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# REVIEW OF AMERICAN CHEMICAL RESEARCH.

VOL. VI. NO. 2.

ARTHUR A. NOYES, Editor.

REVIEWERS: Analytical Chemistry, H. P. Talbot and W. H. Walker; Biological Chemistry, A. G. Woodman; Carbohydrates, G. W. Rolfe; General Chemistry, A. A. Noyes; Geological and Mineralogical Chemistry, W. O. Crosby and M. L. Fuller; Inorganic Chemistry, Henry Fay; Metallurgical Chemistry and Assaying, H. O. Hofman; Organic Chemistry, J. F. Norris; Physical Chemistry, H. M. Goodwin; Sanitary Chemistry, E. H. Richards; Industrial Chemistry, A. H. Gill and F. H. Thorp.

## ANALYTICAL CHEMISTRY.

### ULTIMATE ANALYSIS.

H. P. TALBOT, REVIEWER.

**The Determination of Nickel in Nickel-Steel.** By GEO. WM. SARGENT. *J. Am. Chem. Soc.*, 21, 854-857.—The steel is dissolved in hydrochloric acid, the iron oxidized by nitric acid (the excess of which is removed by evaporation to dryness), and the hydrochloric acid solution of the residue treated with ether, which has previously been shaken with strong hydrochloric acid. A single treatment was found to suffice. The aqueous solution containing the nickel is treated with ammonia and the acidified filtrate with sulphuretted hydrogen to remove any copper. The filtrate is concentrated, ammonia is added in slight excess, then measured quantities of solutions of silver nitrate and potassium iodide, after which a standard potassium cyanide solution is added until the opalescence due to silver iodide just disappears. Compare the method of Chase (Blair: *The Chemical Analysis of Iron, Appendix to Third Edition*); and Campell and Andrews: *J. Am. Chem. Soc.*, 17, 125.

**The Quantitative Estimation of Boric Acid in Tourmaline.** By GEORGE W. SARGENT. *J. Am. Chem. Soc.*, 21, 858-888.—The results of the experiments described in this paper are summarized at its close, and cannot well be further condensed. The paper includes a historical résumé of earlier work in this field, and a bibliography.

**Electrolytic Determinations and Separations.** By LILY G. KOLLOCK. *J. Am. Chem. Soc.*, 21, 911-928.—The methods for

the electrolytic determination or separation of a considerable number of metals have been subjected to examination with a view of defining with exactness the conditions essential for success, especially with respect to amperage, voltage, and current density. Reference to the original article is necessary for the detailed results.

**The Precipitation of Copper by Zinc.** By JOHN C. SHENGLE AND EDGAR F. SMITH. *J. Am. Chem. Soc.*, 21, 932-933.—The authors find that copper precipitated from its solutions by means of zinc always contains small, variable quantities of the latter metal.

**Note: Analysis of Zinc for Cadmium and Lead.** By P. A. MACKAY. *J. Am. Chem. Soc.*, 21, 940-941.—The greater portion of the zinc is dissolved in hydrochloric acid, leaving the lead and cadmium deposited upon the residual zinc. After solution in nitric acid the lead is removed as sulphate, the cadmium thrown down as sulphide, dissolved in hydrochloric acid, a known solution of zinc chloride is added, and the whole titrated with potassium ferrocyanide.

**Notes on the Determination of Sulphur in Pig-Iron.** By M. J. MOORE. *J. Am. Chem. Soc.*, 21, 972-975.—The author finds marked differences in the results obtained by volumetric determinations of sulphur made upon shot samples and sand samples of pig-iron taken from the same mixer. The results obtained from the shot samples are low as compared with the gravimetric determinations.

**Note: The Determination of Chromium in Steel.** By R. W. MAHON. *J. Am. Chem. Soc.*, 21, 1057-1060.—The author states the results of experiments made to determine exact conditions as to volume, quantity of reagents, etc., which are best for use in McKenna's method for the estimation of chromium (*Proc. Eng. Soc. Western Penn.*, 11, No. 6).

**The Direct Determination of Available Phosphoric Acid.** By F. P. VEITCH. *J. Am. Chem. Soc.*, 21, 1090-1094.—The author shows that the discrepancies in the method of Ross (*U. S. Dept. Agr., Div. Chem., Bull.* 38, 17), as compared with the official method, arise from the failure to take account of the phosphoric acid in the washings of the citrate-insoluble material. He also makes a comparison of the official method with the citrate and molybdate methods, as applied to the combined filtrates containing the citrate-soluble and water-soluble phosphoric acid. The molybdate method yields satisfactory results.

