Reports on Special Departments of Chemical Science.

REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY, JULY TO SEPTEMBER, 1879.

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I. ATOMIC WEIGHTS OF THE ELEMENTS.

DUMAS (Compt. Rend., **86**, 65, through Zeitschr. f. anal. Chem., **18**, 508) communicates several very interesting and important facts on *the presence of oxygen in metallic silver.* He finds, namely, that 1 kilo of pure silver, when heated to 500–600° C., gives off 57 c.c. of oxygen. If it has been melted with nitre for 15 minutes, in the air, it gives off 168 c.c. or even 174 c.c. oxygen. In the determination of the equivalent weight of silver, when it has been melted with borax and nitre, and granulated with access of air, the presence of oxygen in the product exerts a material influence on the accuracy of the results. Thus, the proportion of silver to chlorine, determined by Stas, becomes 108 : 35.50, instead of 108 : 35.47.

Dr. TELLEF DAHL (Chem. News, **40**, 25) states *the equivalent of norwegium,* the new metal discovered by him in copper-nickel from Krageró in Skjaergaarden, to be, apparently, 145.9. The color of the metal is white, with a slight brownish cast. It has, when polished, a perfect metallic lustre, becoming tarnished after some time. It has about the hardness of copper, and may be flattened in an agate mortar. Melting point, 350° C.; sp. gr., 9.441. Only one oxide, NgO, has been obtained. With hydrogen it gives a brown sulphide, even in strongly acid hydrochloric solutions, soluble in ammonium sulphide. The solutions of the metal are blue, becoming greenish on dilution. The sulphuric solution turns brown on addition of zinc, and the metal is deposited in a pulverulent state.

A. GUYARD (Moniteur Scientifique, Quesneville, July, 1879, through Chem. News, **40**, 57) has determined *the atomic weight of uralium,* the metal discovered by him, in 1869, in Russian platinum, to be 187.25. It is, next to silver, the whitest metal known, more ductile than platinum, which it equals in malleability, soft as lead, and possesses a melting point near that of platinum, a sp. gr. = 20.25; atomic volume = 6.25. In its chemical properties it is difficult to distinguish from platinum.

II. GENERAL INORGANIC ANALYSIS.

A. Qualitative.

E. PURGOTTI (Gazz. Chim. Ital. and Berl. Ber., **11**, 1,248, through Zeitschr. f. anal. Chem., **18**, 476) recommends *tincture of guaiacum as a reagent for copper.* According to the author, even very dilute solutions of cupric chloride are reduced to cuprous chloride by this reagent, with production of a blue tint. As most oxygen salts of copper undergo double decomposition with formation of cupric chloride, when brought in contact with a solution of an alkaline chloride, the author adds the latter before testing. If 1 c.c. of solution containing $\frac{1}{1000}$ mgrm of cupric sulphate, be mixed with a solution of an alkaline chloride, and allowed to flow down the sides of a vessel containing alcoholic tincture of guaiacum, the blue color is distinctly perceptible. Heinrich Fresenius remarks to the foregoing that Purgotti's observations are not new, having been anticipated by Schoenn (Zeitschr. f. anal. Chem., **9**, 210) in his paper on the use of tincture of guaiacum as a reagent.

E. J. CHAPMAN (Chem. News, **35**, 13, 26, 36, through Zeitschr f. anal. Chem., **18**, 477) has pronounced the well known *Turner's reaction for boracic acid*, by examining the color communicated to the flame by admixture of the substance with a mixture of 4½ parts potassium acid sulphate and 1 part calcium fluoride, to be worthless, as it gives no result with sodium borate, and as the other borates give the reaction without the flux. C. Le Neve Foster (idem, **35**, 127) shows, on the contrary, that even a mixture of 99 parts sodium chloride and 1 part sodium borate, will give a distinct green flame reaction.

B. Quantitative.

ALEXANDER CLASSEN (Zeitschr. f. anal. Chem., **18**, 373) has developed *a new method of quantitative analysis susceptible of numerous applications*, from the principle on which is based his method of separating ferric oxide and alumina from manganese, etc. (idem., **18**, 175 and 189, and this JOURNAL, **1**, 325 and 327).

The estimation of magnesia is based upon the fact that magnesium forms a soluble double oxalate with potassium or ammonium oxalate, which, on the addition of acetic acid, is decomposed, with separation of magnesium oxalate. The quantitative separation of the magnesium is effected by adding to the solution, which should measure about 25 c.c., a hot saturated solution of ammonium oxalate, or by dissolving with the aid of heat the solid ammonium oxalate therein. When sufficient of the reagent has been added, the solution is perfectly clear. On adding to the boiling liquid an equal volume of 80 per cent. acetic acid, and boiling, with stirring, for a few minutes longer, a heavy, crystalline precipitate of magnesium oxalate forms, and settles rapidly to the bottom of the vessel, when the ebullition is discontinued. Small quantities of magnesium precipitate only after some time. In all cases the liquid is kept in a covered beaker for about 6 hours, at a temperature of about 50° C., then filtered, and the precipitate washed completely with a mixture of equal volumes of concentrated acetic acid, alcohol and water. The precipitate is enveloped in the filter, and converted by ignition in a covered platinum crucible into magnesium oxide. The heat employed must be very gentle, as long as vapors are evolved, and must not be raised until the cover has been removed, and the carbon of the filter has become entirely consumed, and the residue white. The conversion to oxide is then completed by exposure to a red heat. A series of 34 closely accordant determinations by the author, using from 0.0377 to 0.2075 grm of pure magnesia, gave an average result of 99.41 per cent. The presence of ammonium chloride materially interferes with the accuracy of the determination, the analytical results of the author showing that the loss of magnesium is, to a certain extent, proportional to the ratio between the ammonium chloride and magnesium present.

The separation of magnesia from the alkalies by this method, may be effected in solutions of the chlorides, sulphates or nitrates; the solutions must, however, be more dilute than in the preceding instance, owing to the tendency to the formation, in concentrated solutions, of double oxalates of magnesium and the alkaline metals, which are not decomposed by the further treatment of the precipitate. If, however, the liquid be diluted to about 50 c.c., and treated in the cold with a cold saturated (1–24) solution of ammonium oxalate, the magnesium oxalate may then be precipitated in such a state of purity, that it is only with the help of spectral analysis that the imponderable traces of the alkaline metals may be detected therein. The precipitation from more dilute solutions cannot, however, be effected without a slight loss. By the addition of about $\frac{1}{3}$ volume of alcohol to the cold liquid, after the precipitation of the magnesium oxalate, almost the whole of the magnesia is obtained. The magnesium oxalate, thus precipitated, does not show in the spectroscope a larger proportion of the alkali metals than that precipitated without alcohol. The analytical results obtained by the author are eminently satisfactory.

The separation of ferric oxide and alumina from manganese, zinc, cobalt and nickel, by the older methods (precipitation as basic acetate or oxide), is tedious, and in the case of the three metals last named, imperfect. It is necessary to repeat the precipitation wherever accuracy is required, but in the case of cobalt and nickel, even after five-fold precipitation, the filtrate still gives the reactions for those metals. In a mixture containing 1.35 grm ferric oxide, and 0.1454 grm metallic cobalt, the author obtained by *double* precipitation with ammonium carbonate, but 98 per cent. of the cobalt taken. A similar experiment with 0.1898 grm nickel and 1.35 grm ferric oxide, gave 99.2 per cent. of the amount of the former taken. In a *double* precipitation with sodium acetate, the loss of nickel amounted to 4.34 per cent. The separation of 0.2245 grm of zinc oxide from 1.35 grm ferric oxide, yielded, by one precipitation with ammonium carbonate, 96.39 per cent. of the former. The separation of 0.2814 grm zinc oxide from 1.35 grm ferric oxide, by one precipitation with sodium acetate, yielded but 94.77 per cent. In the previous communication above cited, the author has minutely indicated the precautions required in the separation of *manganese* as oxalate, with addition of zinc chloride ; his more recent researches have shown that the latter substance may, with the most satisfactory results, be replaced by magnesium, or calcium chloride. In order that the manganese should be present in the residue in the state of Mn_2O_3, it is necessary that for each molecule of manganic oxide, there should be present not less than one molecule of magnesium, or calcium oxide, otherwise the residue of ignition will contain manganoso-manganic oxide. The analytical results obtained by the author are extremely satisfactory ; as an instance, I will state that a mixture containing manganous oxide, 0.05 grm, ferric oxide, 0.36 grm, alumina, 0.21 grm, magnesia, 1.6 grm, lime, 0.0935 grm, and phosphoric anhydride, 0.016 grm, yielded manganous oxide, 0.0507 grm. In the separation of *zinc*, it is indifferent whether the metal be present as chloride, bromide, nitrate or sulphate, although, in the latter case, owing to the low solubility of potassium sulphate, the solution should be more dilute than would otherwise be necessary. In all cases, the free acid must be completely expelled by evaporation, the substance moistened with a few drops of nitric acid, and digested in the water-bath for a short time, to insure the complete oxidation of the iron. About seven times the weight of the oxide of solution of neutral potassium oxalate (1 : 3) is added, and the whole warmed about one-quarter hour on the water-bath. The undissolved residue of ferric oxide is brought into solution by the addition, drop by drop, of acetic acid. If sufficient oxalate has been added, the solution will be perfectly clear. On heating the solution to boiling, and adding, at least an equal volume of acetic acid (80 per cent.), the zinc is entirely precipitated as crystalline oxalate, leaving the ferric oxide and alumina in solution. The beaker is allowed to stand for 6 hours, at the temperature of about 50° C., and the precipitate filtered off while still hot, washed with a mixture of equal parts concentrated acetic acid, water and alcohol, and converted into oxide by ignition. The separation of ferric oxide and alumina from *cobalt*, is conducted in the same manner, as in the case of zinc ; here, also, any free sulphuric acid that may be present must be completely expelled by evaporation. The cobaltous oxalate is ignited, washed with boiling water, and reduced to metal by ignition in a stream of hydrogen, before weighing. The separation of nickel is conducted, also, in the same manner, as that of zinc and cobalt. The nickel oxalate is not so distinctly crystalline, as the oxalates previously enumerated ; hence, too large quantities of substance should not be taken.

The separation and estimation of copper may, also, be made by the foregoing method ; the manipulations recommended in the cases of zinc, cobalt and nickel, however, give a very finely divided precipitate of cupric oxalate, which does not readily subside. When a concentrated neutral cupric solution is treated with a sufficiency of potassium oxalate, and allowed to stand for some time, potassio-cupric oxalate crystallizes in handsome, blue needles ; if now an equal volume of acetic acid be added, and the liquid be allowed to stand for some time, the filtrate will be entirely free from copper. The copper in the residue may be determined, gravimetrically, by ignition, thoroughly washing, and ignition with access of air, until the weight s constant, or by electrolysis of the solution obtained by digesting the cupric oxalate with dilute sulphuric acid. Copper may be separated from ferric oxide and alumina in the same manner as zinc, cobalt and nickel. The author prefers to destroy the oxalates by ignition, dissolve the resulting oxides in dilute sulphuric acid, and determine the copper by electrolysis ; the presence of zinc or magnesium does not interfere with the purity of the electrolytic copper. When antimony or arsenic is present, the foregoing method must be somewhat modified ; the presence of the latter substance tends to retard the precipitation of cupric oxalate, while antimony usually contaminates the electrolytic copper. When arsenic and iron, with but little antimony, are present, the substance is dissolved in nitric acid, evaporated to dryness, the residue treated with excess of solution of potassium oxalate, filtered hot, and the residue washed with water containing some potassium oxalate. If the filtrate is then evaporated to about

50 c.c., the greater part of the potassio-cupric oxalate crystallizes out in blue needles ; the residue may be precipitated from the solution by adding one or two volumes of acetic acid, and proceeding as above described. If antimony is present in considerable quantity, the finely divided substance is mixed with about four times its weight of ammonium chloride, and heated very gently, in a porcelain crucible ; most of the arsenic and antimony, and a large part of the iron, are thus expelled; the residue is dissolved, and treated as above.

Phosphoric acid may be separated by this method from all oxides which form insoluble oxalates (*e. g.*, lime), or which form, with potassium oxalate, soluble, double salts, decomposable by acetic acid. From aluminum and ferric oxalates it may be separated by precipitating the latter by addition of alcohol. The solution may then be directly precipitated with magnesium chloride solution. The author made two series of experiments, using 1st, mixtures of calcium phosphate and ferric oxide, and 2nd, mixtures of calcium and aluminum phosphates, magnesium and ferric pyrophosphates, with ferric, aluminum, calcium and magnesium oxides. The substance was dissolved in hydrochloric acid, evaporated to dryness to expel free acid, about six times as much potassium oxalate added as there were oxides present, digested a short time on the water-bath, the undissolved residue of ferric oxide dissolved by addition of acetic acid, excess of acetic acid added, and then alcohol (95 per cent.), as long as a precipitate forms. The liquid was allowed to stand, warm, for about 6 hours, during which time the precipitate became crystalline, and the liquid colorless ; it was then filtered, the precipitate washed by decantation with alcohol, and the filtrate evaporated to expel alcohol and acetic acid. The evaporation should be conducted in a beaker instead of an evaporating basin, owing to the tendency of the crystals to *creep* over the sides of the latter. The nearly dry residue was then dissolved in water, and, after standing some time, filtered from any silica that may have separated. The filtrate was treated with ammonia, and then precipitated with magnesium chloride. The following results were obtained :

First Series.

TAKEN.				FOUND.	DIFFERENCE.
Calcium Phosphate.	Containing Phosphoric Anhydride.	Ferric Oxide.	$P_2O_5 : MO$	Phosphoric Anhydride.	
0.0343 grm	0.0157 grm	0.194 grm	1 : 13.50	0.0170 grm	+ 0.0013 grm
0.0240 "	0.0110 "	13.290 "	1 : 1216.00	0.0112 "	+0.0002 "
0.1320 "	0.0604 "	0.717 "	1 : 13.00	0.0600 "	− 0.0004 "
0.0463 "	0.0212 "	0.717 "	1 : 35.00	0.0183 "	− 0.0029 "
0.0316 "	0.0145 "	3.542 "	1 : 245.00	0.0160 "	+ 0.0015 "
0.0264 "	0.0121 "	7.195 "	1 : 596.00	0.0128 "	+0.0007 "

Second Series.

MIXTURE TAKEN.	CONTAINING.						FOUND.	DIF'R'NCE.
	Phoshoric Anhydride.	Ferric Oxide.	Alumina.	Lime.	Magnesia.	$P_2O_5 : MO$	Phosphoric Anhydride.	
0.0396 g.	0.0199 g.	0.738 g.	0.429 g.	0.206 g.	0.005 g.	1 : 69.00	0.0260 g.	+0.0061 g.*
0.0197 g.	0.0099 g.	0.736 g.	0.003 g.	0.003 g.	0.290 g.	1 : 104.00	0.0108 g.	+0.0009 g.
0.0230 g.	0.0116 g.	0.737 g.	0.218 g.	0.004 g.	0.004 g.	1 : 83.00	0.0144 g.	+0.0028 g.
0.0330 g.	0.0166 g.	0.005 g.	0.635 g.	0.445 g.	0.445 g.	1 : 92.00	0.0163 g.	−0.0030 g.
0.0472 g.	0.0238 g.	1.470 g.	0.005 g.	0.185 g.	0.185 g.	1 : 77.50	0.0249 g.	+0.0011 g.
0.0254 g.	0.0128 g.	0.163 g.	0.865 g.	0.155 g.	0.075 g.	1 : 98.20	0.0138 g.	+0.0010 g.

Arsenic acid may be separated (*e. g.*, from cobalt, nickel and zinc) in the same manner as phosphoric acid.

In applying the foregoing method to the determination of phosphorus in cast iron, the author recommends to dissolve in a dilute solution of bromine in hydrochloric acid ; the solution is evaporated to dryness, and the silica separated as usual. In order to separate the iron after it has been converted into potassio-ferric oxalate, the hot concentrated liquid is, after addition of acetic acid, allowed to cool ; the greater portion of the ferric double salt thus crystallizes out, the residue is precipitated with alcohol. The operation is much more rapid and convenient than that with ammonium molybdate. The author communicates numerous cases to illustrate the adaptation of his method to special technical problems, for which the reader is referred to the original.

F. Beilstein and L. Jawein (Berl. Ber., **12**, 1,528) communicate the following *new methods for the direct separation of manganese from iron*, which appear to unite the conditions of extreme simplicity and rapidity of execution, with a high degree of accuracy in the results :

First method.—The solution of the two metals is poured, in the cold, into a concentrated aqueous solution of potassium cyanide, the solution is filtered, the slight precipitate of ferric oxide which always remains, and which does not dissolve on further addition of cyanide, is dissolved in a few drops of dilute hydrochloric acid, and added to the original solution, after adding an excess of cyanide solution to the latter. Iodine is added, until the solution assumes a brown color, and then a few drops of alkali to remove the free iodine. The manganese is precipitated, theoretically, according to the equation :

$$4KCyMnCy_2 + 14I + 2H_2O = MnO_2 + 4HI + 4KI + 6CyI.$$

In practice, however, it is obtained as a mixture of various oxides. To ascertain if the precipitation is complete, a little of the clear liquid is treated, first with iodine, and then with potassa or soda solution; it

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* The precipitate contained iron.

should remain perfectly clear. The precipitate is filtered, washed, re-dissolved in chlorhydric acid, precipitated boiling (as Finkener directs) with ammonium sulphide, and weighed as manganese sulphide. From a mixture of 0.1090 grm manganese, and 0.1700 grm iron, the author obtained 0.1098 grm manganese. About 90 per cent. of the iodine used may be recovered by the simple addition of crude nitric acid to the filtrate. It is preferable to have the iron present as a ferric salt.

