

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Use of Miscibility Curves in the Examination of Spirits of Camphor. **H. Rosset.** (*Ann. Chim. anal.*, 1913, **18**, 49-56.)—The author has extended the methods described previously by Louise (*ANALYST*, 1907, **32**, 365; 1910, **35**, 322; 1911, **36**, 556) to the analysis of alcoholic solutions of camphor, and shows that it is possible to estimate both the camphor and the alcohol from the miscibility curves obtained. The alcohol is found from curves plotted from the results given by mixtures of alcohol and standard petroleum spirit, and the amount of camphor from the miscibility curves of mixtures of alcohol and nitrobenzene. W. P. S.

Polenske "Difference Value" for the Detection of Certain Animal Fats and its Theoretical Basis. **A. Bömer and R. Limprich.** (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 367-386.)—The method described by Polenske (*ANALYST*, 1907, **32**, 382; 1908, **33**, 476) is of use for the detection of beef or mutton fat in lard when the quantity of the former is not less than from 15 to 20 per cent., and the method depends on the difference in composition of the glycerides of lard and beef fat. The value is chiefly affected by the palmitodistearin present in the fats, the α -palmitodistearin of lard giving the fat a "difference value" of 18.4, whilst β -palmitodistearin is the source of the "difference value" of 11.8 in the case of beef or mutton fat. No advantage is derived from determining the "difference value" of mixed glycerides which crystallise from an ethereal solution of the fats. W. P. S.

Mixed Glycerides of Palmitic and Stearic Acids Present in Lard. **A. Bömer.** (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 321-353.)—By repeated fractional precipitation and crystallisation of lard from ether, the author has isolated the saturated glycerides of this fat. Tristearin is not present in lard, and in this respect the latter differs from beef and mutton tallow. The insoluble glyceride obtained from lard is palmitodistearin and not heptadecyldistearin, as stated by Kreis and Hafner (*ANALYST*, 1904, **29**, 259). Heptadecyldistearin could not be detected in lard. As the palmitodistearin obtained from lard differs in its melting-point and crystalline form from the palmitodistearin derived from mutton tallow, it is probable that the former is α -palmitodistearin and the latter β -palmitodistearin. Lard was also found to contain a second saturated glyceride—namely, stearodipalmitin. The lard used in the experiments yielded about 3 per cent. of α -palmitodistearin, melting-point, 68.5° C., and about 2 per cent. of stearodipalmitin, melting-point 58.2° C. W. P. S.

Lecithin Preparations and the Estimation of Lecithin. R. Cohn. (*Zeitsch. öffentl. Chem.*, 1913, **19**, 54-62; through *Chem. Zentralbl.*, 1913, I., 1129-1130.)—The estimation of lecithin may be divided into three parts—namely, the extraction of the lecithin, its purification, and the estimation of the phosphorus in the product. For the extraction of the lecithin from 1 to 2 grms. of commercial preparations of lecithin, or from 5 to 20 grms. of a food material containing lecithin, are extracted for several hours with two successive quantities of 100 c.c. of 96 per cent. alcohol, the first extraction being carried out at the ordinary temperature and the second at the boiling temperature of the alcohol, a reflux apparatus being used in this case. The residue is then ground up with sand, extracted once more with alcohol, and then boiled for two hours with about 100 c.c. of chloroform. When dealing with fatty substances, the chloroform extraction may be made with advantage immediately after the first extraction with cold alcohol. The ease with which lecithin may be extracted from a preparation depends largely on the treatment to which it has been subjected during manufacture; prolonged heating or lengthy storage renders the lecithin less soluble, and in certain cases, the extraction with hot alcohol must be continued for, say, twenty hours, before phosphorus compounds cease to be extracted. After the alcohol and chloroform extracts have been evaporated, the residue obtained is boiled for two hours with 100 c.c. of chloroform in order to separate the lecithin from glyceryl-phosphoric acid and free phosphoric acid; the chloroform solution is then filtered and evaporated. The quantity of phosphorus in this residue is then estimated by oxidising it with nitric acid and sulphuric acid, or igniting it with the addition of magnesium oxide, or a mixture of sodium carbonate and potassium nitrate, precipitating the resulting phosphoric acid with molybdic acid solution, and converting the molybdate precipitate into ammonium magnesium phosphate in the usual way.

