

Analytical Chemistry.

A Compensation Method in Gasometry. By CARL VON THAN (*Zeit. physikal. Chem.*, 1896, **20**, 307—320).—The author describes a form of eudiometer in which the corrections for temperature and pressure are eliminated. The principle of the method is the use of a similar tube containing a definite quantity of nitrogen which can be brought to either of four volumes, equal respectively to 1.25, 1.428, 2.0, and 5.0 times the normal volume. Arrangements are described for bringing the two tubes to the desired positions, so that the level of the mercury is the same in both, and corresponds with one of the observation marks in the compensation tube. Experiments are recorded indicating the accuracy of the instrument.

L. M. J.

Volumetric Estimation of Hydrofluoric acid. By KARL F. STAHL (*J. Amer. Chem. Soc.*, 1896, **18**, 415—425).—The strength of the commercial acid, if fairly pure, may be conveniently taken by means of a hydrometer made of platinum, but the following method is preferable.

Total acidity. Two grams of the sample is added to a measured volume of normal soda, allowing the liquid to remain faintly acid; after adding phenolphthalein and warming to 50°, the titration is completed.

Hydrofluosilicic acid. Two grams is diluted in a platinum dish with 5 c.c. of water, and partially neutralised with potassium carbonate, employing 0.05 gram for each c.c. of normal soda used in taking the total acidity; alcohol is now slowly added until the mixture

contains 50 per cent. of it, and the whole is left for some time before the precipitate of potassium silicofluoride is collected; after washing it with 50 per cent. alcohol, the precipitate is put into a platinum dish, mixed with 25 c.c. of water, and titrated with normal soda. 1 mol. of potassium silicofluoride requires for neutralisation 4 mols. of sodium hydroxide; every 6 mols. of the latter correspond with 1 mol. of hydrofluosilicic acid.

Sulphuric acid. Four grams of the sample is evaporated in a platinum dish on the water bath until no more acid fumes are given off. The residue is then diluted with water and titrated with normal soda.

L. DE K.

Estimation of Sulphur in Pyrites. By THOMAS S. GLADDING (*J. Amer. Chem. Soc.*, 1896, 18, 446—449).—A controversy with Lunge (*Abstr.*, 1895, ii, 411). The author again points out that if barium chloride is added all at once to a solution of a sulphate, the precipitated barium sulphate is strongly contaminated with the chloride. If, however, the barium solution is added drop by drop to the boiling solution and excess be avoided, the barium sulphate will be practically pure.

L. DE K.

Detection of Nitrites by means of Cuprous Salts. By PAUL SABATIER (*Compt. rend.*, 1896, 122, 1417—1419).—When a small quantity of a nitrite is dissolved in concentrated sulphuric acid, and a few particles of cuprous oxide are added, a very intense violet-purple coloration is produced. All cuprous salts produce the same result, although in the case of the dry chloride the coloured product forms slowly. With cuprous iodide, the iodine is liberated, but can be removed by means of chloroform, in which the purple compound is insoluble. Finely divided reduced copper dissolves in the sulphuric acid solution of the nitrite, giving the violet coloration; cupric salts show no similar reaction, nor do salts of many other metals.

The violet-purple solution loses its colour slowly, even in sealed tubes out of contact with air; the change is accelerated by heat, and takes place immediately if the liquid is agitated with air or if oxidising agents are added. Water, or sulphuric acid mixed with one-fifth its volume of water, also destroys the violet compound.

In order to apply this reaction to the detection of nitrites, a very small drop of the liquid is mixed on a porcelain plate with a drop of concentrated sulphuric acid, and a few particles of cuprous oxide are added. It is not so sensitive as the metaphenylenediamine reaction.

C. H. B.

A Modified Ammonium Molybdate Solution. By A. L. WINTON (*J. Amer. Chem. Soc.*, 1896, 18, 444—445).—One thousand grams of molybdic acid is dissolved in 4160 c.c. of a mixture of 1 part of ammonia (sp. gr. 0.90) and 2 parts of water. 5300 grams of ammonium nitrate is dissolved in a mixture of 6250 c.c. of nitric acid (sp. gr. 1.4) and 3090 c.c. of water, and the molybdate solution is added to it slowly with constant stirring. After a few days, the clear liquid is poured off for use.

