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A simple method for the measurement of carbonyl sulfide in the presence of hydrogen sulphide

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summary reports . . .

A simple method for the measurement of carbonyl sulfide in the presence of hydrogen sulphide

R. G. CONFER and RICHARD S. BRIEF
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Carbonyl sulfide may be associated with hydrogen sulfide in process streams in refineries, sulfur recovery plants and at other operations. If leaks or accidental releases occur, employees will be exposed to both contaminants. The physiologic response resulting from exposure to these toxic materials is reportedly similar. Thus, they are additive in their effect. In many situations, workers' exposure to hydrogen sulfide only is determined. Since exposure to carbonyl sulfide is not determined, the true exposure risk is not assessed.

A gas chromatographic method can be employed for determining employees' exposure to both hydrogen sulfide and carbonyl sulfide. However, some facilities do not have a GC unit and those that do may not have the proper detector for measuring sulfur compounds.

A simple, inexpensive method has been developed for determining exposure to both hydrogen sulfide and carbonyl sulfide. The method can be employed at locations where gas chromatographic equipment is not available. The method involves the conversion of these materials to sulfur dioxide by thermal decomposition and determination of the components by difference when hydrogen sulfide is absorbed from the sample by lead acetate tape. Carbonyl sulfide is not absorbed by lead acetate tape.

The method is as follows. An air sample is collected in a plastic bag. A 5½ liter Saran plastic bag (The Anspec Company, Inc., P.O. Box 44, Ann Arbor, Michigan 48107) has been found to be effective for this purpose. The sample is then taken to the laboratory and the bag is attached to a Gow-Mac Model 13-760 Selective Combustor (Gow-Mac Instrument Company, Madison, New Jersey 07940),

operated at 15 volts. A Mine Safety Appliances Company Universal Pump equipped with a sulfur dioxide tube (Part No. 92623) is used to draw a sample from the bag through the selective combustor and detector tube. The sulfur dioxide concentration determined is equivalent to the combined concentrations of hydrogen sulfide and carbonyl sulfide present in the sample. The carbonyl sulfide concentration can then be determined by placing a glass tube containing approximately 4 square inches of lead acetate tape (Gelman Instrument Company, 600 South Wagner Road, Ann Arbor, Michigan 48106) between the plastic bag and the combustor and again collecting a sample in the manner described above. The sulfur dioxide level measured with the lead acetate tape upstream of the combustor is equivalent to the carbonyl sulfide concentration. The hydrogen sulfide concentration in the sample is then equal to the difference between the total sulfur dioxide concentration determined and the equivalent level of carbonyl sulfide. An additional check on the hydrogen sulfide level can be made by direct measurement of the H₂S level in the bag sample.

If carbon disulfide is also present with COS and H₂S, the sulfur dioxide level measured with the combustor in line will represent the combined amount resulting from the combustion of hydrogen sulfide, carbonyl sulfide and carbon disulfide. The concentration of each component can be determined by difference by measuring H₂S and CS₂ directly from the sample. The carbonyl sulfide level will be equal to the sulfur dioxide concentration minus the sum of the H₂S concentration and twice the CS₂ level.

We have evaluated this sampling and analysis method in the laboratory and in field

situations at concentrations up to 250 ppm combined hydrogen sulfide and carbonyl sulfide and with 100 ppm CS₂ in the presence of 100 ppm H₂S and 100 ppm COS. The efficiency of conversion of hydrogen sulfide and carbonyl sulfide to sulfur dioxide has been found to be approximately 90% at concentrations below 250 ppm. The efficiency for H₂S-COS-CS₂

mixtures up to 300 ppm (100 ppm of each) was the same.

This system has been employed in field studies where a gas chromatographic instrument was not available. It has provided us a means for assessing exposure to both hydrogen sulfide and carbonyl sulfide so that a true assessment of exposure risk can be determined.

A remote generator control for in-place filter testing

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The DOP aerosol test is a standard method¹ for determining the performance of air cleaning systems which contain high efficiency devices for the removal of particulate matter, *e.g.*, HEPA (high efficiency particulate air) filters and graded fiber or sand beds. This test method consists of the introduction of a challenge DOP aerosol upstream of the air cleaning device and measurement of relative aerosol concentrations upstream and downstream of the device with a light-scattering photometer. If the measured penetration of the air cleaning device by the challenge aerosol is to be considered representative of its overall performance, the aerosol must be introduced in a manner such that it is well mixed with the air in the system when it reaches the device;¹ often, this requirement can be met only by introducing the aerosol at a point remote from the upstream sampling port. In other cases, the physical configuration

of the air handling system itself may prevent generation at a convenient nearby location.

In order to eliminate the need for an extra man to operate the aerosol generator at a remote generation point, a simple control was constructed from commercially available reset-type interval/delay timers and a normally-closed solenoid valve. A pictorial diagram of the device is shown in Figure 1. Timer T1 is a 0-30 minute automatic reset timer used in the delay mode; *i.e.*, it provides 115 VAC continuously following the preset time delay. Timer T2 is a similar 0-5 minute unit used in the interval mode, to provide 115 VAC to the solenoid valve during the preset interval only.

In a typical test, the light-scattering photometer is connected to the upstream and downstream sampling ports, and the aerosol generator is positioned at the remote generation port. The generator is connected to the outlet of the control solenoid valve, and the compressed gas supply is connected to the inlet. The control is temporarily defeated with the "on-off" switch on T2 to allow the operator to set the generator pressure at the desired value; then the control is switched back into the circuit, closing the solenoid valve. Timer T1 is set to provide a delay period sufficient for the operator to return to the aerosol photometer and make background readings on the system. Timer T2 is set to provide a generation time sufficient to make all upstream and downstream aerosol concentration measurements (usually 1 to 2 minutes).

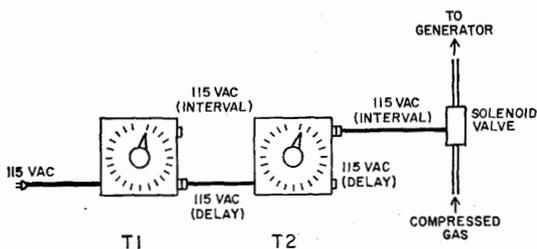


Figure 1—Schematic diagram of aerosol generator control.

Work performed under the auspices of the United States Atomic Energy Commission.

The control is actuated by starting T1. After the preset delay period has elapsed, T2 and the solenoid valve will be actuated, providing challenge aerosol for the interval period selected. At the completion of the interval, the solenoid valve will close and aerosol generation will cease.

This simple control was constructed for under \$100 and it has paid for itself several times over in manpower economy.

Reference

1. American National Standard, Efficiency Testing of Air-Cleaning System Containing Devices for Removal of Particles, ANSI N101-1-1972, American Institute of Chemical Engineers, 345 East 47th St., New York, N.Y. 10017.

