

## Microchemical

**Micro-Density Determination of Gases by Direct Weighing.** E. W. Blank. (*Mikrochem.*, 1933, 13, 149–154.)—The density of gases is determined directly by weighing the gas in a small bulb of 8 to 10 c.c. capacity, on a micro-balance to 6 places of decimals at known temperature and pressure, and comparing the weight with that of the same volume of water. The weighing bulb has a narrow neck, in which is a glass stop-cock grooved to prevent loss of tap grease, which would cause errors in weighing the bulb. The outside of the tip of the neck of the bulb is ground to connect with a double T-piece having two glass stop-cocks; this is used for filling and evacuating the bulb. To find the volume of the bulb it is evacuated, removed from the T-piece and filled by opening the stop-cock while the neck is in distilled water at room temperature. The tap is closed, the neck is dried, and the bulb is carefully wiped (the Pregl technique is used), left for half an hour and weighed against a similar bulb as counterpoise. The bulb is similarly treated when filled with the gas. The double T-piece facilitates filling with the gas, so that, if necessary, only one filling of the bulb may be carried out. If there is sufficient gas, it is best to fill the bulb with the gas, to exhaust and then refill the bulb, the operations being carried out in a thermostat, in which the bulb should remain for half an hour before wiping. The gas can readily be recovered for subsequent work, if necessary. Four determinations of the density of carbon dioxide gave a mean value 1.9788, as compared with the accepted value 1.9768. J. W. B.

**“Spot” Tests for Ammonium Salts.** F. Feigl. (*Mikrochem.*, 1933, 13, 129–135.)—A drop of the test solution is placed at the bottom of a micro test-tube (Feigl, *Qualitative Analyse mit Hilfe von Tüpfelreaktionen*, Leipzig, 1931, p. 121), 2.5 by 1.0 cm., and covered with a drop of a 2 per cent. solution of sodium hydroxide. The test-tube is stoppered either with a ground-glass stopper with a hook on the under side, or with a one-holed rubber stopper through which passes a glass rod reaching nearly to the bottom of the test-tube. The reagent for testing the ammonia liberated is placed either on a paper hung on the hook, or in solution on a drop on the end of the glass rod. The test-tube is heated at 40° C. for 5 minutes on an asbestos plate, after which the apparatus is opened and the drop or paper is examined. The following reagents are used for testing the ammonia:—  
(i) *Litmus paper*.—The moist litmus paper is suspended on the glass hook; alkalinity, due to 0.1γ of ammonia, is easily recognised; as little as 0.01γ in

1: 5,000,000 dilution may be detected by comparing the tint of the litmus paper with a similar unused piece. (ii) *Mercurous chloride*.—A drop of a suspension of mercurous chloride is placed on the tip of the glass rod; when the test is completed, the drop is placed on a piece of filter paper, the darkening being visible with as little as 2.5γ of ammonia in 1: 20,000 dilution. (iii) *Nessler's reagent*.—A drop of Nessler's reagent is placed on the tip of the glass rod. When the test is complete, the stopper is removed and the drop is transferred to filter paper. A yellow colour is visible with as little as 0.1γ of ammonia in 1: 500,000 dilution. (iv) *Silver nitrate and formalin solution*.—The reagent is made from 10 c.c. of a 20 per cent. silver nitrate solution and 5 drops of a 40 per cent. formalin solution with a few drops of dilute sodium hydroxide solution, and the mixture is filtered from the precipitated silver. A drop of this is placed on the tip of the glass rod, and, after the test, examined on paper for reduced silver. As little as 0.05γ of ammonia in 1: 1,000,000 dilution can be detected. (v) *Silver nitrate and tannin*.—The reagent is made up from 5 c.c. of a 20 per cent. silver nitrate solution and 1 c.c. of a 5 per cent. tannin solution, allowed to stand for 24 hours, and filtered; it is freshly prepared for use. The test is carried out as (iv) above. As little as 0.1γ of ammonia in 1: 500,000 dilution can be detected. (vi) *Manganese sulphate (or nitrate) and silver sulphate (or nitrate)*.—The reagent is made from 2.87 grms. of manganese nitrate, dissolved in 40 c.c. of water, filtered and mixed with a solution of 1.69 grm. of silver nitrate in 40 c.c. of water; the mixture is diluted to 100 c.c., and neutralised, drop by drop, with dilute sodium hydroxide until a black precipitate forms. It is filtered from this and kept in the dark. A piece of ash-free filter paper is impregnated with the reagent and placed on the glass hook of the apparatus. When the test is complete the slightly darkened filter paper is removed, and a drop of benzidine in acetic acid is added, a deep blue colour being formed with as little as 0.005γ of ammonia in 1: 10,000,000 dilution. J. W. B.

