

Inorganic Analysis.

Volumetric Benzidine Method for the Determination of Acetic Acid (Acetate Radicle) in Lead Acetate. J. E. S. Han and T. L. Chu. (*Ind. Eng. Chem., Anal. Ed.*, 1931, 3, 379-381.)—The following method depends on precipitating the lead by sulphuric acid in presence of alcohol, removing the excess of sulphuric acid by precipitation as benzidine sulphate, and determining the acetic acid remaining in the solution by titration with sodium hydroxide. The sample of lead acetate (0.94 to 0.95 gm.) is dissolved in 20 c.c. of water, and 26 c.c. of dilute sulphuric acid (0.2 N) are added (both the water and the dilute sulphuric acid must be free from carbon dioxide). Fifty c.c. of neutralised dilute alcohol (prepared by neutralising 96 per cent. alcohol with 0.1 N sodium hydroxide, using phenolphthalein as indicator, and diluting 90 c.c. of this with 81 c.c. of water) are added. The precipitate of lead sulphate is filtered off after 30 minutes and washed with 50 c.c. of the dilute alcohol, followed by 25 c.c. of water. To the filtrate, 5 c.c. of alcoholic benzidine solution (0.921 per cent.) are added; after 5 minutes the precipitate of benzidine sulphate is filtered off (9 cm. paper) and washed with 50 c.c. of water. The filtrate is titrated with 0.1 N sodium hydroxide (free from carbonate), 4 drops of alcoholic phenolphthalein (1 per cent.) being used as indicator. The results of analyses, by this method, of samples of lead acetate from different sources, and also of lead subacetate, show excellent agreement with those obtained by a method involving distillation with phosphoric acid, for the details of which the memoir should be consulted. S. G. C.

Determination of Mercury as Periodate. H. H. Willard and J. J. Thompson. (*Ind. Eng. Chem., Anal. Ed.*, 1931, 3, 398-399.)—The mercury solution (150 c.c.) in dilute nitric acid (not stronger than 0.15 N) or in dilute sulphuric acid (not stronger than 0.1 N) is heated to boiling, and the mercury is precipitated as the orange-red periodate by adding slowly, with constant stirring, a solution of 2 grms. of sodium or potassium periodate in 50 c.c. of water. The cooled liquid is filtered through a crucible with a porous bottom; the precipitate is washed with warm water, dried at 100° C. for 2 to 3 hours, and weighed as $\text{Hg}_5(\text{IO}_6)_2$. Instead of weighing the precipitate, it can be dissolved and the mercury determined in the following manner:—The washed precipitate of mercuric periodate is dissolved in the crucible by adding 2 or 3 grms. of solid potassium iodide and 15 c.c. of water, and stirring. The solution, washed into a flask, is acidified with 10 c.c. of 2 N hydrochloric acid, and the liberated iodine is titrated with 0.1 N sodium thiosulphate solution, with starch as indicator. Iodine is liberated according to the equation



Good results were obtained in test experiments with 0.05 to 0.36 gm. of mercury alone, and in presence of about its own weight of nickel, aluminium, cadmium, zinc and copper. Chlorides interfere. S. G. C.

Electrolytic Determination of Cobalt. D. H. Brophy. (*Ind. Eng. Chem., Anal. Ed.*, 1931, 3, 363–365.)—More or less unsatisfactory results were obtained in the electrolytic determination of cobalt from ammoniacal solution, as recommended by previous investigators. An improvement has been effected by depositing cobalt, from an electrolyte containing 50 c.c. of ammonia (strength not stated), 5 grms. of ammonium chloride and 0.3 gm. of sodium bisulphite in a volume of 100 c.c., upon a platinum-rhodium gauze cathode, employing a platinum anode rotating at 800 to 1000 revs. per minute, and a C.D. of 4 to 7 amps. per sq. dm.; the deposition takes half-an-hour. Good results were obtained with 12.5 to 162 mgrms. of cobalt. For amounts of cobalt below 100 mgrms. it is recommended to use 0.4 gm. of sodium bisulphite in the electrolyte, *i.e.* somewhat more than the normal quantity; apparently, also, it is not clear why more reducing agent is required for the smaller amounts of cobalt. The presence of bisulphite in the solution is essential, but the way in which it acts is not known. A more prolonged period of electrolysis leads to high results, owing to deposition of platinum dissolved from the anode. Cobalt must be separated from “any obviously interfering elements” before the electro-deposition. S. G. C.

