Butyl alcohol (n-butanol)	92	100	117
Butyl acetate (n-butyl acetate)	79	200	127
Ethyl alcohol (ethanol)	76	1000	78.4
p-Tertiary butyl toluene	72	10	200
Diisobutyl ketone	71	50	168.1
Naphtha (coal tar)	66	200	110-60
o-Toluidine	66	5	199.7
Turpentine	66	100	120-80

Table 2. - Organic Liquids Arranged in Order of Vapor Hazard (continued)

Substance	Vapor (a) Hazard	Threshold (b) Limit PPM by Vol.	B. P.(c) °C
Ethyl benzene	63	200	136.2
Nitrotoluene	55	5	222-38
Amyl acetate	53	100	142.0
Hexanone (methyl butyl ketone)	50	100	128
Xylene	50	200	139-44
Methylcyclohexanone	47	100	162-70
Cyclohexanol	47	50	160-1
Cellosolve acetate (hydroxyethyl acetate)	42	100	156.3
Cellosolve (2-ethoxy-ethanol)	41	200	135.1
Octane	34	500	99-125
Isophorone	26	25	215
Phenylhydrazine	11	5	243.5
o-Dichlorobenzene	9	50	179
Diacetone alcohol (4-hydroxy-4-methyl-pentanone-2)	8	50	167.9
Methylcyclohexanol	7	100	165-75
Stoddard solvent	7	500	150-90

(a) Ratio (ppm/ppm) of equilibrium vapor concentration at 25°C to the Threshold Limit Value, computed from vapor pressure data in references (3,8).
(b) From A.C.G.I.H. Threshold Limit Values for 1964.
(c) Boiling point at 760 mm Hg. from references (3,8,9). Observed boiling points in mixtures may be lower due to formation of azeotropes.

Solubility in dimethyl sulfate may be used to distinguish between the aliphatic and aromatic hydrocarbons, as all ordinary solvents are miscible with this reagent except the aliphatic hydrocarbons. Chlorinated hydrocarbons do not give a clear picture in this test since they are partially miscible. In their absence, the difference between the residue immiscible in dimethyl sulfate, and the residue immiscible in strong acid may be taken as the approximate aromatic content. If this value exceeds 25%, the sample should be diluted with pure paraffinic hydrocarbons and the dimethyl sulfate test repeated, as otherwise the results may be too high.

From the three miscibility values the approximate classification of the solvent may be made according to solubility classes. If significant amounts of solvent fall into different classes it is generally advisable to treat a large fraction of the sample with appropriate solutions so that it may be separated into the various groups. Unfortunately, clear cut separations may not be obtained for substances such as halogenated hydrocarbons. However, solubility separations are desirable because of the lack of specificity of many of the procedures. For example, in a fractionation column many azeotropic mixtures can exist and the interpretation of the boiling point is unclear unless a relatively simple mixture is being distilled. Similar considerations apply to other

methods such as spectroscopy, index of refraction, etc.

Another simple preliminary test which should be carried out is the Beilstein test for halogenated hydrocarbons. A drop of solvent is placed on a small loop of copper wire and heated in a flame. A green flame indicates halogenated hydrocarbons. If these are present in sufficient quantities the solvent may not be flammable. A smoky flame would indicate a high aromatic content in many cases.

The odor of the solvent is a simple test which may give an important lead as to the presence of amines, nitro compounds, esters, aldehydes, etc. A more complete collection of organic classification tests may be found in Shriner and Fuson[7].

Analysis of Hydrocarbon Mixtures

Aromatic, aliphatic, and halogenated hydrocarbons may be determined by fractionating the inert oil residue obtained by treating a portion of the sample with 4:1 sulfuric acid. A suitable fractionating column consists of a well-insulated tube, ½ inch in inside diameter and 3 feet long, packed with glass helices, with an accurate thermometer in the top vapor space connection to a special condenser. The sample is placed in a flask at the bottom and is boiled using a Glascol heating jacket with a Variac control. Most of the condensed vapor is returned from the top of the packed column as reflux. A small portion, of the order of a third to a tenth of the condensate is withdrawn as distillate. Such a column effects successive condensations and reevaporations in the streams of rising vapor and descending liquid and is the equivalent of many simple distillations. In this way a separation of closely boiling components in mixtures may be obtained.

Fractionation

Several hundred milliliters of the sample which has been previously dried with anhydrous sodium sulfate or potassium carbonate and decanted is placed in the still flask. The heat is regulated to give the highest distillation rate without flooding the column. The proper operation of the column may be checked at any time by setting the condenser stopcock at total reflux. If the top temperature drops only slightly within a few minutes, the distillate withdrawal rate is not excessive. The distillate is received in a previously weighed glass-stoppered graduate.

A running plot is kept of the cumulative volume distilled (abscissa) versus the temperature at the top of the column (ordinate). This gives the boiling point curve of the sample. The theoretical distillation curve for a mixture of substances is a series of steps. Each horizontal plateau represents the pure component being distilled at the time. When this has been completely removed the temperature rises steeply until it reaches the boiling point of the next component. The best way of carrying out the distillation is to study the running plot of the temperature versus the volume distilled and to change receivers during the steep temperature increases. Thus, the receiving cylinders will each contain a single pure component. In the case of petroleum distillates, so many compounds are present that the individual steps are not resolved, giving a gradually rising plot. In such a case, cuts are made at 95°C, and 125°C, for determination of benzene, toluene, and xylene as described below.

Additional tests. During the course of the distillation other tests may be made, such as a Beilstein test in the event that the presence of chlorinated hydrocarbons are suspected, or an index of refraction and dispersion measurements. These may be made on a single drop of distillate. A sudden change is an indication that the end of that component has been reached and indicates that the cut be taken.

At the conclusion of the distillation the different cuts in graduated cylinders may be weighed. From the volumes, the densities may be computed. Other tests, such as index of refraction may be carried out. Identification of each component is based on the boiling point, density, index of refraction and dispersion, miscibility with dimethyl sulfate, Beilstein test, etc. of the various cuts.

Determination of Aromatics in Hydrocarbon Distillates

The most convenient way of determining aromatics is by index of refraction and dispersion measurements. Specific dispersion, which is the index of refraction for the F (hydrogen) line minus the index of refraction for the C (hydrogen) line, divided by the density, is the most direct measure of aromatic content. If a cut boiling below 95°C is being examined only benzene may be expected. This has a specific dispersion of 0.0190, whereas the specific dispersion for paraffins and cyclic aliphatics in the boiling range below 95°C extends from 0.0096 to 0.0100. By means of a simple proportion the aromatic content may be estimated from the dispersion data. If a cut boiling between 95 and 125°C is being examined only toluene may be expected as the aromatic. The specific dispersion of toluene is 0.0185 whereas the non-aromatics in this fraction range between 0.0096 and 0.0101. The aromatic content of the cut between 125 and 150°C may be estimated using the specific dispersion for aromatics of 0.0174 to 0.0181 for ethyl benzene and the xylenes as compared to 0.0095 to 0.0099 for the non-aromatics. This method is relatively simple and sufficiently accurate in most cases. It requires only reading the instrumental value from the dispersion compensator. The conversion of this number to dispersion is accomplished using tables provided with the refractometer.

The index of refraction may be used also. to determine aromatic content. However, this calculation requires an estimate of the refractive indices of the paraffins and naphthenes (cyclic aliphatics). These refractive indices have a greater variation than the dispersion and are thus more uncertain in unknown mixtures.

Ultraviolet spectrophotometry also may be utilized to determine aromatics which absorb intensely in this region. The peak absorption for benzene occurs at 255 millimicrons, for toluene at 262, for ethyl benzene at 262, for o-xylene at 263, for m-xylene at 265, and for p-xylene at 274 millimicrons. A suitable solvent for these measurements is a paraffin hydrocarbon such as isoctane. For 1 cm cells the working range extends up to concentrations of a few tenths of a gram of aromatic hydrocarbon per liter. Standards may be run using the pure substances in the usual manner. As some of these peaks are quite sharp the resolution of the spectrophotometer will affect the standardization factor considerably.

It is also possible to use the spectrophotometric method on a mixture which has not been distilled and which may contain all of these aromatics. The sample is read at 5 wavelengths, as are each of the pure standards. By means of simultaneous equations the composition of aromatics may be obtained. Wavelengths which have been used are 255.0, 265.0, 268.5, 272.5, and 274.5 millimicrons. The mathematical handling of these equations is somewhat difficult. Furthermore, from the toxicity viewpoint the more volatile aromatics are of the greatest importance. Thus, ordinarily, fractionation is the better procedure.

Aromatic content may be determined on the distillate by dimethyl sulfate miscibility in the same manner as described under preliminary tests.

Analysis of Water and Acid Miscible Fractions

Many solvents, such as lacquer and paint thinners, may contain little or no hydrocarbon. A mixture of alcohols (ROH), esters (RCOOR'), ketones (RCOR') and

ethers (ROR'), may be expected. These may be partially or completely miscible with water.

The water extract may be fractionated as described above for hydrocarbon mixtures. A boiling point curve, temperature versus cumulative distillate volume, should be plotted as before. One of the more toxic substances which should be sought is methanol (B.P., 64.7°C). If there is any sign of a plateau at this temperature the fraction may be analyzed for methanol, using Schiff reagent or chromotropic acid[5,6]. It should be kept in mind that many azeotropic mixtures may be formed which boil at temperatures differing from those of the pure components. Thus, the absence of a plateau at the boiling point of methanol does not exclude this substance. If a pure substance, which might reasonably be expected, cannot be found, tables of azeotropic boiling points[3] should be consulted.

The analysis of lacquer solvents is a rather complex procedure which has been discussed more fully elsewhere[1,2]. Esters may be determined by saponification with standardized, alcoholic potassium hydroxide under total reflux for an hour, after which the unconsumed potassium hydroxide is determined by titration. The most valuable methods for this analysis are based upon the distillation curve and auxiliary physical data, followed by specific chemical tests for type compounds and individual substances.

Tests may be made in the same manner upon the water insoluble-acid soluble fraction. In such a case 4:1 sulfuric acid should be used rather than hydrochloric acid because of its nonvolatility. If it seems appropriate, the acid may be neutralized with sodium hydroxide. This will salt out the organic solvents, which will separate from the aqueous portion. It should be borne in mind that this treatment may serve to hydrolyze the esters and change other components in the solvent. This fraction may then be treated as above.

It is evident that solvent analysis involves a good deal of detective work. When the composition of the solvent is reasonably well known from the industry, the analysis may be relatively simple. In other cases an exact analysis is not required as all the components may have a relatively low order of toxicity. The analyst should make certain that the most toxic substances listed in Table 2 are absent.

Confirmatory Tests Made on Gas Chromatograph Effluent[10]

Instrumental methods available for solvent analysis include gas chromatography, infrared and ultraviolet spectroscopy, and mass spectrometry. The advent of the gas chromatograph has permitted amazing separations of very complex mixtures. The analysis of solvent mixtures such as dry cleaning fluids, paint and lacquer thinners, floor cleaners, degreasing solvents, etc., have been simplified by using this technique.

If the solvent contains dissolved material it is necessary to remove it because the detector may be damaged or the column may be plugged. A 6-inch hypodermic needle will serve as sufficient attachment to the chromatograph exit tubing to collect the components being separated by bubbling the effluent into simple receivers such as 15 ml centrifuge tubes or homemade capillary borosilicate glass tubes.

Ketones

In this test the effluent gas from the column is passed through the hypodermic needle, which is dipped into a solution of 2,4 dinitrophenylhydrazine. A yellow to orange precipitate indicates a positive test for ketones. Table 3 gives the limit of detection and the colors of some ketones tested by this method. The reagent is not specific for ketones since aldehydes also react. However, aldehydes are not likely to occur in the type of material discussed.

Table 3. - Ketone Detection Test

Ketone	Limit of det., μl	Color of ppt.
Acetone	0.1	yellow
Methyl ethyl ketone	0.1	yellow
Methyl isopropyl ketone	0.1	yellow
Methyl isobutyl ketone	0.2	yellow
n-Butyl ethyl ketone	0.2	yellow
Cyclohexanone	0.4	yellow
Mesityl oxide	0.5	red

Esters

For the identification of esters the well known hydroxamic color test is used. The effluent is collected in an alkaline solution of hydroxylamine hydrochloride. Ten minutes after the sample is collected ferric perchlorate reagent is added. A violet color indicates a positive test for esters. The range of detection for some esters is shown in Table 4. Anhydrides, acid chloride, and formic acids may also give a positive test by this method. However, acid chlorides and anhydrides are not likely to appear in solvent mixtures.

Table 4. - Ester Detection Test

Ester	Limit of detection, μl
Methyl acetate	0.04
Ethyl formate	0.04
Ethyl acetate	0.04
Isopropyl acetate	0.06
Ethyl propionate	0.04
n-Propyl acetate	0.06
sec-Butyl acetate	0.06
Isobutyl acetate	0.05
Ethyl n-butyrate	0.06
n-Butyl acetate	0.06
Isoamyl acetate	0.07
n-Hexyl acetate	0.09
n-Heptyl acetate	0.10
Phenyl acetate	0.10
n-Amyl acetate	0.07

Aromatic Hydrocarbons

The effluent gas from the chromatograph is bubbled into a special sulfuric acid-formaldehyde solution. Table 5 lists some aromatic hydrocarbons which give a positive test.

Table 5.-Aromatic Hydrocarbon Detection Test

Aromatic Hydrocarbon	Limit, μl	Color
Benzene	0.5	cherry red
Toluene	0.5	cherry red
o-Xylene	0.6	cherry red
m-Xylene	0.6	cherry red
p-Xylene	0.6	cherry red

Aliphatic Alcohols

The effluent gas is bubbled into a few drops of a ceric ammonium nitrate solution. Table 6 gives some alcohols which give positive identification. A red rather than an amber color may be obtained with a higher concentration of the alcoholic hydroxyl group.

Table 6.-Alcohols Giving Positive Test

Alcohol	Limit, μl	Color
Methyl	0.4	amber
Ethyl	0.4	amber
Isopropyl	0.5	amber
n-Propyl	0.5	amber
n-Butyl	0.8	amber

Halogenated Hydrocarbons

Excellent group specificity and sensitivity can be obtained by using a modified Beilstein test. Column effluent containing chlorinated hydrocarbons is directed through the exit hypodermic needle into a simple burner. The burner is fed with natural gas and the flame is adjusted for minimum luminosity and maximum stability. When chlorinated hydrocarbons are present a green color is imparted to the flame. Table 7 lists chlorinated hydrocarbons with their detectable limits using this test.

Table 7.-Halogenated Hydrocarbon Detection Test

Halogenated Hydrocarbon	Limit, μl
Carbon tetrachloride	0.13
Chloroform	0.20
Methyl chloroform	0.20
Methylene chloride	0.40
Trichloroethylene	0.70
Ethylidene chloride	0.20
Ethyl bromide	0.20

References

1. Gardner, H. A.: Paint, Varnishes, Lacquers, Colors. Henry A. Gardner Laboratory, Inc. Bethesda, Maryland, 10th Ed., 1946.

2. Watts, C. E.: Detection and determination of solvents and volatile liquids in nitrocellulose lacquers and lacquer thinners. Ind. Eng. Chem., Anal. Ed. 6:262 (July 1934).

3. Mellan, I.: Industrial Solvents. Reinhold Publishing Corp., New York, 1939.

4. Scheflan, L., and Jacobs, M. B.: The Handbook of Solvents. D. Van Nostrand Inc., New York, 1953.

5. Jacobs, M. B.: The Analytical Chemistry of Industrial Poisons, Hazards and Solvents. Interscience Publishers Inc., New York, 1946.

6. Goldman, F. H., and Jacobs, M. B.: Chemical Methods in Industrial Hygiene. Interscience Publishers, Inc. New York, 1953.

7. Shriner, R. L., and Fuson, R. C.: The Systematic Identification of Organic Compounds. John Wiley & Sons, Inc., New York, 1948.

8. Jordan, T. E.: Vapor Pressure of Organic Compounds. Interscience Publishers, Inc., New York, 1954.

9. Lange, N. A.: Handbook of Chemistry. Handbook Publishers, Inc., Sandusky, Ohio, 6th Ed., 1946.

10. Dubois, L., and Monkman, J. L. in "Gas Chromatography" (H.J. Noebels, R. F. Wall, and N. Brenner, eds.) pp. 237-46. Academic Press, New York and London, 1961.

ORGANIC EXTRACTION METHODS IN TRACE ANALYSIS

Introduction

Special Problems of Trace Analysis

Trace analysis differs from conventional analytical procedures because of the requirements imposed by the extremely small quantities of substances being determined. Trace analysis methods require special cleaning of glassware, purified reagents and careful attention to small details. The requirements are so exacting that these procedures may be regarded as qualitatively different from the usual type of analysis. For example, in the analysis of urine for lead, the amount normally encountered is of the order of 2 or 3 micrograms. If an attempt were made to determine this small quantity by the usual procedures, such as hydrogen sulfide precipitation, no precipitate would be obtained. Evidently the greatest care is required in such an analysis because the quantity of lead may be less than that in the reagents used and contamination from a single speck of dust may introduce more lead than that present in the sample.

Cleaning of Glassware

As an appreciable amount of glass is dissolved in these chemical procedures, only borosilicate glassware should be used to minimize this contamination. It is advisable to reserve a set of glassware exclusively for trace metal analysis. This glassware is cleaned in the usual manner so that it is visibly clean and then given a rinse with strong acid, such as 1:1 hydrochloric acid or concentrated nitric acid, followed by tap and distilled water. Pipettes and stoppers are never laid on the laboratory bench during the course of the analysis. Special racks for supporting pipettes and stoppers have been found convenient and thereby avoid contamination. If any appreciable period of time is to elapse during the course of analysis, open beakers are covered with watch glasses, parafilm, large sheets of filter paper or special rigid plastic covers.

Preparation of Samples

The preliminary operations require dissolution of the sample followed by destruction of the organic matter. Dissolution is generally effected by heating with strong acid, such as sulfuric, hydrochloric, nitric or perchloric acids. Special techniques, such as fusion, are required for certain minerals and dusts. Ashing, or destruction of organic matter, is then performed by heating with strong oxidizing agents, such as nitric and perchloric acids, hydrogen peroxide, potassium permanganate, etc. Special precautions are required in certain determinations, such as those for mercury, arsenic, and antimony, to avoid volatilization of these elements. In general a recommended ashing procedure is specified for each type of sample and for each determination.

Separations

Because trace analysis involves very unfavorable ratios of other substances to the element being determined, separations are generally required. The extent of the separations necessary depends upon the type of determination. Only rarely is the determination so specific that some separation is not required.

Separation by precipitation, which is the classical method, is generally not applicable to trace analysis. The quantity of the analysis element is so small that it may be within the solubility range of even its most insoluble salts. Procedures have been developed in which another element is added to co-precipi-

Prepared by: Richard E. Kinser and
John L. Holtz

tate the element being determined. However, problems of supersaturation still remain, and, also, such procedures are generally time-consuming.

Distillation is a very effective means of separation and may be applied in certain special determinations such as those for arsenic and fluoride.

In recent years many ion exchange resins have been developed which may be used to separate desired constituents, even at the trace element level, from other substances present in the sample solution by their differential adsorption on the resin. These procedures involve varying conditions of acidity, concentration, effect of mesh size and cross linkage etc. in order to accomplish these separations. Two ion exchange methods of separation involving mercury and fluoride in biological samples have been developed in our laboratory[1,2]. Also an anionic exchange resin is now used in our laboratory to completely remove lead from chemical reagents for the preparation of spectrographic standards and in solutions of normal blood ash. These separations are actually better than dithizone extractions.

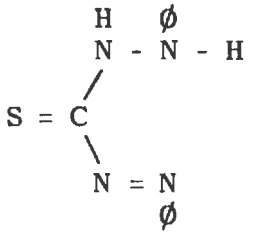
The most generally applicable method for separations in trace metal analysis is by extraction as metallo-organic complexes with organic solvents. These procedures are very convenient and rapid and are very effective in separating even sub-microgram quantities of most elements. As will be seen later, dithizone is a unique reagent in that it may be used not only for determination but also for these preliminary separations.

Determinations

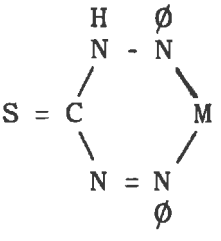
The usual gravimetric and titrimetric determinations ordinarily are not satisfactory for trace analysis due to the extremely small quantities of material involved. The most useful methods for trace metal analysis are colorimetric determinations conducted with organic reagents, emission spectrography and more recently, neutron activation analysis. The sensitivity of the colorimetric methods equals or exceeds that of the spectrographic methods in some instances. Colored metallic complexes are frequently concentrated by extraction with organic solvents.

Dithizone as a Reagent for Trace Metal Analysis

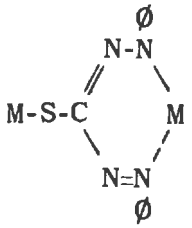
Dithizone is a chelating agent which reacts quantitatively with microgram quantities of a score of metals to produce intense colors, ranging from red through yellow, brown, and violet. The color of dithizone itself is green. Dithizone and its metallic complexes are quite soluble in solvents such as chloroform and carbon tetrachloride. Many separations by extraction procedures can be accomplished. It is a versatile reagent and is used for the determination of many metals. The formula for dithizone is presented below as I. This reagent reacts with monovalent heavy metals to produce the keto form or primary dithizonate II, or the enol form or secondary dithizonate III. The primary and secondary dithizonate formulae for the products with divalent metals are shown in IV and V, respectively. Dithizone is easily oxidized by many substances such as trivalent iron, alkaline copper solutions, halogens, nitrous acid, etc., producing a yellowish brown material as indicated by the structure in VI. This product is soluble in chloroform and carbon tetrachloride but is insoluble in water, whereas dithizone itself is soluble in alkaline aqueous solutions.



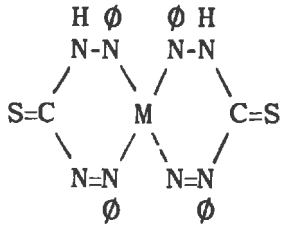
I



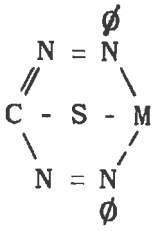
II



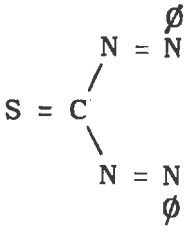
III



IV



V



VI

Table 1 presents a periodic table arrangement showing the elements which react with dithizone. These elements are listed in more detail in Table 2, which gives the pH for the formation and extraction of each complex in carbon tetrachloride and the color in the solvent. It is apparent that, because of the large number of elements which react with dithizone, it is necessary to provide some additional means of making these procedures specific.

NOTE: * Page 141

** Pages 142, 143 and 144 - E. B. Sandell, Ph.D., Chemical Analysis, Vol. 3, Colorimetric Metal Analysis, 3rd Edition, 1959.

Table 1.-Metals Reacting with Dithizone. (The enclosed elements yield dithizonates in CCl_4 or $CHCl_3$)

H																
Li	Be											B	C	N	O	F
Na	Mg											Al	Si	P	S	Cl
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br
Rb	Sr	Y	Zr	Nb	Mo	Te	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	-
--	Ra	Ac	Th	Pa	U											

Table 2. - Elements Reacting with Dithizone (P = primary dithizonate; S = secondary dithizonate).

Metal	Complex	Color (CCl_4)	pH for extraction
Bismuth	P	Orange-yellow	>2 (CCl_4)
	S	Orange-red	Basic soln.
Cadmium	P	Red	Basic soln.
Cobalt	P	Violet-red	8-9 (optimum in CCl_4)
	S?	Brownish	Strongly basic soln.
Copper (I)	P	Brown	Dilute mineral acid soln. (ca.0.1N HCl)
(I)	S	Violet	Basic soln.
(II)	P	Violet-red	Dilute mineral acid soln.
(II)	S	Yellow-brown	Basic soln.
Gold (III)	P	Yellow	Dilute mineral acid medium (e.g., 0.5N H_2SO_4)
	S	Red	(5-6(CCl_4)
Indium (III)	P	Red	(8.3-9.6 ($CHCl_3$)
Iron (II)	P	Violet-red	7 (CCl_4)
Lead	P	Cinnabar red	8-5-11 (optimum in $CHCl_3$)
Manganese (II) 1	P	Violet-brown ($CHCl_3$)	ca.11
Mercury (I)	P	Orange	Dilute mineral acid soln.
(I)	S	Purplish red	Basic medium
(II)	P	Orange-yellow	Mineral acid soln. (even 6 N H_2SO_4)
(II)	S	Purplish red	Basic medium
Nickel	P	Brownish	Weakly basic medium
Palladium (II)	P	Brown	Mineral acid soln.
	S	Red-violet	Mineral acid soln. with excess Pd
Platinum (II)	P?	Light yellow	1:3 HCl ($SnCl_2$ present)
Polonium			95% complete extraction in pH range 0-5, with 0.1% $CHCl_3$ soln. of H_2Dz
Silver	P	Yellow	Dilute mineral acid soln.
	S	Red-violet	Basic soln.
Tellurium (IV)	P?	Maximum absorption at 430 mμ	0.1-1N mineral acid soln.

Table 2. (continued)

Metal	Complex	Color (CCl_4)	pH for extraction
Thallium (I)	P	Red	9-12 (CCl_4)
(III)	P	Yellowish red	3-4 (CCl_4)
Tin (II)	P	Red	4 (optimum 6-9 in CCl_4)
Zinc	P	Purplish red	Neutral or weakly basic soln.

Variables in Dithizone Complex Formation

Hydrogen ion concentration. pH is the most important variable for increasing the specificity of the reactions of dithizone with metals. Each metal has a certain pH range in which the reaction with dithizone is complete as shown in Figure 1, which presents equilibrium curves for some metal dithizonates in chloroform.

It is evident that the noble metals may be extracted at strongly acid pH values, whereas metals such as tin, bismuth, copper, zinc and lead require increasingly higher pH values for effective reaction. Other metals such as thallium and cadmium require extremely alkaline solutions for this complex formation. Many metal complexes are stable in strong alkali. Thus, cadmium, cobalt, nickel, copper, mercury and silver complexes are stable in 1N sodium hydroxide. Other metals, however, are not extracted in strongly alkaline solution as indicated in Figure 1 for tin, bismuth, zinc and lead. This may be due to the amphoteric nature of these elements which exist in other ionic forms in strongly alkaline solution, such as the zincate and plumbite forms. The solvent also has an effect upon the reaction pH. Thus, while the values shown in Figure 1 are for chloroform as a solvent, the values which might be expected when carbon tetrachloride is the solvent are one pH unit lower.

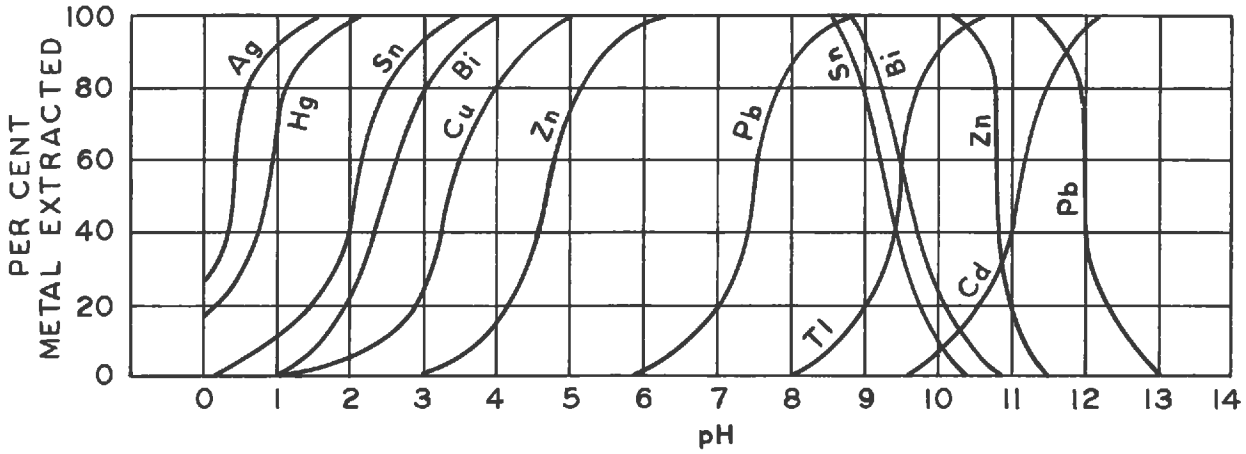


Figure 1. - Equilibrium Curves for Some Metal Dithizonates in Chloroform. (From Welcher, F.J., Organic Analytical Reagents, Vol. III, p. 471, D. Van Nostrand Co., New York, 1947).

Extraction reactions are quite reversible and the complex may be transferred back and forth indefinitely from the aqueous to the organic layer if the pH of the aqueous layer is changed appropriately. In the procedures given below, this reversibility is utilized in conducting separations. A group of metals is extracted by an organic solvent from the aqueous layer which is then discarded. The organic solvent is then stripped of these metals by shaking with a second aqueous solution of appropriate pH. Some metals such as nickel, cobalt and bismuth exhibit a marked hysteresis effect in this regard, requiring a much more strongly acid solution for the stripping operation than would be expected.

Valence. Variation of metallic valence provides another important means of effecting separations in dithizone procedures. This may be applied to metals such as iron, tin and platinum which do not react in their higher valence forms but do complex in the reduced forms. Thus, if these metals are in aqueous solution in their higher valence forms, interfering metals can be extracted without affecting this group. Then, after treatment of the aqueous solution with a reducing agent, these metals may be extracted in a purified state in their lower valence forms.

Complexing agents. Another very important method for making dithizone extractions specific is the use of complexing agents such as iodide, thiosulfate, and cyanide as illustrated in Table 3. These reagents effectively control the interfering metals by forming very tightly bound complexes which prevent these elements from reacting with dithizone.

The procedure for a single metal analysis is based upon the proper combination of these variable conditions so as to favor the reaction with the metal being determined and suppress reactions with the interfering metals likely to be present. The limiting amounts of these interfering metals which can be tolerated are established experimentally.

Table 3. - Complex-forming Reagents in Dithizone Reactions.

Conditions	Metals Reacting
Basic solutions containing citrate and tartrate.	Usually without effect
Basic solution containing cyanide.	Pb, Bi, Sn(II), Tl(I)
Slightly acid solution containing cyanide.	Cu, Hg, Ag, Pd
Dilute acid solution containing thiocyanate.	Cu, Au, Hg
Dilute acid solution containing thiocyanate and cyanide.	Cu, Hg
Acid solution of pH 5 containing thiosulfate, and extracted with CCl_4 solution of dithizone.	Pd, Sn(II), Zn(Cd)
Acid solution of pH 4-5 containing thiosulfate and cyanide.	Sn(II), Zn
Dilute acid solution containing iodide or bromide.	Cu, Au, Pd
Dilute acid solution containing chloride.	Cu, Au, Pd, Hg

Determination of Lead with Dithizone

There are a number of recommended dithizone methods for the determination of lead in air and biological materials[3,4,5,6]. The levels of lead in air samples are generally considerably higher than those found in biological samples, thus higher blanks may be tolerated and reagent purification may be simplified. Reagent purification for biological samples is quite important.

The U.S.P.H.S. procedure for lead in blood and urine is known as a double extraction method[4]. An aqueous solution of the ashed sample is brought to the proper pH by the addition of indicator and distilled ammonia. Lead is quantitatively extracted at a pH of 9 to 11. Since calcium and manganese phosphates will precipitate at the required pH, the prior addition of citrate is necessary to prevent such precipitation and concurrent losses of lead. Hydroxylamine hydrochloride is used to protect the dithizone from oxidation by mild oxidants. Most of the interfering metals are then complexed by the addition of potassium cyanide. When this solution is extracted with a chloroform solution of dithizone, only lead, bismuth, thallium and stannous tin will be transferred to the organic solvent. The aqueous solution containing the ashed salts may then be discarded. The extracted metals are stripped from the organic solvent by shaking with an exact volume of one percent nitric acid. The chloroform layer may then be discarded. Subsequent extraction with standardized dithizone under carefully controlled conditions will yield quantitative results for lead.

Other procedures are used, such as the one for lead in air, in which only a single extraction is made to separate and determine lead[6,7]. These procedures have the advantage of speed and are applicable to special types of samples. Highest accuracy may not be expected with the single extraction procedures. Traces of oxidant in the sample, perhaps formed as a by-product of the ashing process, may partially destroy some of the dithizone. The absorbance of unreacted dithizone at the wavelength used is considerable thus an appreciable error may occur. The single extraction procedures are subject to more interference because the single stage cyanide treatment is not as effective in suppressing interferences as the two stage method.

As stated previously lead, bismuth, thallium and stannous tin are determined by these procedures. Thallium is rarely encountered and does not constitute a practical interference. Any tin in the sample would be oxidized to the stannic state during ashing. Tin, therefore, does not interfere in this procedure. Bismuth does interfere with the determination of lead by this method, but it is rarely found in biological samples. Bismuth present in oral medications generally appears in the feces and does not enter the blood or urine unless taken over a prolonged period[8]. Intravenous injection of bismuth for syphilis therapy is seldom practiced in view of the more modern treatments. If the presence of bismuth is suspected or known, a special procedure may be applied[9]. Lead is stripped from the first extract with a pH 3.4 buffer. The bismuth dithizonate remains intact and is not stripped from the chloroform layer. The pH is quite critical and the buffer has little capacity; therefore, prior washing of the chloroform layer with distilled water is recommended to remove the entrained alkali.

These procedures are known as "Mixed Color Methods" because of the presence of both the red lead dithizonate complex and the unreacted green dithizone.

Microdetermination of Cadmium with Dithizone

Cadmium may be determined specifically with dithizone by extraction at very high pH values. Although cadmium is

40

complexed by cyanide, carefully regulated amounts of cyanide are used in amounts which suppress interfering metals but not cadmium. Under these conditions very few other metals will extract to any great extent. The aqueous layer is then discarded and the chloroform extract is stripped of cadmium by shaking with 2 percent tartaric acid. A second extraction of cadmium is then made under similar conditions with standardized dithizone. The details of this method are given in references[10 & 11].

It will be noted in this procedure that difficulties arise due to the instability of strongly alkaline chloroform solutions. Additional amounts of cyanide may be produced by the reaction of traces of ammonia with chloroform under these conditions, resulting in low cadmium recovery. These difficulties are minimized by using cold solutions and the shortest possible contact time of chloroform with strong alkali. In this manner complete extraction of cadmium is obtained.

Determination of Mercury with Dithizone

Ashing of mercury samples is a special problem because of the volatility of mercury salts. Total reflux procedures are necessary. The treatment of air samples is outlined in the ACGIH method[12]. In urine samples the presence of water reduces greatly the efficacy of the oxidizing action. Satisfactory results have been obtained by digesting 100 ml of urine with 15 ml of concentrated nitric acid and 7½ grams of potassium permanganate under total reflux for one hour. A 500 ml flask is necessary because of foaming. Sulfuric acid is not required. After cooling, the permanganate is reduced by slow addition of hydroxylamine hydrochloride, 5 ml of which is then added in excess. After additional cooling, meta cresol purple indicator is added and the solution is cooled and extracted with dithizone according to the ACGIH method for air samples[12].

The basic principle of the mercury procedure consists of extraction in strongly acid media, followed by shaking the organic layer containing the mercury with an acidified, concentrated, solution of potassium bromide which strips the mercury, leaving copper and noble metals in the chloroform layer which is discarded. The aqueous layer is then brought to pH 6 by the addition of buffer solution. A second treatment with standard dithizone solution then extracts the isolated mercury. It should be noted that mercury dithizonate is sensitive to light and the solution must be kept out of direct sunlight. As stated previously, however, a method has been developed whereby mercury is separated by an ion exchange procedure and then complexed directly with a standard dithizone reagent[1]. The sensitivity is much improved over the dithizone extraction method.

Other Reagents for Organic Extractions

There are naturally, a large number of organic reagents available for the separation and determination of metals. The majority of these reagents form chelate complexes in which one or more of the acidic hydrogens of the reagent have been replaced by the metal. Several of these reagents which have been found useful, are discussed below.

Acetylacetone

Acetylacetone forms chelate complexes with many of the heavy metals. This reagent has been found to be of great value in isolating beryllium from samples down to a small volume after ashing, due to the large amount of calcium present in bone ash. However, by using the acetylacetone extraction method, beryllium acetylacetonate can be extracted by benzene at pH 4.5 and beryllium is thereby separated from the alkaline earths and phosphate. The beryllium is then transferred from the benzene phase to the aqueous phase by shaking with 5N HCl. The amount of

beryllium present is then determined spectrographically.

Bathophenanthroline

There are several good spectrophotometric methods for iron which use 1,10-phenanthroline and its derivatives. The 4,7-diphenyl derivative "bathophenanthroline" has increased the sensitivity and lessened interference in the analysis of iron[14]. Hydroxylamine hydrochloride is added to an aliquot of the ashed sample to reduce all the iron present to the ferrous state. The bathophenanthroline is added and the pH is adjusted to about 5 with buffer solution. Chloroform is added and the samples are shaken. Distilled water is added and again the samples are shaken and are allowed to settle. The chloroform layer is drained off and the samples are extracted for a second time with chloroform. The extracts are combined and made to volume with ethanol and read spectrophotometrically. The standard curve is linear for the range 0- 12.5 micrograms of iron.

References

1. Kopp, J.F. and Keenan, R.G.: Determination of Submicrogram Quantities of Mercury in Urine by Ion Exchange Separation. Amer. Ind. Hyg. Assoc. J. 24:1 (Jan.-Feb. 1963).
2. Talvitie, N. A. and Brewer, L. W.: Separation of Fluoride by IonExchange: Application to Urine Analysis. Amer. Ind. Hyg. Assoc. J. 21:4 (Aug.1960).
3. Committee on Chemical Procedures of the Occupational Health Section, American Public Health Assoc., Inc.: Methods for Determining Lead in Air and in Biological Materials, American Public Health Assoc., New York, 1955.
4. _____: Determination of Lead in Air and in Biological Materials. The "USPHS" Double Extraction Mixed Color Dithizone Method. Recommended Method, American Conference of Governmental Industrial Hygienists, 1963.
5. Keenan, R. G., et. al.: The "USPHS" Method for Determining Lead in Air and in Biological Materials. Amer. Ind. Hyg. Assoc. J. 24:481-491(1963).
6. _____: Determination of Lead in Air. A Single Extraction, Mixed Color Dithizone Method. Recommended Method, American Conference of Governmental Industrial Hygienists, 1951.
7. Woessner, W. W. and Cholak, J.: Improvements in the Rapid Screening Method for Lead in Urine: A.M.A. Arch. of Ind. Hyg. & Occ. Med. 7:249(1953).
8. Elkins, H.B. and Ruotolo, B. P. W.: Notes on Determination of Lead by Dithizone Method. Interference from Bismuth and Tin. Amer. Ind. Hyg. Assoc. Quart. 14:111 (1953).
9. Bambach, K. and Burkey, R.E.: Microdetermination of Lead by Dithizone. Ind. Eng. Chem., Anal. Ed. 14:904 (1942).
10. Saltzman, B.E.: Colorimetric Microdetermination of Cadmium with Dithizone. Anal. Chem. 25: 493 (Mar.1953).
11. _____: Determination of Cadmium in Air. Monocolor Dithizone Method. Recommended Method, American Conference of Governmental Industrial Hygienists, 1957.
12. _____: Determination of Mercury in Air. Dithizone Method. Recommended Method, American Conference of Governmental Industrial Hygienists, 1951.
13. Toribara, T.Y. and Chen, P.S., Jr.: Separation of Beryllium from Biological Material. Anal. Chem. 24: 539 (1952).
14. Reynolds, R. G. and Monkman, J. L.: The Determination of Iron in Dustfall Samples. Amer. Ind. Hyg. Assoc. J. 23:415 (Sept.-Oct. 1962).

FREE SILICA - ITS FORMS, OCCURRENCE, AND DETERMINATION

Introduction

Silica is a term applied to silicon dioxide. The silica of concern in Occupational Health is silicon dioxide uncombined and independent of other elements and is therefore said to be "free silica." Silicon dioxide found in combination with other elements is called "combined" silica. Free silica and combined silica have differences in physiologic action and also in the methods used for their analysis.

Table 1
Examples of Free and Combined Silica

	Free Silica	Combined Silica
Example	Quartz	Feldspar
Formula	SiO_2	$KAlSi_3O_8$

The formula for orthoclase feldspar can be written as $K_2O \cdot Al_2O_3 \cdot 6SiO_2$. This may give the erroneous impression that feldspar contains silicon dioxide in a free form. Chemical formula written as above are forms of combined silica.

The silicon content of rocks for industrial uses is commonly reported in terms of percent silica which represents the "total" silica present. Total silica is the sum of free silica and silicon dioxide present in combined silica. This same misinterpretation may apply to dust analyses unless the reports specify that the silica is "free", "combined" or "total".

It should be noted as a matter of interest that silica gel is precipitated silicic acid in the form of lustrous granules. The approximate formula is H_2SiO_3. Therefore, it is outside the scope of our consideration.

Prepared by: Niilo A. Talvitie
Revised by: John V. Crable

Forms of Free Silica

Silicon dioxide as free silica exists in both the crystalline and amorphous state. A crystal is composed of atoms arranged in a system periodic in three dimensions. Precious gems are perhaps the best known crystals. Solid materials without periodicity of atomic arrangement, i.e., random orientation, are defined as amorphous. Glass is an amorphous material.

Crystalline Free Silica. Free silica has three crystalline forms: quartz, tridymite, and cristobalite. Quartz constitutes about 12% of all rocks and is the most common of the three crystalline forms of free silica.

Sand, sandstone, and quartzite are composed almost entirely of quartz. Significant amounts of quartz are usually found in clay, shale or slate. Since quartz has a widespread distribution, an appreciable quartz content is found in ores, talc, mica, feldspar, perlite, pumice and other industrial minerals.

The cryptocrystalline varieties of quartz are composed of minute grains cemented together with amorphous silica. These include chalcedony, agate, onyx, jasper, siliceous sinter, flint, hornstone, chert, and many others.

Tridymite is present on a large scale in certain siliceous volcanic rocks. The lavas in the San Juan District of Colorado contain large amounts of this form of crystalline silica. Tridymite is the principal constituent of silica refractory brick.

Cristobalite is present also in many siliceous volcanic rocks. It is the principal constituent of calcined

diatomaceous earth and is often found in ceramic materials and refractory bricks. Cristobalite may be produced by the thermal decomposition of some silicate minerals.

Modifications of Crystalline Silica

The thermal conversion of one crystalline form of silicon dioxide to another crystalline form is a sluggish process but may be hastened by traces of a flux. The thermal conversion is not reversible, for once formed the crystalline forms remain stable at ordinary temperatures.

Quartz is the most common form and is stable below 870°C. Above 870°C quartz is converted to tridymite whereas tridymite is converted to cristobalite at 1470°C. A liquid is formed at 1700°C.

Each of the crystalline forms has minor variations called low and high temperature forms or alpha and beta modifications. These changes are rapid and are reversible.

Quartz Alpha $\overset{573^{\circ}C}{\longleftrightarrow}$ beta

Tridymite Alpha $\overset{117^{\circ}C}{\longleftrightarrow}$ beta, $\overset{163^{\circ}C}{\longleftrightarrow}$ beta$_2$

Christobalite Alpha $\overset{200^{\circ}C \quad 272^{\circ}C}{\longleftrightarrow}$ beta

Amorphous Silica. Fused silica and fused quartz are amorphous and differ from crystalline free silica in physiological properties. Opal is a natural hydrated form of amorphous silica whereas silica gel and silica aerogel are artificial products of the same form. Diatomaceous earth is an opaline silica composed of the skeletons of diatoms, but converts to cristobalite when calcined at high temperatures, particularly with a trace of alkaline flux present.

Determination of Free Silica

Chemical Methods

Gravimetric Method. The rate of solubility of silica depends on whether it is combined or free silica. The combined silica is more soluble than the free silica and therefore permits a means of separating silicates from crystalline silica. Quartz has the lowest dissolving rate and amorphous silica the highest with tridymite and crystobalite being intermediate. The rate of solution is also dependent on particle size.

Dust samples can be analyzed for quartz by using the above information. The constituents of a dust sample other than quartz are removed by solution with reagents which have little action on quartz. Hydrofluosilicic acid, fluoroboric acid, fused potassium pyrosulfate, sulfuric acid in combination with sodium hydroxide, and phosphoric acid are suitable reagents for silica determinations. Phosphoric acid is the preferred reagent. When heated at a controlled rate, phosphoric acid dissolves most silicates in 12 to 14 minutes and has little effect on the quartz. Under these conditions the loss of 200 mesh quartz is only about 1 percent. The pyrophosphoric acid, which forms during the heating of the sample with the acid, complexes with both the metallic elements and the combined silica. The quartz residue can be recovered by diluting the acid solution and filtering.

Colorimetric Method. The following is a brief description of a colorimetric method for the determination of total silica and it is usable on small samples. A sample size of 2.5 milligrams or less is the range for this method and it is therefore ideal for determining total silica in air-borne dust samples. Particulate silica and silicates are collected on membrane filters, dissolved in hydrofluoric acid and determined colorimetrically as yellow silicomolybdate or as molybdenum blue after suitable reduction.

Microscopic Methods

A few substances do not dissolve readily in phosphoric acid. It is wise to examine a portion of the residue of quartz microscopically and, if substances other than quartz are found to be present, an attempt should be made to identify these and a correction applied to the analysis on the basis of their chemical composition.

This identification can be carried out most easily by mounting the residue in an oil having the same refractive index as quartz. A mixture of equal parts by volume of d-chloronaphthalene and light mineral oil (liquid petrolatum) is suitable and may be adjusted to a refractive index of 1.548 by means of a refractometer. With ordinary illumination the quartz will be invisible permitting attention to be concentrated on the impurities. A polarizing attachment to the microscope is useful in obtaining a rough estimate of the percentage of extraneous particles since the quartz particles may be made visible when observed between crossed polarizers.

The quartz in the bulk of the residue is volatilized with hydrofluoric acid in the presence of sulfuric acid and any remaining residue interpreted in accordance with the microscopic examination in terms of percentage of undissolved extraneous minerals or other substances.

Differential Thermal Analysis

When a substance is heated, it may undergo chemical or physical changes. These are usually accompanied by the evolution or adsorption of heat. The temperature and the amount of heat involved in any such reaction is characteristic of the material being heated.

Crystallographic inversions may be a source of thermal reaction. As mentioned above, quartz, tridymite, and cristobalite each have thermal modifications. Quartz undergoes a small, precise, reversible thermal modification associated with the latent heat of inversion of α to β quartz at 573°C. This change is used by Differential Thermal Analysis (D.T.A.) for the determination of quartz.

X-Ray Diffraction

Powder Technique. Laue's discovery in 1912 that crystals diffract X-rays also showed that the manner of the diffraction revealed the structure of the crystal. In the beginning, X-ray diffraction was used only for the determination of crystal structure but with experience other uses were developed. X-ray diffraction is now widely used in chemical analysis.

The individuality of a diffraction pattern provides an excellent means by which crystalline compounds can be identified. X-ray diffraction can be applied either qualitatively or quantitatively to the examination of industrial dusts for free silica. The three crystalline forms of free silica i.e., quartz, tridymite, and cristobalite can be distinguished from each other and from the amorphous forms which give no distinct line pattern.

A qualitative pattern of the sample is obtained first, and, if quartz is present as determined by comparison with a pattern of pure quartz, the percentage of quartz is roughly estimated.

A quantitative determination requires an internal standard and the preparation of a standard curve. Beryl can be used as an internal standard in the quantitative determination of quartz. Beryl is similar to quartz in brittleness, fracture characteristics, hardness, and specific gravity. Both quartz and beryl approach the same degree of fineness in grinding.

The standard curve for quartz analysis is plotted from the ratios of intensities of the beryl 3.26 A line to the quartz 3.35 A line which were obtained

from diffraction analysis of a series of beryl and quartz mixtures. These mixtures should include, besides pure quartz and pure beryl, ratios of beryl to quartz of 1:1, 2:1, 3:1, 6:1 and 9:1. Plotting the ratios of intensities against the ratios of weights provides data for drawing the standard curve.

In practice, a qualitative pattern is first determined in order to estimate the percent of quartz present. Beryl is added to a weighed sample in about 3 to 4 times the estimated amount of quartz present. This mixture which should weigh about 0.5 gram is ground and the diffraction pattern obtained. The ratio of the density of the beryl line to the density of the quartz line is computed. Using the standard curve, prepared as outlined above, the ratio of the weights of beryl to quartz can be obtained. The percentage of quartz in the sample can be computed as follows:

$$\frac{\text{Wt. Beryl} \times 100}{\text{Wt. Sample} \dfrac{(\text{Wt. Beryl})}{(\text{Wt. Quartz})}} = \% \text{ Quartz}$$

Other substances may be present in the sample which interfere with the quartz 3.35 A line. Among these are mica, graphite, and cassiterite. In such cases, the 4.26 A line of quartz may be used.

Membrane Filter Technique. This is a method by which small samples can be analyzed by X-ray diffraction. A qualitative analysis can be made or a quantitative analysis can be determined by use of an external standard. A known weight of the sample is suspended in a definite volume of water. A volume of the suspension, containing about two milligrams of the sample, is filtered through a membrane filter. After the filter has dried, it is mounted in the diffractometer. External standards are prepared in a similar manner from finely ground quartz, cristobalite and tridymite.

References

1. Talvitie, N. A. and Brewer, L. W.: X-ray Diffraction Analysis of Industrial Dust. Amer. Ind. Hyg. Assoc. J., 23: 214-221 (1962).

2. Talvitie, N. A. and Brewer, L. W.: Separation and Analysis of Dust in Lung Tissue. Amer. Ind. Hyg. Assoc. J., 23: 58 (1962).

3. Talvitie, N. A. Determination of Quartz in Presence of Silicates Using Phosphoric Acid. Anal. Chem. 23: 623 (1951).

4. Cullity, B. D. Elements of X-ray Diffraction. Addison-Wesley Publishing Company Inc., Reading, Mass., 1956.

5. Hurlbut, C. S. Dana's Manual of Mineralogy. John Wiley and Sons, Inc., New York, 1941.

6. Smathers, W. J. and Chiang, Y. Differential Thermal Analysis: Theory and Practice. Chemical Publishing Company, Inc., New York, 1958.

DETERMINATION OF NITROGEN OXIDES IN AIR

Introduction

Nitrogen oxides occur in chemical manufacturing operations such as those involving nitric acid, nitrates, dyes, explosives, nitro-cellulose paints and lacquers. They are also produced in blasting, welding, electroplating, and metal cleaning operations, as well as in fires, in the exhaust of internal combustion engines and in furnace stack gases. Nitrogen oxides have become very prominent recently as important factors in smog generation.

Properties of Nitrogen Oxides

A series of nitrogen oxides exists. Many discrepancies in reported toxic effects may be due to failure to distinguish between different oxides which have widely varying properties.

Nitrous Oxide (N_2O). Commonly known as laughing gas, this oxide of nitrogen has been used for anesthesia. Concentrations as high as 80 to 86% have been used in dentistry, thus this oxide may be regarded as being of little importance from the industrial toxicology point of view.

Nitric Oxide (NO). This gas has no irritant properties. The effects on the body are probably due to anoxia. It combines with the blood and produces cyanosis caused by methemoglobinemia. No after effects occur unless the exposure is carried beyond the point of asphyxial convulsions and sudden central paralysis.

Nitrogen Dioxide (NO_2). This is the most irritating and toxic of all oxides of nitrogen. Many fatalities have been caused by this gas. Delayed pulmonary edema and sudden collapse are the symptoms to be expected. The present threshold limit value (TLV) is 5 ppm. Nitrogen dioxide may be produced by the trimolecular oxidation of nitric oxide by oxygen of the air:

$$2\ NO + O_2 = 2\ NO_2$$

The half life of this reaction at 25°C is 69 hours divided by the initial parts per million of nitric oxide. Thus if the latter is present at a level of 100 ppm the half life of the nitric oxide would be 41 minutes. After aging only 2¼ minutes the TLV of 5 ppm for NO_2 would be exceeded. Subsequently, the toxicity of the nitrogen dioxide present would greatly outweigh that of the nitric oxide. Thus in a mixture of nitric oxide and nitrogen dioxide, we orginarily need be interested only in nitrogen dioxide levels.

Other Nitrogen Oxides. These are much less important. Nitrous acid anhydride (N_2O_3) and dinitrogen tetroxide (N_2O_4) dissociate to produce nitric oxide and nitrogen dioxide. These dissociations are favored by lower pressures and are complete at concentrations below 100 ppm. Because of the great rapidity of their reactions, these oxides can be eliminated from the picture.

Nitrogen pentoxide (N_2O_5) is a rare and unstable oxide produced only by special procedures such as the reaction of nitrogen dioxide with ozone or the dehydration of nitric acid with phosphorus pentoxide. Its hydration by atmospheric water vapor to produce nitric acid vapor is very rapid. It also decomposes much less rapidly to a mixture of nitrogen dioxide and oxygen, the half life at 25°C being 5.3 hours.

Both nitric acid vapor and nitrous acid vapor may be present in the air at an appreciable level. These are much less toxic than the equivalent amount of nitrogen dioxide.

Another oxide, nitrogen trioxide (NO_3),

Prepared by: Bernard E. Saltzman

may be found in very small amounts in mixtures of nitrogen oxides and ozone. However, these levels would be very low and the situation is so unusual that the formidable analytical problems have not been solved. Ordinarily the major toxic effect may be expected to come from the ozone.

Sampling Methods

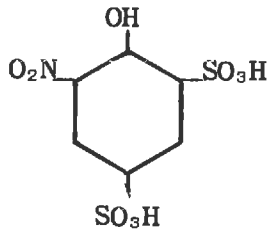
It may be seen from the foregoing that nitrogen oxides may exist in complex mixtures. To these difficulties is added the problem of sampling. Nitrogen dioxide may be sampled by a continuous scrubbing technique with good efficiency even at low concentrations.(1) It is likely that nitrogen pentoxide, if present, would also be sampled quite efficiently by an aqueous solution. Little information is available about continuous sampling methods for other nitrogen oxides.

The efficient continuous absorption of nitric oxide has not been attained. This problem has been investigated in detail by Thomas, et. al.,(2) who recommended the conversion of nitric oxide to nitrogen dioxide and its estimation in that form. Thus, two determinations are necessary. The first gives the total nitric oxide and nitrogen dioxide and the second gives the nitrogen dioxide alone. The difference is the amount of nitric oxide. The oxidation of nitric oxide to nitrogen dioxide was achieved by the addition of small amounts of ozone from air passed near a Westinghouse 794H ultraviolet lamp. Chlorine dioxide and acid potassium permanganate solutions were also effective oxidizers. The Thomas method is applied widely in air pollution studies for continuously recording low concentrations (fractions of a ppm) of these oxides.

If the total nitrogen oxides are to be determined it is generally necessary to use grab sample methods rather than continuous scrubbing devices. This necessitates the use of evacuated bottles or of flushing devices. A rather large sample of the order of several liters is necessary when working with levels close to the maximum allowable concentration of nitrogen dioxide (5 ppm). In the following sections, two methods will be described which are useful for determining total nitrogen oxides, following which the methods for determining nitrogen dioxide specifically will be discussed.

Phenoldisulfonic Acid Method for Total Nitrogen Oxides

Details of this method are given in the ACGIH procedure.(3) A grab sample is taken in a bottle containing a very dilute hydrogen peroxide-sulfuric acid absorbing reagent. After absorption of the sample, the solution is made alkaline and evaporated to dryness, after which it is treated with phenoldisulfonic acid. This reagent is readily nitrated giving the product shown below:



The mixture is then diluted and made alkaline with sodium hydroxide to obtain the trisodium salt of the nitro compound which has an intense yellow color. This color is determined photometrically at 410 millimicrons.

The reagents for this method (with the exception of the absorbing solution) may be kept for months. The nitration step is usually completed within two minutes, although ten minutes are allowed as a safe time. Final calculations are made using the perfect gas laws discussed in the lecture on gas and vapor sampling.

The weakest part of this method appears to be the sampling and absorption portion. When lower concentrations of

nitrogen oxides are determined the recovery of the method decreases.[1] Thus at the 10 ppm level only about 60% may be recovered. The absorption is very slow at these low levels. Even three days time is not sufficient for complete absorption since slightly higher results may be obtained when one week is allowed.

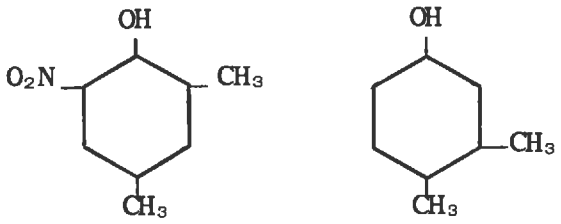
Other difficulties may arise for various reasons. The blank value is usually of the order of several ppm. Most of this is from the hydrogen peroxide used in the absorbing reagent. If the solution is made too alkaline during the initial evaporation step, an appreciable amount of material is dissolved from the dish, producing a turbidity in the final color. These make the method difficult to apply except at rather high levels.

If an appreciable amount of organic matter is present the sample may char, producing an off color and possibly causing loss of nitrate by reduction. In such a case the neutral sample may be ashed with hydrogen peroxide. The presence of chlorides may cause loss of nitrate as nitrosyl chloride. A complete preliminary treatment should consist of removing chlorides by treatment with silver sulfate. Then phosphate is added and the pH is adjusted to 6.5; the solution is filtered and ashed with hydrogen peroxide in the presence of calcium carbonate buffer. When the solution is finally made alkaline for color development, precipitation may occur in some samples. Sequestrene AA has been added to prevent this difficulty.

Xylenol Method for Total Nitrogen Oxides

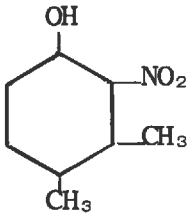
This method[6] may be preferred for certain operational advantages, although it has only about a quarter of the sensitivity of the phenoldisulfonic acid method. A grab sample is absorbed in 5 ml of 62.5% (5:3) sulfuric acid. Potassium permanganate is added to oxidize any nitrite followed by 0.2 ml of 1% m-xylenol in propylene glycol. Nitration is completed in a 10-minute interval, after which the sample is diluted with 100 ml of water and distilled. The nitrated meta xylenol (see I below) is volatile and is absorbed in 1 ml of 2% sodium hydroxide producing an intense yellow color which is determined photometrically at 450 millimicrons in a volume of 25 ml.

In a study of this method,[4] different nitration products and side reactions were shown to occur with the different forms of xylenol. Some products were not steam-distillable while others had low extinction coefficients. Certainly, for quantitative results, a pure reagent is necessary. These authors recommend a reagent of 3, 4-orthoxylenol (II) which gives a mixture of the 2-nitro (III) and the 6-nitro (IV) products.

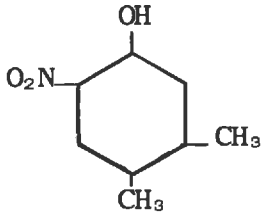


I

II



III



IV

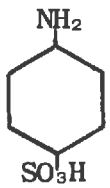
In another procedure,[5] instead of distillation, the nitro-xylenol is extracted from the acid solution with toluene. The aqueous layer is then discarded and replaced with 10 ml of 0.4 N sodium hydroxide. When this is shaken with toluene the salt goes into the aqueous layer, producing the color which is determined photometrically.

The advantages of the xylenol method are that sample evaporation is not required and no precipitation of metals occurs when the solution is made alkaline. The distillation or extraction steps enable one to make a separation from many interfering substances. However, this method has only about one-fourth the sensitivity of the phenol-disulfonic acid procedure.

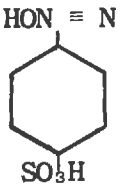
Methods for Nitrogen Dioxide

As indicated above, in most cases the major toxic effect of nitrogen oxide gases may be attributed to nitrogen dioxide. The specific determination of this form therefore is generally more meaningful than the group determination of all the nitrogen oxides by the foregoing procedures. The nitrogen dioxides may be determined simply using the Griess-Saltzman reagent.(1) Levels below the TLV of 5 ppm (1964) may be determined conveniently, using a midget fritted bubbler and sampling at the rate of 0.4 liters per minute. A fritted disc with a maximum pore diameter of 60 microns is recommended as giving good absorption efficiency without having an excessive pressure drop. Higher levels may be determined using evacuated bottles containing the reagent. Fifteen minutes after sampling, a pink color is completely developed and may be read visually or photometrically at 550 millimicrons. The colors are quite stable, only 5% loss in absorbance occurs per day.

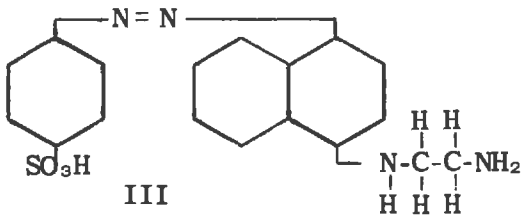
The reaction which occurs is the diazotization of sulfanilic acid (I) to produce the diazo salt shown in (II). This couples with N-(1-Naphthyl)-ethylenediamine dihydrochloride to produce the azo dye shown in (III).



I



II



III

The standardization of this method is empirical. The reaction of nitrogen dioxide gas appears to occur via two pathways, each of which may produce a different amount of color. For a given type of sampling apparatus, the color stoichiometric relationships are quite constant. The color obtained from one mole of nitrogen dioxide when a midget fritted bubbler is used as the sampling device is the same as the color obtained from 0.72 moles of potassium nitrite.

This method is highly specific for nitrogen dioxide. The interference of ordinary amounts of ozone, sulfur dioxide, and other nitrogen oxides is negligible. The only interference of any importance is that due to organic nitrites, which would rarely be found in appreciable amounts.

This reagent may be applied also to the determination of nitric oxide, if this gas is converted to nitrogen dioxide as described above under sampling methods. Commercial recorders are now available for nitrogen dioxide as well as nitric oxide using this reagent.(2) These have been found very useful in air pollution studies to indicate the variation of these gases during a 24-hour period. The reagent has been modified(7) for use in recorders by adjusting the concentrations of ingredients so that color development is twice as fast and the cost is half that of the unmodified reagent.

The Jacobs and Hocheiser method(8) has been applied for air pollution studies using automatic sequence samplers, when it is desirable to store samples for a considerable time without loss. The

color is developed by addition of reagents in the laboratory just before reading. Allowance should be made for low collection efficiency[9] if a midget impinger or coarse fritted bubbler is used for sampling. The absorbing reagent used is 0.1 N sodium hydroxide containing 2 ml of butyl alcohol per liter to promote foaming.

An interesting application of the Griess-Saltzman reagent is the nitrogen dioxide equivalent method for ozone.[10] About 5 ppm of nitric oxide is metered from a tank into the sample air stream. The mixture passes through an empty flask which allows 2/3 minute of flow reaction time, then through the reagent. Ozone stoichiometrically oxidizes the nitric oxide (present in excess) to nitrogen dioxide in the brief time allowed. Thus after deducting the blank obtained by a nitrogen dioxide determination without addition of nitric oxide (plus about 0.01 ppm caused by air oxidation of the added nitric oxide), the result is an analysis for ozone. This method is free from interferences from organic oxidants, or reducing gases such as sulfur dioxide and hydrogen sulfide, all of which interfere seriously with other methods for ozone.

References

1. Saltzman, B. E.: Colorimetric microdetermination of nitrogen dioxide in the atmosphere. Anal. Chem. 26: 1949, (Dec. 1954).

2. Thomas, M. D.; MacLeod, J. A.; Robbins, R. D.; Goettelman, R. C.; Eldridge, R. W.; and Rogers, L. H.: Automatic apparatus for determination of nitric oxide and nitrogen dioxide in the atmosphere. Anal. Chem. 28: 1810, (Dec. 1956).

3. Manual of Analytical Methods. Committee on Recommended Analytical Methods, American Conference on Governmental Industrial Hygienists: Determination of oxides of nitrogen in air. Phenoldisulfonic acidmethod. 1958.

4. Holler, A. C.; and Huch, R. V.: Colorimetric determination of nitrates and nitric acid esters. Anal. Chem. 21:1385, (Nov. 1949).

5. Barnes, H.: A modified 2:4-xylenol method for nitrate estimations. Analyst 75: 388, (July 1950).

6. Yagoda, H.; and Goldman, F.: Analysis of atmospheric contaminants containing nitrate groupings. Jour. Ind. Hyg. and Toxicol. 25:440, (Dec. 1943).

7. Saltzman, B. E.: Modified nitrogen dioxide reagent for recording air analyzers. Anal. Chem. 32:135, (Jan. 1960).

8. Jacobs, M. B.; and Hochheiser, S.: Continuous sampling and ultramicrodetermination of nitrogen dioxide in air. Anal. Chem. 30:426, (March 1958).

9. Perry, W. H.; and Tabor, E. C.: National air sampling network measurements of SO_2 and NO_2. A.M.A. Arch. Environ. Health 4:254, (March 1962).

10. Saltzman, B. E. and Gilbert, N.: Microdetermination of ozone in smog mixtures. Nitrogen dioxide equivalent method. Am. Ind. Hyg. Assn. J. 20:379, (Oct. 1959).

FLUORIDES - THEIR OCCURRENCE AND DETERMINATION

Introduction

The element fluorine is a member of the halogen family. It was first isolated in 1886 by H. Moissan. Fluorine, like other members of the halogen family, is so active chemically that it is found in nature only in combination with other elements. Unlike other members of the halogen family fluorine can have no more than a momentary existence in the atmosphere in the elemental state. It unites with hydrogen, silicon, sodium, calcium, aluminum, and other elements to form fluorides.

Occurrence and Uses

The compounds of fluorine have a widespread use in industrial processes, and because of the low threshold limit values assigned to them, they are in the group of frequently investigated air contaminants. Fluorides are widely distributed in nature. They are found as important constituents of practically all soils and sea water, as well as in the bones of all animals. Fluorides occur naturally in some domestic water supplies and in low concentration either naturally or by introduction in feeds, foods, and vegetation.

Fluorine compounds are liberated by industrial processes which make use of high temperatures in the treatment of fluorine-containing materials. The fluorine may be a natural impurity or may be added in a fluxing process such as fluorspar. The principal fluorine containing minerals of commercial importance are fluorite (fluorspar), cryolite, apatite, and sedimentary phosphate rock.

Fluorspar. Known chemically as calcium fluoride (CaF_2), fluorspar provides most of the fluorine for secondary uses and is mined in Illinois, Kentucky, and some of the western states.

Cryolite. (Sodium aluminum fluoride - $AlF_3 . 3NaF$). The only significant commercial deposit is located in Greenland. Cryolite has a direct use in the aluminum reduction industry as a molten electrolyte and solvent for bauxite, the basic ore of aluminum. Cryolite is also used in crop dusting as an insecticide.

Phosphate Rock. This is primarily calcium phosphate containing fluorine in amounts less than 4 per cent. The deposits in Florida are of sedimentary origin and consist of hydroxyapatite. This material, when treated with sulfuric acid, is converted into a more soluble form suitable for use as a fertilizer and feed supplement. Phosphoric acid, which can be made by treating phosphate rock with sulfuric acid, when treated with phosphate rock dust produces triple super phosphate -- a fertilizer with high phosphate content. The gaseous fluorine compounds, released in these processes, can create air contamination problems both inside and outside the processing plant. Some of the fluorine is recovered and converted into synthetic cryolite and other by-products. The phosphate deposits of the western states are more difficult to process but are being mined at an increasing rate. The hard crystalline apatite of metamorphic origin occurs only in small deposits in the United States.

Secondary Fluorine Compounds

Hydrofluoric acid is used for the etching and polishing of glass, for pickling stainless steel, and also by the petroleum industry in producing aviation gasoline by alkylation. Fluosilicic acid finds use as a disinfectant of copper and brass vessels in the brewing industry. It is used also as a hardener for concrete and in the electroplating of certain metals.

Prepared by: N. A. Talvitie
Revised by: Richard E. Kinser

Bifluorides (acid fluorides) are constituents of welding fluxes used particularly with aluminum and stainless steel. They have been used extensively to frost glass but are being displaced by sand blasting.

Fluoborates have value as fluxes in the casting of aluminum and magnesium. They are used also as electrolytes in plating with lead and similar metals.

The freons (chlorofluoromethanes and ethanes) have a low order of toxicity and are used extensively in refrigeration equipment and as solvent propellants in pressurized spray cans.

Toxic decomposition products are liberated by otherwise stable organic fluorine compounds when they come into contact with a flame or a hot surface. The same applies to plastics made of polymerized chlorofluoroethylenes and tetrafluoroethylene.

Analytical Methods for Fluorides

Sample Collection

Collection of samples for fluoride analysis is very important to the industrial hygienist and should be considered here. Hydrogen fluoride (HF), because of its high solubility, is generally assumed to be collected efficiently by a single standard impinger containing 100 ml. of 0.1N NaOH and operating at one cubic foot per minute (1 cfm) air flow. A midget impinger operating at 0.1 cfm also may be used. A ten to fifteen minute sample is adequate for concentrations near 3 parts per million of HF. Dusts and mists containing fluoride may be sampled with a standard impinger or midget impinger containing the appropriate amount of 0.1N NaOH and operating at the proper air flow. Fumes may be collected on a membrane filter, but if gaseous fluorides are also present the filter should be followed by an impinger. Particulate fluorides may be collected with the electrostatic precipitator or with filter media in a suitable holder. Some difficulty may be encountered with corrosive materials which may adhere too firmly to the precipitator tube.

Sample Preparation

If it is necessary to evaporate or concentrate samples that have been collected in aqueous media, the operation should be carried out in platinum dishes in the presence of an alkali. If this is not done some fluoride may be lost. Sodium hydroxide (low in fluoride) or a slurry of calcium hydroxide are suitable when no organic matter is present. When samples must be ashed to remove organic matter either calcium hydroxide or magnesium acetate should be used. Samples should be ashed at a dull red heat (550°C). Magnesium acetate is soluble in alcohol and is especially suitable for samples washed from electrostatic precipitator tubes with alcohol.

Blank values in fluoride determinations tend to be high. Special selection or purification of reagents is sometimes desirable.

