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PHARMACEUTICAL ABSTRACTS

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ABSTRACTORS

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Eudalene—New Synthesis of. A new synthesis of eudalene has been effected starting from sodium salt of ethyl 6-methylcyclohexanone-2-carboxylate.—NRIPENDRA NATH CHATTERJEE and AMALENDU BOSE. *J. Indian Chem. Soc.*, 18 (1941), 196. (F. J. S.)

6-Hydroxy-3,4-Benzpyrene and 8-Isopropyl-1,2-Benzanthracene—Synthesis of, from 9,10-Dihydrophenanthrene. 4-Keto-1,2,3,4-tetrahydrochrysene has been converted by Reformatskiĭ reaction and dehydrogenation into 4-chryseneacetic acid and the latter cyclized with hydrogen fluoride to 6-hydroxy-3,4-benzpyrene. 8-Isopropyl-1,2-benzanthracene was synthesized by a process requiring a dehydrogenation in the last step; this was accomplished successfully with sulfur without loss of the alkyl group.—L. F. FIESER and W. S. JOHNSON. *J. Am. Chem. Soc.*, 62 (1940), 575–577. (E. B. S.)

Isoquinoline Derivatives—New Method of Synthesis of. It has been found that the Me group of 1-methylnorhydrastinine is reactive to aldehydes and ketones other than nitro aldehydes, under proper conditions.—K. N. GAIND, S. KAPOOR and J. N. RAY. *J. Indian Chem. Soc.*, 18 (1941), 213. (F. J. S.)

Malaria Parasites, Etc.—Compounds Suitable for Combating. Such products are obtained by introducing the 1-dialkylamino-4-pentyl radical into the amino group of 4-amino(benzo-1′,2′,7,8-quinolines) which are substituted in the 3- and 6′-positions by treating with a reactive ester of 1-dialkylamino-4-pentanols (for instance, their esters with hydrohalic acids or with sulfonic acids, or with their salts), or by condensing the 1-dialkylamino-4-pentanols themselves with 4-amino(benzo-1′,2′,7,8-quinolines) in the presence of agents having a condensing action.—HANS ANDERSAG and STEPHAN BREITNER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,231,844, Feb. 11, 1941. (A. P.-C.)

3-Methyl-3,4-Dihydroisoquinolines and 3-Methyl-1,2,3,4-Tetrahydroisoquinolines. A number of 3-methyl-3,4-dihydroisoquinoline and 3-methyl-1,2,3,4-tetrahydroisoquinoline derivatives are described. The substituents are 6,7-dimethoxyl, 6,7-methylenedioxyl and 6,7-dihydroxyl. The compounds are closely related to norhydrastinine and dihydronorhydrastinine. A series of general reactions for their preparation is given.—W. S. IDE and J. S. BUCK. *J. Am. Chem. Soc.*, 62 (1940), 425–428. (E. B. S.)

10-Methyl-1′,9-Methylene-1,2-Benzanthracene—*o*-Halide Synthesis of. A synthesis has been developed for 1′,9-methylene-1,2-benzanthracene and its hitherto undescribed 10-methyl- and 10-hydroxy-derivatives starting with the reaction between *o*-chlorophenylmagnesium bromide and 7-acenaphthenone. A new method was found whereby the ketonic starting material can be produced readily in quantity. This consists in the oxidation of acenaphthene with red lead in acetic acid solution, saponification of the resulting 7-acetoxy compound and oxidation of 7-acenaphthenol to 7-acenaphthenone.—L. F. FIESER and J. CASON. *J. Am. Chem. Soc.*, 62 (1940), 432–436. (E. B. S.)

Oxalenediamidoxime. II. A continuation of a previous communication and some further work is reported on the nickel complex, elucidating its structure. The preparation and properties of different types of complexes of nickel, cobalt, copper and mercury have also been described.—R. CHATTERJEE. *J. Indian Chem. Soc.*, 18 (1941), 19. (F. J. S.)

Phenols—Condensation of α-Substituted Acetoacetates with. III. The Pechmann Condensation of Ethyl α-(α-Hydroxy-βββ-Trichloroethyl)-Acetoacetate. The Pechmann condensation of phenols with ethyl α-(α-hydroxy-βββ-trichloroethyl)-acetoacetate has been studied with a view of finding the effect of —CH(OH)CCl_3 group as an α-substituent on the course of the reaction. Various coumarins containing —CH(OH)CCl_3 in 3 position and their derivatives have been synthesized. **IV. The Condensation of Cresols and Other Less Reactive Phenols with Ethyl α-(α-Hydroxy-βββ-Trichloroethyl)-Acetoacetate.** Ethyl α-(α-hydroxy-βββ-trichloroethyl)-acetoacetate has been condensed with less reactive phenols. The effect of —CH(OH)CCl_3 as α-substituent in acetoacetic ester on the course of the reaction is discussed.—D. R. KULKARDNI, R. L. ALIMCHANDANI and N. M. SHAH. *J. Indian Chem. Soc.*, 18 (1941), 113, 123. (F. J. S.)

Δ^5-Pregnenediol-3,17-one-20—Preparation of, from Δ^5-17-Ethynyl-androstenediol-3,17. Δ^5-17-Ethynyl-androstenediol-3,17 and aniline have been condensed in the presence of ether-boron fluoride and mercuric oxide catalysts to form Δ^5-pregnenediol-3,17-one-20-anil. The anil is partially hydrolyzed by contact with water to form Δ^5-pregnenediol-3,17-one-20. The acetate of this substance has been oxidized to 3-acetoxydehydroandrosterone, thus proving its pregnane structure. By saponification of Δ^5-3-acetoxypregnenol-17-one-20 with methyl alcoholic potassium hydroxide a rearranged product is obtained for which the name Δ^5-3,17-dihydroxy-18-keto-chrysopregnene is proposed. The substance is identical with the ketone obtained by the direct hydration of Δ^5-17-ethynyl-androstenediol-3,17.—H. E. STAVELY. *J. Am. Chem. Soc.*, 62 (1940), 489–491. (E. B. S.)

Procaine Hydrochloride. A method of preparing a particulate procaine hydrochloride which will remain free-flowing after heat-sterilization comprises washing commercial procaine-hydrochloride with a solvent of the group consisting of ether, petroleum benzine, acetone and ethylene dichloride, until the wash leaves no oily residue on evaporation.—WALTER G. CHRISTIANSEN and EDWARD S. HERLONG, assignors to E. R. SQUIBB & SONS. U. S. pat. 2,224,181, Dec. 10, 1940. (A. P.-C.)

Proflavine—Synthesis of, from *m*-Phenylenediamine and Its Derivatives. In a continuation of the investigation of the mechanism of the reaction whereby proflavine is obtained in good yields by condensing *m*-phenylenediamine, glycerol and formic acid. Albert presents evidence that the dihydrochloride of (anhydro)-2:4:2′:4′-tetraaminobenzhydrol is the immediate precursor of proflavine.—ADRIEN ALBERT. *J. Chem. Soc.*, (1941), 484–487. (W. T. S.)

Pseudotropine—Hydrohalides of the Benzilic Acid Ester of. By reaction of pseudotropine with benzilic acid, the ester is obtained, having the formula $C_{22}H_{25}O_3N$, which melts at 156° to 158° C., forms a hydrochloride (melting at about 232° C.), a hydrobromide and a hydroiodide, and is suitable for therapeutic use.—OTTO WOLFES and OTTO HROMATKA, assignors to MERCK & CO., INC. U. S. pat. 2,235,661, March 18, 1941. (A. P.-C.)

Pyrazolone Derivatives—New. In attempting to make acetamino-ethoxyphenyl derivatives of antipyrine, the following new compounds have been prepared: 3,4-dinitrophenetole; 4-acetamino-

3-aminophenetole; 3,4-diacetamino-phenetole; 2-ethoxy-5-nitrophenyl-hydrazine; benzaldehyde hydrazone of the latter; acetone hydrazone; 1-(2-ethoxy-5-nitrophenyl)-3-methyl-5-pyrazolone. Procedure for each of these is described.—FRANCOIS X. DEMERS and E. V. LYNN. *Jour. A. Ph. A.*, 30 (1941), 627. (Z. M. C.)

Pyridine and Quinoline Amines—Sulfanylyl Derivatives of. An improved method of preparation of the 5,7, and 8-aminoquinolines as well as the preparation of certain sulfanilamido derivatives of pyridine and quinoline amines is described.—R. WINTERBOTTOM. *J. Am. Chem. Soc.*, 62 (1940), 160–161. (E. B. S.)

Pyrimidine-Thiazole Chloride Hydrochloride. Vitamin B_1 chloride is produced by condensing a thiazole hydrochloride with a 2-alkyl-6-amino-5-alkyoxymethylpyrimidine hydrochloride or a 2-alkyl-6-amino-5-hydroxymethylpyrimidine hydrochloride. Various examples with details of procedure are given.—OTTO ZIMA, assignor to MERCK & CO., INC. U. S. pat. 2,235,862, March 25,1941. (A P.-C.)

Quinoline Derivatives. VI. A pyridino-quinoline derivative has been synthesized. The condensation product of $\alpha\alpha'$-phenylcarbamylacetone dicarboxylate and an aromatic amine could not be cyclized to a piperidine derivative.—DEBABRATA DAS-GUPTA and TEJENDRA NATH GHOSH. *J. Indian Chem. Soc.*, 18 (1941), 120. (F. J. S.)

Quinoline Series—Basic Double Ethers of the. Various details are given of the production of compounds suitable for combating blood parasites.—HEINRICH JENSCH, assignor to WINTHROP CHEMICAL CO. U. S. pat. 2,228,166, Jan. 7, 1941. (A. P.-C.)

Saponaceous Formaldehyde Preparations of High Stability. Forty per cent aqueous formaldehyde is added to preparations of sulfonated castor oil or sulfonated, acetylated, unsaturated OH-acids which have been neutralized with aqueous ammonia.—G. LUSIGNANI and A. MOSSINI. *Boll. chim.-farm.*, 79 (1940), 101–102; through *J. Soc. Chem. Ind.*, 59 (1940), 427. (E. G. V.)

Spasmolytic and Analgetic Esters. Basic esters of the general formula $R'R''R'''CCO_2\ R''''$ (where R' and R'' stand for aryl radicals, R''' stands for a tertiary amino alkyl radical and R'''' stands for an alkyl or aralkyl radical), being colorless oils which are immiscible with water and which may be transformed into water-soluble salts by means of acids, are obtainable by causing a nitrile of the general formula $R'R''CHCN$ (where R' and R'' stand for aryl radicals, for instance, the diphenyl acetonitrile or the derivatives of such nitriles substituted in the phenyl nucleus) to react with a basically substituted alkyl halide (for instance, piperidylethyl chloride, diethylaminoethyl chloride and the like), in the presence of agents splitting off hydrogen halide (for instance, alkali, sodamide or alkali alcoholate), and by transforming the tertiary nitriles thus obtained into the corresponding esters. The basic esters may likewise be obtained by causing a metal compound of the general formula $R'R''C(M)CO_2R$ (where R' and R'' stand for aryl radicals, which may be connected with each other, M stands for an alkali metal and R stands for an alkyl or aralkyl radical) to react with a basically substituted halogen alkyl, for instance, piperidethyl chloride, diethylaminoethyl chloride, morpholinylethyl chloride and the like. Details are given of the production of a number of such compounds.—MAX BOCKMÜHL and GUSTAV EHRHART, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,230,774, Feb. 4, 1941. (A. P.-C.)

Stilbene—Methods for the Preparation of Derivatives of. II. Unsymmetrical Stilbenes. 4-Hydroxy-4′-methoxystilbene and 4-methoxy-4′-acetoxy-stilbene have been prepared and the estrogenic activity of the former has been compared with 4:4′-dihydroxy-, 4:4′-dimethoxy- and 4:4′-diacetoxy-stilbenes. One free hydroxyl group in the *p*-position only appeared to be essential for activity. Many new compounds were obtained as intermediates, namely, β-[*p*-methoxyphenyl]-*p*-hydroxycinnamic acid and the corresponding methyl methoxy ester and methoxy-acid; *p*-hydroxy-, *p*-acetoxy- and *p*-methoxy-α:β-diethylcinnamic acids and esters; ethyl α:β-diethyl-β-hydroxy-β-[*p*-acetoxyphenyl]-propionate and the corresponding *p*-methoxyphenyl compound; β-bis[*p*-methoxyphenyl]-acrylic acid; and α-bromo-α-ethyl-β-[*p*-methoxyphenyl]-$\Delta^{\beta,\gamma}$-valeric acid. In the stilbene molecule only one hydroxyl in the *p*-position is necessary for estrogenic activity.—W. H. LINNELL and H. S. SHAIKMAHAMUD. *Quart. J. Pharm. Pharmacol.*, 14 (1941), 64–72. (S. W. G.)

Sulfanilamide Derivatives. V. Constitution and Properties of 2-Sulfanilamidopyridine. The preparation of 2-sulfanilamidopyridine and its physical and chemical properties are described. Evidence in favor of the accepted formula for this compound is given.—M. L. CROSSLEY, E. H. NORTHEY and M. E. HULTQUIST. *J. Am. Chem. Soc.*, 62 (1940), 372–374. (E. B. S.)

Sulfanilamidopyridines—Preparation of. A method of preparing sulfanilamidopyridines and *p*-nitrophenylsulfonamidopyridines involves the reaction of an aminopyridine with an *N*-acylsulfanilyl chloride or *p*-nitrobenzenesulfonyl chloride in the presence of dioxane, suitably with heating to 95° C.—ELMORE H. NORTHEY and MARTIN E. HULTQUIST, assignors to AMERICAN CYANAMID CO. U. S. pat. 2,245,292, June 10, 1941. (A. P.-C.)

Sulfonamides. II. Anilino-, toluidino-, anisidino-, phenetidino-, xylidino-, 5-aminoquinolino-, anisidino-acetylamino-*p*-benzenesulfonamides and various allied products have been synthesized for studying their therapeutic activity and toxicity.—K. N. GAIND, R. P. SEHGAL and J. N. RAY. *J. Indian Chem. Soc.*, 18 (1941), 209. (F. J. S.)

Ultraviolet Light—Splitting the CONH Linkage by Means of. The absorption spectra of stearic anilide, benzylstearylamine and β-phenylethylstearylamine have been determined and the molecular areas when spread on hydrochloric acid solution have been measured. These substances as monolayers have been irradiated at 2483 and 2537 Å. and found to undergo photolysis at the CONH linkage. The products of the reaction have been identified by film potential measurements (stearic acid) and colorimetric tests (aniline) and as crystalline picrates (benzylamine and β-phenylethylamine). It seems reasonable to expect that peptides and proteins containing amino acids with light-absorbing side chains may undergo breakage of the adjacent CONH linkages in an analogous manner.—D. C. CARPENTER. *J. Am. Chem. Soc.*, 62 (1940), 289–291. (E. B. S.)

Vitamin E—Chemistry of. Some New *p*-Hydroxy-Coumarans and -Chromans. To study the behavior at the dropping mercury electrode of substances related to tocopherol, the authors have prepared 2 new hydroxy-coumarans and 2 new hydroxy-chromans. 5-Hydroxy-4,6,7-trimethylcoumaran was obtained from this carbinol which in turn was prepared either by treating the corresponding amine (from 3,6-dimethoxy-2,4,5-trimethylbenzylcyanide) with HNO_2 or through the Grignard and ethylene oxide from bromopseudocumohydroquinone dimethyl ether which resulted from the reduction of bromopseudocumoquinone followed by methylation. 5-Hydroxy-2,2,4,6,7-pentamethylcoumaran was prepared by refluxing ethyl 3,6-dimeth-

oxy-2,4,5-trimethylphenylacetate with CH_3MgI followed by decomposition in the usual way. 6-Hydroxy-2,2,5-trimethyl-7,8-benzochroman and 5-hydroxy-2,4-dimethyl-6,7-benzocoumaron were prepared by a series of reactions previously reported (Smith, *et. al., J. Org. Chem.*, 4 (1939), 323 and Smith and MacMullen, *J. Am. Chem. Soc.*, 58 (1936), 629).—LEE IRVIN SMITH, STANLEY WAWZONEK and HENRY C. MILLER. *J. Org. Chem.*, 6 (1941), 229–235. **Oxidation of Hydroquinones, *p*-Hydroxychromans and *p*-Hydroxycoumarans with Ceric Sulfate.** As in the case with hydroquinones, 6-hydroxychromans and 5-hydroxycoumarans may be analyzed volumetrically with ceric sulfate in 50% ethanol. Ceric sulfate also oxidizes these compounds to the corresponding quinones and hydroquinones in good yields.—LEE IRVIN SMITH, P. M. RUOFF and STANLEY WAWZONEK. *J. Org. Chem.*, 6 (1941), 236–241. (W. T. S.)

BIOCHEMISTRY

Acetone Bodies—Formation of, from Acetic Acid. By the use of the heavy isotope of carbon, C^{13}, it has been shown that acetic acid takes part in the synthesis of the acetone bodies in the fasting rat. In a similar manner it has been shown that the carbon of sodium bicarbonate does not enter the acetone bodies formed by the fasting rat.—MARIAN E. SWENDSEID, RICHARD H. BARNES, ALLAN HEMINGWAY and A. O. NIER. *J. Biol. Chem.*, 142 (1942), 47. (F. J. S.)

Adrenal Cortical Hormone—Purification of Extracts Containing. A method of obtaining the adrenal cortical hormone from aqueous extracts containing the hormone together with impurities of the order of lipidlike substances and adrenaline comprises producing in the aqueous extracts a precipitate on which the impurities are adsorbed by introducing into the extracts a ferrous compound, removing the precipitate together with the impurities adsorbed thereon, extracting the aqueous solutions with organic solvents immiscible with water and evaporating the solvent from the hormone dissolved therein.—ALOIS DETZEL, assignor to CHEMISCHE FABRIK PROMONTA G. M. B. H. U. S. pat. 2,228,561, Jan. 14, 1941. (A. P.-C.)

Allantoin—Estimation of, by the Rimini-Schryver Reaction. A method for the colorimetric estimation of allantoin in urine has been described, based on the Rimini-Schryver reaction as applied to glyoxylic acid. Some of the variables in this reaction have been studied and its specificity determined as applied to urine. By this method allantoin added to urine and in pure solution is estimated with an error of ±5% and between 0.02 and 0.4 mg. is required for a determination.—E. GORDON YOUNG and CATHERINE F. CONWAY. *J. Biol. Chem.*, 142 (1942), 839. (F. J. S.)

Allopregnanol-3(β)-one-20—Isolation of, from Human Pregnancy Urine. Allopregnanol-3(β)-one-20 is a constituent of human labor and delivery urine. The presence of limited amounts of 17-ketosteroids in the digitonin-precipitable fraction is indicated. A detailed method for the isolation of allopregnanol-3(β)-one-20 is given.—W. H. PEARLMAN, GREGORY PINCUS and NICOLAS T. WERTHESSEN. *J. Biol. Chem.*, 142 (1942), 649. (F. J. S.)

Allyl-β-Tocopherol. An allyl halide is treated with β-tocopherol in the presence of an acid-condensing agent such as zinc chloride, suitably in benzine at the boiling point of the reaction mixture.—PAUL KARRER, assignor to HOFFMANN-LA ROCHE, INC. U. S. pat. 2,245,480, June 10, 1941. (A. P.-C.)

Amino Acids. A product suitable for parenteral therapeutic administration is prepared by a process which involves treating a protein, such as casein, containing all of the indispensable amino acids with an aqueous solution of a mineral acid under hydrolyzing conditions, separately treating an additional quantity of such protein with an aqueous solution of an alkali under hydrolyzing conditions, combining the reaction mixtures after the hydrolysis has taken place in such proportions that the final product is substantially neutral and separating the amino acids in the reaction product from the mineral acid salt.—MELVILLE SAHUYN. U. S. pat. 2,241,927, May 13, 1941. (A. P.-C.)

Ammonia—Formation of, from Proteins in Alkaline Solution. Data are presented on the rate and extent of the formation of ammonia at 25°, 35°, 68° and 100° in various concentrations of NaOH from egg albumin, β-lactoglobulin and edestin. In all three proteins the amount of ammonia formed exceeded that due to the known sources which are amide groups and arginine. The rate of formation of this excess of ammonia can be most simply accounted for by assuming that it is produced from two independent sources by first order reactions. Only a fraction of the unknown ammonia can be obtained by treating an acid hydrolysate of egg albumin with alkali. It is concluded that the ammonia is not formed by decomposition of any amino acid as such, but it is referable to alkali-labile groups that exist in the protein.—ROBERT C. WARNER and R. KEITH CANNAN. *J. Biol. Chem.*, 142 (1942), 725. (F. J. S.)

Anterior Pituitary—Separation of Thyrotropic and Interstitial Cell Stimulating (Luteinizing) Hormones of. The interstitial cell stimulating hormone can largely be freed from thyrotropic activity when precipitated repeatedly between 0.25 and 0.35 saturation of ammonium sulfate and the thyrotropic factor is precipitated mainly between 0.35 and 0.5 salt saturation. These results speak against the identity of both hormones.—H. JENSEN and SIBYLLE TOLKSDORF. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 223. (A. E. M.)

Antibiotin Factor—Isolation of, from Egg White. An antibiotin factor, a basic protein (isoelectric point *p*H 10) which combines firmly with biotin, has been isolated from egg white. The preparations were 15,000 times more active than egg white and were homogeneous in electrophoresis and sedimentation experiments. Some chemical and biological properties of the protein have been investigated. The protein is similar in biological activity to the substance isolated from egg white by Eakin, Snell and Williams (*J. Biol. Chem.*, 136 (1940), 801) and called by them avidin.—D. W. WOOLLEY and L. G. LONGSWORTH. *J. Biol. Chem.*, 142 (1942), 285. (F. J. S.)

Ascorbic Acid in Blood—Photometric Determination of. A Leifo photometer was used with an error of not more than 1%. The property of ascorbic acid to decolorize methylene blue in the presence of light was used to detect its presence in blood and plasma; and 10% metaphosphoric acid was found preferable to trichloracetic or sulfosalicylic acid for deproteinating blood. The *p*H should range between 2 and 3. A 1:10,000 solution of methylene blue was used as the indicator, and a 1:10,000 solution of ascorbic acid in double distilled water was used as the standard. A 30% solution of lead acetate was used to clear the samples. Yellow light with an irradiation of 6 to 9 min. gave the best results. The coefficient of extinction is in direct proportion to the concentration of ascorbic acid. The mean coefficient for 10γ is 12.7.—EUGENIO E. VONESCH and CARLOS A. ZIMMAN. *Anales farm. bioquím*, 12 (1941), 1 (G. S. G.)

Bile Extracts—Photometric Determination of, and a Standardization of Their Content of Active Prin-

ciples. It was thought interesting to study the determination of bile acids photometrically. This method permits the preparation of extracts of a uniform potency, possessing a constant and well-determined activity. The preparation of an extract merely on a basis of the weight of the bile used without regard to the content of bile salts seemed illogical since extracts prepared according to Swiss Pharmacopœia directions may vary widely. The determination is based on a reaction originally proposed by HERZFELD and HAEMERLI (*Schweiz. Med. Wochschr.*, Nos. 6, 7 (Feb. 1924), 141). In the reaction furfurol is added and reacts with the bile acid in the presence of 84% phosphoric acid. The test is carried out as follows: Into a tube of the proper size with a ground in stopper, are pipetted 1 cc. of an 80% alcoholic solution of the substance to be determined or of the standard, 0.5 cc. of a 1% solution of furfurol in absolute alcohol and then 3.5 cc. of 84% phosphoric acid from a burette directly into the center of the tube without wetting the walls. The mixture is agitated vigorously without inverting the tube and then immersed for 5 min. in a boiling water bath. The tube is then immersed in cold water for 5 min. and 5 cc. acetic acid (glacial) is added to bring the volume up to 10 cc. The mixture is then mixed by inverting the tube 10 to 20 times when a blue-green to blue color is obtained depending on the concentration. The quantities of materials must be carefully followed and should be added from pipettes. At the end, a few minutes are necessary to allow bubbles of air to rise before placing in the photometer. The color is not stable indefinitely and is sensitive to light. Several hours' exposure to sunlight gives nearly complete decolorization whereas an identical sample stored in the dark slowly lost its color over about 15 days. However, since the color is determined immediately after running the test, no difficulty is encountered. Control tests made with other constituents of the bile such as cholesterol, choline, lecithin, oleic acid, stearic acid and biliverdin indicated that these substances even in amounts above normal did not interfere. The author suggests the standardization of bile extracts at about 50% of bile acid derivatives present in fresh bile, the amount of lactose varying with the bulk of the other constituents present. The exact technique of making the photometric measurements is described in detail. The color was found to be proportional to the concentration. The optimum wave length of the light was found to lie in the range 450–510 mμ and a filter was chosen accordingly. The methods of calculating percentage expressed as cholic acid, sodium glycocholate or sodium taurocholate are given. A standard and a modified method of preparing the extract are suggested.—R. FREUDWEILER. *Pharm. Acta. Helv.*, 16 (1941), 21–35. (M. F. W. D.)

Biotin—Isolation of, from Milk. A method for the isolation of pure crystalline biotin from a milk concentrate has been described. The yield of biotin is 25% to 40%. The method is suitable for the preparation of relatively large amounts of pure biotin.—DONALD B. MELVILLE, KLAUS HOFMANN, ELEANOR HAGUE and VINCENT DU VIGNEAUD. *J. Biol. Chem.*, 142 (1942), 615. (F. J. S.)

Blood and Plasma—Military Use of Stored. Blood and blood substitutes made available through a blood bank have a demonstrated value in the treatment of civilians and of wounded soldiers. Blood substitutes have been found wanting in certain conditions, and particularly in shock due to hemorrhage. The author states that dried plasma has been shown to have less value than plasma from which the water has not been removed. Since the primary factor in the treatment of shock is time, blood and plasma should be available in war time farther forward than the casualty clearing station. Whole blood cannot be made available any closer to the front than the last point where refrigeration is possible, and beyond this point some blood substitute must be used. Unmodified plasma would appear to be the simplest and most satisfactory substance to use for this purpose. In the author's opinion, clinical tests have indicated the superiority of plasma with sufficient clearness so that there should be little hesitancy in making a choice.—R. C. HARDIN. *J. Iowa State Med. Soc.*, 31 (1941), 158; through *Abbott Abstract Service*, (1941), No. 940. (F. J. S.)

Blood Coagulation—Delayed, in Methyl Methacrylate (Boilable "Lucite") Vessels. The blood coagulation time in methyl methacrylate tubes was found to be twice as long as found in glass tubes. The coagulation inhibiting effect of this material follows Lampert's rule of surface adhesion.—JOHN S. HIRSCHBOECK. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 311. (A. E. M.)

