

## CHAPTER 11

# INSTRUMENTS AND TECHNIQUES USED IN CALIBRATING SAMPLING EQUIPMENT

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### 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. Aerosols, 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 <sub>2</sub> Permeation Tube, Individually Calibrated, Effective length $\cong$ 10 cm, Permeation Rate $\cong$ 0.28 ug SO <sub>2</sub> /min. @ 25°C.
1626	Same as above, except that effective length $\cong$ 5 cm.
1627	Same as above, except that effective length $\cong$ 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 = $0.0107 \pm 0.0001$ mole %.
1612	Same as above, except that concentration = $0.00117 \pm 0.00001$ mole %.
1613	Same as above, except that concentration = $0.000102 \pm 0.000002$ mole %.
1601	Carbon Dioxide in Nitrogen compressed gas mixture, 68 standard liters @ 500 psi in disposable cylinder. Concentration = $0.0308 \pm 0.0003$ mole %.
1602	Same as above, except that concentration = $0.0346 \pm 0.0003$ mole %.
1603	Same as above, except that concentration = $0.0384 \pm 0.0004$ 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	250 W. 57th Street New York, N. Y. 10019

**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 <sup>(13)</sup>
AIHA	117	Analytic methods for air contaminants	No	Analytic Guides <sup>(14)</sup>
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 <sup>(15)</sup>
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:

$$E_c = [E_1^2 + E_2^2 + E_3^2 + \dots + E_n^2]^{1/2}$$

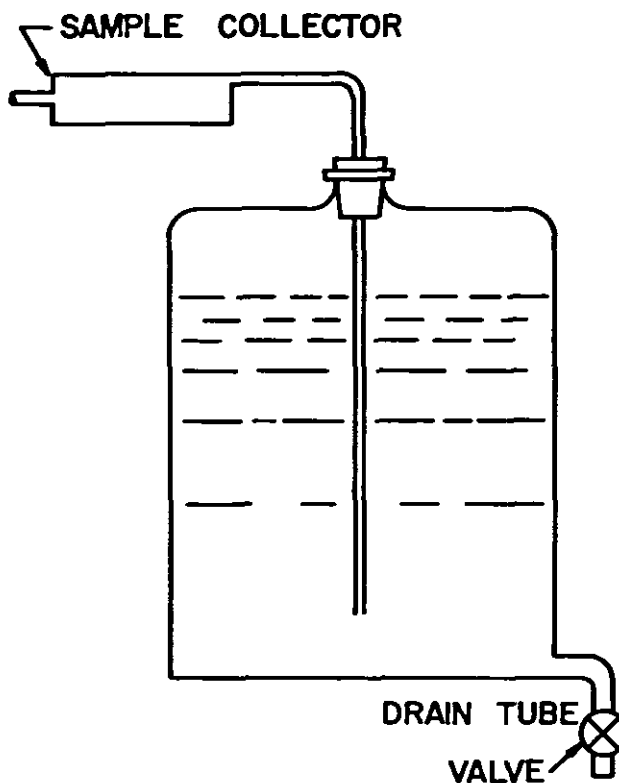
For example, if accuracies of the flowrate measurement, sampling time, recovery, and analysis are  $\pm 15$ , 2, 10, and 10% respectively, and there are no other significant sources of error, then the cumulative error would be:

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

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.

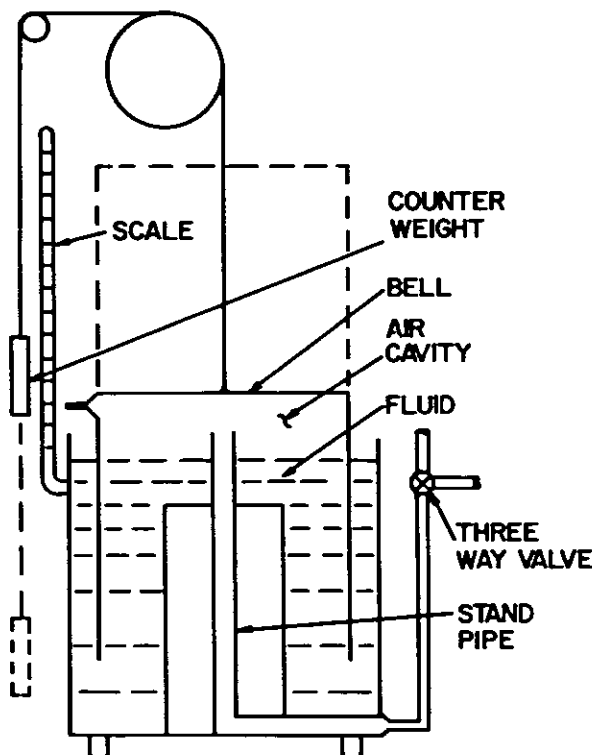
### CALIBRATION INSTRUMENTS AND TECHNIQUES FOR FLOW AND VOLUME CALIBRATION

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. Mariotti 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**

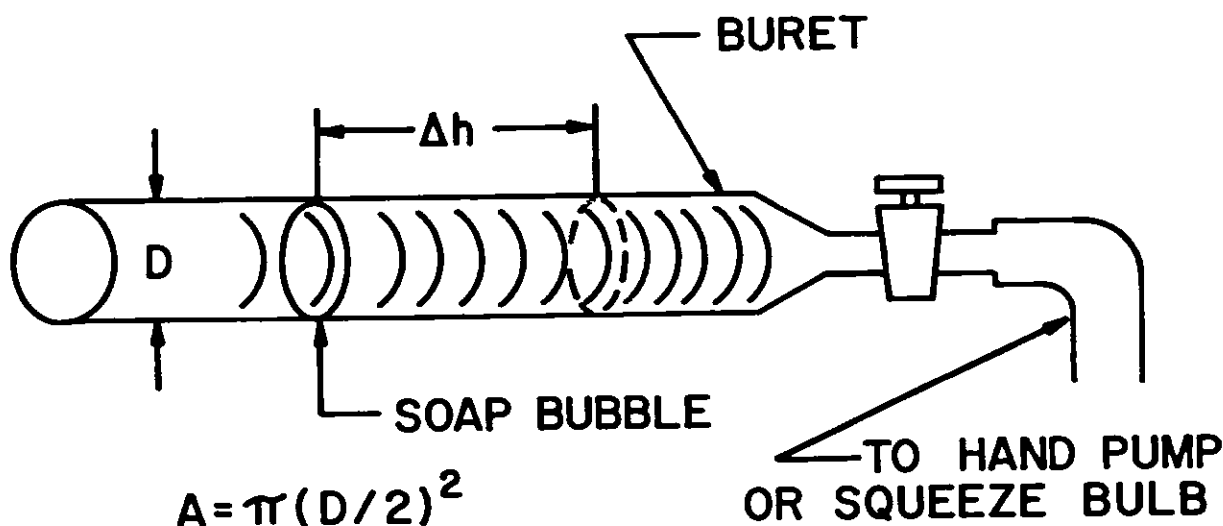
*Water Displacement.* Figure 11-1 shows a sche-

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 cm<sup>3</sup>/min.<sup>1</sup>

*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<sup>1</sup> 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.<sup>(1)</sup> In the mercury-sealed piston, most of the cylindrical cross section is blocked off

