By precipitation of the acid solution, with copper acetate, a light blue, difficultly soluble, amorphous precipitate is obtained; acetate and basic acetate of lead yield white precipitates. The yield of the acid amounts to about 15 per cent. of the crystallized sulphate of quinine employed in the oxidation; small quantities, however, collect in the mother liquors, which are obtained in preparation of the barium salt.

The mother liquors probably contain also other nitrogenous oxidation products of quinine, the purification of which still engages our attention.

We call attention to the fact that Ramsay and Dobbie (Ber. XI, 324) have described an acid obtained in the oxidation of quinine by potassium permanganate, which shows many similarities to ours.

Quinidine (Conchinine of Hesse) and Cinchonine, on oxidation with potassium permanganate, apparently yield the same acid.

The analytical results obtained for the three acids, and the behavior of these acids to salt-solutions, are the same.

Our acid, which, as already mentioned, is tribasic, will probably have to be looked upon as Tricarbopyridinic acid,  $C_5H_2N$  (COOH)<sub>5</sub>.

The observation that the lime salt, on distillation, with an excess of lime, yields an oil, the odor of which reminds of that of Dippel's bases, tends to confirm this view.

We are still engaged upon the further investigation of the acids and their derivatives.

ROTTERDAM, AMSTERDAM, January, 1879.

Reports on Special Departments of Chemical Science.

REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY, JANUARY TO MARCH, 1879.

BY GIDEON E. MOORE, PH. D.

F. KESSLER (Zeits. f. anal. Chem., 18, 1, 1) describes an improvement on his method of determining manganese in alloys of iron (idem, 11, 255), whereby the method which, in its original form, was applicable only to alloys containing not more than 13 p. c. of manganese, is rendered applicable to alloys of every degree of richness. The principles on which the method is based, are : 1st, Precipitation of the iron as basic sulphate in the cold, by addition of sodium carbonate as long as the precipitate first formed dissolves on shaking, and addition of sodium sulphate. 2d, Precipitation of the manganese as dioxide, combined with zinc oxide, by the addition of zinc chloride, sodium acetate and bromine water. 3d, Reduction of the manganese dioxide, thus formed, by a solution of antimonous chloride of known strength. 4th, Titration of the excess of antimonous chloride with potassium permanganate, standardized with manganese pyrophosphate. The author has subjected his new method to an exhaustive series of tests, the result of which would appear to amply justify his claim, that in regard to rapidity and accuracy, the method compares favorably with the best gravimetrical methods. As a matter of general interest, it may be mentioned that out of many hundred specimens of steel examined by the author, but one, viz.: a specimen of genuine East Indian Wootz steel, was found to be free from manganese.

**RICHARD** POPPER (idem, 18, 14) gives the continuation of his paper on the quantitative determination of precipitates without filtration, washing or drying. The principle on which the method is founded, finds its expression in the following equations:

$$G = g + N - \frac{N}{S}$$
 s, and  $N = \frac{S}{S-s} (G-g)$ ,

wherein N is the weight of the precipitate. S is the known specific gravity of the precipitate, s the specific gravity of the liquid in which it was formed, g the weight of the specific gravity bottle filled with the same, and G the weight of the specific gravity bottle containing the precipitate, and filled up with the liquid in which it was formed. The method, hence, requires but two weighings. When the specific gravity of the precipitate is unknown, it may be readily determined by simply taking a known weight of a crystallized salt, from which the weight of the precipitate can be calculated, and transforming the first of the above equations into:

$$S = \frac{N - (G - g)}{N} s$$

C. Wolf (idem, 18, 38) describes methods of quantitative spectral analysis carried out with the apparatus described in his former papers on the spectral analysis of commercial indigo (idem, 17, 65). In the determination of cobalt, he employs the sulphocyanide dissolved in dilute alcohol. In the determination of copper, the metal is precipitated by electrolysis, dissolved in nitric acid, and the solution supersaturated with ammonia before using the spectroscope. Both determinations are characterized by extreme delicacy. The author recommends the method as being specially well adapted for the estimation of minute quantities of copper in articles of food, etc. In the latter case, the organic matter is destroyed by incineration, and the copper determined in the ash by solution in acids and electrolysis, etc., as already described. Metallic iron is determined by dissolving in a solution of cupric salt, and estimating the copper, thus reduced, as above. A number of samples of commercial *ferrum reductum* examined by this method, showed that the proportion of metallic iron contained therein varied from 15.62 to 96.13 per cent.

Dr. C. DOELTER (idem, 18, 50) describes a simple method of determining ferrous oxide in silicates by means of hydrofluoric acid, without the use of a ptatinum retort. It results, namely, from the experiments of E. Ludwig, which were confirmed by those of the author, that commercial hydrofluoric acid, owing to the presence of such impurities as sulphurous oxide and hydrogen sulphide, decolorizes to a marked degree a solution of potassium permanganate. Hence, before using the commercial hydrofluoric acid, it has hitherto been necessary to rectify it over potassium permanganate in a platinum retort. To obviate this inconvenience, the author employs the following method, the principle of which was communicated to him by Prof. v. Pebal. The silicate is mixed in a platinum crucible with a quantity of commercial hydrofluoric acid sufficient for its complete decomposition. The crucible is placed on a flat, round, iron plate, having around its circumference a gutter, about 11 centimeter in depth. A tall beaker glass is inverted over the plate, with its lower edge in the gutter, the joint being made tight with sand, or, better, with mercury. A hole is bored in the bottom of the beaker and a glass tube, passing through it and ending just above the crucible, serves to introduce carbon dioxide, with which the apparatus is kept filled during the decomposition of the mineral. Instead of the beaker, a glass cylinder, closed above with a tight-fitting, perforated glass plate, may be used. After the apparatus has been put together and filled with carbon dioxide, it is placed on the water bath, or over a small lamp flame. In the case of minerals readily decomposable by hydrofluoric acid, decomposition of the mineral and expulsion of the hydrofluoric acid, may be effected in two hours. In the case of difficultly decomposable minerals, more time, and a larger platinum crucible are required. When the decomposition is complete, the contents of the crucible are dissolved in water and titrated with permanganate. with the usual precautions. The results communicated by the author are highly satisfactory, the fact having been established that the impurities of the commercial acid do not effect the reduction of ferric to ferrous oxide under the conditions indicated.

Dr. WILHELM PILLITZ (idem, 18, 58) communicates the results of his analysis of the Zsadányer meteorite, and describes in detail the methods of analysis employed. The latter contain nothing new, and need not therefore be reproduced here. The separation of the metallie from the silicate portion of the mineral, was effected by Boussingault's method of solution with mercuric chloride. The results of the analysis were as follows:

Silicate Part.		Metallic Part.			
FeO	15.054	Cu & Sn	2.323		
$Cr_2O_3$	1.276	Mn	6.047		
$Al_2O_3$	3.027	${f Fe}$	67.219		
MgO	22.343	Ni	10.177		
CaO	4.683	Co	trace.		
K <sub>2</sub> O	<b>5.85</b> 0	$\mathbf{s}$	9.734		
Na <sub>2</sub> O	0.421	Р	1.658		
$SiO_2$	47.346	С	0.774		
		Chromite	2.065		
	100.000		-		
			99,9 <b>9</b> 7		

The author notes, as of special interest, the occurrence of manganese in the metallic state, and the large proportion of potassium oxide. (Note: The figures in the list of metallic ingredients being derived by calculation, should sum up 100.)