Blood Iodine—Fractionation of Cattle, with Alcohol. It was found that that fraction of blood iodine which is insoluble in cold alcohol can be made partially soluble in cold alcohol (*i. e.*, alcohol at room temperature) by the application of dry heat to the residue and completely soluble in cold alcohol after the application of heat and alcoholic sodium hydroxide to the residue. It is suggested that varying degrees of influence of heat and hydrolysis may account at least in part for the discrepancies in reported values of hot alcohol-soluble iodine of blood.—ELDON M. BOYD and ELEANOR L. CLARKE. *J. Biol. Chem.*, 142 (1942), 619. (F. J. S.)

Blood Magnesium—Determination of. A spectrochemical method for the determination of magnesium in blood which is accurate, rapid and simple to perform and requires only 1 cc. of sample is described. The blood samples are first diluted with a potassium alum solution which acts both as a spectroscopic buffer and internal standard and then atomized into a spark between graphite electrodes. Analysis is made by measurement of relative intensities. The average error obtained by this method is less than 3%.—F. W. LAMB. *Ind. Eng. Chem. Anal. Ed.*, 13 (1941), 185–187. (E. G. V.)

Blood Prothrombin—Determination of. This describes the technique of determining the time and concentration of prothrombin necessary for clotting; also the influence of various factors and the reagents employed. Plasma is separated from fresh blood by centrifuging with sodium oxalate. Prothrombin is adsorbed from this plasma by barium sulfate. Thromboplastin extracted from rabbit brain with sodium chloride is added, together with calcium chloride, and the time of coagulation noted. The best results are obtained at *p*H 6 to 8; barium sulfate is a better adsorbent than any of the twelve others tested. The time of coagulation in the presence of thromboplastin and calcium is inversely proportional to the concentration of the prothrombin. Time and concentration are plotted on a curve and an equation based on this curve is used for calculating the concentration of prothrombin in normal or pathologic blood.—C. A. TANTURI and R. F. BANFI. *Anales farm. bioquím*, 9 (1940), 83. (G. S. G.)

Butter—Deterioration of, During Storage. I. Development of Fishiness. Fishiness in butter is due to oxidation of lecithin to yield trimethylamine. The development of the taint is favored by traces of heavy metals and high acidity of butter serum. The mechanism of the oxidizing action is the same as in the Fenton reaction (hydrogen peroxide in the presence of ferrous salts), trimethylamine being formed from the choline residue of lecithin.

Ammonia to the extent of a quarter of the volatile base nitrogen is also formed. The growth of *Oidium* and related molds also cause fishiness in butter. The fishy flavor of milk due to feeding betaine-containing sugar beet by-products is due to reduction of trimethylamine oxide entering the milk.—W. L. DAVIES. *Ind. & News Ed., J. Indian Chem. Soc.*, 4 (1941), No. 1, 1. (F. J. S.)

Carbon Monoxide Concentration of the Blood in Disease. Carbon monoxide concentration for normal blood was found to be 0.3–1.0% of the total hemoglobin concentration. Higher values were observed in some pathological conditions, especially diabetes mellitus (up to 6.23%).—A. GIGON and M. NOVERRAZ. *Schweiz. med. Wochschr.*, 70 (1940), 836–837. (F. S. M.)

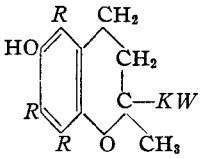
Carbon Suboxide and Proteins. V. Further Study of the Nature of the Reaction. The authors give the following summary: (1) The free amino and tyrosine phenolic groups of serum albumin appear to have completely reacted with malonic acid after the addition of 3 times the theoretical amount of carbon suboxide. (2) Carbon suboxide, unlike ketene, reacts with free amino and tyrosine phenolic groups at approximately the same rate. (3) Malonic acid bound to tyrosyl phenolic groups is labile even in the cold and is quite rapidly hydrolyzed under physiological conditions. (4) Substituted tyrosine derivatives develop with the phenol reagent less than the theoretical intensity of the color equivalent to their tyrosine content.—ANN H. TRACY and WILLIAM F. ROSS. *J. Biol. Chem.*, 142 (1942), 871. (F. J. S.)

Carotene—Determination of Pure, in Green Vegetables and Green Feeds. Considerable loss of carotene occurred when fresh foods (with the exception of parsley, okra and carrots) were ground raw for analysis. This loss was inhibited in most of the foods by cooking them before grinding or by grinding them while they were covered with alcohol. The fresh samples after grinding contained 63% to 127% of the carotene contained in the alcohol-treated samples; cooked samples after grinding contained 37% to 132% of the carotene in the alcohol-treated samples. The carotene content of sweet potatoes, Bermuda grass, 2 out of 3 samples of carrots and 2 out of 5 samples of spinach was less in the cooked than in the raw material. Fresh Bermuda grass and clover stored for 7 days in methanol lost only slight amounts of carotene. The following method is proposed for the preservation and preparation of green foods: for samples to be analyzed soon after gathering (1 to 2 hrs.) place 100 Gm. in a large evaporating dish, soak in 100 cc. of 95% alcohol for 5 min. and cut up with scissors; add 100 Gm. of clean white sand free from organic matter, and grind to a uniform mixture; for samples necessitating shipment or delay in analysis place approximately 100 Gm. in a tared fruit jar with a weighed quantity of alcohol and seal the jar with a jar rubber under the lid; on arrival at the laboratory weigh the jar and contents, subtract the weight of the jar and alcohol to give the weight of the sample, pour the contents into an evaporating dish, cut with scissors and grind with sand as above; decant the liquid from the solid part through cheese cloth and make to definite volume, weigh the solids and take aliquots of both solid and liquid equivalent to 5 Gm. of fresh untreated material, and mix together for carotene determination; saponify by boiling 30 min. in 50 cc. of 12% alcoholic potash, cool, add 50 cc. of petroleum ether, decant the liquid into a separatory funnel, grind the residue in a mortar with 15 cc. of petroleum ether and then with a mixture of 5 cc. of 95% alcohol and 15 cc. of petroleum ether until no more color is extracted; then proceed according to the A. O. A. C. method (*J. Assoc. Official Agr. Chem.*, 22 (1939), 79–81) for crude carotene. For sweet potatoes, boil 30 min. with 50 cc. of 95% alcohol instead of 12% alcoholic potash. For pure carotene, shake 50 cc. of crude carotene solution with 2.5 Gm. of activated magnesium carbonate, X reagent (*J. Assoc. Official Agr. Chem.*, 22 (1939), 190–194). This method is applicable to green leafy vegetables and feeds.—G. S. FRAPS, W. W. MEINKE and A. R. KEMMERER. *J. Assoc. Official Agr. Chem.*, 24 (1941), 739–744. (A. P.-C.)

Chloral Hydrate, Chloroform and Related Substances in Blood—Determination of. Using the Fugiwara color reaction as a basis, the author describes a method for the quantitative determination of chloral hydrate, chloroform and related compounds in blood. This new procedure avoids the simultaneous preparation of standard solutions by using the photoelectric colorimeter and proper calibration curves. Alcohol is used to stabilize the color, making the method rapid and accurate.—LLOYD W. ADAMS. *J. Pharmacol.*, 74 (1942), 11–17. (H. B. H.)

Choline—Importance of, in Synthetic Rations for Dogs. In addition to thiamine, riboflavin, nicotinic acid, pyridoxine and pantothenic acid, young puppies also require choline and probably other factors of the vitamin B complex for normal growth.—A. E. SCHAEFFER, J. M. MCKIBBIN and C. A. ELVEHJEM. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 365. (A. E. M.)

Chromans of Vitamin E Activity—Substituted. A process for the production of compounds of the general formula



(in which R is a methyl radical or two of the radicals R are methyl radicals and the remaining R is hydrogen, and KW is any univalent higher hydrocarbon radical) involves treating a dihydrocoumarin with a mixture of methylmagnesium halide and the Grignard compound from a higher halogenated hydrocarbon, and separating the chroman thus obtained. Various details of procedure are given.—WALTER JOHN and PHILIP GÜNTHER, assignors to MERCK & CO. U. S. pat. 2,245,147, June 10, 1941. (A. P.-C.)

Cortical Hormone—Substances Having the Efficacy of the. *2,230,772*—Compounds such as progesterone, acetoxyprogesterone or pregnenolone are treated (suitably in glacial acetic acid or in benzene) with a reagent such as lead tetraacetate causing the transformation of the methyl group into $HOCH_2$ group, as by heating at 75° to 85° C. for about 7 hrs. Several examples with details of procedure are given. *2,230,773*—This patent relates to effecting similar reactions with other lead compounds such as lead tetrabutyrate, tetrapalmitate or tetrapropionate, tetrabenzoate, tetrakisphenylacetate, tetratoluate or tetraanisate or the like.—MAX BOCKMÜHL, GUSTAV EHRHART, HEINRICH RUSCHIG and WALTER AUMÜLLER, assignors to WINTHROP CHEMICAL CO. U. S. pats. 2,230,772 and 2,230,773, Feb. 4, 1941. (A. P.-C.)

Corticosterone—Studies on the Partial Synthesis of. I. A new route has been suggested to the preparation of sterol derivatives having a hydroxyl group C_{11}. Progress in this direction has been reported. By the debromination followed by saponification of 3-acetoxy-11-bromo-12-ketocholanic acid, an unsaturated acid has been prepared which

proved to be 3-hydroxy-12-keto-9,11-cholenic acid. This acid melts at 173° and possesses the characteristic absorption of the α,β-unsaturated keto derivatives. The crude semicarbazone of the unsaturated keto-hydroxy acid, m. p. 221°, has been reduced according to the Wolf-Kishner method to 3-hydroxy-9,11-cholenic acid of melting point 183–184°.—P. N. CHAKRAVORTY and E. S. WALLIS. *J. Am. Chem. Soc.*, 62 (1940), 318–320. (E. B. S.)

Cosmetic Aspects of Nutritional Deficiencies. A review of vitamin, chemical and mineral deficiencies is presented.—L. STAMBOVSKY. *Drug Cosmetic Ind.*, 50 (1942), 150–153. (H. M. B.)

Cystine—Polarographic Estimation of, in Urine. The author summarizes his work as follows: (1) From 40 to 80 mg. of free cystine are found polarographically in the 24-hr. urine samples of twenty healthy persons. (2) The presence of cystine complex in urine from which cystine is liberated by acid hydrolysis or treatment with ammoniacal cobalt buffers is demonstrated. (3) Polarographic and colorimetric values for cystine show correlation within the limits of error in unhydrolyzed and hydrolyzed urine.—GERALD REED. *J. Biol. Chem.*, 142 (1942), 61. (F. J. S.)

Cytochrome C—Quantitative Determination of. The following summary is given: (1) A new method for the determination of cytochrome *c* in small quantities of tissue from experimental animals has been developed. (2) Existing methods of extraction are used in the method but concentration is effected by precipitation with trichloroacetic acid. (3) The analysis is based on the quantitative spectrophotometric measurement of the change in extinction when cytochrome *c* is oxidized and reduced by specific enzymes. (4) Results obtained with normal rat tissues and chick embryo are given.—V. R. POTTER and K. P. DUBOIS. *J. Biol Chem.*, 142 (1942), 417. (F. J. S.)

Diodrast and Iodides—Direct Photoelectric Colorimetric Method for the Determination of, in Blood and Urine. A method is described for the determination of diodrast and iodides by a simple, rapid, direct colorimetric procedure which possess great sensitivity. In this method diodrast iodine in urine or filtrates of blood plasma is oxidized to iodate by bromine; excess bromine is removed by phenol; elementary iodine is liberated by potassium iodide; and the yellow color of the free iodine, after intensification, is measured in a photoelectric colorimeter. Sensitivity in color absorption is achieved by the use of filters allowing maximum light transmission at either 400 or 365 mμ. By this procedure plasma diodrast iodine concentrations as low as 1 mg. % may be determined with a high degree of accuracy.—JACK FLOX, ISADORE PITESKY and ALF S. ALVING. *J. Biol. Chem.*, 142 (1942), 147. (F. J. S.)

Estrogenic Hormones. A review of the development of hormone therapy, with special reference to ovarian hormones, is presented. The chemistry of estrone, estriol and estradiol is discussed and also the preparation of synthetic hormones or chemical compounds which have hormone activity. Clinical tests indicate that stilbestrol is a satisfactory substitute for natural estrogens, is active orally and is cheaper, although the oral route requires a larger dose than the parenteral. In the human the anterior pituitary stimulates development of two ovarian hormones estrone and lutein relative to the menstrual cycle. These may be administered parenterally or by implantation of crystals, subcutaneously. This latter method is still experimental.—OSCAR A. ROSSI. *Rev. Med. Cienc. Afin.*, 2 (1940), 871. (G. S. G.)

Estrogenic Hormones—Concentration of, in Blood Serum and Blood Cells. The blood serum and cells of a group of 20 non-pregnant women were examined for their estrogenic hormone content after intravaginal application of pellets made from desiccated material. The blood cells contained 2 or more times as many rat units per unit volume as the blood serum.—AMERICO S. ALBRIEUX. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 381. (A. E. M.)

Foodstuffs—Liquefaction of Solid, for Diets. The following conclusions are given: (1) A colloid mill is of inestimable help in the preparation of special diets for sick and convalescent patients. It makes foods more assimilable and a more varied dietary possible. (2) Therapeutic feeding in various conditions is facilitated. (3) The role of the colloid mill in future dietetic research should be an important one.—A. A. NEURWIRTH. *The Military Surgeon*, 89 (1941), 898. (F. J. S.)

Glycogen Content of the Human Liver. The glycogen content of biposy samples of normal livers was highest in patients which were fed glucose prior to operation. When exhaustive liver damage was present, liver glycogen was low despite glucose feeding.—DUGALD S. MACINTYRE, SVEND PEDERSEN and WALTER G. MADDOCK. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 354. (A. E. M.)

Gonadotropic Hormone of Urine of Pregnancy. IV. A highly purified urinary gonadotropin preparation assaying 4000 minimal effective (Friedman) doses per mg. was found to be essentially monodisperse and yielded the following data. The sedimentation constant, $S_{20} = 4.3 \times 10^{-13}$ cm. per second per unit field. The diffusion constant, $D_{20} = 4.4 \times 10^{-7}$ sq. cm. per second. The partial specific volume is 0.76. The molecular weight is in the neighborhood of 100,000.—HAROLD P. LUNDGREN, SAMUEL GURIN, CARL BACHMAN and D. WRIGHT WILSON. *J. Biol. Chem.*, 142 (1942), 367. (F. J. S.)

Inositiol in Chick Nutrition. Lack of inositol in the diet causes inhibition of growth but no other pathological symptoms.—D. M. HEGSTED, G. M. BRIGGS, R. C. MILLS, C. A. ELVEHJEM and E. B. HART. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 376. (A. E. M.)

Insulin—Therapeutic Product from. A product suitable for use by subcutaneous or intramuscular injection is obtained by mixing insulin and the reaction product of formaldehyde or its polymers with guanidine or its salts or decamethylenediguanidine or 4,4′-diguanidodiphenyl dihydrochloride.— GEO. B. WALDEN, assignor to ELI LILLY AND CO. U. S. pat. 2,219,350, Oct. 29, 1940. (A. P.-C.)

Kidney Phosphatases—Note on the Separation of. The acid and the alkaline phosphatases of kidney cortex extracts have been separated as two distinct protein fractions.—GERTRUDE E. PERLMANN and RONALD M. FERREY. *J. Biol. Chem.*, 142 (1942), 513. (F. J. S.)

Lactic Acid Containing Radioactive Carbon in the α or β Position—Metabolism of. The following summary is given: (1) Rats have been fed solutions of lactate containing C^{11} in the α and β positions and the radioactivity of the expired CO_2 and liver glycogen has been measured. (2) The CO_2 expired in 2.5 hrs. accounted for about 10% of the administered radioactivity. (3) The liver glycogen formed averaged 21% of the lactate fed and contained 3.2% of the radioactivity. (4) The results are compared with those obtained with lactate labeled in the carboxyl position and are found to be compatible with the previously suggested mechanism of glycogen formation in the liver after lactate feeding.—BIRGIT VENNESLAND, A. K. SOLOMON, JOHN M. BUCHANAN, RICHARD D. CRAMER and A. BAIRD HASTINGS. *J. Biol. Chem.*, 142 (1942), 371. (F. J. S.)

Lactogenic Hormone. A method of preparing a substantially pure lactogenic product comprises subjecting the pituitary gland of animals to extraction with an aqueous alkaline solution, adjusting the *p*H of the alkaline extract thus obtained to about 8.0 to 8.3 by addition of dilute acid, precipitating the non-lactogenic proteins contained in the extract by addition of an aqueous solution containing a water-soluble alkaline earth salt and separating the supernatant liquid containing the lactogenic hormone from the non-lactogenic precipitate formed upon addition of the alkaline earth salt.—EVERETT I. EVANS. U. S. pat. 2,211,411, Aug. 13, 1940. (A. P.-C.)

Lead and Arsenic Ingestion and Excretion in Man. Report of an investigation to determine the maximal quantities of lead and arsenic ingested and excreted daily by orchardists eating apples that had been sprayed with lead arsenate. Analyses of the fruit showed a daily intake of 1–26 mg. lead and 0.34–6.8 mg. arsenic per person. Daily analyses of urine and feces showed an output of 22.3 mg. and 2.43 mg. lead and arsenic, respectively, per man with wide variability in the quantities excreted by different individuals.—S. H. WEBSTER. *Pub. Health Repts.*, 56 (1941), 1359–1368. (F. S. M.)

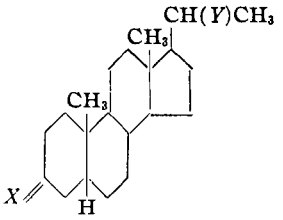
Lead Arsenate Spray Residue—Twenty-Four-Hour Output of Certain Urinary Constituents in Persons Exposed to. The average 24-hr. urine volume for 177 adults was nearly 1.3 L. Large variations in these values were found to occur both between individuals and in identical individuals during different days. Daily calcium and phosphate outputs for all the individuals studied were within the normal range. Lead and arsenic outputs were higher among the orchardists than for the other groups studied. A comparison of urinary lead concentration values for different individuals when derived from fractional-day sample gave little information regarding relative daily outputs. Twenty-four-hour specimens gave more complete information regarding individual daily outputs. First morning specimens are satisfactory substitutes, where it is impossible or impractical to obtain 24-hr. specimens.—S. H. WEBSTER. *Pub. Health Repts.*, 56 (Sept. 26, 1941), 1910–1919. (F. S. M.)

Lead Content of the Liver—Evaluation of. Investigations of Herbivorous Animals Injured by Lead. The average amount of lead in the liver increases in summer, decreases in autumn, rises sharply in November and falls during the winter.—W. GABEL. *Arch. exp. Path. Pharmakol.*, 195 (1940), 383–388. (F. S. M.)

Lead in Soft Tissues (Liver, Kidneys and Spleen)—Deposition and Removal of. Animals kept on a lead and then lead-free diet showed decrease of 50% lead in liver and kidneys but increase of 10% in bone. Blood calcium values were higher in animals after rest from lead diet. Examination showed rather severe damage to the renal convoluted tubule cells but this was greatly repaired after a two week-lead-free period. Regeneration of the spleen was shown.—L. T. FAIRHALL and J. W. MILLER. *Pub. Health Repts.*, 56 (1941), 1641–1650. (F. S. M.)

Lead Porphyrinuria—Contribution to the Problem of. Investigation on the Urine of Herbivorous Animals. The author concludes that the symptom of porphyrinuria is of no value in herbivorous animals.—W. GABEL. *Arch. exp. Path. Pharmakol.*, 195 (1940), 365–382. (F. S. M.)

Male Sex Hormone Series—Sterols of the. *2,226,627*—3(β), 20(β)-Allopregnanediol is prepared by catalytically reducing allopregnanedione which may be obtained by oxidizing a mixture of pregnanediol and allopregnanediol to the corresponding dione compounds and separating the less soluble allopregnanediol from the more-soluble pregnanedione. Androstanedione is prepared by acylating 3(β), 20(β)-allopregnanediol to its diacylated derivative, partially saponifying the latter to its 20-acylate, oxidizing this to the monoacylate of allopregnan-20-ol-3-one, dehydrating the ol-one at its secondary alcohol grouping to the corresponding ethylenic ketone and oxidizing the said ketone to androstanedione. *2,226,628*—This relates to the production of pregnane compounds of the general formula



where X is a ketonic oxygen or equivalent hydrolyzable grouping capable of yielding a ketonic oxygen atom upon hydrolysis, Y is a hydroxyl radical or an equivalent hydrolyzable group capable of yielding a hydroxyl group upon hydrolysis, and in which the C_8 hydrogen atom and Y are present in the cis-steric arrangement. The procedure of the patent may be carried out by starting with a pregnanediol, such as that obtainable from human pregnancy urine, converting the diol to a hydrolyzable derivative wherein the hydroxyl groups are protected against oxidation, such as by formation of a diester derivative, partially saponifying the protected derivative to one in which the protecting group at C_3 only has been removed to regenerate the alcoholic hydroxyl radical and oxidizing the latter compound to convert the secondary alcohol group at C_3 into a ketonic oxygen atom with production of a C_{20}-protected derivative of a pregnan-20-ol-4-one compound. If desired, the resulting 3-keto compound can be hydrolyzed to produce the corresponding pregnan-20-ol-3-one compound, which can be further converted into other hydrolyzable derivatives coming under the general formula given.—RUSSEL E. MARKER, DAVID M. JONES and THOMAS S. OAKWOOD, assignors to PARKE, DAVIS & Co. U. S. pats. 2,226,627 and 2,226,628, Dec. 31, 1940. (A. P.-C.)

Male Sexual Hormones—Compounds Having the Character of the. A process of preparing compounds having the character of male sexual hormones involves subjecting an oxime of the general formula RC(:NOH)CH$_3$ (where R stands for the phenanthrene residue) to transposition by the Beckmann method and treating the amines thus obtained with an oxidizing agent. Various details are described.—MAX BOCKMUHL, GUSTAV EHRHART, HEINRICH RUSCHIG and WALTER AUMÜLLER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,212,363, Aug. 20, 1940. (A. P.-C.)

Methionine—Gravimetric Method for the Determination of. A gravimetric method for the determination of methionine, involving the isolation of methionine sulfur as $BaSO_4$, has been outlined. The method has been tested by applying it to pure methionine and homocystine, to methionine and cystine mixtures and to eight common protein materials. The cystine and methionine contents of arachin, casein, edestin, egg white, gelatin, human globin, lactalalbumin and beef muscle have been determined. The sulfur partitions of these eight protein materials are presented.—ELIOT F. BEACH and D. MAXWELL TEAGUE. *J. Biol. Chem.*, 142 (1942), 277. (F. J. S.)

Munson and Walker's Reducing Sugar Tables—Errors of, and the Precision of Their Method.—RICHARD F. JACKSON and EMMA J. MCDONALD. *J. Research Natl. Bur. Standards*, 27 (1941), 237–255. (W. T. S.)

Nicotinic Acid in Blood—Determination of. Stir 5 cc. of oxalated blood with 8 cc. of water and 5 cc. of 15% trichloracetic acid solution, and add 2 cc. of absolute alcohol; after 10 min. filter or centrifuge the mixture. Heat 8 cc. of the clarified liquid (corresponding to 2 cc. of blood) with 0.25 cc. of hydrochloric acid in boiling water for 5 min., cool and adjust to *p*H 7 with solution of sodium hydroxide. Add the mixture to 1 cc. of alcohol containing 10 micrograms of nicotinic acid, add 2 cc. of absolute alcohol and 8 cc. of a solution of cyanogen bromide prepared by decolorizing solution of bromine with 10% solution of potassium cyanide. Shake for 3 min., add 5 cc. of 2.5% solution of aniline and compare the color produced with that of a standard prepared from 20 micrograms of nicotinic acid in 9 cc. of water, 3 cc. of absolute alcohol, 8 cc. of cyanogen bromide solution and 5 cc. of aniline solution. The non-specific tint of the blood filtrate is corrected for by subtracting 0.3 from the colorimeter reading, the standard being set at 20. Nicotinic acid in blood is present chiefly in the cells, the mean concentration being 368 ± 129 micrograms % for forty-one determinations on students and 356 ± 67 micrograms % for eighteen determinations on in-patients.—B. D. KOCHHAR. *Indian J. Med. Research*, 28 (1940), 385; through *Quart. J. Pharm. Pharmacol.*, 14 (1941), 87. (S. W. G.)

Pantothenic Acid (Blood) Values in Multiple Sclerosis. Blood pantothenic acid values from six normal subjects varied over the usually obtained normal range of from 19.7 to 33.3 micrograms %. These values are extraordinarily decreased in pantothenic acid deficiency. Six patients with multiple sclerosis failed to show any deficiency indicating that pantothenic acid is probably not an etiologic factor in the disease.—LOUIS SIEGEL, TRACY J. PUTNAM and JOHN G. LYNN. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 362. (A. E. M.)

Potassium in Plasma—A New Technique for Identifying. Potassium is precipitated by cobalt nitrate with "Nitroso-R-sal" (Eastman), a sodium salt of 1-nitrous-2-napthol-3,6-disulfonic acid as an indicator. This method eliminates volatile solvents and uses a very small amount of plasma. Comparison of samples with standard is made in a photocolorimeter.—A. D. MARENZI and C. E. CARDINI. *Anales farm. bioquím.*, 12 (1941), 32. (G. S. G.)

Pregnanedione Compounds—Obtaining. A method for the preparation from pregnant mare's urine of pregnane-3,20-dione and allopregnane-3,20-dione comprises separating from the urine a water-insoluble carbinol fraction, removing phenolic substances from such fraction, oxidizing the phenol-free fraction with an oxidizing agent capable of converting the hydroxyl groups of pregnane-3,20-diol and allopregnane-3,20-diol into ketone groups, converting the resulting diketone compounds into hydrolyzable derivatives differing in their solubilities in solvents, separating such derivatives of pregnane-3,20-dione and allopregnane-3,20-dione from one another by means of such differences in solubilities, and hydrolyzing the separated derivatives to obtain pregnane-3,20-dione and allopregnane-3,20-dione separate from each other.—RUSSELL E. MARKER, assignor to PARKE, DAVIS & CO. U. S. pat. 2,246,595, June 24, 1941. (A. P.-C.)

Progesterone. Oxidation of cholestenone is effected in sulfuric acid of over 50% strength, and the progesterone formed is isolated from the resulting reaction mixture, as by dissolving in petroleum ether.—HERRMANN BRETSCHNEIDER and ANDRAS SALAMON, assignors to CHINOIN GYOGYSZERÉS VEGYÉSZETI TERMÉKEK GYARA R. T. U. S. pat 2,246,341, June 17, 1941. (A. P.-C.)