This solution differs from Fresenius's solution in that it contains in 50 c.c. an extra 15 grams of ammonium nitrate. The author

claims that the new solution causes a more rapid and complete formation of the yellow phosphoric acid precipitate. L. DE K.

A Simple Method for determining the Neutrality of the Ammonium Citrate Solution used in the Analysis of Fertilisers.

By NATHANIEL W. LORD (*J. Amer. Chem. Soc.*, 1896, 18, 456—457).—Pure litmus solution is added to 200 c.c. of distilled water until this is faintly coloured. Half of this is then diluted with its own volume of water; three clear 50 c.c. Nessler tubes are taken, and two are filled with the diluted liquid, and the third to the same depth with the stronger solution. To one of the two first, a drop of dilute sulphuric acid is added, and to the other, a drop of ammonia. These tubes are set one in front of the other, so that the light passes through both, thus giving a strictly neutral purple colour. To the liquid in the third tube is now added 5 c.c. of the citrate solution to be tested, and the colour is then compared with the colour shown by the doubled tubes. The slightest acidity or alkalinity is then at once noticed, and the amount of either acid or alkali necessary to make it neutral may be rapidly ascertained. L. DE K.

Separation of the Insoluble Phosphoric acid derived from Bone Phosphate from that derived from Mineral Phosphate.

By A. P. BRYANT (*J. Amer. Chem. Soc.*, 1896, 18, 491—498).—As the value of the phosphate insoluble in ammonium citrate is not the same in these two classes of phosphates, a method for separating and estimating them is desirable. The following separating solution is required:—75 grams of potassium iodide and 100 grams of mercuric iodide is dissolved in 350 c.c. of water and evaporated to a sp. gr. of 2.26. The liquid should be kept in a wash bottle.

The special apparatus required consists (1) of a separating tube, made of two tubes about 13 mm. internal diameter, one being 70 mm. in length, closed at one end, the other 200 mm. long, and open at both ends, and connected by a short piece of stout rubber tubing, sufficiently long to allow of the lower closed tube or "bucket" being separated by a screw pinch-cock from the upper portion. (2) An extraction tube, 25 mm. internal diameter, slightly contracted at one end, which has a rim, over which filter paper and cheese cloth can be tied.

In carrying out the separation, 2 grams of the sample is transferred to the extraction tube, and washed with 100—225 c.c. of hot water. After thoroughly drying, it is most carefully removed and transferred to the separating tube; 15—20 c.c. of the separating solution is then poured into the tube, thoroughly shaken, and the sides washed down with a jet of the solution. After five minutes, the lower part of the tube is tapped smartly with the finger to release any light portion carried down with the heavy, and the matter on the top is stirred up with a jet of solution. After the solution has cleared, the rubber tube is clamped, and a beaker is placed under the "bucket," which is then carefully removed, the fingers being protected by india-rubber finger tips. The solution in it containing the mineral phosphate is filtered into the supply flask, the first washings being again evapo-

rated to a sp. gr. of 2.26, and the insoluble phosphoric acid is treated in the usual way. The light portion from the upper tube is treated in a similar manner. If desired, the heavy and light portions can be treated as for total phosphoric acid, thus determining all the phosphoric acid derived from inorganic and organic sources, except that soluble in water.

L. DE K.

Rapid Estimation of Carbonic Anhydride in the Atmosphere, &c. By HENRIET (*Compt. rend.*, 1896, 123, 125—127).—The method described makes use of the fact that a solution of normal potassium carbonate, coloured by a drop of phenolphthalein, becomes colourless on the addition of sulphuric acid, the instant the first trace of carbonic acid is set free after the neutral carbonate has become converted into the bicarbonate. The carbonic anhydride in a given volume of air is absorbed by potash and the liquid titrated, an equal volume of the original potash solution is also titrated, and the difference between the titrations multiplied by two gives the amount of carbonic anhydride absorbed. It is obvious that the amount of carbonate in the original potash need not be taken into account.

