

INSTRUCTOR MANUAL
INDUSTRIAL HYGIENE CHEMISTRY COURSE

LESSON NUMBER 4

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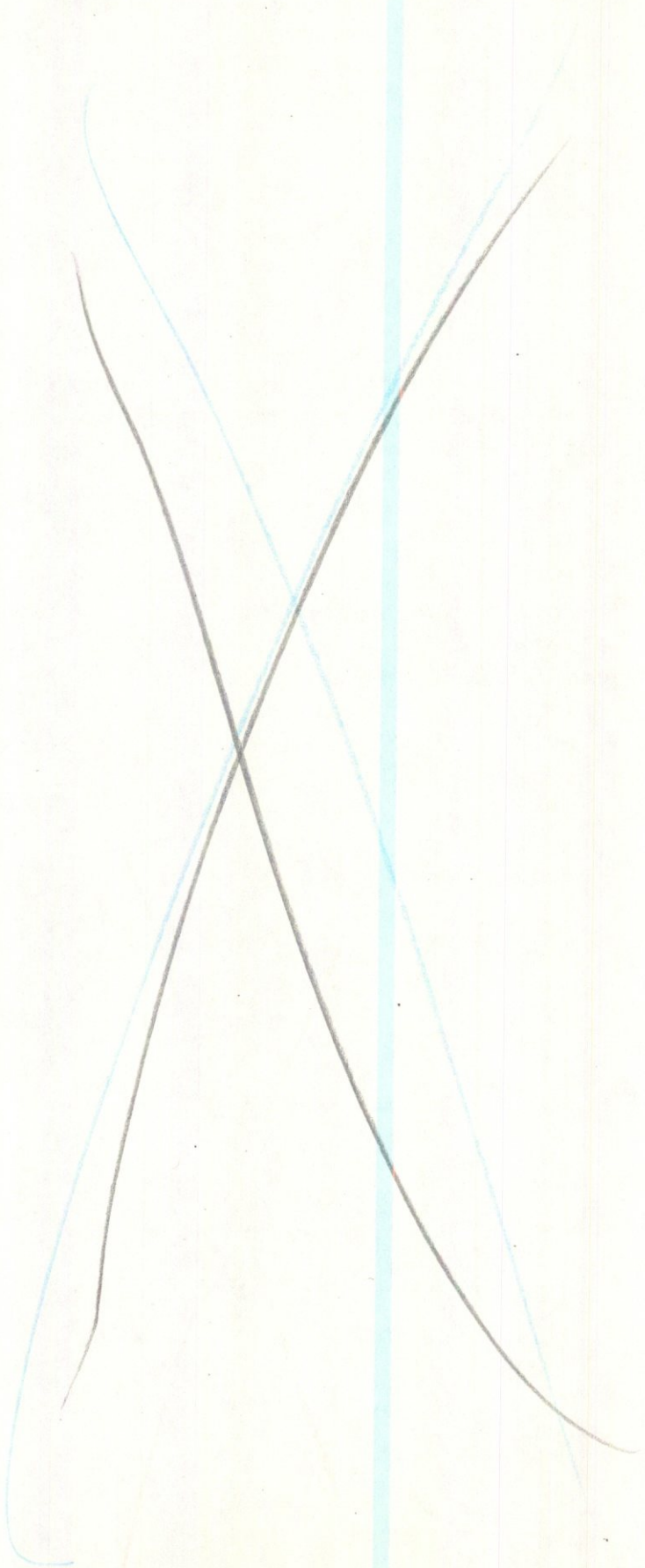
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INTRODUCTION

This Instructor Manual has been prepared for industrial hygienists and analytical chemists participating in the National Institute for Occupational Safety and Health's Regional Training Program. The purpose of this Manual is to assist these professionally qualified, but possibly inexperienced, instructors in the preparation and conduct of a one-week "Industrial Hygiene Chemistry" course. This Manual will guide instructors through both lecture and laboratory lessons. It is complemented by a matching Student Manual. The course is recommended for students having, as a minimum, an undergraduate degree in chemistry (or its equivalent) along with at least one year's experience in instrumental analysis.

It is not necessary for instructors to have had prior teaching experience although such experience would be desirable. All instructors should be thoroughly familiar with industrial hygiene chemistry procedures, instruments and equipment relevant to the subject areas they will teach. In addition, each instructor should attend the course director's orientation seminar presented before the start of each one-week "Industrial Hygiene Chemistry" course.

The remainder of this introduction describes the course objectives, lessons, and the organization and format of the documentation in each lesson, including lecture and laboratory lesson plans.

Course Objectives

The following course objectives will be attained by graduates of this program:

- Given a particular chemical health hazard commonly found in the occupational environment, the trainee will be able to select an appropriate sampling strategy using available sampling techniques and to select a corresponding appropriate analytical method for quantitative characterization of the sample by using his knowledge gained from the course and technical information referenced in the course.

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- Trainee will be able to apply his knowledge of wet chemical and/or instrumental analysis in employment of current methodologies for evaluating the typical work environment.
- Trainee will be able to perform and evaluate quantitative analytical determinations for four classes (types) of hazardous substances using a correspondingly different method for each class or type.
- Given the analytical results obtained through proper measurement procedures, the trainee will be able to define the data in terms of actual environmental concentration levels and to interpret the results in light of existing exposure standards.

Lessons

18 lessons are presented in this course:

- Introduction to Course
- Introductory Topics
- Direct Reading Instruments
- Air Flow Calibration and Sampling
- Ion Selective Electrode Laboratory
- Introduction to Spectrophotometry
- Instrumentation and Application of Spectrophotometry
- Colorimetric Determination of Free Silica (Quartz) Laboratory
- Introduction to Spectroscopy
- Atomic Absorption Spectrometry
- Atomic Absorption Spectrometry Laboratory
- Introduction to Chromatography
- Instrumentation and Application of Chromatography
- Gas Chromatography of Organic Solvents Laboratory
- Titrametric Determination of SO₂ Laboratory
- Colorimetric Determination of SO₂ Laboratory
- Biological Monitoring
- Related Topics

Lectures

Each lesson that is to be presented as a lecture is documented in a standardized format.

A. Lecture Cover Sheet

A cover sheet for each lecture presents the following information:

- . Lesson title
- . Lesson number and length
- . Behavioral objective
- . Scope of the lesson
- . List of visuals
- . List of exhibits
- . List of equipment needed for the lesson

B. References

After the cover sheet, there is a list of references. These references are keyed to the paragraphs within each lesson. The number in parenthesis following each paragraph is the reference number. These references are included so that the instructor, if he wishes, may further research specific instructional subject matter.

C. Additional Readings

Following the reference list, in most lessons, is another listing called "Additional Reading." This bibliography contains books and articles which are generally pertinent to the subject area covered in this lesson. These are considered as important secondary reference sources.

D. Expanded Outline (left-hand page)

On the left-hand page, beginning after the Additional Readings section, is an expanded outline. This outline indicates the information that should be emphasized and covered during the lecture. The sequence of the outline should be followed during

teaching. The expanded outline gives sufficient information to explain the brief outline which is on the right-hand page. All test questions (both self tests and course evaluator) come from the expanded outline. Additionally, there are descriptions of the visuals within the outline.

E. Brief Outline (right-hand page)

This page consists of a notes column and the outline.

1. Notes Column - times (both elapsed and projected) are indicated in this column. The elapsed time designates the time it should take the instructor to reach this point in the lecture starting from 0 at the beginning of each lecture. The elapsed time is in parentheses. The projected time designates the time it should take the instructor to reach the next major portion of the outline. A major portion of an outline is designated by a capital letter in the outline. In addition, transitional phrases connecting the major outline portions are included in the notes column. These phrases are to assist the instructor in bridging from one section of the outline to the next. Notations of what visual, exercise, table, etc., should be introduced at a given point in a lesson and miscellaneous notes to the instructor are contained also in this column.
2. Outline - this is a brief outline corresponding to the expanded outline on the facing page. Words and phrases in the brief outline key the instructor to the lesson's subject content and to the expanded outline on the left-hand page. There is sufficient space between the key words in the brief outline for the instructor to write his own additional notes when he is preparing his lecture.

F. Exercises and Problems

In some lessons, exercises and problems are included. These are given during class time. The answers to the problems are worked out with students after they have had an initial try at completing them on their own. Answers are provided in the Instructor Manual.

G. Self Tests

Self tests are included after most lessons. The Instructor Manual contains the correct answers, whereas the Student Manual does not. The students should first answer the questions, and then the instructor should review the answers, explaining fully the reasons for the correct answers. The self tests are not scored by the instructor and no records are kept of the individual student's performance. The instructor should use the information from the discussion of self tests to remove student misunderstandings or lack of understanding.

H. Copies of Visuals

Copies of visuals that are to be shown in a lecture are included at the end of that lesson documentation. These can be useful in preparing for the lecture presentation.

I. Homework

No specific homework assignments are included within the lesson documentation. However, there is a great quantity of information for the students to absorb during this one-week course. Therefore, students should be urged to review nightly all lessons covered during the day and all lessons to be presented on the following day. In particular, they should become familiar with the laboratory procedures for the following day. There is much to be accomplished in every laboratory and little time to do it. If the students are familiar with the procedure, the laboratory experiments will progress much more smoothly.

Laboratories

Each lesson that is to be presented as a laboratory is documented in standardized format consisting of four elements.

A. Laboratory Cover Sheet

A cover sheet for each laboratory presents the following information:

- . Lesson title
- . Lesson number and length
- . Behavioral objective
- . Scope of the lesson
- . List of equipment, apparatus and forms

B. Special Preparation Section

This section will follow the laboratory cover sheet, and includes specific directions that must be followed prior to actual class time. These instructions are concerned with the preparation of apparatus, facilities, chemicals and materials that are necessary for the laboratory session.

C. Laboratory Procedures

The procedures for performing each laboratory are fully documented on the left-hand page. The elapsed and projected times are indicated for some lessons with the elapsed times appearing in parentheses. The right-hand page is a blank page for notes on specifics of the laboratory to aid the individual instructor in giving an efficient lesson.

D. Figures and Forms

Equipment figures and student forms are included after the procedures. The figures are presented to aid the instructor in setting up the experimental equipment. The forms are to be used by the students during the laboratory to assist them in recording, calculating and analyzing data.

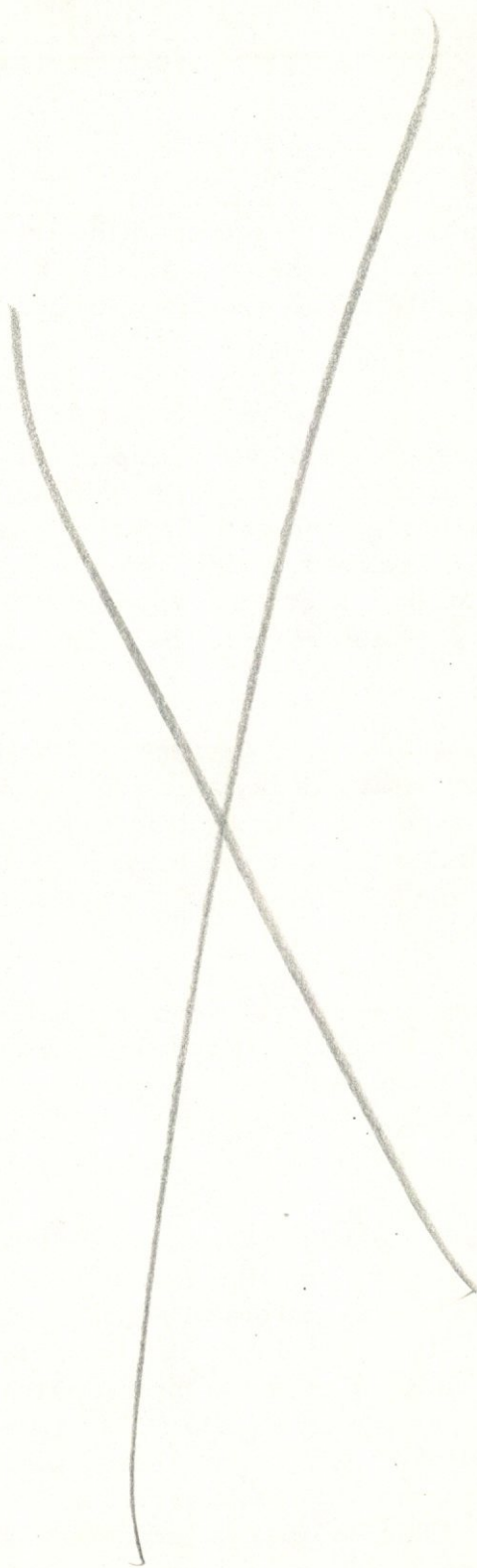
<p>LESSON TITLE</p> <p>Air Flow Calibration and Sampling</p>	<p>LESSON NUMBER</p> <p>4</p> <p>LESSON LENGTH</p> <p>1:30</p>
<p>BEHAVIORAL OBJECTIVE</p> <p>The student will be able to describe general air flow calibration techniques and sampling procedures relevant to industrial hygiene chemistry.</p>	
<p>SCOPE</p> <p>Calibration of sample volumes Calibration of sampling systems Standard mixtures Sample collection</p>	
<p>VISUALS</p> <p>4-1 through 4-24</p>	<p>EXHIBITS</p> <p>None</p>
<p>EQUIPMENT</p> <p>35 mm projector Overhead projector Screen Blackboard Chalk</p>	

REFERENCES

LESSON TITLE

Air Flow Calibration and Sampling

1. Nelson, G. D. Controlled Test Atmospheres--Principles and Techniques, Ann Arbor Science Publishers, Ann Arbor, Michigan (1971).
2. U. S. Department of Health, Education and Welfare, Public Health Service, National Institute for Occupational Safety and Health, Center for Disease Control. The Industrial Environment--Its Evaluation and Control, U. S. Government Printing Office, Washington, D. C. (1973).
3. Linch, A. L. Evaluation of Ambient Air Quality by Personnel Monitoring, The Chemical Rubber Company Press, Cleveland, Ohio (1974).
4. Threshold Limit Values for Chemical Substances and Physical Agents in the Workroom Environment with Changes for 1974, American Conference of Governmental Hygienists, Cincinnati, Ohio. (1974).
5. Katz, Morris, (Ed.) Methods for Air Sampling and Analysis, Intersociety Committee, American Public Health Association, Washington, D. C. (1972).
6. U. S. Department of Health, Education and Welfare, Public Health Service, National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Methods, U.S. Government Printing Office, Washington, D.C.(1974)
7. Air Sampling Instruments for Evaluation of Atmospheric Contaminants, 4th ed., American Conference of Governmental Industrial Hygienists, Cincinnati, Ohio. (1972).



