CHAPTER 10

GENERAL PRINCIPLES IN EVALUATING THE OCCUPATIONAL ENVIRONMENT

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INTRODUCTION

Evaluating the occupational environment requires a multidisciplined approach. A fundamental need exists for the input of the knowledge of engineers, chemists, health physicists, physicians, toxicologists, nurses, production supervisors, and others in the elimination of hazards which threaten workers. The most successful approach coordinates these many disciplines and incorporates effective communication between the employer and employee for the recognition, evaluation and control of potential hazards.

Obviously it is not always practical to enlist such a group of workers, management, and highly skilled professionals in Industrial Hygiene for most plants. It is essential, however, that each person evaluating the work environment be knowledgeable of the contributions of other professions to the solution of specific problems. For example, an engineer studying ventilation control for benzene should know the chemistry of benzene and the influence of benzene upon man. Likewise, the physician studying the work environment should have a knowledge of the engineering requirements for control, as well as the chemical sampling and analytical techniques used.

GENERAL PRINCIPLES

The general principles in evaluating the occupational environment concern recognition of potential hazards, preparation for field study, conducting the field study, and interpretation of the survey results.

The recognition of potential hazards includes becoming familiar with the process 3, maintaining an inventory of physical and chemical agents encountered, periodically reviewing the different job activities of a work area, and studying the existing control measures. The procedures in the preparation for a field study embrace the selection of proper instruments, calibration of equipment, and the development of the required analytical methods. Factors to consider in conducting a field study are all related to sampling; where, when, how long, and how many samples, as well as the merits of general area versus breathing zone sampling should be weighed. Once the survey has been conducted, the industrial hygienist must interpret the results. Health standards and previous data are available for comparison. Knowledge of proper corrective measures is an integral part of the industrial hygienist's responsibilities.

This chapter presents the guidelines to be considered by an industrial hygienist in planning a

strategy for evaluation of the occupational environment.

RECOGNITION OF POTENTIAL HAZARDS

The investigator must become familiar with all processes used in the particular plant or other establishment. He must learn what chemicals or substances are used and produced and the intermediate products, if any. This information may be obtained by asking questions during the survey, by visual observation, and by a study of process flow sheets usually contained in technical books that describe the particular operation. It is of utmost importance that a list of all chemicals and products used be obtained for future reference during the evaluation of the environment. The variety of substances capable of causing occupational diseases increases steadily. New products are constantly being introduced which require the use of new raw materials or new combinations of older substances, and new processes. It has been estimated that new (and some potentially toxic) substances are introduced into industry at the rate of one every 20 minutes and that about 10,-000 such materials are in use today. New uses for physical agents in industrial processes are increasing at a rapid rate. Examples include the use of lasers, microwaves, supersonic welding equipment, and many others. These, too, are potentially hazardous unless proper control measures are insti-

Toxicity of Raw Materials and Products

It is important for the investigator to recognize that the toxicity of a substance is not the sole criterion, or necessarily the most important, of the existence of a health hazard associated with a particular industrial operation. The terms "toxicity" and "hazard" are not synonymous. The nature of the process in which the substance is used or generated, the possibility of reaction with other agents (physical or chemical), the degree of effective ventilation control or the extent of enclosure of the process materials all relate to the potential hazard associated with each use of a given chemical agent (see Chapter 9). Such an assessment must be made along with due consideration of the type and degree of toxic responses the agent may elicit in both the average and, possibly, hypersusceptible workers.

After the list of chemicals used and produced is obtained, it is necessary to determine which of these are toxic and to what degree. Information of this nature can be found in the latest texts and in scientific journals, Hygienic Guides published by

the American Industrial Hygiene Association (A.I.H.A.), the Z-37 Standards published by the American National Standards Institute (A.N.S.I.).² and by correspondence with toxicologists, technical information centers, and manufacturers. Toxic Substances, a recent publication by the National Institute for Occupational Safety and Health^a contains over 8000 substances. The list, which is revised annually, gives the concentration at which each substance is known to be toxic and should serve as an excellent reference source in the area of toxicology. Many companies publish a list of toxic materials used in their plants for use by safety personnel, foremen, and others. Similar information should be obtained also on the potential hazards of physical agents in use. A number of guides, standards, and texts are available for this purpose.

Sources of Air Contaminants

Many potentially hazardous operations can be detected by visual observation during the preliminary survey. The most dusty operations can be easily spotted, although this does not necessarily mean they are the most hazardous. It must be remembered that the dust particles which cannot be seen by the unaided eye are the most hazardous because they are of respirable size. Furthermore, dust concentrations must reach extremely high levels before they are readily visible in the air. Thus the absence of a visible dust cloud does not mean necessarily that a dust-free atmosphere exists. However, those operations that generate fumes, such as welding, can be spotted visually.

Reference to the list of raw materials, products and byproducts will indicate possible air contaminants. In any burning operation, a knowledge of the fuels used will indicate the air contaminants generated. Separation processes can produce chemicals or particulates which are potentially hazardous.

The presence of many vapors and gases can be detected by the sense of smell. Trained observers are able to estimate rather closely the concentration of a limited number of solvent vapors and gases in the workroom air by the odor level. For many substances, however, the odor threshold concentration is greater than the permissible exposure levels. For example, if the odor of carbon tetrachloride vapor is barely perceptible, this indicates the amount is generally too great for a continuous exposure. In fact, concentrations of some vapors and gases may be present in concentrations considerably in excess of the permissible level, but may not be detectable by their odor. New Stresses — Changes in Processes

As indicated above, new chemical products and physical agents are continually being introduced and used in industrial processes. The investigator must be aware of these and must ascertain the potentially hazardous nature of these before they are used so that any necessary safeguards can be inaugurated. Many companies, especially the larger ones, have such information available and will generally make it accessible to the investigator. Furthermore, the employer should inform employees of these potential hazards and should establish controls for their protection.

Job Activities Review

A review of the worker's routine job requirements should be made. Changes in his job requirements or modifications of techniques to accomplish his work can have a profound effect upon his exposure to health hazards. Overtime requirements for particular jobs should be determined so that the contribution of overtime and the related prolonged exposure of workmen to health hazards can be evaluated.

Control Measures in Use

The preliminary survey would not be complete unless the types of control measures in use and their effectiveness are noted. Control measures include local exhaust and general ventilation, protective respiratory devices, protective clothing, and shielding from radiant heat, ultraviolet light, or other forms of radiant energy. General guides to effectiveness include the presence or absence of dust on floors and ledges; holes in ductwork, fans not operating or rotating in the wrong direction (the latter has been found to occur in many plants and it should be noted that, even though a blower is operated backward, it will still exhaust some air, but not the required amount); or the manner in which personal protective devices are treated by workmen. During the preliminary survey it may be desirable to conduct a check on local exhaust ventilation systems in use to determine if sufficient airflow is provided to remove the contaminants from the workers' breathing zone. The manual, "Industrial Ventilation,"4 will serve as a useful guide for this purpose since it describes test procedures and contains examples of many types of systems with the recommended airflows.

Adequate notes must be made during an evaluation of the environment. The speed and accuracy of preparing a report of an investigation will depend largely on information recorded in the form of notes.

SELECTION OF INSTRUMENTS TO EVALUATE THE WORK ENVIRONMENT

Sampling instruments used to evaluate the environment for occupational health hazards are generally classified according to type as follows: (1) direct reading; (2) those which remove the contaminant from a measured quantity of air; and (3) those which collect a known volume of air for subsequent laboratory analysis.

Most of the equipment used by industrial hygienists is found under the first two types. The third group includes various types of evacuated flasks, plastic bags, or other suitable containers for collecting known volumes of contaminated air to be returned to the laboratory for analysis.

The choice of a particular sampling instrument depends upon a number of factors. Among these are: (1) portability and ease of use; (2) efficiency of the equipment or device; (3) reliability of the equipment under various conditions of field use; (4) type of analysis or information required; (5) availability; and (6) personal choice based on past experience and other factors.

No single, universal sampling instrument is available and it is doubtful if such an instrument will ever be developed. In fact, the present trend

is the development of a greater number of specialized instruments such as the direct reading gas and vapor detectors.

In evaluating a worker's exposure or the environment in which he works, an instrument must be used that will provide the necessary sensitivity, accuracy, reproducibility, and, preferably, rapid results. Detailed discussions of instruments used for sampling for particulates are given in Chapter 13, for gases and vapors in Chapter 15, and for direct reading instruments for aerosols, gases and vapors in Chapter 16, as well as in "Air Sampling Instruments for Evaluation of Atmospheric Contaminants" published by the American Conference of Governmental Industrial Hygienists. Instruments for assessing noise exposure and for other physical agents are discussed in subsequent chapters of this manual. One of the older, but still valid, discussions on this subject is "Sampling and Analyzing Air for Contaminants" by Silverman. Those whose responsibilities include the collection and analysis of samples will find this publication a worthwhile reference.

The use of continuous monitoring devices to evaluate the working environment has increased tremendously in recent years. While these devices are normally not designed for field use, many are available in sizes that are convenient for this purpose. In general, however, many industries install these devices in areas where exposures to certain gases or vapors may vary considerably. Examples include the use of continuous monitors for carbon monoxide in tunnels or plant areas where this gas is produced or used, monitors for chlorinated hydrocarbons such as in the production of carbon tetrachloride or trichloroethylene, and monitors for certain alcohols. Many of these continuous detecting and recording instruments can be equipped to sample at several remote locations in a plant and record the general air concentrations to which workers may be exposed during a shift. Many large plants have added computerized equipment to the recorders so that the data may be readily available and summarized for instant review. However, as is the case with other instruments, continuous monitors must be calibrated periodically and the interferences known.

After selecting the instrument, the industrial hygienist, compliance officer, or other person collecting samples must become familiar with the device and its limitations. He must know, for example, whether or not the particular instrument is specific for the contaminant to be determined, what other substances interfere with the test, and the accuracy and sensitivity of the device. He must also be familiar with the response time, which is the time interval from the instant samples are taken to the time the instrument shows a reading or the chemical reaction takes place in a detector tube. Furthermore, in the case of detector tubes, the readings must be made under good lighting conditions, preferably in daylight. 7.8.9.10-11

CALIBRATION OF INSTRUMENTS

Instruments and techniques used in calibrating sampling equipment are discussed in detail in Chapter 11. This brief discussion is included to

stress the necessity for following recommended procedures in order that the data resulting from the analysis of field samples (whether by direct reading devices in the field or by equipment that collects samples for subsequent laboratory analysis) will truly represent concentrations in the environment, and particularly concentrations to which the worker is exposed.