Second method.—This is based on the fact, discovered by the authors, that on boiling a salt of manganese with nitric acid and potassium chlorate, the manganese is entirely precipitated as dioxide. The iron and manganese compound is dissolved in nitric acid (sp. gr. = 1.35), the solution heated to boiling, and potassium chlorate added in small portions. In a short time all the manganese is precipitated, as may be demonstrated by boiling a small portion of the clear liquid with a fresh addition of potassium chlorate. The precipitate is not entirely free from iron; it may either be dissolved in chlorhydric acid, evaporated to dryness, re-dissolved in nitric acid, and, by boiling with potassium chlorate, re-precipitated with only a very slight trace of iron, or the manganese it contains may be determined by the modified Bunsen's chlorimetric method, using sodium hyposulphite. By the latter method the authors obtained from manganous sulphate in two experiments, 0.1047 and 0.1075, mean 0.1061 grm. instead of 0.1058 grm manganese. 3.0068 grms steel gave, when treated by the method with sodium acetate, and weighing the manganese as sulphide, 1.930 per cent. Mn; when treated by the foregoing method by double precipitation, and re-solution and precipitation of the manganese dioxide as manganous sulphide, 1.963 per cent. The precipitation of the manganese as dioxide is complete in a few minutes, and the precipitate filters and washes well.

J. B. SCHOBER (Bay. Industrie- u. Gewerbebl., Nov. and Dec., 1878, through Zeitschr. f. anal. Chem., **18**, 467) has devised the following *method for the volumetric determination of zinc.* The zinc solution is treated with excess of ammonia, and then with standard solution of pure potassium sulphide (free from chlorine), until a drop browns paper saturated with plumbic acetate; the solution is then diluted to a known volume and filtered. A measured volume of the filtrate is then treated with a measured volume of ammoniacal deci-normal silver solution, more than sufficient to decompose the potassium sulphide it contains, the liquid filtered, and in an aliquot part of the filtrate the silver determined by titration with ammonium sulpho-cyanide, with ferric nitrate as an indicator, after the method of Volhard (Ann. d. Chem., **190**, 1, and this JOURNAL, **1**, 272).

If A be the volume of the mixture of zinc and sulphide solutions,

A_1, the volume of the portion of the filtrate from A taken,

B, the volume of the mixture of A_1 and the silver solution,

B_1, the volume of the filtrate from B, taken for titration by Volhard's method,

a, the volume of silver solution, corresponding to 1 c.c. of sulphide solution,

b, the quantity of zinc, corresponding to 1 c.c. of the sulphide solution,

n, the volume of the sulphide solution added,

p, the volume of the silver solution added to A_1,

q, the volume of sulpho-cyanide solution added to B_1,

x, the weight of zinc sought,

then, on the assumption that $2A_1 = A$, and $2B_1 = B$, we have the value of x by the equation:

$$x = \frac{(an - 2p + 4q)\,b}{a}$$

NOTE:—The foregoing method is much more complicated than that of C. MANN (Zeitschr. f. anal. Chem., **18**, 162, and this JOURNAL, **1**, 329) for the volumetric estimation of zinc by Volhard's method, and manifestly inferior thereto in essential particulars, especially in respect to the fact that the precipitation of hydrogen sulphide in acetic acid solution, which forms the first step in the latter method, also insures the separation of the manganese, which is such a frequent ingredient in the zinciferous minerals of this country.

J. HERTZ (Archiv. f. Pharm. [3 R.], **14**, 322, through Zeitschr. f. anal. Chem., **18**, 475) communicates the results of his experience with *Volhard's method for the determination of silver and the halogens.* The author finds the method to be extremely practicable, and that it combines the advantages of great accuracy, with ease and rapidity of execution.

CHARLES A. CAMERON (Chem. News, **38**, 145, and Analyst, Oct., 1878, 338, through Zeitschr. f. anal. Chem., **18**, 475) recommends *the determination of lead as lead iodate.* According to the author, iodic acid and iodates precipitate lead much more completely than sulphuric acid. The estimation may be made either gravimetrically or volumetrically; in the latter case, a normal solution of a soluble iodate, standardized with lead nitrate, is added in excess, the precipitate filtered off and washed, and the excess of iodic acid in the filtrate determined by titration with hydrochloric acid and sodium thiosulphate. The iodate of lead is not insoluble in the alkaline haloid compounds; hence, these compounds must not be present. Hydrochloric acid dissolves it readily.

W. Knop (Landwirthsch. Versuchsstationen, **23**, 199, through Zeitschr. f. anal. Chem., **18**, 462) has, in conjunction with Hazard, devised the following *method for the determination of potassium and sodium in silicates*, which he considers to be preferable to that ordinarily used. The finely pulverized mineral is decomposed, as usual, with hydrofluoric and sulphuric acids; the residue is, if necessary after addition of a few drops of concentrated sulphuric acid, dissolved in warm water, and treated with crystallized barium hydrate to alkaline reaction. The precipitate of silica, alumina, magnesia and ferric oxide, is filtered off and well washed. The filtrate is evaporated to 200 c.c., treated with a few grms of solid ammonium sesquicarbonate, and evaporated to dryness. The residue is extracted with water 15 times, using each time 20 c.c., each time pouring the liquid through a filter 3-4 c.m. in diameter. The filtrate is evaporated to dryness in a platinum dish, the residue extracted as before, and again evaporated, with addition of a few bits of ammonium carbonate, and re-dissolved, and, if no insoluble residue is left, the liquid is neutralized with hydrochloric acid, evaporated, and the mixed chlorides dried, weighed, and separated by the usual methods.

H. Precht (Corr.-bl. des Vereins analyt. Chemiker) states that the method of Carnot (Comptes Rendus, **86**, 478, and this Journal, **1**, 333) for *the volumetric determination of potassium*, based on the precipitation of the latter as potassio-bismuth hyposulphite, does not afford reliable results.

A. Carnot (Bull. Soc. chim. de Paris, **32**, 161) recommends to the attention of chemists *the employment of hydrogen sulphide, in the dry way, in analysis*, which was proposed some time ago by Ebelman (Ann. d. chim. et phys., 1849). At higher temperatures, this gas converts completely to sulphides the metallic oxides, carbonates, sulphates and arseniates, which transformation could only be effected by repeated fusions with sulphur alone. The oxides, sulphates, arseniates and chlorides, of *manganese*, may be readily converted to MnS, of constant composition. *Iron* may also be transformed into sulphide, FeS, but the composition is only constant when the current of hydrogen sulphide is succeeded by one of hydrogen at a high red heat. *Cobalt* does not give a sulphide of definite composition. *Nickel* gives a bronzeyellow NiS. *Zinc* may be converted without loss to ZnS. *Cadmium* undergoes a similar change to CdS. *Copper* gives a sulphide, the composition of which is then only Cu_2S, when the experiment is finished with a current of hydrogen. *Silver* in any form of combination, is readily transformed into Ag_2S. Oxide, chloride, carbonate and sulphate of *lead*, are readily converted into PbS. *Bismuth* is readily converted to Bi_2S_3, by igniting the precipitated sulphide. Antimony sulphide may be freed from free sulphur and converted to Sb_2S_3, by ignition at a temperature below dark redness. The alcohol flame is convenient for this purpose. Tin is converted with greater or less difficulty to SnS_2; the conversion is not, however, always exact, but may serve to simplify other methods, *e.g.*, tin solder is readily transformed to a mixture of tin and lead sulphides, which may readily be separated by means of hydrochloric acid, etc. An analogous process serves for the separation of tin from tungsten, WS_2 being insoluble in acids.

F. A. Gooch (Proc. Am. Acad., Oct. 8, 1879) has, at the request of Dr. Wolcott Gibbs, subjected to an exceedingly thorough investigation, the *method for the determination of phosphoric acid as magnesium pyrophosphate*, with the object of ascertaining the best mode of conducting this determination, concerning which so many conflicting accounts have been published by earlier investigators. The general results attained by the author, in the course of 129 determinations, made under the most diverse conditions, are given in his own words, as follows: "It would appear, from the preceding account, that in determining the phosphoric oxide of alkaline phosphates, free from sulphates, or other substances likely to contaminate a precipitate, accuracy is most conveniently and surely attained, by adding to the somewhat dilute solution of the phosphate, ammonia, in slight, but quite distinct excess, then an excess of magnesic chloride mixture, containing no free ammonia (made by dissolving three parts of crystallized magnesic chloride and eight parts of ammonium chloride, in water, adding water containing ammonia, until the volume of the solution reaches forty-eight parts, filtering and boiling off the free ammonia), and, after the precipitate has settled, ammonia, until the liquid evolves a strong odor of it. In determining the phosphoric oxide of phospho-tungstates, the best results are to be got by adding to the solution containing free ammonia, an excess of either of the magnesia mixtures, washing the precipitate with the precipitant, dissolving in hydrochloric acid, diluting, if necessary, and re-precipitating with a little dilute ammonia, adding strong ammonia after the precipitate has settled; or, when working with small amounts, by proceeding as in the case of the phosphates, taking special care, however, that the solution is distinctly ammoniacal before precipitating, and that no great excess of the precipitant is used. In the determination of the phosphoric oxide of phospho-molybdates, the method recommended for use, in the case of the phosphates, serves very well when the amount of phosphoric oxide does not exceed (let us say) 0.05 grm, but for amounts larger than this, the method of double precipitation recommended for the phospho-tungstates is more accurate.

"As to the time which should be allowed to pass between precipitating and filtering, my experiments support those of Abesser, Jani and Maercker (Zeitschr. f. anal Chem., **12**. 250), in pointing to the conclusion, that a precipitate may be filtered with safety, as soon as it has completely subsided, or after ten or fifteen minutes."

By the use of the author's method of rapid filtration (Proc. Am. Acad., 1878, 342, and this Report), he succeeded, with two perforated crucibles, in making seven determinations of phosphoric oxide, in alkaline phosphates, in four and one-half hours; the ignition of each precipitate requiring less than five minutes.

R. W. ATKINSON (Chem. News, **35**, 127, through Zeitschr. f. anal. Chem., **18**, 477) finds in contradiction to the results obtained by E. H. Jenkins (Journ. f. prakt. Chem. [N. F.], **13**, 237), that in *the determination of phosphoric acid with ammonium molybdate*, the silicic acid, if present, will precipitate with the ammonium phospho-molybdate, and must be separated before proceeding to transform the phosphoric acid into ammonio-magnesium phosphate.

OTTO HEHNER (Analyst, **4**, 23) communicates an *expeditious method for the determination of phosphoric acid as phospho-molybdate.* The author prepares the molybdic solution according to Fresenius, *i.e.*, dissolves 1 part molybdic acid in 4 parts ammonia (sp. gr. = 0.96), and pours the solution slowly into 15 parts HNO_3 (sp. gr. = 1.2), avoiding all rise of temperature. This solution contains 5 per cent. molybdic acid. By carefully neutralizing all excess of free acid, especially HCl, with NH_3, before adding the molybdic solution, and carefully avoiding elevation of temperature above 30–35° C., the author succeeded in avoiding the contamination of the precipitated phospho-molybdate with free molybdic acid, and in obtaining it of constant composition. The precipitation is complete in two or three hours. The precipitate is filtered off, washed with dilute (45 per cent.) alcohol (of which 13,513 parts dissolve but one part of the phospho-molybdate, or 378,000 parts an amount corresponding to one part of phosphoric acid), and re-dissolved in ammonia water. The filtered solution is evaporated to dryness, a little water added, and re-evaporated, the operation being repeated until the weight is constant. The residue then represents a mixture of ammonium phosphate and an acid ammonium molybdate, and contains $\frac{1}{28.5}$ of its weight of phosphoric anhydride. The analytical results communicated by the author, show the method to be as exact as that of conversion to magnesium pyrophosphate, while a determination can be made with it in one day.

Samuel L. Penfield (Am. Chem. Journ., I, 27) has devised *a new volumetric method for the determination of fluorine* based on the conversion of the fluorine into hydrofluo-silicic acid, the conversion of the latter to potassium silico-fluoride by treatment with potassium chloride and alcohol, and titration of the hydrochloric acid thus set free. Hydrofluo-silicic acid cannot be directly titrated, because as soon as an alkaline reaction is reached, the silico-fluoride is decomposed, and the acid reaction reappears. Barium chloride cannot be used in the place of potassium chloride, for the reason that, in the former case, the turbidity produced masks the end-reaction. The potassium silico-fluoride is a very transparent precipitate, and permits the change in color of the litmus to be readily perceived. The author proceeds as follows: the fluoride is weighed into a 150 c.c. flask and, unless it is a silicate, 10 grms of powdered and ignited quartz added. Two or three pieces of quartz of the size of kidney beans are also introduced, to facilitate the mixing when the flask is shaken; 30 to 40 c.c. sulphuric acid, previously heated and then cooled, are added, and the flask tightly closed with a doubly perforated cork. Through one of the perforations is passed a tube, by means of which dry air from a gasometer is conducted to the bottom of the flask; through the other, a tube connected with a U-tube 5 m.m. in diameter, kept cool by immersion in a beaker of water, to retain any sulphuric acid that may come over, conveys the mixture of gas and air to a plain U-tube, 18 c.m. long and 2½ c.m. in diameter, containing a mixture of equal volumes of a solution of potassium chloride and alcohol; after bubbling through this, the gas passes through a second smaller U-tube, where the last traces of acid are absorbed. The decomposing flask is placed on an iron plate, heated by a lamp, and by its side is placed a second flask, containing sulphuric acid and a thermometer so supported that its bulb is immersed in the acid. The lamp under the plate is placed midway between the two flasks, and the temperature maintained constant between 150° and 166° C. In ordinary cases the decomposition lasts two hours, during which time from 5 to 6 liters of dry air are forced through the apparatus.

After the decomposition is finished, the acid may be titrated either in the two large U-tubes, or the contents of the latter may be first transferred into a beaker, and the tubes rinsed out with alcohol and water (equal volumes). In order that the alcohol at the end of the titration may make up one-half of the volume of the liquid, add a few cubic centimeters before titration, or use a standard alkali, of which one-half the volume is alcohol. If the mineral contains chlorine, there is substituted for the empty tube one filled with fragments of pumice impregnated with perfectly anhydrous cupric sulphate. In the calculation of the results, every single equivalent of sodium carbonate is equal to one of hydrofluo-silicic acid, or six of fluorine, according to the proportion:

Mol. wt. of Na_2CO_3		Mol. wt. of 6Fl.		Amt. Na_2CO_3		Amt. Fl.
106	:	114	=	1	:	x

The analytical results obtained by the author with pure fluor spar are in the highest degree satisfactory.

A. Colson (Bull. Soc. chim. de Paris, **32**, 115) recommends that *the determination of sulphur in certain natural sulphurets, etc.*, should be effected by combustion in oxygen, and conduction of the evolved gases into a titrated solution of sodium hydrate contained in Liebig's potash bulbs. The anterior end of the combustion tube is washed, and the washings, together with the contents of the bulbs, are brought to a definite volume; the sulphurous acid in one aliquot part is estimated by titration with iodine, after addition of hydrochloric acid; in another, after addition of sufficient iodine solution to oxidize the sulphurous acid, the excess of free alkali is determined by titration with litmus and standard acids, or the sulphuric acid therein may be determined by titration with barium chloride and sulphuric acid.

Note.—It would seem that the oxidation of the sulphurous acid in the foregoing method might, with great advantage, be effected by hydrogen peroxide, which is now an article of commerce, and has been recommended for similar applications, by G. E. Davis (Chem. News, **39**, 221, and this Journal, **1**, 335).

R. Warington (Journ. Chem. Soc., 1879, 375) has subjected to a thorough investigation the method of Crum & Frankland, for *the determination of nitric acid as nitric oxide, by means of its action on mercury*. The author, in his experiments, followed the directions of W. Thorp (Sutton's Volumetric Analysis, 3d ed., 1876, 316) for the conduct of the method. The nitrate solution and rinsings measured 2 c.c., the oil of vitriol 5 c.c.; these were agitated with mercury in a graduated shaking tube, and the gas produced transferred to the laboratory tube of Frankland's latest modification of Regnault's gasometric apparatus, wherein it was accurately measured. The author concludes from numerous experiments:

1. That in the absence of organic matter, and with proper manipulation of the shaking tube, the method is one of great accuracy, and capable of determining extremely small quantities of nitrates and nitrites.

2. That the natural error of the method is a small one of deficiency. An imperfect reaction in the shaking tube may, however, take place from faulty manipulation or other causes, through which low results may be obtained. In such cases, the missing gas will be slowly evolved in the laboratory vessel.

3. That the presence of chlorides, at least in moderate quantity, is no hindrance, as was supposed, to the success of the method.

4. That, while quantities of organic matter, small in relation to the nitrates, have little or no effect, larger quantities may cause a very considerable deficiency. The presence of chlorides reduces this error, but fails to remove it entirely.

R. Warington (Journ. Chem. Soc., 1879, 578) communicates the results of his investigations *on the determination of nitric acid, by means of indigo, with special reference to water analysis.* The paper is devoted to the enumeration of the precautions essential to the attainment of the highest degree of accuracy of which the method is susceptible, and for this reason does not admit of abstraction. Among the general conclusions attained by the author, he finds that the presence of organic matter greatly affects the accuracy of the method, and that, in the case of liquids in which the quantity of organic matter is large in proportion to that of nitrates, the results are even lower than in the case of the method of Crum and Frankland, which itself errs on the side of deficiency. The indigo method cannot, therefore, be employed with safety, in the case of waters distinctly contaminated with organic matter. In the case of ordinary drainage waters, the method agrees very well with that of Crum and Frankland. The author believes, however, that the indigo method, on account of its simplicity, rapidity and delicacy, is excellently adapted for general use in water analysis, but that accuracy can only be attained by working under the same conditions which obtained when the indigo solution was standardized. In the presence of organic matter, the indications obtained with indigo must be accepted as probably below the truth.