A. Calibration of Sample Volumes

1. All physical measurements must be based upon the international primary standards for length (the meter), mass (the gram), time (the second), and temperature (degrees centigrade) - sometimes referred to as the cgs system.
2. With the exception of two personal monitor pumps, sampling equipment provided with total volume read-out is not currently available from commercial sources. Therefore, the sample volume must be derived from sampling rate and time elapsed during sample collection: $\text{Rate} \times \text{Time} = \text{Volume}$. In order to obtain sample volume within acceptable control limits ($\pm 5\%$) the flow rate meters must be calibrated accurately and frequently. (1)
3. Visual 4-1 shows the fundamental gas laws relating temperature and pressure to volume. This also must be applied to the correction of volume to standard conditions or to normal industrial hygiene conditions (760 mm. of Hg. pressure and 25°C).
4. Most volumetric calibrations are derived from the displacement of water which can be weighed with National Bureau of Standards (NBS) certified weights from a container of fixed volume such as a spirometer, aspirator bottle or a burette (soap film flowmeter). (1)
5. The spirometer - an inverted, floating water sealed bell - is the most accurate standard for flow rate and volume measurement. It is a primary standard. Commercial units are available in sizes from nine to over 1000 liters. The spirometer usually is employed for high range flowmeter (above about 2 liters/min.) calibration. The instrument operates on the principle of displacement. As air enters the inlet, the floating bell of precisely determined volume rises. The bell is supported by a chain, balanced by a counterweight and separated from the stationary tank by a liquid layer (either water or a light oil). The volume is indicated by a pointer carried on the chain moving over a scale affixed to the stationary tank. The pressure drop through this system usually is not more than 50 mm. of water. The assembly is expensive and occupies considerable space. Spirometers are available in most larger industrial hygiene laboratories and since reference to them is required on an annual basis, arrangements can be made for their use. (2)

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<div data-bbox="145 72 305 186"> Times NOTES (elapsed) projected </div>	<div data-bbox="407 103 728 155"> LESSON OUTLINE </div>
<div data-bbox="101 238 189 321"> (---) 0:20 </div> <div data-bbox="29 911 203 942"> Visual 4-1 </div> <div data-bbox="29 1388 211 1419"> Visual 4-2 </div>	<div data-bbox="414 238 1004 279"> A. Calibration of Sample Volumes </div> <div data-bbox="495 352 764 393"> 1. Cgs system </div> <div data-bbox="495 569 939 611"> 2. Rate \times Time = Volume </div> <div data-bbox="495 901 735 942"> 3. $PV = NRT$ </div> <div data-bbox="495 1129 1274 1201"> 4. Volumetric calibrations use displacement of water </div> <div data-bbox="495 1377 1106 1419"> 5. Spirometer -- use and operation </div>

6. In the absence of a spirometer, an aspirator bottle equipped with a needle valve and filled with a known volume of water can serve as a reliable substitute (Visual 4-3). When the valve or stopcock at the bottom of the aspirator bottle, sometimes referred to as a "Marriott Bottle," is opened, water flows out of the bottle by gravity and air is drawn in through the flowmeter to replace the water. The volume of air is equal to the change in water level multiplied by the cross section of the water surface. Rate is determined by dividing the air volume by the time required to displace the water volume as determined by stopwatch.
7. The soap-film flowmeter or bubble meter has become recognized as a primary standard for low flow rates (less than 2 l./min.). All that is required for this assembly is a calibrated (NBS certificate grade) burette and stabilized soap solution (Visual 4-4). A soap bubble is trapped in the inlet of the burette, and the rate of flow through the burette is then determined with a stopwatch. The soap film is formed by either immersing the open end of the burette or by raising the soap solution into the burette by means of an aspirator bulb. The principle is based on the movement of a frictionless piston through a cylinder of known volume. (3)
8. A wet test meter consists of a metal drum which houses a four-chamber inner revolving drum which is approximately two-thirds submerged in water (Visual 4-5). There are openings at the center through which air enters and openings at the periphery through which air vents from each radial chamber in turn as the inner drum rotates. The pressure of the air when reduced turns the rotating drum clockwise. The total volume is recorded by decade dials which are driven by a gear chain attached to the inner drum. The range covers 1 to 80 l./min. with an average accuracy of $\pm 0.5\%$. The volume measured is determined by the fluid level in the meter. A sight gauge indicates the water level and the meter is leveled by positioning screws as indicated by a sight bubble. Potential errors include corrosion and mechanical damage from misuse, frictional losses in the bearings and gear train and inertia which must be overcome at low flow rates (less than 1 RPM). At high flow rates (above 3 RPM) the water may surge and break the inlet or outlet water seal. (1, 2,

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-3</p>	<p>6. Aspirator bottle -- uses and operations</p>
<p>Visual 4-4</p>	<p>7. Bubble meter -- uses and operations</p>
<p>Visual 4-5</p>	<p>8. Wet test meter -- uses and operations</p>

9. Another secondary standard is the dry test meter as shown in Visual 4-6. Commercial dry test meters cover the range 5 to 5000 l./min. with an accuracy of about 1%. The meter contains a sealed outer case, two sliding valves, two bellows and a linkage system connected to the read-out dials. As air enters the meter, the bellows alternately fill and empty as controlled by the slide valve assembly. This action is linked to the gear train that drives the volume recording dials. The dry test meter has disadvantages which include mechanical drag, relatively high pressure drop (ΔP) and leakage. However, the meter can be used under relatively high pressures and volumes which compensates for these disadvantages. The alternate filling and emptying of two chambers as a basis for volume measurement also can be applied to twin cylinder piston and twin-lobed meters. Operational details for both the wet and dry test meters will be found in reference 1. Calibration against a primary standard should be carried out at least annually. (1, 2)
10. The spirometer, aspirator bottle, wet test and dry test meters are based on the transfer of a fluid volume from one position to another. The flow rate meters described in the following paragraphs are based on the principle of the exchange of potential energy for kinetic energy and/or frictional heat. A constriction within a closed tube produces an increase in fluid velocity thereby increasing the kinetic energy with a decrease in potential energy or static pressure. Flowmeters which operate on this principle can be classified in two groups and are tertiary standards:
- Rotameters are variable area meters based on a constant pressure differential by varying the cross section of the flow.
 - Orifice meters, venturi meters and flow nozzles operate on a fixed restriction which produces a differential pressure which varies with flow (variable-head meters).

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div>
<div>Visual 4-6</div>	<div>9. Dry test meter -- uses and operations</div> <div>10. Flow rate meters based on exchange of potential for kinetic energy</div> <div> <ul style="list-style-type: none"> Rotameters Orifice meters </div>

11. The rotameter by reason of its simplicity is the most widely used flow-meter design (Visual 4-7). Most pumps employed for personal monitoring are fitted with a small rotameter as an integral unit in the assembly. Regardless of sizes, all rotameters consist of a slightly tapered precision bore glass or plastic tube which contains a ball, or float which closely fits the bore at the narrower (bottom) end of the tube. When a fluid, air or water, enters the bottom of the tube, the ball rises to a height which is proportional to the volumetric flow rate of the fluid. The pressure drop across the annular area between the float and tube is just sufficient to support the float in the column of fluid at equilibrium. The flow rate is then read at the point of maximum diameter of the float. Rotameters usually have a range of 10:1 between maximum and minimum flows, and the flow rate can be extended by using heavier or lighter floats. Tubes are available for covering the range of 5 ml. to over 28,000 liters per minute. When carefully calibrated, an accuracy of better than $\pm 5\%$ can be attained. The rotameter must be in a vertical position when readings are taken. (1, 2)
12. The rotameter scale may be in millimeters or in actual flow rate units. Rotameters are easily calibrated with either a secondary standard, or even a primary standard when an aspirator bottle assembly is available. Liquids, especially oils, or greases must not be allowed to enter an air flow rotameter, otherwise the float will tend to adhere to the tube and thereby indicate erroneous flow rates. Dust also will foul the meter.
13. The calibration of orifice and venturi meters can be estimated to within $\pm 10\%$ when they are fabricated to standard dimensions from standard equations and published empirical coefficients. A variety of variations of this most useful flow rate measuring instrument have been described. The most useful and easily constructed orifice meter consists of a capillary tube inserted between the two legs of a liquid filled U-tube manometer. The restriction in the air stream presented by the capillary restriction produces a pressure drop which is proportional to the flow rate and is measured by the difference in levels of the manometer fluid in the two arms of the manometer (ΔP). The flow rate ΔP relationship is obtained experimentally and plotted on graph paper. As with the rotameter, liquids and dusts must be removed from the incoming air stream to avoid alteration in the dimensions of the orifice, or actual plugging of the capillary.

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-7, Visual 4-8</p>	<p>11. Rotameter -- uses and operations</p> <p>12. Rotameters calibrated with secondary standards</p> <p>13. Orifice meter -- uses and operations</p>

14. Porous or packed plug orifice meters have been employed for metering vapors at very low flow rates into dynamic gas mixing systems. The equipment is easily assembled from common laboratory glassware (Visual 4-9). An asbestos plug tamped into one arm of a T-bore stopcock usually serves as a porous "graded leak." Flows in the range of 0.02 to 10 ml./min. are attainable. (2)
15. To calibrate, the inlet is connected to a pressurized cylinder containing pure gas or a mixture. The cylinder valve is opened sufficiently to produce a slow stream of bubbles through the fluid in the graduated flask. The height of the liquid above bottom of the pipet determines the static pressure against the asbestos plug and, thereby, the flow through the plug to the outlet. A graduated 1-ml. pipet with the delivery tip cut off and containing a drop of water is connected to the outlet. The movement of the drop past the pipet calibration marks is timed with a stopwatch. The timing is repeated for several different liquid heights obtained by adjustment of the leveling bulb and the results plotted on graph paper (ml./min. vs. mm. liquid height). Care must be exercised to avoid liquid wetting the asbestos plug, otherwise the system must be recalibrated with a new, dry plug. (2)
16. Orifice meters are used to determine the flow rate for high volume samples. When the pressure taps are properly mounted, the calibration can be calculated. However, any orifice meter can serve as a secondary standard when calibrated with a reliable reference standard. (2)
17. All orifice type flowmeters suffer two major disadvantages:
- A relatively high ΔP is introduced into systems which can tolerate only minimal air-flow resistance
 - The orifice dimensions are susceptible to alteration by dust particles, corrosion products, mechanical damage and erosion. (2)

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div>
<div>Visual 4-9</div>	<div> <div>14. Packed plug flowmeter -- description</div> <div>15. Calibration -- packed plug flowmeter</div> <div>16. Orifice meters determine flow of high volume samples</div> <div>17. 2 disadvantages of orifice meters:</div> <div> <div>• High ΔP</div> <div>• Orifice can be damaged</div> </div> </div>

B. Calibration of Sampling Systems

1. A simple sampling method is to take a "grab sample" of the ambient industrial atmosphere in a container of known volume and bring it back to the laboratory for analysis. When ultrasensitive analytical methods such as gas chromatography are available, this technique can be effective. The sample bottle (Visual 4-10) may be evacuated in the laboratory before going into the field or the sample may be taken by flushing the atmosphere through a 250-300 ml. gas bulb fitted with end tubes (Visual 4-11) which are connected to glass stopcocks or flexible tubing and screw clamps. The aspirator bottle previously described as an alternate primary volumetric calibrating device also can be used for sample collection especially for large samples collected over an extended period of time. These techniques are widely used for collecting samples for the determination of O₂, CO₂, CO, N₂ and H₂ or combustible gases for Orsat or similar analysis. Application of this method is limited to those gases which are insoluble and non-reactive with the liquid used for displacement. "Grab sampling" cannot be recommended for general field surveying as the sample fails to represent the overall ambient atmosphere conditions with respect to time or place. (2)
2. The current trend is toward continuous sampling over finite time intervals, usually an entire 8 hour work shift to evaluate conformance with time weighted average (integrated) Threshold Limit Values (TLV's) and to compensate for the heterogeneity of the atmosphere being tested. However, for many substances that have ceiling values, short period sampling is mandatory. (2)
3. A liquid reagent is used in a midget (Visual 4-12) or microimpinger to collect vapors or dusts by impingement. This technique is based upon the collection of the contaminant from a known volume of air in a small volume of liquid absorbant (2 to 10 ml.). Although the impingers were designed for the collection of particles, application to gases and vapor has been extensively documented. (2)

