REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY, APRIL TO JUNE, 1879.

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1. ATOMIC WEIGHTS OF THE ELEMENTS.

DR. F. KESSLER ("Ist das Atomgewicht des Antimons, 120 oder 122?", Bochum. Ad. Stumpf., 1879) has subjected to a critical review the question of the *atomic weight of antimony.* The figures which have been obtained by different analysts are:

Schneider	(1856)	Sb = 120.30
Dexter	(1857)	" = 122.33
Dumas	(1858)	" = 122.00
Kessler	(1861)	" = 122.37
Cooke	(1873–1877)	" = 120.00

The author shows that the results of Schneider (Pogg. Annal., **97**, 483–484; **98**, 293–305) were obtained on a material (native Stibnite, from Arnsberg, Westphalia) which contains impurities of which he took no notice, and that his figures are necessarily too low; that the results obtained by J. P. Cooke, Jr. (Proc. Am. Acad. Sci., **13**, 1–71, and Sill. Jour. [3] **15**, 41–49 and 107–124) are discordant, and the methods employed by him liable to numerous sources of error which have not been duly taken into account in regard to their influence on the question of accuracy. He successfully refutes the objections which have been brought against the results of Dexter, Dumas and himself, by Schneider, Cooke and others, and maintains that the true atomic weight of antimony is 122.0.

LE-COQ DE BOISBAUDRAN (C. R., **86**, 941–943, through Pogg. Annalen. [Beibl.] **3**, 248) has found the *atomic weight of gallium* to be 70.032 and 69.688, mean, 69.865 (H = 1, O = 16). The determination was made by the ignition of gallium alum and of gallium nitrate. This atomic weight agrees with that of a body standing midway between Al and In (69.82). From the consideration of the relative positions of the spectral lines of Al,Ca,Zn on the one hand, and K,Rb,Cs on the other, the atomic weight is calculated at 69.86.

K. SEUBERT (Inaug. Diss. Tübingen, 1878, through Pogg. Ann. [Beibl.] **3**, 322) has determined the *atomic weight of iridium.* The determinations were made on the double salts—iridium-ammonium chloride and iridium-potassium chloride. The mean result attained was 193.220 ($H = 0.9975$), or 192.744 ($H = 1$).

A. TERREIL (Bull. Soc. chim. de Paris, **31**, 153) has redetermined the *atomic weight of aluminum.* The method employed by the author consists in decomposing gaseous hydrochloric acid by the metal, and measuring the volume of hydrogen evolved. The determinations were made at a red heat. The results obtained would indicate that the chloride, formed, possessed the composition :

Al	20.26
Cl	79.74
	100.00

This composition corresponds to the atomic weight 9, if the formula be $AlCl$, or to 13.5, if the formula be Al_2Cl_3. The latter number alone satisfies the law of isomorphism, and accords with the vapor density determinations of Deville and Troost (9,35; $Al_2Cl_3 = 2$ vols.).

II. GENERAL INORGANIC ANALYSIS.

EMIL SCHOENE (Zeitschr. f. anal. Chem., **18**, 133) contributes a voluminous paper on *the quantitative estimation of hydrogen peroxide.* After a description of the author's experience with the previously proposed methods, taken from a brochure printed in the Russian language ("Experimental Investigation on Hydrogen Peroxide," Moscow, 1875), the author proceeds to the description of the colorimetric method employed by him in the determination of small quantities of hydrogen peroxide, such as in atmospheric precipitates (rain, dew, etc.). The method with potassium permanganate rests, according to Aschoff (J. f. prakt. Chem., 1850, **81**, 404), on the reaction :

$$2KMnO_4 + 5H_2O_2 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$$

which was confirmed by the experiments of the author. The reaction can be employed as the basis either for a gasometric method, in which the volume of the oxygen resulting from the decomposition is directly measured, or of a volumetric method, wherein a titrated solution of permanganate is employed. The author did not test the gasometric method, but considers it to be unreliable on account of the variations in the coefficient of absorption of oxygen, and other gases contained in the air ; a simple calculation showing that 1 mgm of hydrogen peroxide cannot by this method be determined with certainty in one liter of liquid. The volumetric method, conducted by adding the permanganate solution from a burette to the solution, previously acidified with sulphuric acid, until a permanent pink tinge appears, was found by the author to be extremely perfect. One millegramme in a liter of liquid is susceptible of perfectly accurate determination, and $\frac{1}{10}$ millegramme with tolerable accuracy. According to all the authorities, hydrogen peroxide acts on hydrogen iodide according to the equation: $H_2O_2 + 2HI = 2H_2O + I_2$. This reaction has been made the basis of two quantitative methods, viz.: the iodometric (Brodie) and the acidimetric (Houzeau). In the former, the amount of iodine, separated, is determined by titration with sodium hyposulphite; in the latter, the amount of undecomposed hydriodic acid is determined by titration with centinormal alkali, after expulsion of the separated iodine by boiling. The former method is inferior to the permanganate method. The acidimetric method may be used with advantage where an error of 1 mgm in the liter is immaterial. Thenard's gasometric method is based on the decomposition of the hydrogen peroxide by heat, or by contact with manganese peroxide, blood-fibrine, etc., and measurement of the volume of oxygen produced. This method is inferior to the permanganate method, and to that of Houzeau. Methods based on the change of color produced in indigo and indigo-carmine, by oxidation and reduction, have also been proposed by J. Assmus and H. Struve, but it is still doubtful whether these methods are capable of yielding accurate results. In the author's determinations of the percentage of hydrogen peroxide in atmospheric precipitates, the quantities present rarely exceeded 1 mgm in the liter. The only one of the preceding methods, viz., that with permanganate, which was sufficiently delicate, was inapplicable, owing to the presence of other substances which also reduce permanganate. The author has, therefore, been led to devise a method, based on the coloration produced by hydrogen peroxide in a neutral solution of potassium iodide and starch. The reaction is so delicate that 0.00008 gm H_2O_2 in a liter, produces a weak violet coloration, which attains its maximal intensity in about 5 or 6 hours. The reaction is only applicable to solutions of not more than .001 gm to the liter. When this limit is exceeded, the color changes are no longer appreciable. The color scale is prepared by ascertaining, by means of the permanganate method, the strength of a solution of pure hydrogen peroxide, and diluting, so as to obtain solutions of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9 and 1 mgm, to the liter. These solutions are treated with potassium iodide and starch solution in the same manner as the liquid to be tested. When preserved in the dark, and in well stoppered glasses, these solutions remain unchanged for three to four weeks. The author prefers to prepare a new scale every ten to fourteen days.

J. Volhard (Ann. d. Chem., **190**, 1, through Zeitschr. f. anal. Chem., **18**, 271) has communicated a voluminous paper on the *application of the silver titration with ammonium sulphocyanide to the determination of the halogens, copper and mercury.* The method involves the use of two standard solutions, viz.: a deci-normal silver solution and a solution of ammonium sulphocyanide of the same strength. The indicator consists of a cold saturated solution of ferric ammonium alum. The titration of *chlorine* is as follows: the substance is dissolved in 200 to 300 cc of water; 5 cc of the iron solution is added, and then nitric acid until the color of the ferric salt disappears. A moderate excess of silver solution is then added from the burette, and then, without the necessity of filtration or even shaking or heating, to cause the silver chloride to agglomerate, the potassium sulphocyanide is added, the flask being continually shaken, to insure the rapid mixture of the sulphocyanide solution with the contents of the flask. As soon as the liquid assumes a light yellowish-brown color which, after shaking and letting stand for 10 minutes, does not disappear, the titration is finished. The difference between the volume of silver solution originally taken and that corresponding to the sulphocyanide added, is the measure of the chlorine in the substance. The titration of *bromine* is conducted in the same manner as that of chlorine. The titration of *iodine* requires certain precautions owing to the facility with which silver iodide carries down with it potassium iodide, silver nitrate, or both, which then enter very slowly into reciprocal decomposition with the silver or sulphocyanide solutions. The iodide is dissolved in two to three hundred times its weight of water, and the silver solution added from the burette. The silver iodide is, at first, in a state of extreme subdivision, and the liquid appears like a yellowish-white milk; as soon, however, as a slight excess of silver has been added, the liquid appears to *curdle*, and the precipitate separates out after a short shaking; $\frac{1}{10}$ to $\frac{2}{10}$ cc more of the silver solution is then added, and the liquid cleared by shaking. 5 cc of the iron solution are introduced and the titration is conducted with sulphocyanide solution, as above indicated. When the color first appears it is dispelled by shaking, owing to the silver nitrate occluded in the precipitate. The addition of small quantities of the sulphocyanide solution, with prolonged shaking, is continued until the color no longer disappears on shaking. The process is well adapted to the determination of the halogens in organic compounds.

When the halogens are present with sulphocyanides, the last named acid must be destroyed; if chlorine alone is sought, the acids are precipitated as silver compounds, and the sulphocyanide of silver is decomposed, in a flask, by warming with a mixture of 2 parts sulphuric acid and 1 part water, and addition of a few drops of nitric acid; if bromine and iodine are present, the method of Carius, or melting with alkaline nitrate and carbonate, should be employed. Chloride of silver may be separated from iodide of silver by treatment in ammoniacal solution with ammonium sulphocyanide. The chloride is completely transformed to sulphocyanide; the iodide remains unaffected. *Cyanogen* may be determined by precipitation with an excess of silver, filtration and washing of the precipitate, and titration of the excess of silver in the filtrate with sulphocyanide. *Copper* is determined by heating the solution to boiling, and then adding an excess of sulphurous acid; on the first addition of sulphocyanide solution, the liquid is colored of a dirty green, owing to the formation of cupric sulphocyanide; on shaking, however, the liquid becomes colorless, and a white precipitate of cuprous sulphocyanide is thrown down. When the copper is entirely precipitated, the last addition of sulphocyanide produces no change of color. The liquid is allowed to cool, diluted to 300 cc, filtered through a dry filter, and 100 cc of the filtrate titrated with silver solution, to determine the excess of sulphocyanide. *Mercury*, when present as a salt of an oxygen acid, may also be approximately determined in the same manner as silver. The author finally calls attention to another application of ammonium sulphocyanide in volumetric analysis, viz: as a measure of the strength of standard solutions of potassium permanganate. The oxidation of sulphocyanhydric acid proceeds according to the equation:

$$S_2C_2NH + 6O = 2SO_3 + C_2NH.$$

The solution of ammonium sulphocyanide possesses the great requisite of entire stability. The author has minutely indicated the precautions to be observed in each of the foregoing methods, and upon which the accuracy of the results largely depend.

W. J. SELL (Jour. Chem. Soc., 1879, **292**) has devised the following method for the *volumetric determination of chromium*. The solution of a salt of chromium is acidified with sulphuric acid, heated to boiling, and treated with solution of potassium permanganate, in small portions, until the liquid retains a distinct purplish tint, after boiling for three minutes. The chromium is thus oxidized to chromic acid, according to the equation:

$$5Cr_2(SO_4)_3 + 3K_2Mn_2O_8 + 6H_2O = 10CrO_3 + 2K_2SO_4 + 6MnSO_4 + 6H_2SO_4.$$

The solution is then rendered slightly alkaline by addition of sodium carbonate ; alcohol is added, to reduce the excess of permanganate, and the manganese removed by filtration. The chromic acid in the filtrate is determined by titration with iodine and sodium thiosulphate. In the analysis of chrome-iron ore, the ore is rendered soluble by fusion with potassium, or, preferably, sodium acid sulphate. Three determinations of chromic oxide in the same sample of chrome-iron ore gave the author 47.11, 47.23, and 47.11 p. c. of Cr_2O_3. The solution of the ore may be more quickly effected by fusion with potassium fluoride, or with a mixture of one molecule of sodium acid sulphate, and two of sodium fluoride.

Wm. Galbraith (Chem. News, **39**, 276) claims priority in the discovery of the foregoing method, and refers to his article in the *Chemical News*, **25**, p. 151, wherein it is recommended for the determination of chromium in iron and steel. The process there described is in daily use, and gives satisfactory results.

Alexander Classen (Zeitschr. f. anal. Chem., **18**, 175) communicates *a new method for the separation of ferric oxide and alumina from manganese.* The author has previously (Berichte d. deutsch. chem. Gesellsch., **10**, 1315) communicated a method for the separation of iron from manganese (nickel, cobalt and zinc), based on the following facts : If to a solution of a salt of manganese, there be added, drop by drop, a solution of neutral potassium oxalate, there is formed a white precipitate of manganous oxalate, which dissolves in an excess of the precipitant as potassio-manganous oxalate. If acetic acid be added to this solution, the double salt is decomposed, with separation of manganous oxalate, which is absolutely insoluble in acetic acid, although soluble in acid oxalates, and in oxalic acid. Ferric salts form, with a sufficient excess of potassium oxalate, potassio-ferric oxalate, which is not decomposed by acetic acid. The presence of the alkaline chlorides exerts an unfavorable influence on the precipitation of the manganous oxalate. When, however, the solution contains a sufficient quantity of a metal, the oxalate of which is not soluble in alkaline chlorides, the entire amount of manganous oxalate precipitates with the oxalate of the metal in question, even when a large quantity of alkaline chlorides is present. Zinc was found by the author to satisfactorily fulfill this condition. The process of the author is to be employed in two modifications, according as the material analyzed has been dissolved in hydrochloric acid, with addition of bromine, or in nitric acid. In the first case the solution is evaporated to dryness, the dry residue digested a few minutes with 5 to 10 cc bromine water, in a covered dish, on the water bath; then about 7 times as much potassium oxalate, as there is of oxides present, is added, and the mass digested ¼ hour on the water bath. Most of the iron and all of the manganese thus dissolve as oxalates. The undissolved residue of ferric oxide is dissolved by the gradual addition of dilute acetic or hydrochloric acid. The intensely green solution is concentrated until green crystals of ferric oxalate begin to separate (to about 20–25 cc, when 0.4 to 0.5 of iron has been taken), poured hot into a beaker of about 100 cc contents, the dish washed with hot water so that the bulk of the liquid is about 40 to 45 cc, a few cc alcohol added, to reduce any higher oxide of manganese which may have formed, and then zinc chloride solution, from a burette, until to 1 part of manganous oxide, 3 to 4 parts of zinc oxide are present. If too little potassium oxalate has been employed, more is added until the precipitate of zinc oxalate, which then forms, is, for the most part, dissolved. An equal volume of concentrated acetic acid is now added to the liquid, and the covered beaker allowed to stand a short time at 50–60° C., until the precipitate has become crystalline, and the supernatant liquid perfectly clear. For exact determinations 6 hours standing is required. The precipitate is filtered, washed, by decantation, with a mixture of equal volumes of concentrated acetic acid, alcohol and water, until the filtrate gives no iron reaction with potassium sulphocyanide (3–4 decantations, using each time 10 cc liquid, are sufficient), brought on the filter, dried and ignited, with free access of air, so as to form the compound of Mn_2O_3 and ZnO, mentioned by Bunsen and Krieger (Ann. d. Chem., **87**, 257, et seq.). The ignited precipitate is finally decomposed with strong hydrochloric acid, and the chlorine thus liberated, titrated with sodium hyposulphite. The precipitate is very apt to retain a small proportion of potassium oxalate, which, by ignition, passes first to carbonate, and then to permanganate. To avoid the excess which would result from this cause, the precipitate should first be heated for a short time only, then washed out with hot water, and again ignited, until the manganese ceases to absorb oxygen, before the chlorimetric titration. If the ferric oxide resulting from the solution of the residue of the first evaporation, has been dissolved in hydrochloric acid, it is necessary to carefully neutralize the excess of oxalic acid thus induced, before precipitation with acetic acid. If the substance analyzed has been dissolved in hydrochloric acid, with addition of nitric acid and evaporation to dryness, nitric acid must be added to bring the residue entirely into solution. The excess of acid must in this case, also, be neutralized with potassa or soda solution before precipitation. The method also permits the simultaneous separation of Co, Ni, Cu and Ca, from a large excess of iron, and these metals may be separately determined in the liquid from the chlorimetric determination. Phosphoric acid, when present, is also thrown down with the metals above mentioned. The author reserves for further investigation the question, whether the separation of this last-named substance is quantitatively exact. The results obtained by the author in the separation of quantities of manganous oxide, varying from 0.0022 to 0.4970 gm, from quantities of ferric oxide, varying from 1.51 to 2.85 gms, show the method to be extraordinarily exact, as well as extremely easy and rapid in execution.

ALEX. CLASSEN (Zeitsch. f. anal. Chem., **18**, 189) describes a method for *the quantitative estimation of cobalt, nickel and zinc, by precipitation as oxalates,* based upon the same principles and manipulation as in the preceeding method, except that no addition of zinc salts is required. The advantages of this method, compared with those previously in use, are, that the oxalates of these metals are more easily obtained in a state of purity than the hydroxides and carbonates precipitated by the older methods, and that these metals may be separated from ferric oxide and alumina without previous precipitation of the latter. The oxalates are converted into oxides by ignition—at first gentle and in a covered platinum crucible, and then intense, with free access of air. Nickel and zinc are weighed as oxides (NiO, ZnO) ; cobalt is reduced to the metallic state by ignition in a current of hydrogen. The analytical results obtained by the author demonstrate that in respect to accuracy and constancy the method leaves nothing to be desired. The presence of ammonia or potassium chloride causes the method to give too low results with cobalt, and of ammonium chloride, with zinc ; sodium acetate is without effect on the results.