Progesterone—Structural Isomer of. A structural isomer of progesterone which corresponds to the empirical formula $C_{21}H_{30}O_2$ is made by treating neopregnenolones or neopregnendiols with an oxidizing agent or a dehydrogenating agent, respectively, and is suitable for use as a therapeutic agent or as an intermediate in making therapeutic products.—KARL MIESCHER and HANS KAEGI, assignors to CIBA PHARMACEUTICAL PRODUCTS, INC. U. S. pat. 2,246,889, June 24, 1941. (A. P.-C.)

Prothrombin Destruction and Vitamin K Storage in the Body. Three patients with bile fistulas were studied and it was shown that vitamin K in some form is stored within the body and that vitamin K permits the elaboration of prothrombin by the liver as long as the stores hold out. In this manner the continued destruction of prothrombin by the body is compensated for. The fact that vitamin K may be stored and that prothrombin is constantly being destroyed has a practical application in surgery. It is a mistake to treat hypoprothrombinemia preoperatively with only enough vitamin K to raise the prothrombin to normal. Treatment must be continued beyond this point so that a reserve of vitamin K is built up and the vitamin K store must be large enough to last until adequate amounts can be taken in the food. The authors believe it would be wise to administer vitamin K preoperatively when there is obvious defective absorption even though the prothrombin content of the blood is not decreased since the reserve may be exhausted in a short time.—J. G. ALLEN and C. BERMEULEN. *Arch. Surg.*, 42 (1941), 969; through *Abbott Abstract Service*, (1941), No. 980. (F. J. S.)

Protoporphyrin IX—Isolation of, from Feces of Normal and Anemic Rats. Protoporphyrin IX has been isolated from the feces of anemic copper-deficient rats, anemic iron-deficient rats and normal rats on a milk diet. It has also been isolated from the feces of normal rats on a solid diet.—M. O. SCHULTZE. *J. Biol. Chem.*, 142 (1942), 89. (F. J. S.)

Provitamin D. A process of producing a provitamin D which in its pure state has an m. p. of about 137° C. and a specific optical rotation in benzene equal to about −124°, and whose acetate has an m. p. of about 135° to 136° C. and a specific optical rotation in benzene equal to about −85.5°, comprises extracting the fatty material from periwinkles, saponifying the fatty material with a saponifying agent and extracting therefrom the unsaponifiable portion with an organic solvent, such as ethyl ether.—ALBERT G. BOAR, JOHANNES VAN NIEKERK, ENGBERT H. REERINK and AART VAN WIJK, assignors to HARTFORD NATIONAL BANK AND TRUST CO., AS TRUSTEES. U. S. pat. 2,216,719, Oct. 8, 1940. (A. P.-C.)

Pyridoxine Deficiency—Studies on Anemia in Dogs Due to. The following summary is given: (1) The blood plasma iron is abnormally high in anemia due to pyridoxine deficiency in dogs. It drops to a low normal level during the remission with pyridoxine therapy. (2) Total blood copper values are at a low normal level during the anemia and increase to normal during pyridoxine therapy. (3) Following the immediate stimulation in blood formation afforded by pyridoxine therapy, there is a lag which may be overcome by the addition of liver extract to the ration. This stimulation is not apparently due to thiamine, riboflavin, nicotinic acid, pantothenic acid or choline.—J. M. MCKIBBIN, A. E. SCHAEFER, D. V. FROST and C. A. ELVEHJEM. *J. Biol. Chem.*, 142 (1942), 77. (F. J. S.)

Radioactive Copper—Use of, in Studies on Nutritional Anemia of Rats. The following summary is given: (1) The radioactive isotope $_{29}Cu^{64}$ is suitable for biological experiments. (2) Copper-deficient rats retained more of the single therapeutic dose of copper than did iron-deficient rats. In both types of animals only a small fraction of the copper fed was retained. (3) The kidney, the liver and the bone marrow show the highest relative retention of copper in 24 to 48 hrs. (4) The entrance of therapeutic copper into the bone marrow of copper-deficient rats has been demonstrated.—M. O. SCHULTZE and S. J. SIMMONS. *J. Biol. Chem.*, 142 (1942), 97. (F. J. S.)

Serum Inorganic Phosphate and "Acid" and "Alkaline" Phosphatase Activity—Estimation of. Micro and macro photometric techniques are described for serum inorganic phosphate and "acid" and "alkaline" phosphatase activity. Sodium β-glycerophosphate is the substrate used. On this basis the unit of phosphatase activity for both enzymes is comparable.—GEORGE Y. SHINOWARA, LOIS M. JONES and HARRY L. REINHART. *J. Biol. Chem.*, 142 (1942), 921. (F. J. S.)

Sex Hormones—Changed Excretion of, in Cirrhosis. Previous experimental work has indicated that the liver plays an important part in the metabolism of estrogenic hormones and it is therefore not surprising that in the presence of severe liver damage this metabolism should be deranged. The authors found in eight cases of cirrhosis that estrogens were excreted in the urine in the free form, and the usual inactivation appeared not to have taken place. In one acute case, the liver damage was very extensive and there was a partial failure to inactivate either androgens or estrogens. It is suggested that the working out of a suitable bioassay method for determining the amounts of estrogens and androgens excreted in the urine might aid in evaluating the degree of liver damage which is present. In male patients who are suffering from cirrhosis, gynecomastia and testicular atrophy are frequently observed clinically and this may be the result of inadequate destruction of estrogens known to be formed in the male.—S. J. GLASS, H. A. EDMONSON and S. N. SOLL. *Endocrinology*, 27 (1940), 749; through *Abbott Abstract Service*, (1941), No. 953. (F. J. S.)

Sterols. LXXXVII. Cholesterol and Sitosterol Derivatives. A short study has been made of the action of potassium permanganate in acetic acid on cholesterol and sitosterol compounds. The preparation and some reactions of 7-keto-sitosteryl chloride are reported.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 62 (1940), 516–517. (E. B. S.)

Sterols. LXXXVIII. Pregnanediols from Sarsasapogenin. The isomerism of sarsasapogenin to pseudosarsasapogenin with acid anhydrides is described. Pseudosarsapogenin on mild oxidation with chromic anhydride is converted into $\Delta^{16,17}$-pregnenedione-3,20. The reduction products of this compound have been studied.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 62 (1940), 518–520. (E. B. S.)

Sterols. LXXXIX. Reactions of Pseudosarsapogenin. Evidence is presented which indicates the probable structure of pseudosarsasapogenin, which is given tentatively.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 62 (1940), 521–525. (E. B. S.)

Succinoxidase System in Riboflavin-Deficient Rats. The work is summarized as follows: (1) The effect of riboflavin deficiency in the rat upon the succinoxidase content of various tissues was studied. (2) The dietary intake of riboflavin was found to have a definite effect upon the succinoxidase content of liver tissue. (3) The results obtained were taken as *prima facie* evidence that one or more components of the succinoxidase system are flavo-proteins.—A. E. AXELROD, V. R. POTTER and C. A. ELVEHJEM. *J. Biol. Chem.*, 142 (1942), 85. (F. J. S.)

Sugar—Dry Reagent for Testing Urine for. A dry reagent adapted to react to a drop of urine to test it without the application of heat other than that of the reaction itself is formed of a water-free bismuth salt such as bismuth oxychloride, caustic alkali and sodium silicate. In the presence of sugar, the reagent gives a black color.—ALEXANDER GALAT, assignor to DENVER CHEMICAL MFG. CO. U. S. pat. 2,210,579, Aug. 6, 1940. (A. P.-C.)

Sulfathiazole Therapy—Reduction of Benedict's Solution by Urine, during a Course of. In addition to comparing the sulfanilamide drugs in numerous respects, the author reports for the first time that the urine of a patient, taking sulfathiazole for a staphylococcus and a streptococcus septicemia, reduced Benedict's Solution although the blood sugar was only 73 mg. The observation is not satisfactorily accounted for.—RICHARD E. STRAIN. *Indian Med. Gaz.*, 76 (1941), 206–208. (W. T. S.)

Thiols—Effect of, on the Reducing Groups of Lactogenic Hormone. The presence of 3.0% cystine in lactogenic hormone has been demonstrated. No cystine was found in hydrolysates nor did the unhydrolyzed protein reduce the usual thiol reagents. It did reduce cystine in strongly alkaline solution. Treatment of lactogenic hormone with thiol compounds causes (*a*) the reduction of disulfide bonds to cystine thiol groups, (*b*) the appearance of groups of unknown nature which reduce phosphotungstate and ferricyanide in neutral solution and (*c*) the probable formation of stable addition compounds between the protein and the thiol compound. Thioglycolic acid was found to cause about twice as much reduction as an equivalent amount of cystine.—HEINZ FRAENKEL-CONRAT. *J. Biol. Chem.*, 142 (1942), 119. (F. J. S.)

Threonine, Serine, Cystine and Methionine Content of Peanut Proteins. Arachin and conarachin were prepared from peanuts; it was purified and analyzed for threonine, serine, cystine and methionine.—W. L. BROWN. *J. Biol. Chem.*, 142 (1942), 299. (F. J. S.)

Tocopherols—Esters of. By reaction of α- or β-tocopherol (suitably in pyridine, with heating to 50° to 60° C.) with stearic or oleic acid chloride or the like, esters are formed which have a vitamin E action. Mention is made of a lauric acid ester and details are given of the production of the stearic and oleic acid esters.—PAUL KARRER, assignor to HOFFMANN-LAROCHE INC. U. S. pat. 2,231,125, Feb. 11, 1941. (A. P.-C.)

Triphosphopyridine Nucleotide—Microdetermination of. A spectrophotometric test, which allows the accurate determination of 2×10^{-2} to 20×10^{-2} γ of triphosphopyridine nucleotide in the course of a few minutes, has been described. Diphosphopyridine nucleotide is inactive in this test.—ERWIN HAAS, CARTER J. HARRER and T. R. HOGNESS. *J. Biol. Chem.*, 142 (1942), 835. (F. J. S.)

Urinary Lead Excretion—Diurnal Variation of. The occurrence of diurnal variation in urinary lead excretion is demonstrated—it appears to occur independent of previous exposure of the individual or the time of year. A wide daily variation is shown for urinary lead concentration measurements as well as for the rate of lead excretion (in micrograms per hr.) and for the total volume with all of the individuals studied. The total daily urinary outputs were, however, remarkably constant for any one

individual. An increase in the rate of urine excretion was generally paralleled by an increase in the rate of lead excretion for those specimens whose specific gravity was greater than 1.010. Marked diuresis was found not to increase greatly the rate of lead excretion nor the total quantity. For a given individual an increase in the amount of urinary phosphorus per sample was usually accompanied by an increase in the quantity of lead excreted. First morning specimens have been shown to be representative of the corresponding 24-hr. specimens. Fractional-day samples give no measure of the total daily outputs. Neither the urinary lead concentrations nor the rate of urinary lead excretion can be used as a measure of lead absorption when they are based on short-time measurements.—S. H. WEBSTER. *Pub. Health Repts.*, 56 (1941), 1834–1848. (F. S. M.)

Vitamin-Bearing Liquids—Protecting, from Oxidation by Use of a Gelatin Matrix. *2,218,591*—There is formed a molded, solid product consisting essentially of a gelatin matrix containing discrete particles of a vitamin-bearing liquid dispersed therein and completely imprisoned and surrounded thereby, whereby no free vitamin-bearing liquid is on the exterior surfaces of the molded product, the vitamin-bearing liquid containing at least vitamin A, and, if desired, various other vitamins. *2,218,592*—Relates to generally similar compositions or products containing, as an additional ingredient, invert sugar, which serves as a plasticizing agent. Honey or molasses may be used, *e. g.*, with gelatin, water and a fish liver oil, etc.—HARDEN F. TAYLOR, assignor to ATLANTIC COAST FISHERIES CO. U. S. pats. 2,218,591 and 2,218,592, Oct. 22, 1940. (A. P.-C.)

Vitamin A Estimation—Photoelectric Photometer for. The instrument described makes possible rapid and accurate estimation of the amount of preformed vitamin A. It has been economically constructed, is readily portable, and does not require a high degree of technical skill in its operation or in the evaluation of the results. By the use of other light sources and filters, the instrument can be employed for making transmission measurements in other ranges of the ultraviolet; hence its scope is greater than for vitamin A assay alone.—A. E. PARKER and B. L. OSER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 260–262. (E. G. V.)

Vitamin A—Physicochemical Assay of. Several solutions of vitamin A which were being assayed in the regular work were split up into two parts. One part was put into an amber test tube, and the other part into a clear test tube of the same dimensions. The tightly stoppered test tubes stood in a glass beaker on the laboratory bench 12 ft. from the window of the room. After a few hours of this exposure to daylight, the solutions were again tested and the potencies of the vitamin A concentrates calculated. Cloudy days during which no sun shone were chosen for these tests, in order to help compensate for the higher actinic power of summer daylight. The solutions contained 20 to 25 units of vitamin A per cc. Results of these tests show that serious losses of vitamin A potency result from exposure of solutions of vitamin A in clear glass test tubes, while only small losses are found if amber test tubes (Kimble) are used.—N. D. EMBREE. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 144–145. (E. G. V.)

Vitamin A Potency—Relation of Chemical Analysis of Butter to Its. From the analyses of 29 samples of butterfat for carotene, spectro vitamin A and vitamin A potency in I. U. (International Units), it was found that the biological potency in I. U. could be calculated from the analysis by the equation I. U. $= 3.2S + 1.7C$ or I. U. $= 4(S - 0.5) + 1.7C$, where S = p. p. m. spectro vitamin A, and C = p. p. m. carotene. The second equation gave better results.—G. S. FRAPS, A. R. KEMMERER and W. W. MEINKE. *J. Assoc. Official Agr. Chem.*, 24 (1941), 731–735. (A. P.-C.)

Vitamin B Complex—Deficiency of, as a Cause of Seborrheic Dermatitis. The disease produced experimentally by feeding rats egg white bears the characteristics of seborrheic desquamative dermatitis, similar to the syndrome in infants and to the seborrheic eczema in adults. Toxicity of egg white is probably due to incompletely digested protein molecules which are absorbed and act as specific poisons. Other proteins may pass through intestinal wall undigested, especially during infancy when there is a high permeability of the intestinal wall and immature digestive power. For the production of egg white injury, a protective factor, biotin, must be lacking. The main sources of biotin in food are liver, kidney and yeast, foods not in the usual diet. Deficiency of other components of the vitamin B complex, pantothenic acid, riboflavin and pyridoxine as well as of biotin may cause seborrhea in adults. The author believes that considerably amounts of liver and yeast than are commonly advocated should be employed to prevent seborrhea.—P. GYÖRGY. *Arch. Dermatol. Syphilol.*, 43 (1941), 230; through *Abbott Abstract Service*, (1941), No. 886. (F. J. S.)

Vitamin B_1—A Reaction of. Vitamin B_1 has few chemical reactions. Those in current use are: (*a*) the formation of thiochrome by oxidation which produces a blue fluorescence. This is used for quantitative determination of thiamine. (*b*) Reactions produced by diazoics: with diazotized sulfanilic acid and with diazotized *p*-amino acetophenone. More sensitive color reactions are produced by the use of diazotized *para*-nitro aniline, which develops a rose color not dissolved out by common organic solvents. The procedure is described.—A. D. MARENZI. *Anales farm. bioquím.*, 11 (1940), 115. (G. S. G.)

Vitamin C Deficiency Associated with Certain Cases of Hematuria. The author has seen cases of hematuria where the bleeding came from the bladder. By very careful investigation he ruled out any involvement of the kidney in these cases and found in all of them a marked deficiency in the amount of urinary vitamin C. Inquiry into the histories showed that all of these patients were on diets extremely deficient in vitamin C. The cystoscopic picture presented by these bladders was that of a patchy purpuric rash. The affected areas were seen to leak or ooze blood into the bladder medium. The diets were suitably altered and vitamin C was administered. Reëxamination a week or ten days later showed the urines to be free from blood cells, and to have normal vitamin C content; cystoscopic examination showed the complete disappearance of the purpuric changes noted before. The author emphasized the fact that vitamin C deficiency as an explanation of hematuria should be accepted only after the most careful investigation of the kidney.—T. J. D. LANE. *Irish J. Med. Sci.*, 180 (1940), 779; through *Abbott Abstract Service*, (1941), No. 883. (F. J. S.)

Vitamin C and Copper—Relation Between, in Oxidation-Reduction of Cells. The activity of pumpkin and cauliflower juice in oxidation of ascorbic acid is not due to an oxidase but to Cu in protein linkage. Proteins influence the catalytic properties of copper considerably. Carrot juice contains ascorbic acid as is demonstrated by the reduction of dichlorophenolindophenol and copper is identified by the pink color developed with Meyer's reagent. Solutions of ascorbic acid do not give the latter reaction. An important role of copper in the

biological processes of the plant cell is claimed.—DIONISIO ECHAVE. *Rev. farm. (Buenos Aires)*, 82 (1940), 219. (A. E. M.)

Vitamin C—Chemical Methods for Determination of. When adequate precautions are taken, the reaction of ascorbic acid with 2,6-dichlorophenol-indophenol can be used in direct titrations or with the photoelectric colorimeter to give relatively satisfactory quantitative analyses. Other methods of analysis may be preferable under special circumstances because of interference by other reducing materials. Methods for the measurement of dehydroascorbic acid, however, are subject to great interference, because many aldehydes, ketones and quinones give rise to an interfering reaction when reduced by hydrogen sulfide. Among the interfering compounds are pyruvic acid, pyruvic aldehyde, glyceric aldehyde, dihydroxyacetone, acetaldehyde, mannosaccharic acid, 5-ketogluconic acid, 1,4-benzoquinone, 1,4-naphthoquinone, and 2-methyl-1,4-naphthoquinone (thyloquinone). Three methods for detecting and avoiding such interference are pointed out.—C. G. KING. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 225–227. (E. G. V.)

Vitamin C Oxidation—Studies in. I. Coexistence of Oxidizing and Protective Factors in Plants for Vitamin C. The existence of a protective factor in vegetables which protects vitamin C from oxidation has been established. The enzyme ascorbic acid oxidase and the protective factor occur together in various vegetables, and a method is described for the separation of the two factors from each other. The protective factor inhibits the copper oxidation while the enzymic oxidation of the vitamin is not influenced by it. The enzyme and the protective factor are more concentrated in the pericarp of the vegetables. The nature and properties of the protective factor have been investigated.—P. V. KRISHNAMURTHY and K. V. GIRI. *J. Indian Chem. Soc.*, 18 (1941), 7. (F. J. S.)

Vitamin C Oxidation—Studies in. II. Influence of Various Substances Occurring in Plant and Animal Tissues on the Catalytic Oxidation of Vitamin C. A study has been made of the influence of various substances on the catalytic oxidation of vitamin C by copper. Among the substances investigated, oxalic acid, xanthine, uric acid, theophylline, creatinine, antipyrine and albumin exert powerful protective action, while tartaric, citric, malic, maleic, malonic, tannic and aspartic acids, glycine, alanine, asparagine, histamine and pyrogallol exert slight protection against the oxidation of the vitamin. Creatine, succinic acid and other compounds investigated exert no protection. The various possible mechanisms underlying the action of these substances on vitamin C oxidation are discussed. **III. Retardation of Vitamin C Oxidation by Oxalic Acid.** The retardation of vitamin C oxidation by oxalic acid has been studied in detail. It has been found that oxalic acid inhibits markedly the oxidation of vitamin C under different conditions: (1) the uncatalyzed oxidation, (2) the oxidation catalyzed by copper and iron and (3) the oxidation catalyzed by the enzyme ascorbic acid oxidase. The mechanism of the inhibition is discussed.—(II) P. V. KRISHNAMURTHY and K. V. GIRI; (III) P. V. KRISHNAMURTHY. *J. Indian Chem. Soc.*, 18 (1941), 191, 201. (F. J. S.)

Vitamin D. Apparatus is described, and a method of producing or increasing vitamin D in ergosterol, comprising: forming a solution of ergosterol in ether (the solution having no more than 1% of ergosterol therein), and exposing the solution to the action of ultraviolet light, about 90% of which is in the range of 2536 to 2540 A. units, at a distance of approximately $^{3}/_{16}$ in. and for about 45 min.—MENFRED L. JOHNSON, assignor to VITAMIN TECHNOLOGISTS, INC. U. S. pat. 2,234,632, May 27, 1941. (A. P.-C.)

Vitamin D—Evaporated Milk Concentrate of. Evaporated milk is used as a carrier for activated ergosterol dissolved in butterfat.—REGINALD C. SHERWOOD and CHARLES G. FERRARI, assignors to GENERAL MILLS, INC. U. S. pat. 2,245,418, June 10, 1941. (A. P.-C.)

Vitamin E as an Accessory Factor in Avitaminosis A. One of the most noteworthy properties of vitamin A is the extent to which it can accumulate in the liver. The authors performed many experiments on animals to discover if in the absence of other vitamins these large reserves of vitamin A would be preserved. As a result of these experiments it was found that the vitamin A reserves of rats which had been kept on a diet deficient in vitamin E were much lower than those of animals receiving equal amounts of vitamin A together with supplements of vitamin E. The authors believe the deficiency of vitamin E may lead to acute secondary deficiency of vitamin A. The deficient rats also developed the whitening of the teeth which is generally held to be an effect of deficiency of vitamin A, but which may be considered a common result of deficiency of either of these vitamins. In human diseases in which the reserves of vitamin A are known to be reduced, vitamin E deficiency should be considered also.—A. W. DAVIES and T. MOORE. *Nature*, 147 (1941), 794; through *Abbott Abstract Service*, (1942), No. 1015. (F. J. S.)

Vitamin E—Chemistry of. The Reaction between Grignard Reagents and Coumarins and Hydrocoumarins. *o*-Hydroxystyryldialkylcarbinols have been obtained by reaction between coumarin and Grignard reagents. The structures of these carbinols have been proved by reducing the diethyl compound to the saturated carbinol and preparing the same substance from dihydrocoumarin and ethylmagnesium bromide. The unsaturated carbinols have been cyclized to 2,2-dialkyl-Δ^3-chromenes but the chromenes are accompanied by large amounts of dark viscous oils, probably polymeric substances. Previous work on the preparation of 2,2-dialkylchromans by action of Grignard reagents on dihydrocoumarins has been extended. At present the series of 2,2-dialkylchromans is complete from $R = CH_3$ to $R = n\text{-}C_4H_9$; the series of saturated carbinols includes those in which $R = CH_3$, C_2H_5 and $n\text{-}C_4H_9$; the series of unsaturated carbinols includes those in which $R = CH_3$ and C_2H_5; the series of Δ^3-chromenes includes those in which $R = CH_3$, C_2H_5; the series of Δ^3-chromenes includes those in which $R = CH_3$, C_2H_5 and $n\text{-}C_4H_9$.—L. I. SMITH and P. M. RUOFF. *J. Am. Chem. Soc.*, 62 (1940), 145–148. (E. B. S.)

Vitamin E—Synthetic. Vitamin E is active in fertilization, as an anti-abortive and a galactagogue. It favors nutrition of the fetus and development of the newborn, and relieves certain neuromuscular distresses such as amiotrophic lateral sclerosis. The synthetic vitamin E (Ephynal Roche) seems to have all the qualities of the natural in wheat germ oil with the added advantage of perfect stability.—A. DABBAH. *Semana mêd.*, 2 (1940), 94; through *Rev. Col. Farm. Nac.*, 7 (1940), 186. (G. S. G.)

Vitamin K Requirement of the Newborn Infant. The vitamin K requirement of the newborn is extremely low, approximately 1 microgram of synthetic vitamin a day. Milk contains enough preformed vitamin K to meet this requirement.—R. L. SELLS, S. A. WALKER and C. A. OWEN. *Proc. Soc. Exptl. Biol. Med.*, 47 (1941), 441. (A. E. M.)

Vitamins—Need for. A discussion stressing the importance of auxiliary vitamins in the maintenance of good health.—L. STAMBOVSKY. *Drug and Cosmetic Ind.*, 50 (1942), 32, 35, 37. (H. M. B.)

ANALYTICAL

Acetanilid—Determination of. In the determination of acetanilid, hydrolysis for 1 hr. on a steam bath with 4 times normal hydrochloric acid can be substituted for the A. O. A. C. of the N. F. sulfuric acid hydrolysis, with the same degree of accuracy. If caffeine is present but need not be determined, the hydrolyzed solution can be titrated directly with the bromide-bromate solution to the appearance of a slight yellow color without removal of the caffeine; indirect titration (addition of excess bromine solution, addition of potassium iodide and titration of liberated iodine) cannot be used to presence of caffeine, as the latter under these conditions can combine with a comparatively large quantity of the free bromine. When caffeine is removed by means of chloroform, the solution of aniline hydrochloride is not sufficient to create an appreciable error in the determination of caffeine. The method is not applicable in the presence of acetophenetidin.—T. W. KETHLEY. *J. Assoc. Official Agr. Chem.*, 23 (1940), 782–787. (A. P.-C.)

Aliphatic Nitro Compounds—Polarographic Study of. The reduction of six aliphatic nitro compounds at a dropping mercury cathode was studied with a manually operated polarograph in the concentration range 0.0005 to 0.017 molar. The deflections of the the galvanometer were proportional to the concentration of the compound within a few per cent error. A 0.05 *M* sulfuric acid solution was used as the indifferent electrolyte. In neutral 0.05 *M* sodium sulfate solution a partial conversion to an acid-nitro form was probably the cause of the deviation from a linear relationship. The half-wave potentials were also determined, but it was not possible to determine the compounds separately in the presence of each other.—T. DEVRIES and R. W. IVETT. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 339–340. (E. G. V.)

Ammoniacal and Nitrate Nitrogen in Decomposed Plant Material—Determination of. The magnesia distillation method is unsuitable for the determination of ammonia in decomposed plant residues because high results are obtained through the concurrent liberation of ammonia from organic nitrogenous substances. The substitution of a phosphate buffer giving a reaction of *p*H 7.4 is recommended. Nitrate nitrogen can be determined on the aqueous extract of such materials by the Devarda reduction method after removing free or liberated ammonia by boiling under alkaline conditions.—J. G. SHRIKHANDE. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 187–188. (E. G. V.)

Arsenic—Determination of Very Small Amounts of, in Organic Material. After destroying interfering materials with hydrogen peroxide and sulfuric acid the arsenic is reduced to arsenic trioxide with hydrazine sulfate and is determined by titration with potassium bromate in hydrochloric acid solution. By electrometric determination of the end-point and the use of 0.01 *N* potassium bromate small quantities of arsenic can be determined within about 1 gamma.—B. BLEYER and H. THIES. *Vorratspflege u. Lebensmittelforsch.*, 2 (1939), 281–290. (F. S. M.)