Volumetric Determination of Manganese after Oxidation by Periodate. H. H. Willard and J. J. Thompson. (*Ind. Eng. Chem., Anal. Ed.*, 1931, 3, 399–401.)—Willard and Greathouse (*J. Amer. Chem. Soc.*, 1917, 39, 2366) employed potassium periodate to oxidise manganese to permanganate, and determined the permanganate colorimetrically. The present authors have extended this method by precipitating the excess of periodate as mercuric periodate, thus permitting the titration of the permanganate in the ordinary way.

Manganese in steel (containing less than 0.1 per cent. of chromium).—One gm. of the steel is dissolved in a mixture of 15 c.c. of water and 15 c.c. of phosphoric acid (85 per cent.). The solution is oxidised with 1 to 2 c.c. of nitric acid, boiled to remove nitrous fumes, and diluted to 100 c.c.; 0.3 gm. of sodium or potassium periodate (sufficient for up to 15 mgrms. of manganese) are added, the solution is boiled gently for 15 minutes to oxidise the manganese, diluted to 150 c.c., and cooled to room temperature. The periodate is precipitated by adding slowly, with constant stirring, a strong solution of 2 to 3 grms. of mercuric nitrate, the liquid is filtered immediately through an asbestos filter into a measured excess of standard ferrous sulphate solution containing 10 c.c. of sulphuric acid (50 per cent.), and the precipitate is washed with water. The excess of ferrous sulphate is titrated with standard permanganate solution.

Manganese in iron (oxide) ore.—One gm. is heated with 15 c.c. of phosphoric acid (85 per cent.) and a few drops of sulphuric acid until fumes of sulphuric acid appear. The cooled residue is dissolved in water (100 c.c.); the rest of the process is the same as for steel.

Manganese in bronze.—One gm. is dissolved in a mixture of 3 c.c. of concentrated nitric acid, 10 c.c. of phosphoric acid (85 per cent.), and 8 c.c. of water. The liquid is diluted to 50 to 75 c.c., and the process is continued as for steel.

Owing to the deep blue colour of the solution, it is recommended to carry out the final titration with permanganate electrometrically or by the use of alphazurine indicator (*cf.* Knop, *Z. anal. Chem.*, 1929, **77**, 125). S. G. C.

Iodimetric Determination of Chromium. E. Schulek and A. Dozsa. (*Z. anal. Chem.*, 1931, **86**, 81–92.)—The cold solution of chromic sulphate (20 c.c.) is treated with 2 to 5 c.c. of fresh (*i.e.* bromate-free) saturated bromine water, followed by 10 c.c. of 30 per cent. sodium hydroxide solution, and heated only to incipient boiling. After thorough cooling, 5 c.c. of 5 per cent. phenol solution are added all at once, which destroys the excess hypobromite. The solution is next transferred to a conical flask provided with a well-fitting glass stopper, and diluted to 400 c.c.; 4 grms. of potassium bicarbonate are dissolved in the liquid, which is then treated with 50 per cent. sulphuric acid in small portions until its yellowish colour changes to reddish, when potassium iodide (2 grms.) is added. This is followed by more of the sulphuric acid (25 c.c. in all); the flask is then stoppered at once. The iodide is made to react near the neutral point, to prevent any reaction of bromide upon chromate in the more acid medium. After 5 minutes' standing, the solution is titrated with thiosulphate solution; 1 c.c. of 1 *N* = 1.733 mgrm. Cr. For a separation from *iron*, the sodium hydroxide precipitate must be dissolved and re-treated, the filtrates being combined; if the chromium is high, a third precipitation should be made. If *nickel* is present, the combined alkaline filtrates should be acidified with 15 c.c. of sulphuric acid and boiled so that any bromate—formed by induced autoxidation of hypobromite in contact with nickelic oxide—may be decomposed. During this boiling, a small fraction of the chromate is reduced by bromide; hence the cooled solution is oxidised once more with 1 to 2 c.c. of bromine water, caustic soda, etc., with phenol treatment as above. The method should not be used for nickel and chromium-bearing materials high in chromium. W. R. S.

