ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of Acetanilide and Phenacetin in Pharmaceutical Preparations. J. L. Turner and C. E. Vanderkleed. (*Amer. Journ. Pharm.*, 1907, **79**, 151-156.)—The method proposed is one that will permit of the estimation of acetanilide or phenacetin in a complex mixture, and consists in the saponification of the acetanilide obtained by extracting the mixture with chloroform, the acetate formed being then distilled with phosphoric acid, and the acetic acid titrated in the distillate. In the case of tinctures containing other drugs the acetanilide is extracted by means of chloroform, and the impure acetanilide is then treated as follows: One gram of the acetanilide is boiled under a reflux condenser with 3 grams of sodium hydroxide, 20 c.c. of alcohol, and 10 c.c. of water for about two hours. The solution is evaporated until the alcohol has been driven off, and the residual liquid is shaken out once with ether to remove the aniline split off from the acetanilide. The ethereal solution is shaken out twice with water in order to recover traces of sodium acetate, which is somewhat soluble in ether, and the washings are united with the main bulk of the aqueous solution. The latter is then acidified with about 25 c.c. of 85 per cent. phosphoric acid solution, and the acetic acid is distilled off in a current of steam. The distillation is continued until the distillate is no longer acid to litmus paper, from 800 to 1,000 c.c. of distillate being collected before this point is reached. The distillate is now titrated with $\frac{N}{1}$ sodium hydroxide, using phenolphthalein as indicator. One c.c. of $\frac{N}{1}$ alkali corresponds with 0·134 gram of acetanilide. As phenacetin is closely related chemically to acetanilide, the above process may be employed for its estimation. One c.c. of $\frac{N}{1}$ sodium hydroxide is equivalent to 0·177 gram of phenacetin. The method is, however, inapplicable to the separate estimation of these two substances when they occur together in a drug. The phosphoric acid used must be free from acetic, nitric, and nitrous acids or their respective salts.

W. P. S.

Determination of the Value of Rhubarb. A. Tschirch and J. Edner. (*Archiv Pharm.*, 1907, **245**, 150-153.)—The active principles of rhubarb are hydroxymethylanthraquinones, and may be precipitated quantitatively by means of diazotised *p*-nitraniline solution. This reagent is prepared by treating 5 grams of *p*-nitraniline with 25 c.c. of water and 6 c.c. of concentrated sulphuric acid; after shaking, 100 c.c. of water and a solution of 3 grams of sodium nitrite in 25 c.c. of water are added, and the whole is diluted with water to a volume of 500 c.c. The reagent should be kept in a dark place. In carrying out the process, from 0·5 to 1·0 gram of powdered rhubarb is thoroughly extracted by boiling with several successive quantities of dilute alcoholic potassium hydroxide solution. The extracts are united, filtered, and the alcohol is removed by distillation; water is added to the residue, and the solution is then acidified with hydrochloric acid. The precipitate which forms on

standing is collected on a filter, washed with water, and dried. The hydroxymethylanthraquinones are extracted from the dry precipitate with chloroform, several hours' extraction in a Soxhlet apparatus being required; the rheotannic acid remains insoluble. After the chloroform has been distilled off, the residue is dissolved by warming it with 10 c.c. of 5 per cent. sodium hydroxide solution, and water is then added to make the volume up to 50 c.c. Twenty c.c. of the above-mentioned reagent are next added, and sufficient hydrochloric acid is run in drop by drop, with constant stirring, to decolorise the solution and render it acid in reaction. At the end of a few hours the precipitate is collected on a weighed filter, washed free from acid, dried at a temperature of 70° C., and weighed. The weight of the precipitate is conveniently expressed in terms of chrysophanol (chrysophanic acid); 447 parts of precipitate are thus equivalent to 254 parts of chrysophanol. The following are the average percentages of chrysophanol found in various samples of rhubarb by this method: Shensi, 3·2; Canton, No. 2, 2·67; Canton, round, 4·24; Canton, flat, 3·35; Shanghai, flat, 2·7; Shanghai, 4·14; English, 2·07; French, 1·58. Experiments with known quantities of emodin show that the method is trustworthy.