ALEX. CLASSEN (Zeitschr. f. anal. Chem., **18**, 194) has investigated the method of Tamm (Fresenius, Quant. Anal., 6 Aufl., p. 575) for the *separation of manganese from zinc,* by the precipitation of the former as manganous carbonate by ammonium carbonate, in the presence of ammonium chloride, and finds that the method gives too high results, owing to the fact that the precipitate always contains more or less zinc carbonate, which cannot be removed by washing with ammonium carbonate.

JOHN PATTINSON (Jour. Chem. Soc., 1879, 365) communicates a *method of precipitating manganese entirely as dioxide, and its appli-*

cation to the volumetric determination of manganese. After a careful study of the conditions under which manganese may be precipitated as dioxide, the author has attained the same conclusion as Messrs. Wright and Luff (idem, **33**, p. 513), viz., that certain methods usually prescribed for producing hydrated manganese dioxide, do not yield this oxide alone, but a mixture of dioxide with various lower oxides. The author has tried other methods usually recommended for this purpose, with similar results. He finds, however, that the manganese may be invariably precipitated entirely as dioxide, *if a certain amount of ferric oxide be present in the solution*, when a sufficent excess of calcium hypochlorite or bromine water, and, after heating the solution to from 60° to 70° C., excess of calcium carbonate is added, and the mixture well stirred. The presence of half as much iron as there is of manganese, is sufficient to determine the precipitation of the latter wholly as dioxide. In practice, however, it is preferable to have equal quantities of each metal—a large excess of iron is no disadvantage. The experiments of the author showed that from 99.95 to 100.12 per cent. of the manganese is precipitated as dioxide under the above conditions. In the analysis of an iron ore, the author dissolves 10 grains in a 20 oz. beaker, in 100 fl. grs hydrochloric acid (sp. gr. 1.180) ; calcium carbonate is added until the liquid is slightly red, and then six or seven drops of hydrochloric acid ; 1,000 grs of a solution of bleaching powder (10,000 grs water to 150 bleaching powder) is now added, and boiling water poured in until the temperature is 60° to 70° C. ; 25 grs calcium carbonate is introduced, and the solution well stirred until the effervescence ceases. The dark brown precipitate settles readily. If the supernatant liquid has a pink color, owing to the formation of permanganic acid, a few drops of alcohol should be added to decolorize it. Instead of the bleaching powder, 500 grs of saturated bromine water may be used. The precipitate is filtered, washed, and placed in 1,000 grs of a solution of 530 grs ferrous sulphate in 10,000 grs of a mixture of 1 part sulphuric acid and 3 parts water, in which it readily dissolves, even in the cold, converting its equivalent of ferrous sulphate into ferric sulphate. Cold water is now added, and the liquid titrated with potassium dichromate, to determine the excess of ferrous sulphate. From the ferrous sulphate oxidized, the percentage of manganese may readily be calculated. If too little iron be present, pure ferric chloride (free from manganese) is added. The method is applicable to the analysis of spiegeleisen, ferromanganse, steel, slags, etc. The special precautions to be used in each case are fully described in the paper. The author claims for this method not only a great saving of time, but, in many cases, more accurate results than the gravimetrical method is capable of affording. The author further states that it is now possible to determine manganese volumetrically by this method, with as great exactitude and almost as speedily, as iron may be determined by Penny's method.

C. ROESSLER (Berl. Ber., **12**, 925) communicates a *new method for the volumetric determination of manganese, with the use of Volhard's method of silver titration.* The method is based on the formation of a compound, first observed by Woehler, and which H. Rose found to possess the composition $Ag_4OMn_2O_3$. The solution of the manganese compound (which must be free from the halogens and from organic compounds capable of reducing silver salts) is placed in a ¼ to ½ liter flask, treated with an excess of a solution, of known strength, of silver nitrate, and heated on the water bath. Sufficient sodium carbonate is added to completely precipitate the silver. A moderate excess of ammonium hydrate is then added, the flask is well shaken, cooled, filled to the mark, and the solution filtered through a dry filter. The precipitate on the filter possesses the above composition. By the determination by titration, by Volhard's method, of the amount of silver remaining in the filtrate, the proportion contained in the precipitate, and hence the weight of the latter, is readily ascertained. The reaction takes place even in the presence of a large proportion of ferric oxide ; on account, however, of the organic compounds resulting from the solution of spiegel, and other varieties of iron, it is necessary to separate the iron as basic acetate. The filtrate from this precipitate is then free from reducing substances, and suited for the foregoing method.

C. MANN (Zeitschr. f. anal. Chem., **18**, 162) has devised *a new method for the volumetric estimation of zinc,* based upon the reciprocal decomposition of hydrated zinc sulphide and silver chloride, and the estimation of the zinc from its equivalent contents in chlorine by means of Volhard's method of silver titration with alkaline rhodanates, using ferric nitrate or ferric ammonium sulphate as an indicator. The zinc is precipitated by hydrogen sulphide in acetic acid solution, the excess of the precipitant expelled by violent ebullition, the precipitate washed by decantation and filtration. The precipitate, with the filter, is then introduced into a flask, water and silver chloride added, and the whole boiled until the supernatant liquid is quite clear; finally 5–6 drops of dilute (1·6) sulphuric acid is added. The precipitate is then filtered off, washed, and the solution, after adding a measured volume of silver solution, titrated, as above. The determination consumes, from the moment of the introduction of the sulphuretted hydrogen to the end of the process, about 1½ hours. The results obtained by the author are exceedingly satisfactory.

Arthur G. Haddock (Chem. News, **39**, 156) has found that in the *titration of sulphuric acid with barium chloride*, as also in other cases where a minute quantity of solid precipitate is to be observed in a drop of liquid, the observation is greatly facilitated by placing the liquid on a mirror, instead of on the black glass hitherto recommended.

F. Beilstein and L. Jawein (Berl. Ber., **12**, 446) communicate *a method for the quantitative determination of zinc by electrolysis.* The solution is prepared by adding sodic hydrate to a solution of zinc nitrate or sulphate, until a precipitate is formed, and then adding potassium cyanide until the solution is again clear. With a current from 4 Bunsen's elements and platinum electrodes (Zeitschr. f. anal. Chem., **8**, 28 ; **11**, 6) the precipitation is at the rate of about 0.1 gm zinc per hour. When the precipitation is finished, the electrode with the adhering zinc is removed, washed with water, then with alcohol, and finally with ether, and dried in the air bath. After weighing, the zinc is dissolved off, the electrode re-weighed and again immersed in the solution to ascertain if the precipitation be complete. The method gives exact results. The authors made an analysis of brass by solution in nitric acid, evaporation to dryness, solution in water, and precipitation of the copper by electrolysis. The platinum spiral forming the positive electrode was coated with lead dinoxide, from which the percentage of lead was determined. The zinc was then determined, as above. The results of the analysis were :

Cu	67.13
Zn	32.93
Pb	0.01
	100.07

F. Beilstein and L. Jawein (Berl. Ber., **12**, 759) have been led by reason of the insufficiency of the methods previously in use, to attempt *the electrolytic determination of cadmium.* The authors employed a method similar to the one used by them in the electrolytic determination of zinc. The solution of cadmium chloride or nitrate was neutralized with potassium hydrate, treated with an excess of potassium cyanide, and subjected to electrolysis, using a spiral of thick platinum wire as the anode, and a cylinder of platinum foil as the cathode. The following results were obtained with electrolytic cadmium, using the current from 4 15-cm Bunsen's cells.

TAKEN.	FOUND.	TIME.
0.2356	0.2344	3 hours.
0.2565	0.2547	3 "
0.2344	0.2324	2 "
0.2547	0.2532	3 "

The authors attribute the slight loss in these experiments to the contamination of the electrolytic cadmium with platinum. The end of the precipitation is ascertained by testing with hydrogen sulphide. The precipitated metal is washed, first with water, then with alcohol, and dried by placing in a heated platinum dish.

E. F. SMITH (Am. Jour. Sci., **117**, 60) communicates the following method for *the electrolytic determination of cadmium.* The solution of *acetate* of cadmium is placed in a platinum crucible, which it should fill to about one-half, and placed in contact with the negative pole of a two-cell Bunsen's battery, the positive pole is formed by a piece of platinum foil dipping into the crucible. In about 3 hours the cadmium (0.1270 gm) was entirely deposited, as a crystalline greyish-white coating on the crucible. The precipitated metal was washed with water, then with alcohol, and finally with ether. The conditions for success are : 1st, to work with concentrated solutions, and 2nd, to have the current of sufficient strength for rapid and energetic action.

PH. DE CLERMONT (Comptes Rendus, **19**, May 12, 1879, through Chem. News, **39**, 251) has investigated the *action of ammoniacal salts on certain metallic sulphides.* Bismuth, cadmium, copper, mercuric and mercurous sulphides, are not affected by boiling with solution of sal-ammoniac. Antimony trisulphide yields antimonous chloride, which dissolves, and ammonium sulphide, which escapes. Stannic and stannous sulphides yield respectively stannic and stannous oxides. Metals precipitable by ammonium sulphide, but not by hydrogen sulphide in acid solutions, behave in a peculiar manner with sal-ammoniac. Manganese, iron, nickel and cobalt sulphides dissolve, the last two more slowly. Zinc sulphide resists longer, but ultimately dissolves. Alumina and chromic oxide are insoluble. On these reactions the author founds a method for the separation of certain metals. The solution containing the precipitate produced by ammonium sulphide with salts of cobalt, nickel, manganese, iron, aluminum, chromium and zinc, is added to a boiling solution of sal-ammoniac, and boiled for a sufficient time. On filtering, the filtrate contains all the iron and manganese, and part of the cobalt, nickel and zinc, while the undissolved portion contains all the alumina and chromium with the residue of the nickel, cobalt and zinc. The analysis is completed by the usual methods. If iron and manganese alone have to be separated from aluminum and chromium, the process is complete and precise.

F. Beilstein (Berl. Ber., **11**, 1,715, through Zeitschr. f. anal. Chem., **18**, 262) has found that *the separation of nickel from zinc* may be effected by precipitating the latter with hydrogen sulphide from solutions containing citric acid or soluble citrates. The precipitation of the zinc is complete after standing 24 hours in the cold, while the nickel can be determined by electrolysis of the filtrate, after evaporation and saturation with ammonia. As ammonium chloride hinders the electrolytic precipitation of the nickel, the metals should be in solution as nitrates. With due observance of the precautions indicated by the author, the method gives extremely accurate results.

Julius Thomsen (Berl. Ber., **11**, 2,043, through Zeitschr. f. anal. Chem., **18**, 263) has found that *the precipitate produced by hydrogen sulphide in solutions of cupric salts*, is not CuS, but a mixture of sulphur with a sulphide, possessing the formula Cu_4S_3.

L. F. Nilson (Fres. Zeitschr. f. anal. Chem., **18**, 165) contributes *a criticism of Bunsen's older method of separating arsenic from antimony*, based upon the reaction between the sulpho-salts of these metals and potassium bisulphite. As Bunsen himself (Ann. Chem. Pharm., **192**, 305) has announced that he no longer employs this method, in its original form, and has substituted a method free from the objections to which Nilson has called attention, it is unnecessary to reproduce this somewhat acrimonious article.

R. Bunsen (Ann. d. Chem., **192**, 305, through Zeitschr. f. anal. Chem., **18**, 264) describes in detail the method at present employed by him in the *separation of antimony from arsenic*. The mixture of sulphides is dissolved on the filter in solution of potassium hydrate (purified by alcohol). The solution and washings are placed in a 150 cc porcelain crucible, and saturated with chlorine gas. The contents of the crucible are then treated with an excess of concentrated hydrochloric acid, evaporated to one-half on the water bath, diluted to their previous volume with concentrated hydrochloric acid, and again evaporated to one-half or one-third, to remove the free chlorine. The solution is mixed, in a suitable vessel, with 100 cc saturated solution of hydrosulphuric acid for each decigramme of antimonic acid it may contain, and after the precipitate of antimony pentasulphide has thoroughly subsided, the excess of gas is removed by causing a rapid current of air to pass through the liquid. The liquid is filtered through a weighed filter with the aid of the water pump, and the precipitate washed, first 8 times with alcohol, then 4 times with carbon disulphide, and, finally, thrice with alcohol, dried, first on the pump, and then at 110° C., and weighed. It then consists of antimony pentasulphide, with possibly a trace of arsenic, from which it may be freed by repetition of the operations of solution, etc., before the washing with alcohol, etc., and drying. The filtrate is treated with a few drops of chlorine water, heated on the water bath, and a stream of hydrosulphuric acid allowed to pass through the warm liquid until it is cold. The liquid is allowed to stand for one day in a moderately warm place, and is then filtered through a weighed filter. The precipitate, which consists of arsenic pentasulphide, with a little sulphur, is washed and dried, as in the case of the antimony precipitate, and then weighed. The analytical results communicated by the author, show the method to be extremely exact. It will be welcomed by all chemists as a solution of one of the most difficult problems in quantitative analysis. From the experiments of the author, it appears that the weight of ammonic di-magnesium arseniate is only then constant, and its formula $2Mgo, NH_4O, AsO_5 + Aq$, after it has been dried by long (44 hours) exposure to the temperature of 102.5–105°. The author has previously (Ann. d. Chem., **106**, 3) recommended the estimation of antimony as antimony tetroxide, Sb_2O_4. The result of his more recent observations shows that the temperature at which antimonic acid is transformed into antimony tetroxide, lies so near to that at which the latter body is decomposed into antimony trioxide and oxygen, that an exact estimation can hardly be made by this method.

A. Millot and Maquenne (Comptes Rendus, **86**, 404, through Bull. Soc. Chim. de Paris, **31**, 544) recommend *the volumetric determination of arsenic*, by titration of arsenic acid in a boiling neutral or acetic acid solution, with uranium acetate, using potassium ferrocyanide as an indicator. The arsenic acid should previously be separated from those bases which would give compounds insoluble in acetic acid. This may be done by transforming it into arsenuretted hydrogen, which is passed into fuming nitric acid, whereby the arsenic is reconverted into arsenic acid.

A. Carnot (Comptes Rendus, **86**, 478) has devised a *method for the volumetric determination of potassium*, based upon the precipitation by alcohol of potassio-bismuth hyposulphite. The determination of the hyposulphurous acid in the latter, is conducted by titration with iodine, the reaction being the transformation of the former to tetrathionic acid, as in the equation :

$$2S_2O_3Na_2 + I_2 = 2NaI + S_4O_6.$$

To prevent the deposition of red bismuthous oxide, the liquid is slightly acidified with chlorhydric acid. In adding the iodine, the liquid assumes first a golden-yellow color, passing to persistent brown when the point of saturation is attained. The potassium is calculated from the amount of iodine added. 1 atom I corresponds to 1 of potassium. The iodine solution contains 12.7 gms dissolved in 18 gms potassium iodide, or it may contain 26.96 gms to the liter, in which case 1 cc corresponds to 0.01 gm potassa (KO).

GASTON BONG (Bull. Soc. Chim. de Paris [N. S.], **29**, 50, through Zeitsch. f. anal. Chem., **18**, 270) recommends that litharage should be used for the decomposition of silicates. As a rule, three times the weight of the substance is sufficient, and the action is very rapid. The decomposition may be effected in a platinum crucible, in an oxidizing flame; but all carbonaceous or other reducing substances must first be removed by roasting. The fusion is dissolved in nitric acid, evaporated to dryness, redissolved in dilute nitric acid, and the silica filtered off. The lead may be removed by hydrogen sulphide or sulphuric acid, and the bases determined by the usual methods. The method is specially recommended for the determination of the alkalies in silicates.

GERHARD LOESEKANN (Berl. Ber., **12**, 56) has investigated *the behavior of alkaline solutions of aluminum hydrate towards hydrogen sulphide.* He finds that aluminum hydrate is completely precipitated by hydrogen sulphide from solution in sodium or potassium hydrate, and that the precipitate redissolves on boiling. If solutions of zinc oxide and alumina be precipitated by hydrogen sulphide, the alumina can be completely dissolved out by caustic alkalies. Alkaline solutions of chromic oxide are also completely precipitated by hydrogen sulphide, but the precipitate does not redissolve on boiling. The precipitate dissolves if soda solution be added immediately after the precipitation, but not after standing 18 hours.