Times NOTES (elapsed) projected	LESSON OUTLINE
<p>(0:20)</p> <p>Transition A. -B.)</p> <p>From a consideration of calibration of air volume to calibration of sampling systems.</p> <p>0:25</p> <p>Visual 4-10, Visual 4-11</p> <p>Visual 4-12</p>	<p>B. Calibration of Sampling Systems</p> <ol style="list-style-type: none">1. Grab samples -- apparatus, advantages, disadvantages2. Continuous sampling for 8 hours3. Midget impinger

4. A high collection efficiency is attained in a midget impinger when a chemically reactive sampling medium is used to collect a contaminant with which it reacts to produce a non-volatile derivative such as dilute aqueous NaOH to collect acidic gases (HF, HCl, SO₂, etc.). Gases and vapors can be collected satisfactorily in solvents that are not reactive if the contaminant is readily soluble and the critical ratio of the volume of air sampled to the volume of the collecting liquid is not exceeded. For acetone in water, this ratio is approximately 50 ml. of air to 20 ml. water distributed between two absorbers connected in series. Other factors include degree of contact between gas and liquid phases, duration of contact between the phases, rate of diffusion of the gas into the liquid, degree of solubility and volatility of the component sought. (3)
5. Although being rapidly superseded by the filter technique for collecting dusts, the midget impinger has been widely used for this purpose, and in some cases remains a useful tool, especially if comparisons with older published data is desired. The impinger is highly efficient for the collection of particles larger than 1 micrometer in diameter, but the efficiency drops off rapidly for smaller particles. (3)
6. Solid adsorbents, such as silica gel or activated charcoal, have established themselves as the medium of choice for collecting organic vapors. Both will adsorb the contaminant quantitatively and release the adsorbed materials completely for analysis, usually by gas chromatography. The granular adsorbent usually is packed in small glass tubes in two or more discreet columns separated by packing and sealed to prevent contamination before use. The individual sections are analyzed separately to determine collection efficiency (amount collected in the second, downstream, section divided by the total collected times 100 to convert to %). (3)
7. Federal Governmental regulations (NIOSH criteria documents and OSHA standards) have in many cases adopted sample collection with the charcoal tube which is now commercially available for personnel monitoring (Visual 4-13). The small size and light weight are ideal for breathing zone sample collection. The tube recommended by NIOSH conforms to the following:
 - . The dimensions of the glass tube is 7 cm. long x 6 mm. O.D. x 4 mm.

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-13</p>	<p>4. Sampling mediums -- reactive and not reactive</p> <p>5. Midget impinger used for particulates</p> <p>6. Solid absorbants -- silica gel and activated charcoal</p> <p>7. Charcoal tube -- NIOSH recommendation:</p> <p>. Dimensions -- 7 x 6 x 4</p>

- The adsorbent consists of two sections of 20/40 mesh-sized activated charcoal. The first 100 mg. section is separated from the second 50 mg. section by a 2 mm. section of urethane foam. A 3 mm. urethane plug retains the backup section and a glass wool plug retains the fore-section.
 - Tubes are flame sealed.
 - The charcoal quality is based on retention of the target material at a concentration two times the TLV from a 10 liter air sample drawn through at a 100 ml./min. rate. Less than 0.1 ng. of the target material should be found in the back-up section. (3)
8. Although indicator papers for the detection of air contaminants have been widely used over a period of many years, only recently has application to quantitative analysis been established. The major advantage is sampling and analysis directly in the field without recourse to laboratory facilities. The first development was the use of piston type pumps to draw a known volume of sample through a fixed area of color-forming reagent impregnated paper for a number of war gases and other highly toxic substances (phosgene, HCN, arsine, SO₂, H₂S, Hg, etc.). The estimation of lead dust collected on filter paper by treatment with a liquid reagent containing tetrahydroxyquinone to form a red stain can be carried out with a commercially available kit. (3)
9. During the past two years, a continuous tape analyzer has become available. Originally designed for general area fixed station monitoring for toluene-di-isocyanate (TDI), the size has been reduced to personnel monitor size, and tapes for a number of gases (H₂S, phosgene, NO₂ and chlorine) have been announced. The instrument can be coupled with a read-out system which records on concentration vs. time strip charts and can integrate the total 8 hour exposure to determine TLV compliance.

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
	<ul style="list-style-type: none"> . Absorbent -- 100 and 50 mg. . Flame sealed 8. Reagent impregnated paper -- uses and application 9. Continuous tape analyzer -- application

10. The most direct, expedient and economical approach to evaluation of the dust hazard problem is filtration of a known volume of the contaminated atmosphere and either count the particles by microscopic examination (Visual 4-15 and 4-16), weigh the dust collected (Visual 4-17), or analyze the sample for specific components such as inorganic lead. Filters are used to count or size particles as well as for performing gravimetric or chemical analysis (Visual 4-18). Uniformly high efficiencies - great than 99% - for all sizes of particles in the same state as they existed in the original atmosphere can be attained routinely. Particles smaller than the absolute size of the surface pores are collected due to electrostatic effects and impaction on the walls of the pore passages formed by the interlocking cell layers. Filter membranes fabricated from polyvinyl chloride (PVC), silver, and fiberglass also are commercially available. (3)
11. Airflow calibration should be carried out by attaching a flow rate meter (rotameter) or spirometer which introduces a minimum ΔP into the system to the inlet connection and a vacuum source to the outlet of the filter holder. A water manometer connected to a "T" tube inserted between the filter outlet and vacuum supply should be included. Leakage should be eliminated by a tight joint between the filter membrane and edges of the retaining rings, and by tight jointed sections which are wrapped with tape. Leakage can be detected by clamping off the inlet tube and outlet tube in the downstream side of the water manometer and noting leakage as mm./min. (3)
12. If the objective is to obtain information relative to the health hazard resulting from the inhalation of airborne particles, the sampling technique must provide data of the contaminant concentration within the size range which reaches the lower lung. The human respiratory tract rejects most particles larger than 5 micrometers (unit density sphere). Miniature cyclone separators have been designed to closely approximate human respiratory particle collection and are installed ahead of the filter as a pre-collector for the non-respirable particles (Visual 4-19).

<div>Times</div> <div>NOTES (elapsed)</div> <div>projected</div>	LESSON OUTLINE
<div>Visual 4-15,</div> <div>Visual 4-16,</div> <div>Visual 4-17,</div> <div>Visual 4-18</div>	<div>10. Dust collection on filters</div>
<div>Visual 4-19</div>	<div>11. Air flow calibration of filters and eliminating leakage</div> <div>12. Respirable particulates -- cyclones</div>

13. A cascade impactor grades an aerosol on the basis of its airborne particle size (Visual 4-20). By passing the dust laden sample through a series of slots, orifices or nozzles of decreasingly smaller size, a spectrum of the range of particle sizes is obtained by impingement on microscope slides or metal plates. Both the slot type with microscope slide collector plates and the orifice plate (multi radial holes in circular discs separated by gaskets) in sizes suitable for personnel monitoring are commercially available. Analysis of the collected dust can be based on gravimetric, counting or chemical analysis procedures. (3)
14. Combinations of any of these collection devices can be adapted to solving multicomponent system problems. For example, for the evaluation of the potential lead hazard encountered in the manufacture of tetra-ethyl lead (TEL), a personnel monitor has been assembled with a pre-filter to collect inorganic lead dust followed by a microimpinger containing aqueous iodine monochloride reagent to collect the TEL vapor. (3)
15. Charcoal traps following liquid reagents are used to protect the pump and flowmeter from physical and corrosion gas damage. (3)
16. Flowmeters or total volume readout meters are used to measure the sample size. (3)
17. A pump or aspirator is used to draw the air sample through the assembly. A relatively constant source of vacuum (suction) is required. The most practical source is provided by an electrically powered pump-piston, diaphragm or rotary - for prolonged periods of sampling. Miniature piston and diaphragm pumps are commercially available for personnel monitoring. Several models are suitable for operation in fire and explosion hazard areas (coal mines). (3)
18. As a general consideration for all sampling systems, the sampled air must follow the shortest possible route to reach the collection medium, and the overall air flow resistance must be held to the absolute minimum. Collection efficiency for the selected flow rate must be known. For collection in a liquid reagent or on a solid adsorbent two collectors may be connected in series and the contents analyzed separately. If more than 10% of the total sample escapes into the second collector, the system is not satisfactory and the flow rate or equipment design must be revised to provide 90% or better performance.

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-20</p>	<p>13. Cascade impactor -- operation and use</p> <p>14. Combination collection devices</p> <p>15. Charcoal traps</p> <p>16. Flowmeters</p> <p>17. Pumps -- uses</p> <p>18. General consideration -- short reach to collection and small ΔP</p>

19. The sample size (volume) must be large enough to provide sufficient material - dust or vapor - to detect the presence, or confirm the absence, of the component sought (benzene or lead dust in air) but not so large that the collection capacity of the collection medium is exceeded. If the sampling rate, or total volume passed through the collector exceeds the critical absorption or adsorption limit, breakthrough can occur and a negative error will be introduced into the final analysis. In dust collection by filtration, excess sample will produce "blinding" of the filter with greatly increased air flow resistance, and frequently, redispersion of the dust.
20. Volumetric calibration (flow rate and sample volume) of the sample collection assembly must be made with all components in place exactly as used in the field. Since rotameters introduce negligible pressure drop into the system, calibration can be carried out quickly by connecting a calibrated rotameter to the sample inlet. Overall system calibration (collection and analysis) with known concentration of contaminants in air also must be carried out with all components in place.

C. Standard Mixtures

1. All evaluation systems require periodic calibration to assure integrity of the overall sampling and analysis operation. For this essential procedure, standard contaminant in air mixtures - known concentrations of the component sought in air - must be available. The background matrix should conform as closely as possible to the matrix which exists in the environment sampled. A bulk sample of the material as it exists in the workplace should be collected and analyzed before the collected air samples are analyzed. Frequently the background matrix will have a significant effect on the analytical results. Either positive or negative interference can be expected. (15)

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>(0:45) Transition B. -C.) from calibration instrumentation to the preparation and use of standards. 0:25</p>	<p>19. Sample size -- enough not too much</p> <p>20. Volumetric calibration with all components in place</p> <p>C. Standard Mixtures</p>

2. For example, a bulk sample of a thinning solvent used in a varnish manufacturing operation might be run through the gas chromatograph to locate the peaks which represent minor components found in commercial grade solvents and to locate the major peaks before the vapors collected on charcoal are analyzed. Alpha-quartz in siliceous dust provides another example of the obscuring effects created by the matrix (silicates) in infrared, x-ray diffraction, and colorimetric methods of analysis. In this case, prudence would indicate analysis by at least two of the available methods for confirmation. (5)
3. The simplest and most direct procedure for the preparation of a known mixture of a hazardous gas or vapor (such as benzene vapor in air) is to add a calculated volume of vapor or gas or readily vaporizable liquid by syringe or pipette into a glass or metal vessel of known volume. (2)
4. Calculations are based upon close agreement with the Perfect Gas Law:

$$PV = NRT \quad \text{where -}$$

P = pressure at 760 mm.

