ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Study of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients: II. (Estimation of Aconitine, Codeine, Cocaine, Morphine, and Strychnine.) J. W. Marden and V. Elliott. (*J. Ind. and Eng. Chem.*, 1914, **6**, 928-934.) (*Cf.* ANALYST, 1914, **39**, 354.)—Chloroform is a much better solvent for aconitine than is ether, which is recommended for the purpose by the U.S. Pharmacopœia. Three extractions, each with 10 c.c. of chloroform, will extract more than 99·9 per cent. of the aconitine from 50 c.c. of an ammoniacal aqueous solution. Six similar washings with ether are necessary to extract even 99 per cent. Chloroform is an even better solvent for codeine, two washings sufficing to remove practically 99·9 per cent., using the quantities stated above. For the extraction of codeine, ether is quite useless, the alkaloid being almost equally soluble in wet ether and in water saturated with ether. On the other hand, ether is nearly as good a solvent for cocaine as chloroform is for codeine, two washings sufficing to extract 99·8 per cent., using the quantities stated above. Two washings with chloroform will remove this percentage of strychnine from aqueous solution, whereas mixtures of ether and chloroform, recommended by many authors, are much less efficient.

The difficulty in extracting morphine completely is discussed, and it is suggested that, if the distribution co-efficient for morphine between water or a saline solution and some immiscible solvent under closely defined conditions were accurately

determined, it would suffice to work with measured volumes, determine the morphine in an aliquot portion of the immiscible solvent, and calculate that in the remainder of the immiscible solvent as well as that remaining in the aqueous layer. Unfortunately, the authors' determinations of the distribution ratios for morphine are not sufficiently concordant to permit them to recommend a method based on this principle, but it is suggested that the principle may have other applications.

It is said that "terpeneless" lemon extract is made by extracting citral from lemon-oil by means of 45 per cent. alcohol. It is shown that alcohol of this strength is a poor solvent for citral compared with the residual terpenes, and that a large quantity of alcohol is necessary to extract 75 per cent. of the citral, even if applied in successive small portions. Alcohol of 50 per cent. strength is shown to be three times as good a solvent for citral, but whether alcohol of this strength dissolves any appreciable amount of terpene as well is not stated. G. C. J.

Method for Quantitative Determination of Resins in Hops. Ö. Winge and J. P. H. Jensen. (*Compt. rend. des trav. du Lab. de Carlsberg*, 1914, **11**, 116-147.)—An error has hitherto been made in considering the γ resin of hops as valueless, since during the brewing process it both imparts a flavour to the wort and assists in the precipitation of the albumens. The resins of hops, which are all soluble in cold ethyl ether, are not all equally bitter and equally valuable, but, as the relation between their bitterness can be expressed by the proportion, $\alpha : \beta : \gamma = 10 : 7 : 4$, the total quantity of resin extracted from the hops by means of cold ether and determined by potash titration is an approximately accurate expression of the bitterness value of hops. The analytical methods hitherto used, involving the use of petroleum ether, being based on a separation of the so-called soft resins from the hard resin, rest on an insecure basis and give misleading results. The following method is recommended: Thirty grms. of hops are pulverised by means of a meat machine, the first 5 grms. are discarded, and the remainder well mixed. About 5 grms. are transferred to a previously weighed 300 c.c. Erlenmeyer flask. The whole is dried in a vacuum for twenty-four hours at 35° C., the loss of weight giving the moisture content of the sample, after which 150 c.c. of ethyl ether free from water and alcohol are added, and the mixture left to stand, with repeated shaking, for one hour. The liquid is filtered and a careful washing with ethyl ether is made by means of a wash-bottle with fine jet. This washing is very important, and requires about 100 c.c. of ether. The solution is then titrated with 0·05 N potassium hydroxide solution in 93 per cent. alcohol, using phenolphthaleïn as an indicator. Since 1 c.c. of $\frac{N}{1}$ potassium hydroxide corresponds to 0·40 grm. of the mixed α, β and γ resins, the desired percentage contents of resins, based on the dry weight of the hops, becomes $x = \frac{2y}{z}$, where y = c.c. of 0·05 N potash required, and z = the dry weight of the hops in grms. The average value of 0·40 grm. of resin for 1 c.c. $\frac{N}{1}$ KOH was found as the result of analysing twenty samples, the titration factors for the three resins being as follows:

0·32 grm. α resin = 1 c.c. $\frac{N}{1}$ KOH
0·40 ,, β ,, = 1 ,, ,,
0·06 ,, γ ,, = 1 ,, ,,

The authors have made a comparison between the ethyl ether cold extraction method above described, the carbon tetrachloride extraction method of Seibriger (*Wochenschr. f. Brauerei*, 1913, **30**, 530), and the ammonia method of Nilson (*Brewers' Congress*, 1911, 104), and reasons are adduced for the preference being given to the ether process. H. F. E. H.