The apparatus suggested consists of a flask of about 6 litres capacity, fitted with a caoutchouc cork, through which pass a dropping funnel, and a tube bent at right angles and provided with a cock. A given volume of potash, coloured with phenolphthalein, and covered with a layer of ether to prevent the absorption of carbonic anhydride from the air, is run into the flask, containing the sample of air, by means of the dropping funnel; the latter is then thoroughly washed out with distilled water, previously boiled to remove carbonic anhydride, and the flask is agitated at intervals during one hour. The potash solution in the flask is then titrated.

J. J. S.

Estimation of Potassium. By CHARLES FABRE (*Compt. rend.*, 1896, 122, 1331—1333).—Potassium platinochloride, precipitated and washed in the usual way, is dissolved in hot water, and reduced to the metallic state by adding magnesium powder previously washed with alcohol and water. The chlorine in the liquid is estimated by means of silver nitrate solution, using potassium chromate as the indicator. A small quantity of insoluble magnesium oxychloride often forms during the reduction, and it is therefore advisable to add a few drops of sulphuric acid; after the reduction is complete, filter, neutralise with calcium carbonate, and then titrate.

C. H. B.

The End Point in Gay-Lussac's Method of Silver Titration. By C. HOITSEMA (*Zeit. physikal. Chem.*, 1896, 20, 272—282).—In the titration of silver by Gay-Lussac's method, the end point of the reaction is, according to Mulder, the "neutral point" at which both silver nitrate and sodium chloride produce a precipitate in the clear, supernatant liquid. The existence of this neutral point is explained by the author by the solubility of silver chloride. As the compound is also present in the solid state, the product of the concentrations of its ions must remain constant, and hence the addition of either silver or chlorine ions must cause a precipitation of the salt. From Stas's

experiments on the sensitiveness of the reaction, the author calculates that in 100 c.c. 23×10^{-5} millimol is the least quantity capable of giving an observable precipitate, *i.e.*, about 1 part in 3,000,000, and from other experiments similar values are obtained. A similar neutral point is not observed in titrating with a bromide or iodide, because the solubility is less than the amount necessary to produce the apparent precipitate. L. M. J.

Solubility of Barium Sulphate. By FRIEDRICH W. KÜSTER (*Zeit. anorg. Chem.*, 1896, **12**, 261—271).—The author criticises the work of Fresenius and Hintz on the solubility of barium sulphate in water. These chemists found the solubility to be 1 in 100,000 parts of water. If, however, a solution of barium sulphate of this strength is prepared in the manner described by Fresenius and Hintz, and is then treated with pure barium sulphate, the electric resistance of the solution gradually decreases from 0.000085 to 0.000020, and then remains constant, and since the electric resistance is proportional to the concentration, it follows that this saturated solution of barium sulphate contains 1 in 425,000 parts. This number agrees closely with that obtained by Kohlrausch, Rose, and Holleman.

The author also criticises the work of Fresenius and Hintz on the solubility of barium sulphate in various salts and acids, and comes to the conclusion that the results obtained by them are only qualitative, and that the solubility varies in accordance with the action of mass, and the reciprocal influence of electrolytes on solution.

E. C. R.

Volumetric Estimation of Lead. By ANTONIO LONGI and L. BONAVIA (*Gazzetta*, 1896, **26**, i, 327—403).—The authors have carefully examined most of the methods suggested for determining lead volumetrically, in order to ascertain whether any of them possess sufficient accuracy to replace the ordinary gravimetric methods.

Trials were made of Domonte's process (*J. pr. Chem.*, 1846, **38**, 306), which consists in titrating a lead salt, dissolved in excess of potash, with sodium sulphide solution, and of Casamajor's modification of the method (*Abstr.*, 1882, 776); the two methods are not to be recommended owing to the uncertainty of the end reaction.

