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**CHEMICAL METHODS
in INDUSTRIAL HYGIENE**

INTERSCIENCE

manual **3**

CHEMICAL METHODS IN INDUSTRIAL HYGIENE

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INTRODUCTION AND SAMPLING

I. Introduction

The analytical problems of the industrial hygiene chemist are somewhat different from those in other specialties. For one thing, the samples are usually dusts, gases, and fumes, coupled with a variety of miscellaneous substances, any of which may be encountered in the industrial environment. The constituents or components of these samples which are to be determined are usually only those of toxic significance and they are generally present in trace amounts. To the industrial hygienist, a large amount means milligrams and small amounts are thought of in terms of micrograms. Often, the samples themselves may be relatively large, but the substances which are to be determined are present only in trace concentrations.

The methods which are described are principally applicable to environmental samples and air contaminants. A few of these methods have also been applied to biological samples such as blood or urine, governed by the frequency with which this type of analysis is required. They are designed essentially for field samples. Problems of a research nature may very well demand methods of greater precision and refinement.

Sampling apparatus has not been described in detail. The preferred sampling device, the rate of sampling, and the collection medium for any given contaminant have however been detailed as necessary and preliminary parts of the chemical procedure. The question of accuracy and efficiency of sampling devices is discussed, but may well be a subject for further investigation.

The calculations and examples which have been included have been so chosen that they will serve not only as a guide to obtaining numerical values, but also to indicate the size of the sample which must be obtained and the lower limits of applicability of the method. Parts per million are calculated at 25°C. unless otherwise specified.

For the purposes of this manual we thought it best to list the references alphabetically at the end of the book. The subject matter of these references has been indexed so they can readily be found. The use of these references will usually lead to a more complete bibliography of the respective subjects.

2. Sampling and Sampling Devices

The industrial environment is usually sampled for the purpose of determining the existence of any real or potential health hazard. Because of the degree of complexity and fluctuation in such an environment, the sample itself is the limiting factor as regards the final accuracy of the analysis. As a rule the laboratory methods available for the examination of environmental samples have a higher degree of refinement and precision than the requirements

of the sample demand. Consequently, in the choice of an analytical procedure, it is often possible to employ less refined methods in the interest of speed and still keep within the limits of accuracy which may be required. However, it is well to insert a few words of caution at this point. Small aliquots involving multiplication by large factors, poor and nonspecific methods, and sloppy technique may easily vitiate the validity of the analytical result and add to the uncertainties of the sample itself. A poor analysis is usually misleading and may be worse than none at all.

The proper collection of the various samples is exceedingly important and must be entrusted to specially trained personnel. In general, samples are collected in the immediate vicinity of the workers in a particular environment, near the source of the contaminant entering the general atmosphere, and in the general workroom atmosphere. It is not always possible to obtain all these types of samples. Nevertheless, whatever type of sample is obtained should be clearly borne in mind so that samples may not be indiscriminately averaged together or unjustifiably compared.

It is a task of no small magnitude to obtain representative samples of the industrial environment. Over and over again the man in the field is confronted with the statement that he is there at the "wrong time." Conditions in the plant are normally either worse or better, usually depending upon the bias of the informant. The inherent difficulties of getting a clear picture of the industrial environment from just a few visits to a plant which has been operating year in and year out are well recognized. Nevertheless, it is well established that when samples are collected and

properly evaluated by trained personnel a good picture of the industrial environment may be obtained. As an analogy samples are as valuable to the industrial hygienist as determinations of body temperature, blood pressure, and urinalysis are to the physician. It enables a proper evaluation or diagnosis to be made, as the case may be.

A number of years ago not many good methods of analysis for "trace" amounts of substances were known. It was, therefore, necessary to sample for long time intervals in order to obtain sufficient sample with which the analyst could work. Today we have many specific and refined methods of analysis which are applicable to industrial hygiene problems. However, as a carry-over from conditions which obtained years ago and as a result of making a virtue of necessity, the statement is often made that what is needed are individual samples which are collected over long time periods. Undoubtedly, this type of sample has its place in providing a ready integration of conditions prevailing throughout the sampling period, but it tells nothing about the maxima and minima which occur during such a sampling period. Actually, the difference between a thirty minute sample and a two hour sample as compared to years for the worker in the plant, is insignificant. The ideal sample is one similar to that obtained with a recording thermometer, a continuous record of the conditions which obtain. In lieu of that, a sufficient number of samples, taken over short periods, will yield a good result. Of course, in environments which are known to be fairly constant it is of no particular consequence whether samples are taken over long or short periods. However, as a general rule, it is better to take a

number of consecutive samples over short periods than one sample over a long period.