Since the amount of sample, whether collected by means of a filter, an impinger, or a bubbler or indicated by a direct reading instrument, depends upon the volume of air sampled and its duration, it is essential that the device operate at a known rate of airflow. Thus, the equipment must be calibrated against a standard airflow measuring device both before and after use in the field. The exact rate of airflow must be recorded so that when it is multiplied by the sampling time, the total volume of air sampled or collected will be known. This volume of air is used in calculating the concentration of contaminant to which the worker was exposed. Furthermore, direct reading instruments and detector tubes must be calibrated against a known concentration of the substance for which they are used. Results obtained during a survey or study are no more accurate than the instruments used to obtain the data. In some situations the investigator must do his own calibrating, but more frequently this is done by others at a central laboratory.

ESTABLISHING PROPER ANALYTICAL METHODS

The use of accurate, sensitive, and reproducible analytical methods is equally as important as the proper calibration of the sampling equipment. In evaluating the occupational environment the concentration of contaminant in the ambient air is generally small. In fact, the direct reading instruments and other devices used to collect samples for subsequent analysis are required to detect quantities of substances in the microgram or part-permillion range. Thus, a sufficient quantity of sample must be collected to enable the analyst to determine accurately this small amount of substance.

When available, standard methods of analysis should always be used. Unfortunately, only a few such methods have been tested under various situations and conditions. In the field of industrial hygiene the A.C.G.I.H. has available a "Manual of Analytical Methods" that contains about 20 methods.12 Many of the nearly 200 A.I.H.A. Hygienic Guides¹ contain recommended methods of analysis. This same organization also publishes Analytical Guides, which to date cover 61 methods of analysis. The American Public Health Association as prime contractor with the National Air Pollution Control Administration, and through the Intersociety Committee, has been developing methods for air sampling and analysis; the first 60 of these have been published as a manual entitled "Methods of Air Sampling and Analysis," American Public Health Association, 1015 Eighteenth Street, NW Washington, D.C., 1972.12 While these methods were developed primarily for the field of air pollution, most of them can be used

for sampling and analysis of environmental con-

ditions in plants.

The American Society for Testing and Materials14 initiated Project Threshold in March, 1971 to validate test methods in the field of air pollution. Many of these methods can be used also for sampling and analyzing contaminants in the workplace. Several methods have been developed and tested, the latest of which is a tentative fluorescent method for beryllium.

The reason for this lack of tested standard methods is the tremendous amount of time and personnel required to perform the necessary testing. There are nearly 500 substances included in the latest TLV list (A.C.G.I.H.) but there are perhaps less than 100 standardized methods available to determine compliance or non-compliance with the U.S. Department of Labor's regulations under the Occupational Safety and Health Act of 1970. This does not mean that methods other than the standard methods cannot be used. However, these other methods must also be tested and calibrated against known concentrations of the substance in question. Many analytical methods have been published in the literature and the methods should be evaluated in the laboratory prior to performing an analysis.

MAKING THE FIELD SURVEY

The nature of the substance or condition to which workers may be potentially exposed will usually have been determined during the preliminary survey. The problem, then, is to determine the intensity of exposure and to do this one must collect samples of the air or use direct reading instruments. Every effort must be made to obtain samples that represent the worker's exposure. To decide what constitutes a representative sample, the investigator must answer these five basic questions: (1) where to sample; (2) whom to sample; (3) how long to sample (sampling duration); (4) how many samples to take; and (5) when to sample — day or night, what month or season.

Where to Sample

Where the purposes of the sampling are to evaluate a worker's exposure and to determine his daily, time-weighted average exposure, it is necessary to collect samples at or as near as practical to his breathing zone and also in the area adjacent to his normal work station, or general room air. Sometimes it is necessary to sample at the operation itself because of the difficulties of placing a sampling device at his breathing zone or attaching it to his person. On the other hand, if the purpose of sampling is to define a potential hazard, to check compliance with regulations, or to obtain data for control purposes, samples representative of the worker's exposure must be collected. In some cases it is necessary to sample the general room air also to define certain exposures. Whom to Sample

Personnel exposure is best determined by monitoring the different job tasks in a suspect area. Personnel monitors properly attached to workers directly exposed should give a representative sample to actual breathing zone exposure. Actual exposure sources can be documented with the proper number of personnel samples combined with the results from stationary sampling points. Job descriptions for area personnel and the time spent at each task are of primary importance in determining potential exposure.

How Long to Sample

The volume of air sampled and duration of sampling is based upon the sensitivity of the analytical procedure or direct reading instrument; the estimated air concentration; and the Standard or TLV of the particular contaminant. Thus, the volume of air sampled may vary from a few liters, where the estimated concentration is high, to several cubic meters where low concentrations are expected. Knowing the sensitivity of the method, the TLV, and the sampling rate of the particular instrument in use, the minimum sampling time can be determined. The above applies to devices that collect a known volume of air for later analysis. The duration of sampling should represent some identifiable period of time — usually a complete cycle of an operation - to determine an operator's exposure. Another technique is to sample on a regular schedule, for example, so many minutes of each hour. This procedure usually requires more samples than cyclic-type sampling but, when used in conjunction with the cyclic-type sampling, gives more confidence in the results and recommendations.

Evaluation of worker's daily time-weighted average exposures is usually best accomplished when analytical methods will permit, by allowing the worker to work his full 7- or 8-hour shift with a personal breathing zone sampler attached to his person. Techniques have been developed in recent years that allow full shift sampling for many dusts, fumes, gases and vapors. These techniques, which include the use of filters and miniature cyclones to sample airborne dusts and activated charcoal to sample many gases and vapors have been developed successfully in recent years for many airborne contaminants. The concept of full shift integrated personal sampling is much preferred to that of short term or general area sampling if the results are to be compared to standards based on time-weighted average concentrations. When methods that permit full shift integrated sampling are not applicable, time-weighted average exposures can be calculated from alternative short term or general area sampling methods by applying the general formula explained in Chapter 3.

The first step in calculating a worker's or group of workers' daily, time-weighted exposure is to again study the job descriptions obtained for the persons under consideration and ascertain how much time out of each day they spend at various tasks. Such information is usually available from the plant personnel office or foreman on the job. In many situations the investigator must make time studies himself to obtain the correct information. Even though this information was obtained from plant personnel, it should be checked by the investigator because in many situations what the investigator observes and the times given by plant personnel do not agree. From this information and the results of the environmental survey, a

daily, time-weighted average 8-hour exposure can be calculated. This assumes that a sufficient number of samples have been collected or readings obtained with direct reading instruments under various plant operating conditions to give a true

picture of the exposure.

Where sampling for the purpose of comparing results with airborne contaminants whose toxicological properties warrant short term and ceiling limit values, it is necessary to use short term or grab sampling techniques to define peak concentrations and estimate peak excursion durations. For purposes of further comparison, the time-weighted average 8-hour exposure can be calculated using the values obtained by short term sampling.

How Many Samples to Take

There is no set rule to determine the number of replicate samples that are necessary to evaluate a worker's exposure, provided that a minimum number are taken to characterize the exposure in time and space. A single sample will never suffice even if the investigator believes that this concentration would be maintained throughout the work shift. If the indicated concentration is near or above the TLV or Standard, repeated sampling should be done. Ordinarily, contaminants are not generated at a constant rate, and the concentration can vary considerably from time to time.

Only rarely does an operation release contaminants into the workroom air at a fairly constant rate. Chapters 13 and 15 describe sampling procedures for particulates, gases, and vapors, including information on the number of samples needed. The concentration found in single sample may have been too high or too low due to a number of factors and if the sample had been collected at another time, the results could very well be considerably different. Several dozen samples may be necessary to define accurately a daily, timeweighted average exposure for a worker who performs a number of tasks during the shift.

When to Sample

Another area to be considered in sampling is when to sample — a determination of the work shift or seasonal period during which samples should be collected. If, for example, an operator continues working for more than one shift, samples should be collected during each shift that he works. It has been found that in many situations airborne concentrations of toxic substances or exposure to physical agents may be different for each shift. Furthermore, and this applies to plants located in areas where large temperature differences occur during different seasons of the year. samples should be collected during summer and winter months. Normally, there is more general ventilation during the summer months than in winter, a factor which tends to dilute the concentration of the contaminant.

INTERPRETATION OF FINDINGS

Interpretation of the analyses of samples collected or from direct reading instruments is the final step in evaluating the environment. A great deal of common sense and judgment must be used in interpreting the results of an environmental study. Before an investigator determines that a worker or group of workers is exposed to a hazard injurious to health, he must have the following facts: (1) nature of substance or physical agent involved; (2) intensity (concentration) of exposure; and (3) duration of exposures, which will have been determined from the preliminary survey and the results of air sampling done during the environmental study. In many cases, adverse effects from exposure to toxic materials or physical agents do not appear until the exposure has occurred for several years. The purpose of TLVs or Standards is to protect against the future appearance of such symptoms.

Comparison of Results with Standards

Results of the environmental study must be compared with standards before an employer can be cited for a violation or control measures can be recommended. It must be emphasized again that the samples collected during the study must be representative of the worker's daily, time-weighted average exposure, if there is a standard for such exposure, before a comparison can be made.

In connection with the enforcement of the Occupational Safety and Health Act, the standards for exposure to gases, vapors, dust, ionizing and non-ionizing radiation are contained in the Code of Federal Regulations (CFR), Title 29, Part 1910, Occupational and Environmental Health Standards. These were first published in the Federal Register, Vol. 26, No. 105, May 29, 1971 and are subject to revision. At this writing, most of these standards are the same as the A.C.G.I.H. 1970 TLVs, but about 20 are the latest A.N.S.I. Z-37 Standards. In 1971, for the first time A.C.G.I.H. published a combined list of Threshold Limit Values for airborne contaminants and physical agents.15 Included in the latter are guidelines (TLVs) for noise, lasers, microwaves, ultraviolet radiation, and heat stress. Basic Radiation Protection Criteria¹⁶ should be consulted for standards on ionizing radiation. Other guidelines in this area include the A.I.H.A. Hygienic Guides¹ A.N.S.I. Standards, 2 and the A.S.H.R.A.E. Guide 17 for temperature and humidity.

Many states have adopted standards in the above area, some of which are more stringent than those referred to above. Since a state may qualify to administer and enforce a State Occupational Safety and Health Program under provisions of the Federal Act, the standards in effect in a particular state must be consulted by the investigator. Federal standards adopted to date (1972) are minimum legal requirements and they will no doubt be modified from time to time as new data become available. State standards must be "at least as effective" as Federal standards. When state standards are applicable to products distributed or used in interstate commerce, they must be such as are required by compelling local conditions and do not unduly burden interstate com-

Comparison of Results with Previous Data

As indicated earlier in this discussion, many of the larger industrial establishments utilize continuous monitors to maintain a record of concentrations of various gases and vapors in certain areas of their plants. These data must be related to worker exposure. Other companies that employ industrial hygienists ordinarily have data on exposures of workmen to various toxic substances as well as certain physical agents. If at all possible, the investigator should make every effort to study these data and compare them with the results of his study. In many cases the data may not be the same and there may be good reasons for the discrepancy. In many instances the data on exposures will be more complete and detailed when taken from company records than when obtained by an investigator from a one- or two-day investigation of certain hazardous operations in that plant. Thus, considerable embarrassment can be avoided if the above suggestion to check other recorded data is followed.