Commercial preparations of lecithin frequently contain less than the guaranteed quantity of lecithin; the author considers that a substance sold as pure lecithin should contain, at the least, from 90 to 95 per cent. of lecithin, and that the quantity of the latter in any preparation should be within 10 per cent. of the amount claimed by the manufacturers to be present. The quantity should be expressed as lecithin itself, and not as lecithin-albumin or lecithin-protein, as these are of variable composition, and may contain from 5 to 30 per cent. of lecithin.

The author does not attempt to solve the question whether yolk of egg contains lecithin in chemical combination, but points out that, as lecithin may be almost completely extracted from yolk of egg by means of cold alcohol, the presence of combined lecithin is improbable. The insolubility of lecithin under certain conditions is possibly due to adsorption by proteins.

W. P. S.

Effect of Boiling on the Physico-Chemical Behaviour of Human Milk, Cow's Milk, and Buttermilk. P. Grosser. (*Biochem. Zeitsch.*, 1913, **48**, 427-432.)—The experiments were undertaken to determine whether the two kinds of milk are affected differently by boiling, and if it is possible to find a difference between raw and boiled milk by physico-chemical methods. The milk, from which the cream was removed by means of a separator, was filtered through a Bechold ultrafilter by

means of compressed nitrogen at 6 atmospheres. The filtrate, which was free from colloids, was examined with regard to depression of freezing-point, and nitrogen, phosphorus, and lime content. The results indicate that boiling does not affect the freezing-point, and in the case of cow's milk scarcely affects the phosphoric acid and nitrogen. In human milk the phosphorus and nitrogen sink considerably on boiling. The lime content of the ultrafiltrate of both milks diminished on continued boiling, the diminution being greater in human milk. In the case of butter milk, boiling produced no change in the values determined. E. W.

Detection of Saffron in Confectionery. C. Martini. (*Staz. sperim. agrar. ital.*, 1912, **46**, 18-24; through *Chem. Zentralbl.*, 1913, I., 1068.)—The finely powdered confectionery is extracted for twenty-four hours in the cold with 70 per cent. alcohol with frequent agitation. Fifty grms. of the powdered residue are then boiled for fifteen minutes with 100 c.c. of 70 per cent. alcohol under a reflux condenser on the water-bath. After filtration, the residue is again boiled with fresh alcohol, the united extracts are concentrated on the water-bath; it is then exhausted with ether. The extract, after evaporating the ether, is boiled with 98 per cent. alcohol, and the latter solution evaporated. The product is then tested by the well-known colour reaction for saffron, with sulphuric and nitric acids. J. F. B.

Chemical Composition of Authentic Vanilla Extracts, together with Analytical Methods and Limits of Constants. A. L. Winton and E. H. Berry. (*U.S. Dept. Agric., Bureau of Chem.*, Bull. No. 152.)—After a discussion of the commercial origin of various kinds of vanilla beans, and the processes of manufacture of vanilla extract, methods of analysis are fully described, including the determination of vanillin and coumarin by the modified Hess and Prescott method (*J. Amer. Chem. Soc.*, 1899, **21**, 256; *ibid.*, 1902, **24**, 1128; *ibid.*, 1905, **27**, 719; *U.S. Dept. Agric., Bureau of Chem.*, Bull. 132, p. 109, and Bull. 137, p. 120); the normal lead number (Winton and Lott method, *U.S. Dept. Agric., Bureau of Chem.*, Bull. 132, p. 110, and Bull. 137, p. 120); the colour value of the extract; the residual colour after precipitation with lead acetate; and the colour insoluble in amyl alcohol (Tolman and Hillyer, *Bull.* 122, p. 206; Bull. 132, p. 90). A large number of samples of known origin were examined in the laboratory by preparing extracts from them, following the method employed by the U.S. Pharmacopœia, in order to learn the influence of variety, grade, and length of bean on the composition of the vanilla extract. In discussing the results of these analyses (all the figures of which are recorded) it is observed that the extracts from Ceylon beans were so abnormal and variable in composition as to indicate either unusual curing or previous extraction.