Separation of Fluoride from Interferences

In fluoride determinations the analyst is quite often faced with the problem of interferences. Phosphate, free chlorine, sulfate, aluminum, iron, and other materials may interfere in many of the standard methods for fluoride. These materials must be removed from the sample before a reliable determination can be made. Fluoride can be separated from its interferences by:

(a) distillation
(b) ion exchange; and
(c) diffusion techniques

Distillation. Fluoride is separated from interferences by distillation as H_2SiF_6 from either a sulfuric or perchloric acid medium.[1] Consideration should be given to the acid to be used when the sample is prepared. If sulfuric acid is to be used, calcium hydroxide

should be avoided because of the insolubility of calcium sulfate. Distillation from sulfuric acid in the range of 135-145°C gives good recovery, but should not be used for samples when the subsequent method for determination is sensitive to small amounts of sulfate. Perchloric acid in the range of 135-140°C is suitable for a greater variety of samples but should be used in an all glass still and every precaution should be taken to avoid contact with organic matter. When chlorides are present they may be held back by the addition of either silver sulfate or silver perchlorate. The temperature of the acid is maintained within the desired limits by adding water through a dropping funnel or by steam distillation. Temperature control is somewhat easier with steam distillation. The steam inlet tube should extend nearly to the bottom of the distillation flask and should not be constricted or bent at the end, otherwise an atomizing action will carry spray into the receiver.

Silica present in the sample or dissolved from the glassware may deposit as a gelatinous mass above the level of the acid. Fluoride can be trapped by this gelatinous material unless the distillation is conducted in such a way as to continuously break up the mass and keep it in contact with the acid. Collecting twice the usual volume of distillate may be necessary for adequate recovery in the presence of gelatinous silica.

Fluoride may be distilled directly from a sulfuric acid water mixture boiling at 180°C.(2) Aqueous solutions containing fluoride are distilled from the mixture until a temperature of 180°C is reached. Distillation is stopped at this point to prevent excessive sulfate carry over. No dilution of the sample is produced therefore preconcentration is unnecessary. This distillation method cannot be used with colorimetric systems that do not tolerate small amounts of sulfate.

Ion Exchange. Ion exchange methods also have been used successfully to separate fluoride from its interferences.(3) Fluoride-containing solutions may be passed through a chromatographic column containing the appropriate resin. If the resin is properly chosen fluoride will be collected on the column and may be eluted from the column with a selected eluting agent. Ion exchange procedures provide a simple, low cost alternative to the distillation process. While ion exchange methods are not rapid in the sense of obtaining individual results quickly, the simplicity of the apparatus and the fact that operations proceed without constant attention allows for processing of a large number of samples simultaneously.

Diffusion. Recently, methods have been described that are based on the diffusion of fluoride as hydrogen fluoride in a confined atmosphere.(4,5,6) Small polyethylene bottles and plastic Petrie dishes have been used as diffusion cells. Fluoride in the sample diffuses to the upper half of the cell, which is thinly coated with sodium hydroxide. Samples are acidified with sulfuric or perchloric acid. As in distillation methods, chlorides are held back by the addition of silver sulfate or perchlorate. This procedure is particularly applicable to very small amounts of fluoride (1-20 micrograms).

Determination of Fluoride

Large amounts of fluoride may be determined by titration with thorium nitrate using monosodium alizarin sulfonate as the indicator and monochloroacetic acid as a buffer. Small amounts of fluoride are best determined photometrically. Several methods exist for the determination of fluoride but only a few will be discussed here.

The method of Megregian has as its basis the effect of fluoride ion on the zirconium lake of eriochrome cyanine R

in acid solution.[7] The method of Bellack and Schouboe is based on the bleaching effect of fluoride on the zirconium lake of 4,5- dihydroxy-3-(p-sulfophenylazo)-2,7- naphthalene disulfonic acid trisodium salt (Eastman Organic Chemicals No. 7309).[8] The two methods are similar except for the dye used. Megregian's reagent is more sensitive but requires a narrow band-pass spectrophotometer. The Bellack and Schouboe reagent is more stable and may be used with filter photometers. Both methods are somewhat temperature sensitive and because they depend on the bleaching of a deep color they require closely matched cells and accurate measurement of the color-forming reagents. The range of both methods is approximately 0.0-1.4 milligrams (mg) of fluoride per liter of aqueous solution. Sensitivity of the methods is about the same (0.05 mg F^-/liter). These methods are indirect methods and depend on the effect of fluoride ion on another color system.

A newer colorimetric method has been reported.[9,10,11] This method is based on the reaction of fluoride with the cerium and lanthanum chelates of alizarin complexone. The procedure makes use of a direct measurement of fluoride and has relatively few interferences. Procedures using the lanthanum chelate of alizarin complexone give linear plots in the range (0.0-0.4 mg F^-/liter). These methods have particular use in fluoride determinations using diffusion techniques for separation, since small samples are adequate.

Additional information on fluorides can be found in a survey of the literature. A few general references on the subject are given below.

References Cited

1. Willard, H. H. and Winter, O. B.: Volumetric Method for Determination of Fluoride. Ind. Eng. Chem., Anal. Ed., 5:7 (1933).

2. Bellack, E.: Simplified Fluoride Distillation Method. J. Amer. Water Works Assoc., 50:530 (1958).

3. Talvitie, N. A. and Brewer, L. W.: Separation of Fluoride by Ion Exchange, Application to Urine Analysis. Amer. Ind. Hyg. Assoc. J., 21: 287 (1960).

4. Singer, L. and Armstrong, W. D.: Determination of Fluoride, Procedure Based Upon Diffusion of Hydrogen Fluoride. Anal. Chem., 26:904 (1954).

5. Frere, F. J.: Specific Microdiffusion Method for the Determination of Fluorine. Anal. Chem., 33:644-5 (1961).

6. Rowley, R. J. and Farrah, G. H.: Diffusion Method for Determination of Urinary Fluoride. Amer. Ind. Hyg. Assoc. J., 23:314-18 (1962).

7. Megregian, S.: Rapid Spectrophotometric Determination of Fluoride with Zirconium - Eriochrome Cyanine R Lake. Anal. Chem., 26:1161(1954).

8. Bellack, E. and Schouboe, P. J.: Rapid Photometric Determination of Fluoride in Water. Anal. Chem., 30: 2032 (1958).

9. Belcher, R. et. al.: A New Spot Test for the Detection of Fluoride Ion. Talanta, 2:92 (1959).

10. Belcher, R. and West, T. S.: A Comparative Study of Some Lanthanon Chelates of Alizarin Complexan as Reagents for Fluoride. Talanta, 8: 863-70 (1961).

11. Yamamura, S. S. et. al.: Direct Spectrophotometric Fluoride Determination. Anal. Chem., 34:1308-12 (1962).

General References

1. Boltz, D. F.: Colorimetric Determination of Nonmetals, Chapt. VIII, Interscience Publishers, Inc., New York, 1958.

2. _____, _____: Back-Titration Method for Fluorides. Methods of Analysis, Assoc. of Official Agricultural Chemists, pp. 389-96, 1950.

3. Talvitie, N. A.: Colorimetric Determination of Fluoride in Natural Waters with Thorium and Alizarin. Ind. Eng. Chem., Anal. Ed., 15:620 (1943).

4. Thatcher, L. L.: Modified Zirconium-Eriochrome Cyanine R Determination of Fluoride. Anal. Chem., 29:1709 (1957).

SOUND AND NOISE

Introduction

Airborne sound refers to rapid pressure variations, that is, alternate increases and decreases in normal atmospheric pressure, caused by a vibratory object, and also the sensation experienced when such pressure changes strike the ear. One complete vibration of the object corresponds to one complete cycle of pressure change. The number of object vibrations per second (or pressure cycles per second) defines the frequency of a sound which is expressed as cycles per second (cps).

The frequency range of audible sounds for healthy young ears is usually considered to extend from 20 to 20,000 cps although there is evidence to indicate that man's hearing extends beyond these limits. The simplest type of sound, called a pure tone, is described as having a single frequency. Sounds, as encountered in nature, rarely consist of a single frequency. Indeed, music, speech and noise are each composed of many frequencies. The frequencies comprising speech are found principally between 250 and 3000 cps. This frequency range is considered most important to man since hearing losses for speech sounds would handicap the individual in most daily activities.

The magnitude of the pressure variations constituting a sound provide a measure of its strength or intensity. Actually the pressure variations producing audible sound are quite small. Normal atmospheric pressure is approximately one million dynes per square centimeter. The faintest sounds which can be detected by the ear are produced by pressure variations of approximately 0.0002 dyne per sq. cm. The pressure involved in producing the background noise levels which we encounter in offices or similar places is of the order of one dyne per sq. cm. or about one-millionth of the barometric pressure. The unit microbar is equal to one dyne per sq. cm. and the terms are used interchangeably.

The Decibel Scale

The ear is able to respond without difficulty over a remarkable pressure range A pressure one million times as great as that of the faintest audible sound can be tolerated before discomfort or pain begins to develop. To eliminate the difficulties arising from handling large numbers, it is customary to employ the decibel system which is logarithmic in nature. The formula for computing sound pressure level in decibels (dB) is

$$dB = 20 \log_{10} \frac{P_1}{P_0}$$

where P_1 is the pressure of the sound being measured and P_0 is a reference pressure. In industrial hygiene work, 0.0002 dynes per sq. cm. is usually the reference pressure. This value corresponds to the weakest sound that the ear can detect under the most ideal listening conditions. Sound pressure level measurements must always state the reference value being used. Unless otherwise stipulated, all sound pressure level measurements in this presentation will be given as re .0002 dynes/sq. cm. The sound pressure and decibel equivalent for some common sounds are given in Table 1.

Combining Decibel Readings

Since the decibel scale is logarithmic, decibel values cannot be added directly. If the values for two sounds measured separately are known, the approximate sound pressure level that would result

Prepared by: Charles D. Yaffe
Revised by: Alexander Cohen

Table 1

Sound Pressures and Decibel Values for Some Everyday Sounds

Sound Pressure (microbars)	Sound Pressure Level (dB re 0.0002 microbar)	Example
0.0002	0	Threshold of hearing
0.00063	10	
0.002	20	
0.0063	30	
0.02	40	
0.063	50	Residence
0.2	60	Conversational speech
0.63	70	
1.0	74	
2.0	80	
6.3	90	Subway
20	100	Looms in textile mill
63	110	Woodworking
200	120	Hydraulic press
2000	140	Jet plane

from combining them is obtained from the following table:

Difference between levels in dB	No. of dB to be added to higher level
0	3
1	2.6
2	2.1
3	1.8
4	1.5
5	1.2
6	1.0
7	0.8
8	0.6
10	0.4
12	0.3
14	0.2
16	0.1

In combining more than two sound pressure levels, the two highest levels should be combined first by this procedure. The total thus obtained is next combined with the highest remaining level, and this procedure is continued to completion.

Loudness Levels

It is important to realize that sounds of equal sound pressure level may not be equally loud. At sound pressure levels near 100 dB, frequencies between 20 cps and 1000 cps sound equally loud. At lower sound pressure levels the lower frequency sounds do not seem as loud as the 1000 cps tone. For example, a pure tone of 70 cps having a sound pressure level of 50 dB does not sound any louder than a 1000 cps pure tone having a sound pressure level of only 10 dB. At sound pressure levels below 40 dB, frequencies below 80 cps are inaudible.

Curves showing the sound pressure levels and frequencies required to produce similar loudness levels have been developed through tests on trained observers. These curves are called loudness-level contours. Loudness level is expressed in phons, and is numerically equal to the sound pressure level in dB, re 0.0002 dynes/sq. cm., of a 1000 cps pure tone which sound equally loud to a normal observer.

The sone is the unit of loudness. By definition a 1000 cps tone 40 dB above the listener's hearing threshold (or any sound having a loudness level of 40 phons) produces a loudness of 1 sone. The loudnesses of other sounds are compared with this value. Thus, a sound which seems twice as loud to that listener is said to have a loudness of 2 sones.

Through the relationships of decibels, phons and sones, it is possible to translate, at least to some extent, sound pressure levels, which can be measured, to the ear's response in terms of loudness.

Noise and Its Effects

Noise is defined as unwanted sound and may be arbitrarily classified into three basic types, namely, wide-band noise, narrow-band noise, and impulse-type noise. The total acoustical energy of a wide-band noise is distributed across a broad range of frequencies. Descriptions of such distributions, called spectra, can be obtained by dividing the noise into eight frequency bands, each one octave wide, and measuring the sound pressure level within each band.

Figure 1 illustrates data obtained from an octave band analysis of two different wide-band noises having the same total sound pressure level (98 dB). The energy contained in one sound appears to be uniformly distributed across the 8 octave frequency bands. The other noise, however, shows the acoustical energy to be concentrated largely within one or two bands. Information about the spectra of these sounds is helpful in making judgments about their loudness. As already noted, the ear does not respond equally to different frequencies. Hence, noises having the same total sound

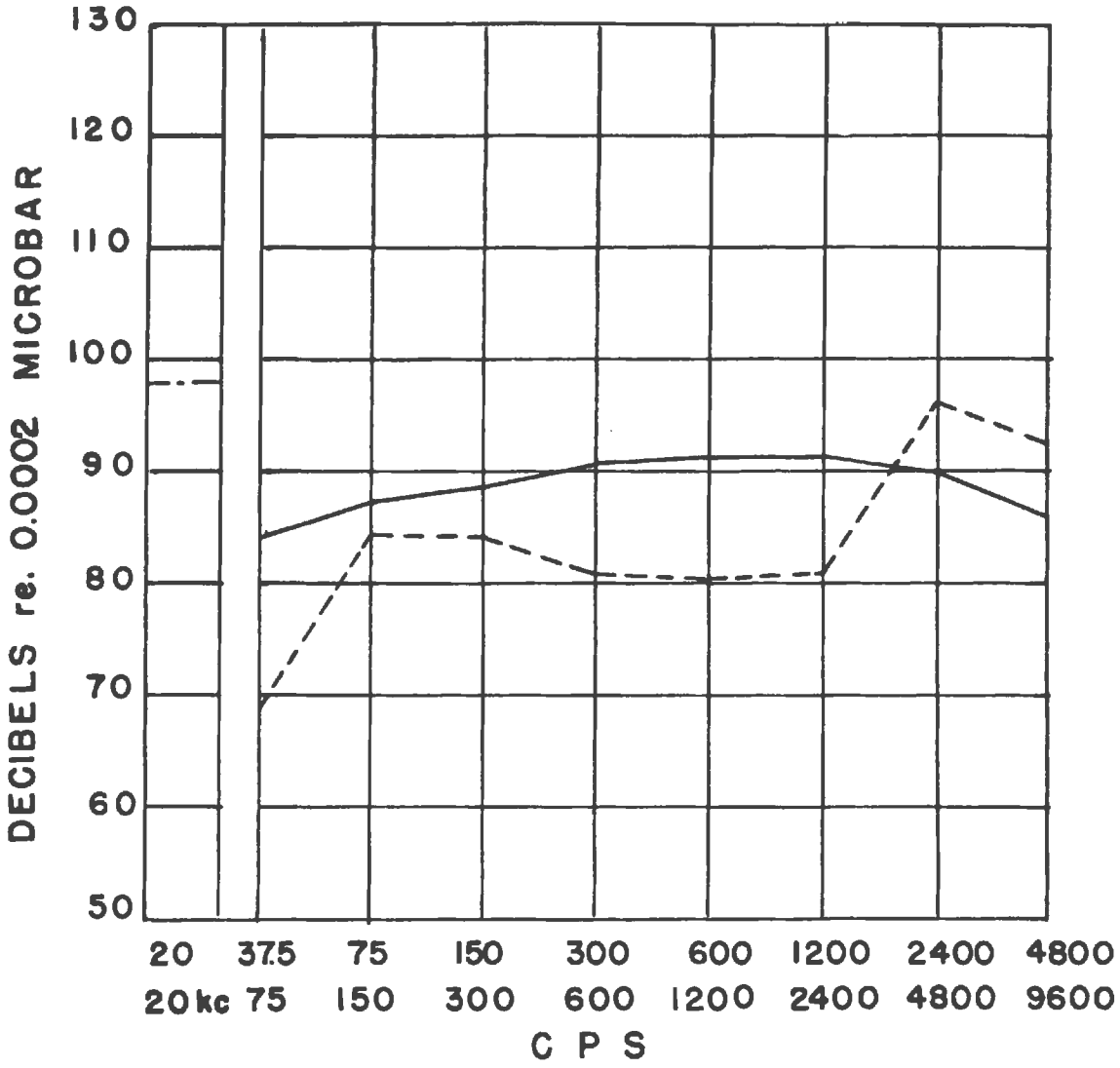


Figure 1. Spectral Differences between Two Sounds Having the Same Overall Sound Pressure Levels

pressure level may not be equally loud due to differences in frequency content or in the manner in which the various frequency components contribute to the total sound pressure level. As will be noted below, knowledge about the spectrum of a noise is also important in evaluating its potential harmfulness.

Types of Noise

Narrow-band noises have most of their energy confined to a narrow range of frequencies or concentrated about single frequencies. Accurate spectral determinations of such noises require an analysis of the total sound pressure-level into frequency bands which are smaller than an octave in width. The noise caused by circular saw, planer or power cutting tool operations is of the narrow-band type.

The impulse-type of noise consists of transient pulses, occurring in repetitive or non-repetitive fashion. Repetitive impulse noise is associated with the operation of a rivet gun or a pneumatic hammer. The impact of a drop hammer and the firing of a gun are examples of non-repetitive impulse noise. Techniques used to describe impulse noise differ from those used for narrow or wide-band types of noise. Repetitive impulse noise which occurs at a rate exceeding 200 pulses per minute, however, can be measured and evaluated in a manner similar to that used for wide- or narrow-band noise.

Effects of Noise

Noise may cause hearing loss, disruption of noise communication, annoyance and some impairment in performance. Losses in hearing may be temporary or permanent depending upon the length and severity of the exposure. Temporary hearing loss, also called auditory fatigue, represents losses which are recoverable after a period of time away from the noise. Such losses may occur after only a few minutes of exposure to intense noise. With prolonged exposures (months or years) to the same noises, there may be only partial recovery of the hearing losses, the remaining loss being indicative of a permanent hearing impairment.

Damage Risk Criteria

There exists a definite need to determine the maximum conditions of noise exposure which are tolerable to the ear. Specifications of such conditions, called "damage risk criteria", would be useful in serving as guides for

(a) hearing conservation programs in industry as well as the military service,
(b) noise control procedures and techniques as applied to machinery and work environments, and
(c) equitable rulings in court cases involving compensation for noise-induced hearing loss.

Many different damage risk criteria for noise exposure have already been proposed but none are considered to be sufficiently valid. Confirmation of these criteria will require, in particular, more knowledge of the effects of different types of noise upon hearing, the time required for the ear to become damaged given either continuous or intermittent types of exposure, the reversibility of any apparent hearing loss, and the importance of individual susceptibility to noise. In the absence of any well established noise criterion, it has been recommended that ear protective devices be used and periodic audiometric examination be administered to all personnel routinely exposed to noise having sound pressure levels of 85 dB or more in the octave bands 300-600, 600-1200, 1200-2400, 2400-4800 cps. Adjustments in the prescribed 85 dB limit are noted in such procedures to take into account short term exposures and noises having predominant concentrations of energy in a narrow range of frequencies or at single frequencies.

Hearing Loss and Aging

It is to be noted that the hearing losses caused by noise are similar to those associated with the aging process. Difficulty in distinguishing between these creates problems especially where compensation issues are involved. Much research has been conducted in recent years to determine the normal deterioration in hearing with increasing age for populations which have experienced only limited amounts of noise exposure. This information can be used to separate the amount of hearing loss due to excessive noise exposures from that due to aging.

Speech Interference

Noise which is not intense enough to cause hearing damage may still disrupt speech communication and the hearing of other desired sounds. While communication is essential in many places of employment, the levels of acceptability vary with the nature of the work involved. Being able to communicate by shouting, for example, may be satisfactory when doing maintenance work on certain machinery. On the other hand, raising the voice slightly to overcome typical office noises may be quite undesirable for a conference room. The average of the sound pressure levels found in the three octave bands 600-1200, 1200-2400, 2400-4800 cps provide a simple measure of the ability of a noise to interfere with speech communication. This average is referred to as the speech interference level which together with subjective ratings of noise has been used to establish guidelines for noise control in various types of work and living areas. Suggested criteria for selective areas in terms of the maximum permissible speech-interference-value are given in the following table:

Type of Room	Maximum Permissible Speech-Interference Level (measured when room is not in use)
Small private office	40 dB
Conference room for 20	30 dB
Conference room for 50	25 dB
Movie Theater	30 dB
Theaters for drama (500 seats, no amplification)	25 dB
Concert halls (no amplification)	20 dB
Secretarial offices (typing)	55 dB
Homes (sleeping areas)	25 dB
Assembly halls	25 dB
School rooms	25 dB

Speech-interference-level has been used also to predict the expected usability of a telephone under given noise conditions. The following schedule has been found generally useful in this regard.

Speech Interference Level	Telephone Usage
less than 60 dB	Satisfactory
60 to 75 dB	Difficult
above 75 dB	Impossible

Annoyance Factors

Perhaps the most widespread reaction to noise is that of annoyance. Some characteristics of sound seem to be more annoying than others. These characteristics are as follows:

(1) loudness - the more intense and consequently louder noises are considered more annoying
(2) pitch - a high pitch noise, that is, one containing predominantly frequencies above 1500 cps is more annoying than a low pitch noise of equal loudness
(3) intermittency and irregularity - a sound that occurs randomly or is varying in intensity or frequency

is believed more annoying than one which is continuous and unchanging.

(4) localization - a sound which repeatedly seems to change in its location relative to the listener is less preferred than one which remains stationary.

A measure of noise intended to describe its annoyance value has recently been developed and found to be highly correlated with observers' ratings of the acceptability of flyover noise produced by various types of aircraft. This measure, called perceived noise level in dB, is derived from calculations based upon the octave band intensity levels of the noise in question together with data showing equal annoyance ratings for different octave bands of noise. While of some value, perceived noise levels and other annoyance measures based upon single judgments of the noise stimulus, are expected to have only limited usefulness in gauging the complaint potential of a noise. This is due to the many non-acoustical considerations which enter into such judgments. Some of these factors are cited below with examples to illustrate each of them:

Factor	Example
1. The sound has unpleasant associations.	The annoyance caused by the intrusion of aircraft noise into communities around airports is based, in part, upon the residents being fearful of the planes crashing into their homes.
2. The sound is inappropriate to the activity at hand.	Music tolerated during waking hours may be annoying during the hours of sleep.
3. The sound is unnecessary.	People may complain of the noise made by the neighbor's pets but not by the delivery trucks in the same neighborhood.
4. The sound has an advantage associated with it.	The comforts derived from air conditioning outweighs the noise of such units. Similarly, the economic value of nearby plants to a community may balance out the noise produced by the plants; annoyance due to military aircraft noise may be offset by the assurance against surprise attack by an enemy.
5. Individual tolerance to noise.	Some individuals complain about all kinds of noise as well as other types of nuisances.

* * * * * * * * *

Several models for predicting the complaint potential of a community noise now exist which take into account acoustical as well as the non-acoustical considerations outlined above. The accuracy of the predictions made by these models, however, is yet to be determined.

Noise and Performance

The effects of excessive noise on efficiency and work output seem to be slighter than is often thought. Performance on tasks involving simple repetitive operations, does not appear to be affected by noise, while apparent losses in efficiency on most complex tasks tend to become dissipated with increased exposure time. The relationship between excessive noise in a work situation and accident rate, absenteeism, and rate of employee turnover has not as yet been clearly determined.

References

1. _____: Industrial Noise Manual. American Industrial Hygiene Assn., 14125 Prevost, Detroit 27, Mich., 1958.

2. Glorig, Aram: Noise and Your Ear. Grune and Stratton, New York, N. Y., 1958.

3. _____: Guide for Conservation of Hearing in Noise. Subcommittee on Noise in Industry, American Academy of Ophthalmology and Otolaryngology, 111 N. Bonnie Brae, Los Angeles 26, Calif., 1957.

4. Exploratory Subcommittee Z24-X-2 of the American Standards Assn.: The Relations of Hearing Loss to Noise Exposure, American Standards Assn., Inc., 10 East 40th, New York 16, N. Y., 1954.

5. Kryter, K. D.: The Effects of Noise on Man. Monograph Supplement 1, Journal of Speech and Hearing Disorders, 1950.

6. Glorig, A.; Wheeler, D. E.; Quiggle, R.; Grings, W. W.; and Summerfield, A.: Wisconsin State Fair Hearing Survey: Statistical Treatment of Clinical and Audiometric Data. American Academy of Ophthalmology and Otolaryngology, 111 N. Bonnie Brae, Los Angeles 26, Calif., 1957.

7. Noise - Causes, Effects, Measurement, Costs, Control. Proceedings, Inservice Training Course on "The Acoustical Spectrum - Sound, Wanted and Unwanted." University of Michigan, Ann Arbor, Mich., 1952.

8. Broadbest, D. E.: Perception and Communication. Pergamon Press, New York, N. Y., 1958.

9. Schneider, E. J.; Peterson, J. E.; Hoyle, H. R.; Ode, E. H.; and Holder, B. B.: Correlation of Industrial noise exposures with audiometric findings. Am. Ind. Hyg. Assoc. J. 22: 245, (1961).

10. Ward, W. D.; Glorig, A.; and Sklar, D. L.: Temporary threshold shift from octave-band noise; applications to damage risk criteria. J. Acoust. Soc. Amer. 31: 522, (1959).

11. Yaffee, C. D. and Jones, H. H.: Noise and Hearing Relationship of Industrial Noise to Hearing Acuity in Controlled Population. U. S. Public Health Publication No. 850, Washington, D. C., 1961.

12. Corso, J. F.: Age and sex differences in pure tone thresholds from 18 to 65 years of age. Research Bulletin No. 25, Department of Psychology, Pennsylvania State University, University Park, Pa., 1962.

13. Riley, E. C.; Sterner, J. H.; Fassett, D. W.; and Sutton, W. L.: Ten years experience with industrial audiometry. Am. Ind. Hyg. Assoc. J. 22: 151-159, (1961).

14. Hinchcliffe, R.: Threshold of hearing as function of age. Acoustica 9: 303-308, (1959).

15. Beranek, L. L.; Kryter, K. D.; and Miller, L. N.: Reaction of people to exterior aircraft noise. Noise Control, 5: 23, (Sept. 1959).

16. Eldredge, D. H.: The problems of criteria for noise exposure. Armed Forces-National Research Council Committee on Hearing and Bio-Acoustics (CHABA), 1960.

17. Kryter, K. D.: The meaning and measurement of perceived noise level. Noise Control, 6: 12, (Sept.-Oct. 1960).

PHYSIOLOGY OF HEARING

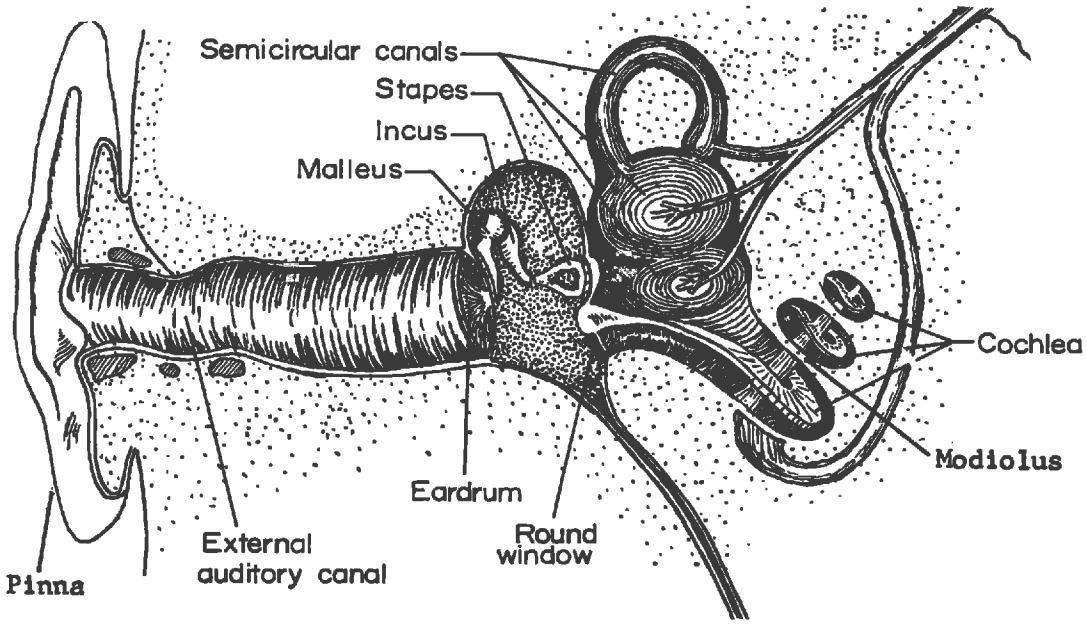


Figure 1.-Drawing of human ear showing three subdivisions in cross section.

Major interest in this section will be given to the structure and function of the ear as regards the hearing process and its impairment due to excessive noise exposure. The brain mechanisms underlying the hearing process will be largely ignored since noise-induced hearing loss seems to be primarily a peripheral as opposed to a central-type of disorder.

Ear Anatomy and Physiology

Anatomically, the ear can be divided into three subdivisions called the external, middle and inner ears (see Figure 1). The external and middle ear functions are principally to collect and transmit sound stimuli to the inner ear where the sensory receptors for sound sensation are located. The pinna of the external ear funnels sound inward through the external ear canal to the tympanic membrane or eardrum. The incoming sound waves strike the eardrum and set it into vibration. Behind the eardrum is the middle ear, an air-filled cavity containing three small bones or ossicles referred to as the malleus, incus and stapes. The handle of the malleus is attached to the eardrum and articulates with the incus which, in turn, is joined to the stapes. The footplate of the stapes fits snugly in the oval window which is one of the two covered openings between the middle and inner ears. The other opening lies just below the oval window and is called the round window. Functionally, the three ossicles form a chain which carry the sound-produced vibrations of the eardrum through the middle ear to the inner ear.

Also located in the middle ear are the intra-aural ear muscles consisting of the tensor tympani and the stapedius. The tensor tympani is attached to the malleus and, when contracted, places the eardrum under greater tension. The stapedius muscle is fastened to the stapes and upon contraction pulls this bone in a downward and outward direction therein affecting the movement of the stapes footplate in the oval window.

Prepared by: Alexander Cohen, Ph.D.

The contractile state of these muscles can be produced by the onset of intense sound and, in effect, reduces the amount of sound energy that is conducted by the ossicular chain to the inner ear. This action is considered protective in nature since it minimizes the potentially damaging effects of high intensity sounds on the hearing receptors in the inner ear.

Another middle ear feature is the Eustachian tube, a passageway leading from the middle ear cavity to the back of the nose and throat. The purpose of the tube is to equalize air pressure on both sides of the eardrum. Equalization in pressure is necessary to make the eardrum more capable of responding to and transmitting the sounds impinging upon it.

Just behind the oval and round windows is the inner ear which consists of three sections known as the vestibule, semicircular canals, and cochlea. Of these, the cochlea is the most important for hearing. The human cochlea is essentially a triple canal coiled up spirally around a bony axis, the modiolus, which is channeled to form the pathway for the auditory branch of the VIIIth cranial nerve. The larger turns of the spiral are at the base of the cochlea, the smaller turns are at the apex. The three canals comprising the cochlea are formed by two partitions (see Figure 2). One partition is composed of a ledge of bone, the spiral lamina, which winds around the modiolus like the thread of a screw and the basilar membrane which extends from the projecting tip of the spiral lamina to the outer wall of the cochlea. The second partition is formed by Reissner's membrane which stretches from the upper surface of the spiral limina to a point on the outer wall of the cochlea, a short distance above the attachment of the basilar membrane. The canal, which lies below the basilar membrane, is called the tympanic canal (scala tympani); the canal enclosed between the basilar membrane and Reissner's membrane is called the cochlear duct (ductus cochlearis), and the canal lying above Reissner's membrane is referred to as the vestibular canal (scala vestibuli). The vestibular and tympanic canals contain perilymph fluid and communicate with one another through a tiny opening at the apex of the cochlea. The base of the vestibular canal is sealed by the oval window into which the footplate of the stapes is lodged. The lower termination of the tympanic canal is sealed by the membrane-covered round window. The cochlear duct contains endolymph fluid and also the tectorial membrane which is attached at one end to the spiral lamina with the other end floating freely in the endolymph just above the basilar membrane.

The basilar membrane is narrowest at the base of the cochlea (i.e., near the oval window) and becomes progressively wider as it extends towards the apex. This is in contradistinction to the dimensions of the total cochlear structure which becomes smaller as the apex is approached. Distributed in four rows (one inner row, three outer rows) along the entire length of the basilar membrane are hair cells which project upward toward the underside of the tectorial membrane. These hair cells are the sensory receptors for hearing which together with supporting cells constitute the Organ of Corti, the auditory sense organ. The hair cells are innervated by nerve fibers which pass through small openings in the spiral lamina and enter the nodiolus of the cochlea where their cell bodies are grouped to form the spiral ganglion. Axons from this ganglion collect at the base of the cochlea and pass out from the bottom of the coil as the auditory branch of the VIII cranial nerve. These axons end upon nerves in the medulla, which, in turn, pass axons to different and higher nerve centers in the thalamus and cerebellum. Axons from these centers finally lead to the temporal lobe of the cerebral cortex.

Although knowledge is still lacking, cochlear function underlying the hearing process has become clearer in recent

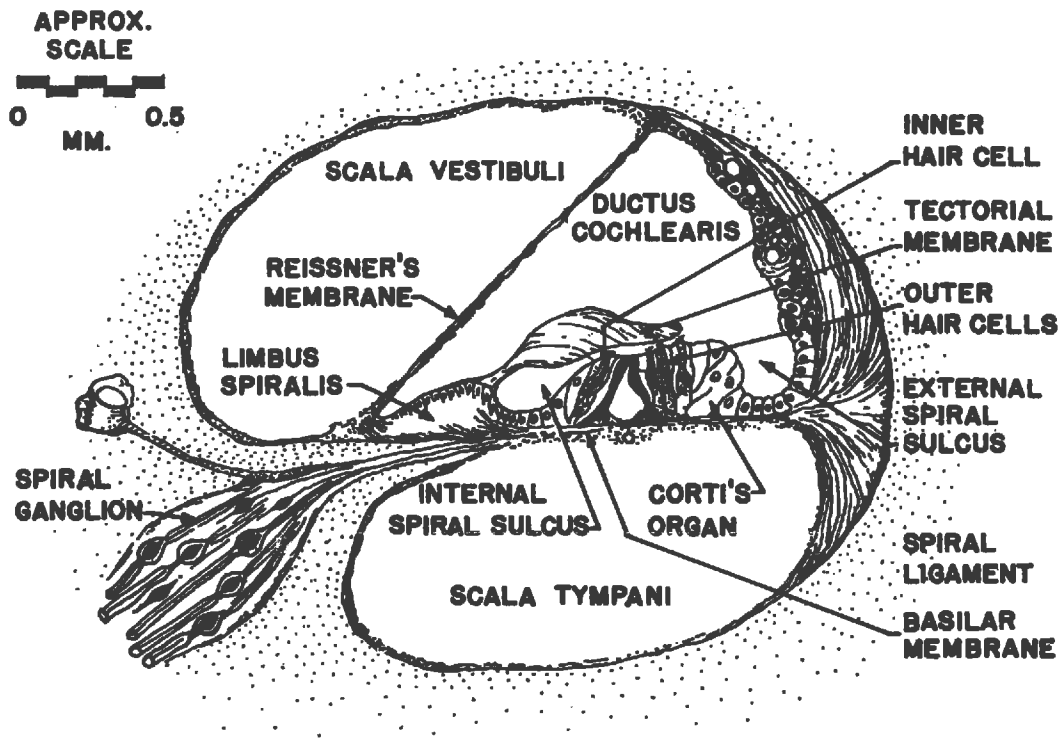


Figure 2.-Enlarged cross-section drawing of the cochlea.

years. As a physical system the cochlea has been described as an enclosed column of fluid bounded at one end by the stapes footplate in the round window and at the other end by the membrane covering the round window. Since fluid is incompressible, the vibrations transmitted by the stapes footplate, similar to a plunger action, cause mass movements of fluids in the cochlear canals. In this process every inward movement of the stapes footplate causes the round window to bulge outward, and every stapes outward movement causes the round window to move inward. The fluid movements in the cochlear canals cause structural displacements along the basilar membrane which take the form of a travelling wave whose crest reaches a peak at different points along the membrane depending upon the frequency of sound stimulation. Generally, the locus of the crest or peak displacement for high frequency sounds is in the basal section of the basilar membrane, the peaks for lower frequencies becoming progressively shifted toward the apex of the basilar membrane.

As a result of the wavelike motions of the basilar membrane, a shearing action develops between the basilar membrane and the tectorial membrane leading to bending and squeezing of the hair cells found on the upper surface of the basilar membrane. This hair cell deformation causes electrical and/or chemical changes which are believed to trigger nerve impulses in the nerve fibers associated with the hair cells. The pattern of neural impulses arising within the cochlea provides the basis for auditory sensation and is transmitted to the cerebral cortex for interpretation.

Hearing Loss from Noise Exposure

Deafness may be classified into three basic types, namely, conductive, perceptive (neural) and functional. Conductive hearing loss is caused by a disorder in the external and/or middle ear which prevents the normal amount of sound energy from reaching the inner ear. This pathology may vary from excessive wax being formed in the external

ear canal to a bony sclerosis or hardening of tissue around the footplate of the stapes in the oval window (called otosclerosis). Most conductive-type impairments are amenable to treatment and can be corrected. Perceptive deafness refers to disorders in the inner ear and/or along the VIII cranial nerve. Such pathology may range from disturbance in the cochlear fluids to degeneration of the hair cell receptors and nerve supply. These types of loss are not capable of being restored through surgical or other medical means.

Functional deafness is applied to a hearing loss that has no organic basis. In other words, the individual does not fully utilize his hearing capacity despite the fact that there is no actual damage to his hearing mechanism.

Hearing loss from noise exposure can be either conductive or neural in nature. Occasionally, it may even be a combination of the two. Noise-induced hearing loss of a conductive type, termed acoustic or blast trauma, can result from an explosion which may rupture the eardrum. The inner ear is infrequently damaged in such instances, but the ossicular chain may be dislodged. The perceptive type of noise-induced hearing loss results from prolonged exposure to excessive amounts of noise such as may be found in industry. The eardrum or ossicular chain is rarely affected in these cases, the site of this disorder is in the cochlea. Initial exposure to excessive industrial-type noise produces a temporary loss in hearing which is recovered after a short time away from noise. With repeated or prolonged exposure for months or years, the likelihood of the ear recovering all of its temporary noise-induced loss is diminished. The residual or non-recovered part of the loss constitutes a permanent hearing impairment due to noise.

The mechanism responsible for deafness from noise exposure remains to be more fully determined. That part of the inner ear which seems first affected by intense noise is the outer rows of hair cells close to the basal end of the basilar membrane. This causes more loss in hearing for high frequency sounds than for low frequency sounds. As

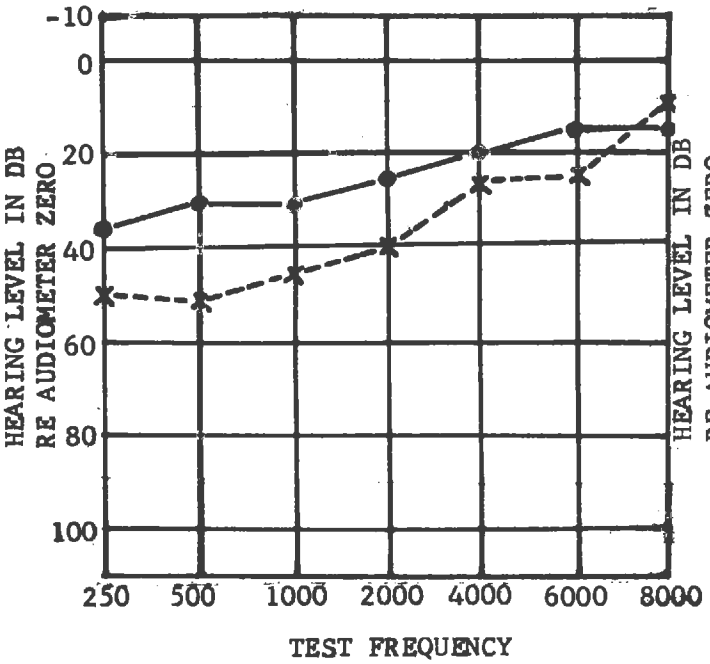


Figure 3.-Audiogram showing conductive-type hearing impairment.

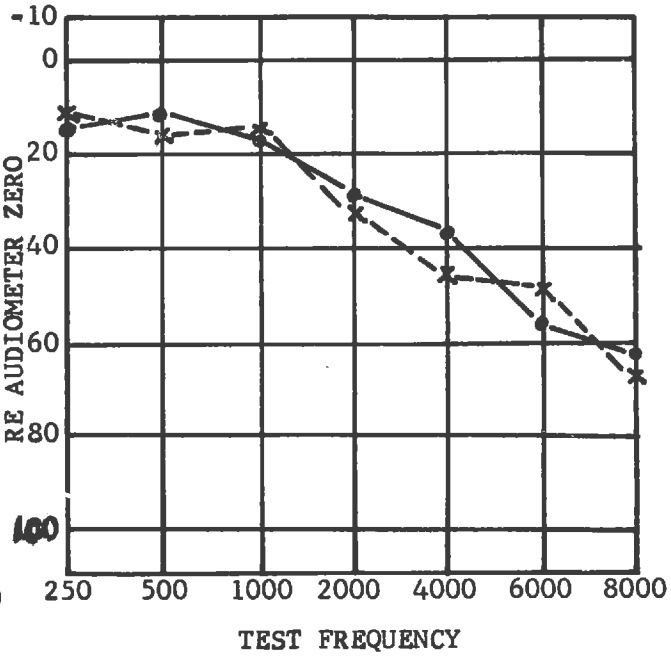


Figure 4.-Audiogram showing perceptive-type hearing impairment.

already mentioned perceptive deafness is not curable. Noise-induced losses of the perceptive-type can be prevented, however, and all feasible efforts must be taken to insure such prevention.

Audiometry

Hearing losses can be measured by a pure tone audiometer which describes threshold intensity levels for hearing different frequency sounds relative to intensity values representing the average hearing of a normal listener population. (Note: these reference settings are not to be confused with the .0002 microbar reference for sound pressure level measurements. Actually, the reference values for normal hearing on the audiometer are different for different test frequencies). Pure-tone air conduction audiometry is most commonly used in hearing testing although bone conduction and other more elaborate tests (e.g., noise-masked thresholds or pure tone discrimination) are also used for purposes of diagnosing a given hearing disorder.

Figures 3 and 4 show typical audiograms for ears with conductive and perceptive hearing losses, respectively. Note that in the conductive case (Figure 3) the losses for low frequency sounds are greater than those for the higher frequencies. In contrast, the perceptive hearing disorder (Figure 4) shows relatively greater losses at the higher frequencies. Figure 5 describes the development of permanent noise-induced hearing loss as a function of exposure time to excessive industrial noise. Early in the development, the losses are most prominent for frequencies of 3000 to 6000 cps and show a peak at 4000 cps. With continued exposure significant losses appear at other neighboring frequencies as well as increases still further in the 3000-6000 cps range.

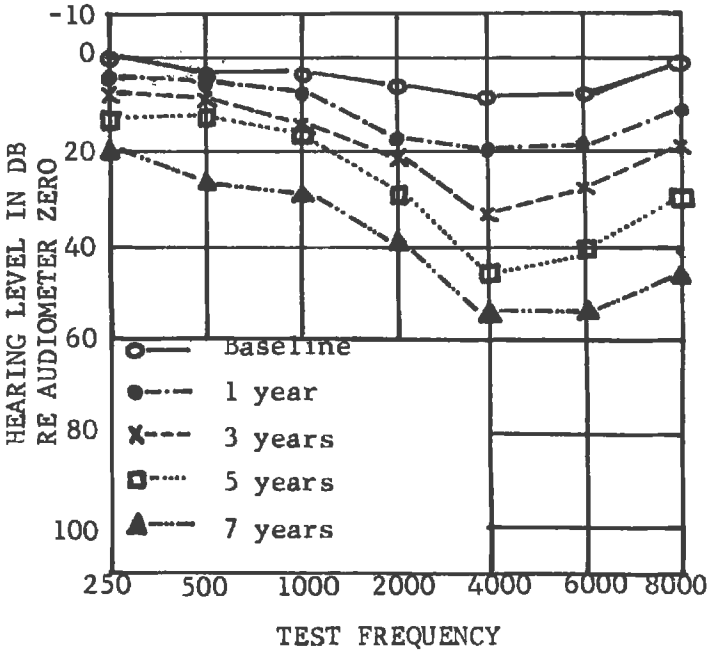


Figure 5.-Hearing loss from noise as a function of exposure time.

Deafness due to aging (presbycousis) also shows losses in the audiogram at those frequencies severely affected by noise exposure. This raises the problem of how much of a given hearing loss at a test frequency is due to noise and how much is due to age. Hearing data acquired from noise-free populations of listeners, classified by age, are presently being used to estimate the extent of hearing loss due to the age factor only. Such losses are deducted from audiometric measurement on noise-exposed individuals, leaving a value which is believed to more accurately reflect the extent of the noise-induced loss.

References

1. _____: Industrial Noise Manual. Amer. Ind. Hyg. Assoc., Detroit, Mich., Chapters 6, 7, April 1958.

2. Davis, H. (Editor). Noise and Your Ear - A Guide for Laymen. Murray Hill Books, Inc., New York, N. Y., Chapters 3, 6, 1947.

3. Glorig, Aram. Noise and Your Ear. Grune & Stratton Press. New York, N. Y., 1958.

4. Sataloff, J. (Editor). Industrial Deafness. McGraw-Hill Book Company, Inc., New York, N. Y., Chapters 2,3, 1957.

NOISE MEASUREMENT

Introduction

The industrial hygienist is being called on more frequently to make noise measurements and to evaluate noise problems. These may be problems of potential hearing loss of employees due to excessive noise exposures, annoyance from a noise in a building or community, compliance with noise specifications, or requirements for a low noise background for audiometric testing. In any of these situations the industrial hygienist will need to determine:

(1) total sound pressure level,
(2) distribution of this sound pressure or energy with frequency, and
(3) distribution of this sound pressure or energy with time.

The type of noise source will dictate which equipment is necessary in order to make an accurate evaluation of these three factors.

Types of Equipment

At present there is a wide assortment of equipment available for noise measurements. The choice of equipment used will depend on the type of noise encountered and the purpose of the measurements. Specific types of equipment will be discussed, including the general features of each type. In most cases commercially available instruments will incorporate all of these features but it should be pointed out that the manufacturer's instructions for the specific instrument being used should be consulted freely. It has been the author's experience that manufacturers modify equipment electronically, which results in changes in operating procedures even though the exterior appearance of the instrument remains unchanged. Therefore, it is essential that the instruction book be used, which was obtained with the instrument.

Prepared by: Herbert H. Jones

Sound Survey Meter

The sound survey meter is a small instrument that usually consists of a nondirectional microphone, an adjustable calibrated attenuator, an amplifier with three weighting networks, and an indicating meter.

The instrument has a function switch with an off position, A, B, and C weighting network positions, and a battery check position. It has either a continuously variable or stepped attenuator. The sound pressure level is the sum of the attenuator setting and the reading from the indicating meter.

Even though the sound survey meter does not meet American Standards Association specifications for sound level meters it is a good instrument for rapid screening and surveying of a large area or number of locations.

Sound Level Meter

The sound level meter is the basic instrument used for noise measurements. It comprises a microphone and an electronic circuit including an attenuator, an amplifier, three frequency response networks (weighting networks), and an indicating meter. The attenuator in the circuit controls the current within limits that can be handled by the indicating meter, which is calibrated in decibels. The value obtained is the root-mean-square sound-pressure level expressed in decibels re 0.0002 dynes per sq. cm.

Sound level meters are available in the range of 20 to 180 decibels (dB) (re 0.0002 dynes/sq. cm.). The newer sound level meters provide an amplifier with a flat frequency response from 20 to

20,000 cycles per second (cps), and the overall response depends on the microphone used. The design criteria for sound level meters (American Standards Association, 24.3 - 1944 and S1.4 - 1961) permit large tolerances at both low and high frequencies. This emphasizes the need for the proper calibration of each instrument.

Three weighting networks, A, B, and C are included. The purpose of these is to give a number which is an appropriate evaluation of the total loudness level. Human response to sound varies with its frequency and intensity. The ear is most sensitive to the low and high frequencies at low sound intensities. On the other hand, at high sound levels there is less difference in human response at the various frequencies. The three weighting networks also provide a means of compensating for these variations in human response.

The A network is less sensitive to low frequencies and is intended for use at sound pressure levels below 55 dB. The B network is an intermediate step for the range of from 55 to 85 dB. The C network has a flat response and is used for everything above 85 dB. The C network is the only one which indicates the actual sound pressure level, since the sound pressure level is based on a flat response.

Microphones

The microphone is by far the most important part of the equipment, since the quality of the final result can be no better than the signal produced by the microphone. The function of the microphone is to pick up the sound energy and convert it to electrical energy, which is fed to the electronic circuit.

The frequency response, sensitivity, directionality, and range of a sound level meter are primarily determined by the microphone; therefore, its importance cannot be overemphasized.

Three major types of microphones are in general use for the measurement of sound. Each type has advantages and disadvantages, and final selection depends on the requirements of the job to be done.

Rochelle salt crystal microphone. The Rochelle salt crystal microphone was supplied as standard equipment on sound-level meters for many years. The reasons for this were its low cost, ruggedness, availability, and high sensitivity. It has a frequency range of 20 to 8000 cps and an intensity range of 24 to 150 dB. Its frequency response varies widely with the incidence of the sound and the response is far from flat above 2000 cps.

One of the disadvantages of the Rochelle salt crystal microphone is its sensitivity to heat and humidity. Temperatures above 115°F may damage the crystal and temperatures above 125°F will destroy it. Also, there is a reversible change in sensitivity produced by temperature change. When an extension cable is used and the input impedance of the meter is high, a temperature correction is necessary.

Ceramic crystal microphone. Since 1963 a PZT (lead titanate-lead zirconate) piezo-electric - ceramic diaphragm type microphone has been regularly supplied with many of the sound level meters. This is a stable and rugged microphone with a smooth frequency response and it is relatively unaffected by normal temperature and humidity changes. It has a frequency range of 20 to 9000 cps and an intensity range of 24 to 150 dB. It will withstand temperatures of 22 to 205°F without damage. A correction is necessary when the PZT microphone is used with an extension cable. A correction of about 7 dB is made when a 25 foot cable is used between the microphone and the instrument.

Dynamic microphone. This is probably the second most commonly used type. It operates on the principle of a coil

moving in a magnetic field, resulting in an induced voltage. This type of microphone has a dynamic range of 20 to 140 dB and a frequency response in the range 40 to 10,000 cps ±5 dB. Its frequency response is less erratic than the crystal microphone; but the frequency response drops off sharply below 40 cps.

The dynamic microphone has the advantage of low self-noise, permitting noise measurements down to less than 20 dB in an octave band. Sensitivity to temperature change is small up to 180°F, and it can be used with long extension cables without correction. This microphone cannot be used in strong magnetic fields such as those encountered around large transformers, generators, or electric arc welders. Care must be taken to avoid getting the microphone wet because of the possibility of internal shorts.

Condenser microphone. This is the least commonly used of the four microphones. It consists of two plates which act as a condenser; one is a thin diaphragm, the other a solid backing. Movement of the diaphragm changes the capacitance of the condenser in proportion to the displacement of the diaphragm. This change in capacitance produces an electrical signal which is proportional to the sound pressure level.

This microphone has the best frequency response of all types in current use. It is available in models which are essentially flat in response to frequencies from 20 to 8,000 and ±3 dB at 15,000 cps to models which are essentially flat from 20 to 40,000 cps. The dynamic range is from 40 to 145 dB in the standard models with special models available with an upper limit of 200 dB. Its temperature sensitivity corresponds to that of a dynamic microphone. It can be used at higher temperatures and with longer cables without corrections.

Disadvantages are its high cost, high self-noise, limited humidity range, and additional electronic complexity due to the preamplifier which is required. High humidities may increase internal noise but this can be corrected by keeping the microphone in a dessicator when not in use.

Noise Analyzers

When the sound to be measured is complex, consisting of a number of tones, or having a continuous spectrum, the single value obtained from a sound level meter reading often is not sufficient for analytical purposes. It may be necessary to determine the sound pressure distribution according to frequency.

In general, there are three types of noise analyzers:

(1) octave, half octave, and third octave bandwidth,
(2) constant bandwidth, and
(3) constant-percentage narrow band.

Octave band analyzer. The most practical and widely used analyzer for industrial noise studies is the octave band analyzer. As indicated by the name, the upper cutoff frequency is twice the lower cutoff frequency. For a half-octave analyzer the upper cutoff frequency is $\sqrt{2}$ times the lower, and for a third-octave analyzer the ratio is $\sqrt[3]{2}$. A commonly used set of octave pass bands in commercial equipment is 20 to 75, 75 to 150, 150 to 300, 300 to 600, 600 to 1200, 1200 to 2400, 2400 to 4800, and in excess of 4800 cycles. This analyzer provides sufficient data on industrial noise to evaluate its physiologic significance and to provide a basis for noise control. The small number of bands makes it possible to perform an analysis in a reasonable length of time, and the bands are wide enough to reduce the problem of transient components affecting stability of the meter.

This analyzer may be equipped with one of two types of filters. In one type there is a choice of eight pass-bands and the selection is made by a single switch. The second type consists of a

low cutoff filter, which filters out all frequencies below the setting of the dial; and a high cutoff filter, which filters out all frequencies aobve the setting of this dial. Measurements can be made in bands of one octave or multiples of an octave to cover the complete spectrum using the second type of analyzer. The attenuator, which is 50 dB in 10 dB steps, and the amplifier serve the same purpose as in the sound level meter. The meter has a 16 dB range. The sound pressure level within the chosen band is the sum of the sound level meter attenuator setting, the analyzer attenuator setting, and the analyzer meter reading. The analyzer has a rheostat for adjusting the indicator to correspond to the meter reading on the sound level instrument when the analyzer is set to the overall position.

All octave band analyzers are equipped with an output jack permitting the use of headphones, a recorder, or an oscilloscope.

At the present time there are being introduced some octave band analyzers using the American Standards Association's preferred frequencies. In these analyzers the octave bands are centered around the following mid frequencies: 63, 125, 250, 500, 1000, 2000, 4000 and 8000 cycles per second. Although there is a shift in octave bands utilized on the standard analyzers, there is no practical difference in the interpretation of data of industrial hygiene significance.

Constant bandwidth. This instrument has a fixed bandwidth that is a specific number of cycles wide. Common bandwidths range between 5 and 200 cps. These are essentially laboratory instruments requiring line voltage for operation. Because of the transient nature of industrial noise and the narrow bandwidth of the analyzer, this instrument is seldom used in industrial noise studies.

Constant-percentage bandwidth. The constant-percentage narrow band analyzer has a bandwidth which is a fixed percentage of the mid-band frequency. Thus, at low frequencies the bandwidth will be only a few cycles, while at high frequencies the bandwidth will cover a wide range of frequencies. This instrument has essentially the same limitations as the constant bandwidth analyzer but it can be used in some noise control problems.

Accessories

A number of accessories are available for use with sound level meters and frequency analyzers to give additional information about the noise situation under study.

Impact noise analyzer. This instrument operates directly from the output of the sound level meter or frequency analyzer. It can be used to measure the peak level and duration of impact noise.

Through the use of electrical storage systems, three characteristics are measured by the analyzer for every impact noise. These are: the maximum instantaneous level, the average level, and a continuously indicating measure of peak sound levels. The values can be read individually on the same meter by means of a selector switch. A reset position of the selector switch restores the meter to its prereading condition.

Cathode-ray oscillograph. An oscillograph affords a useful means for observing the wave form of a noise and also for observing many general noise patterns, particularly short duration or impact noises. The oscillograph can be operated from the output signal of a sound level meter, octave band analyzer, or magnetic tape recorder. It can be used to measure the peak amplitude, the rate of decay, and the shape of a wave.

A cathode-ray oscillograph with a sweep rate extending down to at least two seconds and a long persistence screen is preferable for acoustic measurements. If the wave form is to be photographed

a short persistence screen should be used. A five-inch oscillograph tube is normally used for this latter purpose.

The output of the sound level meter, sound analyzer or magnetic tape recorder is connected to the vertical input of the oscillograph. The controls are set as explained in the instruction manual for the oscillograph. A reference signal of 60 cps, or 400 cps can be fed into the sound level meter to adjust the gain of the vertical amplifier. The reading on the meter is noted and the vertical gain is adjusted to obtain a peak-to-peak deflection according to the table below for a five-inch screen.

Meter Reading dB	Peak-to-Peak Deflection Inches
0	1
1	1.12
2	1.26
3	1.41
4	1.59
5	1.78
6	2.0
7	2.24
8	2.51
9	2.82
10	3.16

Once set, the gain control of the oscillograph should not be changed, adjustments being made with the attenuator on the sound level meter to keep the maximum deflection within the range of the table above.

The peak-to-peak sound-pressure level for a noise is the decibel value obtained from the table above, plus the sound level meter attenuator setting, plus 9 dB.

Magnetic tape recorder. The magnetic tape recorder can be used for recording noise in the field with subsequent analysis in the laboratory. Detailed and repeated studies can be made immediately or the tape can be kept for later analysis or for comparison purposes.

If meaningful measurements are to be made from a recording, a high quality recorder must be used. It should have a flat frequency characteristic over a wide range, low hum and low noise levels, low non-linear distortion, a constant speed drive, and it should be of good mechanical construction. A tape speed of 15 inches per second is recommended, since these characteristics are more readily obtained and maintained at high tape speeds.

The gain control should be set according to instructions supplied with the recorder. After it is set a reference signal should be recorded, such as the signal from an acoustical calibrator. Whenever the gain control is reset, a reference signal should again be recorded.

When an analysis is desired a representative sample of the tape should be selected and that portion of the tape is spliced to form a loop so it can be repeated continuously. If the absolute level needs to be known also, a sample of the recorded reference signal should be measured with the same control settings as were used for the original noise recordings.

It might appear that the only instrument needed in the field would be a good tape recorder; but in many situations it will be better to make the analysis in the field with the usual sound equipment to make certain that the desired data have been obtained.

Graphic level recorder. The graphic level recorder provides a permanent record of the noise characteristics by tracing a graph on a moving paper. The recorder can be operated from the output of the sound level meter or the octave band analyzer to record the level of a noise as a function of time. It is subject to the same limitations as the sound level meter in the analysis of noise of short duration.

When using the graphic level recorder it must be calibrated with an acoustical calibrator. The gain control should be adjusted according to instructions supplied by the manufacturer.

Acoustic calibrator. An overall acoustical check can be made of the sensitivity of a sound level meter, including its microphone by use of an acoustic malibrator. Calibration may be accomplished either by electronic or mechanical devices. The electronic calibrator consists of a small speaker mounted in an enclosure which fits over the microphone of the sound level meter. The enclosure is designed in such a way that the measurements can be repeated with a high degree of accuracy. Signals may be supplied to the calibrator from either an audio oscillator or random noise generator depending on which calibrator is being used. The designated operating voltage of the supplied signal and the sound level output can be found in the operating instructions furnished by the manufacturer when the calibrator is purchased.

Two types of mechanical acoustical calibrators are available commercially. One is a piston activated diaphragm mounted in an enclosure which is placed over the microphone. The other type consists of a diaphragm which is activated by steel balls. It is located a specified distance from the microphone to be calibrated. Each type of calibrator, when operated according to the manufacturer's instructions, will produce sound of a specified level.

Dosimeters. Three different types of noise dosimeters are available at present. These are designed to indicate in some manner the total noise dose during a specified time interval. Each of these uses a different approach; one measures the total amount of sound energy to which a worker is exposed for a workday, another one measures the amount of time that a specified decibel level is exceeded, and the third one measures the rate at which sound energy impinges on the exposed person for selected short-term periods of time. Correlation of these dosimeter readings with hearing loss is lacking. They cannot be used to evaluate damage risk to hearing from variable noise exposure situations at the present time.

Field Measurements

Evaluation of a noise problem depends upon accurate and meaningful measurements. Even though the equipment is electronic and may appear to be simple to operate, one should use it with caution and be on the alert to detect errors in the operation of the equipment and the influence of the environment.

In general, there are three types of noise studies and the equipment used will depend upon the type of study being made. The simplest of the three is a screening survey which can be done with a sound survey meter or sound level meter. This involves making a number of measurements in an area for rapid evaluation of the problem and determining where a more detailed study is needed.

The second type is a study to determine the characteristics of, and to evaluate the potential effects of a noise. This involves the use of a sound level meter and a noise analyzer in order to determine the sound pressure distribution with frequency. For industrial work, an octave-band analyzer would probably be used.

The third type of study is one for research or noise control purposes. This might require a large number of measurements and analyses in the field and also magnetic tape recordings to be analyzed in the laboratory. A sufficient number of samples should be taken to determine the exact nature of the source, including power and directionality.

Operation of Instruments

The sound level meter has an on-off

switch, a means of checking battery voltages, a weighting network switch, a fast-slow meter response switch, an attenuator switch with steps of 10 dB and an indicating meter (16 dB range). The instrument is turned on, batteries are checked, the instrument is permitted to warm up for a few minutes, and the attenuator is adjusted until the indicating meter is on scale. The sound pressure level is the sum of the attenuator setting plus the meter reading.

The octave-band analyzer operates from the output of the sound-level meter. The analyzer has an on-off switch, battery voltage check switch, a fast-slow meter response switch, an attenuator switch and a switch permitting the selection of the band to be measured. The analyzer is connected to the sound-level meter by a cable, both instruments are turned on. The batteries should be checked. The analyzer band selector switch is set at 20 to 20,000 cps and the two meter readings should agree. The overall sound pressure level is recorded, then the band selector switch is set to the 20 to 75 cps octave band, and the attenuator on the analyzer adjusted until the indicating meter is on scale. The sound level in the octave-band is the sum of the attenuator setting of the sound-level meter, the attenuator setting of the octave-band analyzer and the octave-band analyzer indicating meter observing the appropriate signs.

Maintenance of Equipment

Because of the nature of the equipment, one should constantly be on the lookout for faulty operation. The equipment should be checked before it is taken from the laboratory because of the availability of a quiet location and availability of other test instruments if repair is necessary. The following steps should be taken:

1. The instruments should be connected, turned on, and allowed to warm up for a few minutes.
2. Check batteries.
3. Make electrical and acoustical calibrations.
4. Make octave-band measurements of a convenient wide band steady noise.
5. Remove the microphone and make electrical background measurements.
6. Recheck calibration.

If any of the above steps yield unsatisfactory results the instrument instruction manual should be consulted for necessary adjustments or repairs. If the first three steps are completed satisfactorily, this usually means that the equipment is operating properly. Step 4 can expose a defective octave-band which might be overlooked in making measurements in the field. Step 5 will indicate whether there is an excessively noisy tube or faulty oscillating amplifier. This can be corrected by replacing the defective tube. Step 6 could indicate weak batteries if the calibration has changed by more than 1 dB. If it has changed, the batteries should be replaced before making a number of measurements.

Precautions for Handling Equipment in the Field

Most sound level meters available today come equipped with a Rochelle salt crystal microphone which can be damaged by excessive heat and humidity. The microphone should not be subjected to temperatures above 115°F; it should never be left in the trunk or inside a closed automobile on a hot summer day.

Since electronic equipment can be damaged by intense shock, extreme care must be taken in shipping or transporting these instruments by commercial carrier.

Calibration of Equipment in the Field

On reaching the area where the measurements are to be made the six steps listed under Maintenance of Equipment should be repeated, preferably before the equipment is taken into a noisy area. The acoustical calibration should be performed in an area where the

background noise, with the calibrator in place, is 10 dB less than the calibration signal.

The acoustical calibrator can be used also to determine the temperature corrections needed for an extension cable between the microphone and sound level meter. The reading should be taken without the cable and then with the cable inserted. The difference is the correction to be added to the readings obtained with the cable. This should be done after the microphone temperature has reached the room temperature where the measurements are to be made. This correction is a function of ambient temperature of the microphone, but not a function of frequency.

If a large number of readings are made at one time, the batteries should be checked every two hours and the equipment calibrated a minimum of four times a day.

Selection of Sampling Locations

After it has been determined that the equipment is operating satisfactorily, the next step is to determine the sampling locations. This will depend on the reasons for making the measurements and other factors. If it is a preliminary survey to determine if a noise problem exists, then a large number of measurements are taken with the sound survey meter or sound level meter throughout the area, paying particular attention to the noise sources.

In the evaluation of noise exposure to individuals the microphone would be placed as near to the individual as possible without interfering with his work. It is also best to make several measurements with the sound level meter in the area to determine if the sound pressure level is relatively uniform and if the measurements being made represent the individual's exposure.

Another type of survey is for noise control purposes. In this case, measurements would be made to determine the total acoustical output and directional characteristics of the source. Measurements at one point yield information about that point only and in that environment. In order to determine how a source differs in another environment, the acoustic power characteristics must be determined. A number of measurements must be made in a geometrical pattern about the source and at some distance from the source. This type of survey is basically an engineering problem and the measurements are made with particular attention to the physical characteristics of the source and environment and without regard to personnel in the area.

Sources of Error

The reliability of noise measurements depends upon the manner in which the instruments are used and their operating condition. The immediate recognition of faulty equipment is essential if accuracy is to be maintained.

Position of the microphone. In general, the location of the microphone is determined by the type of measurements to be made as was discussed under sampling locations.

The microphone should be located so that it is at the side of the observer or operator and not between the noise source and observer or operator. When making measurements around a single source large errors may result; but, in a large room with a number of sources, measurements are made at quite some distance from the source and the error would be small.

The sound field should be explored to make sure that the measurements are representative. The possible effects of obstacles upsetting the distribution of sound, particularly at high frequencies, should be kept in mind when making this exploration.

Wind will produce a low frequency noise which can be appreciable when using a

microphone that is very sensitive to low frequencies. If it is necessary to sample in the wind, a wind screen can be made out of wire, forming a sphere about 18 inches in diameter.

Circuit noise. All vacuum tubes are affected by mechanical vibration. The tubes used in sound measuring equipment have been selected to be less sensitive to shock than the usual tubes. But at high noise nevels some of these tubes may be vibrated to such an extent so as to produce extraneous noises in the equipment. A test for this is to disconnect the microphone from the sound level meter and observe whether there is a reading on the meter. If there is a reading, the meter can be placed on a rubber pad or rubber-tired cart which should eliminate the trouble. In very intense noise fields it may be necessary to remove the meter from the area and use an extension cable to the microphone.

When making measurements around electrical equipment a check should be made to see if there is an appreciable pick-up of the electromagnetic field by the sound measuring equipment. This check is particularly important when using a dynamic microphone. A pair of good quality earphones can be used to monitor the output of the sound level meter or octave band analyzer. The hum produced by the electromagnetic field will be of the same frequency as the power source, usually 60 cycles per second. The orientation of the instruments and microphone should be changed to determine if there is a difference in the meter reading; also, the microphone should be disconnected to check if the pickup is in the microphone or the instruments. If the pickup is in the instruments they can be removed from the area under study and an extension cable used; if it is caused by the dynamic microphone, a crystal or condenser microphone may have to be used.

When making noise measurements at low levels, the inherent circuit noise may be the limiting factor. A check for this is made by removing the microphone and substituting a capacitor of the proper size. The lowest reading that can be obtained with the sound level meter or analyzer is determined. This will be the lower limit of measurement.

Background. In some cases, when making measurements to determine the amount of noise generated by a particular source, it may be necessary to make corrections for background noise. If there is any evidence that the background noise is contributing to the noise being measured, a background measurement should be made with the noise source turned off. It is possible to make a rough correction for this background noise by applying the factors given in the table below:

Total noise level less background level (dB)	Subtract from the total noise level to get the noise level due to the source (dB)
10	0.5
9	0.6
8	0.7
7	1.0
6	1.2
5	1.6
4	2.2
3	3.0
2	4.3
1	6.9

Recording Data

One important part of making noise measurements is the recording of sufficient data. Data that should be recorded will depend on the purpose of the survey, that is, whether it is a hearing loss, noise control, or nuisance problem. To a certain extent each noise survey is unique, and the report resulting from it must have its own pattern that fits the needs of the particular situation. There are, however, certain basic data which are essential and must be recorded in almost all surveys:

1. Equipment used for the measurements
   - type and serial numbers of all

microphones, sound level meters and analyzers.

2. Corrections for measured values, such as cable, temperature, and acoustical calibration.
3. The time and date that measurements are made and name of person conducting the study.
4. Description of space in which the measurements were made such as, dimensions and nature of ceiling, walls and floor, and locations of windows and doors.
5. Description of the noise source under test (primary noise source). This should include a clear description of the machine as to size, name plate data, speed and power rating. Types of operations and operating conditions and number of machines in operation, locations of the machines and types of mountings.
6. Description of secondary noise sources including location and types of operations.
7. Noise control measures instituted, including the types and effectiveness of ear protectors.
8. Overall and band levels at each microphone position and the extent of meter fluctuation.
9. The meter speed and weighting network used.
10. Position of the microphone and the direction of the sound with respect to the microphone, tests for standing wave patterns and the decay of sound level with distance.
11. Time pattern of the noise, that is, whether continuous, intermittent or impact.
12. Personnel exposed, directly and indirectly.

In recording data it is helpful to have a blueprint or sketch of the building on which locations of measurements can be noted. Frequently, location of columns in a building can be used to form a grid system such as shown in Figure 1.

It is helpful to have a survey data sheet on which the data can be recorded. A typical one is shown in Figure 2 which was provided by the Subcommittee on Noise in Industry of the American Academy of Ophthalmology and Otolaryngology.