E. REICHERT (idem, 18, 68) describes a simplified method of testing butter, based on the principle of Hehner's method (idem, 16, 149). 2.5 grammes of the filtered and dried fat, 1 grm. solid potassic hydrate and 20 cc 80 per cent. alcohol, are placed in a 150 cc flask, and heated in the water bath, with continual shaking, until the soap no longer has the character of a foaming, viscid mass; 50 cc water is then added and, after solution, the mass is decomposed by addition of 20 cc of dilute sulphuric acid (1 cc pure acid to 10 cc water). The mixture is distilled, under employment of the bulb tubes used in determining boiling points to avoid contamination of the distillate with sulphuric acid, a current of air being carried through the boiling liquid to prevent concussive ebullition. The distillate, which may contain solid fatty acids, is filtered directly through a moistened filter into a 50 cc flask; after 10 to 20 cc has come over, the distillate is poured back into the flask and the distillation continued until the 50 cc flask is filled precisely to the mark. The distillate is treated with 4 drops of tincture of litmus and immediately titrated with decinormal soda solution. The titration is finished when the blue color of the solution remains for some time unchanged. The analytical tests show that

87

the method possesses a very high degree of accuracy and constancy. Proceeding as above, the author found different samples of butter and other fats to require the following quantities of decinormal soda solution :

Butter,	No.	114.50 cc	Butter, No. 1014.95 cc
"	"	2	" " 11
"	"	3	<i>" "</i> 1213.00 <i>"</i>
"	"	4	" " 1313.40 "
"	"	514.00 "	Commercial "Butter fat". 10.50 "
"	"	<b>6</b> 14.40 "	Cocoanut fat 3.70 "
"	"	714.00 "	Oleo-Margarine Butter 0.95 "
"	"	8	Lard 0.30 "
"	"	913.80 "	Kidney fat 0.25 "

The author adds that, assuming the specimens of butter Nos. 1 to 13 to have been pure, there results an average of 13.97, or, in round numbers, 14 cc of soda solution, with a probable deviation of  $\pm$ .45 cc, as the alkalimetric titre of the soluble fatty acid from butter. The author concludes that, until the matter has been further investigated, it is safe to assume every butter to be genuine which requires above 13 cc of soda solution, while he should consider a sample of doubtful genuineness, which required less than 12.5 cc. The author proceeds to the development of a formula for the determination, by means of the above method, of the relative proportions of butter and other fats contained in mixtures, and shows that, assuming 12.5 cc as the lowest titre of genuine butter, adulterations exceeding 10 per cent. can thus be detected.

GUSTAVE BISCHOF (idem, 18, 73) describes his experience in the determination of minute traces of lead by the colorimetric process of Pelouze, by sulphuretted hydrogen in acid solution. Hydrochloric acid, or even a not very considerable excess of acetic acid, causes the colorimetric process to give too low results, the difficulty in the first case being that plumbic chloride is not readily decomposed by hydric sulphide. The author overcomes this difficulty by the following modification: Suppose the residue from the evaporation of a mineral water to be under examination; it is ignited and dissolved in the least possible excess of absolutely pure hydrochloric acid, filtered, and the filter washed. Hydric sulphide in moderate excess is then passed through (the author found 30 bubbles generally sufficient), the solution is treated with the least possible excess of ammonia, and then of hydrochloric acid. The advantages of this method are : first, that the ammonium sulphide completely decomposes the plumbic ehloride, and secondly, the excess of acid is nearly constant, both as to quantity and quality. A solution to which 10 drops of hydric ehloride had been added, and which gave too low results by the old process, gave by the modified method 0.0000096 grm. instead of 0.00001 grm. lead. In certain cases it is best to shake the liquid until the plumbic sulphide collects into flakes, then to filter and, after dissolving the precipitate in a few drops of concentrated hydrochloric acid, to proceed as above. As cupric sulphide is not easily soluble even in hot concentrated hydric chloride, this mode of procedure affords a qualitative test of the nature of the precipitate. The standard of colorimetric comparison adopted, is a solution of planibic acetate, of which 1 cc contains 0.0001 grm. lead, diluted to  $\frac{1}{10}$  before use.

L. KLEIN (idem, 18, 76) describes his experiments, made at the suggestion of Prof. Classen, in the determination of carbon in cast iron. The author employed the modification of the Fresenius' or Kolbe's apparatus, recommended by Prof. Classen in his paper on the determination of carbonic acid (idem, 15, 288), the chief advantage of which, over the apparatus of Ulgren, consists in a better construction of the cooler, which is capable of condensing the greater part of the water and acid vapors, so that the complete drying of the carbonic acid may be effected by a single U tube containing glass balls moistened with concentrated sulphuric acid.

The iron is dissolved in the solution of cupric-ammonium chloride, recommended by Creath (Engineering and Mining Journal, 23, 168), and prepared by dissolving 346 grammes of cupric chloride and 214 grammes of ammonium chloride in 1850 cc water; 1 gramme of iron requires 20 to 25 cc of the solution. With iron containing the carbon chiefly in the graphite modification, the solution may be effected in a few hours; iron in which the carbon is chemically combined, requires more time. The carbon is collected on an asbestos filter, washed with concentrated hydrochloric acid and then with alcohol, until the last trace of chlorine is expelled. The filter and its contents are then placed in a flask of about 200 cc capacity, provided with a strong and narrow neck, and first, crystallized chromic acid (about 6 grins, to 1 grm, iron), and then sulphuric acid (2 parts acid to 1 of water) is added. The apparatus is put together and gently warmed, a slow and regular current of air being caused to pass through to prevent stoppage of the funnel tube. The heat is gradually raised to boiling, and maintained at this temperature about  $\frac{1}{4}$  hour, to expel the dissolved carbonic acid. When the soda lime tubes have become entirely cooled, they are disconnected from the apparatus and weighed. The analyses made by this method show slightly more carbon than when the determination is made by combustion in oxygen gas. The method with chromic acid possesses the advantages of ease and rapidity of execution over the latter.

ED. DONATH (idem, 18, 72) describes his method of detecting the presence of alkaline monochromates, and of free chromic acid in solutions of alkaline bichromates. When a neutral solution of manganous sulphate is added to a solution of potassic monochromate, the color of the latter changes from yellow to yellowish-red, and on heating, a heavy, crystalline, blackish-brown precipitate is formed (Mn<sub>s</sub>CrO<sub>s</sub> +2Aq., C. Freese Pogg. Annalen, 140, 253). This precipitate does not form in solution of potassic bichromate. If, therefore, to a boiling solution of potassic bichromate, a drop of moderately concentrated solution of manganous sulphate be added, there will be formed the before mentioned precipitate if monochromate be present. To detect bichromate in the presence of much monochromate, a few cubic centimeters of a moderately concentrated solution of sodic hyposulphite is heated to boiling, and an equal volume of the boiling solution to be tested is mixed therewith; the presence of bichromate is then revealed by the formation of a precipitate, or at least turbidity, from formation of the brown chromic superoxide (Cr.O.H., E. Wintz, Ann. Chem. Pharm., 160, 370). Potassic bi- and trichromates do not separate iodine from potassic iodide; if, however, free chromic acid be present, iodine is separated and may be detected by the color imparted to a few drops of carbon bisulphide shaken therewith.

DR. JACOB GROSSMANN (idem, 18, 79) describes the method employed by him in the *determination of sulphites and hyposulphites*, such determinations being frequently useful in the study of the intermediate products of the soda manufacture. The principles on which the method is based, are as follows: 1st. Titration of the sulphites and hyposulphites with iodine and starch. 2d. Oxidation with iodine or bromine, and determination of sulphuric acid. 3d. Determination of the sulphuric acid originally present, and its deduction from the amount obtained after oxidation. If sulphites are present, the solution is first treated with CdCO<sub>3</sub>. In calculating the results of the analysis, the following factors are employed :

A = grammes of iodine required in the titration of the unit of weight.

B =grammes  $Na_2SO_4$  formed by oxidation.

According to the author, the method yields exact results.

**Prof.** BRONNER (idem, 18, 81) recommends the use of the Hempel's lamp (idem, 16, 454) for the performance of the operation of *cupellation as a lecture experiment*. By the use of a current of oxygen, the time required can be reduced to, at most, a quarter of an hour.

The following notices are condensed from the reports on the progress of analytical chemistry, by H. Fresenius and C. Neubauer (Fres. Zeitschrift f. anal. Chemic, 18, 85 et seq.).