Hempel's method (*Jahresb.*, 1853, 627) of determining lead by adding oxalic acid, separating the lead oxalate, and determining the oxalic acid either in the precipitate or in the solution by potassium permanganate, gives excellent results if certain precautions are taken. Considerable excess of oxalic acid must be added, and the solutions must be concentrated, lead oxalate being slightly soluble in water, but less so in oxalic acid solutions; the presence of even 0.066 per cent. of nitric acid interferes slightly with the determination of oxalic acid by permanganate. Alcohol should be added to the solution in which the precipitation is performed, and the precipitate should be washed with dilute alcohol, decomposed by sulphuric acid, and the oxalic acid titrated with permanganate.

Schwarz proposed (*Dingl. polyt. J.*, 1863, **169**, 284) to titrate lead solutions, in presence of sodium acetate, with potassium dichromate, determining the end of the titration by mixing drops of the super-

nant liquid with silver nitrate solution. Notwithstanding Schwarz's statement to the contrary, the authors find that the precipitate of lead chromate does not settle with sufficient rapidity for volumetric work; they find also that freshly precipitated lead chromate reacts with silver nitrate giving silver chromate, so that the indication of the end of the titration may be masked. The method gives low results.

The process described by Pellet (this Journal, 1877, i, 227), depending on precipitating the lead with a chromate and determining the excess of the latter volumetrically, is in several respects unsound, and therefore gives bad results. The authors propose the following modification of Pellet's method: Sodium acetate solution is added to the concentrated lead solution, and then standard potassium dichromate is run in until the liquid becomes decidedly yellow; the solution is filtered, and the precipitate washed with the least possible quantity of water. The filtrate is conveniently diluted, and 2 parts of 100 c.c. taken; to each of these is added 5 c.c. of sulphuric acid, and then a standard solution of ferrous ammonium sulphate until the solution is green with no trace of the yellow tint of the chromate. The excess of ferrous salt is then titrated with permanganate, and the quantity of lead in the original solution calculated. The test results are very satisfactory.

Diehl's method (Abstr., 1880, 752), in which the excess of dichromate added to a lead solution is determined by sodium thiosulphate, cannot be depended on; the equation $4K_2Cr_2O_7 + 3Na_2S_2O_3 + 13H_2SO_4 = 3Na_2SO_4 + Cr_2(SO_4)_3 + 4K_2SO_4 + 13H_2O$, which Diehl uses, is incorrect, so that the equivalent values of the thiosulphate must be found by titration against the dichromate.

Roux (Abstr., 1881, 849) has devised a method for determining lead volumetrically, which is widely used; it consists in precipitating the lead by potassium dichromate in presence of sodium acetate, and estimating the excess of dichromate by standard ferrous ammonium sulphate, using potassium ferricyanide as an indicator. The authors failed to get trustworthy results with this method as it stands, but the results are much improved by acidifying the filtrate from the lead chromate with sulphuric acid and running it into a definite quantity of standard ferrous salt, until the latter gives no reaction with ferricyanide, or reducing the chromic acid by adding standard ferrous solution and estimating the excess of the latter by titration with standard dichromate; very dilute potassium ferricyanide strongly acidified with sulphuric acid should be used as an indicator. Very satisfactory results are obtained by this modified process in dilute lead nitrate solutions, which are neutral or contain free acetic acid; the solution, however, should not contain acetates.

The method of titrating chromates by adding potassium iodide and hydrochloric acid and estimating the liberated iodine with thiosulphate (Mohr, *Zeits. anal. Chem.*, 1863, 2, 247) can be applied to the volumetric determination of lead; the titration with thiosulphate can be performed immediately after adding the hydrochloric acid. Sodium acetate is added to lead nitrate solution, and then excess of dichromate is run in; the lead chromate is filtered off,

washed, and the filtrate made up to a standard volume, in which the excess of dichromate is determined by potassium iodide and thiosulphate. The chromic acid in the precipitate may also be determined by dissolving it in caustic potash, diluting to a known volume, adding potassium iodide, then hydrochloric acid, and estimating the liberated iodine with thiosulphate; the results are excellent, but N/100 potassium dichromate should be used, or the solution must be so dilute that the green colour of the chromic salt does not interfere with the end reaction (compare Crismer, *Abstr.*, 1884, 1078).