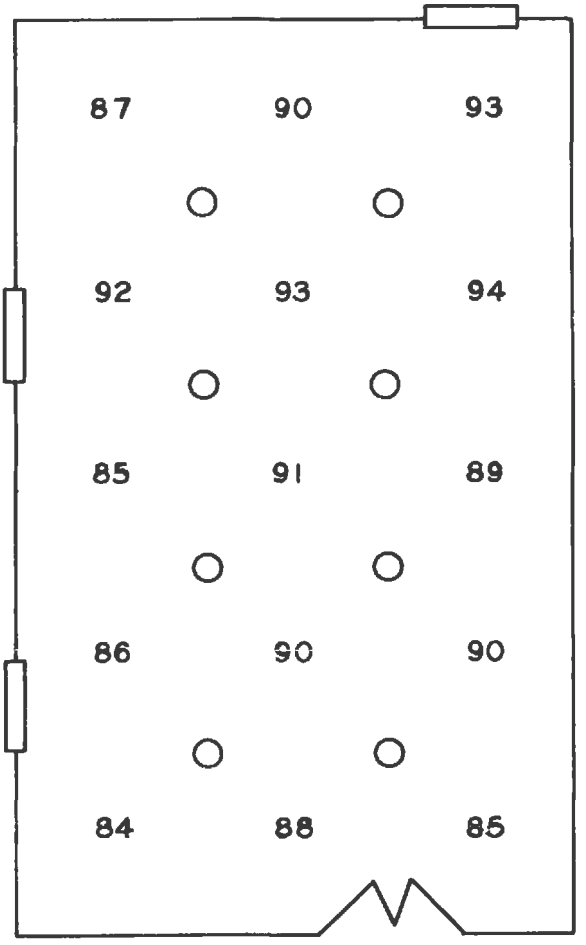


Figure 1.

Analysis of Data

After a survey has been completed and before a report can be prepared, it is necessary to analyze the data. The necessary corrections must be made and the accuracy of the final results determined.

Corrections should be made for any errors produced by an extension cable or any other errors revealed by the acoustical calibration. If a calibration curve is available for the microphone being used, the appropriate corrections should be

SOUND SURVEY

Page ___ of ___

Date: _____ Time: _____

Wind Velocity: _____ Wind Direction: _____

Sound-Level Meter: Type _____ Model _____ Serial No. _____

Microphone: Type _____ Cable Length _____

Analyzer: Type _____ Model _____ Serial No. _____

Other Equipment: _____

_____

_____

Location: _____ Sketch

_____

_____

_____

Calibrated: 60 cps: Acoustic: Corr. Factor:

Location	Weighting Network	Over-all Level	Octave-Band Pressure Levels Re. .0002 microbar							
			200–75	75–150	150–300	300–600	600–1200	1200–2400	2400–4800	Above 4800
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										

Remarks: _____

Recorded By: _____

Figure 2.

made. When measurements have been made to determine the sound pressure level of a particular source, corrections for background noise should be made if necessary.

With proper acoustical calibration the meter should be accurate within ± 1 dB at the calibrated frequency. With electrical calibration, the meter should be accurate within ± 2 dB at the middle frequencies. The average response curves for various microphones are probably within ± 1 dB in the middle frequencies and from ± 2 dB to ± 10 dB in the first and last octave bands. Also, with proper precautions, the readings should be accurate within ± 2 dB in all but the first and last octave bands.

Preparation of a Report

The type of report will depend on the purpose of the survey and for whom the report is prepared. In general, it is better to present the data in the same form that is used in the field, that is, showing overall and octave band levels. This may be plotted on ordinary linear graph paper with the dB as the ordinate scale and the octave band levels as the abscissa, or it may be plotted on special graph paper shown on the next page.

The criteria that apply to the situation should be presented and then the appropriate recommendations made.

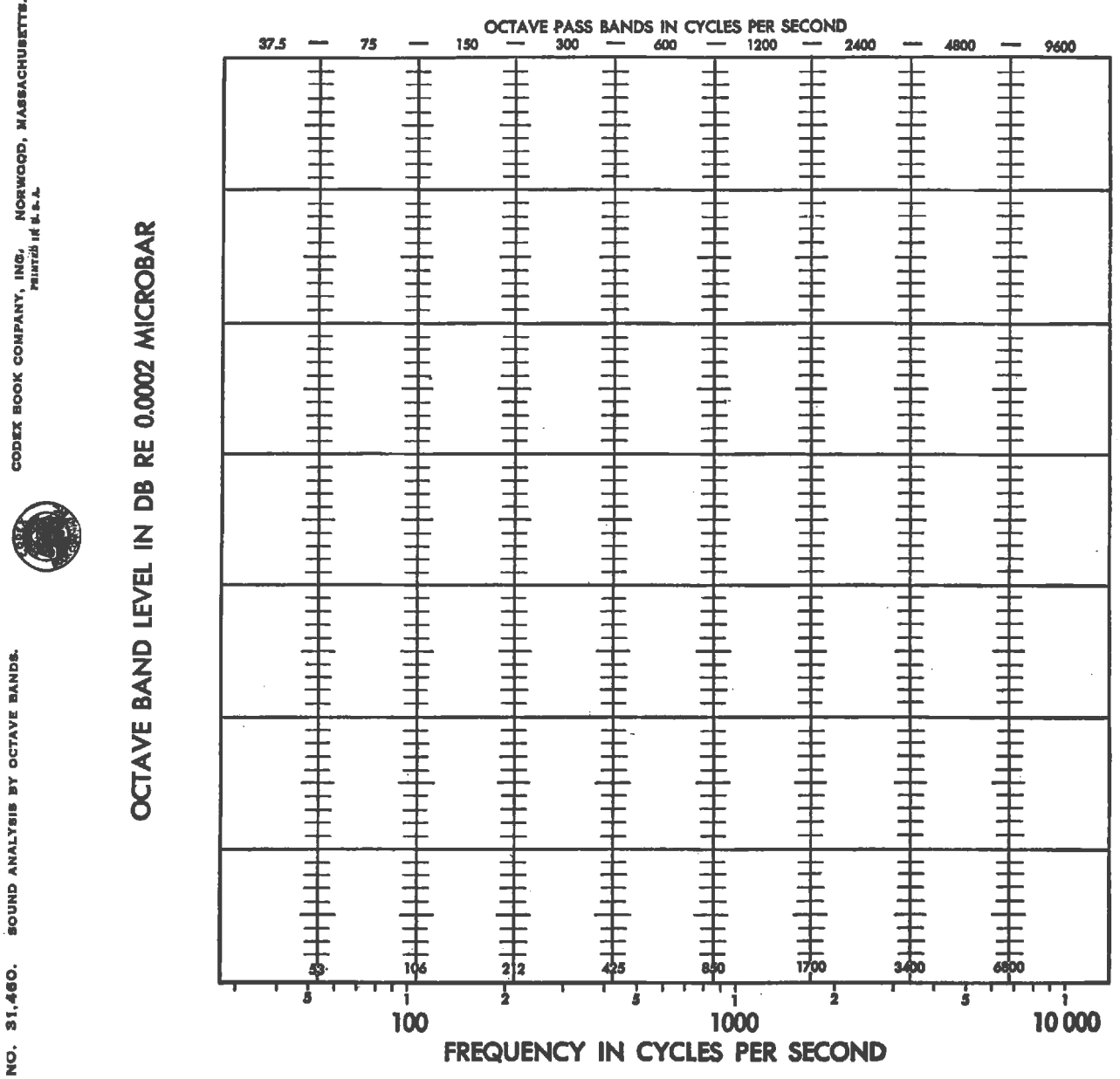
References

1. Peterson, A. P. G. and Gross, E. E., Jr. Handbook of Noise Measurement. General Radio Company. West Concord, Mass., 1963.

2. Harris, Cyril M. Handbook of Noise Control. McGraw-Hill Book Company, Inc. New York, N. Y., 1957.

3. Beranek, L. L. Acoustics. McGraw-Hill Book Company, Inc. New York, N. Y., 1954.

4. Industrial Noise Manual. American Industrial Hygiene Association. 14125 Prevost, Detroit, Mich. 1st Edition 1958, 2nd Edition, 1964.



(Reproduced by permission, the Codex Book Company, Norwood, Massachusetts).

ELECTROMAGNETIC SPECTRUM I

Introduction

There are several kinds of radiation propagated by electromagnetic energy which are included in the electromagnetic spectrum. Radiations from the portion of this spectrum that are of importance and interest to industrial hygienists are: visible light, ultraviolet and infrared radiations, microwaves, and ionizing radiations. Although it has become the custom to speak of ultraviolet and infrared as light there is only one narrow band of these waves in the entire spectrum that can be seen as visible light. All the rest are invisible. Radiations from electromagnetic sources differ in frequency, wave length, energy level, their visual response by the human eye, and their effects on the human body. Exposures to some of the radiations may be harmful to man depending upon the intensity and duration of the exposure. On the other hand, certain curative radiations can be derived from various electromagnetic sources. Ultraviolet radiant energy, visible radiation, and infrared radiation have been employed therapeutically.

Wave length is the one physical factor of these radiations that differentiates one from another. The unit of length adopted by physicists for measuring these extremely small wave lengths of the electromagnetic spectrum is the Angstrom (A) unit. Millimicrons (Mμ), centimeters (cm), and cycles per second (cps) are used also as units of measurement for these wave lengths. Figure 1 gives the types of radiation and the various range of wave lengths in centimeters and cycles per second. (One centimeter equals 10^{8} A; one A equals 0.1 Mμ equals 10^{-8} cm).

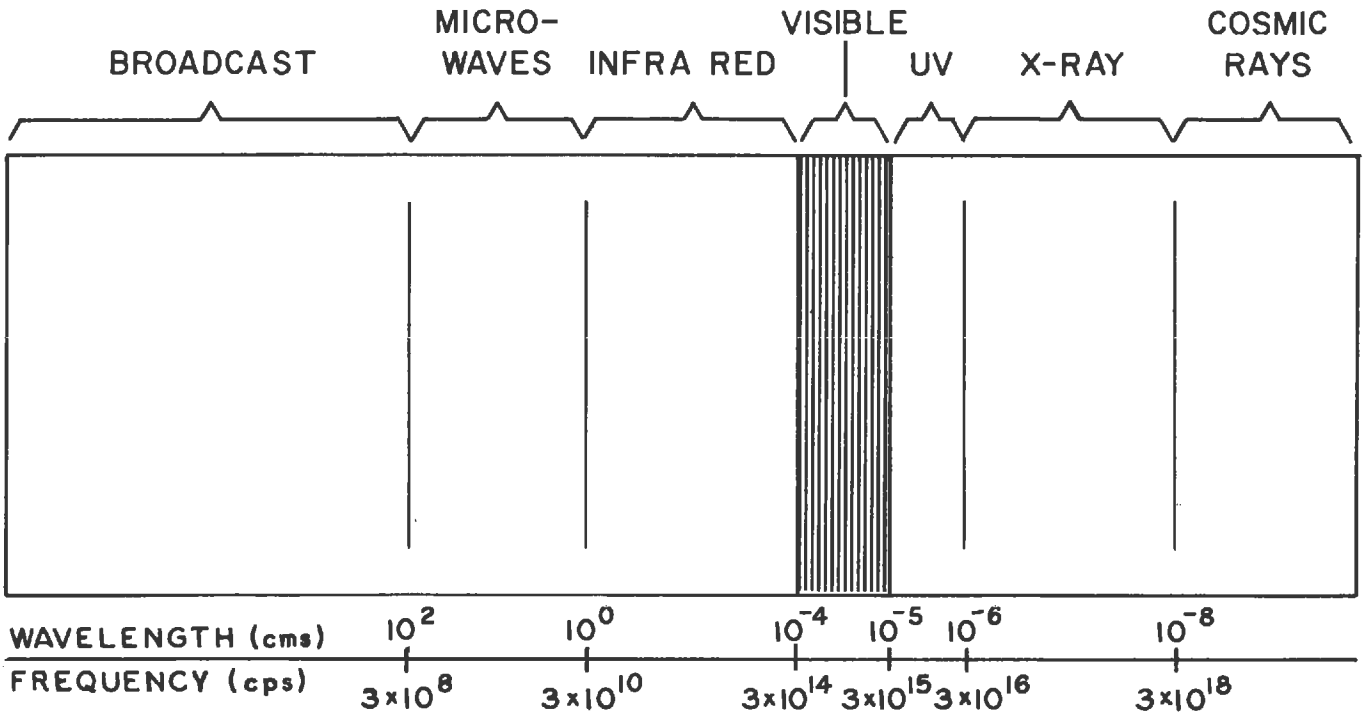


Figure 1. - Electromagnetic Spectrum

Prepared by: E. Elbridge Morrill, Jr.

Illumination in Industry

Illumination is a factor of primary importance in every industrial environment. The ever increasing development of new tools, intricate circuitry in electronics, and complex machines used in space technology require that good illumination be provided for these difficult, precision, and critical seeing tasks. Poor illumination in industry is reported to cause eye fatigue resulting in decreased production, more rejects of finished products, increased accidents, and has an effect on the morale of the worker. Poor housekeeping may result where there is inadequate illumination.

Good Illumination (Quantity and Quality)

The ultimate objective of good illumination is to provide adequate visibility for the task at hand and satisfactory eye comfort. Good illumination is not a means of "protecting the eyes." There is no causative relationship between illumination, either in quality or quantity, and the various sight stealing diseases[1]. Furthermore, although fluorescent lighting is occasionally suspected of possessing harmful qualities not found in other forms of artificial illumination or in daylight, it is not harmful to the eyes. It does not cause visual discomfort if properly installed, maintained and used[2].

There are four fundamental characteristics or factors that determine the quantity of illumination for a person to adequately and comfortably perform a specific task beside his good eyesight. They are: (1) Size of object being viewed, (2) time available to perform the task, (3) contract (brightness ratio) and (4) brightness level to which the object is illuminated[3].

There are many instances where the size, contrast and time are fixed. The only controllable factor is brightness. It might be mentioned that persons with poor eyesight require more illumination than those with average or normal eyesight.

In addition to the requirement for a sufficient quantity of light to permit the necessary details to be seen with ease, the quality of light is highly important in providing good seeing conditions. These quality factors include color, direction, brightness, brightness ratio (contrast), diffusion, uniformity of distribution, and glare. Glare is the least understood of these factors, but all of them have significant effects on visibility, and the ability to see easily, accurately, and quickly.

Glare (Direct and Reflected)

Glare may be defined as any brightness within the field of vision of such character as will cause discomfort, annoyance, interference with vision, or eye fatigue. When the condition is caused directly by the source of lighting, whether natural or artificial, it is described as direct glare[4]. Reflected glare is caused by the reflection of high brightness light sources from shiny surfaces such as ceilings, walls, desk tops, materials, critically machined metal surfaces, and highly polished surfaces within the visual field. Reglected glare is frequently more annoying than direct glare because it is so close to the line of vision that the eye cannot avoid it.

Reflected glare may be of two types: (1) It may occur within the task itself, or (2) within the area immediately surrounding the task[5]. This "reflected glare can be minimized and eliminated by using low brightness light sources or by orienting the work so that reflections are not directed toward the normal line of vision"[4]. Problems of reflected glare may be solved sometimes by checking the angular relationships between the luminaires, the task, and the eye. The location of the task or the luminaire may be of help because of the known fact that the angle of reflection will equal the angle of incidence.

Brightness Ratios

Brightness ratio is the ratio between the photometric brightness of any two relatively large areas in the visual field. The brightness within the environment should be carefully controlled. Although the greater the contrast the more readily the seeing task is performed, the eyes function most comfortably and efficiently when the brightness of the environment is relatively uniform. The recommended maximum brightness ratios[4] are shown below but lower values would be desirable.

5 to 1	Between tasks and adjacent surroundings
20 to 1	Between tasks and more remote surfaces
40 to 1	Between luminaires (or sky) and surfaces adjacent to them
80 to 1	Anywhere within the environment of the worker.

Industrial Lighting Applications

Industrial applications of lighting call for either the direct or semi-direct type of luminaires. Some light should be directed to illuminate the ceiling or upper structure to eliminate the "dungeon" effect. Top openings in luminaires will minimize dirt collection on the reflectors and lamps. Other considerations in selecting and installing luminaires are: (1) The outside of the fixture should be light colored to reduce brightness ratios between the outside of the luminaire and the inner reflector surface and light sources; (2) luminaires should be mounted high enough to raise them out of the normal field of view; and (3) the light source should be shielded with deep reflectors, cross baffles, or louvers. The latter is particularly important with the high wattage filaments or mercury sources and the higher output fluorescent lamps[4]. Supplementary lighting may be required when the object or task is small or of little contrast with adjacent area.

Levels of Illumination

Illumination in an industrial environment may be provided by luminaires (lighting fixtures), by natural light from windows, reflected daylight from walls, ceilings, floors and equipment, or a combination of these sources. The Illuminating Engineering Society has developed lighting recommendations for a wide variety of tasks encountered in industry. In Table 1, a few representative values are presented. Specific levels of illumination currently recommended for individual industrial tasks can be found in Figure 9-53, IES Lighting Handbook, Third Edition, published by Illuminating Engineering Society, 1860 Broadway, New York 23, New York, 1959.

Table 1. - Levels of Illumination Recommended for Typical Work Tasks*

Tasks	Foot-candles on Task**
Industrial Tasks	
Rough, easy assembly work	30
Rough bench and machine work, ordinary inspection	50
Medium bench and machine work, rough grinding, medium buffing and polishing, difficult inspection	100
Color inspection, grading and evaluation, making and finishing shoes, highly difficult inspection	200
Fine bench and machine work, medium grinding, fine buffing and polishing, sewing, very difficult inspection	500
Extra-fine bench and machine work, fine grinding, welding extra-fine assembly work, most difficult inspection	1000

Table 1. (continued)

Task	Foot-candles on Task**
Office Tasks	
Corridors, elevators, escalators, stairways	20
Reading high contrast or well printed material, tasks and areas not involving critical or prolonged seeing such as conferences, interviews, inactive files and washrooms	30
Regular office work, reading good reproductions, reading or transcribing hand writing in hard pencil or on poor paper, active filing, index references, mail sorting	100
Reading or transcribing hand writing in ink or medium pencil on good quality paper, intermittent filing	70
Accounting, auditing, tabulating, bookkeeping, business machine operation, reading poor reproductions, proof reading, rough layout drafting	150
Cartography, designing, detailed drafting	200

* Source: Ref. No. 6
** Minimum on task at any time

The Importance of Maintenance in Lighting

There should be a regularly scheduled system of maintaining the skylight, windows, lighting equipment, lamps, and room surfaces to keep them clean. Luminaires should be kept in proper adjustment, and in good repair. Even if this is done illumination levels will drop 25 to 35% less than the original under normal operating conditions. When illumination has decreased to two-thirds of its initial value, the lighting equipment should be washed. A thorough washing at least twice a year is recommended. In addition, the useful output of some lamps decreases with age long before they burn out. Therefore it is important that a regular maintenance program be set up whereby the lamps and fixtures are cleaned and dim and burned out lamps replaced at suitable intervals.

Instruments for the Measurement of Illumination(7)

Physics of Light

Light as defined by the Illuminating Engineering Society is "visually evaluated radiant energy." This energy with the proper wavelength makes visible anything from which it is emitted or reflected in sufficient quantity to activate the receptors of the eye. Several concepts of the nature of radiant energy have been expressed over the years but the modern idea is that of Einstein - the quantum theory. This theory, briefly stated, is that bundles of energy (photons) are emitted and absorbed in discrete quanta.

In making a lighting study or survey the following units of measurement are employed:

Illumination is measured in "foot-candles" = 1 lumen/square foot (one lumen = intensity of one standard candle).

Brightness is the density of light emitted by or reflected by a surface in a given direction and is measured in "footlamberts."

One footlambert, a unit of photometric brightness (luminance), is equal to $1/\pi$ candle per square foot.

(A theoretical perfectly diffusing surface emitting or reflecting flux at the rate of one lumen per square foot would have a photometric brightness of one footlambert in all directions).

Brightness (B) = illumination (E) x reflectance (r). As reflectance is given in percent the formula for brightness would be:

$$B = \frac{E(\%r)}{100}$$

Light Intensity Meters

Several kinds of light meters are commercially available. The ones most commonly used for lighting surveys are the General Electric (G.E.) pocket sized meters, and the Weston hinged-cell meters. Both types are available with cosine correcting elements. With cosine correction, a light meter responds equally to illumination from all directions instead of primarily to vertically incident radiation. The Weston hinged-cell meters have wider ranges than the G.E. pocket sized meters, but are more expensive. For highly accurate illumination measurements special low-range sensitive and multi-cell light meters are commercially available.

Brightness Meter

The most commonly used brightness meter is the Luckiesh-Taylor instrument made by G.E. It has a battery operated internal light source, making it a self-contained unit. With it, the brightness of any surface can be measured within the range 0.0025 to 50,000 footlamberts. The instrument has two eyepieces. One is a focusing telescope which is sighted on the surface to be measured. In the center of this field are a pair of silvered trapezoids illuminated to a constant brightness by the internal light source. The brightness of the background is adjusted with a circular gradient filter until it matches the trapezoids in brightness. The second eyepiece views a scale which rotates with the gradient filter. At the proper setting of the filter, the brightness can then be read directly from the scale.

Visibility Meter

The visibility meter is a device for determining the relative visibilities of seeing tasks under various conditions of lighting and levels of illumination. It can be used also to determine the relative footcandles required by a variety of tasks to make them equal in visibility. The object whose visibility is being measured is viewed through variable filters which are adjusted until the object is just barely seen. This is the point of threshold visibility.

A new instrument, the visual task evaluator, was developed at the University of Michigan in 1957. However, in its present form it is used for research purposes only. Results of tests with this instrument may bring about future modifications in the visibility meter.

Glare

Measurement of glare, either reflected from surfaces or direct from luminaires, has posed a problem because satisfactory field instruments are not commercially available. Research in this field has progressed and several meters have been developed for this purpose. These should be available in a few years.

Ultraviolet Radiation and Black Light

Ultraviolet radiation is divided into: (1) Long wave, which is continuous to the luminous rays (visible light) and extends from about 3900 to 2900 A; and (2) short wave, which extends from 2900 to 1800 A. A portion of the long waves that ranges from 3100 to 4100 A is known as the black light portion of the ultraviolet region, although this is not a very specific term. The peak transmission of black light occurs around the 3650 A line. Very long ultraviolet wave lengths

from 3200 to 3100 A penetrate deeper into the skin but exert little biological effect. Short rays below 2300 A exert very little biological effects and have practically no penetration. Most biological action occurs in the intervening zone between the long wave lengths and the short rays.

Physiological Effects

The two physiological effects from exposure to ultraviolet radiation are: (1) Photochemical, occurring in the skin; and (2) biological, which occur in the blood or body metabolism. There is usually a latent period of four to eight hours between exposure and development of injury. Radiations below 2950 A are absorbed in the cornea and conjunctiva. This absorption can produce conjunctivitis, corneal ulcers, or iritis. Secondary infection may result if the individual attempts to allay the painful irritation by rubbing his eyes. Prolonged exposure to ultraviolet energy from 3400 A to 2800 A, with the greater potency from 3000 A to 2800 A, is capable of causing skin cancer in certain people.

One of the most prevalent sources of industrial exposure to ultraviolet radiation occurs during welding operations. In particular, the inert-shielded gas welding produces much higher levels of ultraviolet than other types of welding. Other industrial sources may be found in the use of a plasma jet torch and in areas where ultraviolet lamps are used for their germicidal action such as in hospitals, pharmaceutical plants and in the sterilization of food.

The American Medical Association, Council on Physical Medicine, has recommended the following limits for exposure to ultraviolet light: 0.5 microwatts per square centimeter for a 7-hour exposure, and 0.1 microwatts per square centimeter for a continuous 24-hour exposure, both as a tolerance level. These figures are based upon the total intensity of ultraviolet radiation at 2537 A and which is reflected from walls and fixtures eminating directly from the lights and incident upon the occupants. A more detailed discussion of this subject can be found in the book on Ultraviolet Radiation by Koler(8).

Instruments for Measuring Ultraviolet

The measurement of ultraviolet light is considerably more complicated than the measurement of visible light. There are no broad range instruments, and if there were, their readings would have questionable value, since different wave lengths of ultraviolet have different effects. For instance, 2537 A is the most effective bactericidal wave length, while 2947 A has the greatest erythemal effect. Therefore, in the measurement of ultraviolet, it is important to determine both the intensity and wave length of the radiation.

Commercially available instruments include the following intensity meters:

1. Germicidal attachment for the General Electric pocket light meter. This attachment consists of a sandwich of fluorescent phosphor between a glass and a quartz plate. The difference between the readings with and without the attachment is a measure of ultraviolet intensity at 2537 A. It is a relatively insensitive instrument requiring about 35 microwatts per sq. cm. (milliwatts per sq. ft.) to obtain a difference in reading of 1 foot-candle.

2. Germicidal ultraviolet intensity meter (G.E.). This is a sensitive instrument for measuring intensity at 2537 A. It utilizes a cadmium photocell and an electrometer tube circuit. The instrument has three ranges, 1, 10, and 100 microwatts per sq. cm., and is battery operated.

3. RS Sunlamp Tester (G.E.) is used to test the output of sunlamps. It consists of a G.E. pocket sized light meter with a filter which responds to erythemal ultraviolet radiation. It would be difficul to interpret the readings of this meter for uses other than sunlamp testing, but it might conceivably be useful for comparison purposes during a study.

4. Measurement of ultraviolet may be made by the Archer-Reed Ultraviolet Meter manufactured by the Archer-Reed Company, 23874 Kean, Dearborn, Michigan. This instrument is calibrated to indicate eminations representative of 0 to 2000 microwatts per sq. cm. at 2537 A. It responds only to energy in the wave lengths of 2000 to 3000 A.

5. Other methods for measuring ultraviolet include photocells or film sensitive to certain wave lengths. By using a series of such cells or film, the radiation can be broken down into bands, and the intensity of each determined.

Illumination References

1. Foote, F. M.: Ophthalmic Aspects of Light and Vision. Ind. Med. and Surgery 27:546 (Nov. 1958).

2. _____: Council of Industrial Health, Effect of Fluorescent Light on Vision. J.A.M.A. 168:47 (Sept. 6, 1958).

3. Sell, Floyd: Application of the Four Factors of Seeing to a Variety of Industrial Operations. Ind. Med. and Surgery 27:578 (Nov. 1958).

4. Kahler, W. H.: Visual Comfort in the Plant. Ind. Med. and Surgery 27:556 (Nov. 1958).

5. Niedhart, J. J.: Visual Comfort in the Office. Ind. Med. and Surgery 27:558 (Nov. 1958).

6. Guth, S. K.: Lighting Research. Amer. Ind. Hyg. Assoc. J. 23:359 (Sept.-Oct. 1962).

7. Encyclopedia of Instrumentation for Industrial Hygiene. Edited by Yaffe, C.D.; Byers, D.H.; and Hosey, A.D., Univ. of Michigan Institute of Industrial Health, Ann Arbor, Michigan, 1956.

ELECTROMAGNETIC SPECTRUM (II)
(MICROWAVE AND COHERENT LIGHT)

Introduction

Communication and electrical engineers divide the spectrum of electromagnetic radiation into two primary sections[1]. This sectional division is based upon the principal methods of detection of the radiation, and includes, (1) detection of quantum effects in the "optical regions" which extends from the short x-rays to the far infrared (see Figure 1, Section B-26) and (2) the detection of phenomena more closely associated with electrical effects produced in the microwave and radio frequencies of the spectrum, which are beyond the far infrared portion of the spectrum.

The energy from the radiations of all the frequencies of the electromagnetic spectrum have some type of biologic effect[2]. The electromagnetic spectrum can be divided into three sections and considered on the basis of the three primary biological reactions.

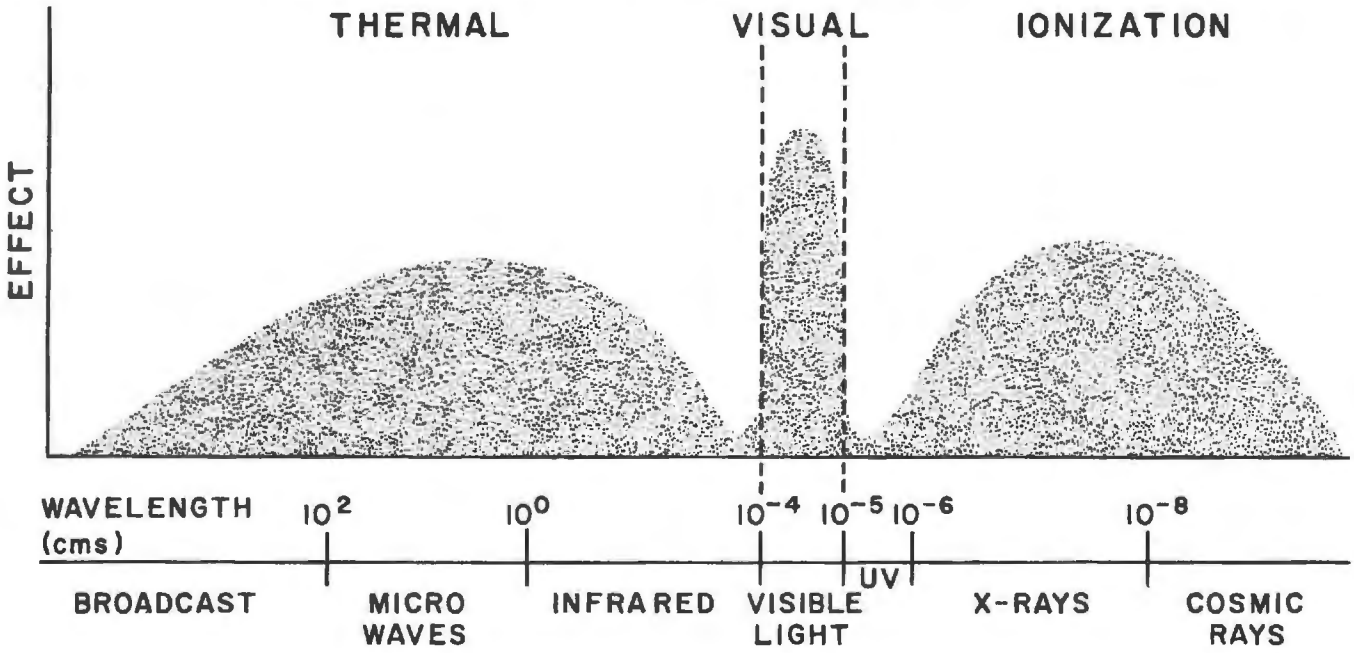


Figure 1. - Effects of Electromagnetic Radiation.

The biological effect upon the retina of the eye which makes sight possible is not an adverse reaction; this however, is not the case with the effect of ionization at wavelengths less than 10^{-6} or the thermal effects found in the microwave, and infrared portions of the spectrum.

A more classical division and the one primarily utilized in industrial health practice, as indicated in Section B-26, divides the spectrum into smaller segments based on not only the wavelength and frequencies, but also on the method of production and application of the radiation. These include microwave radiation, infrared, visible light, ultraviolet light and x-radiation. The recent development of a new type of electromagnetic wave generator, which produces coherent energy also must be included. The biologic effects of microwave radiation and coherent energy of lasers and masers, as well as the poten-

Prepared by: Charles H. Powell, Sc.D.

tial hazard of exposure of the working population to these radiations will be considered in this Section.

Microwave Radiation

The term microwave or radio frequency radiation is generally considered to apply to that portion of the electromagnetic spectrum ranging from 100 to 30,000 megacycles (MC) - wavelength 3 M to 1 cm. This wide range of frequencies includes commercial as well as television and broadcast bands, bands of diathermy and microthermy units, and the X (9000-9500 MC), S (2600-3200 MC), and L (1100-1400 MC) bands of radar.

Biological Effects

The amount of microwave radiation which is absorbed in the body and transformed into some form of energy, usually heat, is dependent upon a number of factors, one of which is the frequency of the radiation. At longer wavelengths the radiation will penetrate the body to a greater depth than at shorter wavelengths or higher frequencies.

The electromagnetic energy is thought to be absorbed and to produce three primary effects, these are: (1) The thermal effect; (2) the electrical effect, and (3) specific biological effects.

Thermal effect. This is produced by the conversion of electrical energy of the radiations into heat. As indicated above the depth of penetration is related to the wavelength of radiation and also differences in the thermal conductance of various tissue. These result in different amounts of energy being absorbed and different degrees of heating of such tissue as fat, bone, and muscle. This is a dynamic situation and as the energy is being absorbed and converted into heat, the heat is dissipated by the blood stream. As long as the intensity of the radiations is comparatively low and the heat generated can be dissipated by normal body functions, the only result will be discomfort. However, when the intensity of electromagnetic energy generated is increased not only is the amount of heat produced increased but also there is the possibility that another biologic effect may occur.

Electrical effect. This is the second primary effect just referred to. It causes an enforced distribution of the electrical charge in the molecules of tissue under the influence of a microwave field. This electrical effect seems to be associated only incidentally with the frequency of the radiation. It is postulated that under the applied electrical fields a complete reorganization of molecules can occur. This seems to be particularly possible in molecules having loose chemical bonds and relatively free electrons, such as hydrogen and unsaturated bonds[2]. Furthermore, the electrical effect is associated with the electrical field and seems to be a field effect. It may be possible to produce the same effect with magnetic fields as well as electrical fields, but magnetic fields are believed to be of only secondary importance in causing reorientation of the molecule since the magnitude of the magnetic fields is somewhat smaller than that of the electrical fields.

Specific biologic effects. Exposures to these radiations have been reported to cause changes in electrophoretic patterns, an increase in antigenic activity, and the production of cataracts in the eyes of experimental animals.

Maximal Safe Exposure Value

Based upon the best information available at that time, primarily experimentation on small fur animals, the United States Air Force several years ago adopted a Maximal Safe Exposure Limit (MSEL) of 10 milliwatts per square centimeter. This limit also has been adopted in a number of other countries. The MSEL does not take into consideration the following:

(1) The duration of the exposure or repeated short term exposure

through any given period. Exposure time is of special concern in the case of pulsated transmissions of radar generators.

(2) The frequency or wavelength of the radiation, even though experimental data have indicated that the coefficient of absorption in tissue varies with frequency.

(3) Exposure to radiation of several different frequencies. This is of prime importance at radar installations where microwave generators operate at different frequencies and could be a source of potential multi-frequency exposure.

(4) Increased dissipation of the heat caused by air movement past the body being exposed to the radiation.

(5) The differences in sensitivity of various organs to the absorption of heat.

(6) Consideration of reflected radiation, and

(7) Environmental temperature and its effect upon the dissipation of heat created by the impingement of microwave radiation on the body.

In 1961 consideration was given to duration of exposure in proposing new limits[3] and later, in 1964, similar limits were adopted by the USAF[6] which also took into consideration intermittent exposures and average power density. These limits are: "A maximum field power density of 0.01W/cm^2 (10MW/cm^2) for any period of time (used as a design criteria). This threshold level is based on an average power density, not peak power. In the case of a continuous wave, radar peak and average powers are equal. In case of intermittent exposures, the time on, time off, may be averaged. However, if the power density is over 0.1W/cm^2 (100MW/cm^2) then averaging cannot be permitted and no personnel exposure will be allowed." At the present time the American Standards Association C-95 committee is considering the establishment of a limit as have other governmental and private organizations[3,6].

Measurement of Power Density

Measurement of free-field power density of microwave radiations may be detected either by electrical or thermal methods. The electrical method uses low-frequency devices for measurements after conversion of the microwave energy to low frequency electrical energy by means of a nonlinear amplifier. The thermal method of detection is based upon the conversion of microwave energy into heat. This method has the disadvantage that it does not differentiate between the microwave and the so-called "optical region" of the electromagnetic spectrum, as is possible with electrical measurement, but it is applicable to the entire spectrum. The thermal method, however, has the advantage of comparative ease of calibration, and is normally the method of choice in industrial health practice.

High power density measurements (greater than 1 W) are normally obtained by using calorimetry to change the microwave radiation into heat and then determining the relationship of the heat produced to the power density. Medium (10 MW to 1 W) and low (less than 10 MW) power density measurements are made by use of a bolometer, which is a temperature sensitive resistive element for the determination of the amount of heat produced by absorption of the radio frequency power. In instruments of this type, the bolometer may be one arm of a Wheatstone bridge which, in the absence of microwave energy, is balanced by bias power. Bolometers are normally sensitive to changes in the environmental temperature, and this sensitivity is usually the limiting factor for measuring minimum power levels with any degree of accuracy. The upper limit of measurable power densities can be extended by the use of attenuators.

In measuring the medium and low power densities, which include the range of most interest in industrial health, two basic types of bolometers are used, these are:

(1) Barreters, made of a fine platinum wire with a positive temperature coefficient of resistance, whose resistance can be impedance matched at the end of the transmission line. These detectors are usually delicate and will burn out quickly in fields of excessive power densities.

(2) Thermistors, which consist of a small bead of semi-conducting material between two fine parallel wires. The primary resistance of a thermistor is in the bead, which has a negative temperature coefficient of resistance. The thermistor is usually mounted at the termination of the transmission line. These devices are usually more sensitive, and less delicate than Barreters. In addition they are less susceptible to damage by high power density levels and, as a result, they are usually superior components for field instruments. In both the barreters and thermistors the resistance change resulting from the microwave energy is measurable as one leg of a Wheatstone bridge. The thermistors or barreters are used with a calibrated antenna or feed horn to measure the radio frequency radiation.

These antennae can be utilized only over a narrow range of frequencies or wave lengths, and the choice of the feed horn or antenna depends upon the frequency of the microwave energy that is to be measured. The microwave antennae used in measurement of medium and low power densities usually are conical feed horns or may be of other configurations. These microwave radiation probes normally are used for a fixed range of frequency but some may be used for pickup over a variable range of frequencies. The antennae are highly directional and the absolute gain of the receiving probe may be changed drastically by the positioning of the antenna in relation to the radio frequency field. Most industrial health organizations will not have a microwave source of the appropriate frequency and intensity for calibration of power density meters, which normally will necessitate return of the densiometer to the manufacturer for periodic calibration.

Engineering Control Methods

Basic engineering control principles used in industrial hygiene are discussed in Section C-1, and are as applicable to the control of microwave radiation as to other potential sources of hazardous conditions. Although there are other industrial applications of microwave radiation, the primary sources are commercial and military radar installations. When selecting engineering control methods the physical characteristics of not only the microwave radiation, but also the location of workers, equipment, buildings and, at radar installations, of the surrounding terrain must be considered.

Several specific methods have been developed and applied to control potential exposure to microwave radiation and should be given careful consideration. These are:

(1) To increase the distance between the source of energy and the workers. Microwave radiation normally can be considered as a point source and the power density at any given location can be estimated using the inverse square law. Care, however, must be exercised in the use of the inverse square law as a control measure near a microwave transmitter, since the generator does not act as a point source in the near field and crossover regions near the antenna or reflector. Reflection of radiation from

buildings and other obstructions can increase and intensify the energy ten to twelve times the emitting power of the generator.

(2) Shielding of occupied areas by the use of wire mesh for reflecting microwave radiation, which has proved to be a satisfactory method of control. A nomogram has been developed for estimating the attenuation of microwave radiation by wire mesh under various conditions[4]. Shielding of this type should be used primarily as a means of control in the far field and preferably in areas of occasional occupancy. Another form of shielding is to blank off certain sectors of the microwave field to assure that personnel will not be exposed while in the sector. This type of shielding may reduce the effective operational capability of microwave generators since the area traversed has been reduced. This type of shielding possibly could be eliminated by the proper positioning of the transmitters.

(3) The use of protective clothing by operational and maintenance personnel. Proper protective clothing has been developed for workers in fields of high power densities. This protective clothing consists of a suit made of a special metalized fabric which is coated with neoprene to protect against the discharge of electrical currents. Special closures are provided since metalized zippers cannot be used. Mittens and boots are fitted to the suit to make it a completely contained unit. The eyes are protected by closely woven copper screening. It is difficult to use a suit of this type, particularly in warm or high temperatures, unless a ventilating or air-conditioning system is provided. Protective clothing as a means of control for microwave radiation is somewhat open to question and its use as a substitute for other engineering controls and safe operational procedures should never be condoned. The primary use of protective clothing at radar installations would be by operating and maintenance personnel. Maintenance often can be accomplished either during shut down periods of the transmitter or when personnel are located in areas which are not in the microwave beam.

With the continued development and increased commercial use of larger microwave transmitters, constant surveillance and control must be exercised to assure that excessive exposures to microwave energy do not occur.

Coherent Energy

In 1954, a new device was developed to generate electromagnetic energy which concentrated the radiations into a comparatively small area. This coherent electromagnetic energy can be generated in several portions of the spectrum at the present time (microwave and visible) and it is anticipated that these radiations can be generated at other frequencies in the future. Industrial, medical, and research applications of these radiations are increasing at a rapid pace.

Coherent energy was first generated in the microwave frequencies by stimulating the emission of energy from molecules of ammonia[7]. From this method of production of coherent energy the acronym Maser was developed from Microwave Amplification by Stimulated Emission of Radiation. The term used for coherent light in the visible section of the electromagnetic spectrum is LASER, or Light Amplification by Stimulated Emission of Radiation. When the part of the electromagnetic spectrum producing the energy is not identified the process of amplification by stimulated emission of radiation may

be referred to as ASER. The waves from any one source have almost the identical wavelength and frequency and can be considered as a monochromatic beam. Coherent energy is emitted from the source as directed parallel beams and it is not discharged as a wider spectrum of frequencies nor does it follow the inverse square law, as does most electromagnetic energy. This coherent radiation may be released as either a pulsed or as a continuous wave.

Biological Effects

Biological effects of exposure to coherent light are still under investigation, however, the magnitude of the biological reaction can, to some extent, be related to several phenomena associated with the energy produced. The intensity and frequency of the emitted energy, whether or not the beam is focused or unfocused, and if it is a pulsed or a continuous wave determines to a varying degree the severity of the biological reaction to coherent light, as does the duration of exposure. There are two primary effects:

(1) Damage to the retina of the eye, which is probably a thermal effect. The eye is particularly sensitive to this type of energy, due to its inability to dissipate heat. In addition, the energy of coherent light is intensified as it passes through the cornea and is focused on a small segment of the retina, thereby increasing the energy per unit area. Other effects have also been postulated[8]. Considerable differences in power densities are required to produce biological effects of the same magnitude in experimental animals exposed to different frequencies and to pulsed or steady state emitters of coherent energy.

(2) Superficial tissue damage from the production of heat by the absorption of radiation on the skin; which is usually considered to be primarily a surface phenomenon. The pigmentation of the tissue affects the magnitude of biological reaction with a greater reaction to areas of hyperpigmentation. This reaction has been observed from unfocused laser beams[8].

Engineering Control Methods

At the present time no maximum exposure limits have been established for coherent energy. The methods of engineering control of exposure to lasers and masers are based upon the same principles as are applied to other electromagnetic radiation. It must be remembered, however, that coherent light does not follow the inverse square law. Thus, the use of distance from the source is not as an effective means of control as it is with other electromagnetic radiation. The necessity for engineering control is based upon the intensity of the energy and the duration of exposure.

Various techniques for engineering control of potential hazardous sources of lasers and masers have been developed and should be given careful consideration. These are:

(1) Avoid exposure to the primary beam and any reflected energy from walls or other surfaces. This is a basic means of control, however, if this is not possible due to operational procedures, consideration should be given to other means of control. It must be remembered that an acceptable maximum exposure limit has not been determined and that one exposure may be sufficient to cause retinal damage.

(2) Visual or audible signals should be used to indicate when the maser or laser generator is operating. These signals should be positioned to call attention away from the light source not toward the source of energy. If the

laser or maser is to be used outside of a building the proposed path of the beam should be well posted and other special precautions as required should be taken to reduce the possibility of exposure, as damage may occur at extended distances from some generators of coherent energy.

(3) Laser and maser operations of high intensity which occur in a room may dictate that the walls and other surfaces be covered, if possible, with suitable material to reduce the reflection of the energy. In addition, the room should be well lighted to reduce the size of the pupil of the eye.

(4) It may be desirable to enclose the operation in a suitable shield to reduce the possibility of exposure to either the operational staff or transient personnel.

(5) The use of protective eye shields has been recommended[8] but the proper lens must be selected for the wavelength of the coherent energy being produced. Some investigators[9,10] have indicated that the transmittance characteristics of the glass may change or fade with repeated use, consequently care must be taken in the use of eye protectors.

(6) One control of prime consideration is the education of the workers regarding the health problems related to the use of coherent light and operational procedures which they must use to assure that they are not exposed excessively to this type of energy. Furthermore, operating personnel should be aware of the necessity of following such industrial medical procedures as reporting immediately to the medical department any exposure. This may be of particular importance if an after image of the light source persists.

References

1. Montgomery, G. C.: Technique of Microwave Measurements. McGraw-Hill Book Company, Inc., New York, 1947.

2. Boysen, J. E.: Microwave Radiation, Proceedings Sanitary and Industrial Hygiene Engineering A.S.T.I.A. No. AD-287799, 65-80, Oct. 1961.

3. Weiss, M. M., and Munford, W. W.: Microwave Radiation Hazards. Health Physics 5: 160-168 (1961).

4. Engelbrecht Fourth Annual Tri-Service Conference on the Biological Effects of Microwave Radiation, Sept. 1960.

5. _____: Health Hazards Information on Microwave Radiation AFP 160-6-13, May 1958.

6. _____: The Threshold Limit Value for Toxic Chemicals and Certain Electromagnetic Radiation AFP 161-2-1, April 1964.

7. Brotherton, M.: Masers and Lasers - How They Work, What They Do. McGraw-Hill Book Company, Inc., 1964.

8. Tebrock, H. E., Young, W. N., and Machle, W.: Laser-Medical and Industrial Hygiene Controls. J. Occ. Med. 5: 564-567 (Dec. 1963).

9. Goldman, L., Blaney, D.J., Freemond, A. and Hornby, P.: The Biomedical Aspects of Lasers. J.A.M.A. 188: No. 3, 302-306 (April 1964).

10. Straub, H. W.: Protection of Human Eye from Laser Radiation, U.S. Army Material Command, Harry Diamond Laboratories TR-1153.

11. Diakun, R.: Laser, Commonwealth of Pennsylvania, Dept. of Health Hygiene Information Guide No. 72, Nov. 1963.

HEAT STRESS

Introduction

Some of the problems that confront man when he is exposed to a hot environment and some of the physiological mechanisms he can bring to bear in meeting the problems will be discussed in this Section.

There are two sources of heat which are important to man working or living in a warm or hot environment. These are:

(1) The internally generated metabolic heat, and
(2) The externally imposed environmental heat.

Metabolic heat is a byproduct of the chemical processes occurring within the cells, tissues, and organs. Under resting conditions the metabolic heat production of an adult is about 75 KCal per hour (300 BTU). Muscular activity is the major source of increased heat production. During very hard physical work heat production may reach 600-750 KgCal per hour (2400-3000 BTU). Thus under conditions of physical work large quantities of heat must be removed from the body if an increase in body temperature is to be prevented. Environmental heat is important because it influences the rate at which body heat can be exchanged with the environment and consequently the ease with which the body can regulate and maintain a normal temperature.

An internal body temperature of 99°F (98.6°F mouth temperature) is usually considered to be "normal". However, body temperature varies from time to time during the day and with changes in physical activity. Consequently body temperatures of 97 to 102°F are frequently and normally obtained. Body temperature over 102°F in otherwise healthy individuals must be viewed with some concern and a temperature over 105°F is critically serious. Consequently, the regulation of body temperature is an important physiological function and the ease with which it can be successfully accomplished is determined by the individuals' ambient environment -- by the air temperature, the humidity of the air, air movement, long wave radiation and solar radiation.

Prepared by: Austin F. Henschel, Ph.D.

Heat Exchange

The metabolic heat produced by the body is exchanged with the ambient environment by the processes of convection (and conduction), radiation and evaporation. If the contact substance, whether it be air, water, clothing, or an external object, is at a lower temperature than the skin, heat will be lost; but if the contacting substance is at a higher temperature, heat will be gained. The rate at which transfer takes place is determined basically by the difference between the two temperatures, but if the contacting substance is fluid, like air or water, movement in the fluid accelerates the transfer. This additional transfer process is termed convection.

Heat Transfer by Conduction-Convection

Nearly all transfer of "sensible" heat between skin and air is by the combined process of conduction-convection, in which convection plays by far the greater part. Heat transfer by conduction-convection from the skin to air may be expressed quantitatively by the equation:

$$(1) \qquad H_c = \frac{K_c\,(t_s - t_a)}{I_a + I_c} \qquad \text{where:}$$

H_c is the rate of heat loss per unit area of exposed surface
K_c is a constant whose value depends upon the units used
t_s is the temperature of the skin surface
t_a is the temperature of the ambient air
I_a is the resistivity of the ambient air to the outward passage of heat
I_c is the resistivity of the clothing to the outward passage of heat.

To this exchange between the skin and the air must be added heat exchanged between the respiratory tract and the inspired air, since the former behaves in this respect simply as an inward extension of the body surface, with a special mechanism--respiration--moving the air away when it is heated.

Heat Loss by Evaporation - Convection

Heat may be lost also from the surface of the body to the air by evaporation of water diffusing through the skin from deeper tissues (perspiration), produced by sweat glands (sweating), or applied from without. The rate of evaporative heat loss is determined basically by the difference between the effective vapor pressure of the water on the skin and that of the air, but once again movement of the air greatly accelerates the rate of loss, so that the combined process is properly termed evaporation-convection.

The vapor pressure of such water as is present on the skin is determined by the temperature of the skin, but the extent of the water film varies between something less than 10 percent and 95 percent of the maximum. The effective vapor pressure of water on the skin is thus a function of these two factors. The extent of the water film is variously termed "skin wetness" or "skin relative humidity". It represents a balance between evaporation on the one hand and addition of water on the other. It is high only when the sweat glands are active, evaporation is inhibited, or water is applied from without.

Humidity. The vapor pressure of the air is determined by the amount of water vapor present in (unit volume of) the air and corresponds closely to the absolute humidity of the air. Unfortunately, atmospheric humidity is usually expressed in terms of relative humidity, which is something quite different, namely, the ratio between the amount of water vapor actually in the air and the amount the air could hold at that temperature. To determine the vapor pressure from the relative humidity one also needs to know the air temperature and to have tables or a graph by which to make the transformation. A great deal of misunderstanding and confusion has arisen from the use of these two measures of atmospheric humidity, between which the relationships are far from obvious. A vapor pressure of 15 mmHg corresponds to 100% relative humidity at 63°F, 50% at 84°F, and 30% at 100°F, since the holding capacity of the air increases with temperature while the amount of water vapor remains the same. Another measure of humidity sometimes used, the dew point, is the temperature at which air, on being cooled, becomes saturated and moisture begins to be deposited from it. Dew point is closely related to vapor pressure and to absolute humidity. The various combinations of dry-bulb temperature and relative humidity just cited as having the same vapor pressure (15 mmHg) also have the same dew point (63°F).

A generalized equation for heat loss by evaporation from the skin to air is:

$$(2)\quad H_e = \frac{K_e\,(P_s - P_a)}{r_a + r_c}\,w \qquad \text{where:}$$

H_e is the rate of heat loss per unit area of exposed surface

K_e is a constant whose value depends upon the units used

P_s is the saturation vapor pressure at skin temperature

P_a is the vapor pressure of the ambient air

r_a is the resistivity of the ambient air to the outward passage of water vapor

r_c is the resistivity of the clothing to the outward passage of water vapor

w is the proportional wetness of the skin.

To this loss must be added that from the respiratory tract into the inspired air.

Heat Exchange by Radiation

Heat will be exchanged by radiation between the surface of the body and all of the surfaces in its surroundings which are at temperatures different from its own. (The term "surface" is easily understood for solid objects, but for such things as the sky it must be regarded as that hypothetical surface which would exhibit the same radiative behavior as the sky is observed to exhibit). The details of radiative exchange can become very complex, but the following simplified explanation will illustrate the principles involved.

The intensity of the energy emitted from a surface by radiation increases as the fourth power of its absolute temperature. The intensity is usually diminished below the theoretical maximum, however, by the physical nature of the surface, the relative effect being known as its emissivity. The wavelengths of the emitted radiation are usually distributed over a range, with a model length which decreases as temperature increases.

Radiation incident upon a surface is absorbed by it in proportion to its emissivity for the wavelength involved. The absorptivity for a particular wavelength is the same as the emissivity for that wavelength. From an opaque surface, the incident radiation which is not absorbed must be reflected, so that its reflectivity is the converse of its absorptivity and thus of its emissivity -- for the particular wavelength involved.

A substance whose surface emits at maximum intensity for its temperature is termed a "black body." (This is an unfortunate term, since it inevitably suggests a visual observation which relates to reflectivity rather than to emissivity, and then only in the portion of the spectrum to which the eye is sensitive). In general, most conventional surfaces other than highly polished metals are classified as "black bodies" in the long infrared; but many of them are obviously far from black bodies in the visible range. Thus, the apparently paradoxical statement can be made that a white shirt may be a black body (in the long infrared).

The surface of the human body and its clothing emit only in the long infrared range; and in this range virtually all such surfaces act as black bodies. Emission from surrounding surfaces, however, is far more complex. Many conventional surfaces at normal temperatures are emitting long infrared radiation as essentially black bodies; but some, at the same temperature, may be emitting less intensely (for example, polished metal surfaces). Some surfaces at higher temperatures may be emitting short infrared radiation, and others at still higher temperatures visible or even ultraviolet radiation. The exchange between the body and surrounding infrared emitters is fairly simple, being complicated only by the geometrical relationships of the opposing surfaces, which often can be approximated by a single sphere at a uniform temperature. For such a situation the appropriate equation for radiant energy exchange is:

$$(3)\quad H_r = K_r (T_w^4 - T_s^4) \qquad \text{where:}$$

H_r is the rate of exchange per unit area of exposed surface
K_r is a constant whose value depends upon the units used
T_w is the absolute temperature of the surrounding sphere
T_s is the absolute temperature of the skin.

Net Heat Exchange

The net heat exchange between the man and his ambient environment can be expressed by:

$$(4)\quad H = M \pm C \pm R_1 + R_s - E \qquad \text{where:}$$

H = net heat gain or loss by the body
M = metabolic heat production
C = heat exchange by conduction-convection

h 7 D

R_1 = heat exchange by long wave radiation
R_S = solar heat gain
E = heat loss by evaporation.

If the body temperature is to be maintained at an acceptable normal level then H must equal zero. Small fluctuations in total body heat are, of course, permissible. They normally occur as a result of rapid changes in metabolic heat production or in the rate of heat exchange with the ambient environment. Metabolic heat production may increase by a factor of 10 within seconds as one goes from a state of rest to maximum physical effort (from 75 to 750 KgCal per hour or 300 to 3000 BTU). Metabolic heat production can be calculated since about 5 Kg Calories are liberated for each liter of oxygen used by the body cells.

The heat exchanged by convection-conduction can be calculated from equation (1). The rate of exchange depends on skin temperature, air temperature and insulation value of the clothing and air surrounding the skin. The insulation value of the air-clothing system will vary with the rate of air movement. For practical purposes these relationships have been derived in simplified form and put in graphic form for conditions of a constant nude skin temperature of 95°F (see Figure 1).

Evaporative heat loss from the clothed man as indicated in equation (3) can be a rather complex phenomenon. However, evaporation of sweat from the skin surface is a very effective means of losing body heat. Each liter of sweat requires 580 Kg Calories to evaporate it. The major factors that influence rate of heat loss by evaporation are the vapor pressure of the ambient air, the saturation vapor pressure of the skin, air movement and the resistance of the clothing to vapor movement. The simplified relationships for conditions of a nude wet skin at 95°F have been derived in graphic form and are presented in Figure 2.

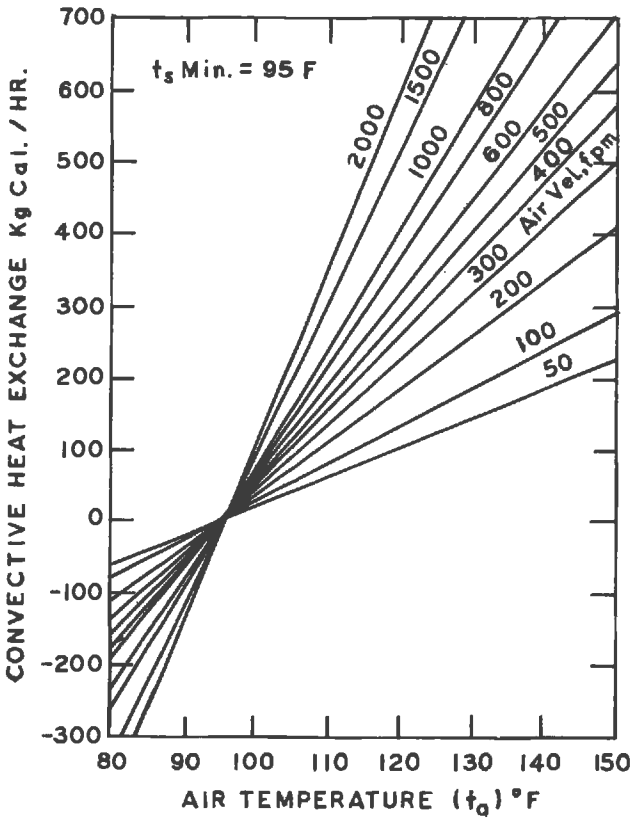


Figure 1. - Heat exchange by convection between man (skin temperature 95°F) and surrounding air.

To calculate radiant exchange from equation (3) requires more complex mathematical manipulation than may appear from the form of the equation. This is due primarily to the complexity of shapes of most surrounding objects. If the surrounding is assumed to be a sphere, the mean temperature of which can be measured, the rate of heat exchange with the nude individual with a skin temperature of 95°F can be obtained readily from a graphic representation of the relationships as shown in Figure 3.

The equation $H = M \pm C \pm R_1 + R_S - E$ can be solved using Figures 1, 2 and 3 provided the data on metabolic rate, air temperature, radiant temperature, vapor pressure and air movement are available. These data can be obtained at the worksite in an industrial environment, in a field situation, and in a controlled laboratory set-up. In Figures 1, 2 and 3 certain basic assumptions were made

in order to simplify the presentation. Interindividual differences in sweating, (vapor pressure, at skin temperature) blood flow to the skin, muscular efficiency, body volume-surface ratio and others make it hazardous to apply data derived from these figures to specific situations.

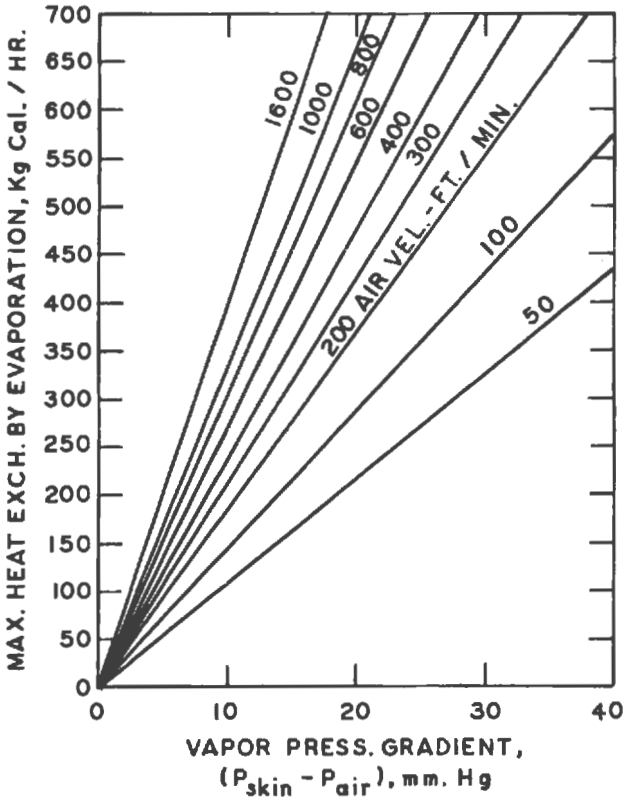


Figure 2. - Maximum evaporative capacity as related to air velocity and vapor pressure gradient.

Responses to Heat

Since radiant energy exchange, air temperature, humidity, and air movement all affect, in quantitative fashion, the same physical process -- heat balance of the body -- their operations are largely interchangeable. An alteration in one can be duplicated or compensated by an appropriate change in another. The effect of a rise in radiant heat gain can be duplicated by that of a rise in air temperature; a rise in humidity may be offset by an increase in air movement; and so on.

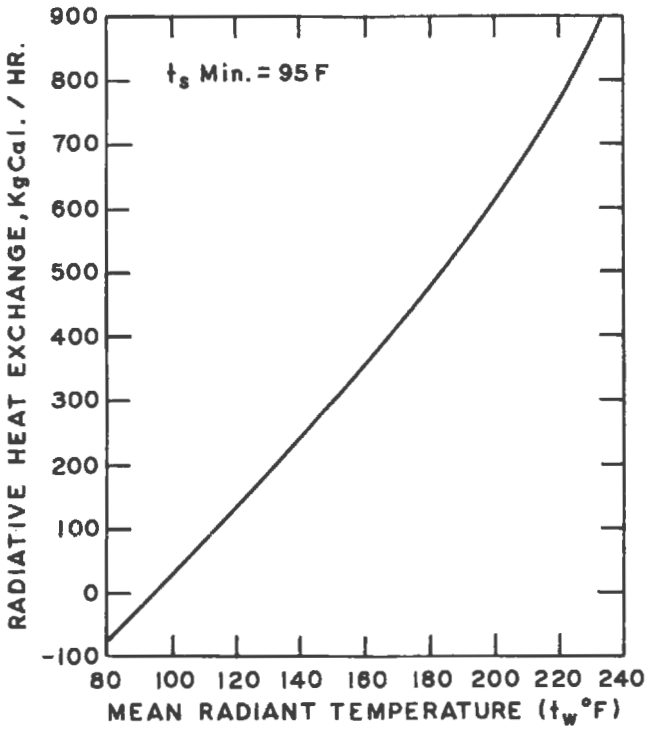


Figure 3. - Heat exchange by long wave radiation.

Certain schemes for assessing the net effect of all four thermal factors will be mentioned in Section B-29; it will suffice at this stage to point out some of the major inter-relationships. At temperatures below 70°F sweating is not called into play, the skin is comparatively dry, and changes in humidity are of little consequence. As temperature rises, sweating is called more and more into play, and humidity becomes of increasing importance. As long as air temperature is lower than skin temperature, all movement will facilitate heat loss by both conduction and evaporation, but when air temperature exceeds skin temperature a mixed situation is created. Air movement will still increase heat loss by evaporation, but it will now also increase heat gain by conduction-convection. The higher the air temperature, the more important the latter will become, until it may actually override the increase in evaporative cooling. For each set of conditions in which air temperature exceeds skin temperature, there will be an optimal air movement.

Lower rates of air movement will result in sweat accumulation; higher rates will result in additional heating and place a bigger burden on compensatory sweating.

To the extent that the body has to take action to step up heat loss in the face of an environmental heat load, a burden is placed upon the body. This burden is represented primarily by the physiological reactions designed to promote heat loss, but these reactions in turn may provoke other changes which add to the total physiological disturbance. The ultimate consequences of this chain of events are illustrated in Figure 4.

Increased Flow of Blood

When the heat loss from the body by radiation and convection becomes less than the metabolic heat production, the first corrective action initiated by the body is a vasodilatation of the blood vessels near the surface of the skin, which results in an increased flow of blood to the area and an increase in skin temperature. This results in an increase in both convective and radiative heat loss from the body when the ambient air temperature and the average radiant temperature of the surroundings are less than skin temperature; if these are higher than the skin temperature the heat gain through these channels is decreased. The flow of blood in the human body carries heat from the interior of the body, where it is produced, to the surface of the skin where heat transfer occurs.

Sweating

Sweating, the second defense mechanism, is brought into action when there is an insufficient flow of blood to the skin to meet the requirements for heat loss. This occurs usually when there is anything more than a minor thermal imbalance. The number of sweat glands activated and the rate of secretion of sweat are graded to meet the magnitude of the imbalance. Sweat production of more than two liters an hour has been observed but continuous sweat rates of about one liter an hour over several hours each day are considered to be maximum production rates. This means that, except for short periods of time, about 600 KgCal per hour is the maximum amount of heat that will be lost from the body surface by sweat evaporation. Sweat that is not evaporated has no value for heat loss.

Sweat production results in a drain on the water and salt in the body. The water lost in sweat is usually replaced by an increase in water intake. In most situations the thirst mechanism is sufficient to keep the water intake and water loss in balance. However, under conditions of heat stress with large sweat production (6-12 liters a day), enough fluids are not voluntarily consumed to replace the water lost in the sweat. This "voluntary" dehydration may amount to two to three liters or more during an eight hour working day. The "voluntary" water deficit is usually replaced during meals and non-working hours if an adequate supply of drinking water is available. Dehydration in excess of three liters may have serious physiological and clinical consequences.

Heat Induced Illness

If the normal responses of increased skin blood flow and sweat production are not adequate to meet the needs for body heat loss or if the mechanisms fail to function properly, physiological breakdown may occur. There are four major categories of heat-induced illnesses: heat exhaustion, dehydration, heat cramps, and heat stroke.

Heat exhaustion. This is a state of collapse brought about by an insufficient blood supply to the cerebral cortex as a result of dilatation of blood vessels in response to hot conditions. The failure here is not one of heat regulation, but an inability to meet the price of heat regulation. A low arterial blood pressure is the critical event,

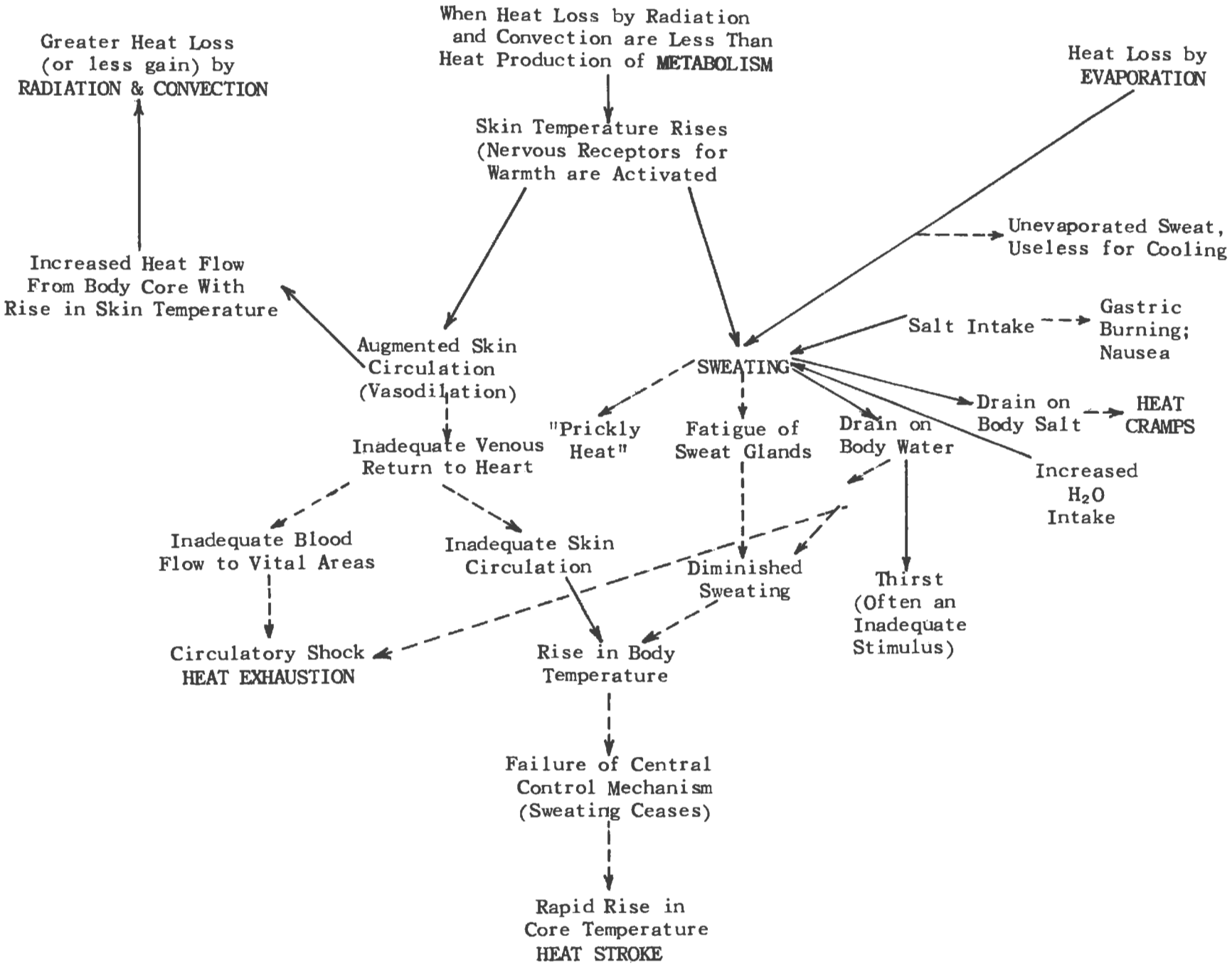


Figure 4. - Physiological effects of heat stress.

resulting partly from inadequate output of blood by the heart, partly from the widespread vasodilation. Inadequate cardiac output results in turn from a fall in the volume/capacity ratio below unity. The chief factors which may bring about this state of affairs may be classified as follows:

1. Increasing vascular dilatation and decreasing the capacity of the circulation to meet the demands for heat loss to the environment, exercise, and digestive activities.

2. Decreasing blood volume by dehydration, gravitational edema, adrenal insufficiency or lack of salt.

3. Reducing cardiac efficiency by emotion, malnutrition, lack of physical training, infection or intoxication, cardiac failure.