W. WEDDING (Verhandl. d. Vereins zur Beförderung d. Gewerbefleisses, 1878, p 104) has constructed an apparatus by which the volume of solids can be determined in cubic millimeters. The weight of the body in milligrammes, divided by its volume in cubic millimeters, gives directly the specific gravity. The apparatus consists of a cylinder of steel, with a cover, the projecting rim of which is cut with the threads of a screw, fitting accurately on the threads cut on the cylinder. The circumference of the cover is divided into 100 parts, permitting by means of a vernier the reading of 1 of a revolution of the cover. The cover bears on its lower surface a cylinder of hard rubber accurately fitting the steel cylinder and terminating in a cone, through the centre of which a narrow glass tube passes, projecting above the cover. If the apparatus be filled with a liquid, such as mercury, and the cover be screwed down, the mercury will rise in the tube. If the number of revolutions required to bring the mercury to a mark on the tube, be noted, and if the cover be removed, the body to be examined introduced and the cover screwed down until the mercury again stands at the mark, the difference between the number of revolutions required in the two cases, will permit the deduction of the volume of the body introduced. The method is rapid and exact, and was used by the author in determinations of the specific gravity of prismatic gunpowder.

A. C. CHRISTOMANOS (Berichte der deutsch. chem. Gesellschaft, 10, 782) has attempted to determine the *specific gravity of iodine trichloride*, by weighing at a very low temperature in an atmosphere of chlorine and then in one of carbonic acid. The method is attended with many sources of inaccuracy.

T. BRUGNATELLI (Gazz. Chim. Ital., 1878, p. 16) effects the *rapid* evaporation of liquids by having lateral openings in the flask in which the evaporation is effected; nuder equal conditions a single tubulature increased the evaporation 30 per. cent., two opposing tubulatures, 50 per cent.

G. QUINCKE (Pogg. Annalen, 160, 118) has made interesting studies on the *imperviousness of glass to gases*. Hydrogen and carbonic acid enclosed for 17 years under pressure of 40 to 120 atmospheres, in glass vessels with walls 1.5 mm thick, showed no perceptible transfusion.

MUNSCHEID (Berg und Hüttenm. Zeitung, 37, 148) has devised a small blowing machine, worked by a treadle or small water-motor, which is sufficient for the fusion of about 1 kilo. steel, bronze, cast or even wrought iron. The machine with the gas furnace belonging to it is furnished by Ludwig, Löwe & Co., of Berlin, at from 172 to 182 marks (\$43 to \$45.50).

ALEX. C. THOMSON (Chem. News, 36, 97) uses air saturated with gasoline as a substitute for gas in the blast lamp.

O. LOHSE (Ann. d. Phys. u. Chem. [N. S.] 2, 479) has succeeded in obviating certain difficulties in the *employment of oil gas in the blast lamp.* He finds it to yield a hotter flame than coal gas.

C. LE NEVE FOSTER (Mineralog. Magazine, 1876, No. 1) recommends a *traveling blowpipe lamp* with wax filling.

G. L. ANDREAE (Ann. d. Phys. u. Chem. [N. S.] 4, 614) describes an exceedingly delicate temperature regulator for use with gas lamps. The mechanism whereby the flow of gas is increased or diminished, is similar to that in the well-known Bunsen, Kemp and similar regulators, the rise or fall of a mercury column being used to enlarge or diminish the aperture through which the gas passes. The point of novelty in Andreae's apparatus lies in the means of influencing the height of the column by variations in temperature. The apparatus resembles an inverted syphon partly filled with mercury. The longer limb is fitted with the usual tubes for the passage of the gas, the shorter limb is drawn out to a point, a small quantity of a volatile liquid whose boiling point lies near the required temperature, is introduced through the point, and the end sealed by melting. The liquids most suitable for temperatures under 110° are ether, methylic alcohol, aceton, certain of the lighter petroleum oils, and water. As the maximal tension of the vapor of a liquid at temperatures near its boiling point is very great, the Andreae regulator is extremely delicate. Using ether, the author maintained the temperature of a water bath constant within 0.04° C. for 5 or 6 hours during the day time. In the evening, the pressure of gas being doubled, the temperature rose about .04 to .05° C.

RUDOLPH GUNSBERG (Dingler's Journal, 228, 457) describes a simple form of colorimeter, which, however, embodies no features of especial novelty.

## 92 REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY.

THORE (Mondes, [2] 42, 587) describes a new succharimeter. Between the polarizing and analyzing Nichols there is placed a thin parallel-sided section of selenite, cut from a twin crystal and cemented with Canada balsam between two parallel-sided glass plates, and fastened to a diaphragm so that the round opening of the latter is halved by the twinning plane of the crystal. The polarizing prism is so placed that its principal section forms an angle of 45° with the twinning plane of the selenite. If now the analyzing prism be turned at right angles to the polarizing prism, both halves of the field of view will appear of the same tint. This is the zero point. If the analyzer be moved as little as 20 minutes from its position, the equality of tint disappears. If the prisms are adjusted to zero, and the substance to be examined be inserted between the analyzer and the gypsum plate, the analyzer must then be rotated to a degree commensurate with the circular polarization of the substance in question, before the two halves of the field of view again appear of the same tint.

ED. DONATH (Dingler's polytech. Journal, 220, 351) has investigated the question of the presence of tarry ingredients in commercial ammonia. Wittstein (idem, 213, 512) has stated that the bases of the aniline series are present therein, and he, as also Kupferschläger, (Bull. Soc. Chim. de Paris, [2] 23, 256) recommended the test of the occurrence of a reddish or brownish color when the ammonia is saturated with moderately dilute nitric acid. A sample of impure ammonia was tested by the author. Treated with an excess of nitric acid, it showed an inconsiderable brownish-red coloration; with an excess of sulphuric acid, a rose red, and with hydrochloric acid, the same color appeared after some time on the surface, the intensity increasing with the time. The sulphuric acid solution showed a peculiar sharp smell, resembling that of commercial napthalin, and possessed reducing properties : 50 cc of the ammonia, after supersaturation with dilute sulphuric acid, decolorized 3.8 cc of a potassic permanganate solution, of which 1 cc corresponded to 0.00449 grm. Fe. Such ammonia is unfit for analytical use, and for the preparation of fine reagents. The author recommends that commercial ammonia should always be tested by saturation with sulphuric acid and addition of permanganate solution. A solution of pure ammonic sulphate does not decolorize this reagent. The author tested the ammonia in question for aniline and toluidine, with negative results. In view of the statement of Bailo (idem, 202, 380 [1871]), that the odor of commercial naphthalin is due to the presence of leucoline oil, as well as of the fact that the preparation of sulphonaphthalic acid is attended with the production of a rose red color, the author is inclined to assume that the impurities in the ammonia are bases of the leucoline series.

HEREMAN W. VOGEL (Berichte der deutsch. chem. Gesellschaft, 10, 1638) in an interesting paper on *the influence of light on chemical* processes, especially on oxidation, contests the views of P. Chastaing (Fres. Zeitschrift f. analyt. Chemie, 17, 205).

G. CIAMICIAN (Sitzungsber. der Wiener Akademie, 1878, p. 158) has investigated the effect of pressure and temperature on the spectra of vapors and gases, including most of the volatile metalloids and the metals sodium and mercury. The spectra of the elements of the halogen group were greatly affected, both as regards the number and intensity of the lines, by changes in these conditions. In the case of every element, the different spectra formed at different degrees of pressure may be termed partial spectra, and from these the complete spectrum may be constructed. Some lines, however, prove to be invariable and, as they are different in the case of nearly every one of the three elements, chlorine, bromine and iodine, constitute the characteristic lines of the element in question. Through the study of the variable lines the following conclusions as to the relations existing between the partial spectra of the different elements are attained. The spectrum of rarified bromine vapor approaches that of chlorine more closely as the rarification of the former progresses, while that of compressed bromine vapor may best be compared with the spectrum of iodine. Conversely, iodine gives a spectrum corresponding to that of bromine only when moderately rarified iodine vapor is employed; if the rarification be carried very far, the resemblance to bromine diminishes, and the similarity to the chlorine spectrum becomes apparent. The spectrum of strongly condensed iodine vapor cannot well be compared with that of any of the other halogens. On the other hand, chlorine gives by compression a spectrum which corresponds well with that of moderately rarified iodine vapor, while the spectrum of the rarified gas cannot be compared with that of any other of the halogens. If the different lines occurring in the different partial spectra of each of these elements, be combined to a complete spectrum, the three spectra so obtained correspond perfectly with each other in all their lines, the wave lengths of the homologous lines diminishing from chlorine to iodine. In regard to the widening of the lines in the spectra of the halogens, it results that, during the compression of a gas or vapor, the lines which predominate during a certain degree of pressure appear, without regard to their intensity,

the least widened and faded, while the lines most liable to alteration at the pressure and temperature in question, experience the greatest widening.