W. P. S.

Estimation of Alkaloids in Nux Vomica, Hydrastis, and Jaborandi by Means of Dinitrophenylmethylpyrazolone. H. Mattheo and O. Rammstedt. (*Archiv Pharm.*, 1907, **245**, 112-132.)—The method proposed is based on the precipitation of strychnine, brucine, hydrastine, and pilocarpine by dinitrophenylmethylpyrazolone (picrolonic acid), and is recommended for the alkaloidal valuation of nux vomica, hydrastis, and jaborandi, and the extracts and tinctures prepared from the same. As applied to the various preparations the method is as follows:

Nux Vomica Extract.—One gram of the extract is dissolved in 5 grams of absolute alcohol and 5 c.c. of water, and the solution is well shaken with 50 grams of ether and 25 grams of chloroform; 10 c.c. of a 50 per cent. sodium carbonate solution are then added, and the mixture is shaken for ten minutes. After standing for twenty minutes, 50 grams of the ether-chloroform solution (equivalent to 0·66 gram of extract) are filtered, the filtrate is evaporated to one-half its volume, and 5 c.c. of $\frac{N}{10}$ alcoholic solution of picrolonic acid are added, the mixture being then placed aside in a cool place for twenty-four hours. The precipitated brucine-strychnine picrolonate is collected on a weighed filter, washed with 2 c.c. of a mixture of 3 volumes of ether with 1 volume of alcohol, dried for thirty minutes at 110°, and weighed. The mean molecular weight of brucine-strychnine being 364·32, and that of brucine-strychnine picrolonate 628·32, the weight of the precipitate obtained, multiplied by 0·5798, gives the weight of the mixed alkaloids.

Nux Vomica Tincture.—Fifty grams of the tincture are evaporated to 10 grams; 5 grams of absolute alcohol, 50 grams of ether, and 20 grams of chloroform are added, and the estimation is then carried out as described above. The 50 grams of ether-chloroform filtered off represent 33·3 grams of the original tincture. An approximately correct estimation may be made by adding the picrolonic acid solution directly to the tincture diluted previously with an equal volume of water.

Nux Vomica Powder.—Fifteen grams of the powder, previously dried at 100°,

are treated with 100 grams of ether and 50 grams of chloroform; the mixture is well shaken, 10 c.c. of 7·5 per cent. sodium hydroxide solution are added, and the whole is shaken for ten minutes. Sufficient water—about 15 c.c.—is then added to cause the drug to collect together, and the ether-chloroform solvent is allowed to separate. Fifty grams of the solution are filtered off, and used for the precipitation with picrolonic acid.

Liquid Extract of Hydrastis.—Fifteen grams are evaporated to a weight of 5 grams, the residue is dissolved in 10 c.c. of water, and the solution is shaken with 10 grams of petroleum spirit, 50 grams of ether, and 5 grams of ammonia solution. Forty grams of the ethereal solution (corresponding with 10 grams of the original extract) are employed for the actual estimation. The molecular weight of hydrastine picrolonate is 647. Eighteen samples of the fluid extract examined yielded from 2·05 to 2·36 per cent. of hydrastine.

Tincture of Hydrastis.—Fifty grams of the sample are evaporated to 10 grams, and then treated as described under the fluid extract. Six samples gave from 0·167 to 0·173 per cent. of hydrastine.

Hydrastis Rhizome.—Six grams of powdered hydrastis are macerated for thirty minutes with 50 grams of ether, 10 grams of petroleum spirit, and 6 grams of ammonia solution; 6 c.c. of water are then added, and the mixture is shaken until the supernatant liquid is clear. Fifty grams of the latter are filtered off and precipitated as described above. From 2·24 to 2·42 per cent. of hydrastine was found in ten samples of the rhizome.

Jaborandi Leaves.—Fifteen grams of the powdered leaves are thoroughly mixed with 150 grams of chloroform and 10 grams of ammonia solution; 100 grams of the chloroform solution are, after filtration, evaporated to a volume of about 20 c.c., 3 c.c. of the picrolonic solution and 60 c.c. of ether are added to the residue, and the mixture is allowed to stand aside overnight. The precipitated pilocarpine picrolonate is collected on a weighed filter, washed, dried, and weighed. Its molecular weight is 472. Six samples of jaborandi leaves yielded from 0·27 to 0·29 per cent. of pilocarpine.