In addition to data available from company records, other sources of such information include the results of previous studies conducted by Federal, state and local agencies, some insurance companies, and consultants. Here again, results of these studies may be different than those obtained by the investigator so great care must be exercised in making comparisons.

SUMMARY

The conduct of environmental surveys and studies is only one phase in the over-all effort in determining occupational health hazards. Such surveys are valuable only if all environmental factors relating to the workers' potential exposures are included. In evaluating workers' exposures to toxic dusts, fumes, gases, vapors, mists, and physical agents, a sufficient number of samples must be collected, or readings made with direct reading instruments, for the proper duration to permit the assessment of daily, time-weighted average exposures and evaluate peak exposure concentrations when needed.

It is essential that the proper instrument be selected for the particular hazard under study and that it be calibrated periodically to insure that it is sampling at the correct rate of airflow and, in the case of direct reading instruments, that they have been calibrated against known concentrations of the contaminant in question. For those samples to be analyzed in the laboratory, a method must be used that is accurate, sensitive, specific, and reproducible for that particular contaminant.

Adequate notes taken during environmental studies are a necessity. An investigator or inspector cannot rely upon his memory after a study is completed to provide the detailed information necessary for the preparation of a report.

Finally, sound judgment should be exercised both during the actual survey and while preparing the report.

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Preferred Reading

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- Archives of Industrial Health.
- Environmental Science and Technology. 3.
- Journal of the Air Pollution Control Association.
- Journal of Occupational Medicine.
- British Journal of Industrial Hygiene.
- Bulletin of Hygiene (London)
- Heating, Refrigeration and Air Conditioning. 8.
- Analytical Chemistry
- 10. Industrial Hygiene Digest.
- 11. Journal of Laboratory and Clinical Medicine.
- Noise Control.
- Toxicology and Applied Pharmacology. 13.
- 14. New England Journal of Medicine.
- Air Sampling Instruments Manual (ACGIH).
- Encyclopedia of Chemical Technology (Interscience Publishers).
- 17. Handbook of Chemistry and Physics (Chemical Rubber Pub. Co.)
- I.E.S. Lighting Handbook (Illuminating Engineering Society).
- 19. Handbook of Dangerous Materials (Sax).

CHAPTER 11

INSTRUMENTS AND TECHNIQUES USED IN CALIBRATING SAMPLING EQUIPMENT

Morton Lippmann, Ph.D.

INTRODUCTION

Importance of Accurate Calibrations and Periodic Recalibration

Air samples are collected in order to determine the concentrations of one or more airborne contaminants. To define a concentration, the quantity of the contaminant of interest per unit volume of air must be determined. In some cases, the contaminant is not extracted from the air; i.e., it may simply alter the response of a defined physical system. An example is the mercury vapor detector, wherein mercury atoms absorb the characteristic ultra-violet radiation from a mercury lamp, reducing the intensity incident on a photocell. In this case, the response is proportional to the mercury concentration and not to the mass flowrate through the sensing zone; hence, it measures concentration directly.

In most cases, however, the contaminant is either recovered from the sampled air for subsequent analysis or is altered by its passage through a sensor within the sampling train, and the sampling flowrate must be known in order to ultimately determine airborne concentrations. When the contaminant is collected for subsequent analysis, the collection efficiency must also be known, and ideally should be constant. The measurements of sample mass, of collection efficiency and of sample volume are usually done independently. Each measurement has its own associated errors, and each contributes to the overall uncertainty in the reported concentration.

The sample volume measurement error will often be greater than that of the sample mass measurement. The usual reason is that the volume measurement is made in the field with devices designed more for portability and light weight than for precision and accuracy. Flowrate measurement errors can further affect the determination if the collection efficiency is dependent on the flowrate

Each element of the sampling system should be calibrated accurately prior to initial field use. Protocols should also be established for periodic recalibration, since the performance of many transducers and meters will change with the accumulation of dirt, corrosion, leaks, and misalignment due to vibration or shocks in handling, etc. The frequency of such recalibration checks should initially be high, until experience is accumulated to show that it can be reduced safely.

Types of Calibrations

Flow and Volume. If the contaminant of interest is removed quantitatively by a sample collector at

all flowrates, then the sampled volume may be the only air flow parameter that need be recorded. On the other hand, when the detector response is dependent on both the flowrate and sample mass, as in many length-of-stain detector tubes, then both quantities must be determined and controlled. Finally, in many direct-reading instruments, the response is dependent on flowrate but not on integrated volume.

In most sampling situations the flowrates are, or are assumed to be, constant. When this is so, and the sampling interval is known, it is possible to convert flowrates to integrated volumes, and vice-versa. For this reason flowrate meters, which are usually smaller, more portable and less expensive than integrated volume meters, are generally used on sampling equipment even when the sample volume is the parameter of primary interest. Normally, little additional error is introduced in converting a constant flowrate into an integrated volume since the measurement and recording of elapsed time generally can be performed with good accuracy and precision.

Flowmeters can be divided into three basic groups on the basis of the type of measurement made; these are integrated-volume meters, flowrate meters, and velocity meters. The principles of operation and features of specific instrument types in each group will be discussed in succeeding pages. The response of volume meters, such as the spirometer and wet-test meter, and flowrate meters, such as the rotameter and orifice meter, are determined by the entire sampler flow. In this respect they differ from velocity meters such as the thermoanemometer and Pitot tube, which measure the velocity at a particular point of the flow cross section. Since the flow profile is rarely uniform across the channel, the measured velocity will invariably differ from the average velocity. Furthermore, since the shape of the flow profile usually changes with changes in flowrate, the ratio of point-to-average velocity will also change. Thus, when a point velocity is used as an index of flowrate, there is an additional potential source of error, which should be evaluated in laboratory calibrations which simulate the conditions of use. Despite their disadvantages, velocity sensors are sometimes the best indicators available, as for example in some electrostatic precipitators where the flow resistance of other types of meters cannot be tolerated.

Calibration of Collection Efficiency. A sample collector need not be 100% efficient in order to be useful, provided that its efficiency is known

and constant, and taken into account in the calculation of concentration. In practice, acceptance of a low but known collection efficiency is a reasonable procedure for most types of gas and vapor sampling, but is seldom, if ever, appropriate for aerosol sampling. All of the molecules of a given chemical contaminant in the vapor phase are essentially the same size, and if the temperature, flowrate, and other critical parameters are kept constant, they will have the same probability of capture. Acrosols, on the other hand, are rarely monodisperse. Since most particle-capture mechanisms are size-dependent, the collection characteristics of a given sampler are likely to vary with particle size. Furthermore, the efficiency will tend to change with time due to loading; e.g., a filter's efficiency increases as dust collects on it, and electrostatic precipitator efficiency may drop if a resistive layer accumulates on the collecting electrode. Thus, aerosol samplers should not be used unless their collection is essentially complete for all particle sizes of interest.

Determination of Sample Stability and Recovery. The collection efficiency of a sampler can be defined by the fraction removed from the air passing through it. However, the material collected cannot always be completely recovered from the sampling substrate for analysis. In addition, the material can sometimes be degraded or otherwise lost between the time of collection in the field and recovery in the laboratory. Deterioration of the sample is particularly severe for chemically reactive materials. Sample losses may also be due to high vapor pressures in the sampled material, exposure to elevated temperatures, or to reactions between the sample and substrate or between different components in the sample.

Laboratory calibrations using blank and spiked samples should be performed whenever possible to determine the conditions under which such losses are likely to affect the determinations desired. When the losses are likely to be excessive, the sampling equipment or procedures should be modified as much as feasible to minimize the losses and the need for calibration corrections. Calibration of Sensor Response. When calibrating direct-reading instruments, the objective is to determine the relationship between the scale readings and the actual concentration of contaminant present. In such tests the basic response for the contaminant of interest is obtained by operating the instrument in known concentrations of the pure material over an appropriate range of concentrations. In many cases it is also necessary to determine the effect of environmental co-factors such as temperature, pressure and humidity on the instrument response. Also, many sensors are non-specific and atmospheric co-contaminants may either elevate or depress the signal produced by the contaminant of interest. If reliable data on the effect of such interferences are not available, they should be obtained in calibration tests. Procedures for establishing known concentrations for such calibration tests are discussed in detail in Chapter 12.

Sampling and Calibration Standards and Errors Use and Reliability of Standards and Standard Procedures. Calibration procedures generally involve a comparison of instrument response to a standardized atmosphere or to the response of a reference instrument. Hence, the calibration can be no better than the standards used. Reliability and proper use of standards are critical to accurate calibrations. Reference materials and instruments available from, or calibrated by, the National Bureau of Standards (NBS) should be used whenever possible. Information on calibration aids

available from NBS is summarized in Table 11-1.

TABLE 11-1
NATIONAL BUREAU OF STANDARDS (NBS) — STANDARD REFERENCE MATERIALS
(SRM's)* INTENDED FOR USE AS PRIMARY INSTRUMENT CALIBRATION
STANDARDS BY AIR POLLUTION LABORATORIES

SRM No.	Description				
1625	SO, Permeation Tube, Individually Calibrated, Effective length ≈ 10 cm, Permeation Rate ≈ 0.28 ug SO,/min. @ 25°C.				
1626	Same as above, except that effective length ≈ 5 cm.				
1627	Same as above, except that effective length ≈ 2 cm.				
1610	Hydrocarbon in Air compressed gas mixture, 68 standard liters @ 500 psi in disposable cylinder. Concentration $= 0.103 \pm 0.001$ mole percent, calculated as methane, as determined by flame ionization.				
1611	Same as above, except that concentration = 0.0107 ± 0.0001 mole %.				
1612	Same as above, except that concentration = 0.00117 ± 0.00001 mole %.				
1613	Same as above, except that concentration = 0.000102 ± 0.000002 mole %.				
1601	Carbon Dioxide in Nitrogen compressed gas mixture, 68 standard liters @ 500 psi in disposable cylinder. Concentration — 0.0308 ± 0.0003 mole %.				
1602	Same as above, except that concentration = 0.0346 ± 0.0003 mole %.				
1603	Same as above, except that concentration = 0.0384 ± 0.0004 mole %.				

^{*} Available from the Office of Standard Reference Materials, Room B 314, Chemistry Bldg., National Bureau of Standards, Washington, D. C. 20234.