Dehydration. In its early stages, dehydration acts mainly by reducing the blood volume and promoting heat exhaustion. But in extreme cases it brings about disturbances of cell function which increase and reinforce each other with worsening deterioration of the organism. Muscular inefficiency, reduced secretion (especially of the salivary glands), loss of appetite, difficulty in swallowing, acid accumulation in the tissues, and nervous irritability followed by depression, will occur in increasing strength; while uremia, fever and death will terminate the picture. A surprising feature is the persistence of urine excretion in small amounts (5 cc/hr.) in the face of dehydration. Water to drink is the primary treatment, clinical experience suggests carefulness in administration, while the addition of chloride, glucose and perhaps alkalies is to be recommended.

Heat cramps. This is a condition of cramp-like spasm in the voluntary muscles following a reduction of the concentration of sodium chloride in the blood below a certain critical level. Just why cramps should follow a fall in blood chloride is not clear, but the association is certain and the relief obtained by the administration of chloride may be spectacular. A high chloride loss is facilitated by high sweating rates and lack of acclimatization. Depletion of chloride reserves is facilitated by low dietary intakes of salt, and by adrenal cortical insufficiency. A high water intake makes dilution of the remaining chloride easier. The actual critical level of blood chloride concentration seems to vary, and may be affected by factors such as general health which are not as yet understood.

The abdominal as well as the limb musculature may be affected, the exact site not necessarily being related to the preceding exercise. Abdominal cramps may simulate acute surgical conditions, while limb cramps may resemble exercise or nocturnal cramps. Their persistence without saline therapy and their abolition by it provide the clue. Heat cramps can be prevented by taking extra salt whenever heavy work is to be carried out in hot dry environments, especially by unacclimatized persons.

Heat Stroke (Hyperpyrexia). This is reached when the mean temperature of the body is such that the continued functioning of some vital tissue is endangered thereby. It represents, of course, a marked failure of the heat regulating mechanism to maintain a proper balance between the two sides of the heat balance. The chief factors which may bring this about may be classified as follows:

1. Reducing heat loss -- lack of sweat glands, inhibition of sweating, inadequate peripheral circulation, high environmental temperature, high humidity with restricted convection.

2. Increasing heat reception -- radiant energy absorption, environmental temperatures above skin temperature.

3. Increasing heat production -- muscular exercise, pyrogenic agents,

over-activity of the thyro-adrenal apparatus, rising body temperature, agitation.

4. Interfering with the heat regulating center -- brain injuries or infections.

The critical body temperature for man lies between 108 and 112°F, depending upon the length of time that the tissues are so exposed. The cause of tissue damage and finally death is probably a mixture of protein denaturation, enzyme degradation, alteration in the physical structure of the cell membranes, and changes in the viscosity of the cellular protoplasm. It is unlikely that this crisis will be reached in a healthy, acclimatized man carrying out normal activities in a natural climate. But under severe emotional and physical stress and very hot conditions, heat production may reach a level high enough to produce heat stroke without prior onset of the usual escape provided to man, that is, heat exhaustion.

Relief is secured only by an early and effective reduction of body temperature. This is usually obtainable by wrapping the patient in wet sheets and playing a fan on him, but it is sometimes necessary to resort to packing in ice. A resistance to gentler cooling methods is particularly likely to occur in cases where the hyperpyrexia is partially due to infection. It sometimes seems in such cases that the effect of the infection is to set the heat regulating "thermostat" at an abnormally high level, so that gentle cooling results only in vasoconstriction, with negation of the cooling effects. When drastic cooling is used, however, care has to be taken that the temperature is not lowered too fast or too far.

Acclimatization to Heat

The fact of acclimatization is well attested by both experience and scientific observations and some of its features are known but as yet much of the basic mechanisms are still elusive. With the onset of a heat wave or when one is suddenly transported to a hot environment, it is common experience to observe impairment in performance capacity and strong heat discomfort and distress. Tasks that were easily performed in a cool environment became difficult and heat discomfort may interfere with rest and eating. If, however, the exposure to the heat is continued for several days, performance gradually returns to normal and heat discomfort subsides at least to some extent; acclimatization to heat occurs. The improvement in performance and sense of well-being is more than accustomization; rather, it is the result of certain well defined and other more obscure, physiological adaptations.

Acclimatization to heat results at least in part from increased protection against hyperthermia since some of the initial distress of heat exposure results from the hyperthermia. However, other adaptations occur; e.g., improved cardiovascular function. The acclimatized individual is able to work in the heat with a lower body temperature, a lower heart rate and a more stable blood pressure than before acclimatization. Some increase in sweat production also may occur. Other alterations that have been implicated but are as yet still not fully proved include changes in adrenocortical activity, blood volume and venomotor tone. Regardless of which changes are most important, the improvement in performance that occurs with heat acclimatization is referable to the increased ability to maintain adequate cardiovascular function in the face of a high heat load.

The fully heat acclimatized individual, then, shows no important decrease in capacity to do physical work in the heat as compared to the amount he can do in comfort temperature conditions. This does not mean, however, that he is insensitive to the heat. Also, there may be some psychological effects even in the heat acclimatized individual including:

(1) some loss of mental initiative,

(2) decrease in accuracy particularly in poorly motivated individuals,

(3) need for greater concentration to do a given task, and

(4) possible personality change.

Climate, however, is a convenient bogeyman to be blamed for psychological and physiological difficulties whose real origin may be much more personal.

Susceptibility to Disease

Early studies of mortality during hot weather in a large American city have been supplemented recently by an analysis of records over nine and a half years, with the following conclusions:

1. Tolerance to climatic change decreases with increasing age past 25;

2. Mortality increases notably in hot summer months;

3. The mortality is at a minimum in normally hot summer months, but high peaks are superimposed by hot spells;

4. Rapid fluctuations in temperature during summer months are accompanied by a significant increase in mortality;

5. In the total period the highest single daily mortality occurred in an exceptionally hot period; and

6. Temperature is the most significant environmental factor in summer mortality.

Taken together with previously familiar evidence, this places the responsibility for increased mortality firmly on temperature fluctuations, and exceptionally hot periods, but absolves continued "normal" heat. It would seem that attempts to control atmospheric conditions should be directed at the rather exceptional peak conditions, and that extension of such controls to lower degrees of heat might not be only unnecessary, but even undesirable, in that it would impose rapid fluctuations upon those who have to alternate between conditioned and natural environments.

MEASUREMENT OF THE THERMAL ENVIRONMENT

Psychrometry

Dry air is a mixture of 21% oxygen and 78% nitrogen, with the remaining 1% made up of trace amounts of a number of other gases. This chemical composition of air remains quite constant in nature.

The atmosphere in which we live is not dry air, but rather, a mixture of dry air and water vapor. The moisture content of the atmosphere is quite variable, and it affects us and the things around us in many ways. For example, humidity is one of the factors which determines human comfort, it causes marked physical changes in the properties of hygroscopic material, and it exerts an important influence on the rate of corrosion of steel.

As a first step in the discussion of psychrometry, some of the terms to be used should be defined:

Dry-bulb temperature. The temperature of a gas indicated by an accurate thermometer after correction for radiation.

Wet-bulb temperature. The temperature at which water, by evaporating into the air, can bring the air to saturation adiabatically at the same temperature. It is the temperature indicated by a wet-bulb psychrometer when properly used and shielded from radiation effect.

Dew-point temperature. The temperature corresponding to saturation (100 percent relative humidity) for a given absolute humidity at constant pressure.

Humidity. Water vapor within a space.

Absolute humidity. Weight of water vapor per unit volume.

Specific humidity or humidity ratio. The weight of water vapor (steam) associated with one pound of dry air.

Relative humidity. Approximately, it is the ratio of the vapor pressure or density of the water vapor in the air, to the saturation pressure or density, respectively, of water vapor at the same temperature.

Vapor pressure. The pressure exerted by the water vapor.

Enthalpy. The thermodynamic property of a substance defined as the sum of its internal energy plus the quantity $\frac{PV}{J}$

where P = pressure of the fluid
V = volume of the fluid
J = mechanical equivalent heat

Enthalpy was formerly called "total heat" or "heat content."

Sensible heat. Heat which is associated with a change in temperature.

Latent heat. The change of enthalpy during a change of state. The heat given up by a vapor condensing to liquid at the same temperature is an example of latent heat.

The properties of moist air may be shown graphically on a psychrometric chart, one form of which is shown in Figure 1. The several scales and sets of lines on the chart may be described briefly as follows:

1. The dry-bulb lines are vertical, and the dry-bulb temperature scale is along the bottom of the chart.
2. The wet-bulb lines slope down to the right, and the wet-bulb temperature scale is along the saturation (100% relative humidity) curve.
3. The curved lines show relative humidity.

Prepared by: Austin F. Henschel, Ph.D.,
Clark M. Humphreys and
Kenneth J. Kronoveter

4. Horizontal lines are lines of constant specific humidity, dew-point temperature, and vapor pressure. Specific humidity (grains per pound of dry air) and vapor pressure (mm of mercury) are read on the two scales along the right side of the chart. Dew-point temperature is read from the same scale as the wet-bulb temperature.

If any two properties of moist air are known which will establish an intersection of lines on the chart, all of the other properties may be determined readily.

Example

Given: Air at 100°F dry-bulb and 75°F wet-bulb temperature.
Find its other properties.

Locate the condition point on the psychrometric chart at the intersection of the 100°F dry-bulb and 75°F wet-bulb lines. From this point, move as indicated by the insert drawing on the chart (top left) to find

Relative humidity	= 31%
Specific humidity	= 91 gr/lb
Dew-point temperature	= 65°F
Vapor pressure	= 15.2 mm of Hg

Figure 1 does not show the enthalpy of air-water vapor mixtures. Lines of constant enthalpy are almost parallel to the wet-bulb lines, and on some charts they are considered to be identical. The process of adiabatic saturation would move upward and to the left along a wet-bulb line; the moisture content of the air would increase and the dry-bulb temperature would increase.

A process involving the addition or removal of sensible heat without change in specific humidity would move horizontally to the right or left, respectively, on the chart. A process involving an increase or decrease in specific humidity without a change in sensible heat would move vertically upward or downward, respectively.

There is a general relationship existing between dry-bulb, wet-bulb, and dew-point temperatures which might be noted. At any point on the saturation (100% relative humidity) curve, all three of these temperatures are the same. At any point to the right of the saturation curve, the dry-bulb is always the highest, the dew-point is the lowest, and the wet-bulb temperature is intermediate.

The chart in Figure 1 was selected primarily for illustrating its use. Temperature lines are drawn only at 5 degree intervals. For any design work it is suggested that a larger chart with temperature lines at one degree intervals be used.

For a much more thorough discussion of the subject, see Reference 1. This reference also contains tabular data on the thermodynamic properties of moist air, and of water at very low pressures.

Thermal Radiation

Whenever temperature differences exist between two or more bodies and create energy exchanges between these bodies, it is said that heat is transferred. Net heat transfer is always from the body (or object) of higher temperature to the body of lower temperature and occurs via one or more of three essentially different mechanisms:

1. Conduction - transfer of heat by intermolecular action without gross displacement of the particles.

2. Convection - transmission of heat by forced or natural motion of a fluid.

3. Radiation - transmission of energy by means of electromagnetic waves.

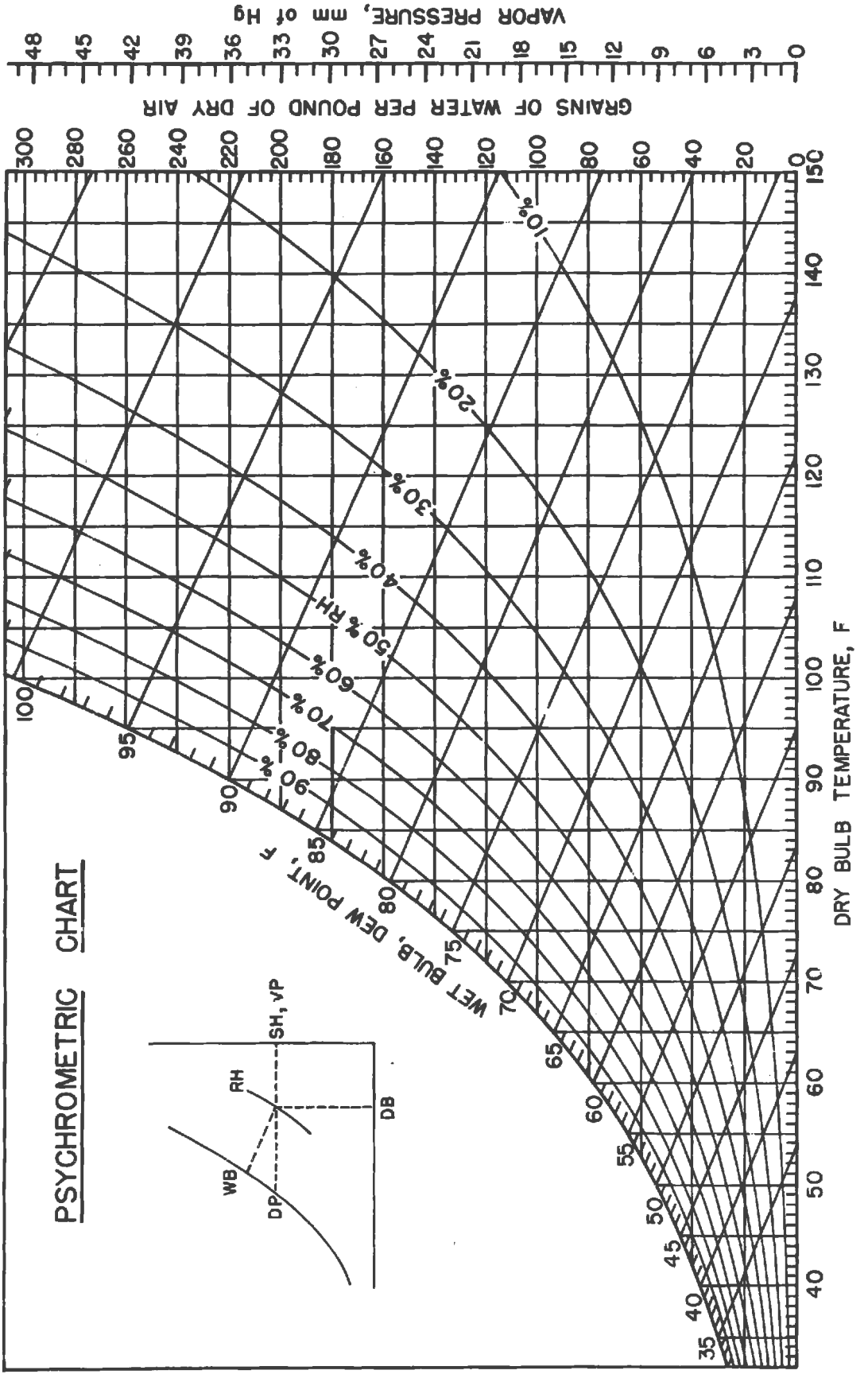


Figure 1. - Psychrometric Chart and Vapor Pressure Nomograph.

Thermal radiation is one of many forms of radiant energy. Radiant energy is exchanged continuously by all bodies and may be considered to be a form of wave motion, known as electromagnetic phenomenon. This type of wave motion should not be confused with such mechanical wave motions as sound waves. Radiant energy passes through air without appreciably heating it and will warm the surface upon which it falls. Radiant energy also will pass through a vacuum and certain other media with a velocity dependent upon the particular medium.

Most thermal radiation encountered in industry is of a longer wave length than visible light and thus lies in the infrared region of the electromagnetic spectrum. The thermal distinction between the various types of electromagnetic radiation is rather indefinite because radiation of all wave lengths will heat bodies.

A "black body" absorbs all of the radiant heat incident upon its surface and reflects none. Most materials reflect a fraction (reflectivity) and absorb the remaining fraction of the incident energy (absorptivity). The radiating power of a black body is proportional to the fourth power of its absolute temperature. Ordinary surfaces radiate less energy than does a black body at the same temperature. The ratio of the radiating power of the surface to that of a black body is its emissivity. The emissivity of a surface is equal exactly to its absorptivity. In other words, a good reflector (or a poor absorber) has a low radiating power. Table I in Section C-3 shows approximate emissivity and reflectivity values for some common surfaces.

A basic equation for radiant heat exchange between two surfaces is:

$$\frac{qr}{A_1} = F_A F_E \sigma(T_1^4 - T_2^4)$$

where:

$\frac{qr}{A_1}$ = rate of exchange, BTU/(hr)(ft^2)

A_1 = area of surface 1, ft^2

F_A = the configuration factor which is dimensionless and ≤ 1 ($F_A = 1$ for infinite parallel planes, infinite concentric cylinders and completely enclosed bodies).

F_E = the emissivity factor which is dimensionless and ≤ 1 (for infinite parallel planes, or for a completely enclosed body which is large compared to the enclosing body)

$$F_E = \frac{1}{\frac{1}{E_1} + \frac{1}{E_2} = 1}$$

where E_1 = emissivity of surface 1

E_2 = emissivity of surface 2

σ = Stefan-Boltzmann radiation constant = 1714×10^{-12} BTU/$(hr)(ft^2)(deg.\ R)^4$

T = absolute temperature, deg. R

Although this equation looks simple enough, evaluation of F_A and F_E can be quite difficult. Equations for evaluating these constants for a number of common radiation systems are given in various references(2,3,4,5,6).

Mean radiant temperature (MRT) is a useful concept when determining radiant heat exchanges in non-uniform environments. MRT may be defined as the temperature of a uniform black enclosure in which a solid body or occupant would exchange the same amount of radiant heat as in the existing non-uniform environment. The MRT in a non-uniform environment is not uniform, but varies from one location to another in the space.

Instrumentation and Techniques for Measuring the Thermal Environment

Human comfort is influenced by the following environmental factors:

1. Ambient air temperature (dry-bulb)
2. Humidity of the ambient air
3. Mean radiant temperature
4. Air velocity

All four of these factors must be measured in any assessment of the thermal environment. The measurement of these variables at some convenient point within a space usually presents little difficulty provided conditions in the space are reasonably uniform and thermal radiation is not excessive. In hot industries the dry-bulb and MRT may vary widely from place to place, and near hot surfaces the radiation intensity is often high enough to complicate the accurate determination of air temperatures.

Measurement of Dry-Bulb Temperatures

Dry-bulb temperatures usually are measured with an ordinary liquid-in-glass thermometer. Thermocouples, thermistors, or resistance thermometers also may be used if available. To obtain accurate results it is necessary to shield[7] the sensing element from radiation from nearby surfaces which are either appreciably hotter or cooler than the air.

Measurement of Humidity

The simplest and most common type of instrument used to determine the humidity of the air is the sling psychrometer. This instrument consists of a frame on which two thermometers are mounted. At one end of the frame is a handle by which the device may be whirled through the air, to obtain an air velocity of 1,000 fpm or more across the bulbs of the thermometers. A cloth wick covers the bulb of one of the thermometers and is wetted with distilled water prior to whirling the psychrometer. Due to evaporation from the wick, the wet-bulb thermometer will indicate a lower temperature than the dry-bulb thermometer, the difference being known as the wet-bulb depression. The whirling of the psychrometer is interrupted at intervals to read the thermometers, and is continued until both thermometer readings become steady. By use of a psychrometric chart or table the relative humidity and other thermodynamic properties of the air can be established from the dry-bulb and wet-bulb temperatures thus obtained. Determinations of wet-bulb temperatures below 32°F require certain precautions which are discussed in reference[8].

Often a small fan or blower is used to produce air movements across stationary thermometers. This arrangement is termed an aspirated psychrometer and several commercial models are available. Thermistors or thermocouples can be used as the sensing elements for dry- and wet-bulb temperatures, and by "feeding" the outputs to suitable recorders, a record can be made of temperature fluctuations.

Relative humidity can be determined with good accuracy with a dry- and wet-bulb type psychrometer if proper techniques are followed. Otherwise results can be grossly in error. A study of a psychrometric chart or table will show that a rather small error in the reading of either the dry- or wet-bulb temperature can result in a rather large error in the relative humidity. A matched pair of accurate thermometers, preferably with ½ degree graduations, should be used.

Generally, it has been assumed that a sling psychrometer is not affected by radiation because of the high velocity air flow over the bulbs. In a recent study[9] in a hot industrial plant, dry- and wet-bulb temperatures obtained with a sling psychrometer were found to be seriously in error because of radiation.

Where radiant heat is likely to be encountered, it is recommended that a properly shielded, aspirated psychrometer, rather than a sling psychrometer, be used.

Some of the other precautions[10] to be taken in using a sling or aspirated psychrometer are listed below:

1. The wick must be clean and in good contact with the bulb.
2. The wick should extend about one bulb length above the bulb to prevent errors due to stem conduction.
3. Use only distilled water on wick.
4. When the wet-bulb depression is large, water at approximately wet-bulb temperature should be applied to the wick.

Measurement of Mean Radiant Temperature

The globe thermometer[8,11] is often used to measure the mean radiant temperature. A conventional globe thermometer consists of a 6-inch hollow, copper sphere painted on the outside and inside with a matte black finish. For ordinary plant measurements a common copper toilet float painted a flat black gives a sufficiently accurate reading, and is cheaper and easier to obtain. A thermocouple, thermistor, or thermometer bulb is fixed at the center of the sphere with the wires or stem protruding to the outside through a sealed opening. The temperature of the air inside the globe at equilibrium is the result of a balance between the heat gained or lost by radiation and the heat gained or lost by convection. In terms of heat transfer relationships:

$$MRT^4 = T_g^4 + 0.103 \times 10^8 \sqrt{V}\,(tg-ta)$$

where:

MRT = mean radiant temperature, deg. R

T_g = globe temperature, deg. R

V = air velocity at the globe, ft/min.

tg = globe temperature, deg. F.

ta = ambient air temperature, deg. F.

The MRT may be approximated by the simplified equation:

$$MRT = tg + C\sqrt{V}\,(tg-ta)$$

where:

MRT = mean radiant temperature, deg. F.

C = a convection coefficient = 0.17 for a 6" globe.

V, tg, and ta are as defined above.

It will be noted from the above equations that the air temperature and air velocity around the globe must be determined. It is the determination of this velocity which presents the greatest problem in the use of a globe thermometer. A period of 15 to 25 minutes usually is required for the globe to reach equilibrium.

A recent development is the two-sphere radiometer[12] which may be used to measure the MRT. Two small spheres, approximately two inches in diameter, one gold plated and the other black, are heated electrically to the same temperature. Since there is no difference in convection losses the difference in energy input required by the two spheres is determined, and from this difference the MRT may be calculated or read from calibration curves.

The MRT at any point in a space can be calculated if the temperature, emissivity, and area of all surfaces, and their orientation with respect to the point of calculation are known. Surface temperatures for such a calculation can be obtained by means of thermocouples, or suitable radiometers or pyrometers.

A somewhat simplified method for calculating the radiant heat exchange between the point of interest and individual

segments of its surroundings is given in reference 13.

Measurement of Air Velocity

Measurement of low air velocity is always difficult. In addition to being low, the air velocity in an open room is usually quite random in direction, and a non-directional instrument should be used for its measurement. Thermal anemometers of the heated-thermocouple, hot-wire, or heated-thermometer types are best suited for such measurements. They will produce useful data if they are maintained in calibration, and the user understands their operation and limitations[14].

The above discussion of instrumentation is far from complete. For further information consult references 8 and 15, or see the manufacturers' literature.

Assessment of Heat Stress

There have been many attempts to devise a system to express as a single number, or at least as a semi-quantitative expression, the significance of a given set of environmental conditions. Efforts have centered around the thermal aspects of the environment because it was thought that the well-known physical laws of heat exchange could be applied to physiological heat exchange. It seemed reasonable to concentrate on the thermal aspects but it is clear that a formulation cannot be simple and accurate at the same time, and a workable scheme must necessarily be something of a compromise. Variables that must be considered include human factors as well as environmental conditions and in addition, physiological effects must be considered. Clearly, it is impossible or at least impracticable to set up a scheme giving full expression to all the variables involved even if the necessary quantitative data were available. Some selection must be made and some condensations effected if the results are to be comprehensible.

Attempts to provide predictive schemes fall roughly into three groups:

1. Instrumental methods, whereby a device was sought which would respond to the major environmental variables conjointly and in a fashion comparable to man's.
2. Physiological methods, at the other extreme, were based on charts or tables prepared from measured human responses, from which a given combination of environmental conditions could be evaluated.
3. More attention has been given recently to formulation methods, which seek to base evaluation on the difficulty of maintaining a thermal balance in a given environment. Some of the better known examples will be described briefly.

Katathermometer

In its earliest and simplest form[16] it was merely a thermometer with alcohol as the liquid, consisting of a bulb measuring 1.5 by 0.75 inches and two marks engraved on the stem corresponding to 100 and 95°F respectively. The time taken for it to cool between these two marks, after it had been heated to a higher temperature, was measured. From this time, and the amount of heat lost from the katathermometer in cooling (the "factor" engraved on the stem), the "cooling power" of the air was determined. When the kata was used dry, the cooling power indicated the combined effect of air temperature, radiant heat, and air movement; when the bulb was wrapped with a wet sock, the cooling power included the effect of humidity as well.

Because the shape, dimensions, and thermal properties of the kata differed markedly from those of the human body, it fell into disuse. A silvered version in three ranges (100-95; 130-125; 150-145°F) has been used frequently to measure turbulent air movements.

Wet Bulb-Globe Thermometer

Minard and coworkers[17,18] developed a simple index which combines the effects of temperature, humidity, radiant heat, and air movement. The index is obtained by combining dry-bulb temperature (DBT), wet-bulb temperature (WBT), and conventional dry globe thermometer temperature (GBT) in the following fashion:

For indoor use: WBGT = .7 (WBT) + .3 (GBT)

For outdoor use: WBGT = .7 (WBT) + .2 (GBT) + .1 (DBT)

A comprehensive table of evaluations has not been published, but the following practice has been reported for U.S. Marine Corps training centers:

WBGT 82 to 84.9 - Green flag. Alert for possible curtailment of drill.

WBGT 85 to 87.9 - Yellow flag. Active drill curtailed for new (less than four weeks) recruits.

WBGT 88 to 89.9 - Red flag. Active drill stopped for all recruits, except those in last quarter of 12 weeks training may continue routine training.

WBGT 90 and over - All training stopped.

It has been found that the globe thermometer readings in gusty wind, or in the vicinity of hot surfaces of limited size are difficult to interpret, so that it would not be expected that the WBGT would be very accurate in many industrial situations. The significance of a given value for WBGT needs to be considered in terms of the activity of the person exposed, his clothing, degree of acclimatization, etc.

Effective Temperature

The earliest and most widely known scheme for indicating the thermal significance of environments based upon physiological reactions, is the "Effective Temperature" scheme, developed by a research team in the American Society of Heating and Ventilating Engineers[19]. Test subjects were exposed serially to atmospheres with different temperatures, humidities, and air movements, and asked to rate their comparative sensations of warmth or coolness. From a large number of records two nomograms were drawn up indicating the combinations which produced comparable sensations: (a) on normally clothed persons, and (b) on persons stripped to the waist. The latter is seldom used. For simplicity of understanding, the significance of any combination of environmental conditions is expressed as the temperature of a still, saturated atmosphere which would produce the same effect. For example, normally clothed persons exposed to a dry-bulb temperature of 76°F, a wet-bulb temperature of 62°F, and an air movement of 100 ft/min, on the average expressed the same sensations as when they were exposed to "still" air (actually an air velocity of 25 ft/min) with both dry- and wet-bulb temperatures at 69°F. The "effective temperature" of the former atmosphere is, therefore, said to be 69°F. From the appropriate nomogram, (Figure 2), the "effective temperature" for any combination of dry-bulb temperature, wet-bulb temperature, and air movement can be determined.

This, like any other scheme, has certain limitations:

1. The basic observations were made on a specific group of subjects -- healthy, young, white men and women, living under the current American conditions of climate, housing, clothing, etc. The results are not necessarily representative of other groups and should not be applied with abandon.

2. The observations relate only to sedentary conditions, although some later modifications were drawn up to make the scheme applicable to greater activities.

Instructions for use: Stretch a thread or place a rule to join dry-bulb and wet-bulb temperatures. Note where this cuts appropriate air velocity line and read effective temperature at this point on grid lines.

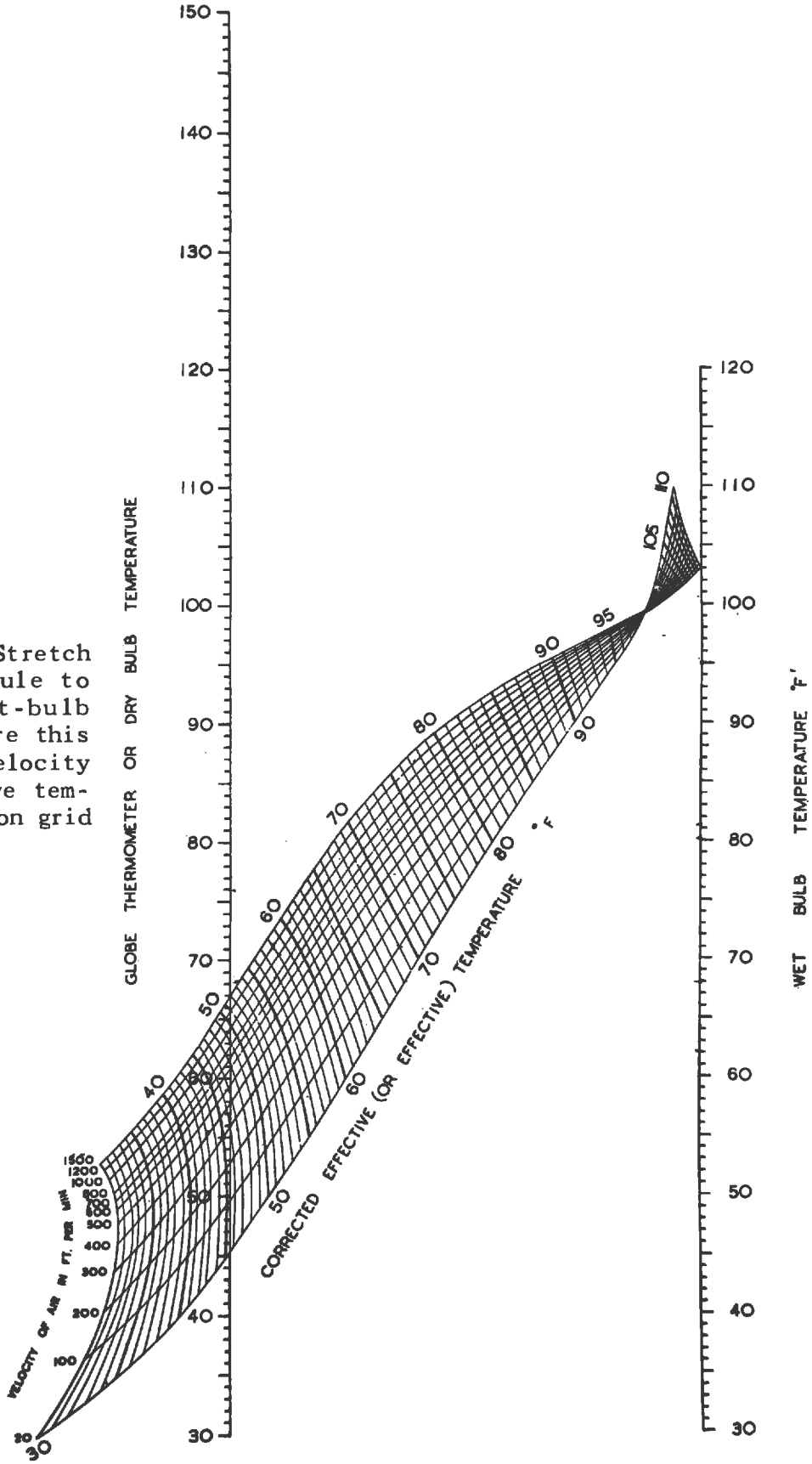


Figure 2. - Chart Showing Normal Scale of Corrected Effective (or Effective) Temperature.

3. The scheme applies only to persons clad as described. Heavy clothing could be expected to reduce the effect of air movement still further.

4. The original scheme applied only to situations in which the mean black body temperature of the surroundings was substantially the same as the air temperature. It has been shown, however, that the scheme can continue to be used where the temperatures of the surrounding surfaces differ from the air temperature, provided that they are fairly uniform, and provided further that if the globe thermometer temperature is substituted for the air temperature.

5. It does not apply at air movements below 25 ft/min.

6. It is not reliable at the upper end of the scale -- E.T. above 90°F -- since sensations of heat cease to be very good guides under conditions as hot as this.

7. The scheme in its original form gives too much weight to changes in humidity at the lower end of the scale.

8. The scheme provides no scale for evaluating the physiological significance of stresses above or below the comfort zone.

EP Index of Physiological Effects

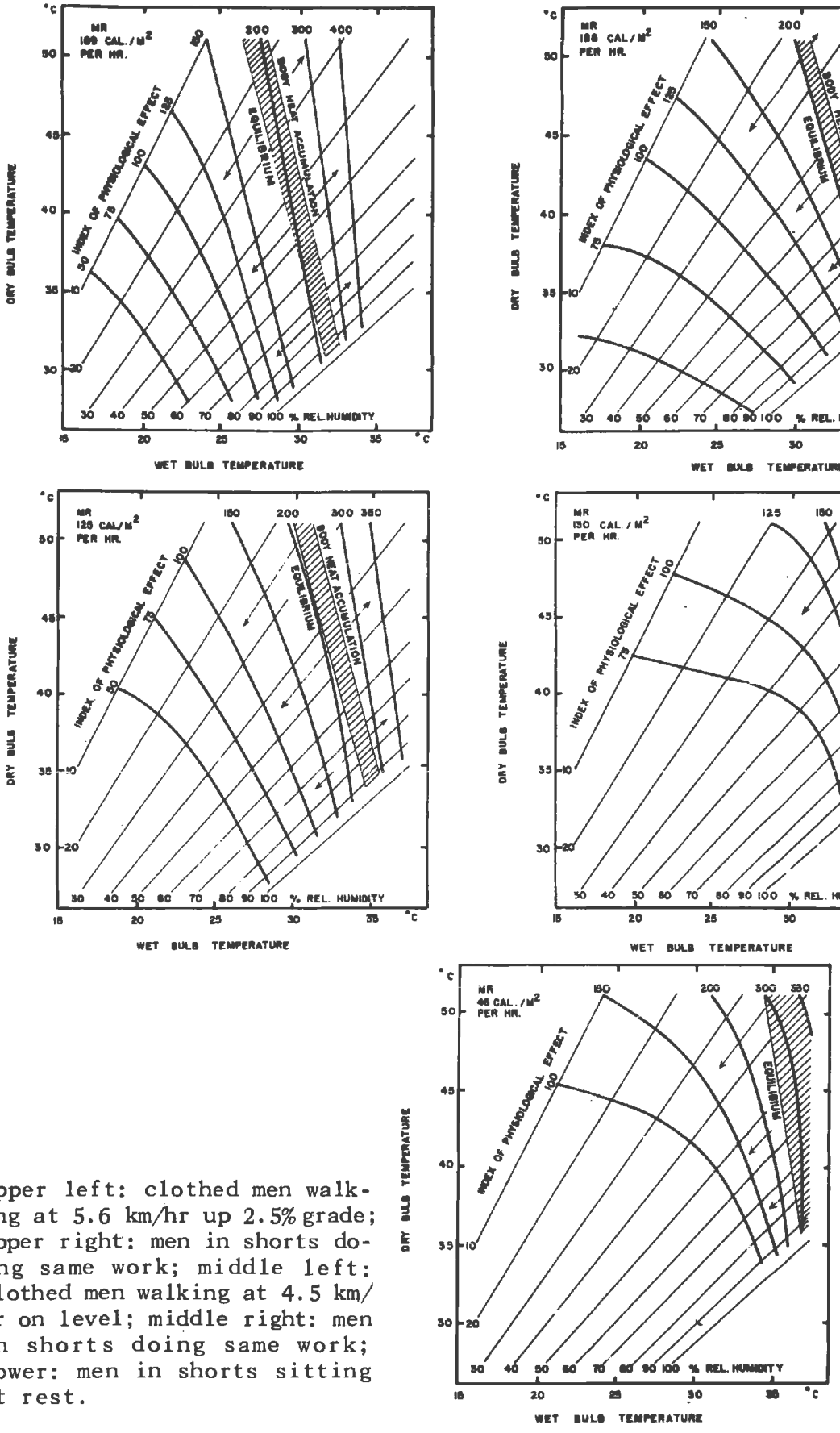
Robinson and coworkers[20] devised an index of environmental stress based on the combined response of rectal temperature, pulse rate, skin temperature, and sweat rate. The concept of relative strain, that is, the deviation from normal as compared with the maximum deviation that the body can tolerate without breaking down, was used here for the first time, although not clearly labelled as such. The relative strains for pulse rate, skin temperature, rectal temperature, and sweat loss were established for a variety of conditions, and the results expressed on dry-bulb, wet-bulb temperature grids (Figure 3). Unfortunately, this scheme, in common with most physiological methods of prediction, does not furnish any basis for extrapolation to sets of conditions other than those used in the original experiments.

Predicted Four Hour Sweat Rate (P_4SR)

Researchers in England[21] developed a nomogram whereby the significance of different atmospheric conditions is expressed in terms of the sweat loss they induce. With a later modification it was made applicable to various combinations of clothing and levels of activity (Figure 4). In using this chart, locate point of intersection of wet-bulb temperature and air velocity lines at right side of diagram. Join this to the globe or dry-bulb temperature reading on the left side of the diagram. The point at which this transverse line cuts the air velocity line in the center of the diagram indicates the basic four-hour sweat rate. The nomogram may be used for higher rates of work by adding to the wet-bulb temperature reading the amount indicated by the small chart at top left. For conditions which are hot enough to induce moderate sweating, but not sufficiently severe to overtax the sweating capability, the index works fairly well. Care is necessary, however, in applying it to people who differ markedly in training or acclimatization from those used by the authors.

Heat Stress Index (HSI)

Belding and Hatch[22] explicitly tackled the concepts of stress and strain. On the former, they reported: "Heat stress is considered to exist whenever, despite vasomotor adjustment, metabolic heat production exceeds the combined losses by radiation and convection." Strain they regarded as the probable consequence of exposure to stress, and by a sequence of reasoning, hit upon expressing strain as the ratio between:



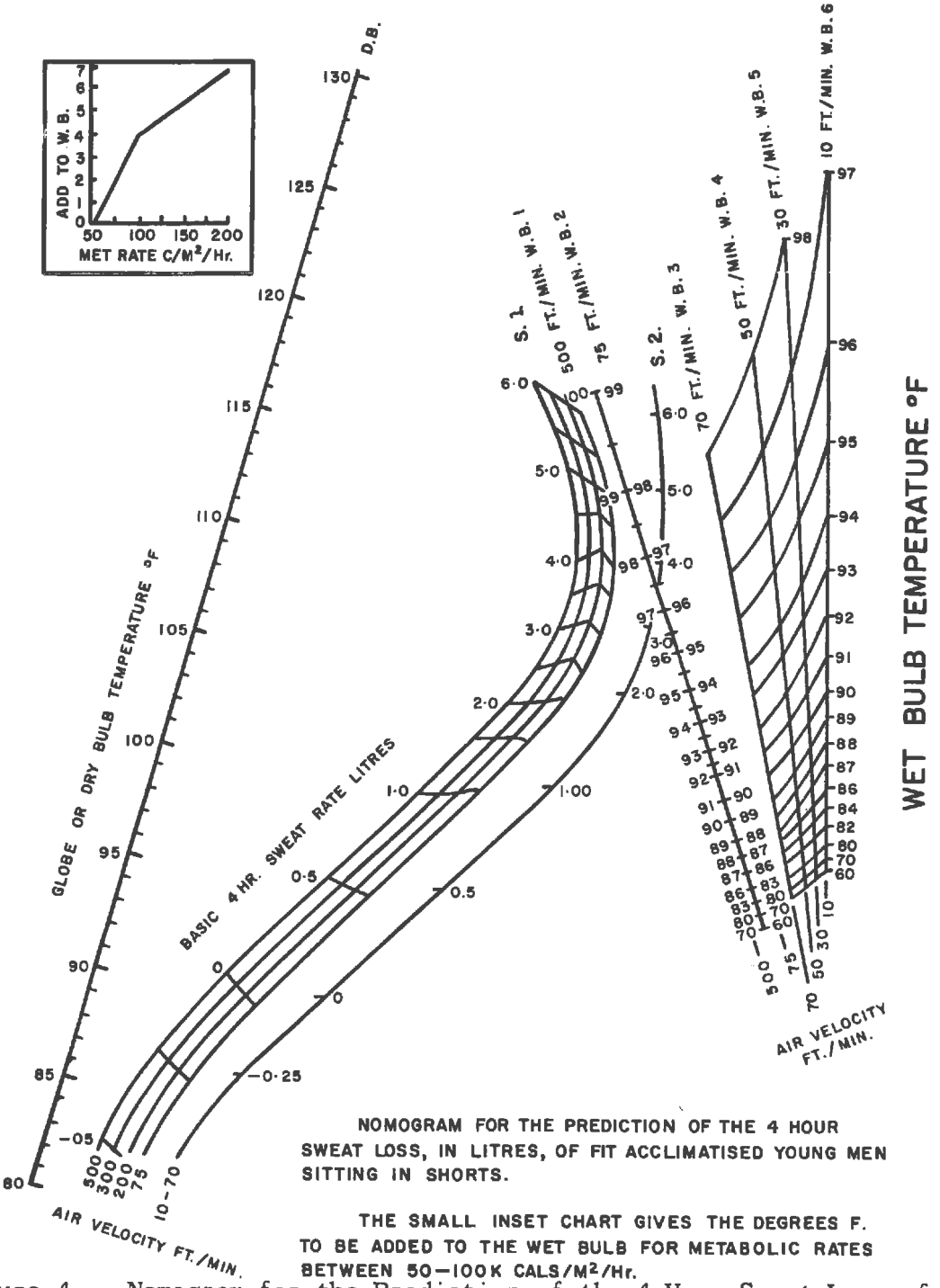
Upper left: clothed men walking at 5.6 km/hr up 2.5% grade; upper right: men in shorts doing same work; middle left: clothed men walking at 4.5 km/hr on level; middle right: men in shorts doing same work; lower: men in shorts sitting at rest.

Figure 3. - EP Index of Physiological Effect of Environmental Conditions.

(a) the rate at which evaporative cooling is needed to balance the stress, and (b) the maximum rate at which evaporative cooling can be maintained in that particular environment.

Strain = evaporative cooling required ÷ maximum evaporative cooling possible.

This scheme was a distinct advance over previous proposals in expressing the unmeasurable strain as a dimensionless ratio between definable quantities. It is unsatisfactory in that it does not include the effects of clothing and ignores physiological reactions other than sweating. Attempts have been made



NOMOGRAM FOR THE PREDICTION OF THE 4 HOUR SWEAT LOSS, IN LITRES, OF FIT ACCLIMATISED YOUNG MEN SITTING IN SHORTS.

THE SMALL INSET CHART GIVES THE DEGREES F. TO BE ADDED TO THE WET BULB FOR METABOLIC RATES BETWEEN 50—100K CALS/M²/Hr.

Figure 4. - Nomogram for the Prediction of the 4-Hour Sweat Loss of Fit, Acclimatized Young Men, Sitting in Shorts.

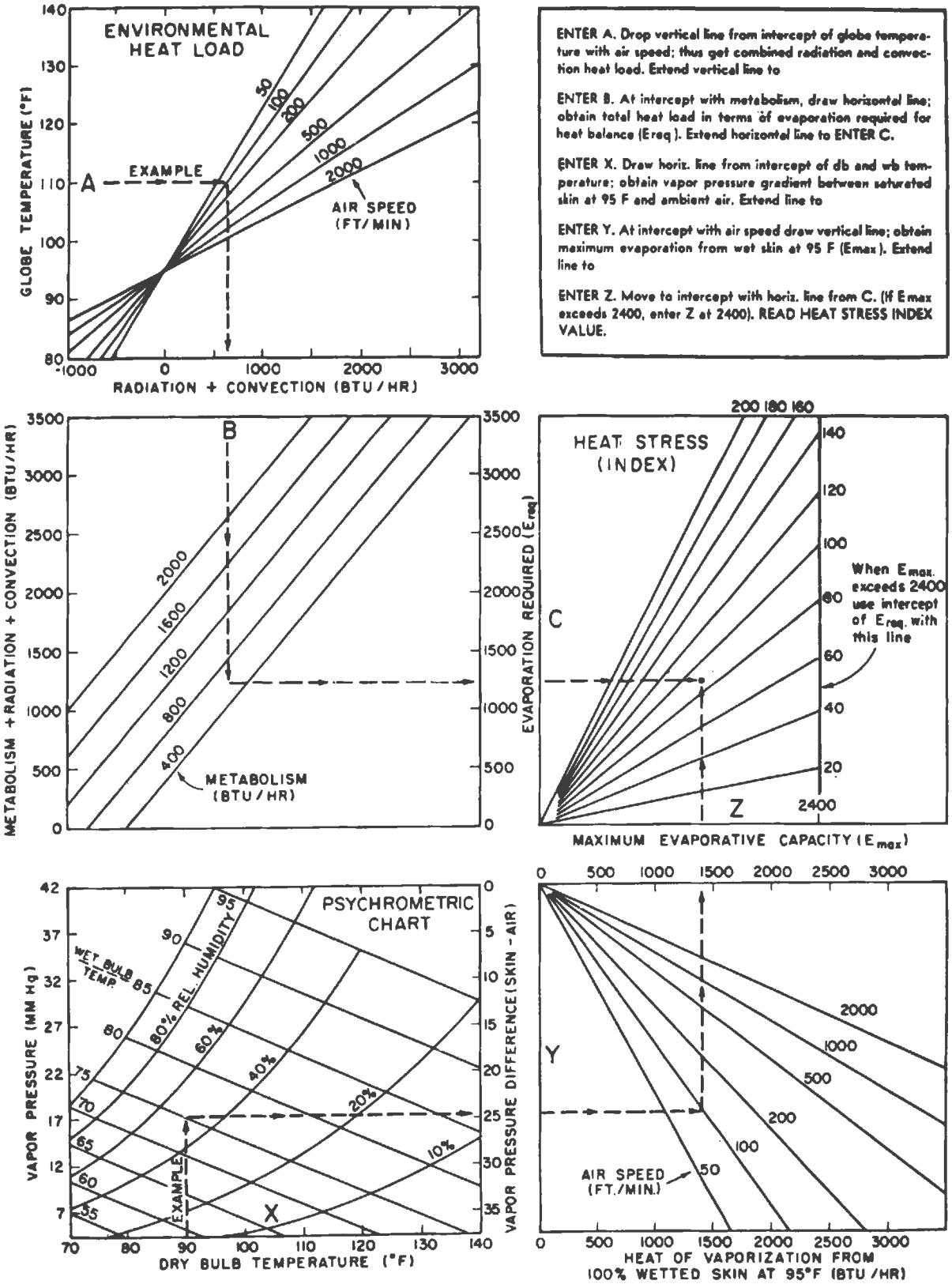


Figure 5. - Flow Charts for Determining Heat Stress Index.

Table 1. - Evaluation of Values in Belding and Hatch H.S.I.

Index of Heat Stress	Physiological and Hygienic Implications of 8-hr Exposures to Various Heat Stresses
-20 -10	Mild cold strain. This condition frequently exists in areas where men recover from exposure to heat.
0	No thermal strain.
+10 20 30	Mild to moderate heat strain. Where a job involves higher intellectual functions, dexterity, or alertness, subtle to substantial decrements in performance may be expected. In performance of heavy physical work, little decrement expected unless ability of individuals to perform such work under no thermal stress is marginal.
40 50 60	Severe heat strain, involving a threat to health unless men are physically fit. Break-in period required for men not previously acclimatized. Some decrement in performance of physical work is to be expected. Medical selection of personnel desirable because these conditions are unsuitable for those with cardiovascular or respiratory impairment or with chronic dermatitis. These working conditions are also unsuitable for activities requiring sustained mental effort.
70 80 90	Very severe heat strain. Only a small percentage of the population may be expected to qualify for this work. Personnel should be selected (a) by medical examination, and (b) by trial on the job (after acclimatization). Special measures are needed to assure adequate water and salt intake. Amelioration of working conditions by any feasible means is highly desirable, and may be expected to decrease the health hazard while increasing efficiency on the job. Slight "indisposition" which in most jobs would be insufficient to affect performance may render workers unfit for this exposure.
100	The maximum strain tolerated daily by fit, acclimatized young men.

recently[23] to meet some of these objections but practical application awaits further development.

The following example illustrates how the chart is used: Globe temperature 110°F, dry-bulb temperature 90°F, wet-bulb temperature 75°F, air speed 100 ft/min, metabolism 600 BTU/hr. For solution follow broken lines. HSI = 90. Table 1 lists the physiological and hygienic implications of exposures to the various heat stress indices.

Relative Strain Index

The evaluation of a thermal environment as a dimensionless ratio between definable quantities has been an attractive concept but ideas have differed on what should go into the ratio. The EP index used the observed responses (pulse, temperature, sweat) in comparison with the maximum response that the body could make without breaking down. The ratio of the opportunity for heat loss to the heat produced by metabolism has

also been suggested. Others have sought to express strain as a ratio between the threatened rate of heat gain and an empirically derived expression of the opportunity for evaporative compensation. The HSI is based on the ratio between the rate at which evaporative cooling is needed to balance the stress and the maximum rate at which evaporative cooling can be maintained in a particular environment. This resulted in an index which expressed the concept of strain as a deformation resulting from stress. It departed from the physical definition of strain in comparing the extent of deformation with the maximum deformation possible without system breakdown instead of with the normal dimensions of the system.

However, it is the ratio of deformation produced to the maximum permissible deformation that is desired. The conflict with the physical practice can be resolved if the nomenclature is changed somewhat and the ratio is termed "relative strain." A relative strain index has been designed for application to the specific circumstances that might ensue in an enclosed space such as a civilian defense fall-out shelter. To extend its applicability to industrial situations will require modifications - unfortunately the data upon which the modifications could be based are at present far from acceptable.

References

1. ASHRAE Guide and Data Book, Fundamentals and Equipment, Chap. 3. American Society of Heating, Refrigerating and Air-conditioning Engineers, Inc., New York, 1963.

2. ASHRAE Guide and Data Book, Fundamentals and Equipment, Chap. 4. American Society of Heating, Refrigerating and Air-conditioning Engineers, Inc., New York, 1963.

3. Brown, A. I.; and S. M. Marco: Introduction to Heat Transfer, 3rd Ed. McGraw-Hill Book Co., Inc., New York, 1958.

4. Hutchinson, F. W.: Industrial Heat Transfer. The Industrial Press, New York, 1952.

5. McAdams, W. H.: Heat Transmission, 3rd Ed. McGraw-Hill Book Co., Inc., New York, 1954.

6. Jakob, M.: Heat Transfer, Vol. II. J. Wiley and Sons, Inc., New York, 1957.

7. Parmalee, G.V.; and R.G. Huebscher: The Shielding of Thermocouples from the Effects of Radiation. ASHVE Transactions, 52:183 (1946).

8. ASHRAE Guide and Data Book, Fundamentals and Equipment, Chap. 16. American Society of Heating, Refrigerating and Air-conditioning Engineers, Inc., New York, 1963.

9. Humphreys, C. M.; and K. J. Kronoveter: Measurement of Environmental Conditions in Hot Industries; to be published.

10. Technical Bulletin, No. 1. Hygrodynamics, Inc., 949 Selim Road, Silver Springs, Maryland.

11. Bedford, T.; and C. G. Warner: The Globe Thermometer in Studies of Heating and Ventilating. J.Hygiene, 34:52 (1934).

12. Sutton, D.J.; and P.F. McNall, Jr.: A Two-Sphere Radiometer. ASHVE Transactions, 60:297 (1954).

13. Longley, M. Y.; Harris, R. L.; and D.H.K. Lee: Calculation of Complex Radiant Heat Load from Surrounding Radiator Surface Temperatures.

J.Amer. Ind. Hyg. Assn., 24:103-112 (March-April, (1963).

14. Tuve, G.L.; Wright, D.K.; and L. J. Siegel: The Use of Air Velocity Meters. ASHVE Transactions, 45:645 (1939).

15. Encyclopedia of Instrumentation for Industrial Hygiene. Publications Distribution Service, University of Michigan, Ann Arbor, Michigan, 1956.

16. Hargood - Ash, D. et al.: The Katathermometer in Studies of Body Heat and Efficiency. Med. Res. Counc. (U.K.), Spec. Rept. Series, 73: 1923.

17. Yaglou, C. P.; and D. Minard: Control of Heat Casualties at Military Training Centers. Arch. Indust. Health, 16:302-316 (1957).

18. Minard, D.: Prevention of Heat Casualties in Marine Corps Recruits. Milit. Med. 126: 261-272 (1961).

19. Houghten, F.C.; and C.P. Yagloglou: Determining Lines of Equal Comfort. J. Am. Soc. Heat. & Vent. Engrs. 29: 165-176 (1923).

20. Robinson, S.; E. S. Turrell; and S. Gerking: Physiologically Equivalent Conditions of Air Temperature and Humidity. Am. J. Physiol. 143: 21-32 (1945).

21. McArdle, B. et al.: The Prediction of the Physiological Effects of Warm and Hot Environments. Med. Res. Counc. (U.K.) RNP Rept. No. 47, p. 391 (1947).

22. Belding, H. S.; and T. F. Hatch: Index for Evaluating Heat Stress in Terms of Resulting Physiological Strains. Heat., Piping, & Air Condit. 27: 129-136 (1955).

23. Belding, H. S.; B. A. Hertig; and M. L. Riedesil: Laboratory Simulation of a Hot Industrial Job to Find Effective Heat Stress and Physiological Strain. Am. Indust. Hyg. Assoc. J., 21: 25-31 (1960).

OCCUPATIONAL DERMATOSES - THEIR RECOGNITION, CONTROL AND PREVENTION

An occupational dermatosis is defined as any inflammation or abnormality of the skin incurred directly from the working environment. The term, "dermatosis," can represent a discoloration, a blemish, a callus, a scar, an inflammatory eruption, or even a tumor. On the other hand, the term, "occupational dermatitis," refers strictly to an acute inflammatory type of occupational dermatosis.

Frequency

Occupational skin disease is the commonest industrial illness that attacks the working man. At least two-thirds of all compensated industrial disease is of dermatologic nature. These figures do not include hundreds of cases of dermatitis which are not compensated because workers do not lose time from work. The frequency of cutaneous disease in industry will vary with the nature of the plant operations and the materials being handled by the workmen. It can be safely estimated, however, that, in the majority of plants, at any given time, at least 1% of the workmen will show some form of an occupational dermatosis.

Periodically, the U. S. Public Health Service and others have assembled statistical data to ascertain the frequency of occupational diseases among the working population. In 1951, the Division of Occupational Health assembled such information from several State and Federal installations. It was shown that in 28 States and among Federal employees, there were 43,307 alleged or suspected cases of occupational diseases reported during a 12 month period. In this analysis, diseases of the skin accounted for 54.3% of all occupational diseases. In some States, the occupational diseases of the skin will account for 80% of all the reported industrial disease.

In brief, there is no need to justify the efforts of State health departments, the Public Health Service, and industry to recognize, control, and prevent these diseases. Concerted effort in this direction during the past 50 years has produced considerable information about industrial illness in general and occupational dermatoses in particular. Today, through industrial hygiene engineering and the in-plant practice of occupational medicine, industrial hazards can be recognized, and their effects upon the skin and other organs can be prevented through modern prevention and control methods.

Function of the Skin

There are numerous reasons why the skin can be injured by occupational contactants. First, the skin is the largest organ of the body and constitutes the largest surface area in close contact with foreign substances in nature as well as in the industrial environment.

Among its various functions, the skin protects, regulates body heat, receives sensations, secretes, absorbs, and manufactures pigment. Each of these physiologic actions is important in maintaining a healthy skin. Any deviation of these functions from normal can alter the health of the skin and sometimes that of the entire body.

The structure or anatomical design of the skin is protective because of its thickness, its resiliency, and the capacity of certain of its layers to inhibit the entrance of water and water-soluble chemicals. Its thickness and elasticity protect the underlying muscles, nerves, and blood vessels. Further, the thickness and color of the

Prepared by: Donald J. Birmingham, M.D.

skin afford protection against the effects of sunlight and other sources of physical energy. Body temperature is regulated by the excretion of sweat, the circulation of the blood, and the central nervous system. The blood is maintained at a relatively constant temperature, despite the fact that the body can be exposed to wide ranges of temperature variations. The sweating mechanism facilitates greatly the cooling of the overheated surface by its contribution to evaporation. At the same time, there is marked dilation of the blood vessels within the skin. Conversely, if the body is exposed to severe cold, the vessels contract to conserve heat within the body.

Many nerve endings and fibers are contained within the skin. These act as a conducting system so that the individual can differentiate between heat, cold, pain, and sense perception. The sensory perception allows one to discriminate as to whether a substance is dry or wet, thick or thin, rough or smooth, hard or soft.

The secretion of the skin occurs through the medium of sweat glands and sebaceous glands. The perspiration or sweat contains products from the body's metabolic function. Excessive or inadequate sweating can be harmful, not only to the skin, but to the general health.

The sebaceous glands, located in the midportion of the skin, manufacture an oily material called sebum. The precise physiologic function of sebum is not known, but when it is present in normal amounts it helps to offer some surface protection to the skin.

Absorption of materials through the skin usually occurs when the continuity of the skin is disrupted such as by a wound, but it also can occur by way of the hair follicle which contains the hair bulb and a portion of the hair shaft. Some substances are absorbed directly through the intact skin.

Pigmentation of the skin is a complex physiologic mechanism which occurs naturally throughout the lifetime of an individual. Sunlight and certain chemicals stimulate pigment formation, and at times its activity can be inhibited. The pigment contained in certain cells of the epidermis is constantly shed by the Keratin (horn) layer of the skin. At the same time, however, pigment serves as a screen to protect against the effects of sunlight. Figure 1 is a schematic drawing of the skin, depicting its defensive capacity against the environment.

The waxy emulsion composed of sebum, breakdown products of keratin, and sweat, impedes somewhat the entrance of water and water-soluble chemicals. Though its actual protective quality is not great, it also assists in maintaining the surface pH of the skin which is generally in the range of 4.5 to 6. This emulsion layer is easily removed by soaps, solvents, and alkalis, but it is physiologically replenished under normal conditions. Keratin, the layer of dead cells beneath the surface emulsion, is produced and shed as part of the metabolic process. It moderately resists mild acids and water and also aids in maintaining surface pH. Ultraviolet radiation causes it to thicken. Conversely, it is susceptible to the action of alkali, strong detergents, solvents, and prolonged exposure to warm water.

In the lower portion of the keratin layer is a highly important barrier which prevents the marked loss of water from the skin and the wholesale entrance of water-soluble materials. It can be altered by injury, strong chemical agents which destroy keratin, solvents, and by internal diseases which disrupt the skin. The skin itself permits the ready exchange of gases except for carbon monoxide. The follicular orifices allow the entrance of lipid-soluble chemicals, fats, and oils. The sweat glands offer little if any avenue for penetration. It is evident that the skin has its own built-in defense, yet there are a number of direct and indirect causes of occupational skin disease which can disturb this normal defense pattern.

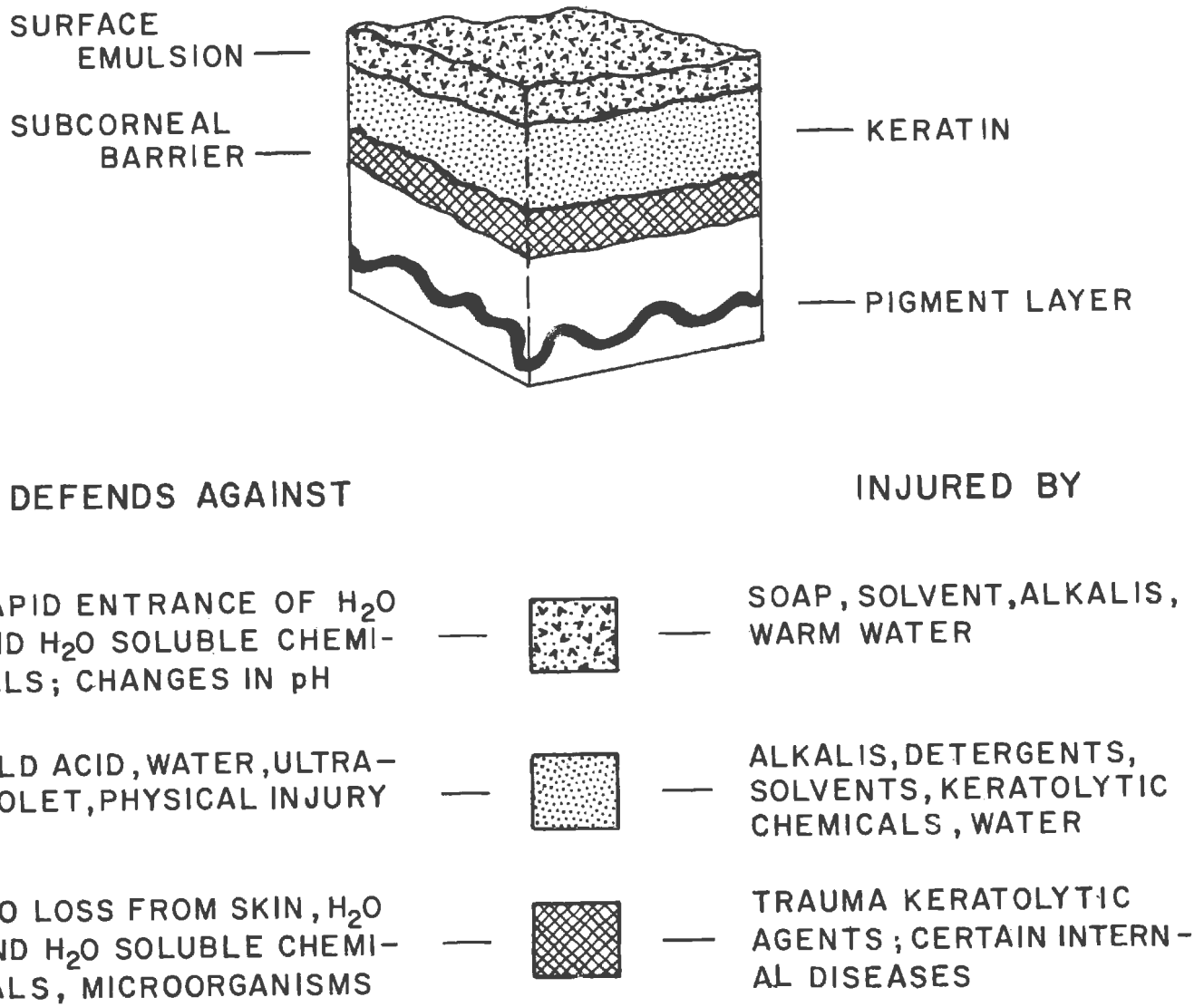


Figure 1. - Protective Mechanisms of the Skin.

Causes of Occupational Dermatitis

Indirect Causes

The indirect or predisposing factors which aid and abet the induction of an occupational skin disease are enumerated as race, age, sex, texture of the skin, perspiration, season of the year, lack of cleanliness, and the allergic state.

Race. It has often been stated that the colored skin is less susceptible to industrial materials than is the light skin. This is true in the case of sunlight and in the handling of certain chemicals, but there is also evidence which shows that the colored skin is not universally resistant to the industrial environment. The tendency toward keloid formation (severe scarring) is a well known problem.

Age. Young workers often develop occupational dermatoses more readily than do the older workers. This is not caused by any peculiar structure of their skin. Rather, it is because they tend to be less cautious and show disregard for the injurious nature of chemicals or other materials at work. When occupational skin disease attacks the older workers,

their period of recovery is sometimes unusually prolonged. Just why this is so has not been completely ascertained.

Sex. Women are as prone to develop occupational dermatitis as are the male workers. The effect of the irritant materials such as solvents contacted at work is often supplemented by cleansers, detergents, waxes, and other agents used in the household. Women tend to be clean about their work and try to avoid prolonged contact with irritant chemicals. They generally seek first aid for minor skin complaints and this may account for their dermatoses being less severe. In hazardous jobs, however, women will display as many, if not more, cases of dermatitis as will be found among an equal number of male workers.

Texture of Skin. Thin, dry skin is less able to withstand the action of solvents and detergents than is the thick, oily skin. Individuals who cannot replace readily the natural surface emulsion which is removed by solvents and detergents, soon discover they cannot handle these materials. Without added protection, dry skin is invariably irritated by solvents, oils, and alkalis. The oily skin is more capable of producing natural oils for surface protection; thus the drying action of the solvents, soaps, and detergents is lessened. Even in these individuals, however, if solvents are handled promiscuously, they will injure the skin. An inherent weakness associated with oily and hairy skin is its susceptibility to various cutting and lubricating oils. These oils collect within the hair follicles and produce an irritation which results in oil folliculitis. This can progress to a severe form known as occupational acne.

Perspiration. Excessive perspiration predisposes to an occupational dermatitis. The increased sweat in contact with the skin irritates and macerates the tissues, particularly in the armpits, the groins, and at sites of friction such as the line of the belt and the collar. A frequent sequel to excessive perspiration is prickly heat. particularly among workers exposed to high degrees of temperature.

Season of the Year. There is more occupational dermatitis noted in the warm months than during the cold season. During warm weather, workers become lax in wearing protective clothing and thereby over expose themselves to the environment. Further, warm weather predisposes to excessive perspiration.

Lack of Cleanliness. Lack of cleanliness is the greatest predisposing factor in the development of an occupational skin disease. Workmen who do not practice cleanliness permit prolonged contact between their skin and harmful materials. These same individuals wear work clothing for prolonged periods of time without laundering.

The Allergic State. Not more than 20% of all occupational skin disease can be attributed to allergy. Some people are born with a predisposition for allergic disease, as asthma, hay fever, hives, and infantile eczema. On the other hand, some individuals without any preexisting history of allergy become allergic to the materials with which they work. If all workmen became allergic to their industrial environment, it would create a most serious complication. Some workers despite having developed an occupational allergy, are able, by good control methods, personal and environmental, to continue their work. However, other workers who develop an allergy must be transferred to a less offensive type of environment.

Direct Causes

The direct causes of occupational dermatitis are: mechanical, physical, biological, plant and wood, and chemical.

Mechanical. Frictional action on the skin, pressure, and other forms of mechanical trauma can produce changes in the skin. Calluses, blisters, and pressure tumors have been seen among

workmen whose duties cause mechanical injury.

Physical. Excessive heat, cold, sunlight, artificial ultraviolet, and ionizing radiation produce harmful effects on the skin. Excessive heat and cold can cause disturbances of the blood vessels. Sunlight can be tolerated by dark complexioned individuals, whereas those with less pigment are also less tolerant of sunlight. Some workmen react peculiarly from exposure to sunlight with various eruptive lesions resulting from certain wavelengths of ultraviolet. Sunlight, accompanied by the action of certain photoreactive chemicals, for example, tars, oils, some dyes, and extracts of a number of plants, vegetables, and fruits, can lead to phototoxic or photoallergic dermatitis. Artificial ultraviolet, if sufficiently intense, will burn the skin. This is associated with radiation produced by hot metals, welding, and the plasma torch, among other applications of ultraviolet. Excessive exposure to x-ray, radium, and nuclear reactor materials produces severe injury to the skin and to the entire body.

Biologic. These effects can be primary or secondary. They can be caused by bacteria, fungi, and parasites attacking the skin or sometimes causing systemic disease. Animal handlers, packing house workers, hide handlers, kitchen employees, agricultural workers, bakers, florists, nurserymen, and laboratory technicians are among the many who can be affected by this form of occupational skin disease.

Plants and Woods. A large number of plants and woods can irritate the skin. Farmers, nurserymen, florists, wood workers, electric linemen, lumbermen, road builders, and others whose occupations allow contact with the various plants and woods can be affected. Some of these plants are also photoreactive.

Chemical. This category covers a vast number of organic and inorganic materials used in industrial plants and agriculture throughout the world. Most of the skin problems produced by chemicals occur on the basis of either primary irritation or sensitization (allergy).

The primary irritant is a substance which, if allowed to remain in contact with the skin in sufficient concentration for a sufficient length of time, will produce a demonstrable effect upon the skin at the site of contact. In short, a primary irritant will affect the skin of anybody. There are different grades of irritation, so that some irritants are absolute, for example, nitric acid, sodium hydroxide, chloride of line. Others are relative irritants and require several contacts before the skin resistance is overcome, for example, prolonged moisture, contact with soaps and mild solvents. Most primary irritants have a direct clinical action on the skin in one of the following ways:

1. Keratin Solvents. Sodium hydroxide, potassium hydroxide, and other alkali materials.

2. Dehydration. Inorganic acids, anhydrides, and alkalis withdraw fluid from the skin. As a result, the skin becomes excessively dry, cracks, and is then subject to secondary infection.

3. Fat and Oil Solvents. Organic solvents dissolve natural skin oils just as they do oily industrial soils. Excessive depletion of the skin oil leads to dryness of the skin, thickening, and sometimes severe inflammation.

4. Protein Precipitants. Arsenic and chrome, among other heavy metals, alter the chemical structure of the skin and cause ulceration, along with other forms of skin injury.

5. Oxidizers. Excessive exposure to bleaches, as the peroxides and chlorine causes disturbed function and a change in the fluid balance of the keratin layer.

6. Reducers. Certain chemicals, as salicylic acid, oxalic acid, and others in excessive concentration, chemically reduce the outer layer of the skin leading to irritation of the underlaying portions.

7. Keratin-Stimulants. Some petroleum compounds and coal tar products, certain of the chlorinated hydrocarbons, arsenic, and aniline are known to stimulate the skin to overproduce the horn layer. This may lead to new growths which assume tumor formation.

Fully 80% of all occupational skin disease is caused by contact with primary irritants. These substances injure the skin of workmen within a few hours to a few days. Deficiencies in the defense of the skin, such as dryness, excessive perspiration, and most of all, uncleanliness, facilitate the action of the primary irritant. Because a primary irritant is handled by a workman does not mean that he will necessarily develop an occupational dermatitis. Exposure to irritants can be controlled when proper precautions are taken. If a dermatitis does occur, it does not mean that the workman must necessarily change jobs, because in most instances, he can return to work if proper precautionary measures are used.