Determination of Gallium. A. Brukl. (*Z. anal. Chem.*, 1931, **86**, 92–94.)—A slight loss in gallium, which has been found to occur when precipitates containing gallium together with organic matter are ignited, has been traced to the formation of the volatile monoxide Ga_2O . This has been prepared by the reduction of the sesquioxide in hydrogen; it begins to sublime at 660°C . at ordinary pressure. The cupferron and tannin precipitates of gallium (ANALYST, 1929, **54**, 64, 267; 1930, **55**, 218) should, therefore, be treated by wet methods for the destruction of the organic matter, and the hydrated sesquioxide precipitated with ammonia. The precipitate is dissolved in nitric acid, the solution is evaporated in a tared platinum crucible, and the residue is ignited to Ga_2O_3 in an electric furnace. The oxide attacks quartz and porcelain at ignition temperatures, and may be partly reduced by diffusing burner gases if the platinum crucible is heated in a gas flame. W. R. S.

Quantitative Determination of Calcium by means of Picrolonic Acid. R. Dworzak and W. Reich-Rohrwig. (*Z. anal. Chem.*, 1931, **86**, 98–113.)—Picrolonic acid (1, *p*-nitrophenyl-3, methyl-4, nitro-pyrazolon 5) gives with solutions

of calcium salts a voluminous, yellowish-green precipitate which changes to a dense, crystalline pale-orange powder, of the composition $\text{Ca}(\text{C}_{10}\text{H}_7\text{N}_4\text{O}_5)_2 \cdot 8\text{H}_2\text{O}$ (Ca factor, 0.05641). The reagent is a 0.01 *N* aqueous filtered solution of the acid, prepared by warming and stirring (2.64 grms. per litre). Its strength may be ascertained by titration with alkali against phenolphthalein. The calcium solution, neutralised to litmus, containing not more than about 0.1 gm. of metal, and not more than 1 gm. of magnesium, alkali, and ammonium salts, is stirred and titrated with the reagent until a precipitate has formed; if it is flocculent, the liquid is warmed and stirred until the precipitate becomes crystalline. Further addition of reagent is regulated by the speed of transformation of the flocculent into the crystalline form, heating and stirring being resorted to if necessary. When the precipitation is complete, an excess of reagent (more than half the volume required for precipitation) is added. After standing several hours, the precipitate is collected in a porous crucible, washed with cold water, dried at ordinary temperature in a current of dust-free air, or by simple standing, and weighed. The salt dried by heating is hygroscopic and contains between 7 and 8 molecules of water. The process is very suitable for micro-work. It provides a quantitative separation of calcium from magnesium, unless the proportion of the latter exceeds 10 times the quantity of the former. For the determination of lime in natural waters, 100 c.c. are treated with the same bulk of reagent. The precipitate forms in the crystalline condition; after some hours it is collected and treated as described.