W. P. S.

The Estimation of Antipyrine. J. D. Riedel. (*Pharm. Zeit.*, 1907, **28**, 290; *Pharm. Journ.*, 1907, **78**, 457.)—The solution of antipyrine (0·5 gram in 50 c.c.) is boiled with 5 or 6 c.c. of N-hydrochloric acid, and shaken while hot with an excess (about 10 c.c.) of a cold saturated solution of picric acid. After standing for a few hours separation of the crystalline picrate is complete, and the precipitate is collected on a tared filter, drained with the aid of a filter-pump, and, without washing, dried at 90° to 95°. An equivalent quantity of $\frac{N}{2}$ sulphuric acid may take the place of hydrochloric acid, but is liable to discolour the final dried compound. C. A. M.

Note on the Determination of the Reichert-Meissl and Polenske Values of Fats. A. Goske. (*Zeit. Untersuch. Nahr. Genussm.*, 1907, **13**, 491-492.)—It is stated that the Polenske value is influenced considerably by the mode in which the heat is applied to the distillation-flask: If the latter rests on a wire gauze coated with asbestos at its centre, the sides of the flask become overheated owing to the

large flame required in order to obtain the requisite volume of distillate in the given time. It is better to avoid the use of a gauze of any description and to heat the flask over a naked flame. The flasks employed should be of Jena glass. W. P. S.

The Influence of Preservatives on the Reichert-Meissl Value of Margarine. E. Bemelmans. (*Zeit. Untersuch. Nahr. Genussm.*, 1907, **13**, 492-493.)—The addition of benzoic acid to margarine causes a marked increase in the Reichert-Meissl value of the fat. This acid is readily soluble in fats and oils, and is distilled over with the volatile fatty acids during the ordinary course of carrying out the determination. 0·1 gram of benzoic acid, when added to 4 grams of neutral lard, gives a Reichert-Meissl value of 1·92. The increase due to this cause is particularly worthy of notice in the determination of the percentage of butter-fat in margarine. Salicylic acid is but seldom used as a preservative, and influences the Reichert-Meissl value to a smaller extent than benzoic acid. W. P. S.

Estimation of Lanoline in Soaps. K. Braun. (*Seifenfabrikant*, 1907, 257; *Chem. Zeit. Rep.*, 1907, **31**, 200-201.)—The following method is recommended as being accurate and rapid: 10 grams of the soap are dissolved in water, the solution is treated with concentrated calcium chloride solution, and the precipitate is collected on a filter. After being dried at a temperature of 60°, the precipitate is extracted in a Soxhlet apparatus with ethyl acetate in order to obtain the lanoline. A soap prepared with 5 per cent. of lanoline yielded 6·75 per cent. when examined by the above method. W. P. S.

Adulteration of Flour with Ivory-nut Meal. E. Bertarelli. (*Zeit. Untersuch. Nahr. Genussm.*, 1907, **13**, 484-488.)—A meal prepared from the residues of ivory-nuts (the seeds of a South American palm, *Phytelephas macrocarpa*) is sometimes used as an adulterant of flour. Its presence in a sample may be detected by steeping a portion of the latter in 3 per cent. sodium hydroxide solution for thirty minutes, then decanting the solution and washing the residue with water. The flour dissolves, and the insoluble residue, consisting of the ivory-nut meal, is then detected with the microscope. Another test consists in moistening the flour with phloroglucinol solution, adding hydrochloric acid, and cautiously warming the mixture. The particles of ivory-nut meal are at first coloured orange, the colour afterwards changing to red. Sawdust, if present, is also detected by this test, but the red coloration at once shows itself. Two samples of flour, containing large amounts of ivory-nut meal, gave the following results on analysis: Water, 8·5 and 9·8: proteins, 6·2 and 6·8; fat, 0·8 and 0·7; carbohydrates (by difference), 45·1 and 30·4; crude fibre, 38·8 and 51·5; ash, 0·6 and 0·8 per cent. W. P. S.

