tem and air is admitted to the gage through the T-tube. The gage is then evacuated again to flush out most of the water vapor. In about 2 minutes, a reading will be found higher than the original before flushing in proportion to the amount of water vapor which was present initially. Further readings gradually become lower again as vapor diffuses back into the gage. Each time a reading close to the true total pressure in the system is desired, the gage must be flushed. This method is not applicable at higher vacuum.

ERRORS OF BOTH CONDENSATION AND OUTGASSING. *Chemical Trap.* This is the simplest method of all.

A glass tube large enough in diameter to cause no undue restriction of flow is filled with a chemical selected on the basis of a vapor pressure well below the minimum to be encountered in the high-vacuum system. By suitable selection, chemicals for vapors, such as alcohol and oil, which cause similar errors may be removed. An indicator chemical may be used, so that time for replacement may be determined readily. At highest vacuum, say at pressures below 0.1 micron, it may be necessary to cool the chemical trap.

Freezing Trap. A low-temperature condenser kept cold by means of dry ice, liquid air, or liquid nitrogen may be used for freezing out the moisture at a properly low vapor pressure. The necessity of continually supplying the refrigerant and frequently removing the condensate makes this method less convenient.

CERTAINTY OF ACCURATE READINGS

The chemical or freezing trap causes true total pressures to be indicated, even though only dry air is in the gage at all times. This air accumulates in quantity, so that its pressure exactly balances the total pressure of water vapor and air in the system on the other side of the trap. Even traces of moisture will cause some degree of error and new gages (or old ones that have just been cleaned) may require several days under vacuum, protected by a moisture trap, for complete removal of moisture in order to ensure accurate readings. The gage must be kept protected and only dry air admitted to it.

Since the wider the range of the gage the higher the condensation point, a moist gage of wide range can be used up to higher pressures than one of the narrow range before errors of condensation arise. A condensable vapor trap is recommended to exclude all vapors permanently, the chemical type being the most practicable. Using two gages of different scale range and obtaining identical readings is the best means of knowing with certainty that readings are accurate. With a McLeod gage of the recording type for producing a continuous record of vacuum, a proper trap for moisture is indispensable.

LITERATURE CITED

(1) Flosdorf, E. W., IND. ENG. CHEM., ANAL. ED., **10**, 534 (1938).
(2) Flosdorf, E. W., U. S. Patent 2,278,195 (Mar. 31, 1942).
(3) McLeod, *Phil. Mag.*, 47, 110 (1874).

Microdetection of Sulfur in Insoluble Sulfates and in Organic Compounds

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THE procedures described are based upon the evolution of nitrogen in the sulfide-catalyzed reaction between sodium azide and iodine (*1, 3, 4*):

$$3I_2 + 6NaN_3 \xrightarrow[\text{or}\ Ag_2S]{K_2S} 6NaI + 3N_2$$

The reaction is catalyzed by metallic thiocyanates, thiosulfates, and sulfides, whether soluble or insoluble in water, and also by organic compounds which contain the group $-\overset{|}{C}{=}S$ or $-\overset{|}{\underset{|}{C}}-SH$, and some others with active sulfur—e.g., sulfathiazole—to which the test may be applied without preliminary treatment (*2, 5*). To apply this test to the detection of sulfur in insoluble sulfates or nonvolatile organic compounds, a minute quantity of substance, in a molten globule of potassium hydroxide on the tip of a piece of fine copper wire, is heated in the reducing zone of a flame, whereby sulfur in any combination is converted to potassium sulfide. Organic compounds whose volatility excludes this procedure are pyrolyzed in a capillary tube internally coated with silver; the resulting silver sulfide serves to catalyze the iodine-azide reaction. It is advisable to test first directly—i.e., without a preliminary decomposition—and then, if the result is negative, to use one of the procedures described below, as in this way some indication as to the manner of combination of sulfur in an organic compound may be obtained (*2, 5*). Application of all three procedures requires only a small amount of substance and can be completed in 5 minutes.

PROCEDURES

REAGENT. Dissolve 1.0 gram of sodium azide in 50 ml. of 0.5 *N* iodine solution.