W. R. S.

Improved Method for the Determination of Magnesium by means of *o*-Hydroxyquinoline. F. L. Hahn. (*Z. anal. Chem.*, 1931, 86, 153–157.)—The reagent is a solution of 38 grms. of the base in 125 to 150 c.c. of 2 *N* hydrochloric acid, made up to one litre. One c.c. precipitates approximately 0.003 gm. of magnesium. The solution (about 20 c.c.) containing 0.005 to 0.1 gm. of metal and contained in a 250 c.c. measuring flask, is treated with 2 to 3 grms. of ammonium chloridé, 2 to 3 c.c. of strong ammonia and 20 c.c. of acetone, heated to water-bath temperature, and titrated gradually with small amounts of the reagent. The precipitate may not form at once; it is very coarse and settles quickly, and hence the colour of the supernatant liquid can easily be observed. The addition of reagent is continued until the hot liquor is as yellow as the precipitate. If much reagent is required, more acetone is advisable (as a solvent for the base). The coloured liquid is diluted with 10 c.c. of acetone, and water to about 200 c.c., stirred, and left for some hours or overnight. It is then made up to the 250 c.c. mark, mixed and filtered, and 100 c.c. are transferred to a porcelain basin. Acetic acid is added until phenolphthalein is decolorised, followed by 5 c.c. of 5 per cent. zinc sulphate solution, which precipitates the excess of base and thus prevents its volatilisation. The solution is heated on the water-bath for half-an-hour to ensure removal of the acetone, and transferred to a conical flask, and the basin is washed with 30 c.c. of strong hydrochloric acid, which is added to the liquid in the flask. This is treated with a known excess of bromate-bromide solution (28 grms. KBrO_3 and

50 grms. KBr per 1000 c.c.), and the excess of bromine is titrated with iodine and thiosulphate (ANALYST, 1927, **52**, 431). For smaller quantities of magnesium (up to 0.01 grm.), 10 c.c. of solution are treated in a 100 c.c. measuring flask, and the quantities of the reagents reduced proportionately.

W. R. S.

Determination of Rubidium and Caesium, particularly in Mineral Waters. L. Fresenius. (*Z. anal. Chem.*, 1931, **86**, 182–190.)—The gravimetric method is based on some recent investigations (ANALYST, 1926, **51**, 162; 1928, **53**, 459; 1930, **55**, 465), combined with an improved process for the elimination of the bulk of the potassium by repeated precipitation with alcoholic hydrochloric acid. An amount of mineral water furnishing 500 to 700 grms. of mixed alkali chlorides is concentrated in a porcelain basin to about 4 litres and boiled with sodium hydroxide and carbonate, the alkaline-earth precipitate being filtered off and washed with dilute sodium carbonate solution. The filtrate is acidified with hydrochloric acid and treated at 80° C. with cobalt nitrate and sodium nitrite. The precipitate is stirred at intervals for a day; after addition of 50 c.c. of acetic acid and another day's standing, it is collected and washed 3 to 4 times with 10 per cent. acetic acid. It contains all the potassium, rubidium, and caesium with a little sodium; it is digested for an hour at 70° C. with ammonia and ammonium sulphide, and the cobalt precipitate is filtered off and washed with dilute ammonium sulphide, and tested for alkali metal. The filtrate is evaporated to dryness with a large excess of hydrochloric acid; this treatment is repeated twice, the last residue being dissolved in very dilute acid and the sulphur filtered off. The boiling filtrate is treated with hot barium chloride solution and filtered, and the excess of barium in the filtrate is precipitated with ammonium carbonate. After another filtration, the liquid is evaporated to dryness, and the residue is gently ignited until the ammonium salts are expelled.

Separation of potassium, rubidium, and caesium.—The residual chlorides are weighed and dissolved in four times their weight of water; the solution is treated with 2.5 times its volume of a mixture of fuming hydrochloric acid and 96 per cent. alcohol (1:2). The precipitate (potassium chloride) is collected after 2 hours, washed 3 times with the alcoholic acid, and treated twice more by the same process. The combined filtrates and washings are evaporated to dryness on a water-bath, the dry residue is weighed, dissolved in 4 parts of water, and the precipitation with the alcoholic acid repeated three times as before. These threefold extractions with alcoholic acid are repeated until the concentrated liquor no longer yields a precipitate of potassium chloride. The solution is then evaporated to dryness, and the residue is dissolved with an equal amount of ferric chloride in a minimum of water; after addition of 50 c.c. of glacial acetic acid per grm. of mixed chlorides, the solution is heated almost to boiling and precipitated with one c.c. of a 40 per cent. solution of antimony trichloride in glacial acetic acid. The precipitate (caesium chloro-antimonite) is left on the water-bath for one hour, and for 12 hours at ordinary temperature. It is collected in a porous-glass crucible, washed with a 5 per cent. solution of antimony chloride in glacial acid, and dissolved in hot water

containing hydrochloric acid. The solution is treated with hydrogen sulphide and filtered, and the filtrate is evaporated with perchloric acid until heavy fumes are given off. When cold, the acid is diluted and stirred with 5 c.c. of 0.3 per cent. alcoholic solution of perchloric acid; after 1 hour, the precipitate is collected in a porous crucible and washed with alcohol, dried at 130° C., and weighed as CsClO_4 , which is tested spectroscopically for purity (especially for potassium and rubidium).