Sensitizers (allergens) cause about 20% of the occupational dermatoses but almost any chemical and many plants can produce allergic dermatitis. Poison ivy is a well known example. Defined, the sensitizer is a substance which does not cause demonstrable change in the skin on first contact, but may affect such specific changes in the skin, that after a period of incubation (five to seven days or more), further contact on the same or other parts of the body will cause a dermatitis. Thus, allergic dermatitis rarely is seen before the fifth or seventh day after a worker starts a new job, whereas primary irritant dermatitis usually occurs within a few hours or a few days. Sometimes, the worker may contact a certain chemical for months or even years before an allergic eruption takes place. Under ordinary circumstances, allergic occupational dermatoses are seen in only a few individuals in a given operation. Conversely, primary irritant chemicals generally affect several people in a work area. Materials known to act as potent sensitizing compounds are found among the dyes and dye intermediates, rubber ingredients, plastics in the unfinished stage, certain poisonous plants, and metals as nickel, chrome, and mercury.

Signs and Symptoms of Occupational Skin Disease

There are only a few instances in the diagnosis of an occupational eruption where the lesions are characteristic of a certain chemical. To make the clinical diagnosis of an occupational skin disease requires a familiarity with the disease. It involves understanding the nature of the lesion, the site of the eruption, the course of the disease, and being able to interpret correctly the results of any clinical tests, if such are found necessary. These cases are best managed by physicians familiar with occupational diseases of the skin.

Prevention of Occupational Dermatitis

Occupational dermatitis is a preventable disease. Ideally, the control programs will protect workmen along the entire production line, from the manufacture of basic chemicals to the completion of fabricated products, but it requires more than wishful thinking to diminish or eliminate occupational skin disease within a plant. The two main approaches are environmental control methods (engineering) and personal hygiene methods.

Engineering Controls

The importance of industrial hygiene engineering in providing a safe working environment needs no defense. The ideal modern factory is now constructed and

equipped with automatic machinery that protects the worker from contact with noxious chemicals from the beginning of the manufacturing process to the very end. Sealed cars or containers transport the raw materials to the plant, where they are emptied into storage bins and then cycled through retorts, filters, and other enclosed equipment. When closed processes cannot be used, collection systems can be installed to control the irritant dusts, vapors, fumes, and mists, keeping the floors, ceilings, walls, and windows clean. This is the engineer's way of protecting the workmen's health. In plants where good engineering controls are used, the prevalence of occupational dermatitis is low. It must be remembered, however, that ventilation systems are expensive and that all industrial plants cannot afford this type of control. In the United States, no more than 25% of the working population is employed in what is called "big industry" where good medical and engineering control programs are in effect. Conversely, 75% of the working population is employed in the small plants, which individually can rarely support good industrial hygiene programs. As a result, workmen in the smaller plants must rely for the greater part upon personal hygiene.

Personal Hygiene

There is no substitute for washing hands, wearing clean work clothes, and keeping clean on the job. To do this, the workmen must be furnished with adequate washing facilities and good cleansing materials. Wash stands must be well designed and conveniently located. When the wash basin is placed 200 yards from a machine or a work operation, it will be used infrequently. Washing facilities should be used at least three times a day during work, before lunch, after lunch, and at the close of the shift. In certain jobs, however, the degree of soilage may require more frequent cleansing, and in some instances, the use of showers.

Industrial hand cleansers are numerous and can be obtained as plain soap powders, abrasive powders, abrasive cakes, liquids, and the waterless cleansers. Mild industrial soaps will generally remove ordinary soils from the skin. When workmen encounter tenacious soils, however, they often use solvents and powerful abrasives which, in effect, are more harmful than the contact agents with which they work. Management's responsibility in eliminating skin disease is to provide good washing facilities, good cleansing agents, and disposable towels. The latter are more practical because they can be discarded after use. The large plant is better able to establish a complete system of environmental and personal control methods to prevent occupational dermatoses, but no industrial plant, however small, can afford to let the employees neglect personal hygiene.

Protective Clothing

There is a large selection of protective garments of rubber, plastic, leather, cotton, or other materials, depending upon what is needed for a particular job. Clothing designed to protect the skin against irritant chemicals or extreme exposures of heat, cold, and moisture is commonly used in many industrial plants. Management should provide the protective gear because better control over the wearing and servicing is facilitated. In addition, exposure of the family's clothing by way of the laundry can thus be avoided. Protective sleeves and gloves are advantageous in certain operations, but it is essential that they be selected properly. A job analysis should be done throughout the plant to ascertain where protective clothing, sleeves, or gloves are needed. Finally, it must never be forgotten that a protective sleeve or glove can constitute a real safety hazard when worn near moving gears.

Protective Ointments

At times, a barrier or protective cream

may be the best method of preventing an occupational dermatitis. As a general rule, however, this method is the least efficient way of protecting the skin. When the face cannot be covered by a shield, or gloves cannot be worn, barrier creams may be helpful. Most workmen seem to like to use protective creams, but these agents often create a false sense of security. There is no one all-purpose protective cream. If these materials are furnished to workmen, an appropriate cream capable of protecting against the noxious agent in the environment must be the one of choice. The hidden advantage attending the use of protective creams is that they must be washed off the skin.

CONTROL OF THE OCCUPATIONAL ENVIRONMENT

Introduction

Control of occupational exposures to injurious materials or conditions may be accomplished by means of one or more of the following methods, not necessarily listed in order of their importance or effectiveness:

1. Substitution of less toxicmaterials, or change of process.
2. Isolation or enclosure of operations or machinery.
3. Control at point of generation or dissemination.
4. Dilution with uncontaminated air.
5. Maintenance, housekeeping, and education.

These general principles of control apply not only to toxic materials such as chemicals and dusts, but also to many of the physical agents.

General Methods of Control

Substitution of Less Toxic Materials or Process Change

The preliminary survey (Section B-1) should have yielded a complete list of the toxic materials used. In each case, the possibility of substituting less hazardous materials should be considered. Substitution is a very important, and often an inexpensive method of control, especially where solvents are involved. Solvents such as methyl chloroform, dichloromethane, and aliphatic petroleum hydrocarbons have been used successfully as substitutes for carbon tetrachloride. Toluene or xylene can be used in place of benzene. It must be remembered, however, that many of these substitute materials are not without some degree of hazard themselves.

Non-silica parting compounds have replaced silica flour in foundry molding operations. Steel shot, ground walnut hulls, carborundum, alundum and other non-toxic materials are employed as substitutes for sand in many abrasive cleaning operations. Titanium and zinc compounds have been used successfully to replace lead pigments, resulting in the elimination of the lead hazard in many painting operations. Fiber glass has been substituted successfully for asbestos in insulating steam lines. A classic example of substitution was the use of non-toxic carroting materials for mercury compounds in the fur hatting industry.

A change of process can be made in many situations to reduce the exposure and thus the hazard. Examples include the application of paints or other protective coatings by dipping rather than spraying; the use of an ultrasonic cleaner instead of trichloroethylene; and use of a hydroblast for cleaning castings in place of sand blasting. Certain manual operations often can be mechanized and thus reduce the exposure. One example was the replacement, many years ago, of hand methods for applying lead oxide to the grids of lead storage batteries. Sometimes, mechanization may aggravate rather than decrease hazardous conditions.

Isolation or Enclosure

Some types of hazardous processes in-including storage may endanger other personnel than those directly concerned with the process. Where the hazard is serious as, for example, in the manufacture or process use of such highly toxic substances as prussic acid or tetraethyl lead, the entire operation

Prepared by: Andrew D. Hosey

is isolated in a separate building or in a carefully sealed-off area. Operations are made automatic so far as possible and all personnel involved are equipped with suitable personal protective equipment and are trained thoroughly in safe practices not only for day-by-day operations, but for emergencies as well. The very thorough application of this approach in the manufacture of explosives has helped bring the over-all injury rate for this industry down to among the lowest. The isolation of hazardous processes is a widespread practice in the chemical industry.

Many processes can be completely enclosed during all or part of the operation and, when maintained under a slight vacuum, will prevent escape of contaminants into the workroom atmosphere. Loading and removing the contents should be mechanical or automatic whenever possible. The means chosen should be as effective as possible, thereby eliminating or reducing the hazard itself rather than depending upon personal protective devices. Examples are the modern bag and drum filling equipment; the fully enclosed and exhausted tumblers and shot blast machines for cleaning castings; radium dial painting glove booths; and the new types of garment dry cleaning machines.

Enclosure is also a method of controlling exposures from such operations as sandblasting, heat treating, mixing, grinding, and screening. The test in each instance is: Does the process at any stage give off any substance that may contaminate the air? If so, can it be completely enclosed? If not, can a partial enclosure combined with exhaust be applied?

Isolation and enclosure are two of the methods employed to reduce noise exposure. In some instances this reduces the number of persons subjected to noise while in others, enclosure can reduce the exposure time where automatic machines need only periodic attention.

Control at Source

The approaches commonly employed to control atmospheric contaminants at their source are:
(1) local exhaust ventilation,
(2) use of wet methods, and
(3) good housekeeping.

Local Exhaust Ventilation. This is perhaps the most important method for controlling atmospheric contaminants at their source. The engineer plays important roles both in designing the exhaust systems and also checking their efficiency. Detailed discussions on this subject are found in other sections of this volume. It should be pointed out here, however, that all ventilation equipment requires systematic checking and maintenance if it is to function effectively. Particular care should be given to equipment on whose faultless functioning the worker's health or safety depends. Without preventive maintenance the first evidence of breakdown or other defects in an exhaust system may be a case, or perhaps even an outbreak of occupational disease among the plant personnel, particularly if the substances involved are relatively toxic.

It is important that medical supervision be provided as a check on the effectiveness of the engineering and operational control measures. When substances that can cause chronic injury by continued exposure to even low concentrations are involved, a more or less continuous medical supervision of all exposed personnel should be maintained. Examples are lead, mercury, and cadmium among the metals, benzol, carbon tetrachloride, and carbon disulfide among the solvents.

The disposal of the air exhausted from production processes presents a problem in the case of toxic substances or substances that may create a nuisance such as dust, odors, shavings, or lint. Merely discharging the exhausted air outside the building may serve only to

transfer the hazard or nuisance to another area. Air currents may also carry the contaminant back into the plant through windows or other openings. This hazard may be lessened by discharging from stacks carried well above the building roofs, but the resultant contamination of the general atmosphere is building up a widespread sentiment against such a practice. Moreover, in some cases, the contaminant may have considerable value. The general desirability of collecting exhaust substances has led to the development of various types of collecting equipment whose purpose is to separate the contaminant from the air exhausted and enable its recovery or harmless disposal.

Wet Methods. These are used successfully in certain foundry operations where, for example, a hydroblast is used in place of sandblasting or tumbling operations for cleaning castings. Dust concentrations can be reduced also if the molding sand is kept moist, if the castings are wet down before shakeout and if the floors are wet down intermittently. In the granite industry wet methods have reduced the dust formerly generated by many operations. In the Vermont granite industry, for example, the average dust concentrations from drilling operations in quarries were reduced from about 130 million particles per cubic foot of air (mppcf) to less than 2 mppcf following the introduction of wet drilling methods. Wire saws and contour grinding wheels, operated with water and artificial abrasive, have replaced most of the former cutting tools used in the granite industry.

Housekeeping. Immediate or periodic cleaning of spilled material is a method for removing the contaminant at its source. For example, unless it is removed immediately, mercury spilled on the floor or workbench may create a mercury vapor hazard not only to workers in the immediate area but also to those located in other areas of the plant. Housekeeping is particularly important when dealing with radioactive materials and certain compounds of beryllium to prevent their spread to other areas of a building or laboratory. Housekeeping at the source should not be confused with general housekeeping, which will be discussed later.

Dilution with Uncontaminated Air

The term, general ventilation, implies changing the air throughout an entire room or building in contrast to exhausting an operation or process in order to trap and carry off the dusts, fumes, or vapors given off. Local exhaust systems contribute to the general ventilation because they draw air from the room thus inducing an inward leakage of air, but in most cases, the volume of air drawn out of a workroom of any size is too small to have much effect on the general ventilation.

Operations may be such as to make the use of local exhaust impracticable as, for example, on the casting floor in the ordinary foundry. Furthermore, some contaminant may escape even the best local exhaust. Occasional operations such as painting, welding, cleaning, brushing, and grinding may produce a considerable degree of air contamination. Heat, humidity, or odors, or a combination of these, may produce an unpleasant or debilitating atmosphere. Therefore, general forced ventilation to provide a continual influx of fresh air is often necessary, or at least desirable.

General ventilation, sometimes referred to as dilution ventilation, should not be relied upon as a control measure where toxic materials are involved unless the degree of air contamination is not great, and unless the contaminant is released at a substantial distance from the worker's breathing zone to allow dilution to a safe level.

Instead of completely removing the contaminant at its source, as in most cases of local exhaust, the volume of air exhausted can be reduced to a level where

the amount of the toxic material escaping into the worker's breathing zone is below the threshold limit value. This technique was investigated a number of years ago and is discussed in Plant and Process Ventilation.[1] Considerable savings in fuel costs can be achieved by using this method, especially in northern climates. It is generally applicable where the exposure is to solvent vapors. A word of caution is indicated at this point. This type of dilution ventilation should not be attempted unless adequate facilities and trained personnel are available for air sampling to assure that the concentration of the toxic materials are maintained below safe levels.

Personal Protective Devices

Personal protective devices have one serious weakness, that is, they do nothing to reduce or eliminate the hazard. They merely set up a defense against it and any failure of this defense means immediate exposure to the hazard. The fact that many protective devices can be or can become ineffective without the knowledge of the wearer is particularly serious.

Standards and specifications for personal protective equipment have been developed as the result of extensive research and testing. Excellent equipment in great variety is commercially available.

Despite its general availability and relatively low cost, however, protective equipment should not supersede the elimination of the hazard. Employers who are not adequately safety-minded are constantly tempted to rely chiefly on personal protective equipment, whereas, these devices are intended for emergency or temporary use only. Respirators, in particular, are frequently resorted to instead of eliminating the air contaminants.

These devices may be divided into four groups: (1) respirators, (2) goggles and face shields; (3) protective clothing; and (4) protective creams and lotions. The safety department, especially in larger plants, is generally responsible for the issuance, maintenance, and educational program of these devices. In many instances, however, the industrial hygienist is asked to advise and make recommendations concerning the choice and use of the protective equipment. For this reason he must be familiar with, and know the limitations of, these items.

Respiratory Protective Devices. All respirators have two drawbacks in common: they are more or less uncomfortable to wear and they reduce the worker's efficiency. If the lessened efficiency could be measured in terms of cost it would probably show that substantial expenditures to eliminate the hazard often are justified.

To insure the effectiveness of respiratory protective devices, the U. S. Bureau of Mines tests and approves such equipment. Only those devices approved by the Bureau of Mines should be used, since such approval is authoritative assurance of their effectiveness, when used under the specified conditions. Lists of approved devices may be obtained upon request from the Bureau of Mines, Washington 25, D. C.

It is important that the type of respiratory equipment used be correct for the hazard involved. Deaths and near deaths have occurred because of dependence in deadly atmospheres on respiratory protective equipment not suited to the hazard. Examples are the use of filter-type respirators in an atmosphere containing carbon monoxide, or an all-purpose canister type when there is an oxygen deficiency.

In the event a hazard cannot be eliminated or reduction of the hazard is impracticable, an orderly procedure should be followed, including at least the following steps:
(a) Identify the substance or substances

against which protection is necessary.
(b) Obtain full knowledge of the hazards that each such substance offers and its significant properties.
(c) Determine the conditions of the exposure that will be involved.
(d) Determine what, if any, personal characteristics and capabilities are essential to the safe use of the protective devices and procedures required.
(e) Determine what facilities are needed for maintenance.
(f) From these considerations, select the type respirator which will provide protection.

Respiratory protective devices are of three types:
(a) Air purifying - which removes the contaminant from the air as it is inhaled by filtering or by chemical absorption, adsorption, or catalytic action.
(b) Air supplying - which supplies respirable air from a compressor, hand or electrically operated blower, or a cylinder of compressed air.
(c) Self contained breathing apparatus which supplies air or oxygen from a tank carried by the wearer, or by a chemical reaction producing oxygen from the moisture in the wearer's exhaled breath.

The correct use of all types of respiratory protective equipment requires careful attention to many details and the unfailing observance of rules for safe procedure. Full details of types approved by the U.S. Bureau of Mines should be obtained from the manufacturers.

No matter how well a respiratory protective device is designed and made, unless it is properly cared for and maintained in good condition, it may fail to give protection. Planning after determining the need for the equipment should provide for careful and unfailing maintenance. This includes inspection, repairs, replacement of used filters, cleaning, and sterilization.

Since a respirator is uncomfortable when worn for extended periods, personnel must realize fully the need of the protection or they will not wear respirators faithfully. Important factors in getting full employee cooperation in this respect are:
(a) Recourse to respiratory protective equipment only after every effort has been made to eliminate the hazard.
(b) Explaining the situation fully to the workmen involved.
(c) Careful fitting of respirators.
(d) Adequate provisions for maintenance and cleanliness.
(e) Careful instruction as to use.
(f) Intelligent, fully informed supervisors.
(g) Sterilization of equipment before reissue.

A more detailed discussion of respiratory protective devices can be found in the Manual of Respiratory Protective Devices, published in 1963 by a joint AIHA-ACGIH Committee.(2)

Goggles and Face Shields. These are used to protect the eyes and face from corrosive solids, liquids, and foreign bodies. Goggles are also used for protection against conjunctivitis or other eye injury resulting from welding or flame cutting operations and ultraviolet and infrared from other sources. Many types are available; the important point is to choose the correct type for the specific job and insist that the workmen wear the goggle or face shield on the job. Many industrial companies now have excellent eye protection programs. One reason for their success is a strict rule requiring that eye protection devices be worn on specific jobs.

Protective Wearing Apparel. In all work with substances that attack through the skin or attack the skin itself, protective clothing may be used to advantage to lessen skin contact. It must be suited to the nature of the substance, the exposure involved, and the kind of work done. The material of which the garment or protective article is made

must be resistant to attack by the substance in question, must not absorb it to any extent or be easily penetrated by it, must be cleanable, and should have reasonable wearing qualities. The garment or article must be as comfortable as possible. For women workers, the factor of appearance is important. Garments and articles of common use include head coverings, coats and jackets, aprons, coveralls, trousers, gauntlets, hand and finger protectors, leggings, boots, and shoes.

Protective Creams and Lotions. There are a number of barrier preparations on the market designed to protect the skin. Their effectiveness varies, but properly selected and correctly used they may be very helpful. They should never be relied on in lieu of positive means of preventing or reducing skin contact; but since in working with any substance some contact with at least the hands and arms is inevitable, this type of protection may constitute a worthwhile aid in prevention.

In each instance it is essential that:
(a) A cream or lotion effective against the substance or substances involved be selected with competent medical advice.
(b) The employees involved be instructed as to the value of the protection and in its proper application.
(c) Adequate facilities be provided for washing and for change of clothes necessary to eliminate all contact with the substance in question during offtime.
(d) The worker exercise careful personal hygiene; at the very least he should wash thoroughly before eating and at the end of each shift.

Maintenance, Housekeeping and Education

These three items are interdependent because it is impossible to have an effective maintenance program unless housekeeping is good and the worker has been informed of the need for these measures. For example, if paint is allowed to accumulate on the blades of the exhaust fan in a paint spray booth, the amount of air exhausted will be reduced thereby creating a possible health or fire hazard. If the thermostat on a degreaser fails or is accidentally broken, excessive concentrations of trichloroethylene might quickly build up in the work area, unless repairs are made promptly. Many machines which may ordinarily be noisy in operation, create even more noise if they are not given proper maintenance.

Housekeeping also is a very important control measure. Dust allowed to accumulate on floors, machinery, and beams can be disseminated to all parts of the plant. In the case of toxic dusts, such as lead or those containing quartz, many more workers may be exposed to hazardous concentrations if periodic cleaning is not practiced. Many plants have inaugurated regular cleanup schedules employing vacuum cleaners or vacuum lines. This is the only truly effective method of removing dust from the work area. The use of an air hose for cleaning purposes should be discouraged.

Education of the workers is important if they are to use effectively the control measures provided for their health and safety. Knowledge of the proper operating practice by the worker is necessary if the engineering controls are to perform the tasks for which they were designed. For example, improper loading and unloading of a degreaser or performance of an operation away from an exhaust hood will defeat the purpose of the controls and cause contamination of an area. Workers should be given reasons for wearing respirators, protective clothing, and goggles. They also should be informed of the necessity for good housekeeping and maintenance. When toxic materials are used in a process it is essential that the workers be given detailed information regarding the hazardous materials and the measures to be used by them to prevent injury. Too frequently this type of information is overlooked with the result that workers are needlessly exposed to toxic materials and are sometimes injured.

Other Control Methods

Many of the general methods cited above, or a combination of them, can be used for the control of most occupational health hazards. There are, however, a few additional situations which deserve special mention.

Ionizing Radiation

The entire field of ionizing radiation is a specialized one and cannot be adequately covered in a short course such as this. However, the same basic principles described in this lecture are involved in the control of ionizing radiation problems. The Atomic Energy Commission and the National Bureau of Standards have published much useful information in this field. If these guides are followed carefully, there should be no difficulty in controlling exposures. Many occupational radiation exposures occur as a result of carelessness and accidents; thus constant vigilance must be maintained.

Excessive Heat and Humidity

Control of these environmental factors may be a problem particularly in operations such as manufacturing steel and processing, heat treating, drop forging and manufacturing glass.

Protection of the worker from excessive radiant heat usually can be accomplished by shielding the source. The glass industry has spent large sums of money shielding the furnaces with aluminum panels, which incidentally reflect about 95 percent of the radiant heat. In the steel industry shielding certain operations such as soaking pits and rolling mills, and air conditioning crane cabs, resulted in reduced radiant heat exposures. Heat treating furnaces can be shielded by means of aluminum or in certain instances by fire chains. The use of steel or sheet metal for shielding is not very effective because a black body absorbs radiant heat and after a short time it will act as a secondary radiator. While shielding will normally result in lowered effective temperatures since the radiant heat is reduced, other measures may be needed to further reduce the effective temperature to comfortable levels. These include the use of general ventilation, local exhaust or floor fans. A combination of two or more of these measures may be necessary for certain operations. An air-conditioned enclosure provides the greatest protection to the worker and, when equipped with suitable windows, it can be used for crane operators, remote control console operators, and for other situations. Windows should be made of heat-reflectant glass and the operator may need tinted glasses where the light is intense or includes injurious wave lengths.

In recent years several types of air-conditioned suits have been developed and they usually provide air for breathing. The fabric can be light as long as it maintains an effective envelope through which the air can be circulated. Aluminized cloth or other materials can be used. These suits are particularly effective where the worker must get close to the radiant heat source and maintain freedom of motion as in stirring molten metal, while making repairs inside a furnace (shut down, of course), around certain types of electrolytic cells, or in test chambers.

Humidity inside a plant can be reduced by installing local exhaust ventilation on all operations generating steam or water vapor. This method is effective in paper manufacture, dye plants, laundries, electroplating and pickling tanks, and similar industrial operations. The application of insulation around steam and hot water pipes in these plants will effectively reduce temperatures.

Many hot operations cannot be controlled because of the cost involved. In these situations the only solution is to reduce

the duration of the work day or in effect, the exposure. This method was successfully employed several years ago at a zinc smelter and also in a large drop forge plant.

Medical Controls

The inauguration of a plant preventive medical program or the availability of medical aid is important in the control of occupational diseases. Preplacement physical examinations are becoming routine in most of the larger and many of the smaller industrial plants. These examinations usually include chest X-rays, and blood and urine analysis, and other clinical laboratory tests as indicated. The modern preplacement physical examination takes into account the capacities of the worker and the demands of the job.

Finally, periodic physical and laboratory examinations are made to determine if the individual is showing systemic effects from exposure to various toxic materials.

Physicians also play an important part in the development of general good health among employees. This may include advice regarding proper diets for specific jobs, or advice on diseases not of occupational origin.

Personal Hygiene

The frequent use of soap and water is the most important method for reducing the incidence of skin diseases. This simple control method frequently is overlooked or used ineffectively by many persons. When the use of epoxy resins became rather widespread in 1956-57, for example, hundreds of workers developed dermatitis from contact with this material, particularly the amine hardeners. The dermatitis was further aggravated by the use of solvents for hand-cleaning purposes. It was found that frequent use of soap and water, and protective gloves largely decreased dermatitis incidence. Workers should be encouraged in the practice of good personal hygiene by the provision of adequate washing facilities, lockers, and clean change rooms.

Summary

General methods for control of the occupational environment were discussed and examples of each were cited. One or a combination of these methods may be necessary to control effectively occupational exposures to hazardous materials or physical agents. In addition, measures were discussed briefly for controlling exposures to ionizing radiation, extremes of temperature and humidity, and radiant heat. The importance of preventive medical programs and personal hygiene were cited.

References

1. Plant and Process Ventilation. Hemeon, W. C. L. The Industrial Press, New York, N. Y., 1955.

2. Respiratory Protective Devices Manual. Joint AIHA-ACGIH Committee on Respiratory Protective Devices. Braun-Brumfield, Inc., Ann Arbor, Mich., 1963.

CONTROL OF NOISE EXPOSURE

Introduction

With the increased mechanization, use of larger and faster machinery, and the speeding up of transportation in the last decade there has also been an increase in noise output. This has resulted in a greater need and demand for noise control and it has become necessary that the industrial hygienist be familiar with some of the principles involved in the control of noise exposures. General acoustical principles of noise control have been fairly well established; however, the complexities of industrial noise sources and their environments, in many cases, require empirical solutions. In general, it will not be possible to use "text book" solutions for a specific noise problem because many situations will require a certain amount of experimental work for a satisfactory answer to a particular problem.

It must be emphasized that in order to control noise large reductions in energy must be achieved. For example, cutting the energy in half reduces the sound pressure level only by 3 dB. For this reason noise control is usually expensive and it is important to study the problem thoroughly before an attempt is made to reduce the noise.

The first step in the control of noise is to secure adequate quantitative and qualitative information on the extent and magnitude of the problem. This requires extensive noise measurements and a complete description of the environment. The next step is to compare the measured levels with acceptable criteria for the particular environment. This determines the amount of reduction which is necessary. When the required amount of noise reduction has been determined the industrial hygienist must then consider various noise control measures such as engineering design, personal protective devices, or limitation of exposure. It should be kept in mind that personal protective devices are not a substitute for engineering control.

Plant Design

Since exposures of workmen to industrial noise is becoming more of a problem with our advancing technology, it is essential that preliminary planning be done prior to the construction of a new plant or to modernizing an existing plant in order to eliminate or minimize excessive noise.

Selection of Building Site

Several factors should be taken into consideration in the selection of a plant site. One of the most important of these is the existing noise level of the community. A noisy industrial plant should be located in an area that has a high background noise level rather than in a quiet area. Conversely, a church, school, or hospital should be located in an area with a low background noise level.

Ofttimes, advantage can be taken of the topography and prevailing winds in controlling noise. There is a tendency for sound waves to be deflected upward so it is better to locate a plant on top of a hill rather than in a valley if there is a possibility of a neighborhood noise problem. Sound waves traveling into the wind tend to be deflected upward while those traveling with the wind tend to be deflected downward. In some situations it may be possible to take advantage of this by considering the prevailing winds.

It is also possible to reduce noise by proper landscaping; using embankments, terraces or shrubbery as soundbarriers. These can be used to isolate a building

Prepared by: Herbert H. Jones

from existing noise or to prevent noise from a building creating a neighborhood problem.

Building Layout

Noisy operations should be isolated from the rest of the work room. When a quiet area is desired or necessary it is better to locate offices, conference rooms, libraries and other areas away from street noises.

Selection of Work Process

Sometimes the work process can be changed with an appreciable reduction in noise level. Several possibilities are as follows:

(a) compression riveting instead of pneumatic riveting;
(b) welding instead of riveting;
(c) belt drives in place of gears;
(d) hot instead of cold working of metals;
(e) grinding instead of chipping;
(f) pressing instead of rolling or forging; and
(g) avoidance of dropping hard materials during handling.

Choice of Equipment

When purchasing new equipment serious consideration should be given to procuring the type with the lowest possible noise level. Engineering specifications should incorporate noise levels desired and requirements for noise performance data. In most cases, the machinery manufacturer is in the best position to reduce the noise of the machine at its sources with proper built-in designs. If there are a number of suppliers of the equipment being purchased, the relative noise outputs of each should be evaluated when considering prices and other factors that determine which product will be chosen. One should consider also how much the application of noise control measures will add to the cost of a less expensive, but noisy machine.

Reduction at Source

If at all practicable, it is usually more economical to reduce noise at its source rather than attempting to either reduce noise levels in a large work area or to provide, and supervise the use of, personal ear protective devices for several workers. Furthermore, reduction or elimination of noise at its source affords protection to the immediate operator(s) as well as to other workers in the area. Several general methods for reducing unnecessary noise are discussed below.

Lost Motion and Rattle

Noises of this nature often are attributed to poor maintenance. Replacement of worn parts, securing, stiffening or damping loose panels usually will eliminate a large amount of this noise.

Friction

Noise is produced by frictional forces generated by grinding and cutting operations, or from portions of machines such as, clutches and brakes. Some of this noise can be eliminated by proper lubrication of moving parts on the machine and the use of cutting oils or coolants on the products being handled. The use of sharp and properly shaped cutting tools will also usually reduce noise at such operations. Substitution of materials used in the manufacture of brakes or clutches may give a reduction in noise.

Impacts

Impact noise is generated when materials are forged, hammered, sheared, tumbled or peened. Noise from such sources is generally difficult to control. One approach is to spread the impact out over a longer time interval. This can be done in some cases by the use of stepped dies, which produce a number of small impacts rather than one large impact. Other approaches may be:

(a) change a punching operation into a shearing operation;
(b) clamping or placing weights on the piece being hammered or peened will reduce materially the noise generated from such operations; and
(c) tumbling barrels can be coated with a mastic material or suitable innerliners can be used to reduce noise levels.

Fluctuations in Gas Flow

If the flow of gas varies irregularly in a piece of equipment noise is usually generated. This may occur, for example, in an internal combustion engine, from the use of a high speed fan or blower, or from jets of compressed air, steam, and air driven tools. Examples for controlling the noise from such sources are:

(a) install proper intake and exhaust mufflers on internal combustion engines;
(b) use a large fan operating at a slow speed rather than a small, high-speed fan;
(c) redesign fan blades to reduce the turbulent flow of air;
(d) equip compressed air jets with mufflers or redesign the jet nozzle; and
(e) redesign the exhaust ports of those tools which produce a siren type of noise or slow down the speed of operation, if possible.

Unbalanced Forces

Reciprocating motion or unbalanced rotation can produce noise or it may excite other structural members and cause them to radiate noise. High speed rotating parts should be balanced properly. Very little can be done about reciprocating parts except proper maintenance and mountings.

Reducing the Flow of Noise Energy

This is a very fruitful field for noise control because many sources of noise energy are small and inefficient sound-wave generators. If this flow of energy can be interrupted and prevented from reaching structural members and panels, which may be more efficient sound generators than the original source, much of the noise can be controlled.

Solid-borne transmission. Vibrations can be transmitted for considerable distances through solid materials with very little attenuation. If some break can be made in this path, the flow of energy will be reduced. This can be done by using soft, flexible material such as, vibration mounts, flexible couplings on shafts or pipes, flexible connectors on ventilation ducts, belt drives, and mastic materials at joints when fastening materials together.

Energy may be transmitted to structural members or panels which in turn are set into vibration and radiate the energy. Structural members and panels can be stiffened to reduce vibration. Vibration-damping materials resist motion in any direction during every cycle of vibration. Thus, they provide definite energy losses during each cycle of vibration. Materials such as mastic, and vegetable and mineral fibers impregnated with mastic, have been used successfully to reduce vibration and in turn reduce noise levels.

Air-borne transmission. When it is not possible to control the noise energy within the machine some other method must be employed. It is usually more economical to construct a partial or total enclosure around the machine rather than installing sound absorbing materials throughout the entire work area involved.

Partial enclosures can be of a number of shapes or designs. They may consist of single panels beside the source, panels on both sides of the source, or tunnels with the work flowing through them. Sound absorbing linings must be used inside these enclosures. The

barrier can be constructed by using plywood, wallboard, sheet metal or almost any material with the requisite mass and strength. This type of enclosure usually does not protect the operator but does produce a shadow effect to give protection outside of the enclosure up to about 15 dB. Machines or noisy operations can be totally enclosed except for small access openings, which are treated by the use of short acoustical ducts. These should be made of an impervious material to stop the flow of energy and should be provided with an acoustical lining to absorb the energy within the enclosure. Properly designed enclosures will give a 20 to 30 dB noise reduction. They do have the disadvantage of interfering with ventilation, handling of materials, and maintenance.

Absorption of Noise

The most common use of sound absorbing material is in general or overall noise control in enclosed spaces. Sound absorbing material has very little effect on the noise level close to a source but will give a considerable reduction some distance from the source in the reverberant field. The limit for most practical installations is a reduction of 10 dB. Sound absorbing materials are usually porous and may be made of vegetable or asbestos fibers, glass or mineral wool. They are available in a number of forms including blocks, panels or blankets which can be fastened to the walls or ceiling, and space absorbers, which can be suspended from the ceiling.

Personal Protection

In some cases it will not be possible to control the noise because of economic limitations or lack of engineering knowledge. If such exposures are of sufficient intensity and duration to cause hearing loss, personal protective equipment should be worn.

Types of Ear Protectors

Ear protectors are usually classified according to the manner in which they are to be worn, and the three usual types are: ear plugs, ear muffs, and helmets.

Ear plugs. These are inserted into the ear canal and are intended to reduce the air-borne sound just before it impinges on the ear drum. They may be made of rubber, plastic, neoprene, or cotton impregnated with wax. Material and shape have very little to do with the effectiveness of commercially available plugs. Contrary to popular opinion, dry cotton gives very little protection.

Ear muffs. The muff-type protector consists of a set of cups designed to cover both ears and to be held snugly to the head by a headband. The cups may be made of plastic or rubber and are designed to encompass the ear without compressing it and yet at the same time to fit so closely to the head that very little sound leaks through.

Helmets. These are designed to cover most of the bony portion of the head to prevent bone conduction, but this has not proved to be completely satisfactory. Actually the whole head would have to be encased to avoid bone conduction effectively.

The amount of protection that can be expected from plugs or muffs in current use is shown in the table on the next page as the average attenuation in decibels at different frequencies.

Selection and Use of Ear Protectors

The ear protection program should be under the supervision of the medical department. An employee's ears should be examined and his hearing tested at the time he is fitted with ear protectors. A number of types and sizes of plugs should be available and plugs should be

fitted individually for each ear. Most of the available ear protectors, when properly fitted, provide about the same amount of protection, therefore the one that fits correctly and is the most comfortable for a particular employee is the best for him. Properly fitted protectors can be worn continuously by most persons and will provide adequate protection against most industrial noises.

As with other types of personal protective devices it may be difficult to convince employees that they shouldwear ear protectors. An educational program will be necessary and employees should be told why it is necessary to wear the plugs or muffs. They should be instructed how to insert the plugs and how to keep them clean. In many plants it is the responsibility of the safety engineer or industrial hygienist to supervise the use of such devices.

Ear Protector	Frequency in Cycles per Second					
	250	500	1000	2000	4000	6000
Typical plug	15	17	20	25	30	30
Typical muff	20	22	30	37	45	40
Typical muff plus plug	33	35	42	47	50	45

References

1. Beranek, L. L. Noise Reduction, McGraw-Hill Book Company, Inc., New York, N. Y., 1960.

2. Bolt, R. H. et al. Handbook of Acoustic Noise Control. Vol. I and II, WADC Tech. Report 52-204, PB 111200, 1952 and 1953.

3. _____. Industrial Noise Manual. Am. Ind. Hyg. Assoc., 14125 Prevost, Detroit 27, Mich., April 1958.

4. _____. Guide for Conservation of Hearing in Noise. Subcommittee on Noise in Industry. American Academy of Opthalmology and Otolarynology, Los Angeles, Calif., 1957.

5. Harris, Cyril M. Handbook of Noise Control. McGraw-Hill Book Company, Inc., New York, N. Y., 1957.

COMFORT VENTILATION AND CONTROL OF THE THERMAL ENVIRONMENT

Introduction

Environmental conditions being maintained in industry today vary widely from plant to plant, and from section to section within a plant. Year-round air conditioning may be installed in some areas while others remain extremely hot. Among the factors which must be considered in selecting the thermal environment for a given area are the following:

1. Population density within the area
2. Type of work being done
3. Heat load in area
4. Environmental requirements of process or product
5. Geographical location of plant.

Both labor and management groups are currently showing increasing concern regarding plant environment, and in most cases management is attempting to provide the maximum degree of comfort consistent with the manufacturing processes involved. A comfortable environment will improve worker morale, reduce absenteeism, and attract better employees. It is also generally believed that improved conditions will increase worker productivity, but conclusive data on this point are lacking.

Year-round air conditioning is commonly used today in factory offices, drafting rooms and similar areas where the population density is high and heat loads are not excessive. Air conditioning is also becoming an accepted practice in many plants doing light manufacturing or light assembly work.

Prepared by: Robert L. Harris, Jr.
Revised by: Clark M. Humphreys

Ventilation Requirements

Ventilation in one of its many forms is frequently relied upon to maintain an acceptable thermal environment in industrial plants. Ventilation may be defined as the process of supplying or removing air to or from a space by either natural or mechanical means. Such air may or may not have been conditioned.

It is only under very unusual circumstances such as men working in closed tanks or similar confined spaces, that ventilation is ever required to supply oxygen to or remove carbon dioxide from a space. It has been demonstrated that these requirements can be met with as little as one cubic foot per minute of outdoor air per person(1).

When human occupants are the only source of contamination, the minimal quantity of outdoor air needed is that required to remove objectionable odors and tobacco smoke, and sometimes heat. The following general recommendations are given in reference 2.

	Ventilation Rate cfm/person	
	Recommended	Minimum
People not smoking	7½	5
People smoking	40	25

The same reference contains a table giving recommended and minimum outdoor air requirements for many different applications or types of occupancy.

It is also recommended(2) that ventilation rates based on the above requirements be checked against the volume of the space. In rooms having a ceiling height of 10 feet or less, the ventilation rate should be not less than one air change per hour. In rooms having ceilings higher than 10 feet, the ventilation rate may be checked against the

volume of the space below an assumed 10 foot ceiling.

In residences, apartments, and similar spaces having very low occupancy, ventilation requirements are usually met by normal air infiltration, and no special provisions for air introduction are required. In theatres or similar buildings having high occupancy, adequate ventilation is an important consideration.

Most cities and states have codes or ordinances governing minimum ventilation rates for schools, theatres, auditoriums and other public buildings. All relevant code requirements should be determined before a system is designed.

Mechanical Ventilating Systems

A mechanical ventilating system[3,4] is one in which air flow is produced by fans. If the fan is delivering air to a space it is termed a "supply" system; if the fan is removing air from a space, it is called an "exhaust" system. Frequently a system will utilize both supply and exhaust fans.

A mechanical system can be very simple or quite complex. It may consist of an exhaust fan in the wall of a building, with air admitted through open windows. At the other extreme, it may consist of a supply fan which delivers air to various points in a building through a system of ducts. The air may be supplied at the existing outdoor conditions, or it may be cleaned and heated or cooled according to the space requirements. Cleaning or otherwise conditioning air is possible only with a mechanical system. The forces producing flow in a natural ventilation system are too small to overcome the resistance of the conditioning equipment.

Natural Ventilating Systems

A natural ventilating system[5] is one in which air flow is produced by natural forces. These forces are (a) wind, and (b) the difference in temperature between the air inside and outside the building. Air movement may be the result of either of these forces acting independently, or of a combination of the two.

The inside-outside temperature difference is effective in producing air movement only when the air linet and outlet openings in the building are at different heights. The stack or chimney effect, thus produced, increases with stack height and temperature differences.

The more common types of inlet openings for natural ventilation are windows, doors, or specially designed openings. Air may leave the building through windows and doors, monitors, skylights, roof ventilators, stacks or openings of special design.

The performance of a natural ventilation system is dependent upon many factors. Among these are:

1. Orientation of the building with respect to the prevailing winds.
2. Interference by nearby buildings or other obstructions.
3. Wind velocity and direction, inside-outside temperature differences, and stack height.
4. Type, size and location of inlet and outlet openings.
5. Adjustment of inlet and outlet openings in accordance with existing wind conditions.

Air flow rates with natural ventilation may vary widely due to changes in wind velocity and/or direction, and with temperature difference. Although maximum ventilation is usually needed in summer, temperature difference, and usually wind velocity, are highest in winter. It is therefore necessary to design a system for severe summer conditions, and reduce its effectiveness at other seasons by reducing the area of openings.

A combination of mechanical and natural ventilation is frequently installed in large industrial plants. Fans, usually arranged to exhaust air from the building, are used to supplement natural ventilation during periods of extreme weather.

Reference (5) contains a good discussion of natural ventilation systems. Equations are included for calculating air flow produced by wind forces and temperature difference, separately or in combination.

Heat Removal by Ventilation

In the hotter industries, where floor areas are usually large and the population density is low, ventilation, with little or no conditioning, is relied upon to remove heat and maintain conditions within tolerable limits[4]. The amount of ventilation air required for the removal of a given amount of sensible heat may be determined by the equation:

$$cfm = \frac{Hs}{c \times \rho \times 60 \ (t_i - t_o)}$$

where: Hs = sensible heat removed, Btu/hr.
c = specific heat of air, 0.24 Btu/lb./°F.
ρ = air density, lbs/cu. ft.
$t_i - t_o$ = indoor-outdoor temperature difference, °F.

For standard air having a density of 0.075 lbs. per cubic foot, this equation becomes:

$$cfm = \frac{Hs}{1.08 \ (t_i - t_o)}$$

Latent heat or humidity also may be removed by ventilation if the dew point temperature of the outdoor air is lower than that in the building. The quantity of ventilation air required to remove a given amount of latent heat (expressed in Btu/hr.) may be approximated by the equation:

$$cfm = \frac{H_1}{\rho \times 60 \ (W_i - W_o) \times 1100 \ / \ 7000}$$

where: H_1 = latent heat removed, Btu/hr.
$W_i - W_o$ = indoor-outdoor specific humidity difference, grains/pound of dry air
1100 = a factor approximating the average enthalpy of water vapor in a warm environment, Btu/lb.
7000 = grains per pound.

For standard air this equation becomes:

$$cfm = \frac{H_1}{0.71 \ (W_i - W_o)}$$

It is always desirable to use local exhaust hoods or enclosures to capture the sensible and/or latent heat given off by hot processes or equipment, and discharge it to the outdoors.

The quantity of supply air required in a plant is frequently determined by the exhaust system requirements[3,6]. Exhaust systems cannot function properly unless proper provisions have been made for supplying make-up air. These provisions should include a means of heating the air in winter. This will not necessarily increase heating costs. If the plant is to be kept comfortable, the make-up air has to be heated, whether it enters through heaters provided for that purpose, or through cracks and miscellaneous openings throughout the building. The latter situation can cause serious drafts and low temperature areas near such openings. Where large quantities of air at relatively high temperatures are being exhausted, some form of heat recovery equipment may be effective in reducing heating costs[6].

Local or Spot Cooling

Local or spot cooling[4] is used frequently to provide more comfortable or tolerable conditions at specific work

stations in a hot industry, without appreciably altering the surrounding environment. To accomplish this, ventilation air, with or without some conditioning, is delivered to the work area through ducts and discharged at or adjacent to the workers.

Discharge outlets should be located as close as possible to the work area, and not more than 7 or 8 feet above the floor. If, for some reason, they must be located remotely, larger air volumes will be required because of the mixing that takes place between the jet air and the hot air in the space.

The allowable air velocity in the occupied zone varies considerably depending upon the air temperature. If the air temperature is high, a rather high air velocity will be comfortable. The same velocity with cooler air will be very uncomfortable. For this reason air outlets should be readily adjustable in direction, so that optimum conditions can be maintained at all times. Several types of adjustable outlets are shown in references 4 and 6.

With a well designed system it is usually possible to maintain the temperature at the work stations within one to three degrees of the supply air temperature, even though the air, when it eventually leaves the building, is at a considerably higher temperature.

Local man-cooling fans are sometimes used for spot cooling. However, if the ambient air is above skin temperature, the additional convective heat gain by the man may seriously increase the demand for sweating.

Evaporative Cooling

Where the high initial and operating costs of mechanical refrigeration cannot be justified, evaporative cooling may provide an acceptable substitute[7,8,9].

When air is passed through a washer, operating with recirculated spray water, the temperature of the spray water and the leaving air temperature approach the wet-bulb temperature of the entering air. The extent of this approach is determined by the saturating effectiveness of the air washer, which is defined as:

$$e_h = \frac{t_1 - t_2}{t_1 - t'} \times 100$$

where: e_h = saturating or humidifying effectiveness, percent
t_1 = dry-bulb temperature of the entering air, °F.
t_2 = dry-bulb temperature of the leaving air, °F.
t' = thermodynamic wet-bulb temperature of the entering air, °F.

The effectiveness of commercial spray-type washers varies from 50 to 95%, depending upon the number of banks of spray nozzles, the direction of the sprays, and a number of other factors.

The following example illustrates the results that may be obtained with evaporative cooling:

Problem: Assume that outdoor air at 95°F dry-bulb and 70°F wet-bulb is passed through an air washer having a saturation effectiveness of 80%. Find the dry- and wet-bulb temperatures and the relative humidity of the air leaving the washer.

Solution: By substituting into the above equation, the leaving dry-bulb temperature of 75°F may be determined. Since the evaporative cooling process takes place at constant wet-bulb temperature the leaving wet-bulb will be 70°F. By referring to the psychrometric chart in Section B-29, a relative humidity of approximately 78% may be read at the intersection of the 75° dry-bulb and 70° wet-bulb lines.

It is evident that for the conditions assumed in the above example, evaporative cooling would make it possible to supply ventilation air to a work space

at 75°F instead of 95°F. This would make a marked improvement in the comfort of the workers. It is also evident from following the above process on the psychrometric chart, that evaporative cooling is most effective when the air is relatively dry. As the relative humidity of the entering air increases, the possible reduction in dry-bulb temperature decreases.

The use of a two-stage evaporative cooling system may provide air at temperatures up to 10 degrees lower than those obtainable from the single-stage system just described. In the two-stage system, water is first cooled in a cooling tower. This cooled water is then circulated through a coil where it reduces both the dry- and wet-bulb temperatures of the air flowing over the coil. Such cooling takes place at constant dew-point temperature. The air thus cooled is then passed through a washer where it is cooled at constant wet-bulb temperature as described in the above example. Further details on two-stage evaporative cooling systems will be found in references 8 and 9.

Control of Heat Exposures

Measures to be taken in the control of heat exposures in hot industries will vary considerably from plant to plant. In "hot-dry" industries such as forging or glass forming, the problem is one of excessive sensible heat. In a "warm-moist" situation such as a laundry or a textile dyeing plant, discomfort is caused primarily by excessive latent heat (moisture).

Control of Heat Source

The first step to be taken in the control of heat exposures is to reduce as far as possible the heat being added to the space[3,4]. Steam and hot process piping should be well insulated. Where practical, insulation should be applied also to other hot equipment surfaces. Where high humidity is a problem, steam leaks should be eliminated and covers should be provided for steaming water tanks, hot water drains and similar sources of water vapor.

Local exhaust hoods or enclosures may be used to capture either sensible or latent heat given off by a process or piece of equipment. The heat thus captured is discharged to the outdoors, and does not raise the temperature or humidity of the plant environment to which the worker is exposed. Much more heat per pound of air can be removed by local exhaust than by general ventilation, because the permissible rise in temperature or humidity is much greater for the local exhaust system.

Radiation Shielding[4]

The fundamental equation for calculating radiant heat exchange is given in Section B-29. The terms emissivity, absorptivity and reflectivity are used to describe surface characteristics which play an important part in radiant heat exchange. Emissivity may be defined as the ratio of the total radiant flux emitted by a surface to that emitted by an ideal black body at the same temperature.

All of the radiant energy falling on an opaque surface is either absorbed or reflected. The absorptivity of a surface may be defined as the fraction of the total incident radiation which it absorbed. It can be shown that the absorptivity of any surface is numerically equal to its emissivity. The reflectivity of a surface is the fraction of the total incident radiation which it reflects and is numerically equal to one minus the absorptivity.

In general, highly polished metal surfaces have low emissivities, while non-metallic surfaces have high emissivities. The emissivity of any surface varies widely with the condition of the surface, and it is therefore impossible to predict its exact value. The values in Table 1 indicate the usual range of emissivities, absorptivities and reflectivities for a few surfaces.

Table 1 - Emissivities, Absorptivities and Reflectivities of a Few Surfaces

Surface	Emissivity and Absorptivity	Reflectivity
Black non-metallic surfaces such as asphalt, carbon and slate	0.90 to 0.98	0.10 to 0.02
Ordinary window glass	0.90 to 0.95	0.10 to 0.05
Brick (any color), concrete, tile, stone	0.85 to 0.95	0.15 to 0.05
Painted surfaces (any color)	0.85 to 0.95	0.15 to 0.05
Oxidized steel sheet	0.75 to 0.80	0.25 to 0.20
Smooth sheet iron	0.55 to 0.60	0.45 to 0.40
Aluminum paint	0.40 to 0.60	0.60 to 0.40
Galvanized steel sheet	0.20 to 0.30	0.80 to 0.70
Dull aluminum sheet	0.10 to 0.20	0.90 to 0.80
Polished aluminum sheet	0.02 to 0.05	0.98 to 0.95
Aluminum sheet with one coat linseed oil	0.56	0.44

The radiant energy from hot furance walls, ovens, and similar surfaces can constitute a major heat load in an industrial plant. Although the air is not directly warmed by the thermal radiation passing through it, the radiation heats the various surfaces on which it falls, and these surfaces in turn heat the air by convection. Thus radiation can be indirectly responsible for an appreciable increase in air temperature in an enclosure. The radiant energy which falls on a workman also adds directly to the thermal stress imposed upon him.

The net radiant exchange between two surfaces is a function of the temperatures and emissivities of both surfaces. A reduction in the temperature of the radiating surface will reduce the radiant exchange, but ordinarily the improvement to be made in this direction is quite limited. Altering the surface to lower its emissivity is usually much more effective. If the radiant exchange between a single pair of surfaces is considered, the same reduction would be accomplished by making either the radiating or receiving surface reflective. However, in an industrial plant, for each high temperature surface there are many lower temperature receiving surfaces, and it is therefore advantageous to treat the radiation at its source.

Instead of altering the existing radiating surface to reduce its emissivity, it is frequently more feasible to place a radiation shield between it and the cooler surroundings. The closer the shield can be placed to the hot object, the more complete the shielding is likely to be. The radiant heat thus reflected back to the primary source can be removed by local exhaust.

Durable and effective radiation shields may be made of highly polished tinplate or stainless steel, or ordinary flat or corrugated aluminum sheets. Since the reflectivity is a surface characteristic and is not dependent upon thickness, a thin foil may be used. Foil-faced building boards of several types are available and will provide good reflectivity from one side. Aluminum foil-faced cloth curtains, which may be raised or lowered on spring rollers, provide a light weight, portable, and readily adjustable type of shield.

Several types of transparent radiation shields are also available. These include reflective tempered plate glass, reflective metal chain curtains and close mesh wire screens.

Reflective shielding is usually located so that it reflects the radiant energy

back to its original source. However, this is not always the case. Before installing reflective shields, a careful study should be made to ascertain that the heat reflected from one area will not create a serious problem in another location.

Personnel Protection

In hot industries there are areas in which environmental conditions are so severe that a man can work in them for only a short period of time. If the work area can be confined, an air-conditioned room or enclosure is the ideal solution. Man can then work in comfort in spite of the severe conditions outside the room. The air-conditioned crane cab is a good example. Where it is not feasible to cool a work area, small air-conditioned recovery rooms located adjacent to the hot areas will provide welcome and rapid relief for the workers during their "off" periods. At the present time data are lacking on the optimum duration of work and rest periods for various hot environments.

Many types of protective clothing have been developed for use in hot environments. The simplest of these utilize thermal insulation and highly reflective surfaces to protect against high air temperatures and thermal radiation. They are usually made of fireproof or fire resistant materials and may include face masks or hoods, gauntlets, etc. This type of clothing will increase the allowable duration of work periods, but is suitable only for relatively short, intermittent exposures.

Several types of cooled suits are also available. In one of these, cool air is supplied to the suit from a remotely located cooler. The large hose required for this type of equipment may seriously limit the movements of the wearer, and the air flowing through it may be warmed considerably before it reaches the man. In a rather recent development, a vortex tube[10,11,12] carried on the belt of the worker and supplied with compressed air, furnishes a source of cool air which is introduced under the outer clothing. The hose required is small, and the worker's mobility is not seriously limited. Both of these air supplied units, when equipped with hoods, can provide a supply of clean cool air for breathing.

Several types of self-contained cooled suits are also available. In one type the cooling is supplied by a tank of liquid air carried on the worker's back. Another utilizes a cooling cartridge which must be periodically regenerated in a low temperature refrigerator. Such units are available for various time periods, and some are said to be satisfactory for ambient air temperatures in excess of 500°F.

References

1. Consolazio, W.V.; and Pecora, L.J.: Minimal Replenishment Requirements for Living Spaces. ASHVE Transactions, 53: 127 (1947).

2. ASHRAE Guide and Data Book, Fundamentals and Equipment. Chapter 26, 1963.

3. Industrial Ventilation - A Manual of Recommended Practice. American Conference of Governmental Industrial Hygienists, P.O. Box 453, Lansing, Michigan, 8th Ed. 1964.

4. ASHRAE Guide and Data Book, Applications. Chapter 34, 1964.

5. ASHRAE Guide and Data Book, Fundamentals and Equipment. Chapter 24, 1963.

6. Clark, J. H.: Makeup Air, Heat Conservation and Recovery, Air Conditioning Heating and Ventilating, 61: 29 (Feb. 1964).

7. ASHRAE Guide and Data Book, Fundamentals and Equipment. Chapter 38,

1963.

8. ASHRAE Guide and Data Book, Applications. Chapter 84, 1964.

9. Evaporative Cooling, A Symposium, ASHVE Journal Section, Heating Piping and Air-Conditioning 27: 141 (Aug. 1955).

10. Fulton, C. D.: Ranque's Tube, Refrigeration Engineering 58: 1 (May 1950).

11. Alexander, J. M.; Croley, J.J.; and Messick, R. R.: Use of Vortex Tube for Cooling Wearers of Industrial Protective Clothing, AEC Res. and Dev. Report DP-861, Office of Technical Services, U. S. Dept. of Commerce, Washington, D.C. 20025, Oct. 1963.

12. Leinhard, W. F.; Hughes, J. P.; and Brassette, T. A.: Man Cooling by a Vortex Tube Device. Archives of Environmental Health 9: (Sept. 1964).

Note: The initials "ASHVE" and "ASHRAE" denote the present organization - American Society of Heating, Refrigerating, and Air-Conditioning Engineers, 345 E. 47th Street, New York, N. Y. 10017

MEDICAL MONITORING OF THE OCCUPATIONAL ENVIRONMENT

Introduction

One traditional definition of occupational medicine is: "that branch of medicine which deals with the relationship of man to his occupation, for the purposes of the prevention of disease and the promotion of optimal health, productivity and social adjustment." The plant physician of a modern industry or the medical director who has workers as patients must first have an awareness that workers are subject to a multitude of materials, many of which can produce disease of one sort or another. This may appear to be a trite statement but physicians in general, and also many industrial medical personnel, are lacking in knowledge of the materials and processes used in the industries they serve. The biological effects of industrial machines, processes, raw materials, and finished products are myriad. Therefore, it certainly behooves the industrial physician to acquaint himself with the actual and potential problems in the industries with which he comes in contact.

Laboratory investigations have provided leads regarding the existence of possible or probable toxic agents or physical hazards. Animals can be given injections of materials; they can be exposed to particulates; they can be sprayed or painted with liquids; they can be subjected to all forms of radiant energy; and they can be put into stressful situations. However, the final test of toxicity or hazard to humans is by subjecting them to that toxic substance or hazard. At least one can qualify this to say that the degree of toxicity for humans must be determined in humans.

Each worker, then, becomes a sort of guinea pig or even a sort of canary when he steps into the industrial environment. Just how he reacts to his environment depends in part on the composition of that environment and in part on the man himself - his physical makeup, his heredity and his previous exposure to toxic materials. It follows that an important part in the attempt by the medical department to understand the effect of an environment on a man or a woman is the knowledge of how materials enter the body and then what effects they might produce in the individual. In the working environment there are four major areas in which the worker makes contact with this environment:

I. the special senses, eyes, ears, nose, mouth;

II. the skin;

III. the digestive tract, and

IV. the respiratory system.

Monitoring the Environment

There are several ways the physician has of monitoring the environment both initially and as a follow-up procedure through the use of the human "canary", that is, the worker in his environment.

Speaking of the canary, and recalling its use in the mines as an indicator for the presence of toxic levels of carbon monoxide, a recent article by T. D. Spender[1] of the British National Coal Board sheds new light on the effect of carbon monoxide on man and canaries. The canary was expected to fall from its perch if the concentration of carbon monoxide met in the mine was dangerous to man. However, at lower levels, especially below 500 ppm, the canary is less sensitive than man to carbon monoxide

---

Prepared by: William S. Lainhart, M.D.

and might give a false sense of security to an observer. The results observed are due to the several considerations: the canary may get dizzy and weak before falling from its perch but we have some difficulty diagnosing these early symptoms in the bird; the canary reaches a state of equilibrium with the carbon monoxide it is breathing much more quickly than does man; it takes two or three times the concentration of carbon monoxide to produce an equivalent reaction in the canary to that seen in man; and human hemoglobin apparently has a marked increase in its affinity for carbon monoxide over that for oxygen when compared with avian hemoglobin. This bit of information only serves to point out that we continuously increase our knowledge of the toxic ingredients of our environment even though that knowledge may appear to be adequate at any one time.

Specific Indicators

Skin

Now that the notion of the human being as a canary has been introduced, what are the ways that the environment is reflected in the human worker? The skin can be considered an excellent mirror for the compounds with which it comes into contact. The contact dermatitis reactions; the flushing of the skin; sweating; depigmentation and other manifestations can indicate exposures to very diverse substances such as gases, chemicals, metals, and physical agents. Thus, the environment can be monitored by observing the reaction of the workers' skins.

Gastro-Intestional System

We are all aware of the effects of the environment on our gastro-intestinal system. Although motion sickness involves more than this system, the diagnosis is primarily a gastro-intestinal one. Reactions to lead poisoning can involve various levels of the gastro-intestinal tract with such manifestations as a metallic taste, nausea and vomiting, abdominal colic and constipation. Through the inhibition of an enzyme system (cholinesterase), organic phosphate poisoning can result in diarrhea, nausea and vomiting. In addition to such specific items, noxious odors in general, tend to produce gastro-intestinal upsets.

Special Senses

The so-called special senses include vision, audition, smell, taste, touch, and a host of others, less generally recognized. The physician in industry can use these senses to get an indication of the working environment. It would be in order to mention here that pre-placement and periodic examinations of workers give baseline information against which future data can be compared. Baseline and periodic examinations are particularly important with regard to eye injuries due to radiant energy and to hearing loss.

Eye Damage

An article by David J. Cogan[2] on the subject of eye damage pointed out the relation between wave length and tissue damage. The wave length determines penetration so that the involved area of the eyes differs with the differing wave lengths. Thus, with the longer infrared waves produced by diathermy machines and radiant heat, there tends to be less penetration and consequently, the concentration of the rays on the lens of the eye can produce cataracts. In the visual or near visual part of the spectrum, scotoma or blind spots on the retina can be produced - i.e., the blindness produced by viewing an eclipse of the sum or carbon arc welding. Lesions in the eye from Grenz or X-rays

again involve cataract production and conjunctivitis or inflammation of the anterior coating of the eyeball. It might be noted that the time of development of the eye lesions is also correlated with the wave length of the radiant energy. Those reactions following exposure to diathermy, infrared and visible rays tend to be almost immediate whereas the latent period between exposure and the development of symptoms in the case of Grenz, X-ray and Gamma rays may extend from hours to days and even months. Although the medical personnel in a factory or office do get involved with the immediate traumatic injuries to the eye, they must also be aware of the potential exposures to radiant energy.

Hearing

Another of the special senses affected by the environment is hearing. The relation between noise exposures and deafness is undergoing critical review and some interesting experimental work is being conducted in this area.

Systemic Reactions

Although general systemic reactions to environmental hazards are somewhat more difficult to define, the blood, bone marrow, urine, sputum, feces, and other materials can be examined to ascertain derangements in the internal body environment as well as to identify toxic materials themselves or by-products produced by the body.

Blood

The blood is composed of the so-called formed elements - white cells, red blood cells and platelets - and the serum. White cells are primarily concerned with ingesting and destroying bacteria but they also appear to have functions relating to the immune and delayed hypersensitivity reactions, an example of which is the tuberculin skin reaction or an attack of hay fever.

Red blood cells contain hemoglobin, which includes a ferrous radical for binding oxygen to the heme. The oxygen is thereby transported from the lungs to the cells throughout the body. If blood is exposed to oxidizing agents such as cyanide, the ferrous iron is changed to ferric iron forming methemoglobin, which, when present causes the skin to take on a dusky discoloration. To return to the canary mentioned near the beginning of this discussion, carbon monoxide reacts with hemoglobin to form carboxyhemoglobin. The increased affinity of hemoglobin for carbon monoxide reduces the oxygen-carrying capacity of the blood and so decreases the available oxygen for tissue use. Anemia, or a paucity of either hemoglobin and/or red blood cells, can be a result of poisoning by certain aromatic hydrocarbons.

The functions of the blood serum are not completely understood. It is known, however, that the quantity of certain enzymes present in the blood serum is changed with exposure to organic phosphate ester insecticides and chlorinated hydrocarbon solvents. Toxic products such as lead may be dissolved in the blood. There is a great deal of work now being directed toward the possibility of developing what might be termed screening tests for certain industrial compounds. Might an industrial physician, for example, draw blood from a potential employee and test that serum against known agents used in his plant? A positive test then could indicate what materials or processes should be avoided by this employee. A test of this sort is still at the investigative stage but there is a promise of success in this area of study.

In the adult, red blood cells and some white blood cells are formed in the bone marrow. Examination of this tissue can give valuable clues to the type and amount of anemia or to the results of exposure to radiation.

Urine

Laboratory tests on urine can provide

knowledge regarding the type and extent of exposure to some toxic materials. Heavy metals such as lead and arsenic may be excreted in the urine. Certain organic compounds are detoxified by the body and these detoxification products (or metabolites) are excreted in the urine. In interpreting the results of urinalyses, especially with regard to the heavy metals, it is most important to remember that absorption can take place without poisoning and that access to the body can be non- as well as occupational.

Sputum

Sputum examinations may be used to rule out certain infections when the diagnosis of pneumoconiosis is being entertained. A more recent development is that an increased number of ambiguous and suspicious cells in the sputum is associated with underground uranium mining experience. A Public Health Service team of workers in Salt Lake City has been investigating the increased incidence of lung cancer among uranium miners. As a part of this study, sputum cytology was introduced in the hope that earlier diagnosis of cancer might be made.

Nervous System

The effects of various toxic compounds on the nervous system vary from such non-specific generalized manifestations of disorder as headache, dizziness, giddiness and silliness to some of the reported dramatic deaths due to the inhalation of rather massive doses of DDT, to the almost specific wrist drop, a form of lead palsy (with atrophy and weakness of the extensor muscles of the forearm). Personality changes, difficult to recognize, have been seen in methyl chloride poisoning while carbon monoxide can produce a whole gamut of neurological and psychological manifestations.

Respiratory Disorders

It should be pointed out also that the environment can contribute to a whole series of respiratory disorders from a mild cold-like catarrh to frank pneumonia, pneumoconiosis or cancer. It is obviously difficult to delineate where a cold begins and pharyngitis or bronchitis begin or, for that matter, when cancer begins. Thus, with occupational respiratory diseases, the symptoms and signs of cough, phlegm production, chest pain, shortness of breath, and the spitting up of blood merely can be considered to be one or more parts of a continuous process -- irritation to the respiratory tract.

Case Histories

When evaluating a case where the respiratory tract may be involved, one must be very careful to inquire into the whole respiratory history in order to assess properly the role the industrial exposure might play. Thus, in a study of pneumoconiosis of any sort, the respiratory signs and symptoms during the employee's entire life are emphasized. A history of asthma may be important; repeated chest illnesses may indicate some type of physiological, anatomical, or immunological malfunction; the history of exposure to irritating gases, smokes, or fumes (including cigarette smoking) may contribute to the development of lung disease.

Afflictions of the respiratory tract cannot always be specifically located anatomically. The effect of arsenic has been reported to be a painless perforation of the nasal septum but some investigators feel that the inhalation of arsenic dust can produce lung cancer. Irritation to the nasal passages and bronchial linings is a common manifestation of exposure to certain chemicals, fumes, and dusts, but some chemicals may affect one part of the respiratory tree, while others may cause generalized responses. Lung cancer has been attributed to arsenic and to other agents, such as beryllium, asbestos, and ionizing radiation. Uranium miners, who apparently inhale rock dust which carries

radon daughters into the lungs, are included in the latter category. Because there appears to be an increased incidence of lung cancer in these uranium miners, the association is made between the inhalation of radioactive dust and the development of cancer of the lung.

Pneumoconioses

What are the dusts which cause signs and symptoms of pneumoconiosis and possible disability from this disease? Johnstone and Miller[3] have offered what appears to be a workable classification -- although other authors have, in effect, "thrown up their hands" and reported that a classification cannot be devised which is wholly satisfactory.

The Major Pneumoconioses

Disease	Dust Incriminated
Silicosis	Free crystalline SiO_2
Asbestosis	Silicate
Talcosis	Silicate
Coal Miners' Pneumoconiosis	Coal Dust

The Minor Pneumoconioses

Anthracosis	Soot and carbon
Diatmoaceous earth pneumoconiosis	Amorphous SiO_2
Silicatosis	Silicates
Mica	
Clay	
Feldspar	
Beryllium Disease	
Vegetable Dust Pneumoconioses	
Mill Fever	Various organic dusts
Byssinosis	Cotton
Bagassosis	Mouldy sugar cane
Farmer's Lung	Mouldy hay, straw
Grain Asthma	Grain
Tamarind Asthma	Tamarind seed
Weaver's Cough	Mouldy cotton yarn

The Benign Pneumoconioses

Baritosis	$BaSO_4$ & BaO	
Siderosis	Fe_2O_3	"pure to denote the absence of silica"
Stannosis	SnO_2	
Titanosis	TiO_2	
Graphosis	Graphite	
Chalicosis	Calcium salts	
Limestone		
Marble		
Cement		

It is quite evident from the above that a myriad of dusty materials can produce demonstrable lung disease - ranging from severely disabling silicosis to the poorly defined byssinosis of cotton mill workers (also called "Monday dyspnea"). The Division of Occupational Health has been involved in the study of several of the pneumoconioses:

diatomaceous earth pneumoconiosis,
silicosis,
coal workers' pneumoconiosis,
asbestosis,
anthracosis, and
bagassosis

These investigations have included working with the metal miners, coal miners, pottery workers, asbestos product workers, processors of diatomaceous earth, carbon plant workers, etc.

The primary epidemiological tools used were:

questionnaires to record occupational and medical histories;
the 14x17 inch chest X-ray;
measurements of pulmonary function; and
tests to determine the work capacity of individuals at work in the various industries studied.

Because of the necessity for using a battery of investigative tools, it is apparent that no single test is completely diagnostic. There are definite correlations between pneumoconioses and age, pneumoconiosis and other-than-occupational environmental factors, and probably also between pneumoconiosis and as-yet-unknown factors. These correlations do not indicate necessarily, a cause-and-effect relationship. Even though some pictures are consistent with pneumoconiosis and the worker has a positive occupational history, the findings cannot be considered to be this clear-cut. In these situations it is possible to make only very general retrospective or prospective statements about individual employees in terms of means, modes, or trends.

The primary interest of the Occupational Medicine Section, Division of Occupational Health, at the moment is an investigation of chronic chest disease in coal miners. The principal cause of disability among coal miners (aside from accidents) appears to be chronic lung disease, especially pneumoconiosis. This disease apparently has two not-very-distinct stages; the inhalation of high concentrations of comparatively pure coal dust which can lead to a localized tissue reaction with fibrosis or scarring. This fibrosis occurs around the respiratory bronchioles and as a result of the subsequent dilation of the bronchioles, focal emphysema occurs. (Emphysema is the ballooning out of the alveoli or air sacs). The nodules seen on X-ray are the small scars produced. The disease may be complicated by bronchitis and general emphysema but simple pneumoconiosis is compatible with good health and there is usually unimpaired work capacity.

The second stage of coal workers pneumoconiosis termed "complicated" is most usually defined by X-ray and is, more often than not, symptomatic with bronchitis (cough and jet black phlegm production) and exertional shortness of breath. It is at this stage that the miner usually thinks toward asking for compensation or at least consulting a physician.

Summary

The human body can be used as a monitor of the environment in diverse ways. The several systems and organs which offer themselves are the skin, the special senses - the eyes, ears, nose, -- the gastro-intestinal tract, internal organs, blood and the respiratory tract. Industrial hygienists and others in this field should remember these possibilities and offer their cooperation to the physician in industry by providing

him with information regarding the industrial processes used and the toxicological agents present in the plant or other operations. This data will be of obvious value to the doctor in his diagnostic workups and his follow-up of the workers under his jurisdiction.

References

1. Spender, T. D.: Effects of Carbon Monoxide on Man and Canaries. Anal. Occup. Hyg. 5:231-240, (1962).

2. Cogan, D. G.: Lesions of the Eye from Radiant Energy. JAMA 142:3, (Jan. 21, 1950).

3. Johnstone, R. T. and Miller, S. E.: Occupational Diseases and Industrial Medicine. L. B. Saunders Company, Philadelphia, 200, 1960.