Evacuated refrigerant container as a source of vacuum for air sampling

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The Los Alamos Scientific Laboratory conducts research on diversified subjects, many of which involve high explosives. Air sampling in an explosives area is complicated by the fact that standard electrical air sampling pumps cannot be used because of the potential explosion hazard. In addition, there are areas within the Laboratory where electrical power is not readily available. Yet there is a need for a sampling system which can be used under these conditions. This system must be portable and designed in such a way as to allow the industrial hygienist a good deal of versatility in regard to the sampling media that he may choose to evaluate exposure.

In view of these problems, an air sampling system was developed which utilizes a single-use evacuated Freon® container as a source of vacuum. This bottle containing the refrigerant, Freon® 12, is shipped to the Laboratory by the DuPont Company. It is a single-use non-refillable container which is 15 inches high and 12 inches in diameter, weighing 12 pounds.

When the container has been emptied, it is sent to the Industrial Hygiene Group for modification. The plastic valve on top is removed and replaced with a brass valve. Then a vacuum-pressure gauge and a hose connector

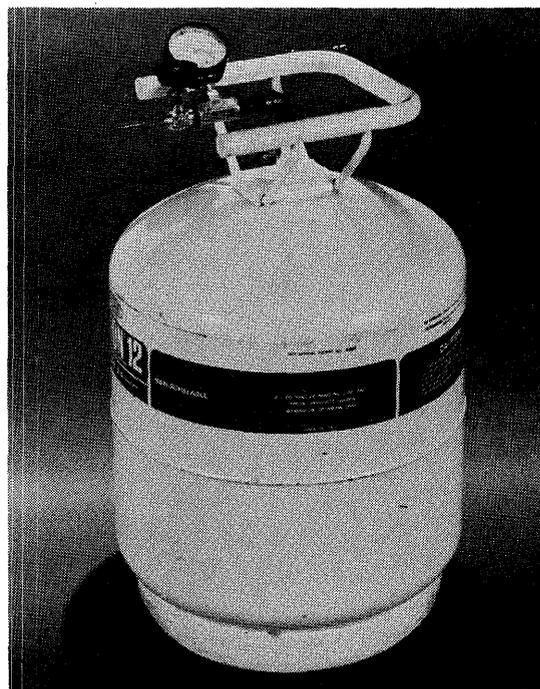


Figure 1—Air sampling device.

are installed to convert the unit into an air sampling device as seen in Figure 1. The container is evacuated and observed for leakage for several days to insure that it is leak tight. It is then evacuated continuously for 3 days to eliminate the Freon® background and any background created by the fitting sealant.

Work performed under the auspices of the U.S. Atomic Energy Commission, Contract W-7405-ENG-36.

Prior to sampling, the container is evacuated with a laboratory vacuum pump for approximately 60 minutes to a negative pressure of 585 mm of mercury, the minimum pressure attainable at the 2195 meter elevation of Los Alamos, New Mexico. If joints are properly sealed, this vacuum can be maintained over a period of one month or longer. Thus, it is possible to have a supply of evacuated containers that are immediately available for sampling, without concern for such factors as deteriorated pump capacity or changes in flow meter calibration.

The sample collection media used to evaluate occupational exposure to organic vapors is the standard NIOSH charcoal tube. The sample that has been collected is then analyzed by gas chromatography. In dusty atmospheres, a membrane prefilter is used to prevent entrance into the charcoal tube of particulate matter which could affect the analysis of the sample.

To sample for particulate matter, the appropriate filter media is chosen, and with a filter head, is connected to the container. Caution must be used when sampling with certain filter media because it has been shown that filter efficiency is dependent, in most cases, on the flow rate through the media. For example, the filter efficiency of Whatman 41 filter paper has been shown to be affected greatly by low flow rates. Therefore, membrane filters are normally used with this system, because it is felt that this medium's efficiency is less velocity dependent than Whatman 41.

By opening the valve as far as possible, an air sample through a membrane filter can be collected in 4 minutes and through a charcoal tube in 6 minutes. However, in the latter case, the high initial flow rate may pro-

duce a pressure drop across the charcoal tube, which could cause the charcoal to be drawn out of the tube. To eliminate this problem and to prolong the sampling time, an orifice was designed and fabricated to limit the flow rate to a maximum of one liter per minute.

The Freon® 12 container represents an improvement over equipment used for grab sampling done in past. The sample volume is relatively large, 24 liters as compared to 100 to 300 milliliters, and the container is rugged compared to glass bottles or flasks. The evacuated glass vessels used in grab sampling present a definite hazard because of the possibility of breakage.

The modified Freon® container used for air sampling is relatively light-weight, is rugged and safe. The unit is readily portable, needs no electricity and is inexpensive. Its volume is relatively large when used as a grab sampler. The airflow and sampling duration can be varied using orifices. The sample volume is known, thus eliminating the need and possible uncertainty in calculating air volumes by flow rates and sampling times. A variety of sampling systems can be employed when sampling for particulate matter, vapors and grab gas samples. The container also needs only one calibration.

Some disadvantages in using this container are that it becomes difficult physically to carry several containers out in the field for multiple samples and the flow rate is not constant throughout the sampling time. There remains the unknown factor involved with chemicals plating on the inside of container walls, although a study at LASL has shown many gases such as CO and CO₂ are stable with time.

A chlorine gas concentration monitor

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Introduction

Gaseous chlorine is used in many industries, and in each case may constitute an industrial hazard to equipment and personnel working in its vicinity. In particular a shrink-resist treatment for wool fabric involves dry chlorination.¹ The development of a new industrial plant for this process² has highlighted a need for the provision of a simple, inexpensive alarm system to warn operators of the existence of unsafe chlorine levels in the working area.

Such an apparatus has been developed and is the subject of this paper. Its operation is based on the change in electrical conductivity of water used to scrub continuously sampled air. It is completely self contained, portable and is not dependent upon mechanical pumps for air sampling.³ A battery power supply is an important feature of the instrument, because electrical power failure in a chlorination plant could be the cause of a dangerous situation and an alarm relying upon mains power would be useless at precisely the time when it was most required.

When present in air to the extent of 5 ppm chlorine has a slight odor and causes a stinging or burning sensation in the eyes, nose and throat. Severe coughing and eye irritation occur within 1 minute of exposure to 10 ppm. Exposure for one half to 1 hour to a concentration of 14 to 21 ppm is dangerous, and the concentration of 100 ppm cannot be borne for longer than 1 minute.^{4,6} One ppm can be detected but values of 10 ppm and 50 ppm were adopted as reasonable for the instrument to satisfy the particular application.

Although originally designed for chlorine gas detection this apparatus could be used to monitor the concentration in air of any gas that dissociates into ions in aqueous solution.