Test atmospheres generated for the purpose of calibrating collection efficiency or instrument response should be checked for concentration using reference instruments or sampling and analytical procedures whose reliability and accuracy are well documented. The best procedures to use

are those which have been referee or panel tested; i.e., methods which have been shown to yield comparable results on blind samples analyzed by different laboratories. Such procedures are published by several organizations, which are listed in Table 11-2. Those published by the individual

TABLE 11-2
ORGANIZATIONS PUBLISHING RECOMMENDED OR STANDARD METHODS AND/OR TEST PROCEDURES APPLICABLE TO AIR SAMPLING INSTRUMENT CALIBRATION

Abbreviation	Full Name	Mailing Address
APCA	Air Pollution Control Association	4400 Fifth Avenue Pittsburgh, Pa. 15213
ACGIH	American Conference of Governmental Industrial Hygienists	P.O. Box 1937 Cincinnati, Ohio 45201
AIHA	American Industrial Hygiene Association	66 South Miller Rd. Akron, Ohio 44313
ANSI	American National Standards Institute, Inc.	1430 Broadway New York, N. Y. 10018
ASTM	American Society for Testing and Materials, D-22 Committee on Sampling and Analysis of Atmospheres	1016 Race Street Philadelphia, Pa. 19103
EPA	Environmental Protection Agency Office of Air Programs	5600 Fischer's Lane Rockville, Md. 20852
ISC	Intersociety Committee on Methods for Air Sampling and Analysis New York	

TABLE 11-3
SUMMARY OF RECOMMENDED AND STANDARD METHODS RELATING TO AIR
SAMPLING INSTRUMENT CALIBRATION

Organization	No. of Methods	Types of Methods	Panel Tested	Reference
ACGIH	19	Analytic methods for air contaminants	Yes	Manual of Analytic Methods(12)
AIHA	117	Analytic methods for air contaminants	No	Analytic Guides(14)
ISC	46	Analytic methods for air contaminants	†	Health Laboratory Science 6(2) (Apr. 1969) 7(1) (Jan. 1970) 7(2) (July 1970) 7(4) (Oct. 1970) 8(1) (Jan. 1971)
ASTM	20	Analytic methods for air contaminants	*	Part 23, Annual Book of
ASTM	5	Recommended practices for sampling procedures, nomenclature, etc.	NA	ASTM Standards(15)
APCA	3	Recommended standard methods for continuing air monitoring for fine particulate matter	NA	J. Air Pollut. Cont. Assoc. 13:55 (Sept. 1963)
ANSI	1	Sampling airborne radioactive materials	NA	ANSI N 13.1-1969
EPA	6	Reference methods for air contaminants	No	Fed. Register 36(84) (April 30, 1971)

[†] All methods will be panel tested before advancing from tentative to standard.

^{*} Seven methods are undergoing panel validation under Phase 1-ASTM Project Threshold. Additional methods will be panel evaluated in subsequent phases.

NA Not applicable.

organizations have been supplemented in recent years by those approved by the Intersociety Committee on Methods for Air Sampling and Analysis, a cooperative group formed in March, 1963, composed of representatives of the Air Pollution Control Association (APCA), the American Conference of Governmental Industrial Hygienists (ACGIH), the American Industrial Hygiene Association (AIHA), the American Public Health Association (APHA), the American Society for Testing and Materials (ASTM), the American Society of Mechanical Engineers (ASME), and the Association of Official Analytical Chemists (AOAC). "Tentative" methods endorsed by the Intersociety Committee have been published at random intervals since April, 1969, in "Health Laboratory Science," a publication of APHA. These "Tentative" methods become "Standard" methods only after satisfactory completion of a cooperative test program. Lists of published "Tentative" and "Standard" methods for air sampling and analysis are summarized in Table 11-3.

Sources of Sampling and Analytical Errors. The difference between the air concentration reported for an air contaminant on the basis of a meter reading or laboratory analysis, and true concentration at that time and place represents the error of the measurement. The overall error is often due to a number of smaller component errors rather than to a single cause. In order to minimize the overall error it is usually necessary to analyze each of its potential components, and concentrate one's efforts on reducing the component error which is largest. It would not be productive to reduce the uncertainty in the analytical procedure from 10% to 1.0% when the error associated with the sample volume measurement is $\pm 15\%$

Sampling problems are so varied in practice that it is only possible to generalize on the likely sources of error to be encountered in typical sampling situations. In analyzing a particular sampling problem, consideration should be given to each of the following:

- a) Flowrate and sample volume
- b) Collection efficiency
- c) Sample stability under conditions anticipated for sampling, storage and transport
- d) Efficiency of recovery from sampling substrate
- e) Analytical background and interferences introduced by sampling substrate
- f) Effect of atmospheric co-contaminants on samples during collection, storage and analyses.

Cumulative Statistical Error. The most probable value of the cumulative error E_c can be calculated from the following equation:

$$E_c = [E_1^2 + E_2^2 + E_2^2 + \dots + E_n^2]^{1/2}$$

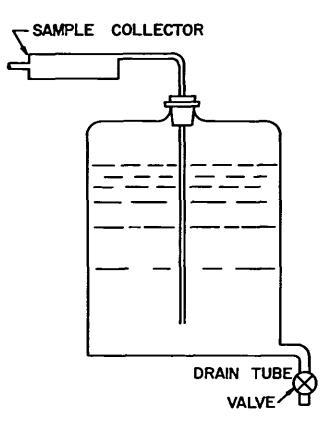
For example, if accuracies of the flowrate measurement, sampling time, recovery, and analysis are \pm 15, 2, 10, and 10% respectively, and there are no other significant sources of error, then the cumulative error would be:

$$E_e = [15^2 + 2^2 + 10^2 + 10^2] - [429]^{1/2} - \pm 20.6\%$$

It should be remembered that this provides an estimate of the deviation of the measured concentration from the true concentration at the time and place the sample was collected. As an estimate of the average concentration to which a workman was exposed in performing a given operation, it would have additional uncertainty, dependent upon the variability of concentration with time and space at the work station.

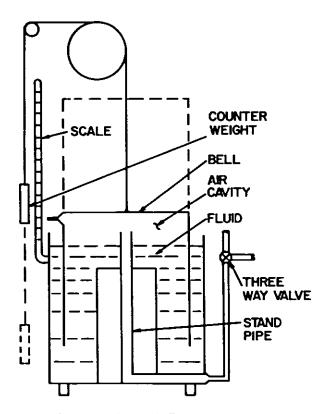
CALIBRATION INSTRUMENTS AND TECHNIQUES FOR FLOW AND VOLUME CALIBRATION

In this section, the various techniques used for measuring sampling rate or sampled volume in samplers and in laboratory calibrations of samplers will be discussed in terms of their principles of operation and their sources of error. Some may be considered primary measurements, while some are secondary or derived. Primary measurements generally involve a direct measurement of volume on the basis of the physical dimensions of an enclosed space. Secondary standards are reference instruments or meters which trace their calibrations to primary standards, and which have been shown to be capable of maintaining their accuracy with reasonable handling and care in operation.



Powell CH, Hosey AD (eds): The Industrial Environment
— Its Evaluation and Control, 2nd Edition. Public
Health Service Publication No. 614, 1965.

Figure 11-1. Marriotti Bottle



Powell CH, Hosey AD (eds): The Industrial Environment
— Its Evaluation and Control, 2nd Edition. Public
Health Service Publication No. 614, 1965.

Figure 11-2. Schematic Drawing of a Spirometer

Instruments which Measure or Are Calibrated in Volume Units

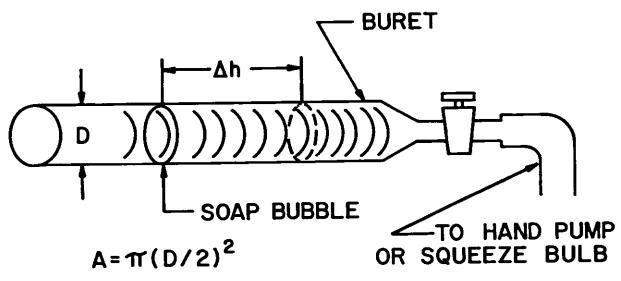
Water Displacement. Figure 11-1 shows a sche-

matic drawing of a Mariotti bottle. When the valve at the bottom of the bottle is opened, water drains out of the bottle by gravity, and air is drawn via a sample collector into the bottle to replace it. The volume of air drawn in is equal to the change in water level multiplied by the cross section at the water surface. The Casella Standard Thermal Precipitation uses a water-filled aspirator with an orifice at the discharge end of the cylinder which limits the flowrate to 7 cm³/min. ¹

Spirometer or Gasometer. The spirometer (Figure 11-2) is a cylindrical bell with its open end under a liquid seal. The weight of the bell is counterbalanced so that the resistance to movement as air moves in or out of the bell is negligible. It differs from the Mariotti bottle in that it measures displaced air instead of displaced liquid. The volume change is calculated in a similar manner, i.e., change in height times cross section. Spirometers are available in a wide variety of sizes and are frequently used as primary volume standards.

"Frictionless" Piston Meters. Cylindrical air displacement meters with nearly frictionless pistons are frequently used for primary flow calibrations. The simplest version is the soap-bubble meter illustrated in Figure 11-3. It utilizes a volumetric laboratory buret whose interior surfaces are wetted with a detergent solution. If a soap-film bubble is placed at the left side, and suction is applied at the right, the bubble will be drawn from left to right. The volume displacement per unit time (i.e., flowrate) can be determined by measuring the time required for the bubble to pass between two scale markings which enclose a known volume.

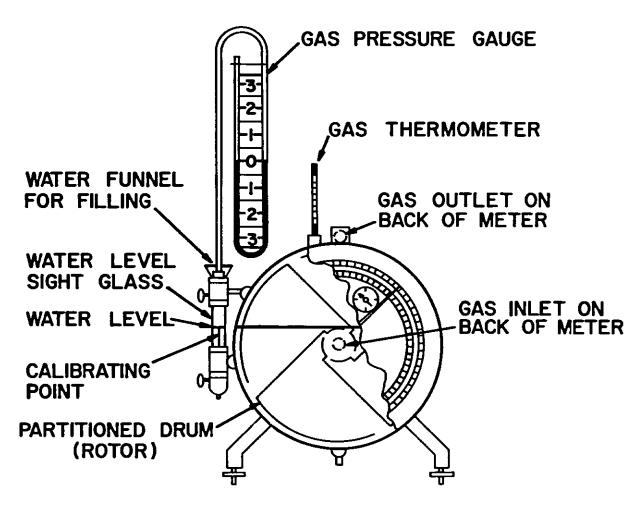
Soap-film flowmeters and mercury-sealed piston flowmeters are available commercially from several sources. (1) In the mercury-sealed piston, most of the cylindrical cross section is blocked off



Powell CH, Hosey AD (eds): The Industrial Environment — Its Evaluation and Control, 2nd Edition. Public Health Service Publication No. 614, 1965.

Figure 11-3. Bubble Meter

by a plate which is perpendicular to the axis of the cylinder. The plate is separated from the cylinder wall by an 0-ring of liquid mercury which retains its toroidal shape due to its strong surface tension. This floating seal has a negligible friction loss as the plate moves up and down.