DILUTION VENTILATION

Introduction

The term general ventilation is accepted to mean the constant exchange (supply or exhaust) of air from a general area, room or space. This exchange of air is for the purpose of both comfort control and reduction of atmospheric contaminants by dilution of contaminated air with uncontaminated air.

While the terms general ventilation and dilution ventilation are used interchangeably, a great many times in this section, the principles of dilution will be considered as applying to such atmospheric contamination as gases, vapors, and particulates. The use of general ventilation to control the comfort (temperature, humidity, and odor) of the worker is covered as Comfort Ventilation in Section C-3.

Classification of General Ventilation Systems

In any building, room, or enclosed area there is continuous air circulation of some degree. This air movement is created by: (1) Differences in pressure or temperature, (2) operation of equipment, or movement of people, and (3) fans or other air movers.

Air circulation within a confined area or space may be classified as natural ventilation or mechanical ventilation.

Natural Ventilation

Natural ventilation is the displacement and infiltration of air through windows, doors, walls, floors, ventilators and other openings by natural forces such as pressure (wind) and temperature (air density) differences between the building and its surroundings. The air movement may be caused by one or the other, or a combination of the two. It must be remembered that air movement by infiltration is not only dependent on differences in temperature and pressure but also on the physical characteristics of the building.

Ventilation due to pressure forces. In the utilization of natural wind forces for producing air movement consideration must be given to the average velocity, direction and variation of the wind. In addition, consideration must be given to natural and man-made objects which may obstruct or change the natural wind conditions. Some engineers assume that natural ventilating systems should prove satisfactory if designed for wind velocities of ½ the average seasonal velocity(1).

Average wind conditions for various localities are usually obtained from local meteorological stations or airports. However, these data must be utilized with care due to variations caused by differences in local terrain and the location of the measuring equipment.

The air volume moved by wind forces can be calculated from the formula:

$$Q = EAV$$

where: Q = cfm of air
A = free area of inlet openings, ft.^{2}
V = wind velocity, lfm
E = coefficient = 0.5 to 0.6 when inlet faces wind and 0.25 to 0.35 when inlet is at angle to wind.

The inlets should face the prevailing wind and the outlets should face the opposite side, or discharge into a low pressure area.

Ventilation due to air density difference. Air density differences are normally found in hot, dry industrial areas

Prepared by: Charles H. Powell, Sc.D.

such as foundries and steel mills. Air movement is effected in this type of industry by (a) the production of heat; (b) the vertical distance between inlet and outlet; and (c) the areas of the inlet and outlet.

The volume of air moved by temperature (density) differences is determined by the draft head and the resistance:

$$Q = \sqrt{\frac{H}{2}\,\frac{(ta - to);}{RT}} \qquad R = \frac{1}{2gA^2C^2}$$

where Q = cfm of air - natural ventilation
H = vertical distance between inlet and outlet
(ta - to) = temperature difference between indoor and outdoor air, ^{o}F
T = average absolute temperature, ^{o}R
R = resistance
g = gravitational constant
A = free area of inlet
C = coefficient = 0.65 in most cases; 0.50 for restricted openings

Using an assumed T of $100^{o}F$ or $550^{o}R$, and C = 0.65 this reduces to

$$Q = 9.4\,A\sqrt{H\,(ta - to)}$$

When solving problems where the inlet and outlet areas are unequal the flow is increased by the excess of one opening over the other. A curve which indicates the percentage of increase may be found in Chapter 24, page 439, of the ASHRAE Guide and Data Book(1). To estimate the combined effect of wind and temperature differences, calculate both by the equations given above and express the temperature difference effect as a percent of the total. When the two pressure heads are about equal, the combined effect will be about 10% greater than either. This percentage decreases as one pressure head increases over the other. The effect of the larger head always predominates. Curves which indicate the actual flow caused by combined forces of wind and temperature may be found in Chapter 24, page 440, ASHRAE Guide and Data Book(1).

Mechanical Ventilation

Mechanical ventilation is the creation of air movement by some mechanical or artificial method, such as by the use of wall, window or portable fans, roof ventilators, or the operation of industrial equipment. This is usually required when contaminant dilution is necessary as infiltration or other natural ventilation normally cannot provide the desired volume of air.

In comparatively small rooms or work areas, window or wall fans are generally used for exhaust. In larger single level industrial buildings roof ventilators, which take advantage of the air temperature difference that exists, may be used as exhausters. Many other types of commercial roof ventilators are available from simple propeller to centrifugal types which are capable of producing high static pressures and efficiencies and thus exhaust a greater volume of air.

Where roof ventilators are not practical, air may be exhausted through grilles to ductwork and fans. General supply or make-up air is usually required to prevent the creation of negative pressures in the building when sufficient dilution ventilation is provided by local exhaust hoods within the building. It should be noted also that while supply air blower and distributor systems are needed with this type of system they may not always be provided. A more detailed discussion

of air distributing systems can be found in the ASHRAE Guides[1,5].

Use of Dilution Ventilation

Distinction Between Dilution and Local Exhaust Ventilation

Control of hazardous contaminants by the action of dilution ventilation is accomplished by diluting the concentration of the contaminant before it reaches the workers' breathing zone with clean or uncontaminated air. Dilution ventilation does not reduce or eliminate the amount of hazardous material released into the workroom air.

The action of local exhaust ventilation is to control the contaminant at its source or point of generation thus preventing the release of the contaminant within the general workroom.

Normally local exhaust ventilation is a better means for industrial hygiene control than dilution ventilation. Local exhaust has the advantage of requiring less air volume for effective control, and of localizing the contaminant. Less air is handled since a smaller volume of air at a higher concentration of the contaminant is removed rather than a larger volume at lower concentration. This is an important consideration if the make-up air must be heated or cooled. In addition, localizing the contaminant has several other advantages. Among these are: (1) it will eliminate nuisance complaints in the general area; and (2) it will prevent the accumulation of particulates or condensable vapors which may create a housekeeping problem even though they are reduced to a hygienically safe concentration by dilution.

The differences between dilution and local exhaust ventilation cannot always be determined with any degree of certainty. If the exhaust system is difficult to classify as one type or the other, the following criterion can be applied. It is a dilution ventilation system when the concentration of the contaminant in the exhaust duct is not significantly higher than that in the general room air; it is a local exhaust system if the concentration of material in the duct is significantly higher than that in the general room air.

When to Use Dilution Ventilation

If the material of concern escapes into the air at a comparatively low rate and more or less uniformly throughout the work area - not at one or more isolated locations - dilution ventilation may be the most practical means of controlling the contaminant at the breathing zone of the worker. However, if the process generates especially toxic or irritating substances, local exhaust should be used. The successful application of dilution ventilation depends upon the following conditions:

1. The material or contaminant generated should not be in excess of the amount which can be diluted with a reasonable volume of air.

2. The distance between the workers and the point of generation of the contaminant should be sufficient to assure that the workers will not be exposed to average concentrations in excess of the current established TLV's.

3. The toxicity of the contaminant must be low (must have a high TLV), and

4. The material or contaminant should be generated at a reasonably uniform rate.

Dilution ventilation is used with the greatest success in the control of contaminants when the rate of consumption can be determined with some degree of accuracy, by a materials balance or some other method, and the volume of dilution air required can be calculated.

It may well be that a health hazard does not exist due to dilution ventilation

created by the construction and other features of the work area because practically all rooms or areas have, to a larger or smaller extent, some amount of natural or mechanical ventilation. On the other hand, only a small amount of material is required to produce a hazardous condition in an air-tight room.

Dilution ventilation is used successfully to control the concentrations in the workroom air of gases and organic vapors, with comparatively low toxic properties, as well as heat and water vapor. The removal of heat and water vapor is discussed in another section.

Several reasons preclude the successful application of dilution ventilation for controlling the concentration of fumes and dusts. The amount of material generated is usually too great, and reliable data on the rate of generation of fumes and dusts are very difficult to obtain. In addition, the material may be quite toxic, which would require an excessive amount of dilution air.

Control by Dilution Ventilation

The rate of flow of uncontaminated air required to reduce the atmospheric concentration of a hazardous material to an acceptable level can be calculated by the following formula:

$$Q = \frac{6.71 \times G \times 10^{e} \times S \times K}{M \times TLV}$$

where: Q = ventilation rate in cubic feet/min.
G = specific gravity of liquid contaminant
S = rate of consumption of the liquid contaminant in pints/hour
M = molecular weight of liquid contaminant
TLV = threshold limit value in ppm
K = design factor.

This equation assumes the vapors act as an ideal gas at normal room temperature and pressure (1 atmosphere and 70°F) and the contaminant is generated at a more or less uniform rate. In addition, the formula assumes complete mixing of the contaminated air with the uncontaminated dilution air throughout the room area if the design factor (K) is equal to one or was not included in the formula.

The "K" factor is based upon three primary considerations, they are:

1. Estimate of the efficiency of mixing and distribution of the make-up air and room air to reduce the level of concentration of the contaminant to the desired level throughout the room.

2. To reduce the concentration of contaminant to some level at or below its threshold limit value. This determination is based upon toxicologic considerations used for the establishment of the TLV. (These may include (a) acute and/or chronic toxic effects and (b) discomfort, irritation and odor).

3. An estimate of any additional variances which the industrial hygienist determines to be of importance based upon his experience and the individual problem. Included in this criteria are such considerations as:
   (1) Seasonal changes in the amount of natural ventilation
   (2) reduction in operational efficiency of mechanical air moving devices
   (3) duration of the process, operational cycle and normal location of workers in relation to sources of contamination
   (4) location and number of points of generation of the contaminant in the room or area, and
   (5) other circumstances which may affect the concentration of the hazardous material in the breathing zone of the worker.

K values for various distributing systems are:

K = 1.2 to 1.5	- perforated ceiling
K = 1.5 to 2	- air diffusers
K = 2 to 3	- trunk line alongceiling with branch jets pointing downward
K = 3 & above	- window fans, wall fans, etc.

Industrial Ventilation[2] on page 2-2 lists air volumes required to dilute the vapors of 39 common organic solvents to the TLV level, based upon the number of pints or pounds of solvent evaporated per unit of time. These values should be multiplied by a K factor to allow for variations in uniformity of air distribution and other considerations. Hemeon[3] includes a table of recommended dilution rates for 53 organic solvents. His "Ventilation Design Concentrations" are not threshold limit values but are based on both limits and odor. All of the concentrations in this table are lower than the threshold limits, but those substances which are especially toxic, or which have a very disagreeable odor, have the greatest safety factors.

A word of caution should be interjected here regarding the use of tables of dilution rates. The date that such information was compiled is extremely important because threshold limit values are subject to revision, and may be changed periodically. If the threshold limit value is lowered, the dilution rate should be correspondingly increased.

Calculation of Contaminant at Any Time

The concentration of a gas or vapor at any time can be determined, assuming that the contaminant is generated continuously, the material is mixed instantaneously with the room air, and the rate of air flow is constant.

Let x = concentration of gas or vapor in air at a time t
G = rate of generation of contaminant
Q = rate of air movement
K = distribution constant
$Q' = \frac{Q}{K}$ = effective rate of air movement
V = volume of room.

The material balance for the contaminant will be:

$$Vdx = Gdt - Q'xdt$$

$$Vdx = (G - Q'x)dt$$

$$\int_{x_1}^{x_2} \frac{Vdx}{(G - Q'x)} = \int_{t_1}^{t_2} dt$$

$$V\left[-\frac{1}{Q'} \ln(G - Q'x)\right]_{x_1}^{x_2} = t_2 - t_1 = \Delta t$$

$$\Delta t = \frac{V}{Q'}\left[-\ln(G - Q'x_1) \ln(G - Q'x_2)\right]$$

$$\Delta t = \frac{V}{Q'} \ln\left[\frac{G - Q'x_1}{G - Q'x_2}\right]$$

Terminology for Dilution Ventilation Rates

Dilution ventilation rates are often expressed as "air changes per hour" or "minutes per air change." These units express the ratio of the volume of air supplied or exhausted to the volume of the room. They are useful when applied to meeting rooms, offices, schools, warehouses, retail stores, laundries, and similar spaces where the purpose of ventilation is simply odor, temperature, and humidity control, and the only contamination of the air is from the activity of people. For purposes of control by dilution, however, such terms are of little value. Air changes per hour are dependent upon the size of the room, while the quantity of air needed to dilute a given contaminant, generated at a constant rate, is not affected by

the size of the room. Dilution ventilation rates should be expressed in cubic feet per minute or some other absolute unit of quantity flow. The room volume will determine the rate at which a concentration of contaminant will build up, and also the time required to purge the room after the generation of contaminant is stopped. A large room will act as a surge tank, so that for an intermittent generation of contaminant, it may be safe to supply air to balance the average rate of generation rather than the maximum rate.

Selection of the Air Movement System

Careful consideration must be given to the selection of the air distribution system to be used in a dilution ventilation problem. Each problem must be carefully studied and evaluated on its own merit. After it has been determined that the amount of air necessary to dilute the contaminant to a safe level is not excessive, the choice of a supply system, an exhaust system or a combined supply-exhaust system must be selected. and the location of the supply and exhaust openings must be chosen[4].

Normally, the supply air should be introduced along one side of the room or work area and the exhaust opening located on the opposite side of the room; however, under some circumstances, the air flow pattern may be changed or short circuited by partitions, large machines, hot processes or other disruptions of the normal air flow. Therefore, care should be taken to avoid short circuiting the dilution air. The supplied air must pass over the point or points where the contaminant is generated and toward the exhaust opening. In most circumstances this should assure that the contaminant is not carried to the breathing zone of the worker.

The air exhausted must be replaced by an equal amount of uncontaminated fresh air, either from a mechanical system or by entry through openings in the building. This will assure that the room is not under excessive negative pressure which will reduce the possibility of workers being exposed to dangerous concentrations of other gases or vapors, principally carbon monoxide. Provision must be made to heat the make-up air during cold weather. Care must be exercised to prevent drafts around windows, doors and other sources of make-up air. This requires special consideration if the temperature of the make-up air is more than 10°F below the temperature of the workroom.

Normally better dilution is possible with a good air supply system because the air can be directed toward the sources of generation of the contaminant. However, if adjacent areas are occupied, the positive pressure created in the room by the excess air supplied will cause the contaminated air to flow into any adjoining occupied areas of lower pressure. This is true also of combined supply-exhaust systems when there is excessive make-up air. In the case of the combined system the adjoining area would not be contaminated if the workroom in which the ventilation was being utilized was under a slight negative pressure caused by an excess of exhaust air.

In a combined supply-exhaust system the ventilation rate is the rate of either the supply or exhaust, whichever has the greatest capacity, and is not the sum of the rates of the two systems.

Consideration must be given to any local exhaust hoods which are located in the work area to assure that the air flow at the face of the hoods is not disturbed by air from the supply inlets.

The location of exhaust openings should be selected to keep the source of contaminant between the operator and the exhaust, and to avoid re-entrance of the contaminated exhaust air into the workroom. This usually requires that the contaminated air be exhausted well above the roof line of the building or above closely adjacent buildings.

References

1. ASHRAE Guide and Data Book - Fundamentals and Equipment - American Society of Heating, Refrigerating and Air-Conditioning Engineers, Inc., New York, 1963.

2. Industrial Ventilation - a Manual of Recommended Practice. ACGIH Committee on Industrial Ventilation, Lansing, Michigan, 8th Ed., 1964.

3. Hemeon, W.C.L.: Plant and Process Ventilation. 2nd Ed. The Industrial Press, New York, 1963.

4. Brandt, A.C.: Industrial Health Engineering. John Wiley & Sons, Inc., New York, 1947.

5. ASHRAE Guide and Data Book - Applications. American Society of Heating, Refrigerating and Air-Conditioning Engineers, Inc., New York, 1964.

PRINCIPLES OF VENTILATION

Introduction

Ventilation is a method of controlling the environment with air flow. This air flow may be used to remove a contaminant, heat or cool a space, or simply supply make-up air. Normally to do this job four basic components are assembled into a ventilation system. They are a hood, duct, air cleaner and a fan.

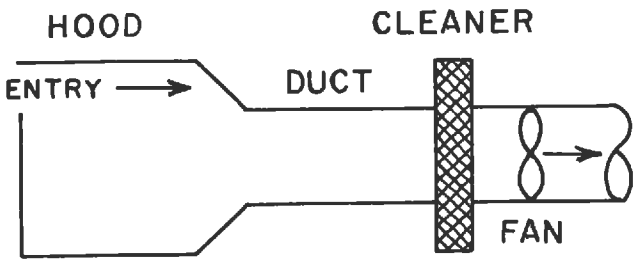


Figure 1. Ventilation System Components

The air flow problem in ventilation design, then, is to make these four components function together to move the required amount of air at a specific speed through the system. The laws that define this air flow and the use of these laws to analyze a system will be discussed in this Section.

Principles of Ventilation Air Flow

The driving force for air flow is pressure difference. A pressure is required to start and to maintain flow. This pressure is called total pressure (TP) and has two components, Static Pressure (SP) and Velocity Pressure (VP).

Static Pressure produces the initial air velocity, overcomes the resistance in the system caused by friction of the air against the duct walls, and overcomes turbulence and shock caused by a change in direction or velocity of air movement. Static pressure is analogous to potential energy in that it exists even when there is no air motion. It acts perpendicularly to the walls of the duct and either tends to collapse the walls of the duct work upstream from the fan or tends to explode the walls of the duct work on the downstream side. Static pressure, then, is negative in sign upstream from the fan and positive in sign downstream. It is measured as the difference between duct pressure and atmospheric pressure, and the most common units are inches of water ("H_2O).

Velocity Pressure maintains air velocity in the duct and is therefore analogous to kinetic energy. It exists only when air is in motion and it acts in the direction of air flow. It is always positive in sign.

Static pressure, velocity pressure and total pressure are all interrelated. If a gas flowing through a duct system undergoes an increase in velocity, a part of the static pressure available at that point is used to create the additional velocity pressure necessary. Conversely, if the velocity is reduced at some point, a part of the kinetic energy or velocity pressure at that point will be converted into potential energy or static pressure. Static and velocity pressure are, therefore, mutually convertible but this conversion is generally accompanied by a loss of energy due to turbulence and shock. This means that the conversion is almost always less than 100% efficient.

At a given point in any duct system, the static pressure, velocity pressure, and total pressure are mathematically related as follows:

$$\text{Total Pressure} = \text{Static Pressure} + \text{Velocity Pressure}$$

Figures 2, 3, 4 and 5 illustrate this relationship at different points in this system.

Prepared by: Ralph G. Edwards, Jr.

Basic Assumptions

Before the principles of air flow can be developed, several basic assumptions must be made. The first of these is that air is an incompressible fluid. For normal flow rates used in ventilation this is a practical assumption.

Also, air flow will be in the turbulent range. The turbulent range is defined by a Reynolds number (R_e) of greater than 4,000. Streamline flow occurs when R_e is less than 2100. Between 2100 and 4000 there exists a transitional region of instability where no accurate prediction of the type of flow can be made. Reynolds number is defined as:

$$R_e = \frac{DV\rho}{\mu} \qquad (1)$$

where: R_e = Reynolds number
D = Pipe diameter
V = Average linear velocity
ρ = Fluid density
μ = Fluid viscosity

The formulae which we will derive and the tables which we will use will assume turbulent flow.

Calculation of Ventilation System Variables

Five variables completely define any ventilation system. They are:

Q - Quantity of air flow in cfm
V - Velocity of air flow in ft/m
SP - Static pressure in $"H_2O$
VP - Velocity pressure in $"H_2O$
A - Area of duct work in ft^2

Quantity

The basic formula used to measure quantity of air flow at any point in the system is the mass or weight continuity equation. This equation states that the mass rate of flow remains constant along the path taken by a fluid. Letting A_1, V_1, σ_1, be the cross sectional area, velocity, and specific weight (lb/ft^3) at point #1 in a duct and $A_2V_2\sigma_2$ be the same variables at point #2; then:

$$A_1V_1\sigma_1 = A_2V_2\sigma_2$$

Recalling that air is incompressible, then $\sigma_1 = \sigma_2$ and the formula becomes:

$$A_1V_1 = A_2V_2 \text{ or } Q = AV \qquad (2)$$

Velocity and Velocity Pressure

Bernoulli's equation is a statement of the conservation of energy for fluid flow. It can be simplified and expressed as:

$$Z_1 + \frac{V_1^2}{2g} + P_1v_1 + W_o =$$

$$Z_2 + \frac{V_2^2}{2g} + P_2v_2 + F \qquad (3)$$

where the terms represent specific energy (ft lb/lb) and:

Z = Height above any arbitrary horizontal datum plane (ft).
V = Linear velocity (ft/sec.)
g = Gravitational acceleration ($ft/sec.^2$)
P = Absolute static pressure (lb/ft^2)
v = Specific volume (ft^3/lb)
W_o = External work put into system (ft lb/lb)
F = Frictional losses (ft of fluid)

From this equation:

$\frac{V^2}{2g}$ represents the velocity head, h

Solving for V and substituting the more convenient units of air velocity (linear ft/min) and pressure (inches of water gauge), the formula becomes:

$$V = 1096\sqrt{\frac{VP}{\rho}} \qquad (4)$$

VP = velocity pressure in inches water gauge ($"H_2O$)

ρ = density of gas stream (lb/ft^3)

Fig. 2

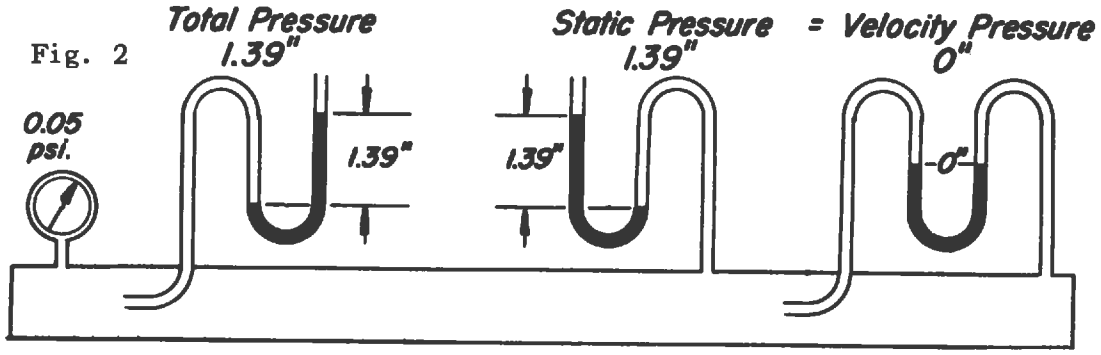
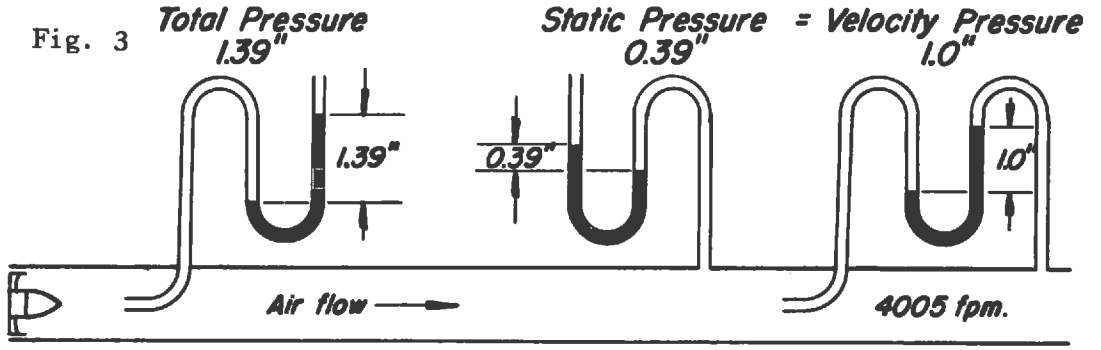
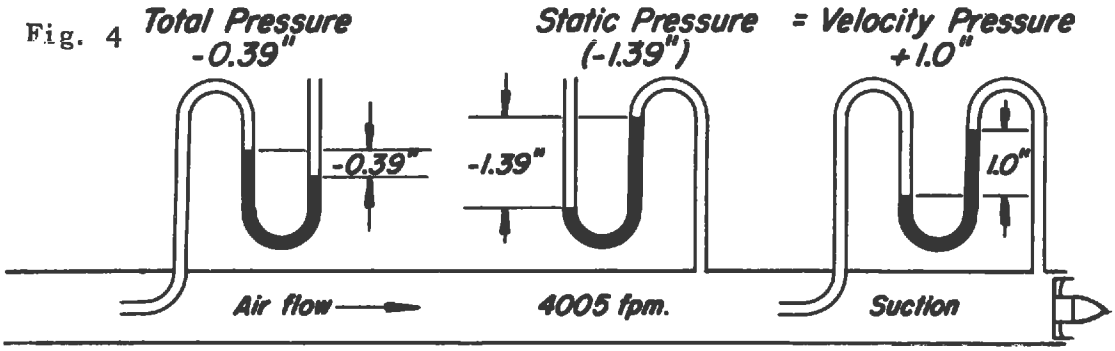


Fig. 3



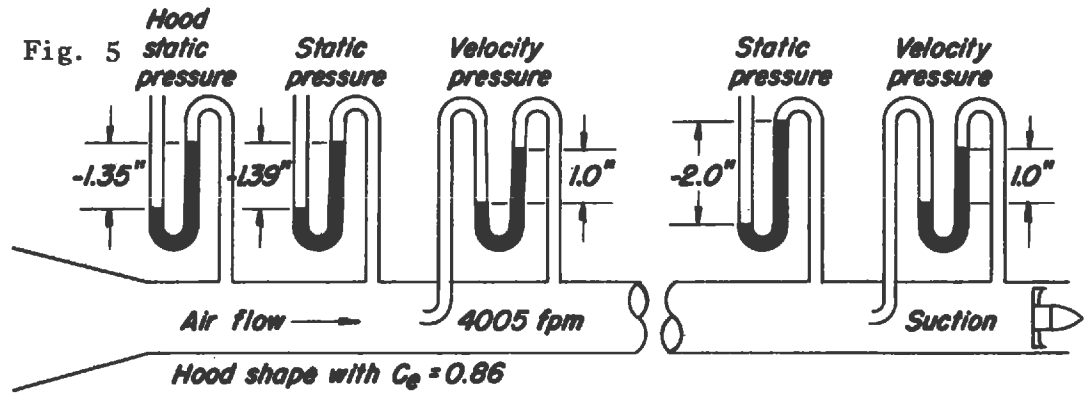
PRESSURE IN THIS PIPE ABOVE ATMOSPHERIC PRESSURE

Fig. 4



PRESSURE IN THIS PIPE BELOW ATMOSPHERIC PRESSURE

Fig. 5



Figures 2, 3, 4 and 5 were reproduced by permission from Industrial Ventilation - A Manual of Recommended Practice, published by the American Conference of Industrial Hygienists' Committee on Industrial Ventilation.

At standard air conditions of 70°F and 1 atmosphere, the formula becomes:

$$V = 4005\sqrt{VP} \quad (5)$$

$$\text{or } Q = 4005\, A\sqrt{VP} \quad (6)$$

Static Pressure

Duct Friction Losses. Air in motion encounters resistance along the surfaces confining the flow. Some of the energy of the air is given up in overcoming this resistance and manifests itself as heat. The rougher the surface or the higher the flow rate or the greater the surface to volume ratio, the higher the duct friction loss will be.

This loss varies directly as the length, inversely as the diameter, and directly as the square of the air velocity. It can be calculated for any fluid flow in circular ducts from the Fanning or Darcy equation:

$$F = 4f\left(\frac{V^2}{2g}\right)\left(\frac{L}{D}\right) \quad (7)$$

where F = Friction loss in feet of fluid flowing

V = Fluid velocity (ft/sec)

g = Gravitational acceleration (ft/sec^2)

L = Duct length (ft)

D = Duct diameter (ft)

f = Friction factor - a dimensionless function of Reynolds number

The Fanning friction factor, f, varies with Reynolds number according to the following graph:

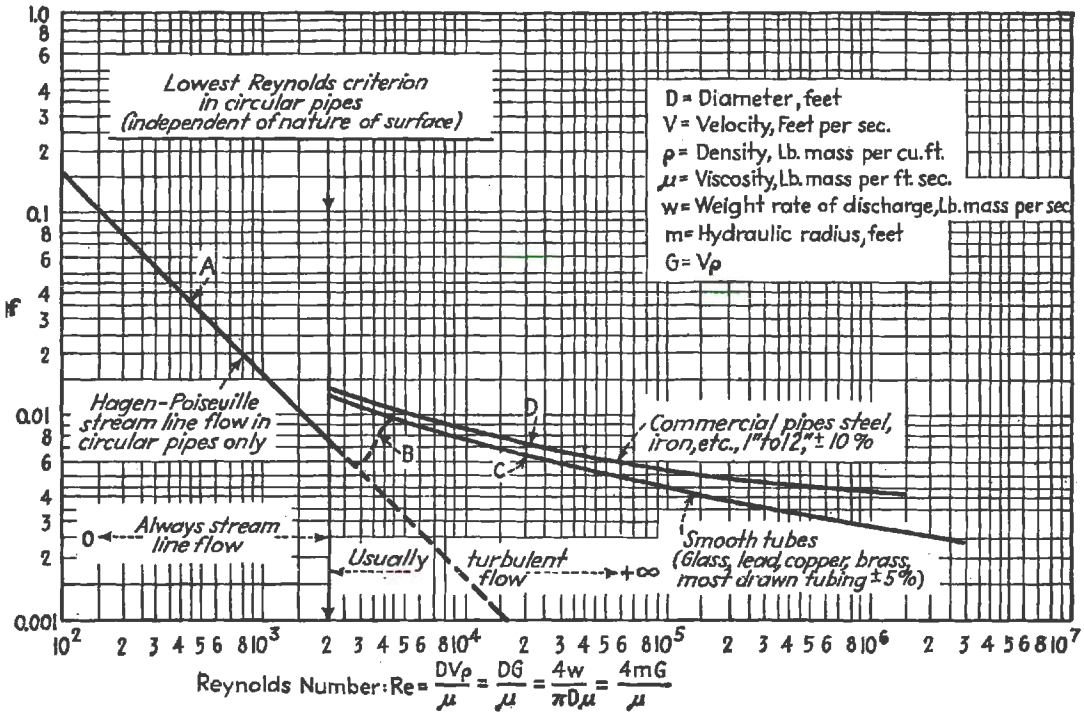


Figure 6. - Fanning Friction Factors, f, in Equations (7) and (8). (Reprinted by permission from CHEMICAL ENGINEERS' HANDBOOK, edited by J.H. Perry. Third Edition, Copyright, 1950. McGraw-Hill Book Company, Inc.)

If air at standard conditions is the fluid under consideration, the Fanning equation can be reduced to the convenient form:

$$h_f = .000298\frac{fV^2}{D} \qquad (8)$$

where: h_f = Friction loss per 100 ft. of duct ("H_2O)

f = Fanning friction factor

V = Air velocity (ft/min)

D = Duct diameter (inches)

Equation (8) has been used to plot friction loss, air velocity, duct diameter, and air volume. This is fortunate because using the charts is much easier than performing the mathematical manipulations for each change of conditions in the air flow through a system.

Figures 6-15A and 6-15B in Industrial Ventilation[1] are charts of this type. Note that if two factors are known the other two can be found.

The effect of air density will introduce errors no larger than 2% for densities corresponding to temperatures between 32°F and 100°F. Uncertainties in f and duct design approximations will obscure such errors.

Dynamic Losses. Another type of loss encountered is dynamic loss (also called turbulent or shock loss). Losses of this type are caused by air turbulence which takes place whenever air enters a duct or whenever air flow through a duct changes direction or velocity.

The first type of dynamic loss, entry loss (h_e), is a drop in pressure caused by air turbulence when air enters a duct. Turbulence losses vary with the type of opening and are defined for ducts and common types of hoods in Figure 6-5, Industrial Ventilation[1].

This entry loss plus an acceleration energy required to get the air moving at a given velocity (one VP) make up Hood Static Pressure (SP_h). SP_h is expressed mathematically as:

$$SP_h = h_e + VP \qquad (9)$$

or can be measured directly three duct diameters down from the hood entry. The calculation of SP_h is the first step in systems analysis or design, which is discussed in Section C-10.

The coefficient of entry C_e is a measure of how well an entry is able to convert SP into VP.

C_e = Actual rate of flow caused by hood static pressure / Theoretical flow if hood static pressure were converted to VP with 100% efficiency

$$C_e = \frac{4005\ A\sqrt{VP}}{4005\ A\sqrt{SP_h}} = \frac{\sqrt{VP}}{\sqrt{SP_h}}$$

substituting ($\sqrt{VP} = C_e\sqrt{SP_h}$) in formula (6) yields another formula for calculating Q:

$$Q = 4005\ A\ C_e\sqrt{SP_h} \qquad (10)$$

A second type of dynamic loss occurs when air is made to change directions in a duct. This occurs in elbows, junctions and weather caps. The pressure drops caused by these losses are expressed in units of "equivalent length". For example, if an elbow of 10" diameter is said to have an equivalent length of 14 (see Industrial Ventilation[1], Figure 6-10), this means that the loss through that elbow will be the same as the loss through 14 feet of straight pipe of the same diameter, operating under the same conditions.

Another system of defining the losses discussed above is to express the losses in terms of velocity pressure. For example, a loss in a 2.00 D elbow of .27 VP (Industrial Ventilation[1], Figure 6-11) means that the pressure drop is equal to (.27) x (VP of the air stream at that point).

Summary

The result of the friction and dynamic losses is to cause a pressure drop in the system. This pressure drop is also defined in a simplified version of the Bernoulli theorem:

$$\text{Letting} \quad \frac{V^2}{2g} = VP$$

$$P_v = SP$$

$$VP_1 + SP_1 = VP_2 + SP_2 + \text{Losses} \quad (11)$$

This simplified equation becomes the basis for all systems design or analysis work, as it enables one to calculate the SP and VP at any point in a system if the SP and VP at another point can be calculated.

References

1. _____: Industrial Ventilation - A Manual of Recommended Practice. Committee on Industrial Ventilation, American Conference of Governmental Industrial Hygienists, 8th ed., 1964.

2. May, James W.: The Physics of Air. American Air Filter Company, Louisville, Kentucky, 1962.

3. _____: The Industrial Environment - Its Evaluation and Control. Public Health Service Publication 614, 1961. (Revision in progress).

AIR-FLOW MEASUREMENTS

Introduction

To determine the effectiveness of environmental control by any ventilation system, an evaluation of the air movement must be made. In some instances, a qualitative determination, such as observing a vapor trail or the smoke from a smoke tube, may be sufficient to evaluate the effectiveness of control. In most cases, however, quantitative measurements are needed for proper evaluation. Various physical properties of moving air are used to make this evaluation.

In many cases the velocity of the air is determined by use of the various instruments. Then the quantity of moving air can be calculated by the formula:

$$Q = AV$$

where: Q = Volumetric rate of flow, cfm
A = The cross-sectional area through which the flow takes place, ft^2
V = average linear velocity in cross-section A, lfm

This formula is applicable where the cross-sectional area A can be defined.

All flowmeters, with the possible exception of well-designed Pitot tubes, should be calibrated against a primary or reliable secondary standard before use, and periodically checked for calibration thereafter.

Air-flow Measuring Devices

Pressure Sensing Instruments

Pitot Tube. The Pitot tube is the standard instrument for measuring the velocity of air. A standard Pitot tube, carefully made, will need no calibration. It consists of two concentric tubes - an impact tube whose opening faces axially into the flow, and the static pressure tube, a larger tube with circumferential openings. The difference between the impact and the static pressures is the velocity pressure. Bernoulli's theorem applied to a Pitot tube in an air stream simplifies to the formula:

$$V = \sqrt{\frac{2p}{\rho}} \qquad (2)$$

where: V = velocity of the airstream
p = velocity pressure measured by the Pitot tube
ρ = density of the air

The pressure, p, may be expressed in terms of a head of some fluid by:

$$p = \rho_f g h_f$$

where ρ_f = density of the fluid
g = acceleration of gravity
h_f = head of fluid

Expressing V in feet per minute, and p in inches of water (h_v), formula (2) becomes:

$$V = 1097\sqrt{\frac{h_v}{\rho}}$$

where ρ = density of air or gas in lbs/ft^3.

If the Pitot tube is to be used with air at standard conditions (70^oF. and 1 atm.), formula (2) reduces to:

$$C\,V = 4005\sqrt{h_v} \qquad (3)$$

where V = velocity in ft/min.
h_v = velocity head in inches of H_2O

The Pitot tube measures pressure in the location in which it is placed. One measurement of pressure is usually insufficient to define the rate of air flow through a duct. However, if a dircular duct is straight for at least

---

Prepared by: Morton Lippmann
Revised by: George W. Fisher

10 diameters upstream from the point of measurement, a single center-line Pitot reading may give a fairly accurate estimate of the flow rate. It has been found that under such conditions, the average velocity is about 90% of the center-line velocity, or:

$$Q = 0.9\ AV_c$$

where V_c = center-line velocity

When accuracy is required, however, a Pitot traverse should be made. In a rectangular duct, the cross-section should be divided into at least sixteen equal areas, and a velocity measurement made at the center of each. The average of the velocities measured is the average velocity in the duct. For a traverse of a round duct, the cross-sectional area should be divided by means of concentric circles into regions of equal area. Measurements should then be taken at radii whose circles divide each region into two equal areas. A ten point traverse is frequently made. It consists of a set of ten measurements along one line, and another set of ten measurements along a line perpendicular to the first line - both lines being perpendicular to the axis of the duct. The velocities are then averaged to get the average velocity in the duct. Tables of distances from pipe wall for 6 and 10 point traverses in round ducts of various sizes are given on page 9-4 of Industrial Ventilation.(1)

The Pitot tube is used largely for measuring air velocity in ducts, or at high velocity supply and exhaust openings. Its accuracy in the low velocity range is limited by the sensitivity of the pressure sensing device used with it. At 3000 1fm, the velocity pressure is 0.56" H_2O; at 2000 1fm, 0.25" H_2O; at 1500 1fm 0.14" H_2O; and at 1000 1fm 0.062" H_2O. Therefore, a vertical water manometer should not be used for velocities below about 2000 1fm. An accurately balanced inclined manometer may be used down to about 600 1fm. Various accurate low pressure measuring devices may be used with a Pitot tube, but their use is usually limited to the laboratory where they can be carefully set up and balanced.

Pitot tubes can be used under a wide range of conditions; there are no moving parts to get out of order, and if made of stainless steel, they are resistant to corrosion and high temperatures. Standard Pitot tubes cannot be used in dusty atmospheres because they will become plugged, but modified Pitot tubes for use in such atmospheres have been made.(1) This type of Pitot tube should always be calibrated before use.

Further discussion of the Pitot tube can be found in many standard references.(2,5,7,8)

Deflecting Vane Anemometer (Velometer). This type of instrument is widely used for air flow and static pressure measurement by industrial hygienists and heating and air conditioning engineers. Various uses of this device are illustrated on page 9-8 of Industrial Ventilation.(1) Commercial Velometers are available with one pressure and one, two, three or four velocity ranges with a variety of jets for each range. The Velometer has the advantage of being a direct-reading instrument with ranges as low as 50 1fm or as high as 24,000 1fm, although readings below 100 1fm are not very accurate.

The Velometer operates by the pressure of an air stream against a spring loaded swinging vane. It is fairly rugged, and if calibrated regularly, the readings are sufficiently accurate for most field work. If the temperature of the air stream is outside the range $70^{\circ}F \pm 30^{\circ}$ or the altitude is greater than 1000 feet, density corrections should be made. Dust or corrosive gases should not be permitted to enter the instrument. Velometers can be ordered with dust filters which will retain light dust loadings. If the instrument is calibrated with a dust filter in place, it must always be used in this manner.

In order to measure air flow or total pressure in a duct with a Velometer, a hole is required in the duct large enough to accommodate the appropriate probe. Such a hole is larger than that required for a Pitot tube and may be difficult to provide. When measuring static pressure, a minimum 3/8-inch diameter hole is needed, and it must be free from burrs in order to obtain accurate measurements.

Rotating vane anemometer. This instrument consists of a propeller connected through gears to a dial which counts rotations. The dial reads in linear feet, which when divided by the time interval of the measurement, gives linear feet per unit time. The instrument is available with 3, 4, or 6-inch wheels.

The rotating vane anemometer is useful for measuring air velocities in the range 200 to 2000 lfm. It is largely self-averaging for fluctuating flows and for traversing large openings. Readings are little affected by deviations in alignment with the direction of air flow up to about 20°. The instrument is delicate and should be calibrated frequently. It cannot be used in dusty or corrosive air. Since this type of instrument is rarely used in temperatures or pressures far from standard, pressure-temperature corrections are seldom needed.

For a more complete discussion of the rotating vane anemometer see Chapter 7, The Measurement of Air Flow.(5)

Other pressure actuated anemometers. One type of commercially available anemometer is a direct reading instrument designed primarily for measuring air velocity at discharge grilles in the range of 30 - 2700 lfm. It has a rotating impeller whose motion is opposed by a spring. When the velocity pressure on the impeller and the spring tension balance, the velocity can be read from the dial indicator. Another type of anemometer available is the cup anemometer used in meteorological work. It consists of four hemispherical cups mounted on light arms from a hub which turns freely on a vertical axis. This type of instrument has little application to ventilation work.

Thermal Anemometers

Heated thermometer anemometer. The heated thermometer anemometer is based on the principle that the rate of heat loss from an object at elevated temperature is a function of air movement over the object. The instrument consists of a pair of thermometers, one with an electric resistance coil about the bulb to which a known voltage may be applied. The other is an ordinary thermometer. In use, the two thermometers are placed in the air stream to be measured, and the applied voltage adjusted to give a difference of 15° to 30° in the two thermometer readings. The thermometers are then allowed to come to equilibrium. Velocity of the air stream can be determined from a calibration chart using the temperature difference between the two thermometers, or by application of the temperature difference in the following equation:

$$\frac{\text{net milliwatts}}{\Delta t} = K + K'\sqrt{V}$$

where: net milliwatts = fixed value for each thermometer at each voltage. A table is supplied with each instrument by the manufacturer.

Δt = temperature difference between thermometers.

K and K′ = constants; values for each supplied by manufacturer.

V = velocity of air stream.

(Instead of constants for the above equations, some manufacturers supply a calibration curve).

The instrument indicates the cooling power rather than the velocity of an

air stream. It is affected by radiant heat and convection. In the absence of radiant heat sources, it is very accurate as a velocity measuring device when carefully used. The instrument is nondirectional and has a range of 15 to 500 lfm. It is fragile and its use is time consuming. A more complete discussion of the heated thermometer anemometer is given by Yaglou.[9]

Heated thermocouple anemometer. This instrument works in the same manner as the heated thermometer anemometer, but uses thermocouples in place of thermometers. The advantages gained are a reduction in size, increased ruggedness of the sensing elements, faster response time, and it can be readily calibrated as a direct reading instrument. Several commercial instruments of this type are available. One has a range of 10 to 2000 lfm. Another has a range of 10-5500 lfm plus a static pressure scale of 0-4" H_2O. A third type has a range of 0-8000 lfm, a static pressure range of 0-10" H_2O, and also a Fahrenheit temperature scale. The calibration of this type of instrument should be checked regularly. Most of these instruments use batteries which must be replaced periodically.

Hot-wire anemometer. A hot-wire anemometer consists of a fine, electrically heated wire which is placed in the air stream to be measured. The cooling effect on the wire will depend on the velocity of the air stream. As the wire cools, its resistance changes, and this change can be measured with a bridge circuit. If calibrated properly, this type of instrument can be used to measure a wide range of air velocities. Pannell[6] gives a detailed discussion of this type of instrument.

Kata thermometer. The Kata thermometer is a special thermometer with a large bulb, containing alcohol, and a stem with marks at 95 to 100°F. It is heated above 100°F. and the time required for it to cool from 100 to 95°F. is a measure of the nondirectional air velocity in the room. It was designed for comfort ventilation measurement, and its surface to volume ratio is similar to that of the human body. The useful velocity range is 25 to 500 lfm. It has the disadvantage of being fragile and having large radiation and convection areas.

Ionization Anemometer

The ionization anemometer consists of a duct section having polonium on one surface and an alpha ray counter on the opposite surface. As the air flow increases, fewer α particles reach the counter, and the instrument can be calibrated in terms of velocity. Extremely low velocities can be measured with this instrument. Lovelock and Wasilewska[4] give further information about this instrument.

Air Quantity Measurement

Orifice Meter

An orifice meter is simply a restriction in a pipe between two pressure taps. There are several types of orifice meters used, but the simplest and most common is the square edged orifice, which is a very short cylindrical passage in a thin metal plate. If it is properly constructed, the orifice plate will be at right angles to the flow and the surface will be carefully smoothed to remove burrs and other irregularities. Since the square edged orifice is easily constructed and the meter as a whole is simple and comparatively inexpensive, it is widely used as an accurate flow meter. Orifice meters are seldom used as permanent flowmeters in ventilation systems because of their high permanent pressure loss. They are more typically used in the ventilation laboratory for calibration purposes. Permanent head loss will vary from 40 to 90 percent of the static pressure drop across the orifice as the ratio of orifice diameter to pipe diameter varies from 0.8 to 0.3. Orifices have been intensively studied and their performance characteristics can be predicted if they are constructed to

standard proportions. Detailed discussions of orifices and orifice equations are contained in standard references.[2,5,7] On pages 191-193 of Industrial Health Engineering[2] Brandt gives a simplified formula for calculating the flow rate through a thin-plate orifice with flange taps for air at standard conditions. In it, Q is a function of the orifice diameter, the pressure drop across the orifice, and a factor K which varies with the Reynolds number and the ratio of orifice to duct diameter. A table of K's is given in this discussion.

Critical Flow Orifice

For a given set of upstream conditions, the discharge of a gas from a restricted opening will increase with a decrease in the ratio of absolute pressures P_2/P_1, where P_1 is the upstream pressure, and P_2 the downstream, until the velocity through the opening reaches the velocity of sound. The value of P_2/P_1 at which the acoustic velocity is just attained is known as the critical pressure ratio. The pressure in the throat will not fall below the throat pressure attained with the critical pressure ratio, even if a much lower downstream pressure exists. Therefore, when the pressure ratio is less than the critical, the rate of flow through the restricted opening is dependent only on the upstream pressure.

It can be shown[7,8] that for air flowing through rounded orifices, nozzles and Venturis, when $P_2 < 0.53\ P_1$, and $S_1/S_2 > 25$, the flow rate of air, w, is determined by:

$$w = 0.533\frac{C_v S_2 P_1}{\sqrt{T_1}} \text{ in lb/sec}$$

where: C_v = coefficient of velocity (normally 1)

P_1 = upstream absolute pressure in lb/in^2

T_1 = upstream temperature in ^oR

S_1 = upstream cross-sectional area in in^2

S_2 = orifice area in in^2

Critical flow orifices are widely used with industrial hygiene sampling instruments.

Flowmeters

The term flowmeter is often used to designate any restricted opening through which the rate of flow has been determined by calibration. One can be made by inserting a restriction in a pipe section, or for smaller flows by using a piece of glass capillary tubing as the restriction. A typical laboratory flowmeter consists of a U-tube manometer with a capillary connected across its legs.

Venturi Meters

A Venturi meter consists of a 25° contraction to a throat, and a 7° re-expansion to the original size. This differs from the orifice meter where the changes in cross section are sudden. The great advantage of the Venturi meter over the standard orifice is that the permanent reduction in static pressure is small, because the velocity head in the throat is largely reconverted to static pressure by the gradual re-enlargement. A well designed and constructed Venturi will have a permanent static pressure loss of only 0.1 to 0.2

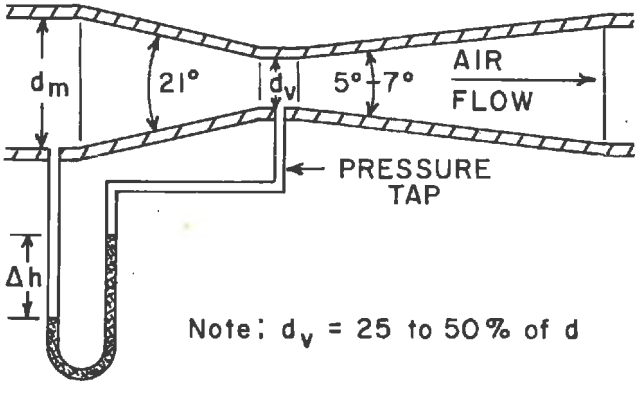


Figure 1. - Standard Venturi Meter.

of the Venturi reading as compared to 0.4 to 0.9 with a square-edged orifice. Standard references[5,7,8] discuss Venturi meters and Venturi equations. In Industrial Health Engineering[2], Brandt gives a simplified formula for flow through a Venturi which is applicable to air at 70°F. and 1 atm. when the ratio of throat diameter to duct diameter is between ¼ and ½ as follows:

$$Q = 21.2\ r^2 d^2\ \ h$$

where: Q = flow in cfm
r = ratio of throat to duct diameter
d = duct diameter in inches
h = pressure drop in inches of water

Rotameters

A rotameter consists of a vertical transparent tube, increasing in cross-sectional area from bottom to top, in which an upward flowing fluid stream supports a float at a level determined by the flow rate. The underlying principle of operation is the same as that of an orifice or Venturi meter. The float is the restriction, but instead of a variable pressure drop and a constant area of opening, there is a constant head (the weight of the float) and a variable area.

Rotameters are available for measuring a wide range of air flow rates, from as little as 10 cc/min. of air, to as much as 350 cfm. They are used rarely to measure ventilation air flow, but find wide use in laboratory calibration and metering.

Thermal Meters

A thermal meter measures mass of air or gas flow rate with negligible pressure loss. It consists of a heating element in a duct section between two points at which the temperature of the air or gas stream is measured. The temperature difference between the two points is dependent on the mass rate of flow and the heat input.

Mixture Metering

The principle of mixture metering is similar to that of thermal metering. Instead of adding heat and measuring temperature difference, a contaminant is added and its increase in concentration is measured; or clean air is added and reduction in concentration is measured. This method is useful for metering corrosive gas streams. The measuring device may react to some physical property such as thermal conductivity, or vapor pressure.

Other Meters

Other meters include wet gas meters, dry gas meters, and spirometers which are discussed in Section B-3, Air Flow Calibrations.

Indirect Measurement of Air Flow

Air volume flowing into a standard exhaust hood can be determined fairly accurately from a measurement of throat suction. The volume of air entering a branch can be calculated from the formula:

$$Q = 1097\ C_e A \sqrt{\frac{h_s}{\rho}}$$

or for air at 70°F. and 1 atm.

$$Q = 4005\ C_e A \sqrt{h_s}$$

where: Q = air flow rate in cfm
C_e = coefficient of entry
A = area of duct in ft^2
h_s = suction pressure in inches of water
ρ = density of air in lb/ft^3

The suction pressure should be measured one to three pipe diameters downstream from the throat of the exhaust inlet in a straight section of duct. Coefficients of entry for standard exhaust hood types are tabulated in various references.[1,2,3] For hoods not listed, an estimate may be made by breaking down the hood into component simple

shapes, and combining the coefficient of the simple shapes. This method is valid if the hood does not accumulate deposits, or become dented before the point of measurement, which would affect the magnitude of the entry loss. Very little expense is involved in making throat suction measurements. It is a very useful method for periodic inspections since it will quickly show how the exhaust rate compares with previous values. Since the only equipment required is a piece of rubber tubing and a manometer or pressure gage, determinations can be made quickly and easily even by nontechnical inspectors.

For further discussion of flow measurement by throat suction see Industrial Health Engineering(2) and Plant Process Ventilation.(3)

References

1. _____: Industrial Ventilation - A Manual of Recommended Practice. ACGIH Committee on Industrial Ventilation, P. O. Box 453, Lansing, Michigan, 8th Ed., 1964.
2. Brandt, A. D.: Industrial Health Engineering. John Wiley and Sons, New York, 1947.
3. Hemeon, W.C.L.: Plant and Process Ventilation. The Industrial Press, New York, 1955.
4. Lovelock, J. E.; and Wasilewska, E. M.: An Ionization Anemometer. J. Sc. Inst., 26:367-370 (Nov.1949).
5. Ower, E.: The Measurement of Air Flow. Chapman and Hall, London, 3rd Ed., 1949.
6. Pannell: The Measurement of Fluid Velocity and Pressure. Arnold, London, 1924.
7. Perry, J. H., Editor: Chemical Engineers' Handbook. McGraw-Hill Book Company, New York, 3rd Ed., 1950.
8. Walker, W.E.; Lewis, W. K.; McAdams, W. H.; and Gilliland, E. R.: Principles of Chemical Engineering. McGraw-Hill Book Company, New York, 3rd Ed., 1937.
9. Yaglou, C. P.: The Heated Thermometer Anemometer. J. Ind. Hyg. & Toxic., 20:497 (1938).

LOCAL EXHAUST SYSTEMS

Introduction

The control of atmospheric contamination in industry is most frequently accomplished by means of local exhaust ventilation. By "local exhaust" is meant the capture and removal of the contaminant close to the point of origin, before the contaminant enters the worker's breathing zone, or is disseminated into the general environment. This type of ventilation is preferred over general or dilution ventilation in most cases for the following reasons:

1. Less air needs to be exhausted to control the contaminant. This means that a smaller fan will be used and the power consumption will probably be lower. Also, the make-up required will be less, and in cold climates or air conditioned shops, this may be an important economic factor.
2. The contaminant is captured before it enters the worker's breathing zone and not after it has been generally disseminated in the room. With some highly toxic and radioactive substances, this is the only method of exhaust possible.
3. The contaminant is more highly concentrated in the conveying air stream. This is an important factor where the air will be cleaned either to salvage some valuable product or to prevent air pollution.

Capture Velocities

The capture velocity is that velocity in front of the local exhaust hood that is necessary to overcome the dispersive forces and room air currents and capture the contaminant. The table below from Industrial Ventilation[1] (page 4-5) gives the range of velocities:

Table 1. - Range of Capture Velocities

Condition of Dispersion of Contaminant	Examples	Capture Velocity fpm
Released with practically no velocity into quiet air.	Evaporation from tanks; degreasing; etc.	50-100
Released at low velocity into moderately still air.	Spray booths; intermittent container filling; low speed conveyor transfers; welding; plating; pickling.	100-200
Active generation into zone or rapid air motion.	Spray painting in shallow booths; barrel filling; conveyor loading; crushers.	200-500
Released at high initial velocity into zone of very rapid air motion.	Grinding; abrasive blasting; tumbling.	500-2000

Capture velocities for specific operations are given in Industrial Ventilation[1] and the ASA Z9.1-1951 for Ventilation and Operation of Open Surface Tanks[2] gives capture velocities for such tanks. For open surface tanks the corners have the minimum surface velocity, but may give off only a small amount of contaminant even if not well controlled.

Prepared by: Howard E. Ayer
Revised by: Jeremiah R. Lynch

Air Flow into Openings

The theory of air flow into round openings as developed by DallaValle is based on the postulation of a point source of suction to which air flows towards from all directions. The velocity at any point a distance X from the source would then equal the quantity of air flowing (Q) divided by the area of the sphere of radius X or,

$$V = \frac{Q}{4\pi X^2}$$

The closest approach to a point source is the end of an open pipe. Air flows into the open pipe from all directions. A diagram of the equal velocity contours for a circular opening is shown below:

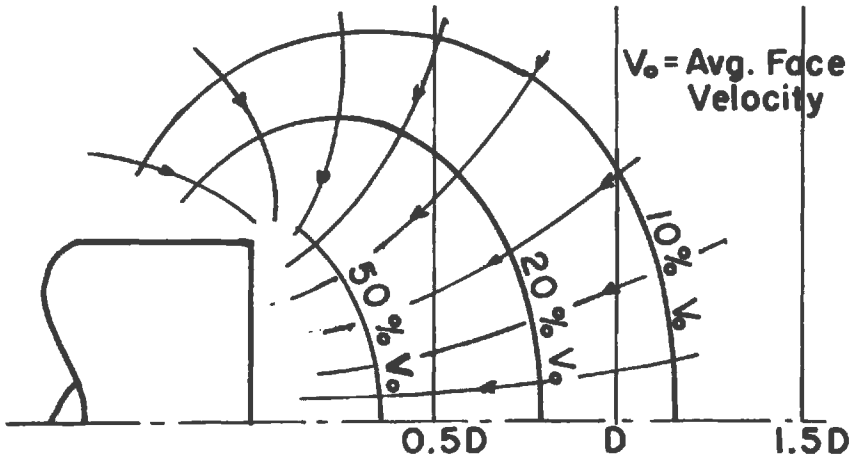


Figure 1. - Velocity Contours for Circular Openings.

These contours do not agree exactly with the theoretical equation but are best described by the following equation:

$$V = \frac{Q}{10X^2 + A}$$

where V = velocity at point X in fpm
Q = air flow in cfm
X = centerline distance in feet
A = pipe area in feet^2

Addition of a flange to the pipe will obviously increase the velocity in front of the opening since it will prevent flow into the pipe from the back. Where the hood can be placed on a table, this will cut off another 90^o sector from which air is drawn, and further increase the effectiveness of the hood. The velocities under these conditions are then:

1. Open pipe end: $V = \frac{Q}{10X^2 + A}$

2. Flanged pipe ends: $V = \frac{Q}{0.75(10X^2 + A)}$

3. Flanged pipe on table: $V = \frac{Q}{0.5(10X^2 + A)}$

Air flow into a square opening will be quite similar to that into a circular opening. For rectangular openings, however, the shape plays a part, and the equation for velocity becomes:

4. $V = \frac{bQ}{X^2 + bA}$ where b is an aspect ratio which depends on the ratio of the sides. A table of b values for different width to length ratios is given in Industrial Ventilation[1] (Fig. 4-4).

Slots

Caution should be used in applying the above equation as the width to length ratio approaches 0.1, since the opening is thus becoming more like a slot. Using the same line of reasoning as DallaValle, Silverman[3] considered the slot as a line source of suction. Ignoring the ends, the area of influence would then be a cylinder, and the velocity would be:

$$V = \frac{Q}{2 \pi X L}$$

where X is the perpendicular distance from a slot of length L. In practice, of course, the theoretical conditions do not apply exactly, and the constant used is thus not 6.28, but 3.7 and the equation becomes:

5. $$V = \frac{Q}{3.7 X L}$$

for freely suspended slots. Flanging a slot will give the same sort of benefits as flanging an open pipe, so that only 75% of the air is required to produce the same velocity at a given point in front of the slot, and thus:

6. $$V = \frac{Q}{2.8 X L} .$$

Where the area of influence is still further reduced because the slot is in a corner, velocity contours will be again improved.

Several important local exhaust characteristics can be deduced from these equations. The control velocity produced by a hood is approximately inversely proportional to the square of the distance from the hood. Also the additions of side and back flanges to a hood reduce the amount of air required for a given velocity. These points argue strongly for good enclosure as a means of obtaining adequate control with minimum air volumes. Another point is the lack of significance of the velocity at the entrance to the pipe. For example, a 6" diameter open pipe with a flow of 350 cfm will produce a velocity of 130 fpm on the centerline 6" from the open end of the pipe. If the pipe size is decreased to 4", the inlet velocity increases 2½ times but the velocity 6" from the open end of the pipe is only 4% higher. However, the static pressure and power required will be 6.4 times higher.

Dispersive Forces

The above discussions have all been based on still air. Actually, in many operations natural forces or air currents will influence the air-flow pattern about the operation to be controlled. Such influences will include the rising current of warm air above a hot process, the aspiration of air downward by falling rocks from a conveyor, the rotative air currents about a grinding wheel, natural air currents caused by room ventilation, and similar external forces. Hemeon[4] discusses these dispersive forces in detail, giving their influences on hoods and air flows, and methods for correcting them. In the design of hoods it is preferable to take advantage of these dispersive forces whenever possible. The standard grinding wheel hood design, for example, takes advantage of the motion of the chips or fragments from the wheel. While these large particles, which are projected long distances, are of no hygienic importance, they do create an aspirated air current which carries small particles along with them into the hood and also their collection aids in housekeeping. In woodworking operations, housekeeping and fire prevention are the major factors in control so that collection of the large, non-respirable particles is most important.

Canopy Hoods

Canopy hoods offer a good example of the advantages of dispersive forces and also of enclosures. Canopy hoods are commonly used over hot processes or heat-generating operations. They may be either natural draft or fan actuated.

There are two basic problems with simple canopy hoods:

(1) The difficulty of exhausting a large enough quantity of air to remove all the smoke, fume or gas from the process, plus the secondary air which is aspirated upward, and
(2) The sensitivity of the simple canopy hood to room air currents such as drafts from man coolers and movement of machinery and personnel past the hood. Canopy hoods cannot be used for toxic vapors if workers must bend over the tank.

A commonly used formula for canopy hoods, given in Industrial Ventilation[1] (page 4-4) is Q = 1.4 PDV where Q = cfm air flow, P is tank perimeter in feet, D is height of the hood above the tank, and V is average vertical velocity of air under the edges of the canopy in fpm. If this formula is used the difficulty is in selection of V, which is said to range from 50 - 500 fpm depending on cross-drafts. The formula does not take into account the process going on underneath it. Hemeon[4] and Silverman have attempted to take into account the process and the amount of hot air which the hood is required to remove. Hemeon's formulae, which require some explanation, are found in Chapter 8, "Exhaust for Hot Processes."[4] Silverman uses the formula:

$$Q = 278\ A \sqrt{H''\ (1 - \frac{Ta}{Ts}\ \frac{Ms}{Ma})}$$

where Q = air volume, cfm
A = tank area, ft^2
H'' = height in inches from liquid level or tank top to exhaust hood
Ta = absolute temperature of air
Ts = absolute temperature of substance
Ma = molecular weight of air
(In practice let $\frac{Ms}{Ma} = 1$)
Ms = molecular weight of substance

This formula for total air to be removed is derived from the theoretical convective velocity, and the form shown here assumes an entrainment ratio of 4.

In many simple canopy hoods material will be observed "spilling" out the sides. One attempt to remedy this has been the so-called double canopy hood where an inner cone or pyramid blocks all but the outer rim of the hood, with the idea that a high velocity at the edges will solve the problem, since spillage is occurring only at the edges. The previous comments about high velocity entrances apply - the double hood does nothing except change air distribution, and if better distribution is needed it may be more easily obtained with simple baffles which distribute the air volume throughout the face area, not just on the outside.

All canopy hoods are very subject to cross-drafts, and should be avoided where possible. Considerable improvement can be achieved by enclosing one or more sides. Enclosure also will reduce air entrainment from hot processes, thus making control possible with a smaller quantity of air.

Where an enclosure can be used, it will benefit any local exhaust system. For some materials such as very finely divided solids, enclosure is the only solution, since any face velocity adequate for control without enclosure would cause loss of product. An example is the highly toxic, finely divided silica with a mean size of less than one micron.

Air Distribution in Hoods

Air flow into a hood should be uniform throughout its cross-section. In the case of slots for lateral exhaust, for example, this may be done by "fish-tail" design. An easier and also effective method of design, however, is to provide a velocity of 2000-2500 feet per minute into the slot, and use a low-velocity plenum or large area chamber behind it. The 2000 fpm velocity will give a static pressure behind the slot of 0.4 inches

of water. This pressure drop will equalize flow along the slot, since the static pressure throughout the plenum will be practically uniform. For large shallow hoods such as paint spray booths, lab hoods, side draft shakeout hoods and the like, the same principle may be used. In these cases, unequal flow may tend to occur, with a concentration in the neighborhood of the take-off. Baffles provided for the hood increase velocity and pressure drop into the hood, giving the plenum effect. Where the face velocity over the whole hood is relatively high, or where the hood or booth is quite deep, no baffles will be required.

Entrance Losses in Hoods

The negative static pressure that exists a short distance up a duct from a hood, beyond the vena contracta, if any, at the duct entry, is called the "hood static pressure" SP_h. The energy it represents has accomplished two things:

1. Accelerated the air from zero velocity to duct velocity.
2. Overcome the friction losses occasioned by turbulence of the air on entering the hood and duct.

Thus: $SP_h = VP + h_e$

Where VP = velocity pressure in the duct

h_e = hood entry loss

The hood entry loss h_e is a function of the velocity pressure and for most common hoods and types of entries the factor F in the equation $h_e = FVP$ is listed in Industrial Ventilation(1) (page 4-11). For hoods such as plain or flanged ducts, canopes or grinding wheel type hoods, the VP referred to is that in the duct and the hood static pressure can be expressed:

$$SP_h = VP_{duct} + h_e = VP_{duct} + FVP_{duct}$$

$$= (1 + F)VP_{duct}$$

However, for slot and plenum hoods there are two entry losses, one through the slot and the other into the duct.

Thus: $SP_h = h_{e_{slot}} + VP_{duct} + h_{e_{duct}} =$

$F_{slot} \times VP_{slot} + VP_{duct} + F_{duct} \times VP_{duct}$

The velocity pressure resulting from acceleration through the slot is not lost as long as the slot velocity is less than the duct velocity, as is usually the case.

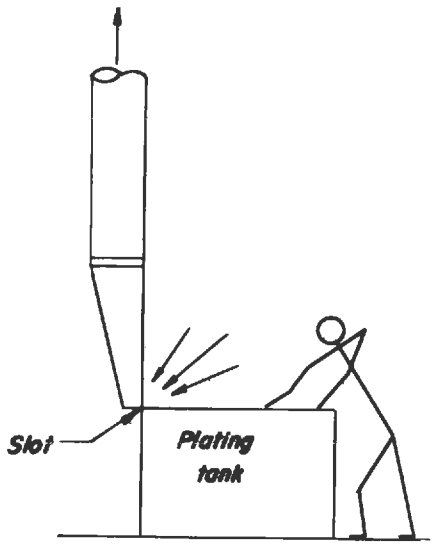


Figure 2. - Example of a hood with slot and plenum. (Reproduced with permission from Industrial Ventilation).

Slot velocity = 2000 fpm
Duct velocity = 4000 fpm

For this type hood the entry loss factors are as follows:

$F_{slot} = 1.78$

$F_{duct} = 0.25$ (90° included angle)

From the equation:

$$VP = \left[\frac{V}{4000}\right]^2$$

$VP_{2000} = 0.25$" water

$VP_{4000} = 1.00$" water

Therefore:

$$SP_h = 1.78 \times 0.25 + 1.00 + 0.25 \times 1.00$$

$$= 0.45 + 1.00 + 0.25$$

$$= 1.70'' \text{ water}$$

This is the pressure that would actually be measured at a hole drilled in the side of the duct three diameters up from the hood.

Another constant used to define the performance of a hood is the "coefficient of entry" C_e. This is defined as the ratio of the actual air flow to the flow that would exist if all of the static pressure were present as velocity pressure. Thus:

$$C_e = \frac{Q_{actual}}{Q_{VP = SP_h}}$$

$$= \frac{4000\ A \sqrt{VP}}{4000\ A \sqrt{SP_h}} = \sqrt{\frac{VP}{SP_h}}$$

This quantity is constant for a given shape of hood and is very useful for determining the flow into a hood by a single hood static pressure reading. The coefficient of entry C_e is related to the hood entry loss factor, f, by the following equation:

$$C_e = \sqrt{\frac{1}{1 + f}}$$

Industrial Ventilation[1] (page 4-11) gives C_e and f for a number of simple hood shapes. Most of the coefficients for more complicated hoods are obtained by combining some of these simple shapes.

Static Suction

One method of specifying air volume for a hood is to specify the hood static pressure and pipe size. It has long been recognized that the hood static pressure at an average grinding wheel hood is 2 inches of water. This reflects a conveying velocity of 4500 feet per minute and an entrance coefficient (C_e) of 0.78. In the same way, for many pieces of machinery where the type of exhaust hood is relatively standard, a specification of the static suction and the pipe size is given in Alden[5] and other reference sources. Specification of the static suction without pipe size is, of course, meaningless, since decrease in pipe size will increase the velocity pressure and thus the static suction, while actually decreasing the total flow and the degree of control. Static suction measurements are quite useful for standard hoods, or for systems where the air flow has been previously measured, to estimate the quantity of air flowing through the hood.

Low Volume High Velocity Systems

As a general rule the dust generated by chipping and grinding operations will travel in the direction of the air stream created by the motion of the tool. For this reason the conventional hoods for such tools are designed to catch this air stream and thus capture the dust. However, for certain types of portable grinding, brushing and chipping tools the usual hoods are not practical since they would get in the way of the work. For these tools special "low volume high velocity" systems have been developed incorporating small closely fitting "extractor" heads operating at a vacuum in the order of 5 inches of mercury. The slot velocities at these extractor heads range from 5000 to 15,000 feet per minute and the control velocities created are sufficiently high to overwhelm the air current caused by the motion of the tool. In these systems the air is conveyed in pipes and flexible hoses rather than sheet metal ducts, and the compressibility of air must be considered in the system design and in the selection of an exhauster.

Examples of Hood Selection

1. Select a hood for an 18" diameter, 3" wide grinding wheel.

Grinding wheel hoods are found in VS-411 in Industrial Ventilation[1] (page 5-41). For an 18" diameter wheel a good enclosure with not more than 25% of the wheel exposed, will require 500 cfm. The required minimum branch duct velocity of 4500 feet per minute will be obtained with a 4½" diameter duct. Using a tapered take-off, the hood entry loss, $h_e = 0.40\ VP = 0.40 \times 1.26 = 0.50"\ H_2O$.

2. Select a suitable hood and the quantity of exhaust ventilation necessary for a 2' x 6' chromic acid plating tank operated at 120°F.