(a) *Measurement of Air*

As a rule atmospheric samples which are to be analyzed subsequently by chemical methods are collected by means of some bubbler device, "grab" sample bottle, impinger, or electrostatic precipitator. In every case, the volume of the air which is sampled must be recorded. Suitably calibrated flowmeters are generally used for this purpose.

The need for periodic systematic calibration, care and maintenance of all field sampling equipment cannot be over emphasized. If these instruments are not operating properly their efficiency will vary, dial readings may be off, and the value of the final results will be nil. It is also particularly necessary to check collecting efficiencies when using old devices for sampling new substances or when changing over to new methods.

Wet Meter: This instrument measures total gas volume. It is designed to operate at close to atmospheric pressure, and is principally used to calibrate other flowmeters. See figure 1.

The meter must be level during use and filled with water up to the indicated mark. The drum of the meter rotates as the gas flows through and operates a revolution counter permitting the gas volume to be read on the face of the dial. Each revolution corresponds to the passage of a definite volume of gas so that the dial may be read directly in units of volume. Wet meters cannot be operated satisfac-

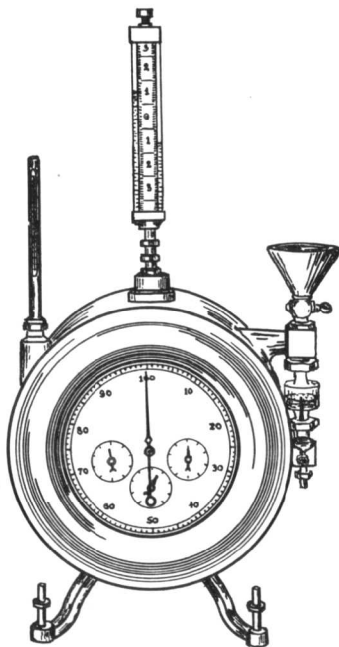


Fig. 1. Wet test meter.

torily at too rapid or even too slow rates of gas flow because of the inherent characteristics of their design. If the flow is too rapid the water will surge within the meter. If the flow is too slow pulsations may occur because of the time lag between the filling and rotation of the compartments in the drum. The manometer is designed to indicate pressures

of just a few inches of water and must never be filled with mercury.

For optimum results a meter of suitable capacity is chosen so that it will work within the limits recommended by the manufacturer. For work in the field of industrial hygiene it is desirable to have one meter whose dial indicates three liters or one tenth of a cubic foot per revolution and perhaps two additional meters, one reading one liter per revolution, and the other, one cubic foot per revolution.

Rotameter: This instrument is illustrated in figure 2. It may be calibrated against the wet meter and will work against varying pressures. The float is interchangeable. Rotameters are available for measuring air flow in various ranges as well as liquid flow. This is a very useful instrument. Once calibrated, it may be used to calibrate other types of flowmeters.

The rotameter consists of a float which moves in a vertical tapered tube. Its height varies according to the air flow necessary to support it. By varying the size of the tube and the weight of the float this instrument may be made to cover great ranges of flow at both high and low pressures. A set of three rotameters will usually be sufficient to cover the needs of the industrial hygiene laboratory.

Orifice Flowmeter: Figure 3 illustrates this instrument. The orifice tubes are interchangeable. Such an instrument may easily be constructed in the laboratory, but it is also available commercially. After calibration against a rotameter or wet meter, the inlet and outlet ends must not be reversed. The indicating fluid may be water which is colored with red ink.



Fig. 2. Rotameter.

This instrument should be checked and cleaned regularly. It is a common occurrence for the orifice to become plugged.

