PHARMACEUTICAL ABSTRACTS

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BOTANY (Continued)

Tannin—Rôle of, in the Living Plant. The author reviews the literature describing studies intended to determine the function of tannins in the living plant.—K. GANZINGER. Scientia Pharm., 8 (1937), 107. (M. F. W. D.)

CHEMISTRY

GENERAL AND PHYSICAL

Calcium Hypochlorite. The equilibrium diagram water-calcium hypochlorite-calcium chloride-calcium hydroxide at 50° is discussed with reference to the production of Ca(OCl)₂.2Ca(OH)₂(I).3Ca(OCl)₂.2Ca(OH)₂.2H₂O(II), and Ca(OCl)₂.3H₂O (III) by chlorination of milk-of-lime. No compounds of calcium hypochlorite with calcium chloride exist in solution (15-50°). I exists only above 20° and in absence of supersaturation. III is obtained by chlorination of a thick suspension of I in presence of crystals (to avoid supersaturation and formation of fine crystals); II is obtained free from III only from dilute suspensions (temperature greater than 35°). After dehydration III contains 75% of active chlorine, and is 20-30 times as stable as bleaching powder. Water and calcium chloride contents, catalyzing impurities and physical form affect its stability. Uses are discussed.—J. Ourisson. Bull. soc. ind. Mulhouse, 103 (1937), 217-226; through J. Soc. Chem. Ind., 56 (1937), 1044.

Carbon—Colloidal, p_H Properties of. On boiling colloidal carbon with distilled water a reproducible p_H is obtained with the sludge. The p_H does not change after organic extraction of the carbon. Impingement carbon and lampblack give low, while thermal-decomposed carbon and furnace carbon give high, p_H values. It is suggested that these properties are related to the presence of the complex C_xO_y , since in the absence of oxygen, alkalinity is always shown provided that the particle size is small enough; with progressive addition of combined oxygen the acidity increases and the minimum $p_H(2.6)$ probably occurs at saturation of the surface with the complex.—W. B. Wiegand. Ind. Eng. Chem., 29 (1937), 953-956. (E. G. V.)

Dextrin—Molecular Weight of. M. discusses critically the molecular weight of dextrin and applies v. Euler's equation to four malt amylase preparations $k \cdot \sqrt{M} = 7$; k is diffusion constant at 20°. The concentration of dextrin in the layers are determined by the polariscope. The median molecular weight of eleven observations is 2540. A corresponding value by a reduction method is 2200, a reasonable agreement at the present stage.—Karl Myrback. Svensk Kem. Tid., 49 (1937), 145–148; through Chem. Abstr., 31 (1937), 6950. (F. J. S.)

Emulsion—Water/Oil or Oil/Water Type, an Apparatus for Demonstrating. A number of methods are available for indicating if the continuous phase of an emulsion is oil or water. A method suggested by Bhatnagar (J. C. S. Abs. (1920), 544) depends on the conductivity of the continuous phase when aqueous, and the increase in resistance to infinity as the emulsion changes to the oil-in-water type, this increase being measured by means of a milliameter in series with it and a source of high potential. A method utilizing the same general principles as Bhatnagar's was developed by the author, using a neon tube instead of a milliameter. The method depends on the production of a glow, caused by insertion of two electrodes in the emulsion to be tested, within the neon tube.—W. J. Pullar. Pharm. J., 140 (1938), 7. (W. B. B.)

Emulsion Particles—Size-Frequency Distribution of. As part of an investigation into the design and operation of emulsifying machinery, size-frequency analyses were carried out on sixty oil-in-water emulsions of the same chemical composition, but treated in a colloid mill under different conditions of rotor-speed, gap and time of action. The emulsions were prepared from a refined paraffin oil of d^{25} 0.8735, and viscosity 0.291 poise at 25°(the temperature of the experiments). This was emulsified with an equal volume of potassium oleate solution, prepared from "oleine B. P." neutralized with caustic potash, and containing 2% by weight of fatty acid. The size-frequency distributions of the emulsions were determined to see how their form was affected by conditions of manufacture. The paper is divided into four sections, dealing with (1) the method used to determine the size-frequency distributions, (2) the statistical methods used to describe and classify the distributions, (3) the results found in practice, and (4) consideration of a general equation for the distributions.—F. A. COOPER. J. Soc. Chem. Ind., 56 (1937), 447T.

(E. G. V.)

Graph—Titration, a Model Logarithmic. It is interesting and instructive to attempt to follow the changes in ionic concentration during the course of a typical neutralization of an acid

and a base. It may be shown that in the earlier stages of the titration the change in p_H is slow. These changes are well shown graphically by plotting p_H against percentage neutralization, and the form of the graph is the familiar sigmoid curve. The calculation of the p_H at the desired stages of neutralization of a weak acid may be calculated from the equation: $p_H = pK_a - \log_a - \log_a$ where K_a = dissociation constant, c = number of cc. alkali added and a = number of cc. alkali still required. In the original presentation of the logarithmic titration graph, the logarithmic titration curves for several typical acids and bases had been calculated, and were shown on the graph.—E. J. Schorn. *Pharm. J.*, 140 (1938), 7. (W. B. B.)

Lead Arsenates—Method for Controlling the Holding in Suspension of. A suitable amount of the arsenate is diluted in a test-tube with water to a height of 30 cm. and allowed to stand for 15 minutes. The upper, middle and lower thirds of the liquid are drawn off into different flasks and the amount of lead is determined in each portion, thus obtaining the ratio of the weights of the active ingredient. Wide variations were observed in different samples studied; e. g., in the upper third, the percentage of lead can vary from 0.1 to 22.5%, while in the middle third it is about 20 to 30%. By means of these figures an arbitrary coefficient can be determined showing the differences in the homogeneity of the samples by taking the ratio between the arsenate content of the middle third to the ideal content of 33.3%.—G. PAULIAN. Bull. Inst. Œnol. Algerie, 9 (1936), 60-65; through Chimie & Industrie, 38 (1937), 162. (A. P.-C.)

Powder Measurements. Methods are described for determining (a) the density of the particles of a powder after delivery into a container and after settling by prolonged shaking, and (b) the limiting surface inclination of a heap before slipping occurs.—H. W. Gonell. Arch. tech. Messen, No. 70 (1937), 47–48T; through J. Soc. Chem. Ind., 56 (1937), 1142. (E. G. V.)

Viscosity Determination—New Method For. An instrument and technic for measuring viscosity are described. A number of cups of equal size but with apertures of different size at their base are placed in the test liquid. The time taken for the liquid to drain from the appropriate cup is noted and viscosity read from a table, or, if relative readings only are required, the time is used directly to give viscosity. The size of the aperture chosen should be such that drainage takes not less than 20 seconds.—E. A. Zahn. Gen. Elec. Rev., 40 (1937), 285–286; through J. Soc. Chem. Ind., 56 (1937), 1142. (E. G. V.)

ORGANIC

Alkaloids

Alkaloids—Action of Bromoacetates on Various. The reaction between alkaloids and sodium bromoacetate may result in: (1) detoxication (e. g., strychnine) where the toxicity appears to lie in the free amine groups; (2) no detoxication (e. g., colchicine), where the toxicity lies in existence of the —NCH₃ group.—L. Espil and G. Mandillon. Compt. rend. acad. sci., 202 (1936), 2177–2179; through Chimie & Industrie, 38 (1937), 527. (A. P.-C.)

Alkaloids—Methods for Titration of. Attention is drawn to inconsistencies in the present A. O. A. C. methods for the titration of different alkaloids and the literature on the subject is briefly discussed.—R. L. Herd. J. Assoc. Official Agr. Chem., 20 (1937), 602-604; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Ergonovine—Determination of, in Ergot Preparations. The colorimetric method is utilized and is based on the principle that ergonovine is precipitated with picric acid only in strong concentrations, whereas the other alkaloids are precipitated completely in weak concentrations. Method: The fluid extract or other commercial preparation is extracted as usual first with ether at an alkaline reaction and again with ether with tartaric acid. The tartaric acid solution is concentrated to any desired volume. A portion of this solution is tested for total alkaloids with Smith's reagent; another portion is tested for ergonovine. To 2 cc. of the tartaric acid solution are added 4 drops of a warm saturated solution (1.5%) of picric acid and 2 drops of dilute hydrochloric acid. The solution is filtered three times on the same filter and to 2 cc. of the filtrate is added Smith's reagent. The ergonovine content is determined by the intensity of coloration. The color can be matched against a known sample of pure ergonovine or against a stable artificial color consisting of trypan blue and Berlin blue. Thus, 2 cc. of tartaric acid extract containing 0.16 mg, ergonovine produces an intensity of color equal to a mixture of equal parts of trypan blue 0.0008% and solution of Berlin blue 0.0002%.—EMILIO TRABUCCHI. Boll. soc. ital. biol. sper., 12 (1937), 232-234; through Chem. Abstr., 32 (1938), 303. (F. J. S.)

Ergot—Alkaloids of. A review.—L. Kofler. Pharm. Monatsh., 18 (1937), 210-212.
(H. M. B.)

Glycols and Alkaloids—Reaction Products of. The reaction product of a glycol with quinine, strychnine, codeine or ephedrine is claimed as a new therapeutic composition.—Samuel Ruben. U. S. pat. 2,099,432, Nov. 16, 1937.

(A. P.-C.)

Hydrastinine, Scopolamine, Hyoscyamine, Eserine and Apiol—Mercurimetric Determination of. New Reaction for Identifying Apiol. Determination of these substances is based on their precipitation by the Mayer-Valzer reagent, destruction of the complex by digesting with sulfuric and nitric acids, precipitation of the mercuric ion with nitroprusside, and titration with standard sodium chloride solution. The new reaction for the identification of apiol is based on the action of phosphomolybdic and concentrated sulfuric acids on apiol in hydro-alcoholic solution; a greenish blue color is produced.—Al. Iomesco-Matiu and C. Popesco. Bull. Soc. Stiinte Farm. Romania, 1 (1936), 61-67; through Chimie & Industrie, 38 (1937), 524. (A. P.-C.)

Papaver Rhoeas—Constituents of. Rhoeadine, kryptopine and chelidonine dissolve in acetic anhydride without coloration. Under ultraviolet light rhoeadine solutions exhibit a slight blue fluorescence. On addition of a few drops of sulfuric acid, the color turns very light blue. On heating on the water-bath, rhoeadine solution progressively turns violet red, while kryptopine and chelidonine solutions turn colorless.—W. Awe. Arch. Pharm., 274 (1936), 439-445; through Chimie & Industrie, 38 (1937), 930. (A. P.-C.)

Salsola Richteri—Alkaloids of. Extraction of Salsola Richteri, 1935 crop, gave a mixture of dl- and d-salsoline. Resolution of the racemate through the tartrate gave the pure d- and l-forms. Salsolidine (l-salsoline methyl ether) was also isolated. Both salsoline and salsolidine are stable toward racemizing agents, and thus racemization takes place in the plant itself and not during the extraction process.—A. P. Orekhov and N. F. Proskourina. Izvest. Akad. Nauk S. S. S. R. (Sér. Chim.), (1936), 957-960; through Chimie & Industrie, 38 (1937), 930. (A. P.-C.)

Essential Oils and Related Products

Black Currant Buds (Ribes Nigrum L.) - Essential Oil of. Cold extraction of black currant (Ribes nigrum L.) buds with benzene gave 2.4 to 3% of semi-crystalline, dark green concrete with acid value 120 and ester value 15.5. Steam distillation of the concrete with cohobation gave 16 to 17% of essential oil having the following characteristics: specific gravity at 15° C. 0.879, optical rotation at 25° C. 1°35', refractive index at 20° C. 1.4870, acid value 1.12, ester value 7, ester value after acetylation 30.16, ester value after cold formylation 40.68; it contained no nitrogenous products, aldehydes nor ketones. The oil contained about 85% of terpenes and sesquiterpenes (among which nopinene, l-sabinene, d-caryophyllene and d-sadinene were identified, 6% of unidentified terpene alcohols, 0.25% of phenols (including carbolic acid and β -naphthol), 0.7% of combined acetic acid and 0.5% of combined higher acids. The non-volatile portion of the concrete contains resinous and coloring matters and a large proportion of a colorless, odorless and tasteless acid, soluble in alcohol, ether, chloroform and benzene, sparingly soluble in petroleum ether, insoluble in water; it melts at 148° C. (corrected), has a specific optical rotation at 20° C. of $-9^{\circ}20'$ (in 10% solution in alcohol), and seems to be a monobasic hydroxy acid in C₁₈.—L. S. GLICHITCH and MME. M. G. IGOLEN. Parfums de France, 15 (1937), 241-244. (A. P.-C.)

Cananga, Patchouly and Vetiver—Dutch East Indian Oils of. From a critical survey of available analyses and inquiry from the principal European consumers, the following specifications are proposed. Java Cananga Oil.—Specific gravity at 15° C. 0.908 to 0.925, refractive index at 20° C. 1.495 to 1.506, optical rotation -15° to -40°, acid value 0.5 to 2, ester value 15 to 35, residue after steam distillation not over 5%, soluble in 1 to 3 volumes of 95% alcohol with opalescence and cloudiness with more alcohol. Achin Patchouly Oil.—Specific gravity at 15° C. 0.950 to 0.990 (for good grade oil, not less than 0.970), refractive index at 20° C. 1.506 to 1.516, optical rotation -40° to -72°, acid value 0.5 to 3, ester value 2 to 10, soluble in 1 to 10 volumes of 95% alcohol (good grade oil should be soluble in 1 to 10 volumes of 90% alcohol). Java Vetiver Oil.—Specific gravity at 15° C. 0.985 to 1.045, refractive index at 20° C. 1.510 to 1.530, acid value 8 to 35, ester value 5 to 25, ester value after acetylation 100 to 150.—D. R. Koolhaas and P. A. Rowaan. Parfums de France, 15 (1937), 245-247. (A. P.-C.)

Celery (Apium Graveolens L.)—Essential Oils of. A review with some original data. Oils obtained in 1937 by distillation of fresh plants: (1) during full bloom (June 20th), and (2)

when the seeds were ripe (Aug. 8th), had the following characteristics: specific gravity at 15° C. 0.9052, 0.9210; optical rotation at 25° C. 35° 5′, 41° 21′, refractive index at 20° C. 1.4877, 1.4881; acid value 4.20, 5.60; ester value 68.03, 85.57.—G. IGOLEN. Parfums de France, 15 (1937), 219-228.

(A. P.-C.)

Chamomile and Peppermint for 1937. Thirty-four samples of chamomile flowers from eight geographical sources were examined for % volatile oil (0.22-79%), color and odor. Thirteen samples of two varieties of peppermint leaves from six regions were examined for volatile oil content (0.85-1.6%).—HANNS WILL. Apoth. Ztg., 52 (1937), 1322. (H. M. B.)

Essential Oil from Atalantia Monophylla. Steam-distillation yielded 0.4-0.6% of an oil (on weight of dry leaves) having d_{30}^{30} 0.8561, n_{0}^{30} 1.4600, α_{0}^{30} -33.2°, acid value 0.8, saponification value 79.3, acetate value 34.2; it was soluble in 1 volume of 90% ethyl alcohol.—M. T. Chobe and B. S. Rao. *Proc. Soc. Biol. Chem. India*, 2 (1937), 16-17; through *J. Soc. Chem. Ind.*, 56 (1937), 1268. (E. G. V.)

Essential Oils—Rotary Dispersion of. The dispersion coefficients of a number of essential oils are recorded; they vary from 1.93 to 2.28.—N. A. VALJASCHKO and J. G. BORISIUK. *Ukrain. Chem. J.*, 12 (1937), 245–247; through *J. Soc. Chem. Ind.*, 56 (1937), 980. (E. G. V.)

Essential Oils—Various. Characteristics of oils from Ocimum basilicum, L., and var. selasih hidjan (Java), O. pilosum, o. canum, Sims, and Java basilicum oil are given. A sample of dill-herb oil (d^{15} 0.8957, $\alpha_{\rm D}$ + 88° 37'; $n_{\rm D}^{20}$ 1.48364) was soluble in 9.5 volumes of 80% ethyl alcohol and contained 37% carvone; it was adulterated with caraway oil. Two samples of commercial sassafras oil consisted of camphor oil fractions containing safrole. A sample of wormwood oil, probably adulterated, had d^{15} 0.9232, $\alpha_{\rm D}$ -12°10', $n_{\rm D}^{20}$ 1.46191, acid value 2.8, ester value 9.3 (after acetylation 37.3), solubility in 80% ethyl alcohol, 1 in 0.9 volume, and contained 60.9% of thujone.—Schimmel and Co. Ann. Rept., 76 (1936), 7-8, 87-88; through J. Soc. Chem. Ind., 56 (1937), 979. (E. G. V.)

Essential Oils of the Western Australian Eucalypts. III. Oils of E. Salmonophloia, F. v. M., and E. Tetragona, F. v. M. E. salmonophloia leaves yielded 1.4% of a pale yellow volatile oil having the following properties: acid number 2.2; saponification number hot, 11.8, cold, 11.1; acetylization number hot, 61.7, cold, 54.1; d_{20} 0.9137, n_D^{20} 1.4731, α_D^{20} -4.12° . The oil contained some d-pinene, 46.4% cineol, geranyl acetate, 4.8% unidentified phenols (one may be australol), 4.5% aldehydes and traces of aromadendrene. E. tetragona leaves yielded 0.48% of an oil having the following properties: d_{20} 0.9390; n_D^{20} 1.4954; α_D^{20} $+3.65^{\circ}$; acid number 1.0; saponification number hot, 6.8, cold, 6.6; acetylization number hot, 107.5, cold, 19.8; soluble in 1 volume 80% alcohol, 10 volumes 70% alcohol. The oil contained geranyl acetate 2.3, cineole 15.4, phenols 3.7, aldehydes 0.80, eudesmol approximately 32% and small amounts of d-pinene, l-phellandrene and aromadendrene.—E. M. Watson. J. Roy. Soc. W. Australia, 22 (1935–1936), 113-118; through Chem. Abstr., 31 (1937), 7194.

Oil of Horse Radish Root. Treatment with benzene of crushed fresh roots of *Cochlearia armoracia* L. yielded 2% of a semi-liquid, brownish concrete. Steam distillation of the concrete yielded 1.57% of essential oil with exceedingly strong, aggressive odor, refractive index at 20° C. of 1.505, acid value of 33.6, ester value of 91 and containing 17.8% of total sulfur.—Etablissements Antoine Chiris. *Parfums de France*, 15 (1937), 228. (A. P.-C.)

Onion Plants—Essential Oil of. Distillation of whole onion plants (Allium cepa L.) gave 0.05% of brown, semi-liquid oil, with characteristic cooked onion odor. It had specific gravity at 15° C. 1.0118, optical rotation at 18° C. 1°30′, refractive index at 20° C. 1.5236, and was soluble in 0.1 volume of 95% alcohol with considerable turbidity and milky precipitate by dilution to 0.5 volume.—ESTABLISSEMENTS ANTOINE CHIRIS. Parfums de France, 15 (1937), 228.

(A. P.-C.)

Seychelles—Essential Oils from. II. Two samples of cinnamon oil had the following constants: specific gravity at 15.5° C.: 1.0142, 1.0160; optical rotation at 20° C.: -2.01°, -1.74°; refractive index at 20° C.: 1.5845, 1.5812; total aldehydes as cinnamic aldehyde (by the hydroxylamine method): 71.1%, 67.3%; solubility in 70% alcohol: both insoluble in 10 volumes; solubility in 80% alcohol: soluble in 0.7 volume, soluble in 0.8 volume. These constants agree with those recorded for the oils distilled in Germany from Seychelles cinnamon bark, but not with British Pharmacopæial requirements. Two samples of oil distilled from a plant known locally as "Toc Maria" and identified as Ocimum basilicum L. had constants in close agreement to

those previously reported. A sample of oil distilled from soft stems and leaves of Ocimum sanctum L. had constants agreeing closely with those previously reported; the phenolic portion of the oil appeared to consist chiefly, if not entirely, of chavibetol. A sample of oil distilled from the same strain of palmarosa plants as had furnished previous samples had an abnormally low ester value and high acid value, indicating that a partial hydrolysis of the esters had probably taken place. Two samples of oil of palmarosa distilled from plants raised from Indian seed had a very high "total geraniol" content, very high ester values falling outside the range recorded for the Indian product and abnormally high specific gravity and acid value, possibly attributable to storage under unsuitable conditions. A sample of oil of Mentha arvensis L. (obtained in a yield of 9.3 liters per ton of fresh material) had the characters of natural Japanese peppermint oil derived from Mentha arvensis.—Anon. Bull. Imp. Inst., 35 (1937), 298-311. (A. P.-C.)

Glycosides, Ferments and Carbohydrates

Enzyme—Hydrolyzing, in the Bark of Periploca Graeca L. By extraction of the powdered bark with petroleum ether and with 3 volumes of water for 48 hours, precipitation and washing with 96% alcohol, 9% of a brown powder is obtained; it is purified by adsorption on aluminum hydroxide at a $p_{\rm H}$ value of 5 and elution with very dilute ammonium hydroxide. The substance converts the periplocoside contained in the bark into periplocomaroside. The enzyme splits also k-strophanthoside, but does not affect ouabain and digitoxoside. The name periplocibiase is suggested.—Th. Solacolu and G. Herrmann. Bull. Soc. Stiinte Farm. Romania, 1 (1936), 53-60; through Chimie & Industrie, 38 (1937), 523-524. (A. P.-C.)

Hyacinthus Orientalis—New Constituents of. The author succeeded in isolating a new compound from Hyacinthus orientalis which was named hyacin chloride (1). It is a coloring material of the leaves of the plant and seems to be identical with the coloring substance obtained from delphinidine diglucoside. In hydrolyzing 1, 2 molecules of glucose and 1 molecule of delphinidine chloride were obtained.—K. HAYASHI. Chem. Zentr., 108 (1937), 618. (G. B.)

Medicinal Plants of Brazil—Biochemical Method of Bourquelot and Its Application to Study of. Classification of plant ferments, and description of methods of isolation of glycosides, by crystallization, by lead, copper, barium, etc., with tables. Details of preparation of enzymes and technic of biochemical investigation. Tabulations of heterosides with name of investigator and year, plant source, chemical formula, rotation and reduction index.—OSEALDO DE ALMEIDA COSTA. Rev. quím. farm., 2 (1937), 71. (G. S. G.)

Oleander Leaves—Glucosides Having an Action on the Heart from. An aqueous extract of oleander leaves is extracted with a chlorinated aliphatic hydrocarbon, and the glucosides having an action on the heart are precipitated by means of a liquid saturated hydrocarbon.—Max Bockmühl and Gustav Ehrhart, assignors to Winthrop Chemical Co. U. S. pat. 2,099,158, Nov. 16, 1937. (A. P.-C.)

Olive—Bitter Principle of. Oleuropein, the bitter principle of the olive, has been isolated and purified by a method giving a product with a higher specific rotation than that previously obtained. It is glycosidal, hydrolyzed by an enzyme found in olive leaves, and by pectinol, a preparation of Penicillium, but only slowly by emulsin and not at all by invertase. Acid hydrolysis yields d-glucose and an ether-soluble product, the optical rotation becoming strongly l-rotatory; this also occurs with pectinol. The bitter ester, which does not exist before hydrolysis, is readily hydrolyzed by alkalies with disappearance of the bitter taste. The glycoside, on hydrolysis with sodium hydroxide, loses its bitter taste, but retains its l-rotation if the treatment is not too severe, and from the products of hydrolysis a crystalline acid, identical, in qualitative tests, X-ray spectrum and m. p., with caffeic acid, has been isolated; the mixed m. p. with true caffeic acid also shows no depression. An unidentified phenol, not pyrocatechin, has also been isolated. The bitter principle probably possesses a glycoside and an ester grouping, the former dissociated by mineral acids, emulsin and similar enzymes, while the latter is decomposed by alkalis and apparently by an enzyme found in the olive. Olive pulp contains about 1\% of the glycoside, but its concentration is greater in the green fruit of the Mission and Manzanillo varieties, and lower in the varie-IV Congr. intern. tech. chim. ind. agr., Brussels, ties of Seville and Ascolano.—W. V. CRUESS. 3 (1935), 638; through Quart. J. Pharm. Pharmacol., 10 (1937), 564. (S. W. G.)

Papain—Crystalline. From the undried latex of green papaya fruit there has been obtained a crystalline material which clots milk, digests casein and splits hippurylamide in the pres-

ence of added cysteine under the conditions usually employed for demonstrating the activity of papain. The activity of the crystals per mg. protein-nitrogen as measured by milk clotting or casein digestion was 25-50% higher than that of any amorphous preparations made in the authors' laboratory and was about twice as great as that of the best commercial preparations. The crystals had protein properties, contained nitrogen which was precipitated from aqueous solution by trichloracetic acid and were inactivated by incubation with dilute hydrogen peroxide. For the preparation, coagulated papaya latex, preserved with toluene, was suspended in four volumes water or two volumes of 0.25 saturated ammonium sulfate. After about one hour the material was filtered and the clear filtrate was made 0.6-0.7 saturated with ammonium sulfate and filtered. The semidry filter cake was suspended in an equal weight of water, the $p_{
m H}$ was adjusted to light green to brom thymol blue, and the solution was cooled slowly to 5°. The solution containing about 15 mg. nitrogen per cc., became turbid on cooling, and in a few days developed a sheen due to formation of small needle crystals. The yield was increased by slow addition of saturated ammonium sulfate. Recrystallization was carried out by essentially the same technic. Recently crystals have also been obtained from commercial papain but have not yet been freed from amorphous material.—A. K. Balls, H. Lineweaver and R. R. Thompson. Science, 86 (1937), 379; through Squibb Abstr. Bull., 10 (1937), A-2019.

Papain and Similar Vegetable Enzymes—Preparation of Dry Active Products of. Papain is mixed with dried yeast or with seeds containing amygdalin and emulsin.—W. Klorz & Co. Belg. pat. 420,750, April 30, 1937. (A. P.-C.)

San-Ch'i (Aralia Bipinnatifida)—Saponins of the Chinese Drug. Two saponins have been isolated, called provisionally arasaponin A and B. Arasaponin A is amorphous, has the formula $C_{30}H_{62}O_{10}$, m. p. 195–210°, rotation 23°. On hydrolysis with dilute sulfuric acid it yields an arasapogenin, $C_{17}H_{20}O_{5}$, glucose and two other crystalline substances, m. p. 244° and 252°. It yields a crystalline acetate, $C_{30}H_{46}O_{10}Ac_{7}$. The lethal dose for mice is 1 Gm. per Kg. Arasaponin B is amorphous, has the formula, $C_{23}H_{36}O_{10}$, m. p. 190–200°, rotation 8°.—T. Q. Chou and J. H. Chu. *Chinese J. Physiol.*, 12 (1937), 59–66; through *Chem. Abstr.*, 31 (1937), 7063.

(F. J. S.)

Scoparoside (Scoparin) of Sarothamnus Scoparius Koch—Constitution of. Scoparoside is a heteroside, C_{2ℓ}H₂₂O₁₁.H₂O, difficultly hydrolyzed, but can be hydrolyzed by the enzyme rhamnodiastase, giving one molecule of rhamnose, C₆H₁₂O₅, and one molecule of scoparol, C₁₆H₁₂O₇, a flavone derivative that is probably a methyl ether of quercitol. Physiologically, scoparin exerts a slight depressant action on blood pressure. The pure product is devoid of the diuretic properties which are generally attributed to scoparin, and which are found to a slight degree in incompletely purified products.—M. Mascre and R. Paris. Compt. rend. acad. sci., 204 (1937), 1270–1271; through Chimie & Industrie, 38 (1937), 929. (A. P.-C.)