Construction

Basically the instrument consists of a gas scrubber and a conductivity cell. An aspirator

bottle, arranged as a constant head device (Marriot Bottle) delivers water through an ion exchange column at a constant rate of approximately 5 ml per minute. At this rate of flow a 5l bottle will provide at least 12 hour operation before refilling becomes necessary. The feed water flows down through a column packed with ion exchange resin and then the deionized water passes upward through the annular space surrounding the resin column and into the gas sampling and scrubber portion of the assembly. The scrubber consists of 1500mm of 4mm glass tubing wound into a helical coil and mounted vertically. For convenience the coil is mounted coaxially with the ion exchange column. Drops of water entering the scrubber draw air behind them. Air trapped between descending slugs of water is scrubbed by contact with the wet wall of the tubing. At the base of the scrubber, residual air is vented to the atmosphere and the water passes through a conductivity cell containing two platinum electrodes. Water leaving the cell can be collected for further analysis, (i.e. by specific ion electrodes), run to waste or saved for reuse. See Figure 1.

The electronic circuitry associated with the conductivity cell is shown in Figure 2. An alternating current resistance measuring circuit is employed to obviate instability due to electrode polarization caused if direct current is used. When the resistance of the solution between the electrodes drops to a selected value (see Calibration), the circuit oscillates and drives the loudspeaker built into the cabinet of the monitor. An external jack is provided for an extension speaker.

Calibration

Marks were made at either end of the coil to define a known volume. The rate of water and gas flow through the scrubber was then determined by timing slugs of water between these marks, the water was collected and measured and the gas volume obtained by difference.

SUMMARY REPORTS . . .

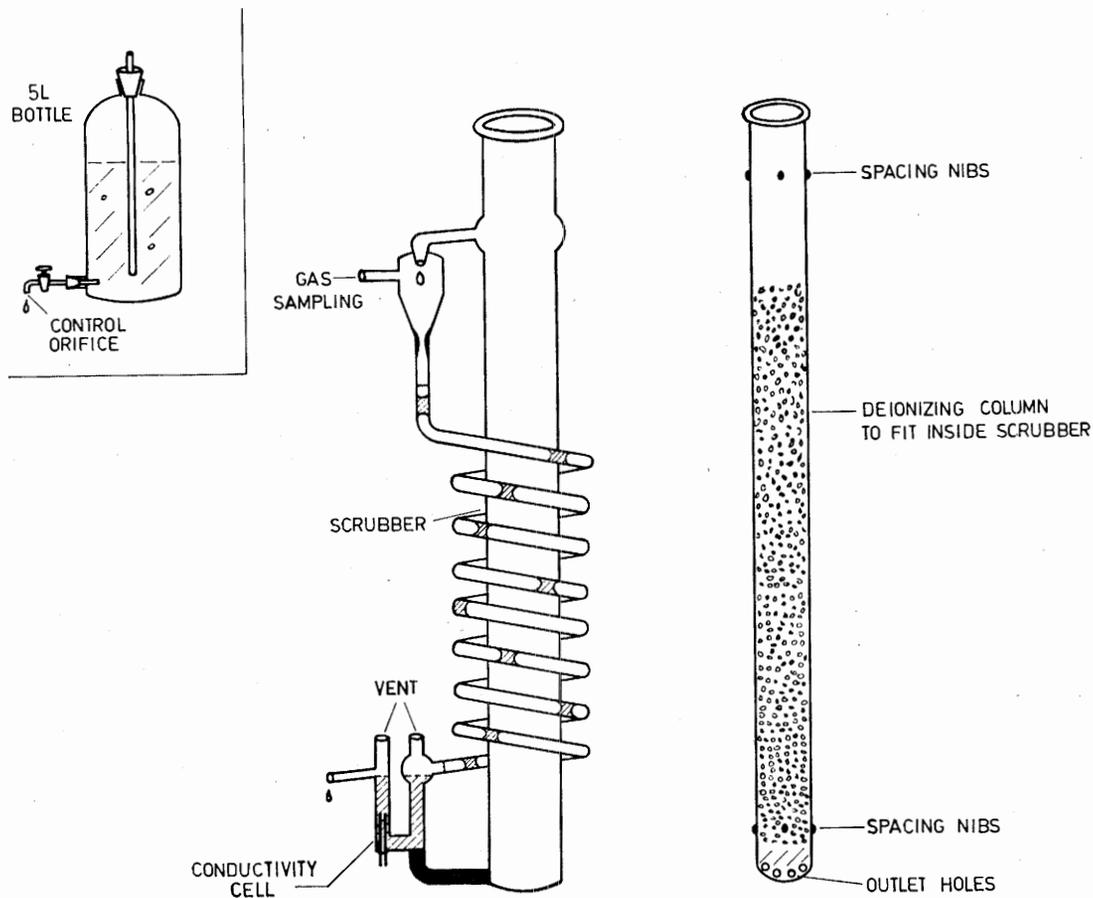


Figure 1—The water feed, deionizing, air induction, scrubbing and conductivity cell of the instrument.

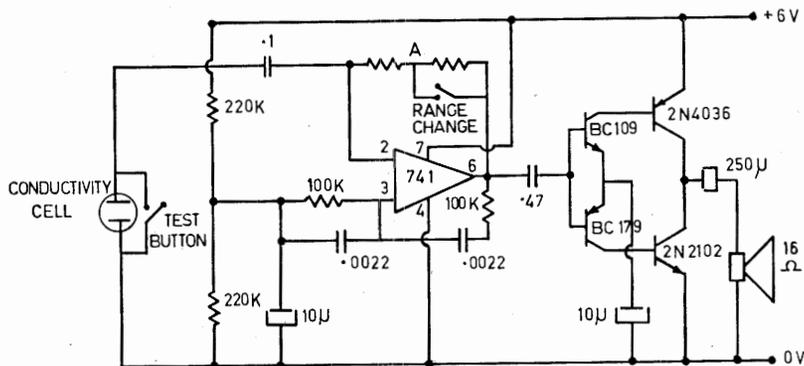


Figure 2—The electronic circuitry, connected to the conductivity cell, which oscillates and sounds the alarm when the resistance of the solution drops to the predetermined level.

In the prototype instrument a gas to water ratio of 12:1 was used to concentrate the soluble gas and thus increased the electrical conductivity of the solution to a level suitable for measurement.

To set the level at which the alarm would sound, the assumption was made that, at the low concentrations involved, chlorine dissolved in water is equivalent to hydrochloric acid. Hence, 10 ppm chlorine in air, concentrated with a gas to water ratio of 12:1 in the scrubber, would result in a 0.0000155 N HCl solution. A standard solution of this strength was placed in the conductivity cell and a rheostat in the circuit (Resistor A in Fig. 2) was adjusted to the threshold value for operation. An equivalent fixed resistor was then substituted for the rheostat. A similar procedure was adopted for calibration to 50 ppm chlorine in the atmosphere. In practice, the resistor A consists of two resistors in series, one for the higher concentration value and the other chosen to give a series resistance appropriate to the lower concentration. Range selection is effected by a shorting switch across the second resistor.

A "TEST" button which short circuits the conductivity cell permits a check on the electronic circuit.

It should be noted that this monitor was designed for use in an area where chlorine was the only water soluble gas expected to be present in appreciable amounts. However, other gases such as carbon dioxide sulphur dioxide

and nitrogen dioxide will also be detected by this system and it could equally well be calibrated for one of these. In situations where several such gases are present, calibration would be impossible, but the scrubber could be used to collect samples for analysis by other means.