Orifice flowmeters are usually employed when sampling with bubblers of various sorts. They are also very useful in the laboratory.

Flow Gages and Sources of Suction: Impingers and

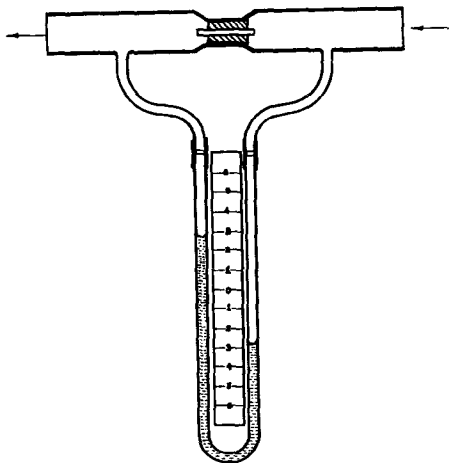


Fig. 3. Orifice flowmeter.

the electrostatic precipitator usually are equipped with special gages and electrically driven suction devices to indicate air flow. The electrically driven suction apparatus for the Greenburg-Smith impinger may also be used in conjunction with glass bubblers for collecting air samples. It is designed to sample at the rate of one cubic foot per minute. The apparatus is commercially available as a single unit. The motor is series-wound, single-phase, 60-cycle, one-fifteenth horsepower, rated at 1.6 amperes, at 110 volts, with a speed of 1,800 revolutions per minute. This motor, being series-wound, operates on either A.C. or D.C. current. The motor is geared to a rotary eccentric positive pressure blower by means of a set of gears having a 1 to 3

ratio. The blower is rated at 4 cubic feet of free air per minute and is used as a source of suction rather than as a source of air pressure. Wound in series with the electric motor is a 98 ohm, 1.6 ampere variable sliding rheostat used for speed control of the motor. By employing such a rheostat a voltage of 110 or 220 volts may be used. To the intake or suction side of the blower is attached a 0.25 inch malleable-iron elbow fitting provided with two inlets. To one of the inlets a 0.25 inch brass needle valve is attached which serves as a by-pass in regulating the rate of suction. The second inlet of the elbow is connected to a construction-type flowmeter the inlet side of which is connected to the sampling flask. A vacuum gage may be used instead of a flowmeter as a measuring device for the air flow.

The hand-operated midget impinger pump may also be used as a means of suction for small sampling flasks and fritted bubblers which require sampling rates of a few liters per minute. Suction is supplied by a small rotary pump of the vane type operated by a hand crank through a set of gears that step up the speed at a ratio of nine to one. A vacuum gage calibrated in inches of water is used to indicate the air flow. A head of 12 inches of water will give a flow of approximately 0.1 cubic foot per minute when the apparatus is used with the midget impinger. With fritted glass bubblers it is usually necessary to increase the head to 16 inches of water vacuum.

(b) Sampling Devices

Sintered Glass or Fritted Glass Bubblers: This type of bubbler (figure 4) may be used in conjunction with vari-

ous liquids or special reagents to collect gases which are fairly soluble in such media. The size of the large bubbler is usually 125 ml. In many cases a midget bubbler (20 ml.) is more convenient. This is particularly true when only the color of the resulting solution is to be estimated. The collecting cylinders may also be stoppered and used as con-

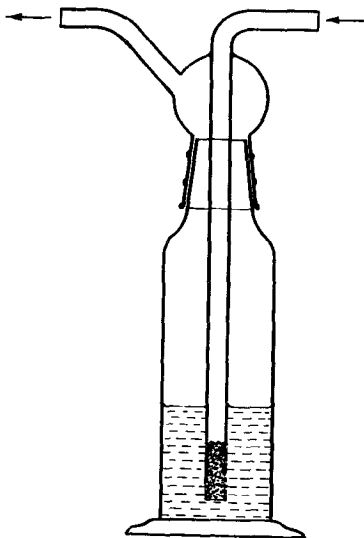


Fig. 4. Fritted glass bubbler.

tainers for the samples. The use of glassware with interchangeable joints is of particular advantage in this connection. Medium or coarse discs should be specified. Discs with fine porosities clog up quickly and offer too great resistance to air flow.