The behavior of the spectra of different elements at high pressure is very diverse. From the comparison of the investigations of the author with those of Wällner, it results that the variations by high pressure and temperature are greatest, and take place most readily, in the case of the spectra of those elements (H, Na, O, Cl, Br, Q) which are characterized by the possession of a large chemical vis viva, while those elements which possess a weaker affinity give the spectra least subject to variations, and these variations consist in the widening of the lines and the appearance of a continuous spectrum. It is to be remarked that in the case of metallic vapors (Na Hg), the widening of the lines predominates; in the case of the metalloids, the continuous spectrum.

J. L. SORET (Arch. d. Geneve, [2] 60, 298) has investigated the ultra violet absorption spectrum of different liquids. The observation is materially facilitated by the employment of the spectroscope with fluorescent eve-piece (Zeitschr. f. anal. Chemie, 13, 442), the source of light being either the sun or the electric spark between magnesium, zine and aluminum electrodes. The chlorides and sulphates of the alkalies show great transparency. The total number of lines shown by the spark from these electrodes is 32. Lavers of 1 centimeter thick of solutions of the following salts, of such strength that the unit of each contains the same quantity of chlorine, will still permit the passage of the following lines : KCl : 27 ; NaCl : 24 ; LiCl : 25 ; MgCl : 28; CaCl: 24; SrCl: 27; BaCl: 28. Water is very transparent for the ultra violet rays. In salt solutions, concentration and thickness of the absorbing layer are equivalent as regards the absorption, In many cases base and acid retain their properties in the solution; thus, aqueous solutions of nitric acid show the same absorption spectra as solutions of nitrates containing the same proportion of nitric acid in the unit of volume. Nitric acid is pre-eminent in a series of substances possessing the capacity of strong absorption of the ultra violet rays. A large number of salt solutions show absorption lines in the ultra violet spectrum ; thus, very dilute chromate and bichromate solutions show two; one between H and N, and the other between the 15. and 20. cadmium line. Cerium sulphate has one between the 17, and 23. and potassium permanganate between the 10, and 17. As Stokes has already shown, the nitrates show an absorption stripe. Didymium sulphate darkens the spectrum between N and O.

H. E. ROSCOE and T. E. THOBPE (Philos. Trans., 167, 207) communicate their observations on the absorption spectrum of bromine and iodine monochloride.

LEO LIEBERMANN (Sitzungsber. d. Wiener Akademie, 75) has found that many salts, by filtration of their solutions through animal charcoal, are not only more or less completely retained, but are, many of them, decomposed. Thus, a neutral solution will often run through acid, all of the base, and a portion of the acid having been retained by the coal.

EMANUEL BORICKY (Arch. d. naturw. Landesdurchforschung von Böhmen, III, V) has published a new process for the microchemical analysis of minerals and rocks. The method is based on the employment of hydrofluosilicic acid for the decomposition of the minerals; the silicofluorides, thus formed, being easily recognized under the microscope (power, 200-400) by their characteristic forms, or by use of other reagents. The modus operandi is as follows: A piece of the substance under examination, of the size of a pin's head, is fastened to the slide, by melting to the Canada balsam, with which the slide is coated, covered with one or two drops of a  $3\frac{1}{2}$  per cent. solution of pure hydrofluosilicic acid, and allowed to dry by spontaneous evaporation. The metal which, in the mineral, may have been combined with any acid, with oxygen, sulphur, selenium, tellurium, arsenic or antimony, is thus converted into silicofluoride, and may be identified by the characteristic crystalline form. The original contains drawings of the very characteristic forms of the silicofluorides of Ka, Na, Li, Ca, Sr, Ba, Mg, Fe, and descriptions of the other known silicofluorides. If the assay has been entirely decomposed, an approximate estimate of the relative proportions of the different constituent metals may be made by noting the relative quantities of the crystals. In the case of the determination of the alkaline metals in silicates, such as amphibole, which are not attacked by hydrofluosilicic acid, the author effects the decomposition with vapor of hydrogen fluoride. The mineral is placed on a piece of platinum foil and suspended in a platinum crucible, the bottom of which contains a layer of about + gramme of barium fluoride, just covered with sulphuric acid, and the cover fastened airtight with wax. After about two days exposure, the foil is removed, the assay boiled out with water, and a drop of the solution placed on the balsam-covered slide; on drying, the slightest trace of alkali will appear in its characteristic form. The paper is of great interest, not only to mineralogists and petrographers, but also to chemists.

SCHLAGDENHAUFFEN (Journ. de pharm. et de chimie, 1878, 373) has investigated the delicacy of the ordinary qualitative reaction for magnesia with sodium phosphate. He finds that on mixing a solution containing one-half per cent. of magnesium sulphate with a solution of sodium phosphate of equal strength, and containing ammonium chloride and hydrate, a crystalline precipitate takes place only after the lapse of half an hour. A solution containing only  $\frac{1}{100}$  of one per cent, will still give a crystalline precipitate. If magnesia is to be detected in a solution containing the metals of the alkaline earths, these latter must be removed before the sodium phosphate test is ap-The anthor has devised the following test, which can be plied. applied in the presence of the metals of the alkaline earths : Potassium or sodium hypoiodate is prepared by dissolving iodine in a two per cent, solution of the caustic alkali until the fluid has just attained a golden yellow tint. If some of this reagent be added to a solution containing one-half per cent. of magnesic sulphate or chloride, there is formed a copious brownish-red precipitate. If the magnesium solution be diluted with 5, 10, 15 and 20 times its volume of water, the precipitate will be formed in gradually diminishing quantity, until, at the last degree of dilution, a distinct reddish coloration alone appears. The immediate brown precipitate, which forms in a solution containing 0.02 per cent, of magnesic subplate or chloride, can be more easily collected than the ammonic magnesic phosphate, formed in the same solution. The brownish red precipitate or coloration, produced in a 0.01 per cent. solution, disappears after a certain lapse of time; the author, nevertheless, considers the test to be of value, on account of the rapidity with which it can be carried out. The author reserves the further investigation of the chemical nature of the magnesia precipitate.

T. M. BROWN (Iron, 1878, 361) recommends pulverized zinc for the reduction of iron in iron ores, and for the reduction of ferric to ferrous oxide. The pulverization of the zinc is effected by pounding in an iron mortar, at the temperature of  $210^{\circ}$  C., and sifting through sieves of 7, 10 and 13 meshes to the square centimeter, and finally bolting through fine cloth; 0.03 grammes of powdered iron ore are mixed with ten times their weight of finely powdered zinc, the mixture placed in a porcelain crucible and covered with an equal weight of powdered zinc. The uncovered crucible is heated for ten minutes to a dark red over the Bunsen's burner. After cooling, the crucible and its contents are placed in a strong flask, covered with hot dilute chlorhydric acid, and heated rapidly to boiling; the zinc and the reduced iron dissolve in a few minutes. The flask is then tightly corked, and, after cooling, the iron titrated with permanganate. Ores containing carbonaceous substances must be roasted before reduction. Even the gas of the lamp may introduce organic matter, and cause the solution of the reduced mass to be dark in color. The remedy for this consists in the use of a thicker layer of zinc, or a cover of powdered borax, during the ignition. Powdered zinc has great advantages over granulated or sheet zinc for the reduction of solutions of ferrie to ferrous oxide.