Sorbitol from Glucose by Electrolytic Reduction. Until recently sorbitol (I) sold for \$300-\$500 a pound and mannitol (II) sold for several dollars a pound. A synthetic process has been developed for large scale preparation of I and II by the electrolytic reduction of corn sugar (d-glucose). The plant, process and properties of the products are described in detail with illustrations and diagrams. The present uses of I are in the medicinal and pharmaceutical fields and in the making of fancy papers and resins. Nitro-mannite has found a use in medicine and is claimed to have exceptionally favorable properties as a non-corroding primer for explosives.—
R. L. TAYLOR. Chem. Met. Eng., 44 (1937), 588; through Squibb Abstr. Bull., 10 (1937), A-2024.

(F. J. S.)

l-Sorbose—Mutarotation of. Optical-rotation measurements are reported on carefully purified l-sorbose at 0.4 and 20° C. In contradiction of the statements in various periodicals and textbooks, the results show that this sugar exhibits a small complex mutarotation.—William W. Pigman and Horace S. Isbell. J. Research Natl. Bur. Standards, 19 (1937), 443; through Squibb Abstr. Bull., 10 (1937), A-2160. (F. J. S.)

Soybeans—Sucrose from, Isolation of. The authors isolated sucrose from soybeans by two different methods. In the first method soybean flakes were extracted with petroleum ether or with ether followed by extraction with 99% alcohol. The alcohol extract was concentrated on a steam-bath until sugar began to crystallize on the sides of the beaker and on a stirring rod standing in the solution. After standing two days the crystals were filtered and washed with ether. In the second method an 80% alcohol extract of soybean flakes was concentrated before

a fan to a thick syrup. Water was added and the solution was treated with lead acetate. The precipitate was filtered and the filtrate was treated with barium hydroxide. The lead and barium were removed with sulfuric acid. The solution was shaken with ether to remove acetic acid, and was then concentrated to a thick syrup. An equal volume of 99% alcohol was added and the syrup settled to the bottom. After stirring the syrup in the alcohol on the steam-bath, the alcohol was decanted and let stand for several days. Crystals appeared in the alcohol extract. The first two fractions were combined and recrystallized. A 5% solution of these crystals gave a specific rotation of 66.57°. The refractive indices were the same as those of pure sucrose.—H. R. KRAYBILL, R. L. SMITH and E. D. WALTER. J. Am. Chem. Soc., 59 (1937), 2470. (E. B. S.)

Strophanthin—Study of. It was previously suggested by Jacobs (J. Biol. Chem., 69, 153) that amorphous strophanthins obtained by the usual extraction methods contained other more glucose-rich strophanthins besides cymarin and k-strophanthin. There has now been isolated a crystalline saccharide, m. p. $217-220^{\circ}$, with the constitution of a glucosido-glucosido-cymarose, by mild hydrolysis of a commercial strophanthin preparation. The empirical formula corresponds to $C_{19}H_{34}O_{14}$. After hydrolysis of the sugar with 2N sulfuric acid at 100° , a reduction value of 68% calculated as glucose was found; the reduction value calculated for the triose is 74%. The compound is not identical with the "methyl- strophantobioside" isolated by Feist (Ber., 31, 534). By acetylation of the same strophanthin preparation there was obtained a new crystalline acetyl-strophanthin, m. p. $216-220^{\circ}$ (uncorrected), differing from the known acetate of k-strophanthin, m. p. 167° . It is not yet known whether this new acetate is a derivative of the new strophanthin.—J. Kraus. Naturwissenschaften, 25 (1937), 651; through Squibb Abstr. Bull., 10 (1937), 4-2025.

Other Plant Principles

"Resins" and "Pitch" from Ancient Egyptian Tombs. Four "dark, somewhat pitchlike substances found in Egyptian tombs" were examined. The conclusions reached were that all four samples were resins of coniferous trees but not of pine or cedar. One showed indication of mixture with mineral bitumen. Quantitative data are given on the composition of the samples.—
J. G. A. Griffiths. Analysi, 62 (1937), 703. (G. L. W.)

Rotenone and Associated Substances. A natural rotenone-containing material, such as derris root and linseed oil, is subjected to distillation without ebullition at a pressure of about 10^{-2} to 10^{-6} mm. of mercury (and suitably at a temperature of about 120° C.) with condensing surfaces placed in close proximity to and substantially coextensive with the evaporating surfaces. A pale yellow waxy solid product is obtained which is suitable for insecticidal uses.—Eric W. Fawcett, assignor to Imperial Chemical Industries. U. S. pat. 2,096,678, Oct. 19, 1937.

(A. P.-C.)

Vanillin—Manufacture of, from Lignin. Vanillin is obtained in 2-3% yield from lignin-containing substances (from which most of the non-ligneous matter has been removed), such as basic calcium ligninsulfonate, by converting the latter (200 Gm.) in water (1 litre) into the sodium salt, separating calcium carbonate, heating with sodium hydroxide (25% on lignin) at 130-200 pounds per square inch for one-half to one and one-half hours, acidifying to $p_{\rm H}$ greater than 7 with sulfur dioxide, sulfurous acid or an alkali bisulfite at about 80-95°, filtering, acidifying the filtrate with sulfuric acid, extracting the vanillin (containing about 25% of impurities), and finally removing the solvent (benzene).—L. T. Sandborn, J. R. Salvesen and G. C. Howard, assignors to Marathon Paper Mills Co. and G. C. Howard Co. U. S. pat. 2,057,117; through J. Soc. Chem. Ind., 56 (1937), 1177. (E. G. V.)

Fixed Oils, Fats and Waxes

Animal Oils—Marine, Component Acids and Glycerides of Partly Hydrogenated. I. General Review of Analytical Procedure Employed. The details of the analytical procedure now employed in the ester-fractionation of the acids from the original and hydrogenated whale and cod liver oils are described, and a description is given of the methods by which the components of ester-fractions containing two saturated and two (or three) unsaturated esters may be determined. The order of accuracy attainable in analyses of this kind is discussed.—D. A. HARPER, T. P. HILDITCH and J. T. TERLESKI. J. Soc. Chem. Ind., 56 (1937), 310-315T. II. Antarctic Whale Oil. The component acids and fully saturated glycerides present in Antarctic whale oil which had

been hydrogenated to various stages have been quantitatively studied. The oil [saponification equivalent 286.5, iodine value 109.3, free acid (as oleic) 0.53%] contained mixed fatty acids (approximate molecular %): myristic 9, myristoleic 4, palmitic 17, palmitoleic 17, stearic 2, C₁₈ unsaturated (mean unsaturation -2.6 H 35, C₂₀ unsaturated (-5.6 H) 12, and C₂₂ unsaturated (-9 H) 4. The oil contains only small quantities of C₂₂ acids and only moderate quantities of C20 acids; the C16 and C14 acids occur in much the same proportions as in many other whale and marine fish oils and, consequently, there is a larger amount than in the latter cases of C_{18} unsaturated acids (about 90% of which is oleic acid). The oil contains a complex mixture of glycerides, but, owing to the presence of only 16-20% of combined C20 and C22 acids, almost half of the oil probably consists of glycerides containing only acids of the C18, C18 and C14 series. The course of hydrogenation is partly selective. Polyethenoid C22, C20 and C18 acids pass toward the monoethenoid condition before any of the respective saturated acids are produced in quantity, but monoethenoid derivatives in one group (C14, C16 and C18) commence to pass into the saturated derivatives before all polyethenoid unsaturation in other groups has disappeared. The formation of fully saturated glycerides depends on the preliminary reduction of poly- to monoethenoid compounds and, subsequently, on the transformation of tri- to di- and di- to mono- unsaturated glycerides by successive, and not simultaneous, stages. Consequently fully saturated glycerides do not augment rapidly until the fat reaches a comparatively low iodine value, the increase then becoming more and more rapid (at iodine values of 50, 30 and 10, for example, the respective contents of fully saturated components are about 12, 30 and 65%). A hydrogenated whale oil of iodine value similar to beef or mutton tallow contains about the same proportion of fully saturated glycerides as the latter, and, in consequence of the relative lack of C₂₀ or C₂₂ saturated acids at the earlier stages of hydrogenation, the component acids of such fully saturated glycerides are more similar to those of the corresponding tallow components than might at first be expected. The liquid components of the rest of the fat (mixed saturated-unsaturated glycerides) differ from those of tallow, owing to the presence of C20 and C22 unsaturated acids and the "iso"-monoethenoid acids of hydrogenation.—T. P. HILDITCH and J. T. TERLESKI. Ibid., 315-322T. III. North Sea Cod Liver Oil. The component acids and the fully saturated glycerides present in North Sea cod liver oil which have been hydrogenated to various stages have been quantitatively studied. The cod liver oil (iodine value 177.7) contained mixed fatty acids (approximate molecular %): myristic 2, myristoleic 2, palmitic 14, palmitoleic 10, stearic 1, C18 unsaturated (mean unsaturation -3.3 H) 26, C_{20} unsaturated (-5.5 H) 25, C_{22} unsaturated (-7.4 H) 20, C_{24} unsaturated less than 1%. The oil has a high content of C22 acids and somewhat less palmitoleic acid than usually found in fish oils. The proportions of some of the acids differ from those recorded for other cod liver oils, but the average unsaturation of the C18, C20 and C22 groups of acids, respectively, seems to vary but little, either as between various cod liver oils, or in the liver oils of gadoid species of fish as a whole. The oil contains a very complex mixture of glycerides and, since 45% of the acids are of the C20 and C22 series, most glyceride molecules will contain at least one acid group of these series. The first phase in the hydrogenation process (80 iodine value units fall) is exclusively devoted to the reduction of polyethenoid unsaturation. After this the C₁₈ acids rapidly reach a state of monoethenoid unsaturation and thereafter the production of stearoglycerides sets in. In the C20 and C22 series the mean unsaturation continues to decrease but the action becomes less marked the lower is the iodine value of the whole fat, and complete reduction to not greater than monoethenoid is not reached until the iodine value of the fat is very small. In these cases reduction of the di- to mono-unsaturated and then to saturated takes place side by side. Saturated compounds are produced more rapidly in those series in which, in the original oil, monoethenoid unsaturation predominates. This, however, except in the case of palmitoleic, does not indicate selective reduction of one monoethenoid acid before another. As the reduction takes place in stages, the production of the fully saturated glycerides in the first half of the hydrogenation is small and negligible. Thereafter production sets in and gradually accelerates, becoming very rapid when the iodine value of the fat is small.—D. A. HARPER and T. P. HILDITCH. *Ibid.*, 322-329T. (E. G. V.)

Cichorium Intybus, L.—Chemical Examination of the Seeds of. Constituents of the Oil from the Seeds. The use khasni (*Cichorium intybus*, L.) by Indian physicians is similar to that of taraxacum in Europe. Exhaustive extraction of 2.5 Kg. of the seeds with benzene gave 118 Gm. of a transparent light brown semi-drying oil, d_{22} 0.9229, n_{22}^{30} 1.3795, m. -11°, acid number 11.2,

saponification number 193.1°, acetylization number 14.8, iodine number 95.6, Hehner's value 93.9°, unsaponifiable 1.7%. The oil is free from nitrogen and sulfur and is optically inactive. The semi-solid fatty acids, molecular weight 291.4, m. p. 35–38°, d_{40} 0.8931, neutralization value 192.5, iodine number 104.8 contain 21.7% of saturated, molecular weight 260.8, iodine number 1.6 and 78.3% unsaturated acids, molecular weight 280.5, iodine number 141.6 and probably consist of 33.5% oleic, 44.8% linolic and palmitic and stearic acids. The unsaponifiable material was identified as a phytosterol, m. p. 131–133°.—RAM NATH MISRA and SIKHIBHUSHAN DUTT. J. Indian Chem. Soc., 14 (1937), 141–143; through Chem. Abstr., 31 (1937), 7274. (F. J. S.)

Fat Absorption and Dialysis of Fatty Acids. Contrary to the findings of Verzar and Kuthy [cf. S. A. B., 2 (1929), 727] saturated fatty acids with sixteen or more carbon atoms, and oleic acid, showed no tendency to diffuse through parchment (Schleicher and Schüll, No. 579) in the presence of bile acid salts, either as colloidal aqueous solutions or as soaps. Sodium oleate, however, diffused through cellophane. Saturated fatty acids with less than sixteen carbon atoms were diffusible through parchment in inverse proportion to their molecular weight. Linoleic and ricinoleic acids were also diffusible. The soaps were more readily diffusible than colloidal solutions of the free fatty acids in bile acid salts. An aqueous colloidal solution of lecithin dialyzed very slowly of itself, but markedly accelerated dialysis of sodium glycocholate. Dialysis of soaps was not accelerated by lecithin and was inhibited by salts of bile acids to some extent. The results are discussed in relation to possible mechanisms of lipoid absorption from the intestine.—F. L. Breusch. Biochem. Z., 293 (1937), 280; through Squibb Abstr. Bull., 10 (1937), A-2008.

(F. J. S.)

Fat Hardening—Problems in. The mechanism of fat hydrogenation is discussed with particular reference to the possibilities of manipulating conditions so as to control the reaction and to produce selective hydrogenation; for example, addition of 10% of carbon monoxide to the hydrogen checks hydrogenation beyond the mono-ethylenic stage.—W. Normann. Fette u. Seifen, 44 (1937), 330-336; through J. Soc. Chem. Ind., 56 (1937), 1232. (E. G. V.)

Fats, Butter, Margarine and Other Substances-Determination of Water in. Small quantities of water in fats, glycerin, acetic esters, etc., can be determined by a modification of Fisher's I-SO₂-C₆H₆N method. Twenty to fifty grams of fat are dissolved in 10-25 Gm. of decalin and well shaken with 25 cc. of absolute methyl alcohol; an aliquot part (20 cc.) of the methyl alcohol layer is then titrated with the iodine solution (254 Gm. of iodine in 5000 cc. of methyl alcohol, to which 790 Gm. of pyridine and 192 Gm. of sulfur dioxide are added). A blank test on the reagents and solvents is needed, and the iodine solution is standardized by means of a weighed amount of water. Fats such as castor oil or cod liver oil emulsion may be dissolved in methyl alcohol and titrated directly. For butter or margarine a solution of 0.5-1.0 Gm. in 5-10 cc. of carton tetrachloride is used. The method is not suitable for the analysis of soaps. Traces of water are determined by the following method: A mixture of fat (20-40 Gm.) with 5 cc. of pyridine is shaken with a measured 10-cc. portion of a 0.5N solution of acetyl chloride in carbon tetrachloride; after setting aside for 15-30 minutes the excess of acetyl chloride is decomposed by addition of 5 cc. of fresh phenyl amine, and the acetic acid liberated by the reaction of acetyl chloride with the water in the fat is titrated with potassium hydroxide. The titre of the reagent solution is checked by a blank test, and a correction applied for any free fatty acids in the fat. The results agree with those by the first method given above, and agreement by duplicates is better than by the first. From 0.03 to 0.19% of water was found in ordinary vegetable and animal fats by these methods.—H. P. KAUFMANN and S. FUNKE. Fette u. Seifen, 44 (1937), 345-346, 386-390; through J. Soc. Chem. Ind., 56 (1937), 1232.

Fats—Improvement Refining of. Bleaching of edible fats should be reduced to a minimum in order to avoid a loss of vitamins or provitamins.—W. Halden. Fette u. Seifen, 44 (1937), 346–348; through J. Soc. Chem. Ind., 56 (1937), 1232. (E. G. V.)

Fats and Oils—Animal and Vegetable, Stabilization of. Sugar amines, particularly secondary amines containing a long-chain (having more than eight carbon atoms) alkyl radical, such as lauryl-glucamine, and their salts are claimed as inhibitors of deterioration of fats and oils. 0.001-0.1% is used.—E. I. Du Pont de Nemours and Co. Brit. pat. 470,573; through J. Soc. Chem. Ind., 56 (1937), 1235. (E. G. V.)

Fatty Acid Groups—Preferential Reduction of, during Hydrogenation of Natural Fats. An attempt has been made to compare the relative rates of hydrogenation of different mono-

ethenoid acids, or di- and mono-ethenoid acids, by a simple mathematical analysis of the data given previously for hydrogenated cod liver and whale oils, and of that given by previous workers for hydrogenated rape, cottonseed and soya-bean oils and their mixed esters or acids. It is shown that the relative rate of hydrogenation for the different acids frequently varies, as between the cases of glycerides and of the corresponding mixed methyl or ethyl ester, and that selective hydrogenation of linoleic to oleic compounds is uniformly more pronounced in the case of simple esters than of glycerides.—D. A. HARPER. J. Soc. Chem. Ind., 56 (1937), 308–310T. (E. G. V.)

Fish Oils—New Zealand. S. reviews the literature on New Zealand fish oils and tabulates their properties in comparison with oils of North Sea and Chilean fish. A high vitamin A content was found in New Zealand groper liver oils in the winter (July and August) but not in the summer (October and November). Winter groper liver oil was exceptional in containing as much as 20% phosphatide. Examinations of other fat depots of this fish failed to reveal the presence of vitamin A except in the stomach oil. Cook Strait elasmobranch liver oils were not rich in vitamin A. Seasonal variation of ling liver oil is not marked.—F. B. Shorland. Nature, 140 (1937), 223; through Squibb Abstr. Bull., 10 (1937), A-1658. (F. J. S.)

Flaxseeds—Superior and Inferior, Chemical Composition of the Oils from. A detailed study made on the unsaturation of two varieties of flaxseed oil (Abyssinian Yellow and Bison) grown in different localities is reported. Tables showing the extent of variation of the iodine number with the change in the index of refraction and the per cent of the unsaturated fatty acids composing the oils are given. Abyssinian Yellow linseed oil is a superior oil having a relatively high iodine number, while Bison linseed oil is an inferior oil and usually has a somewhat lower iodine absorption value.—R. A. Gross and C. H. Bailey. Oil and Soap, 14 (1937), 260; through Squibb Abstr. Bull., 10 (1937), A-2118. (F. J. S.)

Indicators—Brief Study of, in Determining Fatty Acids in Dark Colored Oils. Thymolphthalein or, better, thymol blue is preferred to phenolphthalein as indicator for the titration of dark oils; excess of carbon dioxide should be restricted. Good results were also obtained with methyl blue. The presence of about 10 cc. of light petroleum facilitates the titration.—P. M. Shuby. Oil and Soap, 14 (1937), 232-233; through J. Soc. Chem. Ind., 56 (1937), 1234.

(E. G. V.)

Oil from Seeds of Onguekoa Gore, Engler. The pale yellow oil (acid value 2.2) from hand-picked sound kernels had d_4^{20} 0.973, viscosity (Höppler) 7.30 poises at 20°, saponification value 189, iodine value (Wijs, 2 hours) 184, hydrogen-iodine value 355, unsaponifiable matter 1.1%, hexabromide value less than 1%, hydroxyl value about 60 (the varying results of duplicate analyses by the Verley-Boelsing method suggest that true hydroxyl acids are absent). The high d^{70} suggests the presence of conjugated unsaturated acids (probably an octadecatetraenoic acid) rather than the ethyleno-acetylenic acid postulated by Steger and van Loon. Azelaic and oxalic acids were identified among the oxidation products of the fatty acids.—H. A. BOEKENOGEN. Fette u. Seifen, 44 (1937), 344; through J. Soc. Chem. Ind., 56 (1937), 1233. (E. G. V.)

Oil from Seeds of Ximenia Americana, L. New Unsaturated Fatty Acid, Ximenic Acid. The seed oil consists of the glycerides of stearic, cerotic (I), ximenic (C₂₆H₅₀O₂) (II), oleic and linoleic acids, a little phytosterol, and a large amount of rubber-like substance. No arachidic acid was found. II has not been obtained free from I, but is hydrogenated (hydrogen-platinum) to I and oxidized by cold potassium permanganate-potassium hydroxide-water to dihydroxycerotic acid, C₂₆H₅₂O₄, melting point 118-119°.—S. V. Puntambekar and S. Krishna. J. Indian Chem. Soc., 14 (1937), 268-274; through J. Soc. Chem. Ind., 56 (1937), 1233. (E. G. V.)

Oils, Fats and the Like—Flavor, Stability, Etc., of, Improvement of. The oil (or crude tallow, or oilseeds, prior to the recovery of the oil) is intimately mixed at 20-60° with an aqueous solution of fermentable sugar which has been inoculated with a yeast, or lactic acid, or other acid-producing bacteria, e. g., B. Leichmann, B. bulgaricus. Reaction-controlling substances, activators, e. g., sodium chloride, catalysts, such as tin or nickel salts, nutrient salts and buffers may also be added. The purified fat is finally separated from the products of fermentation. A pretreatment with a structureless ferment, e. g., rennet, may be given, and the process applied to deodorize rancid fats, fish oils, etc.—W. EKHARD. Brit. pat. 465,111; through J. Soc. Chem. Ind., 56 (1937), 1236.

Unclassified

Aldonic Acids—Crystallizing Calcium Salts of. A solution of calcium xylonate (I), obtained by electrolytic oxidation of xylose in presence of calcium bromide and calcium carbonate is evaporated in vacuo to a 70% solution which is seeded with crystals of $Ca(C_bH_9O_0)_2.2H_9O$ obtained by slowly cooling a concentrated aqueous solution obtained by treating basic I with carbon dioxide. Solutions of calcium α -glucoheptonate afford crystals of the trihydrate by prolonged stirring of the syrup at 50° and solutions of calcium gulonate crystallize on slow evaporation at $30-90^{\circ}$.—H. S. ISBELL. U. S. pat. 2,044,793; through J. Soc. Chem. Ind., 56 (1937), 1021.

(E. G. V.)

Alkylchlorohydroxybiphenyls. Antiseptic and microbicidal compounds are prepared by various methods such as by the direct chlorination of an alkylhydroxybiphenyl, or by the reaction of a chlorohydroxybiphenyl compound with an olefine, an alkyl halide or an aliphatic alcohol in the presence of catalysts such as aluminum chloride, aluminum bromide, ferric chloride, zinc chloride, etc. The preparation and properties of a large number of such compounds are described.— Edgar C. Britton, Gerald H. Coleman and Lindley E. Mills, assignors to Dow Chemical Co. U. S. pat. 2,092,724, Sept. 7, 1937. (A. P.-C.)

p-Aminobenzoic Acid—Dialkylaminoalkanol Esters of. Several series of compounds of the

general formula, H₂N——C—O—X—NRR' were prepared. With increase in size of the alkyl groups on the nitrogen, the toxicity increased. The anesthetic value also increased markedly. The anesthetics were prepared by (1) condensation of p-nitrobenzoyl, and (2) condensation of a dialkylamine with an ω-halogen alkyl ester of p-nitrobenzoic acid. Reduction of the nitro compounds gave the anesthetics.—W. B. BURNETT, R. L. JENKINS, C. H. PEET, E. E. DREGER and R. ADAMS. J. Am. Chem. Soc., 59 (1937), 2248. (E. B. S.)

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Antimalarial Substances—Chemistry of. II. Synthesis of 4-Methoxy-2-Aminocarbazole and Its Diethylaminotrimethylene Derivative. o-Amino-p'-methoxycarbazole was prepared from p-chlorobenzoic acid by converting the latter into the azimine and heating the latter in a paraffin bath at 360° C.; the methoxyaminocarbazole thus obtained was condensed with diethylamino-trimethylene hydrochloride, and the condensation product converted into the salt of methylene-bis-salicylic acid. Neither the methylene-bis-salicylate of o-diethylaminotrimethylene-amino-p'-methoxycarbazole, nor the 5% solution of its hydrochloride possessed any anti-malarial activity (as tested on birds).—A. M. Berkenheim and S. I. Lourié. J. Obchtch. Khim., 6 (1936), 1043-1056; through Chimie & Industrie, 38 (1937), 319.

Arseno Compounds—Asymmetric. Arsono-phenoxyacetic acids or arylarsonic acids containing a nitrogenous heterocyclic ring, or other therapeutically active arylarsonic acids, are converted to asymmetric arseno compounds by reduction. Thus, 4-acetylamino-2,3-dimethyl-1-phenylpyrazolone-p-arsonic acid and 1-methyl-2-hydroxyacetic acid-benzimidazole-5-arsonic acid are mixed with sodium acetate and potassium iodide; the mixture is reduced by adding glacial acetic acid and hypophosphorous acid; the precipitated arseno compound is filtered off. Several other examples are given. The preparations are used for intravenous, subcutaneous or intramuscular injections and have a strong stimulatory action.—Karl Streitwolf, Alfred Fehrle and Walter Herrmann, assignors to Winthrop Chemical Co. U. S. pat. 2,095,577, Oct. 12, 1937. (A. P.-C.)

Aryl Compounds—Bactericidal Hydroxy-Halogenated. β -Ethylhexylchlororesorcinol, which melts at 56° C. in the hydrated form, is prepared as follows: slowly add resorcinol to α -ethylhexanoic acid containing dissolved zinc chloride, while maintaining the temperature at 125° C. and stirring the solution, after allowing to stand for 3 hours dilute with water, separate the non-aqueous layer containing the desired product, purify it by distillation (preferably under reduced pressure), reduce with amalgamated zinc and hydrochloric acid, purify the reduction product by crystallization from gasoline, and chlorinate in an inert solvent such as acetic acid or carbon tetrachloride. β -Ethylhydroxybromoresorcinol (melting point 62° C. in the hydrated form), β -ethylhexyliodoresorcinol (melting point about 35° C.), and analogous amyl, heptyl and nonyl compounds are similarly obtained. Such compounds may be used in mouth washes, tooth pastes, or moth, fungus or mold preventives, etc.—Lucas P. Kyrides, assignor to Monsanto Chemical Co. U. S. pat. 2,093,778, Sept. 21, 1937. (A. P.-C.)

l-Ascorbic Acid—Manufacture of. By treating l-2-ketogulonic esters with alkali salts of weak acids in hot alcohols, excellent yields of alkali ascorbates are obtained, and these are converted into the free acid by means of a strong acid. Methyl l-2-ketogulonate (I) (20.8) and sodium carbonate (8.2) in hot methyl alcohol (300) or I (104) and sodium acetate (45) in methyl alcohol (700 parts) give greater than 90% yields of sodium ascorbate.—T. REICHSTEIN. Brit. pat. 469,157; through J. Soc. Chem. Ind., 56 (1937), 1268. (E. G. V.)

Barbituric Acid Compounds. Double compounds of 5,5-dialkyl- and 5,5-alkylaryl-barbituric acids with bromoacylureas are prepared by fusing the components at below the melting point of the higher melting component. The products have therapeutic properties. Examples are described of the fusion of (1) 5,5-diethylbarbituric acid with α-bromoisovalerylurea in the molecular proportions of 1:1, 1:2 and 2:1, (2) 5,5-phenylethylbarbituric acid and α-bromoisovaleryl urea in the molecular proportions of 1:1 and 1:2, and (3) 5,5-diethyl- or 5,5-phenylethylbarbituric acid with bromodiethylacetylurea in the molecular proportions of 1:1 and 2:1.—Werner Ursum, assignor to E. Taeschner Chem. Pharm. Fabrik. U. S. pat. 2,093,120, Sept. 14, 1937.

Benzene—Chlorination of. The velocity of chlorination of ortho-dichlorobenzene is greater than that of para-dichlorobenzene, as a result of which the relative yield of ortho-dichlorobenzene falls with time, and the yield of 1:2:4-trichlorobenzene rises.—G. B. ZILBERMAN and SLOBODNIK. J. Appl. Chem. Russ., 10 (1937), 1080-1085; through J. Soc. Chem. Ind., 56 (1937), 1167.

(E. G. V.)

Camphor—Synthetic, Manufacture of. The principles of the manufacture of synthetic camphor are explained; commercial manufacture of synthetic camphor by the pinene-hydrochloride and by the organic acid processes is described; the two processes are compared and the future of synthetic camphor is discussed from the technical and economical standpoints.—Y. MAYOR. Chimie & Industrie, 38 (1937), 20-26. (A. P.-C.)

