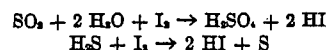


Sulfur dioxide and hydrogen sulfide each require only the usual solution of iodine in potassium iodide, and the reactions are the common oxidation ones:



A similar result is achieved by utilizing the color developed by the solution of the contaminant in the sampling medium. This simple type may be utilized in estimating ammonium picrate. Ammonium picrate in water, with no added reagents, produces sufficient color that 0.01 mg. in 10 ml. of water is readily distinguishable from none and from 0.02 mg. Each sample is collected until a visible color is obtained and then that color is compared with standards, in the field. (Ammonium picrate, TNT, Tetryl, and other contaminants in their class have been successfully collected by means of the midjet impinger regardless of whether they occurred as vapor or particulate matter.) TNT and Tetryl samples may be collected and semiquantitative results may be obtained in the field by the use of diethylaminoethanol²⁰ in the impinger; but, because color develops gradually and continuously over many hours, really satisfactory results must await transportation to the laboratory. However, the use of permanent standards, and possibly an accelerator, would improve the results obtainable at the time of sampling. In its present stage of development the method is satisfactory only in the laboratory where results may be determined with a spectrophotometer and compared with results obtained with standard solutions, due regard being taken of the time factor.

D. HAND PISTON PUMP, LUER-SYRINGE, AND RUBBER-BULB COLLECTION DEVICES

Hand piston pumps and rubber aspirating bulbs have been used for collecting samples. These methods, which aim to avoid the more cumbersome, orthodox methods of sampling, may involve pulling the atmosphere through indicating granules, such as in the MSA hydrogen sulfide detector, the aromatic hydrocarbon detector, and the NBS carbon monoxide indicating tube.²¹ Indicator paper or a scrubber may also be used. The pump may be calibrated and the sample measured by the number of piston strokes. Luer syringes have been used for measuring atmospheric samples and as reaction chambers for the determination of oxygen²² and also as a comparison chamber in the determination of nitrogen dioxide.²³ All examples of this class of sampling have as their purpose the keeping of sampling equipment to a minimum and also the obtaining of direct results in the field. Accuracy in some instances may, and in others may not, equal that obtained by longer and more cumbersome methods.

²¹ F. H. Goldman and D. E. Rushing, *J. Ind. Hyg. Toxicol.*, **25**, 184, 195 (1943).

²² M. Shepard, *A Preliminary Report of the N.B.S. Colorimetric Indicating Gel for the Rapid Determination of Small Amounts of Carbon Monoxide*. Natl. Bur. Standards, June 29, 1948.

²³ Y. Henderson and L. A. Greenberg, *J. Am. Med. Assoc.*, **96**, 1474 (1931).

²⁴ F. A. Patty and G. M. Petty, *J. Ind. Hyg. Toxicol.*, **25**, 361 (1943).

II. Methods Requiring Laboratory Analysis

HALOGENATED HYDROCARBONS COMBUSTION APPARATUS

Several devices have been proposed for the combustion of halogenated hydrocarbon vapors in the atmosphere. Passing the atmosphere through a heated tube containing a platinum foil was first utilized by Pregl²⁴ and several modifications for field use have been made.²⁵ The use of a free flame for the combustion analysis of this class of vapors was introduced by Patty, Schrenk, and Yant.²⁶ A similar plan was followed by Winter,²⁷ using a lamp method. A sulfur lamp apparatus was used by Wirth and Stross²⁸ to determine sulfur and chlorine in gasoline, and since then has been used for the determination of various organic chlorides in inflammable liquids. Elkins²⁹ scrubbed carbon tetrachloride from the air with amyl acetate and burned it in a modified sulfur lamp. In all of these methods combustion was found to be quantitative. The percentage absorption of the products of combustion varies with the type of apparatus employed and may be seriously low when sampling concentrations on the order of 0.2 per cent or higher, and when using a small volume of absorbing liquid. In the combustion-tube method at least 24 hours are required to collect the sample, after which the combustion products must be washed into a suitable flask and taken to the laboratory for titration, which is simple and rapid. The sulfur-lamp method requires more time since it involves scrubbing the vapor from the air before it can be burned in the lamps. The combustion methods are specific only for the class of halogenated vapors and gases (chlorides, bromides, or iodides), and organic halogens cannot be differentiated from acid halogens, unless sampled through a filter to remove acid halogens. Methods employing glass apparatus are not applicable to the determination of fluorides, because the fluoride combustion products attack glass. These combustion methods obviously give an integrated result for the entire period of sampling.

It will be of interest to compare sets of figures obtained independently by two experienced operators, one with the Willson combustion apparatus for halogenated hydrocarbons, and one with the Zeiss gas interferometer. The samples were taken over a 30-minute period in a room of a plant where carbon tetrachloride was being splashed from bottles onto a table top. The samples were drawn through a 2-foot glass tube extension on the combustion tube of the Willson apparatus, the

²⁴ J. Pregl, *Quantitative Organic Microanalysis*. Trans. by Tylemann. 2nd ed., Blakiston, Philadelphia, 1924.

²⁵ W. M. Mallison, *Ind. Eng. Chem., Anal. Ed.*, **7**, 428 (1935); B. D. Tebbens, *J. Ind. Hyg. Toxicol.*, **19**, 204 (1937); A. N. Setterling, *Determination of Chlorinated Hydrocarbons (Except Carbon Halides)*. Illinois State Dept. of Health, Div. of Industrial Hygiene, Chicago, Ill.; H. C. Dudley, *Public Health Repts.*, **56**, 102 (1941).

²⁶ F. A. Patty, H. H. Schrenk and W. P. Yant, *Ind. Eng. Chem., Anal. Ed.*, **4**, 259 (1932); F. A. Winter, *Ind. Eng. Chem., Anal. Ed.*, **15**, 571 (1943).

²⁸ W. Wirth and M. J. Stross, *Ind. Eng. Chem., Anal. Ed.*, **5**, 85 (1933).

²⁹ H. B. Elkins, A. K. Hobby, and J. E. Fuller, *J. Ind. Hyg. Toxicol.*, **19**, 474 (1937).