Ascorbic Acid—Determination of, in Citrus Fruit Juices. Add to 5 cc. of citrus fruit juice 1 cc. of 10% potassium iodide and 2 cc. of 2 *N* sulfuric acid. Titrate the resulting solution with 0.01 *N* iodate, adding the reagent dropwise near the end-point. It is best not to add the starch (made from improved Lintner's soluble starch, using 1 Gm. per 100 cc. with 2 Gm. of potassium iodide added) until very near the end-point. One cc. of 0.01 *N* iodate is equivalent to 0.88 mg. of ascorbic acid.—R. BALLENTINE. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 89. (E. G. V.)

Basic Copper Sulfates Used as Fungicides—Determination of Hydroxide in. A detailed description is given of a method in which the alkalinity is determined by back titration after solution in an excess of acid, using methyl orange as indicator, using a comparison solution containing approximately the same amount of copper and indicator in the same volume of solution to overcome the masking effect of the copper ion on the end-point color. Results are presented showing that, while the errors are in the same direction and tend to increase with the quantity of copper, they are small when compared with the total titration in the case of a basic copper sulfate.—H. BOIS. *J. Assoc. Official Agr. Chem.*, 24 (1941), 766–767. (A. P.-C.)

Benzocaine—Estimation of, and Its Separation from Acetanilid. The bromometric method of Valencien and Deshusses (*Mitt. Lebensm. Hyg.*, 30 (1939), 246) was found to be suitable with slight modification for the volumetric estimation of benzocaine. An attempt to separate benzocaine from acetanilid by extracting the latter with chloroform from a 1 + 1 hydrochloric acid solution of the mixture followed by bromometric determination of the separated ingredients gave 158% to 244% recoveries of benzocaine and 84% to 94% recoveries of acetanilid, probably due to incomplete extraction of acetanilid owing to its partial hydrolysis by the 1 + 1 hydrochloric acid. From a study of the distribution ratios of benzocaine and acetanilid between 1 + 1 hydrochloric acid and 6 times normal sulfuric acid and chloroform and ether, a method of separation was developed (technique described in detail) consisting in dissolving the mixture in 50 cc. of chloroform, passing this solution successively through three separatory funnels containing 30, 20 and 10 cc. of 6 times normal sulfuric acid and one funnel containing 10 cc. of water, passing four 20-cc. portions of chloroform through the same series of funnels, and combining all the chloroform extracts. Water-soluble excipients, if present, can be removed by two washings with 20 to 40 cc. and 10 cc. of water before passing the chloroform solution through the sulfuric acid. A collaborative study was made of the method on a benzocaine-acetanilid-sucrose mixture, the separated benzocaine being titrated bromometrically by the method of Valencien and Deshusses and the acetanilid being determined by the A. O. A. C. method (*A. O. A. C. Methods of Analysis*, (1940), 561, 4(*b*)) or by the Kethley method (*J. Assoc. Official Agr. Chem.*, 23 (1940), 782). Recoveries of 98.2% to 108.0% of benzocaine and 98.9% to 100.2% of acetanilid were obtained.—EUGENE H. WELLS. *J. Assoc. Official Agr. Chem.*, 24 (1941), 736–739. (A. P.-C.)

Calcium and Magnesium—Microdetermination of. By careful attention to detail, 0.1 to 10 mg. of calcium may be precipitated as calcium oxalate and titrated with 0.02 *N* potassium permanganate with an error not exceeding 2%. 0.1 to 2 mg. of magnesium can be precipitated as magnesium ammonium phosphate, centrifuged, dissolved in 0.01 *N* acid, and back-titrated to *p*H 4.5, using a modified achromatic indicator, with the same order of accuracy.—A. W. MARSDEN. *J. Soc. Chem. Ind.*, 60 (1941), 20–23. (E. G. V.)

Calcium—Microdetermination of, by Precipitation as Picrolonate and Estimation of the Precipitated Carbon by Manometric Combustion. A micromethod for calcium is described in which the calcium is precipitated as picrolonate and the precipitate, containing 20 atoms of carbon to 1 of calcium, is estimated from the carbon, which is determined by the rapid manometric wet combustion method of Van Slyke and Folch. Precipitation and combustion are done without transfer in a single centrifuge-combustion tube. The method serves for the esti-

mation of smaller amounts of calcium than can be determined accurately by the usual microprocedures based on titration of the oxalate; 0.2 mg. of calcium or 0.2 cc. of serum suffices for an analysis.—DONALD D. VAN SLYKE and FRANK J. KREYSA. *J. Biol. Chem.*, 142 (1942), 765. (F. J. S.)

Camphorated Menthol—An Assay for. The following methods of assay are offered: *Menthol.*—Into a tared 125-cc. Erlenmeyer flask weigh accurately 2 Gm. of the product. Add 5 cc. accurately measured, of acetylizing agent (1 part acetic anhydride and 4 parts of anhydrous pyridine) and reflux in boiling water for 30 min., using a water-cooled condenser. Through the condenser add about 30 cc. of warm distilled water and continue heating for 15 min. The system is allowed to cool, and before disconnecting the Erlenmeyer flask, the condenser is rinsed once with 15 cc. of water. The contents of the flasks are titrated with 0.5 *N* alcoholic potassium hydroxide using 2 drops of phenolphthalein as an indicator. A blank determination must be carried out and the number of cc. of 0.5 *N* alcoholic potassium hydroxide required to neutralize the acetic acid produced by 5 cc. of the acetylizing mixture is noted. The number of cc. of alcoholic potassium hydroxide required for the blank minus the number of cc. required for the sample, × 0.0781 = Gm. free menthol. *Camphor.*—One Gm. of the product is weighed in a tared 300-cc. Erlenmeyer flask. To this is added 75 cc. of dinitrophenylhydrazine reagent (1.5% in 25% methyl alcohol). The mixture is refluxed on a steam bath for 2 hrs. A Hoffman distilling head is attached to the flask, and the mixture heated at such a rate that 15–17 cc. of alcohol are distilled in 30 min. Fifty cc. of 5% sulfuric acid are added to the flask and the mixture is allowed to cool. An additional 150 cc. of 5% acid is added and the mixture is allowed to stand overnight. The precipitate is collected in a tared fritted-glass funnel No. 3, and dried to constant weight at 100° C.—CHARLES O. WILSON. *Bull. Natl. Formulary Committee*, 9 (1941), 350–353. (H. M. B.)

Capsicum—Chemistry of. A review.—FRANCIS D. DODGE. *Drug and Cosmetic Ind.*, 49 (1941), 516–518. (H. M. B.)

Chlorate Ion—New Colorimetric Method for the. A new colorimetric method is described for the detection and rough estimation of small amounts of chlorates. Pyridine in concentrated sulfuric acid produces a permanent violet color with chlorates.—M. B. ROY. *J. Indian Chem. Soc.*, 18 (1941), 165. (F. J. S.)

Chlorophyll and Carotene—Determination of, in Plant Tissues. Some of the recent literature on the subject is briefly reviewed (22 references). A coloborative study was made by four collaborators using the technique which they desired, the Peterson-Hughes-Freeman method for carotene and the Petering-Wolman-Hibbard method for carotene and chlorophyll being used as standards for comparison. The comments of the collaborators are given and the results are discussed, but no conclusions are drawn, and collaborative effort toward unification of methods available for determining both carotene and total chlorophyll will be continued.—ERWIN J. BENNE. *J. Assoc. Official Agr. Chem.*, 24 (1941), 526–539. (A. P.-C.)

Chromate in Blood Iodine Determinations—Detection and Titration of. After carrying out the oxidation with chromic and sulfuric acids, reduce with phosphorus acid and distill, using 10 cc. of 0.1% potassium carbonate in the receiver. Acidify the distillate with 0.3 cc. of 1 *N* sulfuric acid. Oxidize with bromine and evaporate to 2 cc. Cool. Add 0.2 cc. of buffer, 0.05 cc. of 25% potassium iodide and 1 drop of 1% starch. Titrate the iodine liberated by iodate with 0.001 *N* thiosulfate, then add several drops of 1 *N* sulfuric acid and about 3 mg. of oxalic acid (recrystallized). If chromate is present it will liberate iodine immediately and quantitatively, so that one can titrate again to the starch end-point. The amounts of sulfuric acid and buffer are adjusted so that the solution is certain to be acid during oxidation with bromine and the potassium acetate in the buffer will suffice to convert the excess sulfuric acid to acetic acid. The 25% potassium iodide solution is freshly prepared each time by dissolving 1 Gm. of the salt in 3.7 cc. of iodine-free water. The buffer is prepared by one-twelfth neutralization of glacial acetic acid with a saturated solution of potassium carbonate that has been washed with iodine-free alcohol. Let us suppose that the glacial acetic acid is 17.4 *N*, and the washed potassium carbonate solution 10.8 *N*. To 10 cc. of glacial acetic acid one would add $\frac{10 \times 17.4}{12 \times 10.8}$ or 1.34 cc. of the carbonate solution.—O. H. GAEBLER and M. BATY. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 442–443. (E. G. V.)

Concentration and Acid Content—Influence of, in the Form of Crystalline Precipitates. A special study was made of the precipitation of a number of bases with potassium ferrocyanide in the presence of varying concentrations of the bases and acid. The bases were usually dissolved in varying concentrations of acid. It was of importance to know whether the concentration of acid added to bring about solution, influenced the crystalline form. The freshly prepared solution of the base in hydrochloric acid was treated on the microscope slide with an excess of solid potassium ferrocyanide which was not weighed and no conclusions can be drawn as to its effect on the crystal form. The results of these precipitations with the following bases are described in tabular form: alypin, antipyrine, quinine, quinoline, cocaine, ephedrine, ephetonin, hexamethylenetetranine, larocaine, novocaine, panthesine, pantocaine, percaine, tropacocaine and tutocaine. The organismoid form of precipitate results only with the concentrated solutions and perhaps the viscosity of the solution plays a role. In those cases where the precipitate was crystalline, the concentration of the solution and of the acid are both often factors. For practical purposes in the use of crystals for analytical purposes, care must be taken to maintain comparable conditions.—L. ROSENTHALER. *Pharm. Acta Helv.*, 15 (1940), 257–265. (M. F. W. D.)

Copper—Biguanide Sulfate as a Reagent for the Estimation of. Copper biguanide sulfate which forms a very sparingly soluble inner metallic complex salt of the second order and has been utilized for the estimation of copper. Zinc, cadmium, molybdenum (Mo^{6+}), tungsten (Wo^{6+}), magnesium and alkali metals do not interfere with the estimation. The solution must not contain any nitrate as the corresponding copper biguanide nitrate is also sparingly soluble. The precipitate, dried at 50°–70°, has the composition $[Cu(C_2N_5H_7)_2]SO_4 \cdot 3H_2O]$, and is weighed as such. The estimation can be made on both macro and semimicro scale with very good results, using only a good quality macro analytical balance. In the absence of a large excess of ammonium salt and ammonia the method has also been employed for the volumetric estimation of copper with rubeanic acid as an external indicator.—PRIYADARANJAN RAY and JAMINIBHUSAN ROY-CHOWDHURY. *J. Indian Chem. Soc.*, 18 (1941), 149. (F. J. S.)

Copper—Determination of, in Country Spirits. A method has been worked out for the determination of copper in country spirits (potable spirits made from gur, molasses, mahua, etc., with or without flavoring) by the use of diethyl dithiocarbamate

which has been found very useful especially in cases where the percentage of copper or the quantity of the spirit submitted for test is very small.—H. D. SURI, GURCHARAN SINGH AHLUWALIA and H. B. DUNNICLIFF. *J. Indian Chem. Soc.*, 8 (1941), 326. (F. J. S.)

Copper—Quantitative Determination and Separation of, with Benzotriazole. A new organic precipitant for copper, benzotriazole, is described. Copper is precipitated in a tartaric-acetic acid solution at a *p*H of 7.0 to 8.5. In the absence of silver, nickel, cadmium, zinc, cobalt and ferrous iron the precipitate can be weighed directly. In their presence the reagent can be used to effect a preliminary separation, and the copper subsequently determined by a standard method—for example, electrolytically or by the potassium iodide-thiosulfate method. Benzotriazole is an excellent precipitant for copper in iron and steel preparatory to the analysis by the standard iodide method. It possesses the advantage over the sulfide method that arsenic, antimony and molybdenum are not precipitated. The paper presents data on the proper conditions of acidity, temperature, *p*H, etc., for precipitation. A detailed procedure for determining copper in iron and steel is given.—J. A. CURTIS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 349–352. (E. G. V.)

Drop Analysis—Quantitative. XVI. An Improved Diffusion Method for Total Nitrogen. A rapid and simple method for the analysis of total nitrogen by diffusion is presented. The accuracy and precision were found to be about equal to those of the micro- and macro-Kjeldahl methods. No transfers or other disadvantageous techniques were involved in the procedure. It was possible to determine amounts of nitrogen as low as 1γ with an absolute probable error of not more than about 1% and a precision of about 0.3%. Smaller amounts could be determined with somewhat greater errors.—EDWARD R. TOMPKINS and PAUL L. KIRK. *J. Biol. Chem.*, 142 (1942), 477. (F. J. S.)

Electron Microscope. The construction and operation of an electron microscope are described. There is a possibility of increasing the resolving power of electron microscopes to the point where molecules and atoms may be observed directly.—A. L. G. REES. *Chemistry and Industry*, 60 (1941), 335–337. (E. G. V.)

FD & C Yellow No. 3 (Yellow AB) and D & C Green No. 6 (Quinizarin Green)—Analysis of Mixtures of. From a study of the properties of the two dyes, the following methods were devized for the determination of pure dye in D & C Green No. 6, and for the determination of the two dyes in admixture. *Pure Dye in D & C Green No. 6.*—Transfer or weigh direct into a 23 × 150 mm. test tube an accurately weighed sample of about 0.35 Gm.; wash down the wall of the testtube with 4 cc. of concentrated sulfuric acid, cover with a watch glass, heat 30 min. in the steam bath, wash down the wall of the testtube with 1 to 2 cc. of sulfuric acid, continue heating for 1 hr., cool, pour into 100 cc. of 50% alcohol in a 1-L extraction flask, partly neutralize with 5 Gm. of sodium carbonate and add 150 cc. of 50% alcohol and 15 Gm. of sodium citrate; heat to boiling and titrate with decinormal titanium trichloride (1 cc. = 0.0291 Gm. of pure dye) under an inert atmosphere. *Analysis of Mixtures.*—Dilute 0.5 to 1.0 Gm. of dry mixture or 25 to 50 Gm. of liquid mixture to 150 cc. with 95% alcohol; heat 30 min. on the steam bath, add 80 cc. of water with stirring, and let stand overnight; filter off the D & C Green No. 6 on a tared Gooch crucible, rinse the beaker, and wash the residual FD & C Yellow No. 3 from the crucible with 40% alcohol; dry the precipitate 3 hrs. at 135° C., cool, weigh and calculate the percentage of D & C Green No. 6; make the filtrate to exactly 500 cc. with alcohol; to a 100-cc. aliquot (200 cc. with small amounts of FD & C Yellow No. 3) in a 500-cc. wide-mouthed Erlenmeyer flask add 100 cc. of water, 15 Gm. of sodium bitartrate, and 1 cc. of 1% Light Green SF yellowish indicator solution; heat almost to boiling, titrate with decinormal titanium trichloride (1 cc. = 0.006180 Gm. FD & C Yellow No. 3) under an inert atmosphere, and deduct the blank due to the indicator. In the case of solutions of the mixed dyes in benzyl alcohol, the solubility effect on the green color is perceptible at approximately 25% concentration of the benzyl alcohol, but the precision is satisfactory for dye analysis work.—J. A. KIMR. *J. Assoc. Official Agr. Chem.*, 24 (1941), 751–754. (A. P.-C.)

Ferrous and Ferric Iron in Minerals—Micro-Analytical Method for the Estimation of. A micro-analytical method for the estimation of ferrous and ferric iron in the same sample of mineral has been proposed. The method consists in decomposing the mineral with a mixture of hydrochloric and hydrofluoric acid in a Pyrex glass vessel and titrating the ferrous iron in the resulting solution with ceric sulfate and the total iron subsequently with titanous chloride. Two sets of redox indicators have been used, namely (1) ferrous *o*-phenanthroline complex with methylene blue and (2) *N*-phenylanthranilic acid with potassium thiocyanate. The method has been successfully used for the micro-estimation of ferric and ferrous iron in minerals such as magnetite, ilmenite, etc.—JYOTIRMOY DAS-GUPTA. *J. Indian Chem. Soc.*, 18 (1941), 375. (F. J. S.)

Fluorine Compounds—Determination of, in Insecticides. In using the methods recently adopted by the A. O. A. C. special precautions are required in some cases. In the analysis of silicofluorides that are more or less volatile on heating (*e. g.*, magnesium and sodium silicofluorides) the combined sample and fusion mixture should be covered with a heavy layer (2 to 3 Gm.) of the alkali carbonates to prevent possible loss of fluorine by volatilization before the melt is effected. During the washing of the gelatinous zinc precipitate it should be returned to the beaker 3 times, instead of once or twice. After the lead chlorofluoride is precipitated, it should be allowed to stand overnight in a refrigerator or for 1 hr. in an ice bath in order to reduce its solubility in the aqueous solution; this is particularly necessary in summer and in labs. in which the temperature is above normal.—C. G. DONOVAN. *J. Assoc. Official Agr. Chem.*, 24 (1941), 653–654. (A. P.-C.)

Fluorine—Determination of, in Water. The present A. O. A. C. thorium nitrate titration method (*A. O. A. C. Methods of Analysis*, (1940), 529) was modified as follows: addition of 1 cc. of 0.1% hydroxylamine hydrochloride to the aliquot titrated; use of a standard color comparison tube; running a blank, on each distillation for fluorine; evaporation of the sample before distillation in porcelain or platinum over a Bunsen flame just below the boiling point; preliminary determination of chlorides and their precipitation in the distillation flask with silver perchlorate. The modified method was studied collaboratively, the results justifying the changes. The method is especially applicable to solutions containing 1.00 to 3.00 p. p. m. of fluorine, in which range the expected error will be 3.3% to 4.6% and the expected standard deviation 0.071 to 0.230.—A. E. MIX. *J. Assoc. Official Agr. Chem.*, 24 (1941), 540–544. (A. P.-C.)

Fluorine—Determination of Traces of. A study of the distillation blank or "positive perchlorate error" when pure perchloric acid is carried through a Willard and Winter fluorine distillation indicated

that, even when all possible precautions are taken, there was still a small blank which was taken to be actually fluorine; it did not come from the perchloric acid, but was probably leached from the glass of the still and volatilized during the distillation. Analytical figures are presented, illustrating the necessity of taking the distillation blank into account when small samples of comparatively low fluorine content are analyzed. A similar blank is also present in sulfuric acid distillation. A 100% recovery is practically never obtained when quantities of up to 200γ of fluorine are distilled from perchloric acid and 150 cc. of distillate is collected at 135° C.; errors of the methods used would hardly account for the losses. The case is somewhat better with sulfuric acid distillation, but here also a slight repression is noted. The collection of larger volumes of distillate does not solve the difficulty. When the still is fumed out with boiling sulfuric acid and rinsed thoroughly with water immediately before use, recoveries with perchloric acid distillation are increased, but still do not reach 100%. Dahle's "back titration" procedure for the thorium nitrate method (*J. Assoc. Official Agr. Chem.*, 21 (1938), 468) was used as a standard method by the author, with the only modification that 1 cc. of 0.1% hydroxylamine hydrochloride was added to the distillate aliquot before adding the dye, to eliminate possible traces of chlorine from the perchloric acid distillation. Owing to the fact that introduction of excessive amounts of sodium ion as standard sodium fluoride in the back titration develops a "salt effect" with attendant off shades and sluggish end-points, there is an upper limit to the quantity of fluorine that can be titrated in a single tube, which is about 50γ for a 50-cc. Nessler tube and about 80γ for a 100-cc. tube. An unexplained tendency to undertitrate slightly was noted. As a check upon results by this method, the aluminum-aluminon method has been developed, which is about equal in scope and accuracy to the thorium nitrate method. As with all colorimetric fluorine methods, the quantity of fluorine is derived from its bleaching effect upon a colored complex. In its present form (technique described in detail) the method uses the neutral wedge photometer with a filter centered at 524 mμ and a 100-mm. cell, and has been adapted with success only to the distillates from completely ashed samples. Interferences are phosphates, nitrates, nitrites and other reducing substances such as sulfur dioxide; sulfates do not interfere; free chlorine interferes by bleaching the lake color, and possible traces of chlorine are discharged as in the titration procedure by adding 1 cc. of 0.1% hydroxylamine hydrochloride. In the preparation of samples containing organic matter a fluorine fixative must be used, the most suitable being calcium hydroxide. The danger of loss of fluorine with certain fatty and proteinaceous materials through improper "wetting" by a fixative solution may be eliminated to a large extent by a double distillation, first from sulfuric and then from perchloric acid, the first distillate being treated with excess of the lime-water fixative, evaporated to dryness, ashed for one to two hrs. at 550° to 600° C., taken up and redistilled; adequate amounts of silver sulfate or perchlorate should be used to prevent evolution of hydrochloric acid. The double-distillation procedure is especially indicated with materials high in phosphates in order to minimize distillation of phosphoric acid, which interferes in the determination. Sulfuric acid cannot be used for the first distillation with products high in calcium because of the precipitation of calcium sulfate in the distillation flask; here perchloric acid may often be used, and if the temperature is kept below 150° C. danger of explosion in the distillation is minimized. *Incidental precautions:* Use platinum for all evaporations and ashings as porcelain has been found to contribute small quantities of fluorine. Some material, presumably dissolved aluminum silicate, causes low results in the titration when alkaline solutions are evaporated in porcelain. To avoid contamination in the muffle, cover the dishes suitably with Pyrex petri dishes. Make filtrations through carefully cleaned fritted-glass filters to avoid contamination with hydrofluoric acid-treated filter papers.—P. A. CLIFFORD. *J. Assoc. Official Agr. Chem.*, 24 (1941), 350-363. (A. P.-C.)

Fluorine in Organic Compounds—Assay of. The author gives a new method and describes the apparatus for determining the fluorine content in certain organic fluorine compounds. The sample is completely destroyed by $KNO_3+H_2SO_4$ whereby HF is generated in a stoppered flask. The loss in weight of the etched flask shows the original F content. The method requires no expensive apparatus and may be carried out in 4 hrs.—DIRK H. BRAUNS. *J. Research Natl. Bur. Standards*, 27 (1941), 105-111. (W. T. S.)

Fumigation Residues—Determination of, in Foods. A thorough investigation was made of the phenolphthalein method and of the thiocyanate method for the final determination of hydrocyanic acid, and a detailed description is given of a photocolorimetric technique for each method that was found satisfactory. In the thiocyanate method the use of acetic acid for decomposing the sulfide and separating the sulfur was found to be more satisfactory than that of mineral acid. The only objection found to the method is the high blank obtained on the reagents, which appears to be contributed almost entirely by the sodium sulfide used since all the other reagents were used in the standards and gave no such blank. Plotted results on samples containing known added quantities of potassium cyanide gave a curve as straight as that obtained on the standards and parallel to it. The advantages of the phenolphthalein method over the thiocyanate method are rapidity, ease of application and low blank determination. On the other hand, while it is even more delicate, it is more erratic, and the points do not fall so well on the curve; it has the further disadvantage of not being specific for cyanide. With amounts of hydrocyanic acid greater than 80 to 90 γ the curve produced by plotting the amounts of hydrocyanic acid against photometric readings appears to be parabolic, but up to this amount the curve appears to be practically a straight line. With 13.5 to 81.0 γ hydrocyanic acid the thiocyanate method gave recoveries of 96% to 99.6%; with 8.1 to 54.0 γ hydrocyanic acid, the phenolphthalein method gave recoveries of 88% to 99%.—W. O. WINKLER. *J. Assoc. Official Agr. Chem.*, 24 (1941), 380-383. (A. P.-C.)

Gas Identification. A lecture covering grouping and detection of chemical warfare agents, including chlorine, phosgene, chloropicrin, hydrocyanic acid, chlorarsines, carbon monoxide, mustard gas and Lewisite.—G. LEWI. *Chemistry and Industry*, 60 (1941), 374-377. (E. G. V.)

Glycerol—Effect of, on Distillation Method for Water. The determination of water in soaps by the distillation method is affected by the presence of glycerol. The error is negligible when benzene or toluene is the distillation medium, but appreciable when xylene is used.—R. B. TRUSLER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 509-510. (E. G. V.)

Iodate and Iodine—Photoelectric Microdetermination of. The photoelectric microdetermination of iodate and iodine (Sendroy 1939) has been simplified and made more convenient by increasing the sensitivity of yellow color readings to a point approximating that of blue color readings in very di-

lute solutions. This has been done by the use of several selective filters efficient at the lower wave lengths in the visible or in the near ultraviolet portion of the spectrum. A further increase in sensitivity was obtained by the addition of KI or ethyl alcohol to the solutions analyzed. A simple modification of Evelyn's (1936) photoelectric colorimeter, increasing the range of its readings to the near ultraviolet, is described.—JULIUS SENDROY, JR., and ALF S. ALVING. *J. Biol. Chem.*, 142 (1942), 159. (F. J. S.)

Iodate Ion—Determination of, in the Presence of Cupric Ion. Mixtures containing known amounts of potassium iodate and copper sulfate were prepared. An excess of sodium pyrophosphate (free from reducing agent), acetic acid, and potassium iodide solution were added, in this order, to each of the solutions to be titrated. The iodine was liberated slowly and was titrated against standard sodium thiosulfate solution, using starch as the indicator. After iodine had ceased to separate, the solution was set aside and kept for 24 hrs. in the dark. Under these conditions no more iodine was evolved, showing that the blue copper complex did not decompose to react with iodide.—P. L. KAPUR and M. R. VERMA. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 338. (E. G. V.)

Iodine and Boron—Determination of, in Plants. A collaborative study was made of the determination of boron in various kinds of plant tissues by the quinalizarin method of Berger and Truog (*Ind. Eng. Chem., Anal. Ed.*, 11 (1939), 540–545), slightly modified as follows: ignite a 0.25 to 1.00 Gm. sample to a white or gray ash at 450° in either porcelain or platinum, after cooling dissolve the ash in 5 cc. of approximately 0.36 times normal sulfuric acid (10 cc. of 95% acid per L.), mix thoroughly, allow the residue to settle or centrifuge to clarify; transfer a 1-cc. aliquot of the clear supernatant liquid to a comparison tube, add 9 cc. of acid-indicator solution (98.5% by weight sulfuric acid containing 0.008 Gm. of quinalizarin per L.) and mix thoröughly; stopper, and let stand 30 min. before making a comparison with standards (containing from 0 to 0.005 mg. of boron per cc. in 0.18 times normal sulfuric acid) either visually or by the use of a photoelectric colorimeter. The results showed the method to be quite satisfactory. Some objections may be made because of the very strong sulfuric acid required, but the speed and simplicity of the procedure should offset such objections.—J. S. MCHARGUE and W. S. HODGKISS. *J. Assoc. Official Agr. Chem.*, 24 (1941), 518–520. (A. P.-C.)