**Note upon the Determination of Nitrogen in Fertilizers Containing Nitrates.** By F. P. VEITCH. *J. Am. Chem. Soc.*, 21, 1094-1095.—The author describes a modification of the Gunning method (*U. S. Dept. Agr., Div. Chem., Bull.* 46) slightly different from that described by Fields (*this Rev.*, 3, 32). The fertilizer is treated with the mixture of sulphuric and salicylic acids in the cold.

**The Determination of Graphitic Carbon in Cast and Pig Iron.** By ALLEN P. FORD AND I. M. BREGOWSKY. *J. Am. Chem. Soc.*, 21, 1113-1115.—The use of a platinum Gooch filter instead of a paper filter, and of hydrofluoric acid to remove the silica, is recommended by these authors. They find that graphitic carbon is slowly oxidized by nitric acid of 1.20 sp. gr. on boiling, but is not affected by acid of 1.12 sp. gr.

W. H. WALKER, REVIEWER.

**The Iodometric Determination of Gold.** By F. A. GOOCH AND F. H. MORLEY. *Am. J. Sci.*, 158, 261-266.—In a recent attempt to determine small quantities of gold in solution by allowing auric chloride to react with potassium iodide, and titrating the iodine liberated with sodium thiosulphate, Peterson (*Ztschr. anorg. Chem.*, 19, 63) found that one-half more thiosulphate is consumed than would be required if the thiosulphate were simply converted to tetrathionate in the usual manner. The authors have studied this reaction and find no evidence of any variation from the usual reaction of sodium thiosulphate and iodine. The reduction of the auric salt and the consequent liberation of iodine is, however, dependent upon the volume of the solution, the mass of iodine present, and the time of action. By using one-thousandth normal solutions the process is made fairly accurate.

**On the Volumetric Estimation of Cerium.** By PHILIP E. BROWNING. *Am. J. Sci.*, 158, 451-459.—The iodometric method of Bunsen (*Ann. Chem. (Liebig)*, 105, 49) for the determination of cerium is modified in that a glass-stoppered bottle is used in the place of a flask with the neck drawn out and closed by heat. As a further check on the accuracy of the method, the iodine set free by the reaction of cerium dioxide and potassium iodide was distilled from a retort in a stream of carbon dioxide and received in a solution of potassium iodide. The results obtained by both these methods are very satisfactory. An attempt to estimate cerium dioxide by reduction with arsenious acid proved unsatisfactory. The statement by Stolba (*Ztschr. anorg. Chem.*, 19, 194) that cerium could be estimated in the same manner as calcium oxalate, by oxidizing the precipitated cerium oxalate with standard potassium permanganate is proved correct.

**On the Estimation of Thallium as the Chromate.** By PHILIP E. BROWNING AND GEORGE P. HUTCHINS. *Am. J. Sci.*, 158, 460-462.—The authors have repeated the work of Baubigny (*Chem. News*, 64, 239) on the gravimetric estimation of thallium, and obtained results which confirm his statements. Instead of weighing the thallium chromate precipitate, the thallium may be estimated volumetrically by determining the amount of chromate in the filtrate by means of standard solutions of arsenious acid and iodine, and, by difference, the amount of chromate combined with the thallium. The results compare very favorably with those by the gravimetric method.

#### PROXIMATE ANALYSIS.

A. G. WOODMAN, REVIEWER.

**Directions for Certain Alkaloidal Assays.** By H. M. GORDIN AND A. B. PRESCOTT. *Am. J. Pharm.*, 71, 462-473.—This paper is a résumé of the work done by the authors on the extraction of alkaloids and their volumetric estimation as higher periodides. It contains the directions for the general method of alkaloidal extraction and assay, and for the modification of the general method in special cases.

**Further Work upon the Estimation of Alkaloids and the Assay of Alkaloidal Drugs.** By H. M. GORDIN AND A. B. PRESCOTT. *Am. J. Pharm.*, 71, 514-525.—In this paper the authors present some of the experimental data upon which they have based their methods for alkaloidal assay, discussing the composition and properties of the periodides of emetine and of hydrastine, and giving directions for the assay of hydrastis and the hot extraction of opium. A table is appended showing the comparative merits of the gravimetric and the iodometric methods of assay.

**A Simple Alkalimetric Method for the Estimation of Salt-forming Alkaloids with the Aid of Phenolphthalein as Indicator.** By H. M. GORDIN. *Pharm. Archives*, 2, 313-318.—The direct alkalimetric estimation of alkaloids is in many cases unsatisfactory, owing to the fact that the end-point is not very sharp. In some alkaloidal salts the acid acts as if wholly free, so that indicators show no end-point at all until the acid used is all neutralized. Often it is necessary to make the titration in a solution colored by non-alkaloidal matter. To obviate these difficulties it is proposed to dissolve the alkaloid in an excess of standard acid, precipitate the alkaloid by some neutral alkaloidal reagent, filter, and determine the free acid in the filtrate. As there is no alkaloid in the filtrate to interfere with the indicator, phenolphthalein can be used with satisfactory results.

