

PHARMACEUTICAL ABSTRACTS

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BOTANY (Continued)

Pyruvic Acid—Method of Formation of, by Barley. The following summary is given: (1) Cell-free barley saps were prepared by a simple method. On incubation for 48 hrs. at 30° in the presence of thymol and 1-naphthol-2-sulfonic acid, they gave rise to little or no pyruvic acid. (2) Addition of sucrose did not cause pyruvic acid formation. Glucose and fructose would be present in the sap itself, and therefore were also incapable of giving pyruvic acid. (3) Addition of adenylic acid to starved sap (*i. e.*, sap low in sugar content) gave traces of what was probably pyruvic acid. (4) Addition of sucrose + adenylic acid, or glucose + adenylic acid, led to the formation of readily identifiable quantities of pyruvic acid. (5) Hexosediphosphate and phosphoglycerate were also converted into pyruvic acid. Pyruvate formation from hexosediphosphate was markedly inhibited by *M/40* NaF. (6) Lactic acid was oxidized to pyruvic acid in the presence of ascorbic acid and atmospheric oxygen. These results (1 to 5) indicate a conversion of sugars into pyruvic acid through a phosphorylating cycle similar, at least in outline, to the reactions of yeast.—WILLIAM OWEN JAMES, GLADYS MACPHAIL JAMES and ARTHUR HUGH BUNTING. *Biochem. J.*, 35 (1941), 588. (F. J. S.)

Vanilla Vines from Seed. Well-developed plants grown from vanilla bean seed by Dr. Lewis Knudson, head of the botany department of Cornell University, Ithaca, N. Y., are being sent to Puerto Rico where the Department of Agriculture is introducing commercial cultivation of the vanilla bean. Puerto Rico plantings of vines imported from Mexico and other vanilla bean producing countries are attacked by a fungus and the Cornell experiment of growing vines from seed was undertaken to get plants from which to produce hybrid varieties capable of growing better in a new environment. Dr. Knudson germinates the seed in test tubes plugged with cotton which lets in air but excludes the germs and fungi that otherwise would ruin the seed which are minute in size.—ANON. *Perfumery Essent. Oil Record*, 32 (1941), 347. (A. C. CeD.)

Vitamin B₁—Further Tests with, on Established Plants and on Cuttings. China aster seedlings grown in flats (one flat per treatment) during February and March produced taller plants with a greater fresh weight when the soil was watered weekly with a solution of vitamin B₁ (0.01, 0.1 and 1.0 mg./l.) than when watered only with tap water (control flat). In contrast, there was no influence of vitamin B₁ on stem elongation of potted asters grown individually in pots (20 pots per treatment) during June and July. Stem elongation of potted asters varied significantly for plants located in different rows on the greenhouse bench but not according to treatment with vitamin B₁ in concentrations of 0.1, 0.5, 1.0 and 10 mg./l. These results appear to be typical of the conflicting reports in the literature relating to treatment of the soil with vitamin B₁. However, the more substantial experimental plan for potted asters makes the results more convincing than those obtained with flat-grown asters. Application of vitamin B₁ every three to five days to the rooting medium (sand) or as a deferred treatment to the basal ends of cuttings did not accelerate the development of roots as reported by Warner and Went. Differences in rooting response were characteristic of a root-inducing substance (indolebutyric acid) and not of a root growth factor. In a few cases vitamin B₁ appeared to function as an activator, thus increasing the activity of indolebutyric acid as previously reported for several substances in addition to vitamin B₁. The results of these tests indicate that vitamin B₁ should not be recommended for practical use in treating cut-

tings or as a soil amendment until there is more substantial evidence than at present to show that plants or cuttings can benefit from an external supply of vitamin B₁.—A. E. HITCHCOCK and F. W. ZIMMERMAN. *Contrib. Boyce Thompson Inst.*, 12 (1941), 143-156. (B. M. B.)

Wound Hormones of Plants. V. The Synthesis of Some Analogs of Traumatic Acid. A method for the synthesis of unsaturated acids of the type HOOC(CH₂)_nCH=CHCOOH and HOOC(CH₂)_nCH=CH—CH₂COOH has been developed and several representatives of these types were prepared. Δ⁸-Undecene-1,11-dicarboxylic acid and Δ^{1,7}-octadiene-1,8-dicarboxylic acid have also been prepared. These acids are found to be active plant wound hormones.—J. ENGLISH, JR. *J. Am. Chem. Soc.*, 63 (1941), 941. (E. B. S.)

Yeast—A Preparation from, That Is Active in Breaking the Rest Period of Buds. The results of Bennett, Oserkowsky and Jacobson, which showed that the action of yeast extracts in breaking the rest period is due to some substance or substances other than glutathione, were confirmed. A procedure is given for obtaining a preparation from yeast that is much more active than glutathione. This preparation contained adenine, pentose and phosphorus. It also hastened the reduction of methylene blue by potato juice plus glutathione. Experiments indicated that adenine had some dormancy-breaking action. It is suggested that the effect of adenine and adenine-containing nucleotides on resting buds be investigated further.—JOHN D. GUTHRIE. *Contrib. Boyce Thompson Inst.*, 12 (1941), 195-201. (B. M. B.)

CHEMISTRY

GENERAL AND PHYSICAL

Adhesive Joints—Strength and Thinness of. The strength of paraffin wax joints between metals has been measured for two thicknesses of the wax layer. Thin joints are stronger than thick ones. Two-thirds of the difference observed can be accounted for by the higher probability of a weak spot in a thicker specimen. Hence the rules "the thinner the film the stronger the joint" and "the shorter the fiber the higher the strength" are closely related. A tentative explanation for the remaining third of the difference between thick and thin joints is advanced; it does not involve "molecular forces."—J. J. BIKERMAN. *J. Soc. Chem. Ind.*, 60 (1941), 23-24. (E. G. V.)

Allergens—Chemistry of. IV. An Electrophoretic Fractionation of the Protein-Polysaccharide Fraction, CS-1A, from Cottonseed. In an electrophoretic fractionation of the cottonseed allergenic fraction CS-1A, a protein migrated toward the cathode and a polysaccharidic-protein migrated to the anode. The evidence presented indicates that CS-1A is a mixture containing a specifically active protein and active compounds of this protein chemically combined with varying amounts of polysaccharidic carbohydrate. The cutaneous activity of the cathodic and anodic fraction was not decreased by refluxing with 0.1 *N* acid for four hours. The reagin neutralizing capacity of the fractions was decreased but not destroyed by the acid treatment. A simple apparatus is described for large-scale, high voltage electrophoretic fractionation of water-soluble ampholytes which is suitable for preparative work.—J. R. SPIES, H. S. BERNTON and H. STEVENS. *J. Am. Chem. Soc.*, 63 (1941), 2163. (E. B. S.)

Blood Pressure Principles of Hog Kidney Extracts—Electrophoretic Separation of the. It is known that "renin," a vaso-pressor substance, and a vaso-depressor substance present in kidneys have somewhat similar chemical properties so that separa-

tion is difficult. There is uncertainty of identification of various kidney depressor principles so several different types of kidney extracts were submitted to similar electrophoretic analysis. The experimental work is reported in detail and the apparatus used is illustrated. In conclusion, the authors state that the "vaso-pressor substance" migrates to the cathode at pH values ranging between 3.0 and 6.5 under the conditions of the experiment. "Renin D" and "renin E" have similar behavior under similar conditions. "The vaso-depressor factor" consistently migrates to the anode at pH values ranging between 5.0 and 7.5 and its migratory velocity is much slower than that of the pressor factors.—RAYMOND JONNARD and MARVIN R. THOMPSON. *Jour. A. Ph. A.*, 31 (1942), 19. (Z. M. C.)

Camphor with Phenol and the Cresols—Physical Evidence of. Mixed with camphor, phenol loses its corrosive action, and so they appear to have combined, but chemical analysis gives the total percentage of phenol. An investigation was undertaken to find physical evidence of a combination or lack of it. Camphor with each of the cresols was tried also. These were of interest because no such investigation had been reported and because they have advantages in bactericidal properties. Experimental work is reported in much detail with numerous tabulations and graphs. Physical properties studied include freezing curves, densities, partial solubilities in water, partial vapor pressures, temperature effects on mixing liquid solutions and cryoscopic molecular weight determinations. Melting points for equimolecular complexes were found. A method of measuring partial pressures and a method of interpretation of temperature effects on mixing were devised. Various evidences point to the existence of at least two complexes in each mixture, namely, one mole of camphor with one and two moles, respectively, of phenol or cresol. All complexes are partially dissociated in solution and rapidly decomposed on chemical analysis. The low content of free phenol or cresol probably accounts for the high toleration shown by wounds and tissues. Being equilibrium mixtures, they will liberate free phenol or cresol as fast as that originally present is consumed. This may explain antiseptic and surface anesthetic effects.—ALFRED W. FRANCIS. *Jour. A. Ph. A.*, 30 (1941), 229. (Z. M. C.)

Carbon Dioxide and Carbonic Acid—Physical Chemistry of. A review was given of the equilibrium conditions for systems containing CO_2 , H_2CO_3 , HCO_3^- and CO_3^{2-} , with derivation of the constants for equilibrium and for speed of reaction.—C. FAURHOLT. *Farm. Revy*, 40 (1941), 629. (C. S. L.)

Colloidal Silver Iodine Compound Containing a Colloidal Zinc Compound. A composition suitable for use as an antiseptic is prepared by heating a mixture of gelatin and a water solution of sodium hydroxide to a boiling temperature, cooling the mixture and dividing it into two equal parts, mixing a water solution of a mild silver protein with one part, dissolving iodine crystals in that part, decanting the mixture to remove excess iodine, mixing a solution of zinc chloride with the other portion of the gelatin solution, mixing the two together and adjusting the pH value of the mixture to about 7.0 to 9.0.—MICHAEL G. MINAEFF, JOHN P. GREZE and RUDOLF J. PRIEPKE, assignors to A. C. BARNES CO., INC. U. S. pat. 2,249,023, July 15, 1941. (A. P.-C.)

Colloids—Moving Boundary Methods for the Determination of Cataphoretic Speed of. II. The suitability of the moving boundary methods as developed by Mukherjee for the determination of cataphoretic speeds of concentrated colloids in which the electrical conductivity of the colloid particles is comparable with those of the intercellular elec-

trolytes has been discussed in the light of the electrolytic migration theory of Kohlrausch and Weber. This theory has been verified with mixtures of KCl and KIO_3 . The conditions necessary for reliable cataphoretic measurements have been experimentally obtained with two ferric oxide and two aluminum oxide hydrosols, and cataphoretic speeds generally reproducible within 2% have been obtained. The cataphoretic speeds of the same sols have been obtained by the transport apparatus of Engel and Pauli and the results are also reproducible within about 2%. The results of the moving boundary method have been found to agree with those of the transport method almost within the range of experimental accuracy.—N. C. SEN-GUPTA and P. R. SINHA. *J. Indian Chem. Soc.*, 18 (1941), 489. (F. J. S.)

Colloids—Purification of. Apparatus is described, and a process of purification of colloids to separate them from contaminating matter which comprises placing a solution containing such substances in the cataphoretic compartment of a cell having at least one membrane permeable to the colloid, interposing a buffer salt solution between the compartment and the electrodes of the cell, the pH of this solution corresponding to that necessary to cause selective migration of the colloid, subjecting the colloid-containing solution to the electric current to cause migration of the colloid through the permeable membrane, and maintaining the pH substantially constant during the treatment. Details are given for the purification of the "yellow oxidation enzyme," intermediate respiratory enzymes from blood corpuscles and intermediate respiratory enzymes from yeast, and for the separation of serum albumin and serum globulin.—AXELL H. T. THEORELL. U. S. pat. 2,251,082, July 29, 1941. (A. P.-C.)

Crystalloids—Purification of. An apparatus and mode of operation are applied to the purification of crystalloids to separate them from other crystalloids and mixtures of crystalloids and colloids, details being given of the treatment of active liver preparations.—AXELL H. T. THEORELL. U. S. pat. 2,251,083, July 29, 1941. (A. P.-C.)

Detergent Solutions—Properties of. The pH measurements obtained by the hydrogen electrode, glass electrode and two colorimetric methods of the systems modified soda-water and modified soda-soap-water have been obtained and critically compared. Electrometric measurements were made at 25°, 40°, 60° and 80° C.; colorimetric measurements were limited to 25° C. The modified soda concentrations encompass the full range of detergent interests; soap concentration has been held at 0.1%. Contrary to general acceptance, the modified soda examined raises the pH of soap solutions at the usual white-work washing temperatures. pH measurements, even of the electrometric type, cannot be used as a means of concentration control for modified soda systems with or without soap. Colorimetric measurements are misleading at the lower builder concentrations of laundry interests because of inaccuracy and, further, because of inability to relate the values obtained to those prevailing under washing conditions. A survey of pH measurements made in the field discloses wide errors. Recent advances in the technique and standardization of pH measurements at elevated temperatures are discussed.—L. R. BACON, J. W. HENSLEY and T. H. VAUGHN. *Ind. Eng. Chem.*, 33 (1941), 723-730. (E. G. V.)

Glucose and Iodine—Reaction between, in Alkaline Medium. Effect of Neutral Salts. The addition of neutral salts, viz., chlorides, bromides and nitrates of potassium, sodium and ammonium, retards the speed of oxidation of glucose by iodine, and

the velocity constant of the reaction decreases with increasing concentration of the salts. The variation in the velocity constant with these different salts shows that the value of K for a given concentration of the salt is not independent of the nature of the added salt. Also, the oxidation of glucose by sodium hypoiodite is retarded in the following order: $\text{Cl} > \text{Br} > \text{NO}_3$ and $\text{NH}_4 > \text{Na} > \text{K}$. The decrease in the velocity constant can be attributed to the formation of complex ions between iodine and halide ions.—KESHO DASS JAIN and B. L. VAISH. *J. Indian Chem. Soc.*, 18 (1941), 503. (F. J. S.)

Hydrocarbon Chains—Monolayers of Compounds with Branched. V. Phthiocerol. Phthiocerol forms on different substrates a typical liquid expanded monolayer. At pH 2, the temperature of half expansion is 17° and the smallest area per molecule to which the monolayers can be compressed before collapse is 30 to 31 sq. Å. In the condensed state the monolayers are solid. The apparent surface moment is 880 millidebyes at the limiting area and falls to 450 at the point of collapse. It was not found possible to build perfect multilayers of phthiocerol and X-ray experiments on an imperfect Z-deposited film showed that the multilayer was poorly crystalline. The surface behavior of phthiocerol suggests that it is a very long molecule with only short side chains and with one or more of the polar groups near one end.—STINA STÅLLBERG and EINAR STENHAGEN. *J. Biol. Chem.*, 143 (1942), 171. (F. J. S.)

Hydroxybenzenes—Adsorption of, by Sugar Charcoal. Discontinuities in the Adsorption of Phenol, Resorcinol and Quinol from Aqueous Solutions. Adsorption by sugar charcoal of phenol, resorcinol and quinol from aqueous solutions has been studied.—KESHO DASS JAIN and J. B. JHA. *J. Indian Chem. Soc.*, 18 (1941), 535. (F. J. S.)

Indian Coals—Studies on. VI. Production of Activated Carbon from Indian Coals. The paper is summarized as follows: (1) Samples of activated carbon have been prepared from Indian coals by steam activation method. (2) The properties of the activated carbon samples were studied as regards the absorption of (a) dyes and (b) CCl_4 vapor. (3) Activated carbon samples prepared from non-coking coals were found to be more active than the carbon samples prepared from coking coals and they would appear to be of commercial value both for decolorization and for gas absorption purposes.—K. L. ROY and D. P. GUPTA. *Ind. & News Ed., J. Indian Chem. Soc.*, 4, No. 3, (1941), 189. (F. J. S.)

Iodination. II. Studies on the Iodination of Different Unsaturated Organic Compounds in the Dark in Different Non-Polar Solvents. Investigations on the velocity of addition of iodine to β -amylene and d -pinene in the dark in the presence of non-polar solvents have shown that the reaction is termolecular with respect to iodine but unimolecular with respect to the acceptor, *i. e.*, it is quadrimolecular with respect to both. The rate of reaction is not much affected by increase of temperature or by increase in the reaction surface, but it appears to depend to a great extent upon the nature of the solvent.—J. C. GHOSH, S. K. BHATTACHARYYA, M. M. DUTT and M. J. RAO. *J. Indian Chem. Soc.*, 18 (1941), 245. **III. Studies on the Iodination of Different Unsaturated Organic Compounds in the Dark in Polar Solvents.** Velocity of addition to β -amylene and phenylacetylene in the dark in polar solvents, *e. g.*, ethyl alcohol and acetic acid, has been investigated. The reaction is termolecular with respect to both iodine and the acceptor. The reaction rate appears to be greater in alcoholic solution than in acetic acid. The temperature coefficient of the reaction rate is not high.—S. K. BHATTACHARYYA and M. J. RAO. *Ibid.*, 253. **IV. Studies on the Photoiodina-**

tion of Different Unsaturated Organic Compounds in Light of Different Frequencies in Non-Polar Solvents. Photochemical iodination of phenylacetylene, dicyclopentadiene, dimethylacetylene dicarboxylate, β -amylene and phenylpropionic acid in 546, 436 and 366 μ in solutions of CCl_4 has been described. The reaction is unimolecular with respect to iodine. The rate of reaction has a small temperature coefficient. The quantum efficiency of the reaction has been worked out to be high in the case of phenylacetylene and dicyclopentadiene but small in other cases.—S. K. BHATTACHARYYA. *Ibid.*, 257. **V. Studies on the Photoiodination of Phenylacetylene in Light of Different Frequencies in Polar Solvent.** The kinetics and mechanism of the iodination of phenylacetylene in alcoholic solution in radiations of frequencies 366, 436 and 546 μ have been studied. Results indicate that the reaction, which is unimolecular in nature, is reversible and does not proceed to completion. The temperature coefficient of the reaction is small as also is the quantum efficiency.—S. K. BHATTACHARYYA. *Ibid.*, 269. (F. J. S.)

Magnetism and Catalysis. IV. Catalysis of the Reaction between Ammonium Oxalate and Mercuric Chloride by Ferric Ions. The influence of ferric ions in catalyzing the reaction between ammonium oxalate and mercuric chloride has been studied from magnetic standpoint. Results indicate that a new intermediate compound having a lower paramagnetic susceptibility value than that of ferric chloride is formed. It has not, however, been possible to ascertain definitely whether this intermediate compound is ferrous chloride or ferrous oxalate.—S. S. BHATNAGAR, P. L. KAPUR, ANAND SWAROOP BHATNAGAR and BRAHM PRAKASH. *Jour. Indian Chem. Soc.*, 18 (1941), 371. (F. J. S.)

Nickel—Effect of Colloids on the Electrodeposition of, on Copper. The effect of chromium, molybdenum, tellurium and tungsten sols on the electrodeposition of nickel has been investigated and the nature of the deposits has been studied.—V. S. PURI and S. R. SETH. *J. Indian Chem. Soc.*, 18 (1941), 465. (F. J. S.)

Nickel—Studies of Some Physical Factors in the Electrodeposition of, on Iron. Results are given for the optimum conditions for the production of a smooth, bright and adherent deposit of nickel on iron from a bath containing nickel sulfate, nickel chloride and boric acid. Data are given for the cathode efficiency in respect to nickel deposition under the influence of the electrolyte concentration, the current density, the duration, the inter-electrode distance, the temperature, the agitation of the bath, the superposition of a. c. and that of the addition of a large number of organic and inorganic agents in different proportions, such as aluminum sulfate, chromic acid, cadmium chloride, hydrogen peroxide, gelatin, etc. The possible role of the addition agents is also discussed. Detailed measurements are made of the cathode efficiency of nickel in the presence of methyl and ethyl alcohol, glycerol, mannitol and sucrose whose proportions were varied over a wide range. Data are also given for conductivity, relative viscosity and density.—DUSHYANT NARASINGASA SOLANKI and DAMRI SINGH. *J. Indian Chem. Soc.*, 18 (1941), 423. (F. J. S.)

Nickel Sulfate—Velocity of Hydration and Dehydration of. A method has been developed by which it is possible to determine the composition of the hydrates of inorganic salts by studying the velocity of their thermal dehydration under a fixed experimental condition. The process of hydration seems to be more complex and unsatisfactory.—B. N. GHOSH. *J. Indian Chem. Soc.*, 18 (1941), 472. (F. J. S.)

Oil-in-Water Emulsions—Viscosity of. Identical oil-in-water emulsions of 50/50 phase-volume have been prepared from a number of water-immiscible organic solvents using sodium and potassium soaps and fatty acids as the stabilizer. The viscosity of these was checked and rechecked in a U-tube viscometer at 20° C. From these data the active volume of the internal phase was determined by means of the Hatscheck's formula. An explanation of the observed variations is advanced, and a numerical correction factor in the Hatscheck's equation is given.—B. A. TOMS. *J. Chem. Soc.*, (1941), 542-547. (W. T. S.)

Ovalbumin Molecules—pH at the Surface of, and the Protein Error with Indicators. The following summary is given: (1) pH_s , the pH at the surface of a protein molecule, is different from that in the surrounding bulk phase. pH_s may be calculated either from the Gibbs-Donnan equilibrium, or from the electrokinetic mobility. For ovalbumin the two methods give results in reasonable agreement. (2) At constant bulk pH, pH_s varies with ionic strength, and consequently the acid- and base-binding powers of a protein vary with ionic strength. It is shown that this accounts for the major part of the influence of ionic strength on the titration curve of ovalbumin, between pH 2 and pH 5.5. (3) Indicators of opposite charge to a protein molecule tend to be adsorbed and give values of pH_s rather than bulk pH values. Results are given for ovalbumin solutions with bromocresol green, chlorophenol red and neutral red. It is not clear whether the whole of the protein error is due to the difference between pH_s and the bulk pH. (4) It is suggested that pH_s , and not pH_b , is the significant pH in enzyme activity, and that, to facilitate comparison of results, enzyme activities should be determined in solutions of constant ionic strength.—JAMES FREDERIC DANIELLI. *Biochem. J.*, 35 (1941), 470. (F. J. S.)

Photoelectric Fluorimeter and Some Applications. The fluorimeter is provided with two photocells, one in each light beam, which can be connected to the potentiometer to form a Klett-Summerson type of colorimeter. For this reason, the potentiometer has the logarithmic scale of that colorimeter in addition to the linear scale of the fluorimeter. The colorimeter can be used with the lines in the visible spectrum and the 3660 lines of the mercury lamp which can be isolated by glass filters, or the mercury lamp can be replaced by an incandescent lamp to use the instrument as a colorimeter of the usual type with filters that isolate narrow spectral regions. The colorimeter has the same linear calibration curve for substances that follow Beer's law and the ease of correction for blanks that is characteristic of the Klett-Summerson colorimeter.—F. KAVANAGH. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 108-111. (E. G. V.)

Plastic Substances—New Instrument for Rheological Studies of. In this paper are discussed the aims, details of construction, accessories and procedure of a new instrument for rheological studies. The instrument is to be regarded not as a fixed quantity invariable in make-up but as an embodiment of principles for accomplishing a certain purpose—namely, study of mechanical properties. Suitably modified forms of the instrument and its accessories should be useful in both control and research related to pectins, jellies, gelatins, ceramic slips, pharmaceutical gels, thermoplastics and polymerplastics. In fact, the principles may be employed in studying any materials which may be allowed to set under conditions of controlled past history (or path) with a view to measuring the forces required to produce incipient flow or permanent deformation. However, in this paper the instrument is discussed from

the point of view of its use as a research tool in studying lyogels.—C. R. BAILEY. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 173-177. (E. G. V.)