Carboxylates—Aryl Mercury Aromatic Polybasic. By reacting together a nitro-substituted aromatic polybasic acid, such as 3-nitro-phthalic or naphthalic anhydride or dinitronaphthalic acid, and a mercury compound such as phenylmercury hydroxide (suitably by heating in a common solvent such as alcohol and water), products are obtained such as diphenylmercury 3-nitrophthalate (melting point 197° to 198° C.), diphenylmercury 3-nitronaphthalate (melting point 184.5° C.), and diphenylmercury dinitronaphthalate (decomposes at about 215° C.), which are of high germicidal power and low toxicity, and are suitable for external uses, as in soaps, mouth washes, ointments, etc.—Carl N. Andersen, assignor to Lever Bros. Co. U. S. pat. 2,094,253, Sept. 28, 1937.

Deuterium—Racemic Reaction of, as Indicator. *l*-Mandelic acid was dissolved in deuterium oxide at a temperature of 60°; after cooling the solution the acid crystallized out; two hydrogen atoms were exchanged for the deuterium atom during the reaction. No racemization took place for mandelic acid during this reaction. However, should the *l*-mandelic acid be heated for 51 hours at a temperature of 140°, racemization takes place. In this case two hydrogen atoms were also exchanged for the deuterium atom; other investigators claim that the number of hydrogen atoms are 3 instead of 2. In alkaline solution the exchange of the hydrogen atom is more than two atoms but never three.—H. ERLENMEYER, H. SCHENKEL and A. EPPRECHT. *Chem. Zentr.*, 108 (1937), 323. (G. B.)

Dihydrojasmone—Process of Preparing. An extract of pyrethrum flowers is hydrogenated in the presence of a platinum, palladium or nickel catalyst.—Herbert L. J. Haller and Frederick B. Laforge. U. S. pat. 2,096,715, Oct. 26, 1937. (A. P.-C.)

Eugenol and Diallyl—Catalytic Isomerization of. The first attempt was made to isomerize diallyl and eugenol compounds by using palladium and platinum contacts. The compound diallyl was exposed to a stream of carbon dioxide at a temperature of 200° . The yield obtained during this reaction was about 30%; by adding to the finished product the catalytic agents palladium and platinum and raising the temperature to 300° , the yield was increased to 73.4%. The product obtained was named dipropenyl. Eugenol, however, was completely converted to isoeugenol at 300° and the catalytic agent used was platinum. In both cases where the use of catalytic agents was made, the double bonds were displaced at the α -, β - and ν -positions.—R. J. Lewing. Chem. J. Ser. A. J., 6 (1936), 1092; through Chem. Zentr., 108 (1937), 586. (G. B.)

Glucosyl-(6)-Piperidine—Synthetic Derivatives of. The compound 5,6-anhydro-mono-acetoglucose (1) reacts especially with *primary amines* to yield monoglucosylamine. The reaction must be conducted in the cold and an excess of amine must be prevalent; diglucosylamine is also yielded during this reaction. The excess of amine must be carefully added during the reaction; the temperature must also be kept low, because the heat liberated during the reaction may char the anhydrosugar. The same reaction cannot be conducted in alcohol. The compound monoacetonylglucosyl-(6)-piperidine can be produced by carefully hydrolyzing isodiacetonglucosyl-(6)-piperidine.—H. Ohle, E. Euler and W. Malerczyk. *Ber.*, 69 (1936), 1936; through *Chem. Zentr.*, 108 (1937), 609.

Iodine Compounds, New, and Bactericidal Products Resulting Therefrom. An amount of iodine greater than is necessary for the stoichiometric saturation of the acid is added to a solution of a glycoholic or taurocholic acid.—Wm. R. WARNER & Co., Inc. Belg. pat. 420,730, April 30, 1937.

(A. P.-C.)

Lanosterol and Agnosterol. Proof that these two compounds are identical can be shown by the fact that they give the same ketone, α -dihydroagnostenone upon dehydrogenation with copper at reduced pressure. α -Dihydroagnostenone upon reduction with sodium in isopropyl alcohol gave the original α -dihydroagnosterol. With aluminum isopropylate in isopropyl alcohol the ketone gave a mixture of epimers which did not precipitate with digitonin and which could be separated by crystallization of the acetates into α -dihydroagnosteryl acetate and the more soluble epi- α -dihydroagnosteryl acetate. Reduction of α -dihydrolanostenone with aluminum isopropylate in isopropyl alcohol gave a mixture of epimers which upon crystallization of the acetate gave epi- α -dihydrolanosteryl acetate, and α -dihydrolanosteryl acetate. Both compounds gave the original ketone upon hydrolysis and oxidation.—R. E. Marker and E. L. Wittle. J. Am. Chem. Soc., 59 (1937), 2289. (E. B. S.)

Local Anesthetics—New Group of. I. α-Di-Alkylamino-Acid Anilides. The following amides have been prepared and their toxicity and anesthetizing action investigated: dimethylamino-, melting point 37° (perchlorate, melting point 104-106°), diethylamino-, boiling point 116-117°/0.15 mm. (perchlorate, melting point 159-160°), and piperidino-acetanilide, melting point 99-100° (perchlorate, melting point 188-190°); diethylaminoacetethylanilide, boiling point 135-136°/0.1 mm. (perchlorate, melting point 148-149°); α-diethylamino-, boiling point $140-143^{\circ}/0.4$ mm. (perchlorate, melting point $138-140^{\circ}$), and α -diethylamino-propionanilide, boiling point 126-127°/0.2 mm. (perchlorate, melting point 169-170°); dimethyl-, melting point 61-62° (perchlorate, melting point 178-179°), and diethyl-aminoaceto-toluidide (I), boiling point 159-160°/0.1 mm. (perchlorate, melting point 198-199°); α-dimethyl-, boiling point 190-191°/3 mm. (perchlorate, melting point $169-170^{\circ}$); (hydrochloride, melting point $183-184^{\circ}$), and α -diethyl-aminopropion-o-toluidide, boiling point 126-127°/0.2 mm. (perchlorate, melting point 164-166°); diethylaminoacet-o-anisidide, boiling point 149-150°/0.45 mm. (perchlorate, melting point 148-149°); dimethylaminoacet-p-toluidide, boiling point 117-118°/0.2 mm. (hydrochloride, melting point 184-185°); diethylaminoacet-p-anisidide, boiling point 182-183°/0.1 mm.; dimethylaminoacet-2:4-, boiling point 136-137°/0.4 mm., melting point 45°, and -2:5-xylide, boiling point 153-154°/0.05 mm., melting point 34° (perchlorate, melting point 182-183°); dimethylaminoacet-2:5-dimethoxyanilide, melting point 100-102° (perchlorate, melting point 173-174°). In general, replacement of NMe₂ by NEt₂ and of NHPh by NH.C₀H₄Me-o enhances physiological activity so that I is more powerful than novocaine. Replacement of NHPh by NH.CoHoMe-p is unfavorable owing to increased toxicity. Increase in length of the carbon chain of the NH₂-acid component diminishes the activity and increases the toxicity.—H. ERDTMAN and N. Lofgren. Svensk Kem. Tid., 49 (1937), 163-174; through J. Soc. Chem. Ind., 56 (1937), 1130. (E. G. V.)

Phenol—Action of Chlorine on, in Alkaline Solution, and the Possibility of Preparing Chloranil. Chlorination of phosphorous hydroxide in aqueous sodium hydroxide at room temperature leads to production of mono, di- and tri-chlorophenol (I). Acidification of the reaction solution at the stage of formation of I liberates hypochlorous acid, which reacts with I to yield trichlorophenyl hypochlorite and tetrachlorobenzoquinone (II). The yield of II may be raised to 44% by treating the precipitate obtained at this stage with nitric acid-hydrochloric acid. Further chlorination of II in acid solution leads to complete oxidation.—J. TSCHULKOV, V. PARINI

and E. Staroseletz. Prom. Org. Khim., 3 (1937), 97-101; through J. Soc. Chem. Ind., 56 (1937), 1018.

Phenol—New Derivatives of. In condensing phenol with either amylene hydrate or trimethylcarbinol in the presence of aluminum chloride it was possible to obtain yields as high as 70% consisting of alkylphenol. During the same reaction the by-product alkylbenzol was obtained. The compound alkylphenylether was obtained when isopropyl or butyl alcohol was replaced with phenol in the presence of aluminum chloride. A confirmation of this reaction was made possible when a 70% yield of isopropylanisol was obtained from anisol and isopropylalcohol in the presence of aluminum chloride.—I. Zukerwanik and S. Nasarowa. Chem. Zentr., 108 (1937), 379.

Piperazine—Derivative of. XII. Alpha Amino Ketones Derived from N-Phenylpiperazine and Derivatives. Several α-amino ketones derived from N-phenylpiperazine have been prepared with the object of studying the effects of various reducing agents on these ketones. The syntheses which may be carried out in two ways are described.—B. L. HAMPTON and C. B. POLLARD. J. Am. Chem. Soc., 59 (1937), 2446. (E. B. S.)

Pyramidone—Manufacture of. The free and combined formic acid is removed from water-formic acid solutions of pyramidone, such as those obtained from methylation of aminoantipyrine with formaldehyde in water-formic acid, by evaporating the solution and heating the residue to about 100–140° (and at less than 1 atmosphere), if desired in presence of an inert vapor, such as steam, carbon dioxide or any inert liquid such as toluene or light petroleum.—M. N. DVORNIKOFF. U. S. pat. 2,045,588; through J. Soc. Chem. Ind., 56 (1937), 1270. (E. G. V.)

Pyrimidic Derivatives. A carboxylic acid amidine which can be used in the synthesis of vitamin B, has the general formula $Z_1(Z_2)RY$ in which R is an aliphatic radical, Z_1 and Z_2 each represents a cyanogen, hydroxymethylene, etc., group both attached to the same carbon atom, and Y is a halogen, aralkoxy, alkoxy, etc.—I. G. Farbenindustrie A. G. Belg. pat. 420,740, April 30, 1937. (A. P.-C.)

Quinoline Derivatives. A cyanuric halide is condensed with a primary or secondary diaminoquinoline containing one amino group in the pyridine nucleus and the other in the benzene ring. Alternatively, a benzylaminoquinoline substituted on the nitrogen atom by a cyanuric residue may be treated by standard processes to introduce an amino group into the pyridine nucleus. If the products still contain halogen linked to the cyanuric nucleus, they may be treated to replace the halogen by amino, hydroxyl or an alkoxy or substituted amino group, or as a further alternative one or two halogen atoms of the cyanuric halide may be replaced by hydroxyl, amino, etc. The products are useful as bactericides and in the treatment of diseases due to protozoa.—

Heinrich Jensch, assignor to Winthrop Chemical Co. U. S. pat. 2,092,352, Sept. 7,1937.

(A. P.-C.)

Racemic Trans-Ephedrine, Etc.—3-Coumarincarboxylic Acid Salts of. Aromatic amino alcohols of the general formula ArCH(OH)CH(X)NYZ, in which Ar represents a phenyl or substituted phenyl group, and X, Y, Z each represents hydrogen or an alkyl residue, are treated with coumarin-3-carboxylic acid to form salts useful in medicine for increasing respiration or increasing blood pressure. Thus, a solution of racemic trans-ephedrine in acetone is treated with coumarin-3-carboxylic acid to give a salt that melts at 196° C.—Otto Dalmer and Fritz von Werder, assignors to Merck & Co. U. S. pat. 2,094,000, Sept. 28, 1937. (A. P.-C.)

Sulfonates—Aliphatic Alcohol, from Soap Substitutes to. The properties of the sulfonates depend on the chain length; octyl and lauryl alcohols yield good wetting agents, cetyl alcohol affords a good detergent, stearyl alcohol a good brightening agent. The uses of the sulfonates are described, and formulæ given for mixtures.—C. H. FISHER. Kunstseide, 19 (1937), 186–189; through J. Soc. Chem. Ind., 56 (1937), 1167. (E. G. V.)

BIOCHEMISTRY

Adrenaline—Enzyme Method for the Estimation of, from Suprarenal Glands. A colorimetric method is based on the oxidation of adrenaline to a red compound by the oxidase prepared from the seeds of *Dolichos lablab*. Color formation takes place between $p_{\rm H}$ 4.4–7.6 and is more stable in the acid range; color develops within 20 seconds, and a good proportionality exists between different concentrations of adrenaline and the intensities of the color. The method involves extracting with 0.1N hydrochloric acid the fresh glands from dogs and monkeys, precipi-

tating the protein by adding sodium acetate and heating, filtering, diluting to known volume, and finally treating two cc. of the solution with two cc. 0.5M phosphate buffer $(p_{\rm H}~6.0)$, three drops 1% peroxide and two-cc. enzyme solution. The color, which develops within one minute, is then compared with the standard. The results are in good agreement with those obtained by the blood-pressure method.—Kamala Bhagvat. Current Sci., 5 (1937), 646-647; through Chem. Abstr., 31 (1937), 7193. (F. J. S.)

Ascorbic Acid—Chemical Identification of, in Urine. From 12 liters of urine, 20 mg. of the pure 2,4-dinitrophenylhydrazine derivative of dehydroascorbic acid were isolated by mixing the fresh urine with oxalic acid, evaporating to 2 liters under carbon dioxide, filtering, completing the oxidation of ascorbic to dehydroascorbic acid by hydrochloric acid and iodine, adding 2,4-dinitrophenylhydrazine, incubating four days at 40° and isolating the hydrazone by repeated adsorption on alumina and crystallization from acetic acid and from acetone or acetone-alcohol. The hydrazone was identified by crystalline form, melting point and mixed melting point, color reaction with sulfuric acid and sodium hydroxide, absorption in the visible spectrum and distribution between two immiscible solvents. An unidentified 2,4-dinitrophenylhydrazone, similarly adsorbed, was isolated from the mother liquor of the first hydrazone. The urine used for this study was obtained from persons receiving an ordinary mixed diet without ascorbic acid supplements.—P. J. Drumm, H. Scarborough and C. P. Stewart. Biochem. J., 31 (1937), 1874; through Squibb Abstr. Bull., 10 (1937), A-2139. (F. J. S.)

Biological Measurements—Calculation of Error in.—H. von Schelling. Naturwissen-schaften, 25 (1937), 699; through Squibb Abstr. Bull., 10 (1937), A-2061. (F. J. S.)

Bismuth—Estimation of, in Urine, Rapid Clinical Method for. A short useful clinical method for estimating the bismuth content of urine is described. Ten cubic centimeters of urine are placed in a large test-tube, 0.4 Gm. potassium permanganate and two cc. concentrated sulfuric acid are added and the solution boiled gently over a microburner for approximately two minutes. Four-tenths gram oxalic acid is added and decolorization should take place. After cooling, 0.01–0.04 Gm. sodium sulfite and sodium sulfate and 0.05 Gm. sodium iodide are added. The fluid becomes a yellowish green if bismuth is present. This color is compared with a standard color scale. The results with this method were compared with those of a long oxidation color method (S. A. B., 8 (1935), A-1743) and the estimates of the daily excretion of bismuth agreed well in instances in which the volume of urine was known. The scope of usefulness of this method in oral medication with bismuth and other applications is discussed.—P. J. HANZLIK, A. J. LEHMAN, A. P. RICHARDSON and W. VAN WINKLE, JR. Arch. Dermatol. Syphilol., 36 (1937), 725; through Squibb Abstr., Bull., 10 (1937), A-2000. (F. J. S.)

Carotene Content of Pasture Plants. The contents in fresh and dry samples of 13 plants sampled May to November are given. High contents occur in early summer, but widely varying and tending to decrease in the hot months. Autumn rains raise the contents to summer value except in Andropogon spp.—W. J. Peterson and A. E. Aldous. J. Dairy Sci., 20 (1937), 557-562; through J. Soc. Chem. Ind., 56 (1937), 1265. (E. G. V.)

Fish and Beef—Nutritive Values of. Young rats fed exclusively on beef for 54 days cease to grow, whereas similar rats fed exclusively on sardines grow normally. The % nitrogen absorbed is 97.6 for beef and 96.9 for sardines. Growth is satisfactory when the beef is supplemented with cod liver oil; hence the principal cause of the inferiority of beef is its deficiency in vitamin-A. The possibility of mineral deficiency, in particular calcium, is not excluded.—O. C. Comes. Quad. Nutriz., 3 (1936), 342-350; through J. Soc. Chem. Ind., 56 (1937), 974.

(E. G. V.)

Galactose—Micro Determination of. Method for determining galactose in blood from finger prick. Fermented with yeast suspension, washed with sodium hydroxide and precipitated with zinc sulfate. Filtrate compared with standard as in glucose test.—Br. Della Maggiore. Diagnostica tec. lab., 7 (1936), 273; through Rev. sud-americana endocrinol., inmunol., químioterap., 20 (1937), 617. (G. S. G.)

Indol—Free, Determination of, in Blood. Technic can detect 0.0001 mg. indol per cc. blood. Procedure consists in shaking out in separatory funnel with methanol, extracting with petroleum ether and using acid solution of paradimethylamidobenzaldehyde for color reaction. Compared with standard solution in colorimeter. Technic may also be used for urine, exudates,

bile and fecal material with slight variations.—E. MACCHIA. Diagnostica tec. lab., 6 (1935), 628; through Rev. sud-americana endocrinol., inmunol., químioterap., 20 (1937), 611. (G. S. G.)

Lipase—Detection of, in Serum. Superior method.—E. Bullo and R. Poll. Diagnostica tec. lab., 7 (1936); through Rev. sud-americana endocrinol., inmunol., químioterap., 20 (1937), 616. (G. S. G.)

Lipoids—Precipitating Substances Such as, with Higher Alcohols. Octyl alcohol or other alcohols containing 5 to 11 carbon atoms are used as precipitating agents in operations such as the obtainment of lipoids from liver juices or blood serum.—Hermann E. Schultze, assignor to Winthrop Chemical Co. U. S. pat. 2,094,931, Oct. 5, 1937. (A. P.-C.)

Mercury—Determination of, in Urine. There is introduced into the urine copper wool which decomposes the mercury salt and forms an amalgam. The latter is withdrawn after 24 hours and heated with iodine; the mercury distils with the iodine and forms on the cold wall of the container a red ring which is compared with standard rings. The ring is dissolved in iodine solution which is shaken with a mixture of copper sulfate and sodium carbonate, forming a complex cupro-iodide of mercury which colors the suspension and is compared with standard suspensions.—

E. Peregoud and E. Kuzbuba. Hig. Truda, 14 (1936), 71–72; through Chimie & Industrie, 38 (1937), 240.

(A. P.-C.)

Milk—Freezing Point of, a New Apparatus for the Rapid and Economical Determination of. A description of the apparatus and its advantages over the Hortvet apparatus with a table of typical results.—P. L. TEMPLE. Analyst, 62 (1937), 709. (G. L. W.)

Morphine—Determination of Small Amounts of, in Blood. A simple inexpensive colorimetric method adapted from that of Sanchez (cf. Chem. Abstr., 24, 1724) is described; morphine can be determined in the blood when the concentration is not less than 0.0025 mg. per cc. The method is practical for animal experimentation but is not sufficiently delicate for use with human subjects receiving therapeutic doses of morphine. Whole blood is used, since the drug can be found in both cells and plasma.—James W. Mull. Proc. Soc. Exptl. Biol. Med., 35 (1937), 551-553; through Chem. Abstr., 31 (1937), 7077. (F. J. S.)

Pregnandiols in Pregnancy Urine of Mares. The authors have obtained pregnandiol from mares' pregnancy urine. Pregnandione and its isomer, allo-pregnandione, were obtained by first oxidizing the total carbinol fraction and separating the ketones as their semicarbazones.—R. E. Markerm, O. Kamm, H. M. Crooks, Jr., T. S. Oakwood, E. J. Lawson and E. L. WITTLE. J. Am, Chem. Soc., 59 (1937), 2297. (E. B. S.)

Preservatives for Foods. The esters of para hydroxy benzoic acid are preferable to those of benzoic acid and salicylic; their use is permitted by Norway, Hungary, Jugoslavia, Rumania and Germany.—T. Sabalitschka. Proc. 5th Intern. Cong. Tech. Chem. Agric. Ind., Holland, 2 (1937), 454–455; through J. Soc. Chem. Ind., 56 (1937), 977. (E. G. V.)

Preservatives in Foods. The amount and form of preservatives permissible in sausages, sausage meat, fruit juices, cordials, syrups, etc., in various countries are tabulated. The preservatives are sulfur dioxide, boric acid, sodium benzoate, formic acid and metabisulfite.—T. W. Corran. Food Manuf., 12 (1937), 232-234; through J. Soc. Chem. Ind., 56 (1937), 977. (E. G. V.)

Products for Rendering the Urine Bacteriostatic. A therapeutic agent for oral administration for rendering the urine bacteriostatic comprises esters of simple organic acids (such as acetoacetic, hydroxybutyric, butyric, phenolbutyric, valeric, isovaleric and crotonic) which are known to cause (when administered as acids) the excretion of acetone bodies in the urine.—Theodore H. Rider, Robert Shelton and John Haynes, assignors to The Wm. S. Merrell Co. U.S. pat. 2,101,097, Dec. 7, 1937. (A. P.-C.)

Proteins—Tissue, Composition of. IV. Estimation of Cystine. The principle of Vickery and White [cf. S. A. B., 6 (1933), 260] for the determination of cystine (I) by cupric oxide (II) has been applied to the microdetermination of I in proteins. The analysis requires only about 2 mg. I and can be completed within four hours after the initial protein hydrolysis. The method involves hydrochloric acid-hydrolysis of the protein, reduction of I in the hydrolysate to cysteine (III) with zinc dust, filtration at $p_{\rm H}$ 5 from humin and excess zinc, precipitation of III with II at $p_{\rm H}$ 4, washing the precipitate with dilute citrate-acetate buffer of $p_{\rm H}$ 3.6, and optional ultimate Kjeldahl digestion or ignition for sulfur determination. Evidence is presented demonstrating the equivalence of nitrogen and sulfur in the cuprous precipitate.—S. Graff, E. Maculla and A. M. Graff. J. Biol. Chem., 121 (1937), 81; through Squibb Abstr. Bull., 10 (1937), A-2004. (F. J. S.)

Science and the Conservation of Food. A lecture.—T. Macara. *Proc. Roy. Inst.*, 29 (1937), 657–682; through *J. Soc. Chem. Ind.*, 56 (1937), 977. (E. G. V.)

d-Sorbitol—Sources of. Fruits of the Rosaceæ family are sources of sorbitol. Small quantities of sorbitol have been obtained from the leaves of these plants. Commercially, sorbitol is prepared by hydrogenation of glucose and is sold in the form of a syrup containing about 50% of the reduction products. Pure sorbitol is isolated readily from the syrup by the use of pyridine for crystallization of the residue obtained by evaporation of the water at reduced pressure.—H. H. Strain. J. Am. Chem. Soc., 59 (1937), 2264. (E. B. S.)

Sugar—Determination of, in Urine. Comments are made emphasizing the daily amount of sugar excreted and not the per cent of sugar in the sample.—ULRICH BAUMEISTER. Apoth. Ztg., 53 (1938), 134. (H. M. B.)

Sugar Alcohols. IX. A Physicochemical Study of the Erythritanboric Acid Complex. The erythritan (1,4-anhydroerythritol, I)-boric acid (II) complex increases in hydrogen-ion concentration as the ratio of I to II increases, and as the concentration increases. The dissociation of the complex is not permanently affected by time and temperature. The number of moles of I or levulose (III) combined with one mole of II in the I-II and III-II complexes, respectively, is approximately 1.3.—J. C. Krantz, F. F. Beck, C. J. Carr. J. Phys. Chem., 41 (1937), 1087; through Squibb Abstr. Bull., 10 (1937), A-2064. (F. J. S.)

Tetramethyldiaminotriphenylmethane. Blood Color Reaction with. Describes procedure of color test to detect blood by reaction with malachite green. May be used with old blood as well as fresh. Sensitivity practical at 1: 100,000 dilution.—J. ALVAREZ DE TOLEDO and R. VALERO. Cronica Medica, 39 (1935), 331; through Rev. sud-americana endocrinol., inmunol., químioterap., 20 (1937), 614. (G. S. G.)

Vitamin Control—Difficulties in the, of Proprietary Food Preparations. With rats as test animals for vitamin D, flavoring materials and considerable dilution with coconut cake cause difficulties in controlling the amount of intake and lead to false results, that is, too large an intake leading to large increases in body weight and a rachitic condition, or vice versa. The examination of the diethyl ether extract is preferable, but this does not overcome the effect of essential oils on the ration or their destructive action on vitamin D. Owing to added calcium and phosphorus, chemical analyses are necessary in order to prepare an identical control ration.—M. BAUWEN. Proc. 5th Intern. Cong. Tech. Chem. Agric. Ind., Holland, 1 (1937), 71–78; through J. Soc. Chem. Ind., 56 (1937), 976. (E. G. C.)

Vitamin A—Destruction of, in Cod Liver Oil by Rancidity. Vitamin A was destroyed in cod liver oil as rancidity developed. The vitamin A was destroyed at lower peroxide values when rancidity developed at room temperature than when it was accelerated by aeration at 100° C. It is suggested that not only vitamin A, but other biological properties of oils, are not equally affected by rancidity produced in different ways and at different rates, and that peroxide value may not necessarily be parallel to that of other changes which take place in an oil as rancidity develops.—Dorothy V. Whipple. Oil & Soap, 13 (1936), 231-232; through Chimie & Industrie, 38 (1937), 320. (A. P.-C.)

Vitamin C Potency—Effect of Storage on the, of Foodstuffs. Coriander, tender amaranth and fenugreek leaves lose vitamin C rapidly on storage, the rate of loss at 38° being greater than at room temperature. Mangoes, chillies and bitter gourds lose very little vitamin C if stored while green, but after ripening an appreciable loss occurs.—S. Ranganathan. *Indian J. Med. Research*, 23 (1936), 755-762; through J. Soc. Chem. Ind., 56 (1937), 976. (E. G. V.)

Vitamin D—Relation of, to Skin Respiration. The oxygen-uptake of the skins of rachitic rats, as measured in the Warburg apparatus was only 60-70% of that of the skins of normal control rats of the same age. On realimentation with viosterol in oil the former rachitic animals showed the same skin respiration, as the normal controls. It is not known whether the effect of vitamin D on skin respiration, and thus on skin vitality, is due to a direct effect on the calcium phosphate metabolism of the skin or to an indirect effect via the change in blood calcium or phosphorus or via the thyroid or other internal secretion. The experiments were undertaken in an attempt to explain the favorable effects of the vitamin on skin tuberculosis, wounds, etc.—A. K. Presnell. J. Biol. Chem., 121 (1937), 5; through Squibb Abstr. Bull., 10 (1937), A-2030.

Yeast—Process of Treating Liquid. A 30 to 50% suspension of yeast in the liquid in which it was grown is subjected at a temperature not over 16° C. to the influence of ultraviolet rays of 2300 to 4000 Angstrom units. The treatment is carried out in a closed system and in substantial absence of oxygen until yeast cells of a desired uniform potency are obtained.—ROBERT F. LIGHT, CHAS. N. FREY and GERHARDT J. PATITZ, assignors to STANDARD BRANDS, INC. U. S. pat. 2,099,025, Nov. 16, 1937.

(A. P.-C.)

ANALYTICAL

Acetophenetidine—Determination of, in the Presence of Caffeine and Aspirin. Experiments made on a known mixture to test the effect of sample size (from 0.4 to 1.5 Gm.) on caffeine recovery showed it had practically no influence. Recoveries obtained were: aspirin 98 24-101% (average 99.6%); acetophenetidine 98.4-100% (average 99.4%); caffeine 100-122% (average 110%).—Solomon M. Berman. J. Assoc. Official Agr. Chem., 20 (1937), 574-575; through Chem. Abstr., 32 (1938), 305. (F. J. S.)