For ordinary cases either Mayer's or Wagner's reagent may be used as a precipitant. Berberine and colchicine cannot be estimated by this method.

**Modified Assay Process for Fluid Extracts.** By A. B. LYONS. *Pharm. Rev.*, 17, 558-560.—For the extraction of alkaloids from preparations containing much tannin, such as fluid extract of cinchona bark, it is recommended to shake a definite volume of the extract with a dilute alcoholic solution of ferric chloride, made alkaline with ammonia. The mixture is allowed to settle, is then filtered, and an aliquot part of it is evaporated with dilute hydrochloric acid to drive off the greater part of the alcohol; the acid solution is then shaken with ether to remove the fats and waxes. Finally the solution is made alkaline with ammonia, and shaken out with ether, as usual. The results obtained are satisfactory. If desired, a solution of basic lead acetate may be substituted for the ferric chloride.

**A Note on the Assay of Opium.** By WILLIAM R. LAMAR. *Am. J. Pharm.*, 72, 36-39.—The author has modified the U. S. P. method for the assay of opium by increasing the amount of alcohol which is added to the aqueous extract of the opium, in this way getting rid of the objectionable matters in solution.

**On Acetic Acid as a Substitute for Ethyl Alcohol in Extracting the Active Principles of Some Officinal Drugs.** By EDWARD R. SQUIBB. *Am. J. Pharm.*, 72, 1-9.—In this paper, which is the third in the series, the author presents the results of further experience in the use of acetic acid as a menstruum, and gives in detail the procedure for the assay of belladonna root.

**The Phenylhydrazine Test for Sugar in the Urine.** By I. H. CORIAT. *Boston Medical and Surgical J.*, 141, 518-519.—The author has studied the phenylhydrazine test for sugar with especial reference to the examination of urine, and finds that substances that interfere with Fehling's or Nylander's reagents do not interfere in any way with this test. It is not necessary to remove albumin, and the various sugars can be recognized by their crystalline forms and melting-points. The limit of delicacy of the test is about one part in 10,000.

**Lemon Flavoring Extract and Its Substitutes.** By A. S. MITCHELL. *J. Am. Chem. Soc.*, 21, 1132-1137.—The author describes the methods which he has found most satisfactory in the examination of lemon extract. In the absence of sugar the oil of lemon is readily estimated by polarizing the extract in a 200 mm. tube and dividing the result in degrees by 3.4. The oil may also be separated by the centrifugal machine and meas-

ured in an ordinary Babcock skim-milk bottle. Tests for various minor adulterants are described and a table is given showing the results of examination of several characteristic extracts.

**On Rapid Methods for the Estimation of the Weight of Suspended Matters in Turbid Waters.** By CHARLES L. PARMELEE AND JOSEPH W. ELLMS. *Tech. Quart.*, 12, 145-164.—Many of the surface waters of the central and western parts of the United States contain such quantities of suspended matters, mainly clay, that this question becomes one of considerable importance. The standard method of estimation of the suspended material is a gravimetric one. In order to secure a more rapid method, comparative trials of the following methods were made on Ohio river water: (1) The water was compared with standards containing known amounts of suspended matter; (2) the amount of light which is absorbed in its passage through the water was measured directly; (3) determinations were made of the vanishing-depth of a bright wire; (4) a method was employed based on the use of the diaphanometer. This instrument consists essentially of a tube with a transparent diaphragm at the lower end. Light is reflected up through the tube, water being admitted until an image painted on the diaphragm just disappears. The results of a number of experiments show that by the use of standard clay solutions reasonably close estimations can be made when the suspended matters amount to less than 25 parts per million. When the water contains more than this quantity of suspended matter, the diaphanometer method gives results which agree fairly well with the gravimetric estimations. Greater variations in the character of the suspended particles is found to increase the error in the use of any of the rapid methods.

**Silica Standards for the Determination of Turbidity in Water.** By GEORGE C. WHIPPLE AND DANIEL D. JACKSON. *Tech. Quart.*, 12, 283-287.—The authors have substituted silica from diatomaceous earth for the kaolin previously used, and get a uniformity of results which, taken in connection with its ease of use, lead them to recommend it.

F. H. THORP, REVIEWER.

**Notes on the Analysis of Dynamite and Gelatine-Dynamite.** By F. W. SMITH. *J. Am. Chem. Soc.*, 21, 425-431.—Typical analyses of a dynamite and of a gelatine-dynamite, with notes and precautions, are given in detail. Also various notes on the use of the nitrometer, its standardizing by the use of potassium nitrate, testing for leaky stop-cocks, etc., are communicated.