Ragweed Pollen Extracts—Electrophoretic Isolation of Constituents of. There has been found a major constituent which is negatively charged, unpigmented and migrates more slowly than the pigmented constituents. The unpigmented fraction is highly skin reactive in individuals with ragweed hay fever.—H. A. ABRAMSON, D. H. MOORE, H. GETTNER, J. GAGARIN and L. JENNINGS. *J. Am. Chem. Soc.*, 62 (1940), 1627. (E. B. S.)

Refractive Index and Surface Tension—Relationship between. Lorentz and Lorenz equation for molecular refraction has been combined with Sugden's parachor equation. The resulting equation $(n^2 - 1)/(n^2 + 2) = (R/P)\gamma^{1/4}$ is found to be correct even when R and P are calculated from their corresponding atomic and configurational constants. This equation has been used successfully in calculating values of surface tensions with the knowledge of those of refractive indices, and vice versa.—R. C. TRIPATHI. *J. Indian Chem. Soc.*, 18 (1941), 411. (F. J. S.)

Resins—Adsorptive Properties of Synthetic. V. The adsorption of inorganic acids, bases and salts by various resins has been studied. The acids are adsorbed in the order $HI > HCl > HBr$; $HNO_3 > H_2SO_4 > H_3BO_3$ and $H_2CrO_4 > HClO_4 > H_2SO_4 > H_3PO_4$; and bases $Ba(OH)_2 > Sr(OH)_2 > Ca(OH)_2$; $KOH > NaOH > LiOH > NH_4OH$. In the case of salts the order of adsorption of cations is $Rb > K > Na > Li$ and in anions $I > Br > Cl$. The acid-condensed phenolic resin does not adsorb the acids and salts. The order of sorption is explained on the basis of the polar concept.—S. S. BHATNAGAR and MAHENDRA SARUP. *J. Indian Chem. Soc.*, 18 (1941), 447. (F. J. S.)

Silver Chloride—Rhythmic Precipitation of, in Gelatin Tanned with Chromium Chloride. On allowing a highly concentrated silver nitrate solution to diffuse into a gelatin gel, tanned with chromium chloride of such concentration that all of the available Cl^- is combined with the gelatin in a test-tube, dirty white rings of silver chloride have been obtained. The study of the influence of concentration of gelatin, chromium chloride and silver nitrate on the dimensions of the rings and interspaces shows that they behave differently from those of $Ag_2Cr_2O_7$ rings in gelatin mixed with $K_2Cr_2O_7$.—C. S. NARWANI and G. T. GURSAHANI. *J. Indian Chem. Soc.*, 18 (1941), 531. (F. J. S.)

Spectrophotometers—Survey of. General principles of the color measurement of white and near-white shades are discussed and a few commercially available spectrophotometers described. The use of a polished white glass ("Vitrolite") instead of magnesium oxide as a standard is advocated.—K. S. GIBSON. *Paper Trade J.*, 111 (1940), TAPPI Sect., 135-139; through *J. Soc. Chem. Ind.*, 60 (1941), 47. (E. G. V.)

Sulfanilamide and Related Compounds—Dipole Moment of. Dipole moments of the following compounds were measured in dioxane and found to be: sulfanilamide 6.63, aniline 1.90, benzenesulfonamide 5.09, *p*-(*p*-aminophenyl)-benzenesulfonamide 6.71, xenylamine 2.07, *p*-phenylbenzenesulfonamide 5.20, metanilamide 5.63. Variations in the moments are interpreted as evidence that a form with a separation of charge contributes appreciably to the structure of the molecules. The connection between the contribution of these forms, the therapeutic effect, the rate of oxidation and the stability of the corresponding quinones is discussed.—W. D. KUMLER and I. F. HALVERSTADT. *J. Am. Chem. Soc.*, 63 (1941), 2182. (E. B. S.)

Sulfosuccinic Esters. New data are presented on thirty-six out of an almost unlimited number of possible esters of sodium sulfosuccinic acid. Several show a greater decrease in surface tension and wetting power in more dilute solution than any other wetting agents on the market or in the literature. There is a certain balance between polar and non-polar groups within the molecule that determines wetting power and reduction of surface tension, yet no definite relation has been shown between molecular structure and wetting power. The area and length of a few of these wetting agent molecules have been calculated. Results are within the range of data on similar compounds reported in the literature. The data presented are scarcely more than an introduction to the study of one extensive series of homologous esters; more research is needed.—C. R. CARYL. *Ind. Eng. Chem.*, 33 (1941), 731-737. (E. G. V.)

Sulfuric Acids—Raman Spectra of Substituted. I. The Raman spectra of $\text{NH}_2\text{SO}_3\text{H}$ and $\text{NH}_2\text{SO}_3\text{Na}$ have been photographed under different conditions. It is found that the tetrahedral structure of the sulfates persists in the amino derivative. Force constants have been calculated and their significance discussed.—JAGANNATH GUPTA and ANIL KUMAR MAJUMDAR. *J. Indian Chem. Soc.*, 18 (1941), 457. (F. J. S.)

Tanning Extracts—Diffusion Characteristics of Some Philippine. Tanning consists in treating hide with a substance that will penetrate or diffuse into, and later fix or combine with, the hide substance (collagen). Collagen is similar to gelatin, so tests of tanning materials were on the basis of their diffusion in gelatin jelly. Extracts from barks of anabiong, bitaog, kalumpit, kariskis, bunot, kamachile, bakauan, betel nut and black wattle of Philippine origin were used. Foreign materials tested were extracts of quebracho, black wattle, myrobalan, valonia, hemlock, oak and chestnut. Tanolin was the only chrometan used. Generally the vegetable materials diffused faster with an increasing pH value, while tanolin diffused faster with a decreasing pH value. Native black wattle, kamachile, bakauan and bitaog like quebracho diffused slowly and should be good for light leathers. Kalumpit like hemlock diffuses faster and is good for all kinds of leather. Bunot, kariskis and betel nut should be good for initial tannage as their diffusion rates are greater than hemlock. Slow diffusion and high astringency are combined characteristics. The most astringent of these extracts are native black wattle, kamachile, bakauan and bitaog; kalumpit is intermediate; and bunot, kariskis and betel nut are the least astringent of the substances tested.—LUZ BAENS, L. G. ALEJO and V. G. LAVA. *Philippine J. Sci.*, 72 (1940), 451; through *Rev. Filipina Med. Farm.*, 32 (1941), 21. (G. S. G.)

Vanadic Acid and Tartaric Acid—Studies on the Photochemical Activity of Mixtures of. I. Optical Properties of Mixtures of Vanadic Acid and Tartaric Acid. Reduction of These Mixtures in Light and in the Dark. The relationship between the hydrogen ion concentration and (a) optical rotation, (b) light absorption and (c) dark reaction of mixtures of vanadic and tartaric acids has been investigated. The reduction of vanadic acid by racemic-tartaric acid in the dark has been found to give a higher value for the velocity of the reaction than that for the reduction by *d*-, or *l*-, or *dl*-tartaric acids, at the same hydrogen ion concentration. The photo-reduction of mixtures of vanadic acid and tartaric acid has been studied in the visible and ultraviolet regions.—T. L. RAMA CHAR. *J. Indian Chem. Soc.*, 18 (1941), 507. (F. J. S.)

Vegetable Charcoal—Studies in the Adsorptive Power and Activation of. I. The following sum-

mary is given: (1) That the increase produced in the adsorptive power of special carbon for iodine on heating for two hours on blowpipe flame is equal to 20.5%. (2) That the increase produced by heating special carbon in contact with 10% of zinc and cobalt chlorides is 113.6% and 129.6%, respectively. (3) That the order of activity of the various zinc and cobalt salts on the adsorptive power of carbon for iodine is chloride > sulfate > acetate > phosphate > oxalate. (4) That the limiting values for activating special carbon for iodine are 3% in the case of zinc chloride and 6% in the case of cobalt chloride. (5) That platinum and nickel salts, which are so widely employed for activation purposes, are totally ineffective and do not alter the adsorptive power of carbon for iodine.—S. L. BHATIA and MANOHAR LAL. *Ind. & News Ed., J. Indian Chem. Soc.*, 4 (1942), No. 4, 236. (F. J. S.)

Vitamin E—Chemistry of. XXIX. Studies of the Behavior of Compounds Related to Vitamin E at the Dropping Mercury Electrode. Current-voltage curves at the dropping electrode of 6-hydroxychromans and 5-hydroxycoumarans have been determined in 50% methanol in well-buffered solutions. The half wave potentials of the various compounds were found to be unaffected by the concentration. The difference in half wave potentials of chromans and corresponding coumarans was found to be 10 millivolts, the coumarans being more easily oxidized than the chromans. A reaction mechanism of the electrode reactions has been proposed which accounts for the experimental facts. Current-voltage curves and half wave potentials have been determined for a great number of hydroquinones and quinones. The half wave potentials were found to correspond to the standard oxidation potentials of the various systems. Compounds related to vitamin E can be determined polarographically.—L. I. SMITH, I. M. KOLTHOFF, S. WAWZONEK and P. M. RUOFF. *J. Am. Chem. Soc.*, 63 (1941), 1018. (E. B. S.)

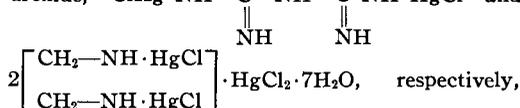
Vycor Brand Glass—Expansivity of. In a study of a new glass (96 per cent silica, glass No. 790) used in heat-resisting glassware, its expansivity is compared with that of fused quartz. For this comparison a modification of Fizeau's method for determining very small differences in linear expansion was used and is presented. This method has the advantage of permitting the use of relatively long samples, the procurement of very sharp and well-defined interference bands and the almost complete elimination of the effect of changes in the refractive index of air on the results. The results indicate that the No. 790 glass has a coefficient of expansion approximately twice that of fused quartz at room temperature, an expansivity equal to fused quartz at approximately 300° C., and an expansivity less than one-half of that for fused quartz, in the neighborhood of 700° C.—JAMES B. SAUNDERS. *J. Research Natl. Bur. Standards*, 28 (1942), 51-55. (W. T. S.)

INORGANIC

Aluminum Chromate—Normal. A method for the preparation of normal aluminum chromate has been described and various properties of this substance have been thoroughly investigated.—PRODOSH CHANDRA RAYCHOUDHURY. *J. Indian Chem. Soc.*, 18 (1941), 573. (F. J. S.)

Biguanide—Complex Compounds of, with Tervalent Metals. IX. Action of Mercuric Chloride and Silver Nitrate upon Chromium and Cobaltic Trisbiguanidinium Hydroxides and the Constitution of Biguanide Metal Complexes. According to the constitution suggested for the biguanide metal complexes in a previous communication it was expected

that the complex metal biguanide bases should behave like ammonia or the amines. Additional evidence in this respect has been brought forward in this paper by a study of the behavior of these complex bases toward mercuric chloride and silver nitrate. By adding mercuric chloride to chromium and cobaltic trisbiguanide bases, the compounds $\text{Cr}(\text{Big}\cdot\text{HgCl})_3$ and $\text{Co}(\text{Big}\cdot\text{HgCl})_3\cdot\text{HgCl}_2$, were obtained, which closely resemble the infusible white precipitate in their constitution and properties. It has also been shown that with AgNO_3 the cobaltic trisbiguanide hydroxide forms the compound, $\text{Co}(\text{BigH}\cdot\text{AgOH})_3$ aq., resembling silver ammine hydroxide. Two more compounds of mercuric chloride with biguanide and ethylenediamine hydroxide, $\text{ClHg}\cdot\text{NH}-\text{C}-\text{NH}-\text{C}\cdot\text{NH}\cdot\text{HgCl}$ and



also of the infusible white precipitate type, have been described. Besides, double salts of the composition, $[\text{Cr}(\text{BigH}_2)_3]\text{Cl}_3\cdot 3\text{HgCl}_2$, $[\text{Co}(\text{BigH}_2)_3]\text{Cl}_3\cdot 3\text{HgCl}_2\cdot 2\text{H}_2\text{O}$ and $[\text{Co}(\text{en})_3]\text{Cl}_3\cdot 5\text{HgCl}_2\cdot 2\text{H}_2\text{O}$ (in two different crystalline forms and color) have been prepared and their properties given.—PRIYADARANJAN RAY and SUSHIL KUMAR SIDDHANTA. *J. Indian Chem. Soc.*, 18 (1941), 298. (F. J. S.)

Cobalt—Magnetic Susceptibilities of Metallic Oxides and Their Molecular Structures with Special Reference to Those of. Magnetic susceptibility values of different samples of cobaltous, cobaltous and cobaltic oxides, prepared in different ways, have been measured. The observed values of μ_b have been utilized to discuss the molecular constitution of the cobalt oxides.—S. S. BHATNAGAR, BRAHM PRAKASH and MOHOMMED ABDUL QAYYUM. *J. Indian Chem. Soc.*, 18 (1941), 540. (F. J. S.)

Hydrogen Peroxide—Continuous Production of, from Potassium Persulfate. An arrangement of apparatus is described, and a process which involves forming a finely divided suspension of potassium persulfate, water and sulfuric acid and passing the suspension through a heated vessel at such a rate that most of the potassium persulfate is converted at least to a less oxidized form of sulfate therein, discharging the mixture from the vessel through a constricted passage of regulatable cross section into a zone of subatmospheric pressure, maintaining the suspension in the vessel at a temperature below its boiling point by correlating the pressure in the vessel, determined by the size of the passage and the heat applied to the vessel, heating the mixture in the zone of subatmospheric pressure to a temperature sufficient to produce and vaporize hydrogen peroxide, and separating the hydrogen peroxide from the residue.—MODESTE SALLERAS, assignor to BUFFALO ELECTRO-CHEMICAL Co. U. S. pat. 2,243,810, May 27, 1941. (A. P.-C.)

Paramolybdates—On the Composition and Constitution of. According to some workers the paramolybdates have the composition, $3R_2O\cdot 7MoO_3$ aq., and should be formulated as $R_6Mo_7O_{24}$ aq., while others represent them as $5R_2O\cdot 12MoO_3$ aq., or more definitely as $R_5H_5[\text{H}_2(\text{MoO}_4)_8]$ aq., due to Rosenheim. Jander in agreement with the latter view represents the paramolybdate as $R_5[\text{HM}_6\text{O}_{21}]$ aq. With a view to a critical examination of the problem, the following paramolybdates of complex metallic biguanide ions of high equivalent weight were prepared and their properties studied: $[\text{Co}(\text{BigH})_3]_2\text{Mo}_7\text{O}_{24}\cdot 9\text{H}_2\text{O}$; $[\text{Cu}(\text{BigH})_2]_3\text{Mo}_7\text{O}_{24}\cdot \text{H}_2\text{O}$; and $[\text{Ni}(\text{BigH})_2]_2R_2\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$, where R is NH_4 or Na . The results are in good accord with the older formula, $3R_2O\cdot 7MoO_3$ aq., showing that the paramolybdate ion is best represented as $[\text{M}_6\text{O}_{24}]^{6-}$.

The conclusion is based mainly upon the analytical composition of the compounds, their dehydration, followed by rehydration and other general chemical properties.—PRIYADARANJAN RAY and SUSHIL KUMAR SIDDHANTA. *J. Indian Chem. Soc.*, 18 (1941), 397. (F. J. S.)

Periodates of Trivalent Metals. Reaction of trisodium paraperiodate on trivalent metallic salts has been studied and also the tetravalent lead periodate and manganese mesoperiodate.—PRODOSH CHANDRA RAYCHOUHDURY. *J. Indian Chem. Soc.*, 18 (1941), 576. (F. J. S.)

Water—Hardness of. A brief review was given of the industrial importance of knowledge of the hardness of water and of methods of softening water by zeolite base-exchange or by adding soda, sodium hydroxide or basic phosphates.—J. QUJST. *Farm. Revy*, 40 (1941), 563. (C. S. L.)

ORGANIC

Alkaloids

Alkaloids—Determination of, by Precipitation with Silicotungstic Acid and Colorimetry. The alkaloids are precipitated in acid solution with silicotungstic acid and the excess of the reagent is determined by measuring the blue color produced by treating the liquid with titanium dichloride which reduces the tungstic acid. Mix two cc. of a 5% solution of silicotungstic acid in dilute hydrochloric acid with 20 cc. of 2% hydrochloric acid. This is used as the standard. Mix 1 cc. of the alkaloidal solution with 7 cc. of 2% hydrochloric acid and 2 cc. of 5% silicotungstic acid. Centrifuge and wash the precipitate with a few cc. of 2% hydrochloric acid, centrifuge again and make the combined liquids up to 20 cc. Mix 5 cc. of each solution with 0.5 cc. of a solution prepared from 1 volume of 15% titanium dichloride with 9 volumes of 2 N hydrochloric acid and compare in a Pulfrich photometer. The extinction curves for the silicotungstic acids are determined separately. The factor for $\text{SiO}_2\cdot\text{WO}_3$ precipitated is 0.4064 for atropine, 0.2925 for pilocarpine and 0.422 for strychnine.—M. MASCRÉ and J. LOISEAU. *Bull. sci. pharmacol.*, 48 (1941), 273–80. (S. W. G.)

Alkaloids from Koto-Tsururafuji (Stephania Sasakii Hayata). Rhizomes of Koto-tsururafuji (*Stephania sasakii* Hayata) are extracted with alcohol. The extract is lixiviated with hydrochloric acid to dissolve the base. The latter is precipitated by rendering the acid solution alkaline with ammonia and is extracted with ether. The ether is removed, the crude alkaloid is dissolved in hydrochloric acid, the solution is rendered alkaline with caustic soda, the precipitate is extracted with ether and the ether is evaporated. The resultant non-phenolic bases are dissolved in acetone with addition of benzene or toluene, and the solution is allowed to stand until the addition product of cepharanthine and benzene compound separates.—HEISABURO KONDO and SYUZI HASEGAWA. U. S. pat. 2,248,241, July 8, 1941. (A. P.-C.)

Alkaloids of Rauwolfia Canescens L. II. Rauwolfscine is hydrolyzed by aqueous alkali to rauwolfscinic acid, $\text{C}_{20}\text{H}_{24}\text{O}_5\text{N}_2$ which contains one CO_2H group. It can be reconverted into rauwolfscine by esterification with methyl alcohol. Ethyl, *n*-propyl and *n*-butyl esters of rauwolfscinic acid have also been described as well as some of their salts. The color reactions of rauwolfscinic acid are similar to those of the parent compound and yohimbine. The ultraviolet absorption spectra of rauwolfscine and yohimbine have been found to be very similar.—ASIMA MOOKERJEE. *J. Indian Chem. Soc.*, 18 (1941), 485. (F. J. S.)

Atropine—Colorimetric (*p*-Dimethylaminobenzaldehyde-Sulfuric Acid) Method for Determining Small Quantities of. A simple colorimetric method for the estimation of small quantities of atropine is described. Conditions for the optimum color intensity have been obtained and under the conditions laid down, the relationship between color intensity and quantity of atropine is shown to be linear.—R. P. DAROGA. *J. Indian Chem. Soc.*, 18 (1941), 579. (F. J. S.)

Ergonovine—Salts of. Details are given of the production of ergonovine maleate and ergonovine succinate, which are suitable for parenteral administration.—MORRIS S. KHARASCH and ROMEO R. LEGAULT, assignors to ELI LILLY & Co. U. S. pat. 2,220,801, Nov. 5, 1940. (A. P.-C.)

Ergot Alkaloids—Colorimetric Determination of. After addition of *p*-dimethylamido-benzaldehyde, add to the alkaloid salt solution traces of an oxidizing action, the action of which manifests itself by the appearance of a blue color; remove the excess of oxidizing agent by a reducing agent. Determine the intensity of the color by means of a photometer. The reaction based on oxidation is purely chemical and can be carried out even in the absence of light, and is not a phot. chemical reaction as Smith had thought.—E. SCHULKE and G. VASTAGH. *Magyar Gyógyszerészeti Társaság Értesítője*, 15 (1939), 322-325; through *Chemie & Industrie*, 43 (1940), 925. (A. P.-C.)

Erythramine and Its Salts. A process for the production of crystalline erythramine, an alkaloid obtainable from certain species of *Erythrina*, in the form of salts from the active free alkaloidal fraction obtained from *Erythrina sandwicensis* Deg., comprises the steps of dissolving the alkaloidal fraction in absolute alcohol, treating the solution with hydrobromic or hydriodic acid and crystallizing the erythramine salt from the liquor. The hydrobromide melts (with decomposition) at 228° C., and the hydriodide at 219° C.—KARL FOLKERS and FRANK KONIUSZY, assignors to MERCK & Co. U. S. pat. 2,252,709, Aug. 19, 1941. (A. P.-C.)

Erythrina Alkaloids. VIII. Studies on the Constitution of Erythramine and Erythraline. Erythraline, $C_{18}H_{19}NO_3$, contains one methoxy group and a methylenedioxy group. The nitrogen atom is tertiary and in all probability is common to two nuclei of the molecule, since it was shown that the tetrahydro derivative was identical with dihydroerythramine. The unsaturation consists of two ethylenic double bonds and one benzenoid nucleus. Erythramine and erythraline appear to contain four nuclei exclusive of the methylenedioxy bridge. Absorption and oxidation studies suggest nuclear formulations for erythramine and erythraline.—KARL FOLKERS and F. KONIUSZY. *J. Am. Chem. Soc.*, 62 (1940), 1673. (E. B. S.)

Erythrina Alkaloids. X. Isolation and Characterization of Erysonine and Other Liberated Alkaloids. The liberated alkaloidal fractions obtained from the seeds of nine species of *Erythrina* have been examined. Erysodine and erysopine were isolated in many cases. One new liberated alkaloid named erysonine was isolated from the seeds of one species. Erysonine has the empirical formula $C_{17}H_{19}NO_3$. Certain preliminary facts about its constitution have been described. Erysonine possesses a curare-like action in frogs when administered by intralymphatic injection. The threshold dose is 100 mg./Kg.—K. FOLKERS, J. SHAVEL, JR., and F. KONIUSZY. *J. Am. Chem. Soc.*, 63 (1941), 1544. (E. B. S.)

Fritilaria Roylei—Alkaloids of. II. Isolation of Peiminine. Peiminine has been obtained in a very pure condition and its properties are recorded. Its formula is $C_{28}H_{43}NO_3$ instead of $C_{27}H_{42}NO_3$, which was previously assigned to it.—YUOH-FONG CHI,

YEE-SHENG KAO and KOU-JEN CHANG. *J. Am. Chem. Soc.*, 62 (1940), 2896. (E. B. S.)

Nicotine Alkaloids from Commercial Nicotine Sulfate. Sodium or potassium hydroxide is added to the commercial nicotine sulfate until at least a slight excess is present, and the resulting mixture is dehydrated by heating with dephlegmation of the vapor under reduced pressure.—KARL B. EDWARDS. U. S. pat. 2,251,041, July 29, 1941. (A. P.-C.)