1. DETECTION OF SULFUR IN NONVOLATILE SUBSTANCES. Clean the end of a piece of thin copper wire (0.1 to 0.2 mm.) with nitric acid or emery paper to remove sulfide, and wash with distilled water. Dip the clean end of the wire into a saturated solution of potassium hydroxide in water, and pass it through the flame of an alcohol lamp, so as to evaporate the water and leave a small bead of molten potassium hydroxide at or near the end. [A gas flame may be used if sufficiently low in sulfur to yield a negative blank test. It is an advantage of the test, especially in mineralogical field work, that an alcohol lamp (whose flame is certain to be sulfur-free) will serve.] If the bead does not form at the tip, the wire beyond the bead may be cut off. Bring the bead of alkali into contact with the substance to be tested. Introduce the bead and adhering sample into the flame, moving the bead slowly into the reducing cone, and after a few seconds allow it to cool near the wick. Use one of the following manipulations to detect the sulfide formed.

Cut off that portion of the wire covered by the bead of alkali and place it on a microscope slide on the stage of a microscope. Cover the bead with a drop of the reagent and observe the result through the microscope. A positive test is an active evolution of nitrogen bubbles.

Cut off the portion of the wire covered by the bead of alkali and drop it into a glass capillary of 1-mm. diameter and sealed at one end—e.g., a short melting point tube. Introduce a droplet of the iodine-azide reagent, and swing the tube rapidly, so as centrifugally to project the reagent to the closed end, where it comes into contact with the test bead. While the tube is held with the closed end uppermost, observe whether or not gas is evolved and collected in the upper end.

Tests of procedure 1 using barium sulfate and sodium naphthoquinone sulfonate showed that less than 0.1 microgram of these compounds gave strongly positive results.

2. DETECTION OF SULFUR IN VOLATILE SUBSTANCES. Coat with silver the inner surface of a piece of ordinary (thick-walled) glass tubing of 0.5- to 2-mm. bore by filling with a dilute reagent containing silver nitrate (about 0.1 *N*), ammonium hydroxide, a soluble tartrate, and sodium hydroxide and allowing to stand overnight at room temperature. Empty the tube, wash the interior with distilled water, and dry by warming while a current of air is passed through the tube. Soften one end in a small flame and pull it out to a fine capillary about 2 to 3 mm. long and of 0.1 to 0.2 mm. diameter. Touch the tip of the capillary section to the liquid substance to be tested (if the substance is solid it

may be used molten or dissolved in a sulfur-free solvent); a small quantity of the substance (some tenths of a milligram to several milligrams) will enter. Melt shut the tip of the capillary rapidly by touching it to a hot flame. Apply heat to the tube at a point 2 to 3 cm. from the sealed end, and move the flame gradually toward the sample so as to volatilize it. A slight crackling sound will be heard when the vaporized substance enters the heated zone and decomposes in contact with the silver coating. Allow the tube to cool, break off the tip of the capillary, and allow a droplet of the iodine-azide reagent to enter, observing under the microscope.

The approximate sensitivity limit of this procedure was determined by use of solutions of sulfur in xylene. The tests were performed as "unknowns", the tubes being charged by a coworker. Xylene gave a negative test. With 0.01 microgram of sulfur (0.3 mg. of 0.0033% solution) the result was doubtful. With 0.013 microgram of sulfur (0.4 mg. of 0.0033% solution) the test was positive but not strong, and the same result was noted with 0.02 microgram of sulfur (0.2 mg. of 0.01% solution). Strongly positive tests were observed with sulfur in amounts from 0.05 to 0.12 microgram taken in both concentrations, and a very strong test was given by 0.26 microgram of sulfur (2.6 mg. of 0.01% solution).

LITERATURE CITED

(1) Feigl, Fr., "Qualitative Analyse mit Hilfe von Tüpfelreaktionen", 2nd ed., p. 305, Leipzig, Akademische Verlagsgesellschaft, 1935.
(2) *Ibid.*, p. 378.
(3) Feigl, Fr., *Z. anal. Chem.*, 74, 369 (1928).
(4) Feigl-Matthews, "Qualitative Analysis by Spot Tests", 2nd ed., pp. 195, 386, Elsevier, Amsterdam, and Nordeman Publishing Co., New York, 1939.
(5) *Ibid.*, p. 291.