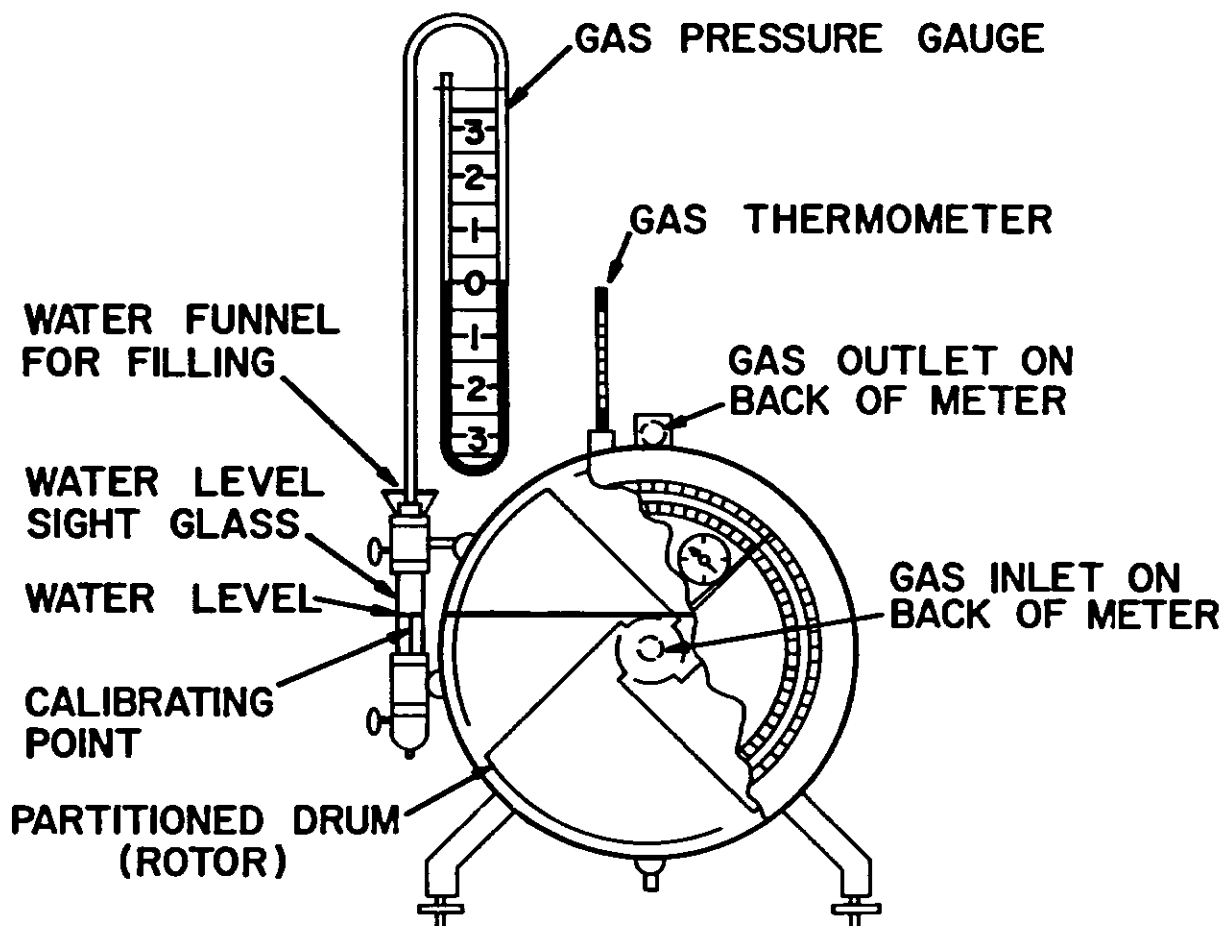


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Figure 11-3. Bubble Meter

by a plate which is perpendicular to the axis of the cylinder. The plate is separated from the cylinder wall by an O-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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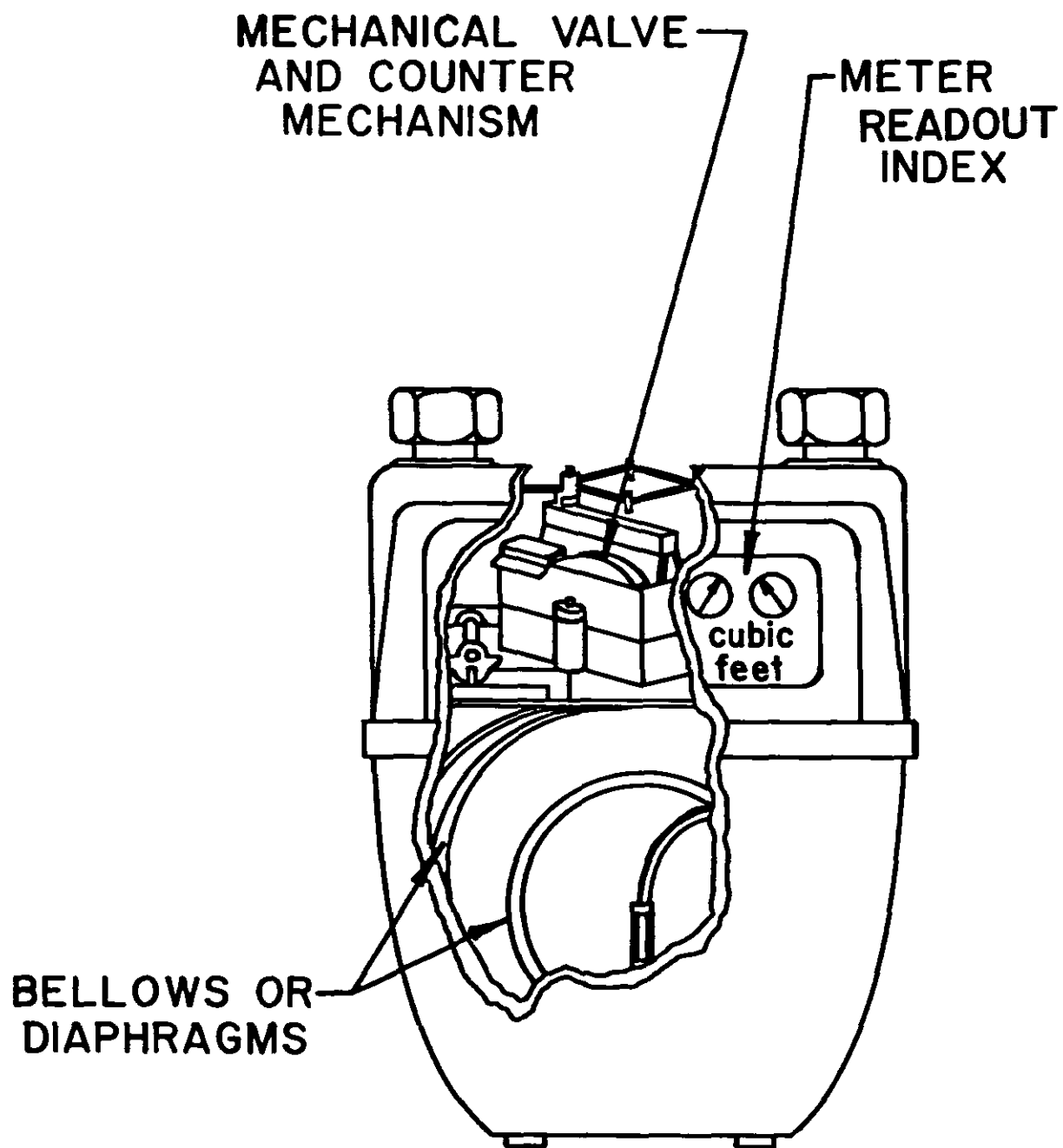
Figure 11-4. Wet Test Meter