Totaquina—Manufacturing Process of. Totaquina is the quinine product produced by the Bureau of Science of the Philippines from cinchona bark grown in the Island of Mindanao. The quills are ground, screened and reground in a ball mill to assure a uniform fine powder. It is mixed with lime and water and charged into percolators. Fresh bark is lixiviated with weak alcohol percolate, while fresh alcohol is passed through spent bark lixiviated the previous day. The strong percolate is charged into a simple batch still to volatilize, the alcohol is recovered and the gummy residue is treated with 5% sulfuric acid to get the alkaloids in solution. Then the remaining gums and resins are filtered on a canvas mat, and the liquid is pumped into a decolorizing kettle where charcoal is added. This mixture is filtered and caustic soda is added to crystallize the alkaloids which are filtered over canvas in the totaquina filter and the filtrate returned to the precipitating tank. Further addition of caustic soda precipitates the amorphous alkaloids which are similarly filtered. The precipitates are dried on plates by a fan circulating warmed air. The dried totaquina is powdered in a pebble mill and fed into a tablet machine. All equipment is stainless steel. This totaquina has become of sufficient importance to break the Dutch monopoly in the Philippines.—RAFAEL SIMPAO. *Rev. Filipina Med. Farm.*, 32 (1941), 80. (G. S. G.)

Glycosides, Ferments and Carbohydrates

Acetoacetic Acid—The Acid-Splitting Reactions of, and the Enzymatic Formation of Acetic Acid from Acetoacetic Acid. The following summary is given: (1) Acetic acid was the only product obtained when acetoacetic acid was hydrolyzed by strong bases. No other catalyst could be found for the reaction. (2) Rabbit muscle mince formed small quantities of acetic acid from acetoacetic acid when incubated at 38° for two hours. (3) Pure cultures of *Escherichia coli* formed large quantities of acetic acid anaerobically from acetoacetic acid. (4) The catalytic system involved in the muscle mince and the bacteria was thermolabile and possessed other classical properties of an enzyme.—ALBERT L. LEHNINGER. *J. Biol. Chem.*, 143 (1942), 147. (F. J. S.)

Aconitic Acid from Sugar Cane Products. The cream-colored sediment occurring in syrup and first and second molasses tanks during recent years in certain areas of Louisiana was studied and found to consist principally of calcium aconitate. The sediment was analyzed, and a method of separating aconitic acid from it and from related materials and of purifying it is described. The isolated acid decomposed at 191.5° C. (194° C. corrected), and other characteristic properties are described which identify it as the *trans* isomeric form of aconitic acid. Refinery pan and evaporator scales were analyzed and found to contain aconitic acid. A simple test for the detection of aconitic acid in sediments and scales is described. The aconitic acid content of syrups made without chemical clarification from juices from two different types of cane grown in different localities was determined and found to range from 0.75% to 1.33% on solids. Two samples of Louisiana final molasses were analyzed and found to contain 1.80% and 2.52%

aconitic acid.—M. A. McCALIP and A. H. SEIBERT. *Ind. Eng. Chem.*, 33 (1941), 637-640. (E. G. V.)

Cellulosic Materials—Hydrolysis and Catalytic Oxidation of. A method is described by which quantitative, hydrolytic decomposition rates of glucose polymers, particularly cellulose, can be determined. The basis of the method is the fact that the normally slow evolution of carbon dioxide from glucose in acid solutions can be increased at will by the use of a suitable catalyst; the accelerated evolution rate is directly proportional to the free glucose in the system. Glucose set free by hydrolysis can be estimated continuously. Experimental conditions and methods of calculation are presented.—R. F. NICKERSON. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 423-426. (E. G. V.)

Collagen—Carbohydrate in. It is surmised that the glucose and galactose in the carbohydrate of collagen are both levorotatory since they are not fermentable with a galactose-active yeast which ferments the dextro-galactose.—J. BEEK, JR. *J. Am. Chem. Soc.*, 63 (1941), 1483. (E. B. S.)

Cottonseed Oil—Sterol Glucosides from. Mixed sterol glucosides were isolated from cottonseed oil by treatment of the oil with an adsorbent and subsequent extraction of the adsorbed material with acetone. The sugar obtained by acid hydrolysis was identified as *d*-glucose. The mixed sterol glucosides isolated from cottonseed oil differ in some respects from those isolated from the cotton plant by Power and Chesnut. These differences are apparent from the higher melting point of the glucosides from the oil and the lower melting point of the sterols which these glucosides yield on hydrolysis.—M. H. THORNTON, H. R. KRAYBILL and F. K. BROOME. *J. Am. Chem. Soc.*, 63 (1941), 2079. (E. B. S.)

Enzymes—Studies in. Amylase from Kaseru (Scirpus Grossus L.). The amylase from Kaseru has an optimum pH between 5.8 and 6.2 and an optimum temperature between 50° and 55° using buffers according to Clark and Lubbs.—J. P. SHUKLA. *J. Indian Chem. Soc.*, 18 (1941), 407. (F. J. S.)

Mucilage from Sugar Cane Juice by Bacteria. In the course of an investigation, at the Imperial Institute of Agricultural Research, Delhi, of the contamination of sugar cane juice, a certain type of bacteria was isolated which is capable of producing gum by artificial incubation at an optimum temperature of 20° C. The organism has been identified as *Leuconostoc mesenteroides*. Of the various preservatives tried at the Institute to ensure the keeping qualities of the mucilage produced, sodium benzoate has been found most effective. The addition of alum and aluminum sulfate has been found to increase the rate of drying.—ANON. *Scientific Rep. Imp. Agr. Res. Inst.*, (1939-1940); through *Ind. & News. Ed.*, *J. Indian Chem. Soc.*, 4, No. 3, (1941), 207. (F. J. S.)

Polyphenoloxidase—Some Properties of a, Present in Cell-Free Kidney Extracts. A cell-free extract has been prepared which is capable of oxidizing certain polyphenols, namely, catechol and hydroquinone. It is ineffective in oxidizing either tyrosine, dihydroxyphenylalanine (dopa), or *p*-cresol. The substance is partially inactivated by heat, is precipitated by either alcohol or acetone without unreasonable loss of activity and is not dialyzable. From the data it appears that the substance is similar to a laccase.—J. F. CADDEN and L. V. DILL. *J. Biol. Chem.*, 143 (1942), 105. (F. J. S.)

Saponins and Sapogenins. XVII. The Structure of the Side Chain of Chlorogenin. Methyl chlorogenoate diacetate reacts readily with ammonia to give a compound whose ultraviolet absorption spec-

trum indicates that it contains a pyrrole nucleus. This is evidence that the original ester contains the 1,4-diketone grouping and favors the ketone spiro-acetal formulation for the side chains of steroid sapogenins.—K. LADENBURG and C. R. NOLLER. *J. Am. Chem. Soc.*, 63 (1941), 1240. (E. B. S.)

Saponins and Sapogenins. XVIII. The Non-Identity of Chlorogenonic, Digitogenic and Digitonic Acids. Chlorogenonic acid is not identical with either digitogenic or digitonic acid. Wolff-Kishner reduction yields gitogenic acid showing that oxidation of chlorogenin has taken place between C-2 and C-3. On the basis of our present knowledge, these facts are in conflict with the location of the hydroxyl groups of chlorogenin at the 3- and 6-positions.—C. R. NOLLER and S. LIEBERMAN. *J. Am. Chem. Soc.*, 63 (1941), 2131. (E. B. S.)

Saponins and Sapogenins. XIX. The Decarboxylation of Echinocystic Acid. Echinocystic acid on vacuum distillation loses a molecule of carbon dioxide and one of water to give norechinocystadienol. The ultraviolet absorption spectrum shows that this compound contains two conjugated double bonds in different rings. These facts are in accordance with the proposed structure of echinocystic acid.—C. R. NOLLER and J. F. CARSON. *J. Am. Chem. Soc.*, 63 (1941), 2238. (E. B. S.)

Starch (Root)—Rapid Determination of, with Sodium Hypochlorite. Weigh out a portion of the sample containing from 1 to 2 Gm. of starch and transfer to a 400-cc. tall-form beaker with about 100 cc. of water or of about 25 per cent alcohol solution in case of dried pulp containing gelatinized starch. After allowing mixture to digest at room temperature for about half an hour, add 10 cc. of a 10% solution of diatomaceous earth and transfer to a Gooch crucible fitted with an asbestos mat prepared in some standard manner. Wash the sample with about 100 cc. of water or dilute alcohol. Transfer the washed sample with asbestos mat quantitatively to the same beaker with from 50 to 75 cc. of water. Add a small quantity of sodium hypochlorite solution and heat on a hot plate; boil the mixture 15 minutes, giving the beaker a rotating motion, or stirring occasionally to keep the insolubles suspended in the water the greater part of this period. Add the main portion of hypochlorite solution at the end of the gelatinization period and continue boiling for exactly 5 minutes, again agitating at times to rinse down the material which may collect on the sides of the beaker. Set aside for a few minutes and transfer while still warm to a 100-cc. volumetric flask. Cool quickly to room temperature; add about 5 drops of concentrated acetic acid (as an aid to clarification and filtration), make up to the mark with water; and add an extra quantity of water equal to the volume of the insoluble matter (chiefly asbestos and diatomaceous earth). In the tests reported, 1 cc. was taken as the correction volume, but each analyst should determine this correction for each type of material or change of procedure which might influence the value. Filter through fluted paper and read the rotation of the clear solution in a 2-dm. tube in a saccharimeter (or polarimeter). Calculate the starch from the reading in degrees Ventzke in accordance with the equation:

$$\% \text{ starch} = \frac{(V. \times 0.0880) 100}{\text{wt. of sample taken}}$$

—R. T. BALCH. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 246-248. (E. G. V.)

Sterols. CXXI. Sapogenins. XLVIII. Bromosarsapogenin and Bromodiosgenin. The oxidation of bromosarsapogenin acetate with chromic acid gave only the C-22 keto acid (3-hydroxy-16-ketobis-nor-cholanic acid). 23-Bromodiosgenin acetate

has been prepared and various reaction products from this are described.—R. E. MARKER, D. L. TURNER, A. C. SHABICA and P. R. ULSHAFFER. *J. Am. Chem. Soc.*, 63 (1941), 1032. (E. B. S.)

Sterols. CXXIII. Sapogenins. XLIV. The Biological Transformation of 4-Dehydrotigogenone to Diosgenin. The authors have successfully tested the possibility of a biotransformation of a Δ^4 -3-keto-steroid to a Δ^5 -3-hydroxysteroid with 4-dehydrotigogenone, the analog of cholestanone in the sapogenin series. Diosgenin was definitely produced and serves as proof of the transformation.—R. E. MARKER, R. B. WAGNER, D. L. TURNER and E. L. WITTBECKER. *J. Am. Chem. Soc.*, 63 (1941), 1769. (E. B. S.)

Sterols. CVI. Sapogenins. XXXV. The Supposed Trillarigenin. Trillarigenin, which was thought to be a diglucoside of a sapogenin from *Trillium erectum* (L.), has been identified as a mixture of diosgenin and trillin, the latter being a glycoside.—R. E. MARKER and J. KRUEGER. *J. Am. Chem. Soc.*, 62 (1940), 2548. (E. B. S.)

Sterols. CVII. Steroidal Sapogenins of Alettris, Asparagus and Lillium. Diosgenin and sarsasapogenin have been obtained from the roots of *Alettris farinosa* (L.) and *Asparagus officinalis* (L.), respectively. A new steroidal sapogenin has been obtained from bulbs of a Japanese lily.—R. E. MARKER, D. L. TURNER, A. C. SHABICA, E. M. JONES, J. KRUEGER and J. D. SURMATIS. *J. Am. Chem. Soc.*, 62 (1940), 2620. (E. B. S.)

Tyrosinase—A Comparison of the Activities of, toward Phenol and *p*-Cresol. The loss in activity toward *p*-cresol occurring when tyrosinase is purified by certain methods, was not observed when phenol was used in place of *p*-cresol as the substrate. The activity of high cresolase preparations toward *p*-cresol is some 4–5 times greater than toward phenol. The oxidation of phenol by means of tyrosinase, like the oxidation of *p*-cresol by the enzyme, requires the simultaneous oxidation of an *o*-dihydric phenol.—D. C. GREGG and J. M. NELSON. *J. Am. Chem. Soc.*, 62 (1940), 2506. (E. B. S.)

Tyrosinase—Action of, on Hydroquinone. Evidence is offered indicating that the action of tyrosinase on hydroquinone resembles the action of the enzyme on monohydric phenols. When hydroquinone is oxidized by tyrosinase in the presence of a small amount of catechol, the reaction and the product formed are different from those occurring when hydroquinone is oxidized by means of laccase. A greater total oxygen uptake occurs in the oxidation of hydroquinone by tyrosinase when the ratio of the activity toward monohydric phenols over the activity toward *o*-dihydric phenols is large.—D. C. GREGG and J. M. NELSON. *J. Am. Chem. Soc.*, 62 (1940), 2510. (E. B. S.)

Tyrosinase. Further Studies on the Enzyme. The following three facts appear to be significant: first, tyrosinase oxidizes *p*-cresol without the simultaneous oxidation of an *o*-dihydric phenol; second, in the presence of *p*-cresol, tyrosinase cannot oxidize catechol any faster than the rate required for priming the enzyme toward *p*-cresol; third, in the case of the high cresolase preparations of tyrosinase, both the activity toward catechol and toward *p*-cresol are proportional to the same copper content. This suggested overlapping of activities is a strong indication that the two activities belong to the same enzyme complex.—D. C. GREGG and J. M. NELSON. *J. Am. Chem. Soc.*, 62 (1940), 2500. (E. B. S.)

Tyrosinase—On the Nature of the Enzyme. II. A method is described for preparing tyrosinase from the common mushroom, *Psalliota campestris*, high in cresolase activity. In the preparations high in

cresolase activity the latter is proportional to copper, and to catecholase activity, both activities being dependent on the same copper content. The Adams and Nelson method of determining catecholase activity does not apply in preparations high in cresolase activity. In high cresolase preparations the volume of oxygen uptake corresponding to the inactivation of an amount of catecholase containing $\gamma/350$ of copper, was found to be 150 μ mm. instead of 100 μ mm. as found by Ludwig and Nelson in the case of high catecholase preparations.—G. G. PARKINSON, JR., and J. M. NELSON. *J. Am. Chem. Soc.*, 62 (1940), 1693. (E. B. S.)

Yeast—Molecular Constitution of an Insoluble Polysaccharide from. The structure of an insoluble polysaccharide isolated from yeast (*Saccharomyces cerevisiae*) was studied. The low specific rotations of the acetylated and methylated derivatives, and the upward mutarotation during hydrolysis suggest that the glucosidic linkages of the anhydroglucose units are predominately of the β -type. On methylation and subsequent hydrolysis of the polysaccharide 2,4,6-trimethylglucose was obtained as the sole product of hydrolysis. No end-group (tetramethylglucose) could be detected. This suggests that the molecule is probably of the closed chain type. The molecular weight of the polysaccharide determined by the Staudinger viscosity method was approximately 6500.—W. Z. HASSID, M. A. JOSLYN and R. M. MCCREADY. *J. Am. Chem. Soc.*, 63 (1941), 295. (E. B. S.)

Other Plant Principles

Campesterol—On the Constitution of. From the chromic acid oxidation products of campesterol acetate were isolated acetone and *d*-methyl isohexyl ketone and from campestanil acetate β -3-hydroxy-*nor-allo*-cholanolic acid. Campesteryl *p*-toluenesulfonate refluxed with methanolic potassium acetate forms a characteristic campesteryl *i*-methyl ether. These facts confirm the empirical formula $C_{28}H_{48}O$ and show that campesterol is an isomer of 22,23-dihydrobrassicasterol which differs only in the optical configuration on C-24.—E. FERNHOLZ and W. L. RUIGH. *J. Am. Chem. Soc.*, 63 (1941), 1157. (E. B. S.)

Cannabinol, Cannabidiol and Quebrachitol—Isolation of, from Red Oil of Minnesota Wild Hemp. Crystalline cannabidiol has been described for the first time. A procedure for isolating cannabinol from red oil of Minnesota wild hemp is given. It was isolated through the 3,5-dinitrophenyl urethan from cannabidiol-free red oil. Cannabinol was obtained crystalline. From red oil by distillation or by water extraction of crude red oil, or from the aqueous layer after steam distillation of the hemp extract, quebrachitol was isolated.—ROGER ADAMS, D. C. PEASE and J. H. CLARK. *J. Am. Chem. Soc.*, 62 (1940), 2194. (E. B. S.)

Cannabidiol—Structure of. IV. The Position of the Linkage between the Two Rings. By absorption spectra studies it was concluded that the methyl residue in tetrahydrocannabidiol dimethyl ether was linked to the olivetol between the methoxy groups. The synthetic compounds were prepared by unequivocal methods. The 2-lithium resorcinol or orcinol dimethyl ethers were condensed with menthone. The tertiary alcohols thus produced were dehydrated to the corresponding olefins and the olefins reduced with platinum oxide and hydrogen. In a similar manner 4-lithium resorcinol dimethyl ether was converted to 4-(3'-menthyl)-1,3-dimethoxybenzene. This compound and the 4-(3'-menthyl)-1,3-dimethyl-5-methylbenzene were also made by direct condensation of 1-menthol and resorcinol or orcinol followed by methylation. Tetrahydrocannabidiol dimethyl ether was prepared

by the reduction of cannabidiol dimethyl ether in glacial acetic acid with platinum oxide catalyst.—ROGER ADAMS, H. WOLFF, C. K. CAIN and J. H. CLARK. *J. Am. Chem. Soc.*, 62 (1940), 1770. (E. B. S.)

Cannabinol—Structure of. I. Preparation of an Isomer, 3-Hydroxy-1-*n*-amyl-6,6,9-trimethyl-6-dibenzopyran. The preparation of 3-hydroxy-1-*n*-amyl-6,6,9-trimethyl-6-dibenzopyran is described. Olivetol was condensed with 4-methyl-2-bromobenzoic acid to give 3-hydroxy-1-*n*-amyl-9-methyl-6-dibenzopyrone. The pyrone was converted to the corresponding pyran by treatment with excess methylmagnesium iodide. The product was not identical with cannabinol. Its derivatives were different from the corresponding derivatives of cannabinol.—ROGER ADAMS, D. C. PEASE, J. H. CLARK and B. R. BAKER. *J. Am. Chem. Soc.*, 62 (1940), 2197. (E. B. S.)

Capsicum and Its Assay. A review of the isolation and chemistry of capsaicin is offered. Proposed methods of assay are discussed and the following assay method proposed: Accurately weigh 2 Gm. of powdered capsicum, which has been previously dried for 24 hours over calcium chloride, place in a glass-stoppered flask and add exactly 100 cc. of ether, allow to macerate with occasional shaking for 1 hour. Pipette off a 50-cc. aliquot portion of the clear supernatant liquid and place in a 100-cc. beaker and allow the ether to evaporate slowly on a warm water bath. Dissolve the residue in 10 cc. of petroleum ether and transfer to a separatory funnel. Wash the beaker with several portions of petroleum ether and transfer the washings to the funnel. Extract the petroleum ether solution of the oleoresin with 10-cc. portions of 60% methyl alcohol 5 times and discard the petroleum ether layer. Should the combined alcoholic extracts contain an appreciable amount of coloring matter, shake further with petroleum ether. Evaporate the alcoholic solution nearly to dryness and place in a desiccator for 24 hours. Dissolve the crude capsaicin in exactly 50 cc. of dry acetone. Take exactly 10 cc. of this acetone solution and exactly 10 cc. of a 0.01% solution of pure capsaicin and add to each 0.3 cc. of freshly prepared 1% vanadium oxychloride solution (to 1 cc. of vanadium oxychloride add enough carbon tetrachloride to make 100 cc.). Mix the solutions thoroughly, immediately place in a colorimeter and make reading. The per cent capsaicin is calculated as follows: reading of the known:reading of the unknown = $x:0.01$ and the value of x is multiplied by 50. Twelve references.—C. B. JORDAN, E. W. REBOL and H. O. THOMPSON. *Bull. Natl. Formulary Committee*, 10 (1942), 49-57. (H. M. B.)

Menthol in Oil of Peppermint—Determination of. One Gm. of the sample, accurately weighed in a tared acetylation flask, is treated with 5 cc., accurately measured by means of a Koch microburette, of a freshly prepared acetylant mixture consisting of 4 parts by volume of *n*-butyl ether and one part of acetic anhydride. A blank is prepared in an identical manner, omitting the oil. The flasks are connected to air condensers and the contents boiled gently for 1 hour on a sand bath. Without removing the flasks from the bath, 20 cc. of hot distilled water are added through the condensers and the contents are boiled vigorously for an additional 30 minutes to convert the excess anhydride into acetic acid. After removing the flasks from the bath and allowing the contents to cool to room temperature, 20 cc. of cold distilled water are added as before. The flasks are removed from the condensers and the ground-glass connections rinsed by means of a water wash bottle, allowing the rinsings to flow into the flasks. At this point the acid strength of the blank is approximately 0.5 *N*. Eight or 10 drops of phenol-

phthalein test solution (dissolve 1 Gm. of phenolphthalein in 100 cc. of alcohol) are added and the excess acid is neutralized with 0.5 *N* alcoholic potassium hydroxide solution. The blank is titrated to the full red color of the indicator and the oil sample matched with the blank. It is necessary to conduct this titration on a 2-phase system, but this presents no difficulty. The difference in the amount of potassium hydroxide indicates the percentage of free alcohols present in the original sample. The calculation of these results is made by the following formula:

$$\% \text{ free alcohol} = \frac{\text{mol. wt. of alcohol}}{20} \times \text{cc. 0.5 } N \text{ KOH} \\ \text{wt. of sample}$$

If the sample to be analyzed is not perfectly anhydrous, it should be dried; allowance should be made for the acid value of those oils containing free acids. If the percentage of total alcohols is desired and ester determination is made by the regular method, then the following formula will effect the necessary conversion:

$$\% \text{ total alcohols} = \% \text{ free alcohols} + \frac{\text{mol. wt. of alcohol}}{\text{mol. wt. of ester}} \times \% \text{ esters.}$$

The procedure permits a more selective esterification of alcohols than the U. S. P. method; it is applicable for the analysis of free primary or secondary alcohols in any of the essential oils.—T. W. BRIGNALL. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 166-169. (E. G. V.)

Monocrotaline—Structure of. IV. Monocrotalic Acid. Ethyl α -carbethoxy- α,β -dimethyllevulinate was prepared and hydrolyzed. Alkali gave the corresponding malonic acid and acid caused the direct formation of α,β -dimethyllevulinic acid. Ethyl β -carbethoxy- α,β -dimethyllevulinate was also synthesized and hydrolyzed. With both acid and alkali α,β -dimethyllevulinic acid was obtained. These results lead to the conclusion that two of the previously proposed structures for monocrotalic acid are probably incorrect since monocrotalic acid loses carbon dioxide when treated with alkali and is stable to acid. α,β -Dimethyllevulinic acid has been synthesized by the two methods indicated above and has been shown to be identical with monocrotalic acid. This was done by comparing and showing to be identical the 2,4-dinitrophenylhydrazones of the methyl esters of the natural and synthetic acids.—ROGER ADAMS and R. S. LONG. *J. Am. Chem. Soc.*, 62 (1940), 2289. (E. B. S.)

Phytosterol—Isolation of a New. Campesterol. A new phytosterol, campesterol, has been isolated from rapeseed oil, soya bean oil and wheat germ oil. The analyses indicate that it has an empirical formula of $C_{28}H_{48}O$. It has not been found in cottonseed oil or in tall oil.—E. FERNHOLZ and H. B. MACPHILLAMY. *J. Am. Chem. Soc.*, 63 (1941), 1155. (E. B. S.)