E. DRECHSEL (Jour. f. prakt. Chem., through Chem. News, **39**, 264) states that the *precipitation of calcium carbonate,* whether effected by sodium or ammonium carbonates, in the presence or absence of ammonia or sal-ammoniac, is complete, in the cold, in fifteen minutes, if the mixture be stirred. The solution of the calcium salt should be poured into the carbonate, pouring in a small quantity of the former at first, and stirring for about five minutes before adding more.

E. F. Smith (Proc. Am. Philos. Soc., **18**, 214) has investigated the question of the limits of delicacy in the *detection of iron by means of salicylic acid.* He finds that the coloration is already very faint when a drop or two of water containing $\frac{1}{32,000,000}$ of a gramme of iron is treated with a few drops of alcoholic solution of salicylic acid. A drop of a solution of potassium sulphocyanide, added to one containing $\frac{1}{80,000,000}$ of a gramme of iron, gave a distinct red color. Salicylic acid is, however, when copper is present, a more delicate reagent for iron than potassium sulphocyanide.

George E. Davis (Chem. News, **39**, 221), in an article on *hydrogen peroxide as a reagent in chemical analysis,* calls attention to the great value of that substance in the analysis of soda ash, which often contains so much sodium sulphite as to make it impossible to determine with accuracy, by titration, the percentage of alkali. It is also useful in the analysis of black ash, to oxidize the sulphites, hyposulphites, etc., contained therein. The author employs the *commercial* article, made from barium peroxide and hydrofluoric acid, and examines the purity and oxidizing strength before using. Its keeping properties are greatly enhanced by the addition of a little ether.

C. Rammelsberg (Pogg. Annalen [N. F.], **6**, 157), referring to his previous note (Monatsber. d. Berl. Akad., 1878, 613 ; and this journal, **1**, 106) *on the precipitation of lithia as phosphate,* states that he has found that the proportion of soda retained by the precipitate varies greatly according to the proportion of sodium hydrate present. In one experiment, wherein the sodium hydrate was present in just sufficient quantity to give a distinct alkaline reaction, the precipitate possessed the following composition :

Phosphoric anhydride	60.41
Lithia	37.14
Soda	1.51
	99.06

The method of the author (Pogg. Annalen, **66**, 85) for the separation of the chlorides of sodium and lithium, with a mixture of ether and alcohol (equal volumes), gave very satisfactory results. Sodium and lithium chlorides, mixed in the proportions 5 : 4 (a), 4 : 9 (b), and 1 : 14 (c), yielded, instead of 100 parts NaCl, a = 103, b = 100.8, c = 106.8.

M. MERGET (Soc. Phys. and Nat. Sciences, of Bordeaux, through Chem. News, **39**, 229) recommends paper steeped in ammoniacal solution of silver nitrate or palladious chloride, as a *reagent for mercurial vapors* much more sensitive than gold foil. A sheet of copper plunged into a solution of 1 part mercury in 10,000, remained bright after immersion, but when exposed to the ammoniacal silver paper gave a characteristic black spot. Even solidified mercury emits vapors in quantity appreciable by this test.

E. BOHLIG (Zeitschr. f. anal. Chem., **18**, 195) discusses the question of the *modes of combination of the elements indicated in water analysis.* In a previous communication (idem, **17**, 301) the author has endeavored to prove that the method hitherto pursued is erroneous, for the reason that it is based on the constitution of water that has been boiled, and is, hence, in a decomposed condition. He has proven by numerous experiments, that at temperatures little exceeding 100° C., magnesium sulphate reacts on calcium carbonate, with formation of magnesium carbonate and calcium sulphate. It is, hence, impossible to decide the question whether the magnesium found in the analysis, exists as sulphate, chloride, nitrate or as carbonate, by the operation of boiling the water, as hitherto practiced, except in the case of magnesian waters free from calcium sulphate, and of such waters as the Hunyadi Jànos, wherein the large contents in sodium chloride and sulphate tend to increase the solubility of the calcium carbonate. The author concludes that the magnesium is present only as carbonate in all natural waters, when the quantity is not greater than is necessary to saturate the normal acidity of the water; only that portion in excess of this is combined with the stronger acids.

III. GENERAL ORGANIC ANALYSIS.

I. *Ultimate Organic Analysis.*

E. PFLUEGER (Pflueger's Archiv f. d. gesammte Physiologie, **18**, 117, through Zeitschr. f. anal. Chem., **18**, 296), in conjunction with D. Finkler and F. Oppenheim, has devised a *new method for the organic elementary analysis of nitrogenous substances,* which permits the simultaneous determination of the carbon, hydrogen and nitrogen, in the same portion of substance. The substance is burned in vacuo with a mixture of cupric oxide and potassium pyrochromate. The gaseous products of combustion are transferred, by means of the Geissler's mercury air-pump, to a Bunsen's eudiometer, wherein they are measured gasometrically. The water formed by the combustion is retained by calcium chloride tubes, introduced between the combustion tube and the pump, and is, of course, determined by weight. The method involves numerous precautions and refinements of manipulation, which do not admit of abstraction. The author has found it necessary entirely to discard the use of rubber connections, all the joints of the complicated apparatus being fitted by grinding ; the reason for this is, that he has found that the white rubber tubing, usually employed, while, in fact, impenetrable to gases, nevertheless absorbs carbonic acid with great energy, even when previously thoroughly saturated with stearine. The results obtained by the author are highly satisfactory, and the method, even in its present, somewhat complicated form, promises to be specially valuable when small quantities of extremely rare substances have to be operated on. The author announces his intention to continue his work in the direction of the further simplification of his method, and the increase of its adaptability to general use.

Hugo Schiff (Ann. d. Chem., **195**, 293), in a paper on the *analysis of organic substances containing the halogens or nitrogen*, describes the method of R. Piria (Lezioni di chimica organica, 1857) for the determination of the halogens in organic bodies, which is similar to, but simpler than the method of Feez, Schraube and Burkhardt, described by J. Volhard (idem, **190**, 40). Piria weighs off the substance in a paper cornet, which is pressed into a platinum crucible of about the size of a thimble. The crucible is then filled up with black flux, inverted in a large platinum crucible, and the latter filled up with black flux, so as to completely cover the smaller crucible. The cover is placed on the larger crucible, and it is heated gradually to redness. The decomposition is complete in 8 to 10 minutes. The fusion is then dissolved in water, and the halogens determined by the usual methods. The process cannot well be used with nitrogenous substances, on account of the formation of cyanogen. It is otherwise much more exact than the method with lime. The author has modified this process as follows : The substance, if solid, is weighed directly into a small, platinum crucible, 3 cm high by 2 cm wide, and intimately mixed with a small quantity of the decomposing agent (in the case of chlorine and bromine, a mixture of 1 part anhydrous sodium carbonate with 4 to 5 parts of lime ; in the case of iodine, with dry sodium carbonate alone), with which the crucible is subsequently entirely filled. The crucible is then inverted into a larger one, 4.5 cm high by 3.5 cm wide, and the latter filled up to within 1 cm of the top with the decomposing agent. The latter has the advantage of being a poorer conductor of heat than black flux, and does not induce the formation of cyanides, when nitrogenous substances are treated; the heating takes somewhat more time. Many liquid substances may be analyzed by this method, but not all. The question of volatility is of less importance than that the body should be readily decomposed with the separation of hydrogen acids. Chlorsalicylaldehyde, volatilizing at little over 100° C., gives satisfactory results; brombenzole does not. Piria's method for the ultimate analysis of nitrogenous substances, consists in burning them in a slow stream of oxygen, in a boat placed in a combustion tube filled to three-quarter with cupric oxide, *without* an anterior layer of metallic copper. In the case of nitro-compounds, the substance in the boat is mixed with finely powdered metallic copper. In the case of many nitro-compounds, however, the author finds it impossible to avoid the formation of nitrogen oxides without the anterior layer of metallic copper. Nitrogenous substances of other description than nitro-compounds, may be safely analyzed by this method, provided the layer of cupric oxide is brought to a *full red heat* before the substance commences to volatilize, and is maintained at that temperature during the entire period of combustion. The observations of Lietzenmayer and Staub (Berl. Ber., **11**, 306), that the anterior layer of metallic copper may, through occlusion of hydrogen, lead to serious errors in the hydrogen determination, for the avoidance of which they recommend special precautions, which take up as much time as the combustion itself, have induced the author to call attention to this simpler method, which is capable of giving accurate results with a large class of substances.

II. Proximate Organic Analysis.

A. QUALITATIVE.

H. R. PROCTOR (Chem. News, **39**, 245) communicates a note on the subject of *Weselsky's reaction for phloroglucine* (Jour. Chem. Soc., 1876, **1**, 964). The reaction consists in the production of a cinnabar-red precipitate, when a trace of phloroglucine is added to a solution of nitrate of aniline or toluidine, containing a little nitrite, and the mixture allowed to stand some hours. According to Hlasiwetz, and others, many tannins, when heated with dilute sulphuric acid, yield glucose and peculiar red bodies, which latter, when fused with potash, yield pyrocatechuic acid and another body, in some cases a fatty acid, in others phloroglucine. It occurred to the author that the phloroglucide tannins would react directly with Weselsky's reagent. This supposition was confirmed by experiment; but, as many other substances give the same reaction, the method is liable to lead to mistaken conclusions.

JACQUEMART (Zeitschr. des oesterr. Apotheker-Vereins, 16, 414, through Zeitschr. f. anal. Chem., 18, 291) employs mercuric nitrate as a *reagent for the detection of alcohol.* 5 or 6 cc of the liquid to be tested are decolorized, if necessary, with bone-black, and a small quantity of the reagent added. If ethylic alcohol is present, reduction to mercurous nitrate ensues, and the subsequent addition of ammonia throws down a black precipitate. Methylic alcohol does not produce the reaction.

A. SIENIER and G. LOWE (Polytechn. Notizblatt, 1878, 303, through Zeitschr. f. anal. Chem., 18, 292) employ the reaction of M. W. Iles for boracic acid, as a *test for glycerine.* Borax moistened with the solution to be tested, gives a green flame when held in the Bunsen's burner, if glycerine be present.

E. W. DAVY (idem, 1878, 300, through Zeitschr. f. anal. Chem., 18, 292) recommends, as a *reagent for carbolic acid*, sulphomolybdic acid dissolved in from 10 to 100 parts of sulphuric acid. 3 or 4 drops of the reagent are added to 1 or 2 drops of the liquid to be tested, in a porcelain dish. If carbolic acid be present, a yellow or yellowish-brown color, which passes first to chestnut or red-brown, and then to a handsome purple, is produced. If the carbolic acid is dilute, the color changes are dark olive-green, passing to dark blue. Wood creosote gives a brown or red-brown color, which becomes lighter by standing, or gentle warming. Small additions of carbolic acid to creosote, may be detected by dissolving 5 to 10 drops in 15 cc water, and distilling. The first fraction of the distillate gives the creosote reaction, the following ones the carbolic acid reaction.

A. OGLIALORO (Gazz. Chim. Italiana, 1879, fasc. 3, through Chem. News, 39, 264) communicates several *characteristic reactions of picrotoxine and of certain of its derivatives.* If a small quantity of picrotoxine be dissolved in two drops of nitric acid, of 1.4 sp. gr., and gently heated, there is obtained an amorphous residue, of a reddish-yellow color. If two drops of potassa are added, a fine bright red is obtained, which, on heating, passes to the color of old blood. If 2 cc of a $\frac{1}{2}$ per cent. solution of picric acid, is mixed in the cold with a 50 per cent. potassa solution, nothing is observed but a yellow precipitate. On heating to boiling, the precipitate dissolves, and the liquid is colored orange. On cooling, small prisms of potassium picrate are deposited, and the liquid remains of a reddish-yellow color. If the experiment is repeated, with the addition of picrotoxine, the color of the solution becomes deeper on boiling, and no crystalline deposit takes place on cooling, the liquid remaining intensely colored. If a little picrotoxine be mixed in a capsule with 4 or 5 drops of concentrated sulphuric acid, there appears a golden yellow coloration, which passes into a saffron yellow; the picrotoxine dissolves, and, on adding a little powdered potassium pyrochromate, there is a violet-green coloration, and, on dilution with water, a clear solution of a yellowish-green color.

DR. J. PETRI (Zeitsch. f. anal. Chem., **18**, 211) communicates the results of his experiments on *the spectroscopic detection of ergot in flour.* Pure ergot is extremely easy of identification. When examined under the microscope, it shows the presence of the minute oval granules, usually with two nucleii, which, as stylospores or sporidiæ, form, so to say, the first phase of the generation of the claviceps purpurea. They are of about the size of the smallest granules of rye starch, and come from the external layers of the grain. The medial layers afford the dark-colored particles, which give the ergot powder its blackish-violet color. The central part of the grain contains the oil cells. The fresh powder of secale cornutum possesses a peculiar odor similar to that of trimethylamine. Hot alcohol extracts the greater part of the fat and resin, and also a brownish coloring matter. If, after exhaustion with hot alcohol, the powder be treated with a few drops of dilute sulphuric, on hydrochloric acid, the color changes from dark, greyish violet to a handsome red. The coloring matter is then readily soluble in cold ethylic alcohol, ether, amylic alcohol, benzole and chloroform. The concentrated solution permits the passage of the red rays only; by progressive dilution, first, the yellow, then a part of the green rays, become visible, and, finally, the whole spectrum, with the exception of two absorption bands, which are visible even in very dilute solutions, and permit the certain detection of even slight traces of ergot. The brown solution, obtained by extracting with alcohol, without acidification, shows the same spectrum, but of lower intensity. The red coloring matter appears, therefore, to preexist in the ergot, although the greater part only becomes visible after treatment with acids. The other methods of detection are defective. The appearance of the trimethylamine odor, with or without the addition of potassa solution, is not characteristic, in view of the fact that the substituted ammonium bases are always present in the products of decomposition of gluten. The yellow coloration produced by the action of caustic alkalies, is also unreliable. The author gives the preference to the following modification of Jacoby's method:

About 20 gms of the flour are boiled for 5 minutes with alcohol, and, after subsidence, the yellow solution decanted, the operation being repeated until the alcohol above the powder is colorless; 20 drops of dilute sulphuric acid are then added, the mass well stirred and allowed to subside; the red solution is then filtered and examined in the spectroscope. About one-qaarter test-tube full of the solution is then mixed with 1 to 2 volumes of water, and the milky liquid shaken with 1 to 2 cc amylic alcohol, a second portion with chloroform, a third with benzole, and a fourth with ether. If ergot be present, these solutions will be reddish, and will show the characteristic spectrum. By this method, 0.2 per cent. of ergot may easily be detected in flour, using 20 gms. By means of colorimetric comparisons with solutions of pure ergot, the author has devised a satisfactory method of quantitative determination. The author's measurements of the ergot spectrum agree satisfactorily with those of C. H. Wolf (Zeitschr. f. anal. Chem., **18**, 119; and this Journal, **1**, 104).

F. SELMI (Berl. Ber., **11**, 1,692, through Zeitschr. f. anal. Chem., **18**, 292) communicates the following *reaction for strychnine:* When moistened with a solution of iodic acid in sulphuric acid, it becomes colored, first yellow, then brick-red, and very gradually passes to brilliant violet-red.

B QUANTITATIVE

OTTO HEHNER (Chem. News, **39**, 197) has subjected to a critical examination the *method of Dr. Pavey for the determination of glucose by means of ammoniated Fehling's solution* (Chem. News, **39**, 77; and this Jour., **1**, 109). The author finds that the method gives variable results, except when the proportion of caustic soda in the test solution, is between 120 to 150 gms per liter, when the results obtained accord with those of Dr. Pavey. When this proportion is observed, the method gives excellent results, the end reaction is beautifully sharp, and the titrations of the same liquid never differ more than 0.1 cc of the sugar solution. It must not be taken for granted that the ratio between the ordinary and ammoniated Fehling's solutions, is valid also in the case of other reducing saccharine substances. Dextrose and laevulose act equally, but milk-sugar and maltose do not. The author's investigation on these points is still in progress.

DR. F. W. PAVEY (Chem. News, **39**, 249) has been led by the foregoing article of Hehner, on the subject of the *titration of glucose with ammoniated Fehling's solution,* to make an explanation of the causes of the discrepancies between the results obtained by the last-named observer and himself. These reside in differences in manipulation, which are corrected, as Hehner has found, by the use of a larger proportion of alkali. The author uses nearly double the amount of alkali existing in the usual Fehling's solution, his formula being :

Cupric sulphate	34.65	gms
Potassio-sodium tartrate	173.00	"
Potassium hydrate	160.00	"

Water to 1 liter.

120 cc of this solution are mixed with 300 cc ammonia (sp. gr. 0.880), and water added to 1 liter ; 20 cc of the liquid correspond to 0.010 gm glucose. The author has made the highly important observation that *uric acid* exerts a reducing action on cupric acid, equally as precise as glucose, and finds that its quantitative determination may be easily and speedily made with ammoniated cupric liquid. Three molecules of cupric oxide are reduced by one of uric acid.