Iodine in Desiccated Thyroid—Determination of. A simplified method for the determination of iodine in desiccated thyroid is reported. A modification of Leipert's wet digestion method is preferable for very accurate work especially if the amounts of iodine are small but for ordinary purposes the suggested method serves. It involves ignition in a simple alkaline fusion mixture free from oxidizing agents. Recoveries of iodine are comparable with those obtained by the U. S. P. XI thyroid assay and within 3% of calculated theoretical values on known mixtures of low iodine content. There is no interference by hypochlorite and nitrate as in U. S. P. XI procedure. Four samples of desiccated thyroid and two standardized mixtures of diiodotyrosine and casein were used and the procedure was compared with three others: U. S. P. XI, the Burnett and Warkow modification and the Manganese Dioxide method. Figures for these are tabulated. The final titration should be performed at a *p*H of 2.20 to 2.50. The assay of thyroid by the proposed method falls within this range. The authors found desiccated thyroid that had been stored for 5 yrs. with no special precautions showed a stable iodine content.—FREDERICK F. JOHNSON and HARRY A. NELSON. *Jour. A. Ph. A.*, 30 (1941), 625. (Z. M. C.)

Iron—Spectrophotometric Determination of. I. Use of Mercaptoacetic Acid. An accurate spectrophotometric method for the determination of iron with mercaptoacetic acid has been developed experimentally.—RUTH ADELE KOENIG and C. R. JOHNSON. *J. Biol. Chem.*, 142 (1942), 233. (F. J. S.)

Iron—Use of Silicomolybdic Acid Indicator before Volumetric Oxidation of. A new method for the volumetric determination of iron uses silicomolybdic acid indicator in showing the beginning of ferrous ion oxidation. N-phenylanthranilic acid indicator shows the end of that oxidation with standard potassium dichromate solution. The volume of solution used between the two color changes is equivalent to the iron being determined. The silicomolybdic acid indicator is prepared by adding a solution of 35 Gm. of ammonium molybdate tetrahydrate to 13.2 Gm. of sodium silicate pentahydrate dissolved in water, then adding 49 cc. of concentrated sulfuric acid and making the whole volume up to 2 L.—A. C. TITUS and C. W. SILL. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 416–418. (E. G. V.)

Lead Oleate Plaster and Ointment of Lead Oleate—Assay of. The following method, embodying features of both the Wetherell (*Quart. J. Pharm. Pharmacol.*, 8 (1935), 461–468) and the Babitsch (*Arch. Pharm.*, 271 (1933), 446–448) methods, was found to give reliable results with both the plaster and the ointment. Accurately weigh approximately 1 Gm. of plaster (2 Gm. of ointment) into a tared 125-cc. Erlenmeyer flask; determine water by drying at 100° to 110° C. for 3 hrs.; add 10 cc. of 36% acetic acid, heat on the water bath until the mass liquefies, add 25 cc. of hot water, shake vigorously, chill, puncture the solidified fatty layer, transfer the aqueous solution of lead salt through a small pledget of cotton to a 250-cc. Erlenmeyer flask, repeat 3 times extraction with 5 cc. of acid + 25 cc. of water; heat the combined aqueous extracts to boiling, add 50 cc. of decinormal oxalic acid, shake, cool, filter through paper, wash with 5 25-cc. portions of water, return the paper to the precipitation flask, add 10 to 15 cc. of 10% sulfuric acid and 75 cc. of distilled water, heat almost to boiling, add 10 cc. of 10% sulfuric acid, and titrate at about 70° C. with decinormal potassium permanganate, of which 1 cc. = 0.01116 Gm. of lead oxide.—HENRY M. BURLAGE. *J. Assoc. Official Agr. Chem.*, 23 (1940), 787–789. (A. P.-C.)

Limonin—Identity of Obaculactone, Evodin and Dictamnolactone with. The identity of obaculactone, evodin, dictamnolactone, citrolimonin and limonin is established by means of physical data.—M. S. SCHECHTER and H. L. HALLER. *J. Am. Chem. Soc.*, 62 (1940), 1307–1309. (E. B. S.)

Mercuric Iodide and Iodine—Recovery of, from Nesslerized Solutions. Mercuric iodide and free iodine were precipitated by adding sulfuric acid and sodium dichromate to the nesslerized solution. The free iodine was then separated from the mercuric iodide by a distillation procedure. The method is simple and inexpensive, and results in the almost quantitative recovery of iodine and of mercury as mercuric iodide.—G. W. SCHIMPFF and R. E. POTTINGER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 337–338. (E. G. V.)

Mercuric Oxide Ointment—Report on the Analysis of. A study of the U. S. P. XI sulfide-precipitation method for the assay of mercuric oxide in yellow mercuric oxide ointment led to the development of the following manipulative technique: weigh the sample on a 2 × 2 in. glassine paper, roll into a

pellet, insert into a horizontally placed separator near the top stopper; immerse the side of the separator held in horizontal position in a water bath at 60° to 80° C. until a portion of the ointment flows; cool to cause the entire sample to adhere to the side near the top of the separator, add about 75 cc. of ether, warm again in the horizontal position (holding the top stopper loosely in the opening) until the ether boils gently; shake vigorously for 5 to 10 sec., release the pressure, and repeat the warming and shaking until the mixture is uniformly cloudy and not lumpy; add the acid immediately; shake the acid-ether mixture 3 times per sec. for 2 min.; wash with 8 to 10 10-cc. portions of water or until the turbidity test with silver nitrate shows less than that caused by 0.15 mg. of mercuric chloride; if a second portion of acid is used to hasten or complete the extraction make four water washings after the first acid portion and at least four after the second; pass in hydrogen sulfide until the supernatant liquid is clear; remove sulfur from the mercuric sulfide by washing as in the U. S. P. XI assay for mercuric chloride. Extraction with two portions of acid gave an average recovery 1 to 1.4% higher than with a single portion and, when corrected for the blank sulfide precipitate obtained on the ointment base, extraction with two portions of acid gave recoveries of 99.9% to 100.0%.—H. O. MORAW. *J. Assoc. Official Agr. Chem.*, 23 (1940), 758–761. (A. P.-C.)

Mercury, Bismuth and Zinc—Separation and Estimation of, in Skin Bleaches. Mercury and bismuth may be separated by precipitation of mercury as mercuric sulfide in 1 + 1 hydrochloric acid. Either bismuth alone or bismuth and mercury together are quantitatively precipitated by hydrogen sulfide in 1 + 3 hydrochloric acid. Mercury is quantitatively separated from zinc by precipitation with hydrogen sulfide in 1 + 4 hydrochloric acid, but in 1 + 19 hydrochloric acid appreciable amounts of zinc are coprecipitated. On the basis of these findings, a technique has been evolved and is described in detail, in which vehicle (usually petrolatum) is extracted with chloroform and weighed, mercury is weighed as mercuric sulfide, bismuth is weighed as the trisulfide and zinc is precipitated as sulfide and determined by the phosphate method.—GEO. MCCLELLAN. *J. Assoc. Official Agr. Chem.*, 24 (1941), 728–730. (A. P.-C.)

Metals in Some Pectinates—Determination of. Procedures are given for the determination of the metal content of bismuth, cobalt and nickel pectinates, comparing the photometric and spectrographic methods.—P. AMBLER and M. A. GRIGGS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 102–103. (E. G. V.)

Methenamine—Quantitative Determination of. The many methods suggested for quantitative determination of methenamine depend on one or both of the following properties: (1) when hydrolyzed with acids it yields formaldehyde and the ammonium salt of the particular acid used; and (2) it forms addition products with the halogens, picric acid and uranyl sulfate. Methods are tedious and not very accurate. Experimental work included the U. S. P. XI method and several modifications of it, that of Brown and Otten, Sugiura and Falk, Korostishev-'ska, a Kjeldahl determination, oxidation with hydrogen peroxide, argentometric method, potassium bromate method, alkaline hypobromite method and a calcium hypochlorite method. The U. S. P. method is slow and use of a smaller sample and tenth-normal solutions gives greater accuracy. Precipitation methods were inaccurate. Both hydrogen peroxide and potassium bromate gave poor results. The alkaline hypobromite method was rapid but not entirely satisfactory. The calcium hypochlorite method seems best suited for methenamine itself and for certain mixtures containing it. It is simple, comparatively rapid and accurate.—EDMUND F. SLOWICK and RAY S. KELLEY. *Jour. A. Ph. A.*, 31 (1942), 15. (Z. M. C.)

Methionine—Determination of, in Certain Mixtures. The purity of methionine can be determined with an accuracy of ±0.1% by oxidation with hydrogen peroxide under specified conditions. The principle of this method is applicable to the determination of methionine in certain other analytical solutions, since other natural amino acids, except trytophane, cysteine and cystine to a small extent, do not seem to interfere. Data on the stability of hydrogen peroxide in 1 to 4 molar perchloric acid solutions are included.—J. J. KOLB and G. TOENNIES. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 723–724. (E. G. V.)

Methoxyl Determination—Studies of. A modified Pregl apparatus is described. Errors in determination are traced to reagents, especially hydriodic acid, the preparation of which is described.—B. E. CHRISTENSEN, L. FRIEDMAN and Y. SATO. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 276–277. (E. G. V.)

Micro and Semimicro Kjeldahl Nitrogen Method—Collaborative Report on. Collaborative results obtained by the method recommended by Clark (*J. Assoc. Official Agr. Chem.*, 24 (1941), 641–647) using 5 to 20 mg. of a variety of compounds are presented and substantiate the claims made for the method by Clark (at least in so far as the compounds analyzed are concerned).—FRED ACREE, JR. *J. Assoc. Official Agr. Chem.*, 24 (1941), 649–651. (A. P.-C.)

Microdiffusion Methods Based on the Bisulfite Reaction. II. Determination of Lactic Acid by Oxidation with Ceric Sulfate. The Conway microdiffusion unit has been applied to the determination of lactic acid in blood filtrates, tissue extracts and urine. The lactic acid is oxidized quantitatively by ceric sulfate in the outer chamber of the apparatus and the resulting acetaldehyde passes by gaseous diffusion into the central chamber where it is absorbed by sodium bisulfite solution. The diffusion is complete in about 5 hrs. at 25° or 2 hrs at 50°. The bound bisulfite is determined iodometrically. **III. Determination of Threonine by Oxidation with Periodate.** A microdiffusion method for the quantitative determination of threonine in protein hydrolysates is described. A sample of hydrolysate is treated with neutral periodate solution in the outer chamber of a Conway unit and the acetaldehyde which results from the oxidation of threonine diffuses into bisulfite solution in the central chamber. After 4 or 5 hrs., the bisulfite which is bound by the acetaldehyde is determined iodometrically. The percentages of threonine in casein, gliadin, lactoglobulin and male and female chimpanzee hair are reported.—THEODORE WINNICK. *J. Biol. Chem.*, 142 (1942), 451, 461. (F. J. S.)

Microkjeldahl Nitrogen Method. A detailed description is given of the apparatus and technique of a microkjeldahl nitrogen determination which has been used successfully over a period of years, in both research and industrial laboratories and which has been checked by means of collaborative study. The apparatus consists of a digester and of the Parnas-Wagner Kjeldahl apparatus (*Biochem. Z.*, 125 (1921), 253–256), and the method is the Gunning-Arnold-Dyer modification of combustion with the boric acid method of titrating ammonia. The system is applicable to practically all classes of animal and vegetable materials, pyridine and quinoline derivatives, purines, pyrimidines, amines, amides, oximes and such substances as carbazole, hydrazobenzene and indigotin; by modifying the method

according to Friedrich, *et al.* (*Z. Physiol. Chem.*, 216 (1933), 68-76), the nitrogen in hydrazines, osazones and nitro, nitroso, azo and even certain diazo compounds may be determined with a high degree of precision. The method is discussed and analytical data are presented to justify it and prove its reliability.—E. P. CLARK. *J. Assoc. Official Agr. Chem.*, 24 (1941), 641-647. (A. P.-C.)

Mono- and Di-Ethanolamines—Detection and Determination of. The following modifications of Rimini's test for primary aliphatic amines and of Simon's test for secondary amines have been developed: to a solution containing about 0.2% of monoethanolamine add an equal volume of an aqueous 1% sodium nitroprusside and 20% acetone solution, add 2% sodium bicarbonate solution to alkaline reaction; a purple color gradually develops in the presence of mono-ethanolamine. The reagent for diethanolamine is an aqueous 1% sodium nitroprusside 10% acetaldehyde solution, and a blue color is formed in the presence of diethanolamine. The quantitative determination (technique of the method described in detail) is based on the conversion of the mono- and di-ethanolamines into corresponding *p*-bromobenzene sulfonyl derivatives (triethanolamine does not give this reaction), extracting the diethanolamine derivative with chloroform from alkaline solution and weighing (weight × 0.324 = diethanolamine; derivative melts at 105° C.); then acidifying the extracted solution, extracting the monoethanolamine derivative with chloroform and weighing (weight × 0.218 = monoethanolamine; derivative melts at 94° C.). A blank determination should be made on the *p*-bromobenzene sulfonyl chloride reagent. For the recovery of mono- and di-ethanolamines from emulsions such as cosmetic creams an aqueous extract may be used; substances extractable with chloroform which would interfere may be removed by preliminary extractions from either acid or alkaline solutions before preparation of the derivatives, the mono- and di-ethanolamines remaining in the aqueous solution; ammonia should not be added since it forms a sulfonamide corresponding to a primary amine derivative. The crystalline precipitates (not described) obtained with Kraut's reagent and diethanolamine, and with phosphotungstic acid and both mono- and di-ethanolamines are useful as microchemical tests. On known quantities of material, the method gave recoveries of 97.0% to 101.0% of di- and of 100.5 to 102.0% of monoethanolamines.—IRWIN S. SHUPE. *J. Assoc. Official Agr. Chem.*, 24 (1941), 754-757.

(A. P.-C.)

Morpholine—Determination of. Morpholine can be quantitatively titrated with acids and methyl red indicator. With excess alkali it can be quantitatively recovered in a small volume by steam distillation or direct distillation. From an aqueous solution containing 20% or more of sodium hydroxide, morpholine can be extracted with ether without removal of any fixed alkali; the ether solution of morpholine can be mixed with water and titrated with standard acid, or may be converted into the dithiocarbamate by treatment with carbon disulfide. Morpholine reacts with silver nitrate similarly to ammonia, forming a precipitate that dissolves in excess of the base; with copper, nickel and cobalt salts it forms no soluble complexes; it does not react with Nessler's reagent. A modified Simon's color test with sodium nitroprusside and acetaldehyde reagent gives a blue color with iodine in dilutions up to 1 in 700; ammonia gives a light orange color with the reagent and decreases the sensitivity of the test. In neutral or slightly acid solution morpholine gives crystalline precipitates useful for microchemical tests with modified Kraut's reagent (dissolve 7 Gm. of bismuth subcarbonate in 20 cc. of concentrated hydrochloric acid, add to a solution of 28 Gm. of potassium iodide in 50 cc. of water and dilute to 100 cc.), platinum chloride, gold chloride, silicotungstic acid, phosphotungstic acid, Reinecke salt (ammonium tetrathiocyano-diammono-chromate). The benzene sulfonyl derivative (m. p. 119° C.) and the *p*-bromobenzene sulfonyl derivative (m. p. 153° C.) are stable and easily prepared (method described in detail), and can be used for the quantitative estimation of morpholine. From either aqueous or ether solution, morpholine reacts with carbon disulfide to form a crystalline dithiocarbamate, practically insoluble in ether, slightly soluble in alcohol, soluble in water, sublimes without melting above 100° C., reduces potassium ferricyanide in aqueous solution with formation of a water-insoluble derivative (probably a thiuram disulfide) which can be recrystallized from hot alcohol and melts at 150° to 151° C.; the same derivative can be obtained directly from an aqueous solution of morpholine and potassium ferricyanide by treatment with carbon disulfide. Isolation of morpholine from creams and lotions is often complicated by the presence of other emulsifying agents, and preliminary separation for their removal may be necessary. Creams may be heated with 10 cc. of 5% hydrochloric acid to break emulsions and decompose soaps, and the oily material may then be dissolved in ether and the aqueous extracts steam distilled. Two types of creams and two of lotions were prepared, and recoveries of 95% to 99% of the morpholine present were obtained.—IRWIN S. SHUPE. *J. Assoc. Official Agr. Chem.*, 23 (1940), 824-831.

(A. P.-C.)

Munson and Walker's Reducing Sugar Tables—Errors of, and the Precision of Their Method. In Hammond's (*J. Research Natl. Bur. Standards*, 24 (1940), 579; *Res. Paper* 1301) revision of the Munson and Walker tables (*J. Am. Chem. Soc.*, 28 (1906), 663) important deviations in many of the copper values were disclosed. The present investigation corroborates Hammond's measurements within the limits of experimental error. The differences between Hammond's and Munson and Walker's values for copper are due almost entirely to the respective methods of estimating copper, the former determining reduced copper by electrolysis and the latter weighing the precipitated cuprous oxide. It is shown that cuprous oxide is contaminated with organic decomposition products even when pure sugars are analyzed, and the amount of contamination is almost exactly equal to the difference between Hammond's and Munson and Walker's copper values. It is shown that reduced copper should be determined by analysis and not by direct weighing of curpous oxide, and that Hammond's tables should be substituted for those of Munson and Walker. Erb and Zerban's (*Ind. Eng. Chem., Anal. Ed.*, 10 (1938), 246) analyses of 0.4 Gm. of sucrose-invert sugar mixtures are in agreement with Hammond's except within a short range of lower concentrations, and analyses are presented that are in agreement with Hammond's within this range. The various methods for the determination of copper are discussed. The main reliance during the investigation was the iodometric method in acetic acid solution with the potassium iodide concentration (4.2 Gm. per 100 cc.) specified by Shaffer and Hartmann (*J. Biol. Chem.*, 45 (1921), 362) with the addition of thiocyanate at the end of the titration as specified by Foote and Vance (*J. Am. Chem. Soc.*, 57 (1935), 845). A method of determining cuprous oxide by oxidation with excess potassium dichromate and back titration with ferrous sulfate to a colorimetric or electrometric end-point is described. The permanganate method as modified by Schoorl and Regenbogen (*Z. Ver. deut. Zuckerind.*, 67 (1917), 563) is shown to give accurate results. The pre-

cision of Munson and Walker's method as indicated by Hammond's and by the present authors' independent analyses is shown to be about 0.2%; if the concentrations of sugar are restricted to the range 69 to 207 mg. of reducing sugar, the average precision appears to be about 0.1%.—RICHARD F. JACKSON and EMMA J. MCDONALD. *J. Assoc. Official Agr. Chem.*, 24 (1941), 767–788. (A. P.-C.)

Muscovite Mica—Microchemical Investigations on Spotted. The chemical nature of some black spots often found in the muscovite mica of Indian origin has been investigated and from their microanalytical study it has been proved that they consist of crystallized magnetite imbedded between the mica sheets.—JYOTIRMOY DAS-GUPTA. *J. Indian Chem. Soc.*, 18 (1941), 381. (F. J. S.)

"Naphthalene"—Determination of, in Denatured Salt. The solvent extraction and picrate methods recorded in literature for the estimation of naphthalene are not suitable for the determination of percentages of commercial "naphthalene" in denatured salt. A method has been suggested which involves the separation of the "naphthalene" by steam distillation, collection of the condensable solids on a tared filter, followed by drying in a desiccator in an atmosphere saturated with naphthalene vapor. The method yields concordant and reliable results even when the drying is done at as high a temperature as 40° (104° F.).—H. B. DUNNICLIFF. *Ind. & News Ed., J. Indian Chem. Soc.*, 4 (1941), No. 1, 26. (F. J. S.)

National Formulary Ointments—Methods of Assay for. The following assay procedures are offered: *Compound Ointment of Benzoic Acid.*—Transfer a sample of the ointment of 2–2.5 Gm. in weight, accurately weighed, into a separator and dissolve as completely as possible in ether. Extract the mixture twice with sodium hydroxide T.S. and 3 times with distilled water. Acidify the aqueous solution with hydrochloric acid and completely extract with chloroform. Evaporate the chloroformic solution on a steam bath with the aid of a current of air and remove the container as soon as dry. Dissolve the residue, using gentle warming if necessary, in 25 cc. of diluted alcohol, which has been previously neutralized with 0.1 *N* sodium hydroxide using 3 drops of phenolphthalene T.S. as indicator. Titrate this solution with sodium hydroxide (0.1 *N*). The amount of alkali represents the equivalent of both benzoic and salicylic acids. Dilute the titrated solution to 500 cc. with distilled water, mix and then dilute 20 cc. of that solution to 250 cc. with distilled water. Add 0.5 cc. of acetic acid and 1 cc. of ferric chloride solution (2%) to 50 cc. of the final dilution. Fifty cc. of a standard solution of salicylic acid (1 mg. per 50 cc.) should be treated in the same manner. Compare the two solutions in a colorimeter and divide the reading of the standard by that of the sample, and multiply this ratio by 0.125 which gives the weight of salicylic acid in the sample. Divide the weight of the salicylic acid by 0.01381 and subtract this figure from that obtained by multiplying the titration value for combined acids by the solution factor and then multiply this figure by 0.01221 to obtain the weight of benzoic acid in the sample. *Ointment of Calamine.*—Transfer a sample of the ointment (2–4 Gm.) accurately weighed, into a separator. Dissolve the base in ether and add 50 cc. *N* sulfuric acid. Shake until the zinc oxide is dissolved, separate the layers and wash the ether layer three times with distilled water. Combine the aqueous extractions, filter if necessary and titrate with *N* sodium hydroxide using methyl red T. S. as indicator. Each cc. *N* sulfuric acid is equivalent to 0.04069 Gm. ZnO. A special apparatus for weighing and transferring the ointment is described. *Ointment of Camphor.*—Place approximately 5 Gm. of the ointment in a dried tared Erlenmeyer flask (120-cc.) and weigh accurately. Connect the flask and tube in an air oven maintained at 110° C., and pass a steady stream of carbon dioxide through the U-tube into the flask for 2 hrs. The orifice of the gas delivery tube should be about 15 mm. above the surface of the melted ointment. Remove the flask, blow out the remaining carbon dioxide with dry air, cool the flask in a desiccator and weigh. *Ointment of Mild Mercurous Chloride.*—Place about 2.5 Gm. of the ointment in a 250-cc. tared glass-stoppered flask and weigh accurately. Add 100 cc. of chloroform, stopper and shake gently until the ointment base is dissolved. Filter the mixture and wash the residue with small portions of chloroform. Place the filter paper and contents in the glass-stoppered flask, add 25 cc. of water and mix well. Add 50 cc. of 0.1 *N* iodine and 5 Gm. of potassium iodide dissolved in 10 cc. water. Stopper the flask, allow the mixture to stand until complete solution has taken place, then titrate the residual iodine with 0.1 *N* sodium thiosulfate solution, using starch T.S. as indicator. Each cc. 0.1 *N* iodine is equivalent to 0.02361 Gm. of HgCl. *Ointment of Mercuric Nitrate.*—Transfer an accurately weighed sample of the ointment (3–5 Gm.) to a Kjeldahl flask and add 40 cc. of nitric acid (1 + 1). Place a funnel in the neck of the flask and heat for 1½ hrs., boiling just enough to maintain agitation. Cool the flask under running water and swirl the flask to prevent the solid matter from solidifying into one large mass. Dilute to 200 cc. and filter through an unwetted filter. Transfer 100 cc. of the filtrate to a Kjeldahl flask, add 10 cc. of sulfuric acid and heat until white fumes of SO_3 are visible. Immediately remove the flame and allow to cool. Add 5 cc. of nitric acid and heat again at a medium rate until the rate of boiling decreases markedly. Cool, dilute to 100 cc. and cool again if necessary. Titrate with 0.1 *N* ammonium thiocyanate, using ferric ammonium sulfate T.S. as the indicator. Each cc. 0.1 *N* ammonium thiocyanate is equivalent to 0.01003 Gm. Hg. *Ointment of Red Mercuric Oxide.*—Transfer the ointment (2–3 Gm.) accurately weighed, into a separator. Dissolve the ointment base in ether and extract the mixture twice with 30-cc. portions of 5% nitric acid and twice with 30-cc. portions of distilled water. Titrate the combined extracts with 0.1 *N* ammonium thiocyanate using ferric ammonium sulfate T.S. as indicator. Each cc. 0.1 *N* NH_4CNS is equivalent to 0.01083 Gm. HgO. *Stainless Iodine Ointment.*—Mix about 2 Gm. of the ointment with 2 Gm. anhydrous sodium carbonate; place the mixture in a small porcelain crucible, and completely fill the crucible with anhydrous sodium carbonate well pressed down; invert the crucible and contents in a larger porcelain crucible, and add sufficient anhydrous sodium carbonate to seal the junction of the two crucibles. Heat rapidly and stongly over a Bunsen burner and continue the heating for 20 min. Allow the crucibles and contents to cool and dissolve the residue in 100 cc. of hot distilled water. Filter the hot solution into a 500-cc. flask and wash the beaker, crucibles and filter with three 10-cc. portions of hot distilled water. Allow the filtrate and washings to cool, and add hydrochloric acid cautiously until effervescence ceases, then add an equal volume of hydrochloric acid, and titrate with *M*/20 potassium iodate, shaking vigorously, until the dark brown solution, which is formed, becomes light brown; add 5 cc. of chloroform and continue the titration until the chloroform becomes colorless and the supernatant liquid is clear yellow. One cc. *M*/20 potassium iodate is equivalent to 0.01269 Gm. I. *Ointment of Lead Oleate.*—Place a sample of the ointment (2–3 Gm.), accurately weighed, in a crucible, and heat until melted. Add shredded ashless filter paper, gradually burn the ointment base and then ignite. Allow the crucible

to cool, add diluted nitric acid (1 + 1) and heat until the residue is completely dissolved. Allow to cool and then add 1 cc. sulfuric acid. Evaporate to dryness and ignite. The weight of lead sulfate × 0.6834 = Pb in the sample. *Ointment of Potassium Iodide.*—Transfer a sample of the ointment (2–3 Gm.) accurately weighed, to a separator. Dissolve the base in ether and extract the mixture with water. Dissolve 2 Gm. of potassium carbonate in the aqueous solution and heat on a water bath until the dissolved ether is expelled and the volume is about 75 cc. Add an aqueous solution of potassium permanganate (1 in 20) until the hot liquid remains permanently pink. Add just enough alcohol to remove the pink tint, cool to 25° C., then add sufficient distilled water, to make exactly 100 cc. Filter the mixture through a filter which has not been previously moistened, rejecting the first 25 cc. of filtrate. To 50 cc. of the subsequently clear filtrate add 10 cc. potassium iodide T.S., acidify with diluted sulfuric acid and titrate with 0.1 *N* sodium thiosulfate. Each cc. 0.1 *N* sodium thiosulfate = 0.002767 Gm. KI. *Alkaline Ointment of Sulfur.*—Transfer from 0.4–0.6 Gm. of the sample, accurately weighed into a Kjeldahl flask. Add 30 cc. of the distilled water, 5 Gm. of potassium chlorate and slowly, 30 cc. of nitric acid. Evaporate the mixture to 5–10 cc. Transfer the residue to a suitable beaker with distilled water and evaporate to 5–10 cc. Add 25 cc. hydrochloric acid and again evaporate to 5–10 cc. Dilute with distilled water, heat to boiling, filter and wash. The filtrate should have a volume of 300–500 cc. Heat to boiling and add barium chloride T.S. slowly. Digest the mixture until the barium sulfate is aggregated and allow to settle. Filter through a Gooch crucible, wash, ignite and weigh. The weight of barium sulfate × 0.1374 = S in the sample, correction being made for any S derived from the reagents. *Compound Ointment of Sulfur.*—Procedure the same as for the Alkaline Ointment. *Ointment of Zinc Stearate.*—Transfer a sample of the ointment (2–4 Gm.) accurately weighed, into a 50-cc. centrifuge tube. Suspend the ointment in petroleum ether and centrifuge until the zinc stearate settles, decant the supernatant liquid and resuspend the zinc stearate in petroleum ether. Repeat this procedure until the compound has been completely separated from the ointment base. Add 25 cc. of *N* sulfuric acid and immerse the tube in boiling water. Heat until a clear oily liquid is on top of the solution and no zinc stearate remains. Cool and transfer to a separator using first distilled water and then ether to rinse the tube. Shake the funnel until the stearic acid is dissolved and then separate the layers. Wash the ether layer 3 times with distilled water. Combine the aqueous extractions and titrate with *N* sodium hydroxide using methyl red T.S. as indicator. Each cc. *N* sulfuric acid = 0.04069 Gm. ZnO.—R. K. SNYDER. *Bull. Natl. Formulary Committee*, 9 (1941), 322–343. (H. M. B.)