Salvia Carnosa (Dougl). II. Carnosol. This new crystalline compound crystallizes in white odorless needles. It is soluble in ethanol, methanol, ether and chloroform but is insoluble in water, dilute hydrochloric acid and a 5 per cent solution of sodium bicarbonate. Because of insolubility in culture media and because of apparent decomposition in alkali, it has been impossible to determine whether it has bacteriological activity or not. The molecular formula $C_{19}H_{26}O_4$ has been assigned to it. Methyl, acetyl and benzoyl derivatives have been made and characterized. It has been shown to have two phenolic OH groups and to contain one ester group. Phenanthrene has been obtained from the compound by zinc dust distillation followed by sulfur dehydrogenation. A tentative partial structural

formula has been proposed.—ALLEN I. WHITE and GLENN L. JENKINS. *J. A. Ph. A.*, 31 (1942), 37. (Z. M. C.)

Sterols. CIV. Diosgenin from Certain American Plants. Diosgenin has been isolated from *Dioscorea villosa* (L.) and from *Trillium erectum* (L.), the rhizomes of the latter yielding much more diosgenin than the *Dioscorea* roots. No trillarigenin was found.—R. E. MARKER, D. L. TURNER and P. R. ULSHAFFER. *J. Am. Chem. Soc.*, 62 (1940), 2542. (E. B. S.)

Fixed Oils, Fats and Waxes

Amaranthus Retroflexus Seeds—Investigation of the Oil from. Physical and chemical constants obtained by semimicro methods are given.—B. E. CHRISTENSEN and L. S. MILLER. *J. Am. Chem. Soc.*, 63 (1941), 2272. (E. B. S.)

Buchanania Latifolia—Seed Fat of. The component fatty acids of *Buchanania latifolia* seed fat were found to be palmitic 29%, stearic 8%, oleic 57% and linoleic 6% (by weight). It contains 4.8% of fully saturated glycerides. The glyceride structure of the fat follows the usual rule of "even distribution."—N. N. GOBBOLE, B. G. GUNDE and P. D. SRIVASTAVA. *J. Indian Chem. Soc.*, 18 (1941), 557. (F. J. S.)

Fats—Antioxidants and the Autoxidation of. XIII. The Antioxygenic Action of Ascorbic Acid in Association with Tocopherols, Hydroquinones and Related Compounds. Ascorbic acid is an effective antioxidant for certain vegetable oils, their hydrogenated products and esters. It enhances the antioxygenic activity of tocopherols, hydroxy chromans, hydroquinones and related compounds.—C. GOLUMBIC and H. A. MATTILL. *J. Am. Chem. Soc.*, 63 (1941), 1279. (E. B. S.)

Martynia Diandra—Composition of Fixed Oil of Seeds of. The light petroleum-extracted oil of *M. diandra* Gl., has a density at 25° of 0.9178, index of refraction at 26° 1.4636, solidifying point -13°, acid value 4.9, saponification value 198.4, iodine value 118 (Hehner 89), acetyl value 31.4 and unsaponifiable matter 2.0%. On hydrolysis it yields palmitic 8.08, stearic 11.25, arachidic 1.34, oleic 35.84 and linoleic acid 32.37.—J. N. TAYAL and S. DUTT. *Proc. Nat. Acad. Sci. India*, 9 (1939), 78; through *J. Soc. Chem. Ind.*, 59 (1940), 804. (E. G. V.)

Paraffin Mollé—Substitutes for. Hardened vegetable oils are being used in India to replace paraffin mollé, 300 tons of which are needed for antimosquito creams.—ANON. *Indian Med. Gaz.*, 76 (1941), 486. (W. T. S.)

Paralithodes Camtschatica—Unsaponifiable Matter of Liver Oil of. In addition to cholesterol and butyl alcohol, three substances of melting points 220–225° (carbon 74.53, hydrogen 10.53%), 65° (carbon 71.14, hydrogen 11.65%), 115–118° (carbon 79.89, hydrogen 11.94%) were obtained. The liquid fraction, after acetylation, hydrogenation and saponification, was a mixture of substances of boiling point 140° (C₁₇H₃₆O₂), melting point 67–68° (C₂₅H₅₀O₂) and melting point 54° (C₂₅H₅₂O₂).—Z. NAKAMIYA. *Bull. Inst. Phys. Chem. Research Japan*, 19 (1940), 790; through *J. Soc. Chem. Ind.*, 59 (1940), 805. (E. G. V.)

Schizandra Chinensis—Fatty Oil from Kernels of. The kernels contain 33.8% of an oil having a characteristic resinous odor. It has d_{4}^{20} 0.9607, n_D^{20} 1.4901, acid value 3.81, saponification value 160.86, ester value 157.05, iodine value (Hubl) 140.74, acetyl value 24.93, Reichert-Meissl value 1.87, Polenske value 0.30, oxyacids 7.2%, unsaponifiables 5.46%. The iodine value is comparable with that of a poppy-type drying oil. The oil contains satu-

rated (3.7) and resin acids (8.7), oleic acid (28.5–34.1), α - (24.7–25.2) and β -linoleic acid (31.2–34.6%).—D. A. BALANDIN. *Compt. rend. acad. sci. U. R. S. S.*, 26 (1940), 584–586; through *J. Soc. Chem. Ind.*, 59 (1940), 545. (E. G. V.)

Vitaminic Oils—Improving the Taste and Odor of. A process of treating tuna liver oil and various other oils for removing substances which cause undesirable odor and taste, whereby the odor and taste materials are obtained as a distillate and the purified vitamin oil is obtained as a distillation residue, comprises subjecting the oil to short-path distillation at a pressure below 1 mm., distilling off from the oil a fraction which contains between 0.5% and 2% of the vitamin content of the oil, condensing this fraction and withdrawing it from the still and withdrawing the purified oil from the still as undistilled residue.—KENNETH C. D. HICKMAN and JOHN C. HECKER, assignors to DISTILLATION PRODUCTS, INC. U. S. pat. 2,249,524, July 15, 1941. (A. P.-C.)

Vitaminic Oils—Vacuum Distillation of. Apparatus is described, and a process of multi-stage distillation which comprises subjecting a liquid organic material to a high-vacuum unobstructed-path distillation while in the form of a thin film, treating undistilled liquid residue to separate mechanically and remove solids contained therein and formed by the distillation and then subjecting the treated liquid to high-vacuum unobstructed-path distillation while in the form of a thin film. Such a process is applicable to the treatment of fish oils containing fat-soluble vitamins, such as cod liver, halibut liver, menhaden, gray fish liver, pollack and other oils, petroleum and petroleum residues and animal refuse, and is also applicable to the distillation of edible oils and glyceride drying oils, such as linseed, corn, sesame, soybean and other oils, and oils or oil-like extracts containing hormones, sterols, hydrocarbons or natural antioxidants. The process is of particular use in the distillation of fish oils to obtain vitamin concentrates.—KENNETH C. D. HICKMAN, assignor to DISTILLATION PRODUCTS, INC. U. S. pat. 2,249,526, July 15, 1941. (A. P.-C.)

Unclassified

Acylated N-Arylglucamines. Products which may be used in the synthesis of riboflavin and other medicinal substances are prepared by a process which involves mixing aldehyde-tetra-acetylribose with a primary amine of the benzene series such as 4-amino-*o*-xylene or 4-carbomethoxy-amino-5-amino-*o*-xylene in methanol solution containing, as a hydrogenation catalyst, platinum, palladium or Raney nickel, and treating the mixture with hydrogen until the calculated amount of hydrogen necessary to reduce the condensation product of the primary amine and the tetra-acetylribose to the ribitylamine has been absorbed (a temperature of 20° to 50° C. being suitable for the catalytic hydrogenation).—Richard Pasternack and Ellis V. Brown, assignors to Chas. Pfizer & Co. U. S. pat. 2,250,909, July 29, 1941. (A. P.-C.)

Alkylcrotylbarbituric Acids and Their Salts. Various compounds are produced of the general formula $R(\text{CH}_2\text{CH} : \text{CHCH}_2)\text{C} \cdot \text{CO} \cdot \text{NH} \cdot \text{CO} \cdot \text{N} \cdot \text{X} \cdot \text{CO}$

in which *R* represents an alkyl group which contains from 2 to 6 carbon atoms and which has no branching of the carbon chain except when there is in the group one straight chain of at least 2 carbon atoms extending from the carbon atoms at which the branching occurs; and *X* represents hydrogen, an alkali metal, an equivalent of an alkaline earth metal, ammonium, monoalkylammonium, dialkylammonium, and alkanolammonium. Their sodium salts are soluble in water and alcohol and serve as

hypnotics when administered either orally or hypodermically.—HORACE A. SHONLE and WILBUR J. DORAN, assignors to ELI LILLY & CO. U. S. pat. 2,250,422 to 2,250,424, July 22, 1941. (A. P.-C.)

Amino Acid Amidines. Compounds which may be used as therapeutic agents are produced by a process which involves treating a reactive ester of a hydroxyalkylimidazolone, obtained by treating the latter with a strong organic or inorganic compound, with an amine. Details are given, or general mention is made, of the production of a large number of such compounds.—KARL MIESCHER, WILLIE KLARER and ERNST URECH, assignors to CIBA PHARMACEUTICAL PRODUCTS. U. S. pat. 2,252,722, Aug. 19, 1941. (A. P.-C.)

4-Aminobenzenephosphonic Acid (Phosphanilic Acid)—Preparation of. The preparation of 4-aminobenzenephosphonic acid or phosphanilic acid has been performed by heating 4-chlorobenzenephosphonic acid with concentrated ammonium hydroxide to 150°, using cuprous chloride as a catalyst. An acetyl derivative has been prepared.—HUGO BAUR. *J. Am. Chem. Soc.*, 63 (1941), 2137. (E. B. S.)

***p*-(*p*-Aminophenyl)-benzenesulfonamide and Derivatives. II.** *p*-(*p*-Acetamidophenyl)-benzenesulfonfyl chloride has been coupled with the following amino compounds and the resulting compounds deacetylated: aniline, benzylamine, cyclohexylamine, *p*-xenylamine, sulfanilamide and *p*-(*p*-aminophenyl)-benzenesulfonamide. The new compounds have been analyzed and reported.—C. T. VAN METER and A. LOWY. *J. Am. Chem. Soc.*, 63 (1941), 1330. (E. B. S.)

5-Androstene-3,16,17-Triol—Synthesis of. Isotriodehydroandrosterone has been converted to 5-androstene-3,16,17-triol by a new method which makes it possible to obtain 16-hydroxytestosterone in yields ten times that previously reported.—FRANK H. STODOLA, EDWARD C. KENDALL and BERNARD F. MCKENZIE. *J. Org. Chem.*, 6 (1941), 841-844. (W. T. S.)

Antimalarial Products of the 8-Aminoalkylamino-6-methoxyquinoline Series—Studies on the Synthesis of. A number of compounds of this series was prepared by a modification of the Gabriel reaction for the formation of amines, the derivatives prepared containing 1, 2, 3, 4 and 5 carbon atoms in the alkyl side chain. The aminomethylamino derivative was found to be inactive; all the other derivatives possessed a high therapeutic activity, approaching in certain cases that of plasmoquin and of plasmocid. Of the new derivatives, the amino-propylamino was the most active.—A. A. BEER. *J. Obchtch. Khim.*, 9 (1939), 2158-2161; through *Chimie & Industrie*, 44 (1940), 52. (A. P.-C.)

Azine Dyes Derived from 9:10-Phenanthrothiophene-2':3'-Dione.—PARESH CHANDRA DUTTA and RAMANI MOHAN SINHA. *J. Indian Chem. Soc.*, 18 (1941), 477. (F. J. S.)

1-Benzylidenecoumaran-2-ones—Some New Reactions of. I. Examination of the reactions of 1-*p*-anisylidene-coumaran-2-one indicates that the reactions characteristic of the keto-ethylene group exhibited by chalcones are unaffected by the cyclic linkage. Thus one of the bromine atoms in its dibromide, like the halogen atom in the chalcone dibromides, is readily replaced by alkoxyl on treatment with methyl or ethyl alcohol. It also undergoes, like chalcones, Michael additions with cyclohexanone and desoxybenzoin and condenses with ethyl acetoacetate.—T. B. PANSE, R. C. SHAH and T. S. WHEELER. *J. Indian Chem. Soc.*, 18 (1941), 453. (F. J. S.)

Cannabidiol—Structure of. VII. A Method of Synthesis of a Tetrahydrocannabinol Which Pos-

sesses Marihuana Activity. The compound prepared by Desai by condensing ethyl cyclohexanone-2-carboxylate with orcinol has been proved to be 1-hydroxy-3-methyl-7,8,9,10-tetrahydro-6-benzopyrone, which with methyl magnesium iodide gives the corresponding pyran. By using ethyl 5-methylcyclohexanone-2-carboxylate, the compound 1-hydroxy-3,6,6,9-tetramethyl-7,8,9,10-tetrahydro-6-dibenzopyran is produced. By condensing ethyl 5-methylcyclohexanone-2-carboxylate and olivetol followed by methylmagnesium iodide, a synthetic tetrahydrocannabinol was formed which had marihuana activity.—ROGER ADAMS and B. R. BAKER. *J. Am. Chem. Soc.*, 62 (1940), 2405. (E. B. S.)

Carcinogenic Hydrocarbons. IV. The Bromination of Hydrindene and a Briefer Synthesis of Cholanthrene. The bromination of hydrindene yields a mixture composed of one-third α and two-thirds β -bromohydrindene when bromine is added to a solution of the hydrocarbon in acetic acid. This mixture has been used to prepare cholanthrene with a yield of 24%, based on the α -isomer. A preliminary experiment shows that addition of hydrocarbon to a solution of bromine gives a much larger proportion of α -isomer than the procedure previously used.—W. F. BRUCE. *J. Am. Chem. Soc.* 63 (1941), 301. (E. B. S.)

Catechol—Antimonial and Thioantimonial Derivatives of. Soluble derivatives of antimonial catechol and antimonial dithiocatechol have been prepared. The yields are reported excellent. Dithiocatechol is more reactive than catechol toward antimony halides and oxides, tending to react with all three rather than only two of the antimony valences. This is in accord with the reactivity of dithiocatechol toward tin and other metals. Monothiocatechol appears to be intermediate in reactivity between catechol and dithiocatechol toward these antimony compounds.—H. P. BROWN and J. A. AUSTIN. *J. Am. Chem. Soc.*, 63 (1941), 2054. (E. B. S.)

Chloral Hydrate—Action of, on Aliphatic Orthoesters. A series of lower alkyl formates were refluxed with chloral hydrate to yield the corresponding hemiacetal with the alcohol and the formate ester as by-products. Ethyl orthothioformate also reacted under the same conditions, and ethyl orthosilicate reacted with the formation of gels. The orthoester did not react with free chloral even in presence of sulfuric acid. Interaction of chloral hydrate and the lower aliphatic alcohols at reflux temperatures gave yields of the corresponding hemiacetals ranging from 28% to 100%. Ethyl mercaptan gave an 8.1% yield. Ethyl and propyl alcohols refluxed with chloral for 2 hrs. gave a 71.3% and a 90% yield, respectively, of the expected hemiacetal. Only certain of the hemiacetals of chloral hydrate undergo a radical interchange with other alcohols depending on the comparative weight of the alcohol employed and the ethereal section of the hemiacetal. A similar radical interchange takes place between the hemiacetals of chloral hydrate and the orthoesters giving yields of the new hemiacetals ranging from 16% to 64%. These reactions do not make for a postulation as to the mechanism of the reaction nor does it prove the dihydroxy structure of chloral hydrate since the water from chloral hydrate could hydrolyze the orthoester to give the alcohol which would then combine with chloral to give the hemiacetal. The hemiacetals are quite stable at their boiling temperatures, and their hydroxyl group is resistant to metathetical reactions with alcohols, ethers and polyethers.—HOWARD W. POSR. *J. Org. Chem.*, 6 (1941), 830-835. (W. T. S.)

7-Cholanthroic Acid. 1-Acetoacnaphthene was

converted to 1-acenaphthylacetic acid *via* 1-acenaphthoic acid by the Arndt-Eistert reaction and, more conveniently, by the Willgerdt process of heating the ketone with yellow ammonium sulfide. The acetic acid was condensed successfully with both *o*-chloro- and *o*-nitrobenzaldehyde, but only the second of the products could be utilized for effecting a phenanthrene ring closure. Although the Pschorr reaction proceeded very poorly, the 7-carboxy derivative of cholanthrene was isolated through the ester in quantity sufficient for an investigation of its possible biological actions. The hydrogen fluoride acylation of acenaphthene with phenylacetic acid and *o*-bromophenylacetic acid does not provide a practical route to intermediates suitable for the synthesis of cholanthrenes.—L. F. FIESER and G. W. KILMER. *J. Am. Chem. Soc.*, 62 (1940), 1354-1360. (E. B. S.)

Cholestenone—Stereoisomeric Oximes of. Two forms of cholestenoneoxime exist. The one, "A," which is relatively more labile, melts at 65°, solidifies again at about 85°, and remelts at 151.8° C. The other, "B," is the ordinary stable form which melts at 151.8° C. The ordinary form can be converted into the less stable form by treatment with glacial acetic acid, propionic or *n*-butyric acid. Either form can exist, without apparent change, in formic acid. The less stable form reverts to the more stable oxime in benzene or carbon tetrachloride, or upon being heated to a temperature above its melting point.—J. O. RALLS. *J. Am. Chem. Soc.*, 62 (1940), 2459. (E. B. S.)

Cyclic Amidines. Compounds, various of which may be used as therapeutic agents for blood pressure regulation, have the general formula $AC(:N-)(R)-$, in which *A* is an aminoalkyl radical containing an aromatic or heterocyclic radical as a substituent at the nitrogen atom and *R* is hydrogen or a hydrocarbon radical, both amidine nitrogen atoms being united together by an alkylene chain as, for instance, in imidazoline or tetrahydropyrimidine rings. They are formed by causing an amino acid having an aromatic or heterocyclic radical as a substituent at the nitrogen atom, or a derivative, to react with an aliphatic diamine. As parent materials, there may be used, besides the acids themselves, derivatives thereof, for instance the imido ethers obtainable from the nitriles, the amidines containing open chains, the imido halides corresponding with the amides or the thio amides. These compounds may be caused by the process to react with aliphatic diamines such as 1,2- or 1,3-diamines to form cyclic amidines. A hydrogen atom of one of the two amino groups of the diamines may also be monosubstituted by a hydrocarbon radical. Details are given, or general mention is made of the production of a number of such compounds.—KARL MIESCHER, ERNST URECH and WILLIE KLARER, assignors to CIBA PHARMACEUTICAL PRODUCTS. U. S. pat. 2,252,721, Aug. 19, 1941. (A. P.-C.)

4,4'-Diaminodiphenyl Sulfones—Substituted. Compounds suitable for therapeutic use in treating streptococcal and pneumococcal infections by oral, parenteral or intravenous administration comprise substituted 4,4'-diaminodiphenyl sulfones in which a hydrogen atom of at least one of the two amino groups is substituted by a radical of the general formula: $-R-S-R'-R_1Ac-X$, in which *R* represents a member of the general class of short-chain alkylidene and alkenylidene groups having not over 6 carbon atoms; *R'* represents a bivalent radical of the class consisting of monocarbon and polycarbon hydrocarbon groups and those substituted polycarbon hydrocarbon groups in which a hydrogen atom on a carbon atom not attached to sulfur is replaced by a hydroxyl or acylated hydroxyl or acylated amino group; *R₁Ac* represents a carboxy or sulfo

group; and *X* represents a member of the class of positive ions consisting of hydrogen, the alkali metals, the alkaline earth metal and the nitrogen hydronium ions derived from ammonia, the alkylamines, the alkanolamines and the polymethylenediamines. Numerous examples with details of manufacture are given.—MORRIS S. KHARASCH and OTTO REINMUTH, assignors to ELI LILLY AND CO. U. S. pat. 2,224,156, Dec. 10, 1940. (A. P.-C.)

Diethylstilbestrol, etc. A method of preparing a compound of the general formula $HO-C_6H_4-C$ (lower alkyl)=C (lower alkyl)- C_6H_4-OH comprises reacting a compound of the general formula (lower alkyl)- $O-C_6H_4-CH_2-CO-C_6H_4-O$ (lower alkyl) with an alkali metal hydroxide and a lower-alkyl halide, reacting the resulting monoalkylated compound with a lower-alkyl magnesium halide and hydrolyzing the reaction product, reacting the resulting dialkylated carbinol with a phosphorus trihalide, reacting the resulting dehydration product with methyl magnesium iodide, phenyl magnesium bromide or ethyl magnesium iodide, and hydrolyzing the reaction product. Details are given of the production of various compounds such as diethylstilbestrol, α -ethyldeoxyvanisoin, and α,α' -diethylstilbene.—WM. BRAKER, MORRIS A. DOLLIVER, EDWARD PRIBYL and FRANKLIN A. SMITH, assignors to E. R. SQUIBB & SONS. U. S. pat. 2,252,696, Aug. 19, 1941. (A. P.-C.)

22-Dihydrostigmasterol and Cholesterol—Dehydration of, with Iodine. Dehydration by iodine of cholesterol and 22-dihydrostigmasterol does not result in unsaturated hydrocarbons but yields instead dicholesterol ether and di-22-dihydrostigmasterol ether and as by-products unidentified bodies. The result thus confirms the theory by Hibbert in 1915, regarding the use of iodine as a dehydrating catalyst.—T. HASSELSTROM and B. L. HAMPTON. *J. Am. Chem. Soc.*, 63 (1941), 111. (E. B. S.)

Enol Derivatives of Cyclopentanophenanthrene Compounds. Therapeutic and intermediate compounds are obtained by heating a 3-keto-10,13-dimethylcyclopentanophenanthrene compound with an acylating agent at a temperature of at least about 100° C. for a sufficient time to form an enol derivative which is acylated at the enolic hydroxyl group. Numerous examples with details are given.—LEOPOLD RUZICKA and WERNER FISCHER, assignors to CIBA PHARMACEUTICAL PRODUCTS, INC. U. S. pat. 2,248,438, July 8, 1941. (A. P.-C.)

Estrone Azobenzene-4-Carboxylate. The publication by Coffman (*J. Biol. Chem.*, 140 (1941), XXVIII) on the preparation and chromatographic analysis of benzeneazo-*p*-benzoyl derivatives of certain sex hormones prompted the writers to publish results from similar experiments with the estrogenic hormones. Estrone azobenzene-4-carboxylate was prepared from the hormone and azobenzene-4-carboxylic acid chloride and then the estrone regenerated by hydrolysis with alcoholic KOH. Some experiments designed to chromatographically analyze the "azo-ester" of certain estrone samples are described. Regeneration of all estrone samples from the "azo-ester" gave products of higher melting points.—F. BERGEL and A. COHEN. *J. Chem. Soc.* (1941), 795-796. (W. T. S.)

Fatty Acids—Halogenation of. III. Reaction between Alkyl Halides with Phenols. Formation of Long Chain Alkyl Ethers. Long chain mono- and dialkyl ethers of β -naphthol, hydroquinone, resorcinol and pyrocatechol with alkyl halides of higher fatty acids have been prepared. The introduction of long chain ether groups to the phenols imparts properties which render them useful for many industrial purposes.—T. N. MEHTA, V. S. MEHTA and V. B. THOSAR. *Ind. & News Ed. J. Indian Chem. Soc.*, 4 No. 3 (1941), 170. (F. J. S.)