V = volume at standard conditions

N = number of gram moles of gas

R = universal gas law constant (at 760 mm. Hg + 24.47 liters)

T = temperature in absolute ($^{\circ}\text{C} + 273^{\circ}$) (2)

5. Dilute gas concentrations may be prepared as a simple ratio of the volume of test gas to the total volume of the mixture.

$$\text{P.P.M.} = 10^6 \frac{v}{V} \quad \text{where -}$$

P.P.M. = parts per million by volume

v = volume of the test gas in liters

V = volume of the mixture in liters (2)

6. For volatile liquids the calculation is based upon the ratio of moles of liquid to moles of gas mixture:

$$\text{P.P.M.} = \frac{10^6 w / \text{M.W.}}{V / \bar{V}} \quad \text{where -}$$

w = weight of liquid in grams

M.W. = gram molecular weight of liquid

V = gram molecular volume of mixture under ambient conditions in liters

$$\text{moles of liquid} = \frac{W}{\text{M.W.}} \quad (2)$$

7. Even when adequate mixing is attained, the disadvantages associated with rigid containers and static systems often outweigh the advantages. Unless two or more such rigid containers are connected in series, the progressive dilution by air drawn in to replace the volume withdrawn becomes a source of significant error. For example, when 5% of the mixture is removed, the concentration is reduced 4%, or when 25% is withdrawn the final concentration of the standard mixture drops 22%. Adsorption or decomposition on the walls of the containing vessel may occur at such a rate as to introduce errors of even greater magnitude. Losses in excess of 50% are common. The half-life of the test gas or vapor should be determined in all static systems. For example, 50% of benzol vapor in the 100-200 P.P.M. range disappeared from a calibration mixture prepared in a borosilicate carboy within 30 minutes after preparation. (2, 5)

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div> <div> <p>Write on blackboard.</p> <p>6. Volatile liquids -- P. P. M. = $\frac{10^6 w/M: / W.}{V/\bar{V}}$</p> <p>7. Disadvantages -- progressive dilution by air, adsorption and decomposition in container walls</p> </div>
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TITLE

Air Flow Calibration and Sampling

LESSON NUMBER

4

8. Notable exceptions to these disadvantages are the preparation of standard carbon monoxide and low molecular weight hydrocarbons such as methane in pressurized steel cylinders. Reliable standards which are stable over long periods of time are commercially available. However, the stated concentration should be confirmed. (2)
9. In all static mixture preparations, when air dilutions of solvents or other combustible substances is made, care must be exercised to avoid explosive concentration ranges during as well as after dilution. This factor is especially critical in the preparation of pressurized mixtures in steel cylinders.
10. Non-elastic flexible bags fabricated from polyvinylchloride (PVC), polyfluoroethylene (PTFE), polyvinylfluoride, polyethylene, etc., eliminate the dilution problem when an aliquot is removed for analysis. Long term stability is generally good only with relatively inert gases and vapors such as halogenated solvents and hydrocarbons. Bags are fabricated from sheets by thermal sealing. Mylar bags combine both strength and inertness. Volatile contaminants can be removed by heating in an oven for several days. A valve of the type used for automobile tires, or a rigid plastic tube fitted with a serum cap can be sealed into one corner to serve as an inlet. Bags designed for this purpose are available commercially. (2)
11. Since the bags are non-elastic, the volume contained at full capacity is reproducible and, therefore, they can be used as a container of known volume for field calibration. The reference sample either as a gas or liquid can be injected with a syringe while air is being pumped into or drawn into the bag. The bag should be filled and emptied several times to flush out impurities. Mixing is accomplished by kneading the bag with the hands. The bags also are a convenient, reliable means for collecting samples for laboratory analysis as they can be transported easily in the deflated condition. These bags also can be inserted into rigid containers as a bladder which can be filled by evacuating the rigid container. A 25-50 mm. ΔP is sufficient to completely fill or expel the air in the flexible bag. (1)

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div>
	<div data-bbox="486 217 1303 300">8. Exception -- CO and low molecular weight hydrocarbons in pressurized steel cylinders</div> <div data-bbox="486 476 997 528">9. Explosive concentrations</div> <div data-bbox="486 735 1288 818">10. Non-elastic flexible bags -- advantages and description</div> <div data-bbox="486 1181 1259 1263">11. Use of bags -- by themselves and in rigid containers</div>

12. However, plastic bags also have disadvantages. In addition to adsorption or decomposition of the contaminant on the container walls, diffusion through the plastic film introduces errors over prolonged storage periods. Negative error results from outward diffusion and positive error from inward diffusion. This deficiency can be corrected to a great extent by aluminizing the plastic film. (5)
13. Diffusion of gases and vapors during storage in non-rigid bags can be utilized as a source of constant rate generation of trace quantities of calibrating gases. Due to a relatively high temperature coefficient of the diffusion rate (rate of change with temperature change), these devices require temperature control within close tolerances. A liquified gas or volatile liquid sealed in a section of PTFE tubing by means of glass balls when placed in a metered air stream at constant temperature serves as a dynamic calibration standard. This technique serves as a primary standard as the diffusion rate is determined by weight loss with time. The National Bureau of Standards now certifies several permeation tubes (SO₂ for example). After a new tube has been prepared, several days or weeks are required to attain a steady permeation rate at constant temperature. Volumetric calibration can be determined in less than an hour with the Gilmont Warburg Compensated syringe monometer which measures the evolved gas to 0.2 microliters. (2)
14. For higher rates of vapor generation, glass capillary diffusion generators (Visual 4-21) have been developed and are commercially available. Calibrated permeation tubes also are available from this source. Gases and vapors will diffuse through tubes at a uniform rate if the temperature, concentration gradients and tube geometry remain constant. The concentration is controlled by varying either the temperature or the flow of the primary or secondary diluent gas. The ratio of the tube area to length should be maintained at less than 0.3 for best results. Diluent gas (air) flow rate should not exceed 1 l./min. Vapor concentrations in the range 0.1 - 100 P.P.M. are easily maintained over long periods. (2)

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-21</p>	<p>12. Bags -- disadvantage -- adsorption, decomposition, diffusion</p> <p>13. Permeation tubes -- benefits, uses</p> <p>14. Diffusion generators -- operation, uses</p>

15. The evaporation technique provides one of the most generally used dynamic dilution procedures (Visual 4-22). The diluent gas (clean air) is passed either through or over the liquid to be vaporized. The gas mixture then is passed through a constant temperature bath held at a lower temperature than the vaporizer to ensure vapor saturation by condensation of excess vapor. The mixture is finally diluted down to the desired concentration with a second clean air supply. (2)
16. Constant rate motor driven syringes have been used to inject either gases or readily vaporizable liquids into controlled air streams to produce known concentrations dynamically. Injection systems for the range 0.5 - 500 ml. /min. are commercially available and are capable of a high degree of precision when good quality, calibrated syringes are employed. Variable speed drives provide flexibility in the choice of delivery rates. From the known gear ratio, drive screw pitch and plunger diameter, the feed rate can be calculated with an accuracy and reproducibility in the parts per thousand range. (Visual 4-23) (2)
17. As indicated under calibration of flow rates, porous plug orifice meters have been used for metering vapors at very low flow rates into dynamic gas mixing systems (Visual 4-24). The equipment is easily assembled from common laboratory glassware. An asbestos fiber (acid washed) plug is packed into one arm of a T-born glass stopcock to serve as a "graded leak." Gas flows in the range of 0.02 to 10 ml. /min. are delivered by this technique. The ΔP which controls the flow rate across the asbestos plug is controlled by a liquid filled leveling bulb connected to the waste vent arm. The height of the liquid head determines the static pressure required to deliver the desired flow rate calibrated. The leveling bulb is connected to a "T" in the diluted gas delivery manifold to compensate for the back-pressure (ΔP) developed by the system into which the flow is delivered. This maintains a constant ΔP across the asbestos plug. (2)
18. Gases such as high purity carbon monoxide, methane and hydrogen, or concentrated mixtures in air are commercially available and can be used to deliver calibration gases into dynamic systems by "step-down" dilution. Only a source of purified air, rotameters or soap film flowmeters and connecting tubing are required. An efficient low volume mixing chamber also should be included to provide homogeneity of the test gas. Such a set-up has been employed for recalibration of CO detector tubes for 4 hour monitoring. (2, 3)

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>Visual 4-22</p>	<p>15. Evaporation technique</p>
<p>Visual 4-23</p>	<p>16. Motor driven syringes in moving air streams at constant rate</p>
<p>Visual 4-24</p>	<p>17. Asbestos plug orifice system</p>
	<p>18. Pressurized gases</p>

19. Preparation of a known concentration of an aerosol mixture is much more complex and difficult than a gas or vapor standard mixture. A major consideration is the size distribution of the particles. Usually a log normal distribution will be found. If the geometric standard deviation is less than 1.1, the particles can be said to be homogeneous or monodisperse. A great variety of particle shapes (spherical, irregular, plates, spiked, rod shaped, fibrous, etc.) as well as a variety of sizes and agglomerates will be encountered. Also electrostatic charges which often are humidity dependent will introduce another variable. (2)
20. One of the most convenient and most widely used techniques provides redispersion of dry powder such as road dust, fly ash, silicates, silica, mineral dust, coal dust, etc. These dusts tend to agglomerate; therefore, the degree of packing of the powder must be controlled and reproducible. A simple method is provided by shaking the powder on a screen into the moving air stream, and feeding the dust cloud into a relatively large chamber which serves to equalize any rapid fluctuations in the dust feed. (2)
21. Compressed air nebulizers are a convenient source of liquid aerosols. The liquid stream is drawn through a capillary tube and atomized into fine droplets by a high velocity air jet. Vaporization and condensation also provides a convenient source of liquid and solid aerosols from low vapor pressure compounds such as stearic acid, lubricating oils, etc. The spinning disc generator has been widely used. This is based upon feeding a liquid continuously onto the center of a high speed rotating disc (60,000 rpm). (2)

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
	<p>19. Known concentrations of aerosol -- difficulties</p> <p>20. Redispersión of dry powder</p> <p>21. Compressed air nebulizers</p>

22. The concentration of calibrating gas in a standard air mixture should be confirmed by independent analysis, e.g., analysis of a CO /air mixture by gas chromatography. A number of tentative consensus standard methods are available for this purpose. Those standard air mixtures derived from gravimetrically calibrated sources such as the permeation tube can be considered primary standards when equilibrium has been reached and "clean" air is used for dilution. The half-life (time required for 50% loss of the calibrating gas) of the standard should be determined also. In most cases, in the dynamic systems this factor can be ignored. However, the investigator should demonstrate that the system reached equilibrium before use as a calibrating mixture. At high dilutions this factor becomes critical for the reactive vapors (phosgene, toluene-di-isocyanate, etc.). In static systems, half-life must be determined as in some cases (benzene vapor in borosilicate glass carboys) loss of the calibrating gas is rapid. Purified dry air must be used in most operations to remove interferences and prevent decomposition (phosgene with water vapor). (5, 6)

D. Sample Collection

1. No analytical result regardless of the accuracy and precision of the method can be any better than the quality of the sample submitted for analysis. Therefore, the primary concern of the industrial hygienist must be directed to the collection of representative samples, maintenance of the integrity of the sample until analyzed, and the homogeneity and accuracy of standard air mixtures employed to calibrate both the collection and analytical systems. The objectives of the air sampling and analysis program must be clearly delineated before sample collection is initiated. The frequency, extent (area to be included in the study), type, duration, and techniques required must be established before sample collection is started.
2. Grab sampling (1-10 min.) is appropriate for scoping surveys (preliminary investigations) and for detection of peak excursions (ceiling TLV's). Direct reading colorimetric equipment, and other portable direct reading instruments are readily available from a number of commercial sources. Collection equipment for laboratory analysis includes evacuated bottles, sampling bulbs, plastic bags, large syringes and impingers containing liquid reagents. Since the industrial environment is so highly variable, this technique is not suitable for 8 hour time weighted average air analysis unless a large number of discrete samples are collected and the results resolved statistically. (3)

<p>Times NOTES (elapsed) projected</p>	<p>LESSON OUTLINE</p>
<p>(1:10) Transition C. -D.) From calibrating to sampling. 0:10</p>	<p>22. Precautions -- independent analysis, half-life</p> <p>D. Sample Collection</p> <p>1. General consideration</p> <p>2. Grab sampling -- uses, disadvantages</p>

3. To determine compliance with 8 hour time weighted average TLV's and OSHA regulations for hazardous substances such as lead dust, continuous sampling or integrated sampling with either work area, or better, personal monitors is required. This approach averages out the concentration peaks and valleys encountered by the worker during his shift assignments. However, unless a continuous sampler such as impregnated paper tape is available, excursions above the TLV are not detected. Therefore, 8-hour samplers are not suitable for ceiling TLV evaluation. With few exceptions, time weighted averages require concentration of the contaminant from a relatively large volume of air in or on a collecting medium. The sampling is selected to provide sufficient quantity of the pollutant for reliable analysis and, frequently, dictates the choice of collection equipment. The sampling rate, in turn, must be within the range of acceptable collection efficiency (above 90%). If the laboratory technician does not collect the samples, these factors must be resolved in detail with the personnel assigned to sample collection and the industrial hygiene surveys. (2, 3)
4. A well designed quality control program must be established for sampling and analysis as a prerequisite to an industrial hygiene study or program for exposure control. Replicate sampling, bulk samples and reference samples as well as volumetric and analytical calibration must be established. The bulk and reference samples will provide information relative to interferences. (2)
5. Collection of gases and vapors from an air sample in a liquid reagent is often made with either the midget or the microimpinger. Some considerations are:
 - Sampling rate - the midget impinger efficiency peaks at 1.0 l./min. and the microimpinger operates most efficiently in the range 400-600 ml./min.
 - Physical absorption occurs as a solution in a solvent.
 - Chemical reaction occurs with the liquid medium to produce a non-volatile derivative.