Acknowledgement

The authors wish to acknowledge the help of Mr. N. A. Michie in designing the electronic circuit.

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Community noise criteria

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Noise in the community is comprised of many sources but transportation noise is the major source. Although automobiles contribute significantly to the overall noise level due to their large numbers, trucks and buses are the worst offenders and are a major target in any noise abatement program. In fact, the first vehicle noise regulations proposed by the EPA were aimed at trucks with a gross vehicle weight in

excess of 10,000 lbs. Railroads and rapid transit systems contribute their share of noise but so far they have not gained as much community attention as truck and bus noise. Aircraft noise impact is a function of peak sound levels and number of occurrences and primarily affects communities in the vicinity of airports. Some other sources of community noise are industrial plants that generate excessive noise

TABLE I
Design Noise Level/land use Relationship for State Departments of
Transportation, Highway Designers and Planners

| LAND USE CATEGORY | EXTERIOR DESIGN NOISE LEVEL ₁₀ | DESCRIPTION OF LAND USE CATEGORY |
|-------------------|---|--|
| A | 60 dBA | Tracts of lands in which serenity and quiet are of extraordinary significance and serve an important public need, and where the preservation of those qualities is essential if the area is to continue to serve its intended purpose. For example, such areas could include amphitheatres, particular parks or portions of parks, or open spaces which are dedicated or recognized by appropriate local officials for activities requiring special qualities of serenity and quiet. |
| B | 70 dBA | Residences, motels, hotels, public meeting rooms, schools, churches, libraries, hospitals, picnic areas, recreation areas, playgrounds, active sports areas, and parks. |
| C | 75 dBA | Developed lands, properties or activities not included in categories A and B above. |
| D | Unlimited | Undeveloped lands. |
| E | 55 dBA (Interior) | Public meeting rooms, schools, churches, libraries, hospitals and other such public buildings. |

at their boundaries, power-driven garden equipment and window air conditioners.

There is a need in the community noise field to describe time-varying noise as single number criteria. Typical criteria take the noise level and time history of the noise level into consideration. These are L_N numbers (noise levels exceeded $N\%$ of the time), L_{eq} (the equivalent continuous sound level in $dB(A)$), LP_{NP} (the Noise Pollution Level), and L_{dn} (the day-night average sound level in $dB(A)$). Common features of these criteria are the use of A-weighted measurements and the fact that all are based on statistical data. The requirements on single number criteria are ease of understanding, simplicity in measurement and good correlation with the subjective response of human beings.

Single number criteria

The Statistical Noise Level, L_N is the noise level exceeded $N\%$ of the time. This criteria is determined from statistical noise data obtained with an amplitude distribution analyzer. Values such as L_{10} , L_{50} , and L_{90} , the sound levels exceeded 10%, 50%, and 90% of the time are the most common L_N numbers. L_{10} represents an average peak noise level, L_{50} a median level and L_{90} an average ambient level.

Several states and two government agencies use L_N numbers in their noise regulations and guidelines. Also, ISO Recommendation R1996 (Assessment of Noise with Respect to Community Responses) specifies L_{95} as the ambient sound level.

Environmental impact statements that are prepared by state highway departments for

proposed highway projects must have a section dealing with the effects of noise on the area in question. The Federal Highway Administration (FHWA) has set down guidelines for the preparations of these statements including a table of design noise levels with respect to various land uses, Table I. Sound levels in this table are L_{10} values.¹

The Department of Housing & Urban Development (HUD) uses amplitude distribution analysis for rating construction sites. Table II shows external noise exposure standards for new construction sites taken from HUD Circular 1390.2. General external noise exposures are given in terms of sound level and time.

More complex single number ratings such as the Traffic Noise Index (TNI) and the Noise Pollution Level (NPL) can be calculated using L_N numbers. TNI is a weighted combination of L_{10} and L_{90} and is a measure of the annoyance level of motor vehicle noise. NPL is a procedure for estimating annoyance caused by any noise source and is discussed in detail later.

The Energy Equivalent Noise Level, L_{eq} , for a stated period is the level of a constant or steady state noise which has an amount of acoustic energy equivalent to that contained in the measured noise; i.e. the rms values.² The value for L_{eq} is calculated from amplitude distributions (statistical variations of noise levels with time) of traffic and community noise using the following equation:

$$L_{eq} = 10 \log_{10} \left[\frac{1}{100} \sum f_i \frac{L_i}{10^{10}} \right] \quad (1)$$

Where

L_i is the sound level in dB(A) corresponding to the class-midpoint of the class i .

f_i is that time interval (expressed as a percentage of the relevant time period) for which the sound level is within the limits of class i .

At an INCE conference early this year on specifications and standards, it was mentioned that L_{eq} is expected to be an EPA criteria for consumer products.

ISO R1996 specifies L_{eq} must be used when a fluctuating noise is being investigated. L_{eq} is also a basic element in the computation of more complex single number ratings such as NPL and L_{dn} .

The NPL is a rating scheme devised to handle fluctuating signals. The data base is the instantaneous A-weighted sound level recorded over a time period (30 minutes minimum) to establish statistics of total noise exposure for a longer time period. If the noise under investigation is non-gaussian then the following equation for NPL must be used.

$$NPL = L_{eq} + 2.56\sigma \quad (2)$$

Where σ = standard deviation and accounts for the variability of noise events. However, if the noise is gaussian, and traffic noise generally is, then the NPL is calculated from the equation:

$$NPL \text{ or } L_{NP} = L_{50} + d + \frac{d^2}{60} \text{ dB(NP)} \quad (3)$$

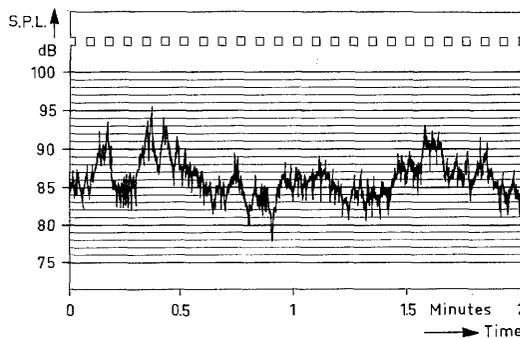


Figure 1—Recording of overall sound pressure level on a busy street.

Where $d = (L_{10} - L_{90})$ dBA. This method is used primarily in England, however, use of NPL may become widespread because it applies to both traffic and aircraft noise. A limited number of studies has shown that NPL has good correlation with the subjective response of human beings to noise.

Lastly, L_{dn} , the day-night A-weighted average sound level, is a rating criteria proposed by an EPA task group as the new measure of community noise. A 10 dB penalty is applied to nighttime (2200-0700 hours) sound levels. This group recommends that L_{dn} be used for all cost-benefit studies, planning, monitoring and enforcement activities under the Noise Control Act of 1972.