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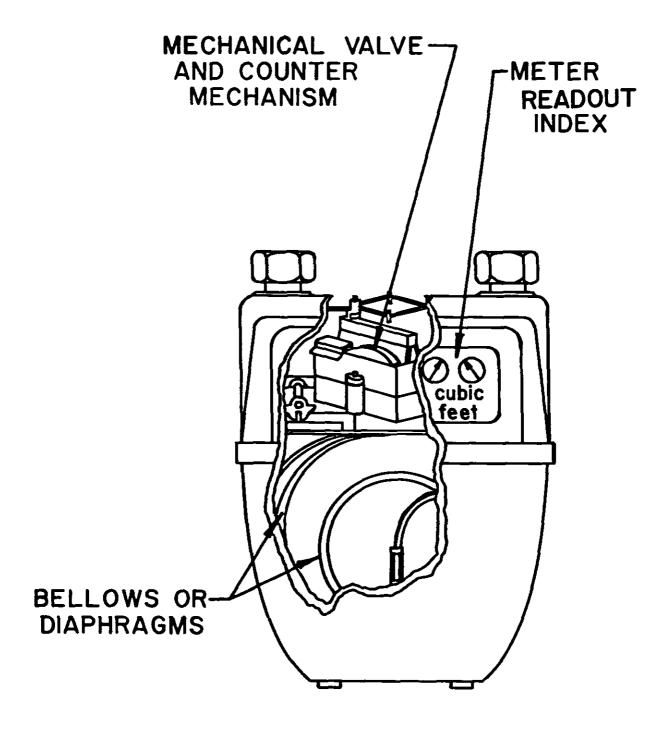
Figure 11-4. Wet Test Meter

Wet-Test Meter. A wet-test meter (See Figure 11-4) consists of a partitioned drum half submerged in a liquid (usually water) with openings at the center and periphery of each radial chamber. Air or gas enters at the center and flows into an individual compartment causing it to rise, thereby producing rotation. This rotation is indicated by a dial on the face of the instrument. The volume measured will be dependent on the fluid level in the meter since the liquid is displaced by air. A sight gauge for determining fluid height is provided and the meter may be leveled by screws and a sight bubble which are provided for this purpose.

There are several potential errors associated with the use of a wet-test meter. The drum and moving parts are subject to corrosion and damage from misuse, there is friction in the bearings and the mechanical counter, inertia must be overcome at low flows (<1 RPM), while at high flows (>3 RPM), the liquid might surge and break the

water seal at the inlet or outlet. In spite of these factors, the accuracy of the meter usually is within one percent when used as directed by the manufacturer.

Dry-Gas Meter. The dry-gas meter shown in Figure 11-5 is very similar to the domestic gas meter. It consists of two bags interconnected by mechanical valves and a cycle-counting device. The air or gas fills one bag while the other bag empties itself; when the cycle is completed the valves are switched, and the second bag fills while the first one empties. Any such device has the disadvantage of mechanical drag, pressure drop, and leakage; however, the advantage of being able to use the meter under rather high pressures and volumes often outweighs these errors, which can be determined for a specific set of conditions. The alternate filling of two chambers as the basis for volume measurement is also used in twin-cylinder piston meters. Such meters can also be classified



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Figure 11-5. Dry Gas Meter

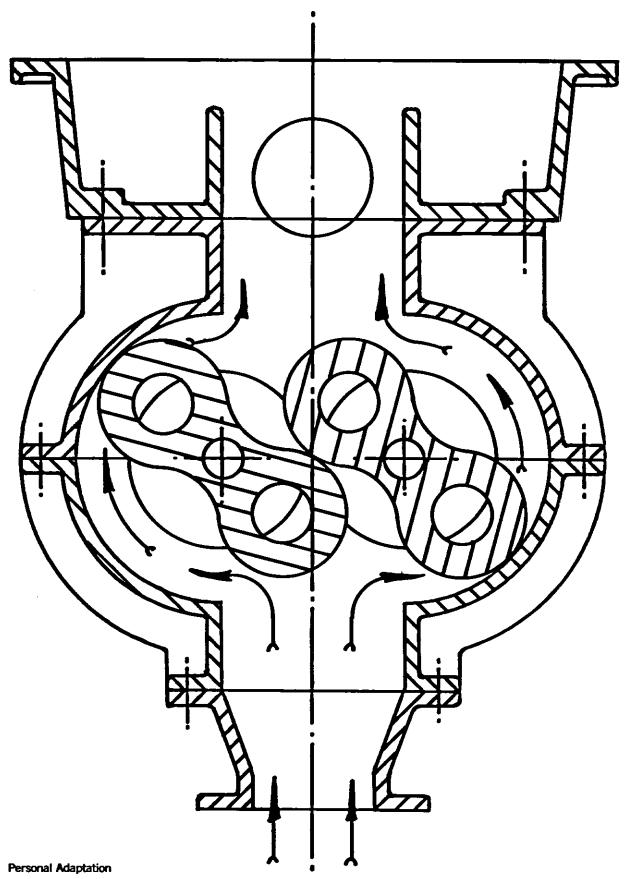


Figure 11-6. Schematic Diagram Showing Principle of Operation of Twin-Lobed Positive Displacement Meter

as positive displacement meters,

Positive Displacement Meters. Positive displacement meters consist of a tight-fitting moving element with individual volume compartments which fill at the inlet and discharge at the outlet parts. A lobed rotor design is illustrated in Figure 11-6. Another multicompartment continuous rotary meter uses interlocking gears. When the rotors of such meters are motor driven, these units become positive displacement air movers.

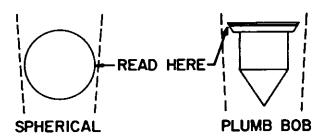
Volumetric Flowrate

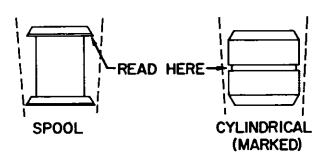
The volume meters discussed in the preceding paragraphs were all based on the principle of conservation of mass; specifically the transfer of a fluid volume from one location to another. The flowrate meters in this section all operate on the principle of the conservation of energy; more specifically, they utilize Bernoulli's theorem for the exchange of potential energy for kinetic energy and/or frictional heat. Each consists of a flow restriction within a closed conduit. The restriction causes an increase in the fluid velocity, and therefore an increase in kinetic energy, which requires a corresponding decrease in potential energy, i.e., static pressure. The flowrate can be calculated from a knowledge of the pressure drop, the flow cross section at the constriction, the density of the fluid, and the coefficient of discharge, which is the ratio of actual flow to theoretical flow and makes allowance for stream contraction and frictional effects.

Flowmeters which operate on this principle can be divided into two groups. The larger group, which includes orifice meters, venturi meters, and flow nozzles have a fixed restriction and are known as variable-head meters, because the differential pressure head varies with flow. The other group, which includes rotameters, are known as variable-area meters, because a constant pressure differential is maintained by varying the flow cross section.

Variable-Area Meters (Rotameters). A rotameter consists of a "float" which is free to move up and down within a vertical tapered tube which is larger at the top than the bottom. The fluid flows upward, causing the float to rise until the pressure drop across the annular area between the float and the tube wall is just sufficient to support the float. The tapered tube is usually made of glass or clear plastic and has a flowrate scale etched directly on it. The height of the float indicates the flowrate. Floats of various configurations are used, as indicated in Figure 11-7. They are conventionally read at the highest point of maximum diameter, unless otherwise indicated.

Most rotameters have a range of 10:1 between their maximum and minimum flows. The range of a given tube can be extended by using heavier or lighter floats. Tubes are made in sizes from about 1/8 to 6 inches in diameter, covering ranges from a few cm³/min. to over 1,000 ft³/min. Some of the shaped floats achieve stability by having slots which make them rotate, but these are less commonly used than previously. The term "rotameter" was first used to describe such meters with





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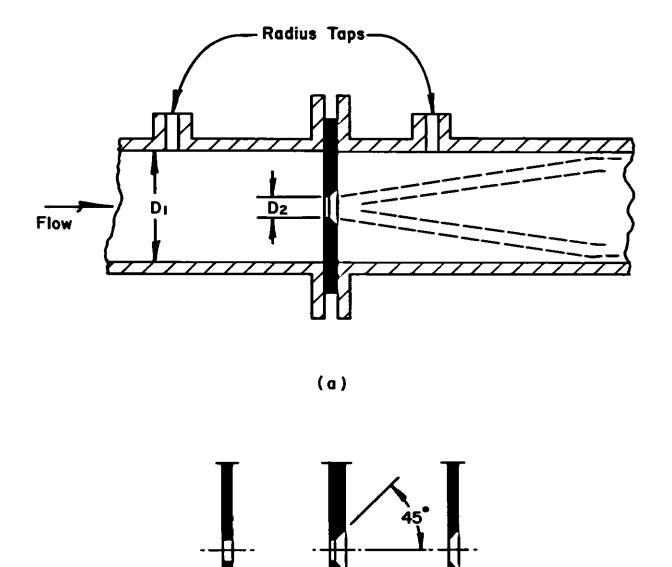
Figure 11-7. Typical Rotameter Floats

spinning floats, but now is generally used for all types of tapered metering tubes.

Rotameters are the most commonly used flowmeters on commercial air samplers, especially on portable samplers. For such sampler flowmeters, the most common material of construction is acrylic plastic, although glass tubes may also be used. Because of space limitations, the scale lengths are generally no more than four inches and most commonly nearer to two inches. Unless they are individually calibrated, the accuracy is unlikely to be better than $\pm 25\%$. When individually calibrated, ± 5% accuracy may be achieved. It should be noted, however, that with the large taper of the bore, the relatively large size of the float, and the relatively few scale markers on these rotameters, the precision of the readings may be a major limiting factor.

Calibrations of rotameters are performed at an appropriate reference pressure, usually atmospheric. However, since good practice dictates that the flowmeter should be downstream of the sample collector or sensor, the flow is actually measured at a reduced pressure, which may also be a variable pressure if the flow resistance changes with loading. If this resistance is constant, it should be known; if variable, it should be monitored, so that the flowrate can be adjusted as needed, and appropriate pressure corrections can be made for the flowmeter readings.

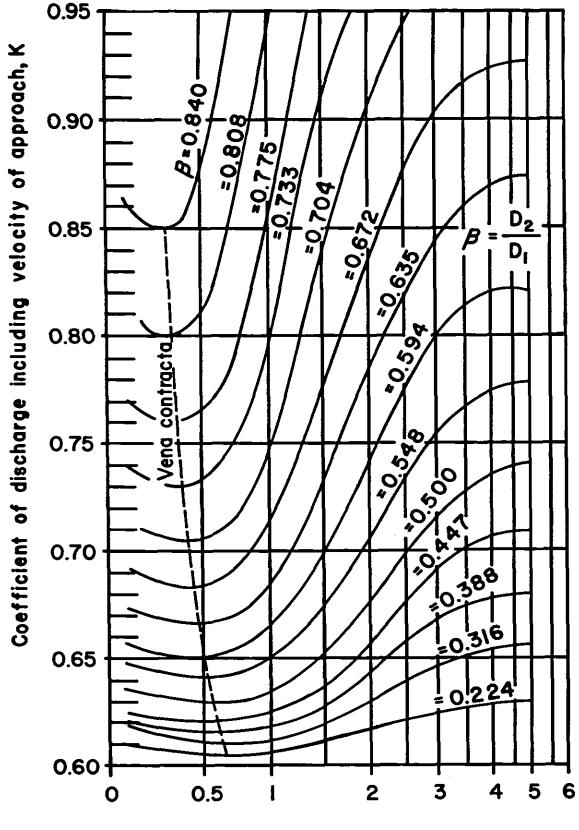
Variable-Head Meters. When orifice and venturi meters are made to standardized dimensions, their calibration can be predicted with $\sim \pm 10\%$



(b)

Perry JH, et al: Chemical Engineering Handbook, 4th Edition. New York, McGraw-Hill, 1963.