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div> <div> <p>3. Integrated sampling -- uses, disadvantages</p> <p>4. Quality control</p> <p>5. Sampling gases and vapors with liquid reagent</p> <ul style="list-style-type: none"> • Sampling rate • Physical absorption • Chemical reaction </div>
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6. The impinger originally was designed for dust collection by impingement in a liquid trapping agent. For this purpose, the sampling rate must be controlled within narrow limits (1.0 ± 0.1 l./min.) for reproducible results. Although this method has been superseded by filter collection, several standards are based on this method. The spill-proof modification of the microimpinger has been employed successfully for personnel monitoring. (3)
7. Many pollutants - gases, vapors and aerosols - can be collected with a high degree of efficiency (95 + %) in liquid reagents. If the collection involves simple solubility, the air sample volume is limited to the escape concentration; i. e., - evaporation from the scrubber solution becomes the controlling factor. When chemical reaction occurs as in the collection of toluene-di-isocyanate (TDI) in an aqueous solution of acetic and hydrochloric acids, the sample size is sometimes limited by the stoichiometry of the reaction, i. e., capacity to hydrolyze the isocyanate to the parent diamine. The collected material may be estimated directly in the field with commercially available kits, or brought back to the laboratory for quantitative analysis. "Pocket" or "suitcase laboratories" are offered for TDI, tetra-alkyl leads, inorganic lead dust, perfluoroisobutylene (PFIB), Cr, Cu, CN, H₂S, Fe, Mn, Ni, -NH₃, -NO₃, -HO₂, pherrol, -PO₄, etc. (3)
8. Although current technology favors activated charcoal as an adsorbent base, silica gel possesses certain distinct advantages. While silica gel does not have as great an affinity for organic molecules as does charcoal, it does have distinct properties which recommended its use as an analytical tool in that adsorbed materials can be removed easily, consistently and quantitatively. Silica gel is the preferred carrier for direct indicating colorimetric applications. (3)
9. Most sampling procedures currently written into governmental standards and recommended procedures (OSHA and NIOSH) specify collection of organic vapors on activated charcoal. This solid adsorbent is non-polar and consequently is capable of adsorbing organic vapors in preference to atmosphere moisture. One of the major advantages involves the ease with which the sample can be transferred to a gas chromatograph by either thermal desorption or by displacement with carbon bisulfide. (3)

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div>
	<div>6. Impinger</div> <div>7. Use of reagents in collecting gases and vapors</div> <div>8. Silica gel -- advantages</div> <div>9. Activated charcoal -- advantages</div>

10. Since the human respiratory system rejects most particles larger than 5 micrometers aerodynamic diameter (unit density sphere) the aerosol collection device should possess similar discrimination. Miniature cyclone separators suitable for use with personnel monitors such as the certified coal dust sampler designed for use in underground mines in accordance with the Coal Mine Safety Act are commercially available. These units are attached to the assembly ahead of the total collection filter. The applicable standards have been delineated by the ACGIH's TLV committee. (2, 3)
11. In the past dust was collected by impingement in a trapping liquid (water or alcohol) in the Greenburg-Smith, or later, the midget impinger. However, this procedure is not quantitative over the entire particle size range and has been superseded by filter collection. The impinger dilutes highly efficient recovery of particles larger than 1 micrometer, but the efficiency drops off rapidly for smaller sizes. However, size selection of dusts by cascade impactors which do not employ a trapping liquid are an important phase of aerosol sampling. (7)
12. Modern technology almost exclusively employs filters for the isolation of both solid and liquid particulates. Filter media in common use include destructable cellulose acetate membrane filters with controlled pore size (most popular is the 0.8 micrometer pore size) and polyvinyl chloride (PVC) membrane filters, and the non-destructable silver membrane and fiberglass bases. The choice of filter medium, to a great extent, is predicated on the analytical method to be applied to the collected sample. Pre-weighted membrane filters for the determination of total dust gravimetrically are commercially available from several sources. Most methods for analysis of specific components, such as lead, in the dust sample require total destruction of the filter matrix by wet or dry ashing. In this case either the cellulose acetate or PVC membranes are appropriate. Membrane filters are recommended for visual particle counting as the dust does not migrate below the surface into the pores to any great extent, and the cellulose acetate membrane can be "cleared" by dissolving in a mixture dimethyl phthalate and diethyl oxalate for visual counting of asbestos fibers. (2, 3)

<div>Times NOTES (elapsed) projected</div>	<div>LESSON OUTLINE</div> <div> <p>10. Sampling aerosols -- respirable by cyclone</p> <p>11. Sampling dusts -- filters and cascade impactors</p> <p>12. Filters -- uses, types</p> </div>
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13. Personal monitoring is intended to determine the inhaled dose of an airborne toxic material, or by the continuous collection of samples in the breathing zone, or other appropriate exposed body area, over a finite period of time. A personal monitor is a self-powered device worn by the monitored individual to collect a representative sample for laboratory analysis. Most NIOSH criteria documents recommend personal monitoring, and future OSHA regulations will probably require evaluation of occupational exposure by personal monitoring as is already required in the coal industry.

E. Self Test

1. Test instructions and review of questions are presented.

<div>Times</div> <div>NOTES (elapsed)</div> <div>projected</div>	LESSON OUTLINE
<div>(1:20)</div> <div>Self Test</div> <div>0:10</div> <div>(1:30)</div>	<div>13. Personnel monitoring -- description</div> <div>E. Self Test</div> <div>1. Instructions and review</div>

LESSON TITLE

Air Flow Calibration and Sampling

LESSON NUMBER

4

1. Fill in the blanks:

- a. The spirometer is an inverted, floating water sealed bell and bell and is the most accurate standard for flow rate and volume measurement.
- b. The soap-film flowmeter or bubble meter has become recognized as a primary standard for low flow rates (less than 2l. /min.).

2. Match the following devices with their description:

Wet test meter ————— A secondary standard which measures volume by the fluid level in the meter by a sight guage.

Aspirator bottle ————— A primary standard.

Rotameter ————— A tertiary standard.

A secondary standard which measures volume by the fluid level in the meter and is feasible for only low flow rates.

3. Compare grab sampling and continuous (or integrated) sampling: (briefly)

See D. 2 - D. 3

LESSON TITLE

Air Flow Calibration and Sampling

LESSON NUMBER

4

4. What are the basic differences between static and dynamic systems for preparing standard mixtures?

See C

5. What device is used in sampling systems for respirable dust?
- a. Silica gel
 - ☒ b. Cyclones
 - c. Charcoal tubes
 - d. Midget impingers

VISUALS, TABLES, FIGURES AND EXHIBITS

CORRECTION FOR TEMPERATURE AND PRESSURE

$$PV = NRT$$

P = PRESSURE AT 760 MM.