Gluconic Acid Production. In the semicontinuous method of producing gluconic acid from glucose by *A. niger*, the mycelia were recovered by pressure filtration and re-used in nine successive fermentations of media containing 16 Gm. of glucose per 100 cc. In the fermentation of a solution containing 16 Gm. of glucose and about 2.6 Gm. of calcium carbonate per 100 cc., the uncombined gluconic acid is present in sufficient concentration to prevent the formation of calcium gluconate crystals which tend to mat with the mycelial growth and thus hinder filtration. This filtration permits the utilization of a full charge of fresh medium of higher glucose concentration than was feasible with the flotation method previously reported. In the cases where the free acid concentration was low, centrifugal filtration was the more efficient method of removing the mycelia from the fermented liquor.—N. PORGES, T. F. CLARK and S. I. ARONOVSKY. *Ind. Eng. Chem.*, 33 (1941), 1065–1067. (E. G. V.)

N⁴-d-Glucosidosulfanilamide—Structure of. Sugar derivatives of sulfanilamides are generally considered to be glycosides, but the anil structure has also been claimed for them (Gray, *et al.*, *Biochem. J.*, 31 (1937), 724). The writers synthesized N⁴-tetra-acetyl-d-glucosidosulfanilamide (I) from β -acetobromo-d-glucose and sulfanilamide. Upon deacetylation of I the N⁴-d-glucosidosulfanilamide obtained was identical with the sulfanilamide-d-glucoside of Kuhn and Birkofer (*Ber.*, 71 (1938), 621) obtained from glucose and sulfanilamide. Since I failed to yield a picrate, a picramide, a substituted thiourea, and was negative with Ehrlich's reagent it was concluded that the glucoside is on the N⁴-nitrogen. Synthesis of I from β -acetobromo-d-glucose indicates its β -linkage. The toxicity of I is less than sulfanilamide, but so is its action. The solubility of I in water is appreciable.—CHARLES E. BRAUN, JACK L. TOWLE and S. H. NICHOLS. *J. Org. Chem.*, 7 (1942), 19–22. (W. T. S.)

Glycerol—Purification of, by Crystallization. Synthetic glycerol, produced by hydrogenolysis processes, is highly impure and the isolation of C.P. or even U. S. P. grades has heretofore proved extremely difficult. A new approach to the problem—namely, crystallization in the presence of suitable solvents—readily yields glycerol of any degree of purity required. As a result of this process it is now possible to produce by a simple one-step treatment with hydrogen any desired quantity of high-quality glycerol from such abundant carbohydrate materials as starch and dextrose.—H. B. HASS and J. A. PATTERSON. *Ind. Eng. Chem.*, 33 (1941), 615–616. (E. G. V.)

o-Hydroxy Aldehydes from Phenols and Hexamethylenetetramine. By heating phenols with hexamethylenetetramine in anhydrous glycerine and glycerylboric acid, the writer has improved over the Reimer-Tiemann method for preparing phenolic aldehydes. Of the sixteen phenolic aldehydes prepared, three (3:5-dialdehyde-o-cresol, 6-chloro-2:4-dialdehyde-m-cresol and 2-chloro-4-aldehyde-m-xylene) are new. The o-hydroxy aldehydes were steam distilled from the reaction mixture in the presence of sulfuric acid. A possible mechanism of the reaction is given.—JAMES C. DUFF. *J. Chem. Soc.* (1941), 547–550. (W. T. S.)

Hydroxystilbene Series—Esters of the. By treating a hydroxy compound of the stilbene series containing at least one hydrocarbon substituent at the ethylene bridge with an inorganic esterifying agent such as a sulfating or phosphating agent, esters are formed which have an effect in the test for estrus and uterus growth and may be used in therapeutics. The initial stilbene derivatives can be prepared as described by E. C. Dodds, *et al.* (*Nature*, 141 (1938), 247–248). Suitable esterifying agents are

inorganic acids, their halides, anhydrides or esters, such as phosphorus oxytrichloride, phosphorus pentachloride, phosphorus pentoxide, metaphosphoric acid, chlorosulfonic acid, toluenesulfonic acid. Known methods of esterification may be used and condensation agents such as, for instance, pyridine, quinoline or the like. The Schotten-Baumann method also can be used. For producing the monoesters, the diesters may be partially saponified; or the monoesters can be obtained by partial esterification. Also mixed diesters may be made by methods in themselves known. Details of the production of some of these esters are given.—KARL MIESCHER and JULES HERR, assignors to CIBA PHARMACEUTICAL PRODUCTS, INC. U. S. pat. 2,234,311, March 11, 1941. (A. P. C.)

16-Ketotestosterone—Partial Synthesis of. In the first of a series of papers dealing with nuclear hydroxylated steroids, the authors describe a method for preparing 16-ketotestosterone acetate starting with androstene-3,17-dione-3-enol ethyl ether. Physiological studies of 16-ketotestosterone acetate indicate that this compound is not responsible for the action of the amorphous fraction of adrenal cortex extract on metabolism of certain inorganic electrolytes.—FRANK H. STODOLA and EDWARD C. KENDALL. *J. Org. Chem.*, 6 (1941), 837–840. (W. T. S.)

Long-Chain Acids—Studies in. III. Bis-Nor-Oleic Acid. Δ^9 -Hexadecenoic acid (bis-nor-oleic acid) has been synthesized from oleic acid by subjecting it to a two-stage Wieland degradation.—P. C. MITTER and PHANINDRA NATH BAGCHI. *J. Indian Chem. Soc.*, 18 (1941), 461. (F. J. S.)

α -Methoxymercurials from Ethylenes—Catalysis in the Formation of. Three concomitant reactions occurring during addition of the methoxymercuric acetate elements to ethylenes have been observed, two of which have been identified as oxidation to the dimethoxyethane and as dismutation to the diorganomercury derivative. Nitriles, pyridine, diethylsulfide and an unidentified impurity in mercuric acetate have been found to retard the addition reaction. The reaction is accelerated by peroxides, and by boron trifluoride etherate.—A. M. BIRKS and G. F. WRIGHT. *J. Am. Chem. Soc.*, 62 (1940), 2412. (E. B. S.)

9-Methyl- and 10-Methyl-1,2-Benzanthracene. Using a previously described method for the synthesis of *meso* substituted anthracene hydrocarbons, two carcinogenic compounds, 9-methyl- and 10-methyl-1,2-benzanthracene have been prepared.—C. K. BRADSHAW. *J. Am. Chem. Soc.*, 62 (1940), 1077–1078. (E. B. S.)

2-Methyl-3,4-Benzphenanthrene—Synthesis of. The preparation of 1,2,3,4-tetrahydro-4-keto-1-phenyl-2-naphthaleneacetic acid by ring closure of β -benzohydroxyglutaric anhydride using aluminum chloride or by direct cyclization of β -benzohydroxyglutaric acid using anhydrous hydrogen fluoride is described. The keto group of this keto acid was reduced by the Clemmensen method and the reduced acid cyclized to 1,2,9,10,11,12-hexahydro-2-keto-3,4-benzphenanthrene. By reaction with the appropriate Grignard reagents followed by dehydration and dehydrogenation 2-methyl-3,4-benzphenanthrene and 2-ethyl-3,4-benzphenanthrene were prepared from the above hexahydroketoone.—M. S. NEWMAN and L. M. JOSHEL. *J. Am. Chem. Soc.*, 62 (1940), 972–974. (E. B. S.)

5-Methylchrysenes. Chrysenes-5-carboxylic acid, obtained by the Pschorr synthesis, has been converted through the aldehyde (Sonn-Müller reaction) to the 5-methyl compound. In attempts to reduce the ester with sodium and ethanol or by high pressure hydrogenation, the aromatic nucleus was in-

variably attacked.—L. F. FIESER and L. M. JOSHEL. *J. Am. Chem. Soc.*, 62 (1940), 1211-1214.

(E. B. S.)

5-Methylchrysene and Related Compounds—Synthesis of. The synthesis of 5-methylchrysene, 5-ethylchrysene, and 5,6-dimethylchrysene is described, together with a new and more generally applicable synthesis for α,γ -diphenylbutyric acid. The recovery of a considerable amount of 1,2,3,4-tetrahydro-1-keto-2-phenylnaphthalene from the Reformatsky reaction between this ketone and ethyl α -bromobutyrate despite a large excess of reagents is noted. An explanation is offered involving reaction of the enol form of the ketone with an organozinc intermediate to produce a bromozinc enolate and ethyl butyrate. This explanation is supported by the isolation of ethyl butyrate from the reaction mixture.—M. S. NEWMAN. *J. Am. Chem. Soc.*, 62 (1940), 870-874.

(E. B. S.)

4,5-Methylenchrysene and 1',9-Methylene-1,2-Benzanthracene from 4,5-Methylenephenanthrene—Synthesis of. By the Friedel and Crafts condensation of 4,5-methylenephenanthrene with succinic anhydride the 1-keto acid is obtained in 45% yield and affords a suitable intermediate for the synthesis of the new 4,5-methylenchrysene. 9,10-Dihydro-4,5-methylenephenanthrene undergoes succinoylation exclusively in the 2-position, and after reduction ring closure can be effected at the 3-position, providing in moderate yield a ketone which can be converted into the known 1',9-methylene-1,2-benzanthracene. γ -(4,5-Methylene-2-phenanthryl)-butyric acid is obtained in good yield by dehydrogenating the dihydro intermediate as the ester and it cyclizes exclusively at position 1, affording a very satisfactory route to 4,5-methylenchrysene in which the overall yield from 4,5-methylenephenanthrene is 30%. The structures of the different products are fully established by the transformations involved. In all of the inter- and intra-molecular acylations observed in the 4,5-methylenephenanthrene series a strong *para* directive influence of the 4-methylene group is discerned.—L. F. FIESER and J. CASON. *J. Am. Chem. Soc.*, 62 (1940), 1293-1298.

(E. B. S.)

Monocrotaline—Structure of. V. Retronecine, a Derivative of 1-Methylpyrrolizidine. A critical discussion is given of the experiments of Menshikov from which he deduced that heliotridane was 1-methylpyrrolizidine. The synthesis of 1,3-dimethyl-2-*n*-propyl-pyrrolizidine was completed. The product was shown to be identical with *dl*-dihydro-des-*N*-methylheliotridane, by comparison of the picrates, picrolonates and methiodides. The structure of heliotridane is thus definitely established as 1-methylpyrrolizidine. Heliotridane was prepared by two methods with identical results.—ROGER ADAMS and E. F. ROGERS. *J. Am. Chem. Soc.*, 63 (1941), 228.

(E. B. S.)

Monocrotaline—Structure of. VI. The Structure of Retronecine, Platynecine and Retronecanol. By catalytic reduction under specified conditions, retronecine is converted into retronecanol or platynecine, and monocrotaline is converted to retronecanol or desoxyretronecine. Desoxyretronecine was shown to be a molecule in which merely the easily removed (β) hydroxyl of retronecine was replaced by hydrogen. Platynecine was formed by reduction of the double bond in retronecine without elimination of either hydroxyl. This establishes the relationship of platynecine and retronecine and indicates that the hydroxyls are in comparable positions. On the basis (1) of the stability of the hydroxyls in platynecine to reduction and the failure of monobenzoyl platynecine to hydrogenolyze in contrast to the ease of reduction of one of the hydroxyls in retronecine and the ease of hydrogenoly-

sis of the monesters of retronecine, and (2) of the difference in the ease of esterification of the two hydroxyls in retronecine or platynecine, structures have been deduced for retronecine, desoxyretronecine, platynecine and anhydroplatynecine, which agree with all the available experimental facts.—ROGER ADAMS and E. F. ROGERS. *J. Am. Chem. Soc.*, 63 (1941), 537.

(E. B. S.)

Nitrosation of Phenols. XVII. *o*-Fluorophenol, and a Comparative Study of the Four *o*-Halogenophenols. Nitrous acid reacts with *o*-fluorophenol to form a little 2-fluoro-6-nitrophenol, but mainly 2-fluoro-4-nitrosophenol, of which the quinoneoxime modification, like those of the other 2-halogenoanalogues, only exists in derivatives and not in the free state. In its properties and derivatives it resembles the other 2-halogeno-4-nitrosophenols now described. Further examples of the rule that nitroso-compounds melt at a lower temperature than the isomeric quinoneoximes are given.—HERBERT H. HODGSON and DONALD E. NICHOLSON. *J. Chem. Soc.*, (1940), 810-812.

(W. T. S.)

Organometallic Compounds—Coördinate Compounds in the Color Test for. A 1:1 mixture of Michler's ketone and phenylmagnesium bromide in ether forms a coördination compound, which on hydrolysis gives an 88% recovery of ketone. However, the coördination compound is sufficiently unstable so that there is no interference with the color test for organometallic compounds. The stability of the complex is markedly decreased in an ether-benzene mixture, and in addition to a 45% recovery of ketone there was isolated a 42% yield of *p,p'*-tetramethyldiaminotriphenylcarbinol. A 1:1 mixture of Michler ketone and phenyl-lithium, in either benzene or ether, forms no stable complex, and the only product isolated in excellent yields is the carbinol, which is an intermediate in the color test. Other aspects of coördination compounds in organometallic chemistry are considered.—H. GILMAN and R. G. JONES. *J. Am. Chem. Soc.*, 62 (1940), 1243-1247.

(E. B. S.)

Oxymethylene Methyl Ethyl Ketone and Oxymethylene Methyl- β -Phenyl Ethyl Ketone—Structure of. By the reaction of self-condensation and by condensation with cyanoacetamide it is shown that oxymethylene methyl ethyl ketone is $\text{CHO}=\text{C}(\text{CH}_3)-\text{CO}-\text{CH}_2$ and oxymethylene methyl- β -phenyl ethyl ketone is $\text{C}_6\text{H}_5-\text{CH}_2-\text{CH}_2-\text{CO}-\text{CH}=\text{CHOH}$. The points of attack of formic ester on the ketone $\text{R}-\text{CH}_2-\text{CO}-\text{CH}_3$ are discussed.—S. N. JOSHI, R. KAUSHAL and S. S. DESHPANDE. *J. Indian Chem. Soc.*, 18 (1941), 479.

(F. J. S.)

Phenols—Bromination of, by Means of Bromide-Bromate Solution. Phenol itself and phenols which contain substituents in the *meta* position react with acid bromide-bromate solution, causing quantitative substitution at the *ortho* and *para* positions. Certain phenols which contain secondary or tertiary alkyl groups in the *ortho* or *para* position also brominate quantitatively in this manner. Under the same conditions, phenols which contain primary alkyl groups in the *ortho* or *para* positions give results which may be 10% to 50% high, depending on the nature of the substituents. Dihydroxydiphenylmethane also requires somewhat more than the theoretical quantity of bromine solution. Fresh samples of saligenin require almost exactly three moles of bromine. The methylol group behaves as though it were not present. *p,p'*-Dihydroxybenzophenone alone, of the phenols examined reacted with less than (82% of) the calculated quantity of bromination solution. Certain phenols having primary alkyl substituents in the *para* position can be estimated accurately by bromination at low temperature. The conditions required for quantitative* bromination must be worked out for

each individual alkylated phenol. Interpretations of the structure and molecular weight of phenolic resins, or related substances, based upon bromination procedure, should be critically reexamined.—M. M. SPRUNG. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 35-38. (E. G. V.)

5-Phenylhydantoin—Preparation of Certain 5-Acetates and 5-Acetamides of. The synthesis has been reported of 5-phenyl-hydantoinacetic acid and sixteen of its derivatives. The latter include examples of esters, amides and nitriles. The compounds are intended to be tested for hypnotic and anticonvulsant activity.—B. G. ROGERS and H. R. HENZE. *J. Am. Chem. Soc.*, 63 (1941), 2190. (E. B. S.)

N¹-Phenylsulfanilamide—Derivatives of. II. The preparation of a number of disubstituted derivatives of N¹-phenylsulfanilamide and N⁴-acetyl-N¹-phenylsulfanilamide in which two hydrogen atoms of the phenyl group are replaced by nitro and amino, nitro and hydroxy, amino and hydroxy, nitro and acetoxy, hydroxy and acetyl amino, acetoxy and acetyl amino, two acetyl amino or two amino groups is described. Eight disubstituted phenyl-4-acetyl amino (or amino)-benzenesulfonates are described.—G. L. WEBSTER and S. D. GERSHON. *J. Am. Chem. Soc.*, 63 (1941), 1927. (E. B. S.)

Polyhydrocyclopentanophenanthrene Series—Tertiary Alcohols of the. A method employed for producing tertiary alcohols of the polyhydrocyclopentanophenanthrene series in which the tertiary carbon is situated in the nucleus, involves causing acetylene or a substituted acetylene capable of combining with keto groups to react on compounds of the polyhydrocyclopentanophenanthrene series containing a keto group in one of the 3 and 17 positions and then hydrogenating the ethynyl compounds so formed. Starting materials which may be used include: the follicle hormones estrone, equilin and equilenin, the male sex hormones androsterone, dehydroandrosterone, their isomers and other cyclopentanophenanthrene compounds containing a keto group. Several examples with details are given, and products are obtained which either themselves possess a high physiological activity or may be used as intermediates in the manufacture of physiologically active compounds.—JOSEF KATHOL, assignor to SCHERING CORP. U. S. pat. 2,251,939, Aug. 12, 1941. (A. P.-C.)

Polyhydrocyclopentanophenanthrene Series and Their Derivatives—Acids of the. A method of producing acids of the polyhydrocyclopentanophenanthrene series and their derivatives involves treating a 13-methylpolyhydrocyclopentanophenanthrene having a keto group in one of the 3- and 17-positions with a halogen-substituted lower aliphatic monocarboxylic acid ester in the presence of a metal capable of taking part in a Reformatskii type of reaction, such as zinc or magnesium. Details are given of a number of such reactions giving products suitable as therapeutic agents.—HANS-GEORG ALLARDT and LOTHAR STRASSBERGER, assignors to SCHERING CORP. U. S. pat. 2,254,407, Sept. 2, 1941. (A. P.-C.)

Pyrazine Series—Synthesis in. III. Amination of 2,5-Dimethylpyrazine. The Synthesis of 3-Sulfanilamido-2,5-dimethylpyrazine. 3-Amino-2,5-dimethylpyrazine was prepared in yields of 35% with the aid of sodamide in dimethylaniline as a solvent at 165°. 3-Sulfanilamido-2,5-dimethylpyrazine was prepared.—R. R. JOINER and P. E. SPOERRI. *J. Am. Chem. Soc.*, 63 (1941), 1929. (E. B. S.)

Retene Field—Investigations in the. XII. The Synthesis of 10-Phenanthr-(2,3-β)-azepine Derivatives by the Beckmann Rearrangement of a Tetrahydrobenzoretene Ketoxime. 3-Isopropyl-7-methyl-8-keto-8,9,10,11-tetrahydrobenz(α)an-

thracene ketoxime suffers a normal Beckmann rearrangement under the influence of 50% sulfuric acid, with formation of a 10-phenanthr(2,3-β)azepine, the first representative of a new tetracyclic system. With phosphorus pentachloride, the same ketoxime underwent a Beckmann rearrangement accompanied by the addition of two molecules of hydrogen chloride to the azepine produced. The structure of the azepine was proved by its hydrolysis to an aminoretylbutanoic acid. The nitrogen of the azepine cycle is therefore an immediate union with the phenanthrene nucleus.—S. A. CASSADAY and M. T. BOGERT. *J. Am. Chem. Soc.*, 63 (1941), 1452. (E. B. S.)

Skraup's Synthesis of Quinoline—An Improved Modification of. By substituting acetylated amines for the free bases, the authors have prepared better yields of pure quinoline by the Skraup synthesis with a less violent reaction and fewer tarry by-products.—RICHARD F. H. MANSKE, FRANK LEGER and GEO. GALLAGHER. *Can. J. Research B*, 19 (1941), 318-319. (W. T. S.)

Soaps—Invert. Quaternary Ammonium Salts of Derivatives of Long Chain Phenols. Diisobutylphenol was methylated, nitrated, reduced to the amine and the quaternary ammonium salt made. Salts prepared were: 2-trimethylammonium-4-diisobutylphenol methyl ether iodide and methosulfate.—J. B. NIEDERL and M. I. DEXTER. *J. Am. Chem. Soc.*, 63 (1941), 1475. (E. B. S.)

Soaps—Invert. Quaternary Morpholinium Salts. N-Ethyl- and N-ethylmorpholine were converted into N-long chain substituted morpholinium salts upon treatment with lauryl, myristyl and cetyl bromide, respectively. All were water-soluble.—M. E. MCGREAL and J. B. NIEDERL. *J. Am. Chem. Soc.*, 63 (1941), 1476. (E. B. S.)

Soaps—Phenolic Invert. p-α,α,γ,γ-Tetramethoxybutylphenol was condensed with formaldehyde and a series of secondary amines. The resulting tertiary amines were then converted into the respective phenolic invert soaps upon treatment with methyl iodide.—J. B. NIEDERL and F. A. ABRUSCATO. *J. Am. Chem. Soc.*, 63 (1941), 2024. (E. B. S.)

Sterols. CII. Chlorogenin. Evidence is presented which indicates that the hydroxyl groups of chlorogenin are 3-beta and 6-alpha.—R. E. MARKER, E. M. JONES and D. L. TURNER. *J. Am. Chem. Soc.*, 62 (1940), 2537. (E. B. S.)

Sterols. CXI. Sapogenins. XL. The Conversion of Chlorogenin to Tigogenin. Chlorogenin has been converted to tigogenin, by means of the sodium ethylate reaction which converted the dimethylcarbazone of chlorogenone to tigogenin.—R. E. MARKER, D. L. TURNER and P. R. ULSHAFFER. *J. Am. Chem. Soc.*, 62 (1940), 3009. (E. B. S.)

Sterols. CIX. Sapogenins. XXXVIII. The Preparation of Dihydro-isoandrosterone from Diosgenin. Dihydro-isoandrosterone and related compounds were made from tigogenin prepared from diosgenin; details are given.—R. E. MARKER and D. L. TURNER. *J. Am. Chem. Soc.*, 62 (1940), 3003. (R. B. S.)

Sterols. CXV. Sapogenins. XLIV. The Relation between Diosgenin and Cholesterol. Diosgenin has been converted to cholesterol. Various reactions of cholesterol have been extended to diosgenin.—R. E. MARKER and D. L. TURNER. *J. Am. Chem. Soc.*, 63 (1941), 767. (E. B. S.)

Sterols. CXVIII. The Action of Selenious Acid on Δ⁶-Pregnenediol and on Δ⁴-Androstenediol. Oxidation of Δ⁶-pregnenediol with selenious acid, followed by dehydration with hydrochloric acid and oxidation of the resulting product, gave progesterone. Oxidation of Δ⁴-androstenediol with selenious acid, followed by dehydration with hydrochloric

acid gave testosterone.—R. E. MARKER, H. M. CROOKS, JR., and E. L. WITTEBECKER. *J. Am. Chem. Soc.*, 63 (1941), 777. (E. B. S.)

Sterols. CIII. The Oxidation of Pregnanetriols. The oxidation of pregnanetriol-3,16,20 and allo-pregnanetriol-3,16,20 by the method of Oppenauer (aluminum isopropoxide) has been studied.—R. E. MARKER and D. L. TURNER. *J. Am. Chem. Soc.*, 62 (1940), 2540. (E. B. S.)

Sterols. CV. The Preparation of Testosterone and Related Compounds from Sarsasapogenin and Diosgenin. Testosterone and related compounds have been prepared from sarsasapogenin and diosgenin, the reactions being given in detail for the conversions.—R. E. MARKER. *J. Am. Chem. Soc.*, 62 (1940), 2543. (E. B. S.)