The volumetric estimation of lead by titration with standard potassium ferrocyanide has been studied by Graeger (*Zeits. anal. Chem.*, 1865, 4, 438) and Yvon (*Abstr.*, 1889, 549). The authors add to a solution of lead nitrate not more than 3 per cent. of potassium nitrate, and then run in an excess of standard potassium ferrocyanide; the precipitate is filtered and washed with 3 per cent. potassium nitrate solution until the filtrate gives no reaction with ferric chloride. The ferrocyanide in the filtrate is determined by permanganate after acidification with sulphuric acid; the precipitated lead ferrocyanide, Pb_2FeCy_6 , also may be dissolved in sulphuric acid and titrated with permanganate. Care must be taken to avoid atmospheric oxidation of the ferrocyanic solution, but otherwise the method is a practicable and accurate one.

The authors have made many trials of Haswell's method (*Dingl. polyt. J.*, 1881, 241, 393), modifying it in various ways, but do not find it suitable for the volumetric estimation of lead. W. J. P.

Electrolytic Estimation of Iron, Nickel, and Zinc. By HUDSON H. NICHOLSON and S. AVERY (*J. Amer. Chem. Soc.*, 1896, 18, 654—659).—Iron when deposited from an alkaline tartrate solution generally contains a trace of carbon, which compensates somewhat for the incomplete precipitation. When citric acid is used, the amount of carbon is still larger. Formates or oxalates do not deposit any carbon. The author has found that, when using Classen's well-known ammonium oxalate process, the electrolytic precipitation of the metal is much facilitated by the addition to the solution (150 c.c.) of a saturated solution of borax (5 c.c.). Nickel has no tendency to carry down with it any carbon, and its electrolytic estimation is, on the whole, satisfactory. As regards zinc, the authors got very satisfactory results in the presence of formic acid partially neutralised with sodium carbonate; a compact, well adhering, evenly distributed, and metallic looking deposit was obtained.

L. DE K.

Sources of Error in Volhard's and similar Methods of Estimating Manganese in Steel. By GEORGE AUCHY (*J. Amer. Chem. Soc.*, 1896, 18, 498—511).—The author has made a large number of experiments on this process, and comes to the conclusion that the following are sources of error in Volhard's process.

(1) The incomplete neutralisation by zinc oxide, usually giving too high results. (2) The too sudden addition of the necessary excess of zinc, frequently giving results which are too low. (3) The titra-

tion in nitric acid solution, giving results about 0.01—0.02 per cent. too high. (4) Neutralisation by zinc oxide in hot solution, giving results which are too high.

In every case a blank experiment should be made, to ascertain the amount of manganese contained in the zinc oxide or other reagents.

The author prefers Stone's modification of Volhard's method as being easier and more rapid than the original. L. DE K.

Estimation of Lactose in Milk by double Dilution and Polarisation. By HARVEY W. WILEY and ERWIN E. EWELL (*J. Amer. Chem. Soc.*, 1896, **18**, 428—433).—The authors have endeavoured to determine the exact correction to be made for the volume occupied by the coagulum when clarifying the milk for polariscopic purposes.

Two portions of milk, of 65.82 grams each, are treated with 10 c.c. of solution of mercuric nitrate, prepared by dissolving the metal in twice its weight of nitric acid (sp. gr. 1.42), and diluting with 5 vols. of water. The one mixture is diluted to 100 c.c., the other to 200 c.c., and, after filtering, their respective rotations a and b are observed at 20°. The corrected rotation a' corresponding with a , allowance being made for the volume of the coagulum, is calculated from the equation

$$a' = \frac{ab}{a - b} \quad \text{The volume } x \text{ of the precipitate is calculated from the equation } x = \frac{100(a - 2b)}{a - b}.$$

L. DE K.

Toxicological Examination for Mercuric Cyanide. By DIOSCORIDES VITALI (*L'Orosi*, 1896, **19**, 80—87).—Mercuric cyanide would not be detected by the ordinary toxicological methods for detecting cyanides, because it is not decomposed by the feeble acids, such as tartaric acid, used to liberate the hydrogen cyanide.