F. SOXHLET (Chem. Centralbl., 1878, 218 and 236, through Zeitschr. f. anal. Chem., **18**, 348) has investigated *the quantitative reducing effect of the different kinds of sugar on alkaline copper solutions.* The author finds that the effect cannot be expressed by a definite equivalent proportion, as one part of sugar will reduce very different quantities of copper, according to the strength of the copper solution. The results obtained by titration with Fehling's solution are purely empirical, and have nothing to do with a fixed stœchiometric ratio. The method is, however, correct up to 0.2 per cent., when the same concentration is observed in standardizing the solution and in the analysis proper. The difference between a determination wherein a barely perceptible excess of copper solution is used, and one in which the amount is doubled, may reach 6.5 to 7.5 per cent. He believes, therefore, that a gravimetrical estimation is absolutely impossible.

R. ULBRICHT (Chem. Centralbl., 1878, 392) and M. Maercker (idem, 584, through Zeitschr. f. anal. Chem., **18**, 349) confirm the foregoing observations of Soxhlet. Maercker, however, contests the correctness of Soxhlet's conclusion of the impossibility of a gravimetric determination. The following process is very exact : 34.632 gms of pure cupric sulphate, on the one hand, and 63 gms caustic soda with 173 gms potassic-sodic tartrate, on the other, are each dissolved in 1 liter of water, and preserved separately. The sugar determination is made as follows : 25 cc of each of these solutions are placed in a beaker, the sugar solution (containing not more than 0.12 gm dextrose) is added, and then water to a mark on the beaker indicating 100 cc. The beaker is then allowed to stand in boiling water for 20 minutes. The liquid is then filtered, and the percepitate washed with 300 cc of boiling water. The filter is then burned in a platinum crucible (sic), and the copper reduced to the metallic state by a stream of hydrogen, with the aid of the Rose's crucible cover. The circumstance that the filter paper is liable to absorb some copper, was not found to materially effect the accuracy of the method. The fact that copper, when reduced by hydrogen, retains a certain proportion of that gas, is immaterial, for the reason, that it equally affects the determination of the reduction equivalent. This latter datum was determined by the use of pure dextrose. Proceeding as above, the authors obtained the following results :

0.1111 gm dextrose gave........... 0.196 gm Cu.
0.1000 " " " ....0.182 " "
0.0667 " " " ....0.130 " "

These figures, when transferred to a system of coordinates, give a curve which, calculated by the equation :

$$(y = a + b + bx + cx^2)$$
$$196 = a + 111.1\ b + 111.1^2\ c.$$
$$182 = a + 100.0\ b + 100^2\ c.$$
$$130 = a + 66.7\ b + 66.7^2\ c.$$

gives the constants :

$$a = -\ 19.26$$
$$b = +\ 2.689$$
$$c = -\ 0.006764,$$

from which the following figures are calculated :

MILLEGRAMMES OF REDUCED COPPER.	MILLEGRAMMES OF DEXTROSE.	MILLEGRAMMES OF REDUCED COPPER.	MILLEGRAMMES OF DEXTROSE.
196.0	111.1	152.5	80
194.7	110.0	144.4	75
188.5	105.0	135.8	70
182.0	100.0	127.0	65
175.1	95.0	117.8	60
167.9	90.0	108.2	55
160.4	85.0	98.3	50

The intermediate members of this series may readily be determined by interpolation. The authors find it convenient to arrange these figures on " millemeter paper," and combine them so that the results of the analysis may be read off directly from the curve thus constructed. The accuracy of the method may be seen from the following table of analytical results :

DEXTROSE TAKEN. MILLEGRAMMES.	REDUCED COPPER. MILLEGRAMMES.	DEXTROSE FOUND. MILLEGRAMMES.	DIFFERENCE. MILLEGRAMMES.
50.0	100.0	50.85	+ 0.85
65.0	126.5	64.83	— 0.17
75.0	144.0	74.80	— 0.20
100.0	182.0	100.00	— 0.10
100.0	181.5	99.65	— 0.35
100.0	182.5	100.35	+ 0.35
111.1	197.0	111.90	+ 0.80
111.1	195.8	111.00	— 0.10

The authors have also made several control experiments with starch, which they inverted by the method of Sachsse (2.5 gms with 200 cc water and 20 cc chlorhydric acid), with the following results: The starch contained 78.92 per cent. of dry starch, free from ash. Two determinations yielded the figures 78.90, 78.80, 78.94, 79.00 and 78.95 per cent.

Heinrich (Chem. Centrallbl., 1878, 409, through Zeitschr. f. anal. Chem., **18**, 352) has investigated, anew, the method of Sachsse (Zeitschr. f. anal. Chem., **16**, 121; **17**, 231) for the *determination of dextrose and inverted sugar in the presence of saccharose*, and is led to ascribe the unfavorable results attained by Strohmer and Klaus with this method, to the large amount of alkali contained in Sachsse's reagent. The formula employed by the author directs that 18 gms mercuric iodide, 25 gms potassium iodide, and only 10 gms potassium hydrate, be dissolved to 1 liter. This reagent is without action on saccharose. The mercurial solution is heated to boiling, and the sugar solution run in so gradually as not to interrupt the boiling. The solution must be at a full boil before taking a sample for testing.

M. H. Pellet (Comptes Rendus, **86**, 604, through Bull. Soc. Chim. de Paris, **31**, 94) recommends the following *new carbonated cupric solution for the estimation of glucose*:

Cupric sulphate (pure cryst.)	68.70 gms.
Seignette salt	200.00 “
Ammonium chloride	6.87 “
Sodium carbonate (dry and pure)	100.00 “

The ammonium chloride serves to render the solution more stable. The titre of the liquid is 10 cc for 0.05 gm saccharose, or 0.0526 gm glucose; but it must be verified. The titre is only then exact when 30 cc of the saccharine liquid is taken to 20 cc of the cupric solution. If, for instance, 60 cc of the sugar solution is taken to 20 cc of the copper solution, the titre for sugar becomes 0.0533 for sugar, and 0.0561 for glucose.

E. Frémy (Comptes Rendus, **83**, 1,136, through Zeitschr. f. anal. Chem., **18**, 354) has devised *a general method for the analysis of vegetable tissues.* After extraction with neutral solvents, vegetable tissues consist essentially of the following substances :

1.) *Cellulose*, which name the author, following Payen's example, applies to that cellular substance which dissolves directly in Schweitzer's reagent (ammoniacal solution of cupric oxide), and which forms the bulk of the hair of the seeds of the cotton plant, and the cellular structure of certain fruits.

2.) *Paracellulose*, which dissolves in the copper reagent only after treatment with acids, and which constitutes the cellular substance of certain roots and of the epidermal cells of leaves.

3.) *Metacellulose*, corresponding to Braconnot's *fungine*, which does not dissolve in the copper reagent, and which occurs chiefly in the structure of the fungi and mosses.

4.) *Vasculose*, which forms the chief constitutent of the vessels and canals. It contains less hydrogen and more carbon than the several varieties of cellulose, and is the substance which binds the cells and vessels together. It represents the heavy part of woody tissues, the gritty concretions in the pear, and forms more than one-half of the substance of the shells of wall, and hazel nuts, and the stones of apricots. It is insoluble in bihydrated sulphuric acid (in which all varieties of cellulose dissolve), and also in alkalies, except under pressure. It dissolves readily in oxidizing agents, such as chlorine water, nitric, chromic and permanganic acids, which, before dissolving it, convert it into a resinous acid, soluble in alkalies. The author separates this substance from the cellular substances, by dissolving out the latter with bihydrated sulphuric acid, or, if it is desired to weigh the cellular substances directly, the tissue is treated, in the cold, for several hours, with a mixture of equal volumes of nitric acid and water ; this does not act in a marked degree on the cellular substances, while it converts the vasculose into a yellow resinous acid, which is extracted by potassa solution.

5.) *Cutose ;* this substance forms the fine skin which covers all the external parts of plants. In combination with vasculose, it is what Chevreul has termed *suberine.* It possesses several properties in common with vasculose, including the power of resisting the action of bihydrated sulphuric acid, but differs therefrom in being soluble in dilute caustic and carbonated alkalies, at ordinary pressures. It contains more hydrogen and carbon than vaculose, and differs furthermore therefrom in yielding suberic acid, when treated with nitric acid. In separating cutose from the cellular substancès, Fremy employs, first the copper reagent, then potassic hydrate at ordinary, and later, at higher, pressures. In the first stage, the cellular substances dissolve, in the second, cutose, in the third, vasculose.

6.) *Pectose*, which occurs in the cellular tissue of fruits and roots, is not soluble in water, but is converted by dilute acids into the soluble *pectine*. The quantitative determination is made by digesting the organic tissue in dilute hydrochloric acid. The *pectine* dissolves, and may be reprecipitated by alcohol.

7.) *Calcium pectate* serves to cement the cells together, as in the case of certain trees. It is detected by treating the tissue with dilute acids, when the tissue disintegrates, and the cells are set free. It is determined by treatment of the tissue with cold dilute hydrochloric acid, which dissolves the lime and leaves the pectic acid insoluble. The latter is extracted with dilute potassa, and precipitated out with acids. Nitrogenous substances and inorganic ingredients are determined by the usual methods.

The investigation of a complicated vegetable tissue, such as wood, is conducted as, follows :

1.) Cold dilute hydrochloric acid decomposes the calcium pectate, and the free pectic acid is dissolved out with alkalies.

2.) Hot dilute hydrochloric acid converts the pectose into pectine, which is precipitated with alcohol.

3.) Schweitzer's reagent dissolves the cellulose.

4.) Boiling hydrochloric acid renders the paracellulose soluble in Schweitzer's reagent.

5.) Bihydrated sulphuric acid dissolves the collective cellular substances.

6.) Hot dilute potassa dissolves the cutose.

7.) Potassa, under pressure, dissolves the vasculose.

8.) Dilute nitric acid renders the vasculose soluble in alkalies.

W. Meyeringh (Berl. Ber., **10**, 1,940, through Zeitschr. f. anal. Chem., **18**, 345) has discovered that *the volumetric estimation of hydroxylamine* may be effected by adding to the solution of the base, a sufficient quantity of magnesia or sodium phosphate, and a slight excess of iodine solution, and titrating the excess of iodine with hyposulphite solution. The reaction, involved, finds its expression in the equation :

$$2NH_3O + 2I_2 = N_2O + H_2O + 4HI.$$

The determination may also be accurately made with ferric sulphate and permanganate, if the ferric salt be made to act on the base at a temperature of 80–90°. The reaction is then :

$$2Fe_2(SO_4)_3 + 2NH_3O, HCl = 4FeSO_4 + 2H_2SO_4 + N_2O + H_2O + HCl.$$

The determination may also be made with Fehling's solution, according to the equation :

$$3NH_3O + 4CuO = N_2O + 2Cu_2O + 3H_2O.$$

IV. PHYSIOLOGICAL AND PATHOLOGICAL ANALYSIS.

E. W. **Hamburg** (Zeitschr. f. physiol. Chem., 2, 197, through Zeitschr. f. anal. Chem., 18, 363) employs the following *method for the quantitative determination of iron in organic substances, such as urine, flesh, faeces, etc.* The substance is evaporated to dryness, and completely carbonized in a platinum dish. The coal is warmed on the water bath with fuming hydrochloric acid, to decompose any iron silicate which may have formed. The coal is then washed with hot water to the disappearance of the acid reaction, treated with a little dilute sulphuric acid, to decompose any ferric chloride it may retain, dried and incinerated. The ash is added to the acid solution, and the whole, after adding sulphuric acid, is evaporated on the water bath, and finally heated, to expel the excess of sulphuric acid. The residue is ignited, to destroy any organic substance that may still be present, and boiled out with a mixture of 8 parts sulphuric acid and 1 of water (A. Mitscherlich, Journ. f. prakt. Chem., 81, 110), to dissolve the ferric oxide. The ash of urine and flesh gives a sufficiently clear solution, that of faeces contains so much sulphate of lime that filtration is necessary. The solution is placed in a two-necked ½ liter flask, and the ferric reduced to ferrous oxide, by boiling with sulphurous acid, while a brisk current of carbonic anhydride is passed through the flask. The tubes for this purpose are ground into the necks, as the sulphurous acid attacks rubber stoppers, and impurity is thus introduced. To test the complete expulsion of the excess of sulphurous gas, the carbonic anhydride is conducted from the flask through a dilute permanganate solution. When the color of the latter remains unchanged for 20 minutes, the operation is at an end. The carbonic acid is made from marble and hydrochloric acid, and is washed first with cupric sulphate, and then with sodium carbonate solutions. The solution is allowed to cool in the stream of carbonic acid, and then titrated with permanganate solution. In the case of urine, the latter has a strength of 0.5 gm in 2 liters; in the case of faeces, it may be thrice as strong. The titre is fixed with pure iron wire (0.4 per cent. being allowed for carbon), dissolved in pure sulphuric acid, and reduced with sulphurous acid as above. The end of the titration is indicated by a *permanent*, weak, rose-red coloration, changes being noted by comparison with an equal volume of water colored with a drop or two of the permanganate solution. The hydrochloric acid used is obtained free from iron by Otto's method. 10 parts concentrated acid are gently warmed with 6 parts calcium chloride, and the washed gas absorbed in water; by long keeping in glass the acid takes up iron. The sulphuric acid was freed from iron by distillation. The experiments of the author show the method to be very exact.

PAUL FUERBRINGER (Berliner klin Wochenschr., 1878, No. 7, through Zeitschr. f. anal. Chem., **18**, 366) contributes very important observations on *the question whether albumen in acid urine is necessarily precipitated by boiling*. Nearly 30 years ago Bence Jones remarked that albumen, in strongly acid urine, does not always coagulate on boiling: The author cites three cases of this nature which have come under his observation. The *first* was that of a young girl suffering from chlorosis, whose urine was pale, perfectly clear and distinctly acid. It remained perfectly clear on boiling. After the addition of acetic acid, it gave a considerable, flaky precipitate. Addition of a few drops of nitric acid, produced only a slight, hardly flaky, precipitate. The *second* case was that of a male adult, with pronounced nephritic symptoms, whose urine was turbid from granulated cylinders, circular cells, and detritus. Color, reddish-yellow; strongly acid; without abnormal odor; on boiling, a slight turbidity, turning flaky on adding a little nitric acid, but hardly increased thereby, while addition of excess of nitric acid produced a considerable precipitation. The same quantity of albumen precipitated at once, in flakes, on adding a few drops of dilute acetic acid, but dissolved rapidly when an excess of the latter was added. The *third* case was that of a man of forty-six, whose urine presented the following characteristics: Without unusual odor; color, yellowish-red (No. 5 on Vogel's scale); sp. gr., 1.018 (normal); slightly cloudy; reaction, moderately acid. No separation of the large amount of albumen it contained, on boiling, or even after slight acidification with nitric acid, although it came down on the addition of a large excess of the latter, or of the slightest quantity of acetic acid. The special reactions of the filtrate were as follows (each portion taken measured

20 cc ; the acetic acid was the officinal acid. acet. dilut. ; the nitric acid, acid. nitric. pur.) :

Simple boiling: Slight opalescence, no definite precipitate.

1 *drop acetic acid:* The turbidity changes to a very considerable flaky precipitate (at least 1 per cent. albumen).

Excess of acetic acid (1 cc): The turbidity completely dissolved, no new precipitation.

1 *drop nitric acid:* The opalescence changed to solitary, slight, hardly noticeable flakes.

Excess of nitric acid (1 cc) : The same precipitation as above.

When the urine is treated *in the cold,* with the above reagents, in the same quantities, and afterwards heated to boiling, the same effects are produced, only the urine treated with one drop of nitric acid, remains clear while boiling, and only on cooling becomes turbid from minute, hardly recognizable, feathery coagula. The addition of one drop of nitric acid to the cold urine, produces no effect. A strong milky coagulation or a broad ring on the sides of the test-tube, is produced when a large excess of the acid is added or poured in a layer on the surface of the sample. The addition of a layer of alcohol produces a thin, sharp ring on the plane of contact of the two layers (after precipitation of the earthy phosphates with soda, and titration back to the original acidity with hydrochloric acid). From these reactions, the author is led to assume the presence of two albuminoids, or, at least, two modifications of the albumen group, viz. : serum-albumen, containing urinary salts, which is present in the traces precipitated by heating and by alcohol ; while the other, which is very closely related to the former, is distinguished by *solubility in alcohol,* and by the fact that it is *not precipitable from the acid urine by boiling.* These two varieties were easily separated, by adding a few drops of nitric acid to the boiling urine, and filtration. The filtrate contained the second variety. If the nitric acid in the filtrate be neutralized with alkali, to the restoration of the original degree of acidity, even long-continued boiling produces no trace of a precipitate, while the additions of larger quantities of nitric acid, whether in the heat or in the cold, produce more and more precipitate, which does not redissolve even in the largest quantity of this acid. A single drop of acetic acid sufficed to precipitate from 20 to 30 cc of the boiling filtrate, almost the same amount of albumen that was observed in the original urine. Alcohol produced absolutely no precipitate, only a turbidity due to the earthy phosphates. In the cold, when treated with acetic acid and potassium ferrocyanide or sodium sulphate, the filtrate showed the same reaction on boiling as does ordinary albuminous urine, and comported itself similarly under polarized light and in the color reactions (HCl and Fehling's solution). The microscopic examination of the sediment from the urine, showed the ordinary ingredients of the nubecula, besides a few pale hyaline cylinders, which established the existence of a renal albuminurea. The author points out that the opinion of Bence Jones, that this peculiar substance is a compound of serum-albumen with hydrochloric or nitric acids, cannot apply to the material found in the third case above cited, wherein the albumen was not coagulated by addition of an excess of alcohol. On the other hand, the author notes the marked similarity of this substance to serum-albumen free from salts, which is not precipitable either by boiling or by addition of alcohol, although it is difficult to understand how an albumen free from salts, could be present in a strong saline solution, such as urine. That Edelfsen's paraglobuline was was not present, was easily demonstrated by Senator's test (dilution to sp. g. 1.002, and passage of CO_2). The same was found to be true of the other hitherto observed albuminoids. The conclusions to be drawn from the foregoing observations are, that even *acid* urine must be treated with acid, after boiling, even when no turbidity is produced. They show, anew, the danger of the least excess of acetic acid, even when added to urine that has already been boiled, as well as the disadvantage of using too little nitric acid. Finally, they show that the quantitative method of Liborius, of determining albumen by precipitation with alcohol, may give very erroneous results.