Nessler's Reagent—Preparation of. The reagent should have 10 mols of potassium hydroxide for each equivalent of mercuric ion in solution with 5% excess of potassium iodide over and above that called for by the hypothetical compound potassium mercuric iodide. Furthermore, it should stand several days before being used, to permit any precipitate to settle out. It is recommended that 45.5 Gm. of mercuric iodide and 34.9 Gm. of potassium iodide be dissolved in as little water as is needed, 112 Gm. of potassium hydroxide (140 cc. of an almost saturated solution, specific gravity 15°/4° = 1.538) added, and the whole diluted to 1 L. This solution is 0.2 *N* with respect to the mercury content. In the Nessler test, 5 cc. of this reagent to 100 cc. of final volume are used, and the color comparison with the standards are made 30 min. after mixing.—A. P. VANSELOW. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 516–517. (E. G. V.)

Nicotine—Turbidimetric Determination of, as Phosphotungstate. A satisfactory procedure has been worked out for the turbidimetric determination of nicotine as phosphotungstate, in conjunction with the warming and cooling technique used by Kozu (*J. Agr. Chem. Soc. Japan*, 7 (1931), 977–983) in connection with nicotine silicotungstate. Sulfuric acid sensitizes the test, the optimum amount being 0.2 to 0.4 cc. of 1 + 5 acid per 10 cc.; without sulfuric acid the range of the test is 3 to 10γ nicotine; with sulfuric acid it is 1 to 6γ, and the reproducibility of the results is improved. To 10 cc. of solution add 2 cc. of 1 + 5 sulfuric acid and 0.2 cc. of 10% phosphotungstic acid ($P_2O_5 \cdot 24WO_3 \cdot 24H_2O$); clear the suspension by warming (not boiling), restore it by cooling (shaking) in cold water and measure light transmission in a photoelectric photometer using a 7 by $^7/_8$-in. test tube as absorption cell through which the beam of light passes horizontally. For the evaluation of unknown solutions a standard curve is prepared from known nicotine solutions by plotting concentrations against logarithms of photometer readings; this curve is a straight line, indicating a behavior similar to that shown by solutions that follow Beer's law; since the nicotine precipitate has a certain solubility, the transmission remains at 100% (water blank = 100%) up to about 0.5γ of nicotine per cc. The temperature of the cooling water (about room temperature) is not important so long as there is no change (1° to 2°) during a series of determinations. Readings should be made without delay after cooling as that is the moment of greatest turbidity. The secret of obtaining uniform results appears to lie in not shaking the tube too vigorously during cooling—only enough to cool the contents in 1 min.; stronger agitation tends to induce aggregation and the photometer reading will be too high. The solution to be tested should preferably contain only nicotine, as in the distillate from a nicotine distillation. Organic bases and alkaloids, proteins and amino acids and certain metals, such as lead, are precipitated with phosphotungstic acid and should therefore be absent. Organic solvents (alcohol, acetone and ether) exert a solvent action on the precipitate and must be absent. Certain inorganic salts, such as sodium and magnesium sulfates, may be present if the standard solutions also contain them in uniform quantities.—L. N. MARKWOOD. *J. Assoc. Official Agr. Chem.*, 23 (1940), 800–804. (A. P.-C.)

Ointment of Red Mercuric Iodide—Assay of. The method is essentially as follows: Weigh 6 to 10 Gm. of sample on cellophane or glassine paper, place in a separatory funnel, add 50 cc. of ether to dissolve the ointment base, extract with three or four 10 cc. portions of 5% potassium iodide solution, filter each extract in a 250-cc. beaker, add 0.25 Gm. of powdered zinc, let stand 15 min. with frequent stirring, decant through a Gooch crucible and wash by decantation, place the crucible in the original beaker and dissolve the zinc with small portions (about 10 cc. in all) of 1 + 5 nitric acid, when the zinc is nearly all in solution add 10 cc. of nitric acid and heat on the steam bath to complete solution of the mercury; add 3% to 4% potassium permanganate solution until the purple color persists 5 min.; add just sufficient 3% hydrogen peroxide to discharge the color and dissolve the manganese dioxide, cool, add 50 cc. of water and titrate with decinormal ammonium thiocyanate using ferric alum indicator; 1 cc. of decinormal ammonium thiocyanate = 0.02272 Gm. of mercuric iodide. In a collaborative study of the method 97.8% to 100.3% recoveries were obtained.—RUPERT HYATT. *J. Assoc. Official Agr. Chem.*, 23 (1940), 774–775. (A. P.-C.)

Organic Compounds—Identification of. I. Chlorosulfuric Acid as a Reagent for the Identification of Aryl Halides. A method for the identification of aryl halides by means of chlorosulfonylation with chlorosulfuric acid has been shown to yield excellent results. Of thirty-two aryl halides studied, thirty yield characteristic arylsulfonyl chlorides readily converted to the corresponding arylsulfonamides and the other two yield reaction products characteristic of the original compound. Three trichlorobenzenes have been characterized by mono-, di- and tri-nitration and the reaction products of the dinitrotrichlorobenzenes with aniline have found useful derivatives.—E. H. HUNTRESS and F. H. CARTEN. *J. Am. Chem. Soc.*, 62 (1940), 511–514. (E. B. S.)

Organic Compounds—Identification of. II. Piperidyl Derivatives of Aromatic Halogenonitro Compounds. Two standard procedures for the preparation of piperidyl derivatives of aromatic halogenonitro compounds have been developed. All possible piperidyl derivatives of the compounds studied have been isolated and the most favorable conditions for their preparation determined. The structures of certain derivatives have been proved.—M. K. SEIKEL. *J. Am. Chem. Soc.*, 62 (1940), 750–756. (E. B. S.)

Organic Microanalysis—Systematic Qualitative. The general applicability of decigram, centigram and milligram pipettes has been demonstrated for the determination of specific gravities of liquids and solids. The accuracies obtainable under ordinary laboratory conditions are sufficiently high for a safe identification of unknown liquids or solid organic compounds in qualitative organic microanalysis.—H. K. ALBER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 764–767. (E. G. V.)

Organic Reagents and Methods Involving Their Use. A bromate-arsenite procedure has been tested for the estimation of hydroxylamine and found accurate. The hydroxylamine that is liberated from nickel dimethylglyoxime or from copper α-benzoin oxime may be determined by the bromate-arsenite method to give an indirect estimation of copper or nickel. Copper salicylaldoxime is oxidized in a reproducible manner under strictly controlled conditions by the bromate-arsenite procedure. One gram-atom of copper requires 14 equivalents of standard bromate in the titration. The method is suitable for the estimation of semimicroquantities of copper.—N. H. FURMAN and J. F. FLAGG. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 738–740. (E. G. V.)

Paraffin Test as Used in Legal Chemistry. In cases of suspected homicide by shooting, the hands or clothing of the suspect may contain particles of gunpowder. Whether old type or modern smokeless powder, the composition of all of them includes nitrites. Paraffin impressions are made of the hands and sleeves of the suspects, the blocks of paraffin are taken to the laboratory where they are touched with Guttman's reagent, a sulfuric solution of diphenylamine. If nitrites are present a blue color results. Records of 52 cases so tested and verified are presented.—ISRAEL CASTELLANOS. *Vida Nueva (Habana)*, 48 (1941), 155. (G. S. G.)

Phenobarbital—Separation and Determination of, in Solutions. All aqueous solutions of barbiturates may undergo appreciable decomposition, and their analysis requires that the barbituric acid found be free from its products of decomposition. The N. F. VI or the A. O. A. C. method eliminates the neutral decomposition product (acetylurea) by discarding it along with "aromatics." A method is proposed (technique described in detail) which eliminates the possibility of hydrolyzing some phenobarbital during the evaporation of the alcohol. It consists essentially in diluting the solution (elixirs, etc.) with an equal volume of water, saturating with sodium chloride, extracting to completion with chloroform (or ether if emulsions form, as with some syrups), evaporating to small volume, extracting with half-normal sodium hydroxide saturated with sodium chloride (the chloroform will retain nonvolatile aromatic compounds plus any neutral decomposition products or drugs, as acetanilid, acetophenetidin, antipyrine, caffeine, etc.); acidifying the alkaline salt solution with dilute hydrochloric acid, adding an excess of dry sodium bicarbonate, extracting completely with chloroform, evaporating the solvent with the usual precautions to give a dry, granular residue having an m. p. very close to the theoretical for phenobarbital. The residual sodium bicarbonate solution will contain acidic decomposition products or drugs (saccharin, aspirin, salicylic, benzoic or cinchoninic acids), which can be extracted with an appropriate solvent after acidifying the solution. Recoveries of 98.3% to 100.3% were obtained of the amount of phenobarbital added to "elixir" base. The m. p. of the residue extracted from the acidified sodium bicarbonate solution should be taken, and if this does not indicate the substance suspected of being present, appropriate separations and determinations should be made.—FELICE A. ROTONDARO. *J. Assoc. Official Agr. Chem.*, 23 (1940), 777–782. (A. P.-C.)

***p*-Phenylenediamine—Separation and Estimation of, in Mixtures.** The method is as follows: to 50 cc. of an aqueous solution containing 0.02 to 0.05 Gm. of *p*-phenylenediamine, neutral to methyl red, add 25 cc. of reagent (120 Gm. of $SiO_2 \cdot 4H_2O \cdot 12WO_3 \cdot 22H_2O$ dissolved in water to 1 L.), stir until precipitation takes place, let stand over night in a refrigerator, filter into a tared Gooch crucible transferring as much of the precipitate as possible, transfer the remainder of the precipitate to the crucible with the aid of the filtrate, wash with two 5-cc. portions of ice water, dry, ignite at 500° C. to complete destruction of organic matter and weigh; the residue $\times$ 0.0760 = *p*-pheneylenediamine. Recoveries of 99.19% to 99.74% were obtained from pure solutions. Metol, *p*-aminophenol and sodium sulfite do not interfere. Collaborative study of the method gave excellent and closely agreeing results.—JONAS CAROL. *J. Assoc. Official Agr. Chem.*, 23 (1940), 821–823. (A. P.-C.)

Podophyllum—Determination of the Resin in. Boil 2 Gm. of the powdered rhizome with 20 cc. of anhydrous acetone at a reflux condenser and pour then through an extracting tube closed at the lower end with a cotton filter. Wash repeatedly with warm acetone until a few drops of the filtrate give no blue color with phosphotungstic reagent. Measure the filtrate and remove the starch by centrifuging at high speed. Replace with acetone any loss in volume caused by evaporation and evaporate half of the liquid to dryness. Dry completely at 80° and weigh. This procedure is quicker than the official method and requires less substance.—ALFREDO JOSÉ BANDONI. *Rev. farm. (Buenos Aires)*, 83 (1941), 402. (A. E. M.)

Pyrethrum—Analysis of. A collaborative study was made of the determination of pyrethrin I by the mercury reduction method and by Seil's method, and of pyrethrin II by Seil's method and also (following determination of pyrethrin I by the mercury reduction method) by a modification of Seil's method. The results for pyrethrin I were in good agreement by both methods; those for pyrethrin II were in good agreement by the Seil method, but not so good by the modified Seil (following determination of pyrethrin I by mercury reduction).—J. J. T. GRAHAM. *J. Assoc. Official Agr. Chem.*, 24 (1941), 651–653. (A. P.-C.)

Pyridine Bases—Detection and Determination of, in Denatured Spirit. Three methods have been

described for the detection and determination of pyridine bases in denatured spirit depending upon the use of 6% $CdCl_2$ solution, titration with H_2SO_4 with Congo red as an external indicator and colorimetric experiments with the use of cyanogen bromide-aniline reagent.—H. D. SURI, GURCHARAN SINGH AHLUWALIA and H. B. DUNNICLIFF. *J. Indian Chem. Soc.*, 18 (1941), 273. (F. J. S.)

Pyridoxine—Adaptation of the Scudi Colorimetric Method for. The method is rapid and requires no complicated manipulations. It has proved to be particularly valuable for routine assay of natural materials as well as pharmaceutical products whose vitamin B_6 content has been augmented by the addition of crystalline pyridoxine.—O. D. BIRD, J. M. VANDENBELT and A. D. EMMETT. *J. Biol. Chem.*, 142 (1942), 317. (F. J. S.)

Radioactivity—Measurement of. The present status of radioactivity measurements (more particularly those of β and γ rays) are briefly outlined.—ARTHUR WOLF. *J. Assoc. Official Agr. Chem.*, 24 (1941), 578–581. (A. P.-C.)

Silver Nitrate and Thiocyanate Standard Solutions—Standardization of. A preliminary study of the standardization of silver nitrate solutions by Fajans' direct titration using fluorescein as adsorption indicator, by Mohr's direct titration using potassium chromate indicator, and by Volhard's indirect titration using ferric indicator, showed all three methods give satisfactory results if caution is used in observing end-points and the proper end-point corrections are applied where necessary. The Fajans' method works equally well with either potassium chloride with fluorescein indicator, or with potassium bromide with eosin indicator, but the precipitated silver bromide with adsorbed indicator is extremely sensitive to light; potassium chloride (reagent grade recrystallized thrice from water, dried at 110° C. and heated to constant weight at 500° C.) was used as primary standard for all three methods. A collaborative study of the three methods indicated that the normality figure obtained by any of these can be expected to deviate from the true value by not more than 1 part in 1000. Preliminary investigation (not described) indicated that use of purified mercuric sulfate as primary standard for thiocyanate solutions is promising.—E. C. DEAL. *J. Assoc. Official Agr. Chem.*, 24 (1941), 631–635. (A. P.-C.)

Standard Solutions of Iodine and Arsenite—Standardization of. A collaborative study was made of the preparation of arsenite solution from U. S. Bur. of Standard arsenic trioxide and the preparation of iodine solution and its standardization against the arsenite. The technique (described in detail) involved slight modification of the method recommended by the U. S. Bur. of Standards. The results were good, the greatest variation from the figure calculated by weight being 2 parts in 1000 and the average deviation 1 part in 1000. No difficulty is experienced in dissolving the arsenic trioxide (5 Gm. in 50 cc. of decinormal sodium hydroxide) if this is done by heating on the steam bath. Adoption of the methods as official is recommended.—GEO. M. JOHNSON. *J. Assoc. Official Agr. Chem.*, 24 (1941), 639–641. (A. P.-C.)

Succinic Acid—Determination of, in Plant Tissues. Succinic acid is extracted from plant tissue with ether, freed from contamination from other substances by oxidation, converted into its anhydride and condensed in toluene solution with *p*-toluidine to the insoluble crystalline succinyl-*p*-toluide. The properties of this substance are such as to permit of substantially quantitative isolation, and of identification by means of the melting point and crystalline form. An empirical solubility correction and a conversion factor are provided that lead to average recoveries of the order of 99%, and single determinations can be made within 5% over the range from 1 to 20 mg. of succinic acid. The only known interfering substance is α-ketoglutaric acid.—G. W. PUCHER and H. B. VICKERY. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 412–415. (E. G. V.)

Sulfonamides in the Cobalt Color Tests for Barbiturates—Notes on the Behavior of. It has been found that the Koppanyi test for barbiturates is also positive to the sulfonamides. Experimental work indicates, however, that presence of sulfonamides does not interfere appreciably with the assay of barbiturate preparations if the Dille-Koppanyi procedure is strictly adhered to. It is unlikely that toxic amounts of barbiturate would be taken by patients receiving much sulfonamide medication nor likely that enough sulfonamide would be taken alone to produce acute collapse. The diagnosis of an occasional case of acute sulfonamide poisoning might be complicated by earlier barbiturate medication. In saturated chloroform-alcoholic solution, all three of the sulfonamides studied give positive reactions with barium, lithium and isopropylamine tests, but positive barium and lithium tests are not obtainable if the sulfonamides are extracted from 2% aqueous media with chloroform. Maximum concentration of free sulfonamide in urine or tissue fluid would not exceed 2%, so urine from a patient receiving sulfonamides alone would be negative with barium and lithium tests and positive with isopropylamine. To distinguish barbiturate poisoning from sulfonamide intoxication, the barium and lithium tests should be made first on body fluids of suspected cases, and only if these are positive is it necessary to proceed with the isopropylamine tests.—THEODORE KOPPANYI, MELVIN W. GREEN and CHARLES R. LINEGAR. *Jour. A. Ph. A.*, 30 (1941), 246. (Z. M. C.)

Sulfur in Organic Compounds—Procedure for Semimicrodetermination of. The paper describes a Parr sodium peroxide bomb assembly suitable for the decomposition of semimicrosamples of organic compounds, a gravimetric semimicro procedure for determination of sulfur in organic compounds by combustion in the semimicrobomb, and a procedure for removal of silicic acid introduced when liquid samples in glass ampuls are decomposed in the sodium peroxide bomb. Tests indicate that in the semimicro method the digestion period required to render the precipitate of barium sulfate filterable is shortened when precipitation is effected in the presence of picric acid.—R. M. LINCOLN, A. S. CARNEY and E. C. WAGNER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 358–361. (E. G. V.)

Sulfuric Acid—Standardization of. A collaborative study was made of the simultaneous preparation and standardization of sulfuric acid solution by the Pickering-Marshall method, and of standardization by the borax and the sodium carbonate methods. Assuming the value by the Pickering-Marshal method to be correct, the greatest error observed in the results of any collaborator would be not more than 2 parts per 1000 by the borax method, and 3 parts per 1000 by the sodium carbonate method. Adoption of the three methods as official is recommended.—H. W. CONROY. *J. Assoc. Official Agr. Chem.*, 24 (1941), 636–649. (A. P.-C.)

Thallous Salts—Permanganate Titration of. Having prepared a solution to a total volume of approximately 60 cc. containing 6 cc. of hydrochloric acid (specific gravity 1.2) and thallous ion between 0.006 and 0.1 Gm., add 3.0 Gm. of powdered sodium fluoride. (Instead of powdered sodium fluoride, a filtered solution containing 7.0 Gm. of potassium fluoride dihydrate may be used.) Titrate at room

temperature with 0.005 *M* potassium permanganate solution to a faint pink color which should persist for several minutes. A faintly brown coloration may appear as the titration progresses but the permanganage end-point is clearly visible. The normality of the permanganate solution, when used with fluoride ion, is four times the molarity. Thus, if the permanganate has been standardized by sodium oxalate, the normality so obtained should be multiplied by 0.8 to obtain the normality for titration in presence of fluoride ion.—R. S. BEALE, A. W. HUTCHINSON and G. C. CHANDLER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 240–242. (E. G. V.)

Theobromine in Tablet Mixtures—Determination of. Ordinary shakeout methods using two immiscible solvents are unsatisfactory where other nitrogenous organic compounds are combined with theobromine. The A. O. A. C. volumetric method is not satisfactory if the other nitrogenous compounds form silver salts easily. Preliminary tests indicated that phosphotungstic acid would precipitate the theobromine and not phenobarbital, so this led to the method suggested which briefly is gravimetric precipitation of the insoluble compound by phosphatododecatungstic acid on hot strongly acidified solutions of the tablet mixtures containing theobromine or its salts. The following reaction probably occurs: $P_2O_5{\cdot}24WO_3 + 7H_2O + 6C_7H_8O_2N_4 \rightarrow 2H_7P(W_2O_8)_6{\cdot}(C_7H_8O_2N_4)_3$. The method is rapid and reasonably accurate. Common tablet diluents do not interfere. If iodides are present the method is unsatisfactory.—A. G. RICHARDSON and Y. C. CAMPBELL. *Jour. A. Ph. A.*, 31 (1942), 24. (Z. M. C.)

Theobromine or Theobromine Salts and Phenobarbital in Mixtures—Method for the Quantitative Determination of. The literature for methods of determining theobromine and phenobarbital individually is briefly reviewed. Following the usual procedure for determination of two or more organic salts by means of an immiscible solvent a method for determination of theobromine in mixtures with phenobarbital was devised. It consists essentially of removing the phenobarbital, forming silver theobromine and liberating nitric acid quantitatively by the use of a silver nitrate solution. The acid is then titrated with a standard alkali. To determine phenobarbital in mixtures with theobromine or theobromine salts the method consists of separating phenobarbital, using a silver nitrate solution to form insoluble silver phenobarbital which in a dilute sodium hydroxide solution is weakly dissociated so that the remainder of the sodium hydroxide solution causes a separation of silver oxide gradually and completely. Experimental work is reported in some detail. Solubilities of theobromine and of phenobarbital in various solvents are tabulated. Dependability of procedures are shown by tabulation of the results of a series of determinations. Eighteen determinations of phenobarbital averaged 99.75% recovery and a like number of determinations of theobromine averaged 99.90% recovery.—C. W. BELL. *Jour. A. Ph. A.*, 30 (1941), 240. (Z. M. C.)

Titania—Recovery of, from Bauxite Wastes. Bisulfate treatment in dry and semi-dry processes for the recovery of titania from bauxite waste has been worked and the authors recommend the semi-dry method as a better commercial process on account of its simplicity in operation without involving any engineering difficulties in large scale practice.—PARESH CHANDRA DAS-GUPTA and H. N. DAS-GUPTA. *Ind. & News Ed., J. Indian Chem. Soc.*, 4 (1941), No. 1, 16. (F. J. S.)

Titanous Chloride—Use of, in the Determination of Lead in Lead Salts and in Certain Metal-Copper Alloys.—H. N. RAY. *Ind. & News Ed., J. Indian Chem. Soc.*, 4 (1941), No. 2, 111. (F. J. S.)

Water in Benzene—Determination of. A solution of sodium triphenylmethyl in ethyl ether has been investigated as a reagent for the analysis of water in solution in benzene. The analyses have been made on samples containing a relatively small concentration of water. The color change of the reagent is used as the indicator, and relatively high precision is obtainable on low water concentrations. The reagent is suitable for the analysis of other substances.—J. H. SIMONS and E. M. KIPP. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 328–330. (E. G. V.)

Zinc—Determination of, in Plants. A collaborative study was made of the previously described (*Ind. Eng. Chem. Anal. Ed.*, 13 (1941), 145–149) photometric dithizone method. The results agreed well for duplicate determinations on the same ash solution, but the variations were quite appreciable on different ash solutions. It is believed the accuracy will be increased when the analysts have mastered the technique. The method is considered to have several advantages over the present A. O A. C. method, and its adoption as tentative is recommended.—HALE COWLING. *J. Assoc. Official Agr. Chem.*, 24 (1941), 520–526. (A. P.-C.)

PHARMACOGNOSY

A. VEGETABLE DRUGS

Canadian Rhubarb—Chemical Composition and Content of Cathartic Substances in. Canadian rhubarb compares favorably with Chinese rhubarb as regards ash content, free and combined oxymethylanthraquinones, anthrones and anthranols. It contains, however, small quantities of rhaponticin which reduces its value as a cathartic drug.—JOS. RISI. *Naturaliste Canadien*, 67 (1940), 233–252. (A. P.-C.)

Drug Cultivation at Nottingham. The horticultural experimental grounds of Boots Pure Drug Co., Ltd., Nottingham, have been established some little time on a plot which forms part of the estate at Lenton House, Lord Trent's residence. This year five acres have been planted with drugs, and it is hoped that the area devoted to these crops next year will be ploughed for the first time in December, 1940, and planted with hyoscyamus, stramonium, belladonna, digitalis and valerian. All the crops will be used, when dried, to replace supplies formerly bought from abroad.—ANON. *Chemist and Druggist*, 135 (1941), 150. (A. C. DeD.)

Drug Supplies in War-Time. An account of the normal sources of supply of vegetable drugs, which may be suitable for cultivation within the Empire, is given. In introducing drug plants to new areas, the cultural conditions should usually reproduce those under which the plant grows in the wild state. For planting, high-yielding strains should be selected where possible. The time of harvesting is important, and while it varied with the part of the plant producing the drug, consideration of other factors such as weather, light and temperature may be necessary. Generally the drying of the plant material should be carried out immediately after harvesting. With certain drugs, however, preliminary treatment is required and the enhancement or prevention of enzyme action during drying may be important. Details of the methods suitable for drying different plant organs are given. The essential requirements for the production of a number of drugs are discussed. Cascara will grow vigorously as coppice and, in view of the activity of the thin bark of the twigs, their utilization entirely may be advantageous. Digitalis is intolerant of calcareous soils and different strains vary widely in activity. Organ-

ized collection of wild varieties of ephedra growing in the Himalayas should meet the Empire's requirements. October and November appear to be the most suitable time for collection. Ipecacuanha can be successfully grown only under its native conditions; propagation is carried out from fragments of roots or from stems and, after harvesting, it is inadvisable to use the land again at once for this drug. Poor soil, resulting in very slow growth, and a dry sunny climate are necessary in the culture of rhubarb. Valerian requires a moist, rich soil; seeds or side-branches of older rhizomes are used for propagation; ridging is advised to promote rhizome formation. After harvesting in late summer or autumn, drying should be thorough and rapid.—M. ASHBY. *Bull. Imp. Inst., Lond.* (1941), 39, 106; through *Quart. J. Pharm. Pharmacol.*, 14 (1941), 298. (S. W. G.)