The filtrate from the caesium chloroantimonite is precipitated with hydrogen sulphide, the antimony sulphide is filtered off, and the filtrate is evaporated to dryness. The residue is dissolved in four parts of water, and the solution is diluted with $2\frac{1}{2}$ parts of alcoholic hydrochloric acid (*supra*) and precipitated with 1 c.c. of a boiling 40 per cent. solution of stannic chloride in absolute alcohol. The rubidium chlorostannate is collected after 12 hours in a porous crucible, washed with absolute alcohol, and dried at 110° C. to constant weight. Alternatively, it may be converted into, and weighed as, perchlorate in the same manner as the caesium chloroantimonite. The weighed rubidium compound is tested spectroscopically for purity (potassium and caesium).

W. R. S.

Determination of Fluorine in Phosphate Rock and Phosphate Slag.

D. S. Reynolds and K. D. Jacob. (*Ind. Eng. Chem., Anal. Ed.*, 1931, **3**, 366–370.)

—The fluorine in highly phosphatic, calcareous materials, such as phosphate rock, cannot be brought into solution by a single fusion with an alkaline flux. Three fusions usually fail to convert more than about 90 per cent. of the fluorine into a soluble form. It has been found that during the extraction of the melt with water, fluoride ions react with the other products of the fusion to form insoluble fluoro-phosphates of the fluorapatite type. By extraction of this insoluble residue with dilute nitric acid, however, the fluorine which it contains can be brought into solution, together with calcium and phosphoric acid. The method described below for the analysis involves (a) obtaining the fluorine in solution, (b) separating the silica, etc., (c) determining the fluorine volumetrically by means of the lead chlorofluoride method (the latter two parts of the process are an adaptation of Lundell and Hoffman's method—*Bureau of Stds. J. Research*, 1929, **3**, 581). One gram. of the finely powdered phosphate rock (100 mesh), 0.5 gram. of silica, and 2 grms. of sodium carbonate, are fused in platinum at 900°–950° C. for 1 hour (in the case of phosphate slags, 1 gram. is fused with 5 grms. of sodium carbonate without added silica). The cooled melt is digested overnight on a steam-bath with 50 to 75 c.c. of water, and the liquid is decanted on to a filter. The residue is broken up, digested with 50 c.c. of dilute sodium carbonate solution (1 per cent.) on a steam-bath for 15 minutes, the residue is filtered off on the filter previously used, washed several times with hot water, and the combined filtrates are evaporated to about 50 c.c. volume (solution A, reserved). The residue is washed back into the digestion vessel with about 50 c.c. of water; 3 c.c. of concentrated nitric acid are added; the mixture is kept for 1 hour, with frequent stirring (solution B); any residue is filtered off, fused with sodium carbonate and the extract of this fusion is added to solution A. Fifty c.c. of dilute oxalic acid solution (5 per cent.) are

added to solution B, and the calcium oxalate is precipitated by adding dilute sodium carbonate solution (10 per cent.), drop by drop, until the liquid is neutral to methyl orange indicator. The liquid is boiled for 1 minute, cooled, and filtered, and the precipitate is washed 4 or 5 times with cold water; the precipitate is rejected. The filtrate is acidified with an excess of 4 c.c. of nitric acid, 10 c.c. of saturated potassium permanganate are added; the liquid is warmed, and when the permanganate colour has disappeared, more permanganate solution is added, drop by drop, until the liquid is permanently coloured or a brown precipitate forms. The excess of acid is then neutralised by adding solid sodium carbonate (until frothing ceases), followed by 2 grms. in excess (if the precipitate is light coloured, permanganate solution is added until it becomes dark brown); the liquid is boiled and filtered, and the precipitate is washed with hot sodium carbonate solution (1 per cent.). The filtrate is united with solution A, and the total volume is adjusted to 250 c.c.