To determine L_{dn} for a given location the L_{eq} for the daytime period (0700-2200) and the L_{eq} for the nighttime period (2200-0700) must be determined. L_{dn} is calculated using the following equation:

TABLE II
External Noise Exposure Standards for New Construction Sites
 (measurements and projections of noise exposures are to be made at appropriate heights above site boundaries)

| GENERAL EXTERNAL EXPOSURES | ASSESSMENT |
|--|-------------------------------------|
| Exceeds 80 dBA 60 minutes per 24 hours Exceeds 75 dBA 8 hours per 24 hours CNR Zone 3, NEF Zone C (airport environs) | Unacceptable |
| (Exceptions are strongly discouraged and require a 102 (2) C environmental statement and the Secretary's approval) | |
| Exceeds 65 dBA 8 hours per 24 hours Loud repetitive sounds on site CNR Zone 2, NEF Zone B (airport environs) | Discretionary-Normally Unacceptable |
| (Approvals require noise attenuation measures, the Regional Administrator's concurrence and a 102 (2) C environmental statement) | |
| Does not exceed 65 dBA more than 8 hours per 24 hours | Discretionary-Normally Acceptable |
| Does not exceed 45 dBA more than 30 minutes per 24 hours CNR Zone 1, NEF Zone A (airport environs) | Acceptable |

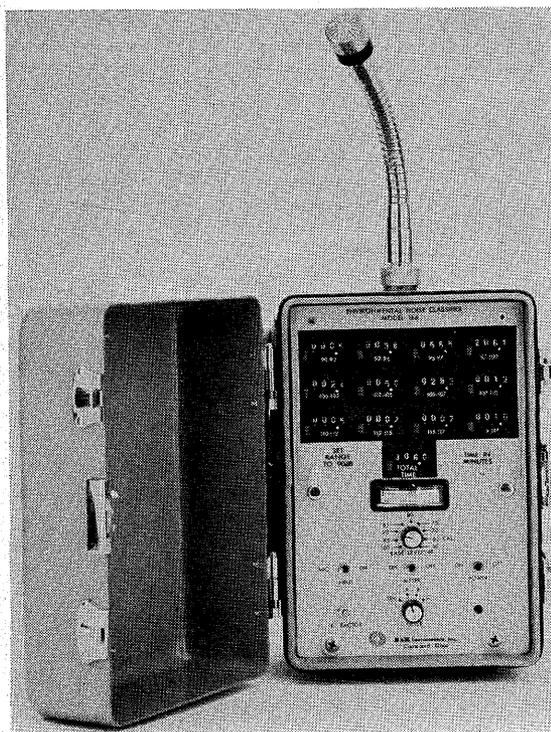


Fig. 2—Type 166 Environmental Noise Classifier provides time vs. amplitude history.

$$L_{dn} = 10 \log \frac{1}{24} \left[15 \left(\frac{L_{d1}}{10^{10}} \right) + 9 \left(\frac{L_{d2}}{10^{10}} \right) \right] \quad (4)$$

Where $L_d = L_{eq}$ for 0700-2200 hours
 $L_n = L_{eq}$ for 2200-0700 hours

Since L_{eq} is calculated from amplitude distributions of community noise the need for statistical analysis instrumentation is again emphasized.

Measurement & determination of criteria

AMPLITUDE DISTRIBUTION

Since the area of interest in community noise has shifted from detailed frequency analysis to statistical variation of noise levels with time, the concept of amplitude distribution has come into widespread use. Amplitude distribution is a technique used to analyze noise based on the statistics of the noise signal under investigation.

Figure 1 is a typical recording of the irregular phenomenon of traffic noise. It can easily be seen that it is meaningless to quote

a sound level at a single instant in time. The sound level varies with time over a 17 dB range so it appears that a statistical approach is proper. This takes the form of plots of probability of exceeding successive dBA levels. Instruments available to obtain the amplitude distributions of a variable, non-periodic signal are an Environmental Noise Classifier, a Statistical Distribution Analyzer and a Digital Data System.

INSTRUMENTATION

The Environmental Noise Classifier (Figure 2), measures time spent in a series of amplitude bands. There are 12 bands, the top band having no upper limit. If the level goes below the baseline, its time will be represented by the difference between the total time meter and the sum of the 12 individual windows.

Consider the histogram in Figure 3 derived from the Environmental Noise Classifier. Time spent in each amplitude band may be plotted in minutes or percent of total time. The cumulative distribution curve (probability of exceedance) indicates the levels exceeded any percentage of the time. In this example; $L_{10} \approx 71$ dBA, $L_{50} \approx 67$ dBA & $L_{90} \approx 60$ dBA

Histograms show the profile of community noise and how the profile changes with time. Histograms also permit the prediction of the impact of *e.g.* a new freeway, based upon prior determination of baseline environmental noise.

At times, complete time histories are required in addition to statistical analyses in order to show when noise intrusions occurred. For such cases a graphic level recorder and statistical distribution analyzer are available to produce both a card copy record and its statistical distribution analysis.

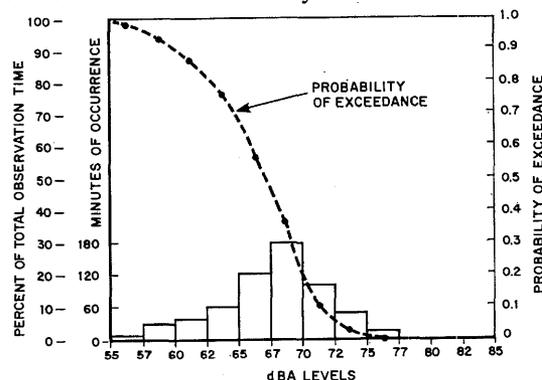


Figure 3—Histogram of community noise.

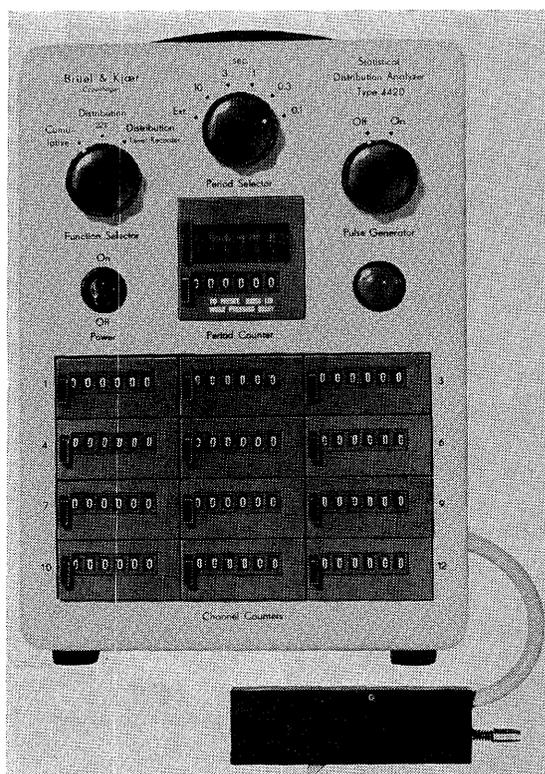


Figure 4—The 4420 Statistical Distribution Analyzer resolves the writing width of B & K's Level Recorders into 12 class intervals, and presents a digital display of the distribution of recorded levels.