Vanillin.—The range in vanillin content ran from 0.11 to 0.31 grm. per 100 c.c., and is somewhat greater than has usually been thought possible; but the figure 0.25 stated by Leach to be regarded with suspicion refers to extracts on the commercial scale and not in the laboratory. The minimum figure (0.11) was found in Tahiti extracts prepared from undried beans.

Normal Lead Number.—The variation in this value (0.4 to 0.74) is less than that of any other constant. The constituents yielding the precipitate appear to be more easily soluble than the vanillin and the colour. This determination not only serves to distinguish a true extract from a solution of vanillin, but is also of value taken along with a coumarin estimation as a means of detecting the presence of Tonka extract.

Colour Values of Extract and Lead Filtrate.—The colour is the most variable and difficultly extractable constituent. As a means of detecting caramel, the percentage of colour left in the lead filtrate is most significant. The maximum found in these samples (excluding Ceylon) was 8 per cent. red and 10 per cent. yellow, these figures being far surpassed by caramel-coloured extracts. As regards length of bean, the differences found, although not marked, point to a slight inferiority of the shorter beans.

The next section deals with vanilla extracts prepared with different solvents and solvent mixtures in the laboratory, the standard solvent being 60 per cent. alcohol and sugar, the other solvents examined being (1) 60 per cent. alcohol alone; (2) 60 per cent. alcohol and glycerol; (3) 35 per cent. alcohol alone; (4) 35 per cent. alcohol and sugar; (5) 35 per cent. alcohol and glycerol. The 35 per cent. extracts were highly unsatisfactory, owing to the extraction of gelatinous material which clogged the percolators. With the same strength of alcohol the only noteworthy difference was found to be that the glycerol extracts were more strongly coloured, thus explaining the popularity of glycerol for commercial extracts.

Standard Vanilla Extract.—The following tentative limits of composition for standard vanilla extract (10 grms. of beans to 100 c.c.) appear to be warranted by the results obtained:

Vanillin, 0.10 to 0.35 grm. per 100 c.c.

Normal lead number, 0.40 to 0.80.

Per cent. of total colour in lead filtrate, not more than 10 per cent. red, or 12 per cent. yellow.

Ratio of red to yellow in the extract, not less than 1 : 2.2.

Colour insoluble in amyl alcohol, not more than 40 per cent.

(*Cf.* O. Folin and W. Denis, *ANALYST*, 1912, **37**, 501.)

H. F. E. H.

Direct Estimation of Water in Foods, etc., by Distillation. F. Michel. (*Chem. Zeit.*, 1913, **37**, 353-355.)—This method has been described previously by Aschmann and Arend (*ANALYST*, 1907, **32**, 21) who have employed it for the estimation of water in butter and other fats, and the author now shows that it may be applied to almost all classes of foods. He recommends the use of a mixture consisting of 1 volume of toluene and 2 volumes of xylene, about 150 c.c. of this mixture being employed for 20 grms. of the substance under examination. Ordinary paraffin, having a boiling-point above 100° C., may also be used. The distillate is collected in a narrow graduated cylinder, and, at the end of the distillation, any drops of water remaining in the condenser may be transferred to the receiver by means of a rubber-tipped rod or feather dipped in xylene. In order that the volume of the

water in the receiver may be determined accurately, the contents of the receiver may be submitted to centrifugal action. A correction must be made on the volume of water observed owing to the change of meniscus caused by the hydrocarbon layer above the water layer; for instance, if a quantity of xylene be poured on the surface of 5 c.c. of water contained in a narrow cylinder, the water will then show an apparent volume of 4.85 c.c. Consequently, 0.15 c.c. is added to the volume of water found. The author finds that a further quantity of 0.03 c.c. must be added for each c.c. of water collected in the receiver in order to allow for loss during the distillation. When these allowances are made, the error of the method is not greater than ± 0.2 per cent.

W. P. S.