OTTO LINDEMANN (Inaugural Dissertation, Jena; Goslar, E. Brückner, 1878) has devised a new method for the volumetric determination of vanadic acid. The method is based on the following reaction, which takes place when soluble vanadates, or vanadic anhydride dissolved in sulphuric acid, are added to a solution of ferrous sulphate, acidified with sulphuric acid or hydrochloric acid:

$$2\text{FeO} + \text{VO}_5 = \text{Fe}_2\text{O}_3 + \text{VO}_4.$$

One grm. iron is, hence, equivalent to 1.630357 grms. VO<sub>5</sub>. The author employs a standard solution of ferrous ammonic sulphate, strongly acidified with sulphuric acid, the titre of which is determined every time before use, by means of deci or centi-normal solution of potassic bichromate. The usual mode of conducting Penny's process is reversed, the ferrons solution being added gradually to a measured volume of the bichromate solution, in order to make the manipulation accord with the vanadic acid titration. The vanadic acid solution is weakly acidified with sulphuric or hydrochloric acid, and properly diluted (especial attention must be paid to the freedom of the acid last named from Cl or So.). The ferrous solution is then added from the burette, under constant stirring, until the indicator (potassic ferricyanide, used as in Penny's well known process) shows the reaction to be complete. When sulphuric acid is used, the color of the solution changes gradually from yellow to the light blue of vanadium tetraoxide, which does not, however, interfere with the determination of the end reaction. Hydrochloric acid solutions appear light green.

The experimental results of the author demonstrate the accuracy of the method, and show that the presence of ferric oxide, alumina, chromic oxide, alkaline chlorides and ammonium salts, is without influence thereon. The author recommends the following method for the detection of vanadium: About 5 grammes of the finely pulverized substance is mixed with two to three parts of anhydrous sodium carbonate, and the same quantity of flowers of sulphur, and heated in a covered porcelain crucible, over the lamp, until the mass

is entirely melted, and no more sulphur sublimes. When cold, the mass is leached out with water, and the reddish-brown, filtered solution treated with a slight excess of dilute sulphuric or hydrochloric acid. If the separated sulphur has a brownish color, the presence of vanadium is probable. The dried precipitate is roasted in a porcelain erucible, with free access of air, and the small quantity of darkcolored residue, which will be left if vanadium is present, is either warmed with a few drops of concentrated sulphuric acid, or it is melted with some sodic carbonate and potassic nitrate, the melted mass dissolved in a little water and slightly acidified. In both cases the presence of vanadium is indicated by the characteristic red coloration on addition of hydrogen peroxide. The author endcavored by this method to effect a quantitative separation of the vanadium, but failed, owing to the fact that the decomposition of the mineral by melting with sulphur is not complete, and that from a solution of a vanadium sulphur salt it is impossible to separate all of the vanadium by addition of acids.

The old method of melting with alkaline carbonates and nitrates gives satisfactory results, but requires special precautions : Thus, about 25 grms, of the extremely finely pulverized substance is mixed with concentrated nitric acid to a thin paste; this is digested some time on the water bath, then carefully neutralized with solution of sodium carbonate, mixed with equal parts of potassium nitrate and potassic sodic carbonate, and the mass dried with stirring. The fusion may be effected in a wrought iron crucible over the blast lamp. The decomposition may also be effected when the ore, mixed with four times its weight of potassic sodic carbonate, is melted, in a platinum vessel, for half an hour in the nuffle of an assay furnace. The melted mass from either of the foregoing operations, is softened in hot water, and washed until the alkaline reaction disappears from the washing water (a troublesome and tedious operation). The liquid is evaporated with excess of hydrochloric acid, the silica separated in the usual manner, the filtered solution warmed to 60°-70° C., saturated with hydrogen sulphide, allowed to stand 24 hours in a moderately warm place, and filtered. The bluish filtrate is evaporated to a small volume, oxidized with potassium chlorate, evaporated until the chlorine odor disappears and the salts commence to crystallize out, water added until the salts again dissolve, and then a slight excess of ammonium hydrate to destroy every trace of free chlorine, the liquid acidified with hydrogen chloride, and titrated with ferrous solution, as already described.

In this manner the author found 0.237, 0.216 and 0.224 in the mean, 0.226 per cent. of vanadic acid in the pisolitic limonite, from the neighborhood of Salzgitter.

F. W. CLARKE (Am. Journal of Science, 16, 200) communicates the results of his experience in the *electrolytic estimation of mercury*. The author connected a platinum dish, containing a solution of mercuric chloride, acidified with sulphuric acid, with the zinc pole of a six-cell Bunsen's chromic acid battery, the positive electrode consisting of a strip of platinum foil dipping into the liquid. At first mercurous chloride precipitated, but within an hour was completely reduced, ammonia producing no turbidity in the supernatant liquid. To prevent the corrosion of the mercury the solution was removed and replaced with water, without interrupting the current. When, by repatition of this treatment, the liquid had nearly lost its acid reaction, the current was interrupted, the metal thoroughly washed, dried and weighed. The results of two experiments were 73.76 and 73.85, calculated 73.80 per cent. of mercury. Similar experiments by the author on cadmium yielded an unsatisfactory result.

H. VON JUPTNER (Ber. der Wiener Akademie, 1878, 161) recommends the following process for the separation of gold and silver and their determination in alloys. The assay is alloyed with five to eight times its weight of zinc, and dissolved in nitric acid. There remains, undissolved, if present, gold, the platinum metals, and some tin as oxide. The alloy can be made over a Bunsen's burner, or even over an alcohol lamp. To prevent oxidation, it is best to cover with rosin, and to avoid a too high temperature. If neither tin nor platinum metals are present, the gold may be ignited and weighed. If the presence of platinum metals or tin is suspected, the metal is dissolved in aqua regia, precipitated with ammonium chloride, the free chlorine expelled by boiling, the gold reduced with a known volume of standard ferrous-ammonium sulphate solution, and the excess of the latter determined with permanganate. From the amount of the ferrous solution consumed, the amount of gold reduced may be calculated. The author obtained the following results :

By fire	assay,	29.4 §	gold;	by the	author's	method,	29.8	per cent.	gold.
"	"	43.1	"	"	"	"	43.1	"	"
"	"	53,55	"	"	"	"	53.6	"	"
"	"	58.3	"	"	"	"	58.25	j 44	"

LEO LIEBERMANN (Ber. d. deutsch. chem. Gesellschaft, 11) has found that sulphur is, to a considerable extent, soluble in warm concentrated acetic acid, from which it separates on diluting with water.

99

## 100 REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY.

E. A. GRETE (Ber. d. deutsch. chem. Gesellschaft, II, 557) has studied the behaviour of nitric acid when heated, under various conditions, with soda line. An attempt to promote the reduction and formation of annonia by addition of zine and iron filings, did not succeed, even when much organic matter was present. The author, however, found the xanthogenic salts, or those which, when heated, evolve hydrogen sulphide in statu nescendi, are adapted to the purpose. The reduction of pure potassium nitrate, mixed with xanthogenates and soda lime, was so perfect, that almost the exact amount of nitrogen was obtained as ammonia. The author promises further contributions, especially in relation to the reduction of the organic nitro-compounds.

LORIN (Pharm. Zeitschrift für Russland, 17, 372) has found that the action of oxalic anhydride on the different sugars may serve as a means of determining their chemical character. While duleite, mannite, quereite and all sugars of alcoholic nature, furnish, when treated with oxalic anhydride, formic and carbonic acids, cane sugar, glucose, milk sugar and sorbine show an essentially different behaviour. Cane sugar and sorbine blacken easily, glucose and milk sugar less so, and the effect is purely destructive. The author subjected inosite to this reaction, and finds that it belongs to the class of polyatomic alcohols, and stands next to quercite. The reaction serves for the detection of a mixture of cane sugar with milk sugar, for while a mixture of milk sugar and oxalic anhydride remains white in the water bath, the presence of one per cent. of cane sugar causes distinct blackening.