The 2305 recorder divides a 25 or 50 dB range into 10 equal intervals. Two additional intervals represent signals above the top line of the chart and below the base line. Each interval is represented by a counter in the Statistical Distribution Analyzer (Figure 4). This unit generates pulses at a selected rate. When each pulse occurs, it is directed to the counter assigned to the amplitude band within which the sound level falls. From this data, histograms as shown in Figure 3 can be drawn. The continuous chart record from the graphic level recorder shows when and how often maximum noise levels occurred. Recording is done with a sapphire stylus on wax paper so that there are no ink problems. The chart line is very fine and the chart can be run at a slow speed so that the user does not have an excessive length of chart paper to edit.

A complete noise survey of a community requires a large number of measurement locations. This means a considerable quantity of data must be collected and processed. The

Environmental Noise Classifier or the Graphic Level Recorder and Statistical Distribution Analyzer will do the job but to save time and money a Digital Data System (Figure 5) can be used.

This system is comprised of a Digital Data Recorder, Digital Data Translator and Digital Interface coupled to a Tektronix 31 Programmable Calculator. The Digital Data System has wide application in environmental noise work. Community noise analysis, highway noise, noise from construction sites, plant boundary noise and ambient noise can all be measured and statistically analyzed using the Digital Data System.

The Digital Data Recorder is battery-powered and has adjustable base lines of 35 dB, 50 dB, and 65 dB with a 60 dB dynamic range, providing recording range of 35-90, 50-110 and 65-125 dBA. Noise levels are recorded digitally on an incremental cassette at sample rates of 1, 3 or 10 seconds corresponding to 1, 3 or 7 days respectively.

The Digital Data Translator plays back the previously recorded data at an accelerated rate so that several complete tapes can be analyzed in an 8 hour day. Two outputs are available, a digital output for computer interface and an analog output for connection to a graphic level recorder to obtain time history charts. A Tektronix Model 31 Programmable Calculator is utilized as the data processor. The programs are conversational with the calculator's printer. For example, with the Statistical Distribution Program (Community Noise)

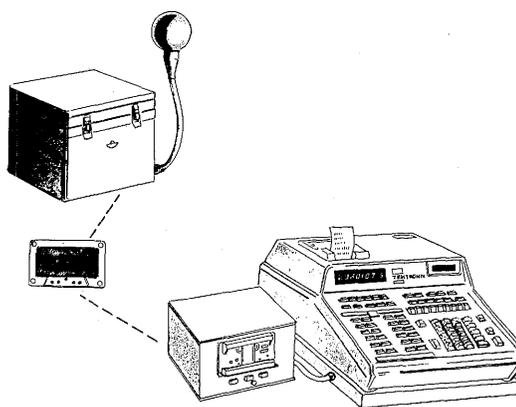


Fig. 5—B & K's SP-321 Digital Data System for community noise analysis records data digitally in the field and then quickly analyzes the data in the laboratory utilizing the Data Translator & Programmable Calculator.

the user can request several L_N values (noise level exceeded N% of the time), in addition to the regular printout of L_{eq} (energy equivalent noise level), mean, standard deviation and Noise Pollution Level (NPL). The calculator prints a pseudo-graph of statistical data by offsetting the printed sound levels across the paper tape of the printer.

Special purpose instruments could be devised for direct readout of any of the single number criteria in use today. The technology exists but there are no commercially available instruments for direct readout because no one is sure which descriptor is best. Manufacturers are reluctant to make instruments that could be obsolete overnight or within a few months.

Bruel & Kjaer makes two special-purpose instruments that produce a noise dose count that can be converted to an L_{eq} dBA reading. The first is the Noise Dose Meter which operates in conjunction with sound measuring equipment for assessment of L_{eq} according to requirements of ISO Recommendation R 1996.

The Noise Dose Meter operates on the equal energy principle (the product of intensity and duration are constant). The integration is performed through a voltage to frequency converter, and the noise dose count counter (NDC). The measuring time T is recorded on the elapsed time counter. At the end of a measurement the NDC and the elapsed time are read from the counters.

In the connected sound level meter, the attenuator setting (ATT.) is noted. L_{eq} is then found from the equation:

$$L_{eq} = ATT. + x \quad (5)$$

where x is obtained from a special slide rule supplied with the Noise Dose Meter. The NDC and T are used to enter the slide rule to find x.

The second instrument for L_{eq} calculation is a Personal Noise Dose Meter. Basic operation is that of a typical noise dose meter how-

ever L_{eq} can be derived from the displayed % figure of noise exposure. A conversion table is included with each dose meter to ease the derivation of L_{eq} . The noise dose meter has an accelerated measurement mode providing more than 100 times faster indication so that short duration measurements can be made and converted to L_{eq} . The major drawback in using the Noise Dose Meter to determine L_{eq} for the community situation, is that it has limited dynamic range and cannot measure below 85 dBA.

Summary

In the fields of community noise abatement and municipal planning, there are new requirements to define an environment of varying noise level by a single number rating method that correlates well with the subjective response of human beings.

Typical criteria, either in use or proposed, are L_N numbers (noise levels exceeded N% of the time), L_{eq} (the equivalent sound level in dB(A)), L_{NP} (the Noise Pollution Level), and L_{dn} (the day-night average sound level in dB(A)).

Instruments available include an environmental noise classifier, a statistical distribution analyzer/recorder combination, and a digital sampling system for field measurements for subsequent interrogation by a programmable calculator.

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X-ray diffraction characteristics of some silicates

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Introduction

X-ray diffraction has recently been applied in the qualitative and quantitative characterization of mineral dusts of interest in the occupational health field.¹ It is also useful in the electron microprobe technique for the detection of dusts in atmosphere, water, foods and tissues.² As a part of research on the chemical aspects of the toxicity of silicates³⁻⁵ the present authors have examined the x-ray pattern of 6 Indian silicates in this investigation.

Materials and Methods

Dust samples of amosite, anthophyllite and chrysotile below 30 μ and that of kaolin, mica and talc below 5 μ were prepared as reported earlier.³⁻⁵

X-ray diffraction pattern

The x-ray diffraction pattern of the silicates was examined using a Debye Scherre circular powder camera of 114.6 mm diameter (Philips Norelco) and Indu X-ray films.

The respective dusts were uniformly filled in Lindemann glass capillaries 0.4 mm diameter to an effective length of 1 mm. The specimen was mounted in the camera and properly centered. The film was tightly held against the inside of the camera. Finally the camera was placed on its track on the x-ray unit. The x-ray tube was a Cu target (Philips) and the unfiltered $K\alpha$ -radiation of copper used for the diffraction photographs. The tube was

operated at 30 KV-10 mA (half-wave rectified). The exposure time ranged from 4-5 hrs. The film was developed for 4 min at 180°C, using May & Baker "Solidex" x-ray developer and fixed by May & Baker "Amfix" high speed fixer. The film was washed for 30 min in running water and dried in a dust-free room.