The author proposes to use a method of detecting mercuric cyanide based on the fact that this salt is converted by magnesium into magnesium cyanide, which, when heated in aqueous solution, is wholly decomposed into magnesia and hydrogen cyanide. The material to be investigated is put into a retort, mixed with the liquid which accompanies it or, in default of this, with water, and magnesium dust is added; the retort is then heated on the water bath and the vapours caused to pass through very dilute caustic soda. If it is permissible to evaporate the contents of the retort to dryness, all the hydrogen cyanide can be driven off, but, if this be inadvisable, dilute acetic acid may be added to drive off the last traces of hydrogen cyanide. W. J. P.

Analysis of Wool-grease. By FERDINAND ULZER and HEINRICH SEIDEL (*Zeit. angew. Chem.*, 1896, 349—350).—The authors recommend the following process: 20 grams of potassium hydroxide is dissolved in 20 c.c. of water contained in a hemispherical dish of about 350—500 c.c. capacity. The solution is heated to boiling, and 20 grams of melted wool-grease is introduced with vigorous stirring. After boiling for another minute, the dish is placed on the water bath

until saponification seems complete; to make sure, however, the dish is placed for another two hours inside the water oven. The soap is then dissolved in 250 c.c. of boiling water, restoring from time to time the water lost by evaporation, and when completely dissolved 40 c.c. of hydrochloric acid, slightly diluted, is added. When the fatty mass is perfectly melted, it is, after cooling, removed, and then repeatedly boiled with water until the washings no longer show an acid reaction. After cooling, the fatty cake is removed, dried between blotting paper, and finally dried in the water oven. The total acidity number is then taken by means of Benedikt and Mangold's process. Three samples of Australian wool-grease gave, respectively, the figures 100.2, 102.9, and 101.9. Three South American samples gave the figures 96.4, 96.7, and 96.9.

To form an opinion on the genuineness of wool-grease, the authors also recommend taking the actual acidity number and the Reichert-Meissl number. Its iodine absorption may also be determined.

L. DE K.

A New Test for Asparagine. By L. MOULIN (*J. Pharm.*, 1896, **3**, 543).—"Saccharin," warmed with sulphuric acid and a little resorcinol, turns yellowish-green, and if water is now added, and the mixture neutralised with ammonia or aqueous soda, a strongly fluorescent liquid is obtained. The author has found that a similar fluorescence of a fine green tint is produced when asparagine is treated in this way. The same reaction is also given by a crystalline compound, which the author has separated from a cold infusion of liquorice by means of dialysis.

L. DE K.

Test for Cinchona Alkaloids. By JAWOROWSKI (*J. Pharm.*, 1896, **3**, 553; from *Pharm. Zeits. Russ.*, 1896, **6**).—A mixture is made of equal parts of a 10 per cent. solution of sodium thiosulphate and a 5 per cent. solution of copper sulphate, and at once added drop by drop to 5 c.c. of the liquid to be tested. In presence of quinine, quinidine, cinchonine or cinchonidine, a yellow, amorphous precipitate is formed; if no precipitate is formed after the lapse of a minute, quinine, &c., is absent; the reagent itself giving a precipitate after a time.

The reaction takes place not only in aqueous solutions, but also in solutions made with chloroform, ether, or amylic alcohol.