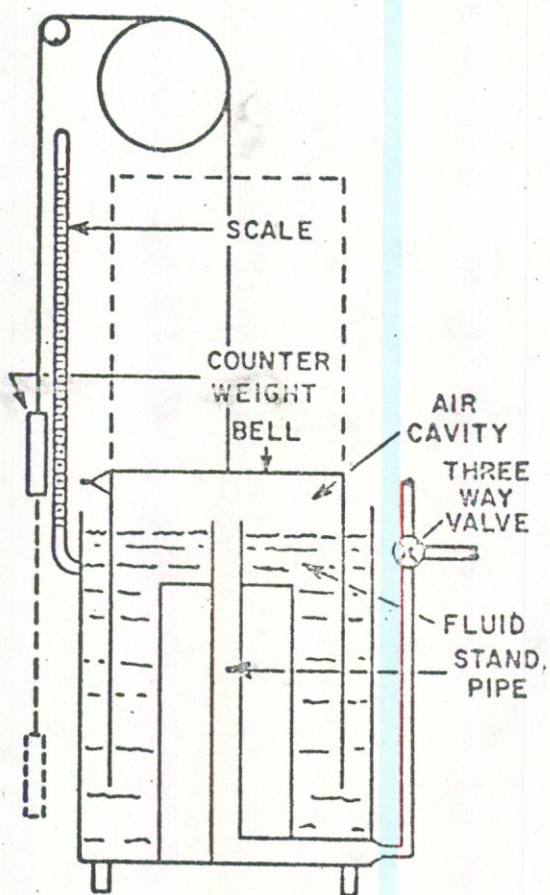
V = VOLUME OF STANDARD CONDITIONS

N = NUMBER MOLES OF GAS

R = GAS LAW CONSTANT

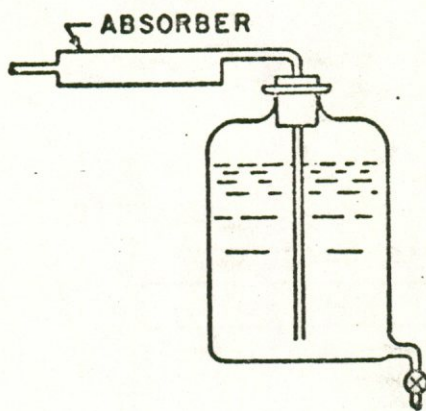
T = TEMPERATURE IN ° ABSOLUTE

Visual 4 - 1



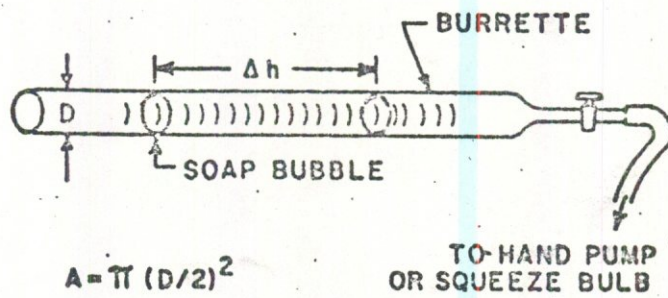
Schematic Drawing of a Spirometer

Visual 4 - 2



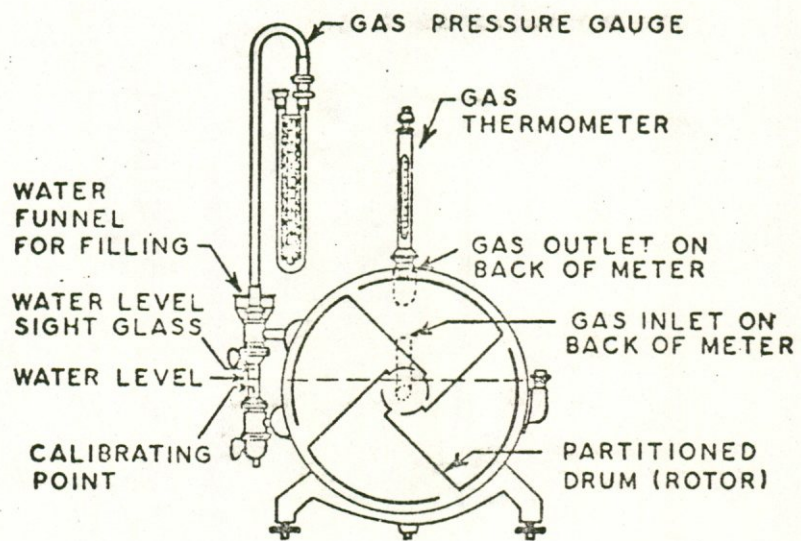
Aspirator Bottle

Visual 4 - 3



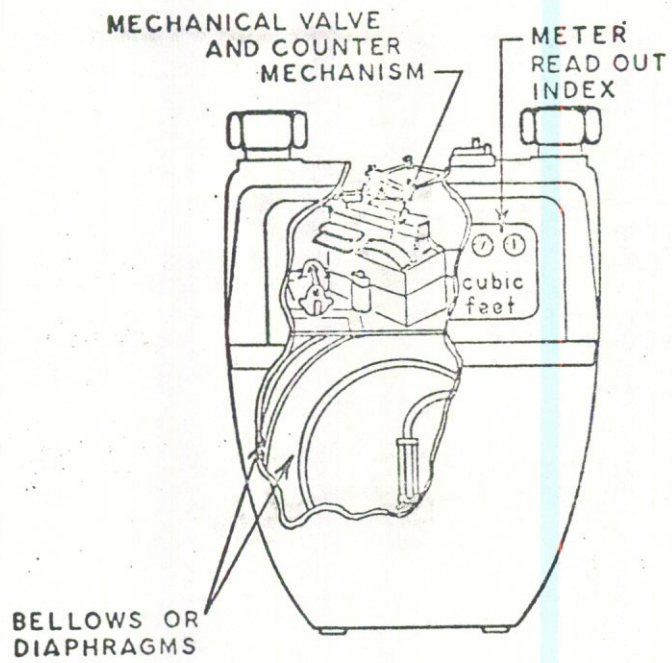
Bubble Meter

Visual 4 - 4



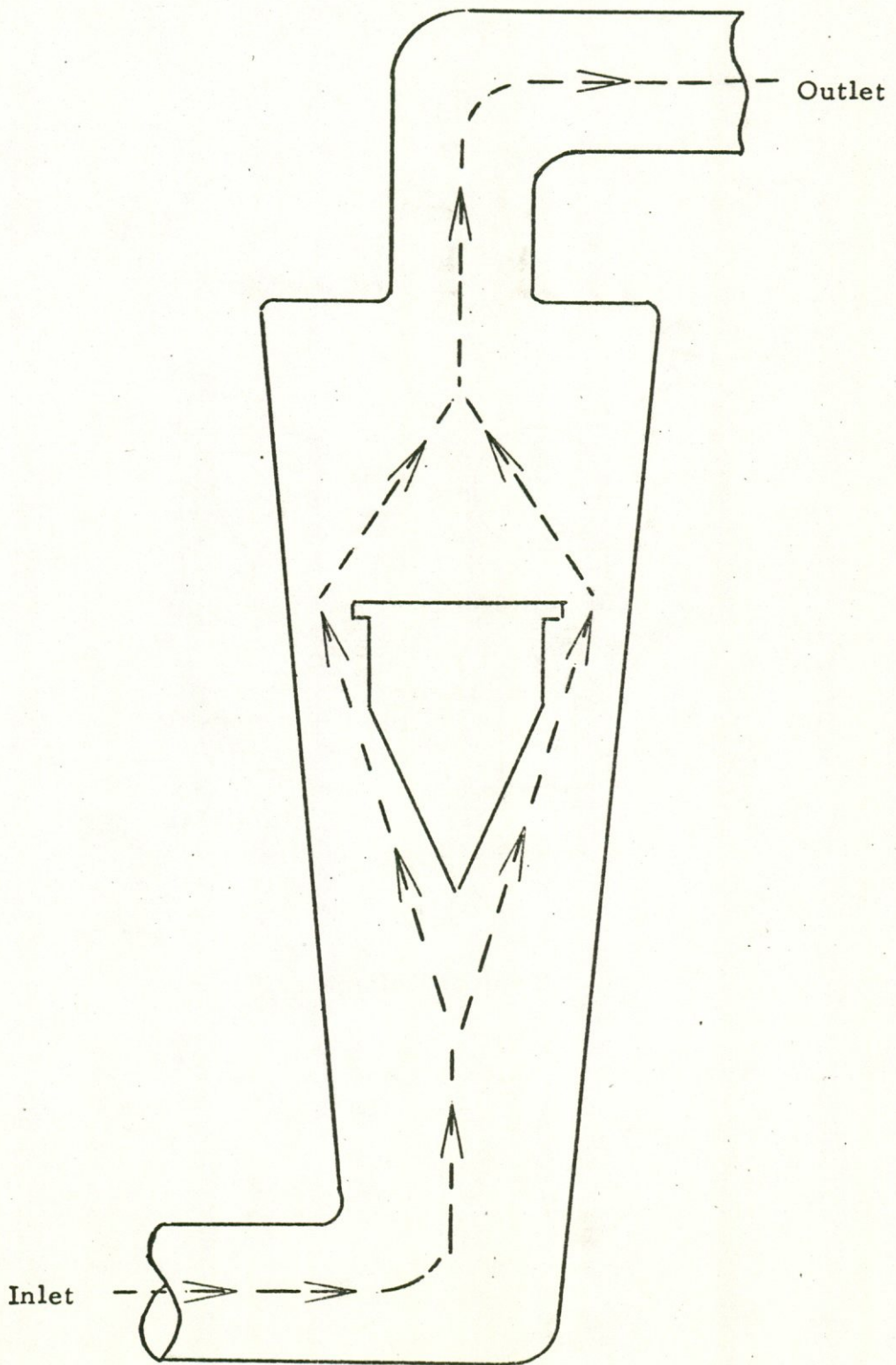
Wet Test Meter

Visual 4 - 5



Dry Gas Meter

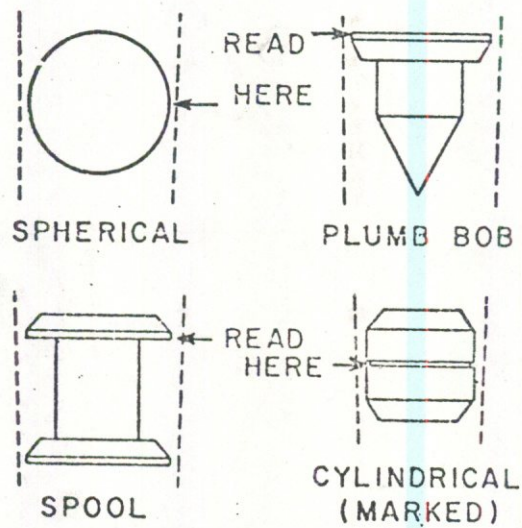
Visual 4 - 6



Rotameter

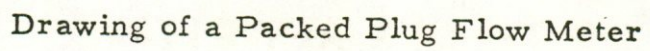
4-57

Visual 4 - 7



Typical Rotameter Floats

Visual 4 - 8

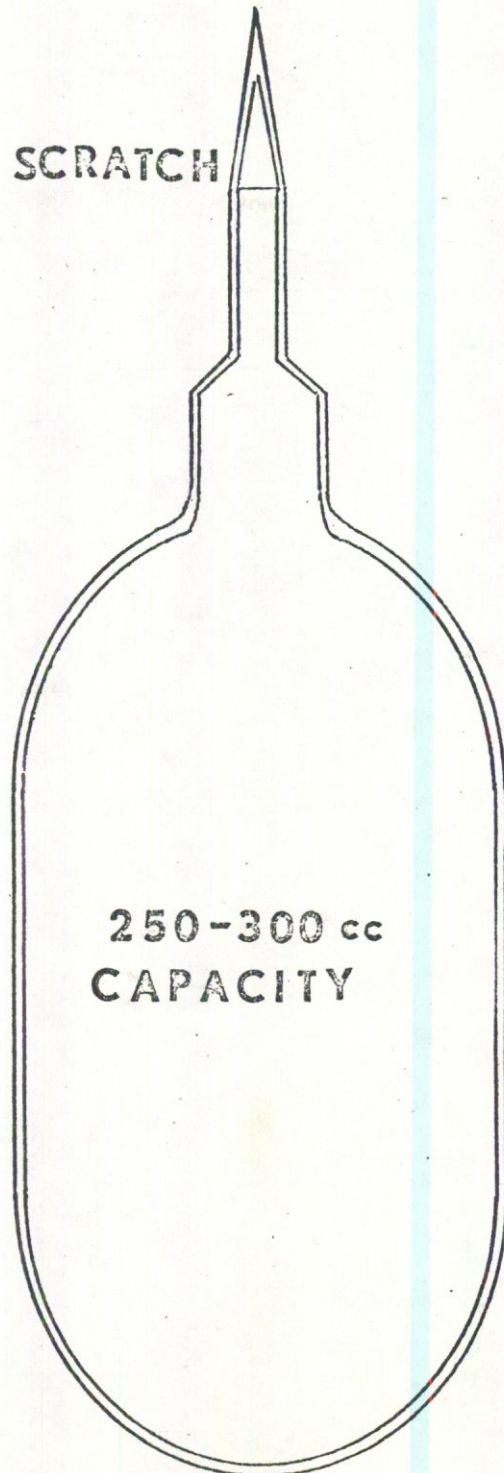


4-29

BREAKING SCRATCH



WAX

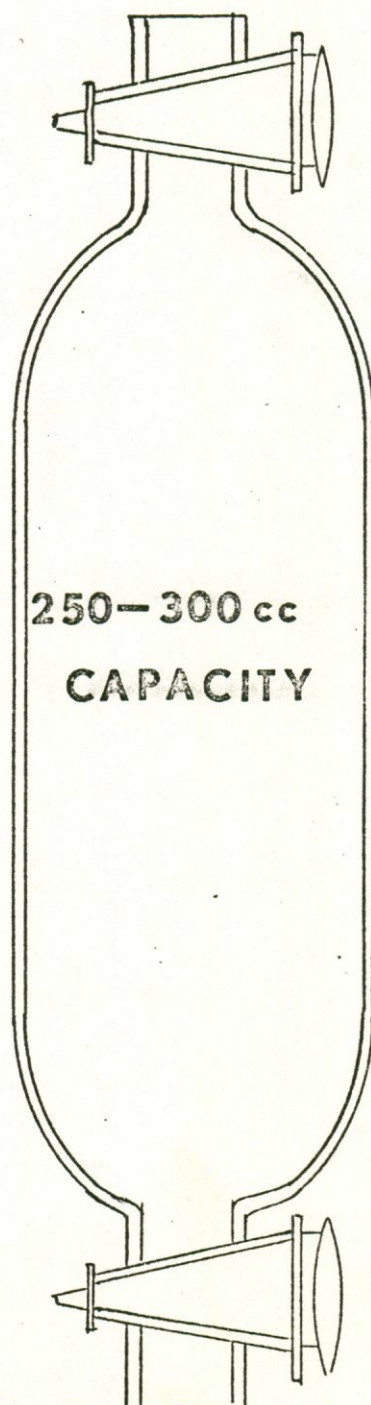


**250-300 cc
CAPACITY**

**GRAB SAMPLE BOTTLE -A
EVACUATED FLASK**

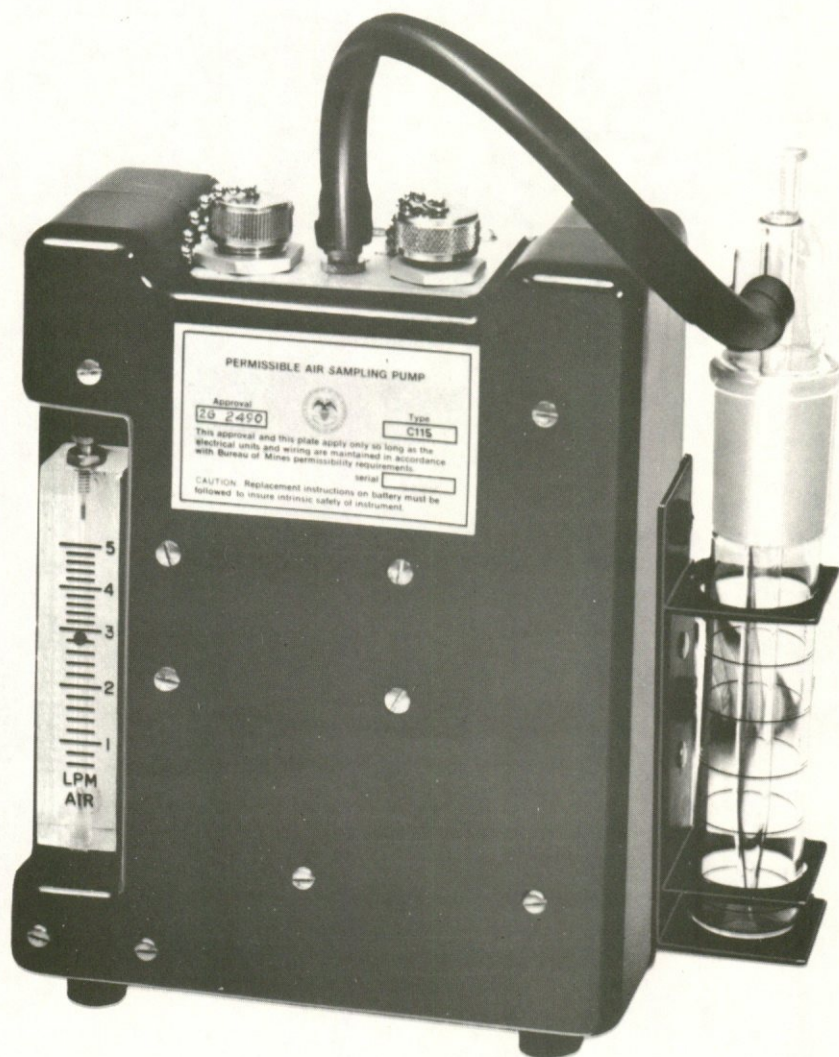
4-60

Visual 4 - 10



GRAB SAMPLE BOTTLE-B

GAS OR LIQUID DISPLACEMENT TYPE

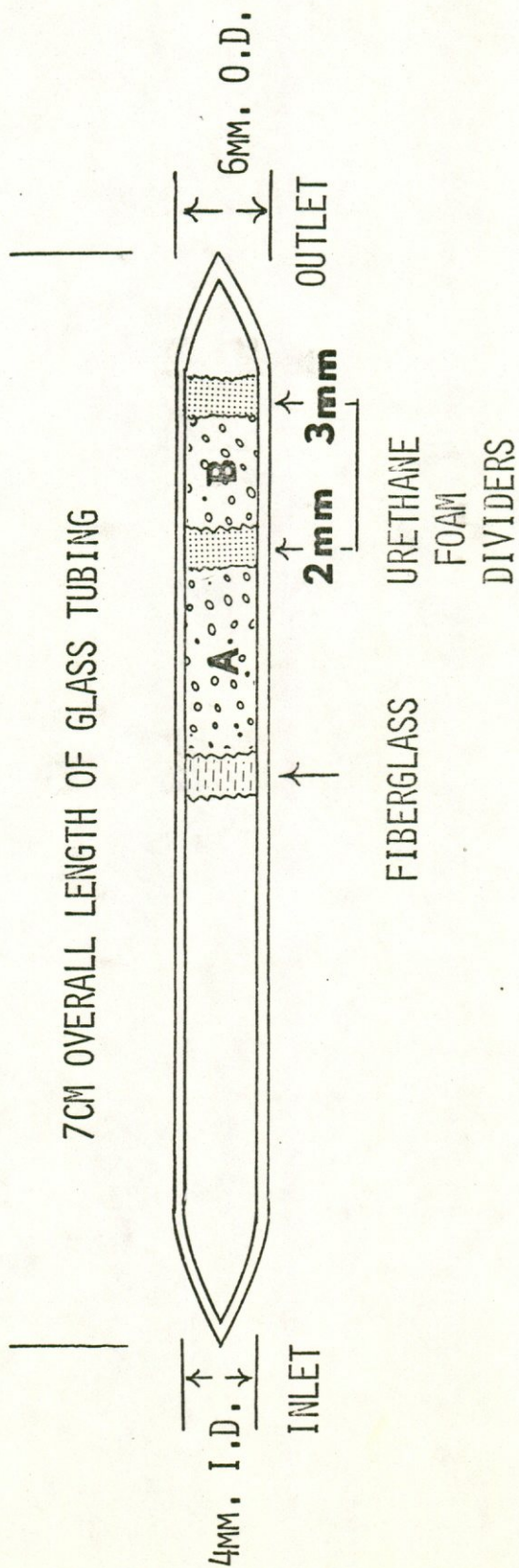


Midget Impinger with Personnel Sampling Pump

Illustration Courtesy of Bendix
National Environmental Instruments, Inc.
Warwick, Rhode Island