Acetyl—Determination of, Especially in O-Acetyl Compounds. The general method for the determination of acetyl is based upon the principle of an alkaline, alcoholic hydrolysis of an acetyl compound, followed by acidification and distillation of the liberated acetic acid. For the distillation the alkali is neutralized with enough strong magnesium sulfate solution containing sulfuric acid, so that the reaction mixture has a volume of 20 cc. This is steam-distilled at constant volume until 50 cc. of distillate are obtained. The acetic acid which comes over under these conditions represents 95.7% of the total acetic acid formed by the hydrolysis of the acetyl compound. If distillation of the liberated acetic acid is conducted at such a rate that the reaction mixture is concentrated to approximately 15 cc. during the collection of the 50 cc. of distillate, the entire quantity of acetic acid is found in the distillate.—E. P. CLARK. Ind. Eng. Chem., Anal. Ed., 9 (1937), 539.

Alkaloids—Microchemical Methods for the Identification of. Aqueous 5% potassium iodide is satisfactory as a microchemical reagent for the identification of apomorphine. Addition of 1 drop 5% hydrochloric acid and one drop of freshly prepared 5% aqueous potassium ferrocyanide is satisfactory for hydrastine. Freshly prepared 2% solution of silver nitrate in 5% ammonium hydroxide is suitable for theophylline, and 5% aqueous mercuric chloride can be used as a confirmatory test. The technic of the test and the description of the crystals are given in J. Assoc. Official Agr. Chem., 20 (1937), 79-80. Collaborative study of the tests showed they were reliable.—C. K. GLYCART. J. Assoc. Official Agr. Chem., 20 (1937), 551-553; through Chem. Abstr., 32 (1938), 305. (F. J. S.)

Aminophylline—Analysis of. Theophylline can be determined in aminophylline by extraction with a 3:1 chloroform-isopropyl alcohol mixture, the solution being slightly acidified; this extraction can be applied to tablets and other preparations containing theophylline. The ethylenediamine in aminophylline can be titrated using standardized acid with bromocresol green as indicator ($p_{\rm H}$ about 4.6). In drying at ordinary temperature the loss in weight is due to evaporation of both ethylenediamine and water. These two substances can be removed completely by drying at 125°; consequently, this drying, in connection with a titration of ethylenediamine, can be used for determining the percentage of water and, in connection with an examination of the purity of theophylline, for determining the percentage of theophylline.—F. Reimers. J. Assoc. Official Agr. Chem., 20 (1937), 631–635; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Ammoniated Mercury. Excessive Requirements of the Hungarian Pharmacopæia IV. According to this pharmacopæia the filtrate of the water-soluble as well as the alcohol-soluble extract should yield upon evaporation "no weighable residue." The following method was used to test this requirement on 10 commercial samples: "Add 10 cc. water to 1 Gm. of the substance in a test-tube and shake vigorously for 1 minute. Filter through a double filter washed previously with water; rinse the test-tube and filter with 5 cc. water; evaporate the clear filtrate in a glass dish to dryness on a water-bath, dry the residue for 30 minutes at 100° C. These samples yielded from 0.34-1.2% water-soluble residue and 0.22-0.80% alcohol-soluble residue. Samples prepared according to the German Pharmacopæia did not meet the Hungarian Pharmacopæial requirement.

—C. A. ROJAHN and FRANZ SLANINA. Apoth. Ztg., 53 (1938), 39-40. (H. M. B.)

Ascorbic Acid—Chemical Determination of. Various methods are discussed. The method of Fujita, Iwatake and Miyata (*Biochem. Z.*, 227 (1935), 296–304) is not specific for ascor-

bic acid. The dichlorophenolindophenol method is the most reliable.—P. MANCEAU, A. A. POLICARD and M. FERRAND. Bull. soc. chim. biol., 18 (1936), 1369-1386; through Chimie & Industrie, 38 (1937), 937. (A. P.-C.)

Benzene—Certain Physicochemical Constants of, Measurements of. A series of preparations of benzene of a high degree of purity was made by fractional distillation, by azeotropic distillation with ethanol and by crystallization followed by centrifuging. The following chemical constants were measured: boiling point $80.094 + 0.002^{\circ}$ C.; freezing point, $5.51 \pm 0.01^{\circ}$ C.; refractive index n_D^{25} , 1.49807 ± 0.00006 ; and density at 25° C., 0.87366 Gm./em³ ± 0.00002 . Previous determinations of these constants, as reported in the literature, are reviewed.—M. WOJCIECHOWSKI. J. Research Natl. Bur. Standards, 19 (1937), 347; through Squibb Abstr. Bull., 10 (1937), A-1920. (F. J. S.)

Benzoic Acid. This paper is one of a series of reviews of the pure organic substances of the Belgian Pharmacopœia. The subject is discussed at length under the following headings: synonyms; etymology; occurrence; history; preparation (from benzoin, from hippuric acid and synthetically); commercial varieties; quality; physical properties; chemical properties; identification; reactions; official requirements; assay and use (internally; externally; in the pharmacy; in the laboratory; in pharmaceutical industry and technically). The review closes with a bibliography of 23 references.—V. Evrard. *Pharm. Tijdschr.*, 14 (1937), 185. (E. H. W.)

Benzyl Compounds—Determination of, in Medicinal Preparations. A method has been evolved, and is described in detail, for determining benzyl benzoate in solution in oil. It consists essentially in saponifying with alcoholic sodium hydroxide or potassium hydroxide, removing most of the alcohol, dissolving in sufficient water to keep the benzoic acid in solution, acidifying with sulfuric acid, separating the fatty acids, extracting benzoic acid from an aliquot of the aqueous solution with chloroform in presence of sodium chloride, evaporating the solvent, dissolving in alcohol and titrating with 0.1N alkali. When the product consists only of benzyl benzoate and olive oil, the percentage of benzyl benzoate = 100-1000 (1.567-r), where r is the n_{25} ; the results are accurate to within 1-2%.—S. Reznek. J. Assoc. Official Agr. Chem., 20 (1937), 560-562; through Chem. Abstr., 32 (1938), 305. (F. J. S.)

Bismuth—Sulfates of. In a study of the sulfates of bismuth at the Pharmaceutical Faculty at Prague University the existence of compounds of formulæ Bi(OH)SO₄.H₂O, BiH(SO₄)₂.H₂O, BiH(SO₄)₃.3H₂O and Bi₂(SO₄)₃.3H₂O is confirmed, while the following new salts are described: Bi(OH)SO₄, Bi(OH)SO₄.4H₂O, Bi₂(SO₄)₄.7H₂O and Bi₃H(SO₄)₅.6H₂O. Phytomicrographs are reproduced to show the characteristic appearance of the different salts. Details of the methods of preparation and the precautions (e. g., temperature limits) taken to ensure pure products are given. Thus, the anhydrous basic salt is obtained from a solution of 20 Gm. Bi(NO₃)₃ in 60 cc. water by adding slowly 120 Gm. of concentrated sulfuric acid. The mass finally warms to 130–140° C., at which temperature the anhydrous basic salt as the solid phase can be removed, drained on a porous plate, washed with alcohol and ether (or glacial acetic acid) and dried in a desiccator over sulfuric acid or calcium chloride. The white micro-crystalline mass is stable in air, water hydrolyzes it, but hydrochloric acid and nitric acid dissolve it completely, sulfuric acid only partially.—S. SKRAMOVSKY and O. VONDRASEK. Coll. Czech. Chem. Comm., 9 (1937), 329; through Pharm. J., 140 (1938), 57. (W. B. B.)

Butter and Ghee—Routine Test for the Detection of Highly Hardened Oils and Mutton and Beef Fats in. One cubic centimeter of the filtered melted sample is treated in a test-tube 160 x 16 to 18 mm. with 15 cc. of a mixture containing 650 cc. of dry acetone with sufficient absolute alcohol to make 1000 cc. The tube is warmed and stoppered and maintained at 30° C. for 3 hours. On examination in transmitted light, the presence of crystals indicates the presence of mutton fat, beef fat or a hardened oil.—V. VENKATACHALAM. Analyst, 62 (1937), 732.

(G. L. W.)

Camphor, Menthol and Methyl Salicylate—Determination of, in Mixtures. The mixture is distilled in steam, and the distillate extracted with ether, which is then evaporated off. The residue is saponified and the salicylic acid determined bromometrically. Menthol and camphor are extracted from the hydrolysate with ether and determined by acetylation and formation of the 2:4-dinitrophenylhydrazone, respectively.—K. K. Andersen. Dansk Tids. Farm., 11 (1937), 208-216; through J. Soc. Chem. Ind., 56 (1937), 1168. (E. G. V.)

Carbon and Hydrogen—Determination of, Content by Combustion. By simple improvement of the customary combustion apparatus, and patient burning of the sample in air instead of oxygen, results are obtained for carbon and hydrogen which are accurate to 0.01%. A value of 12.005 for the atomic weight of carbon, instead of 12, gives closer agreement between actual and theoretical calculations; this atomic weight is in keeping with recent atomic weight determinations.—M. W. Renoll, T. Midgley, Jr., and A. L. Henne. Ind. Eng. Chem., Anal. Ed., 9 (1937), 566.

Cellulose Wool—Detection of, in Bandage Materials.—A technic is described for detecting cellulose wool by reaction toward dyes. A useful comparison table including the reaction of six different fibers of which two are cellulose fibers made by the usual commercial methods toward solutions of eight coloring matters.—Liesche. Apoth. Ztg., 52 (1937), 1577-1578. (H. M. B.)

Chlorides and Bromides—Determination of. Add 5 cc. of 6N nitric acid to 25 cc. of 0.03658N potassium chloride solution, and then 25 cc. of 0.1N silver nitrate. Stir to coagulate the precipitate, filter and wash the precipitate of silver chloride with 60 cc. of 1N nitric acid. To the combined filtrate and washings add 3 cc. of 0.5% starch solution and 0.1 cc. of 0.1N ceric ammonium sulfate solution. Titrate the silver with 0.1N potassium iodide solution to the appearance of a permanent blue-green color. Subtract 0.1 cc. from the burette reading as an indicator blank. (Procedure 2): Add 15 cc. of 6N nitric acid to 25 cc. of 0.03658N potassium chloride solution, and then 25 cc. of 0.1N silver nitrate. Dilute to the mark in a 100-cc. volumetric flask, mix thoroughly, filter through a dry filter and reject the first portion of the filtrate. Titrate 50 cc. of the subsequent filtrate with 0.1 potassium iodide solution as above.—L. A. Reber and W. M. McNabb. Ind. Eng. Chem., Anal. Ed., 9 (1937), 529. (E. G. V.)

Chloroform—Test for Phosgene in. Treat 1 cc. of a 1% solution of p-dimethylaminobenz-aldehyde and diphenylamine in acetone with chloroform containing 0.01% of phosgene; after 15 minutes a strong yellow color has developed. Chloroform containing 0.005% of phosgene shows a faintly yellow color after 30 minutes. The test, long in use for the detection of phosgene in air, is more sensitive than the silver nitrate test of the Pharm. Helv.—V. L. ROSENTHALER. Pharm. Acta Helv., 12 (1937), 6-7; through Chimie & Industrie, 38 (1937), 929. (A. P.-C.)

Cinchophen and Sodium Bicarbonate Tablets—Assay of. Cinchophen tablets can be assayed by dissolving and titrating the cinchophen in neutral alcohol (N. F. VI procedure); sodium bicarbonate, which is often compounded in cinchophen tablets, causes erroneous results. Collaborative study showed that excellent results are obtained by dissolving such tablets in alcohol + 10 cc. of 4% sodium hydroxide, acidifying with hydrochloric acid, extracting with 1+1+2 ethanolether-chloroform mixture, evaporating the solvent and titrating cinchophen with 0.1N alkali. Details of the technic are given in J. Assoc. Official Agr. Chem., 20 (1937), 83.—R. L. VANDAVEER. J. Assoc. Official Agr. Chem., 20 (1937), 589-590; through Chem. Abstr., 32 (1938), 307.

(F. J. S.)

Citric Acid, Tartaric Acid and Cream of Tartar—Detection of, in the Presence of Sugar. Citric and Tartaric Acids.—Shake 5 Gm. of the sample in a test-tube with 10 cc. of methylated ether and, after about 1 minute, filter into a small flask. Evaporate off the ether, and to the residue add about 1 cc. of water. Test 2 or 3 drops of this solution for tartaric acid by the resorcinol and sulfuric acid test. Wash the rest of the solution in the flask into a boiling tube with a few cc. of water and test for citric acid by the mercuric sulfate and potassium permanganate test. The solubility of these acids in ether is sufficient to give a good positive reaction. Cream of Tartar.—If there is a negative or only a slight positive reaction for tartrate by the test described above, the following test should be applied: Shake the same portion of the sample with 10 cc. of acidified ether (1 drop of dilute sulfuric acid in 20 cc. of ether) and proceed as before. In the presence of cream of tartar a good positive reaction will be obtained.—R. DeGiacomi. Analyst, 62 (1937), 731.

Coconut Shells—Composition of. Coconut shells have been analyzed by the standard methods employed for woods. The results of the analysis are recorded. Higher percentages of lignin, total pentosans and pentosans in the cellulose were found in the coconut shells than in the woods. The percentages of the cellulose and the holocellulose from the shells are considerably lower than the values of the corresponding materials in the woods. The acetic acid content and the metoxyl content of the shells are about the same as those of the woods.—L. C. Fleck, W. G. VAN BECKUM and G. J. RITTER. J. Am. Chem. Soc., 59 (1937), 2279. (E. B. S.)

Codeine—Colorimetric Microdetermination of. Morphine (I), codeine (II) and narceine are isolated from opium, and I is determined by the methods of Ginzberg and Juraschevski. The I + II content is determined as follows: 1 cc. of 2% hydrochloric acid and water to 10 cc. are added to 5 cc. of solution, and to 3 cc. of standard 0.05% I solution of the same (acetic acid) as the test solution. Four cubic centimeters of bromine are added to 5 cc. of 10% sodium hydroxide, and water is added to 50 cc.; 2 drops of this solution are added to each solution, followed by 2 drops of 3% hydrogen peroxide. One cubic centimeter of 25% aqueous ammonia is added to each solution, after 25 seconds 5 cc. of test solution are added to the standard, and 5 cc. of water to the test solution (to compensate for coloration of the extract), and the colorations are compared. The actual I content is deducted from the apparent I content. The difference times 1.83 equals the II content. A modification for analysis of poppy heads is described.—N. Juraschevski. Prom. Org. Khim., 3 (1937), 29-32; through J. Soc. Chem. Ind., 56 (1937), 1132. (E. G. V.)

Cod Liver Oil Emulsions—Evaluation of. By mixing a weighed sample (15 Gm.) with fresh anhydrous sodium sulfate (40 Gm.) and extracting (Soxhlet) with light petroleum, the oil can be recovered quantitatively in an unaltered condition suitable for further examination. The color test for liver oils is improved as follows: a solution of 0.06 Gm. of oil in 2 cc. of chloroform is poured into a mixture of concentrated sulfuric acid (0.06 cc.) and chloroform (2 cc.) and well shaken; the blue color which appears immediately with cod- or other fish-liver oil changes to violet-blue in 10 seconds and to brown after 1 minute. Free aldehydes and ketones (if present) can be detected in cod-liver oil emulsions by the ordinary methods. The Kreis reaction for rancidity [epihydrinaldehyde (I)] fails with the emulsions, but the "cotton-wool" test is satisfactory. I and the aldehydes and ketones present in other fats as well as in cod-liver oil are not responsible for the toxicity of some specimens of the oil. The vitamin-A activity of cod-liver oil was reduced to one-tenth when the oil was emulsified by the ordinary technical process, working in air; the vitamin-D activity was unaffected. The administration of the oil or the emulsion to calves after feeding produced no better growth than did coconut oil; if administered with drinking water before food the emulsification of the oil is necessary in order to avoid inferior results,—H. WERNER and H. Schmalfuss. Fette u. Seifen, 44 (1937), 348-351; through J. Soc. Chem. Ind., 56 (1937), 1235. (E. G. V.)

Colorimetric Measurements as—Methods of Rapid and Micro-Determinations. Photoelectric methods are applied to the determination of: (1) gluten quality; (2) maltose, by Berliner
and Schmidt's method; (3) phosphoric acid, by the molybdenum-blue method; (4) starch in bran
and feeding-stuffs, by the color produced with iodine; (5) bromates, by the zinc-iodide starch reaction; (6) phytosterol, by action of acetic anhydride and sulfuric acid on the chloroform extract.
Owing to the constancy of the phosphoric acid content of cereal ashes, (3) supplies a rapid method of
ash determination, particularly valuable when added sodium chloride interferes with the usual
methods. (6) is useful for determining fat in maize flour. (4) is a micro-method.—E. Berliner
and W. Kranz. Mühlenlab., 7 (1937), 89-94; through J. Soc. Chem. Ind., 56 (1937), 1073.

(E. G. V.)

Cubeb—Analysis of. A collaborative study of the previously described method for the analyses of species gave good results when applied to cubeb.—J. F. CLEVENGER. J. Assoc. Official Agr. Chem., 20 (1937), 602; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Cyanide—Determination of Small Quantities of, in Substances Treated with Insecticides. Evaporate the solution on a water-bath in the presence of an excess of sodium hydroxide to 1–2 cc. Add two drops of 1% sodium hydroxide and 0.5 cc. of 0.5% ferrous sulfate. Heat the resulting solution at 60–80° for 2–3 minutes. Cool to room temperature. After five minutes add 3 drops of 0.1% ferric chloride and 5 cc. of 1% hydrochloric acid and heat until colorless. Set aside for 1–2 hours until the appearance of a blue color. Dilute to volume and compare with a standard in the differential photocolorimeter. Separation of hydrogen cyanide from insecticide-treated substances by passing air through the solution and absorption with 1% alkali is recommended. Eighteen references.—M. M. Raines and A. I. Krupkin. J. Applied Chem. (U. S. S. R.), 10 (1937), 960–962 (in German 962); through Chem. Abstr., 31 (1937), 7001. (F. J. S.)

Derris Root—Rotenone Determinations in. The various methods of determining rotenone (I) are reviewed. It is concluded that it is best extracted with cold chloroform, the crude I weighed as the carbon tetrachloride complex, and the purity checked by the alcohol recovery

method.—P. A. Rowaan. Chem. Weekblad, 34 (1937), 605-606; through J. Soc. Chem. Ind., 56 (1937), 1267. (E. G. V.)

1,8-Dimethylxanthine Ethylenediamine. The product is very soluble in water, is hygroscopic and readily transformed by carbon dioxide into an insoluble carbonate. It gives a characteristic violet color when brought into contact with copper. It has an alkaline reaction which varies according to its ethylenediamine content. The commercial products practically never contain less than 70% of theophylline. The control of the manufacturing process is maintained by determining the ethylenediamine content by treatment of a weighed amount of the product in aqueous solution with excess of decinormal sulfuric acid and back-titration with decinormal sodium hydroxide in the presence of methyl orange.—A. Mossini. Boll. chim.-farm., 75 (1936), 557-558; through Chimie & Industrie, 38 (1937), 930. (A. P.-C.)

Dinitrophenol—Determination of, in Medicinal Preparations. A critical investigation of the excess bromination method for dinitrophenol was made and the conditions within reasonable limits for accurate quantitative results were determined. The quantity of excess bromine plays only a subordinate rôle; the bromination period is more important, a longer time than 1-3 minutes gives an error of about +4%; the acid concentration is most important, a lower acid content than 5-10 cc. hydrochloric acid for a total volume of 90-140 cc. may produce an error of up to -80%. Based on these findings a technic was developed (described in detail in J. Assoc. Official Agr. Chem., 20 (1937), 82-83) which was shown by collaborative study to give accurate results for the determination of dinitrophenol or its salts, singly and in admixture.—W. F. Kunke. J. Assoc. Official Agr. Chem., 20 (1937), 592-598; through Chem. Abstr., 32 (1938), 307. (F. J. S.)

Ergot Alkaloids-Report on Chemical Assay for. The investigation included a study of the Hampshire-Page method adapted to the assay of fluidextracts; the application of the wedge photometer devised by Clifford; a comparison of the results obtained by the photometer and the micro-colorimeter and also the separation and estimation of water-soluble alkaloids in tablets by means of chloroform and carbon tetrachloride. The latter procedure is carried out as follows: A sufficient number of tablets to contain 0.005 Gm. of the alkaloidal salt are treated with 20 cc. of a 1% solution of tartaric acid, filtered, transferred to a separator, made faintly alkaline with ammonia and extracted with five successive portions of carbon tetrachloride. The combined solvent is shaken with 1% solution of tartaric acid and made up to volume. The aqueous solution remaining in the separator is transferred to a volumetric flask, made up to volume with 1% tartaric acid solution and ergotoxine determined by comparison with a standard solution. For conversion to ergometrine the result is multiplied by 0.538. The obtained results indicated that the neutral wedge photometer was adaptable to the determination of ergot alkaloids. It is recommended that the methods for ergot be further studied .- C. K. GLYCART. J. Assoc. Official Agr. Chem., 20 (1937), 566; through Squibb Abstr. Bull., 10 (1937), A-2109. (F. J. S.)

Esters, Ethers or Ketones-Colorimetric Limit Test for Free Alcohol in. Qualitative Test.—Five cubic centimeters of the sample are mixed with 10 cc. of a freshly prepared mixture of 5 volumes of concentrated nitric acid made up to 100 volumes with glacial acetic acid. One-tenth cc. of a 15% aqueous potassium chromate is added and the solution shaken. The yellow color persists for at least 30 minutes if alcohols are absent. In the presence of 0.5% of proof spirit in the sample the color is quickly changed to a clear blue. Quantitative Test.—In adapting the reaction as a quantitative limit-test, it is preferable always to compare the sample with controls made up from a purified stock of the compound under test. Esters and ethers may be sufficiently purified by repeated washing with brine. Acetone is best purified by distilling with an oxidizing mixture (5 Gm. of potassium dichromate and 12.5 cc. of concentrated sulfuric acid, made to 100 cc. with water) and redistilling from alkali. The purified stock should give a negative result in the qualitative test already described. Five cubic centimeters of the purified stock are taken, and 0.5 cc. of aqueous ethyl alcohol of 20% proof strength is added. To a 5-cc. portion of the sample under test, 0.5 cc. of water is added. To each are then added 10 cc. of the fresh nitric and acetic acid reagent, and then 15% potassium chromate solution (as nearly simultaneously as possible) 0.1 cc. at a time, shaking after each addition, until the sample retains a distinctly orange tint for several minutes. If the color in the control is then still a clear blue, the alcohol in the sample is less than that in the control, i. e., less than 2% of proof spirit. Where the limit is higher than this, a smaller quantity of the sample should be taken; for example, for a 5% limit, 2 cc. of the sample are sufficient. By using a range of controls containing 0.1-0.5 cc. of alcohol solution, with 0.4-0.0 cc. of

water, a quantitative estimation of spirit within 0.2% of proof strength can be made.—E. G. Kellett. Analyst, 62 (1938), 728. (G. L. W.)

Ficus Carica—Constituents of.—From an aqueous extract obtained from the leaves of the *Ficus carica* a new substance was isolated; this compound had the composition of $C_{11}H_6O_3$ and was named ficusin. Apparently it has all the characteristics of a lactone.

The 3 oxygen-atom seems to react ethereal. When an alkali and dimethyl sulfate are added to it, a new compound takes place, namely, methyletherficusinic acid $C_{10}H_6O(OCH_3).CO_2H$. By oxidizing ficusin, furan-2,3-dicarbonic acid was the result. From these results the compound has the constitution formula 1(R-H); it almost resembles (in behavior) the isomer angelicin. Besides ficasin the author isolated from an acetone solution of the leaves, a small amount of bergapten $(R-OCH_3).-K$. OKAHARA. Chem. Zentr., 108 (1937), 364. (G. B.)

Ginger—Examination of. The authors examined numerous authenticated samples of ginger and some samples of commercial exhausted ginger in an attempt to set values by which adulteration of powdered ginger with exhausted ginger could be detected. It is their opinion that microscopic detection of such adulteration is impossible as only three of thirteen samples of exhausted ginger showed gelatinized starch grains. Separation of the outer layer of whole ginger and separate examination of the two layers showed the outer layer to be richer in alcohol and ether-soluble extractive. The difference was not as great in the case of Jamaica ginger as in the cases of African and Cochin gingers since no unscraped ginger is exported from Jamaica. Ground ginger rapidly loses alcohol-soluble extractive. The water-soluble extractive was much the same in the outer and inner portions. The conclusion reached from the work reported was: "any ginger having an alcoholic extract less than 3% is adulterated. One with an alcoholic extract between 3% and 4.5% may be genuine but the fiber should then be low (less than 3%), and the aqueous extract not less than about 12%."—G. D. Elsdon and C. Mayne. Analyst, 62 (1937), 836.

(G. L. W.

Glutathione—Isolation of, from Wheat Germ. Glutathione was isolated from wheat germ in yields of from 0.1–0.2 Gm. by treating 2 Kg. of fresh wheat germ with 5 liters of water and 50 Gm. of sulfosalicylic acid followed by filtering, centrifuging and precipitation of the supernatant liquid with neutral lead acetate. The lead precipitate was ground with 0.5N sulfuric acid, centrifuged and filtered and the copper, mercury and silver salts precipitated successively in the usual way. The final silver salt precipitate was treated with hydrogen sulfide and filtered. Absolute ethanol was added to the filtrate and the solution evaporated rapidly to dryness in a vacuum desiccator over phosphorus pentoxide.—B. Sullivan and M. Howe. J. Am. Chem. Soc., 59 (1937), 2742.

Glycerol in Fat and Phosphatides—Micro-Determination of. Lipins are dissolved in benzene, and a measured volume is transferred to a modified micro-Zeisel apparatus (described). Benzene is evaporated, and the fat hydrolyzed by heating at 120–125° with hydrogen iodide. The iodine derivative formed from the glycerol liberated is then titrated.—G. BLIX. Mikrochim. Acta, 1 (1937), 75–77; through J. Soc. Chem. Ind., 56 (1937), 1365. (E. G. V.)

Glycerol Samples—Electrometric Titration of Dichromate. Glycerol is oxidized with potassium dichromate. Excess dichromate is then titrated with ferrous sulfate solutions. A platinum anode and tungsten cathode, dipping into the solution, together with a galvanometer form the secondary circuit of a 1000-ohm potentiometer connected to four dry cells. The galvanometer reading is adjusted to zero before titration is started and the end-point is indicated by the swinging of the galvanometer to the side of the scale.—Chemical Division, Proctor and Gamble Co. Ind. Eng. Chem., Anal. Ed., 9 (1937), 514.

Gossypol—Extraction of, with Different Ethers. A peroxide-free ether containing 2.3 to 2.5% of alcohol (by weight) and 1 to 1.2% of water, having a density of 0.724 to 0.726 at 15.6° C., is necessary for the optimum extraction at 45° C. of gossypol from cottonseed meal containing ap-

proximately 22% of moisture and with 5 cc. of water added to the ether in the receiving flask.— J. O. HALVERSON and F. H. SMITH. *Ind. Eng. Chem., Anal. Ed.*, 9 (1937), 516. (E. G. V.)

Gossypol—Revised Method for the Estimation of, in Cottonseed Meal. A complete method for the extraction of gossypol with peroxide-free ether and for its precipitation as dianiline gossypol, which is weighed as such, is described.—F. H. SMITH. Ind. Eng. Chem., Anal. Ed., 9 (1937), 517. (E. G. V.)