**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

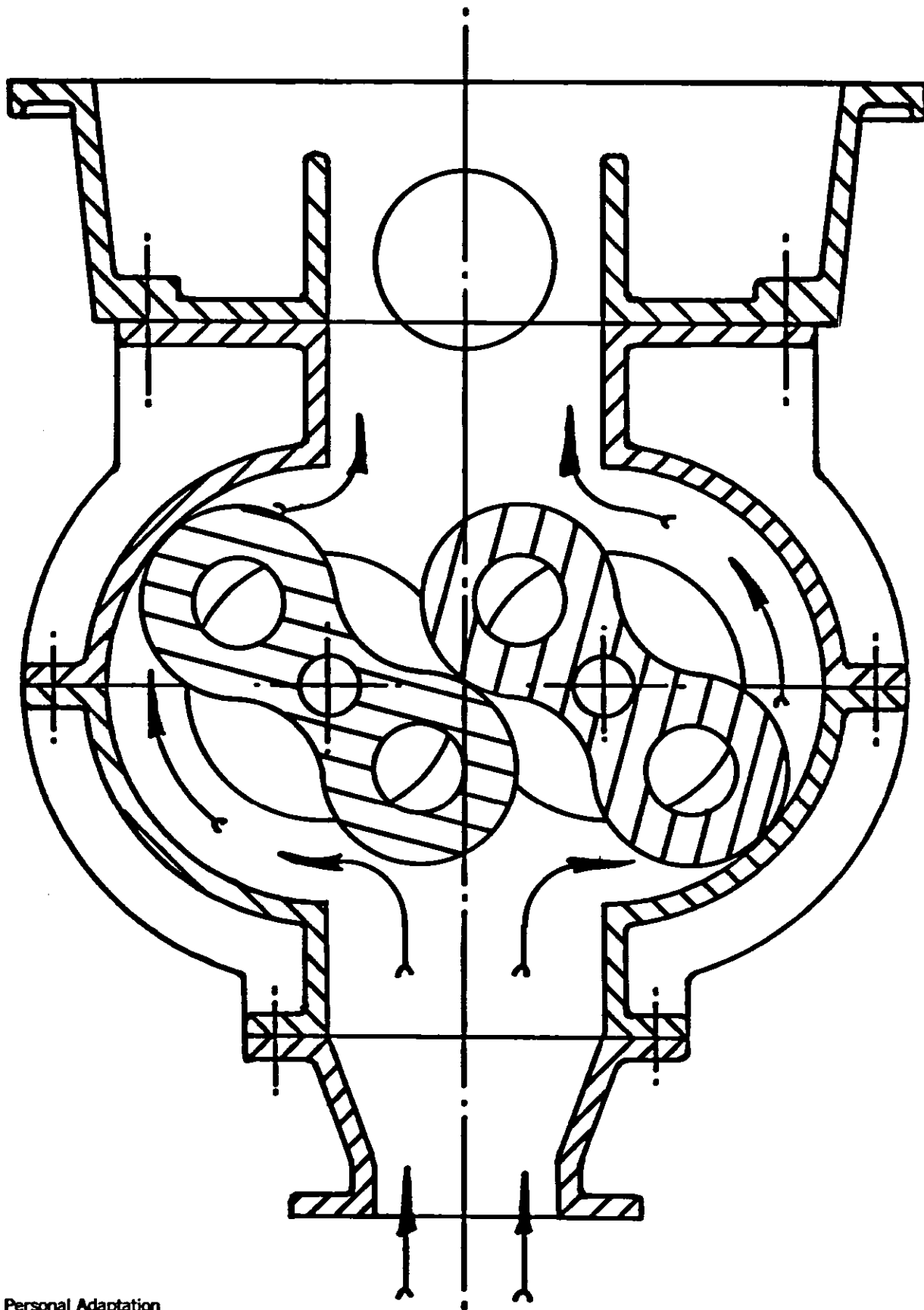
water seal at the inlet or outlet. In spite of these factors, the accuracy of the meter usually is within one percent when used as directed by the manufacturer.

**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



Personal Adaptation

**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.

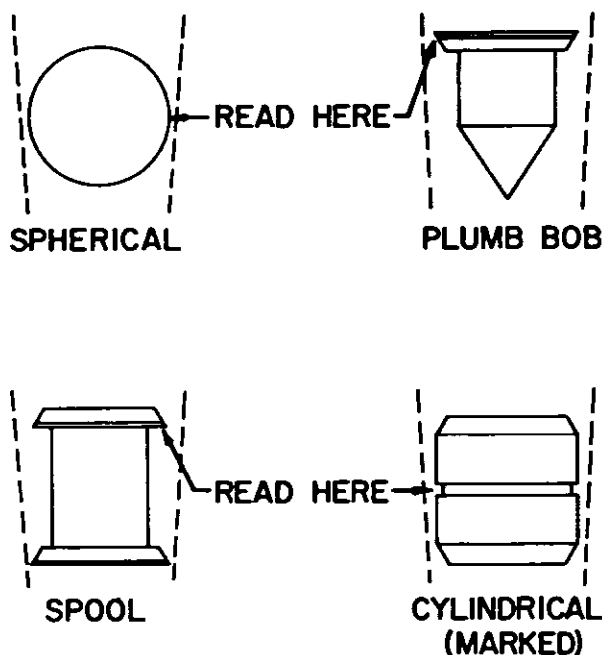
#### Volumetric Flowrate

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  $\frac{1}{8}$  to 6 inches in diameter, covering ranges from a few  $\text{cm}^3/\text{min.}$  to over 1,000  $\text{ft}^3/\text{min.}$  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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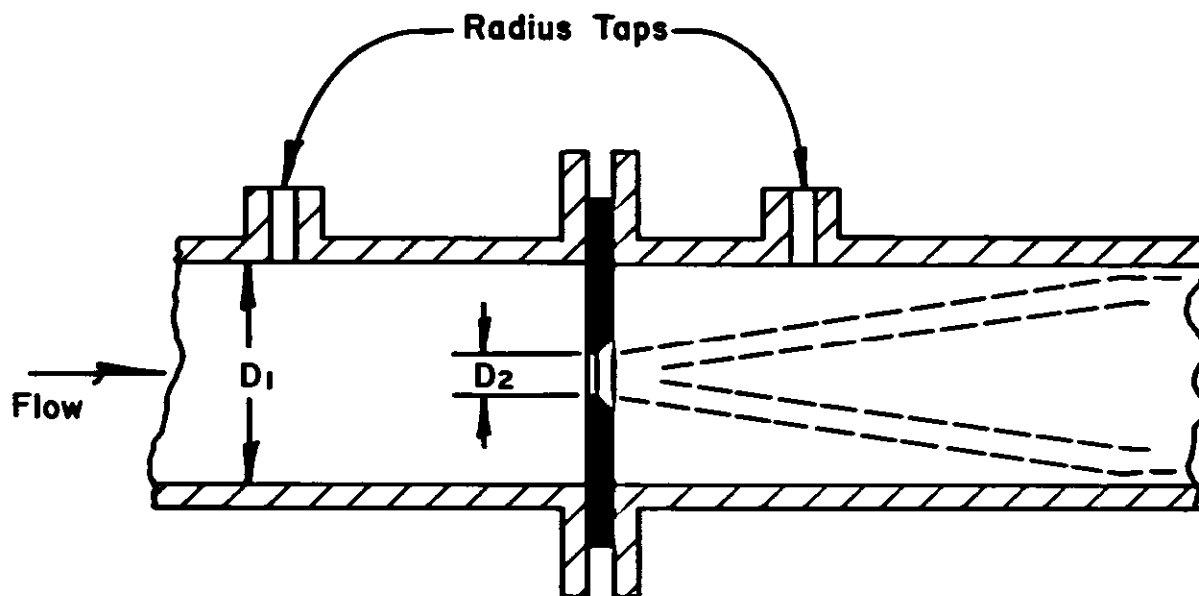
Figure 11-7. Typical Rotameter Floats