III. GENERAL ORGANIC ANALYSIS.

I. *Ultimate Organic Analysis.*

E. A. Grete (Berl. Ber., **11**, 1,558, through Zeitschr. f. anal. Chem., **18**, 486) recommends that *the determination of nitrogen, in such organic substances as scraps of horn, wool, etc.,* should be preceded by the treatment of the substance with concentrated sulphuric acid, with the aid, if necessary, of heat. The comminution thus attained is very perfect, and any excess of sulphuric acid is readily neutralized with soda-lime, so that the mass becomes quite dry. This preliminary treatment increases the percentage of nitrogen yielded by the analysis—a matter of great importance in the case of the albuminoids. Alkaline xanthogenates also increase the yield of nitrogen from albuminoids. The author announces that he will continue his investigation in this direction.

John Rufflé (Chem. News, **40**, 17) communicates the results he has obtained in *the determination of the total nitrogen, including that in nitrates and nitro-compounds, by combustion,* by a modification of the method of combustion with soda-lime, which the author promises to communicate when his experiments are concluded. The results, so far communicated, are eminently satisfactory.

Tschelzoff (Proc. Russian Physico-Chemical Society, 3–15, May, 1879, through Berl. Ber., **12**, 1,486) has a note on *the determination of nitrogen in the explosive nitric ethers.* The author has modified the method of Champion and Pellet (Berl. Ber., **9**, 1,610) to the extent of conducting the decomposition in a stream of carbon dioxide, and titrating the resulting ferric chloride by the method of Fresenius, with stannous chloride and iodine.

B. Delachanal and A. Mermet (Bull. Soc. chim. de Paris, **32**, 50) interpose a claim of priority in the discovery of *the method of C. Fahlberg and M. W. Iles, for the determination of sulphur in organic compounds, etc.* (Berl. Ber., **11**, 1,187), which method consists in fusing the substance with potassium hydrate in a silver crucible, and after dissolving the fusion in water, oxidation with bromine-water and subsequent addition of hydrochloric acid. The claim of priority appears to be based solely on the fact that the authors, D. and M., were the first (Ann. phys. et chim., **12**, 88) to recommend potassium hypobromite as an oxidizing agent in the determination of sulphur in metallic sulphurets, etc.

J. A. Wanklyn and W. C. Cooper (Chem. News, **38**, 133, through Zeitschr. f. anal. Chem., **18**, 486) have experimented on *a method of* *organic analysis by the humid process.* The authors have oxidized such substances as cane sugar, glucose, benzoic, lactic and acetic acids, and alcohol, with potassium permanganate in alkaline solution, and weighed the resulting oxalic acid as calcium oxalate. The experiments are not yet completed, and have not yet developed into a definite analytical method.

FR. LANDOLPH (Berl. Ber., **12**, 1,586) recommends the following *method for the analysis of organic bodies containing fluorine and boron.* In the combustion with cupric oxide, of substances containing these two elements, the greater part of the resulting boracic acid collects in the calcium chloride tube, while some seems even to pass into the potassa solution. It may, however, be completely retained by placing in front of the cupric oxide a layer, a few centimeters in length, of fused and powdered lead chromate. This must only be moderately heated, otherwise boracic acid will volatilize. The determination of fluorine and boron is conducted as follows: the substance is enclosed in a small tube, drawn out at both ends and sealed. One of the ends is broken off, and the tube at once plunged to the bottom of a somewhat wide test tube, containing a solution of calcium chloride. By cautiously heating, the contents of the tube are made to mix with the calcium chloride solution. Decomposition ensues, and calcium fluoride precipitates. The contents of the test tube are then placed in a porcelain dish, diluted with distilled water, neutralized with ammonia, and boiled for some time to ensure complete decomposition. The insoluble calcium fluoride is then filtered off, washed with water, to which a little acetic or nitric acid may be added to dissolve any calcium carbonate that may be present, dried, ignited and weighed. After removal of the lime from the filtrate by ammonium carbonate and oxalate, the boracic acid is determined, by Marignac's method, as basic magnesium borate. Results accurate.

II. *Proximate Organic Analysis.*

A. QUALITATIVE.

ADOLPH BEYER (Berl. Ber., **12**, 1,311) communicates *a characteristic reaction for isatine.* If isatine be mixed with benzole and concentrated sulphuric acid, an intense dark blue coloration is produced, due to the formation of indophenine ($C_{20}H_{15}NO$); $\frac{1}{20}$ mgrm isatine, treated as above, in the cold, gives an intensely dark blue solution. When the mixture is warmed, $\frac{1}{2000}$ mgrm gives a distinct greenish-blue coloration.

L. CLAISEN (idem, **12**, 1,505) remarks that the foregoing reaction, which had already been observed by him, is only one member of a series of extremely beautiful *color reactions for phenylglyoxylic acid* and its derivatives. If concentrated sulphuric acid be added to a solution of phenylglyoxylic acid in benzole, the mixture assumes, after short shaking and standing, first a deep red and then an intense bluish-violet color; on addition of water, the layer of benzole, which separates, shows an intense carmine color. The derivatives of the acid, its amides, ethers, and even benzole cyanide, show a similar behavior. Meta-nitrophenylglyoxylic acid shows a magnificent carmine, ortho-nitrobenzoyle cyanide, a blue-green color.

WATSON SMITH (Chem. News, **40**, 26) describes *certain characteristic color reactions produced by the action of aromatic hydrocarbons and vegeto-alkaloids on fused antimony and bismuth trichlorides.* The general mode of procedure of the author is to fuse, in a small porcelain crucible, 1½ to 2 grms of the chloride, and then to allow a small particle of the substance under investigation to fall on the inner side of the crucible, which is then inclined so as to bring the chloride in contact therewith. With $SbCl_3$, fused at a moderate heat, *diphenyl, triphenyl-methane, phenyl-naphthalene, dinaphthyls*, give no reaction. *Stilbene* gives a fine orange, vanishing on stronger heating. *Naphthalene*, when absolutely pure, gives no reaction; when less pure, a splendid crimson or rose color. *Anthracene*—clear greenish-yellow or yellowish-green; colorless needles form on cooling. *Chrysene*—golden-yellow. *Phenanthrene*—faint greenish tint if quite pure. *Pyrene*—green or greenish tint. With $BiCl_3$, *anthracene* gives a purplish-black. *Phenanthrene*—brown or greenish-brown. With moderately heated $SbCl_3$, the following reactions were obtained with the alkaloids: *coniine, nicotine, morphine, apomorphine, codeine, papaverine, strychnine, quinine, cinchonine, atropine*, give no color; *aconitine*, a bronze-brown. $SbCl_3$, fused and heated somewhat further, gives, with *thebaine*, a red ; with *narceine*, with little more than a trace, a yellow ; *brucine* gives a fine purple-red ; *veratrine*, a brick-red color. If the $SbCl_3$ be fused and heated still further, *thebaine* gives no color; the reactions of *brucine* and *veratrine* are the same as in the last case; *narcotine* gives a dark olive-green ; *santonine*, when more than a trace is present, a dark bluish-olive-green. When $SbCl_3$ is heated to incipient ebullition, *thebaine* gives no tint, or one disappearing instantaneously ; heated more strongly, an olive-greenish-brown ; *veratrine*—a flush of red color, disappearing more slowly than with thebaine ; with more alkaloid, a reddish tint remains, changing to brown if further heated : *brucine*, a trace, gives a fine red stain, disappearing slowly ; with a larger quantity, the color does not disappear, but changes later to purple-brown.

J. TATTERSALL (Chem. News, **40**, 126) communicates the following *new test for papaverine.* The substance to be tested is warmed with a few drops of concentrated sulphuric acid in an evaporating basin, until complete solution is effected. The light pink-violet color, which at first appears, thus changes to light grey, or disappears completely. If now a fragment of sodium arseniate be added, and the dish warmed over a small flame, inclining so as to obtain as much surface as possible, the original color re-appears, and by continued heating changes to cherry-red, and, finally, when vapors of sulphuric acid begin to appear, to dark bluish-violet. If, when the contents of the dish are quite cold, 10 c.c. water are added, and the orange liquid, thus obtained, is placed in a flask, diluted with water, and treated with caustic soda, it darkens rapidly in color, and when an excess has been added, is almost black ; it is violet-red by reflected, and pink-straw color by transmitted light. Strychnine, brucine, morphine, salicine, atropine, narcotine, narceine, digitaline, picrotoxine, curarine, colchicine and cantharidine, become, on addition of alkali, light orange or dirty yellow. Codeine, when heated with concentrated H_2SO_4 and Na_3AsO_4, gives a fine, deep blue color, much darker than that with Fe_2Cl_3 under similar circumstances ; on addition of water and alkali, this becomes orange, and is characteristic of the alkaloid.

F. SELMI (Bull. delle Scienze di Bologna, Ser. **6**, Vol. **I**, through Corr.-blatt d. Vereins anal. Chem., **2**, 79) has investigated the *behavior of strychnine with iodic acid,* concerning which a note is also to be found in Dragendorff's Gerichtlich-chemische Ermittelung von Giften, p. 161. If a drop of strychnine solution be made just perceptibly acid with four per cent. sulphuric acid, and evaporated to dryness on the water-bath, the residue develops, with a drop of a dilute solution of iodic acid, a weak rose color which, in twenty-four hours, passes to a beautiful rose-red. Sodium bicarbonate changes the color to yellow; acids restore the original color. If a drop of a solution of iodic acid in sulphuric acid (prepared by dissolving the former in the latter to saturation, decanting the clear solution and diluting it with 6 volumes of sulphuric acid) be added to a drop of strychnine solution, there is produced a yellow color, passing rapidly to red, and showing the next day a tinge of violet. Excess of the reagent prevents the reaction. If the *violet* liquid be saturated with sodium bicarbonate, and then again acidified, a beautiful rose-red is obtained. The reaction succeeds best when the proper proportion between alkaloid and reagent is attained.

GEORG FRAUDE (Berl. Ber., **12**, 1,558) has a note on *perchloric acid as a reagent for the alkaloids.* If small quantities (0.05 grm) of strychnine, brucine or apidospermine (the alkaloid of the *Apidosperma quebracho* "blanco"; see Berl. Ber., **II**, 2,189, and **12**, 1,560), are boiled for a few minutes with a few c.c. of an aqueous solution of perchloric acid (sp. gr. = 1.13–1.14), the strychnine solution assumes a reddish-yellow color, that of brucine becomes of a dark madeira, and that of apidospermine intensely red. These solutions show strongly marked absorption spectra which resemble each other closely, and are represented, graphically, in the original memoir. Quinine, quinidine, cinchonine, cinchonidine, morphine, codeine, papaverine, veratrine, caffeine, atropine, nicotine and coniine, give no color-reactions with this reagent. If, to the above named quantity of the alkaloids, there be added an excess (30 c.c.) of perchloric acid, and the mixture boiled for several hours, the color becomes lighter, and passes finally to a more or less intense yellow. Similar reactions are produced by boiling the solutions of the alkaloids in dilute sulphuric acid, with small quantities of potassium chlorate, but the oxidation is easily carried too far. 1 c.c. of a solution containing 0.0003 grm apidospermine, boiled with 2 c.c. perchloric acid, shows a distinct red coloration; 0.5 c.c. solution of brucine containing about 0.0006 grm, becomes distinctly reddish-brown with 1.5 c.c. acid; 1 c.c. of hot strychnine solution containing 0.0004 grm, shows, with 2 c.c. acid, a distinct yellow. A solution of iodic acid in sulphuric acid, which Selmi (Berl. Ber., **II**, 1,692, and this JOURNAL, **I**, 545) has recommended as a reagent for strychnine (the alkaloid, when moistened with a small quantity of the reagent, becomes, first yellow, then brick-red, passing very gradually to a brilliant violet), gives, when triturated with brucine, an intense orange-yellow color; with morphine, first deep violet, and then light brown. If a few drops of the reagent be added to one of a solution of curarine, the solution assumes a beautiful rose color.

E. HOFFMANN (Chemiker Zeitung, through Chem. News, **40**, 141) recommends the following *test for phenol*: 1 or 2 c.c. pure concentrated sulphuric acid are placed in a small test glass, and the suspected aqueous liquid poured carefully in, so as to form a separate layer. On adding a few granules of potassium nitrate, violet streaks will be, at once, produced by each particle, if as little as 1 mgrm phenol be present.

B. Quantitative.

ROBERT SACHSSE (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 15, 21, 25) communicates an interesting paper on the subject of the *quantitative determination and formula of starch*. As the result of numerous quantitative estimations, the author has been led to adopt the formula of Naegeli (Beiträge z. Kentnisse d. Stärkegruppe, p. 83), $C_{36}H_{62}O_{31}$, in the place of the older formula, $C_6H_{10}O_5$. This change makes 1080 parts of dextrose equivalent to 990 parts of starch, instead of 180 to 162, or 100 to 90, and involves a difference of 1 to 2 per cent. in the amount of starch found by analysis. The method employed by the author for the estimation of starch, is as follows: 2.5 to 3 grms of the sample, previously completely dried at 100–110° C., are placed in a flask with 200 c.c. water and 20 c.c. hydrochloric acid (sp. gr. = 1.125), the flask connected with a reflux Liebig's condenser, and the contents heated for three hours in a water-bath, in which the water is maintained in violent ebullition. The conversion of the starch is then complete. The liquid, if turbid, is filtered through a weighed filter, the clear filtrate and washings neutralized with potassium hydrate, brought to the volume of 500 c.c., and the dextrose determined in an aliquot part, either by Fehling's method (gravimetrically), or by the method of the author (Zeitschr. f. anal. Chem., **16**, 121, and **17**, 231) with mercuric iodide. The analytical results obtained by the author by the foregoing method, and the use of the formula $C_{36}H_{62}O_{31}$, are very satisfactory. The analysis of a sample of potato starch, for instance, showing the figures:

Starch	99.4
Insoluble residue (cell membranes)	0.2
Ash	0.2
	99.8

Air-dried potato starch loses on the average 17.7 per cent. of water at 100–110° C. From the strong evolution of heat which attends the mixture of the dried starch with water, the author is led to consider the air-dried material to be a definite hydrate, having the formula $C_{36}H_{62}O_{31} + 12H_2O$, which would require exactly 17.9 per cent. of water.

J. STEINER (Chem. News, **40**, 139) communicates the results of his experience with *the ammoniacal copper test* of Pavey (Chem. News, **39**, 77, and this JOURNAL, **1**, 109).

HUGO SCHIFF (Berl. Ber., **12**, 1,531) communicates the following note on *the determination of acetyl, by means of magnesia*, in compounds in which the acetyl group has been introduced into alcoholic hydroxyls. The magnesia is prepared by precipitation from magnesium sulphate or chloride (free from iron) with caustic alkalies, is thoroughly washed, and preserved as a paste, under water. About 5 grms of this paste are placed, with 1.0 to 1.5 grm of the finely pulverized acetyl derivative, in a flask of green glass (not easily attacked by reagents), mixed by shaking, 80 to 100 c.c. water added and, after connecting with a reflux Liebig's cooler, boiled for 4 to 6 hours. The contents of the flask are then evaporated to about one-third, filtered on the pump into a conical flask, and the precipitate washed. The filtrate is treated with ammonium chloride, ammonia and ammonium phosphate, and after twelve hours' standing, the precipitate is filtered off, re-dissolved in hydrochloric acid, re-precipitated with ammonia, and weighed as pyrophosphate. The author ascribes the unfavorable results obtained with this method by Klobukowski (rufigallic acid), and Barth and Goldschmidt (ellagic acid), to neglect of the conditions essential to success. If the action of caustic alkalies on the substance examined is not otherwise disadvantageous, the method of decomposition with normal alkalkies and titration of the excess, is to be preferred to that with magnesia.

A. Loesch (Pharm. Zeitschr. f. Russland, **18**, 545) has devised the following *methods for the quantitative determination of the alkaloids in plants.* The author has, also, subjected to a critical examination, the different methods hitherto in use, with the results communicated below.

A. Methods for the determination of the solid alkaloids.—1. The method of Stas, perfected later by Erdmann and Uslar, Dragendorff and others, and used for chemico-legal investigations, consists in extraction with water acidulated with hydrochloric acid, addition of ammonia to alkaline reaction, repeated extraction with continually renewed portions of ether, chloroform, amylic alcohol or benzole, separation of the watery liquid from the solution of the alkaloid, extraction of the latter by shaking with acidulated water, and repetition of the treatment, until the solution of the alkaloid is colorless. It is then evaporated in a weighed vessel, and the residue dried at 100–110° C. Instead of treating the plant with acidulated water, the author treated it with 99 per cent. alcohol, acidulated with hydrochloric, oxalic, tartaric, phosphoric or sulphuric acids, and evaporation and re-solution in water, before treating with ammonia, etc. By this means, the mucus, starch, gum and albuminoids remained undissolved in the residue from the extraction with alcohol. While this method affords the alkaloids in a very pure state, it is attended with a large loss, and is, in the presence of certain viscid substances and albuminoids, both difficult and tedious.

2. The method of Claus (Vierteljahresschrift f. prakt. Pharm., **13**, 414) for the estimation of theine and quinine. In the first case,

the tea leaves are extracted with ether, the latter removed, for the most part, by distillation, and the residue extracted with water acidulated with sulphuric acid, until it no longer tastes bitter. The acid liquid is mixed with excess of burned magnesia, evaporated to dryness in the water-bath, the residue pulverized and extracted with ether. In the determination of the quinine, the pulverized bark is thoroughly exhausted with water acidulated with sulphuric acid, the extract evaporated to dryness with excess of burned magnesia, and extracted with ether. This method gives satisfactory results and can, without doubt, be employed in the quantitative determination of all other solid alkaloids ; it does not, however, yield the alkaloids in a pure condition.