**"Spot" Test to Distinguish Calcite and Aragonite. F. Feigl and H. Leitmeier.** (*Mikrochem.*, 1933, 13, 136–138.)—The test depends on the slightly greater solubility of aragonite in water, and the reaction of the hydroxyl ion formed according to the equations:— $\text{CaCO}_3$  (dissolved)  $\rightleftharpoons \text{Ca}^{++} + \text{CO}_3^{--}$ , and  $2\text{H}_2\text{O} + \text{CO}_3^{--} \rightleftharpoons \text{H}_2\text{CO}_3 + 2\text{OH}^-$ ; and  $\text{Mn}^{++} + 2\text{Ag} + 4\text{OH}^- \rightarrow \text{MnO}_2 + 2\text{Ag} + 2\text{H}_2\text{O}$  [see Test (vi), previous abstract]. *Reagent*.—Solid silver sulphate is mixed with a solution of 11.8 grms. of manganese sulphate ( $\text{MnSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 100 c.c. of water, boiled, cooled and filtered. One or two drops of dilute sodium hydroxide solution are added, and, after one or two hours, the mixture is filtered. The solution must be kept in the dark. *Procedure*.—A little of the powdered mineral is placed on a watch glass on white paper, or on a "spot" plate, and a drop of the reagent is added. If, after two minutes, a blackening is perceptible, aragonite is present; calcite will cause only a slight grey discoloration after 6 or 10 minutes. J. W. B.

**"Spot" Test for the Detection of Free Basic Oxides in Glass. F. Feigl.** (*Mikrochem.*, 1933, 13, 139–140.)—The reaction for hydroxyl ions, used as a test for ammonia, and to distinguish calcite and aragonite (see preceding abstracts),

is used to detect free basic oxides in glass. The manganese sulphate and silver sulphate reagent (see preceding abstract) is used, and a few mgrms. of glass powder are treated with a drop of this. The development of a gray colour, due to the reduced silver and manganese dioxide, indicates the presence of basic oxides; this may then be confirmed by placing the drop on filter paper impregnated with an acetic acid solution of benzidine, when a more or less deep blue colour is formed according to the amount of hydroxyl ions formed from the basic oxides.

J. W. B.

**Micro-determination of Iodine in Eggs.** H. J. Almquist and J. W. Givens. (*Ind. Eng. Chem., Anal. Ed.*, 1933, 5, 254.)—To the liquid contents of a number of eggs are added an equal volume of ethyl alcohol (95 per cent.) and 10 grms. of potassium hydroxide for each egg, and the mixture is gently boiled under reflux for 16 to 24 hours. An amount of the dark brown liquid produced, equivalent to one egg, is evaporated to dryness in a 500-c.c. nickel or pyrex dish, and the residue is ashed at about 600° C. for 4 hours. The ash is extracted with 50 c.c. of hot water and filtered, and the residue is washed and rejected. The filtrate is neutralised to methyl red indicator with 6 *N* sulphuric acid, 5 drops in excess being added, and sufficient bromine water is added to give the liquid a strong yellow colour. The excess of bromine is boiled off, and the solution is evaporated to about 15 c.c. and transferred to a small separating funnel, any crystalline deposit being removed. A crystal of potassium iodide is added, and the iodine formed is extracted with five 1-c.c. portions of pure carbon tetrachloride. The iodine in the extract is determined colorimetrically against a standard solution of iodine in carbon tetrachloride. Corrections for the iodine-content of the reagents should be carried out, following the same procedure. Direct ashing of eggs without the alcoholic potash digestion was found to yield low results, owing to volatilisation of iodine. The method was tested on eggs to which were added 3 to 100γ per egg of iodine in the form of potassium iodide, iodosalicylic acid, or desiccated thyroid gland, and recoveries of very nearly 100 per cent. were obtained. Iodine-contents of 3 to 1750γ per egg have been obtained with untreated eggs.

S. G. C.