E. SCHMIDT (Pharm. Centralhalle, 1878, 258) has established the *identity of mercurialine* (CH<sub>5</sub>N) with methylamine, and thus, for the first time, established the fact of the occurrence of the latter in the vegetable kingdom.

DRAGENDORFF (Archiv der Pharm., 9, 209) describes a new brucine reaction with chromic acid. It is well known that brueine dissolved in concentrated sulphuric acid, and treated with potassic bichromate, gives a dark green solution, which is not obtained when the sulphuric acid solution is first treated with nitric acid, and the color thus produced allowed to disappear before the bichromate is added. It is also known that potassic bichromate produces in aqueous solutions of brueine sulphate, a crystalline precipitate, and that a similar crystalline mass is obtained by pouring a dilute solution of chromic acid over brueine. To produce the new reaction, brueine is dissolved in a mixture of one volume of pure sulphuric acid and nine volumes of water; if, by means of a glass rod, there be introduced into this colorless solution, a small quantity of a very dilute aqueous solution of potassic bichromate, the entire liquid is colored for a few seconds, of a beautiful red, then changing, first to a reddish orange, and then to brownish orange. Warmth promotes the reaction, the best temperature being the ordinary temperature of the room. The reaction is very delicate, a solution of 1 in 1,000 shows, at once, a color similar to that of raspberry juice, a solution of 1 in 10,000 shows a very distinct coloration. The reaction is one of oxidation.

H. G. H. FENTON (Berichte d. deutschen chem. Gesellschaft, II, 1263) has investigated the *action of hypochlorites on urea*. If urea be treated with a hypochlorite in the presence of caustic alkalies, only half of the nitrogen is set free. The residual nitrogen exists as cyanate, the reaction being

 $2N_{2}H_{4}CO+3NaClO+2NaOH=2NaCNO+N_{2}+3NaCl+5H_{2}O.$ If, however, alkaline carbonate be employed, all of the nitrogen is set free, and the author finds that this method of determining urea, is decidedly more accurate than that with hypobromites.

F. MUSCULUS and D. GRUBER (Zeitschr. f. physiol. Chemie, 2, 177) have studied, anew, the action of diastase, or dilute sulphuric acid, on starch. According to the duration of the action, the following bodies were obtained :

1. Soluble starch. Insoluble in cold water, soluble at 50-60° C. In solution, is colored by iodine wine-red, blue when dry. Specific rotation of  $\alpha = +218^{\circ}$ . Reducing power, 6.

2. Erythrodextrine. Distinguished from the preceding by solubility in water. In solution or dry is colored red by iodine. Has not yet been obtained in a state of purity.

3. Achroodextrine  $\alpha$ . Not colored by iodine. Rotation,  $\alpha = +210^{\circ}$ . Reducing power, 12. Less readily changed by diastase than the two preceding.

4. Achroodextrine  $\beta$ . Rotation of  $\alpha = +190^{\circ}$ . Reducing power, 12. Not acted on by diastase.

5. Achroodextrine  $\gamma$ . Rotation of  $\alpha = +150^{\circ}$ . Reducing power, 28. Unacted on by diastase.

6. Maltose. Formula  $C_{12}H_{23}O_{11} + H_2O$ . Rotation of  $\alpha = +150^{\circ}$ Reducing power, 66. Fermentescible. Unacted on by diastase.

7. Grape sugar. Formula  $C_6H_{12}O_6 + H_2O$ . Rotation of  $\alpha = +56^\circ$ . Reducing power, 100. Fermentescible.

The figures for the rotatory power of the dextrines are only approximative, but show that it diminishes with the progress of the

saccharification. The authors assign to starch the formula n  $(C_{12}H_{20}O_{10})$ , n being not less than 5 or 6.

F. HESS and J. SCHWAB (Berichte d. deutsch. chem. Gesellsch., 11, 192) have investigated the question of the amount of *nitrogen in nitroglycerine*. They have shown that the denitration of nitrocompounds of the ether class with alcoholic potash solutions, is attended with a danger of loss of nitrogen, and have thus been led to give preference to the method of Dumas & Stas (Ann. de chim. et de phys., 73, 148). As the result of numerous closely accordant determinations, they conclude that not all of the nitroglycerine of commerce is pure trinitrine.

A. SCHEURER-KESTNER (Compt. rend., 86, 1024) communicates the following method for the estimation of tartaric acid in crude The occurrence of neutral calcic tartrate in crude argols, and argols. the advance of the commercial value of this substance, render insufficient the old method of determination of the percentage of potassic bitartrate by titration with alkalies. It is necessary to calcine the argols and titrate the alkaline carbonate with a normal acid. The tartrate of lime is determined by solution in hydrochloric acid and precipitation with caustic potassa or soda. This method is exact only in the comparatively rare case of the absence of calcic sulphate, which would form with the potassie tartrate, potassic sulphate and calcie tartrate. The method of calcination, solution in water and titration of the solution for potassie, and the residue for calcie curbonate, is also open to the objection of double decomposition between the calcie sulphate and potassic carbonate. The method of the author consists in the estimation of the potassic bitartrate by titration, and the determination of the collective tartaric acid by solution of the argols in hydrochloric acid, neutralization with sodic carbonate, precipitation with calcium chloride, filtration, washing, ignition and weighing of the precipitate as calcium carbonate.

FRANZ KATHREINER (Dingler's polyt. Journ., 227, 481), for the purpose of answering the question, which method of tunnin estimation is to be recommended for general acceptance in the leather manufacture, has subjected to a careful comparative investigation the methods of Carpene-Barbieri (Zeitschr. f. anal. Chem., 16, 123), Löwenthal (idem, 16, 33), Hammer, Clark and Jean (idem, 16, 123). The method first named is attended with difficulties which preclude its employment in practice. The author recommends the method of Löwenthal as being rapid, easy and affording satisfactory results. In carrying out the method special attention must be paid to the following points :

## REPORT ON THE PROGRESS OF ANALYTICAL CHEMISTRY. 103

1.) The permanganate solution should not contain more than from 1 grm. to 1.333 grm. to the litre.

2.) The indigo solution should not be stronger than 20 cc to 10 cc of permanganate, and must be filtered and at once treated with the requisite quantity of acid.

3.) The salted glue solution must be kept well corked, and be filtered previous to use.

4.) The concentration of the tannin solution, to be tested, must be such, that the portion taken for titration shall not require more than  $\delta$  cc of the permanganate solution of 1.333 grm. to the litre, or 8 cc of that of 1 grm. to the litre. There must be added so much indigo solution that at least  $1\frac{1}{2}$  times as much permanganate is consumed, as in the preceding; in the first instance, therefore, 9 cc; nor should this quantity be greatly exceeded. The quantity of dilute sulphuric acid (1:5) required, under the foregoing conditions, to retain the manganous oxide in solution, is 10 cc.

5.) The duration of the titration, under these conditions, is about 4 minutes for the original solution, 6 for the "filtrate."

6.) It is desirable to compensate for the presence of oxidizable substances in the glue, in the final titration of the "filtrate" with permanganate.

The other methods named are all open to objection, as far as employment in practice is concerned.

C. HIMLY (Chronik d. Univers. Kiel, v. J. 1876; Kiel, 1877, 18) employs the following method of *testing water for bacteria*. The flask used in the experiment, is washed out with concentrated sulphuric acid, and then thoroughly with the water under examination. A small quantity of extract of meat is dissolved in a considerable quantity of the water, and the solution boiled for  $\frac{1}{4}$  hour. The flask is filled to about  $\frac{3}{4}$  with the water, to which a small quantity of the extract solution has been added, closely corked with a rubber stopper which has previously been kept in absolute alcohol, and the whole kept for 24 to 48 hours at a blood-heat. The degree of turbidity, and the thickness of the scum on the surface, produced by the development of the bacteria, indicate the proportion originally present in the water. A control experiment should be made with distilled water, which should show no turbidity.