3. The methods of R. Wagner, Sonnenschein (Zeitschr. f. anal. Chem., 4, 387), Husemann, Marmé and Schultz, are based on the precipitation of the alkaloids by iodine and potassium iodide, phospho-molybdic and phospho-tungstic acids, or potassio-mercuric iodide, from the extract obtained with acidulated water. The precipitates, according to their nature, are decomposed with barium hydrate, hydrogen sulphide, or a mixture of stannous chloride and caustic alkali, and warming, and the alkaloids dissolved out with ether. These methods are not only attended with a large loss, but yield impure products.

4. F. J. Mayer recommends titration with a standard solution of potassio-mercuric iodide. This method gives inexact results, owing to the necessity of decolorizing the solution, and consequent loss of substance, and to the difficulty of determining the end of the titration.

The method of the author is as follows : the cut, or coarsely-powdered, vegetable substances are twice warmed for three hours in the water-bath, with 90 per cent. alcohol acidulated with hydrochloric acid, then pressed and washed with 90 per cent. alcohol. The volume of the collective alcoholic liquids is then reduced, by distillation, to two-thirds, the cold residue filtered, washed with alcohol, and the filtrate concentrated on the water-bath to the consistence of an extract. The residue is warmed with twice the weight of the original substance of water acidulated with sulphuric acid, cooled and filtered. The filtrate is mixed with thrice its volume of cold, saturated solution of alum, ammonia added in slight excess (*i. e.*, more than necessary to precipitate the alumina), evaporated to dryness on the water-bath, the residue pulverized, and exhausted with the solvent suited to the alkaloid to be extracted, *e. g.*, quinine is extracted with ether, and then cinchonidia with 90 per cent. alcohol.

The residue from the leaves and roots of *Atropa belladonna*, from the leaves and seeds of *Hyoscyamus n.*, and the leaves of *Aconitum N.*, with ether; that from *R. Ipecac*, with 90 per cent. alcohol, etc. The ethereal or alcoholic solutions of the alkaloids are evaporated to dryness, dried at 110° C., and weighed. The alkaloids are thus obtained as perfectly colorless crystals, leaving no residue when heated on platinum foil. The results obtained by the author, with the foregoing methods, are given in the following table:

MATERIAL.	ALKALOID.	PERCENTAGE EXTRACTED.				
		METHOD I.	METHOD II.	METHOD III.	METHOD IV.	METHOD OF THE AUTHOR.
Peruvian Bark, *yellow*,	Quinine,	2.735	3.175	2.460	2.570	3.250
	Cinchonidine,	0.194	0.250	0.187	0.175	0.285
" " *red*,	Quinine,	1.105	1.195	1.085	1.005	1.235
	Cinchonidine,	0.425	0.500	0.400	0.395	0.525
" " *brown*,	Quinine,	0.895	0.950	0.825	0.800	0.975
	Cinchonidine,	2.485	2.975	2.350	2.300	3.075
Leaves of Hyoscyamine	Hyoscyamine	0.099	0.145	0.085	0.074	0.175
Seeds of "		0.197	0.225	0.180	0.100	0.285
Leaves of Atropa belladonna	Atropine	0.115	0.197	0.100	0.090	0.225
Roots of " "		0.300	0.325	0.275	0.225	0.375
R. Ipecac,	Emetine,	0.720	0.800	0.550	0.475	0.875
Folia Aconiti,	Aconitine,	0.300	0.395	0.265	0.220	0.425

In the case of substances of unknown nature, the residue of the evaporation with ammonia and alum must be successively treated with the different solvents, *i.e.*, first with ether, then with chloroform, then with amylic alcohol, and finally, with 90 per cent. alcohol. It is by this means not only possible to extract the entire amount of alkaloids, but also to effect a separation of those of different solubilities in cases where more than one are present.

The special precautions for the successful conduct of the foregoing method are: 1. The alcohol must be completely expelled from the alcoholic extract, by evaporation on the water-bath. 2. Both the alcoholic and aqueous liquids must be filtered *cold*. The author finds, as the result of numerous experiments, that the best solvent for the alkaloids, in general, is amylic alcohol, then follow ether, chloroform and benzole, which latter, except in a few cases, dissolves the smallest quantity.

B. Methods for the determination of the liquid alkaloids.—In the methods hitherto in use, the vegetable substances are either distilled with potassium or calcium hydrates, the distillate saturated with hydrochloric acid, evaporated to dryness on the water-bath, and the residue distilled with potassium hydrate; or they are exhausted with acidulated water, the filtered liquid evaporated, and then distilled with potassium or calcium hydrate. As all plants contain not only ammonia, but nitrogenous substances which evolve ammonia when warmed with alkalies, the distillate, after neutralization with hydrochloric acid, is evaporated to dryness, and the residue exhausted with strong alcohol; the ammonium chloride remains undissolved, the alcoholic solution is evaporated to dryness, and the residue exhausted by shaking with solution of potassium hydrate and ether. The ethereal solution is then evaporated, at first at ordinary temperatures, and then over calcium chloride, or hydrate, to remove the moisture. In quantitative estimations, the clean ethereal solution is shaken with a measured volume of standard hydrochloric acid, the ether evaporated off at the lowest possible temperature, and the excess of acid determined by titration. The disadvantages of the foregoing method are: 1. Ammonium chloride is not absolutely insoluble in strong alcohol. 2. A certain proportion of the hydrochloric acid volatilizes with the ether, at temperatures even below 50° C. The error of the method is, therefore, one of excess.

The following method of the author permits an easy and exact determination of the liquid alkaloids:

A weighed quantity of the substance is boiled out with water acidulated with hydrochloric acid, the residue pressed, and washed with water. The collective solutions are evaporated to one-quarter, and the residue distilled with calcium hydrate (under careful cooling of the distillate). The distillation is continued until the last portions of the distillate give no alkaline reaction with sensitive litmus paper. Potassium and sodium hydrates cannot be used, owing to their destructive effects on the alkaloids. The colorless distillate is *exactly* neutralized with sulphuric acid, evaporated to dryness in the water-bath, the residue pulverized and exhausted with 90 per cent. alcohol. The ammonium sulphate remains entirely undissolved, the alkaloid sulphates pass into solution. The solution is evaporated to dryness, the residue shaken out three times with ether and solution of potassium hydrate, the ethereal solution treated with a measured quantity of standard sulphuric acid, the ether distilled off, and the excess of sulphuric acid determined in the residue by titration.

The following determinations of coniine and nicotine were made by the foregoing methods:

	Method with HCl.	Method of the author.
Leaves of *Nicotiana t.* ..	5.750 per cent.	5.250 per cent.
Plant of *Conium m.*	0.075 "	0.060 "

Edwin Johanson (Pharm. Zeitschr. f. Russland, **18**, 97 and 129) communicates the results of his experiments on *the determination of the alkaloids in cinchona bark*, by the method of J. C. B. Moens (Nieuw Tydschrift voor de Pharmacie in Nederland, 1875, 161). The method in question is as follows: The powdered bark is sifted through silk gauze, and the moisture determined in 2 grms at 125° C. For the determination of the alkaloids, 25 to 50 grms of the powder are mixed with 25 grms freshly burned lime, and exhausted with 94 per cent. alcohol (in all 500 c.c.). The extract is neutralized with sulphuric acid, allowed to stand for 24 hours, and then filtered. The alcohol is removed by distillation, the residue is filtered, the precipitate washed with 2 per cent. sulphuric acid, and the alkaloids precipitated from the filtrate with sodium hydrate. After standing for 24 hours, the precipitated alkaloids are filtered off, washed, and the filtrate exhausted by shaking with ether. The alkaloids in the precipitate, and those from the ethereal extract, are dissolved in hydrochloric acid, and the separation of the individual alkaloids effected by treatment with solution of sodio-potassium tartrate, on the principle that cinchonidine tartrate is almost insoluble; quinine tartrate soluble in 1,500 parts, chinidine tartrate in 30 parts, and cinchonine tartrate in about 35 parts of the liquid in question. The solution, therefore, retains but traces of the two first-named alkaloids, the whole of the last two, an amorphous alkaloid and chinamine. Chinidine is readily separable from cinchonine by potassium iodide, as the hydriodate of the latter is readily soluble in water and alcohol. The solution containing the cinchonine and the amorphous alkaloid, is precipitated with sodium hydrate, and the dried and weighed mixture of alkaloids exhausted with 40 per cent. alcohol, which dissolves the amorphous alkaloid, leaving the cinchonine. The cinchonidine is separated from the quinine by taking advantage of the difference in the solubility of the two alkaloids in ether (1 part cinchonidine requires 170 parts of ether). The quinine is freed from the last portions of cinchonidine, by conversion into sulphate and crystallization. The sulphate is dried at 120° and weighed; it contains 86.86 per cent. of pure quinine. The author's experience leads to the conclusion, that in spite of the favorable results obtained by Moens on the Java bark, the method in its present form is not sufficiently exact in other cases, and that further improvements are needed before it can be considered as generally applicable to the analysis of Peruvian barks.

W. Chandler Roberts, C. R. Adler Wright and A. P. Luff (Chem. News, **40**, 94) have reported, as committee of the British

Association for the Advancement of Science, on the *chemistry of some of the lesser-known alkaloids, especially veratria and bebeerine.* The following are among the more interesting results obtained: the materials experimented on were *Veratrum album*, and *V. viride.* The materials were percolated with alcohol acidulated with tartaric acid. The residue of evaporation is diluted with water and filtered to separate resin, rendered alkaline, shaken out with a large volume of ether, the bases removed from the ether with tartaric acid and purified by repetition of the treatment. A certain amount of flocculent alkaloidal matter was in each case left by the ether; this was termed *pseudojervine.* By treatment of the tartrates with soda and about an equal bulk of ether, there was left undissolved, from *V. album*, pseudojervine with a little jervine, and a large quantity of uncrystallizable base, sparingly soluble in ether, to which the name *veratralbine* was given; this base is not present in any considerable proportion in *V. viride* roots. The second ethereal solutions obtained as above, deposited crystals of jervine and a little of a new base, *rubijervine.* The mother liquors from these crystals gave dry, varnish-like residues; that from *V. album* consisted essentially of veratralbine with a minute quantity of an alkaloid yielding veratric acid on saponification with alcoholic potassa, presumably veratrine, such as was extracted by the committee last year from *V. sabidilla* seeds, being powerfully sternutatory (neither jervine, pseudojervine, rubijervine, nor veratralbine, produce sneezing). The product from *V. viride* roots was powerfully sternutatory, and consisted almost wholly of *cevadine* (the second crystallizable alkaloid from *V. sabidilla* seeds). The quantities of alkaloids extracted from 1 kilo of each root was:

	V. album.	V. viride.
Jervine	1.30 grm.	0.20 grm.
Pseudojervine	0.40 "	0.15 "
Rubijervine	0.25 "	0.02 "
Veratralbine	2.20 "	trace
Veratrine	0.05 "	trace
Cevadine	absent (?)	0.43 "

The following are the chief characteristics of the new alkaloids:

Jervine, crystallized=$C_{26}H_{37}NO_3$, $2H_2O$, becomes anhydrous at 100° C.; melting point, 237°–239° (corr.); sulphate almost insoluble; nitrate and hydrochlorate sparingly soluble; with strong sulphuric acid dissolves slowly to a yellow liquid, quickly darkening to greenish-brown, and becoming fine green by absorption of water from the air; not sternutatory nor saponifiable.

Pseudojervine, crystallizes anhydrous, $C_{29}H_{43}NO_7$; resembles jervine; melts at 299° (corr.); sulphate crystallizable, soluble in water, especially when hot; hydrochlorate sparingly soluble hot or cold, if free hydrochloric acid is absent: with sulphuric acid same reactions as jervine: not sternutatory nor saponifiable.

Rubijervine, crystallizes anhydrous, $C_{26}H_{43}NO_2$; resembles jervine; melting point, 236° (corr.): sulphate and hydrochlorate crystallizable; readily soluble in water, especially if warm; with strong sulphuric acid, yellow solution, changing to brownish-yellow, brownish-orange, brownish-blood-red, and ultimately brownish-purple, by absorption of moisture; by cautious dilution with water, the brownish-blood-red solution becomes successively crimson, purple, dark lavender, dark violet, light indigo; neither sternutatory nor saponifiable.

Veratralbine, amorphous; approximately $C_{28}H_{43}NO_5$: no crystallizable salts yet obtained: with sulphuric acid, a yellow liquid, changing to brownish-orange and brownish-blood-red, with strong green fluorescence, resembling cevadine which only differs in giving clearer tints; a crimson-magenta liquid of beautiful and permanent shade being produced by absorption of a trace of moisture: veratrine (of Couerbe) gives precisely the same colors as cevadine, but the dark red solution does not fluoresce before the crimson tint appears through absorption of moisture. Veratralbine is not sternutatory nor saponifiable.

IV. SPECIAL METHODS OF TOXICOLOGICAL AND CHEMICO-LEGAL INVESTIGATION.

F. SELMI (Monit. Scientif. [3 Ser.], **8**, 499, through Zeitschr. f. anal. Chem., **18**, 506) has made an extended series of investigations on the *alkaloids found in the human cadaver*, and their importance in toxicology. The author finds that:

1. From animal substances, in a state of greater or less putrescence, substances may be extracted which possess the character of alkaloids, and show the usual reactions of the vegetable alkaloids; some of them have a reducing action, especially on iodic acid, gold trichloride, etc.

2. Several volatile and non-volatile alkaloids are formed from putrescent animal substances; some are soluble in ether, others in amylic alcohol, and not in ether; others insoluble in both of these liquids.

3. The non-volatile alkaloids give precipitates with nearly all the general alkaloid reagents; some are precipitated by platinic chloride, by potassio-argentic cyanide, and by potassium pyrochromate.

4. These alkaloids may give, with per-iodic acid, crystallized compounds, frequently similar to those afforded by the vegetable alkaloids.

5. They show the following color reactions : with dilute sulphuric acid, red-violet ; with hydrochloric and sulphuric acids, and warming, red-violet ; with sulphuric acid and bromine-water, a more or less distinct red color, which gradually disappears ; warmed with nitric acid, and afterwards treated with potassium hydrate, golden-yellow ; with iodic and sulphuric acids, and sodium acid carbonate, more or less distinct rose-violet.

6. They oxidize readily, and decompose in the air, turning brown; some possess the odor of urine, others an odor similar to coniine, or certain flowers.

7. They possess frequently a piquant taste, and exert a benumbing effect on the tongue, which is more or less persistent ; they often taste bitter.

8. Of those soluble in ether, and those insoluble therein and soluble in amylic alcohol, some do not affect the animal organism ; others are very energetic poisons.

9. The poisonous symptoms are, transitory dilation of the pupil, weak and irregular pulse, convulsive movements, retreat of blood from the heart, with strong contraction of the latter after death.

F. SELMI (Accad. d. Lincei, **3**, through Berl. Ber., **12**, 1,699) recommends the following *modification of Schneider's process for the separation of arsenic in toxicological investigations:* three parts of the substance under examination are heated with four parts of 80 per cent. sulphuric acid, in a retort in the oil-bath, to 130°, and finally to 150° C., and a stream of washed hydrochloric acid conducted through the mass. Only arsenious chloride condenses in the receiver, the antimony and other metals being retained in the retort. In the decomposition of arseniuretted hydrogen, the author uses a tube of only 4 to 5 m.m. width, which is heated to redness for the length of at least 25 c.m. The author has thus obtained an arsenic ring from only $\frac{1}{100}$ mgrm of arsenious acid. By reducing, under precisely equal conditions, the different fractions of a millegramme of arsenious acid, the author has prepared a scale of arsenical mirrors, by means of which an approximate quantitative estimation of minute quantities of arsenic may be made. The original memoir is stated to be rich in the results of practical experience.

J. GUARESCHI (Accad. d. Lincei, **3**, through Berl. Ber., **12**, 1,699) has investigated *the oxidation of thialdine and similar bodies, with nitric acid,* and finds that hydrocyanic acid is formed, as he had previously found to be the case when potassium permanganate was used; the sulphocyanates and mustard oils also furnish hydrocyanic acid by oxidation.

P. C. PLUGGE (Zeitschr. f. anal. Chem., **18**, 408) has investigated the subject of *the decomposition of mercuric cyanide by dilute acids, and the influence of sodium chloride thereon.* It is generally stated in the text books on toxicology, that mercuric cyanide is not readily decomposed by dilute acids, and that hydrocyanic acid cannot be obtained by distilling substances containing mercuric cyanide with acids. The author, however, obtained distinct reactions from the distillate from a mixture of food-pulp and tartaric acid containing mercuric cyanide, which led to an investigation of the circumstances under which the decomposition of the latter substance was effected. By distillation of solutions of 25 grms mercuric cyanide in 125 c.c. of water, with small quantities of sulphuric, tartaric and oxalic acids, until four-fifths of the liquid had passed over, the author obtained from 1 to 5 per cent. of the amount of hydrocyanic acid taken. When hydrochloric acid was used, as much as 78.88 per cent. of the hydrocyanic acid was obtained from the distillate. When a mixture of sodium chloride and oxalic acid was taken, the yield was 46.0 per cent. This latter mixture leaves the residue of the operation in a suitable state for further investigation, which is not the case when, even dilute, hydrochloric acid is used. 23.9 per cent. of the hydrocyanic acid originally present comes over in the first one-fifth of the distillate. The author's experiments show that a solution containing but $\frac{1}{25000}$ of hydrocyanic acid will yield, by distillation with sodium chloride and oxalic acid, a solution in which uncontrovertible traces of hydrocyanic acid may be detected.