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

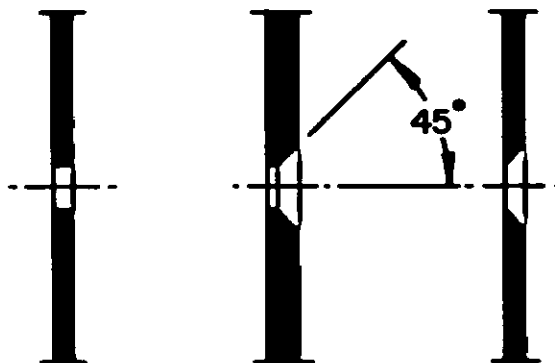
Rotameters are the most commonly used flowmeters on commercial air samplers, especially on portable samplers. For such sampler flowmeters, the most common material of construction is acrylic plastic, although glass tubes may also be used. Because of space limitations, the scale lengths are generally 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,  $\pm 5\%$  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  $\sim \pm 10\%$



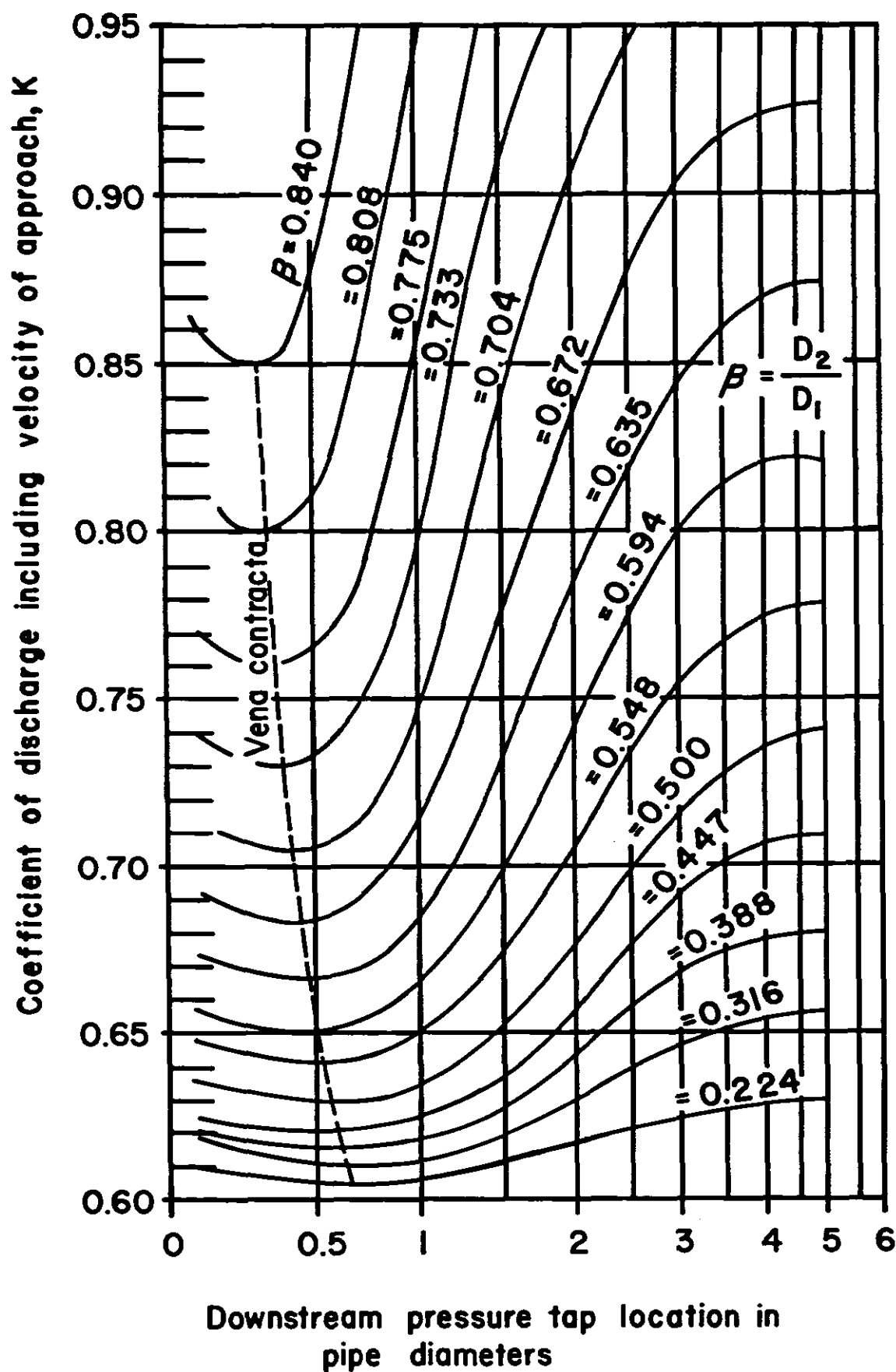
(a)



(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.



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  $N_{Re} > 30,000$  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 equation<sup>2</sup> for this type of meter is:

$$W = q_1 P_1 = KYA_2 \sqrt{2g_c (P_1 - P_2) \rho_1} \quad (1)$$

where:  $K = C / \sqrt{1 - \beta^4}$

$C$  = coefficient of discharge, dimensionless

$A_2$  = cross-sectional area of throat — ft<sup>2</sup>

$g_c$  = 32.17 ft/sec<sup>2</sup>

$P_1$  = upstream static pressure — lb/ft<sup>2</sup>

$P_2$  = downstream static pressure — lb/ft<sup>2</sup>

$q_1$  = volumetric flow at upstream press. & temp.—ft<sup>3</sup>/sec.

$W$  = weight-rate of flow — lb/sec.