Drugs—Cultivation and Drying of. Information on the cultivation and drying of the following drugs: calumba, caraway, chamomile, colchicum, digitalis, licorice, lobelia, psyllium and valerian, is given.—M. ASHBY. *Bull. Imp. Inst.*, 36, II, 106; through *Chemist and Druggist*, 135 (1941), 94. (A. C. DeD.)

Sunflower. The sunflower, *Helianthus annus*, belongs to the family of *Compositæ*. Its chemical composition includes oil, wax, Ca, K, Si and vitamins. The seeds are the most valuable part. They produce oil rich in calories; and their residue pulverized and mixed with wheat flour, makes excellent bread, rich in proteins and carbohydrates. The seeds, whole or residue, are also used for livestock and poultry feed, and the green leaves serve as forage. The plants themselves have a sanitary use in malarial terrain. Wax from the flower is used in soap. There is no scientific medicinal use, but popular therapy includes: concoctions of seeds as a sedative; seeds with wine as an appetizer and with salt as a gargle; infusion of the leaves as a diuretic; a mixture of sunflower oil with cod liver oil for rickets, and with white of egg for local skin affections.—JUAN M. CUADROS. *Rev. farm. Peruana*, 9 (1941), 107–111. (G. S. G.)

Vanilla—Curing of. The curious traditional methods of processing vanilla beans to produce the required aroma were found to cause a marked increase in the rate of evolution of carbon dioxide from the tissues. Freezing the beans produced a contrary effect. Oxidation during the curing process does not necessarily lead to carbon dioxide, however, and may be carried on by enzymes of the peroxidase type. Vanilla beans contain a large amount of peroxidase, and a complex peroxidase system (phenol, peroxide and enzyme) is present even after the curing process is finished. It is suggested that vanillin may be an intermediate product in the development of the desired aroma, and that oxidation products of vanillin may contribute thereto.—A. K. BALLS and F. E. ARANA. *Ind. Eng. Chem.*, 33 (1941), 1073–1075. (E. G. V.)

B. ANIMAL DRUGS

Chinese Blister Beetles. In the News and Notes Section, the zoölogical name, the Chinese character for and the description of the five Chinese blister beetles which are employed in medicine are given. Two are of the genus *Epicauta*, two of the genus *Meloe* and one of the genus *Mylabris*.—ANON. *Chinese Med. J.*, 60 (1941), 92. (W. T. S.)

PHARMACY

GALENICAL

Adrenaline—Stability of, in Solutions of Procaine and Adrenaline. II. Of the substances tried, sodium metabisulfite, cysteine, phenylhydrazine, and protocatechuic aldehyde have been found to act as stabilizers of adrenaline, and of these, only sodium metabisulfite prevents coloration of the solutions when stored or heated. It is cheap and easily obtainable, of low toxicity and has some antiseptic value. These properties make it the most suitable of the tested substances for inclusion in solutions for injection. The amount necessary to stabilize the adrenaline in solutions of procaine and adrenaline varies with the *p*H of the solution. In a further paper it will be shown that in an unadjusted solution of procaine hydrochloride and adrenaline (such as that made to the formula suggested for inclusion in the next British Pharmacopœia) 0.1% of sodium metabisulfite is sufficient to prevent decomposition of the adrenaline on heating or on storing, heated solutions retaining their full adrenaline activity after storage for several months.—G. WOOLFE. *Quart. J. Pharm. Pharmacol.*, 14 (1941), 56–63. (S. W. G.)

Ascorbic Acid—Stable Aqueous Solutions of. Solutions such as those of *l*-ascorbic acid, suitable for injection, are prepared with use of an ethanolamine such as triethanolamine to give the solution a *p*H of between 4 and 6.5, and are filled into ampuls with an inert gas such as nitrogen, hydrogen or carbon dioxide. Sodium carbonate, magnesium hydroxide, etc., also may be used.—CARL L. LAUTENSCHLÄGER and FRITZ LINDNER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,249,903, July 22, 1941. (A. P.-C.)

Drug Extraction. XXIV. The Effect of the Length of Drug Column on the Efficiency of Percolation of Cinchona. Research has been conducted to determine the effect of variations in length of drug column on efficiency of extraction. Cinchona was chosen because it is an important drug and because it presents difficulties in extraction. Apparatus and procedure are described, all experimental data tabulated and discussed. In general, the greater the length of the column the higher the per cent of total alkaloid extracted but length of column does not seem to be the greatest factor in extraction of cinchona. The longest column yielded about 58% of the total alkaloid in a percolate representing 1 cc. for each 1 Gm. of drug; in similar experiments with belladonna root, podophyllum and ipomea more than 99 per cent of the active constituents were obtained in percolate representing 1 cc. per 1 Gm. of drug.—WILLIAM J. HUSA and CLIFFORD T. PACENTA. *Jour. A. Ph. A.*, 30 (1941), 635. (Z. M. C.)

Formaldehyde Solutions—Stabilizing. One % to 10% of melamine or (hydroxymethyl)-melamine is added as a stabilizing agent to an aqueous solution containing at least 35% of formaldehyde, which is otherwise unstable on storage at low temperatures.—ROBERT C. SWAIN and PIERREPONT ADAMS, assignors to AMERICAN CYANAMID CO. U. S. pat. 2,237,092, April 1, 1941. (A. P.-C.)

Hydrastis Canadensis Linne—Percolation of. The technique of sectional percolation was applied to the extraction of this drug and an examination of percolates for specific gravity, total alcohol-soluble extractive and alkaloidal content in each of ten series of percolates reveals that the anomalies reported by Wruble and Powers on cinchona and by Parks on uva ursi are apparently absent.—ELMER L. HAMMOND. *Pharm. Arch.*, 13 (1942), 1–16. (H. M. B.)

Injectionable Solutions—Sterilizing. Solutions are prepared containing the hydrochloride of *p*-butylaminobenzoyldimethylaminoethanol, the hydrochloride of *o*-dihydroxyphenylpropanolamine or the sodium salt of dimethylaminomethylphenylphosphenic acid, and a salt selected from the group consisting of salts of weak acids and strong bases

and salts of strong acids and weak bases, which salt imparts to the solution at sterilization temperature a *p*H value at which it is stable but permits the solution to revert to a *p*H value appropriate to physiological compatibility at atmospheric temperature. Salts may be used, such as ammonium chloride and secondary sodium phosphate.—JOSEPH EISENBRAND and HERMANN PICHER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,251,042, July 29, 1941. (A. P.-C.)

Oleum Arachidis Hydrogenatum Swiss Pharm. V—Absorption Capacity of, for Some Salt Solutions. It is a well-known fact that the various ointment bases do not absorb salt solutions as well as pure water. The author was interested in studying the absorption ability of oleum arachidis hydrogenatum for potassium iodide and other salt solutions of varying concentrations. The stability of the ointments can be explained on the basis of theories of emulsions and depends upon the smallness of the surface tension of the solution. An effort was made to determine whether there was any correlation between the water number (Gm. water held by 100 Gm. ointment base), the viscosity and surface tension of the solution, and whether the absorption capacity could be calculated from this data. The ointments were prepared in a standard fashion and the water content determined by the method of Pritzker. The solution number of an ointment was defined as the number of Gm. of solution absorbed by 100 Gm. of ointment. The viscosity and surface tension were determined for varying concentrations (1% to 60%) of aqueous solutions of potassium and sodium chlorides, bromides and iodides. The solution numbers of these solutions in oleum arachidis hydrogenatum were determined. These results were tabulated. The surface tension of all of the solutions steadily increased with increasing concentration. The viscosities of the solutions of the sodium salts showed a steady increase with increasing concentration whereas the potassium salts showed decreasing viscosity to a minimum value and then increasing viscosity. In contrast to the viscosity and surface tension, the solution numbers showed no regularities with increasing concentration and a simple mathematical relationship cannot be set up. The solution number rose and fell with increasing concentration quite irregularly and the inconsistencies were shown not to be in the experimental procedure. There is also no correlation between the normality of the solutions and the solution numbers. Apparently other factors not yet studied are involved. The data further indicate that the ointment of potassium iodide as prescribed by the Swiss Pharmacopœia can never be stable. Each solution and each ointment base is an individual case.—H. MÜHLEMANN. *Pharm. Acta Helv.*, 15 (1940), 30–40. (M. F. W. D.)

Pepsin—Stabilization of Liquid Preparations Containing. Stability studies of pepsin preparations have been inconclusive due to unsatisfactory methods of assay. Pepsin possesses properties of a typical primary protein and its proteolytic activity is generally associated with the arrangement of the constituent amino acids. A major source of inactivation of pepsin is denaturation of the enzyme protein. Factors affecting activity of a particular enzyme are hydrogen ion concentration, temperature, purity of the enzyme, presence of added electrolytes and other substances as well as length of exposure to these conditions. So a study was made of individual factors and variables affecting peptic activity. They include temperature, *p*H, antioxidants (maleic acid, hydroquinone, resorcinol), preservatives (glycerol, alcohol, hexylresorcinol, "Merthiolate"), protective agents (acacia), low concentrations of amino acids (tyrosine), agitation and storage under nitrogen. Preparations were made with a single variable factor and with several, and all were assayed at regular intervals. Assay results are summarized in a table and discussed, and formulas are suggested. The formulas may be varied slightly but *p*H must be about 4.5 to 5.5 and glycerol 20% or more. Alcoholic content above 15% should be avoided. If suction filtration is not available, filtration by gravity can be used if filter paper is washed thoroughly with distilled water. Preparations should be assayed before putting into permanent containers.—C. J. KLEMME and C. L. BOSWELL. *Jour. A. Ph. A.*, 30 (1941), 249. (Z. M. C.)

Stibophen Solutions—Stabilization of. The following summary is given: (1) The stability of stibophen (sodium antimony *bis*-pyrocatechol-3:5-sodium disulfonate) solutions over a *p*H range from 6.0 to 9.0 has been studied, and attempts have been made to find a better stabilizer than sodium bisulfite, which is used in fouadin. (2) In the absence of a stabilizer, colors develop in stibophen solutions when the *p*H is greater than 7. Ascorbic acid (0.1%) was somewhat more effective than sodium bisulfite (0.1%) in inhibiting color formation in alkaline solution. (3) After storage for several months, some of the trivalent antimony complex is oxidized to the pentavalent state, the change being retarded equally by sodium bisulfite and ascorbic acid. Solutions in rubber-capped bottles deteriorated more rapidly than solutions sealed in ampuls.—F. A. ROBINSON and J. E. PAGE. *Quart. J. Pharm. Pharmacol.*, 14 (1941), 40–44. (S. W. G.)

Tincture of Digitalis—Deterioration of. The two factors studied, temperature of storage and alcoholic concentration, both have important effects on the rate of deterioration of Tincture of Digitalis as measured by the over night frog method of assay. The stability of Tincture of Digitalis would be significantly increased by using U. S. P. Alcohol as the menstruum and storing in a refrigerator at a temperature below 5° C.—R. E. THOMPSON. *Pharm. Arch.*, 12 (1941), 58. (A. C. DeD.)

Vitamin B_1 Content of Tikitiki Extract—Stability of. Samples of tikitiki extract and a syrup preparation of vitamin B_1 previously assayed were assayed again after one year, and one and a half years of storage to determine deterioration. Tikitiki extract deteriorated 50% in one year and the syrup deteriorated 60% in a year and a half. Deterioration is not due to fermentation or changes in *p*H, but further study is necessary to determine the cause of destruction of vitamin in these products. It is recommended that expiration dates be put on vitamin products.—M. GUTIERREZ. *Acta Med. Philippina*, 2 (1940), 189; through *Rev. Filipina Med. Farm.*, 31 (1940), 371. (G. S. G.)

Zinc Oxide—Soft Paste of. It is apparent from the experiments conducted that a slight modification of the formula for this paste will eliminate the separation occurring in the official product. When 2% or less of a stablizing agent (potassium hydroxide or triethanolamine) is used the weight can be deducted equally from the linseed oil and lime water without any apparent detriment to the quality of the paste. The use of 0.5% potassium hydroxide, 2% bentonite, 1% gelatin or 1.5% cholesterol produced very good products; those containing gelatin were most satisfactory in appearance and texture; wool fat and glyceryl monostearate were unsatisfactory. Triethanolamine causes gradual darkening of the paste. The following formula is recommended: Zinc oxide in fine powder 25 Gm.; precipitated calcium carbonate 25 Gm., oleic acid 2.5 Gm., linseed oil 24.5 Gm., solution of calcium hydroxide 22.0 Gm., gelatin 1 Gm.; preservative (propyl *p*-hydroxybenzoate) 0.05 Gm. Mix the zinc oxide and the calcium carbonate thoroughly in

a mortar. To the oil in a beaker, add the acid and preservative and heat to about 60° C. Add the gelatin to the solution of calcium hydroxide and let stand until a thin mucilage is formed. Add this mixture to the oily mixture and stir until saponification is complete; then add this to the mixed powders and triturate until a smooth product is obtained.—PAUL V. MANEY and J. W. JONES. *Bull. Natl. Formulary Committee*, 9 (1941), 346–350. (H. M. B.)

PHARMACOPŒIAS AND FORMULARIES

Aqua Conservans of the New German Formulary. The suggestion is made that Nipagin-Nipasol be used as a preservative in the water used to prepare decoctions and infusions. The oxybenzoic acid esters may be used to preserve other pharmaceuticals from unsightly fermentations, etc.—J. THOMANN. *Schweiz. Apoth.-Ztg.*, 79 (1941), 181. (M. F. W. D.)

British Pharmacopœia—Fourth Addendum to. The General Medical Council will publish shortly a Fourth Addendum to the British Pharmacopœia, 1932, in which certain new monographs, and certain modifications of existing monographs, will be included. Like the Second and Third Addenda, the Addendum has been prepared to deal with conditions arising from the present emergency, and it is expedient that it should be published, and thus made official, without delay. For this reason it has not been found possible to follow the procedure adopted in normal conditions, when a new Pharmacopœia or an Addendum to a Pharmacopœia has been ready for publication, of giving facilities for advance copies to be inspected during the three months immediately preceding publication, by medical practitioners, pharmacists, analysts, manufacturers and others who may be interested. Arrangements have been made, however, for a limited number of advance proofs to be available for supply, on application, to manufacturers of the preparations described therein. A number of new monographs which will be included in the Addendum are listed. The Addendum will also contain certain monographs, or amendments to monographs, of the British Pharmacopœia, 1932, which have become official by virtue of notices of London, Edinburgh, Belfast and Dublin "Gazettes" of February 28, 1941.—ANON. *Chemist and Druggist*, 135 (1941), 130. (A. C. DeD.)

British Pharmacopœia, 1932—A Fourth Addendum to the. This Addendum became official from October 1, 1941. Benzyl benzoate and light liquid paraffin are additions. The former must contain not less than 99% $C_{14}H_{12}O_2$; supplies of course, are at present reserved for medical purposes. The paraffinum liquidum leve is almost odorless when cold and miscible with fixed and volatile oils. An amendment of the B. P. monograph on menthol admits the synthetic racemic variety (optically inactive). The Addendum is published for the General Medical Council by Constable & Co., Ltd., 10, Orange Street, Leicester Square, London, W. C. 2, price 5*s*.—ANON. *Perfumery Essent. Oil Record*, 33 (1941), 332. (A. C. DeD.)

Glycerin Substitutes. In a Fourth Addendum to the British Pharmacopœia, 1932, which is to be issued shortly, amendments made at the request of the Ministry of Supply, relating to glycerin, will be included. These amendments have been made in accordance with the recommendation of the Committee on Substitutes for Glycerin and Sugar.—ANON. *Perfumery Essent. Oil Record*, 32 (1941), 223. (A. C. DeD.)

National Formulary—Additions and Deletions to the. Ninety-six drugs and preparations have been added and 71 of these have been official in U. S. P. XI. Forty-three monographs included in N. F. VI have been dropped; thirty-three of these because they will be added to U. S. P. XII.—ANON. *Bull. Natl. Formulary Committee*, 9 (1941), 344–345. (H. M. B.)

Pharmacopœia—A War. Announcement has been made of a "War Pharmacopœia" compiled by the chairman of the drug committee of the New Sussex Hospital, Brighton, and unanimously adopted for use in the ward and "outpatients" department. The formulas are compiled exclusively from drugs in Class A of the Medical Research Council's War Memorandum No. 3, *i. e.*, drugs readily available or regarded as essential. Any prescriptions not included in the war pharmacopœia are dispensed only when the order is signed by an honorary physician or surgeon. The formulary includes the gargles, drops, draughts, lintuses, liniments, lotions, mixtures, etc.—ANON. *Chemist and Druggist*, 135 (1941), 133. (A. C. DeD.)

Pharmacopoeia—Question of Modernizing the. The author discussed the change which has come about in the nature of the drugs included in modern pharmacopœias, citing with discovery dates the entry of insulin, liver and stomach preparations, vitamins, estrogens, barbiturates, analeptics, diuretics, sulfonamides, pressor amines, antimalarials, ergometrine and newer digitalis glucoside offerings. A codex of purity standards and tests was proposed, with relegation of compound formulas to a dispensatory. The scope that these books should have was discussed.—H. BAGGESGAARD RASMUSSEN. *Arch. Pharm. Chemi*, 48 (1941), 503. (C. S. L.)

U. S. P. XII—War and Patents Change the. The changes involved in the special Interim Announcement No. 4 are discussed.—ANON. *Am. Professional Pharmacist*, 8 (1942), 23–24. (H. M. B.)

NON-OFFICIAL FORMULAS

Baby Preparations. The following formulas are offered: *Powder.*—(1) Talc 50, colloidal clay 35, borax 15; (2) Talc 85, zinc oxide 5, colloidal clay 10. *Oils.*—(1) Mineral oil 80, sesame oil 20; (2) Mineral oil 90, lanolin 3, ethyl stearate 7; (3) Mineral oil 97, cetyl alcohol 3. The mineral oil should be a highly purified grade of 65/75 viscosity. *Creams.*—(1) Mineral oil 50, peanut oil 10, petrolatum 30, paraffin 10; (2) Mineral oil 59, lanolin 5, cetyl alcohol 3, petrolatum 25, paraffin 8.—JOSEPH KALISH. *Drug Cosmetic Ind.*, 49 (1941), 394–395. (H. M. B.)

Bentonite. The uses of bentonite are discussed. Five formulas and twenty-three references.—M. A. LESSER. *Drug Cosmetic Ind.*, 49 (1941), 390–393. (H. M. B.)

Foot Powders. The constituents and formulation of this class of preparation are discussed and five formulas offered.—JOSEPH KALISH. *Drug Cosmetic Ind.*, 49 (1941), 522–523. (H. M. B.)

War-Time Cosmetic Formulas. XII. Shaving Creams. Formulas for the original types of shaving creams and the brushless shaving creams are given.—ANON. *Chemist and Druggist*, 135 (1941), 169. (A. C. DeD.)

DISPENSING

Amaranth—Substitution of, for Cudbear as a Coloring Agent in National Formulary Preparations. A monograph for Compound Solution of Amaranth is given.—ANON. *Bull. Natl. Formulary Committee*, 9 (1941), 312–313. (H. M. B.)

Chemical Incompatibilities. II. The following prescription was received by a pharmacist: sodium citrate 10 Gm., strontium lactate 10 Gm., urotropin 10 Gm., syrup of orange peel 75 cc., infusion uva

ursi *q. s.* 300 cc. This formed a white precipitate of strontium citrate. Neither acidifying nor alkalizing disposed of this precipitate. Since the sodium citrate and strontium lactate are given principally as diuretics they could be given separately. A second similar prescription read: sodium citrate 15 Gm., strontium bromide 16 Gm., urotropin 8 Gm., tr. valerian 20 cc., tr. uva ursi 8 cc., orange water *q. s.* 300 cc. The same white precipitate of strontium citrate formed. Either the sodium citrate or the strontium might be omitted. Citrates of the alkaline metals precipitate soluble salts of strontium producing the insoluble strontium citrate.—JOSE ML. TREJOS F. *Rev. Cien.* Costa Rica, 1 (1940), 79. (G. S. G.)

Compound Elixir of Almond and Spirit of Bitter Almond—Replacement of, with Compound Elixir of Benzaldehyde and Spirit of Benzaldehyde in N. F. VI Preparations. Monographs for the new products are offered.—ANON. *Bull. Natl. Formulary Committee*, 9 (1941), 314–315. (H. M. B.)

Ephedrine Sulfate Solutions—Isotonic, Buffered and Preserved Intranasal. Reference is made to the objections to an oily base and to the faults in some formulas that have been submitted for aqueous solutions for nasal use. An ideal preparation "should contain an effective vasoconstrictor in aqueous solution; the tonicity should approximate that of the blood stream thus producing more effective absorption of the active ingredients as well as partially eliminating its irritant properties; the preparation should be buffered to the *p*H level that is consistent with comfort and effective treatment; and the solution should be preserved to prevent growth of various microörganisms." In the work reported ephedrine sulfate was the vasoconstrictor. The preservative chosen was methyl *p*-hydroxy benzoate and propyl *p*-hydroxy benzoate. To obtain an isotonic solution, buffered at the proper *p*H level an isotonic alkaline buffer solution, buffered with dibasic sodium phosphate and an isotonic acid buffer solution, buffered with monobasic potassium phosphate were used. Tonicity of the solution was adjusted by the method suggested by Nicola. Details of preparation and formula for adjusting tonicity are given. Following is the formula for a 0.5% solution: ephedrine sulfate 0.5 Gm., potassium phosphate monobasic 0.5 Gm., sodium phosphate dibasic 0.5 Gm., potassium chloride 0.15 Gm., sodium chloride 0.15 Gm., dextrose, anhydrous 0.9969 Gm., preserved water, a sufficient quantity to make 100.00 cc. Weights must be made on an analytical balance and a volumetric flask used. The authors recommend inclusion in the next edition of the National Formulary, the formulas for one-half, one and two % solutions.—G. A. TOZER and LOUIS ARRGONI. *Jour. A. Ph. A.*, 30 (1941), 189. (Z. M. C.)

Glycerides of Fatty Acids as Emulsifiers for Ointments—Suitability of Partial. It is often desirable to incorporate aqueous solutions in ointments. The methods of doing this are reviewed and the theories with regard to the dispersion of water in ointment bases are discussed. Some previous work with partial glycerides did not indicate which of these are suitable emulsifying agents. The author has set out to systematically study pure mono- and diglycerides to determine which would produce W/O emulsions and whether there was a certain optimum amount of emulsifying agent. In most of the work, vaseline was the base investigated. The acyl groups to be studied were selected to determine the effects of increasing molecular weight, of spatial configuration and of degree of saturation. Because of the easy rearrangement of mono- and diglycerides under certain conditions, it was necessary to use indirect methods for obtaining the specific glyceride desired. The α-monoglycerides were prepared by treatment of the condensation product of acetone and glycerin with the acid halide and then hydrolysis of the acetone. The diglycerides were prepared by treatment of the monotrityl (triphenylmethyl) ether with acid chlorides and removal of the trityl group. The details of the preparation of undecylic, erucic, behenic, arachidic, brassidic acids, the acid chlorides and the monoglycerides of these and lauric, myristic, palmitic, stearic, pelargic and heptylic, propionic, butyric, isovaleric, caproic and oleic acids are given. Likewise, the preparation of monotritylglycerin and the diglycerides of stearic, palmitic, myristic, oleic, erucic, brassidic are stated. The emulsifying agent and the vaseline were melted together on a water bath and the water incorporated in a standard fashion to insure complete saturation. After a suitable period for the establishment of equilibrium, the water content was determined in Pritzker's apparatus and expressed in terms of Gm. of water taken up by 100 Gm. of ointment base (water number). It was found that among the monoglycerides of saturated acids, the water number increased with increasing molecular weight, and that 4% of 6% of emulsifying agent was optimum. The saturated monoglycerides were not as effective as the unsaturated (monolein) and that the *cis*-configuration (oleic and erucic) was more effective than the *trans* (brassidic). The diglycerides also showed increasing effectiveness with increasing molecular weight but were considerably less efficient than the monoglycerides. The increasing effectiveness is not a function of molecular weight alone but also of the declining solubility in water. Also the chemical properties such as unsaturation in the chain and steric configuration of the molecule play an important role. The emulsifying agents are not of uniform effectiveness in different bases. Forty-eight references.—H. MÜHLEMANN. *Pharm. Acta Helv.*, 15 (1940), 1–30. (M. F. W. D.)

Ointment Bases for Eye Ointments. A brief consideration of the requirements of ointment bases for eye ointments is given. Three ointment bases which have been tested clinically are given. Absorbable ointment bases are not desirable when the medicament is not absorbed, since the solid left behind irritates the eye. Water-soluble substances are more readily absorbed from the eye and should preferably be dispersed in water in oil emulsion bases. Ointments containing free alkaloids in oil solution are less stable than water-in-oil emulsions prepared with the sulfuric or hydrochloric acid salts of the alkaloids in aqueous solution.—H. LEHMANN. *Schweiz. Apoth.-Ztg.*, 79 (1941), 41–43. (M. F. W. D.)

Ophthalmic Ointments—Alkaline. The preparation of alkaline eye salves for use in the treatment of eye irritations produced by acid war gases is discussed. The technique of preparing the salves to avoid crystalline particles is described. The methods of testing the ointments for crystalline particles, for boric acid, for water content and for total alkali are given. A complete monograph for Unguentum Ophthalmicum Alcalinum including directions for testing its properties is outlined. It should be stored protected from light in well-closed containers.—J. THOMANN. *Pharm. Acta Helv.*, 15 (1940), 265–271. (M. F. W. D.)

Pharmaceutical Germicidal Preparation Suitable for Use in Aqueous Solution. Chloramine-T is used with a water-soluble metal iodide such as potassium iodide, both in solid state, and with a water-soluble organic substance oxidizable in aqueous solution by iodine, such as glucose.—PIERO M. SALERNI. U. S. pat. 2,250,504, July 29, 1941. (A. P.-C.)