WALTHER HEMPEL (Zeitschr. f. anal. Chem., **18**, 399) communicates a very interesting paper on the *limits to the detection of carbonic oxide.* The method of H. W. Vogel (Berl. Ber., **10**, 794, and **11**, 235) possesses over that with sodio-palladous chloride, and that with cuprous chloride, the advantages that it is not influenced by the presence of hydrocarbons, or of oxygen. It consists in introducing into a 100 c.c. flask, filled with the air under examination, 2 to 3 c.c. of blood, previously diluted with water to the extent that it only shows a reddish tinge, but still shows in a test-tube the ordinary spectral absorption bands. If carbonic oxide be present, the blood at once shows a change to a rose color and, on the addition of a few drops of strong ammonium sulphide, the absorption bands do not change, whereas in blood free from carbonic oxide, they are, under these circumstances, replaced by a broad band with indistinct boundaries. Vogel states that the limit of delicacy, by this method, is 0.25 per cent. The author has succeeded in greatly increasing the delicacy of the reaction, by causing the air under examination to be first respired by a mouse, and then testing the blood obtained from the animal. The mouse was enclosed in a cage, made by placing two glass funnels base to base, and covering the line of junction with a rubber band. The air to be tested was caused to pass through the apparatus at the rate of ten liters in one to two hours (the gas issuing from the apparatus contained then from 0.3 to 2.8 per cent. carbon dioxide, rarely, however, above 1 per cent., resulting from the respiration of the animal). The mouse was killed by immersing the funnels in water, and the blood taken from the neighborhood of the heart for examination. The animals were subjected to the action of air containing quantities of carbonic oxide, increasing progressively from 0.022 per cent. to 2.9 per cent. Slight symptoms of poisoning, accompanied with difficulty in breathing, were first noticed, after half an hour's exposure to air containing 0.067 per cent. of carbonic oxide. After three hours exposure, the blood gave a distinct carbonic oxide reaction. The same reaction was obtained from a tube containing fresh blood, diluted according to Vogel's directions, and through which the air was passed before entering the apparatus. In another experiment, with air containing 0.127 per cent. of carbonic oxide, strong symptoms of poisoning ensued in seven minutes; after two hours, a carbonic oxide reaction was obtained from the blood of the animal, as well as from that in the tube. In another experiment, with air containing 2.9 per cent. carbonic oxide, the animal died in convulsions in one to two minutes. The blood showed a strong, carbonic oxide reaction. The author concludes:

1. That by the employment of a volume of air of at least ten liters, 0.05 per cent. of carbonic acid admits of certain detection, both in the dilute blood, and in that of the animal.

2. That the limits of delicacy are, with the mouse, 0.03 per cent., with the diluted blood, 0.05 per cent.

3. That symptoms of poisoning commence to make themselves apparent when the percentage reaches or exceeds 0.05.

LONGUET (Pharm. Centralhalle, **19**, 395, through Zeitschr. f. anal. Chem., **18**, 507) describes the following *method for the examination of spots supposed to have been formed by the spermatic fluid*, whereby the nature of even old spots may be ascertained:

1. There is cut from the fabric a small square, in the middle of which the spot should, if possible, be situated.

2. This piece is placed in distilled water, which contains five to six drops of ammoniacal carmine solution to each five grms of liquid.

3. The piece is allowed to remain immersed for thirty-six to forty-eight hours; longer immersion does no harm.

4. The fabric is carefully pulled apart, thread by thread.

5. The ravelings are moistened with glycerine, and examined, each separately, under a magnifying power of five hundred diameters.

If the spermatic fluid is present, there will be seen around the colorless, strongly refracting vegetable fibres, groups of spermatozoids, of which the heads appear bright red, while the tails are uncolored.

V. SPECIAL METHODS OF TECHNICAL ANALYSIS.

J. FELS (Dingl. polytech. Journ., **224**, 86, through Zeitschr. f. anal. Chem., **18**, 498) has subjected to a comparative investigation the following *methods for the disintegration and solution of chromic iron*, in order to ascertain the advantages, if any, which the more modern possess over the older methods :

1. P. Hart (Journ. f. prakt. Chem., **67**, 320) directs to bring into melted borax one-eighth of its weight of the finely powdered ore ; stir the mixture, and maintain the platinum crucible for one-half hour, at a light, red heat ; sodium carbonate is then added, as long as effervescence is produced, and then three times the weight of the ore, of a mixture of equal parts of nitre and sodium carbonate, stirring well with the platinum wire. After a few minutes melting, the whole of the chromic oxide is transformed into chromic acid. The author obtained satisfactory results with this method, but finds it tedious, and leading easily to mechanical loss.

2. F. Calvert (Dingl. polytechn. Journ., **125**, 466) directs to mix the finely powdered ore with three to four times its weight of soda lime, then to add to the mixture one-quarter of its weight of sodium nitrate, and heat to redness for two hours. The heating must be conducted in a porcelain, not a platinum, crucible, but where chromium is the only element to be determined, the results are satisfactory, especially as the decomposition does not require the blast lamp.

3. The method of J. Blodgett Britton (Zeitschr. f. anal. Chem., **9**, 487) is rapid and sure, and deserves the greatest attention of analytical chemists.

4. The method of F. H. Storer (idem, **9**, 71 and 108) is not to be recommended for technical use, as it yields the chromium in the state of nitrate, in which it cannot be determined volumetrically.

5. The method of A. Mitscherlich and F. C. Phillips (idem, **12**, 189) is too complicated.

6. H. Hager (Untersuchungen, **1**, 163) places a mixture of one part of mineral with three parts sodium fluoride in a graphite crucible, covers the mixture with twelve parts potassium bisulphate, and heats; after five to six minutes, during which the mass boils up, the decomposition is complete. Fels finds these statements completely confirmed; he recommends that the mixture should be cautiously warmed in a platinum crucible, and the heat raised as the foaming moderates ; the mass flows quietly in five or six minutes and, after five minutes more, becomes thick, which the author considers the sign of the termination of the reaction. The green mass contains chromium fluoride which the author prefers to convert to chromic acid by cooling, gradually adding potassium chlorate, and heating anew for a few minutes.

7. The method of W. Dittmar (idem, **18**, 126, and this JOURNAL, **1**, 104) requires the blast lamp (twenty minutes suffice for 0.5 grm ore) for the attainment of perfect decomposition. The author specially recommends it, together with that of Britton.

8. The method of R. Kayser (idem, **15**, 187) permits the attainment of complete decomposition.

9. The same is also true of the method of J. Clouet (idem, **17**, 249), but it is tedious and disagreeable.

On the whole, the author gives the preference to the methods of Calvert, Britton and Dittmar, for technical use.

JOH. STINGL (Oesterr. Gewerkszeitung, 1878, **1** and **2**, and Zeitschr. f. d. gesammte Thonwaarenindustrie, **3**, 86, through Zeitschr. f. anal. Chem., **18**, 501), in an extended work on lime and its use in mortar, gives the following *method for the technical valuation of quick-lime:* the numerical expression for the value of the lime is derived from the following factors :

1. The weight of water which a weighed quantity of quick-lime (the density of the latter being assumed to be 2.3) will take up, while still furnishing a paste of such consistence that a glass rod will stand upright therein.

2. The density of the paste.

From these data the increase in volume of the lime by hydration, and hence its capacity for taking up water, may be easily derived. The author proceeds as follows :

50 grms lime are gradually mixed with water in a 750 c.c. beaker, until a paste of the above character is attained. By subtraction of the weight of the beaker and the lime from the weight of the beaker

and the paste, the weight of the water added is ascertained. The density is determined as follows: a wooden cross with a wire projecting from its centre is placed, with the wire pointing perpendicularly downwards, on top of a 750 c.c. beaker, and the weight of the whole (a) noted. The beaker is then filled with water until the surface of the latter just touches the point of the wire, and the weight (b) again noted. The beaker is then emptied, wiped carefully dry, and the paste of lime and water poured in cautiously with the help of a glass rod, until the (horizontal) surface touches the wire, and the weight (c) noted. Then the weight of the water will be $b - a = w$, that of the paste, $c - a = k$, and the density of the latter, $\frac{k}{w} = d$. If we let P represent the weight of the water taken to form the paste with the fifty grms of lime, and V the volume of the paste yielded by the latter, we have:

$$V = \frac{P}{d}$$

A. Rollet (Dingl. polytechn. Journ., **233**, 124) describes the *method for the determination of sulphur in the products of the iron industry, ores and fuel*, which has, for the past two years, been in use, with highly satisfactory results, at the works at Cruzot (France). The methods hitherto in general use for the determination of sulphur in iron, etc., are based on the solution of the metal in a non-oxidizing acid, and the transformation of the hydrogen sulphide in the evolved gases into other forms, in which the sulphur can be weighed or otherwise estimated. Whatever may be the reliability of the method with metals free from carbon, it is, in the case of substances rich in the latter, subject to serious errors due to the formation of sulphuretted carbon compounds which do not afford sulphides with metallic salts. Thus it commonly happens, that in cast iron a smaller percentage of sulphur is found than in the steel made therefrom, and, in general, the richer the substance analyzed, in carbon, the smaller will be the proportion of the true percentage of sulphur found by the foregoing methods. The method described by the author is based on the conversion of the sulphur to hydrogen sulphide, by heating the substance to redness in a current of three-quarter hydrogen and one-quarter carbonic acid, passing the resulting gas through a dilute solution of silver nitrate, and weighing the silver sulphide thus produced. While the desulphurization of rolled iron proceeds with rapidity and completeness, in a current of pure hydrogen, the action in the case of irons richer in carbon is slower in proportion to the amount of the latter, and is only complete after the carbon has been carried off by the hydrogen. The sulphur in ores, slags, limestones, etc., is not completely transformed. The addition of carbonic acid causes a complete desulphurization in a very short time, without danger of decomposition or oxidation of the hydrogen sulphide formed. The carbon is either completely burned off, as in the case of coal, coke, etc., or, almost so, as in the case of iron and steel. In the case of very rich sulphurets, but a small portion of substance should be taken. The hydrogen used is generated from pure zinc and dilute sulphuric acid ; the carbonic acid, from marble and very dilute hydrochloric acid. The carbonic acid is washed with lead acetate, and then enters a washing-bottle containing silver nitrate, where it mixes with the hydrogen ; the mixture being carried through a wash-bottle containing pure water, and then through the porcelain tube containing the boat with the substance under examination. The gaseous products of the reaction are then conducted into a small flask, where water deposits, and then into the silver nitrate solution (sixteen to eighteen grms to one liter water, neutralized with ammonia and re-acidified with one or two drops of nitric acid). In the analysis of *cast iron*, *iron* and *steel*, two to at most four grms, previously pulverized to pass a sieve of nine hundred meshes to the square centimeter, are placed in a platinum or porcelain boat, 10 c.m. long and 15 m.m. wide, and the latter introduced into the porcelain tube. A tolerably rapid current of the mixed gases, in the proportions stated, is conducted through the apparatus and, after the air has been expelled, the tube is gradually heated to redness. At the end of two to two-and-a-half hours, almost all the sulphur is expelled as H_2S, and it is only necessary to heat half an hour longer to insure the expulsion of the last traces. The precipitated silver sulphide is collected on a double, weighed filter, dried at 100°, and the weight estimated from the difference in weight of the two filters ; or the two may be separately burned, and the difference in weight of the two residues of metallic silver and ash, multiplied by 0.148, to obtain the corresponding weight of sulphur. If the temperature cannot be regulated so as to prevent the sintering of the substance under examination, this must be prevented by the addition of pure alumina (one grm to two grms of metal). In the case of *ores* and *slags*, a mixture of two grms alumina to four grms substance is needed to prevent sintering in the case of the more fusible substances ; blast-furnace slags and calcareous and siliceous fluxes require no such additions. In the estimation of sulphur in *coke*, 0.5 grm substance, mixed with 0.2 grm alumina, is sufficient. In the case of *coal*, 0.5 grm is sufficient, but it is best to fill the tube behind the boat with porcelain fragments, to retain the tarry ingredients, and at the end of the operation, to heat this part strongly, to complete the decomposition of the latter. In the case of *gases*, they are mixed either with carbonic acid or hydrogen, according to their reducing or oxidizing character. In the case of certain ores and combustibles, the silver sulphide may be contaminated with silver chloride or cyanide; it may then be purified by washing with weak ammonia, or may be reduced in hydrogen, and the gas conducted into ammoniacal silver solution.

A. H. Allen (Chem. News, **40**, 135) effects *the estimation of nitrogen in steel*, by dissolving the sample in hydrochloric acid, distilling the solution with excess of lime, and nesslerizing the distillate. The results of repeated analyses of the same sample gave substantially accordant results.

A. H. Allen (Chem. News, **40**, 65), in a note on *the determination of silicon in iron and steel*, calls attention to a defect in the method of Thos. M. Drown (Journ. Franklin Inst., **107**, 361 ; Am. Chem. Journ., **I**, 417, and this Journal, **I**, 371) for this purpose. According to the author, the light flocculent residue left on dissolving siliceous iron in hydrochloric or sulphuric acids, consists chiefly of a lower oxide of silicon ("Lenkon" of Woehler), the formula of which has been variously stated at $2SiOH_2O$ and $SiOH_2O$, and which, on ignition, gives off hydrogen silicide, which latter is decomposed in its turn, depositing more or less silicon. He states that five years of additional experience have resulted in strengthening his confidence in the method previously published by him (Chem. News, **29**, 91).

C. M. Balling (Oesterr. Zeitschr. f. Berg- u. Hüttenwesen, **27**, 27) communicates the results of his experience in the *direct determination of silver in galena, by the method of J. Volhard* ("Silbertitrirmethode mit Schwefelcyanammonium," Leipzig, 1878 ; Journ. f. prakt. Chem. [N. F.], **9**, 217 ; Zeitschr. f. anal. Chem., **13**, 171). The author recommends to intimately mix in a porcelain mortar, two to five grms (according to richness) of galena, with three to four parts of a mixture of equal parts of sodium carbonate and potassium nitrate. The mixture is heated to melting in a porcelain crucible over the lamp, and stirred well with a glass rod, cooled, and crucible and contents placed in water in an evaporating dish. When the mass has softened, the crucible is removed and washed, the contents of the dish heated, the aqueous solution filtered off, and the mass on the filter well washed. It is then washed back into the dish, pure, dilute, nitric acid added, and the mixture evaporated to dryness. The dry residue is dissolved in water acidulated with nitric acid, warmed over a lamp, the solution filtered into a flask, and the residue on the filter thoroughly washed with hot water. To the filtrate, when cold, ferric sulphate, or ferric ammonium alum, is added, as an indicator, and the titration conducted, as directed by Volhard. The sulphocyanide is standardized with pure silver, so that 1 c.c. corresponds to 0.001 grm silver. Small quantities of copper do not affect the test. The presence of iron, in considerable quantity, is injurious, as it makes the solution so dark, that the end of the titration becomes difficult of recognition. Portions of five grms each of pure galena gave 0.574, 0.572, 0.574, 0.574; portions of two grms, each of the same, gave 0.575 and 0.575 per cent. of silver; the fire assay gave 0.572 and 0.578 per cent. Portions of two grms each of a similar galena yielded, by the foregoing method, 0.565 and 0.560 per cent.; the fire assay, 0.560 and 0.564 per cent. Two grms portions of a third sample yielded, 0.445 and 0.450; by fire assay, 0.452 and 0.459. The allowance made at Przibram for errors in the fire assay of ores containing 0.30 to 0.60 per cent., is 0.03 per cent. It is evident, therefore, that the errors incident to the method just described, are far within the limits of those pertaining to the dry method. The method is equally exact for ores of every degree of richness, and may be carried out in three hours. Volhard has indicated the modifications necessary in the presence of larger quantities of copper. The author has found no means of counteracting the evils of the presence of larger quantities of iron.

Otto Schott (Zeitschr. f. anal. Chem., **18**, 443) recommends the following method for the *determination of iodine in varec (kelp):* The alkaline iodides therein may be brought into solution either by extraction of the collective soluble constituents with water, or by extraction with alcohol, which dissolves the iodides, leaving most of the other salts undissolved. On account of the cost of alcohol, the author adopts the method first named. 25 to 50 grms of substance are exhausted with water, and the solution brought to the bulk of 1 liter, 100 c.c. of the solution are acidified with a few drops of sulphuric acid, and allowed to stand, warm, for twenty-four hours, to remove the hydrogen sulphide, evaporated to one-half or one-third, passed through a small filter, and the latter washed with the least possible quantity of water. The filtrate is placed in a small, plain retort, a solution of ferric sulphate or ferric-ammonium alum added, and the retort placed with the neck inclined upward at an angle of about 45°; the neck is connected by means of a short rubber tube, or collar, with a wide glass tube which is bent downward and inserted through a cork into a U-tube, the bend of which is filled with a solution of potassium iodide; the delivery tube dips a few millemeters beneath the surface of the solution. The U-tube stands in a beaker of cold water. The liquid in the retort is heated to boiling and, after ten to fifteen minutes, the whole of the iodine will usually have passed over into the potassium iodide solution, wherein it is determined by titration with sodium hyposulphite, using starch solution as an indicator. It must, however, never be omitted to test the solution in the retort for iodine, by shaking with a little carbon disulphide or chloroform, after the distillation is finished, for a prolonged distillation is often necessary to insure the complete expulsion of that substance. A sample of varec from Cudillero (North coast of Spain), which yielded 0.376 per cent. iodine by the estimation with palladous chloride, yielded by the foregoing method, 0.328, 0.338, 0.305 and 0.338 per cent. Two specimens of varec which were carefully prepared in the author's presence at Obiñana, yielded: 1st, 1.701 and 1.676; 2nd, 1.426 and 1.426 per cent.