**Tannin Determination—Estimation of Reds without the Use of Filter-paper.** By J. N. HURTY. *Leather Manufacturer*, 10, 33.—Two methods were tried. First, simple filtration through ignited asbestos in a Gooch crucible was used. Second, the material was subjected to treatment in a centrifugal machine running 2000 revolutions per minute, for 30 minutes, to separate the reds in the extract. With chestnut-oak extract, an addition of barium sulphate is necessary, to cause complete precipitation of the reds. Test analyses of quebracho, chestnut-oak extract, and spent tan (oak bark) are given, and compared with the results obtained by filtering through paper. It is concluded that neither asbestos nor barium sulphate absorbs tannin.

A. H. GILL, REVIEWER.

**The Determination of Carbon Monoxide, Methane, and Hydrogen by Combustion.** By L. M. DENNIS AND C. G. HOPKINS. *J. Am. Chem. Soc.*, 21, 398-410.—The apparatus employed is a modified Winkler grisoumeter, with an adjustable leveling bulb, using mercury as the confining liquid. The gas to be determined is introduced into the grisoumeter, the spiral brought to a suitable temperature, and a quantity of oxygen more than sufficient to burn it passed in at a rate not exceeding 20 cc. per minute. The combustion takes place quietly and without the appearance of a flame. The results upon the single gases were correct within 0.1 per cent., and upon mixtures within 0.1-0.2 per cent. The apparatus is simple and easy of construction and manipulation, and will be welcomed by all having occasion to make gas analyses.

**The Preservation of Hübl's Reagent.** By R. BOLLING. *Am. Chem. J.*, 22, 213-214.—The results indicate that the alteration in the solution is rapid, and is retarded but slightly by the use of absolute alcohol or by the exclusion of air and light.

J. F. NORRIS, REVIEWER.

**The Valuation of Saccharin.** By E. EMMET REID. *Am. Chem. J.*, 21, 461-471.—The amount of saccharin in a commercial sample, which always contains *p*-sulphamibezoic acid, can be determined by saponifying the benzoic sulphinate and estimating the ammonia formed. The saponification is effected by heating at 100° for several hours with 71 per cent. sulphuric acid, or by boiling with dilute hydrochloric acid under definite conditions. The latter method gives more trustworthy results, as sulphuric acid causes a slight decomposition of the sulphamic acid. The methods of analysis were applied with excellent results to a number of samples of commercial saccharine and to mixtures of known composition. Boiling sodium hydroxide

solution readily converts benzoic sulphinide into the sodium salt of *o*-sulphaminebenzoic acid, but does not break this acid down. This fact renders a determination of saccharine in the presence of ammonium salts possible. The nitrogen in *p*-sulphaminebenzoic acid was determined by heating the acid with concentrated sulphuric acid at 230°-260°, making alkaline, and titrating the ammonia set free.

**Hydrastine Hexaiodide, and the Assay of Hydrastis Canadensis, by Means of Standard Iodine for Hydrastine and of Standard Potassium Iodide for Berberine.** By H. M. GORDIN AND A. B. PRESCOTT. *J. Am. Chem. Soc.*, 21, 732-741; *Am. J. Pharm.*, 71, 257-267.—When a dilute solution of hydrastine is added to a large excess of a solution of iodine in potassium iodide a hexaiodide is precipitated. By determining the excess of iodine the amount of the alkaloid can be estimated. If iodine is added to a solution of hydrastine, a triiodide is formed. Berberine is quantitatively precipitated by potassium iodide. A detailed account of the assay of hydrastis canadensis is given.

## INDUSTRIAL CHEMISTRY.

F. H. THORP, REVIEWER.

**Action of a Hard Water on Certain Metals.** By JAS. LEWIS HOWE AND J. L. MORRISON. *J. Am. Chem. Soc.*, 21, 422-425.—The experiments of the authors show that hard water containing an excess of carbon dioxide and considerable magnesia, acts readily upon zinc, corrodes brass, and attacks lead; but that aluminum is not attacked, and in the absence of air iron is only slightly dissolved.

**Glass-making in the United States.** By ROBERT LINTON. *Eng. Min. J.*, 68, 454.—Having described briefly the methods and materials used in glass-making, the writer gives calculations of the cost of plate, window, flint, and bottle glass-making, comparing with these the cost of glass-making in Germany. A noticeable variation between the costs of fuel and labor in the two countries is shown. An interesting review of the progress of the industry in this country is included, and it appears that machines are being rapidly introduced for making fruit-jars, lamp chimneys, and similar articles, thus replacing the costly manual labor. Continuous tank furnaces are displacing the old pot furnaces, and automatic weighing and mixing machines are replacing the often careless "boss mixer." For fuel, gas is almost universally used; natural gas still plays an important though steadily diminishing part, while producer gas, although more troublesome to use than natural gas, has steadily replaced