L. DE K.

Estimation of Caffeine in Tea. By AUGUSTE PETIT and P. TERRAT (*J. Pharm.*, 1896, **3**, 529—534).—The authors have found that the use of magnesia or lime is superfluous, and that the alkaloid may be completely extracted from tea by direct treatment with chloroform, provided the tea is moist; dry tea yielding but very little caffeine to chloroform. The following method is recommended. 25 grams of powdered tea is treated with thrice its weight of boiling water and, after 15 minutes, the whole is evaporated on the water bath until the liquid has disappeared, but the residue remains still visibly moist. It is then introduced into a percolator and repeatedly exhausted with

chloroform to extract the caffeine. The chloroform is distilled off, and the residue taken up with boiling water, which, on evaporation, will generally yield a residue sufficiently pure to be at once weighed.

If the alkaloid should be contaminated with chlorophyll, it must be treated with dilute sulphuric acid (1 : 10), filtered, neutralised with ammonia, and evaporated completely to dryness; the residue is then extracted with chloroform, and, after distilling off the latter, the pure caffeine can be weighed.

L. DE K.

Estimation of Nicotine and Ammonia in Tobacco. By VIKTOR VEDRÖDI (*Zeits. anal. Chem.*, 1896, **35**, 309—311).—A continuation of the controversy between the author and Kissling (see this vol., ii, 401). The author states that he used light petroleum and ethylic ether indifferently, having assured himself that the former "gave just as accurate results" as the latter. He reaffirms the fact of soda being carried over when Kissling's own apparatus is employed. The fact that the addition of ammonium chloride did not influence the nicotine titration when working in the manner he proposes (*Abstr.*, 1895, ii, 541) he attributes to the complete volatilisation of the ammonia during the extraction with ether, the ether used being as free from water as possible, and the condenser being cooled with water of ordinary temperature.

With regard to the objection that the amido-compounds in tobacco would yield ammonia when boiled with soda, he considers that his experiments on this subject (*Abstr.*, 1894, 371) show that the decomposition of proteids by soda is too imperfect to influence the estimation of nicotine and ammonia in tobacco. Moreover, in the fermentation which occurs during the manufacture of tobacco from the leaves, most of the amido-compounds and proteids are destroyed. With regard to the errors of calculation imputed to him by Kissling, he ranges his "practical" results against the "theoretical" arguments of the latter.

M. J. S.

Distinction between Magenta and "Acid Magenta." By PAUL CAZENEUVE (*J. Pharm.*, 1896, **3**, 595—597).—A well-known and very delicate reaction for the presence of aldehydes in commercial alcohol is based on the violet colour produced when the sample is mixed with a solution of a rosaniline salt which has been previously decolorised by the addition of sulphurous acid.

The author points out that magenta and "acid magenta" do not behave in the same way. The article consisting of rosaniline hydrochloride gives the coloration, whereas the sulphonated (S) compound does not, a fact which has been overlooked by Lefèvre.

L. DE K.

Examination of Red Wines for Foreign Colouring Matters. By ALBIN BELAR (*Zeits. anal. Chem.*, 1896, **35**, 322—323).—Most of the coal tar dyes give coloured solutions with nitrobenzene, whilst the natural blue and red vegetable colours are insoluble. To make the test, about 5 c.c. of the wine is shaken in a test tube with an equal volume of pure nitrobenzene, first gently, then violently, and if an emulsion

is produced it is caused to separate by warming. Rosaniline, purpurin, and safranin dissolve unchanged; methylene-blue is partially dissolved with an emerald-green coloration; eosin and rosolic acid both leave yellowish insoluble portions. Indigo carmine (sodium indigotin-disulphonate) is quite insoluble. M. J. S.

