

C. CONDENSATION AT LOW TEMPERATURES

Most vapors may be collected successfully by drawing them through a suitable chamber immersed in a low-temperature bath, the temperature required being that at which the vapor pressure of the liquid or solid to be collected approaches zero. A metering device for the air mixture is necessary, and the rate must be slow enough to permit condensation and avoid loss of mist or dust of the condensed material. The design of the condensation chamber must be such as to avoid choking with either ice or solid contaminant condensed from the air sampled. The condensed material may be weighed, measured by volume, or determined by quantitative chemical analysis. Ice, solid carbon dioxide, and liquid air have each been employed as the refrigerating medium.

D. COLLECTION BY ABSORPTION OR ADSORPTION FOR LABORATORY ANALYSIS

The collecting device here may be any of several types of scrubbers or impingers, and the collecting medium may be granulated silica gel or a liquid of low volatility, either of which must be a tenacious adsorbent or absorbent for the contaminant or else it must fix the contaminant by chemical combination. The collectors may be used singly or in series depending upon their established efficiency. The collected samples are transported to the laboratory and evaluated by various means, which include colorimetric, volumetric, polarographic, or spectrographic analyses, the use of the ultraviolet or infrared spectrometer, and combustion (see also pages 182 and 189).

E. SAMPLING IN EVACUATED BOTTLES

1. Vacuum Bottle Samples

Samples are conveniently collected in vacuum bottles for the determination of oxygen, carbon dioxide, and combustible gases in the analysis of mine gases by means of the Haldane⁸⁸ or Orsat¹⁰ gas burets; for the determination of radon by means of especially constructed electrometers (page 731); for the determination of nitrogen oxides by the phenoldisulfonic acid method; and for many other determinations. Usually this type of sample is not larger than 1 liter. The sample is readily taken upon opening the bottle to the atmosphere, either by opening a glass stopcock or a pinchcock on a short length of heavy-walled tubing, or by breaking off the tip of a sealed inlet orifice, being careful not to warm the bottle by contact with the hands, and then closing or sealing the orifice.

2. Samples in Partially Evacuated Bottles

Samples may be conveniently taken in partially evacuated bottles of any size up to 20 liters. A measured volume of a suitable absorbent liquid is added to a

⁸⁸ L. B. Berger and H. H. Schrenk, U. S. Bur. Mines Circ. No. 7017, 1938.

calibrated bottle and the bottle is partially evacuated by a hand pump or other means, the residual partial pressure being recorded. The bottle is opened in the atmosphere to be sampled. It should then be taken to the laboratory, or to an uncontaminated zone, shaken well, and the contents washed into a suitable container with 2 or 3 successive portions of wash solution. After this, analysis may be made by appropriate methods.

The volume of the sample is computed as follows:

$$V_s = (V - A) \frac{P_2 - P_1}{P_2}$$

where V_s = sample volume, V = volume of bottle, A = volume of absorbent added to the bottle, P_1 = residual partial pressure in the bottle, and P_2 = atmospheric pressure at the time and place of sampling.

F. SPECTROMETRY

1. Infrared

The infrared spectrometer⁸⁴ is a relatively new tool with great possibilities for use in industrial hygiene as a means of recognizing and evaluating organic materials in the air. The basis of application is that practically all organic substances possess selective absorption at certain frequencies in the infrared portion of the spectrum. The essential parts of the apparatus are a heat source emitting a continuous range of the wave lengths desired; a means of dispersing the radiation and providing narrow wave length bands at accurately known wave length positions (for this purpose prisms of sodium chloride, potassium bromide, and so forth are supplied); cells for placing a suitable thickness of material for analysis in the path of the radiation; and a detector, such as a sensitive thermocouple and amplifier for accurately measuring the radiation. Absorption cells of various lengths have been used, from 0.05 mm. for liquids and solids to several meters for gases. The size, weight, and complexity of the apparatus do not make it attractive as a portable instrument; and the length of absorption path must be many meters in order to make the instrument suitable for measuring a few p.p.m. of most gases and vapors in air. However, when the material to be analyzed is absorbed or dissolved in a liquid, analysis is readily accomplished, providing a liquid vehicle has been chosen that has no interfering absorption bands in the significant range for the material to be analyzed. The absorption pattern furnishes positive identification of single compounds having a known pattern; furnishes helpful clues to the composition of complex mixtures (superimposed patterns); and identifies linkages, bonds, and groups in obscure compounds. It is a quantitative tool as well, since Beer's law has been found true for this region of the spectrum (the absorption is proportional to the concentration of absorbent in the solution). Many ranges of wave lengths in the spectrum, between visible and ultra short radio waves, have

⁸⁴ B. Barnes, U. Liddel, and V. Z. Williams, *Ind. Eng. Chem., Anal. Ed.*, 15, 659 (1943).