Gums in Drugs—Identification of. When separated from drug mixtures certain gums fail to yield the reactions observed in pure gum suspensions in water. Microscopical examination of the gum after precipitation with alcohol gave promising results. The special microscope accessories found most useful were the polarizer and the dark field illuminator; reagents used were limited to 95% ethanol saturated with sodium chloride and zinc chloro-iodide solution. The technic followed is described in detail. Direct examination with dark field illumination shows two general types of precipitate: (1) definite stringy structure and (2) tiny noncrystalline particles of uniform size and consistency. Of the gums studied only agar and acacia fall in (2). Tragacanth, quince seed and Irish moss appear stringy. Examination in polarized light before and after treatment with zinc chloro-iodide reveals structural differences between tragacanth, Irish moss and quince seed which apparently can be made the basis of positive identification of these gums.—

J. H. Cannon. J. Assoc. Official Agr. Chem., 20 (1937), 588-589; through Chem. Abstr., 32 (1938), 307.

(F. J. S.)

Halogen—Determination of Alkyl and Aryl, in the Presence of Each Other. A simple and rapid method for the determination of alkyl and aryl halogen in the presence of each other has been developed. It appears to be applicable to a variety of problems and may be used on a micro, semimicro or macro scale. The procedure may be used qualitatively.—W. H. RAUSCHER. Ind. Eng. Chem., Anal. Ed., 9 (1937), 503. (E. G. V.)

Homatropine Tablets—Assay of. The A. O. A. C. tentative method for atropine in tablets, modified in a few minor details, is applicable to the determination of homatropine in most types of commercially available tablets of homatropine or its salts. If gelatin and (or) glycerol is present, as is the case in Brit. Pharm. (1932) lamella, the same method is used except that the chloroform extract is extracted with acid, the acid solution is made ammoniacal and extraction takes place as in the original method.—EDWARD M. HOSHALL. J. Assoc. Official Agr. Chem., 20 (1937), 599–602; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Honey for Extracting Active Constituents of Fresh Plant Materials. Materials such as sage or peppermint are disintegrated in admixture with honey (suitably in equal proportion with the plant material). A very small proportion of formic acid may be added in the case of peppermint, and the products may be dried or filled into starch capsules for use.—Gerhard Madaus, assignor to Madaus and Co. U. S. pat. 2,100,081, Nov. 23, 1937. (A. P.-C.)

8-Hydroxyquinoline—Bromatometric Determination of. Dissolve 20 to 40 mg. of 8-hydroxyquinoline in hydrochloric acid in a specially designed flask, add sodium hydroxide, 0.5 Gm. of potassium bromide and water to make about 50 cc. Add excess of decinormal potassium bromate and 10 cc. of concentrated hydrochloric acid, stopper the flask and let stand in the dark for 5 minutes; add potassium iodide solution, dilute with water and titrate excess iodine with decinormal sodium thiosulfate. 8-Hydroxyquinoline can be separated from drug mixtures either by distillation or by extraction. Its isolation in presence of phenacetin, dimethylaminoantipyrine, sulfosalicylic acid and hexamethylenetetramine, and its determination in ointments and suppositories are described in detail.—E. Schulek and O. Clauder. Z. Anal. Chem., 108 (1937), 385–396; through Chimie & Industrie, 38 (1937), 932.

Hypophosphites—Determination of, in Pharmaceutical Preparations. A small amount of collaborative work was done on the method of Jenkins and Bruening (Chem. Abstr., 30, 2702). It is concluded that the N. F. VI assay for ammonium hypophosphite is probably preferable to the method studied because of the longer reaction period allowed (3 hours instead of 2). The accuracy of the method studied depends on the composition of the product involved when an attempt is made to utilize the method on materials other than the simple salt or a mixture of hypophosphites only. The presence of other ingredients appears to have a pronounced effect on the rate of oxidation. Whether an increase in the time of reaction and (or) the amount of excess bromine is necessary must be determined before the practicability of the method may be assured. B. considers that, until the method studied is sufficiently modified, the present A. O. A. C. tentative

method for the determination of hypophosphites in the absence of phosphites may be generally applied with more accurate results in the assay of syrups of hypophosphites.—Henry R. Bond. J. Assoc. Official Agr. Chem., 20 (1937), 555-558; through Chem. Abstr., 32 (1938), 304.

(F. J. S.

Insecticides—Chemical, Scientific Progress in the Region of. The properties required in effective insecticides and modern methods of testing them are described.—L. Sprengel. Angew. Chem., 50 (1937), 560-569; through J. Soc. Chem. Ind., 56 (1937), 959. (E. G. V.)

Insecticides—Liquid, Official Method for Evaluating. The 1937 Official Method of the National Association of Insecticide and Disinfectant Manufactures (December 1936) for evaluating household fly-sprays by comparison with the official control insecticide in paired Peet-Grady tests is detailed and explained. Fairly concordant results (agreeing qualitatively at least) have been obtained in collaborate tests of an unknown sample by the method.—W. A. SIMANTON. Soap, 13 (1937), 103–107, 115; through J. Soc. Chem. Ind., 56 (1937), 1281. (E. G. V.)

Iodates—Improved Method for the Determination of. The method depends on the titration of an iodate with thiosulfate, after having added an iodide. A weight burette and special titration cell are used; illumination of the latter allows the color of the free iodine to serve as indicator.—V. J. Anhorn and H. Hunt. Ind. Eng. Chem., Anal. Ed., 9 (1937), 591.

(E. G. V.)

Iodine—Determination of, in Extracts from Brine. Iodine liberated with nitrite from the acidified brine is extracted with activated carbon, ground, extracted with sodium sulfite, filtered, made up to a definite volume and titrated (eosin) with silver nitrate in presence of ammonium carbonate. Iodine is determined in waste liquors by oxidizing to iodate with bromine, removing the excess of bromine with phenol, and titrating iodine liberated from adding potassium iodide with 0.001 N-sodium thiosulfate. Chlorine is determined by boiling with nitric acid and titrating by the Volhard method. The acids in the crude iodine are extracted with light petroleum, shaken with standard alcoholic sodium hydroxide and the excess of alkali is titrated.—I. Orlov and T. Kaganova. Chim. Farm. Prom., No. 1 (1935), 44; through J. Soc. Chem. Ind., 56 (1937), B., 778.

Iodine Ointment-Assay of. Free iodine distillation and subsequent titration of the halogen gives low results. Direct titration with 0.1N sodium thiosulfate or a potassium arsenite (containing sufficient potassium bicarbonate to neutralize the hydrogen iodide formed) in presence of the base and sufficient chloroform to dissolve the base gave fairly satisfactory results. Potassium arsenite is considered rather more satisfactory than sodium thiosulfate as it may be used directly as primary standard. Potassium Iodide.—The following four methods were studied: (a) Volatilization of free iodine by boiling, liberation of combined iodine by addition of sulfuric acid and ferric ammonium sulfate, distillation and titration of iodine; (b) distillation without previous volatilization, titration of iodine and calculation of potassium iodide by difference; (c) removal of uncombined iodine and potassium iodide from the base by repeated washing, volatilization of free iodine and titration of iodide by the Volhard method; (4) similar to (c) except that the iodide is titrated with 0.05M potassium iodate. The technic of (c) is described in detail. Both (c) and (d) yielded results that were in good agreement, but about 5% high, which was shown to be due to reduction of some of the free iodine to hydrogen iodine by constituents of the base. Organically Combined Iodine.—Free iodine and potassium iodide are removed by repeated washing and iodine is determined in the separated base by the U. S. P. XI method for total iodine in iodine ointment. The amount of iodine absorbed by the base is small, but increases with the age of the ointment.—W. F. REINDOLLAR. J. Assoc. Official Agr. Chem., 20 (1937), 572-574; through Chem. Abstr., 32 (1938), 306.

Iodine Tincture—Assay of, According to the Supplement to the German Pharmacopæia VI. Various methods are reviewed and it is concluded that the method of determination outlined by the Supplement with slight changes is satisfactory for the manufacturer and the pharmacist.—Paul Runge. Apoth. Ztg., 52 (1937), 1591-1592. (H. M. B.)

Iodine and Iodides—Microdetermination of. Iodine oxidizes an excess of hydroxylamine in sulfanilic acid to form a diazonium salt. The salt is coupled with α -naphthylamine to produce a red dye, which can be estimated with the Pulfrich photometer. Iodides are first oxidized to iodate with bromine water, and iodine is liberated by addition of potassium iodide and distilled. The iodine in the distillate is determined as above. Amounts of 5γ of iodine or 1γ of iodide are de-

termined with an accuracy of 2%.—G. Endres and L. Kaufmann. Hoppe-Seyler's Z. Physiol. Chem., 243 (1936), 144-148; through Chimie & Industrie, 38 (1937), 864. (A. P.-C.)

Iron—Rapid Determination of, in Pharmaceuticals with the Aid of Cupferron and an Immiscible Solvent. The technic of a new method is described, which consists essentially in treating the iron solution with cupferron, extracting with chloroform, evaporating the extract to dryness, igniting to ferric oxide and weighing; or determining iron volumetrically by the Zimmerman-Reinhardt method as defined by Jones and Jeffery (Chem. Abstr., 3, 2660). Comparative assays carried out on 12 common pharmaceutical preparations by the present and a standard method showed the former to be satisfactory.—S. M. Berman, J. J. Chap and D. M. Taylor. J. Assoc. Official Agr. Chem., 20 (1937), 635-638; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Lead—Colorimetric Determination of. By discarding the treatment of the extract of lead diphenyl thiocarbazone with acid as used in the method of H. Fischer (Angew. Chem., 47 (1934), 90-92) a pink solution is obtained which can be compared with the colored standard scale for the determination of lead. Experimental details are given and data are tabulated.—A. IEVLANOVA. J. Prikl. Khim., 9 (1936), 1690-1696; through Chimie & Industrie, 38 (1937), 865. (A. P.-C.)

Lead—Determination of, with 8-Hydroxyquinoline. The determination of 0.04-0.1 Gm. of lead with oxine usually gives values which are about 1% low. If precipitation takes place from a hot solution the results are a little lower than when the precipitation takes place in the cold. In the latter case, filtration is more difficult and there is detectable adsorption of other ions. For the precipitation, 2.5 moles of oxine are sufficient for one atom of lead. Make the solution slightly acid with acetic acid, add the reagent in the form of a solution saturated at room temperature, and then add ammonium hydroxide until the solution has a slight odor of ammonia. After two hours filter off the oxine precipitate, wash with half-saturated aqueous oxine solution, then with cold water, dry at 105° and weigh. The solubility of the precipitate in ethanol, 10% ammonia, 2.5% ammonia, ammoniacal solution of oxine and half-saturated solution of oxine decreases in the order named. The procedure can be applied to the determination of lead in insoluble precipitates which can be dissolved in hot ammonium acetate solution and then treated as above.—V. Hovorka. Collection Czechoslov. Chem. Commun., 9, 191-206; through Chem. Abstr., 31 (1937), 6997.

(F. J. S.)

Lead—Titrimetric Method for the Quantitative Estimation of, in Biological Materials. All the reagents used in the determination were especially purified. Distilled water, hydrochloric acid, nitric acid and ammonium hydroxide were redistilled in glass stills. Ten cubic centimeters of blood in a fifty-cc. silica dish are dried and charred beneath a radiant heater, then ashed 2 hours at 475°, moistened with 2 cc. nitric acid and placed beneath a radiant heater until all action ceases, then returned to the muffle for 30 minutes. The ash is then dissolved in 15 cc. 20% hydrochloric acid and washed into a 125-cc. separatory funnel with 20 cc. of hot water; 10 cc. 20% sodium citrate and 3 cc. ammonium hydroxide are then added to the silica dish, and this solution is transferred to the separatory funnel with the aid of enough water to make a total volume of 75 cc. One cubic centimeter 25% hydroxylamine hydrochloride and one drop of phenol red indicator are added, and ammonium hydroxide is added from a burette to attain a ph of 8.0. Five-tenths cubic centimeter 10% potassium cyanide is then added drop by drop (with shaking), and the solution immediately extracted with 0.5 cc. dithizone solution (10 mg. in 100 cc. chloroform) and 4 cc. chloroform. If the chloroform layer does not contain excess uncombined dithizone, 0.2-cc. portions are added until an excess is present, after shaking. Extraction of the aqueous phase is repeated twice with 0.2-cc. portions of dithizone and 2 cc. chloroform. The excess dithizone used is then removed by two extractions of the combined chloroform layers with 1.5 volumes of 0.5% of potassium cyanide, any lead which may have entered the aqueous phase is extracted with 2 cc. chloroform, which is added to the main chloroform layer. The lead is then separated from the dithizone complex by shaking for 15 seconds with two volumes of 0.5% hydrochloric acid. The acid layer is then extracted with 1 cc. chloroform and the chloroform fractions are combined. Fivetenths volume of 0.5% potassium cyanide solution is added to the combined chloroform extract of dithizone, which is then titrated with standard lead nitrate (0.01 mg. lead per cc.) until only a faint color remains in the aqueous phase. The red chloroform phase is then discarded and the aqueous layer washed with 2-cc. portions of chloroform until the chloroform layer remains colorless after shaking. Extraction of the aqueous layer with 2 cc. chloroform to which 1-2 drops of lead solution have been added is then continued until further addition of lead gives no pink color

to the chloroform. The end-point is a slight pink in the chloroform solution. Addition of one more drop results in a colorless solution. Modifications for the analysis of urine and bone are described. In the analysis of foodstuffs, the procedure is adapted to the nature of the sample.—

M. K. HORWITT and GEORGE R. COWGILL. J. Biol. Chem., 119 (1937), 553-564; through Chem. Abstr., 31 (1937), 7081.

(F. J. S.)

Licorice Root—Saccharine Constituents of. The formula of glycyrrhetinic acid has been established as the aglucone of glycyrrhizine, C₈₀H₄₈O₄, differing from that of Ruzicka and Leuenberger which has 4 hydrogen atoms less. This acid is obtained by boiling potassium glycyrrhizinate with 10% sulfuric acid. It melts at 287° to 292° C.; has iodine number 0; reduces permanganate and bromine very slowly; yields a monoacetate that melts at 319° to 321° C., and unsaponifiable esters. By dehydrogenation with selenium it gives chiefly 1,2,7-trimethylnaphthalene and 2,7-dimethylnaphthalene.—T. Kariyone and O. Nonaka. J. Pharm. Soc. Japan, 57 (1937), No. 2, 20-24; through Chimie & Industrie, 38 (1937), 929. (A. P.-C.)

Lupines-Alkaloidal Content of, New Method for Determination of. The inadequacies of previous methods of determination of the alkaloid content of the lupines are pointed out, and a new method is proposed. Since the two alkaloids, sparteine (I) and lupinine (II) found in yellow lupines differ markedly in properties, they are determined separately. For the determination, the drug is ground with sodium hydroxide and gypsum and the alkaloids are extracted with etherchloroform and transferred to aqueous hydrochloric acid solution. Both alkaloids are precipitated with 10% silicotungstic acid (III), the precipitates are dissolved in sodium hydroxide, and the alkaloids are extracted with chloroform and transferred to hydrochloric acid solution. I is then precipitated with acetone-picrolonic acid solution after exact neutralization. The I content equals the weight of picrolonate times 0.3072. The II is freed from the filtrate with ammonium hydroxide, extracted with chloroform, transferred to hydrochloric acid solution and precipitated with III. The II content equals the weight of precipitate times 0.2376. Modifications are given for bitter and sweet lupines. The I, II and total alkaloid contents are given for various varieties of lupine.—Z. Wierzchowski. Biochem. Z., 293 (1937), 192; through Squibb Abstr. Bull., 10 (1937), A-2015. (F. J. S.)

Magnesium Acetylsalicylate in Headache Powders. Three powders, Stellapirol (1), Heacyl (2) and Velo-powder (3) stated to contain this compound were examined. (1) was a mixture of acetylsalicylic acid and magnesium compounds, (2) mostly magnesium salicylate and small amounts of free acetylsalicylic acid and (3) contained only acetylsalicylic acid.—C. Dultz. Apoth. Ztg., 52 (1937), 1453-1454. (H. M. B.)

Manganese—Determination of Small Quantities of, in Organic Products. Manganese is determined colorimetrically in feces as follows: destroy organic matter by incineration in a muffle at dull red heat; treat the ash with hydrochloric acid on the water-bath till decolorized, add sulfuric acid and evaporate to white fumes to remove hydrochloric acid; oxidize with concentrated nitric acid in presence of silver nitrate and ammonium sulfate; heat on the water-bath till the color has been completely developed, make to definite volume and filter. Prepare a standard solution by dissolving 0.2877 Gm. of potassium permanganate in 100 cc. of water, acidifying with sulfuric acid, decolorizing with oxalic acid and making to 1 liter; 1 cc. of 1 mg. of manganese. For each determination a known volume of the standard solution is oxidized in the same way as the sample and the colored solutions are compared in a Leitz colorimeter or by means of a Pulfrich photometer.—M. M. Retortillo and J. D. Gallego. Rev. Sanidad, 11 (1936), 85–103; through Chimie & Industrie, 38 (1937), 38.

Medicaments—Use of Drop Reactions for Investigation of. I. Aldehyde and Amine Reactions for Identification of Essential Oils. Color reactions of numerous aldehydes and natural oils on spotting with a solution of O-dianisidine (I) in acetic acid are described. Amines may be detected by their reaction with furfuraldehyde, which gives a highly sensitive violet coloration with I and other amines, and especially with derivatives of anthranilic acid which can occur in essential oils.—R. Wasicky and O. Frehden. Mikrochim. Acta, 1 (1937), 55-63; through J. Soc. Chem. Ind., 56 (1937), 1407. (E. G. V.)

Menthol, Menthone and Menthol Esters—Micro-Determination of, in Oil of Mint. The oil is isolated in a micro-Clevenger apparatus (a 2-Gm. sample is sufficient). The menthone is determined by the use of hydroxylamine, and menthol plus menthone by oxidation of the former with chromic-sulfuric acid and determined as menthone. Esters are determined by saponifica-

tion and titration of residual sodium hydroxide.—H. ULLRICH and M. SCHNEIDER. Hoppe-Seyler's Z. Physiol. Chem., 245 (1937), 181-184; through Chimie & Industrie, 38 (1937), 323.

(A. P.-C.)

Methanol and Ethanol—Analysis of Mixtures of. The procedure described by the author is as follows: To about 20 cc. of the solution to be analyzed, containing 0 to 5×10^{-4} Gm. mol. of alcohols, is added a measured excess of N/5 dichromate solution (e.g., 15 cc.) and 10 cc. of 10%sulfuric acid. The mixture is heated in a pressure-bottle in a steam-bath for about 25 minutes and then allowed to cool. The excess of dichromate is determined by adding a weighed amount of ferrous sulfate and then titrating back with dichromate, an external indicator being used. The amount of dichromate used in this oxidation is four equivalents per mol. of alcohol (either methyl or ethyl; the presence of propanol introduces an error, as it requires about five equivalents per mol.). If insufficient dichromate was used, a further quantity may be added and the heating repeated. After this oxidation the solution contains formic and acetic acids from the alcohols. formic acid is oxidized in a subsequent operation, a further quantity of dichromate being added (this need not be more than half the previous amount) and 10 cc. of concentrated sulfuric acid. The mixture is refluxed for about 15 minutes, and then allowed to cool. After dilution and partial neutralization of the acid the excess of dichromate is determined by adding potassium iodide and titrating the liberated iodine. The methyl alcohol orginally present now required two more equivalents of oxygen per mol. If desired, the two oxidations may be carried out on separate aliquot parts of the mixture; the second portion should then receive 3/2 times as much dichromate as the first. The excess of dichromate after both oxidations may be determined iodimetrically, thus avoiding the inconvenience of the iron titration.—E. J. HARRIS. Analyst, 62 (1937), 729.

G. L. W.)

Microgasometric Analysis with the Dilatometer. The dilatometer consists of a graduated horizontal tube of small bore, closed by a droplet of mercury which is free to move until the internal and external pressures are equalized. This movement, over the graduated scale, indicates the sense and the magnitude of the volume change. Applied to micromethods, such an arrangement possesses particular merit. Its construction is simple, permitting a reduction of the apparatus capacity to a point commensurate with the small volumes dealt with. Errors attendant upon measure of the evolved gas by liquid displacement are not present in the dilatometer method. The authors have applied such a dilatometer to the determination of carbon dioxide in the samples of corrosion product weighing only a few milligrams.—B. I., Clarke and H. W. Hermance. Ind. Eng. Chem., Anal. Ed., 9 (1937), 597. (E. G. V.)

Moisture Content of Dried Fruit—Changes of, during Storage. A procedure for the determination of moisture content of dried fruits is described. A description is given of the inexpensive form of vacuum oven that is used. Results are given showing the equilibrium values of moisture content of the fruit and atmospheric humidity.—W. B. Brown. J. Soc. Chem. Ind., 57 (1938), 31. (E. G. V.)

Morphine—Accuracy of the Determination of, in Opium. The various methods in use are discussed and compared. It is concluded that none of the methods are accurate because the quantities of morphine which are lost vary with the quality of the opium. The error, however, remains below the limits permissible in pharmacy.—E. Léger. Bull. sci. pharmacol., 44 (1937), 214–223; through Chimie & Industrie, 38 (1937), 932. (A. P.-C.)

Nicotine—Determination of, in Tobacco Smoke. A simplified method for the determination of nicotine in tobacco smoke is described.—ADOLF WENUSCH. Z. Untersuch. Lebensm., 74 (1937), 46-51; through Chem. Abstr., 31 (1937), 7192. (F. J. S.)

Nitrogen in Potato Tubers—Use of Selenium in the Determination of. Twenty minutes boiling after the mixture has cleared was found necessary for good results. The total time required for the digestion of one Gm. of dried tuber was 30 minutes. The optimum quantity of catalyst was 0.3 Gm. of selenium in conjunction with 9.7 Gm. of potassium sulfate and 25 cc. of concentrated sulfuric acid.—A. M. Smith and W. Y. Patterson. Analyst, 62 (1937), 786.

(G. L. W.)

Nitroglycerin—Report on, in Mixtures. The present report covers work on the determination of nitroglycerin in the presence of caffeine, reduced iron, and tinctures of digitalis, nux vomica, belladonna, hyoscyamus and strophanthus, each singly and in various combinations. In each case the amount used was equal to or greater than the amount that would be present in a sample of the complex tablets. In the case of tinctures the alcohol was not evaporated because it does not interfere with the determination. The acid distillation method was applied and gave promising results in most cases. Reduced iron in the presence of the tinctures gave high results. It is recommended that the work be continued.—OMAR C. KENWORTHY. J. Assoc. Official Agr. Chem., 20 (1937), 569; through Squibb Abstr. Bull., 10 (1937), A-2079. (F. J. S.)

Orpiment—Determination of, in Shellac. Triplicate portions (0.5-1.0 Gm.) of shellac in a No. 100 powder are weighed into the three digestion flasks of a specially designed apparatus (pictured) and a wet combustion with arsenic free sulfuric acid (2-3 cc.) and nitric acid is completed. The residual nitric acid is expelled by boiling and the resulting solution of arsenic acid diluted to 50 cc. with water is reduced with a crystal of potassium iodide. The iodine is boiled off until the solution is pale yellow, the solution diluted to 50 cc. again and the remaining iodine decolorized with N/100 sodium thiosulfate solution (starch indicator). The solution is immediately neutralized with sodium bicarbonate and an excess of 2-3 Gm. of the latter added. The mixture is titrated with N/100 iodine solution (starch indicator). For samples containing less than 0.1%, the following modification is recommended: Five grams of the representative sample are dissolved in 100 to 125 cc. of 95% alcohol on a water-bath, and the solution is filtered through a filter-paper in a hot-water funnel, the stem of which is fitted into a Büchner funnel connected with a filter pump. The filter-paper, containing the orpiment and other impurities, is folded, dried and introduced into the digestion flask of the apparatus for further treatment as described above. M. RANGASWAMI and H. K. SEN. Analyst, 63 (1937), 36. (G. L. W.)

Oxygen in Organic Substances—Direct Microdetermination of, by Hydrogenation. A micromethod, based on ter Meulen's hydrogenation procedure, has developed for the direct determination of oxygen in volatile organic compounds containing only carbon, hydrogen and oxygen. A study was also made of the blank determination involved in this method. It was found that, in the analysis of pure compounds which distil or sublime, results can be obtained which agree with theory to within $\pm 0.1\%$, provided a more or less empirical blank value is deducted. If the manipulative blank value is deducted the results all tend to be low, which indicates that a hidden compensative error is involved in the method. The same technic when applied to sucrose, which leaves a voluminous deposit of carbon in the boat, has so far yielded results which are consistently low.—W. R. Kirner. Ind. Eng. Chem., Anal. Ed., 9 (1937), 535. (E. G. V.)

 $p_{\rm H}$ of Sulfonated Oils—Determination of. The $p_{\rm H}$ of an emulsion of a sulfonated oil, determined electrometrically, is affected by the mode of preparation of the emulsion, but not by the temperature or time of keeping. The degree of dispersion and the $p_{\rm H}$ are influenced by addition of small amounts of potassium chloride to concentrated emulsions of the oil. The $p_{\rm H}$ values determined by the quinhydrone electrode are in agreement with those given by the glass electrode, and those by the colorimeter are 0.5 less than the latter; this difference is attributed to "salt error." The $p_{\rm H}$ values obtained electrometrically on different concentrated emulsions prepared in the same way indicate their comparative "relative activities."—G. Parsy. J. Soc. Leather Trades Chem., 21 (1937), 261–274; through J. Soc. Chem. Ind., 56 (1937), 942. (E. G. V.)

Phenolphthalein and Acetylsalicylic Acid—Determination of. The following method gave good preliminary results: Mix 1 Gm. of the material with saturated sodium bicarbonate solution, extract the phenolphthalein with 2:1 chloroform-ether mixture, acidify the sodium bicarbonate solution and extract the acetylsalicylic acid with chloroform; determine phenolphthalein by weight and as the iodo compound, and the acetylsalicylic acid by weight and by double titration. Collaborative study of the method gave disappointing results and it will be studied further.—Geo. M. Johnson. J. Assoc. Official Agr. Chem., 20 (1937), 598-599; through Chem. Abstr., 32 (1938), 307. (F. J. S.)

Phenols—Sensitivity of Certain Tests for. Of phosphomolybdic acid, phosphotungstic acid, Millon's reagent and sodium nitroprusside, the first is the most sensitive color reagent for phenols; in the presence of ammonia it can detect 1 part of phenol, of hydroquinone or of cresol in 2,000,000. Compounds of mixed function (adrenaline, vanillin, isoeugenol, guaiacol, cresols), as well as α- and β-naphthol and thymol, give color reactions with phosphomolybdic acid in the presence of ammonia; these compounds do not give a color with phosphotungstic acid. Millon's reagent and sodium nitroprusside give colors only with certain phenols; they do not give colors with the above-mentioned compounds of mixed function.—V. M. PLATKOVSKAIA and S. G. VATKINA. J. Prikl. Khim., 10 (1937), 202–207; through Chimie & Industrie, 38 (1937), 867. (A. P.-C.)

Phosphatides. The occurrence, properties and commercial uses of phosphatides are described. Methods of determination by the use of solvents and lecithin-kephalin separations are given.—B. REWALD. Proc. 5th Intern. Cong. Tech. Chem. Agric. Ind., Holland, 1 (1937), 400-408; through J. Soc. Chem. Ind., 56 (1937), 976. (E. G. V.)

Phosphorus—Photometric Determination of Added, in Oils. A reliable photometric method for the determination of phosphorus, particularly adapted for the control of small amounts of added phosphorus in oils, is described. An accuracy of 0.001% has been demonstrated. Less than 1 hour is required for a single complete analysis.—P. Goodloe. Ind. Eng. Chem., Anal. Ed., 9 (1937), 527. (E. G. V.)

Piscidiæ Erythrinæ—New Constituents of. From a petroleum-ether extract of the root of Piscidiæ erythrinæ a stearin was isolated. After the separation of the stearin an oily substance separates out. This substance is extracted with petroleum-ether; a yellowish amorphous optically inactive powder is obtained, m. p. 72° Upon saponifying this powder with alcoholic alkali small crystals, m. p. 78-79° separate. Saponification lessens the activity of the compound.—F. Hauschld. Chem. Zentr., 108 (1937), 108. (G. B.)