Sterols. CI. The Structure of Pseudosarsasapogenin. The structure of pseudosarsasapogenin was further investigated, the evidence supporting the previously suggested structure, which is given with its reactions. Proof is dependent on hydrogenation and oxidation reactions.—R. E. MARKER, E. M. JONES and J. KRUEGER. *J. Am. Chem. Soc.*, 62 (1940), 2532. (E. B. S.)

Stilbestrol—Synthesis of Tricyclic Hydrocarbons Related to. 1-Ethyl-2-phenyl-3,4-dihydronaphthalene, 3-methyl-1-ethyl-2-phenylindene and a number of intermediate compounds were synthesized by a method which establishes their structure. The method developed in the preparation of these hydrocarbons is of such a nature that it should be applicable to the preparation of those corresponding methoxy and phenolic compounds believed likely to possess estrogenic activity, and experimental work in this direction is already under way.—A. A. PLENTL and M. T. BOGERT. *J. Am. Chem. Soc.*, 63 (1941), 989. (E. B. S.)

Sulfanilamide Compounds. VI. N⁴-Acyl-N¹-Heterocyclic Sulfanilamides and N¹-Heterocyclic Sulfanilamides. A series of N⁴-acyl-sulfanilyl- and sulfanilyl derivatives of the three mono amino pyridines, 2-, 3- and 4-(pyridyl)-methylamines and 2-, 3- and 4-(pyridyl)-1-ethylamines have been prepared and are undergoing biologic assessment. Details have been given for the preparation of 4-picoline through its oxalate. A convenient method has been described for the preparation of 2-, 3- and 4-(pyridyl)-methylamines and for 3-(pyridyl)-1-ethylamine. Brief description has been given of two new heterocyclic amines, believed to be 2- and 4-(pyridyl)-1-ethylamines.—H. G. KOLLOFF and J. H. HUNTER. *J. Am. Chem. Soc.*, 63 (1941), 490. (E. B. S.)

Sulfanilamide—N¹,N⁴-Pyrazinoyl Derivatives of. The name pyrazinoic acid is proposed for pyrazine monocarboxylic acid. Five new pyrazinoyl derivatives of sulfanilamide have been prepared and described. The compounds were obtained by treating (impure) pyrazinoyl chloride with the appropriate sulfanilamide derivative. There is reason to expect that some of the compounds may be of pharmacologic interest.—T. C. DANIELS and H. IWAMOTO. *J. Am. Chem. Soc.*, 63 (1941), 257. (E. B. S.)

Sulfanilamide—New Synthesis of. The new process involves only half as many steps as have hitherto been required and starts with a cheaper chemical, namely, chlorobenzene instead of aniline. Chlorobenzene is first treated with sulfonyl chloride; the resultant compound is then heated at a temperature of 150° with aqueous ammonia in the presence of a copper catalyst. The present process starting with aniline involves four separate operations.—*Chemical Age*, 44 (1941), 289; through *Ind. & News Ed. J. Indian Chem. Soc.*, 4, No. 3 (1941), 197. (F. J. S.)

2-Sulfanilamidopyrimidines—Substituted. The preparation of six new 2-sulfanilamidopyrimidines and their N⁴-acetyl derivatives is reported. The synthesis of 2-amino-4-methyl-5-n-amylyl-pyrimidine is described and evidence for its structure is presented.—W. T. CALDWELL, E. C. KORNFELD and C. K. DONNELL. *J. Amer. Chem. Soc.*, 63 (1941), 2188. (E. B. S.)

2-Sulfanilamido-Thiophene—Synthesis of. A method is described for the synthesis of (1-acetylaminobenzene-4-sulfonamido)-2-thiophene and of (1-aminobenzene-4-sulfonamido)-2-thiophene, also called 2-sulfanilamido-thiophene in a special apparatus designed to exclude air. The acute toxicity of the above compounds has been determined and an evaluation made of their chemotherapeutic activity in experimental infections of white mice with pneumococcus Type I; 2-sulfanilamido-thiophene shows no chemotherapeutic activity.—C. VON SEEMANN and C. C. LUCAS. *Can. J. Research B*, 19 (1941), 294-295. (W. T. S.)

Sulfathiazole—Dimorphism of. The new drug, sulfathiazole (2-sulfanilamidothiazole), exists in dimorphic forms. Data have been presented giving methods of preparation, melting points, optical properties and photomicrographs.—D. C. GOVY and G. L. KEENAN. *J. Am. Chem. Soc.*, 63 (1941), 97. (E. B. S.)

Sulfonamide. II. Diphenyl Derivatives. The preparations of *p*-(*p*-aminophenyl)-benzenesulfonamide, *p*-(*p*-nitrophenyl)-benzenesulfon-N-phenylamide and *p*-(*p*-aminophenyl)-benzenesulfon-N-phenylamide are described.—A. NOVELLI and J. C. SOMAGLINO. *J. Am. Chem. Soc.*, 63 (1941), 854. (E. B. S.)

Sulfur-Containing Chemotherapeutic Compounds—Synthesis of. I. II. 4,4'-Nitroaminodiphenyl sulfoxide and 4,4'-nitroaminodiphenylsulfone were synthesized by acetylation followed by oxidation of the corresponding sulfide and saponification of the resultant product with dilute hydrochloric acid. The yields were 50% to 60% for the sulfoxide and 70% to 75% for the sulfone. 4,4'-Nitroacetylaminodiphenyl sulfide was synthesized by condensation of *p*-acetylaminophenylmercaptan with *p*-nitrochlorobenzene in alkaline solution. The *p*-acetylaminophenylmercaptan is obtained by reduction of the chloroanhydride of acetylsulfanilic acid with zinc and hydrochloric acid in presence of alcohol.—(I). IOU, O. GABEL and F. L. GRUNBERG. (II). IOU, O. GABEL and A. L. SPANION. *J. Prikl. Khim.*, 12 (1939), 1481-1484, 1485-1489; through *Chimie & Industrie*, 44 (1940), 52. (A. P.-C.)

Sulfur Studies. XVII. Synthesis of Sulfathiophene, 2-Sulfanilamidothiophene. A synthesis of sulfathiophene is described. The solubility of acetyl-2-sulfanilamidothiophene and 2-sulfanilamidothiophene in certain solvents is given.—R. W. BOST and C. F. STARNES. *J. Am. Chem. Soc.*, 63 (1941), 1885. (E. B. S.)

Tetrahydrocannabinol Analogs with Marihuana Activity. XI. A satisfactory method for condensing ethyl acetoacetate or ethyl *n*-butylacetoacetate with orcinol or olivetol is described. The resulting coumarins were converted into pyrans by means of excess methylmagnesium iodide. The pyrans from olivetol both showed marihuana activity.—ROGER ADAMS, C. K. CAIN and S. LOBWE. *J. Am. Chem. Soc.*, 63 (1941), 1977. (E. B. S.)

9-Thiophenanthrene and Some of Its Derivatives. A discussion.—PARESH CHANDRA DUTTA. *J. Indian Chem. Soc.*, 18 (1941), 469. (F. J. S.)

Vinyl Barbituric Acids—Substituted. IV. Derivatives Containing a Primary 1-Alkenyl Group. A series of fifteen substituted vinyl alkyl-barbituric acids has been prepared in which the substituted

vinyl groups are of the type $RCH=CH-$ or $R_2C=CH-$. The chemical and pharmacological properties of the compounds are recorded.—A. C. COPE, W. H. HARTUNG, E. M. HANCOCK and F. S. CROSSLEY. *J. Am. Chem. Soc.*, 62 (1940), 1199-1201.

(E. B. S.)

Vitamin B₆—Chemistry of. II. Reactions and Derivatives. Acetylation of vitamin B₆ with acetic anhydride gave a triacetate which in turn gave a crystalline hydrochloride. Vitamin B₆ dibromide hydrobromide yielded a diacetate when treated with silver acetate which also gave a crystalline hydrochloride. Reduction of the dibromide gave 2,4,5-trimethyl-3-hydroxypyridine. Catalytic reduction of vitamin B₆ or its 4-ethoxymethyl derivative gave 2,4-dimethyl-3-hydroxy-5-hydroxymethylpyridine. Methyl alcoholic solutions of vitamin B₆ when heated at 125° gave a small yield of the 2-methyl-3-hydroxy-4-methoxymethyl-5-hydroxymethylpyridine.—S. A. HARRIS. *J. Am. Chem. Soc.*, 62 (1940), 3203.

(E. B. S.)

Vitamin E—Chemistry of. XXX. Condensation of Butadiene and of Crotyl Systems with Trimethylhydroquinone. Pseudocumohydroquinone is condensed with crotyl alcohol, butadiene, crotyl chloride, methylvinylcarbinyl chloride and crotyl bromide with some anomalous results. The relation of these facts to the mechanism of the condensations is discussed.—L. I. SMITH and J. A. KING. *J. Am. Chem. Soc.*, 63 (1941), 1887.

(E. B. S.)

BIOCHEMISTRY

Alcaptonuric Urines—Chemiluminescence of. Alcaptonuric urines, as well as solutions of homogentisic acid or lead homogentisate, give off light when made alkaline and shaken with air.—LYNN D. F. ABBOTT, JR. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 14.

(A. E. M.)

Alcohol in Blood—Specific Methods for Determining. The ethyl dinitrobenzoate derivative is prepared, identified, saponified, and the amount of ethanol is determined. Heat just to boil 0.2 Gm. of recrystallized 3,5-dinitrobenzoyl chloride and 5 cc. of benzene in a 100-cc. Erlenmeyer flask under reflux. In a 100-cc. flask add 30 cc. of benzene, 2 Gm. of anhydrous copper sulfate and dropwise, while shaking, 1 cc. of sample (blood, serum, urine, etc.). On lumping, disintegrate the copper sulfate with a thick glass rod and treat the mixture with an additional 2 Gm. of copper sulfate. Stopper, shake 30 min., centrifuge, pour 25 cc. of the clear solution down the reflux condenser into the 5 cc. of benzene-dinitrobenzoyl chloride mixture, add 0.1 cc. of pyridine, let stand 30 min., bring to boiling and allow to stand 30 min. at room temperature; boil and let stand 30 min. again, then reflux gently for 4 hrs., during which add dry benzene to replace any loss during the process. Remove the reflux, cool in a water bath and pour into a separatory funnel containing 10 cc. of 10% hydrochloric acid. Shake well, remove the aqueous layer and wash twice with water. Filter the reaction mixture through a fluted filter into a measuring cylinder. Determine the alcohol in the aliquot (about 24 cc.) by Liebesny's method as described by Heiduschka and Steulmann (*Pharm. Zentralhalle*, 77 (1936), 405-409). With known samples the method proved accurate and also checks other known methods on blood samples. For qualitative identification evaporate the reaction mixture after it has been washed with dilute hydrochloric acid and water, and filter; take up the residue twice with petroleum ether and evaporate. Take up with ether again, wash with 1% sodium hydroxide solution, filter and evaporate. The crystals obtained with samples containing alcohol should melt at 93° C. Other criteria for identification are crystal form, solubility, mixed

melting point with pure ester, determination of carbon, hydrogen, nitrogen and oxygen, and color reaction with α -naphthylamine. The latter is used for small yields of crystals. The procedure is more tedious and time-consuming than other common methods, but since it is specific it should serve that purpose in forensic chemistry.—H. KLUGE. *Z. Unters. Lebensm.*, 78 (1939), 449-458; through *Chimie & Industrie*, 44 (1940), 20.

(A. P.-C.)

Allergens—Chemistry of. III. The Solubility Behavior of an Active Protein Picrate from Cottonseed. A new method of fractionating the allergenic protein picrate from cottonseed has been developed. The active protein combines with picric acid in true salt combination. Chemical and clinical evidence is presented showing that the active picrate fraction CS-5 contains more than one allergenic component, probably very closely related structurally.—J. R. SPIES, H. S. BERTON and H. STEVENS. *J. Am. Chem. Soc.*, 62 (1940), 2793.

(E. B. S.)

Amino Acids—Aromatic Sulfonic Acids as Reagents for. The Preparation of *l*-Serine, *l*-Alanine, *l*-Phenylalanine and *l*-Leucine from Protein Hydrolysates. On the basis of the solubility products of their amino acid salts, several sulfonic acids derived from diphenylamine, anthraquinone and azobenzene have been found to be of potential value for the isolation, purification and determination of amino acids. The utilization of sulfonic acids for the preparation of amino acids is exemplified by the procedures described for the isolation of *l*-phenylalanine and *l*-leucine from hydrolysates of hemoglobin, and of *l*-serine and *l*-alanine from hydrolysates of silk fibroin.—WILLIAM H. STEIN, STANFORD MOORE, GUIDO STAMM, CHI-YUAN CHOU and MAX BERGMANN. *J. Biol. Chem.*, 143 (1942), 121.

(F. J. S.)

Para-Aminobenzoic Acid in Vitamin B Complex Studies in Mice. Aminobenzoic acid has anti-gray-hair activity in the nutritional achromotrichia of the mouse. It is a dietary factor necessary for the demonstration of the anti-alopecia effect of inositol.—GUSTAV J. MARTIN and S. ANSBACHER. *Proc. Soc. Exptl. Biol. Med.*, 48 (1941), 118.

(A. E. M.)

Ariboflavinosis—Excretion Test for Measure of. The authors advocate a riboflavin excretion test as a measure of riboflavin deficiency. Lack of riboflavin in the diet is reflected in a diminished urinary excretion of flavin, but it is apparent that complete absence of riboflavin in the urine does not necessarily indicate the presence of ariboflavinosis, and on the other hand ariboflavinosis has been observed to develop when the 24-hr. specimen contained from 24 to 119 mgm. of flavin. In the technique used by the authors, an intravenous injection of 0.016 mg. per Kg. of body weight of riboflavin dissolved in distilled water is given. Before the injection, urine is collected for a control period of 1 hr. After the injection, the excretion in the urine is followed for half-hourly and then for hourly periods for 4 hrs. Measurements of riboflavin are made by the fluorometric method.—V. A. NAJJAR and L. E. HOLT, JR. *Bull. Johns Hopkins Hosp.*, 69 (1941), 476; through *Abbott Abstract Service*, (1942), No. 1056.

(F. J. S.)

Ascorbic Acid Content of Varieties of Pepper, Tomato and Their Hybrids. The author utilized for measurement ascorbic (vitamin C) alcoholate a method based upon the photochemical reduction of methylene blue by this substance. More so for the tomato than for the pepper the content of ascorbic acid, which increases with the state of maturity of the fruit, appears more strongly in the varieties of small fruits; it is independent of the red or yellow color of the ripened fruit. Usually ascorbic acid is found in the pericarp, in the leaves, peduncles and flowers.—C. SOSA-BOURDOUIL. *Acad. Sci.*, Nov.

11, 1940; through *Presse méd.*, 95-96 (1940), 979.

(W. H. H.)

Ascorbic Acid—Stability of, in Some Acids. Metaphosphoric acid has the best stabilizing effect on the ascorbic acid extracted from plant and animal tissues, but the precipitation of the proteins is not complete; they cause excessive foaming with carbon dioxide or hydrogen sulfide and form stable adsorption products with the latter. Trichloroacetic acid oxidizes ascorbic acid. Best results were obtained with 2% metaphosphoric and 4% of trichloroacetic acid. The stabilizing influence of other acids was studied. With 0.01 mg. of copper per 100 cc. of a 3% to 4% ascorbic acid solution, 0.15 times normal sulfuric acid is best. In the presence of trichloroacetic acid, hydrochloric acid is best. Copper retards the decomposition of ascorbic acid by trichloroacetic acid. The protective influence of hydrochloric acid is confirmed in experiments with tissues. The most stable ascorbic acid is in the brain, keeping for 4 hrs., while that of the liver keeps only 2 hrs. In the extraction of ascorbic acid it is possible to replace metaphosphoric acid with 0.15 times normal hydrochloric acid and 4% trichloroacetic acid or to reduce the concentration of metaphosphoric acid to only 0.002%, with 4% trichloroacetic acid.—M. M. EIDELMANN. *Biokhimičnyi J.*, 13 (1939), 715-732; through *Chimie & Industrie*, 44 (1940), 51.

(A. P.-C.)

Aspartic Acid and Glucosamine—Relation of, to Growth. By the use of diets devoid of proteins, but containing mixtures of highly purified amino acids, aspartic acid and glucosamine have been shown to be dispensable components of the food. Rats deprived of either of these compounds increase in body weight just as rapidly as do litter mate controls receiving a like ration supplemented with the compound in question.—WILLIAM C. ROSE and SCHEURING S. FIERKE. *J. Biol. Chem.*, 143 (1942), 115.

(F. J. S.)

Biochemical Oxygen Demand—Determination of. A comparative study of the azide and the Rideal-Stewart modifications of the Winkler method. Either may be expected to give reliable results.—O. R. PLACAK and C. C. RUCHHOFT. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 12-15. (E. G. V.)

Biological Materials—Mineral Analysis of. The Lundegardh air-acetylene burner used for the excitation of the emission spectra of metallic substances in solution is described, together with the Lundegardh method of calculation of the concentration of these ions in the solutions used. A series of results for calcium, copper, iron, magnesium, manganese, potassium and sodium shows a very satisfactory recovery of these elements when solutions containing known concentrations are analyzed. Methods of drying the biological materials are given, followed by a discussion of methods of preparation of these substances for analysis. Complete oxidation with nitric acid and Perhydrol at 120° to 140° C. is recommended. Recovery of metals added to these materials before the above treatment is excellent. The wide applicability of the method is evident from its successful use in the analysis of such substances as oranges, beans, pectinates, aquarium water, rat lenses, bones and blood.—M. A. GRIGGS, R. JOHNSTIN and B. E. ELLEDGE. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 99-101.

(E. G. V.)

Biometry in the Service of Biological Assay. The all-or-none effects of drugs and graded response are discussed. The characteristics of a valid biological assay are: (1) different samples of the same drug must show the same relative potencies in biological assay as under clinical test; (2) on the coordinates used for biological assay, the curve relation response to log-dose should be a straight line and relatively

steep when compared with the variation about the line; (3) the potency of the unknown or sample should be determined by comparative test with a stable reference standard and expressed in units of this standard; (4) the living material exposed to different doses of a standard and an unknown must be as nearly equivalent as possible; (5) a determination of potency should always include an estimate of its error, computed as an integral part of the assay.—C. I. BLISS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 84-88. (E. G. V.)

Blood—Rates of Hemolysis in Human, Stored in Dextrose Solutions and in Other Mixtures. The addition of dextrose to blood-citrate mixtures markedly inhibits hemolysis during storage at 2° C. Sucrose is not so effective and NaCl and KCl increase the rate of hemolysis. The optimal dextrose concentration is 3%. It permits storage for 30 days or more.—ELMER L. DEGOWIN, JOHN E. HARRIS and JOY BELL. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 481. (A. E. M.)

2-Bromocholestanone—Collidine Treatment of. In the preparation of steroids for photochemical study, the collidine method was employed for the splitting of hydrogen bromide from 2-bromocholestanone to form 1-cholestenone and cholestanone.—R. P. JACOBSEN. *J. Am. Chem. Soc.*, 62 (1940), 1620-1621. (E. B. S.)

Butter—Deterioration of, During Storage. II. Development of Tallowiness. Tallowiness is a taint produced by the oxidation of the fat proper of butter usually following the intermediate taint of fishiness. The development of tallowiness and the oxidation of butterfat are strongly catalyzed by traces of copper, iron, manganese, silver, nickel, cobalt and chromium but not by aluminum and tin. In the presence of traces of these metals, free fatty acidity has only a minor effect in catalyzing oxidation. For detecting incipient tallowiness the sense of taste is by far the most sensitive; other tests in decreasing order of sensitivity are: peroxide content, Schiff's test, oxidizability values and Kreis test. The presence of traces of heavy metals prevents the development of butyric rancidity in fresh butter and reduces greatly the amount of free volatile and total free fatty acids formed in stored butters. **III. Free Acidity.** Freshly made butter has a titratable acidity less than 2 but moldy butters may have an acidity up to 20 cc. *N* alkali per 100 Gm. The free fatty acids are mostly C₁₆ and C₁₈ acids, oleic acid appearing in the free form in greatest equivalent. The development of acidity increases progressively with time and leads to a condition of an acid tallowiness. **IV. Moldiness and Ketonic Rancidity.** Mold growth in butter causes both an acid-oxidative rancidity and a ketonic rancidity, the former being due to molds containing lipases and able to metabolize glycerol (*Oidium*) and the latter due to dry molds fructifying on the surface of the butter and also causing discoloration (*Penicillium*). The acid-producing molds also cause fishiness to develop before tallowiness sets in. The dry molds act mostly on the water-insoluble volatile fatty acids. The coconut flavor of sterilized milk is caused by a *Cladothrix* whose spores survive sterilization temperatures and act on butterfat in a similar manner.—W. L. DAVIES *Ind. & News Ed. J. Indian Chem. Soc.*, 4, No. 3. (1941), 150, 175, 179. (F. J. S.)

C-Avitaminosis—Contribution to the Prophylaxis of, in the Population. The scarcity of fresh citrus fruits and other natural sources of vitamin C is leading to an increasing number of cases of vitamin C deficiency both in the population and in the army. The expense of synthetic vitamin C as well as the general aversion on the part of the public to synthetic chemicals make it difficult to adequately supplement the diet. The author reviews the litera-

ture on other sources of vitamin C and points out the instability of vitamin C in extracts prepared from unedible fruits or parts of fruits. For example, it is shown that a greater concentration of vitamin C is present in the rind of oranges than in the pulp and that an average of 100 mg. ascorbic acid is lost in discarding the rind. Some little known but relatively rich sources of vitamin C are pointed out in the hope that they may provide auxiliary supplies of the vitamin to help prevent C deficiency.—A. MIRIMANOFF. *Schweiz. Apoth.-Ztg.*, 79 (1941), 333. (M. F. W. D.)

Cereal Products. Vitamin and Mineral Restoration and Fortification from the Viewpoint of the Manufacturer. The cereal industry is an important factor in any program of national nutritional improvement. The problem of fortification and restoration is presented by comparing the mineral and vitamin composition of cereal products with that of the corresponding grains. The degree of fortification is discussed from the viewpoint of the average adult and child daily requirements of these essential nutrients. The various sources used to bring about such a restoration are presented, with their merits and limitations. This includes a discussion of pure vitamin and mineral sources, cereal fractions rich in the essential nutrients and concentrates of a non-cereal origin.—R. T. CONNER. *Ind. Eng. Chem.*, 33 (1941), 711-713. (E. G. V.)

Cerebrosides in Nerve Tissue—A Micromethod for the Estimation of. A colorimetric method is given for the quantitative estimation of cerebrosides by the use of the carbazole reaction of Dische. After hydrolysis of the cerebrosides the fatty acids and the glycerol are removed. The fatty acids are separated through their insolubility in water and the glycerol is taken up in ethyl acetate. The method can be applied to 0.2 to 0.6 mg. of cerebrosides.—P. V. EDMAN. *J. Biol. Chem.*, 143 (1942), 219. (F. J. S.)

Choline Metabolism. VI. Hemorrhagic Degeneration and the Labile Methyl Supply. Hemorrhagic degeneration is the result of a dietary deficiency of choline and of the labile methyl supply. Betaine, like methionine, contributes to the labile methyl supply of the body and may be substituted for choline. The effectiveness of betaine corresponds to the utilization of only one of the three methyl groups. Creatine does not contribute to but does spare the labile methyl supply. Cysteine, homocystine and glutathione, but not taurine, increase the severity of hemorrhagic degeneration.—W. H. GRIFFITH and D. J. MULFORD. *J. Am. Chem. Soc.*, 63 (1941), 929. (E. B. S.)