C. H. WOLFF (Pharm. Ztg., 23, 532) has investigated the spectroscopic reactions of ergot (secale cornutum), with special reference to the detection of that substance in flour; using the extract obtained by shaking the flour with acidified alcohol or ether. The darkcolored solution, employed in layers 2 to 3 cm thick, shows extinction of the refrangible part of the spectrum nearly up to D; solution of light color show a lighter color in the parts previously absorbed, and three distinct absorption lines, somewhat faded on their margins, of which two, very characteristic ones, lie in the green, and a third, weaker line, in the blue. The observations were made with an apparatus provided with a single flint glass prism of  $60^{\circ}$ , and the positions of the lines were as follows :

I. II. III. Prismatic spectrum, D64E—D97E E46F—E83F F30G. Typical spectrum, wave-

lengths in 1000.000 mm, 506.5 - 492.3546.8-528.5 466.70.1 grm. of powdered secale cornutum digested with 5.0 grms. ether, and 5 drops of a mixture of 1 part sulphuric acid and 3 parts alcohol, showed, in a layer 5 cm thick, the characteristic lines in the green, when examined with a delicate pocket spectroscope. E. Hoffman having shown that the spectrum of ergot resembles that of a faded chlorophyll solution, the author was led to make a comparison, with similar results. To separate the coloring matter peculiar to flour, the latter is extracted with ether alone before extracting with acidified ether. The method of Hoffman (Pharin, Ztg., 23, 726 and 742), consisting in the extraction of the flour or bread with acidified ether, and the treatment of the filtered etherial solution with a few drops of a saturated solution of sodic bicarbonate, is especially well adapted to the detection of ergot,  $\frac{1}{5}$  to  $\frac{1}{10}$  per cent, of the latter causing the bicarbonate solution to show a distinct violet tint. In the case of bread, the digestion must be continued for at least 24 hours, with continual shaking.

W. RIGGS, Jr., and R. GIEBERMANN communicate a translation of the methods of determination of silicon and phosphorus in iron and steel, employed by ANDREW A. BLAIR, chemist to the U. S. Board, appointed to test iron, steel and other metals. (REPORT OF A. A. BLAIR, CHEMIST TO THE BOARD, TO J. J. S. LARDLEY, COLONEL OF ORDI-NANCE, PRESIDENT.) The paper being generally accessible to American readers, and not adapted for abstraction, need not be reproduced here. The practical directions for the conduct of these difficult and delicate determinations, are exceedingly full and explicit, and the paper is one of much interest to analytical chemists.

W. DITTMAR (Dingler's polytech. Journal, 221, 450) employs the tollowing method for the disintegration of chromic iron ore: 0.5 grm.

of the finely pulverized ore is mixed with 5 to 6 grammes of a flux, prepared by melting 2 parts glass of borax with 3 parts sodium, potassium carbonate, and heated to redness for 5 minutes in a covered platinum crucible over the Bunsen's burner. The crucible is then uncovered, placed in an inclined position, heated to the highest temperature the lamp will give, stirred with a platinum wire until the ore is completely dissolved, and the heat continued for about threequarters of an hour longer. The mass is then placed in water, heated on the water bath, with addition of a few drops of alcohol to reduce any manganates which may be present, and the heat continued until the excess of alcohol is expelled. The solution is filtered, the residue washed, and the filtrate, which contains all the chromium of the ore, as alkaline chromate, is treated by the usual methods for the determination of chromium.

JAILLARD (Journ. de pharm. et de chim., 1878, 205) communicates the following method for the detection of adulterations in oil of geranium. The adulterations consist usually of fatty oils, hydrocarbons, especially oil of copiava, etc. The test consists in shaking 6 drops of the oil in 5 cc 70 per cent. alcohol. The pure oil gives, on shaking, a clear solution; the impure oil a turbid mixture.

P. GRIESS (Ber, d. deutsch. chem. Gesellsch., II, 629; compare, also, Zeitschr. f. anal. Chem. 17, 629) has discovered that metadiamidobenzole (fusing point 63°) is an exceedingly delicate reagent for nitrous acid, 10 of a milligramme in a litre being distinctly recognizable by the yellow coloration imparted to this reagent. The metadiamidobenzole is dissolved in an excess of dilute sulphuric acid, and decolorized with bone black. The solution thus prepared may be kept for months in well closed bottles, without perceptibly darkening in color. The author used this reagent to decide whether the substance found in the saliva, mucus, etc., and which blues a mixture of potassic iodide and starch, is nitrous acid, as Schünbein states, or hydrogen peroxide, as Meissner supposed. He finds the substance in question to be nitrous acid. By comparison of the intensity of color, the reagent may serve for a quantitative method. The author found in his own saliva the proportion of nitrous acid to be 1 mgr. to the litre; ten times this amount was found in the saliva of a 14-year-oldboy.

A. CASALI (Centralbl. f. d. med. Wissensch., 1878, 583) has found that the changes of color produced by oxidation, afford a characteristic reaction for glycochollic acid, and has based thereon a method for the detection of gall in urine. By oxidation with ferric or antimonic chloride, or barie or plumbic peroxide, a solution of glycochollic acid passes through the following series of color changes : Yellow, red, wine-red, violet, and finally a rather permanent bluish violet. The last named coloring substance may be isolated with ethylic or amylic alcohol, and is an extremely beantiful purple, resembling unrexide. In testing for gall in arme, the latter is precipitated with a solution of basic plumbic acetate and anumonia, the precipitate collected and shaken with ether, the etheriai extract is divided into three portions, which are evaporated in separate porcelain dishes. The first residue is oxidized with baric peroxide and sulphuric acid; the second is treated with stannic chloride and sulphuric acid; the third with antimonic chloride and sulphuric acid. If the urine contains gall, all three portions will show the abovementioned colors.

VAN MELCKEBEKE (Archiv. d. Pharm., 10, 172) has found that oxalic acid is produced by the destruction of animal substances with potassic chlorate and hydrochloric acid.

E. DANNENBERG (idem, 7, 2. Heft) has investigated the determination of colchicine in toxicological cases, and finds that chloroform and amylic alcohol are the best solvents for its extraction from acid solutions. In the examination of beer, the author has found it best to redissolve the first chloroform extract in water, and precipitate with tannin. The precipitate is washed, decomposed with plumbic oxide, and the pure colchicine extracted with dilute alcohol.

RICHARD GODEFFROY (Ann. d. Chem. u. Pharm., 181, 176) has redetermined the atomic weight of caesium and found it to be 132.557, or, in round numbers, 132.6. Bunsen found 132.99; S. W. Johnson and O. D. Allen 133.036; and Mercer, the same. The author found the atomic weight of *rubidium* to be 85.476. Bunsen found 85.36, and J. Piccard 85.41.

C. RAMMELSBERG (Jour. Chem. Soc., 1879, 18) has had occasion to investigate, anew, the *determination of lithia as phosphate*, and has obtained results identical with those from his earlier experiments (Pogg. Ann., 66, 79, and 102, 441), which show that the precipitate consists of a mixture of sodie and lithic phosphates, in varying proportions, and that the method is therefore unreliable.

C. M. TIDY (Jour. Chem. Soc., 1879, 46) communicates a voluminous and interesting paper on the determination of the organic purity of potable waters. After a critical review of the different methods

which have been proposed to this end, the author gives the preference to a special modification of the permanganate process, which he terms "the oxygen process," and which consists in adding to a known volume of the water, measured quantities, first of dilute sulphuric acid, and then of permanganate solution. The author takes two equal portions of the same water, adds equal volumes of permanganate and sulphuric acid to each, allows the one to stand one, and the other three hours. The amount of permanganate remaining is then measured by means of potassic iodide and sodic hyposulphite. The results are expressed in the quantities of oxygen required to oxidize the organic matters in one gallon of water. The relation of the result obtained in one, to that in three hours' time, permits a conclusion as to the nature of the organic matter, i. e., its susceptibility to oxidation, or of the relation between the readily oxidizable and putrescent, and the less easily oxidizable or non-putrescent matters. The conclusions attained by the author are as follows, viz.:

1st. As regards the *ammonia* process, an absolute, or nearly absolute, freedom from albuminoid ammonia is, for the most part, an indication of organic purity. Nevertheless, very impure waters sometimes give only a trace of albuminoid ammonia, and some very pure waters give large quantities of it. Its results are therefore inconsistent.