The diffraction photographs were used to determine principal lattice spacing. Bragg's angles (θ) were calculated from the distance between the corresponding area (x) on the photograph since

$$X/2\pi r = \frac{4\theta}{360}$$

x was measured by the special film measuring device. From the camera radius r, θ and λ of the radiation used, d values were calculated by substitution in the Bragg's equation

$$2d \sin \theta = n\lambda$$

The x-ray diffraction pattern of the dusts are presented as 2θ values.

Results

The data for asbestos principal lattice spacing are given in Table I.

The data for kaolin, mica and talc principal lattice spacing 3 are given in Table II.

Discussion

The x-ray diffraction pattern of the various silicates may help in the identification of the minerals and their characterization in comparison to authentic samples. Indian chrysotile differed from Rhodesian and Canadian chryso-

TABLE I
Principal Lattice Spacing (Cu $K\alpha$ radiation) for
Various Asbestos Dusts

| | AMOSITE | | ANTHOPHYLLITE | | CHRYBOTILE | |
|---|----------|-----------|---------------|-----------|------------|-----------|
| | ANGSTROM | 2θ | ANGSTROM | 2θ | ANGSTROM | 2θ |
| 1 | 1.307 | 72.21 | 1.498 | 61.88 | 1.526 | 60.62 |
| 2 | 1.529 | 60.51 | 1.583 | 58.23 | 1.70 | 53.89 |
| 3 | 1.703 | 53.80 | 2.121 | 42.59 | 2.59 | 35.79 |
| 4 | 2.441 | 36.82 | 2.512 | 35.74 | 3.64 | 24.44 |
| 5 | 3.57 | 24.94 | 2.786 | 32.13 | 4.37 | 20.30 |
| 6 | 4.4 | 20.13 | 3.57 | 24.86 | 7.25 | 12.21 |
| 7 | 6.96 | 12.72 | 4.37 | 20.29 | — | — |

TABLE II
Principal Lattice Spacing (Cu K α radiation) for
Kaolin, Mica and Talc Dusts

| | KAOLIN | | MICA | | TALC | |
|----|----------|------------|----------|------------|----------|------------|
| | ANGSTROM | 2 θ | ANGSTROM | 2 θ | ANGSTROM | 2 θ |
| 1 | 1.30 | 72.64 | 1.505 | 61.55 | 1.487 | 62.39 |
| 2 | 1.389 | 67.38 | 1.641 | 56.00 | 1.635 | 56.21 |
| 3 | 1.523 | 60.76 | 1.953 | 46.45 | 2.31 | 38.98 |
| 4 | 1.65 | 55.67 | 2.532 | 35.45 | 2.501 | 35.90 |
| 5 | 2.095 | 43.13 | 3.24 | 27.46 | 2.778 | 32.23 |
| 6 | 2.195 | 41.08 | 4.44 | 20.00 | 3.55 | 25.21 |
| 7 | 2.452 | 36.65 | 4.88 | 18.17 | 5.35 | 16.55 |
| 8 | 2.584 | 34.72 | — | — | 6.945 | 12.75 |
| 9 | 3.06 | 29.18 | — | — | — | — |
| 10 | 3.42 | 26.04 | — | — | — | — |
| 11 | 4.412 | 20.07 | — | — | — | — |

tile in having one additional prominent arc 2 θ value 53.9, the other characteristics being similar.⁶ However, Crable and Knott¹ reported 2 θ values of 24.5, 18.8 and 12.3 to be the chief characteristics of chrysotile. In the case of anthophyllite, the Indian sample had four arcs of 2 θ values above 35° unlike the U.I.C.C. reference sample while arcs with 2 θ below 20° were not prominent.⁶ With amosite, the variety found in India showed several differences, especially in possessing additional x-ray diffraction arcs with 2 θ of 53.8, 60.5 and 72.2. Isomorphous substitution of the main constituents by other metals may lead to differences in x-ray diffraction patterns.⁷

The above minor differences in x-ray diffraction patterns may be due to the minor variations in chemical composition.³⁻⁵ The differences in infrared spectra⁸ also support this conclusion. However, since the relative intensities of the various arcs could not be quantitatively assessed in the equipment used in the present experiment, interpretation of the x-ray characteristics in terms of the structural aspects of the silicates⁷ is not attempted in this investigation. Nevertheless, it is apparent that like other physico-chemical properties studied, in x-ray diffraction patterns also, there were differences among the various silicates. This may have a bearing in understanding of the chemical basis of the relative toxicity of the various silicates. Even more significant is the fact that the x-ray characteristics of the silicates will be helpful in the electron microprobe detection of the dusts which is a recent advancement in the field of research on industrial lung diseases.²

Acknowledgement

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Calibration of low velocity windtunnels (0-300 fpm)

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Background

The NIOSH Maintenance and Calibration Lab is responsible for servicing industrial hygiene equipment used by OSHA of USDL and NIOSH of USDHEW. Among these instruments are air velocity measuring devices.

Calibration standards were necessary to check the accuracy of these devices. NBS recommended pitot tubes be used as the standard for high velocity. However, in 1971, they could not recommend a standard for low velocity calibration. The uncertainty involved with pitot tubes at velocities under 200 fpm is much too great. NBS now has a laser system for low air velocity.

After several ideas were tried, it was decided to use smoke in the calibration method. Smoke particles should move at the same velocity as air. The particles are visible, and the time required to pass two points at a known distance could be measured. The advantages of such a method are that it is simple, cheap, and very basic. Another advantage of using smoke is that turbulence is readily detected by observing smoke patterns.

Calibration

PRELIMINARY STEPS

These include eliminating turbulence and checking velocity profile. The windtunnel used had a bell mouth entrance to an 18 inch square measuring section which is 5 feet long. Then there is a transition to a 6 inch round duct with a 4 inch measuring orifice.

Early tests with smoke patterns (at a nominal 150 fpm) revealed:

1. very high velocities along the tunnel walls;
2. room turbulence appeared to be entering the tunnel;
3. flow straighteners would be necessary.

In order to cure the above mentioned problems it was necessary to:

1. eliminate the bell mouth and substitute a 3 foot long 18 inch square entrance section;
2. cover the new tunnel inlet with 30 mesh screen;

3. install straighteners immediately downstream from the screen.

Before proceeding to the actual calibration, tests with a hot-wire anemometer showed a flat velocity profile. Smoke patterns showed little turbulence, and the smoke traveled in a straight line through the measuring section.

EQUIPMENT REQUIRED

1. movie camera capable of 56 FPS, with zoom lens and capable of using high speed color film (Ektachrome 160).
2. timer with a light emitting display capable counting at 100 Hz. The display must be large and bright enough to be photographed at a setting of f/1.8 on the camera.
3. windtunnel with . . .
 - a. measuring section at least 2 feet long by 18 inches square,
 - b. down stream orifice (for resetability),
 - c. inclined manometer.
4. film viewer.