Visual 4 - 12

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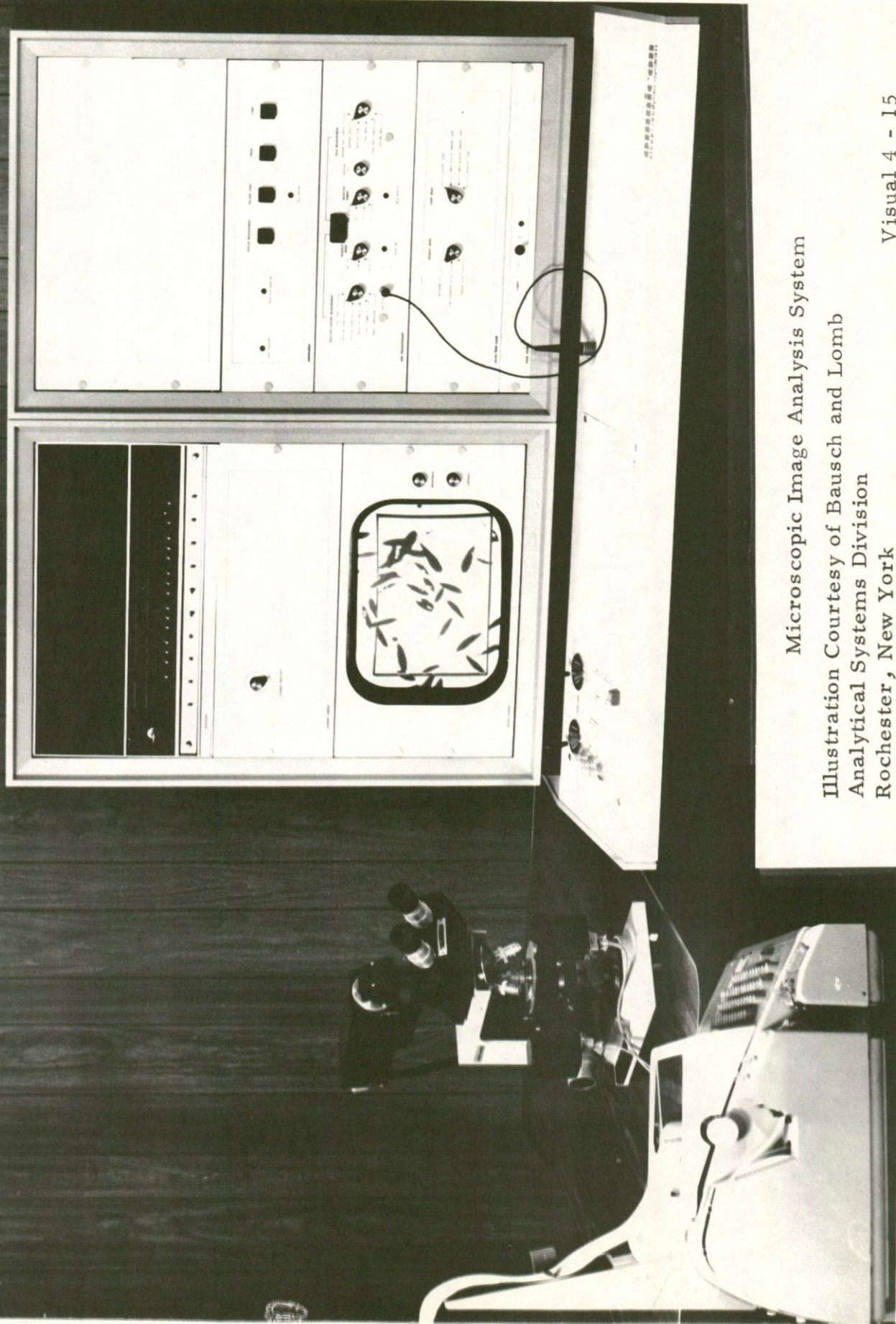
CHARCOAL TUBE



4-64

Reagent and Sampling Equipment

Illustration Courtesy of Mine Safety Appliances Company
Pittsburgh, Penna.



Microscopic Image Analysis System

Illustration Courtesy of Bausch and Lomb
Analytical Systems Division
Rochester, New York



Microscopic Image Analysis System

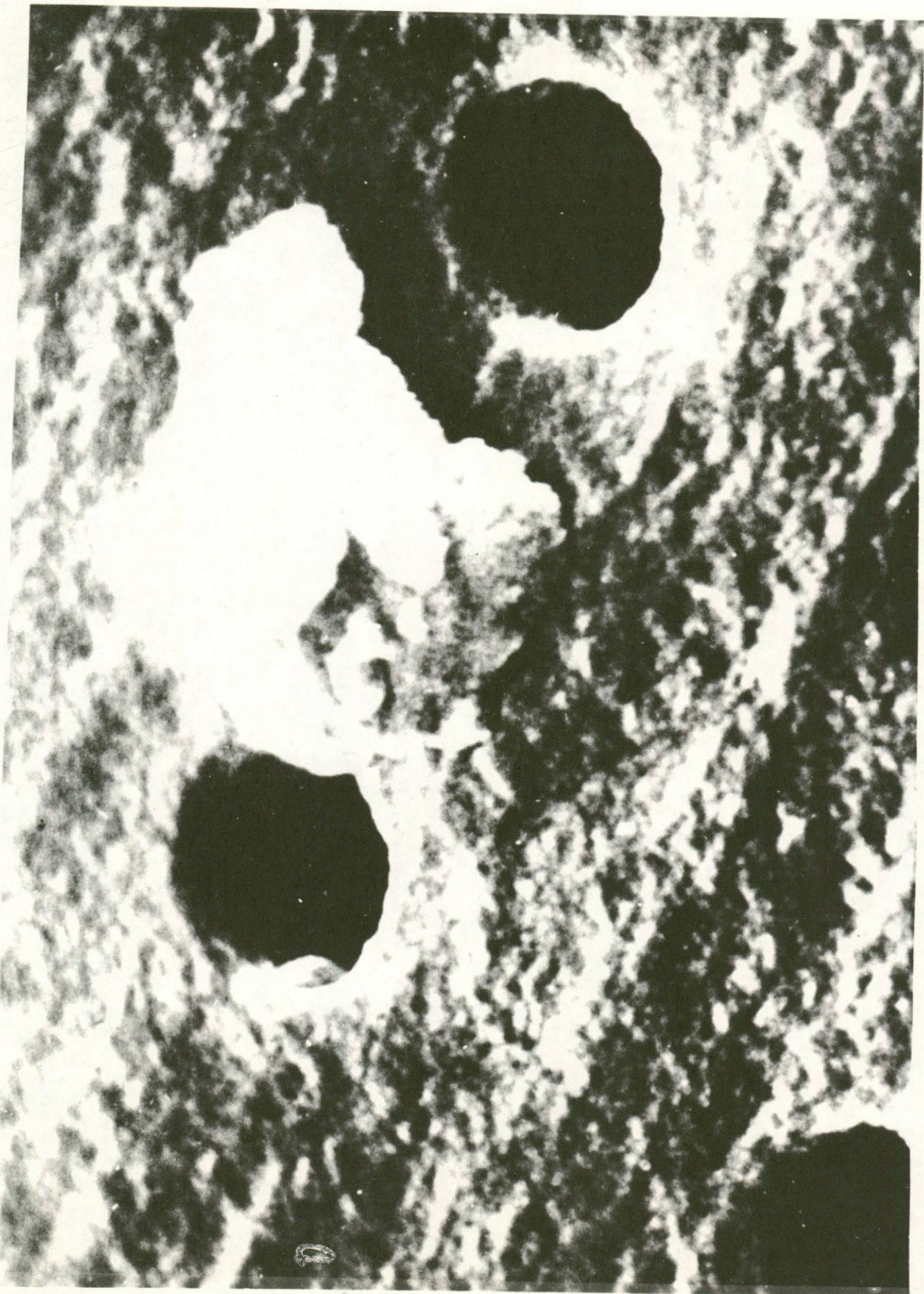
Illustration Courtesy of Bausch and Lomb
Analytical Systems Division, Rochester, New York

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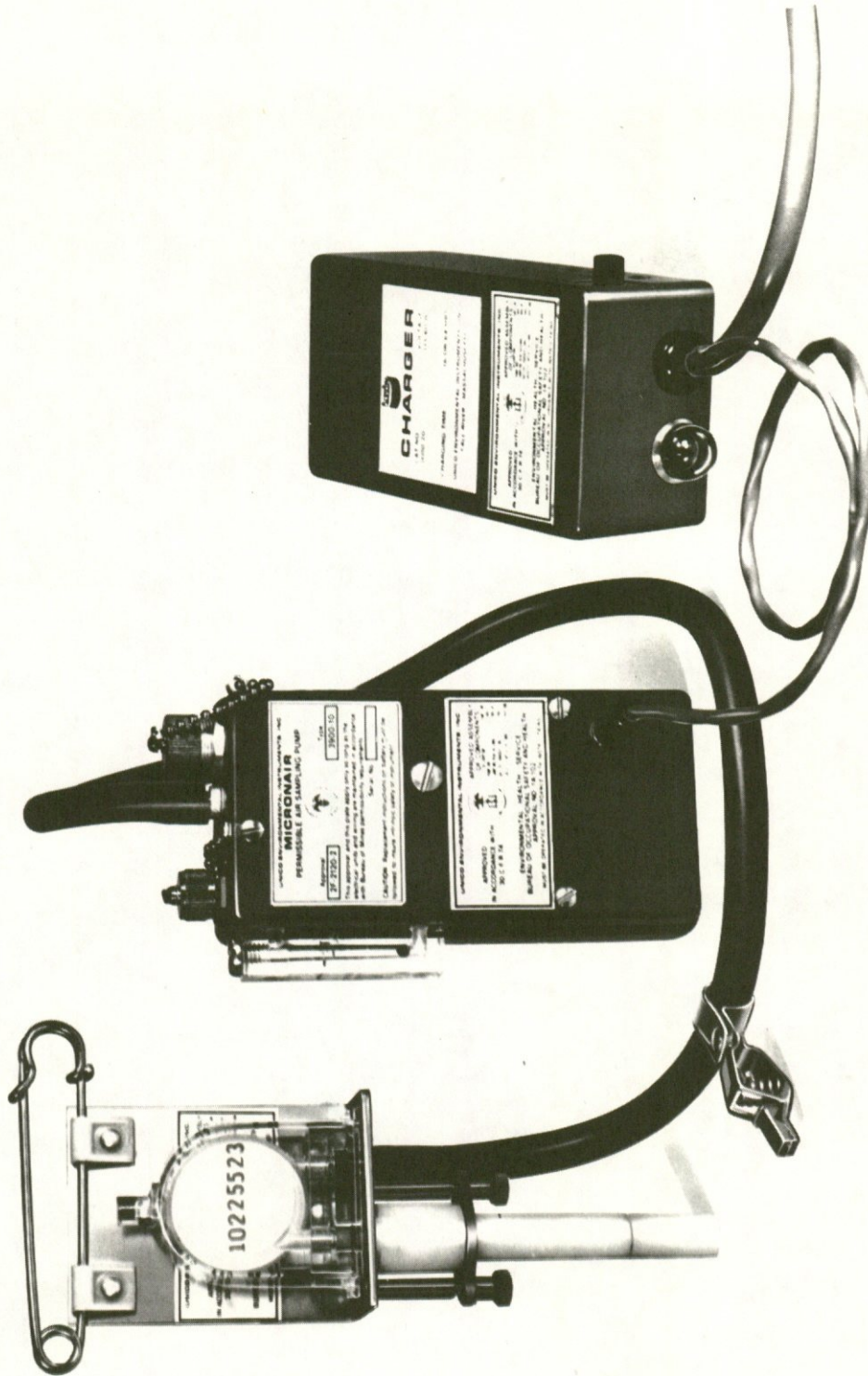