Separation of silica, etc.—This solution is heated to boiling, and 25 c.c. of zinc nitrate solution [(5 grms. of zinc oxide dissolved in 100 c.c. of dilute nitric acid (1:9)] are added, with continuous stirring. The liquid is filtered through a rapid-filtering 15 cm. paper; the precipitate (containing a part of the silica) is washed and discarded. The filtrate (400 c.c.) is neutralised to methyl red indicator with nitric acid, 25 c.c. of ammoniacal zinc oxide solution [5 grms. of zinc oxide dissolved in warm water containing 10 per cent. of ammonium carbonate and 10 per cent. of ammonia (sp. gr. 0.90)] are added, and the liquid is evaporated to a volume of 50 c.c. The precipitate (containing the rest of the silica) is filtered off, washed with cold water and discarded.

Determination of the fluorine.—To the filtrate (250 c.c.) 2 drops of brom-phenol blue indicator are added; it is rendered slightly acid with nitric acid and then just alkaline with sodium hydroxide, and 3 c.c. of sodium chloride solution (10 per cent.) and 2 c.c. of dilute hydrochloric acid (1:1) are added, followed by 5 grms. of solid lead nitrate. The liquid is heated on a steam-bath to dissolve the lead nitrate, 5 grms. of sodium acetate are added, and the solution is heated for 30 minutes and kept overnight. The precipitate of lead chlorofluoride is filtered off, washed once with cold water, five times with a saturated solution of lead chlorofluoride, and finally once with water. The precipitate is dissolved in 100 c.c. of warm dilute nitric acid (1:19); a slight excess of 0.1 N silver nitrate solution is added (a total of 20 c.c. is usually sufficient); the liquid is heated on a steam-bath for 30 minutes, cooled and filtered, and the precipitate of silver chloride is washed with cold water. The excess of silver in the solution is titrated with 0.1 N potassium thiocyanate solution, whence the amount of silver required to combine with the chlorine in the lead chlorofluoride is obtained; 1 c.c. of 0.1 N silver nitrate solution = 0.00190 gram. of fluorine. S. G. C.

Colorimetric Method for the Determination of Carbon Dioxide. E. M. Emmert. (*J. Assoc. Off. Agr. Chem.*, 1931, 14, 386–389.)—When carbon dioxide is shaken with a solution of the sodium salt of phenolphthalein the red colour

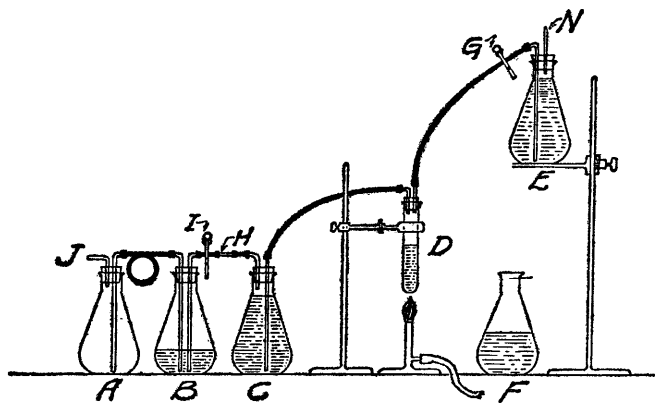
diminishes in intensity in proportion to the amount of carbon dioxide present, owing to the formation of sodium bicarbonate, which removes the sodium from the coloured salt and leaves the phenolphthalein colourless. In using this reaction to determine carbon dioxide, entry of other acidic or basic substances must be prevented by bubbling the gas through 1 per cent. sulphuric acid (10 c.c. of the concentrated acid diluted to 1 litre). The following reagents are used: (1) Neutral alcohol, prepared by adding, to 95 per cent. alcohol, a few drops of 0.5 per cent. phenolphthalein solution and then *N* sodium hydroxide solution dropwise until a faint pink colour persists. (2) Solutions of the coloured sodium salt of phenolphthalein. In each case, 100 c.c. is used, and the strength varies with the amount of carbon dioxide to be determined, as follows:

Amount of carbon dioxide. Mgrms.	Concentration of the sodium salt.
2 to 9	0.0025 <i>N</i>
9 to 18	0.0050 <i>N</i>
18 to 35	0.0100 <i>N</i>
35 to 50	0.0150 <i>N</i>
50 to 70	0.0200 <i>N</i>
70 to 90	0.0250 <i>N</i>
90 to 120	0.0300 <i>N</i>

Slightly more than the amount of phenolphthalein required for 2 litres of solution is dissolved in the neutral 95 per cent. alcohol, made up to 1 litre with the alcohol, and mixed with 1 litre of aqueous solution of the required amount of sodium hydroxide. For accurate work the solutions thus made, which must be kept from the air, should be standardised either by titrating against standard acid or by using the sodium carbonate solution (3) and following the procedure described below. (3) 12.05 grms. of pure anhydrous sodium carbonate are dissolved in 200 c.c. of water and made up to 500 c.c.; 1 c.c. = 10 mgrms. of CO₂. (4) 50 per cent. sulphuric acid (500 c.c. of the acid + 500 c.c. of water).

The liquid or solid, containing 5 to 120 mgrms. of carbon dioxide, is placed in the test tube D (this should be large for large samples), which is filled to within 10 to 15 c.c. with water. One hundred c.c. of the appropriate solution (2) are placed in flask B, which is filled to within 10 to 20 c.c. with 1 per cent. sulphuric acid. F contains reagent (4) and stopcock I should be open. When all connections are made air-tight, the stopper and tubes from E are transferred to F and 10 to 15 c.c. of the reagent (4) forced into D by blowing at N. The cock G is then closed and the stopper and tubes are replaced in E without destroying the siphon formed. If much CO₂ is present, the acid should be blown over slowly. The liquid in D is boiled for several seconds, but not boiled over into C to any extent. The flame is then withdrawn, G is at once opened, and the gases forced from D and C by the 1 per cent. sulphuric acid from E until the liquid reaches the glass tube H (4 to 5 inches long), the clip I being then closed tightly and no acid solution allowed to enter B. The connection at H and the other tubes leading to B should be washed out after each determination. Flask B is disconnected from C at H and is lowered

below A and shaken vigorously until its contents show no further change in colour. If the solution in B becomes colourless or nearly so and much coloured solution has been forced into A, most of the liquid from B is drawn into A by applying suction at J. The suction is then released and the vacuum in B allowed to draw back all the solution it can. If B contains but little solution, this will probably be decolorised several times, repetition of the mixing by suction being thus rendered necessary. If the liquids in both A and B become nearly or quite colourless, either the sample contained too much carbon dioxide or acid has passed into B.



APPARATUS FOR COLLECTING CO_2 OVER THE SODIUM SALT OF PHENOLPHTHALEIN

When change in colour intensity has ceased, the tubes are removed and the solutions in A and B mixed and compared, as regards colour, with the particular reagent used. The quantity (mgrms.) of CO_2 in the sample taken is given by $\frac{Y - YR}{U}$, where Y is the theoretical or determined number of mgrms. of carbon

dioxide equivalent to 100 c.c. of the reagent (2) used, R the original colorimetric reading of the reagent (2), and U the colorimetric reading of the reagent after being shaken with the carbon dioxide. The carbon dioxide of the air enclosed in the shaking flasks introduces an error of 0.1 to 0.2 mgrm., which is of importance when from 2 to 5 mgrms. is to be determined.

The carbon dioxide in a gas may be determined by introducing a known volume of the gas (at definite temperature and pressure) over the reagent (2) by displacement. If the amount of carbon dioxide in the gas is small, it should first be absorbed in concentrated sodium hydroxide solution.

T. H. P.