2d. The ammonia process does not allow a sufficiently large scale for the classification of different grades of purity, the extreme range between waters of extraordinary organic purity and dirty waters being only from 0.05 to 0.1 parts in one million.

3d. The *combustion* process is objectionable, owing to the necessity of evaporation, and the danger of loss by volatilization, or destruction, of the subtile organic matter specially active in producing disease. Barring this objection the process is trustworthy.

4th. The estimation of the organic nitrogen is by no means so constant, owing to the possibility of the nitrate being incompletely reduced, of impurities in the reagents, and necessary errors of experiment and, hence, untrustworthy, unless the organic nitrogen be beyond a certain quantity.

5th. That the oxygen process, when properly controlled by the natural history, and general physical and chemical examination of the water, indicates with sufficient precision the comparative quantity of organic matter present, likely to be injurious to health. That the results of the process are of extreme delicacy, admitting of a wide scale of classification, and permitting a sharp distinction to be drawn between putrescent or readily oxidizable and probably pernicious organic matter, and that which is probably harmless, as regards its action on the human body.\*

A. DUPRÉ and H. W. HAKE (Journ. Chem. Soc., March, 1879, 159) communicate two new methods for the gravimetrical determination of minute quantities of carbon. The first process consists in combistion with oxygen gas and oxide of copper, the products of combustion being caused to pass through a Pettenkofer's tube filled with baryta water. At the end of the combustion, the precipitated baric carbonate is collected, converted into sulphate and weighed. The analytical tests of the authors show the method to be very exact, and they consequently recommend it for the determination of carbon in potable waters. The second method, which the authors term a new Nephelometrie ( $v \varepsilon \varphi \varepsilon \lambda \eta = a$  cloud) method, rests upon the observation of the varying degrees of turbidity caused by the action of varying quantities of carbonic anhydride in a two per cent. solution of basic plumbic acetate. The method is employed in water analysis, as follows : The water is evaporated in a dish of silver foil (capacity, 50 cc), after the addition of an excess of phosphoric acid. When 200 cc have been thus evaporated, the dish is crushed and introduced, with the residue it contains, into a combustion tube, where the residue is burnt with oxygen and plumbic chromate. The carbonic anhydride is conducted into basic plumbic accetate; the turbidity compared, in the colorimeter, with that resulting from the combustion of a known quantity of sugar. The results obtained by the authors are very exact. They recommend the method for the determination of small quantities of carbon in certain metals, and for the determination of carbonic acid, and of volatile and suspended organic matter in air.

FERD. HURTER (Chem. News, 39, 25) communicates a simple method of estimating cyanogen compounds in soda lyes. The ones occurring in crude lyes are sodic ferrocyanide, sulphocyanide and cyanate. The two last named are unimportant, as they yield colorless products of decomposition by the further technical treatment; the former is important for the reason that it may introduce enough iron to discolor the product. The author's method is as follows: 100 cc of the strong lye is oxidized by chlorine, sodic hypochlorite, or bleaching powder solution, until the sulphides, etc., are converted

<sup>\*</sup> The foregoing paper was made the subject of an interesting discussion at the meeting of the Chemical Society, on Thursday, Feb. 6th. The discussion is reported in detail in the *Chemical News*, **39**, 67.

into sulphates, and the ferrocyanide into ferricyanides. The solution is acidified, freed from chlorine by warming, and titrated with  $\frac{1}{20}$  normal copper solution, using drops of ferrous sulphate on a porcelain plate as an indicator. The copper solution is added, until the indicator no longer shows a blue color, but the pure purple of cupric ferrocyanide, the reaction being similar to the following :

 $Cu_3Fe_2Cy_{12} + 2HCl + 2FeCl_2 = Cu_3H_2Fe_2Cy_{12} + Fe_2Cl_6$ . In the author's opinion, the method, when conducted with the indicated precautions, is not rivalled in accuracy by any other volumetric method for this purpose.

T. TATTERSALL (Chem. News, 39, 66) describes the following *test for cobalt*: If cyanide of potassium be added to a solution containing cobalt, until the precipitate first formed redissolves, and then a few drops of *yellow* ammonium sulphide, a blood-red color will be obtained, even in the presence of nickel, or any soluble cyanide except that of copper. Na<sub>2</sub>SO<sub>3</sub> or SnCl<sub>2</sub> will produce the color, but not with the same degree of delicacy.

F. W. PAVY (Chem. News, 30, 77) communicates a method for the volumetric estimation of sugar by an ammoniated cupric test, giving reduction without precipitation. In a process introduced by Bernard, a few years back, for physiological purposes, the use of a large excess of potash in a liquid containing complex organic constituents, was attended with absence of precipitation of cuprous oxide, thus giving decoloration without precipitation. The author was led to assign this peculiarity to the development of ammonia, by the action of the excess of potassa on the nitrogenous substances in the liquid. By adding ammonia to the ordinary Fehling's solution, he obtained a liquid which was decolorized without separation of cuprons oxide. The test solution is prepared by taking 120 cc Fehling's solution, 300 cc of ammonia (sp. gr. 0.880), and diluting to 1 As, in the presence of ammonia, 6 molecules of cupric oxide are litre. appropriated by 1 molecule of sugar, instead of 5, as in the case of the ordinary Fehling's solution, this solution possesses the strength of  $\frac{1}{10}$ Fehling's solution. 20 cc of this solution, corresponding to 0.010 grm. sugar, are placed in a flask, the flask adapted to the cork attached to the delivery tube of the burette containing the sugar solution, the liquid in the flask heated to ebullition, and allowed to boil a few minutes, to expel the air, when the sugar solution is added until the liquid is decolorized. It is convenient to have the sugar solution of such strength that 20 cc will about decolorize 20 cc of the test solution. The cork of the flask must, of course, have an aperture for the

escape of steam. The annuoniated solution possesses greater permanence than the ordinary Fehling's solution. Results accurate.

M. D. PENNEY (Chem. News, **39**, 80) has made a number of *determinations* of the proportion of aluminal contained in pure flour, and attains the conclusion that it is impossible to fix apon a standard of natural alumina, and that it is erroneous to regard its amount as in any degree a measure of the presence of alum in bread.

GREVILLE WILLIAMS (Chem. News, **39**, 98) communicates the results of his experiments on the use of the "orange 3" of Porrier, and the "orange 4," or "tropacoiin," as a substitute for litness as an indicator in alkalimity. The author has specially studied the "orange 3," that being the most delicate of two, and found it to be fully twice as sensitive as the litnus with which he compared it ; being sensitive to 1 part sulphuric acid in 100,000 water. The author employs 1 part of the color dissolved in 10,000 parts water, the liquid being of a full orange color. The amount of this solution ased is 0.4 to 0.2 cc. This indicator is entirely unaffected by carbonic acid.

A. DUPAE (Chemical Review, 8, 55, from <sup>6</sup> The Analyst") communicates the results of the examination of many samples of flour to ascertain the relations between the succents of silica and alumina in pure wheat. While no uniform relation exists, the author believes that a relation, sufficiently close for practical purposes, may be made out, and would be specially useful, in connection with the logwood test, in testing for alum in bread. A better test with flour is to shake up 100 grammes with 300 to 500 cc chloroform. The flour rises to the surface on standing, while the mineral impurities sink to the bottom, and may be drawn off by the lower tap and subjected to further tests. The anthor has based on this principle a quantitative method, the results of which, while somewhat too low, are, nevertheless, more satisfactory than might be expected. From his experience, the author concludes that no sample of flour need be examined for alum, if the logwood test yields no indication of its presence.