Influence of Fineness on the Availability of Bone-Meal. S. S. Peck. (*J. Ind. and Eng. Chem.*, 1914, **6**, 922-924.)—Since there is a limit to which bone-meal can be ground to permit of its convenient handling, fine bone-meal could possibly be defined as that which passes through a 50-mesh sieve. It is not commercially practicable, however, to prepare so fine a product without also including a considerable proportion of very fine dust, so a standard of fine bone-meal of 65 per cent. to pass a 50-mesh sieve, and at least 90 per cent. of the remainder to pass a 25-mesh sieve, is suggested as one to which no reasonable objection can be offered by the dealers, and from which satisfactory results will accrue to the crops. W. P. S.

Examination of Meat Extract. J. Smorodinzew. (*Zeitsch. Physiol. Chem.*, 1914, **92**, 214-221.)—A comparison was made of the yield of bases obtained by the following methods: (1) Treatment with a 10 per cent. mercuric sulphate solution in 5 per cent. sulphuric acid and precipitation of the filtrate after removal of mercury with phosphotungstic acid. (2) Treatment with neutral lead acetate and precipitation of the filtrate, after removal of lead, with phosphotungstic acid. (3) Precipitation with a solution of phosphotungstic acid in 5 per cent. sulphuric acid after previous treatment with lead acetate. (4) Direct precipitation with phosphotungstic acid without addition of sulphuric acid. The best yields of purine bases and carnosine were obtained from the precipitation with mercuric sulphate. The yield of carnosine is considerably reduced by the addition of sulphuric acid in the phosphotungstic acid precipitation. The addition of lead salts reduces the yield of carnitine and only slightly improves that of methylguanidine. The preliminary treatment with lead acetate is objectionable, because acetic acid hinders the precipitation with phosphotungstic acid and necessitates the use of a much larger quantity of this acid: the quantity of this acid required is considerably reduced by the addition of sulphuric acid. Purine bases are not completely precipitated by phosphotungstic acid, and their precipitation is apparently not affected by lead salts or sulphuric acid. E. W.

Carnosine, Methylguanidine, and Carnitine in Mutton. J. Smorodinzew. (*Zeitsch. Physiol. Chem.*, 1914, **92**, 221-228.)—The extract of mutton is poorer in nitrogen than that of beef. Extract of mutton was found to contain twice the quantity of purines and nearly twice the quantity of carnitine, but only one-third the quantity of carnosine and one-half the quantity of methylguanidine present in beef extract. Mutton also contains more purines and carnitine and less carnosine and methylguanidine than is contained in horseflesh. E. W.

Specific Colour Reaction of Marine Animal Oils and their Hydrogenated Products. M. Tortelli and E. Jaffe. (*Annali Chim. Applic.*, 1914, **2**, 80-98.)—Marine animal oils and their hydrogenated products contain a chromogenic substance which reacts with bromine to form a compound which imparts a bright green coloration to a chloroform solution of the oil. One c.c. of the sample is shaken with 6 c.c. of chloroform and 1 c.c. of glacial acetic acid in a glass cylinder, and the homogeneous solution treated with 40 drops of a 10 per cent. solution of bromine in chloroform, vigorously shaken, and allowed to stand. In the presence of a marine animal oil a fugitive rose coloration, changing to bright green, will be obtained, and will persist for at least an hour. Vegetable oils under the same conditions usually remain colourless or assume a yellow tint, which does not alter within an hour. In the case of hemp-seed oil, however, the chloroform solution becomes green on the first addition of bromine and then changes to yellow. Certain animal oils—*e.g.*, neat's-foot oil—give a slight rose coloration, changing to yellow within an hour; but in no instance can the reaction be confused with that given by marine animal oils. Hydrogenated fish and marine animal oils give a rose coloration, changing within a minute to pale green, and after another minute to emerald green, whereas hydrogenated vegetable oils at first remain unaffected or assume a light yellow tint, which becomes yellow or brown after an hour, whilst hydrogenated terrestrial animal oils immediately assume a yellow or light brown tint, changing to deep brown after an hour. As little as 5 per cent. of a hydrogenated marine animal oil may thus be detected in vegetable lard substitutes, etc. Ordinary butter gives a yellowish tint, appearing slightly green by reflected light, but not such as could be confused with the distinctive reaction of marine animal oils. C. A. M.