Figure 11-8. Square-Edged or Sharp-Edged Orifices. The plate at the orifice opening must not be thicker than 1/30 of pipe diameter, 1/8 of the orifice diameter, or 1/4 of the distance from the pipe wall to the edge of the opening. (a) Pipe-line orifice. (b) Types of plates.



Downstream pressure tap location in pipe diameters

Perry JH, et al: Chemical Engineering Handbook, 4th Edition. New York, McGraw-Hill, 1963.

Figure 11-9. Coefficient of Discharge for Square-Edged Circular Orifices for $N_{\rm Bez}{}^{>}30,000$ with the Upstream Tap Location between One and Two Pipe Diameters from the Orifice Position

accuracy using standard equations and published empirical coefficients. The general equation² for this type of meter is:

W =
$$q_i p_i = KYA_2 \quad \sqrt{2g_c (P_1 - P_2)\rho_i}$$
 (1)
where: K = C/ $\sqrt{1 - \beta^2}$

C = coefficient of discharge, dimensionless

 A_2 = cross-sectional area of throat — ft^2

 $g_c = 32.17 \text{ ft/sec}^2$

P₁ = upstream static pressure - Ib/ft²

 $P_z = downstream static pressure — Ib/ft^2$

q₁ — volumetric flow at upstream press. & temp.-ft²/sec.

W - weight-rate of flow - Ib/sec.

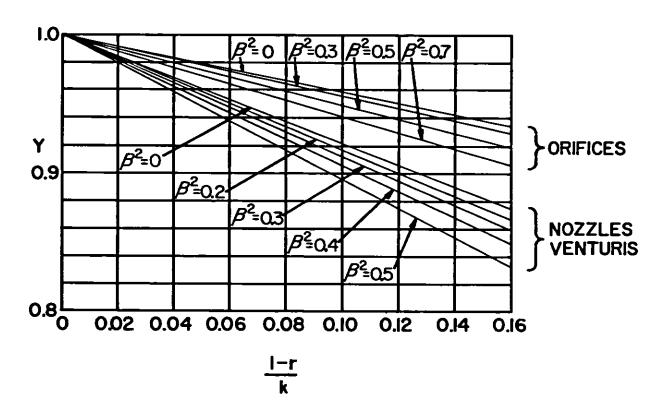
Y = expansion factor (see Figure 11-10)

 β = ratio of throat diameter to pipe diameter, dimensionless

Orifice Meters. The simplest form of variablehead meter is the square-edged, or sharp-edged orifice illustrated in Figure 11-8. It is also the most widely used because of its ease of installation and low cost. If it is made with properly mounted pressure taps, its calibration can be determined from equation (1) and Figures 11-9 and 11-10. However, even a non-standard orifice meter can serve as a secondary standard, provided it is carefully calibrated against a reliable reference instrument.

While the square-edged orifice can provide accurate flow measurements at low cost, it is inefficient with respect to energy loss. The permanent pressure loss for an orifice meter with radius taps can be approximated by $(1 - \beta^2)$, and will often exceed 80%.

Venturi Meters. Venturi meters have optimal converging and diverging angles of about 25° and 7° respectively, and thereby have high pressure re-



Perry JH, et al: Chemical Engineering Handbook, 4th Edition. New York, McGraw-Hill, 1963.

Figure 11-10. Values of Expansion Factor Y for Orifices, Nozzles, and Venturis

coveries, i.e., the potential energy which is converted to kinetic energy at the throat is reconverted to potential energy at the discharge, with an overall loss of only about 10%.

For air at 70°F and 1 atm. and for $\frac{1}{4} < \beta < \frac{1}{2}$, a standard venturi would have a calibration described by:

$$Q = 21.2 \, \beta^2 \, D^2 \, \sqrt{h}$$
 (2)

where $Q = flow - ft^3/min$.

β — ratio of throat to duct diameter, dimensionless

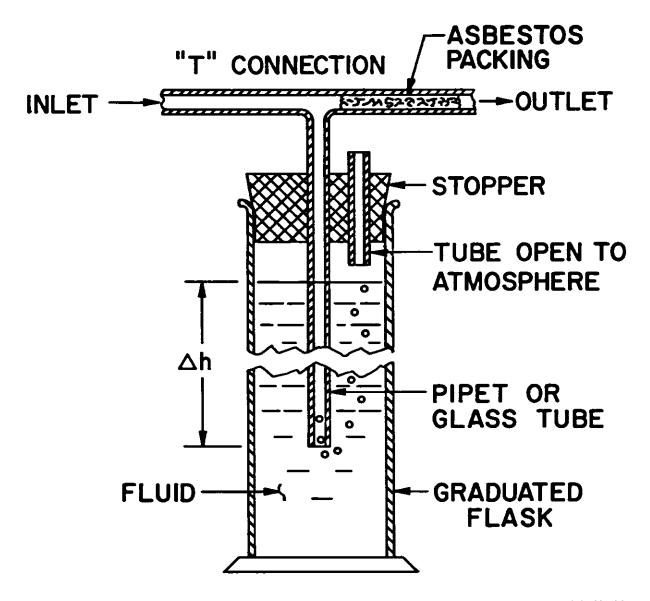
D = duct diameter - inches

h - differential pressure - inches of water.

Other Variable-Head Meters. The characteristics of various other types of variable-head flowmeters, e.g., flow nozzles, Dall Tubes, centrifugal flow elements, etc. are described in various standard engineering references.²⁻³ In most respects they have similar properties to the orifice meter, venturi meter, or both.

One type of variable-head meter which differs significantly from all of the above is the laminarflow meter. These are seldom discussed in engineering handbooks because they are used only for very low flowrates. Since the flow is laminar, the pressure drop is directly proportional to the flowrate. In orifice meters, venturi meters and related devices, the flow is turbulent and flowrate varies with the square root of the pressure differential.

Laminar flow restrictors used in commercial flowmeters consist of egg-crate or tube bundle arrays of parallel channels. Alternatively, a laminar flowmeter can be constructed in the laboratory using a tube packed with beads or fibers as the resistance element. Figure 11-11 illustrates this kind of homemade flowmeter. It consists of a "T"



Powell CH, Hosey AD (eds): The Industrial Environment — Its Evaluation and Control, 2nd Edition. Public Health Service Publication No. 614, 1965.

Figure 11-11. Drawing of a Packed Plug Flow Meter

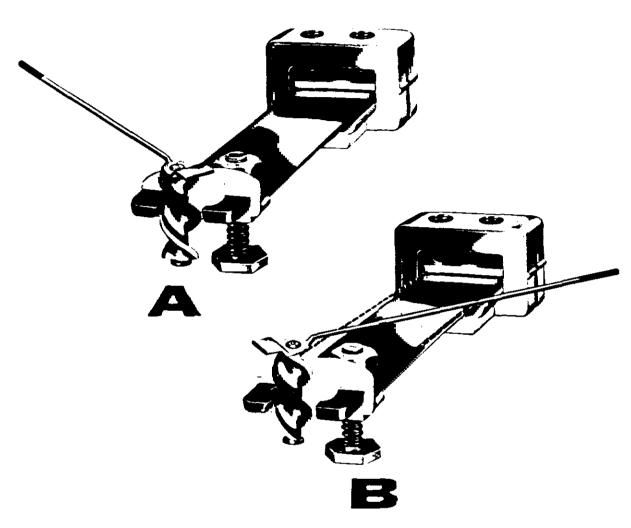
connection, pipet or glass tubing, cylinder and packing material. The outlet arm of the "T" is packed with material, such as asbestos, and the leg is attached to a tube or pipet projecting down

into the cylinder filled with water or oil. A calibration curve of the depth of the tube outlet below the water level versus the rate of flow should produce a linear curve. Saltzman has used tubes

filled with asbestos to regulate and measure flow-rates as low as 0.01 cm³/min.

Pressure Transducers. All of the variable-head meters require a pressure sensor, sometimes referred to as the secondary element. Any type of pressure sensor can be used, although high cost and fragility usually rule out the use of many electrical and electro-mechanical transducers.

Liquid-filled manometer tubes are sometimes used, and if they are properly aligned and the density of the liquid is accurately known, the column differential provides an unequivocal measurement. In most cases however, it is not feasible to use liquid-filled manometers in the field, and the pressure differentials are measured with mechanical gages with scale ranges in centimeters or inches



Dwyer Instruments, Inc.: Bulletin #A-20. Michigan City, Indiana.

Figure 11-12. How the Magnetic Linkage Works*

*From I. W. Dwyer Co. Literature

12A—At zero position, pressures on both sides of the diaphragm are equal. The support plates of the diaphragm are connected to the leaf spring which is anchored at one end. The horseshoe magnet attached to the tree end of the spring straddles the axis of a helix but does not touch the helix. The indicating pointer is attached to one end of the helix.

The helix, being of high magnetic permeability, aligns itself in the field of the magnet to maintain the minimum air gap between the magnet's poles and the outer edge of the helix.

12B—When pressure on the "high" side of the diaphragm increases or pressure on the "low" side of the diaphragm decreases, the diaphragm moves toward the back of the case. Through the linkage, the diaphragm moves the spring and the magnet. As the magnet moves parallel to the axis of the helix, the helix turns to maintain the minimum air gap.

Movement of the diaphragm is resisted by the flat spring which determines the range of the instrument. Precise calibration is achieved by varying the live length of the spring through adjustment of the spring clamp.

of water. For these low pressure differentials the most commonly used gage is the Magnehelic[®], whose schematic is illustrated in Figures 11-12A and 11-12B. These gages are accurate to \pm 2% of full scale and are reliable provided they and their connecting hoses do not leak, and their calibration is periodically rechecked.

Critical-Flow Orifice. For a given set of upstream conditions, the discharge of a gas from a restricted opening will increase with a decrease in the ratio of absolute pressures P_2/P_1 , where P_2 is the downstream pressure, and P_1 the upstream pressure, until the velocity through the opening reaches the velocity of sound. The value of P_2/P_1 at which the maximum velocity is just attained is known as the critical pressure ratio. The pressure in the throat will not fall below the pressure at the critical point, even if a much lower downstream pressure exists. Therefore, when the pressure ratio is below the critical value, the rate of flow through the restricted opening is dependent only on the upstream pressure.