Quantitative Separation of Proteids in Beer Wort. By N. C. HENRIK SCHJERNING (*Zeits. anal. Chem.*, 1896, **35**, 285—296).—The extended experience which the author has by this time acquired in the method of wort analysis suggested by him in 1894 (*Abstr.*, 1894, ii, 371; and 1895, ii, 428) has enabled him to systematise and simplify the operations so as to form a process of practical value. Six reagents are now employed, namely, stannous chloride, lead acetate, acetic acid, uranium acetate, ferric acetate, and magnesium sulphate. The stannous chloride is prepared by dissolving 50 grams of tin in hydrochloric acid, evaporating to 130 grams, diluting to 1 litre, and rapidly filtering. It must be kept in small stoppered bottles. The lead acetate solution is approximately a 10 per cent. one, containing 10—12 drops of 45 per cent. acetic acid to the litre. The acetic acid is made by diluting 15 c.c. of the 45 per cent. acid to a litre. The uranium acetate solution is a cold saturated one, obtained by cooling and filtering one prepared at a higher temperature. "Scale" ferric acetate is preserved in the dry state, and dissolved just before use, taking always 0.8 gram to 40 c.c. of the above acetic acid diluted with 100 c.c. of water.

The stannous chloride precipitate, which is regarded as albumin, is obtained by adding 5 c.c. of the reagent to 25 c.c. of wort, allowing the mixture to repose for 4—6 hours at the ordinary temperature, collecting on an 11-cm. extracted filter, washing twice with cold water without suction, and estimating the nitrogen according to Kjeldahl's method. No correction for solubility is needed. Lead acetate precipitates the albumin and denuclein; about 6 c.c. of the reagent is added to 25 c.c. of wort, the mixture heated to boiling, and the precipitate collected immediately and washed with cold water. In consequence of its solubility, a correction of 0.15 c.c. of N/10 acid must be applied for each 100 c.c. of filtrate + washings. For the ferric acetate precipitation, the reagent prepared as above is heated to boiling and mixed with 20 c.c. of wort. After boiling again, the precipitate is collected and washed with boiling water. No correction for solubility is required. The precipitate contains the albumin, denuclein, and propeptone. The uranium precipitate contains the above proteids together with the peptone; 20—25 c.c. of the reagent is added to 25 c.c. of wort, the mixture boiled, and then left to cool until next day in a dark place. The precipitate is washed with cold 1—2 per cent. uranium acetate solution; an addition of 0.1 c.c. of N/10 acid is made for each 100 c.c. of filtrate and washings. In cases where the ferric acetate precipitate is obtained with difficulty, the albumin and propeptone are thrown down by magnesium sulphate; 20 c.c. of the wort, mixed with 5 drops of 45 per cent. acetic acid, is warmed in a water bath to 36°, and 18—20 grams of pulverised crystalline magnesium sulphate is dissolved in it by stirring. It is then left at the ordi-

nary temperature for $\frac{1}{2}$ —1 hour, filtered, and the precipitate washed with a cold saturated solution of magnesium sulphate containing 4—5 grams of 45 per cent. acetic acid to the litre. Zinc sulphate gives identical results (see Bömer, this vol., ii, 83). An experiment made with the dialysed solution of the magnesium sulphate precipitate showed that the presence of saline substances was essential to the precipitation by stannous chloride and ferric acetate, and yielded, moreover, some anomalous results, which up to the present the author has not been able to interpret.

M. J. S.

Estimation of Phosphorcarnic acid. By BALKE and IDE (*Zeits. physiol. Chem.*, 1895, **21**, 380—386).—In estimating phosphorcarnic acid by precipitation as carniferrin (see Abstr., 1895, i, 313) the use of ferric chloride entails the possibility of dissolving some of the precipitate by excess of the reagent; and ferric ammonium alum cannot be employed, as the aqueous solution, when boiled, deposits a small quantity of insoluble nitrogenous matter. The following process is found to yield concordant results.

The dilute meat-extract is heated, to coagulate albumin, and filtered, the phosphates present being then precipitated with calcium chloride and ammonia. The filtrate is neutralised, heated to boiling, and mixed with a 1 per cent. solution of ferric chloride, the latter being carefully delivered from a burette; when a slight excess has been added, the whole is boiled for about two minutes, the addition of ferric chloride being discontinued if the excess be permanent; the liquid is then neutralised with a few drops of ammonia, and finally separated from the precipitate by decantation, the latter process being employed in washing the sediment. The total nitrogen in the dry carniferrin is then estimated and calculated as carnic acid.

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