Characteristics of Olive Oils extracted with Carbon Disulphide. F. Canzoneri and G. Bianchini. (*Annali Chim. Applic.*, 1914, **2**, 1-9.)—Olive oils extracted with carbon disulphide differ from expressed oils: (1) In the higher specific gravity (0·920, Fritsch; 0·927, Klein at 15·5° C.); (2) the lower solidification-point of the fatty acids (*e.g.*, 17·5° to 19·7° C.), their lower iodine value (77·5 to 80·2), and their higher acetyl value; (3) the lower refractometer reading (59° to 61° Zeiss), except in the case of oils bleached by oxidation, in which the refractometer reading is higher (63°); (4) the saponification value, which is lower than normal; (5) the lower iodine value; (6) the presence of considerable quantities of sulphur. Traces of sulphur in olive oil may be detected by shaking 10 c.c. of the oil with a globule of mercury or a strip of copper, and allowing the tube to stand for some time, or heating it for fifteen minutes at 100° C. Mercaptans, which are invariably present in commercial extracted oils, may be detected by distilling the sample in a current of steam, and testing the distillate with mercuric chloride. In recent methods of refining, industrial olive oils are heated at 38° to 45° C., and submitted to the prolonged action of steam at a low temperature to remove sulphur compounds of unpleasant odour. Colza and ravison rape oils do not give the mercury reaction, and only contain traces of sulphur, especially when refined. They may be distinguished from extracted olive oils by their high refractometer reading (70°). C. A. M.

Notes on Halphen's Reaction for Cottonseed Oil. E. Gastaldi. (*Annali Chim. Applic.*, 1914, **2**, 203-207.)—The coloration obtained in Utz's modification of Halphen's test (ANALYST, 1914, **39**, 92) when the temperature exceeds 160° C., as is usually the case, is also given by fatty acids (oleic, palmitic, stearic acids), and the reaction is therefore not characteristic of cottonseed oil. The following rapid modification of Halphen's test will detect small quantities of cottonseed oil: Five c.c. of the sample are heated for four to five minutes in a tube over a direct flame with 5 to 6 drops of a solution of sulphur in carbon disulphide, and 3 to 4 drops of pyridin, care being taken that the temperature of the fat does not rise above 140° C.

C. A. M.

Estimation of Camphor and of Certain Essential Oils when in Solution in Alcohol. W. B. D. Penniman and W. W. Randall. (*J. Ind. and Eng. Chem.*, 1914, **6**, 926-928.)—The method proposed depends on the fact that camphor and the essential oils of peppermint, lemon, orange, anise, and nutmeg, are liberated from their alcoholic solution when this is mixed with from four to ten times its volume of concentrated calcium chloride solution, and that the separated camphor or oil is soluble in petroleum spirit (b.-pt., 40° to 60° C.). Within certain wide limits the increase in volume of the petroleum spirit is equal to the volume of the camphor or essential oil. A measured quantity of the alcoholic solution under examination is mixed with calcium chloride solution in a flask having a graduated neck (a Babcock milk-bottle), a definite volume of petroleum spirit is added (about equal to the volume of the oil to be dissolved), more of the calcium chloride solution is introduced so as to bring the mixture into the neck of the flask, the whole is submitted to centrifugal action, and the increase in the volume of the petroleum spirit is noted. W. P. S.

Proposed Specification for Turpentine. (*Report of the American Society for Testing Materials*, 1914, 91-118.)—As the result of the analysis of authenticated samples of turpentine, the following specification is suggested:

1. The specification applies both to the turpentine which is distilled from pine oleo-resins, and commonly known as gum turpentine or spirits of turpentine, and to the turpentine known as wood turpentine, which is obtained from resinous wood, either by extraction with volatile solvents, or by steam, or by destructive distillation. No attempt is made to differentiate between the two. The purchaser, when ordering, should specify whether gum or wood turpentine is desired.
2. The turpentine must be clear and free from suspended matter and water.
3. The colour shall be to standard.
4. The sp. gr. at 15·5° C. shall be not less than 0·860 or more than 0·875.
5. The refractive index at 15·5° C. shall be not less than 1·468 or more than 1·478.
6. The initial boiling-point must lie between 150° and 160° C.
7. Ninety per cent. of the turpentine shall distil below 170° C.
8. The polymerisation residue shall not exceed 1 per cent., and its refractive index at 15·5° C. shall be not less than 1·500.

The polymerisation test is carried out as follows: Twenty c.c. of sulphuric acid

(100·92 per cent.) are placed in a graduated Babcock flask, and cooled by placing the flask in ice-water. Five c.c. of the turpentine are then added slowly and mixed with the acid, the temperature not being allowed to rise above 60° C. When the mixture no longer warms up on shaking, it is agitated thoroughly, the flask is placed for ten minutes in a water-bath at 60° C., and shaken occasionally to prevent separation. After cooling to room temperature, the flask is filled with concentrated sulphuric acid until the unpolymerised oil rises into the graduated neck; it is then subjected to centrifugal action (1,200 revolutions per minute) for five minutes, and, after standing for twelve hours, the volume of the unpolymerised residue is read off. W. P. S.