P. CAZENEUVE (Comptes Rendus, **88**, 595) in a note on *the determination of glucose in blood*, formulates the following objections to the method of analysis pursued by Claude Bernard :

1.) The manipulation is defective and incomplete.

2.) The limit of the reduction with cupric-potassic solution is difficult to fix with precision.

3.) The cupric-potassic solution is reduced by other substances present in the blood.

The auther has improved the process by washing the coagulum obtained with sodium sulphate, crystallizing out the latter by concentration, reducing the liquor to a fixed volume, and decolorizing with acetate of lead, before titration with Fehling's solution. 500 cc blood from the femoral artery of a dog, treated by this method, and evaporated to 100 cc, yielded, with Fehling's solution, 3.00 gms glucose per liter, while in the polarimeter of Laurent, it showed but 2.38 gms per liter. A second experiment, made on the serum of human blood, showed 1.58 gm per 1,000 with the cupric-potassic solution, and 1.12 gm per 1,000 in the polarimeter. These latter results harmonize with those of Musculus and Mering (idem, **83**, 87), who also found in blood, substances, other than glucose, which reduced the Fehling's solution.

D'ARSONVAL (idem, **88**, 753) and P. PICARD (idem, **88**, 755) take exception to the foregoing views of Cazeneuve, and while admitting the existence of reducing substances, other than glucose, in blood, contend that the method of Bernard is more exact than the polariscopic method, and sufficient for all physiological purposes. These remarks have drawn out a reply from P. Cazeneuve (idem, **88**, 864), wherein he maintains the correctness of his first conclusions.

V. SPECIAL METHODS OF TECHNICAL ANALYSIS.

S. W. JOHNSON and E. H. JENKINS (Report Conn. Agr. Expt. Station, 1878, 111, and Am. Chem. Jour., **1**, 77) have made *the determination of nitrogen in the analysis of agricultural products* the subject of careful investigation, and have attained results of great value and interest to all chemists working in this field. They have demonstrated that the method of combustion with soda-lime, when properly conducted, gives results closely accordant with those obtained by the *absolute* method, the material experimented on being egg-albumen, maize-fibrine, fish scrap and peruvian guano, and the maximal and minimal differences between the two methods, in a series of 24 consecutive analyses, being respectively 0.17 p. c. and zero. The authors recommend the following extremely simple and convenient method of preparing soda-lime: Equal weights of sal-soda, in clean (washed) large crystals, and of good white and promptly slacking quick-lime, are separately pulverized, so as to pass a sieve with holes of $\frac{1}{16}$ inch, well mixed and heated in an iron pot, which should not be more than half full, gently, and at first without stirring. The lime soon begins to combine with the crystal water of the soda, the mass heats strongly, swells up, and yields, in a short time, a fine powder, which may then be stirred to effect intimate admixture, and dry off the excess of moisture, so that the mass may not be perceptibly moist, yet short of the point at which it rises in dust on handling. When cold, it is bottled for use. In regard to the conduct of the soda-lime method, the following rules are given:

1.) Contrary to what is commonly stated, it is not necessary that the substance analyzed should be finer than to pass through a sieve of 1 mm mesh.

2.) Neither the highest heat of the Erlemeyer combustion furnace, nor a long layer of strongly heated soda-lime, nor these two causes combined, occasion any appreciable dissociation of the ammonia formed by combustion.

3.) The use of pure sugar or of oxalic acid, as a diluent, does not in any way effect the result.

4.) Iron tubes of proper length (22 inches) may be substituted for glass. The results are as satisfactory, but more time is required.

5.) To get a good result, the anterior layer of soda-lime must be of sufficient length. With 0.5 gm of substances containing less than 8 per cent. of nitrogen, a glass tube 12 to 14 inches long is sufficient. As the contents of nitrogen increases to 10 per cent., the tube should be several inches longer. In the combustion of dried blood or egg-albumen, the tube should have a length of 25 to 30 inches, less than half of which should be filled with the mixture of substance and soda-lime, a layer of pure soda-lime 12 or more inches in length being essential for the perfect destruction of volatile organic matters.

6.) The long anterior layer of pure soda-lime must be brought to a full red heat before heating the mixture, and must be so kept during the combustion.

7.) No fumes or tarry matters, indicative of incomplete combustion, should appear in the bulb-tube, or receiver.

8.) When the combustion, proper, is begun, under the conditions above described, it can be carried on quite rapidly until completed, without the contents of the tube then showing any sign of unburned carbon.

9.) The mixture may be made either intimately in a mortar, by stirring with a spatula, or in the tube with a mixing wire.

10.) The authors allow the tubes to cool somewhat, before passing air through them to sweep out the ammonia.

11.) The products of combustion are passed through standard hydrochloric acid in a bulb-tube or flask, and the acid afterwards titrated with standard ammonia and tincture of cochineal.

S. W. JOHNSON and E. H. JENKINS (Report Conn. Agr. Expt. Station, 1878, 117, and Am. Chem. Journ., **I**, 84) communicate *a rapid and exact method for the determination of phosphoric acid,* based upon Mayer's modification of Otto's method (Ann. d. Chem., **101**, 164) of precipitation as ammonium-magnesium phosphate, and F. Stolba's method for the determination of ammonium-magnesium phosphate by titration (Zeitschr. f. anal. Chem., 16, 100). The authors employ a nearly saturated solution of ammonium tartrate, free from carbonic acid, and a solution prepared by dissolving 70 gms magnesium sulphate and 195 gms ammonium chloride, in 1 liter of water ; 10 cc of this last solution contain twice the amount of magnesium necessary to precipitate 0.1 gm phosphoric anhydride. The operation is conducted as follows : 1 gm of the phosphate is dissolved in hydrochloric acid, nearly neutralized with ammonia, and ammonium tartrate added, 10 cc at a time, until the solution remains perfectly clear when slightly alkaline. Add then a suitable quantity of the magnesium mixture, and stir or shake vigorously. When the precipitation is nearly complete, add ammonia to strong alkalinity, and let stand 6 to 12 hours. Filter, preferably on the pump, and wash with equal parts of 85 to 90 per cent. alcohol and water. The precipitate adhering to the glass, need not be detached. The washing of the vessel and precipitate is continued until the liquid runs off neutral. The precipitate and filter are placed in the precipitating vessel, a *little* water and a few drops of cochineal tincture added, and then titrated by adding, first, an excess of standard acid, and then titrating back with standard alkali. The authors have subjected their method to an extensive series of comparative trials, side by side with the molybdic method, and have obtained results which amply justify their conclusion, that the former method is hardly less accurate than the latter, while it requires less than half the time and labor, and appears to be generally applicable. They find that while ammonium-magnesium phosphate is totally insoluble in a very large excess of ammonium tartrate, it is soluble in excess of ammonium citrate and, when iron and aluminum are present, also in *insufficient* ammonium tartrate.

E. F. Teschemacher and J. D. Smith have published a pamphlet (London : Hardwicke & Bogue, 1879), on *the estimation of phosphoric acid by magnesia, for commercial purposes,* which is largely devoted to a discussion of the causes of the discrepancies which frequently occur in the results obtained by different chemists on identical samples of commercial phosphates. One of the most frequent causes is neglect of proper precautions in the determination of moisture. As the authors are analytical chemists in large practice, in England, a detailed description of the method employed by them may not be without interest for American readers. The authors recommend that the bulk-sample be brought as quickly as possible to the crusher, and a coarse sample of some 3 lbs. be taken and at once put up in a tight vessel for a "moisture-sample." The remainder of the bulk-sample is thoroughly ground for a "strength-sample," the object of taking two samples being to avoid the loss of moisture in grinding. The moisture having been determined in the "moisture-sample," the "strength-sample" is passed through a sieve of 224 meshes to the square inch, and thoroughly mixed. Four portions of 250 grains are taken, two of which serve for the determination of moisture at 100° C.; the remaining two are placed in the hands of separate analysts, who proceed as follows: The 250 grs are mixed in a covered porcelain basin with 1,000 fl. grs of hydrochloric acid, sp. gr. 1.165, and gently boiled for 20 minutes, to dissolve the phosphate, and expel the fluorine which may be present. The contents of the dish are placed in a 5,000 grain measure, when cool, and water added to the 5,000 gr mark. After mixing, the liquid is filtered and the insoluble matter washed, dried at 100° C., and weighed. The washings are not added to the solution. 1,000 fl. grs of the solution are measured into a flask, and boiled with 250 fl. grs of solution of ammonium sulphite, to reduce the ferric to ferrous oxide. The liquid is then transferred to a basin containing 80 grs oxalic and 100 grs citric acids, previously dissolved in 250 fl. grs ordinary acetic acid. In this, the precipitation of the oxalate of lime is completed, by adding *very slowly* and in a fine stream, about 1,000 fl. grs of "ammonia water" (1 of ammonia, sp. gr. 0.880, to 20 of water), and then rendered alkaline with stronger ammonia, added in the same gradual manner, the solution being just boiling throughout. Excess of acetic acid is now added, the solution filtered, and the precipitated oxalate of lime washed with boiling water, dried and weighed. It is rarely entirely free from phosphoric acid, hence the authors recommend that it should be converted into carbonate by ignition, dissolved, the phosphate of lime precipitated by ammonia, filtered off, redissolved in hydrochloric acid, and the lime removed by the foregoing method, using 3 grs of oxalic and citric acids; the filtrate is added to that from the first precipitation.

To the acetic acid filtrates and washings, $\frac{1}{10}$ volume of ammonia, sp. gr. 0.880, is added; and then, gradually, about 350 fl. grs of magnesium solution (200 grs magnesium chloride and 100 grs ammonium chloride, in 1,000 fl. grs water), with constant stirring. Should the precipitate not immediately appear on addition of the magnesium solution, but only commence to form when the whole 350 fl. grs have beer gradually added, the mixture must be set aside for 12 hours before filtering; if, on the other hand, it commences to form at once, it may be collected after the lapse of one hour. The precipitate is washed with "ammonia water," redissolved with chlorhydric acid, and reprecipitated by adding 50 grs citric acid, 30 fl. grs magnesium solution, and first, slowly, dilute ammonia, and then $\frac{1}{10}$ volume of strong ammonia, as above described. In about an hour, the precipitate is transferred to a filter, washed with "ammonia water," dried, introduced into a porcelain crucible, and heated, with gradually increasing temperature, for about 40 minutes; then subjected for about 4 minutes to a bright red heat, cooled, and the pyrophosphate thus formed, weighed. The results of the analysis of the "strength-sample" are then adjusted, by calculation, to the percentage of moisture found in the "moisture-sample."

H. Albert and L. Siegfried (Zeitschr. f. anal. Chem., **18**, 220) have subjected to the test of further experiment, their *method of determining the reverted phosphoric acid in superphosphates*, by means of ammonium tartrate (Zeitschr. f. anal. Chem., **16**, 182), and have attained the conviction that this method is inferior to the methods of Fresenius, Neubauer and Luck, with ammonium citrate.

B. E. Dietzell and M. G. Cressner (Zeitschr. f. anal. Chem., **18**, 225) have investigated the subject of *the determination of phosphoric acid in fish guano*. The authors note that the percentage of phosphoric acid yielded by the ash, or the solution of the guano in acids, is considerably lower than that obtained by fusing the guano with sodium carbonate and potassium nitrate and chlorate. They conclude from this, and other reasons, that there is present in this substance a considerable proportion of organic phospho-compounds (nuclein ?), in which the phosphorus can hardly have the same fertilizing value as that in the form of phosphate, for the reason that it is not converted into phosphoric acid even by such energetic oxidizing agents as nitric acid. The authors are, therefore, opposed to the determination of the phosphorus by the method of fusing with oxidizing agents, and believe the direct determination in the acid solution of the guano, or in the liquid obtained by the evaporation of the ash with concentrated nitric acid, is the only one which can be accepted as a satisfactory basis for commercial valuation.

Robert R. Tatlock (Chem. News, **39**, 268) has investigated, anew, the question of *the determination of the nitric nitrogen in guano*. The author calls attention to the fact, that it is not uncommon for natural guanos to contain as much as 1 to 2 per cent. of nitrates, and that the error due to the partial or imperfect decomposition of the latter by combustion with soda-lime, is often considerable. While pure nitre does not evolve ammonia, with soda-lime, the presence of organic substances causes this transformation to take place, to a greater or less extent, proportional to the amount of organic matter present, but incomplete even when the ratio of organic substance (starch) to the nitre is 30 to 1. The practice, followed by many chemists of large experience, of adding the amount of nitric nitrogen, found by direct determination, to the nitrogen obtained by combustion, has, therefore, the consequence that the purchaser pays for a large proportion, or even the whole of the nitric nitrogen, twice over. Of the different methods which may be used for the determination of the nitric nitrogen, the author gives the preference to that of Crum (Proc. Phil. Soc. Glasgow, 1848, 162). The author's investigation is still in progress.

Note: It may not be out of place, in this connection, to call attention to the results obtained by Grete (Berl. Ber., **11**, 557, and this Journal, **1**, 100) by adding the xanthogenates to the mixture of soda-lime and substance. According to this author, the reduction of potassium nitrate, under these conditions, is so perfect, that the whole of the nitrogen was obtained as ammonia.

BEAUMONT J. GROSJEAN (idem, **39**, 281), in relation to the foregoing article, calls attention to his previous communication (idem, **25**, 205) on the same subject, wherein he recommends the distillation of the nitre, with iron filings, and caustic alkaline solutions, to a pasty state, in a retort; the residue solidifies on cooling, and is then powdered and burned with soda-lime.

A. REMONT (Bull. Soc. Chim. de Paris, **31**, 242) states that *the determination of the total nitrogen in manures containing nitrates*, may be satisfactorily made by adding to the substance, before mixing with soda-lime, ten parts of powdered sugar to each part of nitrate which may be present.

ANTON PAVEC (Listy Chemické, **1**, 213, through Zeitschr. f. anal. Chem., **18**, 360) has investigated *the volumetric determination of phosphoric acid in superphosphates and bone-charcoal, by means of uranium solution.* The results obtained by him were always too low when the solution was standardized with sodium phosphate. Proceeding from the fundamental rule in volumetric analysis, that "the titre of the test solution must always be determined under the same circumstances that the analysis itself is made," the author fixed the titre of his uranium solution by comparison with a superphosphate, the percentage of phosphoric acid in which had been determined gravimetrically. The results obtained with other superphosphates were then as closely accordant with the gravimetric results, as could be desired, the difference never exceeding 0.2 per cent. In the same manner, the titre of the solution used in the analysis of bone-black, must be determined by comparison with that substance. The author calls attention to the importance of properly preparing the ferrocyanide solution, used as an indicator. When the crystals are superficially effloresced, the reaction is never sharp. The author takes a large crystal, washes off the superficial layers, and dissolves the inner part of the crystal for use. The solution must be freshly made each day, and must not be allowed to stand in the sun. The uranium solution must also be carefully preserved from direct sunlight, even when in the burette, and should be kept in a dark place. The titre must be redetermined after 2 to 3 months.