It can be shown,² that for air flowing through rounded orifices, nozzles and venturis, when P_2 < 0.53 P_1 , and $S_1/S_2 > 25$, the mass-flowrate w, is determined by:

$$w = 0.533 \frac{C_x S_z P_1}{T_1} lb/sec$$
 (3)

where: $C_r = \text{coefficient of discharge}$ (normally ~ 1)

 S_1 = duct or pipe cross section in square inches

S₂ = orifice area in square inches

 P_1 = upstream absolute pressure in lb/sq. in.

T₁ = upstream temperature in °R

Critical-flow orifices are widely used in industrial hygiene instruments such as the midget impinger pump and squeeze bulb indicators. They can also be used to calibrate flowmeters by using a series of critical orifices downstream of the flowmeter under test. The flowmeter readings can be plotted against the critical flows to yield a calibration curve.

The major limitation in their use is that the orifices are extremely small when they are used for flows of 1 ft³/min or less. They become clogged or eroded in time and, therefore, require frequent examination and/or calibration against other reference meters.

By-Pass Flow Indicators. In most high-volume samplers, the flowrate is strongly dependent on the flow resistance, and flowmeters with a sufficiently low flow resistance are usually too bulky or expensive. A commonly used metering element for such samplers is the by-pass rotameter, which actually meters only a small fraction of the total flow; a fraction, however, which is proportional to the total flow. As shown schematically in Figure 11-13, a by-pass flowmeter contains both a variable-head element and a variable-area element. The pressure drop across the fixed orifice or flow restrictor creates a proportionate flow through the

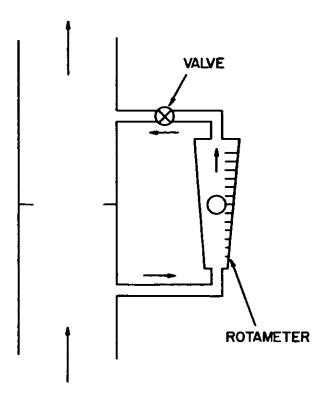


Figure 11-13. Schematic of By-Pass Flow Indicators

parallel path containing the small rotameter. The scale on the rotameter generally reads directly in ft²/min or liters/min of total flow. In the versions used on portable high-volume samplers there is usually an adjustable bleed valve at the top of the rotameter which should be set initially, and periodically readjusted in laboratory calibrations so that the scale markings can indicate overall flow. If the rotameter tube accumulates dirt, or the bleed valve adjustment drifts, the scale readings can depart greatly from the true flows.

Flow Velocity Meters

As discussed previously, point velocity is not the parameter of interest in sampling flow measurements. However, it may be the only feasible parameter to measure in some circumstances, and it usually can be related to flowrate provided the sensor is located in an appropriate position and is suitably calibrated against overall flow.

Velocity Pressure Meters. The Pitot tube is often used as a reference instrument for measuring the velocity of air. A standard Pitot, carefully made, will need no calibration. It consists of an impact tube whose opening faces axially into the flow, and a concentric static pressure tube with 8 holes spaced equally around it in a plane which is 8 diameters from the impact opening. The difference between the static and impact pressures is the velocity pressure. Bernoulli's theorem applied to a Pitot tube in an air stream simplifies to the dimensionless formula

$$V = \sqrt{2g_c P_v} \tag{4}$$

where: V = linear velocity

g = gravitational constant

 P_v = pressure head of flowing fluid or velocity pressure. Expressing V in linear feet per min., P_v in inches of water (h_v) ,

and
$$g_c = 32.17 \frac{\text{(lb.-mass) (ft.)}}{\text{(lb.-force) (sec.}^2)}$$
:

$$V = 1097 \sqrt{\frac{h_v}{\rho}} \tag{5}$$

where: $\rho = \text{density of air or gas in lb./ft.}^2$

If the Pitot tube is to be used with air at standard conditions (70°F and 1 atm.), formula (5) reduces to:

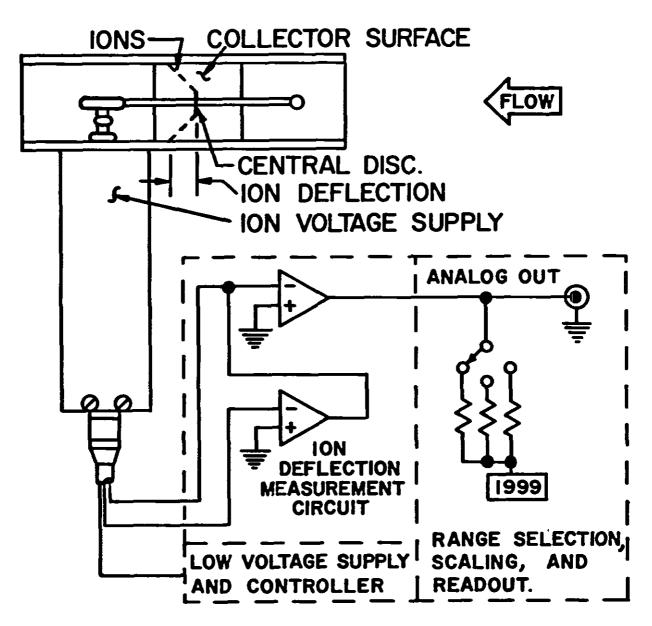
$$V = 4005 \sqrt{h_{\tau}} \tag{6}$$

where: V = velocity in ft./min.

 $h_v = velocity$ pressure in inches of H_vO

There are several serious limitations to Pitot tube measurements in most sampling flow calibrations. One is that it may be difficult to obtain or fabricate a small enough probe. Another is that the velocity pressure may be too low to measure at the velocities encountered. For example, at 1000 ft./min., $h_v = 0.063 \text{ inches of water, a low value, even for an inclined manometer.}$

Heated Element Anemometers. Any instrument used to measure velocity can be referred to as an anemometer. In a heated element anemometer, the flowing air cools the sensor in proportion to the velocity of the air. Instruments are available with various kinds of heated elements, i.e., heated thermometers, thermocouples, films, and wires.



Thermo-Systems, Inc.: leaflet "TSI-54100-671." St. Paul, Minnesota.

Figure 11-14. Ion-Flow Mass Flowmeter

They are all essentially nondirectional, i.e., with single element probes, they measure the airspeed but not its direction. They all can accurately measure steady state airspeed, and those with low mass sensors and appropriate circuits can also accurately measure velocity fluctuations with frequencies above 100,000 Hz. Since the signals produced by the basic sensors are dependent on ambient temperature as well as air velocity, the probes are usually equipped with a reference element which provides an output which can be used to compensate or correct errors due to temperature variations. Some heated element anemometers can measure velocities as low as 10 ft./min. and as high as 8,000 ft./min.

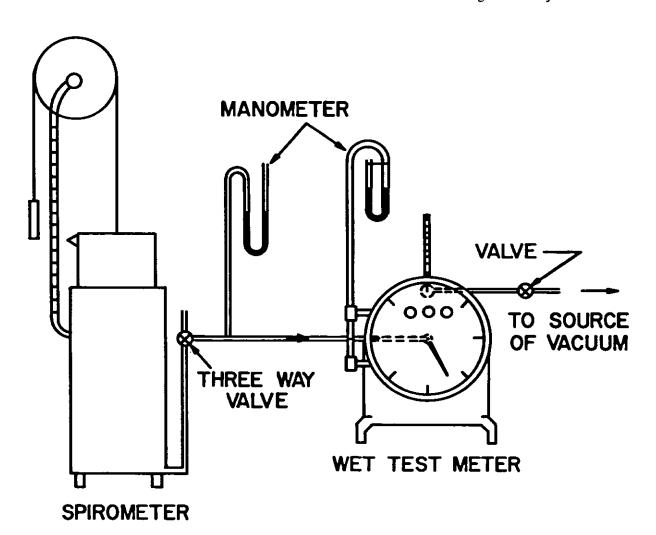
Other Velocity Meters. There are several other ways to utilize the kinetic energy of a flowing fluid to measure velocity beside the Pitot tube. One way is to align a jeweled-bearing turbine wheel axially in the stream and count the number of rotations per unit time. Such devices are generally known as rotating vane anemometers. Some are very small and are used as velocity probes. Others are sized to fit the whole duct and become

indicators of total flowrate and sometimes are called turbine flowmeters.

The velometer, or swinging vane anemometer described in Chapter 40, is widely used for measuring ventilation air flows, but has few applications in sample flow measurement or calibration. It consists of a spring-loaded vane whose displacement is indicative of velocity pressure.

Mass Flow and Tracer Techniques

Thermal Meters. A thermal meter measures mass air or gas flow rate with negligible pressure loss. It consists of a heating element in a duct section between two points at which the temperature of the air or gas stream is measured. The temperature difference between the two points is dependent on the mass rate of flow and the heat input. Mixture Metering. The principle of mixture metering is similar to that of thermal metering. Instead of adding heat and measuring temperature difference, a contaminant is added and its increase in concentration is measured; or clean air is added and the reduction in concentration is measured. This method is useful for metering corrosive gas streams. The measuring device may react to some



Powell CH, Hosey AD (eds): The Industrial Environment — Its Evaluation and Control. 2nd Edition. Public Health Service Publication No. 614, 1965.

Figure 11-15. Calibration Setup for Calibrating a Wet Test Meter

physical property such as thermal conductivity or vapor pressure.

Ion-Flow Meters. In the ion-flow meter illustrated in Figure 11-14, ions are generated from the central disc and flow radially toward the collector surface. Airflow through the cylinder causes an axial displacement of the ion stream in direct proportion to the mass flow. The instrument can measure mass flows from 0.1 to 150 standard ft.3/min., and velocities from 1 ft./min. to 12,000 ft./min.

Procedures for Calibrating Flow and Volume Meters

In the limited space available, it is not possible to provide a complete description of all of the techniques available, or to go into great detail on those which are commonly used. This discussion will be limited to selected procedures which should serve to illustrate recommended approaches to some commonly encountered calibration procedures.

Comparison of Primary and Secondary Standards. Figure 11-15 shows the experimental set-up for checking the calibration of a secondary standard (in this case a wet-test meter) against a primary standard (in this case a spirometer). The first step should be to check out all of the system elements for integrity, proper functioning, and interconnections. Both the spirometer and wet-test meter require specific internal water levels and leveling. The operating manuals for each should be examined since they will usually outline simple procedures for leakage testing and operational procedures.

After all connections have been made, it is a good policy to recheck the level of all instruments and determine that all connections are clear and have minimum resistance. If compressed air is used in a calibration procedure it should be cleaned and dried.