Copper Content of the Milk of Sheep and of Cows. In Western Australia the copper content of the milk of normal ewes (which ranges from 0.01 to 0.64 mg. copper per liter) is dependent on the pasturage and the period of lactation (higher in early periods). There is no correlation in the blood copper levels and the milk copper variations. Copper variations in the cow were greater.—A. B. BECK. *Australian J. Exptl. Biol. Med.*, 19 (1941), 145-150. (W. T. S.)

Cottonseeds—Composition of. III. Solubility of Proteins in Alkaline Solutions of Neutral Salts. At pH 9.0 small amounts of sodium chloride markedly depress the solubility of cottonseed protein, as determined by extraction studies with cottonseed meal. Greater or smaller concentrations of salt permit the peptization of larger amounts of total nitrogen. The alkaline earth salts are particularly effective precipitants; under the conditions used, 0.03-0.05 N calcium or magnesium salt solutions suppress the solubility of the globulin at pH 9.0 completely. The effects of several other salts on the solubility of the nitrogenous constituents of

cottonseed meal have been determined. That the observed phenomena can be ascribed to the globulins and not to other constituents of the meal is shown in experiments with isolated globulin preparations. Analogous results have been obtained with hempseed meal. The data of others have been interpreted to indicate that the influence of salts on the solubility of soybean proteins in the neutral pH range is a parallel phenomenon. The observations cited are exceptions to the hitherto-accepted generalization that the solvent action on proteins of salts and alkali is additive.—H. S. OLCOTT and T. D. FONTAINE. *J. Am. Chem. Soc.*, 62 (1940), 1334-1339. (E. B. S.)

2,4-Dibromo- α -Estradiol. α -Estradiol is converted smoothly and almost quantitatively into 2,4-dibromo- α -estradiol on standing overnight in alcohol solution with the theoretical quantity of N-bromoacetamide.—R. B. WOODWARD. *J. Am. Chem. Soc.*, 62 (1940), 1625-1626. (E. B. S.)

Diffusing Factors. VII. Concentration of the Mucinase from Testicular Extracts and from *Crotalus Atrox* Venom. $(\text{NH}_4)_2\text{SO}_4$ -fractionation of aqueous testicular extracts, followed by lead acetate precipitation under controlled pH, gives a convenient purification of mucinase. This enzyme can also be purified by dilution of NaCl extracts of testicle with water. Both mucolytic and diffusing activities are in the precipitate. The lecithinase and protease of *Crotalus atrox* venom are more thermostable than the mucinase. Concentration of the mucinase from this venom has been carried out by $(\text{NH}_4)_2\text{SO}_4$ -fractionation and by adsorption methods.—J. MADINAVETIA. *Biochem. J.*, 35 (1941), 447. (F. J. S.)

Digitonin—Effect of, on Solubility of Phospholipids. Mixed phospholipid from blood plasma or egg yolk is rendered quantitatively insoluble in petroleum ether by the use of digitonin. Lecithin and cephalin obtained from egg yolk behaved in a similar manner.—H. D. McEWEN and P. L. MACLACHLAN. *Proc. Soc. Exptl. Biol. Med.*, 48 (1941), 195. (A. E. M.)

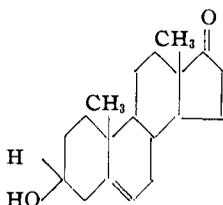
Dihydroequilin and Estradiol—Separation of. A mixture containing these two compounds is made to react with a saponin, such as digitonin, and the saponin reaction products and unreacted products are separated, as by a selective solvent, and the separated saponin compounds are subsequently split up, as by heating with pyridine, for recovery of the substances combined with the saponin.—ARTHUR SERINI and WILLY LOGEMANN, assignors to SCHERING CORP. U. S. pat. 2,221,340, Nov. 12, 1940. (A. P. C.)

3 : 5-Dinitrobenzoyl Derivatives of the Amino Acids and Their Use in Separating the Isomers of Leucine and Valine. It has been confirmed that 3 : 5-dinitrobenzoyl chloride is a very valuable reagent for the identification of amino acids. A table of melting points of those derivatives which the author has been able to prepare is given. The melting points found for some derivatives, notably those of phenylalanine, serine and valine, differ considerably from those quoted by Saunders. There have been isolated two derivatives from the specimens of valine available, and three derivatives from the samples of leucine at the author's disposal. These are thought to be the derivatives of valine and norvaline, and of leucine, isoleucine and norleucine, respectively. The identity of the norvaline and norleucine derivatives is subject to confirmation. It has been found possible to separate the components in mixtures of the various isomers of valine and leucine by fractional precipitation of their derivatives at differing pH. This is important, as no means of separating mixtures of such isomers has hitherto been available. A derivative

has been isolated from aspartic acid, of which the Na salt is readily salted out at pH 4. The aspartic acid derivative is easily hydrolyzed in solutions more acid than pH 2. No derivative could be isolated from tyrosine.—BERNARD WILLIAM TOWN. *Biochem. J.*, 35 (1941), 578. (F. J. S.)

Estradiol 17-Monoesters. Partially esterified compounds of the dihydroestrin series having a free phenolic hydroxyl and intended for therapeutic purposes are obtained when completely esterified compounds of this series are carefully treated with either acid or alkaline hydrolytic agents.—KARL MIESCHER and CAESAR SCHOLZ, assignors to CIBA PHARMACEUTICAL PRODUCTS, INC. U. S. Pat. 2,233,025, Feb. 25, 1941. (A. P.-C.)

Male Hormones, Etc.—Reduction Products of the. A method of improving the physiological action of keto-group-containing male hormones of the general structural formula



(the dotted line indicating the probable position of the double bond in the unsaturated hormones) involves subjecting the starting material to the action of agents capable of reducing the keto group to the secondary alcohol group and of saturating a double bond when present, whereby compounds of the general formula $C_{19}H_{32}O_2$ are obtained. The reduction may be effected by the use of sodium.—WALTER SCHOELLER, assignor to SCHERING CORP. U. S. pat. 2,232,734, Feb. 25, 1941.

Erogens with Proteins—Conjugation of. I. Estrone *p*-nitrophenyl ether was prepared and reduced to estrone *p*-aminophenyl ether. The latter on diazotization coupled with phenolic compounds, and with casein to form an orange-yellow azoprotein. Phenyl-*p*-nitrobenzyl ether was reduced to phenyl *p*-aminobenzyl ether and the diazotized amine was coupled with casein giving an orange azoprotein. Reduction of estrone *p*-nitrobenzyl ether and 4,4'-di-(*p*-nitrobenzyloxy)- α,β -diethylstilbene failed to yield the corresponding estrogen amino ethers.—L. F. KING and W. R. FRANKS. *J. Am. Chem. Soc.*, 63 (1941), 2042. (E. B. S.)

Estrone—Experiments on the Synthesis of. I The 2-(β -Phenylethyl)-Furans as Components in the Diene Synthesis. The synthesis of 2-(β -phenylethyl)-furan and 2-(β -(*m*-methoxyphenyl)-ethyl)-furan has been accomplished. The Diels-Alder reaction has been applied with success to these substances.—R. B. WOODWARD. *J. Am. Chem. Soc.*, 62 (1940), 1478-1482. (E. B. S.)

Estrone—Oxidation of, by Hydrogen Peroxide. The oxidation of estrone in an alkaline solution with H_2O_2 produces a hydroxy acid in which Ring D of the estrone molecule is ruptured at the carbonyl group. The hydroxy acid rapidly loses the elements of H_2O and is isolated as the lactone. The lactone gives a negative reaction in the Kober and Zimmermann tests and is one-fourteenth as active in spayed mice as estrone. A similar oxidation of cyclopentanone with H_2O_2 gave δ -valerolactone.—W. W. WESTERFELD. *J. Biol. Chem.*, 143 (1942), 177. (F. J. S.)

Fibrinogen—Determination of, with Protamine. The following summary is given: (1) A method is described for the quantitative determination of fibrinogen in plasma with the aid of protamine.

(2) Heparin does not interfere with this determination when it is present even in the higher amounts shown to occur in anaphylactic shock (25 units per cc. of blood). (3) Fibrinogen values secured by this technique are about 20% higher than fibrin values obtained by the methods of Cullen and Van Slyke and others that require high plasma dilution. (4) These differences are reduced to about 5% if the comparative fibrin determination are made with smaller plasma dilutions. (5) The discrepancies are not explained by the assumption of a soluble form of fibrin. (6) After the addition of protamine to plasma the enzymatic fibrinogenolysis is markedly increased. About 35% of the fibrinogen is split off after 3 hrs.' incubation of the fibrinogen-protamine precipitate.—E. MYLON, M. C. WINTERNITZ and G. J. DE SÜTÖ-NAGY. *J. Biol. Chem.*, 143 (1942), 21. (F. J. S.)

Fluorides in Mixed Foods—Aliquot Samples in the Determination of. Suggestions are given for grinding the food sample, treating with magnesium peroxide, taking aliquots, ashing, distilling the ash with perchloric acid to obtain the fluoride and then back titration.—E. J. LARGENT. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 93. (E. G. V.)

Fortified Foods—What the Consumer Should Know about. The attitude of nutritionists on fortification of foods has changed in two years from disapproval to limited recommendation under proper control. It is realized that education in food values cannot alone solve all our nutrition problems. Consumers want to know why bread and flour have been "enriched" and what other foods may be considered for similar treatment. They want facts and not extravagant claims in vitamin and mineral advertising. They need to know that no one food, either in its natural or enriched state, will ensure an adequate diet. They will be confused by too many figures, but they can understand what proportion of the day's requirement is obtained from a slice or a loaf of bread or a quart of vitamin D milk. They may want to know exactly what vitamins and minerals have been retained or added, and why. They need to be reassured that added vitamins or minerals are just as well utilized as the same factors in the natural foods.—H. S. MITCHELL. *Ind. Eng. Chem.*, 33 (1941), 716-717. (E. G. V.)

Gastric Juice—Nitrogen Content and Total Osmotic Activity of. Pilocarpine stimulated gastric pouch juice has a lower osmotic activity and higher total nitrogen content than histamine stimulated juice. Fasting juices show variable relations between nitrogen content and osmotic activity. Pyloric pouch fasting juice has a higher nitrogen content than greater curvature pouch juice. The osmotic activity of the former is nevertheless hypertonic with respect to blood. There appears to be a partial correlation between total nitrogen content and osmotic activity of gastric juice.—NATHAN LIFSON, RICHARD L. VARCO and M. B. VISSCHER. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 410. (A. E. M.)

Germinal Gland Hormones—Reducing Unsaturated. A method of reducing keto-group-containing unsaturated germinal gland hormones to corresponding unsaturated hydroxy compounds involves treatment with hydrogen in the presence of a skeleton catalyst capable of promoting reduction of keto groups, such as Raney catalyst, at a temperature below 50° C. and under approximately atmospheric pressure.—LOTHAR STRASSBERGER and ERWIN SCHWENK, assignors to SCHERING CORP. U. S. pat. 2,223,393, Dec. 3, 1940. (A. P.-C.)

Gonadotropic Hormones—Carbohydrates of the. Hormones obtained from the pituitary gland contain mannose and hexosamine in equimolecular proportions. The gonadotropines of pregnant mare serum

and of human pregnancy urine appear to contain galactose rather than mannose. In these preparations the ratio of hexose to hexosamine is 2:1.—SAMUEL GURIN. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 48. (A. E. M.)

Hemin—Reinvestigation of the Configuration of. It is suggested that the synthesis of the unsymmetrical tetramethylmethene used in the synthesis of deuterio-porphyrin and hemin may be another example of an anomalous aldehyde synthesis of dipyrromethenes, since the melting points of the symmetrical and unsymmetrical isomers are so close. Synthesis proved that these melting points are not so close as reported in the literature, and the configuration assigned by Fischer's laboratory is confirmed. A new method for the proof of structure of dipyrromethenes is presented.—A. H. CORWIN and R. H. KRIEBLE. *J. Am. Chem. Soc.*, 63 (1941), 1829. (E. B. S.)

Histidine—Preparations of, by Means of 3,4-Dichlorobenzenesulfonic Acid. When a liberal excess of 3,4-dichlorobenzenesulfonic acid is added to a decolorized hydrochloric acid hydrolysate of crude hemoglobin at pH 1.2 to 1.6, histidine slowly separates in the cold as the relatively insoluble di-3,4-dichlorobenzenesulfonate. The histidine salt is at times accompanied by a considerable quantity of impure leucine mono-3,4-dichlorobenzenesulfonate, but the mixture can readily be separated by recrystallizing and taking advantage of the marked capacity of the impure leucine salt to form a supersaturated solution. Most of this can be removed from the crystals of histidine salt by decantation under the correct conditions; the histidine salt is then readily purified by recrystallization. Histidine is recovered in satisfactory form, either as free base or as the monohydrochloride, after removal of the sulfonic acid as its insoluble barium salt. The yields depend upon the purity of the starting material with respect to its content of hemoglobin; from fairly pure preparations of red blood cells, the equivalent of over 6% of histidine has been obtained.—HUBERT BRADFORD VICKERY. *J. Biol. Chem.*, 143 (1942), 77. (F. J. S.)

Hypoxanthine—Estimation of. Hypoxanthine (1 mg. or more of nitrogen) is determined by a volumetric method with an error of about 1%. It is precipitated as the crystalline argentipicrate. This is separated from silver chloride by extraction with hot concentrated nitric acid and the silver is determined by titration with standard thiocyanate solution.—GEORGE H. HITCHINGS. *J. Biol. Chem.*, 143 (1942), 43. (F. J. S.)

Intravenous Nourishment with Protein, Carbohydrate and Fat in Man. Parenteral nutrition was carried out over a 17-day period in an adult male by means of intravenous injection of dextrose, casein digest (amino acids) and emulsified fat.—DWIGHT E. CLARK and ALEXANDER BRUNSWIG. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 329. (A. E. M.)

Keto Derivatives of Cholic and Desoxycholic Acids—Determination of the, in Bile. A method described previously for the determination of highly purified ketosteroids has been applied to the analysis of 3-hydroxy-12-ketocholic, 3,12-diketocholic, 3-hydroxy-7,12-diketocholic and 3,7,12-triketocholic acids added to bile. The recovery of these compounds from the bile ranged from 97.1% to 101.2% of the theoretical amounts.—HETTIE B. HUGHES. *J. Biol. Chem.*, 143 (1942), 11. (F. J. S.)

Lactose and Arakawa's Reaction of Human Milk. Human milk positive to Arakawa's reaction is higher in lactose content than the Arakawa negative milk (colostrum excepted). Lactose content is slightly lower in cold seasons than in other seasons. Lactose content in human milk is generally the

highest in the middle mature period of lactation. Lactose content of human milk is almost constant in every age of mother, and it is also equal on both sides of the breast in the average. In Arakawa positive milk, cases of abundant secretion are more numerous than in Arakawa negative milk. One and the same mother can secrete a milk of different quantities of lactose content on different sides of the breast. The lactose content of human milk can be different according to different factors, but such a remarkable difference of the content as existing between Arakawa positive and Arakawa negative mothers is not seen. Lactose-poor milk seems to be a "bad" milk, both from descriptions in literature and also from my own result of experiment. Arakawa positive milk, which has been shown to be a "good" milk generally in a number of papers, is rich in lactose.—K. SHODA. *Tohoku J. Exp. Med.*, 39 (1940), 124. (A. C. DeD.)

Nicotinic Acid. A review.—ALFRED CLARK. *J. Trop. Med. Hyg.*, 44 (1941), 126-129. (W. T. S.)

Nicotinic Acid and Vitamin B₃—Chemical Estimation of. The nicotinic-acid methods now available to the investigator leave something to be desired. However, accepting the limitations of the cyanogen bromide method in general, it can be applied with a fair amount of success to the determination of nicotinic acid in animal tissues, urine and blood. The analysis of plant materials must await further investigation before acceptable values are obtained. For practical purposes, however, the relative amounts of nicotinic acid in a variety of samples are important and the absolute values although desirable are not necessary. The liberation of vitamin B₃ from naturally occurring materials depends on a pepsin digest followed by tungstate precipitation for removal of proteins and silver nitrate precipitation for the removal of purines, pyrimidines and amidazole bases. The vitamin was absorbed, then eluted and further treated to remove interfering materials. The pH was adjusted to 7 and the color developed with the diazotized sulfanilic acid. This method is open to criticism since the reaction is not specific.—H. A. WAISMAN and C. A. ELVEHJEM. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 221-225. (E. G. V.)

Nicotinic Acid of Animal Tissues and Blood. By use of a previously developed method for estimating nicotinic acid by absorbing it on charcoal, the writers have determined the content of this vitamin in sheep, cow, pig and fowl tissues, and in the blood of normal persons, pellagrins and lepers. The values for the animal tissues agree with those previously reported. The blood of lepers and pellagrins contain less nicotinic acid than that of normal persons.—K. V. GIRI and B. NAGAMA. *Indian J. Med. Research*, 29 (1941), 585-590. (W. T. S.)

Ninhydrin Reaction—Increasing the Sensitivity of, by Traces of Ascorbic Acid. The ninhydrin reaction with amino acids, peptones, etc., can be made more sensitive by the addition of small amounts of a freshly prepared *l*-ascorbic acid solution. A pure blue color is obtained.—E. TETZNER. *Mikrochemie*, 28 (1940), 141-144. (R. H. B.)

Non-Benzenoid Steroid—Total Synthesis of. The synthesis of 15,16-dihydro-17-cyclopenta(α) phenanthrene is described briefly and the analysis given.—L. W. BUTZ, A. M. GADDIS, E. W. J. BUTZ and R. E. DAVIS. *J. Am. Chem. Soc.*, 62 (1940), 995-996. (E. B. S.)

Nucleoproteins—Physical Chemical Investigation of Certain. I. Preparation and General Properties. Certain striking physical and chemical properties of the calf thymus nucleohistone protein have been presented. The phosphorus and nitrogen contents were accurately determined. The specific volume, refractive index increment and viscosity of

this protein in solution, as well as a titration curve are given. The effect of certain preparational methods on the centrifugal and diffusion properties of the hog thyroid nucleoprotein has been widely discussed.—R. O. CARTER and J. L. HALL. *J. Am. Chem. Soc.*, 62 (1940), 1194-1196. (E. B. S.)

Nutritional Requirements of Man. The nutritional requirements of man can be expressed in chemical terms to a greater extent than ever before. Individuals suffering from certain deficiencies may be treated successfully through the administration of a single chemical compound. The yearly supply of most of our essential vitamins can be purchased at a wholesale price approaching the cost of our energy foods. In spite of all this information we should not feel too confident that adequate nutrition involves merely the supply of a few chemical compounds. Figures on the daily requirement of the better known nutrients are given. They are limited in value for many reasons. There are additional essential nutrients to be isolated and studied quantitatively. The requirement differs for different age groups and for different individuals in the same age groups. The quantity of one dietary constituent may affect the requirement of another factor. Much of our information has come from animal work and we must, therefore, recognize species differences.—C. A. ELVEHEJEM. *Ind. Eng. Chem.*, 33 (1941), 707-710. (E. G. V.)

Pantothenic Acid Absorption in Pernicious Anemia. In patients with pernicious anemia, the daily excretion of pantothenic acid both before and after administration of 100 mg. of its calcium salt was slightly, but not significantly less than that of healthy individuals. This indicates that there is no impairment of absorption in patients with pernicious anemia although it is always associated with achlorhydria. The possibility, however, still exists, that pantothenic acid contained in food is less available because of less efficient digestion.—CURTIS E. MEYER, IRVING F. BURTON and CYRUS C. STURGIS. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 363. (A. E. M.)

Pantothenic Acid—Biological Activity of Synthetic. It has been found that synthetic *d,l*-pantothenic acid contains 36 units per mg. Natural pantothenic acid has been stated to contain 71 chick units per mg. This points to the inactivity of one enantiomorph in the synthetic preparation.—S. H. BABCOCK, JR., and T. H. JUKES. *J. Am. Chem. Soc.*, 62 (1940), 1628. (E. B. S.)

Pantothenic Acid—Resolution of Racemic, by Means of Quinine Methohydroxide. *d,l*-Pantothenic acid has been resolved by means of its quinine metho salt. The resulting quinine metho salts of (+)- and (-)-pantothenic acids were identical with specimens prepared from the pure acids. The enantiomorphous acids were also compared as their *s*-benzylthiuronium salts with authentic samples. *d,l*-Pantothenic acid has also been resolved by means of its cinchonidine salt. The (+)-salt was identical with a specimen from calcium pantothenate (+). The (-)-salt was not isolated. The (+)-salts from both resolutions showed full growth stimulation activity when assayed with *Lactobacillus casei*. The (-)-salts had practically no activity when assayed by the same method.—E. T. STILLER and P. F. WILEY. *J. Am. Chem. Soc.*, 63 (1941), 1237. (E. B. S.)

Photoelectric Vitamin A Photometer. A new photoelectric instrument has been developed for the measurement of vitamin A, using a zinc vapor lamp as light source and a single sodium photocell. The zinc arc spectrum contains six strong lines between 3282 and 3346 Å. When used with a sodium photocell, a single Corning No. 597 filter effectively isolates these six lines, since the sodium photocell

does not respond to the zinc arc lines above 5000 Å. which are transmitted by the filter. The transmission of this filter for the isolated group of six lines is about 50%.—B. DEMAREST. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 374-376. (E. G. V.)

Placental Blood. Placental blood is valuable for transfusion purposes because it is particularly rich in hemoglobin, red cells, bilirubin, salts of potassium, calcium and copper, and low in cholesterol, albumen and globulin. These constituents make it useful in cases of anemia and shock. Objectionable factors are minor, such as temporary urticaria and fever, which may also accompany the common type of transfusion. Blood is collected in sterile flasks with an anticoagulant preservative of mixed sodium chloride (7 Gm.), sodium citrate (5 Gm.), potassium chloride (0.2 Gm.), magnesium sulfate (0.0049 Gm.) and redistilled water 1000 cc. Two samples tubes are taken, one for a Wassermann test and one for typing. Placental blood may be used only if it comes from healthy women who have come to full term, and with membranes unruptured before the fetus is expelled. Studies were made on three lots of blood after 15 days' refrigeration at 4° to 6° C. The blood appeared hemolyzed but the red count was undiminished, though the red cells were much more fragile. There were indications of poikilocytosis, anisocytosis and macrocytosis. The number of leucocytes had diminished, particularly the polymorphonuclears. There were also fewer platelets, but the thromboplastic potency was normal. It was concluded that the cellular elements and thromboplastic potency of placental blood make it acceptable for transfusion after fifteen days' refrigeration.—R. S. DIAZ RIVERA. *Puerto Rico J. Pub. Health and Trop. Med.*, 16 (1941), 523. (G. S. G.)

Plant Nutrient Deficiency Symptoms. A fairly large and apparently increasing number of chemical elements is coming to be recognized as necessary, or in some cases beneficial if not essential, to the normal growth and reproduction of higher plants. The quantitative requirements of the respective elements vary from minute traces to comparatively large amounts, and the concentrations tolerated by plants are limited to a narrow range with some elements but extend over wide ranges with others. The toleration of a wide range of concentrations of the major nutrient elements is an important factor enabling plants to adapt themselves to varied soil environments. In spite of this adaptability, symptomatic indications of maladjustments in crop plants have become increasingly prevalent in recent years. Biochemical studies of normal and affected plants, considered in connection with growth and yield response to soil treatment, have contributed to an understanding of the relation between plant symptoms and the disturbances in plant metabolism brought about by malnutrition. The results lead to more accurate diagnosis of plant malnutrition by aid of symptoms.—E. E. DETURK. *Ind. Eng. Chem.*, 33 (1941), 653. (E. G. V.)