R. HORNBERGER (Landwirthschaftl. Versuchsstationen, Bd. 20, through Zeitschr. f. anal. Chem., **18**, 361) communicates his experience in *the determination of the alkalies in the ashes of plants.* The method usually followed, is based on the separation of all the other ingredients, the weighing of the alkalies as chlorides, and the separation of potassium with platinic chloride. The method of Wolf consists in the precipitation of the sulphuric acid with barium chloride, digestion, at a gentle heat, with ammonia, ammonium carbonate and some ammonium oxalate, filtration, evaporation of the filtrate, and expulsion of the ammonium salts by ignition. The residue is moistened with oxalic acid, evaporated to dryness, and ignited, this operation being repeated once. The mass is then boiled out with hot water, the filtrate acidulated with hydrochloric acid, evaporated, ignited, and the alkaline chlorides weighed. From the experiments of the author, this method is liable to the objection that a partial double decomposition of the calcium phosphate and ammonium earbonate ensues, so that ammonium phosphate passes into the filtrate ; the amount of phosphoric acid which thus passes into the akalies, being sometimes as great as 20 per cent. of the whole amount of the former, and is in direct proportion to the excess of ammonium carbonate present. Nearly the whole of this phosphoric acid is weighed with the alkaline chlorides. The author, therefore, recommends that the phosphate of lime be first precipitated with ammonia, filtered off, ammonium carbonate and oxalate added to the filtrate, etc., as above. If sufficient lime be present, the error above noted is thus entirely avoided.

H. BERING (Corr. Blatt des Vereins Analyt. Chemiker, **2**, No. 7, April 1, 1879) effects *the determination of the solids in milk,* by weighing off 0.09 gm of calcined magnesia in a platinum capsule, and then introducing carefully, without touching the sides, an accurately weighed portion of the milk (1 to 2 gms). On evaporation over a small open gas flame, placed about 40 cms below the capsule, the sample is obtained perfectly dry in 2 to 3 hours. The dry mass is very feebly hygroscopic.

F. FISHER (Hannov. Monatsschr. Wieder Nahrungs-Faelscher, **2**, No. 2, Feb., 1879, through Chem. News, **39**, 265) states that *the examination of butter by polarized light*, under a magnifying power of 200 to 300 diameters, affords a much more certain criterion of purity, than the determination of its specific gravity. Fictitious butter shows not only the globular drops and salt crystals peculiar to genuine butter, but also more or less perfectly developed crystals. The author also finds that the different fats show characteristic colors in polarized light. Mutton tallow always gives a blue tone. Cocoa butter shows colors passing from the deepest red to the brightest green. The fat of oxen shows merely green and white luminous effects; small semilunar and vermicular bodies of a bright green color, appear in common light. Hogs' lard displays many colors, especially red and blue, yellow, which is very conspicuous in cocoa butter, being wanting. These optical reactions are available for the detection of foreign fats fraudulently added to chocolate or cocoa.

F. SOXHLET (Dingler's Journ., **232**, 461) recommends that *the gravimetrical determination of the fat in milk* should be made as follows: 10 cc of milk, measured and weighed, are intimately mixed with 20 gms burned gypsum, in a 100 cc platinum dish. The mass is dried on the water bath, with occasional stirring, and breaking of the lumps with a spatula; in about 30 minutes it is sufficiently dry for extraction with ether. The last-named operation is very expeditiously conducted in a special apparatus devised by the author, a description of which could not well be made intelligible without the drawing. The etherial solution of the fat is evaporated, and the weight of the latter determined. The whole determination may be finished in less than two hours.

H. GEISSLER (Dingler's Journ., **231**, 56) describes *a method for the determination of water in milk.* The milk is placed in a burette, which is connected with a retort-shaped vessel, which, in its turn, is connected air-tight with a receiver in the shape of a graduated tube. A few drops of water are introduced into the retort and receiver, and converted into steam, so as to expel the air. The receiver is then plunged into a vessel of water, the retort is placed in a water bath, and the milk introduced drop by drop from the burette. The distillation proceeds very rapidly in the partial vacuum in the apparatus. The volume of the distillate is measured in the graduated receiver.

J. KOETTSTORFER (Zeitschr. f. anal. Chem., **18**, 199) communicates *a new method of testing butter for foreign fatty bodies.* Investigations made by the method of Hehner (idem, **16**, 145, and **17**, 151, 287), show that butter contains a much larger quantity of fatty acids of the series below C_{10} than was previously supposed. As most of the fatty bodies contain only the higher fatty acids, and as the lower fatty acids possess a smaller molecular weight than the former, butter must contain a larger number of molecules of acids than the same weight of other fats, and the difference in the number of molecules must be considerable. The author has, thus, been led to devise a method, based upon the determination, by titration, of the quantity of potassium hydrate necessary to the complete saponification of the sample of butter, and comparison with the quantity required by other fatty substances. The author employs solutions of ½ normal hydrochloric acid, and a solution, of the same strength, of potassium hydrate, in highly rectified alcohol. 1 or 2 gms of the fat, previously purified by melting and filtration, are placed in a high beaker holding about 70 cc; 25 cc titrated alcoholic solution of potassa are added, and the mixture warmed on the water bath, with constant stirring, until solution is effected. The glass rod is washed off with alcohol and removed, the beaker covered with a watch glass, and warmed for 15 minutes. The watch glass is then washed with alcohol and removed, the glass rod introduced, and the liquid stirred therewith, to saponify any adhering fatty acids. The beaker is removed from the water bath, 1 cc of alcoholic solution of phenole-phtalein is added, as an indicator, and the contents titrated with ½ normal hydric chloride. The end of the titration is sharply defined, the color changing to a pure yellow when incipient acidity is attained. The results of the analysis are expressed in the weights of KHO required to saponify 1,000 parts of the fat. The rèsults of the analysis of 13 samples of butter, from the neighborhood of Fiume, showed the quantity of KHO required to saponify 1 gm of butter, to vary from 221.5 to 232.4 mgms. The materials usually employed as adulterants, contain stearine, olein and palmitine, which, according to theory, require the following quantities of KHO :

1 gm stearine	188.8	mgm KHO
1 " olein	190.0	" "
1 " palmitine	208.0	" "

The following experimental results were obtained:

1	gm	tallow	196.5	mgm	KHO
1	"	" commercial	196.8	"	"
1	"	kidney fat of swine	195.8	"	"
1	"	bacon fat	195.7	"	"
1	"	lard (said to be American)	195.4	"	"
1	"	suet	197.0	"	"
1	"	olive oil	191.8	"	"
1	"	rape-seed oil	178.7	"	"

A sample of a mixture of 50 kilos oleomargarine and 1 liter milk, containing, therefore, 1 per cent. butter, required for 1 gm, 195.8 mgms KHO.

It appears from the foregoing, that the number 195.5 may be accepted as the *titre* of the fats generally employed as adulterants of butter, and that a butter may be assumed to be genuine, which requires as much as, or more than, 221.5 mgms of KHO. In calculating the percentage of adulteration, it is more convenient to take the mean of the foregoing determinations, or 227 as the titre of pure butter. If x represents the percentage of adulteration, and u the quantity of KHO, expressed in milligrammes, we have

$$(227-195.5) : (227-u) = 100 : x$$

and

$$x = (227-u)\ 3.17$$

In the case of a mixture of butter and lard, of which 1 gm required 214.1 mgms KHO, if we take the lowest figure for pure butter, viz.: 221.5, x will equal 29 per cent.; if we take the highest, viz.: 232.4, x will equal 50 per cent., and if we take the mean, 227, x will equal 40 per cent. The mean, hence, involves a possible error of 10 per cent. In the most unfavorable case, 29 per cent. of adulteration might escape detection. Hehner's method involves a possible error of 40 per cent. The author reserves the right to investigate the application of his method to the discrimination of other fats and oils possessing different alkalimetric titres.

J. DAVID (Comptes Rendus, **86**, 1,416, through Dingler's Journ., **231**, 64) has devised the following method for *the quantitative separation of oleic acid from stearic acid.* The method rests on the fact, that if acetic acid be added, drop by drop, to a solution of oleic acid in alcohol, a point is attained when the oleic acid suddenly separates on the addition of even as little as 0.1 cc of acetic acid. Stearic acid, on the contrary, commences to separate when the first drop is added. The author makes, first, a mixture of 1 liter acetic acid and 1 liter water ; he then places in a cylinder, graduated into $\frac{1}{10}$ cc, first 1 cc pure oleic acid, then 3 cc 95 per cent. alcohol, and finally, 2.2 cc of the dilute acetic acid. Up to this point no precipitation should take place, but an addition of even 0.1 cc more of the acid, should separate the whole of the 1 cc oleic acid, which then rises to the surface. If this be not the case, the proportions must be varied until the precipitation is induced by an addition of 0.1 cc of the dilute acetic acid. Alcohol and acetic acid are then mixed, according to the indications of this test, e. g., 300 of alcohol and 200 of acetic acid. A weighed portion of the mixture of fatty acids is taken, finely divided, and placed in a sufficient quantity of the mixture, in a stoppered flask, well shaken, and allowed to stand 24 hours in a cellar, at 15° C. The oleic acid is then entirely dissolved. The residue of stearic acid is dried in the the water bath, and weighed.

Max Buchner (Dingler's Journ., **231**, 272) calls attention to the frequency of *the adulteration of beeswax.* The materials hitherto employed have been tallow, rosin, or stearic acid. A new adulterant is *cerasine*, a mixture of refined ozocerite with carnuaba wax. One means of detecting this last-named adulterant consists in the determination of the specific gravity. The author has made the following determinations :

Yellow cerasine, from Stockerau	0.876
White " " "	0.898
Yellow, hard " " Frankfort a. O	0.901
White " " " "	0.891
White, soft " " "	0.883
Yellowish (not colored) cerasine, from Gratz	0.886
White " "	0.859
White " "	0.858
Semi-refined ozocerite, brown	0.888
Yellow ozocerite, entirely genuine	0.959
White " " "	0.955
Adulterated yellow wax, from Croatia	0.937
Carnuaba wax	0.999

R. v. Wagner recommends to place the wax in alcohol of 0.945 to 0.950 sp. gr., in which, if genuine, it will sink ; while wax containing cerasine will float. For a chemical test, the wax may be boiled with a solution of 1 part of potassium hydrate, in $2\frac{1}{2}$ to 3 parts of 90 per cent. alcohol. If the mixture be kept some time in the water-bath, to prevent solidification, it will remain clear, if genuine ; while, if cerasine be present, an oily layer will form on top of the alkaline solution.

JOSEF M. EDER (Dingler's Journ., **231**, 445 and 526) contributes a very interesting article on *the characteristics of Chinese tea, and methods of detection of adulterations.* The substances with which teas may be adulterated, may be divided into four groups :

1.) Mineral substances, to increase the weight.

2.) Mineral coloring matters.

3.) Organic substances, to increase the weight ; chiefly the leaves of other plants.

4.) Organic coloring matters and astringent substances, to conceal the loss of extractive matter in *spent* tea leaves.

Adulterations of the third and fourth class are the most common : the latter especially so. Owing to the variable percentage of thein in natural tea, the determination of this substance cannot be employed as a criterion of purity. The author's mode of investigation involves the determination of : 1. The extractive matters with boiling water. 2. The percentage of tannin in the decoction. 3. The percentage of ash. 4. The percentage of ash which is soluble in water. The tannin is determined, by the method of the author (idem, **229**, 83), with cupric acetate. The following table contains the average of numerous determinations by the author :

VARIETY.		TANNIN.	EXTRACTIVE.	TOTAL ASH.	ASH SOLUBLE IN WATER.
		Per cent.	Per cent.	Per cent.	Per cent.
Black Tea.	Souchong and Pouchong	9.18	38.3	5.88	2.85
	Congo	9.75	37.7	5.70	2.41
	Blossom tea	11.34	40.0	5.27	2.59
Yellow Tea		12.66	40.8	5.68	2.64
Green Tea (Hyson and Gunpowder)		12.14	41.8	5.79	2.95

Good tea should contain : 1.) Not less than 30 per cent. of extractive, which is the minimum fixed by English law. 2.) At least 7.5 per cent. tannin. 3.) Not more than 6.4 per cent. ash. 4.) Not less than 2 per cent. of ash soluble in water (the English law requires 3 per cent.). Spent tea is frequently colored, and rendered astringent, with catechu and logwood. To detect the former, 1 gm of the sample is boiled out with 100 cc water ; the decoction boiled with an excess of lead acetate, and the clear filtrate treated with silver nitrate. If catechu be present, a copious yellowish-brown, flocculent precipitate is produced. Pure tea gives only a slight greyish-black turbidity of metallic silver. Logwood imparts to cold water, in which the tea has been softened, a blackish tone of color, which changes to light green with sulphuric acid ; potassium chromate colors it blackish-blue. The following results were obtained on adulterated teas :

VARIETY.	TANNIN.	EXTRACTIVE.	TOTAL ASH.	SOLUBLE ASH.
" Russian tea " (probably Souchong)	6.60	18.4	4.76	0.85
" Blossom tea "...................	4.91	15.3	3.34	0.54
" Blossom tea "....	5.13	14.6	4.51	0.90
" Black tea " (probably Souchong)..	19.77	22.4	3.07	1.12

The three first samples are simply *spent* tea. The last one has received an addition of catechu.

SKALWEIT (Jahresber. d. Untersuchungsamtes f. Nahrungsmittel, &c., in Hannover, 1877–78, through Zeitschr. f. anal. Chem., **18**, 357) recommends that *every beer analysis should be supplemented by a polariscopic test.* The carbohydrates in beer, which possess circular polarization, viz., maltose and dextrine, are both strongly dextrorotatory. If A be the angle of rotation of the original wort, and B the angle after fermentation, B will, of course, be less than A, and the difference will be proportional to the extent of the fermentation. It is interesting to note that the diminution in the percentage of extractive matters in the beer, keeps pace with that in the angle of rotation, so that the coefficients

$$\frac{A}{E} = \frac{B}{E'} = \frac{C}{E''}$$

wherein A B C are the angles of rotation, and E E′ E″, the corresponding percentages of extract, are equal. Although his work is not yet completed, the author finds reason to believe that the, optically weaker, maltose ferments more readily than the dextrine, and in such proportion that the increase of rotatory power, thus induced, keeps pace with the diminution in the percentage of the, optically stronger, dextrine. This assumption is confirmed by the fact, that a beer, prepared from sugar and glycerine, gave already in the first observation in the polariscope, a result sufficiently suspicious to indicate the course of analysis necessary to be followed.

H. W. LANGBECK (Corr. Blatt. d. Vereins Analyt. Chemiker, **2**, No. 7, April 1, 1879) has investigated *the bitter principle which forms in unhopped fermented liquors, when the fermentation has been* *neglected.* By fermentation at 18° to 20°, of a mixture of glucose with small quantities of tartar, tartaric acid, kino, and a few drops of formic and oenanthic ethers, the author succeeded in preparing the substance in question. It is a crystallizable solid, by no means innocuous, and may be separated by Dragendorff's methods I and II.

L. Weigert (Zeitschr. f. anal. Chem., **18**, 207) communicates the following *method for the determination of acetic acid in wine*: The exact determination of acetic acid in wine, by simple distillation, is by no means easy. E. Kessel (idem, **8**, 420) has improved the method, by raising the boiling point of the liquid by addition of phosphoric acid. Even with this improvement, the method requires unremitted attention, and the observance of numerous precautions, to insure success. The method of the author is as follows: 40 or 50 cc of the wine under examination, are introduced into a strong, long-necked, 220 to 250 cc flask; the flask connected with a Liebig's cooler and receiver, and the latter with a Bunsen's pump. The flask is placed in a bath of saturated solution of common salt, contained in a copper vessel of suitable form. When the pump has been started, heat is applied to the bath, and 40 to 50 cc distilled over; the same quantity of water is then added to the contents of the flask, and the distillation in vacuo repeated as above, this process being repeated three times. The five distillates are then united, and titrated with normal potassa solution and litmus. The operations consume about three-quarters of an hour. The results are exact.

J. Nessler (Zeitschr. f. anal. Chem., **18**, 230) communicates an article on *the detection of free tartaric acid and on the sulphuric acid in wine.* The method of the author for the detection of the first-named substance, consists in saturating the wine with potassium acid tartrate, and then adding to 100 cc of the solution, 5 drops of a concentrated solution of potassium acetate. After standing a quarter of an hour, there forms, in solutions containing as much as 0.10 per cent. of free tartaric acid, a notable quantity of crystals of potassium acid tartrate. In wine, or in other solutions containing an organic acid, or concentrated to one-half by evaporation, 0.05 per cent. may be detected with absolute certainty. The remainder of the article is devoted chiefly to a criticism of the strictures of Claus (idem, **17**, 344) on a report by the author on the subject of the detection of sulphuric acid in wine. The author insists that the basis of a method for the detection of such addition, must consist in the determination of the percentage of tartaric acid thus set free. He also gives the following test for the presence of *free* sulphuric acid, or potassium acid sulphate, in wine : If a piece of filter paper be suspended, for several hours, with the lower end dipping into the wine, so that the latter may rise by absorption, and then dried at 100° C., the paper will blacken and become brittle at the upper edge of the part saturated with the wine, if the wine contain free sulphuric acid, or potassium acid sulphate.