Final Report on 15-Year Collective Index

The last of the page proof was sent back to the printer on February 12. As this marks the completion of my part in the index, some statistics may not be out of line in this last progress report, as they may be of help in estimating on future indexes.

Job	Subject Index Started	Completed	Hours
Card-making	March 2, 1944	August 1	354
Editing of cards	August 22	October 13	146
Galley proof came in	December 17	January 8, 1945	
Reading and some checking of galleys	December 17	January 14, 1945	105
Page proof came in	February 8	February 9	
Checking page proof	February 8	February 11	20
		Total	625
Work on Author Index			228
		Grand total hours for C.L.B.	853
Work by Mrs. Bernier, checking galleys			97
Work by Mrs. Swank, making Author Index cards, alphabetizing both indexes, reading and checking galley proof on part of Author Index			275
		Total hours for assistants	372

There are 14,500 cards (entries in the Subject Index) and 2250 cross references. The completed Author Index consists of 6750 cards, including cross references.

As to the quality of the index, we who have worked on it feel it to be high—judging from the few corrections on proof, the fact that about 300 references looked up disclosed no mistakes, and because it was designed with the needs of analytical chemists in mind. Because this index contains more information on methods and reagents, it should prove not only the fastest way, but also the surest way, of getting information out of the first 15 years of the ANALYTICAL EDITION. The fact that the index is built very much like *Chemical Abstracts* indexes makes it possible to turn from one to the other with a minimum of shift in search plan; searchers for organic compound names will especially appreciate this. Mistakes and errors occur in every work of this type; to neutralize these, errata published in the ANALYTICAL EDITION—say once a year—would be a big help. I am, of course, wholly responsible for the correctness of the index and will be glad to help in locating bad references and in straightening out other troubles. I anticipate few mistakes or errors.

I want to express here my appreciation for the skillful work of Mrs. Bernier and Mrs. Swank on this index. We are fortunate in having the help of people with the training and experience of these two.

The counsel of M. G. Mellon and E. J. Crane was invaluable. I wish to record my gratitude not alone for the answers and advice which have so greatly improved the index, but also for the kindness with which these men supplied this information.

CHARLES L. BERNIER

BOOK REVIEW

Introduction to Quantitative Analysis. *Saul B. Arenson* and *George Rieveschl, Jr.* 386 pages. Thomas Y. Crowell Co., 432 Fourth Ave., New York, N. Y., 1944. Price, $2.75.

The book contains chapters on fundamentals, chemical equilibrium, acid-base titrimetry, oxidation-reduction titrimetry, precipitation titrimetry, gravimetric analysis, some applications of electrochemistry, photometric methods, and a small section on the mathematical treatment of analytical results. Scattered throughout the text are 79 laboratory experiments and 291 problems. Decimal points in answers to problems are intentionally omitted, and the problems are apparently taken from actual examples met in the classroom and laboratory.

The presentation of the subject matter is good, although it is occasionally wordy and naively informal, as is exemplified in the following introductory sentence to Overtitration, on page 106: "At the beginning of your careers as volumetric analysts you very seldom 'hit the end point on the head'." In other words, the language of the book strongly reflects the language of the laboratory. Questions which may arise in the minds of critical readers are whether potassium tetraoxalate dihydrate is actually a commonly used standard (p. 96), what constitutes a "volumetric analyst" (p. 106), whether it would not be better to give the students the more recent information on standardizing permanganate (p. 164), whether the decomposition temperature of $BaSO_4$ is actually 800° C. (p. 272), and whether the book gives a reasonably strong picture of 8-hydroxyquinoline (p. 277).

The style of the book conveys the feeling of an unusual frankness between student and teacher. Certain rather lengthy direct quotations from chemical journals may have the good effect of arousing the students' interest in such literature and of overcoming the resistance most students have for reading anything outside their own texts. The explanations of the principles of physical chemistry applicable to analytical chemistry are clear and as simple as possible. Reasonable explanations of simple things are given. For example, the desiccator is defined and its limitations are pointed out, so that the student need not make the mistake of assuming that placing an object in the desiccator affords total protection.

As a text for beginners in analytical chemistry, the book deserves consideration by students and teachers because of the unusual method of presentation.

JAMES I. HOFFMAN