$Y$  = expansion factor (see Figure 11-10)

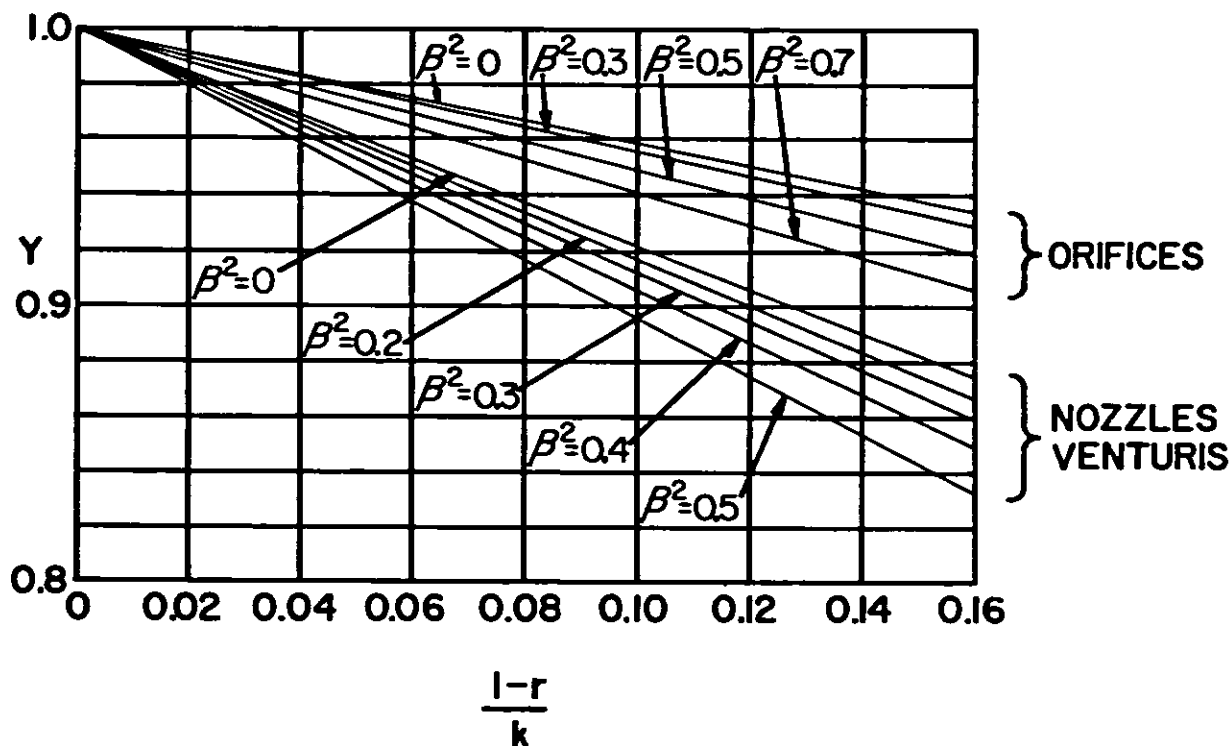
$\beta$  = ratio of throat diameter to pipe diameter, dimensionless

$\rho_1$  = density at upstream press. & temp. — lb/ft<sup>3</sup>.

**Orifice Meters.** The simplest form of variable-head 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:

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

where  $Q$  = flow — ft<sup>3</sup>/min.

$\beta$  = ratio of throat to duct diameter, dimensionless

$D$  = duct diameter — inches

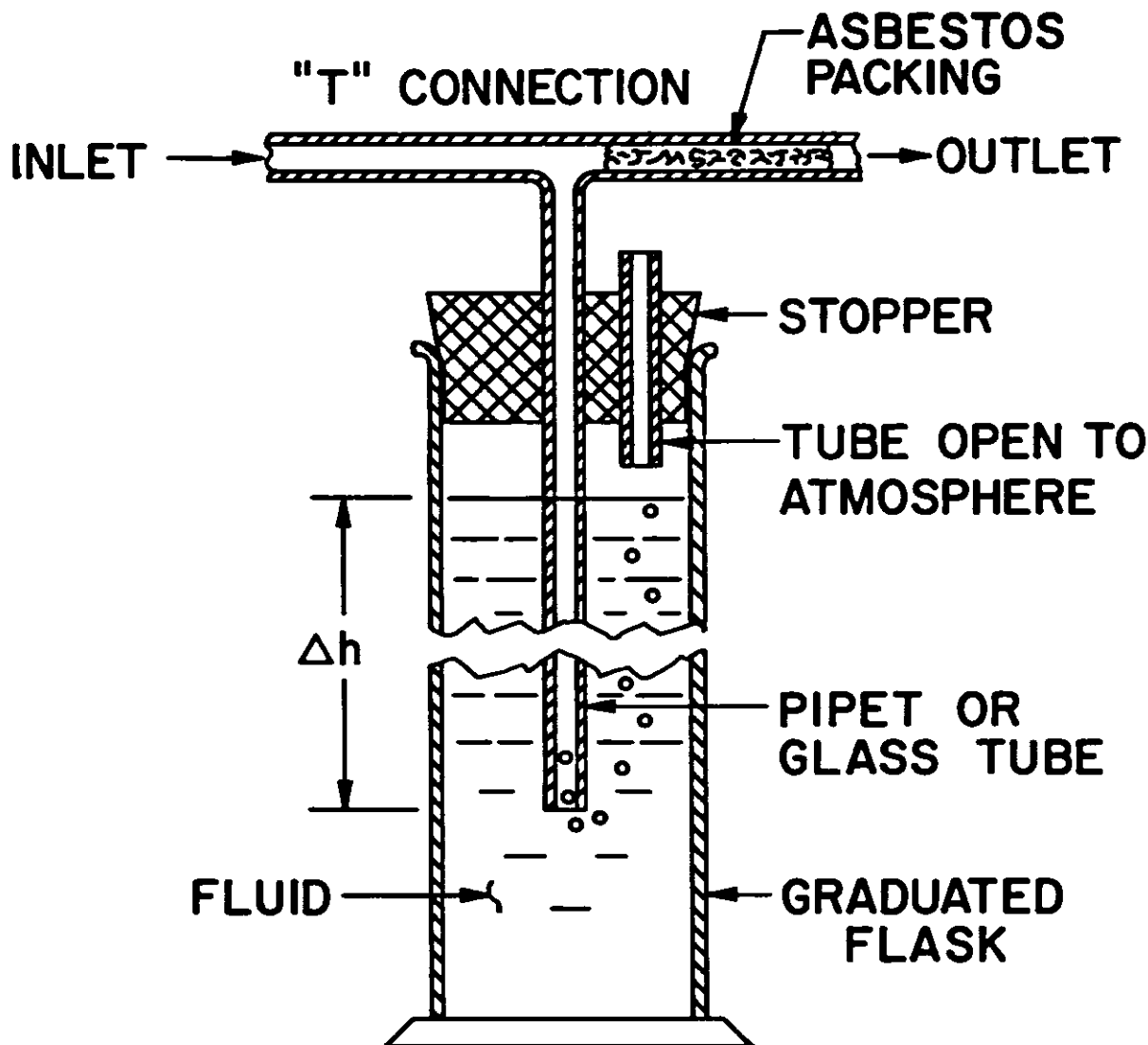
$h$  = differential pressure — inches of water.