Polybromides—Determination of, in Fats and Oils. A critical experimental study is made of the sources of error in the determination of polybromides in fats and oils, as a result of which the following technic is recommended. Saponify 5 Gm. of the oil by refluxing 30 minutes with 25 cc. of alcoholic potash shaking at the beginning to avoid superheating of the oil; add 50 cc. of water, transfer to a separatory funnel using an additional 80 cc. of water for washing, add 50 cc. of ether and decompose the soap with a slight excess of normal hydrochloric acid using helianthin as indicator; shake vigorously, separate the ether extract, re-extract with 50 cc. of ether, wash each extract with 3 times 50 cc. of 10% sodium chloride solution and make the combined extracts to exactly 100 cc. with ether; dehydrate with anhydrous sodium sulfate; if bromination is not carried out immediately, displace the air in the flask with carbon dioxide and keep the flask away from the light. In a tared 80-cc. centrifuge tube containing about 0.1 Gm, of finely powdered polybromides (preferably obtained from the same or a similar oil) place 20 cc. (accurately measured) of the ether solution of the fatty acids, shake vigorously, place in melting ice for about 15 minutes, add in small successive portions 20 cc. of a 4% solution of bromine in ether which has been cooled to 0° C.; the bromine solution is delivered from a burette surrounded by ice and the fatty acids solution is stirred with a thermometer so that it may be cooled to 0° C. whenever the temperature rises to 1° C.; at the same time run a blank on 0.1 Gm. of polybromides and 40 cc. of ether. After allowing the tubes to stand in ice for 3 hours, centrifuge for 1 minute at 3000 to 4000 r. p. m.; decant the clear liquid; add 20 cc. of ether saturated with polybromides at 0° C., shake thoroughly, cool to 1° to 2° C, and centrifuge, decant and repeat the washing with 20, 10 and 10 cc. of ether saturated with polybromides. After the final decantation place the two tubes in an oven, gradually raise the temperature to 100° C., hold at this temperature for 30 minutes, cool and weigh. The gain in weight of the tube containing the sample plus the loss in weight of the tube containing the blank gives the weight of polybromides per Gm. of oil; the polybromide value may be expressed per Gm. of oil or calculated to the fatty acids basis. Experimental data are presented in justification of this technic.—Vizern and Guillot. Ann. fals., 30 (1937), 329-339. (A. P.-C.)

Pycnometric Analysis—New Method of. A simple and rapid method of pycnometric analysis is provided, in which a purified precipitate is quantitatively transfered to a small pycnometer and weighed in the presence of a liquid of known density. Analysis involving such physically different precipitates as silver chloride, barium sulfate and ferric hydroxide indicates that the method is capable of good accuracy.—W. W. Russell. Ind. Eng. Chem., Anal. Ed., 9 (1937), 592.

(E. G. V.)

Qualitative Separations on a Micro Scale. III. Analysis of the Selenium Group of Noyes and Bray. A procedure for the qualitative microanalysis of the selenium group of Noyes and Bray has been carried out, permitting the isolation and approximate estimation of selenium, germanium and arsenic when starting with a solid sample of 1 mg. weight. Five micrograms of any of the three elements can safely be detected, even when accompanied by a hundredfold excess of the other two elements of this group. A screw clamp for microcones enables the simple performance of pressure digestions. A buzzer is used to accelerate the formation of precipitates.—A. A. BENEDETTI-PICCHLER and J. R. RACHELLE. Ind. Eng. Chem., Anal. Ed., 9 (1937), 589. (E. G. V.)

Quillaja—New Constituents of. The sapogenin which was obtained from the root of Quillaja saponaria was named gypsogenin and had the formula $C_{28}H_{44}O_{5}$ (1). Although the dehydrated products which are obtained from 1 are similar to those obtained from other sapogenins, yet the formula in 1 was well established by the authors as being the correct formula. In purifying the compound through fractional crystallization, several new compounds were obtained; these were acetylated and two acetate isomers separated out. The formula of the soluble acetate (acetylgypsogenin) was established after the compound had been titrated. In the same manner the following compounds were also obtained: methylesters, bromlactons and semicarbazons. The replacement of the semicarbazone with sodium ethylate (at a temperature of 160°) and saponification, made possible the formation of oleanolic acid.—L. Ruzicka and G. Giacomello. Chem. Zentr., 108 (1937), 616. (G. B.)

Quinic Acid in Admixture with Shikimic Acid—Detection of, in the Capsules of Illicium Verum Hooker, and Preparation of Quinic Acid Derivatives. Quinic acid can be identified as its triacetyl lactone in the presence of shikimic acid. Quinic acid was found in the capsules of Illicium verum. Quinic acid lactone can be obtained in a yield of 75% by heating quinic acid in acetylene tetrachloride. The triacetyl lactone was prepared in a 95% yield by heating quinic acid in freshly distilled acetic anhydride. As a further derivative of quinic acid the hitherto unknown triacetyl quinic acid was prepared and described.—Anne-Marie Boldt. Pharm. Zentralhalle, 78 (1937), 157-166; through Chimie & Industrie, 38 (1937), 929-930. (A. P.-C.)

Resins and Essential Oils—Analysis of. The composition of resin acid mixtures 1-(I) and d-pimaric acid (II) (but no abietic acid) can be derived from observations of the change in $[\alpha]_D$ which accompanies isomerization of the acids by ethereal hydrochloric or acetic acid. When abietic acid is present, I may be determined by applying Kaufmann's method for obtaining the diene value since it had been found that I combines with maleic anhydride at room temperature (or at 80° in the test), whereas II, pro-abietic and abietic acids react only at temperatures not less than 135° (each yielding the same crystalline product, melting point 227°); pyroabietic acid (III) does not react at all. By a combination of the optical and chemical methods, mixtures containing all four acids can be analyzed. III in colophony may be determined by performing the diene test at 160° (for example, in xylene under pressure), but some error is introduced if reactive rosin oil is present. The diene method can be applied also to the analysis of ethereal oils; thus α -phellandrene reacts in solution at temperature less than 100°, while at 135° β -phellandrene reacts also (yielding an amorphous derivative). The diene values of a number of ethereal oils and balsams are given.—W. Sandermann. Seifens.-Ztg., 64 (1937), 402-403, 421-422; through J. Soc. Chem. Ind., 56 (1937), 1370. (E. G. V.)

Rosin in Soaps—Determination of. Separate the total fatty matter from about 5 Gm. of soap by dissolving in hot water, acidify with dilute sulfuric acid, cool and wash the cake of fatty matter with water until the aqueous washings are free from acid. Weigh about 2 Gm. of the total fatty matter into a 150-cc. flask. Dissolve it in 20 cc. of naphthalene-2-sulfonic acid solution (4% w/v in anhydrous methanol) and boil under a reflux for 30 minutes. A blank determination is carried out simultaneously. Cool and titrate both with N/5 alcoholic potassium hydroxide using 0.5 cc. of 0.5% solution of phenolphthalein as indicator. The difference between the two titrations is a measure of the resin acids. One cubic centimeter of N/5 alcoholic potassium hydroxide is equivalent to 0.0652 Gm. of resin acids. Calculate the resin acids as a % of total fatty matter and subtract 1% from the result so obtained. For the purpose of calculating the approximate proportion of rosin in soap it may be assumed that rosin contains 92% of resin acids.—Report No. 5, Subcommittee on Methods of Soap Analysis, Analytical Methods Committee. Analysi, 62 (1937), 868.

Rubber—Natural and Synthetic. XVIII. The Protein from Natural Rubber and Its Amino Acid Constituents. The object of this work was to isolate a product which would be as nearly pure natural rubber protein as possible, to analyze it and to separate and identify the individual amino acids resulting from its hydrolysis.—T. MIDGLEY, A. L. HENNE and M. W. RENOLL. J. Am. Chem. Soc., 59 (1937), 2501. (E. B. S.)

Santonin, Phenolphthalein and Calomel—Determination of, in Tablets. A collaborative study was made on the following method: Weigh 4 Gm. of powdered material into a Caldwell crucible and wash with about 200 cc. of hot alcohol, collecting the filtrate (I) in a 250-cc. volumetric flask, using a bell-jar and suction and make to volume at 20°; determine santonin in a 25-cc. ali-

quot of I by the tentative A. O. A. C. method (A. O. A. C. Methods of Analysis (1935), 570); evaporate a 50-cc. aliquot of I to dryness and determine phenolphalein by the A. O. A. C. method (A. O. A. C. Method of Analysis (1935), 570); wash the residue in the Caldwell crucible with three 15-cc. portions of cold water, using suction and determine calomel in the residue by the A. O. A. C. method (A. O. A. C. Methods of Analysis (1935), 595). The method gives excellent results for santonin and calomel; phenolphthalein does not interfere in the determination of santonin as the dinitrophenylhydrazone. Most analysts obtained high results for phenolphthalein. The precipitation of phenolphthalein as the tetraiodo compound does not effect a sharp separation from santonin.—Harry J. Fisher. J. Assoc. Official Agr. Chem., 20 (1937), 558-560; through Chem. Abstr., 32 (1938), 305. (F. J. S.)

Shellac—Iodine Value of. Iodine values on a sample of shellac were found to vary with the concentration of the acetic acid used in the Wijs-Langmuir method. Acetic acids of melting points 11.8°, 14.95° and 16.4° C. gave solutions which resulted in iodine values on the same shellac of 14.67, 17.59 and 20.48, respectively. Reference is made to the satisfactory specification of the A. S. T. M. (1930), 299, which requires a melting point of the acetic acid used to be 14.8° C.—R. W. Aldis. Analyst, 62 (1937), 792. (G. L. W.)

Silver—Determination of, in Colloidal Silver Ointments. Destruction of fatty material by calcination as described in the Estonian and Swiss Pharmacopæias is too tedious. Addition of 20 Gm. of dilute (1:1) nitric acid to 1 Gm. of ointment and heating for 30 minutes on a waterbath gives quicker results. Then 20 cc. of water is added and the mixture heated for five minutes until the fat seps. After thorough cooling the liquid is filtered through cotton and the fatty layer washed with small amounts of water, the washings are filtered and added to the rest to make 100 cc. To oxidize any nitrous acid formed during the process and to prevent it from reacting later with the thiocyanate, 0.1N potassium permanganate is added, drop by drop, to a slight excess, and the pink coloring decolorized by adding a few crystals of ferrous sulfate. Then 5 cc. of ferric ammonium sulfate is added and the solution titrated with 0.1N ammonium thiocyanate. The colloidal silver dissolved as nitrate; any silver chloride present (up to 3%) does not interfere with the titration. The amounts detected by this method agree closely with those obtained gravimetrically and by the Pharmacopæial Method.—August Sim. Pharmacia, 17 (1937), 251-253 (in Estonian); through Chem. Abstr., 32 (1938), 303.

Silver Nitrate—Reduction of, by Arsine, and Its Use for Estimating Minimum Amounts of **Arsenic.** Numerous experiments were performed with solutions known to contain 0.1 to 1.0 mg. of dissolved arsenic trioxide. The trivalent arsenic was reduced by pure zinc and sulfuric acid. The liberated arsine was absorbed in either neutral or ammoniacal silver nitrate, the resulting precipitate of silver was filtered off, dissolved in nitric acid and the silver titrated with ammonium thiocyanate by the Volhard method or determined in the nephelometer with sodium chloride as the precipitant. It has often been assumed that when arsine is absorbed in silver nitrate the arsenic is converted to arsenite and an equivalent quantity of silver is precipitated or, in other words, that 1 arsenic = 6 silver, corresponding to the oxidation of arsenic with a negative valence of three to a positive valence of three. The results of at least fifty experiments prove that this assumption is not quite true, because a little silver is reduced by the stream of hydrogen and this quantity is smaller when an ammoniacal silver solution is used than with neutral silver nitrate solution. The silver precipitate occludes some arsenic which causes error in the opposite direction. Twenty-nine references.-J. H. KREPELKA and J. FANTA. Collection, Czechoslov. Chem. Commun., 9, 47-67; through Chem. Abstr., 31 (1937), 6995. (F. J. S.)

Sodium Thiosulfate—Stability of Solid. Sodium thiosulfate decomposes slowly in the solid state to sulfur and to some substances giving the analytical test for sodium sulfite. The deterioration of solutions of freshly recrystallized thiosulfate is not more rapid than that of solutions prepared from crystals not recrystallized recently.—V. K. LA MER and H. M. TOMLINSON. Ind. Eng. Chem., Anal. Ed., 9 (1937), 588. (E. G. V.)

Solanum Nigrum L.—Chemical Examination of the Fruits of. I. The Composition of the Oil from the Seeds. An account of the physical and chemical properties of the fixed oil from the seeds of Solanum nigrum Solanaceæ). The berries of this plant are used in Hindu medicine as a tonic, in heart diseases, in fevers, diarrhea, eye diseases and chronic enlargement of the liver.—G. P. Pendse. J. Indian Chem. Soc., 14 (1937), 367; through Squibb Abstr. Bull., 10 (1937), A-1895.

(F. J. S.)

Solvent Power—Kauri Butanol Test for. II. A microprocedure for the determination of kauri butanol solvent power has been developed and the solvent powers of a number of hydrocarbons have been determined. A method for correlating the solvent power results with the structure of the hydrocarbons has been worked out which provides an additional tool for the analysis of hydrocarbon mixtures.—E. L. Baldeschwieler, M. D. Morgan and W. J. Trobller. Ind. Eng. Chem., Anal. Ed., 9 (1937), 540. (E. G. V.)

Spirit of Camphor—Assay of. The present U. S. P. XI assay for spirit of camphor is both inaccurate and time consuming. A modified method based on the procedure of Randall (Chem. Abstr., 18, 2408) is described in detail; it yields results within ±2% of the theoretical value and good agreement is readily obtained in duplicate determinations.—Samuel W. Goldstein and Wm. F. Reindollar. J. Assoc. Official Agr. Chem., 20 (1937), 887-889; through Chem. Abstr., 32 (1938), 308. (F. J. S.)

Spirits of Turpentine—Composition of Some, from the Dutch East Indies. Spirits of turpentine from three varieties of pines of the Dutch East Indies were analyzed by Darmois' polarimetric method and by determination of the Raman spectra. *Pinus insularis* Endl.: the terpene fraction consists of d-pinene practically free from nopinene and other terpene hydrocarbons. *Pinus merkusii* Jungh and de Vr.: this turpentine contains pinene and nopinene, its quantitative composition and optical rotation being very similar to those of turpentine of *P. maritimus*. *Pinus khasya* Royle: is characterized by a high nopinene content.—G. Dupont. *Bull. Inst. Pin* (1937), 133-134.

Starch Solution—Preservation of, as Indicator. Necessary to have starch solution prepared in advance. Paste of starch and mercuric iodide stirred into boiling water; on cooling, supernatant liquid is decanted or filtered off, and is ready for use. Sulfuretted hydrogen or ionized sulfur may be substituted for mercuric iodide.—General Notes. Rev. quim. farm., 2 (1937), 92. (G. S. G.)

Strychnine—Determination of Small Quantities of. Strychnine in amounts as low as 5 mg. can be determined by precipitating with a standard KI-HgI₂ solution and titration of excess iodide with standard potassium iodate. The technic of the method is described in detail.—A. G. Murray. J. Assoc. Official Agr. Chem., 20 (1937), 638-645; through Chem. Abstr., 32 (1938), 308.

(F. J. S.)

Sulfate—Benzidine Method for the Microdetermination of. A quantity of solution containing 0.05 to 2.0 mg. of sulfate is pipetted into a small hard glass dish, 0.5 cc. of concentrated nitric acid is added, and the solution evaporated to dryness on a steam-bath. The residue is moistened with a few drops of nitric acid and again evaporated to dryness. To destroy the resistant organic matter the dish is placed in an electric oven at 400° for about 1 hour. When cool, the residue is moistened with a few drops of nitric acid and evaporated to dryness on a steam-bath. Finally, a few drops of hydrochloric acid are added and the mixture is again evaporated to dryness to remove the nitrates. To the dry residue are added 2 cc. of distilled water and 1 drop (0.05 cc.) of 0.1N hydrochloric acid. The mixture is gently warmed on the steam-bath to dissolve the calcium sulfate flakes and transferred to a pointed centrifuged tube of 8 cc. capacity, with marks at the 4- and 5-cc. levels. The dish is rinsed out three times with 0.5 cc. of water and the contents of the tube are made up to 4 cc. The $p_{\rm H}$ value of the solution prepared thus is about 3. contents of the tube is added 1.0 cc. of the benzidine reagent (8 Gm. of benzidine hydrochloride in 1 liter, freshly prepared and filtered) and 5 minutes afterward the tube is placed in a mixture of crushed ice and water for 10 minutes. The tube is then centrifuged at 3000 r. p. m. for 5 minutes, the supernatant liquid decanted, care being taken not to disturb the precipitate, and the tube is washed with 5 cc. of 80% alcohol, the precipitate being stirred with a thin glass rod. The tube is again centrifuged for 5 minutes at 3000 r. p. m. and the decantation, washing and centrifuging are repeated once more. The tube is placed in a beaker of hot water to drive off the last traces of alcohol, 5 cc. of 0.5% potassium hydroxide are added, and when the precipitate has dissolved the contents are transferred to a 100-cc. Erlenmeyer flask. The tube is rinsed out 4 times with 5 cc. of water and the washings are collected in the flask. After the addition of 1 cc. of concentrated sulfuric acid, the flask is placed on a steam-bath. When the solution is hot, 0.05N potassium permanganate is run in from a burette until the apparent end-point is reached. An excess of onefourth of the volume used and an extra 1.0 cc. are added, and the flask is returned to the steambath for 10 minutes. 2.0 cc. of 0.05N sodium oxalate are added, and when the precipitated manganese had dissolved, the titration is completed with permanganate. The total number of cc. of 0.05N permanganate less 2 cc. (the oxalate back-titration) multiplied by 0.118 gives the quantity of sulfate present in miligrams.—A. W. Marsden and A. G. Pollard. J. Soc. Chem. Ind., 56 (1937), 464T. (E. G. V.)

Sulfur—Determination of Small Portions of. Refinements of the standard lamp method are described which extend its range to the analysis of 0.0001% of sulfur, in liquids or gases. Purified air for combustion, and turbidimetric estimation of sulfur as barium sulfate, permit high accuracy and reproducibility.—V. Zahn. Ind. Eng. Chem., Anal. Ed., 9 (1937), 543. (E. G. V.)

Sulfur—Microdetermination of, in Organic Compounds. Modifications of the Pregl and Carius methods. In the former, the incineration products are oxidized with bromine water, instead of perhydrol. In the Carius method no barium chloride is added to the contents of the combustion tube; this offers the advantage of easier removal of the incinerated residue from the tube than when barium sulfate is present.—E. EISENSTADT. Prom. Org. Khim., 2 (1936), 165-166; through Chimie & Industrie, 38 (1937), 866.

(A. P.-C.)

Tablets—Compressed, Contribution to the Qualitative Investigation of. The following tests are described for tablets of Dover's powder: the presence of alkaloids, presence of morphine and presence of emetine (according to Wollmann). However, the author found that Wollmann's test for emetine was not sufficiently specific in that it is also given by extract of opium. As a more reliable method for determining the presence of ipecac alkaloids, the capillary analysis method of Platz was modified. The method, requiring only one tablet, is described in detail and the results tabulated. The best combination for the extraction of the alkaloids from the tablets is chloroform and 0.1N sodium hydroxide. The method also allows a quantitative approximation. While typical pictures are obtained for the tablets in question, no proof is offered that the same capillary picture may not be given by some other material, since this would be established only by a thorough study of a large number of drug preparations.—F. Wiesmann. Pharm. Acta Helv., 12 (1937), 321.

(M. F. W. D.)

Tannins—Determination of, in Cacao Kernel. The author summarizes his investigation as follows: Chapman's cinchonine sulfate method for determining tannin was investigated with regard to its application to fresh and fermented cacao beans. Cold 40% acetone was found to be a better extracting agent than hot water, and extraction over night was adopted as a routine procedure. Stiasny's reaction for catechol tannins, i. e., precipitation with a mixture of formaldehyde solution and hydrochloric acid, was applied quantitatively, and gave consistent results. The filtrate from the cinchonine tannate precipitate gave a precipitate by Stiasny's method, and it is suggested that this may be a measure of the catechin and similar phenolic compounds, for the value obtained decreases markedly when beans are fermented. On applying Stiasny's reaction to a 40% acetone extract of fresh or fermented cacao beans, the figure obtained was in agreement with the sum of the tannin precipitated as cinchonine sulfate and the Stiasny precipitate obtained from filtrate.—D. W. Duthie. Analyst, 63 (1938), 27. (G. L. W.)

Terpineol—Distillation of. Decomposition of terpineol (I) to hydrocarbons during distillation is prevented by adding a little alkali. Thus distillation of I (200 parts), containing 14% of hydrocarbons and 1% of phenols, with 50% sodium hydroxide (1 part) first to about 86°/6 mm. and then to about 94°/6 mm., gives 80% of pure I, having a delicate lilac odor.—C. H. Bibbs, Assr. to NewPort Industries, Inc. U. S. pat. 2,052,743; through J. Soc. Chem. Ind., 56 (1937), 1270. (E. G. V.)

Thyroxin—Formation of, from Albumin by Iodination. Thyroxin can be synthesized in vitro by iodination of non-specific albumin. Under definite conditions there is obtained an iodoalbumin which is converted into thyroxin by the usual methods.—W. Ludwig and P. Mutzenbecher. Hoppe-Seyler's Z. Physiol. Chem., 244 (1936), Nos. 1-2, 4; through Chimie & Industrie, 38 (1937), 931.

(A. P.-C.)

Trichloroethylene Vapor—Rapid Determination of, in the Air of Rooms. Approximately 30 liters of air from different parts of the room were passed, at a uniform and regulated rate, through an aspirator-bottle containing alcohol, an aliquot part by weight of the resulting alcoholic solution was burned in an ordinary spirit lamp, and the products of the combustion (hydrochloric acid, chlorine and phosgene, which is decomposed by water into hydrochloric acid and carbon dioxide) were collected in a standard solution of silver nitrate. The apparatus consisted of a Pyrex boiling tube having a side tube, 10 inches long and 5 mm. in internal diameter, about $1^1/4$

inches from the closed end of the tube. The side tube was attached to the condenser by means of a rubber stopper. The lamp was placed below the mouth of the boiling tube, its wick being adjusted to give a flame of about an inch high, and the combustion was continued for an hour. It was found that 50 cc. of N/50 silver nitrate solution, divided between two scrubbers, were sufficient for small quantities of trichloroethylene, but for amounts exceeding 0.1 Gm. N/10 silver nitrate solution was used, and subsequently titrated with N/10 or N/50 potassium thiocyanate solution.—A. Dargie. Analyst, 62 (1937), 730. (G. L. W.)

Tutocaine. Methods of determination are discussed.—MIGUEL DERITO. Rev. centro. estud. farm. bioquím., 26 (1937), 763-776; through Chem. Abstr., 32 (1938), 302. (F. J. S.)

Wine—Value of Undetermined Content of. The difference between the total reducing power, and the aggregate oxygen equivalent of the separately determined lactic, malic, tartaric and volatile acids, glycerol, sulfur dioxide, ethyl alcohol, sugars and ferrous iron, is termed the undetermined content, values of which are tabulated for several wines.—G. Ghimicescu. *Mikrochem.*, 22 (1937), 321–326; through *J. Soc. Chem. Ind.*, 56 (1937), 1258. (E. G. V.)

Zinc Phenolsulfonate—Analysis of. The following method was developed: Dissolve 1.5-2.0 Gm. of zinc phenolsulfonate in water to 250 cc.; to a 25-cc. aliquot in a glass-stoppered iodine flask add 45 cc. of approximately 0.1N sodium carbonate, heat for a few minutes to hasten the later reaction with iodine; gradually add 35 cc. 0.1N iodine with continual shaking of the flask, heat the flask over a 1.5-inch Bunsen flame for about 15 minutes with the stopper kept wet with potassium iodide solution to prevent escape of iodine vapors, let stand 10 minutes, cool, acidify with sulfuric acid (1:1), and titrate the excess of iodine with 0.1N sodium thiosulfate, 1 cc. of which = 0.0051465 Gm. of anhydrous or 0.0069465 Gm. of crystalline zinc phenolsulfonate. The method gives results that check to 0.2%. It allows rather wide limits in detail of procedure, 10-cc. variance in the amount of sodium thiosulfate, and at least 15 cc. in excess iodine; the solution must be heated at least 10 minutes after the addition of the iodine.—Mildred W. Evans. J. Assoc. Official Agr. Chem., 20 (1937), 645-648; through Chem. Abstr., 32 (1938), 308.

(F. J. S.)

PHARMACOGNOSY

VEGETABLE DRUGS

Aconite. In the Alpine regions grow more generally three species of Aconite: napellus, variegatum, lycoctonum, but that used in medicine is Aconitum napellus L. It is an herbaceous, perennial plant of Ranunculaceæ family which grows abundantly in woods and in Alpine pasture. It is a rhizome formed from two fusiform tubers. The plant acquires a height of one meter and has many lanceolate leaves. The flowers are of a copper-blue color. The tubers are picked in the autumn, freed from rootlets, washed and dried in the shade. The principle constituents (aconitine, napelline, aconine, atesine) and the properties of the extract, powder, tincture, etc., are very noted, especially to the pharmacist.—A. Peloni. Farm. ital., 14 (1936), 338. (A. C. DeD.)

Belladonna Root—"Bulgarian," Pharmacognosy of. A sample of "Bulgarian" belladonna root was examined by the author. Its morphological and histological characters differ only slightly—if at all—from those of the ordinary belladonna root of commerce. It has a much lower alkaloidal content than B. P. belladonna root, but chemical qualitative tests, and a physiological test, indicate that their chemical constituents are the same. Further investigation is being made.—A. E. Bailey. *Pharm. J.*, 140 (1938), 77. (W. B. B.)

Cascara Bark from Kenya. A sample received in June 1936 was rather thin in character (as might be expected of the product of young trees), had an ash content (6.5%) slightly greater than specified by the Brit. Pharm. (6%), and had an aqueous extract satisfactory in amount (26.6%). A somewhat larger lot of the same bark is being stored for one year, after which an extract will be prepared and tested physiologically.—Anon. Bull. Imp. Inst., 35 (1937), 424-427.

Chinese Drugs—Famous Old. Ginseng, chaulmoogra oil, ephedrine, sinomenin, algæ, toads and hartshorne are discussed as they appeared in ancient Chinese materia medica.—A. Göhring. Apoth. Ztg., 52 (1937), 1511-1515. (H. M. B.)

Digitalis. Digitalis purpurea grows in Sicily, Corsica and Sardinia. It is known as thimble of the Madonna, herald herb and belongs to the Scrophulariaceæ family. Planted bien-

nially, the sowing is done in March-April, or May according to conditions of temperature, since the plant grows rapidly and cannot stand the cold. The seed is very small, and for uniform sowing, is mixed with dry fine sand. The digitalis of the first year, has only a rosette of leaves; in the second year, the stem becomes as high as one meter. It has alternate leaves, oblong, margin crenate. The flowers are unilateral bunches of beauty and of the form of a thimble, purple or pale rose in color; gamopetalous corolla, in the neck of the flower are many violet spots. It has five sepals, five petals, four stamens: two short and two long, pistil with two stigmas. The fruit is a capsule. The drug is the leaf which is picked the second year. The digitalis in commerce is easily adulterated with leaves of Verbascum thapsus and Verbascum blattaris. The price of the drug varies according to the year from 300 to 480 lira per hundred weight. The leaves are gathered in June and in much sun, that is, when the leaves are very dry; the leaves are dried on bow nets in the shade; it is thought that a small part of the glucoside is lost during drying. Upon aging of the drug some of the enzyme is lost and for this reason the Italian Pharmacopæia considers the leaves inactive after being preserved for one year. From 1000 Gm. of dried leaves of highest quality, 3 Gm. of purified cardio-active substance can be extracted. For the preservation of the drug, it is necessary to keep the leaves in a dry glass container, tightly covered, or in a carton lined with absorbent paper.—A. LISANTI. Farm. ital., 14 (1936), 331. (A. C. DeD.)