From the ASA Standard Table 5-5-6 given in Industrial Ventilation[1] (page 5-55), the major contaminant released by such a tank is chromic acid mist. The threshold limit for chromic acid mist is 0.1 mg/m.. Again using the ASA 9.1 code of Industrial Ventilation[1] the hazard potential is A (Table 5-5-1, page 5-53), the rate is 3 (Table 5-5-2, page 5-53), so the hood is class A-3. Use a hood similar to VS-503A (page 5-49), From Table 5-5-3 (page 5-53) the control velocity for lateral exhaust is 75 feet per minute. Using Table 5-5-4 (page 5-54), with a tank width to tank length ratio of 0.33 and a minimum control velocity of 75 fpm, 110 cfm is required per square feet of tank area, or 110 x 12 = 1320 cfm. The slot area will then be 1320/2000 = 0.66 square foot, and the slot width will be

$$\frac{0.66}{6} = 0.11 \text{ foot or } 1.3 \text{ inches.}$$

Using a duct velocity of 2000 fpm, the hood entry loss for a 450° take-off will be 1.78 x slot VP + 0.25 duct VP or (1.78 x 0.25) + (0.25 x 0.25) = 0.45 + 0.06 = 0.51 inches of water and the hood static pressure will be 0.51 + 0.25 = 0.76" H_2O.

References

1. _____: Industrial Ventilation - A Manual of Recommended Practice. ACGIH Committee on Industrial Ventilation, P. O. Box 453, Lansing, Michigan, 48902 8th ed., 1964.

2. _____: ASA Z9.1-1951 - Ventilation and Operation of Open-Surface Tanks. American Standards Association, 10 E. 40th St., New York 16, N. Y.

3. Silverman, L.: Velocity characteristics of narrow exhaust slots. J. Ind. Hyg. & Toxicol., 24:267 (1942).

4. Hemeon, W.C.L.: Plant and Process Ventilation. The Industrial Press, New York, 2nd ed., 1963.

5. Alden, John L.: Design of Industrial Exhaust Systems. The Industrial Press, New York, 1949.

Other References

_____: ASA Z9.2-1960 - Fundamentals Governing the Design and Operation of Local Exhaust Systems, American Standards Association, 10 E. 40th St., New York 16, N. Y.

FANS AND BLOWERS

Introduction

Fans are the mechanical air moving devices which make most industrial exhaust systems operate. Although the industrial hygiene engineer is not generally concerned with the design of fans, it is important to understand enough of their theory and performance to be able to select, evaluate, and modify industrial exhaust fans.

Fans are usually divided into two groups, centrifugal fans and axial-flow fans. The distinction between the two is the direction of air flow produced. Air enters a centrifugal fan parallel to the fan shaft, and is discharged in a plane perpendicular to it. Axial-flow fans create a flow parallel to the fan shaft.

To be effective, any fan must increase the static pressure in the air passing through it. Potential energy due to the static pressure produced can then be utilized to move air. The increase in pressure developed in a fan arises from two sources: one is the centrifugal force caused by the rotation of an enclosed volume of air, and the other is the increased velocity in the direction of flow imparted to the air by the blades. The centrifugal force causes a compression of the gas, or static pressure, which is dependent on the increase in the centrifugal component of the velocity of the air. The longer the blades, the greater the static pressure developed. Increased velocity in the direction of flow on the other hand, causes an increased velocity pressure, which must be converted to static pressure in order to be effective. Except for friction losses, total pressure remains constant in such a conversion since it is the algebraic summation of static and velocity pressures. Conversion in both centrifugal and axial-flow fans can be performed by using a constantly diverging discharge duct at the fan outlet, and is aided by an efficient impeller arrangement and shape. With centrifugal fans, scroll, or increase in cross section, also aids conversion.

Prepared by: Morton Lippman
Revised by: Jeremiah R. Lynch

Fan Types

Axial-Flow Fans

By definition, axial-flow fans create air flow parallel to the fan shaft. Within this group are low pressure propeller and duct fans, and vane-axial fans which can develop higher pressures.

Propeller Fans. These fans consist of a simple propeller, usually with three or four blades mounted in a ring or cage. Since they have no way of confining the flow, conversion of velocity pressure to static pressure is minimal and usually they are not used against more than one inch of water static pressure. In addition, due to the waste of energy in the useless helical and unconverted radial motion of the air, they are very inefficient.

Tube-Axial Fans. If a propeller is placed in a duct a part of the energy wasted in radial motion can be converted to velocity and static pressure. This tube-axial type fan is able to operate against slightly higher resistances than a propeller fan but is still sensitive to variations in resistance. They are suitable for air containing materials that tend to collect on fan blades.

Vane-Axial Fans. Tube-axial fans with guide vanes either ahead of or following the impeller are classed as vane-axial fans. These vanes straighten the helical flow pattern and convert velocity pressure into useful static pressure, increasing the efficiency. The impeller blades usually have an airfoil shape.

Vane-axial fans are best suited to clean air applications. They are far more efficient, and can operate at much higher pressures (up to 11" H_2O) with lower noise levels, than propeller fans. Vane-axial fans are competitive with centrifugal fans for most industrial operations. At static pressures below about 4" H_2O they are quieter than centrifugal fans. At higher pressures they may be noisier, but are not necessarily so. Their efficiency may be as high or higher than centrifugal fans and their compact design and simplicity of mounting and erection make them a good choice when conditions permit their use. In the very high capacity range, vane-axial fans are superior to centrifugal fans, possibly except for the production of more noise.

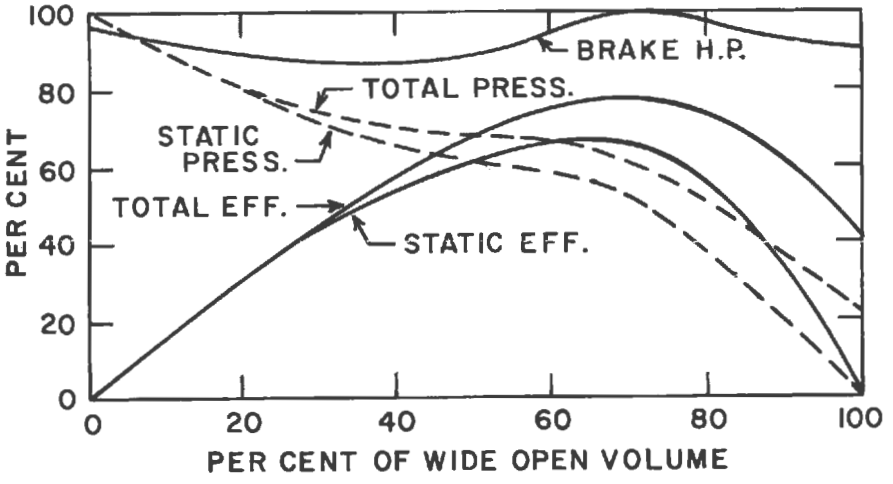


Figure 1. - Typical performance curves for an axial-flow fan.

Figure 1 shows characteristic curves for an axial-flow fan. The actual characteristics for a given fan would depend to a great extent on the size and speed of the fan and the type of blades. Axial-flow fans have their highest horsepower requirement and static pressures at the blocked-tight position.

Centrifugal Fans.

Centrifugal fans are most commonly used for industrial supply and exhaust systems, pneumatic conveying, or forced draft applications. In these fans the air enters parallel to the fan shaft, turns 90°, passes between the blades of the fan wheel and discharges at right angles to the inlet. The increase in velocity and static pressure is principally caused by centrifugal force. The spiral shape of the case or scroll determines the conversion of velocity pressure to static pressure. Centrifugal fans are classified as forward curved, radial, or backward curved according to the shape of the blade.

Radial Blade or Paddle-Wheel Fans. Paddle-wheel fans were the first type developed, and are the only type that should be used for handling dirty air. Centrifugal force tends to keep the blades clean, permitting applications where suspended solids must pass through the fan. The rotors usually have between 5 and 12 blades, and the blades and housing are ruggedly fabricated. Characteristic curves for this type of fan (Figure 2) show the kind of performance that can be expected. The pressure characteristic is stable and shows only a minor reduction from the peak pressures at the blocked-tight position. Horsepower required is almost a straight line function of air volume. Maximum static efficiency for this type of fan is about 65% to 70%.

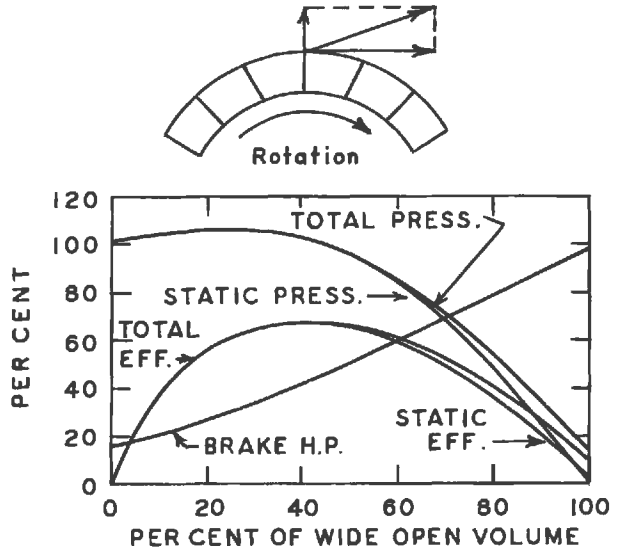


Figure 2.-Curves for radial-blade fans.

Forward-curved Blade Fans. These fans usually have a large number of closely spaced shallow blades. They are the standard "utility" or "squirrel cage" blower used for a number of general ventilation applications where erosion of the blades or the accumulation of solids is not a problem. The forward curve of the blade builds up a high velocity which is partially converted to static pressure by the scroll.

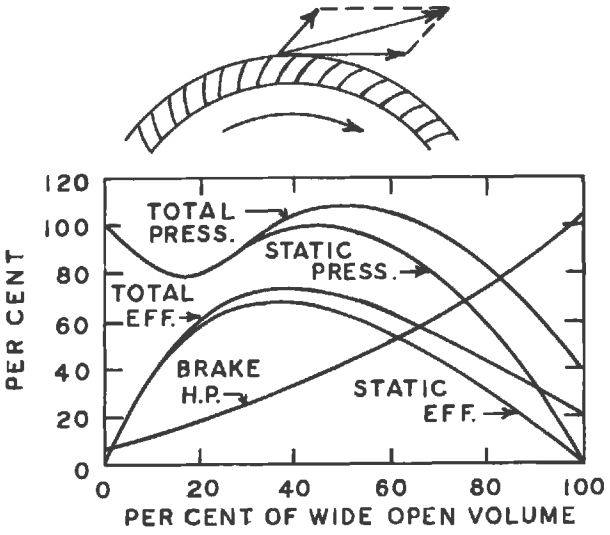


Figure 3. - Forward-curved blade fans.

Figure 3 shows the performance characteristics of this type of fan. Because of the characteristic dips in the pressure curves, stability of operation is uncertain unless the operating point is on the wide-open side of the pressure peak. Maximum efficiencies may be about 70%, but because of possible instability, the selected point of operation is usually in the lower efficiency range. Usual operating static pressures in ventilating applications are from 2 to 4" H_2O, although forward-curved fans are available that will develop higher pressures.

The horsepower requirement for this type of fan rises rapidly with increasing volume. Unless pressure requirements can be estimated accurately, the use of oversized motors is necessary.

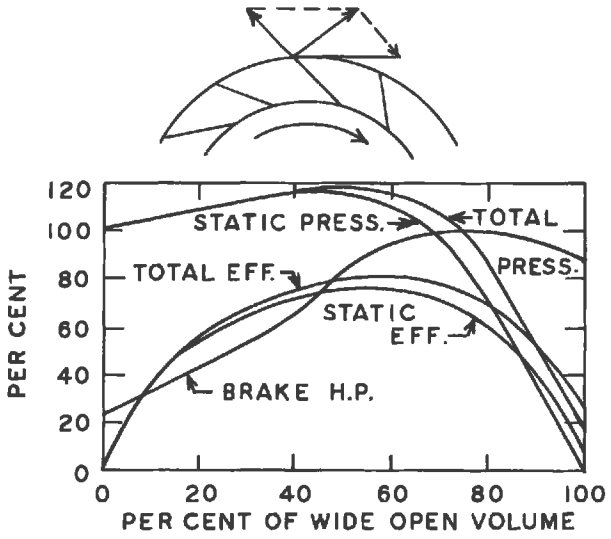


Figure 4. - Backward-curved blade fans.

Backward-curved Blade Fans. These fans have a few flat, curved or airfoil shaped blades. The shape of the blades is such that the air tends to follow the blade even at high volumes. Consequently, the static pressure and efficiency are high at high volumes and the horsepower decreases as the volume approaches wide-open. This eliminates the necessity for over-sized motors. Because of the backward curve of the blades, higher speeds are needed, and this necessitates more rugged construction. The noise will also be higher although this may not be a problem in industrial applications.

Comparison of Fan Types

The table below is a general comparison of fan types used in ventilation. Numerous exceptions to the listing will be found in individual cases, but these data serve as an indication of comparative characteristics.

	Centrifugal Fans			Axial-Flow Fans	
	Backwardly Inclined	Radial Blade	Forwardly Curved	Vane-Axial	Propeller and Tube-axial
First Cost*	High*	Medium*	Low*	Low	Lowest
Efficiency	High	Medium	Medium	High	Lowest
Stability of Operation	Good	Good	Poor	Good	Poor
Space Required	Medium	Medium	Med-Small	Small	Small
Tip Speed (Noise)	High	Medium	Low	Medium	High
Resistance to Abrasion	Medium	Good	Poor	Medium	Medium
Ability to Handle Sticky Materials	Medium	Good	Poor	Medium	Medium

*On normal duty applications, first cost is essentially the same for all three types of centrifugal fans. Heavy duty applications reflect the comparison listed.

Rating Point of a Fan

A fan running at a constant speed can operate at any volume static pressure combination along its characteristic curve. The actual point it will operate at is determined by the resistance of the duct and hood system it is connected to. The fan will operate at the intersection of the fan characteristic curve and the system volume versus static pressure curve (see Figure 5). For most systems, this curve has a parabolic shape given by an equation in the form $p \propto Q^2$. However, certain systems contain some viscous resistance such as filters which introduce a $p \propto Q$ component to the system characteristic. To increase the rate of flow of a fan operating on a given system, it is necessary to increase the speed of the fan. The effect will be to produce a new rating point at high volume and higher static pressure, as predicted by the fan laws.

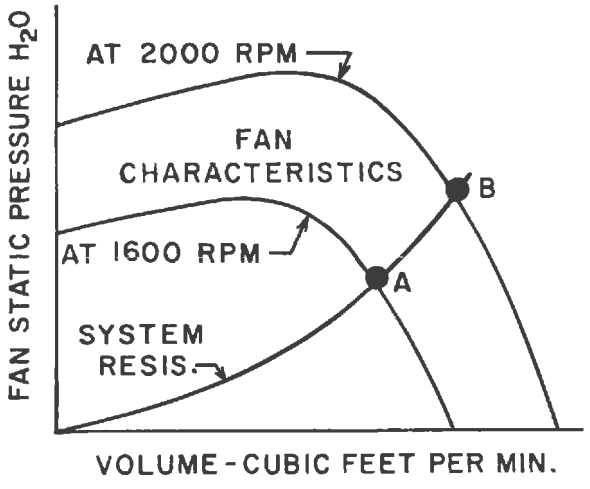


Figure 5. - Rating points of a fan.

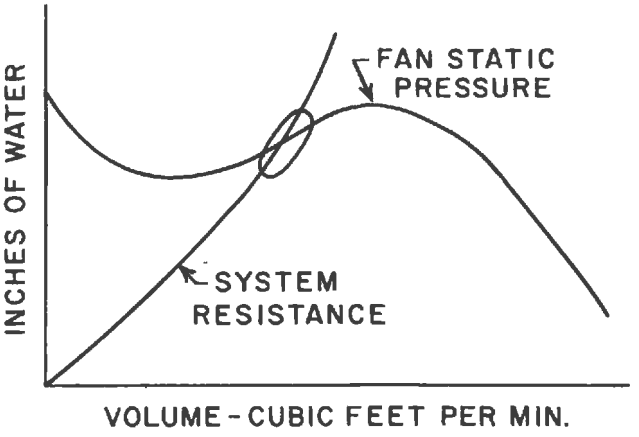


Figure 6. - Point of rating showing unstable condition.

Figure 6 is the same type of curve for a forward-curved fan operating on the blocked-tight side of the pressure curve. Because the pressure characteristic in this case follows the system characteristic closely, a small change in flow would cause a comparatively

large change in pressure drop, resulting in "hunting" and instability of operation. This would be magnified if there were long duct lengths or plenums in the system. A momentary increase in volume would not be compensated immediately by a corresponding pressure rise. Following this lag there would be an overcompensation, and the flow would begin to surge. This effect is most pronounced with forward-curved fans, but can occur with radial and backward-inclined fans also.

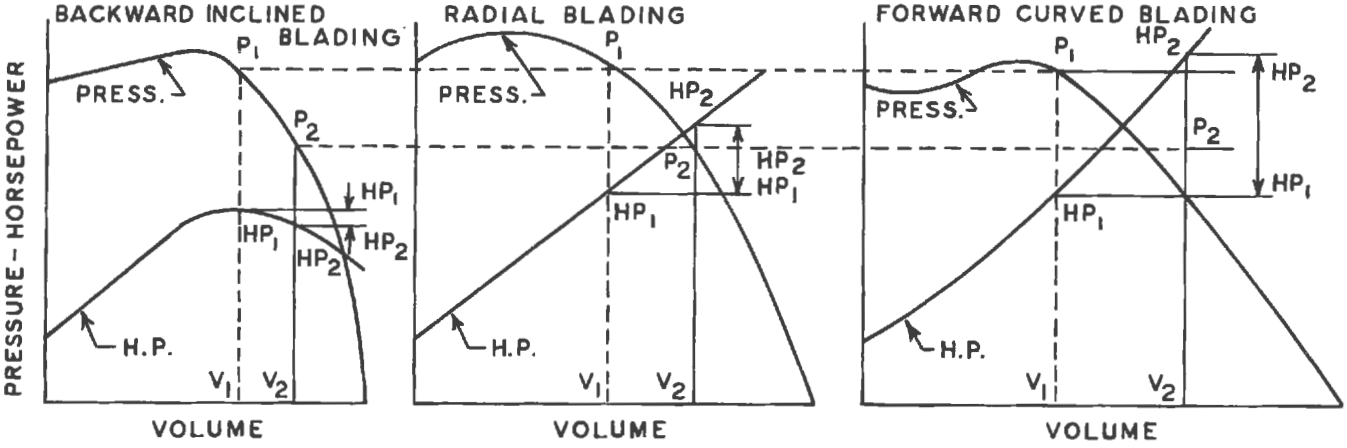


Figure 7. - Effect of Power Change on Power Consumption. (Reprinted by permission from Air Conditioning, Heating, and Ventilating, May 1957, Copyright - The Industrial Press).

Figure 7 demonstrates the effect a change in system resistance can have on power consumption on the three types of centrifugal fan. In this example, P_1, P_2, and V_1 are equal for each fan type, and except for efficiency difference HP_1 is approximately equal. A reduced system resistance requires a new point of operation, and in the case shown in Figure 7 results in a slight decrease in power required from a backward-inclined fan, an increase in a radial fan, and a considerably larger power increase in a forward-curved fan. From this example, and the discussion on fan rating, and the inaccuracies in calculating system resistance that are normally encountered, the importance of selecting a fan that will be stable throughout a fairly wide range near the design point can be seen.

Fan Laws

The fan laws are a series of relationships which permit the prediction of a new operating point of a fan when some factor is changed. Given the volume, pressure and horsepower of a fan operating at one rating point it is possible to determine the same quantities for the new rating point obtained by changing the fan size or speed or the air density. It is assumed in these equations that the efficiency remains constant, and in the case of variation in fan size, that they are fans geometrically similar and members of the same homologous series. Only a few of the more useful laws will be given here. A complete listing can be found in Chapter 8 of reference 1.

Q = volumetric flow rate
P = pressure
HP = horsepower
N = fan speed in RPM
S = fan size - expressed as some linear dimension of the fan
D = density of the air.

Three basic equations and situations are as follows:

1. Variation in fan speed with constant fan size, air density and system.

$$\frac{Q_1}{Q_2} = \frac{N_1}{N_2};\ \frac{P_1}{P_2} = \left(\frac{N_1}{N_2}\right)^2;\ \frac{HP_1}{HP_2} = \left(\frac{N_1}{N_2}\right)^3$$

All three of the above quantities vary simultaneously as the fan speed is changed. It is important to note that this fan law predicts a new ratingpoint which is also on the system resistance curve if it is of the form $P \propto Q^2$. Consequently, if a change in fan speed is made on a fan connected to a constant system, with no other changes, the new volume, pressure and power will be predicted by this law. The same is not necessarily true with the other fan laws.

2. Variation in fansize within a homologous series with constant speed and air density.

$$\frac{Q_1}{Q_2} = \left(\frac{S_1}{S_2}\right)^3;\ \frac{P_1}{P_2} = \left(\frac{S_1}{S_2}\right)^2;\ \frac{HP_1}{HP_2} = \left(\frac{S_1}{S_2}\right)^5$$

In homologous series of fans, the dimensions of each size of fan are proportional to the same dimensions of the other fans in the series. Therefore, any dimension, or a number proportional to a dimension, may beused as "size" in these relations. This fan law is useful in developing a multi-rating table for a series of fans from tests on one fan. The point on the new fan characteristic predicted is not on the system characteristic.

3. Variation in air density with constant volume, system, fan size and speed.

$$Q = \text{constant};\ \frac{P_1}{P_2} = \frac{D_1}{D_2};\ \frac{HP_1}{HP_2} = \frac{D_1}{D_2}$$

This last law illustrates that fans are essentially constant volume machines. When other factors remain constant and the density of the air changes, a fan will deliver the same volume rate of flow, rather than the same mass rate of flow.

Calculating Fan Efficiency

Mechanical (Total) Efficiency

$$\text{M.E.} = \frac{\text{Horsepower output}}{\text{Horsepower input}} \times 100$$

Horsepower output = Air Horsepower (AHP)

$$\text{AHP} = \frac{Q(\text{ft}^3/\text{min}) \times P_t(\text{in. } H_2O)}{12\ (\text{in/ft})} \times \frac{62.4\ (\text{lb/ft}^3\ H_2O)}{33{,}000\ (\frac{\text{ft-lb}}{\text{min}}/\text{HP})}$$

$$\text{AHP} = \frac{QP_t}{6350}$$

where Q = cfm

P_t = total fanpressure ininches of water

Horsepower input = Brake Horsepower(BHP);

$$\therefore \text{M.E.} = \frac{\text{AHP}}{\text{BHP}} \times 100 = \frac{QP_t}{6350} \times \frac{100}{\text{BHP}}$$

Static Efficiency (S.E.)

This is always less than mechanical efficiency because all velocity pressure cannot be converted to static pressure.

$$\text{S.E.} = \frac{QP_s}{6350} \times \frac{100}{\text{BHP}}$$

(P_s = fan static pressure in inches of water).

Fan Standards and Definitions

Most fan manufacturers belong to the "Air Moving and Conditioning Association, Inc." (AMCA) formerly known as the "National Association of Fan Manufacturers, Inc." (NAFM). This group has established fan standards, definitions, and test codes, which are summarized in their Bulletin 210, available on request. When comparing fans of different manufacturers it should be notedwhether the AMCA test code was used.

The AMCA pressure definitions are:

1. The total pressure (P_t) of a fan is the rise of pressure from fan inlet to fan outlet as measured by two impact tubes, one in the fan inlet duct and one in the fan discharge duct, corrected for friction to the fan inlet and outlet respectively.

2. The velocity pressure (P_v) of a fan is the pressure corresponding to the average velocity determination from the volume of air flow at the fan outlet area.

3. The static pressure (P_s) of the fan is the total pressure (P_t) diminished by the fan velocity pressure (P_v).

$$\therefore P_s = P_t - P_v$$

Confusion often arises between the "fan static pressure" and the static pressure in the duct on the inlet or outlet side of the fan. Fan static pressure as defined by the equation above is the change in total pressure from inlet to outlet minus the outlet velocity pressure. This is the same as the total change in static pressure from inlet to outlet minus the inlet velocity pressure. This second form is used in the Industrial Ventilation Manual.

Fan Selection

General Considerations

In order to select the proper fan for a given purpose the following information is required:

1. Air volume to be moved.

2. Fan static pressure required.

3. Cleanliness of the air. A radial-bladed centrifugal fan will be needed if the air stream contains a high concentration of particulates.

4. Direct or belt drive. Belt drive can be changed for changes in air volume handled. Direct drive is inflexible but occupies less space and requires less maintenance.

5. Noise level. Noise level is a function of tip speed. It is not usually a limiting factor in industrial applications.

6. Special conditions such as high operating temperatures, corrosive, inflammable or explosive materials, and unusual space limitations.

Use of Fan Catalogs

Based on the information listed above a decision must be made as to what type of fan is to be used so that the proper fan catalog may be selected. Fan catalogs contain information on the performance, construction, dimensions, modifications and accessories of one or more homologous series of different sizes of a type of fan. The performance data is most generally presented in the form of "multi-rating tables." Each of these tables gives the RPM and horsepower required to move a volume of air against a fan static pressure for one size of fan. The figures for points of maximum efficiency are sometimes underlined or in bold face type. The ideal fan size for a given task is the one where the point of highest efficiency coincides with the desired flow and static pressure. If this point falls between two fan sizes in a series, the smaller fan is usually selected, since it will cost less and will operate on a steeper sloped portion of its characteristic curve, where the stability is better. In most cases interpolation between the points in the tables will be necessary. A static pressure interpolation should be made first, followed by the volume interpolation. Arithmetic interpolations are usually satisfactory. The variations in the procedure required for conditions other than 70°F and one atmosphere are discussed in the section on system design.

Estimating Capacities of Fans

It is sometimes desirable to know the capacity of an operating fan or estimate the performance characteristics of an available fan for which no rating table is available.

For an in-use fan where a Pitot traverse of the duct would be impractical, it is often possible to obtain the inlet and outlet static pressures and RPM. With this information and the fan rating table, the flow may be estimated. Other methods involving measurement of the motor current also are used.

When an available fan is lacking sufficient name plate data, or when the rating tables for such a fan are missing, it is possible to estimate the fan capacity with reasonable accuracy.

Hemeon describes a method in his book, Plant and Process Ventilation[2], of plotting an approximate fan characteristic curve for centrifugal fans from two simple pressure measurements. These measurements are the static pressure at no flow and the velocity pressure at wide open flow. The curve obtained by this method is a fair approximation of the actual characteristic, at least on the right-hand side of the pressure peak. Since the right-hand side is the proper operating region, this should be sufficient.

Fan Location and Maintenance

When air movers are used in conjunction with air cleaning devices, good practice dictates that they be located downstream from the cleaner. This will minimize erosion, abrasion, and the building up of sticky deposits on the fan blades. The pressure drop through the air cleaner will be the same whether up or downstream of the fan. The fan should be located so that the inlet and exhaust ducts go through the minimum number of bends, and it should have an anti-vibration mounting. Another important consideration in the location of a fan is accessibility for inspection and servicing. However, all ducts inside the building should be on the inlet side of the fan under negative pressure to avoid leakage of contaminant into occupied spaces.

When a fan is installed it should be checked for proper mounting, grounding, and correct direction of rotation. The last is an easy error to make since a fan will move some air even when rotating backward. In addition to this initial inspection, a regular schedule of inspection should be established to include the following points:

1. Tightness of mounting bolts and ground connections
2. Tension and condition of belts
3. Lubrication of bearings, etc.
4. Abrasion or accumulation of material on rotor
5. Proper static pressure
6. Vibration check.

Other Air Moving Devices

Injectors (ejectors)

Injectors operate by inducing a large volume of secondary air flow by means of a small volume of high velocity air flow, or jet. The air jet expands in a duct and entrains and mixes with surrounding air. A cylindrical injector has its jet in a simple round duct and has a maximum efficiency of about 5%, while in a Venturi injector the jet discharges into a Venturi throat, and can develop efficiencies as high as 12%.

Although injectors are highly inefficient compared with fans, and may require 5 to 10 times the power for the same purpose, there are many instances where an injector would be the air mover of choice. The advantages of injectors include:

1. Lower first cost

2. Simplicity of installation

3. They can exhaust an air stream containing sticky or highly corrosive material which would rapidly foul a fan. The injector would also become fouled, but at a much slower rate, and it would be much easier to clean.

4. They can be used in areas where electric power is not available or in areas where an explosive atmosphere may be encountered.

In instances where the power cost would be negligible, or where the demand is intermittent, or where an existing compressed air source is available, the lower first cost of an injector would overbalance its higher operating cost and it would be a good choice.

In Plant and Process Ventilation[2], Hemeon[2] discusses injectors and injector design. Brandt[3], in Industrial Health Engineering, also discussed injectors. A more complete discussion of this subject may be found in Design of Injectors for Low Pressure Air Flow, by McElroy[4] and Perry's Chemical Engineers' Handbook[5] contains a discussion of steam jet ejectors.

References

1. Jorgensen, R.: Editor. Fan Engineering. Buffalo Forge Co., Buffalo, New York, 6th ed., 1961.

2. Hemeon, W.C.L.: Plant and Process Ventilation. The Industrial Press, New York, 2nd ed., 1963.

3. Brandt, A.D.: Industrial Health Engineering. John Wiley & Sons, Inc., New York, 1947.

4. McElroy, G.E.: Design of Injectors for Low Pressure Air Flow. Technical Paper 678, U. S. Bureau of Mines, 1945.

5. Chemical Engineers' Handbook. John H. Perry, Editor. McGraw - Hill Book Company, Inc., New York, 4th ed., 1963.

DESIGN OF VENTILATION SYSTEMS

Principles employed in the design of ventilation systems should be known by all industrial hygienists. In this section only the methods of selecting proper pipe size, fan characteristics and principles of good practice are discussed. Construction specifications may be found in Industrial Ventilation[1] and other design manuals.

General Principles of System Design

The most important decisions in the design of local exhaust systems are (1) what operations are to be exhausted, (2) what type of hood will be used at each operation and (3) what quantity of air will be exhausted from each. The chief objectives of ventilation are to protect the health, safety and comfort of employees at the lowest possible cost, and it is the duty of the industrial hygienist to see that these objectives are not sacrificed for lower cost.

Individual Exhaust Fans vs Multibranch Systems

When the operations to be exhausted and their hood types and exhaust volumes have been determined, the number and arrangement of exhaust systems must be decided upon. One or more large systems may be used, a number of individual exhaust fans may be used, or a combination of individual and multibranch systems may be selected. Some advantages of each system are as follows:

Individual system, including a fan for each operation:

1. Where operations are intermittent and contamination occurs only while operations are being performed, fans will be run only part time resulting in a smaller amount of air being exhausted, thus saving on make-up air and power costs.
2. More flexible - operations may be added or removed as desired, provided suitable adjustment is made in make-up air.
3. Exhaust fan may be readily interlocked with operation so that operation cannot be conducted without exhaust.
4. Operations producing non-toxic, non-irritating dusts may be connected to a unit collector and exhausted back into the work room.
5. Optimum velocities may be used for different operations.

Multiple branch system:

1. The total initial cost will usually be lower than for an individual fan for each operation.
2. Where operations are conducted in series, exhaust for all steps is assured as long as the system is operating.
3. Design of make-up air system is simplified.
4. There are fewer collectors and fans to maintain.
5. This system may be the only feasible arrangement in large, multistory buildings.

General Considerations

Whether individual or multibranch systems are used, there are a number of good practices which should be followed. Many of these are illustrated in Industrial Ventilation[1] and include the following:

1. The materials of construction, type of fan and collector must be suitable for the systems employed. When contaminants are

Prepared by: Howard E. Ayer

corrosive, dusty, linty, sticky, explosive, hot, hygroscopic or present any other special problems, these factors must be considered and the systems designed with them in mind.

2. The system should be designed for easy maintenance. Where particulates are handled there should be provision for cleaning out any section of duct work. Fans and collectors should be placed so as to be readily accessible for maintenance.

3. Long radius elbows and 30° or 45° entrances should be used. Miter elbows and tee entrances should be avoided.

4. The system should be designed for a constant velocity throughout, with entrances at enlargements, so that the total area of a main duct is essentially that of the entering branches. In particulate carrying systems, however, the branch velocity is often higher than the main, to reduce likelihood of plugging in the smaller branch ducts.

5. Systems should be designed with some ability to handle increased resistance in the event that additions are made or an existing installation is changed so as to cause higher resistance than anticipated.

6. Where density of air differs appreciably from standard conditions (70°F, 760 mm Hg) density corrections should be made. Practically, variations of less than ±30°F or 1000 feet elevation may be ignored.

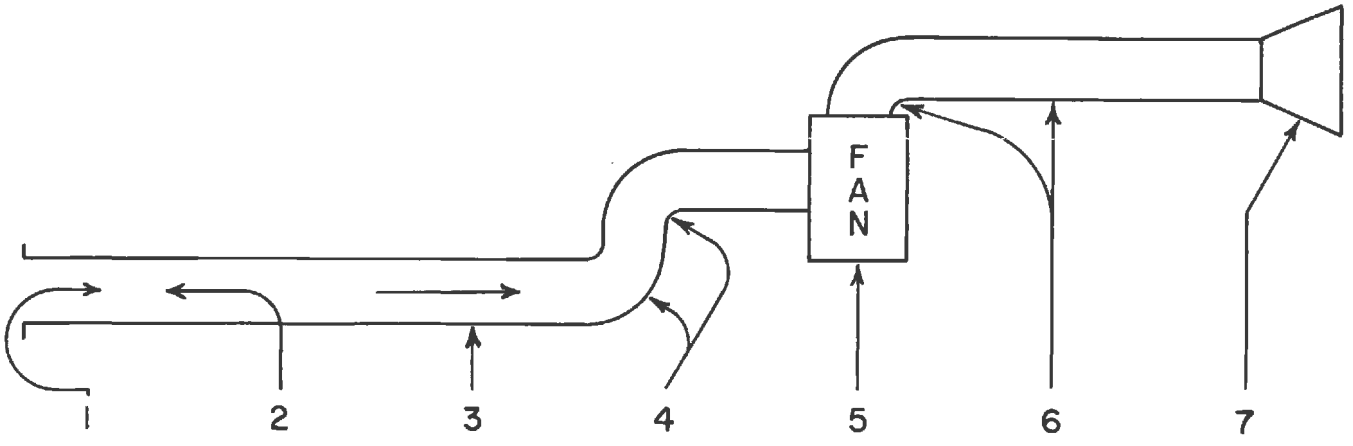


Figure 1. - Pressure changes in a ventilation system.

1. Entrance loss (fraction of v.p. to twice v.p. depending on characteristics of opening).

2. Acceleration (= v.p.)

3. Friction loss in straight pipe. Proportional to length of pipe and velocity squared; inversely proportional to pipe diameter.

4. Turbulence loss in elbows.

5. All pressure losses and velocity pressure supplied by fan.

6. Friction and turbulence losses on outlet calculated same as on inlet side.

7. Velocity pressure loss can be reduced by gradual expansion at outlet.

Pressure Losses

Pressure changes occur in any ventilation system as shown in Figure 1. In the diagram shown, the first loss is at the entrance. This loss is caused by turbulence as the air is forced into the hood by the higher pressure existing in the room. For a well-rounded, bell-mouth type of entrance this loss will be negligible. Where the air enters the duct in such a way that the velocities are higher than required at one portion and are quite low in another portion, losses will be higher. For a given entrance shape and duct size the pressure loss will vary directly with the velocity pressure or velocity squared. For a flanged entrance shown in Figure 1, for example, the entrance loss will be one-half of the velocity pressure in the duct downstream. This would result in entrances losses for various velocities as follows:

Average Duct Velocity (Feet per minute)	Velocity Pressure (Inches of water)	Pressure Loss Flanged Entry (Inches of water)
1000	0.06	0.03
2000	0.25	0.13
4000	1.00	0.50
6000	2.24	1.12
8000	4.00	2.00

In Industrial Ventilation[1] this would be shown by the formula:

$$h_e = 0.49 \text{ v.p.}$$

Similarly, if this were a plain, unflanged duct opening, the entrance loss would be almost as great as the velocity pressure ($h_e = 0.93$ v.p.), and for duct velocities of 2,000 fpm, 4,000 fpm and 8,000 fpm the losses would be 0.23 inch of water, 0.93 inch of water and 3.72 inches of water respectively.

Losses by air turbulence and air friction must be overcome by greater pressure differences, so that the losses may be determined by measurement of the static pressure difference between the air inside the duct and the air outside the duct. A simple water manometer with one side connected to the duct and the other open to the air will measure this difference. If such a manometer is connected just downstream from the entrance of the duct in the first example of the preceding paragraph when average duct velocity equals 4,000 fpm so that velocity pressure = 1 inch of water, the pressure difference is 1.5 inches of water (s.p. = $-1.5''$ H_2O). The difference between the $0.5''$ H_2O entry loss and the $1.5''$ H_2O static pressure is the pressure difference required to accelerate the air to the mean duct velocity and is equal to the velocity pressure. Similar hood static pressures at various velocities would be as follows:

Average duct velocity, V (fpm)	Velocity pressure, v.p.	Entry loss, h_e (inches of water)	Static pressure at hood, $s.p._h$ (inches of water)
1000	0.06	0.03	0.09
2000	0.25	0.13	0.38
4000	1.00	0.50	1.50
6000	2.24	1.12	3.36
8000	4.00	2.00	6.00

Just downstream from the entrance, therefore, [s.p.] = [h_e] + [v.p.]

A special case, as mentioned in the section on hood design (Section C-8-10) is the slot and plenum hood. Such hoods usually include a slot or an orifice discharging into a plenum from which the air is withdrawn by a straight or tapered take-off. The two hood entry losses are additive, but it is important to remember that the slot velocity and duct velocity are not usually the same. Where a slot or orifice enters a plenum, this portion of the hood entry loss is calculated from the slot velocity pressure. It is (1.78)(slot v.p.). The entry loss from the plenum to the duct is calculated as though it were directly from room air. A straight take-off, for example, is equivalent to a flanged entry, so that this entry loss is (0.5) (duct v.p.). The duct velocity pressure is added as before. In the usual type of double-entry hood, the slot velocity is equal to or less than the duct velocity. Although the air is accelerated to slot velocity, and pressure is required for this acceleration, the slot velocity is usually retained in the air flow through the plenum, and for convenience the acceleration pressure is added at the entrance from the hood to the duct. If slot velocity is higher than the duct velocity, the difference in velocity pressure will be lost, and this additional loss should be included in a summation of losses.

Straight Duct Friction Losses

Frictional losses in straight pipe are proportional to the length of pipe, and velocity squared, and are inversely proportional to pipe diameter. The losses also vary, of course, with pipe roughness. For other than the usual steel duct work, references such as Industrial Ventilation[1] or the ASHRAE Guide should be consulted. For the usual steel duct work, frictional losses may be obtained in terms of inches of water per 100 feet or fraction of velocity pressure per 100 feet from charts in Industrial Ventilation[1] (pages 6-24 and 6-25).

If, in the friction equation (see Section C-6-4, one substitutes Q/A for V and $\pi/4\ D^2$ for A, one obtains:

$$h = K \frac{LQ^2}{D^5} K$$

Where: h = the friction loss in inches of water
L = length of pipe
Q = air flow in cfm
D = duct diameter
K = constant

This indicates that for a given air flow and duct length, the frictional loss is inversely proportional to the 5th power of the diameter. A 10% decrease in diameter for the same flow and length will thus result in a 60% increase in duct friction.

Elbow Losses

Elbows create turbulence which results in pressure losses. The loss increases as the radius of the elbow is decreased, with highest loss for mitered elbows. The spin imparted to the airstream by an elbow causes an additional temporary loss which is recovered in a few duct diameters of straight duct downstream. If there are not several duct diameters (about 5) downstream from an elbow, the loss may be considerably higher than predicted from tables such as those in Industrial Ventilation[1] (page 6-20).

Entrance Losses

Where two ducts are joined there is a change in direction for at least one of the air streams, with a loss similar to that caused by an elbow. If the two air streams are of comparable size a loss will occur in both. Where they are sufficiently different in quantity of flow so that one may be considered a "branch" and the other a "main," the loss may be taken to be in the branch only. "Tee" connections create very high losses and should not be used. 45° or 30° entries are preferred. They can be installed conveniently at expansions so that a constant velocity is maintained.

Expansion Losses

Static and velocity pressure changes occur at expansions where no additional air is brought in and velocity decreases. The velocity pressure will decrease as the square of the velocity as shown in the formula:

$$v.p._2 = v.p._1\left(\frac{V_2}{V_1}\right)^2$$

Velocity pressure is converted into static pressure, but turbulence losses prevent a perfect conversion. For the commonly used enlargement ratio of 1-inch diameter increase for each 5 inches of length, the static regain will be about 7/10 of the velocity pressure difference, so that:

$$s.p._2 = s.p._1 + 0.7(v.p._1 - v.p._2)$$

For example:

A 6" duct is enlarged to an 8" duct, with a 10" long taper.

Q = 790 cfm, s.p. in 6" duct = -3" water

$$v.p._1 = \left(\frac{790}{0.1964 \times 4000}\right)^2 = 1" \text{ water}$$

$$v.p._2 = \left(\frac{790}{0.349 \times 4000}\right)^2 = 0.32" \text{ water}$$

$$s.p._2 = -3.0 + 0.7(1.0 - 0.32)$$

$$= -3.0 + 0.48 = -2.52" \text{ water}$$

Although there is little advantage to such an enlargement in a duct system, an enlargement at the system outlet (called an evase) can reduce the loss of velocity pressure, resulting in increased system efficiency.

Losses on Outlet Side of Fan

Losses on the outlet or pressure side of the fan are calculated in the same manner as those on the inlet side. Certain types of weather caps or outlet configurations result in losses at the outlet. An example of a simple system is shown in Figure 2.

Data: System to carry dust; minimum conveying velocity = 3500 fpm.
Q = 750 cfm.

To maintain a 3500 fpm minimum velocity,

$$\text{maximum area} = \frac{750}{3500} = 0.214 \text{ ft}^2$$

A 6" round duct has an area of 0.196 ft², a 7" duct is 0.267 ft², so a 6" duct must be used.

$$V = \frac{750}{0.196} = 3820 \text{ fpm;}$$

v.p. = 0.91 inch of water

From a table in Industrial Ventilation[1] (page 6-14) the entry loss for a 60° included angle, round tapered hood is 0.08 v.p. From the velocity pressure friction chart, friction loss for 3800 fpm in a 6" duct is 4.2 v.p. per 100 feet.

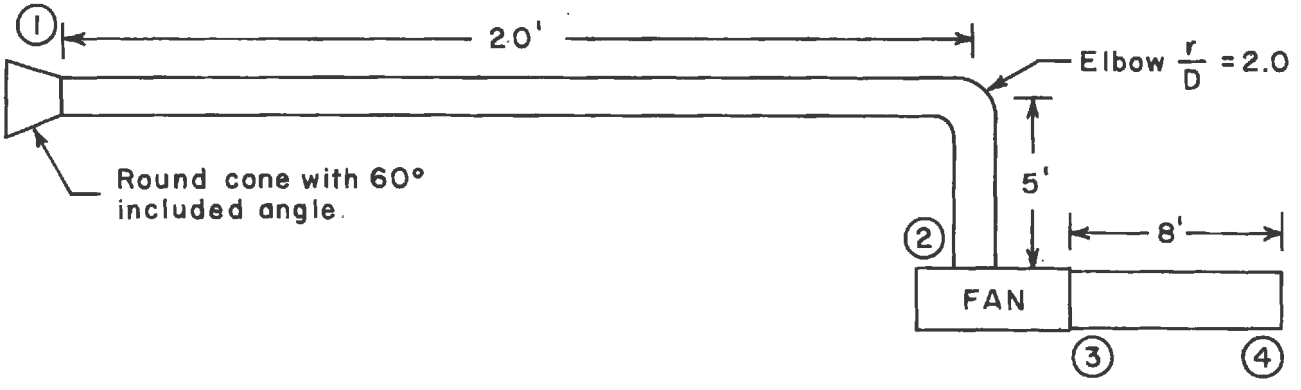


Figure 2. - Example of a simple exhaust system.

From the elbow table, elbow loss is 0.27 v.p. for a 90° elbow with R = 2D. Static pressures would then be:

At (1) $s.p. = -(h_e + v.p.)$

$= -(0.08 \text{ v.p.} + 1.0 \text{ v.p.})$

$= -1.08 \text{ v.p.} = -1.08(0.91)$

$= -0.98$ inch of water

At (2) Duct loss $= \frac{4.2 \text{ v.p.}}{100 \text{ ft.}} \times 25 \text{ ft.}$

$= 1.05$ v.p.

Elbow loss $= 0.27$ v.p.

Loss (1) to (2) $= (1.05 + 0.27)$v.p.

$= 1.32(0.91)$

$= 1.20$ inches of water

At (3) Duct loss (3) to (4) =

$\frac{4.2 \text{ v.p.}}{100 \text{ ft.}} \times 8 \text{ ft.} = 0.34$ inch of water

$\therefore s.p._{(3)} = 0.34$ inch of water

Fan static pressure =

$$TP_{(3)} - TP_{(2)} - VP_{outlet} =$$

$$(0.34 + 0.91) - (-2.18 + 0.91) - 0.91$$

$= 1.25 + 1.27 - 0.91$

$= 1.61$ inches of water

From the other formula given in Industrial Ventilation[1] (page 6-5)

Fan SP =

$[s.p._{inlet}] + [s.p._{outlet}] - v.p._{inlet}$

$= [2.18] + [0.34] - 0.91$

$= 2.52 - 0.91$

$= 1.61$ inches of water

The fan would thus be rated at 750 cfm, 1¾" fan s.p.

Duct Velocity

In the example just cited a conveying velocity of 3500 fpm was said to be required. Such velocities are required for systems carrying particulates to prevent plugging. Particulate conveying velocities, as determined by experience, are listed with the hood designs for specific operations in Industrial Ventilation[1] and range from 3000 to 4500 fpm. Where conveying is not a problem, velocities may be selected as desired. A design velocity of 2000 fpm is often selected as a reasonable compromise between low initial cost (small duct) and low operating cost (low pressure drop). Where space is a problem, or duct work is rather expensive, smaller diameter ducts and higher velocities may be used. Where a natural draft system is used, or a system is to use duct-axial fans, a low velocity (about 1000 fpm) will be selected.

Design of Multibranch Systems

For an exhaust ventilation system with more than one hood connected to it, the static pressure to be overcome by the fan must be that of the branch of greatest resistance. Where two branches join, or where a branch joins a main, only one static pressure exists, so that the upstream resistances of the two branches are equal. Thus, although design procedures appear to be equalizing resistances, what the procedures actually do are to assure the proper flow from each branch when the resistances are equal as they are in every operating system.

Two basic procedures are used for assuring proper flow with equal resistance. One, probably the more common, uses the branch of highest resistance to determine the static pressure required of the fan; the quantity of air is determined by adding the flow from all the branches. When the system is installed, a blast gate or damper is included in each branch, and each is adjusted to the proper flow rate. The second method uses no blast gates, but resistances and flows are adjusted in the design procedure by judicious selection of duct sizes, elbows and other sources of resistance. Each system has advantages and disadvantages which are listed in Industrial Ventilation[1]. The balance by static pressure design procedure will be used in this section.

The design of a multibranch exhaust ventilation system requires a methodical approach with intelligent use of design criteria. A number of formats may be set up to assist in the design, two such are given in Industrial Ventilation[1] (pages 6-1 through 6-13). In order to go through the design procedure, the quantities of air required to be exhausted from each operation must be determined, and the layout must be known, including the length of each pipe run and the number of elbows. Where the number of elbows and length of runs cannot be determined in advance, it may be necessary to use a balance-by-blast gate system.

Once this information is available it should be tabulated with type of hood, air flow, length of run and number of elbows for each branch. (See Section VI, Industrial Ventilation[1]). Starting with what appears to be the branch of greatest resistance, the static pressure is calculated to the junction. Static pressure in the next branch is then calculated to the junction. The two calculated static pressures are compared and if the ratio of the higher to the lower is less than 1.05 (the static pressures are within 5%), the agreement is considered perfect and the higher static pressure is carried forward in the calculation. If the ratio of the two static pressures is greater than 1.05 but less than 1.20, the higher calculated static pressure is used, but the flow expected in the lower resistance branch is increased by the formula:

$$Q_{1\,\text{corrected}} = Q_1\sqrt{\frac{\text{s.p.}_2}{\text{s.p.}_1}}$$

With the above criteria, this correction will result in no more than a 10% increase in flow in the branch of lower resistance. If the ratio of calculated static pressure at the junction is greater than 1.20, then the branch of lower resistance is redesigned. Recalling that the resistance of duct to a given air flow varies as the fifth power of duct diameter, it is evident that the use of the next smallest available pipe size will often be sufficient to bring balance within an acceptable range.

The above limits are design criteria for consistent, rational design. On occasion, it will become evident that some change other than increasing resistance in the branch of lower resistance would be more reasonable. If, for example, a small branch in an otherwise low resistance system turns out to have a high resistance so that it would be necessary for balance to add several inches of water to the static pressure of the entire air volume, obviously some other change should be made. A different type of hood with a larger duct, lower resistance and higher air flow might be substituted, or a unit collector used on this particular operation, or a separate system installed for it. As another example, where going to the next smaller size duct for balancing gives too high a resistance, it may be more feasible to increase the air flow even though it results in more than a 10% increase in branch flow. The point being emphasized is that criteria given are design guides and should be used with intelligence and judgment.

Summary of Design Procedure - Multibranch System - Velocity Pressure Method

The procedure for the design of each branch would then be:

1. Select hood, obtain entrance losses in terms of velocity pressure from Industrial Ventilation[1] (page 6-14).

2. Select air flow from Specific Operation print in Industrial Ventilation[1] (Section V) or other design guide.

3. Select pipe size for desired velocity (closest pipe size for gas and vapor systems); next

smaller size for particulate systems with minimum conveying velocity. Use ½-inch increments in diameter to 5½ inches, 1-inch increments in diameter to 22 inches, and even inches for diameters greater than 22 inches.

4. Calculate actual velocity for air flow with given pipe size (steps 3 and 4 can be done with sufficient accuracy from the duct friction charts in Industrial Ventilation[1], (pages 6-24 and 6-25).

5. Enter velocity pressure corresponding to 4.

$$v.p. = \left(\frac{V}{4000}\right)^2$$

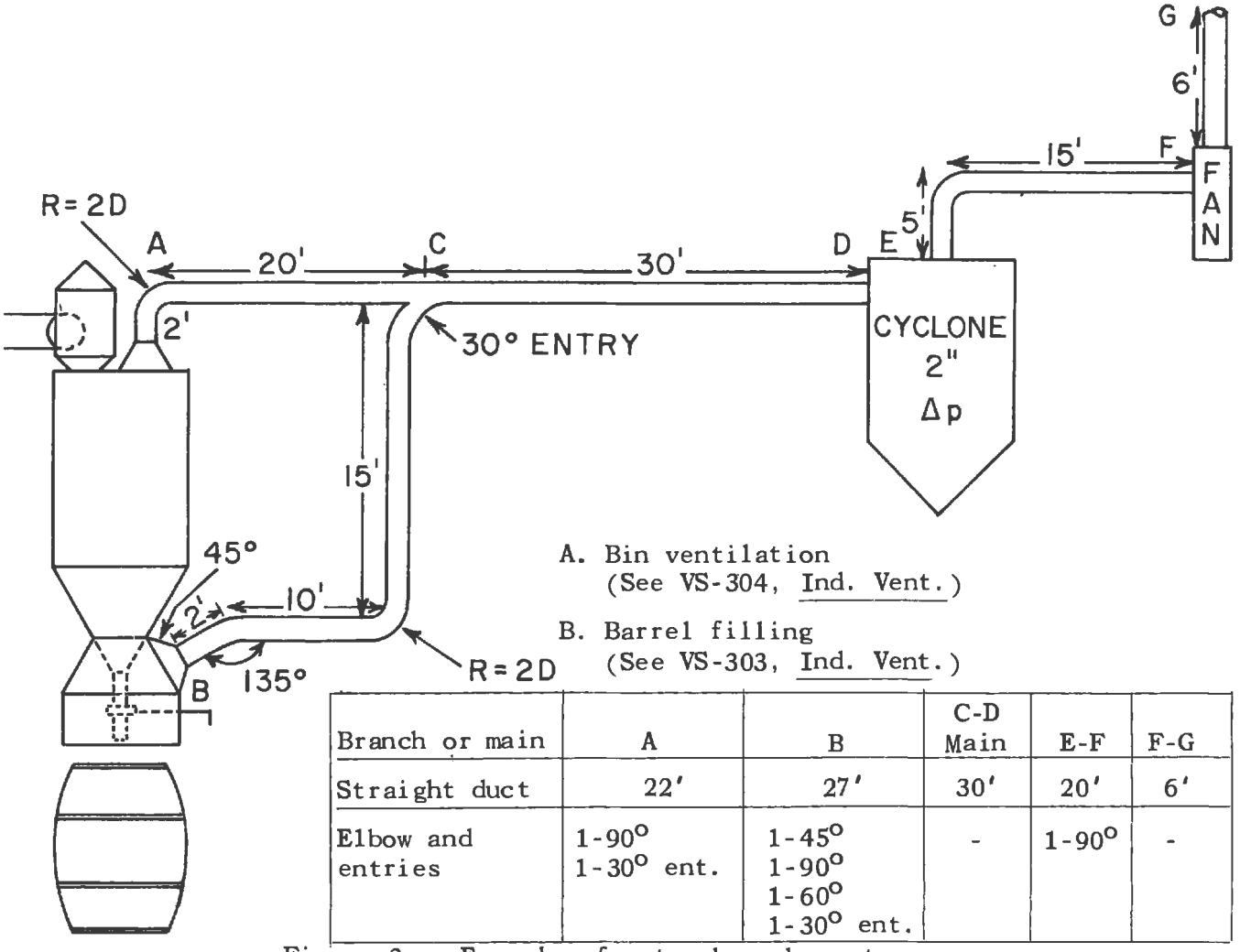
or may be obtained from table in Industrial Ventilation[1] (page 6-26).

6. Calculate slot loss in inches of water (if double entry hood).

7. Enter entrance loss into duct in terms of fraction of duct velocity pressure.

8. Add static pressure equal to duct velocity pressure (for acceleration).

9. Determine (from chart) friction loss (in terms of v.p. per 100 feet) of straight duct.



A. Bin ventilation (See VS-304, Ind. Vent.)

B. Barrel filling (See VS-303, Ind. Vent.)

Branch or main	A	B	C-D Main	E-F	F-G
Straight duct	22'	27'	30'	20'	6'
Elbow and entries	1-90° 1-30° ent.	1-45° 1-90° 1-60° 1-30° ent.	-	1-90°	-

Figure 3. - Example of a two-branch system.

10. Enter friction loss in terms of v.p. for actual length of straight duct.

11. Determine fraction of v.p. lost (from chart in Industrial Ventilation[1] (page 6-20) for each elbow.)

12. Enter total fraction of v.p. lost for all elbows in branch.

13. Determine fraction of v.p. lost in entry.

14. Add fractions of v.p. in 7, 8, 10, 12, and 13 and multiply by v.p. to obtain static pressure in inches of water.

15. Add inches of water slot loss from 6.

16. Compare static pressure at junction with static pressure calculated in same manner from other branch or main to the same point. Correct air flow or redesign as necessary.

An example of a two branch system is shown in Figure 3.

1. Branch or Main No.	A-C	B-C	A-C re-design	B-C re-design	C-D	Dust Collector	E-F	F-G
2. Air volume (cfm)	1000	400	1000	480	1580		1580	1580
10. Branch duct diam. (inches)	7	4.5	6	5	9		9	9
11. Branch duct area	0.267	0.1105	0.1964	0.1364	0.442		0.442	0.442
12. Branch duct vel.	3750	3620	5090	3500	3570		3570	3570
13. Branch duct v.p.	0.88	0.82	1.62	0.76	0.79		0.79	0.79
14. Straight duct length	22	27	22	27	30		20	6
15. Straight duct friction (v.p./100')	3.5	6.2	4.1	5.3	2.6		2.6	2.6
16. Straight duct loss factor	0.77	1.67	0.90	1.43	0.78		0.52	0.16
17. Hood entry loss factor	0.25	0.25	0.25	0.25	--		--	--
18. Acceleration factor	1.00	1.00	1.00	1.00	--		--	--
19. Elbow loss factor	0.27	0.59	0.27	0.59	--		0.27	--
20. Entry loss factor	0.18	0.18	0.18	0.18	--		--	--
22. Duct loss factor	2.47	3.69	2.60	3.45	0.78		0.79	0.16
23. Duct s.p. loss	2.17	3.03	4.21	2.62	0.62		0.62	0.13
24. Other losses	--	--	--	--	2.62	2.00	5.24	5.86
25. Branch or main s.p. loss	2.17	3.03	4.21	2.62	3.24		5.86	5.99
26. Governing s.p. at junction	2.62	3.03	4.21	2.62				
27. Corrected air volume	1100	Not within 20% of A-C	Not within 20% of B-C	Correct flow in A-C $Q = 1000\sqrt{\frac{2.62}{2.17}} = 1100$				

Fan static pressure = 5.86 + 0.13 - 0.79 = 5.20 inches of water

The fan rating should thus be 1580 cfm at 5.2 inches of water

In the example on page C-10-9, the calculated static pressure did not agree within 20% at the first junction. If the next smaller pipe size was used for the bin exhaust (Branch AC), a velocity of over 5,000 feet per minute resulted and the calculated static pressures again did not agree within 20%. If a 5-inch duct was used for the barrel filling exhaust with a 3,500 fpm velocity so that the flow was increased to 480 cfm, calculated static pressures at the junction differed by only 20%, so that the bin exhaust flow is increased by 10%. At this point in the design, some knowledge of the process would be desirable. If, for example, the product were a fine powder, the excess exhaust from barrel filling might result in excess pick-up of product and it might be necessary to increase the open area of the hood to reduce face velocities while still providing a quantity of air sufficient to control the process.

Systems which have a slot entry for distribution of flow and use lower velocities are usually easier to balance. The same flows and duct lengths as in the previous example, but with slot hoods for vapor exhaust, will be used in the following example:

Operation	Vapor exhaust A	Vapor exhaust B	Main C-D	Main D-E
Entry loss (slot + 90° included angle taper take-off)	1.78 slot v.p. + 0.25 duct v.p.	1.78 slot v.p. + 0.25 duct v.p.	--	--
Q	400	1000	--	--
Straight duct	27′	22′	30′	30′
Elbows	2-90°, 1-60°	1-90°	--	1-90°
Entries	1-30°	1-30°	--	--

Slot velocity is 2,000 fpm; all elbows have R = 2.0 D.
Entry is 30°; use design velocity of 2,000 fpm.

As in the example on page C-10-9, a redesign was required at the junction. At the lower velocities, however, and with the slot resistance tending to reduce the percentage difference, a reduction in pipe size for B-C brought the two branches into good agreement with only 0.4 inch additional static pressure.

In the two examples on pages C-10-9 and C-10-11, the velocity pressure method of calculation has been used. This is one of several systems which may be used to tabulate the calculations. Another method given also in Industrial Ventilation[1] (see section VI) uses elbows and entries in terms of lengths of straight pipe with equivalent pressure drop. Hood entry and acceleration pressures are tabulated in terms of inches of water. These two methods of calculation are based on the same data and will give equivalent results.

Another method of calculation often used is to ignore velocity pressure and calculate only in terms of static losses. Such a method has several disadvantages. For example, where there are pronounced velocity pressure changes, as in many dust systems, losses may be underestimated. Also, it does not give static pressure which is readily checked by a manometer. For this reason, it is recommended that v.p. be included even when designing a system to be balanced by a blast gate.

1.			Redesign		
1. Branch or Main No.	A-C	B-C	B-C	C-D	D-E
2. Air volume (cfm)	400	1000		1460	1460
3. Slot area (sq. ft.) (Note 1)	0.2	0.5	Same		
4. Slot velocity (fpm)	2000	2000			
5. Slot v.p. (inches of water)	0.25	0.25	as		
6. Slot entry loss factor (fraction of v.p.)	1.78	1.78	before		
7-8. (Not used when slot velocity is $\leq$ duct velocity.)					
9. Slot s.p. loss	0.45	0.45			
10. Branch duct diam.	6"	10"	9"	12"	12"
11. Branch duct area	0.1964	0.545	0.442	0.785	0.785
12. Branch duct vel.	2040	1830	2260	1860	1860
13. Branch duct v.p.	0.26	0.21	0.32	0.22	0.22
14. Straight duct length	27	22	22	30	30
15. Straight duct v.p./100 ft.	4.4	2.3	2.7	1.9	1.9
16. Straight duct loss factor (14 x 15)	1.19	0.51	0.59	0.57	0.57
17. Hood entry loss factor	0.25	0.25	0.25	--	--
18. Acceleration factor	1.00	1.00	1.00	--	--
19. Elbow loss factor (no. x factor)	0.73	0.27	0.27	--	0.27
20. Entry loss factor	0.18	0.18	0.18	--	--
22. Duct loss factor	3.35	2.21	2.29	0.57	0.84
23. Duct s.p. loss	0.87	0.46	0.73	0.13	0.18
24. Other losses	0.45	0.45	0.45	1.32	1.45
25. Branch or main s.p. loss	1.32	0.91	1.18	1.45	1.63
26. Governing s.p. at junction		1.32 Note 2	1.32 Note 3		
27. Corrected air volume			1060		

Fan static pressure = 1.63 - 0.22 = 1.41 inches water

Notes:

1. Slot area determined by Q and slot velocity .
2. $\frac{1.32}{0.91} = 1.45$. Redesign branch B-C for balance.
3. $Q_{corrected} = 1000\sqrt{\frac{1.32}{1.18}} = 1060$ cfm

Corrections for Density

Where corrections for density are required because of high or low temperatures, high altitudes or pressurized operations, corrections are made as follows:

1. The amount of air entering a hood is kept the same in terms of weight. For a lower density air, this means that the cubic feet of air called for in the hood design is increased by dividing by the ratio of the lower density to the density of air at standard conditions. A "Density Correction Factor" table, giving the ratio of densities to "standard" density for a wide range of temperatures and elevations is presented in the "Duct Design Procedure" section of Industrial Ventilation[1] (page 6-23).

2. Static pressure losses and required velocities and pipe sizes are calculated for the corrected volumes of air as though it were air at standard conditions. The fan size and speed are selected as though these calculated static pressure losses existed. For motor selection, however, the horsepower is decreased by multiplying by the density factor.

3. Static pressures actually observed in the system will be decreased directly as the density factor. Hood static suction, for example, will be:

$$s.p._h = dx\left[\frac{V}{C_e(4000)}\right]^2$$

Where:

d = density factor,
V = velocity, and
C_e = coefficient of entry as described in the previous sections.

For example, the system on page C-10-2 is to be designed for an elevation of 6000 feet and a temperature of 70°F.

The density factor from Industrial Ventilation[1], (page 6-23) is 0.80. The exhaust volume required will thus be:

$$\frac{750}{0.80} = 935 \text{ cfm}$$

Pipe diameter for 3500 fpm will be 7".

$$V = \frac{935}{.267} = 3500$$

$$v.p._{(\text{std. conditions})} = 0.77$$

Entry loss, as before = 0.08 v.p.
Acceleration factor = 1.00 v.p.
Duct length = 33 feet

From v.p. friction chart,
Factor = 3.5 v.p./100 ft.

$$\text{Duct friction loss} = \frac{3.5}{100} \times 33$$

$$= 1.06 \text{ v.p.}$$

$$\text{Elbow loss} = 0.27 \text{ v.p.}$$

$$\text{Total loss} = 2.41 \text{ v.p.} \times 0.77$$

$$= 1.86" \text{ water}$$

$$\text{Fan static pressure} = 1.86 - 0.77$$

$$= 1.09" \text{ water}$$

The fan is then selected for 935 cfm, 1.1" fan static pressure. The brake horsepower listed in the fan catalog for this value, however, is multiplied by the density factor, 0.80, to obtain actual horsepower for motor selection.

If static pressures are to be measured to check performance, the static pressure 1 to 3 diameters downstream from the hood would be: $(v.p. + h_e)(0.80) = 1.08(0.77)(0.80) = 0.66"$ water

This compares with a hood static pressure of 0.97" water for the original system with a volume of 750 cfm in a 6" diameter pipe.

Reference

1. _____: Industrial Ventilation -- A Manual of Recommended Practice. Am. Conf. of Gov. Ind. Hyg., 8th Ed., 1964.

METHODS OF AIR CLEANING

Introduction

Air cleaning is the operation of removing contaminants from an air or gas stream. This job of removal can be simple and straight-forward, or complicated and difficult, depending on the number, nature and concentration of the contaminants that must be removed, and the degree of removal required. Some air cleaning mechanisms are effective on particular kinds of contaminants and less effective, or ineffective, on others. The selection of air cleaning equipment to remove a given contaminant, or group of contaminants, must be based on an evaluation of the components of the air stream to be cleaned, and on the efficiency of the various kinds of air cleaning equipment for these components (see Table 1).

Air cleaning equipment is usually installed for one or more of the following reasons:

(1) For compliance with an air pollution control ordinance

(2) For reclaiming or classifying product material

(3) To prevent a nuisance or damage to property

(4) To prevent re-entry of contaminants into workroom air.

Definitions

A definition of terms at this point will help clarify the discussion that follows:

Gas is a substance which is in the gaseous phase at ordinary temperatures and pressures. Gases diffuse.

Vapor is the gaseous form of a substance which is normally in the solid or liquid state at ordinary temperatures and pressures. Vapors diffuse.

Aerosol is a general term referring to a suspension of a foreign material in air. This material may be in the form of dust, fume, smoke, mist, or fog.

Dust is formed in processes of disintegration, e.g., grinding, drilling, blasting, and crushing of organic and inorganic materials. The size range of a dust usually ranges from the submicron sizes to particles visible to the naked eye. No chemical change takes place in the production of dust. Dusts do not tend to flocculate except under electrostatic forces; they do not diffuse in air but settle under the influence of gravity.

Fume results from condensation from the gaseous state, generally after volatilization from molten metals, and often accompanied by chemical reaction such as oxidation. The particle size is usually more uniform and much smaller than that of a dust. Fumes flocculate and sometimes coalesce.

Smoke is the result of combustion of organic matter. Particle size is generally below 1 micron and relatively uniform.

Mist or fog is a liquid particulate. It may be generated by atomization of a liquid or condensation of vapor on airborne nuclei.

Review of Stokes' and Cunningham's Law

The relative effectiveness of all particulate air cleaning equipment becomes clear when the principle of collection and the application of Stokes' Law is understood. Sedimentation, inertial, precipitation and filtration devices all depend wholly or in part on Stokes'-Cunningham Law. This law was derived on the basis that, as spherical particles settle through a medium, they encounter

Prepared by: Paul F. Woolrich

Table 1. - Particle Size Range of Various Aerosols

Type of Aerosol	Size Range of Particles (microns)
Raindrops	7,000 - 500
Natural mist	500 - 40
Natural fog	40 - 1
Dusts	
Cement	150 - 10
Fly ash	80 - 0.1
Silica dust (in silicosis)	10 - 0.5
Pollens	60 - 20
Plant spores	30 - 10
Bacteria	15 - 1
Fumes and Mists	
Metallurgical fumes	100 - 0.1
H_2SO_4 concentration mist	10 - 1
Alkali fume	2 - 0.1
SO_3 mist	3 - 0.5
NH_4Cl fume	2 - 0.1
Zinc oxide fume	0.3 - 0.03
Electric furnace	72% less than 6
Gray-iron cupola	71% less than 44 18% less than 6
Smokes	
Oil smoke	1.0 - 0.03
Rosin smoke	1.0 - 0.01
Carbon smoke	0.2 - 0.01

a molecular resistance. Equilibrium is quickly reached between the resistance and the force-producing motion (gravity), and the particles assume a constant velocity known as "Terminal Velocity, V."

This resistance is expressed:

$$Fd = \frac{C\ d_g\ V_2^2\ \pi\ D_p^{\ 2}}{8} =$$

Where: C = drag coefficient (dimensionless)
d = density of gas
D_p = diameter of the particle
V = terminal velocity

The drag coefficient, C, is a function of the Reynold's Number, and is equal to

$$\frac{24}{\text{Reynold's No.}} \text{ or } \frac{24N}{D_p\ V\ d\ G}$$

Where: N = the viscosity of the gas.

By substitution, the settling or resistance force, F_d is given by Stokes' Law:

$$F_d = 3\ \pi\ N\ D_p\ V$$

By equating the force of gravity, F_g, to drag resistance, F_d:

F_g = particle mass x acceleration of gravity =

$$\frac{4}{3}\pi\frac{D^3}{8}\,d\,G$$

Stokes' Law equation for gravitational settling becomes:

$$V = \frac{D_p^2\,d\,G}{18N}$$

Where: d = density of particle

For submicron particles, there is a tendency for particles to "slip" between the molecules, therefore, Cunningham proposed a correction for Stokes' Law, "A":

$$A = 1 + \frac{KL}{D_p}$$

Where: K = 1.72 for air
L = 0.1 micron for air (mean free path)

Stokes'-Cunningham's Law, in terms of settling force becomes:

$$F_d = \frac{3\pi N D_p V}{A}$$

Since F_d is directly related to collection efficiency, collection efficiency decreases as particle size decreases (Cunningham's Correction increases).

Air Cleaning Equipment for Particulates

The choice of a particular type of air cleaner or collecting equipment depends upon several factors. Among these are: (1) the amount of material to be collected or gas loading, (2) nature of the material to be collected, and (3) the efficiency of collection required. It must be borne in mind that particulates less than 10 microns in size are of hygienic significance and are more difficult to collect than particles larger than 10 microns, which create a nuisance problem only.

Types of Equipment

In the discussion to follow various types are briefly described including their uses, advantages and disadvantages.

Settling chambers. Sometimes referred to as chip traps, these are enclosed spaces where the force of gravity is exploited to achieve separation.