**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.<sup>2,3</sup> 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 laminar-flow 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

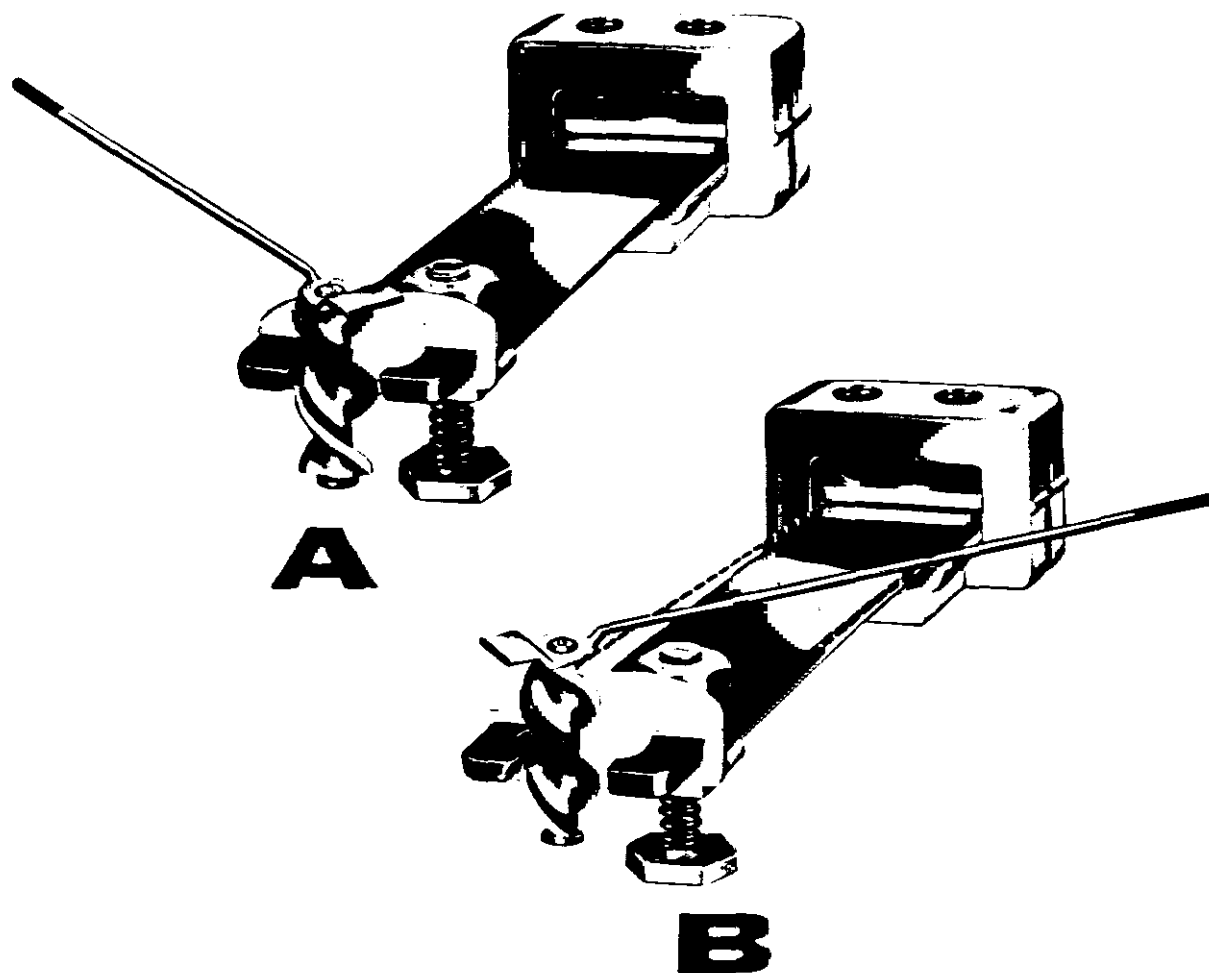
connection, pipet or glass tubing, cylinder and packing material. The outlet arm of the "T" is packed with material, such as asbestos, and the leg is attached to a tube or pipet projecting down

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<sup>4</sup> has used tubes

filled with asbestos to regulate and measure flow-rates as low as 0.01 cm<sup>3</sup>/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 free 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,<sup>2</sup> 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:

$$w = 0.533 \frac{C_v S_2 P_1}{T_1} \text{ lb/sec} \quad (3)$$

where:  $C_v$  = coefficient of discharge  
(normally  $\sim 1$ )

$S_1$  = duct or pipe cross section in square inches

$S_2$  = orifice area in square inches

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

$T_1$  = upstream temperature in °R

Critical-flow orifices are widely used in industrial hygiene instruments such as the midjet 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 ft<sup>3</sup>/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

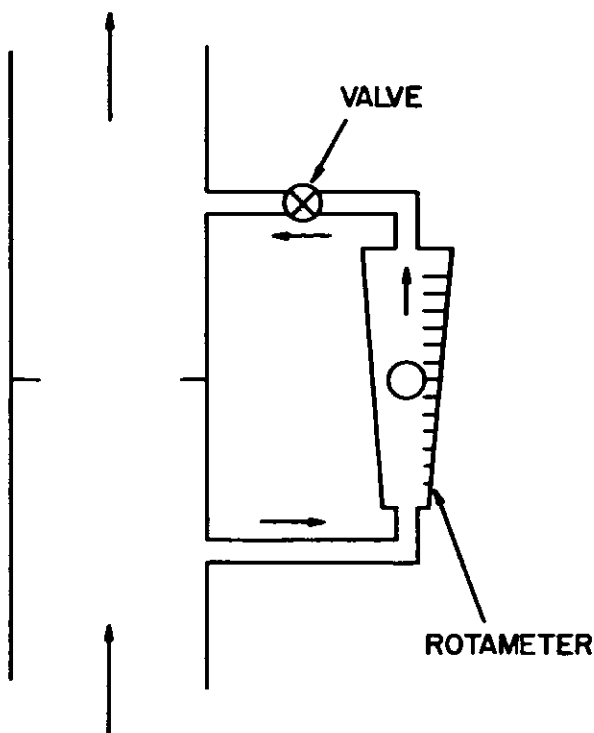


Figure 11-13. Schematic of By-Pass Flow Indicators

parallel path containing the small rotameter. The scale on the rotameter generally reads directly in ft<sup>3</sup>/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