G. LUNGE (Berl. Ber., **12**, 928) takes exception to the views propounded by Nessler (in the foregoing article) on the subject of *the normal percentage of sulphuric acid in wine.*

J. NESSLER (Berl. Ber., **12**, 1,161) answers the criticism of G. Lunge (idem, **12**, 928) on his communication relating to *the presence of sulphuric acid in wine.*

G. WOLFRAM (Jahresb. d. kgl. chem. Centrallstelle f. oeffentliche Gesundheitspflege in Dresden, 1878, through Zeitschr. f. anal. Chem., **18**, 346) has devised *a method for the quantitative determination of theobromine in cocoa and chocolate,* based upon the fact that theobromine is precipitated from its acid solution by sodium phospho-tungstate. The reagent consists of a solution of 100 gms sodium tungstate and 60 to 80 gms sodium phosphate, in 500 cc water acidified with nitric acid. The precipitate only forms in solutions containing at least 6 per cent. of sulphuric or nitric anhydride, and when a large excess of sodium phospho-tungstate has been added. 10 gms of cocoa beans, which have been rubbed to a thick paste in a hot mortar, or twenty to thirty gms of chocolate, are treated for some time with boiling water, a slight excess of ammoniacal lead acetate or basic lead acetate added, the solution filtered hot, and the residue washed with hot water until the acidified filtrate gives no trace of a precipitate when treated with sodium phospho-tungstate and cooled. The washing is rapid, and requires about 700 to 800 cc water. The solution which, when an excess of lead acetate has been used, is as clear as water, is treated with sodium hydrate solution, and evaporated to 50 cc, acidified strongly with sulphuric acid, and the lead sulphate filtered off. The filtrate is treated with a large excess of sodium phospho-tungstate, and the separation of the slimy, yellowish-white precipitate in flakes, is promoted by gentle warming and stirring. After a few hours, the cold solution is filtered, and the precipitate brought on the filter, and washed with 6 to 8 per cent. sulphuric acid. The filter and precipitate are then placed in a beaker, barium hydrate solution added to strong alkalinity, heated to promote the decomposition, the excess of baryta neutralized with sulphuric acid, and a possible excess of the latter removed with barium carbonate. The solution, which contains the theobromine, is filtered hot, the filtrate evaporated to dryness in a platinum dish, dried and weighed. To determine the slight proportion of barium carbonate which is usually dissolved with the theobromine, the contents of the dish are incinerated, the residue moistened with ammonium carbonate, ignited and weighed. The difference between the two weighings is theobromine. The method is very exact. The author has made by this method the following analyses of the different kinds of commercial cocoa. The fat was determined by solution with gasoline, which is preferable to ethylic ether for this purpose :

	BEANS (dry, at 100°).				HUSKS (dry, at 100°).		
	Ash.	K_2CO_3 (in Ash).	Fat.	Theobromine.	Ash.	K_2CO_3 (in Ash).	Theobromine.
Caracas cocoa	3.68	0.51	53.8	1.63	13.32	2.00	1.11
Guayaquil "	3.81	0.61	50.6	1.63	5.99	2.29	0.97
Domingo "	3.02	0.74	51.5	1.66	10.61	0.75	0.56
Bahia "	3.35	0.41	51.7	1.64	5.13	1.94	0.71
Puerto Cabello "	3.59	0.54	49.9	1.46	9.28	1.35	0.81
Tobasco "	4.33	1.22	52.6	1.34	5.87	1.61	0.42

The preparation known as Holland cocoa, contains 1.65 per cent. of theobromine.

F. HESS ("Mittheilungen über Gegenstände des Artillerie und Genie-Wesens," from Zeitschr. f. anal. Chem., **18**, 352) has devised *a method for the separation of nitro-glycerine from nitro-cellulose*, especially adapted to such mixtures as Nobel's "explosive gelatine." The gelatinous mixture is digested with 20 times its weight of methylic alcohol, at a temperature of 40° to 50° C., with frequent stirring and kneading—more methylic alcohol being added when necessary, to replace loss by evaporation. All the nitro-glycerine, and a considerable portion of the nitro-cellulose, evidently chiefly the portions of the lower degrees of nitration, dissolve. An excess of chloroform is then added to the mixture, whereby the dissolved nitro-cellulose is entirely precipitated as a slimy mass. After subsidence, the liquid is filtered through a weighed linen filter, the precipitate washed with chloroform, under continual kneading with a glass rod, dried at 100° C., and weighed. The nitro-glycerine is obtained by slow evaporation of the filtrate, with the precautions usual in such cases. An analysis, by the author, of a mixture composed of 93 parts nitro-glycerine and 7 parts nitro-cellulose, yielded :

Nitro-glycerine	92.53
Nitro-cellulose	7.61
Water	.15
	100.29

C. O. HARZ (Zeitschr. f. Biologie, **12**, Heft I, u. Zeitschr. d. allgem. oesterr. Apothekervereins, **14**, 353, through Zeitschr. f. anal. Chem., **18**, 359) calls attention to the great importance of *the microscopic examination of well water*, and gives detailed directions therefor.

F. DOTTO-SCRIBANI (Gazetta Chimica Italiana, through Chem. News, **39**, 206) has devised the following method for *the determination of nitric acid contained in commercial lime-juice:* 100 cc of the juice are boiled and neutralized exactly with barium hydrate; barium citrate precipitates, while barium nitrate remains in solution. The precipitate is filtered off, after cooling, and the barium nitrate in the filtrate is decomposed with sulphuric acid. The weight of the barium sulphate multiplied by 0.4635, represents the quantity of nitric acid in 100 cc of the sample.

H. R. PROCTOR (Chem. News, **39**, 246) recommends that *the titration of free acid in tan liquors*, should be made with lime-water, the point of saturation being indicated by a turbidity, owing to the precipitation of calcium tannate when the stronger free acids are saturated. The chief object of the determination being to ascertain the amount of lime the acid will dissolve, the question of the nature of the acid is of subordinate importance.

GOTTLIEB STEIN (Berl. Ber., **12**, 1,175) determines *the amount of water in the so-called Turkish-red-oil* (sulpho-castoroleate and sulpho-pyroterebinate of ammonia), by heating, in a porcelain dish, 10 gms of the oil with 75 cc of saturated sodium chloride solution, and 25 gms of dry beeswax. The oil, as it is salted out, unites with the wax, and after cooling, may be weighed therewith.

A. STROMEYER (Correspondenzbl. d. Vereins analyt. Chemiker, **2**, No. 7, through Chem. News, **39**, 264) determines *the percentage of acetic acid in crude pyrolignate of lime*, by distilling, almost to dryness, 5 gms with 50 cc of a solution of phosphoric acid, of 1.2 sp. gr. The residue is again distilled, first with 50 cc of the phosphoric acid, and then with 50 cc of water. The distillates are united and titrated with normal alkali.

R. ANGUS SMITH (Chem. News, **39**, 267) has devised a very simple and ingenious *method for the detection of fire-damp.* The instrument used is modelled after the ordinary "compression syringe," but the brass tube is provided, at its lower extremity, with a small glass window. A mixture of air with but 5 per cent. of marsh gas, will explode when violently compressed in the instrument, and by introducing a very small quantity of platinum black, an explosion can be obtained from air containing but 2½ per cent. The flash of the explosion can plainly be seen through the window in the tube. The tube and piston are lubricated with soap. The author has also improved the diffusion method of Geo. F. Ansell (idem, **22**, 280), by filling the porous vessel with nitrogen (made from chloride of lime and sal-ammoniac). The delicacy of this test is, however, inferior to that of the preceding, being limited to 5 per cent.

Skalweit (Jahresb. 1877–78, d. Untersuchungsamts f. Lebensmittel, etc., zu Hannover, through Zeitschr. f. anal. Chem., **18**, 358) recommends the following *methods of testing the essential oils for adulteration*. The qualitative test for alcohol consists in shaking, with a fragment of fuchsine; the quantitative, by shaking with a sufficient quantity of water, in a graduated stoppered cylinder, and noting the alteration in volume of the water. This last-named operation should precede the determinations of specific gravity and boiling point. A further test consists in noting the angle of rotation in the polariscope. Although this angle is not absolutely constant in different samples of the same oil, it is, nevertheless, a valuable indication of the nature of the oil which may have been used as an adulterant, as the coefficients of rotation differ very considerably in different oils. The following measurements were made by the author with extreme care. The numbers obtained are all calculated for the specific rotation by the formula:

$$(\alpha)\ j = \frac{\alpha}{pl}$$

Oil of lemons (from an apothecary)	+ 164°
“ “ (from a prominent dealer)	+ 145°
“ “ from a bakery, after purification from alcohol	+ 139°
Oil of oranges (peel)	+ 64°
“ fennel	+ 42°
“ pine	+ 39°
“ bergamot	+ 38°
“ “	+ 33°
“ myrrh	+ 24°
“ rosmary	+ 18°
“ cassia	0°
“ olives	0°
“ cajaput	— 3°
“ roses	— 7.5°
“ lavender	— 25°
“ “	— 32°
“ turpentine	— 40°
“ cloves	— 96°
“ peppermint	— 105°

Beaumont J. Grosjean (Journ. Chem. Soc., 1879, 341) communicates a voluminous paper on *the determination of tartaric acid in lees and inferior argols.* The "lees" or deposit which falls to the bottom of fermenting wine vats, consists of calcium and potassium bitartrates, with large quantities of fermentative matters, and some phosphoric acid, iron, alumina, &c. "Argol" is the crystalline crust from the sides of the vats, and contains much less impurity than the former. Tartar is the product of superficial purification of lees and argol. According to the author, the method now exclusively employed in fixing the value of the lees and inferior argol, sold in the London market, is the "oxalate method" of Warrington (idem [2], **13**, 973). Good tartar gives, by the indirect methods, described as C and D on p. 959 of Warrington's paper, the same results as by the "oxalate" method, and, on account of the easier manipulation, the methods first-named are, in this case, preferred by the author. By the direct oxalate method, as originally described, the finely powdered lees or argol was heated with a little water to 100° C., and excess of neutral potassium oxalate was added, the mixture digested for one-quarter hour, nearly neutralized with caustic potassa, digested for a short time longer, with frequent stirring, filtered, and the residue washed on the vacuum filter. The filtrate concentrated, and the tartaric acid precipitated as potassium acid tartrate by the addition of an excess of citric acid, the precipitate filtered off, washed, and titrated with standard alkali. The modifications of this method which the author employs are, in brief, as follows :

1.) The proportion of lime in the sample is approximately determined by treatment with an excess of potassium oxalate, and the excess determined by titration with calcium chloride, in the presence of acetic acid. The acidity of the sample is also determined by titration with sodium hydrate. A portion of the sample containing about 2 gms of tartaric acid, is then taken, softened in water, and mixed with about 1½ gms more of neutral potassium oxalate than is necessary to decompose the calcium tartrate.

2.) The mixture is *nearly* neutralized by the addition, drop by drop, of a 3½ per cent. solution of potassium hydrate, the quantity being regulated by the results of the preliminary titration. If an excess of alkali has been used, a decided excess of hydrochloric acid must be added, and the neutralization repeated.

3.) The nearly neutral liquid, which should not measure over 30 cc, is filtered and washed ten times, using each time 3 cc water, on the vacuum filter. The author prefers the filter of Casamajor (Chem. News, **32**, 45), using disks of platinum, lead or pumice.

4.) The filtrate and washings from the previous operations, which should not measure more than 50 cc, are treated with 5 gms of potassium chloride.

5.) Citric acid is then added to the extent of 1 to 1.5 gms in excess of what is required by the equation :

$$3K_2\bar{T} + H_3\bar{Ci}Aq = 3KH\bar{T} + K_3\bar{Ci}Aq,$$

that is, if 2 gms of tartaric acid are present, there must be added 2 to 2.5 gms of citric acid.

6.) The citric acid may be added either in crystals or as a 50 per cent. solution. It is preferable to precipitate in the granular form, by stirring, rather than to permit the formation of large crystals.

7.) The precipitation may be *completely* effected, by stirring for ten minutes, although, for the sake of safety, the author allows the liquid to stand for half an hour after the stirring.

8.) The bitartrate is filtered and washed on the vacuum filter with a five per cent. solution of potassium chloride, saturated with potassium acid tartrate. This solution must be freshly made, and its acid strength determined by titration. The end of the washing is attained, when the acid strength of the washings becomes the same as that of the washing liquor used. The washed bitartrate is removed, with the paper, to a beaker glass, and titrated with standard soda solution.

Two experiments, by the author, yielded 99.96 instead of 100 of tartaric acid. The determination may be made in four hours.

George Attwood (Journ. Chem. Soc., 1879, 207) describes methods for *the quantitative blow-pipe assay of ores and compounds of mercury.* The treatment varies according to the nature of the substance. Substances belonging to class "A," including metallic mercury, cinnabar, tiemannite, the oxides of mercury and mixed sulphurets, may be assayed by mixing the finely powdered substance with five to ten times its weight of finely powdered litharge, and distilling from a miniature glass retort, over the spirit lamp. Substances belonging to class "B," comprising compounds of mercury with the halogens, are mixed with one volume of neutral potassium oxalate and one volume of potassium cyanide, and distilled from a small glass retort, as above. Class "C" comprises the amalgams. These must be distilled from small steel retorts, which are placed in a charcoal blow-pipe furnace, similar in principle to the Plattner's furnace, and heated with the blow-pipe flame. With this last-named class of substances, the author recommends a control experiment, by determining the loss by ignition before the blow-pipe. The paper is illustrated by drawings of the very simple and practical forms of apparatus employed by the author.

T. M. **Drown** (Journ. Franklin Inst., **107**, 361, and Am. Chem. Journ., **1**, 147) describes the method employed by him in *the determination of silicon in pig-iron and steel.* About 1 gm of the material is dissolved, in a platinum or porcelain dish, with 25 cc nitric acid (sp. gr. 1.2). When the action has ceased, 25 to 30 cc dilute sulphuric acid (1 of acid to 3 of water) is added, and heat applied to expel the nitric acid. Water is added to the cooled contents of the dish, and heat applied until the ferric sulphate dissolves. The solution is filtered hot, the residue washed, first with hot water, then with 25 to 30 cc hydrochloric acid (sp. gr. 1.12), and then with hot water. Results accurate.

V. **Deshayes** (Bull. Soc. Chim. de Paris, **31**, 40) has made a series of extended studies on the subject of *the colorimetric method of Eggertz for the determination of carbon in iron and steel.* The results otained by the author, demonstrate that, while the method affords excellent results with low steels, it is quite inapplicable to those of extra hardness, and to the metals occupying a position between steel and cast iron, with 2.0 to 2.5 per cent. of carbon. The presence of phosphorus or maganese has a strong influence on the color of the solution, and in such cases the author employs a special series of types. The author finds that a material error is also introduced, when there is a very great difference between the percentage of carbon in the type, and that in the sample under examination. He recommends the employment of a series containing 0.150 ; 0.500 ; 0.750 ; and 1.000 per cent.

VI. GASOMETRIC METHODS.

Walter Hempel (Berl. Ber., **12**, 636) describes *a method for the determination of hydrogen in gaseous mixtures, by absorption.* The author employs as an absorbent, palladium sponge, or palladium black, which is slightly oxidized by heating to redness and slow cooling. When introduced into a gaseous mixture containing hydrogen, the thin layer of oxide is reduced, and the reduction is attended by an elevation of temperature sufficient to initiate the absorption of hydrogen, which then proceeds, until less than one-tenth per cent. remains. Beyond this limit, the absorption proceeds very slowly. The author describes a special form of absorption apparatus. The results obtained are very satisfactory.

WALTER HEMPEL (Berl. Ber., **12**, 1,006) communicates *a method for the fractional combustion of hydrogen in mixtures of hydrogen, methyl hydride and nitrogen.* By the passage of mixtures of the above gases, with an excess of oxygen, or air, over palladium sponge, at ordinary temperatures, the author finds that the hydrogen is completely burned, while at temperatures not exceeding 100° C., the methyl hydride remains unaffected. The author proposes the method for use in gasometric analysis. The experimental results, communicated in the paper, show the method to be very exact.

DR. OTTO LINDEMANN (Zeitschr. f. anal. Chem., **18**, 158) describes a simple method for *the volumetric estimation of free oxygen in gaseous mixtures, by means of phosphorus.* The lack of a simple method of estimating the percentage of free oxygen in the gases of the leaden chambers, used in the sulphuric acid manufacture, and the very satisfactory results obtained by P. Hart (idem, **8**, 482), by using phosphorus as an absorbent, led the author to devise a simple apparatus, based on the principle of Orsat's apparatus, in which the absorption by phosphorus may be effected, even in gases rich in oxygen, in, at most, three minutes. The apparatus consists of a pipette, with a glass cock at the lower end, and attached, at the upper end, to a T tube, which communicates, in its turn, with an ordinary burette, and also with the air. The pipette is filled with thin sticks of phosphorus, and the volume of the gas is measured before, and after the absorption, in the burette, as in Orsat's apparatus. Comparative experiments with a Winkler's gas burette, using alkaline pyrogallate solution, have convinced the author of the accuracy of his method.