Retention is a function of the dimensions of the chamber and the flow rate:

$$\text{Retention} = \frac{(h.b.L.)}{Q} = \frac{h}{V_g}\frac{V_g}{V_a} = \frac{h}{L}$$

$$V_a = \frac{Q}{hb}$$

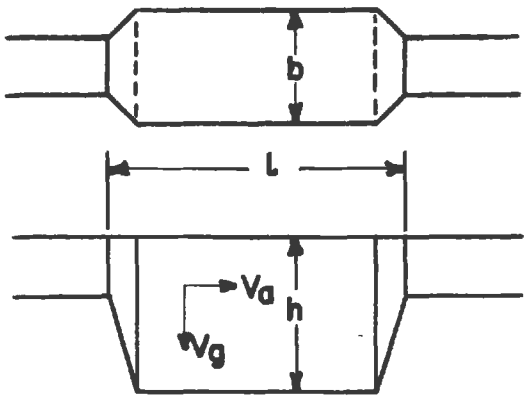


Figure 1. - Settling Chamber

Such a device is efficient only on particles larger than about 50 microns. If a series of horizontal trays is used instead of one large chamber, the device becomes a Howard Dust Chamber. This may remove particles as small as 10 microns. If a baffle is placed in the entrance of a settling chamber, it makes the chamber an inertial collector.

Inertial Separators

When the path of an aerosol is changed, the inertia tends to "throw it from the airstream." The radial velocity of a particle is obtained by equating the centrifugal force to the Stokes' Resistance:

$$V = \frac{D_p^2 \, d \, V'^2}{18 \, N \, R_c}$$

Where: d = density of particle
V' = tangential velocity of gas stream
R_c = radius of curvature of gas stream

By equating centrifugal force to Stokes' Resistance, it can be seen that reducing the radius of curvature of the gas stream increases its tangential velocity which increases the removal capabilities as the square of this tangential velocity.

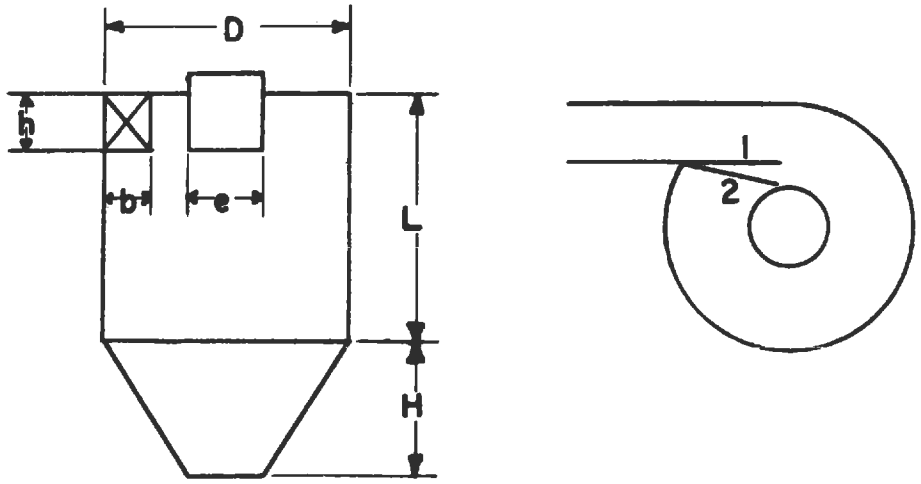


Figure 2. - Schematic Drawing of a Cyclone Collector. Top View Shown at Right.

The gas stream enters the separator tangentially and spirals along the walls and exits at the top (see Figure 2). The particles strike the wall and fall through the core into a collecting hopper. Cyclones handle gas volumes ranging from 30 to 25,000 cfm. Dust loadings should be over 10 grains per cubic foot for efficient operation.

Pressure drop through a cyclone is usually kept below 4" H_2O gauge. It can be computed from the following equations (see Figure 2):

$$F = K \frac{bh}{e^2}$$

$$K = \frac{\frac{12}{k}}{\left(\frac{L}{D}\right)^{1/3} \left(\frac{H}{D}\right)^{1/3}}$$

Where: F = inlet velocity head
k = ½ with no inlet vane
k = 1 with inlet vane (1)
k = 2 with inlet vane (2)

The ordinary cyclone efficiency begins to drop off appreciably with particulate less than 40μ. It operates on a principle of differential pressures which removes the impinged particulate to a collecting hopper. By utilizing a large number of small diameter inertial units in lieu of one large one, effective size range of collection can approach 10μ.

Wet inertial collectors. Particulates impinge on wetted surfaces to facilitate removal. In this type, the spray admixes with the particulate material, agglomerates it and causes it to coalesce to aid in its removal. Advantages of this type of collector are:

(1) There is no secondary dust disposal problem.

(2) It has the ability to clean high temperature gases.

(3) Moisture laden gases cause no problem.

(4) The efficiency curve drop-off indicates that about 5μ minimum size particles are collected.

(5) This collector can be used with sticky or linty materials.

Venturi and Cyclonic Scrubber. Contaminated gas enters the collector and is accelerated at high velocity where it impinges upon the liquid stream introduced at the throat of the Venturi. This results in the atomization of the liquid into a tremendous number of fine droplets. The high differential velocity between the gas and atomized droplets results in the impaction of the particles with the fine droplets. As the gas decelerates, further impaction and agglomeration of the droplets takes place. After the particles have been trapped within the droplets, the resulting agglomerates are readily removed from the gas stream in the Cyclonic Separator. The scrubbed gas enters the Cyclonic Separator tangentially, spinning the liquid droplets against the wall and leaves at the upper part of the unit. The particle-laden liquid drains by gravity from the bottom and may be recycled, sewered, or reclaimed.

Examples and Applications of Inertial Collectors

The following examples illustrate eight different operations for which air cleaning equipment was required and the type of inertial collector that was used:

(1) Operation: Metal Cutting
Collection Device: Cyclone (Single)

A rough metal cutting operation, which gave rise to the emission of relatively large quantities of metal dust of large particle size, made it necessary that the company install some type of air cleaning equipment. It was recommended that they consider the installation of a medium efficiency cyclone of simple design because of the particle size distribution. This application of a simple collector to a simple job was successful.

(2) Operation: Asphalt Plant
Collection Device: Multiple Cyclones

A large asphalt plant purchased a multicyclone type collector (a bank of small diameter cyclones) and applied it to its rotary dryer and fugitive dust operation, (screen, elevator, mixing platform, and transfer points). The effluent plume was relatively dark and performance testing of this equipment indicated low collection efficiencies. Maintenance was difficult and within a month after the installation was completed it was found necessary to discontinue use of the equipment because of its susceptibility to plugging, particularly on cooler days.

(3) Operation: Buffing and Polishing (Automatic)
Collection Device: Wet Collector (Inertial)

Production buffing and polishing operations are characterized by easily collectible contaminants containing fine to coarse dust and sticky, linty materials. Wet collectors are successfully applied to such operations and there have been a few cases of success with recirculation of air. Three large capacity wet inertial type collectors were used for production buffing operations which were equipped with adequate hoods and duct systems. The system did not work as expected. The basic problem was in design of the air cleaning equipment which enabled the linty buffing contaminants to build up as a floating mat in the wet collector. Passage of

the inlet air stream through the mat was not accomplished with the result that dirty air flowing into the collector bounced off the floating mat and passed directly through the collector to the outdoors, creating a severe neighborhood air pollution problem.

Wet centrifugal or inertial collection equipment is apparently satisfactory for the contaminants given off by production buffing and polishing operations. This is true providing there is continuous agitation of the collecting media, making certain that there is water-air stream intermixing, and that the build up of a floating blanket of linty contaminant does not prevent agitation and intermixing.

(4) Operation: Aluminum Anodizing
Collection Device: Wet Collector, Centrifugal

A typical aluminum anodizing process includes the use of alkaline solutions for metal cleaning and etching purposes, sulfuric acid for the electrolytic anodizing operation, and a mixture of phosphoric and nitric acid for bright dipping purposes. The effluents from all of the tanks are corrosive but the bright dip tank in particular is very bad because of the continuous emission of nitric acid and oxides of nitrogen, which cause serious neighborhood pollution problems. A wet centrifugal scrubbing unit consisting of a cylindrical fiberglass tank with sprays introduced at a tangential-type inlet was installed and an attempt was made to use, on an uncontrolled solution basis, alkaline wash water as scrubbing media.

(5) Operation: Iron Ore Improvement
Collection Device: Wet Collector, Centrifugal

A wet collector was installed as the secondary collection device at an iron ore drying operation. The rotary dryer was used essentially to remove moisture from the ore and thereby upgrade the iron content. The cyclone collector originally installed was not successful in application because of the unfavorably particle size range. A collector study indicated a cyclone outlet loading of eight to twelve grains per standard cubic foot per minute (scfm) with a geometric mean particle size of 1.5 microns. Maximum emission was 50 tons per 24 hours and complaints of excessive pollution were received from residents in a city eight miles from the plant. Additional air cleaning equipment was recommended and the company installed a multi-stage collector equipped with a self-contained fan unit and a mist eliminator. The installation proved to be satisfactory and the wet slurry from the collector is piped to the company's nearby iron ore beneficiation plant.

(6) Operation: Foundry Sand Handling Operations
Collection Device: Wet Collector, Packed Tray Type

Various types of tray packing materials including Berl saddles, marbles, and different plastic shapes have been used for creating a satisfactory air stream to liquid contact. Experience with several of these collectors in one large foundry indicated satisfactory performance where the iron content in the inlet air stream is negligible. But it causes a severe maintenance problem and unsatisfactory

performance where iron is present in the inlet in moderate concentrations. A cementing action takes place resulting in solidified trays of the contact media.

(7) Operation: Foundry Cupola
Collection Device: Venturi Scrubber

A large foundry installed a high pressure drop type venturi scrubber on a cupola. This initial installation was to serve as a guide toward control measures to be applied to other cupolas on the premises. The performance testing technique used by the purchaser of the equipment varied somewhat from that used by the supplier. In any event, there were differences in results and there was some question concerning satisfactory air cleaning performance. Aside from disappointing results, this application of a high pressure drop type air cleaning device points out the need for some agreement on the manner in which performance testing is to be done, since there are different approaches to the problem. Further, it confirms the thought that the successful application of air cleaning equipment to cupolas is in no way simple and requires the expenditure of relatively large sums of money. In some locations of the country, depending upon the requirements or air pollution control ordinances, fabric filters have been used successfully, but not without the installation of well-designed controls.

(8) Operation: Open Hearth Furnace
Collection Device: Venturi Scrubber

The venturi scrubber has been applied to a 200-ton capacity open hearth furnace with apparent success even during oxygen lancing phases. It is interesting to note that in this 40,000 scfm system, fan operating costs increased with pressure drop ranging from $35,000 a year at a 26" pressure drop to $75,000 at a 38" pressure drop (based on a power cost of $1.00 per 100 horsepower hours). Particle size analysis of the collected material indicated 46% below 5 microns for a composite sample and it was also found that increasing the scrubbing water rate above 500 gallons per minute had no effect on cleaning performance. The effluent concentration ranged from 0.01 to 0.1 grains per scfm which is good for this type operation.

Fabric Filters

Filters take advantage of inertial forces, diffusion, electrical charging, and straining. The effect of inertial forces and diffusion can be predicted with some accuracy from theoretical considerations. Electrical forces on the particles and/or the filter surface play an important part in filtration, but their effect in a given case is difficult to predict. Straining plays an insignificant role for dusts of hygienic significance.

The two most common types of commercial cloth filters are the "bag" type and the "screen" or "envelope" type. The difference between the two types is in the manner of support for the filter. The sleeves or bags are usually contained in an enclosure known for bag filters as a "bag house." The bag house has an inlet for the dusty air, a manifold to distribute the air to the bags, an outlet, and a hopper to catch the collected dust. Most also contain some kind of shaking device to dislodge the collected dust when the critical resistance is reached.

One type of bag filter, the "Reverse Jet" type, performs at constant resistance. A traversing ring moves continuously up and down the bag blowing off the dust with a high velocity air jet.

The efficiency of a cloth filter increases with dust load since most of the filtering is done by the accumulated dust.

High efficiency filters must be used for air cleaning tasks where efficiencies by count greater than 99.5% are required. One type is a cellulose-asbestos paper. The asbestos does most of the filtering and the cellulose gives the necessary support. Crushed flint, sand, and coke in deep beds make high efficiency filters. Glass and resin wool fibers are used also for this purpose. High efficiency filters are expensive and take up large amounts of space.

While high collecting efficiencies (about 99.9%) can be obtained with the use of certain types of filter media, it should be stressed here that air bearing toxic materials, such as lead, beryllium, or radioactive materials, should not be recirculated. Most states prohibit this practice because of possible failure in the collecting equipment.

Major problems encountered in the use of fabric filters may be due to high temperature, moisture, acidity or alkalinity of the gas stream. Many of these difficulties can be overcome by selecting the proper filter media or a combination of types.

Examples and Applications of Fabric Filters

(1) Operation: Brass Melting
Collection Device: Fabric Filter

A large brass foundry found it desirable to install air cleaning equipment on two electric furnace melting operations in which large quantities of oil-coated brass scrap were melted. A fabric collector with cotton bags was installed and placed in use following a thorough investigation of the air cleaning problem by company personnel. A water-cooled heat exchanger was used to cool the inlet gases but no provision was made for an automatic temperature controlled shutdown or for the introduction of dilution air. Fire destroyed the collector on two occasions. Despite the detailed initial studies preceding the selection of air cleaning equipment for this job, there appears to be several factors which were not studied thoroughly enough, including the temperature cycle and vulnerability of the fabric to the inherent ignition sources such as glowing carbon, hot metal particulates, and pyrogenic oxides given off. Also, instrumentation and dilution control of inlet air stream temperature were not provided.

(2) Operation: Foundry Sand Handling System
Collection Device: Fabric Filter

A large production iron foundry purchased new fabric type collection equipment, installed it outdoors, and applied it to various sand handling operations, excluding the shakeout operation. A screw conveyor system was included in the installation for continuously unloading the hoppers and dumping the collected material into a chute from where it dropped in a dusty manner into ground level bins, which were then placed on trucks and hauled away. Two problems developed, not with the basic air cleaner, but with the accessory screw conveyor. The first unfortunate incident was due to a broken drive shaft which was apparently under-designed for the job. In the second instance,

cold weather resulted in stiffening and setting the material being conveyed with the result that there was a complete screw conveyor breakdown.

What should be noted in this case is that a very important part of the equipment sold to the user failed because of poor engineering design and misapplication (moisture in sand does freeze) and a secondary dust problem developed as a result of dropping the collected contaminant into the bin located at ground level.

(3) Operation: Foundry Shakeout
Collection Device: Fabric Filter

A foundry decided on installation of the fabric type collector for application to its production shakeout operations. Because of the high moisture burden in the shakeout exhaust products and the susceptibility of fabrics to plugging because of condensation, fabric collectors are not usually applied to this type operation. When they are, temperature controls are usually necessary. Selection of equipment was based upon the desirability of recirculating air from the collection equipment back into the plant as a heat-saving measure and to help overcome negative pressure conditions.

(4) Operation: Uranium Refining
Collection Device: Fabric Filter, Reverse Jet

A number of reverse jet fabric collectors using wool felt as the collection media have been used successfully in uranium refining facilities. Such collectors enable a high air to cloth ratio, up to 15 cfm per square foot of cloth. Each collector is provided usually with a pressure control which activates cleaning only when needed and provides a record of operation. Also, dust detectors, high limit pressure switches, process interlocks, and temperature alarms are used as needed.

(5) Operation: Charcoal Briquet Manufacture
Collection Device: Fabric Collector

Fabric collectors using cotton type cloth media are necessary for recovery of about 20% of the product in a charcoal briquet manufacturing plant. The charcoal dust is collected in the air cleaning equipment, dumped, and pressed into briquet form. This is one type of installation in which air cleaning equipment of an efficient type is required in order to make a process economically successful. In this case the air cleaning equipment is essentially a basic part of the process. The same holds true in the carbon black and diatomaceous earth industries where fabric type collectors are used as part of the process.

(6) Operation: Drying and Cooling of Superphosphate Chemical Fertilizer
Collection Device: Wet Collector (Rotating Impeller): Fabric Filter

In recent years, most superphosphate chemical fertilizer plants have converted to the manufacture of granulated or pellatized phosphate fertilizer in addition to or in place of the dust. Several chemical processes have been developed, all of which cause the emission of relatively large quantities of ammonium chloride in the submicron size range. In this case, application of wet "dynamic" type

collection equipment was not successful. Perhaps it represented a successful application of equipment in terms of product recovery since the slurry could be pumped back into the process. However, the neighborhood air pollution problem was severe. This is a characteristic problem of many superphosphate fertilizer plants throughout the country. The ammonium chloride plume given off during the cooling of granulated superphosphate fertilizer has been discussed above where a wet "dynamic" collector could not successfully handle the effluent. Fabric-type air cleaning equipment has been worked successfully in terms of air cleaning effectiveness. However, it has been subject to frequent plugging. There are several reasons for this problem. Among these are that no primary cleaning device was installed to peel off a large part of the very high inlet loading, and temperature control was not applied to minimize the possibility of condensation and plugging of the fabric cleaning media.

Electric Precipitation

When a high voltage is placed across two electrodes in air, an ionizing current will begin to flow between the electrodes. The stresses established against the molecules of air surrounding the ionizing electrode literally strip negative ions or electrons from the air molecule, and this ion migrates to the collecting (positive or grounded) electrode. Too great a voltage across the electrodes produces complete ionization or "arcing," but somewhere between the two extremes, a condition known as "corona discharge" exists.

The precipitating force in electrostatic precipitation is given by the equation:

$$F_p = KQE$$

Where: K = constant
Q = developed electrical charge
E = field strength

Equating this to Stokes' Resistance,

$$V = \frac{EQ}{3 \pi N D_p}$$

NOTE: The smaller the particle, the higher the efficiency.

Industrial uses include the collection of fly ash, metallurgical fume, acid mists, cement dust, and other materials. Two types are in common use - the Cottrell and two-stage types.

Cottrell precipitators find use in metallurgical and chemical industries to collect oxide fumes and acid mists. Utilities use them to collect fly ash. Two-stage precipitators are used as air conditioning precipitators to remove oil and tobacco smoke, and in collecting dusts and smokes from foundries; they have separate ionizing and collecting fields, and are used for light dust loadings. Both types run on rectified a.c.: Cottrell at 25,000 volts and over, and the two-stage with 12,000 volts ionizing stage and 6,000 volts collecting. Cottrells have visible coronas and present a shock hazard.

Electrostatic precipitation is the most sophisticated, expensive, and yet the most efficient air cleaning device known. Its high efficiency is maintained regardless of the size of particles in the gas stream. Furthermore, it can handle gases which are hot (up to about 1100°F), cold, wet, or dry.

Some of the disadvantages of this method are: High initial cost; it cannot be used for combustibles or in explosive atmospheres; a precleaner is often required to prevent overloading the precipitator; and it is not economical for low air flows.

Examples and Applications of Electrostatic Precipitators

(1) Operation: Cement Manufacture
Collection Device: Electrostatic Precipitator

Much improvement has been noted recently with the installation of electrostatic precipitation equipment on the cement kilns.

(2) Operation: Coal Burning Power Plant
Collection Device: Multiple Cyclones and Electrostatic Precipitators (in series)

Three new steam boilers, spreader stoker type, were installed by a power company on a contract to supply process steam to a neighboring automobile manufacturer. Soon after the new boilers were put in operation, it was observed that black, sooty particles, relatively large in size, were settling out in the neighborhood, on new cars waiting for pickup, and on other cars and trucks. The sooty particles were highly acidic and etched painted surfaces. Damage was costly and attempts at correction included insulating the breechings, using very low sulfur coals (less than 1%), bypassing sections of the economizers to elevate the effluent gas temperature above the dew point, and finally by injecting anhydrous ammonia into the effluent gas streams, ahead of the economizer sections. The problem was caused by particulates sloughing off from the precipitator plates and a low effluent temperature. This is due to the fact that the exit gases, after passing through the air cleaning equipment, rise up a tall masonry chimney. The interior surfaces are sufficiently cool to cause condensation of acid gases and the condensate in turn, concentrates on particulate material, which eventually sloughs off. The anhydrous ammonia (½ pound per ton of coal) reduces the SO_3 concentration and lowers the dew point of the effluent gases. The difficulty has been minimized since the ammonia injection process was started.

Some Characteristics of Dust Collectors

Table 2 lists some of the important characteristics of collectors for particulates. A more detailed comparison of dust collector characteristics can be found in Figure 11-1, Industrial Ventilation, 8th Ed., 1964.

Collection of Gases and Vapors

Removal of gases and vapors from gas streams may be accomplished by absorption in liquids or solids, by adsorption on solids, and for vapors by condensation, and by catalytic combustion and incineration. In absorption the gas or vapor becomes distributed in the collecting liquid or solid. Adsorption is a surface phenomena. Both processes are diffusional operations. A theoretical discussion of these processes is beyond the scope of this outline, and belongs in the study of Chemical Engineering. However, a brief discussion is given below.

Absorption

Equipment for absorption includes absorption towers, such as bubble-cap plate columns, sieve plate columns, packed towers, spray towers, etc.; washers, such as the wet-cell washers described for dust collection; and special devices such as Venturi injectors. Selection of equipment for a particular task is dependent on the operational variables such as vapor pressures, solubilities, and efficiency of removal required. Successful methods for the

Table 2. - Some Characteristics of Dust Collectors

Type of Collector	Particle Size Range Readily Collected (Microns)	Pressure Drop (Inches Water)
A. Settling Chambers		
1. Simple	40-100	0.1-0.5
2. Multiple Tray	10-50	0.1-0.5
B. Centrifugal (Dry)		
1. Baffle Chamber	20-50	1-3
2. Louvre	15-50	1-3
3. Single Cyclone	15-50	1-2
4. Multiple Cyclone (High Eff.)	5-20	3-6
5. Dynamic (Mechanical)	5-20	---
C. Scrubbers		
1. Spray Tower	5-10	0.5-1.5
2. Wet Centrifugal	2-5	2-6
3. Wet Dynamic	2-5	---
4. Wet Orifice	2-5	3-10
5. Venturi	<1-5	10-30
6. Packed Tower (Trays of solid packing)	3-10	1.5-4
7. Jet	1-5	---
D. Fabric Filter		
1. Tubular and Envelope	<1-5	2.5-6
2. Reverse Jet	<1-5	2.5-8
E. Electrostatic		
1. Single Stage	1-3	0.25-0.5
2. Two-Stage	1-3	0.25-0.5

removal of sulfur dioxide, hydrogen sulfide, fluorides, and nitrogen oxides have been developed.

Adsorption

For the adsorption of gases and vapors, solid adsorbents have been developed which have an affinity for certain substances. Various clays, chars, activated carbons, gels, aluminas, and silicates have been used. They are usually granular in form and are made up in beds or columns through which the gas passes. Adsorption may be practically complete even with very low vapor content, so the procedure is readily adaptable to solvent recovery operations. Adsorbents can collect from 8% to 25% of their weight in vapors. The vapors can be removed from the beds and the beds reused.

Charcoal, for example, will adsorb the following:

acetic acid	carbon disulfide
benzene	diethyl ether
ethyl alcohol	ammonia
carbon tetrachloride	hydrochloric acid
methyl alcohol	nitrous oxide
chloroform	carbon dioxide
acetone	acetaldehyde

Catalytic Combustion and Incineration

In catalytic combustion, a platinum alloy-alumina catalyst is used to burn

hydrocarbons, reducing them to less nocuous compounds. Catalytic combustion is a low-temperature oxidation process by which many gases and vapors from industrial processing are converted to an odor-free, color-free gas. The catalyst simply provides an activated surface on which the reaction proceeds more readily than in its absence. Where large amounts of particulate are present, these unburnable solids must be removed by dust collectors prior to catalytic incineration. Minimum catalytic ignition temperatures may vary from 350°F to 600°F depending upon the character of the gas or vapor.

Many types of incinerators or boilers may be used to control emissions of noxious gases or vapors. Oil refineries usually "flare" hydrogen sulfide by burning it with other waste gases. In this case, toxic hydrogen sulfide is converted to less toxic sulfur dioxide. Where carbon monoxide is generated as a by-product in a process it may be burned in waste heat boilers to convert CO to CO_2 and thus gain some heat, usually in the form of process steam.

Example and Application of Catalytic Combustion

Operation: Phthalic Anhydride Manufacture

Collection Device: Catalytic Combustion Type

A severe air pollution problem existed in the neighborhood of a plant producing phthalic anhydride starting with naphthalene. Residents of the neighborhood complained of the pungent odors attributable to the presence of alpha-naphthaquinone. Wet collection equipment was applied to the job (high pressure fog spray) but the water effluent had a very low pH, making neutralization too expensive. Catalytic combustion equipment was finally applied and worked successfully apparently because of the presence of oxidizable organic materials including pyroligenous acids.

Summary

Each air cleaning problem is unique. From an evaluation of the nature of the aerosol, gas, or vapor, and the performance required, the appropriate equipment can be selected.

The job does not end with the selection of the equipment. Its performance must be checked after installation, and proper modifications made if needed. Furthermore, a good maintenance program is necessary to insure continued good service.

CONVERSION FACTORS AND EQUIVALENTS

1 m^3 = 35.32 ft^3
1 cm^3 = .0610 in^3
1 ft^3 = 28.32 L.
1 ft^3 = 7.48 gal. (U.S.)
1 L = .0353 ft^3
1 L = 1000 cm^3
1 L = 1.057 qts. (U.S. Liquid)
1 m = 3.28 ft.
1 m = 39.37 in.
1 m = 10^6 microns
1 in = 25.40 mm.
1 in = 25,400 microns
1 mile/hr = 88 ft/min.
1 cm/sec = 1.9685 ft/min.
1 Kg = 2.20 lbs.
1 lb = 453.6 grams
1 oz = 28.35 grams
1 gram = 15.432 grains
1 gram = 10^3 milligrams
1 milligram = 10^3 micrograms
1 ft^3 of water @ 62°F. weighs 62.32 lb.
1 gal. (U.S.) of water weighs 8.33 lb.
1 ft^3 of air at STP weighs 0.075 lb.
1 lb/in^2 = 2.30 ft. H_2O
1 $gram/cm^3$ = 62.43 lbs/ft^3
1 $gram/cm^3$ = 8.345 lbs/gal

1 in. of Mercury (Hg.) = 13.57 in. H_2O
1 in. of Hg. = 0.49 lb/in^2
1 mm. of Hg. = 1.36 cm. of H_2O
1 atmosphere = 14.70 lb/in^2
1 atmosphere = 29.92 in. Hg.
1 atmosphere = 760 mm. Hg.
1 Btu = 0.25 large calories
1 Btu = Heat req'd to raise temp. of 1 lb. of H_2O 1°F.
1 Btu = Heat req'd to raise (approx.) 55 ft^3 of air 1°F.
1 $Btu/hr\text{-}ft^2$ = 3.154 x 10^{-4} $watts/cm^2$
1 $Cal/sec\text{-}cm^2$ = 13,263 $Btu/hr\text{-}ft^2$
1 $Cal/sec\text{-}cm^2$ = 4,183 $watts/cm^2$
1 H.P. = 550 ft. lbs/sec.
1 H.P. = 0.75 Kilowatt
1 H.P. = 2,545 Btu
°F = 1.8 (°C.) + 32
°C = (°F - 32) ÷ 1.8
°C = 273° Kelvin (°K)
°F = 460° Rankine (°R)
π = 3.1416
Circumference of circle = πd.
Area of circle = $\pi d^2/4$
Surface area of sphere = πd^2
Volume of sphere = $\pi d^3/6$

* *

PROBLEMS

1. a. Calculate a worker's daily 8-hour exposure to a solvent with a current TLV of 100 ppm if he spends 1 hour at an operation where the concentration is 250 ppm; 4 hours at 200 ppm; and 3 hours at 100 ppm.

   b. If you returned to this same plant two weeks later and found similar exposures at the same operation, would you recommend that control measures be instituted? If so, why?

2. You have collected three samples for a given fume using a portable electrostatic precipitator which you assumed was operating at 3 cfm. Each sample was collected for 30 minutes and on this basis the laboratory reported the following results; No. 1 - 0.085 mg/m^3; No. 2 - 0.070 mg/m^3; and No. 3 - 0.040 mg/m^3. However, upon recalibrating the precipitator just after you returned from the field trip, you found that it was operating at 2.75 cfm. What are the correct values for these three samples? (1 cu. ft. = 0.0283 cu. m).

3. Several samples were collected on different days in a zinc smelter to determine a worker's exposure to lead fume. These samples were collected on molecular membrane filters at 0.5 cfm for 20 minutes. The laboratory reported these results: (1) Drawing metal, 1st draw: 17, 18, 14 micrograms; (2) drawing metal, 2nd draw: 22, 26, 32 micrograms; (3) Drawing metal, 3rd and final draw: 38, 35, 48 micrograms; (4) General room air: 4, 6, 2 micrograms. This worker spends 1½ hours for each draw and the balance of an 8-hour day is spent watching the furnaces. Assume for this period, he has the same exposure as the general room air. (One microgram = 0.001 milligram).

   a. From the above information calculate this worker's average 8-hour daily weighted exposure in mg/m^3.

   b. Is this exposure above or below the current threshold limit value for lead?

4. If the results in Problem 3 were five times those found, how would you recommend that this worker's daily exposure be reduced to a safe level under the following conditions: (1) At present no mechanical means has been devised to reduce the concentration of lead fume during drawing; (2) because of the extreme heat, it is not practical for the workers to wear respirators.

5. Calculate the diameter for a constant flow orifice to provide an air flow of 1.0 cfm (for a membrane filter sampler). Use the following formula:

$$W = \frac{0.53\ (C_v)(A_2)(P_1)}{\sqrt{T_1}}$$

   Assume the following:
   (1) 2 cmHg resistance through filter at 1.0 cfm.
   (2) Source of suction capable of delivering 28" Hg vacuum.
   (3) Barometric pressure equals 30" Hg.

   Given the following:
   W = mass of air in lbs/sec (density of air at 70°F - .074 lbs/ft^3).
   C_v = constant = 1.
   A_2 = orifice area in sq. in. = ?
   T_1 = absolute temperature in °R at 70°F is 530° R.
   P_1 = upstream pressure in lbs/sq. in. (1" Hg = .4912 lbs/sq. in.)

6. A two-pound cylinder of chlorine gas fell off a lab table and broke permitting the gas to escape into a closed room of 50 by 27 by 15 feet.

   a. Calculate the concentration in ppm. The barometric pressure is

760 mm of Hg and the temperature is 22° C.

b. On the basis of the current TLV's would it be dangerous to remain in this room for three hours?

7. In a test set-up, assuming standard temperature and pressure, a concentration of benzol (benzene) is desired at the TLV. What volume of liquid benzol must be vaporized to give this average concentration in a room 10 x 12 x 12 ft.?

8. An analytical procedure requires a minimum of 5 mg of offending material in a total sample in order to obtain satisfactory analytical accuracy. The TLV of this material is 75 ppm. It is suspected that the air concentration is three times greater than the TLV. The molecular weight of the material is 138. The sampling rate for the collecting device is 0.1 cfm. The temperature is 22°C and the barometric pressure is 750 mm Hg. What minimum time is necessary to collect one air sample?

9. A surge tank, used for line storage of a liquid (at room temperature) in a manufacturing process, is located in an occupied building separate from the process. The tank breathes through a vent in the top and during a 10-hour filling cycle fills at a rate of 15 gallons per minute. The vapor pressure of this liquid at room temperature is known to be 84.50 mm of Hg. Barometric pressure is 760 mm Hg. What concentration of the vapor might be expected in the tank? Express answer in both percent and ppm.

10. a. The vapor pressure of mercury at 79°F and 760 mm Hg pressure is 0.0020 mm Hg. If the mercury vapor were allowed to reach equilibrium at this temperature in an enclosed space, what would be the vapor concentration?

b. By what factor is the current TLV exceeded?

11. Acrolein has the formula CH_2:CHCHO. At an air concentration of its TLV, how many liters of air must be sampled to collect 1 mg. of acrolein? Assume 760 mm Hg and 25°C.

12. A dust sample was taken with a Greenburg-Smith impinger for 15 minutes at a sampling rate of 1 cfm. After dilution of the sample to 500 ml, a dust count was made by means of a microscope using a Dunn cell. The field counted was determined by the use of a square ocular grid which measured 0.54 mm on a side using a stage micrometer. Dust counts obtained were as follows: 1st slide: 71, 66, 57, 68, 61; 2nd slide: 78, 60, 72, 67, 73. The blank count was 7. Calculate the dust concentration in millions of particles per cubic foot (mppcf).

13. If the sample from problem 12 had been taken in a foundry and the analysis of the air-borne dust showed 60% free silica, does the dust concentration exceed the current TLV as set forth by the ACGIH?

14. The following data were obtained upon sizing a sample of dust, using a Porton Eyepiece graticule. Plot the data on log probability paper and determine the median size and geometric standard deviation of the dust.

Number of Particles	Size in Microns
10	0.48
22	0.68
26	0.96
29	1.36
37	1.92
28	2.72
22	3.84
14	5.44
8	7.68
4	10.88

15. Calibration of a filar micrometer eyepiece with a 90X objective and a stage micrometer shows that 515 filar units represents 0.05 mm. What is the value in microns of one filar unit? (1 micron = 0.001 mm.)

16. Calibration of a Porton eye graticule shows that the 100 L width of the graticule rectangle represents 0.025 mm. What is the geometric deviation for a sample which yields the following size distribution data?

$$D = L\sqrt{2}^{N}$$

where: D = circle diameter in microns (μ)

L = Porton linear unit in microns

N = circle number

Circle Number	Number of Particles
1	12
2	24
3	30
4	42
5	36
6	28
7	17
8	6
9	3
10	1
12	1

17. Using the results of problem 16, determine the geometric mean diameter by weight.

18. Air-borne particulates settle according to Stokes' Law with a velocity, as stated in the following formula:

$$V \text{ cm/sec} = \frac{(g)(d^2)(\sigma_p - \sigma_a)}{18\mu}$$

Where:

g = acceleration of gravity (981 cm/sec^2)

d = diameter of particle (cm)

σ_p = sp. g. of particle

σ_a = sp. g. of air (neglected)

μ = viscosity of air (poises) = 181 x 10^{-6} poise

a. Using the above information, calculate the rate of settling of a material having a sp.g. of 1 for 12, 7, and 2 micron-diameter particles.

b. What is the settling velocity of a 2 micron particle of quartz (sp.g. = 2.75)?

19. The following octave band analyses are obtained in two plating rooms. (A and B) thus:

cps	20 75	75 150	150 300	300 600	600 1200	1200 2400	2400 4800	4800 & over
dB (A)	86	90	92	98	96	92	89	84
dB (B)	70	72	75	85	89	93	95	92

What are the over-all noise levels in each of the plating rooms?

20. Using the octave band analysis (A) from problem 19, determine the loudness level in the plating room. The graph for computing loudness level (see page D-2-5) will aid in solving this problem. Center frequencies for the octave bands are:

Octave Bands	Center Frequency	Octave Band (A) Analysis
37.5-75	53	86
75-150	106	92
150-300	212	94
300-600	424	98
600-1200	849	96
1200-2400	1700	92
2400-4800	3390	89
4800-Over	6790	84

21. Two adjacent machines operate intermittently in a room. Each machine was studied individually and the over-all noise level due to machine Y alone was 99 dB. The over-all noise level due to machine Z alone was 97 dB.

    a. What over-all noise level is to be expected when both machines operate simultaneously?

    b. If machine Z were treated so that the over-all noise level due to Z alone was 87 dB, what over-all noise level would then result when both machines are operated.

22. A fan is purchased having the following characteristics: Direct drive, 4800 RPM. to deliver 2400 cfm at ¼", backward-curved blades, 30 blades. What predominate frequency can be expected from the fan?

23. The acoustical treatment of a room has reduced the over-all noise level by 3 dB. By what factor has the acoustical power been reduced?

24. The following information on the thermal environment has been reported:

    Globe temperature = 105° F
    Dry-bulb or air temperature = 84° F
    Wet-bulb temperature = 70° F
    Air velocity = 100 fpm

    What is the effective temperature corrected for radiation?

    (Use Psychometric Chart on page B-29-3 and Figures 3-4 in Industrial Ventilation).

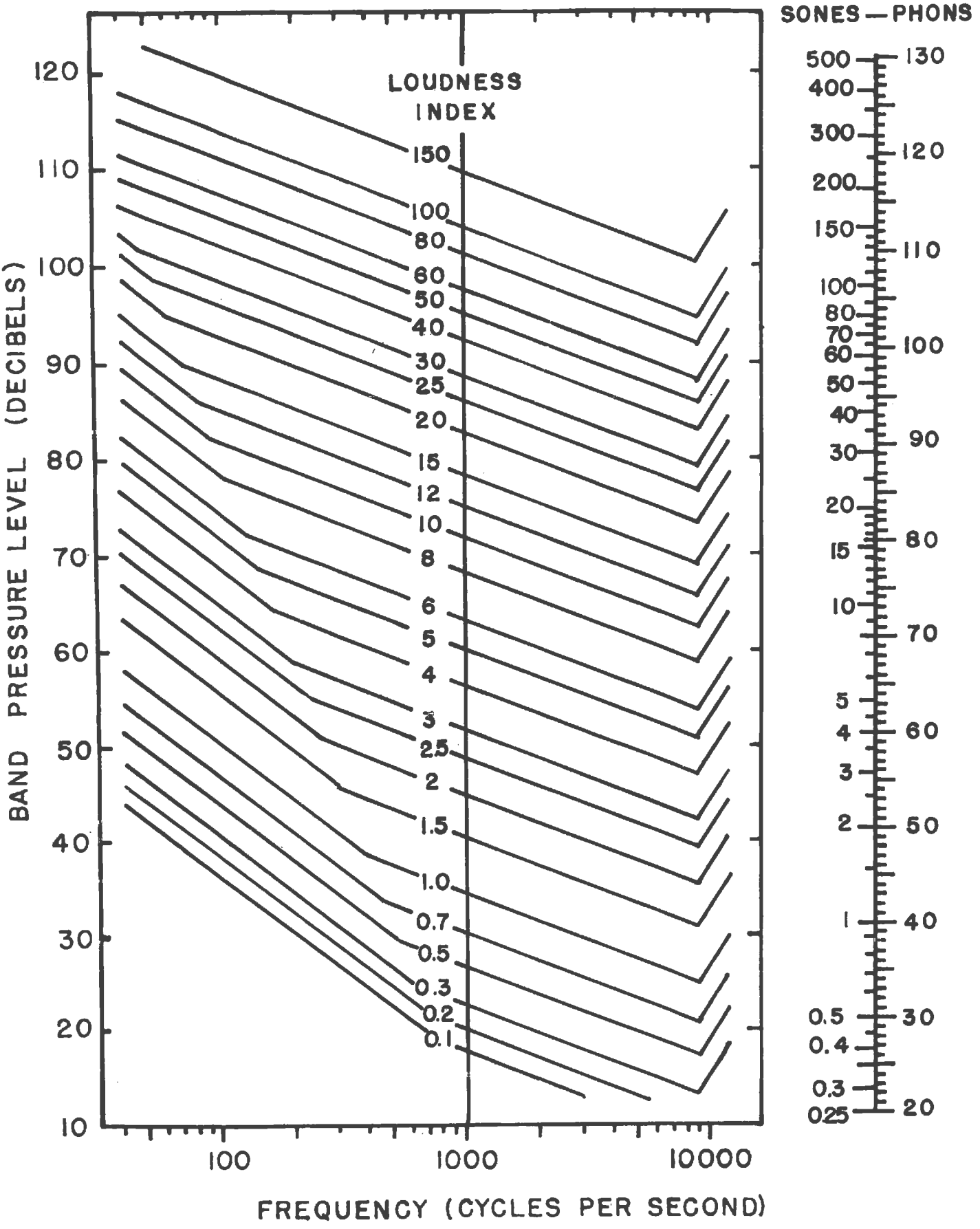
25. A material balance study indicates that two pints of toluene (toluol) are evaporated per eight hour shift in a processing room at a plasticizer plant. What volume of dilution ventilation is required per shift to maintain the general air concentration at the current TLV of 200 ppm (assume a K factor of 4)?

26. Using the result of problem 25 what would be the required flow rate of the ventilation in cubic feet per minute (cfm)?

27. a. 1250 cfm is to be exhausted through a round, freely suspended duct (6" diameter). Calculate the expected velocity at 6" from the duct opening along the center line of the duct.

    b. A 6" flange is attached to the above duct. What center line velocity then results at 6"?

28. a. A freely suspended hood, 2" wide and 72" long, exhausts 600 cfm.


LOUDNESS INDEX
150
100
80
60
50
40
30
25
20
15
12
10
8
6
5
4
3
2.5
2
1.5
1.0
0.7
0.5
0.3
0.2
0.1
BAND PRESSURE LEVEL (DECIBELS)
120
110
100
90
80
70
60
50
40
30
20
10
100
1000
10000
FREQUENCY (CYCLES PER SECOND)
SONES — PHONS
500
400
300
200
150
100
80
70
60
50
40
30
20
15
10
5
4
3
2
1
0.5
0.4
0.3
0.25
130
120
110
100
90
80
70
60
50
40
30
20

What is the velocity at a point 12" from the opening?

b. What is the velocity at this same point after flanging?

29. a. The static pressure immediately downstream from a 24"-wheel grinder hood was measured to be 1.75" H_2O. The throat diameter was 6" and the hood shape was that of a standard grinder hood (see Figure 6-10 of Industrial Ventilation). What air flow would this reading indicate?

b. In a straight section of 6" duct following the above hood a Pitot traverse was made and the average velocity was found to be 3000 fpm. Explain the discrepancy.

30. Air velocity measurements in three ducts indicate the following velocities: 1266 fpm, 4005 fpm, and 5665 fpm.

Calculate the velocity pressure (VP) for each of the given velocities.

31. A 10-point Pitot tube traverse for air at 70°F and normal barometric pressure flowing in a 12" circular duct gives the following data:

Traverse Point - " From Wall	Vertical Traverse Δh ("H_2O)	Horizontal Traverse Δh ("H_2O)
3/8	0.10	0.14
1	0.11	0.18
1-3/4	0.19	0.22
2-3/4	0.27	0.30
4-1/8	0.35	0.35
7-7/8	0.34	0.36
9-1/4	0.29	0.24
10-1/4	0.22	0.19
10-7/8	0.16	0.14
11-5/8	0.13	0.11

How many cfm are flowing through the pipe?

32. a. Using the data of problem 31 and also knowing that the throat suction, h, at the inlet of this pipe is 0.44 H_2O", what is the coefficient of entry, C_e, of the inlet?

b. A subsequent throat suction measurement gives a reading of 0.28 "H_2O. It is ascertained that the inlet is unchanged. What is the quantity of air flowing through the pipe under these conditions?

33. Room air at 70°F is exhausted at a rate of 100 cfm per enclosure through each of 10 enclosures where enamel frit is fused. The air temperature rises to 500°F before leaving the enclosure. The duct work is insulated to the blower housing. What volume of air must the fan be capable of handling?

34. A fabric dust collector handles 400 cfm with a dust loading of 10 gr/ft³. Its initial resistance is 1" of water gauge. At the end of 6 hours it reaches the maximum permissible resistance of 5" of water.

How soon would 5" water gauge be reached for:

(a) a dust loading of 20 gr/ft³ and a flow rate of 400 cfm?

(b) a dust loading of 10 gr/ft³ and a flow rate of 800 cfm?

SOLUTIONS OF PROBLEMS

1. a. Calculate 8-hour exposure to a solvent with a current TLV of 100 ppm if:

   1 hour @ 250 ppm
   4 hours @ 200 ppm
   3 hours @ 100 ppm

   $$\frac{(1)(250)+(4)(200)+(3)(100)}{8} =$$

   $$\frac{250 + 800 + 300}{8} = \frac{1350}{8} = 169 \text{ ppm}$$

   b. Control is necessary since the 8-hour exposure exceeds the TLV of 100 ppm.

2. Three fume samples collected at an assumed flow of 3 cfm for 30 minutes.

   Results reported 1. = 0.085 mg/m³
   2. = 0.070 mg/m³
   3. = 0.040 mg/m³

   Actual flow = 2.75 cfm

   Corrected results:

   1. $0.085 \times \frac{3}{2.75} = 0.093$ mg/m³

   2. $0.070 \times \frac{3}{2.75} = 0.076$ mg/m³

   3. $0.040 \times \frac{3}{2.75} = 0.044$ mg/m³

3. Lead fume samples

   0.5 cfm for 20 min. =
   10 ft³ air sample = 0.283 m³ of air.

   a. Drawing metal

   1st Draw: 17, 18, 14 micrograms (1½ hr. per day); ave. = 16 micrograms.
   2nd Draw: 22, 26, 32 micrograms (1½ hr. per day); ave. = 27 micrograms.
   3rd Draw: 38, 35, 34 micrograms (1½ hr. per day); ave. = 40 micrograms.

   General Air

   4, 6, 2 micrograms (3½ hr. per day); ave. = 4 micrograms.

   Weighted Exposure =

   $$\left[\frac{3}{2}\right]\left[\frac{0.016}{0.283}\right] + \left[\frac{3}{2}\right]\left[\frac{0.027}{0.283}\right] +$$

   $$\left[\frac{3}{2}\right]\left[\frac{0.040}{0.283}\right] + \left[\frac{7}{2}\right]\left[\frac{0.004}{0.283}\right] \div 8 =$$

   $$\frac{0.048 + 0.081 + 0.120 + 0.028}{(2)(0.283)(8)} =$$

   $$\frac{0.277}{(16)(0.283)} = 0.061 \text{ mg/m}^3$$

   b. Check this answer against the current list of TLV's.

4. Assume exposures in problem 3 were 5 times as great as those found and

   (1) no practical ventilation is possible, and
   (2) it is too hot to wear respirators.

   Weighted exposure would be 5(0.06) = 0.30 mg/m³ which is greater than the 1964 TLV of 0.20 mg/m³.

   a. The weighted daily exposure could be lowered by reducing the work time of the individual; for example, a man could work at either of the following:
   During 1st and 2nd draws and spend 6½ hours in general air = 8 hours

   $$(3)(0.016)(5) + (3)(0.027)(5) + (10)(0.004)(5) \div (2)(0.283)(8) = 0.188 \text{ mg/m}^3$$

   or during 3rd draw and spend 6½ hours in general air = 8 hours

   $$\frac{(3)(0.040)(5) + (13)(0.004)(5)}{(2)(0.283)(8)} =$$

   $$0.190 \text{ mg/m}^3$$

b. Urinalysis may be run for Pb to see if the urine Pb levels are elevated appreciably above the normal range. Currently (1965) the safe range is from 0.2 mg/l (urine) to 0.08 mg/l. As values approach and become greater than 0.08 mg/l it may be assumed that the man is being exposed to a hazardous concentration of lead.

5. Constant flow orifice calculation for flow of 0.5 cfm

$$W = \frac{0.533\ C_v A_2 P_1}{\sqrt{T_1}}$$

where: W = lb/sec of air @ STP
C_v = constant = 1
A_2 = orifice area in in^2
T_2 = 530°R (70°F)
P_1 = upstream pressure in psi

$$W = (1.0\ ft^3/min)(0.074\ lb/ft^3)(1\ min/60\ sec)$$

$$= \frac{(1.0)(0.074)}{60} =$$

$$= 0.00123\ lb/sec$$

$$A_2 = \frac{(0.00123)(\sqrt{530})}{(0.533)(1)(14.696-0.387)}$$

$$= 0.00371\ in^2$$

$$D_2 = \sqrt{\frac{4A_2}{\pi}} = \frac{(4)(0.00371)}{3.14}$$

$$= \sqrt{0.00473}$$

$$D_2 = 0.069\ inch$$

6. Cylinder of chlorine -

a. $$ppm = (weight)(22.4L)\left[\frac{760}{P}\right]\frac{(273 + T°C)}{273} \times 10^6 \div (mw)(gm\text{-}mole)(volume)$$

$$= \frac{(2\ lbs)(454\ gms)(22.4L)}{(70.9gm)\ (lb)\ (gm\text{-}mole)} \times \frac{(760)(295)}{(760)(273)(10^6)} \div \frac{(50ft)(27ft)(15ft)}{(0.035ft^3)}$$

$$= 536\ ppm$$

b. The 1964 TLV for chlorine gas is 1 ppm. Therefore, it would be very dangerous.

7. Prepare benzol concentration at the TLV.

TLV = 25 ppm
Volume of room = 1440 ft^3

Assume standard temperature and pressure

ml benzol =

$$\frac{(ppm)\ (gm)\ \ (volume)(28.3L)}{(22.4)(gm\text{-}mole)\ (Sp.G.)\ (ft^3)(10^6)}$$

$$= \frac{(25)(78)(10)(12)(12)(28.3)}{(22.4)(10^6)(0.879)} = 4.04$$

8. Calculate the time necessary for sampling given the following:

TLV = 75 ppm
M.W. = 138

Sampling rate = 0.1 cfm
Temperature = 22°C
Pressure = 750 mm Hg
Minimum amount of material required = 5 mg

$$t = \frac{\left[\text{minimum amt. of material}\right]\left[\frac{22,4L}{gm\text{-}mole}\right]}{(\text{sampling rate})(TLV)(MW)} \times \frac{(10^6)\left[\frac{760}{P}\right]\left[\frac{273 + °C}{273}\right]}{\frac{(28.3L)}{ft^3}}$$

$$= \frac{(5\ mg)\left[\frac{0.001\ gm}{mg}\right]\left[\frac{22.4L}{gm\text{-}mole}\right]}{(0.1\ ft^3)(75)\left[\frac{138\ gm}{gm\text{-}mole}\right]} \times$$

$$\frac{(10^6)\left(\frac{760}{750}\right)\left(\frac{295}{273}\right)}{\frac{(28.3L)}{ft^3}} = 4.19 \text{ min.}$$

Always sample to be able to at least detect the TLV, and longer if practical; not on the basis of the suspected air concentration.

9. Surge tank at room temperature breathes through a vent during 10 hr. filling at rate of 15 gal./min.

Vapor pressure = 84.50 mm Hg. at room temperature.

Concentration in tank = saturation concentration.

$$= \frac{84.50}{760} \times 100\% = 11.12\%$$

$$= \frac{84.50}{760-84.50} \times 10^6 \text{ ppm} = 125{,}091 \text{ ppm}$$

10. Mercury vapor

a. Vapor pressure Hg at 79°F = 0.0020 mm Hg; M.W. = 200.6.

Equilibrium concentration =

$$\frac{0.0020 \text{ mm Hg}}{760 \text{ mm Hg}} \times \frac{200{,}600 \text{ mg/mole}}{0.0244 \text{ m}^3\text{/mole}}$$

$$= \frac{2.0}{760} \times \frac{200{,}600 \text{ mg/m}^3}{24.4}$$

$$= 21.6 \text{ mg/m}^3$$

b. The 1964 TLV for Hg = 0.1 mg/m³; the TLV is exceeded 216 times.

11. Acrolein: $CH_2;CHCHO$

a. 1964 TLV = 0.1 ppm; M.W. = 56; assume 760 mm Hg and 25°C. How many liters of air at 0.1 ppm are needed to collect 1 mg?

Volume of air

$$= \frac{\left(\frac{\text{amount}}{\text{collected}}\right)\left(\frac{24.4L}{\text{gm-mole}}\right)(10^6)}{(\text{TLV})(\text{MW})}$$

$$= \frac{(1 \text{ mg})\left(\frac{.001 \text{ gm}}{\text{mg}}\right)\left(\frac{24.4L}{\text{gm mole}}\right)(10^6)}{(0.1)\left(\frac{56 \text{ gm}}{\text{gm-mole}}\right)}$$

$$= 4357 \text{ liters}$$

12. Dust sample by Greenburg-Smith impinger for 15 min. at 1 cfm. Dilution to 500 ml., counted in Dunn cell - grid was 0.50 mm on a side.

Dust counts: 71, 66, 57, 68, 61; 78, 60, 72, 67, 73: Average = 67.3; Blank = 7.

$$\text{mppcf} = (N_s - N_b) \times \frac{1000}{(\text{field area})(\text{cell depth})} \times (\text{sample volume}) \div \text{Air volume}$$

$$\text{mppcf} = \frac{(67.3-7)(0.500)(\text{cm}^3)(10^{-6})}{(15)(\text{ft}^3)(0.290)(10^{-3}\text{cm}^3)}$$

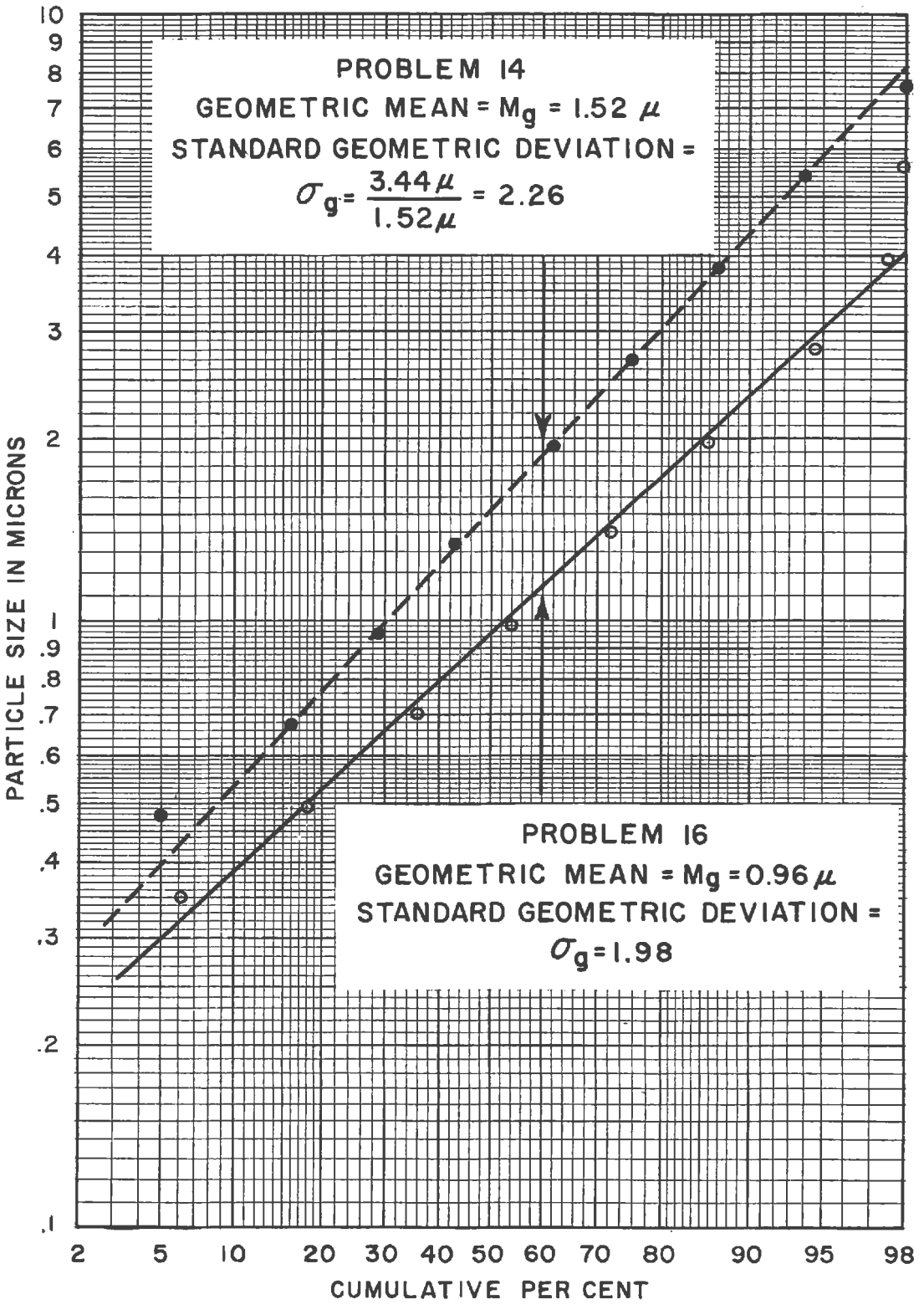
$$= \frac{(60.3)(0.5)}{(15)(0.29)} = 6.9 \text{ mppcf}$$

13. The TLV for a mineral dust containing 60% free silica can be computed from the following formula:

$$\text{TLV} = \frac{250}{\%SiO_2 + 5} = \frac{250}{60 + 5}$$

$$= 3.85 \text{ mppcf}$$

Since the answer to problem 12 is 6.9 mppcf it does exceed the current TLV.


PROBLEM 14
GEOMETRIC MEAN = M_g = 1.52 μ
STANDARD GEOMETRIC DEVIATION =
$\sigma_g = \frac{3.44\mu}{1.52\mu} = 2.26$
PROBLEM 16
GEOMETRIC MEAN = M_g = 0.96 μ
STANDARD GEOMETRIC DEVIATION =
σ_g = 1.98
PARTICLE SIZE IN MICRONS
10
9
8
7
6
5
4
3
2
1
.9
.8
.7
.6
.5
.4
.3
.2
.1
2
5
10
20
30
40
50
60
70
80
90
95
98
CUMULATIVE PER CENT

14. Given a dust sample with the following size analysis, find the median size and geometric standard deviation.

Circle Number	Circle Diameter	Number of Particles	Cumulative Total	Cumulative Percent
1	0.48	10	10	5
2	0.68	22	32	16
3	0.96	26	58	29
4	1.36	29	87	43.5
5	1.92	37	124	62
6	2.72	28	152	76
7	3.84	22	174	87
8	5.44	14	188	94
9	7.68	8	196	98
10	10.88	4	200	100

From the plot of the data (see graph on page D-3-4):

Geometric Mean = Mg = 1.52μ

Standard Geometric Deviation = $\sigma_g = 2.26$

15. 515 filar units represent 0.05 mm, therefore

$$1 \text{ filar unit} = \frac{(0.05 \text{ mm})(\frac{1000 \text{ mm}}{\text{mm}})}{515 \text{ filar units}} = 0.0974\mu$$

16. Find the geometric mean and the standard geometric deviation.

Given: Number of particles corresponding to various circles as shown on page D-3-6 and that 100 L units = 0.025 mm.

$\therefore$ 100 L units = 25μ so 1L unit = 0.25μ.

Substituting this in the formula, $D = L\sqrt{2^N}$,

$D_1 = 0.25\sqrt{2^1} = (0.25)(1.42) = 0.355\mu$

$D_2 = 0.25\sqrt{2^2} = (0.25)(2) = 0.50\mu$

D_3, D_4, etc., are shown on page D-3-6.

Circle Number	Circle Diameter	Number of Particles	Cumulative Total	Cumulative Percent
1	0.355	12	12	6
2	0.50	24	36	18
3	0.707	30	66	33
4	1.0	42	108	54
5	1.412	36	144	72
6	2.0	28	172	86
7	2.82	17	189	94.5
8	4.0	6	195	97.5
9	5.65	3	198	98
10	8.0	1	199	99.5
11	11.30	1	200	100
12	16			

See the plot of this data on graph (page D-3-4) following the solution to problem 14.

Geometric mean = $M_g = 0.96\mu$

Standard geometric deviation = $\sigma_g = 1.98$

---

17. Geometric mean diameter by weight from problem 16.

$$\text{Log } M'_g = \text{Log } M_g + 6.908 \log^2 \sigma_g$$

Where M'_g = Geometric mean by weight

$$= \text{Log } 0.96 + 6.908 \times \text{Log}^2 198$$

$$= \text{Log } 0.96 + 6.908 \times (0.297)^2 = 0.018 + 6.908(0.088) = 0.590$$

$\therefore M'_g = 3.89$

18. Stokes' Law:

$$V \text{ cm/sec} = \frac{(g)(d^2)(\sigma_p - \sigma_a)}{18\,\mu}$$

where:

g = gravitational acceleration = 981 cm/sec^2

d = particle diameter (cm)

σ_p = particle specific gravity

σ_a = specific gravity air

μ = viscosity of air equal to 181×10^{-6} poise

18. continued

$$V = \frac{(981)(d^2)(\sigma_p - \sigma_a)}{18(181 \times 10^{-6})} =$$

$3.0 \times 10^5 \, d^2\sigma_p$ cm/sec

a. For particles of $\sigma_p = 1$, settling velocity for various diameters are:

12μ: $V=3.0 \times 10^5 \times (12 \times 10^{-4})^2 = 0.432$ cm/sec

7μ: $V= 3.0 \times 10^5 \times (7 \times 10^{-4})^2 = 0.147$ cm/sec

2μ: $V=3.0 \times 10^5 \times (2 \times 10^{-4})^2 = 0.012$ cm/sec

b. For 2μ quartz ($\sigma_p = 2.75$)

$V = 3.0 \times 10^5 \times (2 \times 10^{-4})^2 \times 2.75 = 0:033$ cm/sec

19. Determine the over-all noise levels in each of the plating rooms.

cps	20-75	75-150	150-300	300-600	600-1200	1200-2400	2400-4800	4800 over
dB(A)	86	90	92	98	96	92	89	84
dB(B)	70	72	75	85	89	93	95	92

Noise level (A)

98dB	98dB	
96	98	+ 2.1 = 100.1*
94	100.1	+ 0.9 = 101.0
92	101.0	+ 0.5 = 101.5
90	101.5	+ 0.3 = 101.8
89	101.8	+ 0.2 = 102.0
86	negligible	
84	nnegligible	
	102.0 dB	

Noise level (B)

95dB	95dB	
93	95	+ 2.1 = 97.1*
92	97.1	+ 1.2 = 98.3
89	98.3	+ 0.5 = 98.8
85	98.8	+ 0.2 = 99.0
75	negligible	
72	negligible	
70	negligible	
	99.0 dB	

*Combination of dB from table on B-23-3 or Appendix II in General Radio Handbook of Noise Measurement.

20. Determine the loudness level in the plating room A.

1. From the graph on page D-2-5 determine the loudness index for each of the Band Pressure Levels.
2. The calculated loudness index can be computed either from the following equation or from the table on page D-2-8:

$$\text{Calculated Loudness Index} = S_m + 0.3\,(\Sigma S - S_m)$$

Where: S_m = Maximum band loudness index

ΣS = Summation of band loudness indexes

The largest loudness index is multiplied by 1.0 and the remaining ones by 0.3. These are added to obtain the calculated loudness index in sones.

3. From the nomograph on the right of the graph on page D-2-5 obtain the loudness level by entering the calculated loudness index in sones and reading the loudness in phons.

Center Frequency	Band Pressure Level	Loudness Index	Multiply Times	Calculated Loudness Level
53	86	9	0.3	2.7
106	90	17	0.3	5.1
212	94	28	0.3	8.4
424	98	49	0.3	14.7
849	96	49	0.3	14.7
1700	92	43	0.3	12.9
3390	89	50	1.0	50.0
6790	84	40	0.3	12.0
				120.5

Total calculated loudness index = 120.5 sones
From nomograph, loudness level = 109 phons

21. Two adjacent machines operate intermittently in room

machine Y alone = 99 dB over-all
machine Z alone = 97 dB over-all

(a) Combined noise level: 99 + 2.1* = 101.1 dB

(b) If Z is reduced to 87 dB, combined noise level is 99 + 0.3* = 99.3 dB

* Combination of dB from Table on B-23-3 or Appendix II in General Radio Handbook of Noise Measurement.

22. A fan has the following characteristics:

direct drive - 4800 rpm
delivers 2400 cfm @ ¼" H_2O
30 blades - backward curved

Find: Predominant frequency (Frequency for which attenuation design should be calculated when duct transmission is a problem).

$$\frac{4800 \text{ rpm} \times 30 \text{ blades}}{60 \text{ sec/min}} = 2400 \text{ cps}$$

23. Reducing the over-all noise level 3 dB reduces the acoustical power by one-half:

$$W = 10 \log_{10} \frac{P_1}{P_2}$$

$$3 \text{ dB} = 10 \log_{10} \frac{P_1}{P_2}$$

$$\frac{3}{10} = \log_{10} \frac{P_1}{P_2}$$

$$\frac{P_1}{P_2} = 10^{0.3}$$

$$\frac{P_1}{P_2} = 2 \text{ or } P_2 = \frac{P_1}{2}$$

24. Effective temperature corrected for radiation. Given:

Globe temperature = t_g = 105°F

Dry-bulb or air temperature = t_b = 84°F

Wet-bulb temperature = t_w = 70°F

Air velocity = 100 fpm

24. continued -

Procedure:

1. Determine absolute humidity using t_b and t_w from psychometric chart. Absolute humidity = 84 grains per lb. dry air.
2. Determine pseudo wet-bulb temperature using absolute humidity and t_g from psychometric chart. Pseudo wet-bulb = 75.8°F.
3. Determine effective temperature corrected for radiation by using pseudo wet-bulb temperature as t_w and t_g as t_d on the effective temperature chart on Fig. 3-5 in Industrial Ventilation. Effective temperature corrected for radiation = 84°F.

25. The volume of dilution ventilation required can be found by using the following formula:

$$\text{Ft}^3 \text{ of air} = \frac{(403)(\text{Sp.Gr.})(\#\text{ of pints})(10^6)(K)}{(mw)(TLV)}$$

$$= \frac{(403)(.87)(2)(10^6)(4)}{(92)(200)}$$

$$= 152{,}360 \text{ ft}^3/\text{shift}$$

26. The required flow rate would be: (answer to problem 25 is 152,360 ft³/shift).[3]

$$\text{cfm} = \frac{152{,}360 \text{ ft}^3}{(8 \text{ hr})\left(\frac{60 \text{ min.}}{\text{hr.}}\right)}$$

$$= 317.4 \text{ cfm}$$

27. a. Exhaust 1250 cfm through a round, freely suspended 6" duct. Find velocity at 6" from duct opening along centerline

$$V = \frac{Q}{10x^2 + A} \qquad Q = 1250 \text{ cfm}, \quad A = 0.1964 \text{ ft}^2$$

$$\text{At 6"}: V = \frac{1250}{(10)(\frac{1}{2})^2 + 0.1964}$$

$$= \frac{1250}{2.6964} = 464 \text{ ft/min.}$$

b. Same, flanged:

$$V = \frac{Q}{(0.75)(10x^2 + A)}$$

At 6":

$$V = \frac{464}{0.75} = 619 \text{ ft/min.}$$

28. Freely suspended hood 2" wide and 72" long.

$$Q = 600 \text{ cfm}$$

Find velocity 12" from opening - flanged and unflanged

a. Unflanged:

$$V = \frac{Q}{3.7xL} = \frac{600}{(3.7)(1)(6)}$$

$$= 27.0 \text{ ft/min.}$$

b. Flanged:

$$V = \frac{Q}{2.8xL} = \frac{600}{(2.8)(1)(6)}$$

$$= 35.7 \text{ ft/min.}$$

29. a. 24" wheel grinder - 6" duct-hood is standard grinder type.

S.P. = 1.75" H_2O; C_e = 0.78

$$Q = 4005\,(A)(C_e)\sqrt{SP}$$

$$Q = 4005\,(0.1964)(0.78)\sqrt{1.75}$$

$$Q = 812 \text{ cfm}$$

b. Possible explanations for discrepancy with Pitot velocity measurement:

(1) Obstruction in duct

(2) Deformed or crushed duct

(3) Error in static pressure reading.

30. Velocity pressures (VP) for the given velocities can be calculated using the following equation:

$$VP = \left(\frac{V}{4005}\right)^2$$

$$= \left(\frac{1266}{4005}\right)^2 = 0.1" \ H_2O$$

$$= \left(\frac{4005}{4005}\right)^2 = 1" \ H_2O$$

$$= \left(\frac{5664}{4005}\right)^2 = 2" \ H_2O$$

31. Given: Ten Point-Pitot Traverse in 12" Circular Duct (70°F) (1 atm)

Δh	$V=4005\sqrt{h}$	Δh	$V=4005\sqrt{h}$
0.10	1266	0.14	1498
0.11	1328	0.18	1699
0.19	1746	0.22	1879
0.27	2081	0.30	2193
0.35	2369	0.35	2369
0.34	2335	0.36	2403
0.29	2157	0.24	1962
0.22	1879	0.19	1746
0.16	1602	0.14	1498
0.13	1444	0.11	1328
	18207		18575

$18207 + 18575 = 36782$

$$\text{average} = \frac{36782}{20} = 1839 \text{ fpm}$$

$Q = VA = (1839)(.785)$

$Q = 1444$ cfm

32. a. Finding C_e knowing the velocity and the throat suction, h, at the inlet.

$$V = 4005 \ C_e\sqrt{h}$$

$$C_e = \frac{V}{4005\sqrt{h}} = \frac{1839}{(4005)\sqrt{0.44}}$$

$$C_e = \frac{1839}{2660} = 0.69$$

b. Finding new air flow when throat suction, h, is found to be 0.28" H_2O.

$$Q_2 = 4995 \ C_e\sqrt{h}(A)$$

$$= (4005)(0.69)(0.53)(.785)$$

$Q_2 = 1150$ cfm

33. Room air at 70°F exhausted - 10 enclosures at 100 cfm each. Temperature rises to 500°F - Assume ideal gas.

$$(10)(100 \text{ cfm})\left\{\frac{960^\circ R}{530^\circ R}\right\} = 1811 \text{ cfm}$$

∴ The fan must handle 1811 cfm at 500°F.

34. Fabric Dust Collector. Given:

Dust Loading = 10 gr/ft³
Q = 400 cfm
Initial resistance = 1" H_2O
Maximum allowable resistance = 5"H_2O
Time to reach maximum = 6 hours

(a) How long to reach 5" H_2O if Dust Loading = 20 gr/ft³ and Q = 400 cfm?

In given case: Resistance builds at rate of 4" per 6 hrs. or 2/3" per hr.

Since: Flow through bags is in the streamline range; resistance is directly proportional to flow rate. If there is twice as much dust, the given resistance will be reached in ½ the time.

6 hours x ½ = 3 hours

(b) How long to reach 5" H_2O if Dust Loading = 10 gr/ft³ and Q = 800 cfm?

Initial resistance is doubled to 2", leaving 3" for build up. Twice the dust is caught per minute, doubling build up rate. Twice the flow doubles the build up rate.

$$\frac{3" \ H_2O}{2/3" \ H_2O/hr.(2)(2)} = \frac{9}{8} =$$

1.13 hours

*U.S. GOVERNMENT PRINTING OFFICE : 1970 O - 411-012

15