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

where:  $V$  = linear velocity

$g_c$  = 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 ( $h_v$ ),

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

$$V = 1097 \sqrt{\frac{h_v}{\rho}} \quad (5)$$

where:  $\rho$  = density of air or gas in lb./ft.<sup>3</sup>

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

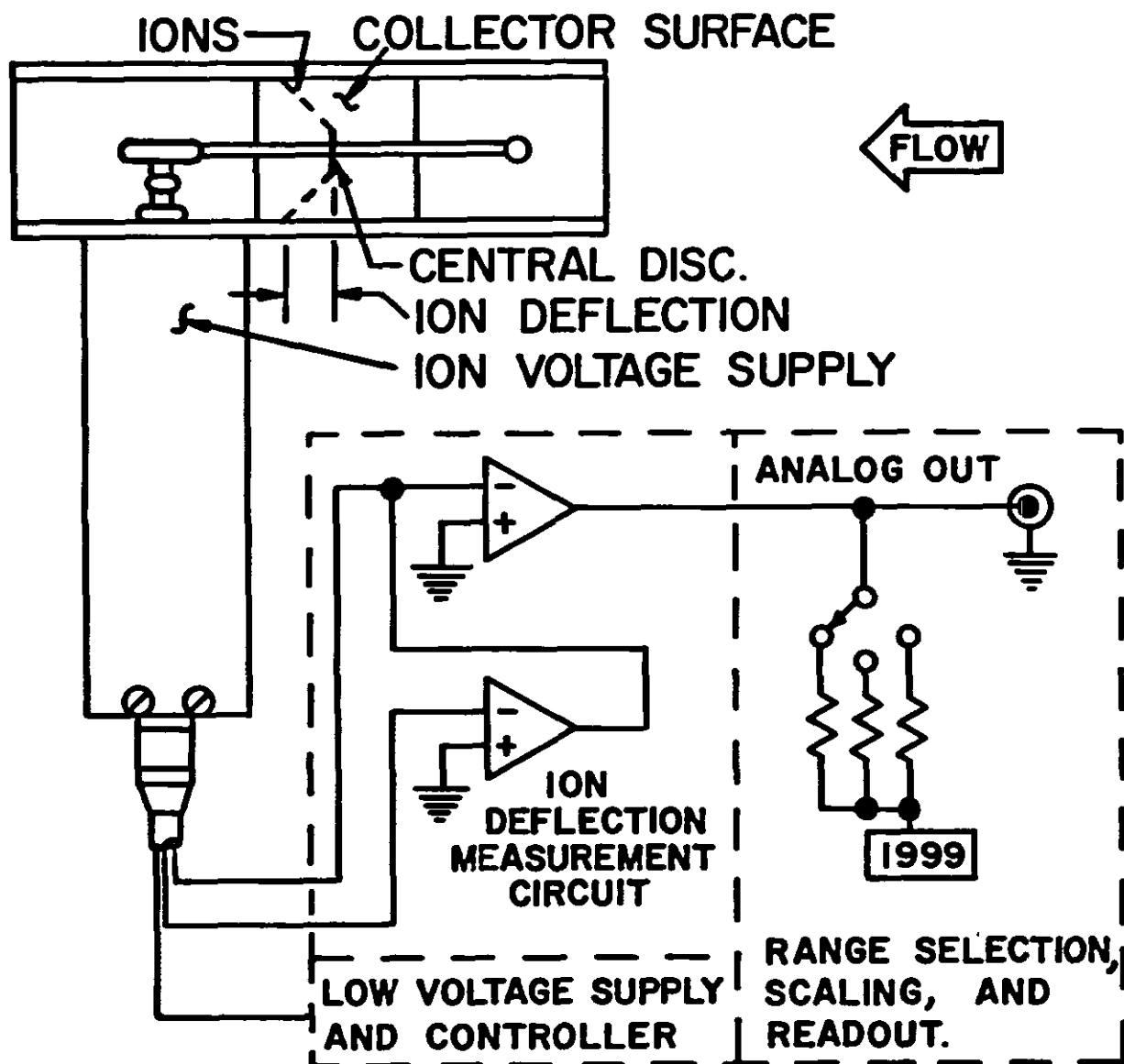
$$V = 4005 \sqrt{h_v} \quad (6)$$

where:  $V$  = velocity in ft./min.

$h_v$  = velocity pressure in inches of H<sub>2</sub>O

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 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

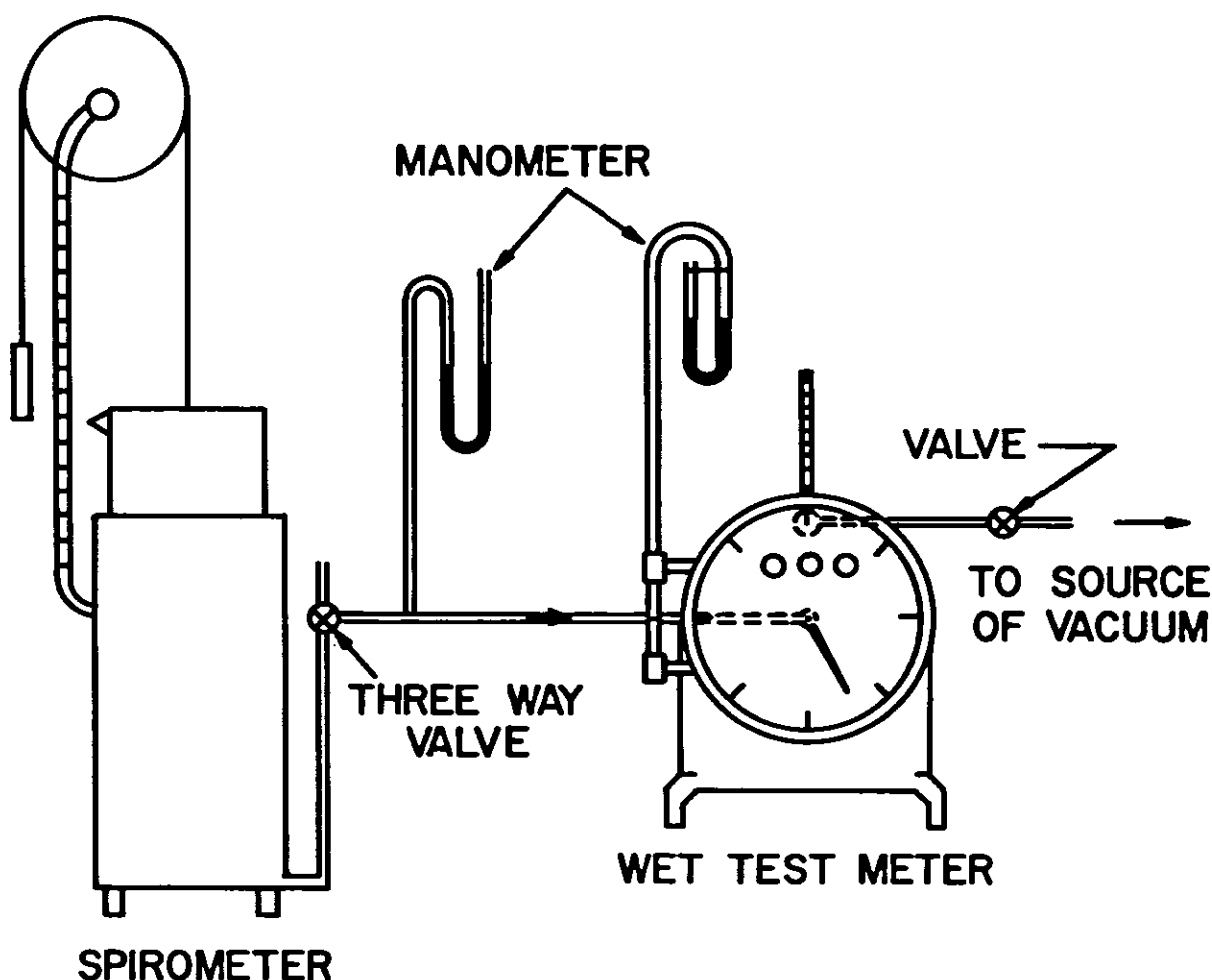
indicators of total flowrate and sometimes are called turbine flowmeters.