The Use of Mercuric Chloride as a Preservative of Milk reserved for Analysis. P. Grélot. (*Journ. Pharm. Chim.*, 1907, **25**, 423-428.)—The addition of 0·2 gram of mercuric chloride to a litre of milk enables the sample to be kept for several days without interfering with the analytical results. The solution of the antiseptic may be facilitated by the addition of a fourth of its weight of ammonium

chloride, which also has no appreciable effect upon the physical characteristics of the milk. The presence of mercuric chloride does not interfere with the detection of formaldehyde. C. A. M.

The Amount of Ash contained in Cayenne Pepper (Paprika). R. Windisch. (*Zeit. Untersuch. Nahr. Genussm.*, 1907, **13**, 389-398.)—The author considers that the amount of ash contained in Cayenne pepper should be from 7 to 8 per cent., this conclusion being arrived at from the examination of eighteen samples ground by himself. The calyx and stem were removed from the fruit before the latter was ground. The amount of sand in the samples varied from 0·09 to 0·26 per cent. (*cf.* ANALYST, 1904, **29**, 158). W. P. S.

Researches on the Anaerobic Microbes in Drinking Water. H. Vincent. (*Ann. Inst. Pasteur*, 1907, **21**, 62.)—A method is described for distinguishing between facultative and obligate anaerobes. The nutrient medium employed is prepared by dissolving from 10 to 15 per cent. of gelatine in peptone-bouillon and adding 1 per cent. of glycerol. After this mixture has been neutralised and sterilised, a sterile solution of sodium sulphindigotate is added, together with from 15 to 20 per cent. of skimmed milk, previously filtered and sterilised. The medium is melted at a temperature of about 35° C., mixed with a portion of the water under examination, and then drawn into sterilised tubes, the latter being 50 cm. long and having a diameter of 3 to 4 mm. Both ends of the tubes are now fused up, and the medium is solidified by placing the tubes under a stream of cold water. The colonies which form are easy to count, and the strictly anaerobic growths are distinguished from the sharply defined facultative anaerobes by their diffused boundaries. They are slightly cloudy, flaky, or granular, sometimes exhibiting thin, delicate projections. W. P. S.

Application of Chemical Analysis to the Examination of Commercial Spirits. E. A. Mann and C. E. Stacey. (*Journ. Soc. Chem. Ind.*, 1907, **26**, 287-289.)—The authors describe in detail the methods which they have adopted. The acidity was determined by titration with $\frac{N}{10}$ barium hydroxide with phenolphthalein as indicator, the fixed acids being calculated from the difference between the total and the volatile acidities, and not by the titration of the residue after distillation, owing to the colour of this residue. For the estimation of aldehydes, furfural and ethers, 195 c.c. were distilled off from 200 c.c. of the sample, and the distillate, diluted to 200 c.c., was employed for the analysis. In the colorimetric estimation of aldehydes by Gayon and Schiff's reagent, the chief difficulty was to obtain the alcohol free from aldehyde for purposes of dilution. This was effected by carefully fractionating spirit and redistilling the best fractions with Hewitt's reagent (sodium phenylhydrazine sulphonate). Standard solutions prepared from aldehyde-ammonia were very insensitive to Schiff's reagent, and the authors were compelled to use Kahlbaum's aldehyde. In the estimation of furfural with aniline acetate the standard solutions were distilled and treated in precisely the same manner as the test spirit. When the standard differs too widely from the test, the

weaker solution gives a coloration with a yellow shade which prevents accurate comparison. For the saponification of the ethers 100 c.c. of spirit were neutralised with barium hydroxide and boiled for one hour with 25 c.c. of $\frac{N}{10}$ sodium hydroxide. For the higher alcohols the authors at first used Marquardt's process as modified by Allen and Chattaway (ANALYST, 1891, **16,** 102), but subsequently they found that the results were obviously unreliable, and another process (*Journ. Soc. Chem. Ind.*, 1906, **25,** 1125) was adopted. For the colouring matter, Leach's fuller's-earth test was condemned as unreliable, since it removes the colour from genuine spirits; the ether-extraction test was therefore employed, 25 c.c. of spirit and about the same volume of ether being used. The authors give the results of the analysis of a large number of samples which, judged by authenticated standards, were condemned on the analytical numbers as doubtful, blended, or fictitious. J. F. B.