Actual calibration of the wet-test meter shown in Figure 11-15 is accomplished by opening the by-pass valve and adjusting the vacuum source to obtain the desired flowrate. The optimum range of operation is between one and three revolutions per minute. Before actual calibration is initiated the wet-test meter should be operated for several hours in this setup to stabilize the meter fluid as to temperature, absorbed gas, and to work in the bearings and mechanical linkage. After all elements of the system have been adjusted, zeroed and stabilized several trial runs should be made. During these runs, should any difference in pressure be indicated, the cause should be determined and corrected. The actual procedure would be to instantaneously divert the air to the spirometer for a predetermined volume indicated by the wettest meter (minimum of one revolution), or to near capacity of the spirometer, then return to the by-pass arrangement. Readings, both quantity and pressure of the wet-test meter, must be taken and recorded while it is in motion, unless a more elaborate system is set up. In the case of a rate meter, the interval of time that the air is entering the spirometer must be accurately timed. The bell should then be allowed to come to equilibrium before displacement readings are made. A sufficient number of different flowrates are taken to establish the shape or slope of the calibration curve with the procedure being repeated three or more times for each point. For an even more accurate calibration the setup should be reversed so that air is withdrawn from the spirometer. In this way any unbalance due to pressure differences would be cancelled.

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

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

$$V_a = V_1 \times \frac{P_1}{760} \times \frac{273}{T_1}$$
 (7)

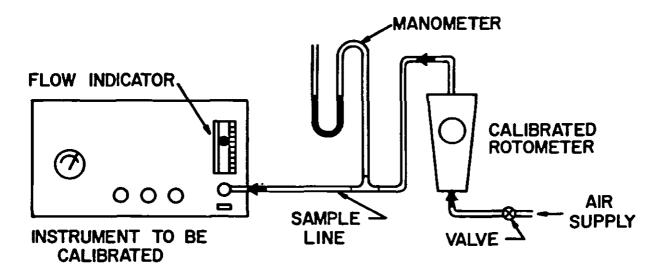
where V_{\bullet} = volume at standard conditions (760 mm & 0°C)

 V_1 = volume measured at conditions P_1 and T_1

 T_1 = temperature of V_1 in °K P_1 = pressure of V_1 in mm Hg

In most cases the water vapor portion of the ambient pressure is disregarded. Also, the standard temperature of the gas is often referred to normal room temperature, i.e., 21°C rather than 0°C. The manipulation of the instruments, data reading and recording, calculations and resulting factors or curves should be done with extreme care. Should a calibration disagree with previous calibrations or the supplier's calibration, the entire procedure should be repeated, and examined carefully to assure its validity. Upon completion of any calibration the instrument should be tagged or marked in a semi-permanent manner to indicate the calibration factor, where appropriate, date and who performed the calibration.

Reciprocal Calibration by Balanced Flow System. In many commercial instruments it is impractical to remove the flow-indicating device for calibration. This may be because of physical limitations, characteristics of the pump, unknown resistance in the systems or other limiting factors. In such situations it may be necessary to set up a reciprocal calibration procedure, that is, where a controlled flow of air or gas is compared first with the instrument flow, then with a calibration source. Often a further complication is introduced by the static pressure characteristics of the air mover in the instrument. In such instances supplemental pressure or vacuum must be applied to the system to offset the resistance of the calibrating device. An example of such a system is illustrated in Figure 11-16.



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Figure 11-16. Setup for Balanced Flow Calibration

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

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

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

$$C_t = C_0 e^{bt} \tag{8}$$

where: C_t = concentration of agent in vessel at time, t

 C_0 = initial concentration at t = 0

e = base of natural logarithms

b = air changes in the vessel per unit time

t - time

The concentration of the gas in the vessel is determined periodically by an independent method. A linear plot should result from plotting concen-

tration of agent against elapsed time on semi-logpaper. The slope of the line indicates the air changes per minute (b) which can be converted to the rate (Q) of air withdrawn by the instrument from the following relationship: Q = bV; where V — volume of the vessel.

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

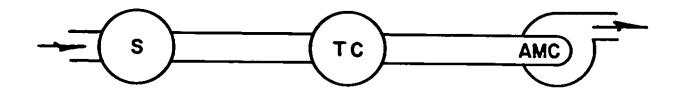
CALIBRATION OF SAMPLER'S COLLECTION EFFICIENCY

Use of Well Characterized Test Atmospheres

In order to test the collection efficiency of a sampler for a given contaminant it is necessary either: 1) to conduct the test in the field using a proven reference instrument or technique as a reference standard, or 2) to reproduce the atmosphere in a laboratory chamber or flow system. Techniques and equipment for producing such test atmospheres are beyond the scope of this chapter. They are discussed in detail in Chapter 12 and in various other sources. **10**11**11** In the discussion to follow, it will be assumed that appropriate test atmospheres are available.

Analysis of Sampler's Collection and Downstream Total Collector

The best approach to use, when it is feasible, is to operate the sampler under test in series with a downstream total collector, as illustrated in Figure 11-17. The sampler's efficiency is then determined by the ratio of the sampler's retention to the retention in the sampler and downstream collector combined. When the penetration is estimated from downstream samples there may be additional errors if the samples are not representative.



S=Sample Under Test
TC=Total Collector
AMC= Air Mover, Flowmeter
and Flow Control

Figure 11-17. Sampler Efficiency Evaluation with Downstream Total Collector: Analysis of Collections in S and TC

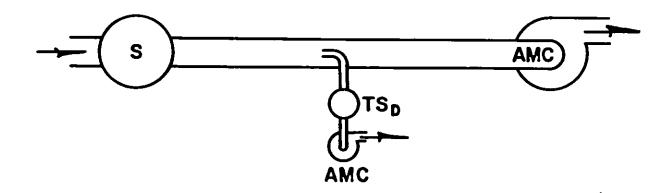
Analysis of Sampler's Collection and Downstream Samples

In some situations it is not possible or feasible to quantitatively collect all of the test material which penetrates the sampler being evaluated. For example, a total collector might add too much flow resistance to the system, or be too bulky for efficient analysis. In this case, the degree of penetration can be estimated from an analysis of a sample of the downstream atmosphere, as illus-

trated in Figure 11-18. When this approach is used, it may be necessary to collect a series of samples across the flow profile rather than a single sample, in order to obtain a true average concentration of the penetrating atmosphere.

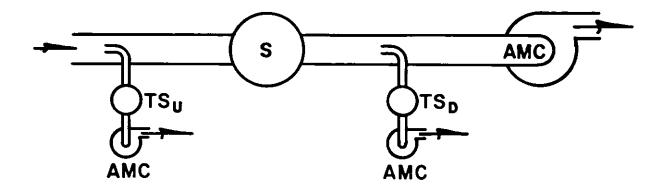
Analysis of Up-and Downstream Samples

In some cases, it may not be possible to recover or otherwise measure the material trapped within elements of the sampling train such as sampling probes. The magnitude of such losses



TS_D= Downstream Sampler Total Collector

Figure 11-18. Sampler Efficiency Evaluation with Downstream Concentration Sampler: Analysis of Collections in S and TSp



TS_U= Upstream Sampler Total Collector

Figure 11-19. Sampler Efficiency Evaluation with Upstream and Downstream Concentration Samplers: Analysis of Collections in TS_u and TS_D

can be determined by comparing the concentrations up-and downstream of the elements in question as illustrated schematically in Figure 11-19.

DETERMINATION OF SAMPLE STABILITY AND/OR RECOVERY

For trace contaminants the stability and recovery from sampling substrates are difficult to predict or control. Thus, these factors are best explored by realistic calibration tests.

Analysis of Sample Aliquots at Periodic Intervals after Sample Collection

If the sample is divided into a number of aliquots which are analyzed individually at periodic intervals, it is possible to determine the long term rate of sample degradation or any tendency for reduced recovery efficiencies with time. These analyses would not however provide any information or losses which may have occurred during or immediately after collection which had different rate constants. Such losses should be investigated using spiked samples.

Analysis of Spiked Samples

If known amounts of the contaminants of interest are intentionally added to the sample substrate, then subsequent analysis of sample aliquots will permit calculation of sample recovery efficiency and rate of deterioration. These results will be valid only insofar as the added material is equivalent in all respects to the material in the ambient air. There are two basic approaches to spiked sample analyses: 1) the addition of known quantities to blank samples, and 2) the addition of radioactive isotopes to either blank or actual field collected samples.

When the material being analyzed is available in tagged form, the tag can be added to the sample in negligible or at least known low concentrations. If there are losses in sample processing or analysis,

the fractional recovery of the tagged molecules will provide a basis for estimating the comparable loss which took place in the untagged molecules of the same species.

CALIBRATION OF SENSOR RESPONSE

Direct-reading instruments are generally delivered with either a direct-reading panel meter, a set of calibration curves, or both. The tendency of the unwary and inexperienced user is to believe the manufacturer's calibration, and this often leads to grief and error. Any instrument with calibration adjustment screws should of course be suspect, since such adjustments can easily be changed intentionally or accidentally, as in shipment.

All instruments should be checked against appropriate calibration standards and atmospheres immediately upon receipt and periodically thereafter. Procedures for establishing test atmospheres are discussed earlier in this chapter and in Chapter 12. Verification of the concentrations of such test atmospheres should be performed whenever possible using analytical techniques which are refereetested or otherwise known to be reliable.

With these techniques, calibration curves for direct reading instruments can be tested or generated. When environmental factors such as temperature, ambient pressure, and radiant energy may be expected to influence the results, these effects should be explored with appropriate tests whenever possible. Similarly, the effects of cocontaminants and water vapor on instrument response should also be explored.

SUMMARY AND CONCLUSIONS

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

- Use standard devices with care and attention to detail.
- All standard materials and instruments and procedures should be checked periodically to determine their stability and/or operating condition.
- Perform calibrations whenever a device has been changed, repaired, received from a manufacturer, subjected to use, mishandled or damaged and at any time when there is a question as to its accuracy.
- 4. Understand the operation of an instrument before attempting to calibrate it and use a procedure or setup which will not change the characteristics of the instrument or standard within the operating range required.
- When in doubt about procedures or data, assure their validity before proceeding to the next operation.
- All sampling and calibration train connections should be as short and free of constrictions and resistance as possible.
- Extreme care should be exercised in reading scales, timing, adjusting and leveling, and in all other operations involved.
- Allow sufficient time for equilibrium to be established, inertia to be overcome and conditions to stabilize.
- Enough points or different rates of flow should be obtained on a calibration curve to give confidence in the plot obtained. Each point should be made up of more than one reading whenever practical.
- A complete permanent record of all procedures, data and results should be maintained. This should include trial runs, known faulty data with appropriate comments, instrument identification, connection sizes, barometric pressure, temperature, etc.
- When a calibration differs from previous records, the cause of change should be determined before accepting the new data or repeating the procedure.
- Calibration curves and factors should be properly identified as to conditions of calibration, device calibrated and what it was calibrated against, units involved, range

and precision of calibration, date and who performed the actual procedure. Often it is convenient to indicate where the original data is filed and to attach a tag to the instrument indicating the above information.

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