PROCEDURE

Mark the rear of the tunnel (away from the camera) at one inch intervals. The front of the tunnel should be marked at two inch intervals.

Align the camera so the entire rear of the measuring section and the timer display are in the picture.

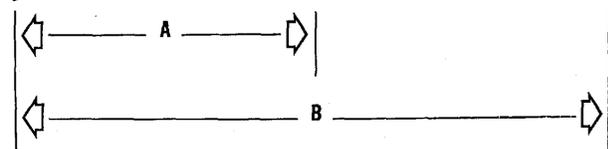
Set the blower speed so the downstream orifice is at an appropriate pressure drop.

Inject the smoke and start the camera. The smoke which we found to be suitable was cigarette smoke cooled by a one foot length of copper tubing. Smoke tubes were tried, but the smoke seemed to get heavier after the first few puffs. The smoke is injected at right angles to the flow and about 1/2" in front of the screen.

Analysis of the film: Pick a point on the smoke and measure the time it takes to pass several marked points on the tunnel. By making several determinations of velocity, we may judge whether the smoke is accelerating or decelerating.

SUMMARY REPORTS . . .

| | | | | | | | | | | | | | |
|----|----|----|----|-----|----|-----|-----|-----|-----|-----|-----|-----|--------------------------|
| 4" | 5" | 6" | 7" | 8" | 9" | 10" | 11" | 12" | 13" | 14" | 15" | 16" | marks on rear of tunnel |
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | |
| 1 | | | | 1 | | | | 1 | | | | 1 | marks on front of tunnel |
| 8" | | | | 10" | | | | 12" | | | | 14" | |



For smoke down the center of the tunnel.

$$A = \frac{(8 - 4) + (10 - 8)}{2} = 3" \quad B = \frac{(12 - 4) + (12 - 8)}{2} = 6"$$

NOTE: This diagram is only for illustration purposes. Since the camera is about 4 feet from the tunnel front, the numbers will not align as shown.

When the camera is properly aligned, the film will appear as shown in the accompanying diagram which includes a sample calculation of the determination of the distance between two points.

Overall system accuracy depends mainly on the film reading, and also on the blower

speed being constant. With a little practice analyzing the film, the reading technique can be refined by looking for "blobs" in the smoke, estimating the actual time for the smoke "blob" to pass the measuring lines, etc. This technique is good for velocities in the neighborhood of 50 to 300 fpm with an accuracy within 10%.

Adapting the noise dosimeter to extended workdays

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Noise dosimeters are designed to measure, accumulate, and display an individual's noise exposure for an eight-hour period in terms of percentage of the daily permissible limit in accordance with OSHA standards. The dosimeter automatically calculates and displays the solution to the following equation:

$$D = 100 \sum C_i / T_i \quad (1)$$

where *D* is percent of the daily permissible limit, *C_i* equals the actual time of exposure to a noise level, and *T_i* equals the allowable time of exposure at that level.

Based on the current OSHA noise standard, exposure to a sound level for an eight-hour period should result in the dosimeter displaying the percentage of the daily permissible limit shown in Table I.

Noise dosimeters now in use are designed to measure noise levels over an eight-hour period. However, special consideration must be given for workdays which exceed eight hours. OSHA or NIOSH have not yet defined permissible noise exposure limits for workdays exceeding eight hours. In view of the lack of any better information, the following equation may be used to determine the permissible duration (*T*) to a noise level (*L*):

$$T = 16 \div 2^{(L-85)/5} \quad (2)$$

From equation (2), the permissible sound levels for 10 and 12-hour workdays are 88.5 dBA and 87 dBA, respectively. The occupational noise exposure standard recommended by the OSHA Standards Advisory Committee on Noise takes into account all sound levels

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TABLE I

| ACTUAL SOUND LEVEL (dBA) | PERMISSIBLE DURATION PER DAY PER OSHA STANDARDS (HOURS) | ACTUAL DURATION PER DAY (HOURS) | PERCENT OF DAILY PERMISSIBLE LIMIT % |
|--------------------------|---|---------------------------------|--------------------------------------|
| 90 | 8 | 8 | 100 |
| 95 | 4 | 8 | 200 |
| 100 | 2 | 8 | 400 |
| 105 | 1 | 8 | 800 |
| 110 | 1/2 | 8 | 1,600 |
| 115 | 1/4 | 8 | 3,200 |

TABLE II

| ACTUAL SOUND LEVEL (dBA) | PERMISSIBLE DURATION PER DAY PER RECOMMENDED OSHA STANDARD (HOURS) | ACTUAL DURATION PER DAY (HOURS) | PERCENT OF DAILY PERMISSIBLE LIMIT % |
|--------------------------|--|---------------------------------|--------------------------------------|
| 85 | 16 | 8 | 50 |
| 87 | 12 | 12 | 100 |
| 88.5 | 10 | 10 | 100 |
| 90 | 8 | 8 | 100 |

between 85 and 115 dBA by application of equation (2). If the recommended standard is adopted by OSHA, dosimeters now in use will have to be modified to change the dBA cutoff to just below 85 dBA and to integrate on a 16-hour basis. After these modifications are made, the dosimeter would display the percent of daily permissible limit for workdays exceeding eight hours as shown in Table II.

If the current OSHA noise standard remains unchanged, dosimeters which can be *field modified* to trigger at sound levels between 85 and 90 dBA can be used for work periods exceeding eight hours by multiplying the dosimeter readout by an appropriate factor. This is illustrated in Table III for a 12-hour workday.

For a 12-hour period, the correction factor is thus $\frac{2}{3}$. This leads to a useful two-

step technique for modifying dosimeters to correct for greater than eight-hour exposures:

- (1) Set the lower cutoff at a level determined by equation (2) for the time period in question.
- (2) Multiply the resultant readout by $8/T$
 T is the daily time period.

In summary, the noise dosimeter should be adaptable to extended workdays. If the noise exposure standard recommended by the OSHA Standards Advisory Committee on Noise is adopted, all noise dosimeters will have to be modified to measure noise levels between 85 and 90 dBA and to integrate on a 16-hour basis. Further modification to account for extended workdays would not be necessary. If the current OSHA noise standard is maintained, dosimeters now in use can be modified and used for extended workdays.

TABLE III

| ACTUAL SOUND LEVEL (dBA) | PERMISSIBLE DURATION PER DAY (HOURS) | ACTUAL DURATION PER DAY (HOURS) | PERCENT OF DAILY PERMISSIBLE LIMIT | |
|--------------------------|--------------------------------------|---------------------------------|------------------------------------|---------------------|
| | | | Dosimeter Readout | Calculated (Actual) |
| 87 | 12 | 12 | 150 | 100 |
| 90 | 8 | 12 | 225 | 150 |
| 95 | 4 | 12 | 450 | 300 |
| 100 | 2 | 12 | 900 | 600 |
| 105 | 1 | 12 | 1,800 | 1,200 |
| 110 | 1/2 | 12 | 3,600 | 2,400 |
| 115 | 1/4 | 12 | 7,200 | 4,800 |