The Use of Carbon Bisulphide in the Estimation of Salicylic Acid in Wine. W. L. Dubois. (*Journ. Amer. Chem. Soc.*, 1906, **29**, 293-294.)—In extracting, by means of ether, salicylic acid from wines, tannin, colouring matters, etc., are simultaneously removed, and on separating these by extracting the dried ethereal residue with petroleum spirit only a small proportion of the salicylic acid is dissolved. Thus, out of 17·5 mgms. added to a wine, only 1·5 mgms. were obtained in the final petroleum spirit extract. The author has tried various other solvents (benzene, carbon tetrachloride, carbon bisulphide, ethyl ether) for separating the salicylic acid from the ethereal residue obtained in the usual way, and has found carbon bisulphide to be the most satisfactory. By means of it he recovered from 61 to 79 per cent. of the salicylic acid added to white wines, and from 75 to 88 per cent. in the case of red wines. C. A. M.

The Estimation of Cane-sugar, Reducing Sugars, and Dextrin in Wines. H. Pellet. (*Bull. Assoc. Chim. Sucr. et Dist.*, 1907, **24**, 1213-1215.)—The method adopted by the French Technical Commission (clarification of the wine with sodium bicarbonate and basic lead acetate, decolorisation with animal charcoal if required, and estimation of reducing sugars with Fehling's solution) is open to the objection that it does not yield comparable results in the hands of different chemists, apart from the fact that clarification with basic lead acetate is unsuitable for liquids containing reducing sugars. The author therefore prefers the following method:

Reducing Sugars.—The wine is heated, without previous clarification, upon the water-bath with Fehling's (or Violette's) solution, and the resulting precipitate washed, ignited, and weighed as cupric oxide. Sufficient wine should be taken to reduce about half the copper in the Fehling's solution used, and the total volume should be double that of the Fehling's solution.

Cane-sugar.—The wine is boiled on the water-bath for five minutes (or longer in the presence of much sucrose) with 2·5 c.c. of 10 per cent. hydrochloric acid, the reducing sugars estimated as before, and the difference between the two results calculated into sucrose.

Dextrin.—A definite quantity of the wine is heated for two or three hours on the boiling water-bath beneath a reflux condenser, with an equal volume of 8 per

cent. sulphuric acid, and the amount of reducing sugars once more determined. The difference between this result and that obtained after the inversion with hydrochloric acid gives the quantity of reducing sugars corresponding to the dextrin. The use of calcium or sodium hypochlorite is recommended for decolorising wine for examination in the polarimeter. C. A. M.

Determination of Sugars in Honey by Means of the Polarimeter. P. Lehmann and H. Stadlinger. (*Zeit. Untersuch. Nahr. Genussm.*, 1907, **13**, 397-419.) — Saccharose may be determined accurately in honey by the formula $y = \Delta.5{\cdot}725$, where y is the percentage of saccharose, Δ the difference between the readings, observed in the polarimeter, of a 10 per cent. honey solution before and after inversion, using a 200 mm. tube. The readings are taken in angular degrees at a temperature of 20° C., and 10 grams of honey diluted with water to a volume of 100 c.c. constitute the 10 per cent. solution. A formula—$x = 0{\cdot}256\ (P + 30)$—is also given for the determination of starch-syrup in honey, where x is the percentage of starch-syrup and P the polarimeter reading of a 33·33 per cent. solution of the sample, the reading being expressed in Soleil-Dubosq degrees (*i.e.*, angular degrees multiplied by 4·588). W. P. S.

The Terpene Oils of Manila Elemi. A. M. Clover. (*Philippine Journ. Sci.*, 1907, **2**, 1-40.) — The results yielded by the examination of 21 individual samples of the resin are given with a view of establishing the composition of elemi oil. The resin is obtained from trees of which the species are unknown, but it is probable that they all belong to the general order of *Burseraceæ*. On distillation, 10 of the samples gave pure dextro-limonene, and 9 of the remaining ones more or less phellandrene, whilst 2 gave almost optically inactive oils, which were found to be terpinene and terpinoline. The total amount of terpene oil obtained from the various samples of resin varied from 9·7 to 16·2 per cent. The quantity of higher boiling oils was from 6·2 to 15·8 per cent. The variations in the composition of the oils is due to the different sources from which the resins were obtained, and also to the age of the resin. W. P. S.