J. RIBAN (Bull. Soc. Chim. de Paris, **31**, 385) finds that *the determination of phosphuretted hydrogen* may be made by absorption with acid solution of cuprous chloride. The absorption is instantaneous, and is attended with the formation of crystallizable compounds. Arseniuretted and antimoniuretted hydrogen, are also absorbed by cuprous chloride, forming black precipitates, and this reagent should be preferred to silver nitrate, when it is necessary to work with the mercurial trough.

J. W. THOMAS (Journ. Chem. Soc., 1879, 213) communicates a paper on *some points in the analysis of combustible gases.* The apparatus employed by the author, is an improvement on McCleod's modification of the apparatus of Frankland and Ward. By shortening the eudiometer, and availing himself of the fact that by exploding the contents under reduced pressure, a much smaller addition of non-explosive gas is sufficient to ensure the safety of the apparatus, the author has succeeded in greatly improving the accuracy of the method, especially when small quantities of material are operated upon.

A. V. E. Young (Am. Chem. Journ., 1, 105) describes *an apparatus for gas analysis, from simple laboratory material*, which appears to unite the conditions of extreme simplicity and ease of construction and manipulation, with a high degree of accuracy in the results which it is capable of affording. The author employs the Bunsen's eudiometer, for the purposes of measurement and explosion, in conjunction with the principle of measurement by comparison with a standard volume of air, as described by Williamson and Russel (Proc. Royal Soc., 9, 218). A detailed description would not be intelligible without reference to the drawing. The analytical results obtained by the author are eminently satisfactory, and the rapidity of the method is such, that an analysis of air can be made with it in about three-quarters of an hour.

A. Vernon Harcourt (Chem. News, 86, 103, through Zeitschr. f. anal. Chem., 18, 249) recommends *a new unit for photometric measurements.* According to the author, the method of determination by means of standard candles is subject to variations, which depend on the purity of the substance of which the candles are composed, the nature of the wick, the temperature and the height of barometer, and may amount to as much as 6 per cent. The author, while retaining the system of indicating the result of the test in candles, proposes to use, as a standard of comparison, the flame of a mixture of air and pentane (that portion of the lighter products of the distillation of American petroleum, which has a constant boiling point of 50° C.). The air is measured in a gasometer over water, the pentane is added in a liquid form. The mixture is burned under fixed conditions of temperature, pressure, and size of burner.

VII. MISCELLANEOUS PHYSICAL METHODS, APPARATUS AND REAGENTS.

J. N. Lockyer (Chem. News, 39, 243) communicates *a note on the spectrum of sodium.* The vapor given off by the metal, after prolonged and slow distillation in vacuo, shows the red and green lines, without any trace whatever of the yellow one. Hydrogen is given off in large quantities, and, at times, the C line and the red "structure" are alone seen. After this treatment, the metal volatilizes with great difficulty, even when red hot.

J. LANDAUER (Berl. Ber., **11**, 1,772, through Zeitschr. f. anal. Chem., **18**, 250) has investigated *the spectra of the salts of saffranine*, and attained results which harmonize with the views of the latest investigators, that the absorption-spectra of colored substances cannot be held to be absolutely characteristic of the substances affording them. As Hoffman and Geyger (Berl. Ber., **5**, 531) have shown, the red color of the solution of salts of this base, on addition of strong acids, especially sulphuric acid, changes to violet, indigo, bluish-green and, finally, emerald-green. Each of these colors possesses a separate spectrum, which is represented, graphically, in the original paper. The author finds that the changes of color are due simply to the formation of different hydrates, and argues that such an essential modification of optical properties from such slight causes, is opposed to the assumption of the characteristic nature of the absorption-spectra.

H. SETTEGAST (Pogg. Annalen [N. F.], **7**, 242) presents a series of *contributions to quantitative spectral analysis.* Among other interesting observations, the author has, at the suggestion of Prof. Lothar Meyer, devised a method for the quantitative estimation of nitric acid, based on the well-known reaction with diphenylamine, dissolved in sulphuric acid. The intensity of the blue coloring matter thus formed, is proportional to the amount of nitric acid present and, as its coefficient of absorption, is constant, when the proper precautions are observed, the method gives exceedingly exact results, especially when very minute quantities are operated upon, and is, hence, especially well suited to the *estimation of the nitrates in potable waters.* The author obtained 0.00002018, 0.00002021, 0.00001976 and 0.00001980 gm NO_3H, instead of 0.000020136 gm. He has also devised a method for the quantitative spectral estimation of phosphoric acid, based on the use of salts of uranous oxide (prepared by reduction of uranic sulphate with zinc dust), which, however, is of subordinate value only.

B. O. PEIRCE (Pogg. Annalen [N. F.], **6**, 599) has investigated *the emission-spectrum of the halogen compounds of mercury.* A small portion of the salt is placed in a Geissler tube and heated, while the current from an induction coil is passed through the tube. At a moderate heat, the mercury spectrum makes its appearance ; as the heat increases, each salt gives a characteristic spectrum.

Mercuric chloride shows bands between the orange and green, reaching from 108½ to 110½, on the scale of the author's instrument ; at a higher heat, a continuous spectrum appears to the right of the bands, the green part of which is traversed by numerous very fine black lines.

Mercurous chloride shows the same bands, whence the author infers decomposition during the volatilization.

Mercuric bromide shows a band between 131 and 135, and a weak continuous spectrum when the tube is hot.

Mercuric iodide shows a blue band between 168 and 172, and, at high temperatures, a continuous spectrum in the violet, to the right of Hgβ.

The central points of the bands of mercuric chloride, bromide and iodide, correspond to the wave lengths 443, 500 and 558. It is interesting to note, that the bands of mercuric bromide lie in the mean between those of the chloride and iodide, thus :

$$558-500=58. \qquad 500-443=57.$$

H. KOEHLER (Zeitschr. f. anal. Chem., **18**, 239) describes *an improved apparatus for the determination of solubilities.* The apparatus is based on the principle of that of Victor Meyer (Berl. Ber., 8, 998), but possesses the advantage that the determination may be made at ordinary pressures, without contact of air, and is, hence, specially suitable in the case of readily oxidizable substances, or of hygroscopic solvents. The apparatus is of very simple construction, and permits the operations of saturation and filtration to be conducted at a constant temperature, in dry air, or in any indifferent gas. Supersaturation is prevented by the passage of a current of dry air, or gas, during the operation of solution. A detailed description would not be intelligible without the figures.

A. GAWALOVSKI (Zeitschr. f. anal. Chem., **18**, 244) describes a convenient *adaptation of the Scheibler's calcimeter for use as an azotometer.* The description is illustrated with drawings of the apparatus, which latter will be manufactured at the establishment of Alt, Eberhardt & Jaeger, in Thuringia.

A. TERREIL (Bull. Soc. Chim. de Paris, **31**, 155) recommends that *the determination of the fusing points of organic bodies*, should be made by placing a drop of the melted substance on the bulb of a thermometer, and by alternate cooling and heating, noting the points of solidification and fusion, respectively.

NOTE.—The foregoing method has long been employed in certain of the German laboratories, and originated with Bunsen.

WILHELM HANKÓ (Berl. Ber., **12**, 451) describes *a new form of apparatus to be used in the determination of nitrogen by Dumas'* *method.* The arrangement is designed primarily to avoid the use of mercury.

H. FRESENIUS (Zeitschr. f. anal. Chem., **18**, 260) calls attention to *the variations in the quality of Swedish filter paper*, and especially of that bearing the mark J. H. Munktell. The quantity of ash found by various analysts, varies from 0.200 to 0.597 per cent., while the great fluctuations in the composition of the ash may be illustrated by the following analyses :

	V. WIEN.	BOHUSLAV BRAUNER.
SiO_2.	31.876	30.63
CaO.	21.384	26.28
MgO.	11.301	7.84
Al_2O_3.	14.364	14.86
Fe_2O_3.	9.367 and PO_5	5.08
Mn_3O_4.	7.637	—
MnO.	—	1.74
Alkalies.	2.162	13.57
SO_3.	1.684	—
PO_5.	0.238	—
Cl.	trace	—
CuO.	trace	—
	100.000	100.00

A. GAWALOVSKI (Zeitschr. f. anal. Chem., **18**, 246) in continuation of his investigation on *the comparative value of the different brands of filter paper* (idem, **16**, 59–61), has examined several brands of Swedish and Austrian filter paper, and finds that manufactured by A. Schmidt, of Gr. Ullersdorf, in Austria, to be equal in purity to the best Swedish paper, while costing less than one-fourth as much as the latter.

A. VAN HASSELT (idem, **18**, 251, from Maandblad voor Natuurwetenschappen, **6**, 77 and 113) has devised *a method for the determination of boiling points on small quantities of substance.* The principle on which the method is based, consists in noting the temperature at which the tension of the vapor becomes equal to 760 mm of mercury.

A. WEINHOLD (idem, **18**, 252, from Carl's Rep. f. exp. Physik, **15**, 1) describes *an improved form of apparatus for the distillation of mercury.* The distillation takes place in vacuo. The apparatus is, in appearance, somewhat complicated, but, in reality, the method is extremely convenient, and affords a product of such purity and dryness, that it deserves the preference over all the other methods of purification.

L. PFAUNDLER (Berl. Ber., **12**, 165) communicates a brief description of his *method of determining the density at high temperatures, of vapors of substances which attack mercury.* The method was originally published in the reports of the Scientific and Medical Society of Innsbruck, for 1870. The apparatus consists of two vessels united by a capillary tube. The substance is introduced into the lower vessel through an aperture, which is then sealed. The upper vessel is connected with an ordinary mercurial manometer. After drying the vessels, filling them with dry air or other gas, and determining their cubical contents, they are heated to the temperature required to volatilize the substance under examination. During the heating, the pressure of the mercury in the manometer is increased, so that no air can escape from the upper vessel. The temperature is noted on an air thermometer of similar construction and capacity to the apparatus. The difference between the pressure in the apparatus and that in the air thermometer, gives the pressure of the vapor with great exactitude. The temperature is given by the air thermometer; the volume by the manometric operations and a previous calibration of the capillary tube connecting the vessels with the manometer. We have, therefore, the four data from which the vapor density may be calculated. The method is somewhat similar to that of Victor Meyer (idem, **12**, 1867), but differs therefrom in the fact that it is based on the measurement, not of the volume of air displaced, but of the tension of the vapor by constant volume. It is susceptible of greater accuracy than the former, but is less simple, and, hence, less suitable for ordinary molecular weight determinations.

J. W. BRUEHL (Berl. Ber., **12**, 204) communicates *a new method of purifying mercury,* which will be welcomed by all chemists familiar with the difficulties of the methods hitherto in use. The author employs a solution of 1 liter water, 5 gms potassium pyrochromate, and a few cubic centimeters of sulphuric acid. The mercury is shaken vigorously with an equal volume of this solution. It separates readily into small globules, while a small portion is temporarily converted into red mercuric chromate. The flask is shaken until this red powder disappears, and the solution appears of a pure green color from chromic sulphate. A powerful stream of water is then conducted through the bottle, to wash out the fine grey powder which surrounds the globules of mercury and consists of the oxidized impurities. The operation is repeated once or oftener, according to the degree of impurity of the metal, and the metal is finally shaken with distilled water, until the latter removes no more grey powder, but remains entirely clear. As an instance of the perfection of the action, the author states that a lot of some 25 kg mercury, which had become contaminated with a considerable quantity of Wood's fusible metal, and which had resisted the most effectual of the older methods of purification, was, by this method, completely freed from its impurities in about two hours. The loss of mercury is exceedingly small.

LOTHAR MEYER (Berl. Ber., **12**, 487) describes *an improved method of purifying mercury.* The apparatus consists of an open glass tube, about 1½ meters long and 3 cm wide, filled with a solution of ferric chloride, and placed in a glass jar, about 150 cc in height, and filled with pure mercury. The mercury to be purified, is allowed to fall in a fine spray from a separatory funnel through this ferric chloride, to the bottom of the longer tube, where it unites with the mercury in the cylinder, from which, as it overflows, it is conducted by a spout to the bottle placed to receive it. The mercury in the jar counterbalances the pressure of the column of ferric chloride solution in the cylinder. If the metal is not completely purified by one passage through the apparatus, it may be passed through again. Repeated treatment is rarely necessary. The metal so treated was found to be so pure, that Prof. G. Quincke used it for capillary measurements.

J. W. BRUEHL (Berl. Ber., **12**, 576), referring to the foregoing method for *the purification of mercury,* states that the purification by chromic acid, proposed by him (idem, **12**, 204), can only be conducted in the Meyer's apparatus when the mercury is sufficiently pure to flow in a thin stream. When the amount of impurity is sufficient to cause the mercury to be thick, or even pasty, the method can only be used as he has indicated, viz., by shaking the acid with the mercury.

R. OTTO (Berl. Ber., **12**, 215) makes a highly important communication on the subject of *the preparation of hydrogen sulphide, for use in toxicological investigations.* The author finds, as the result of numerous experiments, that hydrogen sulphide, made by the action of pure sulphuric acid on ferrous sulphide contaminated with arsenic, is always contaminated with hydrogen arsenide. He insists, therefore, that *hydrogen sulphide, for forensic investigations, must always be made from materials free from arsenic,* and recommends that for such applications, the gas should be made by the action of hydrochloric acid on calcium sulphide (made by heating calcium sulphate with charcoal). The gas so obtained is entirely free from hydrogen, and, hence, is free from hydrogen arsenide, even if the acid used is contaminated with arsenical compounds. The author furthermore concedes the possible correctness of the views of Meyers (Ann. d. Chem., 159, 124 and 127), that the arsenic reported to have been found in the normal animal organism, may be referred to the employment of hydrogen sulphide made from materials contaminated with arsenic.

FR. RUEDORFF (Berl. Ber., 12, 249) describes an extremely *simple and practical form of volumometer*, based on the same principle as the instruments of Say, Kopp and Regnault. In testing the accuracy of the method, the author found the volume of a solid glass body to be 6.4525 cc, while the hydrostatic method yielded the figure 6.453 cc. The volume of a hollow glass body was, according to the volumometric method, 3.385 cc, by the hydrostatic balance, 3.382 cc. By means of his new apparatus, the author made the following specific gravity determinations, viz.:

Cupric sulphate (cryst.)	2.330	Hydrate of chloral (cryst.)	1.901
Rock salt from Wiclizka	2.137	Fuchsine (cryst.)	1.220
Oxalic acid (cryst.)	1.531	Toluidine	1.046
Benzoic acid (sublimed)	1.337	Thymole (cryst.)	1.069
Salycilic acid (dialyzed)	1.443	Asparagine (cryst.)	1.552
Trinitrophenole (cryst.)	1.813		

The apparatus is manufactured by the glass-blower, Fl. Mueller, of Berlin, Prussia.

F. v. LEPEL (Berl. Ber., 12, 263) describes a new *universal support for the pocket spectroscope.* The apparatus is specially designed for use in laboratories not provided with illuminating gas, and, hence, of subordinate interest to American readers.

A. HOUZEAU (Comptes Rendus, 88, 747) describes, under the name of gravivolumeter, *a new form of dropping-glass*, which yields drops of uniform size, and permits the flow to be regulated with great accuracy. The author claims for the instrument numerous important advantages as an adjunct to volumetric analysis.

JAMES BOTTOMLEY (Chem. News, 39, 276), in a *note on colorimetry*, calls attention to a source of error in the use of those colorimeters, in which the depth at which white disks appear of uniform intensity of color, when immersed in colored solutions, is employed as a basis for the determination of the relative proportions of coloring substance they contain. The light which illuminates the disks has first to pass through the solutions, and is, hence, itself more or less colored. The author gives a formula for correction of the indications of such instruments.

E. Schulze (Berl. Ber., **12**, 249) has determined *the specific rotation of isocholesterine* (found by the author, associated with cholesterine, in the fat from sheep's wool). This substance is dextrorotatory, whereas cholesterine is laevo-rotatory. An etherial solution of isocholesterine, containing 6.453 gms in 100 cc, showed in the 200 mm tube of a Soleil-Ventzke polariscope, a rotation of 22° ; a solution of half the strength rotated 11.25°. A solution of another preparation, containing 7.344 gms in 100 cc, showed a rotation of 25.5°. From these data, the author deduces for isocholesterine the specific rotation $[\alpha]_D = + 60°$.

J. W. Bruehl (Berl. Ber., **12**, 197) discusses *the limits to the employment of the method of determination of vapor densities in the barometric vacuum.* From experiments, conducted with improved methods and the greatest care, he is led to the conclusion, that it is useless to employ a much higher temperature than the boiling point of aniline. The highest temperature which can usefully be employed, is about 220° C., as the rapid increase in the tension of mercury vapor beyond this temperature, completely neutralizes, through the much more rapid increase of pressure, the advantages sought to be attained by the conversion to a gaseous state, by increase of temperature.