H. Precht (Zeitschr. f. anal. Chem., **18**, 438) has devised the following *method for the volumetric determination of magnesium:* 10 grms of the substance under examination are dissolved in a 500 c.c. flask, semi-normal potassa solution is added in quantity more than sufficient to precipitate all the magnesium as hydrate. The flask is then filled to the mark, well shaken, and allowed to stand for thirty minutes; by this time the precipitate will have in so far subsided, that 50 c.c. of clear liquid can be removed with the pipette, and the excess of potassa therein titrated with normal sulphuric acid. An experiment, conducted in the foregoing manner, yielded 48.54, instead of 48.78, per cent. $MgSO_4$. A specimen of kainite, K_2SO_4, $MgSO_4$, $MgCl_2 + 6H_2O$, gave, by the foregoing method, 16.32 MgO, and by the pyrophosphate method, 16.50. According to Fresenius, one part MgO dissolves in 53,368 parts of water; according to the author, in 62,000; hence, the 50 c.c. of the solution used, contained 0.8 mgrm = 0.08 per cent. MgO, or 0.24 per cent. $MgSO_4$, which should be added to the results obtained by titration. The error induced by the presence of the precipitate, and consequent concentration of the solution, is also negative, and amounts, in the case of fifty per cent. $MgSO_4$ (in the substance), to 0.08 per cent. of $MgSO_4$. With these corrections, the results are exact.

E. Mylius (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 11) recommends a *volumetric method for the determination of zinc in potable water,* based on the comparison of the turbidity produced by a solution of potassium ferro-cyanide, in the sample, with that produced by the gradual addition of a standard solution of zinc sulphate to pure spring water, to which the same volume of ferro-cyanide solution had been previously added. The method is designed simply to save the trouble involved in the evaporation of the several liters of the sample, required by the methods previously in use. As a matter of general interest, it may be mentioned that the author found 0.49 grains of zinc oxide to the gallon, in the water of a spring at Tuttendorf, which had been used by the population for drinking, for about a century. This is in manifest contradiction to the popular prejudice as to the injurious effects of small quantities of zinc salts in food or drink.

F. P. PERKINS (Analyst, **4**, 66) recommends the following *mode of conducting the ammonia test in potable waters:* Into a half liter retort, place 25 c.c. of a solution of potassium permanganate, prepared by the usual formula; add 100 c.c. of pure water, and distil until no reaction is obtained with Nessler's test in the distillate; add then 250 c.c. of the water to be examined, and distil again until free from ammonia. The distillate contains albuminoid and free ammonia. The free ammonia alone is determined in another 250 c.c. of the water. All traces of ammonia are thus expelled from the reagents, and results attained, as near perfection as the process will allow.

FAUSTO SESTINI (Le Stazione Sperimentale Agrarie Italiane, 1877, and Landwirthschaftl. Versuchsstationen, **23**, 305, through Zeitschr. f. anal. Chem., **18**, 503) has investigated the subject of *the determination of the albuminoids in food for cattle.* It is well known that the old method of calculating these substances, by multiplying by 6.25 the percentage of nitrogen found by analysis, is very inexact, and that the different parts of vegetables, etc., contain nitrogenous substances which have nothing in common with the albuminoids, as regards their nutritious properties. Schulz and Ulrich (Landw. Versuchsstationen, **20**, 193) have, for instance, shown, that in the beet but one-fifth to at most two-fifths of the nitrogen is present as albuminoids. Church (Agricultural Student's Gazette, **1**, 3), therefore, has proposed to precipitate the albuminoids with phenol, wash the mass well, and determine the albuminoid nitrogen in the residue. The author employs the following method which, from comparative experiments, he considers to be superior to the method of Church, and to that of Richard Wagner (Zeitschr. f. anal. Chem., **17**, 515):

The finely cut materials are boiled for one hour with water, whereby the *coagulable* albuminoids are coagulated by the heat at

100° C. After the first half hour's boiling, the mass is acidified to strong reaction with litmus paper, with concentrated lactic acid. The mass, while still hot, is treated with neutral lead acetate, which throws down the non-coagulable albuminoids. It is then filtered, the residue washed, and subjected to a nitrogen determination. The nitrogenous substances in the filtrate are also determined, and, finally, the total nitrogen in the original substance, as a control.

J. Carter Bell (Analyst, 4, 126) has an interesting paper on the subject of *the analysis of flour and bread*, wherein he communicates the results of numerous analyses made during the past two years. The following matters are of special interest to analytical chemists: The author finds the logwood test for alum to answer admirably, if conducted as follows: 16 grms of freshly cut logwood are digested in cold "methylated spirit" for 8 hours, with occasional shaking; the clear solution is then poured off for use. For *bread*, 10 grms of the crumbs are placed in a porcelain dish, and a mixture of 30 c.c. water, 5 c.c. logwood solution and 5 c.c. saturated solution of ammonium carbonate, poured over them. The bread is then washed and *dried*. If a blue lavender color remains, the author always finds alum to be present. For *flour*, 10 grms are mixed with 10 c.c. water, 1 c.c. logwood and 1 c.c. ammonium carbonate; if the flour is pure, a pinkish color is obtained; if alum is present, the pink is changed to lavender. The author has thus detected as little as three grains alum to the four pound loaf. The logwood solution will keep for several months. The author is accustomed in his bread analyses, to deduct ten grains of alum from the total quantity calculated from the phosphate of alumina found in a four pound loaf, before using the quantity of alumina as an indication of addition of alum. Of one hundred samples of pure bread analyzed by him, but one gave as much as eleven grains of alum to the four pound loaf.

W. C. Young (Analyst, 4, 6), in a note on *the detection of alum in flour by the logwood test*, finds that the delicacy and certainty of the test may be greatly increased, by first dissolving the flour to a thin paste in boiling water, previous to adding the ammoniacal logwood tincture; a bluish-grey color is then developed, which is unmistakable, even when the proportion of alum does not exceed five grains to four pounds of flour.

W. W. Stoddart (Analyst, 4, 6) employs *the logwood test for alum in bread*, in the following modification suggested some time since by Mr. Horsley: With or without alum, bread is colored by decoction of logwood of a strong and distinct purple color; if, however, it be exposed in a porcelain capsule to the atmosphere for a few hours, the beautiful color disappears if alum be absent, changing to a dull brown; if alum is present, a conspicuous and beautiful blue is the permanent result. The logwood used must be freshly cut, and kept in a stoppered bottle, secure from the atmosphere. The decoction or tincture must also be freshly prepared. A few crystals of fresh hæmatoxylin give the same result.

J. KOETTSTORFER (Zeitschr. f. anal. Chem., 18, 431) gives the continuation of his investigation on his *new method of testing butter for foreign fatty bodies* (idem, 18, 199, and this JOURNAL, I, 359). The author has examined a number of samples of commercial butter and butter-fat, with results confirmatory of those obtained in his previous investigation. He finds, however, that the extreme limit for the alkalimetric titre of pure butter, must be raised from 232.4 to 233. The titre of rancid butter is lower than that in its fresh state, evidently owing to the volatilization of a certain proportion of the fatty acids thus set free, the difference being, in two cases, 1.5 and 1.4 mgrm. The author recommends the following method for the estimation of the percentage of free acid in butter: 3 to 10 grms of the melted and filtered sample are dissolved in ether in a 50 c.c. flask (the free acid which commercial ether usually contains is first neutralized by adding a few drops of an alcoholic solution of phenol phtalein solution, and then, drop by drop, an alcoholic potassa solution, until a permanent violet coloration is produced). The ethereal solution of the fat is then titrated with alcoholic potassa. The titration of thirty-three samples showed the percentage of free acid to range from 1.0° to 41.6°, the number last named being obtained from a rancid fat, liquid at 10° C., which had evidently resulted from the separation, by crystallization, of the solid, fatty acids from melted butter. The acidity of a good butter-fat (butter, melted, and then solidified by cooling) should not exceed 8°.

A. H. ALLEN (Analyst, 4, 162) communicates the following suggestion concerning *the expression of the results obtained by Koettstorfer's method of butter analysis:* The author proposes that instead of expressing the results of the titration in millegrammes of KHO used per gramme of fat, they should be expressed by figures representing the combining equivalents of the fats present. Thus: 56.1 parts of KHO will, theoretically, saponify 296.7 parts tri-stearin; 292.0 parts tri-olein; 296.0 parts tri-palmitin; or, 100.67 parts tri-butyrin. Koettstorfer's results can be translated into equivalents, by dividing the millegrammes of KHO into the number 56.1. They thus become as follows:

Beef drippings	285.5
Commercial tallow	285.1
Lard, from kidneys	286.5
Lard, from unsmoked bacon	286.7
Commercial lard	287.1
Mutton drippings	284.8
Average	286.0

Olive oil, 292.5; colza oil, 313.9; butter, 253.3 to 241.4; the average being 247.1. Hence, a rise of 0.39 in the equivalence of the sample represents one per cent of *probable* adulteration, although a butter should not be condemned unless its equivalent exceeds 255.

W. G. CROOK (Analyst, 4, 111) has devised *a new method of distinguishing butter from some other fats.* Ten grains of the sample to be tested, previously freed from water, salt, etc., by melting and filtration, are placed in a graduated test tube, and liquified by placing the tube in water at 150° C.; add then thirty minims of carbolic acid (one pound Calvert's No. 2, and two fluid ounces water), shake the mixture, place in the water-bath until transparent, and set aside for some time. Pure butter will give a clear solution; with beef, mutton or pork-fat, two layers of solution are formed. In the case of beef-fat the lower layer is about 49.7, lard, 49.6, mutton, 44.0 of the entire volume. With olive oil the substratum is fifty per cent. Castor oil dissolves completely. With some solid fats, not likely to be used fraudulently, no separation takes place. A minute addition of alkinet root will render the reading of the scale extremely distinct by artificial light. The method is a convenient "first step" in butter analysis.

G. W. WIGNER (Analyst, 4, 192) has carefully tested *Koettstorfer's method for testing butter for foreign fats* (Zeitschr. f. anal. Chem., 18, 199, and this JOURNAL, 1, 359), and finds it to give excellent results. He recommends that after saponification, the soap should be dissolved in hot water and titrated hot; the point of neutralization is thus more easily observed.

F. P. PERKINS (Analyst, 4, 142) recommends the following method for *the analysis of butter-fat:* One to two grms of butter-fat are saponified in a beaker in the usual way, the alcohol expelled by heating, and the fatty acids liberated by adding a slight excess of a cold, saturated solution of oxalic acid. The liquid is passed through a small, wet filter, the insoluble fatty acids washed thorougly, first by decantation with cold water, then on the filter with hot water. The filtrate is brought, with water, to 200 c.c., 100 c.c. distilled to dryness, in a small retort, and the distillate, after the addition of a few drops of litmus, titrated with one-tenth normal KHO. The result is calculated as butyric acid on 100 grms fat. The insoluble acids are dissolved in hot alcohol, and the filter washed with the same until the acid reaction of the washings disappears. The filtrate is made up with alcohol to 100 c.c., and 50 c.c. titrated with deci-normal alkali and acid, as a preliminary test. The remaining 50 c.c. are then carefully titrated to the same color, and the result calculated as stearic acid in 100 grms fat. The results obtained on two samples of purified fat from good farm butter, were :

	I.	II.
Butyric acid	6.7	6.8
Stearic "	92.0	91.7

The results calculated in percentages of potassium hydrate agree closely with those of Koettstorfer :

	Percentage of KHO. I.	II.
Volatile fatty acids	4.31	4.42
Non-volatile fatty acids	18.14	18.00
	22.45	22.42

E. Meissl (Dingl. polytechn. Journ., **233**, 229) communicates the results of his *investigations on the adulteration of commercial butter-fat*, by Reichert's modification of Hehner's method (Zeitschr. f. anal. Chem., **18**, 68, and this Journal, **1**, 86). The author uses the following mode of conducting the examination: five grms of melted and filtered fat are saponified with two grms solid potassium hydrate and 50 c.c. seventy-five per cent. alcohol, in a 200 c.c. flask on the water-bath, and the *clear* soap evaporated until the alcohol is *completely* expelled, dissolved in 100 c.c. water and decomposed with 40 c.c. dilute (1:10) sulphuric acid, a few pieces of pumice stone, of the size of hemp seed, being added, to prevent concussive ebullition, and distilled until 110 c.c. have passed over. The distillate is filtered and 100 c.c. titrated with litmus and deci-normal potassium hydrate, until the blue color does not change on shaking. The percentage of pure butter-fat in a given sample is obtained by multiplying by 3.875 the number of c.c. less 3, of deci-normal alkali consumed in the titration. As the result of some eighty-four determinations, the author concludes that a butter-fat, the distillate of which requires as much as, or more than, 27 c.c. deci-normal alkali, must unconditionally be declared to be genuine;

one of 26 to 27 is suspicious; one below 26 c.c. may be accepted as certainly adulterated. He recommends, however, that, for the sake of safety, adulteration not exceeding ten per cent. should be disregarded. He considers the foregoing method to be one of the most reliable at our disposal.

W. H. WATSON (Chem. News, **40**, 102), in a note on *the detection of milk adulteration*, states that from analyses of milk obtained from different dairies, and comparison of the results with the conditions existing as to character and quantity of food, idiosyncracies of the cows, and their state of health at different periods, he is led to conclude that the present limits adopted by public analysts for genuine milk, should be reconsidered. He has found in milk from healthy, well-fed cows as little as 10.5 per cent. of total solids, and 8.5 to 9.0 per cent. of solids other than fat.

FESER (Pharm. Centralballe, **19**, 21, through Zeitschr. f. anal. Chem., **18**, 492) describes his patented lactoscope for *the optical estimation of the fat in milk.* The instrument is a cylindrical vessel of something over 100 c.c capacity. The lower extremity is narrowed and contains a scale on a milk-glass rod. 4 c.c. of the milk are introduced and water added, with continual shaking, until the milk becomes so transparent that the divisions on the milk-glass rod can be counted. A scale on the outside of the cylinder permits the number of c.c. of water added, and the corresponding percentages of fat, to be read off directly. The instrument is made by Johannes Greiner, Neuhauserstrasse 49, Munich.

MAX BIECHELE (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 79) calls attention to *the importance of the percentage of extractive substances soluble in alcohol, as a criterion of the purity of spices.* The author employs the following method: A flask of about 120 c.c. contents, is fitted with a cork, through one of the two holes in which passes the stem of a funnel of about 7 c.m. diameter; through the other, a tube leading to the top of an upright Liebig's condenser. Five grms of the spice to be tested, previously dried at 30° C., are placed on a filter which should not quite fill the funnel, covered with a disk of filter paper, and absolute alcohol poured through until the flask is half full. The funnel is then covered with an inverted funnel, the stem of which has been broken off, leaving an aperture, through which is passed the lower end of the condenser. The alcohol in the flask is then heated to boiling, and maintained at that temperature, until the prolonged percolation of the condensed alcohol through the spice has removed all soluble matters, and the filtrate is colorless.

The filter, with its contents, is then partially dried at 100° C., the contents removed to a weighed porcelain dish, thoroughly dried at 100° C., and again weighed. The volatile character of many of the extractive substances (essential oils, etc.) renders it impossible to estimate the residue from the evaporation of the alcohol. Proceeding by the foregoing method, the author has obtained the following percentages of extract from pure spices :

Cloves	33.50	Red pepper	18.13
Cassia bark	26.60	Coriander seed	14.88
Cinnamon (Ceylon)	23.90	Star-anise "	25.68
Caraway seed	33.87	Anise seed	36.24
Fennel "	38.20	Clove pepper	22.68
Black pepper	19.87	Mace	37.60
Long "	37.00	Nutmegs	32.70
White "	16.87		

L. Medicus and E. Schwab (Berl. Ber., **12**, 1,285) have investigated *the quantitative determination of starch in sausages.* The qualitative test is easily made by moistening a slice of the suspected material with solution of iodine, and observing, with the aid of a lense, whether a blue coloration ensues over a larger or smaller portion of the surface. The slight coloration from the starch of the pepper is thus easily distinguished from that resulting from direct additions. The starch cannot be extracted with boiling water, without bringing into solution other substances which, by the subsequent inversion with acids, tend to form amido-acids, and thus give solutions in which no end-reaction with Fehling's solution can be obtained. The authors recommend that the starch in the sausage-meat be brought into the state of paste, and that to 20 grms of substance there be added 15 c.c. of a solution of diastase, obtained by digesting 5 grms malt with 5 c.c. water, for one-and-a-half hours, at 30 to 40° C. The mixture is diluted to 100 c.c., and warmed for two hours to 40 to 50° C.; it is then allowed to stand eighteen hours at ordinary temperatures. The filtrate and washings of the mass are heated to boiling, for a short time, to coagulate the albumen, and then re-filtered. The inversion of the maltose and dextrine is effected by warming with hydrochloric acid, and the solution titrated with Fehling's solution. 15 c.c. of the malt extract are then treated in the same manner, and the necessary deduction made for the glucose contained in the 5 c.c. originally added. The percentage of starch is obtained from the glucose resulting from it, by multiplying the latter by 0.9. In a mixture of starch and sausage-meat, the authors obtained, by the foregoing method, 84.6, instead of 100.0 of starch. If it be desired to make a correction for the starch contained in the pepper, in the sausage-meat, the authors recommend a deduction of 1.0 per cent. of the weight of the latter, as being, certainly, in excess of the true amount.

P. Waage (Zeitschr. f. anal. Chem., **18**, 417) communicates a series of *studies on the ebullioscope.* The instrument has been subjected to thorough investigations by Dumas, Dessains and Thénard (Comptes Rendus, **80**, 114) and by Griessmayer (Dingl. polytechn. Journ., **218**, 262), who have reached the conclusion that it is the most exact of all the hitherto known methods of alcohol estimation. The author's investigations, made on mixtures of alcohol and water of respectively 3.80, 9.17 and 16.34 per cent., showed the maximum errors in a series of eight experiments to be respectively, — 0.10, — 0.07 and — 0.14, the mean errors being + 0.006, — 0.037 and — 0.085, and would thus seem to justify the conclusions of the investigators first named. With beer, however, the results obtained were uniformly too high, the error in a mixture containing 9.1 per cent. of alcohol and 10.8 per cent. of extract, being as much as + 1.05 per cent. The mixture was made from alcohol, and beer extract free from alcohol. The error increases with the percentage of the alcohol, and also with that of the extract, although the increase is greatest in the first case. As the ratio between the percentage of alcohol and that of extract varies within narrow limits only, the author suggests that the error may be compensated for with reference solely to the percentage of alcohol contained in the beer. From a series of forty-five experiments made on different varieties of beer from Swedish breweries, the author finds that with Bavarian beer, containing over six volume per cent. there must be deducted from the percentage of alcohol shown by the ebullioscope, 0.216 per cent.; the probable error will then be 0.035 per cent. Bavarian beer, showing in the ebullioscope from 5 to 6 volume per cent., requires a deduction of 0.159 per cent.; probable error, 0.028 per cent. The constant error for (obergähriges) beer of 4 to 5 volume per cent., is 0.11 per cent., and for beer of 2 to 4 volume per cent., 0.02. With these corrections, the results of forty-two ebullioscopic estimations did not, in any case, differ as much as 0.1 per cent. from the true percentage. In conclusion, the author suggests several improvements in the construction of the instrument.