Fava de St. Ignatius of Brazil. Study of Facillea trilobata or nhandiroba. Pharmacognostic description, and method of extracting alkaloid from seeds, with microphotographs of crystals obtained.—Carlos Stellfeld. Tribuna Farm., 5 (1937), 122. (G. S. G.)

Marihuana—Field Tests for. A number of field tests both chemical and biological are discussed.—A. Viehoever. Am. J. Pharm., 109 (1937), 589. (A. C. DeD.)

Matricaria Discoidea DC.—Pharmacognosy of. The production, drying and storage of this drug are described. It is strongly aromatic, somewhat reminiscent of apple and lavender, although slightly musty. The viscous oil therefrom, owing to the high paraffin content, is only moderately soluble in 70-90% alcohol, and is brownish green in color. There is great similarity in the constituents of discoidea and chamomilla.—P. N. Schürhoff and K. Hartwich. Arch. Pharm., 275 (1937), 256-268; through Chem. Abstr., 32 (1938), 301. (F. J. S.)

Nutmegs from Granada. Two samples of Granada nutmegs were found to contain more volatile oil (11.2%) than East Indian nutmegs (about 5.9%). The volatile oil obtained by steam distillation of the Granada nutmegs had the following constants: specific gravity at 15.5° C. 0.8666, 0.8682; optical rotation 48.7° (at 25° C.), 48.4° (at 24° C.); refractive index at 20° C. 1.4728, 1.4736; soluble in 4 volumes of 90% alcohol at 15.5° C. with slight opalescence. These constants differ from those of East Indian nutmeg oil and from the requirements of the Brit. Pharm., and indicate that the larger yield is due to a higher content of terpenes which do not contribute to the spice value of the oil. Four further samples of Granada nutmegs showed a yield of oil that was much lower and fairly close to that from East Indian nutmegs; and the constants of the volatile oil were also closer to those of East Indian oil and to the requirements of the Brit. Pharm.—Anon. Bull. Imp. Inst., 35 (1937), 289-297. (A. P.-C.)

Philippine Ginger—Effect of Decortication on the Constituents of. Decortication of the rhizomes of Zingiber officinale Rosc., grown in the Philippines, was tried in an effort to alter the chemical composition sufficiently to meet the requirements of the United States standards. After washing, the whole rhizome was split longitudinally in halves. One-half was decorticated and the other half was not, being used as a control. Care was taken not to cut deeply and destroy the subepidermal cells which contain much of the oil. The peeled half was washed. Both halves were sun dried and analyzed according to methods of analysis of the Association of Official Agricultural Chemists. A table of results for twenty-two samples is given. Decortication greatly increased the amount of starch and lowered the crude fiber and ash content. Water-soluble ash was slightly reduced. Changes in other constituents were not consistent. Decortication did not produce sufficient change to make ginger conform to the United States standards.—Joaquin Maranon and Luz Ll. Cosme. Philippine J. Sci., 63 (1937), 405. (P. A. F.)

Psyllium Seed and Its Substitution by Native Plantain Seeds. The swelling factors of 11 species of plantago seed, linseed and aquilegia are reported using the Youngken method. The various species are described. A mucilaginous epidermis is present on the seeds of imported P. psyllium as well as on those of the native plantains. This may be proved microscopically and by the swelling factor. The mucilaginous content of the German varieties differ greatly. Species

growing in alpine and shore regions were comparable to imported sorts; crowfoot and sand sorts yield somewhat smaller amounts of mucilage. W. recommends that the native species may be used as mild laxatives.—ULRICH WEBER. Apoth. Ztg., 52 (1937), 1619-1623. (H. M. B.)

Rhubarb (Rheum Hybridum)—Seasonal Changes in the Organic Acids of. Data are given showing the marked changes in concentration and content of citric, malic, oxalic and unknown acids in rhubarb root at different periods of the year. During the summer all acids increase greatly.—A. Allsopp. *Biochem. J.*, 31 (1937), 1820; through *Squibb Abstr. Bull.*, 10 (1937), A-2122. (F. J. S.)

Saffron—Investigation of. Analyses of 36 samples are given.—Thure Sundberg. Tek. Tid., 67, Uppl. A-C, Kemi (1937), 41-44; through Chem. Abstr., 31 (1937), 7193. (F. J. S.)

Tikitiki or Rice Bran. Pharmacognostic study, with micro photographic plates of structures. A light yellowish orange or yellowish brown powder. Standard of quality dependent on variety of rice from which it is prepared, kind of rice-mill used, adulteration or accidental admixture, amount of embryo and aleurone layer present.—Jose K. Santos. Rev. Filip. Med. Farm., 28 (1937), 337. (G. S. G.)

Tonka Bean. The bulk of the tonka beans of commerce comes from the tree *Dipteryx odorata* (Willd), but some confusion has been caused through the use of the old name Coumarouna. It is probable that more than one species of Dipteryx provide beans for the market. For instance, *Dipteryx rosea* (Spruce) and *Dipteryx punctata* (Blake) are said to contain coumarin. Other species, *D. polyphylla* Hub. and *D. oppositifolia* (Willd), are reported to be odorless.—Anon. *Pharm. J.*, 140 (1938), 74. (W. B. B.)

PHARMACY

GALENICAL

Aromatic Principles—Extraction of, by Means of Volatile Solvents. VI. A review dealing with the preparation of concrete essences.—R. Y. Naves. Riechstoff-Ind. Kosmetik, 12 (1937), 175–178. (H. M. B.)

Ascorbic Acid—Stability of Galenical Preparations of. The stability of ascorbic acid in tablets and in solutions for injection is studied. The stability in tablets is excellent. Tablets containing as excipients lactose, arrowroot starch and talcum, which initially contained 101.3 mg. ascorbic acid per tablet, after seven months contained 101.0 mg. Ampuled solutions for injection containing 50 mg. ascorbic acid per cc. lost on autoclaving at 120° C. for 20 minutes, about 5%. On further keeping at room temperature, an additional loss of 15% in seven months was observed. If the solutions were made with sodium bicarbonate, 0.45 Gm. for each gram of ascorbic acid, with resulting $p_{\rm H}=6$, the loss on autoclaving was only 3% and, on standing, the autoclaved solutions lost only a further 2% in seven months. The results confirm those reported by Kubli (Festschr. für E. C. Barrell, Basel, -vide, Chem. Ztg. (Oct. 14, 1936).—I. Bennekou and S. A. Schou. Dansk Tids. Farm., 11 (1937), 349. (C. S. L.)

Chemicals, Drugs and Galenical Preparations—Stability of. A review.—KARL BECKER. Apoth. Ztg., 52 (1937), 1470-1472. (H. M. B.)

Emulsifiers—Addenda on. Emulsifying equipment is described.—Francis Chilson. Drug and Cosmetic Ind., 41 (1937), 774-775, 779. (H. M. B.)

Emulsions—Dehydration of Oil. Oil De-emulsification. Petroleum oil emulsions are dehydrated by mixing with them a demulsifying agent consisting of a mixture of higher alcohols obtained by fermenting a carbohydrate solution until it contains 8–16% by volume of mixed alcohols, destroying the microörganisms by adding a cyanide salt combined with a metallic base, such as potassium thiocyanate or sodium thiocyanate, and separating the liquid from the fermented solution, this liquid serving as the demulsifying agent. An aqueous thio-salt solution is mixed with a distributing agent, such as, a thiosulfate with a glycol ether, or a thiocyanate with an alcohol or a combination of alcohols; small amounts of acetic and lactic acid may be added if desired. A suitable mixture consists of sodium thiosulfate 1, diethylene glycol 2.43 and water 41.6 pounds.—G. D. BAVIN, M. POWELL. U. S. pat. 2,056,668–669; through J. Soc. Chem. Ind., 56 (1937), 1304.

Ether for Anesthesia. The best method of protecting anesthetic ether from decomposition consists in keeping it in bottles wrapped in black paper, in presence of alkaline reagents, preferably

potassium carbonate. Metallic iron, while it retards the appearance of peroxides, increases slightly the proportion of aldehydes formed.—A. Mankov and Z. Larionov. *Prom. Org. Khim.*, 1 (1936), 161–162; through *Chimie & Industrie*, 38 (1937), 523. (A. P.-C.)

Lobeline Hydrochloride-Stability of Solutions of. For measurement of the degree of decomposition of lobeline in solution, the content of acetophenone is determined spectrophotometrically. The acetophenone is isolated by ether extraction from acid solution and determined from the absorption at 2750 Å (maximum). Control experiments show that the method is highly accurate. The influence of heat sterilization on the decomposition of lobeline hydrochloride in solution is studied. In aqueous solution at $p_{\rm H}$ 5.8 the loss from a 1% solution is 9.3% after 3 hours at 80° C., 23% after 1 hour at 100° C. and 29% after 20 minutes at 120° C. In hydrochloric acid solution at ph 3.3 decomposition is lessened but is still too great (7.5% loss in 20 minutes at 120° C.). Studying the losses in commercial, ampuled preparations of the 1% solution of various ages on heating for 2 hours at 80° C. from 1 to 1.9% loss is seen. A more dilute acidified solution (0.3% lobeline) showed smaller percental loss than the 1% solution. The acid solutions are colored yellow by sterilization. The keeping qualities of lobeline hydrochloride solutions are followed at intervals over 18 months. In pure aqueous solution decomposition is rapid (2.9% in 3 weeks) and the solution becomes colored. Acidified solutions in ampuls are more stable, showing not over 1% loss in the whole period of test. Solutions partly decomposed by warming are stable practically without change in four months.—F. REIMERS. Dansk Tids. Farm., 11 (1937), 296. (C. S. L.)

Malt—Extract of, Manufacture of. The starting point in the manufacture of extract of malt is malted barley. Due to the lack of favorable climatic conditions in Great Britain, English barley malt is stated to be unsuitable where a high standard of feeding quality is demanded. Suitable malted barley is crushed in a double roll malt mill, from which it is delivered to a grist case. From here it passes into a mashing machine, in which it is infused with hot water at a temperature of 139° F. This machine passes it to the mash tun where it remains for a number of hours, during which digestion takes place, the diastase in the malt acting upon the starch and converting it into sugars (maltose, dextrose, etc.). In the tun the malt is sprayed with hot water, and later the thin liquor is drawn by vacuum into evaporating pans. The spent mash is retained in the mash tun. The syrup is evaporated in a rapid circulating evaporator fitted with a thermographic temperature control. The liquid is maintained at a temperature of 115° F., and the syrup concentrated until the specific gravity reaches 1.4. The spent mash is removed from the tun and utilized for cattle feeding purposes. The extract is run into storage tanks, from which, after testing in duplicate, it is conveyed to the bottling department, where it is mixed with cod liver oil, halibut liver oil, etc. The extract, when tested, should possess a Lintner value of 60 (a measure of the diastase content), and when examined in accordance with the directions given in the B. P., 1932, it should contain not less than 4.5% w/w of protein. Photographs of apparatus used in the manufacture of extract of malt are shown, including a grist case, a mash tun, a tandem vacuum pumping engine, a rapid circulating evaporator; the wet grains prior to being dried for cattle feeding purposes are also shown.—H. DAVIS. Pharm. J., 139 (1937), 358. (W. B. B.)

Phenobarbitone—Sodium, Sterilization of Aqueous Solutions of. An investigation was carried out in order to find the loss of sodium phenobarbitone solutions after subjection to the methods of sterilization. The degree of decomposition depends on the temperature, the time of exposure to heat, the alkalinity, and to a lesser degree on the concentration of the solution. A 20% solution of sodium phenobarbitone loses 20% on autoclaving, 10% on steaming, 6 to 7% on tyndallization, and 1% when the solution is sterilized by the emergency method of the B. P. Solutions of sodium phenobarbitone for injection should be prepared from a sample yielding a solution of the $p_{\rm H}$ value as low as possible, and should be sterilized by filtration only. They should be stored only for a short period and in a cool place.—H. W. Tomski and L. J. Waller. *Pharm. J.*, 139 (1937), 421. (W. B. B.)

Sterilization of Aqueous Pharmaceutical Solutions and Apparatus by Chemical Means. L.'s experiments confirm the report of Bonde and Velthorst whereby the germicidal action of the esters of p-hydroxy benzoic acid and their salts toward cocci and non-spore-forming bacteria was shown to be satisfactory but not true toward the spore-forming bacilli; their use, therefore, as agents for sterilization in pharmacy must be avoided.—Lühr. Apoth. Ztg., 53 (1938), 146-148.

(H. M. B.)

Tincture of Cinchona—Stabilization Experiments with. The precipitates occurring in official tincture of cinchona are transformation products of cinchona tannic acids and in these precipitates alkaloids (9-13%) are occluded. Their formation is due to oxidation phenomena and may be prevented if the tincture is cooled to a low temperature after preparation, then filtered and stored in well-filled bottles. The addition of such acids as acetic, formic and hydrochloric retards the formation of these precipitates. Tinctures to which hydrochloric or acetic acids have been added show a loss of alkaloidal content on storage; those containing formic acid show a stable alkaloidal content. Extraction of the drug with 42% alcohol containing 1% formic acid yields a preparation very rich in alkaloids and which, when stored in well-filled bottles, yields no precipitates and the alkaloidal content remains constant.—Hans Wojahn. Apoth. Ztg., 52 (1937), 1485-1488. (H. M. B.)

PHARMACOPŒIAS AND FORMULARIES

Dental Formulæ. A description of the Pharmacopœia of the Royal Dental Hospital, London. The Pharmacopœia contains mostly by formulæ for preparations used in oral hygiene. The names of the ingredients are set out in full Latin, and in the main the nomenclature of the B. P. is followed.—Anon. *Pharm. J.*, 140 (1938), 4. (W. B. B.)

National Formulary—1936. A review with comments.—Hanns Will. Apoth. Ztg., 52 (1937), 1390-1392, 1423-1425. (H. M. B.)

Pharmacopæia—New. Criticisms of the U. S. P. XI, based largely on a difference of opinion as to the purpose of the publication. C. suggests that it be more all-embracing, more useful to the practicing physician, less a selected list of drugs used. C. protests against the growing tendency for the physician to prescribe ready-made patent and proprietary preparations. More instruction in materia medica and therapeutics in medical schools is advocated. The inclusion in U. S. P. of certain drugs whose action has not been proved on laboratory animals and of a reasonable number of long-approved formulæ is suggested. The purposes of the N. F. are discussed very briefly.—Warren Coleman. New York State Journal Med., 37 (1937), 1643; through Squibb Abstr. Bull., 10 (1937), A-1933. (F. J. S.)

U. S. P. XI—Supplement to the. Regular interim revisions of the United States Pharmacopæia have been arranged. The first supplement to the U. S. P. XI was issued in August, and becomes official on December 1, 1937. More important changes in the U. S. P. XI are made in the following: acetophenetidinum, aconitum, acriflavina, aqua, carbromalum, codeinæ phosphas, dextrosum, ephedrinæ sulfas, erythritylis tetranitras dilutas, fluidextracta, hydrargyrum cum creta, iodophthaleinum solubile, liquor pituitarii posterioris, massa hydrargyri, menthol, morphinæ sulfas, oleum morrhuæ, rheum, spiritus ammoniæ aromaticus, tabellæ glycerylis trinitratis, theophyllina cum æthylenediamina, tinctura nucis vomicæ, tinctura opii camphorata, unguentum hydrargyri fortum, unguentum hydrargyri mite, unguentum zinci oxidi.—Anon. Pharm. J., 139 (1937), 394.

Non-Official Formulæ

Calamine in Pharmacy and Cosmetics. A discussion. Formulæ for use of zinc carbonate in lotions, liquid face powders, ointments, etc., are given.—F. J. Bolton. *Pharm. and Cosmetics* (1935), 153-154; through J. Soc. Chem. Ind., 56 (1937), 1406. (E. G. V.)

Shampoos—Preparation of. A shampoo powder "Ellas" was found to contain fatty acid 50.6%, pure soap 54.58%, sodium tetraborate 30.6%, sodium bicarbonate 7%, water 2.7%. The following types are discussed and formulæ offered: Shampoo Bases.—(1) Palm oil 150 Kg., tallow 100, lard 50, coconut oil 200. (2) Palm oil 100 Kg., solid fat 100, coconut oil 250, olive oil 50. (3) Palm oil 200 Kg., tallow 150, coconut oil 150. Simple Shampoos.—(1) Soap powder 600 parts, borax 200, ammonia-soda 200. (2) Soap powder 600 parts, trisodium phosphate 100, borax 150, sodium bicarbonate 150. (3) Soap powder 600 parts, sodium bicarbonate 150, borax 250, sodium carbonate 50. Bleaching Shampoos.—(1) Soap powder 300 parts, borax 75, sodium carbonate 100, Henna-Keng extract 5. (2) Soap powder 250 parts, sodium bicarbonate 75, borax 125, sodium perborate 50. Lustre Rinse.—Alum 60 parts, tartaric acid 30, citric acid 30. Soapfree Powder Shampoos.—(1) Borax 150 parts, sodium carbonate 100, saponin 5, talc 10. (2) Boric acid 100 parts, saponin 300, talcum 50, sodium bicarbonate 50, saponin 200, orris 100. Liquid Soap-free Shampoos.—(1) Saponin 70 parts, alcohol (30%) 110, sodium carbonate 10,

ammonia (d = 0.91) 10. (2) Ammonia (d = 0.91) 5 parts, potassium carbonate 5, sodium carbonate 5, saponin 10, water 475. Triethanolamine Soap.—Olein 220 parts, coconut oil fatty acids 160, triethanolamine 200, alcohol 110, water 110. Shampoos with Soaps and Fatty Alcohol Sulfonates.—(1) Soap powder 500 parts, gardinol R sodium laurylsulfonate 150, borax 300, sodium bicarbonate 50. (2) Soap powder 300 parts, gardinol Ca (sodium cetylsulfonate) 50, borax 100, sodium bicarbonate 50. (3) Soap powder 50%, sodium laurylsulfonate 10, sodium sesquicarbonate 40. (4) Fatty alcohol sulfonate 350 parts, sodium sulfonate 30, sodium bicarbonate 45. (5) Borax 75 parts, lorolsulfonate 400, borax 50, sodium metaphosphate 50. (6) Soap powder 375 parts, borax 25, sodium bicarbonate 25, Lamepone B-100 75. (7) Soap powder 300 parts, Igepone 50, borax 35, sodium bicarbonate 30, Lamepone B-100 85. (8) Soap powder 250 parts, borax 80, sodium carbonate 25, sodium bicarbonate 90, dry Henna extract 5, sodium laurylsulfonate 50. Liquid Shampoos.—(1) Water 150 parts, sodium sulforicinate 150, perfume 1-2. (2) Lorolsulfonate 160 parts, borax 20, sodium bicarbonate 20, liquid soap (about 25%) 425, Lamepone PH-A 35, special turkey red oil 25. (3) Alcohol (50%) 326 parts, sapamin citrate 60, camomile extract 6, lemon juice 6. (4) Liquid soap (25-30%) 563 parts, triethanolamine laurylsulfonate 113, turkey red oil 37, Lamepone PH-A 37. Powder Shampoo with Sapamine .-- (1) Sapamin acetate 100 parts, tatarus depuratus 98, saponin 2. (2) Sapamin citrate 50, saponin 1, boric acid 49.—Ekmann. Riechstoff-Ind. Kosmetik, 12 (1937), 201-205. (H. M. B.)

DISPENSING

Albumen Ovi Siccum. The (Dutch) Pharmacopæia defines Albumen Ovi Siccum as the dried, uncoagulated whites of fresh hen's eggs but gives no method to identify this albumen as that of hen's eggs. De Graff mentions (in the Commentaar) that this may be identified by a biological method, but no pharmacist could carry out such a method. Neither the Pharmacopæia nor the literature gives methods to demonstrate that the albumen has been derived from fresh eggs. The author therefore suggests that fresh egg albumen be used in the manufacture of preparations and gives methods for preparing Solutio Ferri Albuminata and Tannalbuminum.—H. T. LIEM. Pharm. Tijdschr. Nederland-Indië, 13 (1936), 1. (E. H. W.)

Bases—Absorption, Pharmacy's Newer. A description is given of newer absorption bases, comprising the wetting and emulsifying agents Areskap, Aresket and Aresklene (sodium salts of alkylated aryl compounds), Santomorse and triethanolamine. Triethanolamine is especially valuable for cosmetic use because of its complete lack of any alkali-irritating effect. When it is present in even very low concentrations it makes cosmetic creams completely removable by washing. A vanishing cream formula containing this agent is given.—Gordon A. Bergy. Am. Professional Pharmacist, 3 (1937), 21; through Squibb Abstr. Bull., 10 (1937), A-2048. (F. J. S.)

Decoctum Chinæ Cum Senegæ. The solubility relationships of the quinine alkaloids during aqueous extraction from quinine bark are studied. Practically all the water-soluble alkaloids are removed in the first extract (53.9-64.9% of the total alkaloid of the drug) while three further extractions only raise the total extracted to 59.8-69.5%. The fineness of pulverization, variation of time of extraction or quantity of fluid used have no effect within the limits studied. Acid extraction is also considered. If 4-5 cc. of 1N hydrochloric acid are used for the extraction of 10 Gm, of bark (\$\rho_H 2.4-2.6\$) practically all the alkaloid of the drug is extracted, if one takes into account the quantity retained in the fluid absorbed in the drug residue. Fractional extraction first with water, then with hydrochloric acid is compared with the extract made with four portions of water only, and with a determination of total quinine content of the drug (by the oxalate method). No fractionation of the alkaloids is observed. In the Danish Pharmacopæia directions for making Decoctum Chinæ cum Senegæ, one adds the senega root during the last five minutes of the extraction. Experiments indicate that this fails to extract much of the senega saponin. One reason for this is precipitation of the saponin by the tannic acid derivatives of the bark. If these compounds are treated with alkali, it is evident that the senega saponin has not lost its hemolytic power.—C. J. T. MADSEN. Dansk Tids. Farm., 11 (1937), 221.

Drugs—Alkaloidal, Ease of Extraction of, and Testing with Mayer's Reagent. The question of the ease of extraction of alkaloidal drugs becomes important in the preparation of solid extracts, fluidextracts and tinctures, since the total active constituent of the drug is required in the finished preparation. Therefore, the extraction of the drug must be carried to complete exhaustion. This is entirely logical since the partial extraction may stop with the incomplete removal of

certain constituents, giving a different composition in the preparation than in the drug. The Swiss Pharmacopœia V requires various alkaloidal drugs to be percolated until 10 cc. of the percolate when evaporated to dryness with 3 drops of dilute hydrochloric acid on a water-bath, dissolved in 5 cc. of water, filtered and the filtrate treated with Mayer's reagent gives only a faint opalescence. The extraction of the following drugs was studied as to the ease of percolation, quantity of menstruum for complete extraction using the Swiss pharmacopæial test, and the quantity of percolate necessary for economic extraction: belladonna leaves, cinchona bark, coca leaves, hydrastis rhizome, hyoscyamus herb, ipecac root and nux vomica seeds. The sensitivity of Mayer's reagent for the alkaloids which would be extracted was also investigated. The studies on the ease of extraction of alkaloidal drugs showed that the individual drugs exhibited a wide variation in extractability during the percolation. Hyoscyamus herb, belladonna and coca leaves were most easily extracted, hydrastis rhizome and nux vomica seeds were more difficultly extracted while ipecac root and cinchona bark were very difficult to extract. The test of the Swiss Pharmacopæia V for the complete exhaustion of the drugs was shown to be too sensitive. Such extraction would require the pharmacist to use too much solvent to obtain the last traces of alkaloids, making the extraction cumbersome and impractical. The following modification of the pharmacopoeial test is suggested: The alkaloidal drugs (with the exception of ergot) should be extracted until the volume of percolate, specified under each drug, when evaporated with 3 drops of dilute hydrochloric acid on a water-bath, dissolved in 5 cc. of water, filtered and tested with Mayer's reagent gives an opalescence but no precipitate. For preparing extracts of the individual drugs the following volumes of percolate were specified: belladonna 5 cc., cinchona 0.1 cc., coca 2.0 cc., hydrastis 2.5 cc., hyoscyamus 5.0 cc., ipecae 0.3 cc. and nux vomica 2.5 cc. By this test 2 to 3% of alkaloids are still obtained in the last percolate.—J. BÜCHI. Pharm. Acta Helv., 12 (1937), 326-335.

(M. F. W. D.)

Quinine, Calcium, Magnesium and Camphosulfonic Acid—Hypodermic Solutions of the Salts of. The characteristic of salts of camphosulfonic acid is that a group of basic function is attached to a group that has an acid function. They have given satisfactory clinical results in cardiopathy, the stimulative action of the camphor complex on the nervous system, circulation and respiration being advantageous. Directions are given for preparing concentrated solutions of salts of camphosulfonic acid for hypodermic injection, consisting of quinine camphosulfonate (neutral), quinine camphosulfonate (basic), calcium camphosulfonate and magnesium camphosulfonate.—Anon. *Pharm. J.*, 140 (1938), 53. (W. B. B.)

Sodium Benzoate-Calcium Chloride—Incompatibility of. The formation of a precipitate of calcium benzoate, as observed in "formulas magistrales," is caused by a lack of solvent. The precipitation is prevented if both salts are dissolved separately before mixing. A relative excess of benzoate requires larger quantities of solvent.—EMILIO A. DEL CARLO. Rev. farm. (Buenos Aires), 79 (1937), 247. (A. E. M.)

PHARMACEUTICAL HISTORY

Danish Pharmacy—History of, in the 20th Century. A review of the development of Danish pharmacy since 1900 and of the Danish Pharmaceutical Society from its inception in 1912.—H. Barroed. Dansk Tids. Farm., 11 (1937), 249. (C. S. L.)

Pharmacy—Military, in Various Countries. Brief reviews of the military pharmacy organizations of Switzerland and of France. The section concerning France cites statistics concerning the quantities of various drugs used during the World War.—A. CEDERGREN. Farm. Revy, 36 (1937), 709, 733. (C. S. L.)

Pharmacy in the Past. Illustrations of cuts and engravings of historical scenes pertaining to pharmacy are shown, and a description of each illustration is given. The following illustrations are given and described: (1) A miniature painting from a seventeenth century German manuscript, (2) a pharmacy of the Rococo period, (3) the center panel from a window blind of the shop of an English chemist, (4) a view of a seventeenth century apothecary's laboratory, (5) the title page of the municipal "Pharmacopœia Augustana," 1640.—Anon. Pharm. J., 139 (1937), 360. (W. B. B.)

Pharmacy and Physic—Historical Relations of. Pharmacy was necessarily part of primitive medicine, and its separation, like that of nursing, has been a result of evolution and specialization. The evolution of pharmacy, and the relation of pharmacy to grocers, barber-surgeons and the medical profession is discussed.—Anon. Chemistry and Industry, 56 (1937), 1022. (E. G. V.)

Pharmacy—Seventeenth Century. A picture is reproduced which appears to be the earliest oil painting in existence, representing, to all intents and purposes, a modern chemist's shop. The date on the base of one of the large mortars is 1652. The details of the fittings, the mortars, the paneling of the counter, the jars and boxes are represented with the meticulous accuracy characteristic of the Dutch painters of the seventeenth century, and it is possible to read on the jars the names of their contents. Careful study of the original picture reveals the names of forty-two drugs, which appear on the drawers, jars, etc.—Anon. Pharm. J., 139 (1937), 363.