Potassium Iodide Ointment—Absorption Studies of, on Healthy and Abnormal Skin. The absorption

of ointments on healthy and abnormal skin (antipyrine eczema) are discussed. A new method was developed whereby the amount of ointment absorbed was determined from the weight of the ointment before and after application to the skin without determining the excreted products. A special container with a capacity for 15–20 Gm. of ointment was fastened to the skin in such a fashion that no ointment could be lost. An 8-hr. absorption period was used. All data were accumulated on one patient. A 10% potassium iodide ointment in the most important ointment bases was chosen for study. The moisture and potassium iodide contents were determined before and after application to the skin. These methods are described in detail in the paper. Nine ointments containing potassium iodide were prepared and studied. It was shown that the skin exhibits a very definite selectivity, absorbing various components of the ointments to varying degrees. Studies were made on the absorption of the ointment bases alone and vaseline, unguentum cetylicum anhyd., and anhydrous lanolin are scarcely absorbed whereas hydrogenated oleum arachidis is absorbed. Except for vaseline, these bases when hydrated are better absorbed. Studies also were made on the absorption of an aqueous solution of potassium iodide. Vaseline and unguentum cetylicum were found to be poor bases for the ready absorption of potassium iodide through the skin. The results also indicate that it is somewhat better to use an ointment containing a solution of potassium iodide rather than the finely powdered salt. The large differences in absorption observed in simultaneous individual studies may depend upon a varying disposition of the individual, a partial saturation of the body which disappears only after a long rest period, in the nutrition of the patient and in various atmospheric changes. Quantitatively the absorption from the abnormal skin is greater than from healthy skin. Qualitatively, the absorption is similar, that is, when the healthy skin absorbs relatively large amounts from a particular base, the abnormal skin also absorbs a larger amount.—H. MÜHLEMANN. *Pharm. Acta Helv.*, 15 (1940), 40–54. (M. F. W. D.)

Prescription Ingredient Survey. The survey involves the ingredient analysis of 10,715 prescriptions representing a year's business in 2 pharmacies in a western state, one in an agricultural area and the other in a college town.—ELMER M. PLEIN and L. WAIT RISING. *Bull. Natl. Formulary Committee*, 9 (1941), 353–373. (H. M. B.)

Quinine Solutions Suitable for Parenteral Use—Stable. Quinine monohydrochloride is dissolved in water containing an equimolecular proportion of ascorbic acid. Calcium gluconate and calcium glucoheptonate may also be conjointly used.—ARNOLD SALOMON, assignor to N. V. ORGACHEMIA. U. S. pat. 2,251,526, Aug. 5, 1941. (A. P.-C.)

Suppositories without Cacao Butter. Because of the scarcity of cacao butter, several mixtures of tallow and lard were investigated as possible substitutes for cacao butter. A mixture containing about 50% lard and 50% tallow produces a base from which acceptable suppositories may be made by the hot method. These mixtures cling to the mold if the suppositories are made by the compression method.—ANON. *Pharm. Tidschr.*, No. 9/10, (1941); through *Schweiz. Apoth.-Ztg.*, 79 (1941), 152. (M. F. W. D.)

Syrup of Rose Hips. Syrup of rose hips is prepared from ripe red rose hips and contains not less than 200 or more than 250 mg. of ascorbic acid per 100 cc., and contains added sugar sufficient to produce a specific gravity in the finished syrup of 1.325. It may vary in color depending on the kind of hips used and the method of manufacture, but should be filtered and free from hairs. The limit of sulfur dioxide is 350 parts per million. Citric acid may be added, if necessary, to bring the acidity to a minimum of 1.1%. This is a provisional specification adopted for the experimental manufacture which has been undertaken this year by a small number of firms. The syrup which it is hoped will be available on the market shortly and which will be sold by chemists will be labeled: "Rose Hip Syrup. This syrup contains approximately 200 mg. ascorbic acid (vitamin C) in each 100 cc. This product conforms to the standard approved by the Ministry of Health. This syrup contains five times the ascorbic acid content of fresh orange juice. Dosage for a child: 1–2 teaspoonfuls a day."—*Chemist and Druggist*, 136 (1941), 215. (A. C. DeD.)

Yeast Syrup High in B Vitamins—Process of Producing. A mixture of 300 lbs. of water, 10 lbs. of fresh compressed yeast, 100 lbs. of dried brewers' yeast, 100 lbs. of sugar and 10 Gm. of papain is heated to 40° C. for 10 to 15 min. and then to 60° C. for 15 min. Sufficient water is added to render suitable for filtering or centrifuging; after heating to 100° C. the water-soluble liquid is filtered or centrifuged and then condensed *in vacuo* to a consistency of approximately 80% solids. The resultant syrup is high in vitamins B_1 and B_2.—NATHAN M. CREGOR, FREDERICK E. TIMMER and ROBERT M. ALLEN, assignors to VEGEX INC. U. S. pat. 2,235,827, March 25, 1941. (A. P.-C.)

PHARMACEUTICAL HISTORY

Banting—Sir Frederick. An editorial.—ANON. *Chemistry and Industry*, 60 (1941), 151–152. (E. G. V.)

Bismuth—Origin of the Word. The word Wissmut (Ger.) or Vismut (Swed.) has been claimed to have arisen from the Arabic uthmud, a sort of antimony. However an old Germanic form of the word, Wissmât, has been said to signify a white or light mass or metal. Some say the second syllable, -mat, is from German Matte, a mat (or grassmat or meadow) because of the play of colors shown by the metal like a meadow spotted with flowers. The author considered it more likely that the word arose from the fact that the chief site of ancient bismuth mining in Europe was "in the meadow" (In den Wiesen) at Schneeberg in the Erzgebirge mountains. Although Agricola did not use the word (he called the metal plumbum cinereum), it appeared in the translation of Agricola's *De Re Metallica* into German by Philip Bechius (*Bergwerck Buch*, Basle, 1557 and 1621) as Wissmut. The word Wisemât, a grassy meadow, is traceable back to 1368. The first syllable came from German Wiese, a meadow, the second from maien or maejen, to mow. After 1450 the word was spelled Wiesemât, and one finds use of Wiesemât-grabe, as term for a bismuth mine. It was Latinized as: Bisemutaria, and Bechius turned it back into Wissmut. So the name probably arose from the fact that the ancient mine at Schneeberg was in a meadow. The "ore from the mine in the meadow" was changed by Paracelsus to Wissmât and by Bechius to Wissmut.—L. GENTZ. *Farm. Revy*, 40 (1941), 501. (C. S. L.)

Coffea Arabica—Brief Description of. The history of the use of coffee is lost in antiquity. It probably originated in Abyssinia. It was used in Egypt in 1576, passed to Persia and was brought to Amsterdam in 1690. It was brought to America first by the Dutch to their West Indies in 1718, thence to Surinam, to French Guiana and to Brazil in 1723, where it has become a major product. The first "coffee house" serving the infusion was established in Constantinople in 1551; Venice and London adopted the idea within the century. Rambosson produced the epigram "Wine stimu-

lates the heart and love, coffee inspires the spirit, the intellect." The value of coffee is due to its active principle caffeine.—MEIRA PENNA. *Gaz. Pharm.*, 9 (May, 1940), 18. (G. S. G.)

Elements—History and Etymology of the Names of the. The author gives a short account of the discovery and origin of the names of the elements.—M. CRABBE. *Schweiz. Apoth.-Ztg.*, 79 (1941), 38, 59, 81. (M. F. W. D.)

Erik Ohlsson. On the occasion of his 50th birthday the Swedish pharmaceutical journal gave a brief biography of Erik Ohlsson, Ph.D., M.D. (Lund), who is professor of chemistry and chemical pharmacy (1932–) at the Royal Pharmaceutical Institute, Stockholm, and who has been director of that institute since 1933.—ANON. *Farm. Revy*, 40 (1941), 517. (C. S. L.)

Old Compounds—Recent Utilization of. Numerous chemical compounds synthesized from 30 to 140 years ago have only recently found therapeutic use. A list of twenty-eight of these is described both as to chemical structure and therapeutic use. A table lists these compounds with their originator and date of origin together with the investigator who made therapeutic application and that date. The earliest mentioned is allantoin by Buniva-Vauquelin in 1800 and given therapeutic application by Robinson in 1935; and mandelic acid by Winckler in 1830, used by Helmholtz and Osterburg in 1936. Among the more recent are guanidine by Hoffman in 1866 developed by Minot and collaborators in 1939; and the latest, *para*-aminobenzoic acid by Fischer in 1863, applied by Ansbacher 1941.—QUINTINO MINGOJA. *Arq. Biol. (São Paulo)*, 25 (1941), 217. (G. S. G.)

Pharmacy—Upward Road of. The progress of pharmacy in the past one hundred years is briefly reviewed.—G. A. Burbidge. *Merck Rept.*, 50, No. 4 (1941), 12. (S. W. G.)

Sertürner, Frederich Wilhelm, Adam—Biographical Sketch of. A brief biographical sketch of Sertürner, the discoverer of morphine.—J. A. HÄFLIGER. *Schweiz. Apoth.-Ztg.*, 79 (1941), 137–142. (M. F. W. D.)

Wormwoods—Fragrant. An interesting survey of the bitter herbs of the scripture.—ANON. *Perfumery Essent. Oil Record*, 32 (1941), 238. (A. C. DeD.)

PHARMACEUTICAL EDUCATION

British Hospital Pharmacy. A review of a booklet dealing with hospital pharmaceutical practice issued jointly by the Council of the Pharmaceutical Society of Great Britain and the Guild of Public Pharmacists.—ANON. *Am. Professional Pharmacists*, 8 (1942), 36–38. (H. M. B.)

Equipment Progress—1940. Development of equipment and instruments in 1940 is described.—D. H. KILLEFFER. *Ind. Eng. Chem.*, 33 (1941), 9–15. (E. G. V.)

1940—Over the Shoulder. A review of the developments, new chemicals, new plants and scientific awards for 1940.—ANON. *Ind. Eng. Chem.*, 33 (1941), 3–8. (E. G. V.)

Organometallic Problems—Research in. Equipment used for research in organometallic problems is pictured.—W. L. GILLILAND. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 474–475. (E. G. V.)

PHARMACEUTICAL ECONOMICS

Bleaching Earths—Some Northern India. It has been found that natural bleaching earths of good quality are available in northern India. These earths on activation by acid treatment show as good bleaching power as that of any imported material. If supplies of acid at cheap cost are available, activated clays can be produced profitably in the country on a commercial scale.—J. L. SARIN and I. S. KUKERAJA. *Ind. & News Ed. J. Indian Chem. Soc.*, 4, No. 3 (1941), 184. (F. J. S.)

British Pharmacy under War Conditions. A review of the steps that have been taken by British pharmacists during the first two years of the present war as an indication of what might take place in this country is offered.—ANON. *Am. Professional Pharmacist*, 8 (1942), 31–33, 42, 44, 50, 59. (H. M. B.)

Canadian Chemicals Production. Canadian production of chemicals and allied products during 1940 is estimated to have totaled $184,152,900, or 15% more than the previous year.—ANON. *Chemist and Druggist*, 135 (1941), 172. (A. C. DeD.)

Chinese Menthol Production. More than a dozen factories in China are understood to have been producing menthol crystals for export during 1940. The average price ranged between 30 and 40 yuan per lb., though much higher prices prevailed at some periods.—ANON. *Chemist and Druggist*, 135 (1941), 172. (A. C. DeD.)

Cocoa and Chocolate—Romance of. A lecture covering source, treatment and food value.—G. R. MAYBEE. *Chemistry and Industry*, 59 (1940), 460–461. (E. G. V.)

Drug Manufacture in India. To replace the boric acid recently imported to India from England, it is proposed to purchase borax in Tibet and manufacture the acid. Arsenic tri-iodide, magnesium sulfate and peptone powder are three other items now being made in India.—ANON. *Indian Med. Gaz.*, 76 (1941), 301. (W. T. S.)

Drug Purchase—Economy of, by the Hospital Pharmacy. The economics of drug purchase for hospital pharmacies in Sweden was considered. Specialties whose therapeutic properties can practically be duplicated by prescription of standard drugs should not be used in a hospital. Price differences among commercial offerings of digitalis preparations, antacids, analgesics, soporifics, laxatives, vitamin preparations, etc., were considered. Frequently the more costly exerted no better therapeutic effect than the cheaper preparations. A fluid preparation form was often cheaper than pills or tablets. For injection medicines, membrane-capped bottles which could be used over repeatedly were far cheaper than ampuls. Items prepared in the hospital pharmacy were often less expensive than commercial offerings.—G. LINDGREN. *Farm. Revy*, 40 (1941), 405. (C. S. L.)

Economy in the Use of Drugs in War Time in India. The Bombay Government has arranged for the production of P, tartaric acid, $KClO_4$, I, CS_2 and $SrCO_3$ under the guidance of their research chemist. Economy in the use of drugs in war time has provoked the tabulation of practically all essential drugs under these classifications: (1) essential and readily available drugs; (2) essential for only certain purposes; (3) non-essentials; (4) identical substitutes of British manufacture.—ANON. *Indian Med. Gaz.*, 76 (1941), 551–554. (W. T. S.)

Insulin Requirements in India. In reply to letters from physicians concerning the availability of insulin in India at the present, the editor states that the British and American products are preferred, but in view of the emergency, a firm in Bombay is exploring the possibilities of manufacturing insulin.—ANON. *Indian Med. Gaz.*, 76 (1941), 573. (W. T. S.)

International Medical Science and the War. A review of the new responsibilities of medicine and pharmacy.—CHARLES HILL. *Am. Professional Pharmacist*, 8 (1942), 28–30. (H. M. B.)

Perfumery Chemist's Notebook—From a. Cosmetics of the future and other topics are discussed.—S. P. JANNAWAY. *Perfumery Essent. Oil Record*, 32 (1941), 349. (A. C. DeD.)

Stains for Medical Work Being Manufactured in India. Congo red, chrysoidin, methyl violet, brilliant green, eosin and Leishman's stain, all formerly imported from Germany, are now being produced in India by a coöperative endeavor.—ANON. *Indian Med. Gaz.*, 76 (1941), 486. (W. T. S.)

Toilet Preparations Production, 1941. A critical and supplementary survey of the articles which have appeared in *Perfumery and Essential Oil Record* during 1941.—FRANK ATKINS. *Perfumery Essent. Oil Record*, 32 (1941), 354. (A. C. DeD.)

MISCELLANEOUS

Bi-Oro Suntan Cream. Bi-Oro is a new preparation made by the Gesellschaft für Chemische Industrie in Basel, Switzerland. In thin layers it completely protects against the harmful ultraviolet rays and promotes the natural tanning of the skin. It remains in the top layer of the skin for several hours. It also protects the skin against climatic changes, helping it to maintain its normal function.—ANON. *Schweiz. Apoth.-Ztg.*, 79 (1941), 210. (M. F. W. D.)

Buto Depilatory Cream. Buto Depilatory is a preparation made by the Gesellschaft für Chemische Industrie in Basel, Switzerland. It is a salt of an organic acid and has a kerolytic activity which is slower than that of the alkali sulfides but produces no undesirable irritation of the skin.—ANON. *Schweiz. Apoth.-Ztg.*, 79 (1941), 208. (M. F. W. D.)

Cleansing and Disinfecting—Compositions Suitable for. A process involving the solution of a dry solid mixture of calcium hypochlorite and an alkali metal metaphosphate, present in an amount sufficient to render the mixture soluble in an aqueous solution of the class consisting of soap solutions, solutions of alkaline detergents and hard waters, in an aqueous medium, comprises intimately admixing with the solid mixture prior to its solution a proportion of sodium chloride about 15% to 35% by weight of the mixture, and adding the resulting mixture to the aqueous medium, whereby the tendency to form sticky, difficultly soluble agglomerates is minimized.—JAMES D. MACMAHON, assignor to THE MATHIESON ALKALI WORKS. U. S. pat. 2,242,315, May 20, 1941. (A. P.-C.)

Cold Creams. A discussion.—JOSEPH KALISH. *Drug Cosmetic Ind.*, 49 (1941), 634–635. (H. M. B.)

Copper Fungicide. Use is made of a double salt of tribasic copper phosphate and disodium phosphate in the form of crystals about 1 to 2 microns in size.—CHAS. F. BOOTH and HERBERT J. KRASE, assignors to MONSANTO CHEMICAL CO. U. S. pat. 2,237,045, April 1, 1941. (A. P.-C.)

Cosmetic Substitutes. A discussion of substitutes for raw materials which are apt to be short due to war conditions.—JOSEPH KALISH. *Drug Cosmetic Ind.*, 50 (1942), 148–149. (H. M. B.)

Dental Cream. A salt of the sulfuric acid ester of a fatty acid monoglyceride is used with a polishing agent such as calcium carbonate and a flavoring material, etc.—FRED W. MUNCIE, assignor to COLGATE-PALMOLIVE-PEET CO. U. S. pat. 2,236,-828, April 1, 1941. (A. P.-C.)

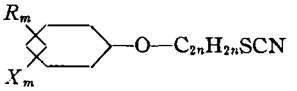
2,2′-Dihydroxy-3,5,6,3′,5′,6′-Hexachlorodiphenylmethane. This compound, which melts at 161° to 162° C. is suitable for use as an antiseptic, bactericidal, fungicidal or preserving agent, as in tooth powders and tooth pastes, ointments, "creams," cosmetics or rubber goods. It is made by treating a solution containing two molecules of 2,4,5-trichlorophenol, 1 molecule of formaldehyde, and methanol at a temperature of 0° to 5° C. in the presence of sulfuric acid, and may be purified by crystallization from benzene, toluene or ethylene dichloride.—WM. S. GUMP, assignor to BURTON T. BUSH, INC. U. S. pat. 2,250,480, July 29, 1941. (A. P.-C.)

Face Powder. For improving the covering power of talc powder when used as a basis for face powder, it is treated with a fatty acid metal salt such as zinc stearate or other zinc, strontium or aluminum salt of a fatty acid containing at least 10 carbon atoms while the salt is in the liquid phase (suitably dissolved in xylene).—MATTHIAS QUAEDVLIEG, assignor to WINTHROP CHEMICAL CO. U. S. pat. 2,218,586, Oct. 22, 1940. (A. P.-C.)

Hair—Removing Artificial Colors from. A composition for removing color is used which comprises a sulfonated oleaginous material containing free sulfonic acid groups but no free sulfuric acid, such as sulfonated castor oil with ethylene glycol and salicylic acid or the like.—ABRAHAM R. GOLDFARB, assignor to LAWRENCE RICHARD BRUCE INC. U. S. pat. 2,236,970, April 1, 1941. (A. P.-C.)

Halophenolcarboxylic Acids and Their Salts—Substituted. Compounds are produced having the general formula $C_6H(3-n)Yn(OH)(CO_2Z)X$, in which $C_6H(3-n)$ may be a benzene nucleus, X a halogen and Y an alkyl, aralkyl, aryl, alkoxy or cycloalkyl group of which there may be 1, 2 or 3 for each molecule, the number of these substituent groups present per molecule being given by n. These alkyl groups need not be the same, but there may be 2 or more different substituent groups in the same molecule. Z may be a hydrogen or a nitrogen-containing basic radical or a metal not lower than zinc in the electromotive series and n an integer not greater than 3. The alkyl, aralkyl, aryl, alkoxy and cycloalkyl halophenolcarboxylic acids and especially their salts are especially useful as antiseptics and disinfectants. The preferred compounds in this class are those in which the substituent alkyl, aralkyl, aryl, alkoxy or cycloalkyl group or groups have a total of not less than 3 nor more than 12 carbon atoms per molecule. Details are given of the production of several such compounds suitable for use in lotions, antiseptic solutions, ointments, hair tonics, etc.—GEO. L. DOELLING. U. S. pat. 2,244,-769, June 10, 1941. (A. P.-C.)

Insecticidal Sprays. *2,239,079*—Sprays suitable for combating household insects comprise a noncorrosive organic solvent such as a petroleum distillate in which there is dissolved an extract of pyrethrin- or rotenone-bearing plant, together with a halophenoxyalkyl thioeyanate such as β-(4-chlorophenoxy)ethyl thiocyanate which serves both as an active toxicant and as a stabilizer. *2,239,080*—This relates to similar compositions containing, as the auxiliary toxicant and stabilizer, a compound of the general formula



where R represents a hydrocarbon radical; X represents alkyl, cycloalkyl, aralkyl, aryl, aryloxy, lower alkoxy, alkenyl, halogen or hydrogen; n represents an integer from 2 to 6, inclusive; each m represents an integer not greater than 2; and the nuclear-substituted phenoxy group contains at least 8 carbon atoms.—GERALD H. COLEMAN, CLARENCE L. MOYLE and JOHN E. LITVAK, assignors to THE DOW CHEMICAL CO. U. S. pats. 2,239,079 and 2,239,080, April 22, 1941. (A. P.-C.)

Insecticides, Fungicides and Caustic Poisons—Analysis of. Recommendations are made regarding modifications in the A. O. A. C. methods.—J. J. T. GRAHAM. *J. Assoc. Official Agr. Chem.*, 23 (1940), 546–547. (A. P.-C.)

Iodoform—Electrolytic Production of. Details of the process are given.—*Chemist and Druggist*, 135 (1941), 102. (A. C. DeD.)

Lozenges—Medicinal. Lozenges are formed including petrolatum dispersed in an aqueous phase containing substances including sugar in quantity substantially less than that of the petrolatum to sweeten slightly the same and constituting a viscous mass, the mass being molded to shape and constituting a core or body, and a relatively hard, thin, uniform coating enclosing such viscous core and serving to hold the core to produced shape, the coating having a composition rendering the same soluble to a degree commensurate with the dispersiveness of the core. *E. g.*, the coating may contain sugar, gum arabic and corn starch.—ELIZABETH MYERS, JULIUS ALSBERG and ADOLPH GOTTFURCHT, ALSBERG and GOTTFURCHT, assignors to ELIZABETH MYERS. U. S. pat. 2,253,800, Aug. 26, 1941. (A. P.-C.)

Nails. The physiology, functions and disorders of nails are discussed. Fifteen references.—M. A. LESSER. *Drug Cosmetic Ind.*, 49 (1941), 513–515, 519. (H. M. B.)

Nasal Drops. A major proportion of a mineral oil and a minor proportion of a glyceride oil such as cottonseed oil is used with a small proportion of 1-phenyl-2-amino-1-propanol and a blending agent, such as thymol or eucalyptol, which is readily soluble in the glyceride oil and is a solvent of the 1-phenyl-2-amino-1-propanol. Numerous examples are given.—SERECK H. FOX, assignor to SHARP & DOHME, INC. U. S. pat. 2,222,976, Nov. 26, 1940. (A. P.-C.)

Parasiticide. As an active constituent, use is made of a composite oily to resinous distillate product resulting from condensing an aromatic hydrocarbon or nuclear-halogen substitution product thereof with an alkylene halide, such as the product formed from benzene and ethylene chloride.—WM. P. TER HORST, assignor to U. S. RUBBER CO. U. S. pat. 2,243,543, May 27, 1941. (A. P.-C.)

Perspiration-Inhibiting Composition. Use is made of aluminum sulfate or other nontoxic water-soluble protein-coagulating metallic salt of a strong acid and a neutral, water-soluble amino compound from the group consisting of the aliphatic amides and the aliphatic amino acids and having at least one intact, reactive amino group, such as urea.—JOHN H. WALLACE, JR., and WILFRED C. HAND, HAND assignor to WALLACE. U. S. pat. 2,236,387, March 25, 1941. (A. P.-C.)

Pharmaceutical Products—Intermediates for. Pentamethylene oxide compounds and pentamethylene sulfide compounds are prepared by treating a compound of the general formula $HlgCH_2CH_2XCH_2CH_2Hlg$ (where X stands for oxygen or sulfur and Hlg stands for halogen) with an arylacetonitrile in the presence of an agent capable of splitting off hydrogen halide. In this manner there are obtained compounds of the general formula $ArC(CN)\cdot CH_2CH_2YCH_2CH_2$ (where Y stands for oxygen or sulfur). The compounds containing sulfur may be oxidized with the aid of oxidizing agents into the corresponding amides, acids or esters. The reaction is suitably carried out in a solvent such as benzene, toluene or xylene, at about 25° to 70° C. As agent capable of splitting off hydrogen halide there may, for instance, be used sodium or active sodium compounds such as sodium amides or phenolate. The new compounds are for use as intermediate products for the manufacture of new remedies.—OTTO EISLEB, assignor to WINTHROP CHEMICAL CO. U. S. pat. 2,242,575, May 20, 1941. (A. P.-C.)

Preservatives. Alkyl phenols, which are relatively nontoxic and may be used for the preservation of cosmetics, food products, etc., are produced by a process involving condensing a monohydric alcohol and a phenol in an acid solution of a condensing agent, such as a solution of zinc chloride and hydrochloric acid. Numerous examples are given.—ROLAND R. READ, assignor to SHARP & DOHME. U. S. pat. 2,242,325, May 20, 1941. (A. P.-C.)

Salve-Like Composition for Coating the Skin. A miscible animal oil having an iodine number not greater than 125, such as a fish oil, is used with a cellulose organic acid ester, such as cellulose acetate-stearate, having an acyl content of at least 50% of the weight of the ester of which a major portion is fatty acid groups of at least four carbon atoms.—GORDON D. HIATT, assignor to EASTMAN KODAK CO. U. S. pat. 2,249,523, July 15, 1941. (A. P.-C.)

Shampoo. As the active ingredient, use is made of a water-soluble salt such as the ammonium or monoethanolamine salt of the sulfonic esters of monoethanolamide of coconut fat acids in an aqueous vehicle, having high foaming power due to the absence of formation of calcium or other insoluble salts, the property of remaining liquid in concentrated solution, high detergent power, and being noninjurious to the scalp or hair due to low imbibition and the absence of the formation of free alkali.—JOHN W. ORELUP. U. S. pat. 2,237,629, April 8, 1941. (A. P.-C.)

Sieves for Coarse Aggregates. Comparative tests have been made on woven wire and perforated plate sieves ranging in size from $^1/_8$ in. to $^3/_8$ in. Woven wire sieves were found to be considerably less accurate in aperture size and shape than perforated plate throughout the range of size examined. A woven aperture had an effective size larger by about 4% than a perforated plate of equal projected size. The effective sizes of $^3/_8$-in. woven and perforated sieves, obtained from sieving tests on hand gaged samples, were analyzed and the differences between the effective and the average sizes of aperture were accounted for by correcting for the effect of woven aperture and by making allowance for the variability in aperture size. The differences are much less for perforated than for woven sieves. They may cause serious errors, particularly in the case of $^1/_2$-in. material where the use of a combination of sieves of both types is now specified. It is suggested that woven sieves could therefore be replaced with advantage by perforated sieves down to and including the $^3/_{16}$-in. size, where the sieves designated in fractions of an inch join the fine-mesh series and a discontinuity therefore already exists.—A. H. D. MARKWICK. *J. Soc. Chem. Ind.*, 59 (1940), 88–92. (E. G. V.)

Sifting Machines. Two types of motor driven sifting machines available in Sweden for the preparation room of the apothecary shop were described and depicted.—S. KJELLMARK. *Farm. Revy*, 40 (1941), 569. (C. S. L.)