G. Dahm (Ann. d. Oenologie, **8**, 4, 1 and 2, through Corr.-blatt d. Vereins analytischer Chemiker, **2**, 67), calls attention to the fact, that *the conversion of the percentage by volume of alcohol in wine, into* *the percentage by weight*, cannot be effected by simple reference to the tables in the text-books. The specific gravity of the wine itself is a factor which may influence the result to the extent of over one per cent. The author employs the following rule: "Multiply the percentage by weight stated in the tables to correspond to the specific gravity of the distillate, by the specific gravity of the distillate, and divide the product by the specific gravity of the original wine."

G. Holzner (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 79) has been led by the foregoing article, to describe *the method of determining the percentage of alcohol in fermented liquors*, devised by Dr. Leyser, in the laboratory of the late Prof. Reischauer. 76 grms of the fermented liquor (which correspond to about 75 c.c.) are distilled, on the oil-bath, and the distillate collected in a 50 c.c. specific gravity bottle, with narrow neck. If D be the weight of the 50 c.c. of distillate (*e. g.*, D = 48.905 grms), then 0.02 × D is the specific gravity (s) of the distillate (*e. g.*, 0.02 × 48.905 = 0.9781). The percentage of alcohol corresponding thereto (S), at 17.5° C., is S = 14.56. The quantity of alcohol in the distillate (A_1), is $\frac{D.S}{100}$, for $100 : S = D : A_1$.

$$A_1 = \frac{D.S}{100} \ (e.\ g.,\ A_1 = \frac{48.905 \times 14.56}{100} = 712.057).$$

This proportion of alcohol results from G parts by weight of fermented liquor; hence, $G : \frac{D.S}{100} = 100 : A$.

$$A = \frac{D.S}{G} \ (e.\ g.,\ A = \frac{712.057}{76} = 9.369),$$

wherein A is the percentage by weight of alcohol in the liquid under examination. As S is a function of s, and this again of D, *i. e.*: As each particular quantity of distillate has its individual, fixed percentage of alcohol, the products, D and S, may be arranged in a table. Such a table for 60° F., after Fownes (up to 6.074 per cent.), has been published by Leyser (Bayer. Bierbrauer, **8**, 121). The author has published such a table up to S = 14.98 (*i. e.*, 18.42 vol. per cent.), at 14° R. (17.5° C.). (Holzner, Attenuationslehre, Berlin, 1875–'76, table VIII.) The product (D.S) from the table must be divided by G. If G be constant (= 76 grms), this division may be made once for all, and it is only necessary to look for D in the table, to find the percentage by weight.

Fleury (Archiv. d. Pharm., **213**, 176, through Zeitschr. f. anal. Chem., **18**, 487) employs the following *method for the approximate* *estimation of alcohol in wine, etc.* The liquid under investigation is shaken up with a mixture of four volumes amylic alcohol and one volume washed ether, and the alcohol estimated from the resulting diminution in volume of the original liquid. The process is less exact when the proportion of alcohol exceeds forty-two per cent. In this case, the liquid must be diluted with twice or thrice its volume of water before the shaking. The method requires special tables, stating the relation between the diminution in volume and percentage of alcohol under the conditions of the experiments.

J. C. Turesh (Chem. News, **38**, 251, through Zeitschr. f. anal. Chem., **18**, 487) recommends the following *method for the detection and estimation of small quantities of alcohol*: 100 c.c. of the liquid under examination are mixed with 2 c.c. of saturated potassium pyrochromate solution, 8 c.c. dilute (1:1) sulphuric acid, a few fragments of pumice stone added, and the mixture distilled until 20 c.c. have come over. The distillate, after adding 3 c.c. concentrated sodium hydrate solution, is boiled for a few seconds. If the original liquid contained 0.1 per cent. of alcohol, there is obtained by the foregoing method a yellow liquid, from which aldehyde-resin deposits. If the alcohol is but 1.05 per cent., no resin deposits, but the liquid is dark yellow and opalescent. With 0.01 per cent. alcohol, the yellow color is just perceptible. For more exact estimations, the author prepares a solution for colorimetric comparison as follows: One part of pure aldehyde is diluted with two hundred parts water, thirty parts concentrated sodium hydrate solution are added, and the mixture warmed for some time. After two hours, two hundred parts of warm methylic alcohol are added, and the mixture made up with water to five hundred parts. The solution is then clear, of a reddish-yellow color and, if kept in a dark place, will last a long time. 5 c.c. of this solution mixed with 45 c.c. water, afford the test liquid for comparison; as, however, this dilute solution does not keep long, it is best to substitute therefor a solution of potassium pyrochromate of the same intensity of color. In quantitative estimations, the distillate is mixed with warm wood-spirit until it has become clear, and then diluted with water to 50 c.c. The quantity of this solution which is necessary, in order when diluted with water to 50 c.c., to give a liquid of the same color as the standard solution, is then determined, and from this the percentage of alcohol may be calculated.

M. Fordos (Bull. Soc. chim. de Paris, **26**, 487, through Zeitschr. f. anal. Chem., **18**, 493) recommends the following *method for the detection of fuchsine in wine*, as being the easiest and most reliable :

10 c.c. of the wine are shaken up with twenty drops of ammonia solution, 10 c.c of chloroform are then added, and the closed test-tube, containing the liquid, is inverted back and forth a few times without shaking. The chloroform is then transferred, by means of a separatory funnel, to a porcelain dish containing a little piece of white silk, and evaporated on the sand-bath. As the chloroform evaporates, the fuchsine, if present, tinges the silk of a rose color. When the evaporation approaches its end, water is added, and the heat continued. To prove that the color is fuchsine, the silk is moistened with ammonia, when, if that substance be present, it will lose its color, but recover it if the ammonia be expelled by heat. The author states that one-tenth mgrm fuchsine, in a liter of water, may be detected by the foregoing method.

Yvon (Répert. d. Pharm., **176**, 223, and Archiv. d. Pharm., 1877, 272, through Zeitschr. f. anal. Chem., **18**, 493) effects *the detection of fuchsine in wine*, by shaking 30 c.c. of the suspected sample with one to two grms bone black, filtering through asbestos, washing the bone black with water, and then pouring alcohol over it. If fuchsine be present, the alcohol will become instantly red. Alcohol does not extract the natural coloring matter of wine from its combination with bone black.

E. Bouilhon (Pharm. Zeitschr. f. Russland, **16**, 80, through Zeitschr. f. anal. Chem., **18**, 494) employs the following *method for the detection of small quantities of fuchsine in wine:* 500 c.c. of the wine are evaporated in a dish to about 125 c.c.; the dish is then removed from the fire, and 20 grms crystallized barium hydrate is added, and the mass thoroughly mixed by stirring. When cold, the liquid is filtered off, and the precipitate washed with water until the filtrate and washings measure 125 c.c. A few crystals of barium hydrate are then added to the filtrate, to ascertain if the precipitation of coloring matter is complete; if not, the treatment with barium hydrate must be repeated. The filtrate is then shaken violently in a 250 c.c. flask, with 50 to 60 c.c. of ether. The ethereal solution is separated, transferred to a porcelain dish, one drop of acetic acid and 3 to 4 drops of distilled water added, and a bunch of fine, white silk threads immersed in the liquid. If the quantity of fuchsine be not too small, the characteristic rose color will at once appear, otherwise the ether is allowed to evaporate, and the watery residue cautiously warmed to facilitate the fixing of the coloring matter on the silk. According to the author, $\frac{1}{100,000,000}$ of fuchsine may thus be detected in wine.

CH. GIRARD (Bull. Soc. chim. de Paris, **26**, 520, through Zeitschr. f. anal. Chem., **18**, 494) has devised the following *method for the detection of certain of the aniline colors in wine*: 150 c.c. wine are made distinctly alkaline with solution of barium or potassium hydrate, and then extracted with 25 to 30 c.c. of acetic ether or amylic alcohol. The filtered extract is rapidly evaporated over a woolen or silken thread, or better, the two together. If the thread has assumed a red color, it is moistened with a few drops of concentrated hydrochloric acid. *Fuchsine* is thus discolored and assumes the color of dead leaves; *safranine* passes through violet and blue, ending in a clear green. If small portions of water be successively added, these color changes take place in inverted sense, a large addition restoring the original color. The *violet* aniline colors which are soluble in water, give, with hydrochloric acid, first a bluish-green, then a yellow color. Water, in excess, restores the violet color. *Mauve aniline* gives with hydrochloric acid, first an indigo blue, then a yellow, finally, like fuchsine, the color of dead leaves; excess of water changes the solution to reddish-violet. Chryso-toluidine is very slightly decolorized by hydrochloric acid; on boiling the solution with zinc dust, colorless leuco-derivatives result, while the color of chryso-toluidine reappears by exposure to the air. *Aniline brown* is fixed on the fibre with a reddish-yellow color; in contact with air or with a drop of dilute hydrochloric acid, this passes to deep brownish-red. The somewhat concentrated acetic acid solution dyes uniformly brownish-red; in dilute solution, yellowish-brown. *Fuchsine* may be distinguished from cochineal by treating the dyed fibre with a drop of sodium bisulphite; the former is completely decolorized, the latter very slowly.

GUYOT and BIDAUX (Compt. Rend., **83**, 982; Chem. Centralbl. [3 F.], **8**, 71, and Pharm. Centralhalle, **18**, 114, through Zeitschr. f. anal. Chem., **18**, 495) effect *the detection of coralline with fuchsine in wine*, by making the wine alkaline with ammonia, and shaking out with ether. Fuchsine passes into ethereal solution, and may be identified by the production of a red color, with acetic acid. Coralline, if present, remains in the aqueous solution which, after being freed from ammonia by evaporation on the water-bath, then gives, with acetic acid, the characteristic yellow color, passing to rose color, on addition of ammonia.

G. CHANCEL (Compt. Rend., **84**, 348, through Zeitschr. f. anal. Chem., **18**, 496) communicates the following *systematic course for the detection of the coloring matters chiefly employed in the coloration of wine:* 10 c.c. of the wine are warmed with 3 c.c. of a 5 per cent.

solution of basic lead acetate (or as much more as may be necessary to constitute an *excess*), filtered, and the precipitate washed with warm water. If the filtrate be colored, it contains fuchsine, which may be extracted by shaking out with amylic alcohol. Extremely small quantities of fuchsine may, however, remain in the precipitate. The precipitate is treated, on the filter, with a two per cent. solution of potassium carbonate, which is poured back, and allowed to run through several times. By this means, any precipitated fuchsine, cochineal and indigo, may be extracted, while logwood and alkanna-reds, remain in the precipitate. With natural wines, the alkaline filtrate is yellow, or weak greenish-yellow.

Cochineal.—The alkaline liquid is acidified with acetic acid, and freed from fuchsine, by shaking out with amylic alcohol. The potassium carminate and indigo sulphate remain undecomposed, in the acetic solution. On adding to the latter sulphuric acid, these acids are set free and, by extraction with amylic alcohol, the carmine may be removed. If the wine be rich in cochineal, the amylic solution is red, otherwise resource must be had to the spectroscope.

Indigo.—The sulph-indigotic acid remains in the acid liquid from the last extraction, and communicates to it a *blue* color.

Logwood.—If the lead precipitate, containing logwood and alkanna, be shaken with solution of potassium sulphide, the coloring matters of logwood and of natural wine, dissolve. The former may be directly tested for in the wine, as follows: If a few cubic centimeters be heated with one to two drops of lime-water, the filtrate will appear of a greenish-yellow, if the natural, wine-coloring matter alone be present; of a handsome red, if logwood has been added.

Alkanna-red may now be extracted with alcohol, from the residue of lead sulphide left from the preceding operation, and identified by its red color.

A. Dupré (Analyst, 1877, 186, through Zeitschr. f. anal. Chem., 18, 497) employs the following *method for the detection of foreign coloring matters in wine:* From a jelly prepared from 5 grms gelatine and 100 c.c. hot water, and cooled, the author cuts cubes of about three quarters of an inch (18 m.m.), and immerses one of these for twenty-four to forty-eight hours, in the suspected wine. It is taken out, washed, and a section cut through the middle, parallel to one of the sides. This section is placed on a piece of white paper, or glass, and the color examined. If the wine be pure, the cube will only be colored to the distance of about one-sixth to one-eighth inch (15 to 3 m.m.) below the surface. Foreign coloring matters usually penetrate deeper, and show their characteristic *nuances*. According to the author, the coloring matters of pure wine and of the ratanhy root, penetrate the gelatine cube very slowly; fuchsine and the coloring matters of cochineal, logwood, Brazil wood, indigo, litmus, red cabbage, red beet, *Malva sylvestris* (mallow flowers) and *Althaea officinalis*, on the contrary, rapidly.

A. BAUDRIMONT (Comptes Rendus, **84**, 1,228, through Zeitschr. f. anal. Chem., **18**, 496) tests for *fuchsine in wine*, by simply spreading a drop of the suspected liquid on the hand, and allowing it to evaporate. If fuchsine be present, a red spot, which will not be removed by washing with water, will be left.

LEO LIEBERMANN (Ber. d. Wiener Akad., 1878, 43, through Zeitschr. f. anal. Chem., **18**, 497) has investigated *the spectrum of fuchsine*. He finds the very intense and characteristic absorption lines, to be between 130 and 138 (the sodium line being placed at 120); also, between D and E, nearer to E, between the yellow and the green. One part of fuchsine, in 500,000 of red or white wine, may be detected by the spectroscopic method.

The following papers, on the subject of *the detection of artificial coloring matters in wine*, are mentioned, by title only, in Heinrich Fresenius's Report, on the progress of analytical chemistry (Zeitschr. f. anal. Chem., **18**, 497):

V. Griessmayer, *studies on the coloring matter of wine, and on the coloration of wine* (Dingl. polytechn. Journ., **223**, 531). A. Wilger, *the properties of the coloring matter of genuine red wine, and those of mallow flowers, blue berries, fuchsine, etc.* (Archiv. d. Pharm. [3 R.], **9**, 481). F. v. Lepel, *the spectroscopic detection of the juice of red beets and the coloring matter of red poppies, in wine* (Berl. Ber., **11**, 1,552), and Hermann W. Vogel, *investigations on the adulterations of wine* (Berl. Ber., **9**, 1,906).

AUBRY (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 34) recommends the *testing for salicylic acid in beer, by dialysis*. It is by this method easy to separate the salicylic acid from the greater portion of the coloring matters, etc. In testing for small quantities, the dialysate is evaporated to dryness, and the residue extracted with a little alcohol.

H. ZERENER (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 34, 37, 41) urges the indispensability of a *microscopic examination, as an adjunct to the analysis of potable water*.

F. BECKER (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 58) recommends the following *method for the investigation of bees-wax*, based on the method of Koettstorfer (Zeitschr. f. anal. Chem., **18**, 199, and this JOURNAL, I, 359) for the investigation of butter. 2 grms of the re-melted and filtered wax are placed in a 150 c.c. flask; 25 c.c. normal or semi-normal alcoholic solution of potassium hydrate are added ; the flask closed with a rubber stopper, through which passes a safety tube, containing mercury, and of such size that a mercury column of 5 c.m. can be maintained therein. The flask is then placed in a water-bath, the water in which is gently boiling. As soon as the wax is melted, the flask is shaken, to promote the solution, and then allowed to remain for half an hour in the water-bath. The pressure of the 5 c.m. of mercury is sufficient to prevent the contents of the flask from boiling, and the saponification, which does not proceed readily at ordinary pressures, is quite perfect. The flask is then removed from the bath, 50 c.c. of absolute alcohol and the necessary quantity of phenol phthalein (as an indicator) added, and the excess of potassium hydrate titrated with normal hydrochloric or nitric acid. The addition of cold alcohol and normal solution causes the solution to become turbid, whence it must be returned to the water-bath until it becomes again clear. From the experiments of the author, it appears that 1 grm of pure bees-wax requires from 97 to 107 mgrms of KHO. The author finds the materials usually employed for adulteration of bees-wax to possess the following alkalimetric titres, viz.: Rosin = 194.3, Japan wax = 222.4, carnuaba wax = 93.1, spermaceti = 108.1. Koettstorfer finds that of tallow to be = 196.5. Cerasine and paraffine do not, of course, act on the normal alkali. In using the method of Koettstorfer, on butter, the author finds that the normal alcoholic potassa solution may be replaced by 10 c.c. normal KHO and 50 c.c. absolute alcohol, to 1 to 2 grms butter.