(W. B. B.)

Miscellaneous

Acetylsalicylic Acid Composition. Aspirin is dissolved in an inert solvent which is free from water, acids and alkalis (such as, glycol oleate, o-methyl salicylate, pine and other essential oils, fatty oils) to yield stable solutions suitable as external medicaments.—E. B. PUTT. U. S pat. 2,056,208; through J. Soc. Chem. Ind., 56 (1937), 1408. (E. G. V.)

Balances and Weighing. The importance of this operation in perfume manufacture is stressed. Several types of balances suitable in this industry are described.—Anon. Riechstoff-Ind. Kosmetik, 12 (1937), 214-217. (H. M. B.)

Beauty Creams. A number of creams including cleansing, tonic, cold-cream and foundation are described. Typical formulas are given for a few of these creams.—G. Delfini. Farm. ital., 14 (1936), 333. (A. C. DeD.)

Blow-Fly Repellent—New. The oil from a weed which grows widely in the Union of South Africa is suggested as a fly repellent. The plant, Tagetes minima, popularly known in South Africa as "khaki bush," grows on poor, dry soil, and yields by steam distillation about 0.5% volatile oil. Soap emulsions of the oil were found to be unsatisfactory, as the free alkali of the soap was irritating to the sheep on which they were tried, and best results were obtained by using an emulsion made from the following: Wool fat 60 Gm., carbon tetrachloride 200 cc., tagetes oil 50 cc. and water 700 cc. Add about 20 cc. of carbon tetrachloride and 100 cc. of water to the wool fat; form a primary emulsion and gradually incorporate the rest of the ingredients. Neutralize with 10% sodium hydroxide solution to phenolphthalein. It was found that 5% of the tagetes oil could be replaced by 10% oil of tar, but the results were not so satisfactory.—H. O. Mönnig. Onderstepoort J. Vet. Sci. Animal Ind., 7 (1936), 419; through Pharm. J., 140 (1938), 57.

(W. B. B.)

Brushes—Self-Sterilizing. Bristles of tooth brushes are treated with a solution of a phenyl mercuric salt such as the nitrate or chloride and with an agent such as sodium iodide adapted to react with the phenyl mercuric salt to form a phenyl mercuric salt less soluble in water.—Warren E. Hill and Frederic A. Parkhurst, assignors to Pro-phy-lac-tic Brush Co. U. S. pat. 2,099,888, Nov. 23, 1937. (A. P.-C.)

Carotin, a New Material for Cosmetics. A review.—Erman. Riechstoff-Ind. Kosmetik, 12 (1937), 244-245. (H. M. B.)

Cosmetic—Colored Make-Up, in Dry Cake Form. A mixture is used containing about 70 to 97% of colors, pigments and fillers, about 1 to 26% of oils and waxes, and 1 to 13% of water-soluble dispersing agents.—Frank Factor and Paul E. Fisher, assignors to Max Factor and Co. U. S. pat. 2,101,843, Dec. 14, 1937. (A. P.-C.)

Cosmetic Products. Products such as toilet soaps, shampoos, shaving soaps and creams, salves, toothpastes, etc., are prepared containing albumin decomposition products acylated at the nitrogen with higher fatty acid residues such as those of lauric, palmitic, stearic, oleic, ricinoleic, linoleic or linolenic acid, or the acids of soybean or täll oil (albumin decomposition products of the type of lysalbinic and protalbinic acids being suitable).—Fritz Sommer and Max Nassau, assignors to Chemische Fabrik Grünau Landshoff & Meyer A.-G. U. S. pat. 2,100,090, Nov. 23, 1937. (A. P.-C.)

Cosmetics and Adolescence. The functions of the skin, its glands and layers are discussed. It is concluded that acne of an adolescent nature is due to a faulty lipid metabolism which might be readjusted to a more natural basis by cosmetics similar to the skin pattern. However, such factors as age, sex, diet, climate and environment must also be considered.—Mary Imogene Shepherd.

Drug and Cosmetic Ind., 42 (1938), 57-58. (H. M. B.)

Depilatories. Most depilatories depend for their action upon the presence of a sulfide, which may be barium, strontium, calcium or sodium. Barium sulfide should be avoided, and sat-

isfactory results have been claimed from the use of strontium sulfide. About 20% is needed, with the same proportion of zinc oxide, together with precipitated chalk and talc. If it is desired to supply the depilatory as a paste—the usual form—then a small quantity of soap and sufficient water to produce the right consistence can be added. Alternatively, the compound tragacanth paste of the Codex, diluted to the right consistency, could be employed. The addition of solution of witch-hazel helps to mask the unpleasant odor of the sulfide.—Anon. Pharm. J., 140 (1938), 64.

(W. B. B.)

Diarrhoea—Remedies Used in the Treatment of. These agents are divided into (1) organic substances active after resorption such as opium and its preparations, coto, uzara, allium, allisatin from Allium sativum, comallysatum, a dialysate of Allium ursinum, (2) astringents including bismuth salts, tannic acid and its derivatives, rhatany, tomentilla rhizome, noventeral and gelonida aluminum subacetate, (3) disinfectants such as yatren, phenyl salicylate, xeroform, (4) adsorbents including charcoal in various combinations, bolus alba, kieselguhr, aluminum hydroxide, (5) those both adsorbent and disinfectant such as adsorgan and silargel, a silver chloride-silicic acid gel. Prescriptions are given for the various agents. 13 references.—Erich Herrmann. Apoth. Zig., 53 (1938), 22-26. (H. M. B.)

Emulsions—Petroleum, Breaking of. The emulsions are treated with a heat-polymerized, blown, sulfonated vegetable oil, or a mixture of such products, such as a sulfonated mixture of 65% by weight of polymerized cottonseed oil and 35% by weight of polymerized castor oil.—E. E. CLAYTOR. U. S. pat. 2,060,281; through J. Soc. Chem. Ind., 56 (1937), 1304. (E. G. V.)

Emulsions—Petroleum, Chemical Methods for Separating. The properties of a good emulsion-breaking compound are outlined. Since these are partly conflicting a compromise is necessary in the choice of a suitable compound. A knowledge of the properties of the water is desirable and should be correlated with those of the emulsion. For alkaline water of high $p_{\rm H}$, neutral fatty acid soaps should be used. For alkaline soaps of low $p_{\rm H}$ best results are given by neutral or weakly acid ricinoleate or by Turkey-red oil. Acid waters require naphthenic acid soaps or sulphonated fatty acids and petroleum products.—G. W. van Dedem. Oil and Gas J., 36 (1937), 65-68; through J. Soc. Chem. Ind., 56 (1937), 1297. (E. G. V.)

Ferrous Compounds—Manufacture of Durable. The manufacture by standard methods of ferrous compounds of "enolized sugar derivatives" [for instance, compounds containing the group .C(OH):C(OH).CO.], said to have therapeutic value, is claimed. Examples are the ferrous compounds of ascorbic acid (made by metathesis or by dissolution of iron in aqueous acid), glucoreductone and dihydroxy maleic acid.—A. G. Bloxam. Brit. pat. 472,531; through J. Soc. Chem. Ind., 56 (1937), 1408. (E. G. V.)

Fungicides—Cupriferous. Fungicides suitable for use on vegetation are obtained by a process which comprises preparing an aqueous gel of the nature of those used in making base-exchanging zeolites by mixing a solution of sodium silicate and a solution of aluminate or alum, causing the copper sulfate solution to react upon the aqueous gel prior to final drying, the amount of copper sulfate being sufficient to furnish cupric oxide to the gelling mixture in more than an equimolecular ratio to the alumina. The gel is dried, washed and reduced to a fine powder suitable for fungicidal purposes. Products may be prepared containing copper and aluminum oxides in molecular ratios of between 1.1 and 10.1.—RAY RILEY and WM. M. BRUCE, assignors to PERMUTTI Co. U. S. pat. 2,099,623, Nov. 16, 1937.

Germicidal and Fungicidal Compositions. Saponifiable germicidal and fungicidal compositions comprising synthetic fatty acids derived from an oxidized petroleum hydrocarbon material are used with sufficient alkali metal hydroxide to give a $p_{\rm H}$ of 4.5 to 5.5.—Cornelia Burwell. U. S. pat. 2,100,469, Nov. 30, 1937. (A. P.-C.)

Hair Shampoos. A detailed procedure is offered.—BERT. PÖLL. Riechstoff-Ind. Kosmetik, 12 (1937), 242. (H. M. B.)

Insecticidal Oil. A new insecticidal oil that is nontoxic to vegetation consists of a distillate fraction from crude petroleum oil which is characterized by having a viscosity of from 65 to 200 sec. Saybolt at 100° F., a specific gravity between 0.9000 and 0.9600, distilling between 5000° F. and 800° F., and yielding an unsulfonated residue of between 60 and 85%.—Lysle R. Coleman and Gerald L. Cowley. U. S. pat. 2,105,856, Jan. 18, 1938.

(A. P.-C.)

Insecticidal Oil Spray. A tree spray composition comprises about 95 to 99% of paraffinic mineral oil, about 0.1 to 2.0% of toxic naphthenic acid, and about 0.5 to 4.5% of oil-soluble

emulsifier.—Hugh Knight, assignor to Emulsoids, Inc. U. S. pat. 2,103,196, Dec. 21, 1937.

Insecticide. A monomolecular combination of rotenone and dichloroacetic acid is claimed as a new chemical compound having insecticidal properties.—Howard A. Jones, dedicated to the free use of the people of the U. S. A. U. S. pat. 2,103,195, Dec. 21, 1937. (A. P.-C.)

Insecticides. The product contains an alkali metal or alkaline-earth metal polyselenide.

—Charles B. Gnadinger. U. S. pat. 2,105,727, Jan. 18, 1938. (A. P.-C.)

Insecticides and Fungicides. The product consists of a water-dispersible condensation product of an inorganic sulfide of selenide with an aliphatic halide containing either two halogen atoms on adjacent carbon atoms or a halogen atom and a hydroxyl group on adjacent carbon atoms.—ROBERT J. BONSTEIN. U. S. pat. 2,102,564, Dec. 14, 1937. (A. P.-C.)

Lyophilic Biologically Active Substances—Sterilizing. A true organic mercury compound such as sodium ethylmercurithiosalicylate, sodium oxymercuri-o-nitrophenolate, diacetoxymercuri-4-nitro-2-cresol, o-chloromercuriphenol, tolyl mercuric nitrate, phenylmercuric nitrate, phenylmercuric acetate, tolyl mercuric acetate, or their homologues, is added in small proportion (as little as 0.01%) shortly before the freezing treatment in the production of solid lyophilic biologically active substances by rapid freezing of liquid biologically active materials and removal of water therefrom while in a solid frozen state under a high vacuum, as described in U. S. pat. 2,066,302.—

JOHN REICHEL, assignor to SHARP & DOHME. U. S. pat. 2,099,659, Nov. 16, 1937. (A. P.-C.)

Odor and Chemical Constitution. A Study for Perfumes. A discussion. A family relationship between the odors for homologous series of compounds (esters, aldehydes, etc.) is noted.—I. Herold. Seifens.-Ztg., 64 (1937), 639-641, 689-690, 709-710; through J. Soc. Chem. Ind., 56 (1937), 1407. (E. G. V.)

Odors—in Perfumery and Nature. A discussion.—Anon. Riechstoff-Ind. Kosmetik, 12 (1937), 199-200. (H. M. B.)

Patents. Are Patents on Medicinal Discoveries and on Foods in the Public Interest? A symposium presented before the Division of Medicinal Chemistry, Biological Chemistry and Agricultural and Food Chemistry of the American Chemical Society. Medical Patents.—Morris Fishbein. Ind. Eng. Chem., 29 (1937), 1314. Society's Need for Patents to University Research Workers, Especially on Food and Drug Inventions.—George B. Schley. Ibid., 1319. Are Patents on Foods and Medicines in the Public Interest? H. L. Russell. Ibid., 1322. Discussion.—H. B. Haas. Donald K. Tressler. Ibid., 1325. (E. G. V.)

Pepper Products—Bleached. For bleaching a viscous or semi-solid oleoresin of pepper, it is mixed directly with an organic peroxide such as benzoyl peroxide in the absence of other material to dissolve the oleoresin, and the mixture is heated sufficiently (suitably to about 90° C.) to effect solution of the peroxide in the oleoresin, and the mass is cooled after bleaching is effected.—LLOYD A. HALL, assignor to GRIFFITH LABORATORIES, INC. U. S. pat. 2,097,405, Oct. 26, 1937.

Pests of the Apothecary and Their Control. The following Lepidoptera (butterflies) are described: (1) fur moths (*Tinea pellionella L.*), (2) clothes moths (*Tinea biselliella Hummel*) and (3) dried fruit moths (*Plodia interpunctella Hb.*).—W. MADEL. Apoth. Ztg., 52 (1937), 1528–1529. (H. M. B.)

Pharmaceutical and Technical Problems in Fermentation Chemistry. A review with eleven references.—Horst Böhme. Apoth. Ztg., 52 (1937), 1247-1250. (H. M. B.)

Plastics—Chemistry and Manufacture of. Pharmaceutical Uses of Resinoids. Resinoids are formed by reacting three molecules of phenol with two of formaldehyde in the presence of a catalyst, acid or alkali, e. g., dilute hydrochloric acid or weak solution of ammonia. Heat is afterward applied, and eventually a complete change is effected. Among the many pharmaceutical uses of resinoids are included the uses in toilet ware, packages for creams and ointments, bottle tops, water-proof bandage, etc.—Anon. Pharm. J., 140 (1938), 54. (W. B. B.)

Shop Windows—Steamy, Prevention of. In general, methods for preventing steamy windows must air either at raising the temperature of the inner surface of the glass above the dewpoint of the aim in the window case or at lowering the dew-point of the air in the window case below the temperature of the inner surface of the glass. The first method of preventing the "steaming" is to place tubular heaters at the bottom of the window. In the second method, which aims at lowering the dew-point of the air in the window case, two lines of approach are possible. First, desic-

cating agents may be used to remove moisture. This is an expensive method unless the desiccating agent can readily be regenerated, and even then, the inconvenience of the manipulation involved would probably cause the process to be neglected. Also, experiments show that shop windows can be kept clear by adequate ventilation with air from the street. For efficient natural ventilation, the window case must have ventilators both at the top and at the bottom.—H. A. Stevenson. *Pharm. J.*, 139 (1937), 655. (W. B. B.)

Specialties and Investigations of 1937. New specialties and investigations under the following headings are reviewed: (1) vitamins, (2) diabetes preparations, (3) hormones, (4) new productions with various applications, (5) known remedies with new applications, (6) chemo-therapeutic agents, (7) technical antifreezes, (8) determinations of alcohol in the blood, (9) helium, (10) from the trade to the profession and (11) sessions and congresses. Forty-nine references are given.—Konrad Schulz. Apoth. Ztg., 53 (1938), 87-90, 101-103, 118-120. (H. M. B.)

Tooth Brush—Antiseptic. The brush head and bristles are immersed in a solution containing phenyl mercuric nitrate, paraffin wax and toluene or like materials.—WARREN E. HILL and CLIFFORD L. McArthur, assignors to Pro-Phy-Lac-tic Brush Co. U. S. pat. 2,099,688, Nov. 23, 1937.

(A. P.-C.)

Violet Essence Research. A review of recent developments.—L. RUZICKA. Drug and Cosmetic Ind., 41 (1937), 766-767, 781. (H. M. B.)

Violet and Violet Perfume. For the preparation of this odor the aromatics recommended as a basis are α-ionone, irone, methyl ionone, oil of iris concrete, concrete and absolute violette feuille, methyl octincarbonic acid; for bouquets, hydroxycitronellal, rhodinol, benzyl acetate, ylang ylang oil, jasmine, phenyl ethyl alcohol and cyclamine aldehyd are used; as fixatives musk ketone, muskene, heliotropin, cinnamic and anisic alcohols and styrax absolute are recommended.

—Anon. Riechstoff-Ind. Kosmetik, 13 (1938), 8-9. (H. M. B.)

PHARMACOLOGY, TOXICOLOGY AND THERAPEUTICS

PHARMACOLOGY

Acetylcholine—Influence of, on Glucemia Effects of Intravenous, Subcutaneous and Intramuscular Injections. In dogs the rapid intravenous injection of 2 to 3 mg. per kilo of acetylcholine produced a 20 to 60% increase in glucemia in 15 minutes with a return to normal in less than an hour after the injection. The intramuscular or subcutaneous injection of 4 mg. per kilo caused a slightly decreased glucemia lasting an hour or longer.—F. JOURDAN, P. GALY and L. GALLONI. Compt. rend. soc. biol., 123 (1936), 604-605; through Chimie & Industrie, 38 (1937), 934.

(A. P.-C.)

Adrenaline and Nicotine—Absorption of, by the Pericardium. When adrenaline is placed in the pericardial cavity of the dog it is absorbed very slowly. It is not oxidized and can be detected in the pericardial fluid several hours after its introduction. Nicotine is absorbed rapidly by the pericardium. Unusually large doses are tolerated when given in this way.—G. Baltaceanu, C. Vasiliu and A. Novac. Compt. rend. soc. biol., 123 (1936), 833-836; through Chimie & Industrie, 38 (1937), 934. (A. P.-C.)

Antirachitic Potency of New Sterol Derivatives—Evaluation of the, by Tests on Rats and on the Cock's Comb. By the cock's comb test, the antirachitic potency of tuna liver oil is the same as that of vitamin D_3 and of 7-dehydrocholesterol. Vitamin D_3 is the "natural" vitamin D_3 of tuna liver oil; its provitamin is 7-dehydrocholesterol. The antirachitic potency of irradiated 22-dehydroergosterol is close to that of vitamin D_3 (determined by the cock's comb method); it may therefore be supposed that the active principle of this compound is closely related chemically to vitamin D_3 . Sterols in C_{29} cannot be considered as being natural provitamins of vitamin D_3 , as the antirachitic potency of 7-dehydrositosterols and of 7-dehydrostigmasterols are too low.—W. Grab. Hoppe-Seyler's Z. physiol. Chemie, 243 (1936), 63-89; through Chimie & Industrie, 38 (1937), 320.

Antuitrin S Intradermal Pregnancy Test. The authors report that the antuitrin S intradermal test for pregnancy, which has been so enthusiastically acclaimed, has proved, in their hands, to be valueless.—A. M. Gill and J. Howkins. *Brit. Med. J.*, 4012 (1937), 1069. (W. H. H.)

Barbituric Acid Derivatives—Addiction to. Four cases of barbiturate addiction are described. The development of tolerance, while not as marked as with some other addiction-produc-

ing drugs, leads the addict to increase the dose until clinical evidence of toxicity and pathological changes in the brain both occur. The effect of sodium-5-ethyl-5-α-methylbutylbarbiturate (pentobarbital sodium) is shown by animal experiment to be cumulative; other barbiturates presumably act in the same way. The four eases include addiction to this drug in conjunction with chloral and bromide, and following phenobarbital; to sodium-5-ethyl-5-isoamyl barbiturate (sodium amytal) following barbital; and to barbital itself. Legal control of the barbiturates to prevent self-prescription, is urged. Eight references.—G. W. Robinson, Jr. J. Missouri M. A., 34 (1937), 374; through Squibb Abstr. Bull., 10 (1037), A-1919. (F. J. S.)

Benzedrine Sulfate—Effect of, on Stomach Activity and Emptying Time. An increased activity and tonus appear after 8 minutes. The secondary effect is a marked inhibition, even cessation of activity, which sets in after 40 minutes. This may or may not be accompanied by a slight loss of tonus.—Karl H. Beyer and W. J. Meek. *Proc. Soc. Exptl. Biol. Med.*, 37 (1937), 74. (A. E. M.)

Benzedrine Sulfate—Stimulant Effect of. Benzedrine sulfate was found to depress spinal reflexes in brain-pithed frogs and to depress skeletal muscle in vitro. In doses at which epinephrine depresses respiration, benzedrine had no depressant effect.—Eldon M. Boyd. Proc. Soc. Exptl. Biol. Med., 37 (1937), 127. (A. E. M.)

Blood Pressure—Substance for Increasing. Material of kidneys or spleens is extracted with an aqueous solution of an electrolyte such as 5% sodium chloride solution and the substance reducing the blood pressure is removed from the extract by dialysis with distilled water, precipitated by saturating the solution with an albumin-precipitating salt such as sodium chloride at a $p_{\rm H}$ of 3 to 5 and extracted from the precipitate with water; pigments in the solution are precipitated by acidifying the solution, and the latter is then treated with an adsorbing agent such as kaolin and the pressor substance is extracted from the adsorbing agent by use of an aqueous solution of an alkaline substance such as secondary sodium phosphate.—Georg Hessel and Hans Maier. U. S. pat. 2,100,593, Nov. 30, 1937.

Blood Transfusions—Heparinising the Donor in. This paper is based on the result of one hundred and fifty transfusions carried out after the intravenous injection of heparin into the donor, as previously described. Such an injection reduces the coagulability of the donor's blood, but not that of the recipient. The method described fulfils all the requirements of an ideal blood-transfusion method. It allows the use of whole blood in its natural state and does not involve the use of any substances foreign to the body. It has all the advantages of the citrate methods but none of the disadvantages, such as a possible excess of anticoagulant when the expected amount of blood is not obtained. The transfusion can be performed with any equipment available and the technic should prove of value particularly in performing transfusions away from hospitals.—P. Hedentus.

Lancet, 233 (1937), 1186. (W. H. H.)

Bromide Medication. A detailed pharmacological and clinical study is reported concerning the properties of a synergistic bromide combination (neurosine). This sedative and hypnotic was chosen for investigation because of its very extensive use by physicians in sympyomatic treatment of neurasthenia, hysteria, insomnia, epilepsy, alcoholism and menopausal neuroses. Animal tests showed neurosine to be of exceedingly low toxicity, corroborating clinical reports of its safety even when abused. In a series of sixty-seven cases, relief of disturbing symptoms effected by the medication was striking. The data are tabulated and discussed in the text. Tests on the knee-jerks by an original method are described. These tests demonstrated a definite reduction in the amplitude of the knee-jerk commencing one hour after a single large dose and disappearing entirely within four hours. Contrary to reports in literature concerning the slow prolonged action of single bromides it is believed that the action of neurosine is quick and temporary. There is no "hang-over." Bromides cannot be detected in the urine after a single dose of neurosine until three hours or longer.—F. Damrau. Med. Record, 246 (1937), 445. (W. H. H.)

Cardiotoxic Vegetable Substances. XI. Constitution of Thevetin. The composition of thevetin corresponds to the formula $C_{42}H_{66}O_{18}$. It contains 3 molecules of sugar, bound to the genin. Of these sugars, 2 molecules were identified as glucose and one as a methyl ether. The glucone of thevetin, thevetigenin, $C_{23}H_{34}O_4$, is probably an isomer of digitoxygenin and of uzarigenin. From the standpoint of toxicity, digitoxygenin is the most active, followed in order by thevetigenin and then uzarigenin.—R. TSCHESCHE. Ber. deutsch. chem. Ges., 69 (1936), 2368-2372; through Chimie & Industrie, 38 (1937), 931.

(A. P.-C.)

l-Carnosine—Alleged Oxytocic Activity of. No oxytocic effect could be observed.—WILLIAM T. McClosky, Lloyd Miller, Madison Hunt and Vincent du Vigneaud. Proc. Soc. Exptl. Biol. Med., 37 (1937), 60. (A. E. M.)

Digitalin and Ouabain—Absorption of, by the Pericardium. The drugs are absorbed slowly by the pericardium. The effects of a single dose last several hours. Large doses are tolerated without toxic symptoms.—G. Baltaceanu, C. Vasiliu and A. Novac. Compt. rend. soc. biol., 123 (1936), 837–839; through Chimie & Industrie, 37 (1938), 934. (A. P.-C.)

Digitalis—Sensitivity of the Diphtheritic Heart to. The authors reviewed briefly earlier work, and by experiments upon cats verified previous observations to the effect that diphtheria toxin renders the heart more susceptible to digitalis. They point out that if it is to be employed at all in the treatment of cardiac disorders occurring early in diphtheria, it should be used with extreme caution.—Charles W. Edmunds, Ralph G. Smith and Carl A. Moyer. J. Pharmacol., 61 (1937), 286. (H. B. H.)

Ephedra—Sardinian. Sardinian ephedra contain various active principles: those of Ephedra altissima have no mydriatic action on the pupil of the dog or cat but are toxic for the frog; those of Ephedra vulgaris and Ephedra nebrodensis present the biological characteristics and chemical reactions of substances of the type of ephedrine. Chen's method is not well adapted for the extraction of the active principles of the three species. During extraction the active principles undergo marked changes.—Maria Mulas. Boll. soc. ital. biol. sper., 11 (1936), 743-744; through Chimie & Industrie, 38 (1937), 934-935.

(A. P.-C.)

Ergot—Determination of the Quality of. A critical investigation of the methods for the assay of ergot led the authors to suggest certain modifications in the Wirth method (Arch. Pharm., Vol. 1, 1936). The modification involves a more rapid procedure whereby the frequent trouble with colloids is absent. It is difficult to state specifically just what the degree of accuracy really is as the method itself is liable to certain subjective errors. During the work the authors observed that defatted ergot retains its activity while the undefatted powder deteriorates rapidly. Attention is called to the economic problem involving the increasing scarcity of ergot. The importance of the investigation of galenicals from the standpoint of the alkaloids of the ergometrine group is mentioned and the author states his hope to investigate this problem. Discussion of the various assay methods and tables of data are given.—R. Verbeke. Pharm. Tijdschr., 14 (1937), 229. (E. H. W.)

Ergotamine Tartrate—Influence of, upon Peripheral Blood Flow in Subjects with Liver Disease. Single doses of 0.5 mg. ergotamine tartrate, injected subcutaneously, produce definite vasoconstriction as indicated by a decrease in peripheral blood-flow. Less commonly, vasodilatation is produced, with increased blood-flow. No difference was observed between healthy persons and patients with liver disease.—David I. Abramson and S. S. Lichtman. *Proc. Soc. Exptl. Biol. Med.*, 37 (1937), 262. (A. E. M.)

Ethyl Alcohol and Sodium Pentobarbital—Synergism of. A potential synergism exists between pentobarbital and ethyl alcohol. The average potentiation of alcohol depression by pentobarbital is greater in small doses than with large. The rate of elimination of ethyl alcohol is unaffected by the presence of pentobarbital and the rate of elimination of pentobarbital is unaffected by the presence of alcohol. Rabbits were used as the experimental animals. The use of alcohol in the treatment of acute barbiturate poisoning appears to be not only ineffective but dangerous.—

James M. Dille and Raymond P. Ahlquist. J. Pharmacol., 61 (1937), 391. (H. B. H.)

Eupaverine in Embolism. The author states that eupaverine was introduced by Denk in 1933 for the treatment of embolism in the limbs and the pulmonary artery. Out of his twenty-five cases of embolism in the limbs, seventeen were cured, three were improved and five were failures. The site of the embolus in the seventeen successful ones was the axillary artery in one, the brachial artery in four, the aorta in one, the femoral artery in one and the popliteal artery in ten. In seven out of nine cases of pulmonary embolism complete success was obtained. The drug was given intravenously in doses of 30 mg. for peripheral embolism and 60 mg. for pulmonary embolism. According to Denk, the effect of the injection is almost immediate in pulmonary embolism, and in peripheral embolism it appears in about half an hour and lasts for about three hours, after which it is necessary to repeat the dose. The author records six cases of pulmonary embolism, of which four died, and two cases of peripheral embolism, of which one died. He concludes that eupaverine should only be used when immediate embolectomy is impossible.—P. VALDONI. Policlinico, Sez. Prat. (August 16, 1937), 1557; through Brit. Med. J., 4008 (1937), 888B. (W. H. H.)