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



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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.<sup>3</sup>/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 wet-test meter (minimum of one revolution), or to near capacity of the spirometer, then return to the by-pass arrangement. Readings, both quantity and pressure of the wet-test meter, must be taken and recorded while it is in motion, unless a more elaborate system is set up. In the case of a rate meter, the interval of time that the air is 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:

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

where  $V_s$  = volume at standard conditions (760 mm & 0°C)

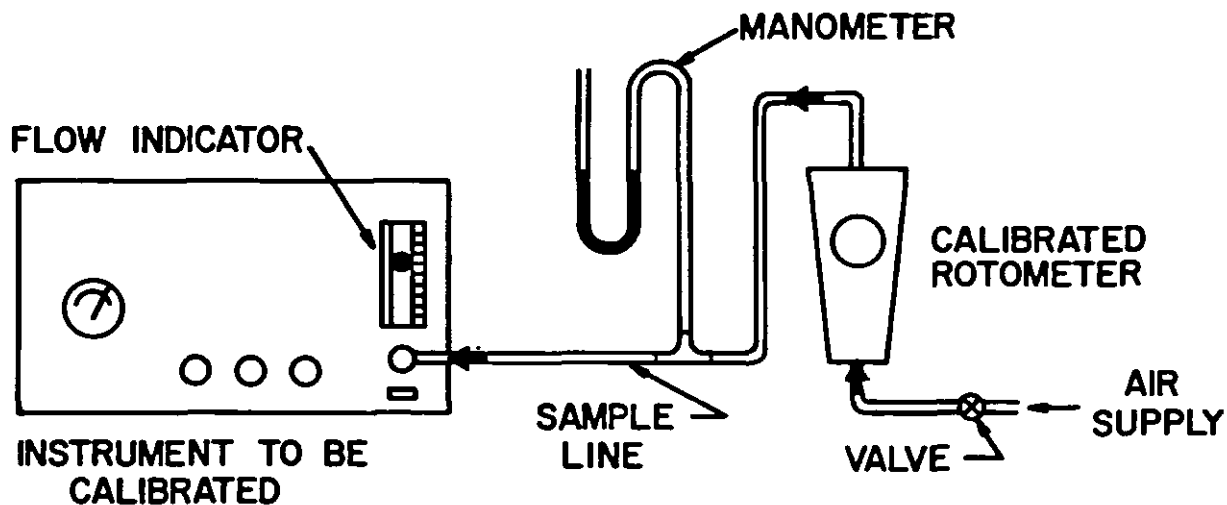
$V_1$  = volume measured at conditions  $P_1$  and  $T_1$

$T_1$  = 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 system<sup>4</sup> 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.<sup>4</sup> 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<sup>6,7,8</sup> 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:

$$C_t = C_0 e^{-bt} \quad (8)$$

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-log-paper. The slope of the line indicates the air changes per minute ( $b$ ) which can be converted to the rate ( $Q$ ) of air withdrawn by the instrument from the following relationship:  $Q = bV$ ; where  $V$  = volume of the vessel.

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

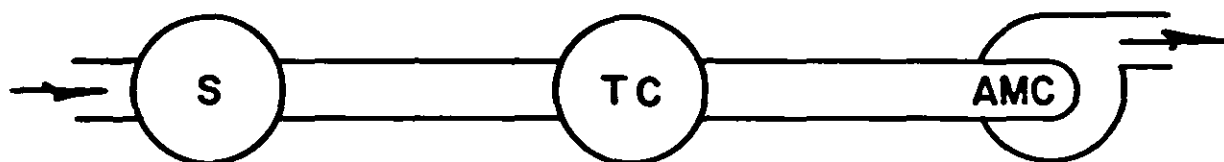
### CALIBRATION OF SAMPLER'S COLLECTION EFFICIENCY

#### 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.<sup>9-10, 11, 12</sup> 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**

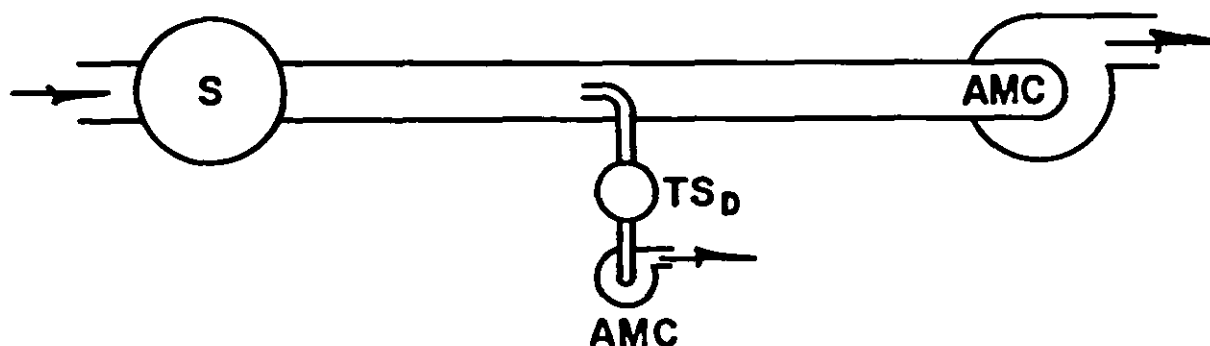
**Analysis of Sampler's Collection and Downstream Samples**

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.

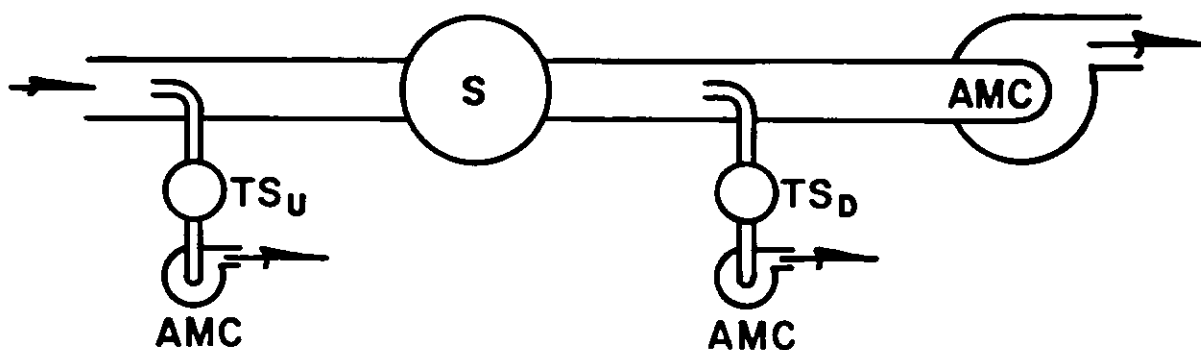
**Analysis of Up-and Downstream Samples**

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<sub>D</sub> = Downstream Sampler**  
**Total Collector**

**Figure 11-18. Sampler Efficiency Evaluation with Downstream Concentration Sampler: Analysis of Collections in S and TS<sub>D</sub>**



**TS<sub>U</sub> = Upstream Sampler  
Total Collector**

**Figure 11-19. Sampler Efficiency Evaluation with Upstream and Downstream Concentration Samplers: Analysis of Collections in TS<sub>U</sub> and TS<sub>D</sub>**

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 referee-tested 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 co-contaminants 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 sample 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:

1. Use standard devices with care and attention to detail.
2. All standard materials and instruments and procedures should be checked periodically to determine their stability and/or operating condition.
3. 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.
5. When in doubt about procedures or data, assure their validity before proceeding to the next operation.
6. All sampling and calibration train connections should be as short and free of constrictions and resistance as possible.
7. Extreme care should be exercised in reading scales, timing, adjusting and leveling, and in all other operations involved.
8. Allow sufficient time for equilibrium to be established, inertia to be overcome and conditions to stabilize.
9. Enough points or different rates of flow should be obtained on a calibration curve to give confidence in the plot obtained. Each point should be made up of more than one reading whenever practical.
10. A complete permanent record of all procedures, data and results should be maintained. This should include trial runs, known faulty data with appropriate comments, instrument identification, connection sizes, barometric pressure, temperature, etc.
11. When a calibration differs from previous records, the cause of change should be determined before accepting the new data or repeating the procedure.
12. Calibration curves and factors should be properly identified as to conditions of calibration, device calibrated and what it was calibrated against, units involved, range

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

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