CH. BONDY and L. BORDET (Bull. Soc. chim. de Paris, **32**, 4) communicate a paper on *the determination of methylic alcohol, in commercial wood-spirit.* The method employed by the authors is essentially that of Krell (Berl. Ber., **6**, 1,310), *i. e.*, transformation of methylic alcohol into methyl iodide, by treatment with phosphorous iodide (PhI_2) and hydriodic acid, and distillation. The authors place 15 grms of phosphorous iodide in a flask, to which are attached, by a ground glass stopper, a stoppered pipette and the inner tube of a Liebig's condenser, the capacity of which has been increased by blowing three bulbs on the part within the outer condenser tube. The condenser is reversed, and 5 c.c. of wood-spirit placed in the pipette, and allowed to fall slowly, drop by drop, on the iodide. When the action is finished, 5 c.c. of a solution of one part iodine in one part of hydriodic acid (sp. gr., 1.7), are introduced, and the flask heated in the water-bath to 80°–90° C., for a few minutes. The condenser is then righted, and the contents of the flask distilled into a graduated tube ; 5 or 6 c.c. of water are then introduced into the flask and, after emptying the outer condenser tube of its water, caused to boil violently for two or three minutes ; the vapor cleanses the condenser from the phosphonium iodide which it usually contains. The graduated tube is then detatched, 7 or 8 c.c water introduced, and the distillate well shaken therewith. After standing, the volume of the separated methyl iodide is then read off on the graduations of the tube. With pure methylic alcohol, the authors obtained satisfactory results. As, however, acetone is always present, and tends to increase the volume of the methyl iodide, the authors have been led to devise the following mode of correction : If the water, with which the distillate is first shaken, be pipetted off, and the distillate shaken again with the same quantity (7 or 8 c.c.) of fresh water, the diminution in volume will be proportional to the percentage of methyl iodide in the mixture of that substance and acetone. From the results of their experiments, the authors have constructed the following table :

Percentage of Diminution in Volume, on Shaking with Water.	Percentage of Methyl Iodide, in the Mixture of Iodide and Acetone.	First Column Continued.	Second Column Continued.
(*n.*)	(*p.*)	(*n.*)	(*p.*)
18.7	70	8.3	86
18.0	71	7.7	87
17.3	72	7.1	88
16.6	73	6.5	89
15.9	74	5.9	90
15.2	75	5.3	91
14.6	76	4.7	92
13.9	77	4.1	93
13.2	78	3.5	94
12.5	79	2.9	95
11.8	80	2.4	96
11.2	81	1.8	97
10.6	82	1.3	98
10.0	83	1.0	99
9.5	84	0.8	100
8.9	85		

The data furnished by the operation are : V, the volume of the iodide, after the first washing ; V_1, the volume of water used in this washing ; n, the diminution in volume from the second washing ; p, the percentage of methyl iodide corresponding to n in the table. The total quantity of iodide produced in the analysis, is, therefore, obtained by the following formula, wherein a is the constant error peculiar to the apparatus used, and determined, once for all, by experiment with pure methylic alcohol : $V_{,}p + V_1 \times 0.008 + a$. The factor, 0.008, represents the coefficient of solubility of methyl iodide, in the liquid of the first washing. The authors find that the *best* commercial wood-spirit contains but 94 to 95 per cent. of methylic alcohol, while inferior grades may contain only 35 to 40; the average article contains from 75 to 90 per cent.

PAUL DEGENER (Journ. f. prakt. Chem. [N. F.], **17**, 390, through Zeitschr. f. anal. Chem., **18**, 488) recommends the following *method for the volumetric estimation of phenol*: On addition of bromine water to a five per cent. solution of phenol, the liquid remains at first clear, owing to the fact that tribrom-phenol is somewhat soluble in water; it then turns milky and, by continued shaking, deposits a voluminous, snow white, flocculent precipitate of very thin interlaced needles. As soon as enough bromine has been added to satisfy the equation:

$$C_6H_5OH + 3Br_2 = C_6H_2Br_3OH + 3HBr$$

the slightest excess may be detected with potassium iodide and starch paper, or, after a little practice, by the yellowish coloration, which is best observed on placing the vessel on a light-blue surface.

If the liquid under examination contains up to five per cent. of phenol, a single drop of a solution of 40 grms bromine to the liter, added in excess of the quantity required to transform the phenol to tribromide, will show the reaction, provided the volume of the liquid does not exceed 40 to 50 c.c. To prepare such a solution, the bromine is dissolved with half its weight of potassium bromide. The titre of the solution must be determined the day it is used, by titration with potassium iodide and sodium hyposulphite. If an excess of bromine has been added to the phenol solution, it may be titrated back with potassium iodide and sodium hyposulphite, or with pure phenol solution. If the reaction with potassium iodide and starch paper be employed, only an instantaneous and distinct blue coloration is decisive. A slight coloration usually soon disappears again. The amount of phenol solution taken for examination should not be less than 10 c.c. nor more than 30 c.c., unless it be very dilute. Compounds which react with bromine, *e. g.*, sulphurous acid, must be removed before proceeding to the titration.

NOTE.—The foregoing method is substantially identical with that devised by Elwyn Waller, and published in *The Sanitarian* November, 1874, 337.

A. STROMEYER (Corr.-blatt d. Vereins analytischer Chemiker, **2**, 26) states that *the valuation of crude pyrolignate of lime* must be effected by determination of the percentage of acetic acid (distillation with phosphoric acid), and not, as is frequently done, by determination of the percentage of lime. The results obtained by the method last named are 4 to 5 per cent. too high.

L. FLEISCHMANN (Polytechn. Notizblatt, **34**, 47, through Zeitschr. f. anal. Chem., **18**, 479) recommends that the *testing for alcohol in ethereal oils and chloroform*, should be conducted as follows: The oil is well shaken with water; the watery layer is then removed, and treated with a few drops of potassium pyrochromate and an excess of concentrated sulphuric acid. If alcohol be present, it will be oxidized to aldehyde by the free chromic acid, and the solution will assume a green color.

VI. MISCELLANEOUS PHYSICAL METHODS, APPARATUS AND REAGENTS.

F. A. GOOCH (Proc. Am. Academy, Feb. 13, 1878) has devised the following *method for the separation and subsequent treatment of precipitates in chemical analysis*: A platinum crucible of ordinary size, and preferably of the broad, low pattern, is chosen, and the bottom perforated with fine holes (the finer and more numerous, the better) by means of a steel point; or, the bottom may be made of fine platinum gauze. The crucible is suspended in a Bunsen funnel by the plan used by Munroe (Am. Journ. [3], **1**, 1) for supporting his porous cones, *i. e.*, a short piece of rubber tubing is passed over the top of the funnel, and drawn down until it arranges itself at right-angles to the stem, and the crucible suspended in the opening. The funnel being connected with the pump, and the latter started, a small portion of white, silky, anhydrous asbestos previously scraped to a fine, short down, boiled with hydrochloric acid, to separate all soluble matters, washed by decantation and suspended in water, is poured into the crucible. The water is almost instantly drawn through, and the asbestos is deposited in an uniform layer, the thickness of which may be increased, at pleasure, by further additions; a little distilled water is then run through to remove loose filaments of asbestos, and the filter is ready for the ignition and weighing preliminary to use. If the precipitate is to be heated for a very long time, it is best to enclose the filter crucible in another, because in such cases the asbestos is apt to curl at the edges, and some of the precipitate might drop through the perforations and be lost. For ordinary ignitions, this is unnecessary; the ignition of readily reducible substances must, of course, be conducted with avoidance of a reducing atmosphere. In using the filter, the vacuum pump must be started before pouring on the liquid. For turbid liquids or gelatinous precipitates, the author prefers to use a cone, of which the lower part is of fine platinum gauze, the upper part, where it is embraced by the rubber collar, of foil. The asbestos felt is prepared in the cone, in the manner already described. The experiments of the author show that the filter is not exposed to loss in weight from the prolonged filtration of strong mineral acids, or from mechanical loss, even in filtering viscid liquids. The method appears to be a neat, convenient and very acceptable addition to our experimental resources.

E. CAPPELL (Naturforscher, 12, 29, through Zeitschr. f. anal. Chem., 18, 466) has been led by his investigations on *the spectrum of calcium* to the conclusion previously reached by H. Fleck (Zeitschr. f. anal. Chem., 11, 73), *i. e.*, that lime is no simple alkaline earth, but a group of several similar substances. The author dissolved calcite from several different localities, in nitric acid and examined, by Vierordt's method, the relative luminosity of the Caδ line in the spectra yielded by the induction spark. The differences noted in this respect were so serious, that the author is led to conclude that the most characteristic of all the lines in the calcium spectrum (Caδ), represents an element which is present in very variable quantities in calcite from different sources, and from different geological periods; the luminosity being the greater, the older the formation from which the specimen is derived. He furthermore advances the hypothesis that the true number of elements is as great as that of the metallic spectral lines, and that every body in its *elemental* state is represented by a single spectral line.

G. HUEFNER (Journ. f. prakt. Chem. [N. F.], 16, 290, through Zeitschr. f. anal. Chem., 18, 451) describes *a new form of spectrophotometer* which is designed to avoid the objection to the, otherwise excellent, apparatus of Vierordt, that the alteration in the width of a single half of the slit in the spectroscope, alters the *quality* as well as the *intensity* of the spectral lines. The author has combined the simplest form of Zöllner's astrophotometer (Zöllner, Photometr. Bestimmungen u. s. w., Leipzig, 1865) with a spectroscope, in such a manner, that the light passing through the upper half of the slit is previously polarized by reflection from a system of plane mirrors in front of the slit, while the lower half transmits ordinary light from the same luminous source. After passing through the prism of the spectroscope, the rays traverse a Nichol's prism, by the rotation of which the intensity of the polarized half of the spectrum may be diminished or increased at pleasure, while the other half remains unaffected. The loss of light caused by the polarization, is compensated for by means of a sliding prism of neutral tinted glass in front of the half of the slit which receives the ordinary light. The light transmitted by the two halves having thus been brought to the same intensity, the Schulz's vessel containing the solution is placed between the instrument and the luminous source, and the two halves of the spectrum brought again to the same intensity, by rotation of the Nichol's prism; the angle of rotation read on the graduated arc attached to the latter, affords the basis for the calculation of the coefficient of absorption. The author has made, with the foregoing apparatus, a number of determinations of the oxygenized coloring matter of blood, and found the mean error to be + 1.2317 per cent., and the probable error, + 0.83 per cent. The apparatus is manufactured by Mechanikus Albrecht, in Tübingen.

A. WAGNER (Carl's Repert. f. Experimental Physik, **15**, 199, through Zeitschr. f. anal. Chem., **18**, 460) describes the following *automatic regulator, for the operations of evaporation and distillation:* The dish or retort is suspended to one arm of a balance, the other arm carries a scale-pan, on which is placed a weight equal to that of the vessel and the amount of liquid it should retain, at the end of the operation. When the evaporation is terminated, the scale-pan descends, and sets in motion an arrangement which extinguishes the flame under the vessel.

G. BROESICKE (Med. Centrallbl., **16**, 833, and Chem. Centralbl., **10**, 7, through Zeitschr. f. anal. Chem., **18**, 460) recommends the following *mode of coloring microscopic preparations:* Smaller fragments of tissues or sections of fresh, or recently dried preparations, are placed for about an hour, in a one per cent. solution of perosmic acid; they are then removed, carefully washed, to remove the excess of acid, and placed for twenty-four hours, in a cold, saturated (1 : 15) solution of oxalic acid. On examining the preparations in water, or glycerine, they will then be found to manifest peculiar colorative effects. While, namely, certain animal tissues (specified in the original) appear colorless, or hardly perceptibly tinged, some appear of a bright, and others of a dark, carmine-red, while still others appear, more or less, of a wine-red, and almost every structure possesses a special shade which renders it, even for a beginner, easy of identification. Another series of tissues appear of a more or less, dark yellow, yellowish-brown to inky blackness.

C. Liebermann (Berl. Ber., 12, 1,294) recommends that *the evaporation of solutions in ether, chloroform, benzole and carbon disulphide*, be effected in an ordinary dessicator, charged with paraffine (preferably crude). The evaporation is very rapid. The paraffine soon deliquesces, but still retains its absorptive capacity. From the experiment of the author, it results that paraffine will absorb its own weight of:

Carbon disulphide	in	4	to	5	hours.
Ether	"	8	"	9	"
Chloroform	"	9	"	11	"

The absorption of benzole proceeds considerably less rapidly. Paraffine will absorb more than thrice its weight of carbon disulphide and more than twice its weight of ether. The solvents may be recovered by distillation. The author suggests the application of these facts to the purification of gases, *e. g.*, purification of coal gas from carbon disulphide.

Ferd. Fischer (Berl. Ber., 12, 1,696) describes an *apparatus for the determination of oxygen in air*, based on the principle of the method of Jolly (Pogg. Annalen [1879], 6, 538). The air is contained in a globe, of known capacity, communicating at its lower extremity, by means of a glass cock, with a mercurial manometer, and closed at the top by a cap containing another stop-cock. Through this cap pass stout metal rods, connected within the globe by a thin spiral of copper wire. The stop-cocks being closed, the rods are connected with the poles of a battery, and the copper spiral brought to a red heat by the passage of a galvanic current. The oxygen combines with the copper, and the diminution in volume thus produced, is estimated from the diminution in pressure indicated by the manometer.

W. v. Beetz (Sitzungsber. d. k. b. Akad. d. Wissensch. in München, 7, 92, through Zeitschr. f. anal. Chem., 18, 457) has investigated *the electro-motive force and internal resistence of several forms of thermo-electric batteries*, especially those of Noë (Zeitschr. f. anal. Chem., 17, 205) and of Clamond and Koch (idem, 14, 350, and 15, 333). While, for technical applications, the latter is, on account of its greater solidity of construction, to be preferred, the Noë battery possesses, for laboratory use, the great convenience, that by coupling several elements a strong current of very constant electro-motive force can be readily obtained. Through recent improvements, it has been rendered much more durable than in its old form.

Walther Hempel (Zeitschr. f. anal. Chem., 18, 404) describes *a gas furnace, with arrangements for oxidation*, which is a modification of the furnace previously (idem, **16**, 454) described by him. The new furnace is very simple in construction, is heated by a single Bunsen's burner, and can be used for cupellations and other oxidizing operations. It will be manufactured by C. Desaga, of Heidelberg.

Ferd. Fischer (Berl. Ber., **12**, 1,694) describes an *apparatus for the determination of the heat of combustion*, which he considers to possess advantages over the calorimeter of Fabre and Silbermann.

F. **Tschaplowitz** (Zeitschr. f. anal. Chem., **18**, 440) describes *a new form of volumometer* similar to, but somewhat simpler in construction than, the one described by Rudorff (Berl. Ber., **12**, 249, and this Journal, **1**, 379). The same author (idem, **18**, 440) describes an *apparatus for the estimation of fatty bodies*, for which he claims certain advantages over the forms hitherto in use.

Marx (Zeitschr. f. anal. Chem., **18**, 442) describes a somewhat complicated *arrangement for moving the rider-weight in weighing*, which, however, appears to possess no advantages over the simpler form on the Becker's balances, in general use in this country.

J. S. **Thomson** (Chem. News, **40**, 18) effects *the preparation of distilled water, free from ammonia*, by the following device: The vapors from the still or boiler are passed to the bottom of an iron drum of, say, ten gallons capacity; the top of the drum is connected with the worm, or other form of condenser. The water which gradually condenses in the drum, being maintained in continual ebullition by the steam blowing through it, is absolutely free from ammonia and, hence, suitable for such applications as the Wanklyn and Chapman method of water analysis. The water condensed in the worm is rich in ammonia.

Franz Stolba (Sitzungsber. d. Kgl. Böhm. Akad. d. Wissensch., through Corr.-blatt d. Vereins analytischer Chemiker, **2**, 24) recommends *lead oxalate as a standard for fixing the titre of solutions of potassium permanganate*, in the place of oxalic acid. The titration is conducted precisely as in the case of oxalic acid in sulphuric solution. The lead oxalate is prepared by precipitating lead acetate with oxalic acid, washing the precipitate, and drying at 120° C. It possesses the advantages that it is not hygroscopic, and has a high molecular weight (1 part = 0.4273 oxalic acid).

J. **Fletcher** (Chem. News, **40**, 154), under the title of *a new method of preparing hydrogen sulphide*, communicates the results of his experience with the method of Walter Skeye (Chem. News,), by heating roll sulphur with paraffine in a glass flask, and conducting the products into the solution to be tested. The gas which is at first given off is not sulphuretted, but when the mass becomes thoroughly mixed, hydrogen sulphide is given off abundantly. The evolution ceases at once when the heat is withdrawn. The method is a cleanly, simple and elegant substitute for the older methods, and is specially well suited for small laboratories. The "bumping" to which the mixture is liable, may be prevented by introducing a few pieces of broken tobacco-pipe. Too strong heat should be avoided to prevent volatilization of the contents of the flask. Care must also be taken, that on withdrawing the heat, the solution under examination is not drawn back into the flask.

NOTE: The foregoing process seems well adapted for the preparation of hydrogen sulphide, for use in toxicological investigations.

E. DUVILLIER (Compt. Rend., **84**, 444, through Zeitschr. f. anal. Chem., **18**, 461) employs the following *method for the recovery of platinum, from platinum residues:* 100 grms of potassio-platinic chloride are added, in small portions at a time, to a boiling solution of 50 grms sodium formiate and 50 c.c. sodium hydrate (30° Bé.), in one liter water; the reduction is attended with strong effervescence, whence the addition of platinic salt must be cautiously made. After the effervescence ceases, the mixture is boiled for fifteen minutes, the liquid poured off from the precipitated metallic platinum, and the latter washed several times by decantation, with hot water acidified with hydrochloric acid. Waste solutions of platinic chloride are first distilled, for the recovery of the alcohol, then treated, first with excess of sodium hydrate, and then with sodium formiate, in small portions, and the operation finished, as above described. Another method for the same object has been described in the *Zeitschr. f. anal. Chem.*, **13**, 51.

Reports on Foreign and American Patents Relating to Chemistry.

Foreign Patents.

Condensed from R. BIEDERMANN's Report to the German Chemical Society, by H. ENDEMANN.

ERNEST SOLVAY, Brussels: *Improvements in the manufacture of soda by the ammonia process.* (Germ. P., No. 8180, July 5, 1879.)—These concern mainly the decomposition of the ammonium chloride by lime. The lime, as formerly used in its doughy consistency, reacted but slowly. If quick-lime be slacked in a solu-