Microbalance

Illustration Courtesy of Cahn Instruments
Cerritos, California

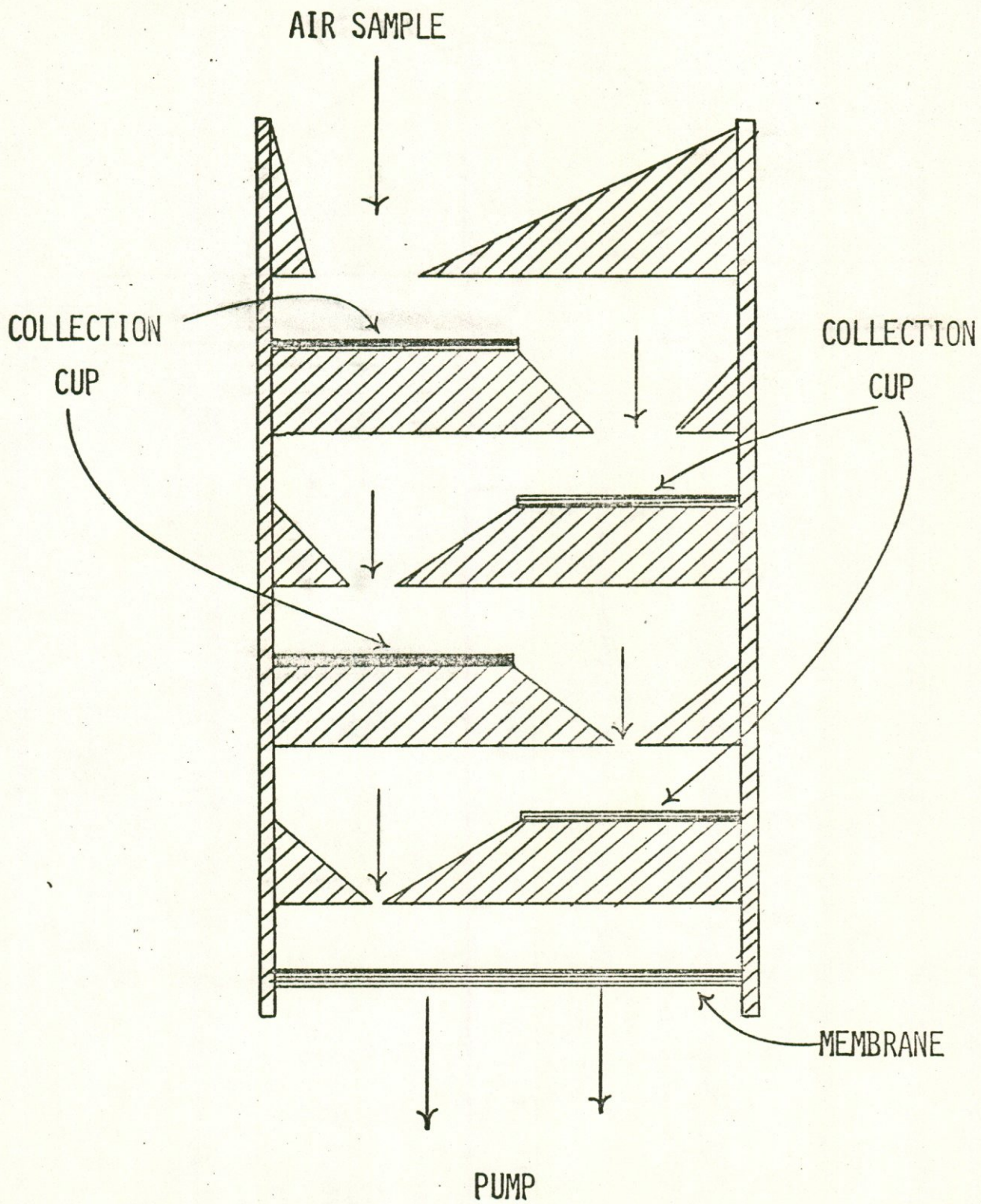


Scanning Electron Micrograph (pores are 1 μm . in diameter)
Illustration Courtesy of Nuclepore Corporation

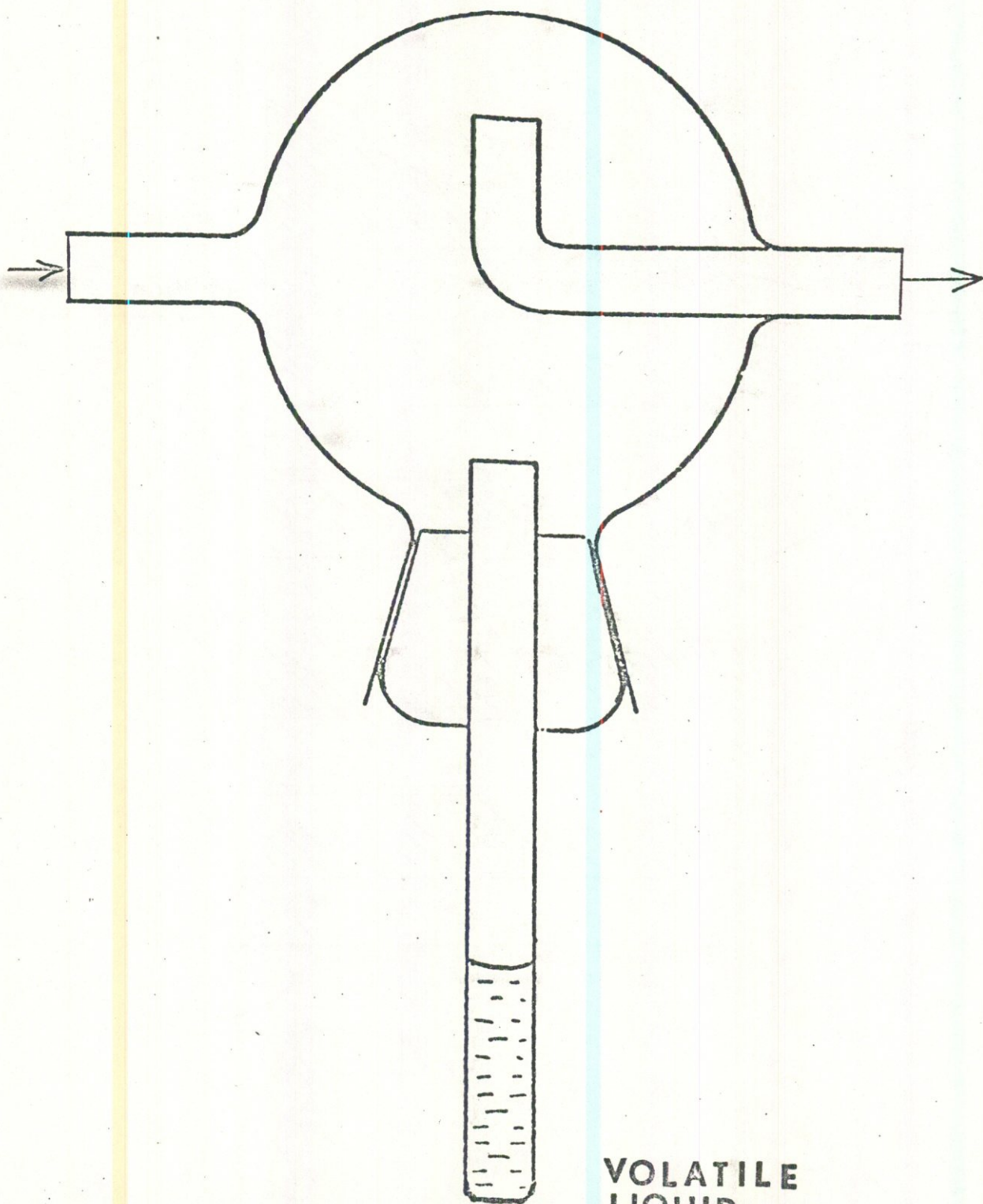


Cyclone Separator Attached to Pump with Battery Charger

Illustration Courtesy of Bendix
National Environmental Instruments, Inc.
Warwick, Rhode Island



A CASCADE IMPACTOR

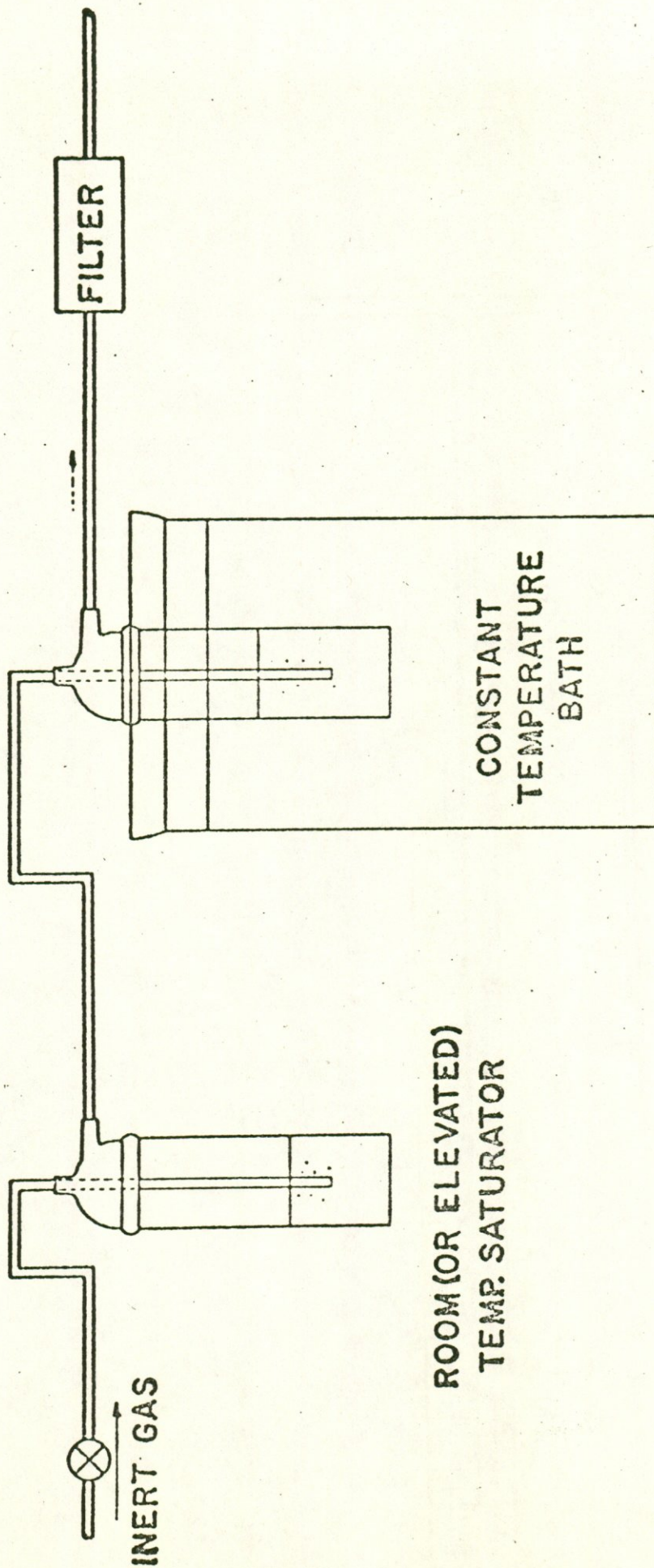


**VOLATILE
LIQUID**

D I F F U S I O N S Y S T E M

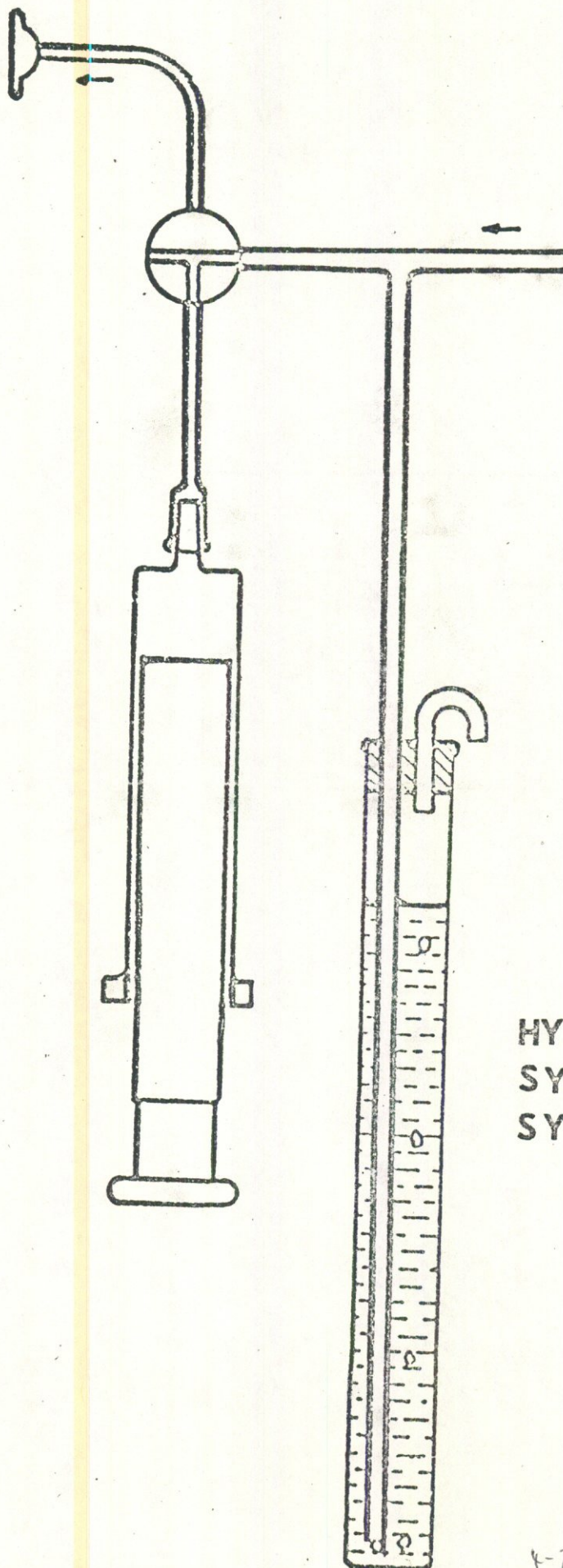
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Visual 4 - 21



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VAPOR SATURATOR



**HYPODERMIC
SYRINGE
SYSTEM**

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