Potassium and Sodium—Determination of, in Blood Serum. Three cc. of serum are digested with sulfuric acid and hydrogen peroxide; the potassium and sodium separated as iodides by means of isobutyl alcohol and ethyl ether. The iodides are titrated according to L. W. Winkler's method, which consists of oxidizing to iodate by chlorine water, adding excess of potassium iodide and titrating the excess of iodine with thiosulfate. Tables giving corrections for potassium and sodium are included. By adherence to the described method, the authors claim that deviations from the mean values of $\pm 3\%$ for potassium and $\pm 1\%$ for sodium can be attained.—M. HEGEDUS and I. JUVANCI. *Mikrochemie*, 28 (1940), 113-124. (R. H. B.)

Potassium—Microdetermination of. A method

for the determination of potassium is described which is based on precipitation as potassium dipicrylamine; the precipitate is collected, washed and dissolved in alcohol, and the potassium content determined by measurement of the color of the resulting solution. The application of the method to the determination of blood serum potassium is described.—C. R. HARRINGTON. *Biochem. J.*, 35 (1941), 545. (F. J. S.)

Pregnenediones—Derivatives of. Compounds suitable for use in the preparation of therapeutic agents having the action of the cortical suprarenal hormone may be produced by treating a saturated 10,13-dimethylpolyhydrocyclopentanophenanthrene compound having a keto group at C₃ and a side chain at C₁₇ of the form $-\text{CO}\cdot\text{CH}_2\text{R}$ (in which R indicates a radical from the class consisting of hydroxyl, halogen, acyloxy and alkoxy) with a halogen such as bromine, and thereafter splitting off hydrogen halide from the reaction product to produce a double bond in ring A of the compound. Various details of procedure are given in examples.—TADEUS REICHSTEIN, assignor to ROCHE-ORGANON, INC. U. S. pat. 2,232,730, Feb. 25, 1941. (A. P. C.)

Provitamin A Not Found in American Wholewheat Flour. For the second time it has been shown that American wholewheat flours, and bread therefrom, are of no importance as a source of provitamin A in human nutrition. Samples of freshly milled, unbleached flour were found by the Tswett chromatographic method to contain from 0.02 to 0.06 mg. β -carotene per kilo.—L. ZECHMEISTER and R. B. ESCUE. *Proc. Nat. Acad. Sci. U. S.*, 27 (1941), 528-532. (W. T. S.)

Serum Electrolytes—Studies in. XIII. Estimation of Total Base in Serum. A method is given by which the concentration of total base of serum may be calculated from measurements of specific conductance and either specific gravity or serum protein. The method is simple and economical of both time and material, so that it would seem to afford a method of choice for clinical studies.—F. WILLIAM SUNDERMAN. *J. Biol. Chem.*, 143 (1942), 185. (F. J. S.)

Sex Hormones—Studies on the Preparation of Synthetic. I. Hexoestrol. A new method for the preparation of hexoestrol, m. p. 185° has been described. The starting material is *p*-hydroxypropionophenone and the yields are better than for previous methods.—S. BERNSTEIN and E. S. WALLIS. *J. Am. Chem. Soc.*, 62 (1940), 2871. (E. B. S.)

Sex Hormones—Synthesis of Analogs of. An Analog of Equilenin Lacking the Phenolic A Ring. The synthesis of the *cis* and *trans* forms of 3'-keto-2-methyl-1,2-cyclopentano-1,2,3,4-tetrahydronaphthalene from 1-tetralone is described. These compounds possess the B, C and D rings of the sex hormone equilenin including the angular methyl group, but lack the phenolic A ring. In addition, a homolog possessing a six-membered D ring has been synthesized.—W. E. BACHMANN and D. G. THOMAS. *J. Am. Chem. Soc.*, 63 (1941), 598. (E. B. S.)

Sex Hormones—Synthesis of Compounds Related to. A Homolog of Equilenin Containing an Angular Ethyl Group. The synthesis of the *cis* and *trans* forms of a homolog of the sex hormone equilenin is described. The homolog contains an ethyl group in the place of the angular methyl group. The estrogenic activity of one of the racemic forms is of the same order as that of racemic equilenin.—W. E. BACHMANN and D. W. HOLMES. *J. Am. Chem. Soc.*, 63 (1941), 595. (E. B. S.)

Sterols. CXX. Anterior Pituitary Gland Extracts. Anterior pituitary extract has been partially studied. (1) The only sterol isolated was chol-

esterol. (2) Sodium stearate was found. (3) A water-soluble nitrogenous product, C₂H₁₀N₄O₄, or C₁₀H₁₈N₆O₆, has been isolated. (4) The hydrocarbon found in pregnancy urines also was obtained.—R. E. MARKER and E. L. WITTEBECKER. *J. Am. Chem. Soc.*, 63 (1941), 1031. (E. B. S.)

Sterols. C. Diosgenin. The reactions of diosgenin have been studied, there being presented a chain of reactions showing the relationships of the members of this general series to diosgenin.—R. E. MARKER, T. TSUKAMOTO and D. L. TURNER. *J. Am. Chem. Soc.*, 62 (1940) 2525. (E. B. S.)

Sterols—Extraction of. A lipoidal substance separated from a fungoid organism such as yeast is treated with an aliphatic alcohol containing 4 to 6 carbon atoms as a solvent, a binary mixture containing solvent and water is removed, the lipoidal substance is saponified in the presence of solvent, as by sodium hydroxide solution, and, after evaporation, water is added to dissolve the solvent with subsequent precipitation of sterols, and final separation of the precipitated sterols.—WM. G. BENNETT, assignor to STANDARD BRANDS, INC. U. S. pat. 2,223,398, Dec. 3, 1940. (A. P. C.)

Sterols. XCIX. Sterols from Various Sources. The androstane and pregnane content of cantharides, Mexican flies, ant eggs, sheep feces, chicken feces and mares' non-pregnancy urine were investigated.—R. E. MARKER and A. C. SHABICA. *J. Am. Chem. Soc.*, 62 (1940), 2523. (E. B. S.)

Sterols. CVIII. The Preparation of Dihydroandrosterone and Related Compounds from Diosgenin and Tigogenin. Dihydroandrosterone was obtained from diosgenin. The intermediates in this preparation are described.—R. E. MARKER. *J. Am. Chem. Soc.*, 62 (1940), 2621. (E. B. S.)

Succinic Acid—Formation of, in Yeast. The following summary is given: (1) The amount of succinate formed in the resting suspensions of baker's yeast, brewer's yeast and *Torulopsis utilis* is proportional to the amount of sugar fermented. The yield of succinate varies between 1.35 and 3.96 Gm. mol. per 100 Gm. mol. of sugar. The quantity of succinate can exceed the dry weight of the fermenting yeast. This shows conclusively that most of the succinate formed during fermentation is derived from the fermented sugar. Ehrlich's view, according to which succinic acid is derived from the glutamic acid of autolyzed yeast cells, is therefore to be discarded. (2) The formation of succinate depends on the presence of bicarbonate in the medium. No succinate is formed in 1% KH₂PO₄; between 0.45 and 1.29 mg. succinate are formed when 50 mg. glucose are fermented by yeast suspended in 0.5% NaHCO₃. (3) The amount of succinate formed from various fermentable sugars is of the same order. (4) Oxaloacetate, *l*-malate and *l*-aspartate increase the yield of succinate in the presence of glucose by about 20%, α -ketoglutarate and α -hydroxyglutarate about 30%, *l*-glutamate about 200%, while various other substances had no effect. (5) No succinate is found when glucose is fermented by growing yeast cells. (6) Fumarase is absent from baker's yeast. (7) The mechanism of succinate formation from glucose is discussed.—ARNOŠT KLEINZELLER. *Biochem. J.*, 35 (1941), 495. (F. J. S.)

Sulfanilamide—Oxidation Products of. On oxidation sulfanilamide yields the corresponding azo- and azoxy-benzenes which may be reduced to the hydrazo compound. It can be chlorinated readily by a mixture of hydrogen peroxide and hydrochloric acid.—M. K. SEIKEL. *J. Am. Chem. Soc.*, 62 (1940), 1214-1216. (E. B. S.)

Sulfur—Use of, as a Reagent for the Estimation of the Sulfhydryl Groups of Egg Albumin. Using

sulfur as a reagent for the determination of SH groups in egg albumin, the SH content of native egg albumin was found to be 0.60 in terms of cysteine per cent. After denaturation with NaOH and KCN the SH content was found to be 1.29 cysteine %. This later value accounts for all of the cysteine plus cystine present in egg albumin. Assuming a molecular weight for egg albumin of 40,000, the above values indicate the presence of two SH groups and one S-S group in the egg albumin molecule.—JOHN D. GUTHRIE and JOSEPH ALLERTON. *Contrib. Boyce Thompson Inst.*, 12 (1941), 103-109. (B. M. B.)

Thiamine—Determination of, by the Thiochrome Reaction. A study of the thiochrome method for determining thiamine showed that, by the procedure developed, extraction and hydrolysis of the sample may be carried out in the same vessel, thereby eliminating any error due to transfer of the extract inherent in previous methods. For the enzymatic hydrolysis of cocarboxylase, the enzyme clarase has been introduced. From a study of the oxidation of thiamine to thiochrome, it was found that the most important factor was the amount of sodium hydroxide employed. One cc. of a solution of sodium hydroxide-potassium ferricyanide containing 0.45 Gm. of sodium hydroxide and 0.002 Gm. of potassium ferricyanide gave an optimal oxidation of thiamine to thiochrome for solutions of thiamine chloride containing from 0.5 to 2.0 mgm. of the vitamin. These amounts of sodium hydroxide and potassium ferricyanide gave an optimal oxidation of the thiamine to thiochrome, irrespective of the pH of the thiamine solution or of the presence or absence of potassium chloride. The optimal conditions for the extraction of the thiochrome formed in the oxidation of thiamine chloride solutions containing 0.5 to 2.0 mgm. of the vitamin have been found to be a 1-min. mechanical shaking in the presence of 20 cc. of iobutyl alcohol. For amounts of thiamine chloride ranging from 0.5 to 2.0 mgm., the conversion of thiamine to thiochrome is approximately 67% under the conditions studied in this paper. The method is in close agreement with biological assays, and has been applied to various types of natural products.—R. T. CONNER and G. J. STRAUB. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 380-384. (E. G. V.)

Tissue Water—Studies on. I. The Determination of Blood Water by the Distillation Method. A method is described for the determination of water in blood by distillation with toluene. The analysis requires one hour and has a reproducibility of 0.2%. The results are uniformly higher than by the oven drying method (2.3% for whole blood, 1.6% for plasma and 3.3% for cells (calculated)). The sources of error in the oven drying method are discussed and reasons are given for believing that lower results by this method are due to incomplete extraction of water.—A. T. MILLER, Jr. *J. Biol. Chem.*, 143 (1942), 65. (F. J. S.)

Tobacco Mosaic Virus—An Analysis of, for Biotin, Riboflavin and Pantothenic Acid. The presence of riboflavin, pantothenic acid and biotin could not be demonstrated in significant amounts in moderately concentrated solutions of purified tobacco mosaic virus.—HERBERT SPRINCE and EMANUEL B. SCHOENBACH. *Proc. Soc. Exptl. Biol. Med.*, 49 (1942), 415. (A. E. M.)

α -Tocopherol and α -Tocopherylquinone—Interrelation of. A procedure for isolating α -tocopherylquinone from reaction mixtures has been developed which provides a method of preparing pure synthetic α -tocopherol.—M. TISHLER and N. L. WENDLER. *J. Am. Chem. Soc.*, 63 (1941), 1532. (E. B. S.)

Urinary 17-Ketosteroids—Elimination of Errors in the Colorimetric Assay of Neutral, by Means of a

Color Correction Equation. Evidence is presented which shows that interfering chromogenic substances may cause significant and variable errors of over-estimation in the colorimetric assay of neutral urinary 17-ketosteroids. Except in unusual instances, these errors may be largely eliminated by means of a simple color correction equation without preliminary chemical purification of the crude neutral extract.—N. B. TALBOT, R. A. BERMAN and E. A. MACLACHLAN. *J. Biol. Chem.*, 143 (1942), 211. (F. J. S.)

Vitamin A and Carotene—Estimation of, in Human Blood. The methods of estimating vitamin A and carotene in human plasma are discussed. The reasons for using a photoelectric colorimeter for the measurement of the colors are given. A parallel series of results obtained by the use of four different methods of extraction shows that the simple extraction from serum or plasma precipitated with alcohol is the most satisfactory. A preliminary series of normal readings is given.—SIMON YUDKIN. *Biochem. J.*, 35 (1941), 551. (F. J. S.)

Vitamin A Esters—Purifying. A process of purifying a concentrate of a carboxylic acid ester of vitamin A which contains impurities of the kind normally present in a reaction mixture from an esterification of vitamin A alcohol or normally present in a vitamin A concentrate derived from a fish oil, involves extracting the impurities from the concentrate by bringing it into contact with methanol or alcohol and separating the alcohol containing the extracted impurities from the concentrate being purified, the impurities present being substantially soluble in methanol or alcohol.—KENNETH C. D. HICKMAN, assignor to DISTILLATION PRODUCTS, INC. U. S. pat. 2,249,525, July 15, 1941. (A. P.-C.)

Vitamin A in Oils—Spectrophotometric and Biological Assay of. The details of a spectrophotometric method of assay of vitamin A in fish liver oils and the results of the biological and physical assay of 53 such oils are recorded. In all cases assays were made on fresh oils and all biological assays were made on the whole oils. The average conversion factors computed from the measurements on 22 cod liver oils yield values of 2700 and 2370 for the unsaponifiable fractions and whole oils, respectively. The average conversion factors for oils of higher potency are 2260 for tuna liver oils, 2250 for halibut liver oils, and 2270 for miscellaneous oils as listed, giving an average of 2260 for these oils of higher potency. Studies on the U. S. P. reference standard have shown that the *E* value gradually decreases when the oil remains in partially filled bottles, even though they have been flushed with carbon dioxide and stored in a refrigerator.—N. H. COY, H. L. SASSAMAN and A. BLACK. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 74-76. (E. G. V.)

Vitamin A in Shark and Saw Fish Liver Oils. The writer describes a spectrophotometric and a tintometric method which was applied to 24 Indian fish liver oils and their non-saponifiable fractions to determine vitamin A contents. The ratio I. U./blue values averaged 53, with less variation in the ratio when non-saponifiable matter was used. The ratio, blue values/ $E_{1\text{ cm}}^{1\%}$, 328 $\mu\mu$, was found to be 23.6 for whole oils and 28.5 for non-saponifiable matter. The color in the tintometric method using SbCl_3 was on the average 1.60 times more intense on the non-saponifiable matter. Thirty-seven samples of shark and saw fish liver oils assayed, respectively, 13,600 and 8000 I. U. per gram.—K. RAJAGOPAL. *Indian J. Med. Research*, 29 (1941), 575-583. (W. T. S.)

Vitamin A Synthesis. A process employed involves condensing α -ionylideneacetaldehyde, in the

absence of oxygen and in the presence of a salt of a secondary amine such as piperidine acetate, with β -methyl-crotonaldehyde, to ϵ -(β -ionylidene)- β -methylsorbalddehyde, and reducing the aldehyde group of the latter to the alcohol group by the action of aluminum isopropylate. Various details of the procedure are described.—RICHARD KUHN and COLLIN J. O. R. MORRIS, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,233,375, Feb. 25, 1941. (A. P.-C.)

Vitamin B Complex—Different Constituents of, in Tikitiki. Rice bran contains other vitamins of the B complex in addition to thiamine (vitamin B₁). These are riboflavin (B₂) 100 gammas, pyridoxine 60 gammas (B₆), nicotinic acid 100 gammas and pantothenic acid 13 Filtrate Factor value.—PATROCINIO VALENZUELA, JESUSA CONCHA and ESTER ANASTACIO. *Rev. Filipina Med. Farm.*, 32 (1941), 52. (G. S. G.)

Vitamin B—Effect of, on Urine of Arakawa Negative Mothers. In mothers with negative Arakawa's reaction, the administration of vitamin B will shift the reaction toward positive, and increase the whole day chlorine excretion and the urine output. In the mothers with positive Arakawa's reaction, the administration will make the positive reaction possibly more intense, and may cause a small increase of the whole day chlorine excretion and the urine output. Specific gravity will generally be made lower except for cases in which it is already low before the administration of vitamin B. This will further prove that human milk with positive Arakawa's reaction is generally a good milk, and that Arakawa negative milk is generally a bad milk. The general impression may be expressed in this way: Arakawa negative mother + vitamin B = Arakawa positive mother.—H. UMEMURA. *Tôhoku J. Exp. Med.*, 39 (1940), 227. (A. C. DeD.)

Vitamin B Factors—Inadequacy of a Synthetic Diet Supplemented with All Known. When fed a basal diet containing extracted casein, sucrose, salts, cod liver oil and butter fat together with supplements of thiamine chloride, riboflavin, pyridoxine, nicotinic acid, calcium pantothenate, choline hydrochloride, ascorbic acid, inositol and *p*-aminobenzoic acid, young mice grew at a good rate but did not preserve a clean and good looking fur. Variation of the constituents of the diet did not produce improvement but fresh beef liver immediately increased the growth rate and improved the pelage.—ELIZABETH TROESCHER-ELAM and HERBERT M. EVANS. *Proc. Soc. Exptl. Biol. Med.*, 48 (1941), 549. (A. E. M.)

Vitamin B₁. 2-Methyl-4-amino-5-thioformamido-methylpyrimidine is subjected to the action of 2-methyl-2-hydroxy-3-halotetrahydrofuran in the presence of salts of weak bases with strong acids such as pyridine hydrochloride or dimethylaniline hydrochloride or the like (numerous examples with details being given).—ZOLTAN FOLDI and ARPAD GERECs. U. S. pat. 2,252,921, Aug. 19, 1941. (A. P.-C.)

Vitamin B₁—Amino Analog of. Two analogs of vitamin B₁, one having a cyano and the other an amino group in the side chain of the thiazole nucleus have been prepared. The latter was assayed and found to be inactive.—D. PRICE and F. D. PICKEL. *J. Am. Chem. Soc.*, 63 (1941), 1067. (E. B. S.)

Vitamin B₁—Chemical Methods for the Determination of. Accurate and rapid chemical methods are available for the determination of vitamin B₁. The Prebluda-McCollum reagent used in the manner prescribed by Melnick and Field is practical for such determinations on materials of moderate and high potency. The thiochrome method, having considerably greater sensitivity than the colorimetric methods, may also be used for the assay of

materials of low potency. The use of the synthetic zeolite, Decalso, as a preliminary step in both procedures effectively eliminates interfering materials. Recent modifications of the thiochrome method have made it suitable for routine analysis.—D. J. HENNESSY. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 216-218. (E. G. V.)

Vitamin B₁—Inactivation of, by Raw Fish. Foxes fed raw fish develop a deficiency disease which was proved to be caused by a destruction of vitamin B₁ in the food mixture. The level of inactivation depends on the time that the fish is in contact with the B₁ containing food factors. The reaction seems to be of enzymatic nature.—E. H. SPITZER, A. I. COOMBS, C. A. ELVEHJEM and W. WISNICKY. *Proc. Soc. Exptl. Biol. Med.*, 48 (1941), 376. (A. E. M.)

Vitamin B₁—Recent Advances in Our Knowledge Concerning. A review of vitamin B₁ divided into the subheads: (1) isolation and identification; (2) nomenclature; (3) principal effects of deficiency; (4) cause of deficiency; (5) increased demand; (6) physiological role; (7) relationship with thyroid; (8) interaction with acetylcholine; and (9) therapeutic uses.—N. C. DATTA. *Indian Med. Gaz.*, 76 (1941), 615-619. (W. T. S.)

Vitamin B₂ (Riboflavin)—Determination of. A comparison has been made of four methods for determining vitamin B₂ (riboflavin): biological rat growth; visual fluorescence; photoelectric fluorescence; and microbiological, by both culture turbidity and acidimetry. The four methods gave similar results; the greatest differences were with the low-potency samples. In a further study the microbiological method showed excellent specificity and reproducibility. The results were almost identical whether measured as culture turbidity at the end of 24 hrs. or acidimetry at the end of 72 hrs.' incubation.—A. D. EMMETT, O. D. BIRD, R. A. BROWN, G. PEACOCK and J. M. VANDENBELT. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 219-221. (E. G. V.)

Vitamin B₆. A process is employed which involves treating ethoxyacetylacetone and cyanoacetamide to form 3-cyano-4-ethoxymethyl-6-methyl-2-pyridone, hydrolyzing the latter compound to form the lactone of 3-carboxy-4-hydroxymethyl-6-methyl-2-pyridone, treating the latter with nitric acid to form the lactone of 3-carboxy-4-hydroxymethyl-5-nitro-6-methyl-2-pyridone, chlorinating to form the lactose of 2-chloro-3-carboxy-4-hydroxymethyl-6-methylpyridine, reducing the latter to form the lactone of 3-carboxy-4-hydroxymethyl-5-amino-6-methylpyridine, diazotizing the latter compound to form the lactone of 3-carboxy-4-hydroxymethyl-5-amino-6-methylpyridine and reducing the latter (as by acetic acid and sodium amalgam) to form vitamin B₆.—STANTON A. HARRIS, assignor to MERCK & Co. U. S. pat. 2,248,078, July 8, 1941. (A. P.-C.)

Vitamin B₆—Chemistry of. I. Tautomerism. Chemical and absorption spectra data are given for vitamin B₆ reactions. Methylation of the nitrogen atom of vitamin B₆ destroyed its biological activity.—S. A. HARRIS, T. J. WEBB and K. FOLKERS. *J. Am. Chem. Soc.*, 62 (1940), 3198. (E. B. S.)

Vitamin B₆—Estimation of, in Urine. S.'s method for estimating vitamin B₆, published in 1940, consists of absorption on Fuller's earth, elution, removal of impurities, reabsorption, elution and diazotization for colorimetry. By the use of this method with five human subjects, S. found that absorption and elimination of vitamin B₆ is rapid, with the normal daily output ranging from 400 to 560 mgm., and that about 5% of a test dose is eliminated within 3 hrs.—M. SWAMINATHAN. *Indian J. Med. Research*, 29 (1941), 561-566. (W. T. S.)

Vitamin C—Isolation of, from Lemon Juice. Lemon juice is decitrated and the vitamin C is precipitated out with a copper-free basic lead compound such as the basic carbonate and acetate. Oily impurities are extracted from an acid solution of the vitamin C precipitate by means of butyl alcohol. Ethanol is added to the extracted solution to precipitate insoluble physiologically inert lead salt, and after separating the salt acetone is added to produce a further substantially inert precipitate, which is filtered off. The filtrate is evaporated to dryness with enough barium carbonate to neutralize the acid therein. The dry product is extracted with a non-reactive solvent (*e. g.*, methanol, ethanol, propanol, ethyl acetate, acetone and combinations of these with petroleum ether and dioxane) and the physiologically active principle is reprecipitated. The dry product is extracted with acetone, the acetone is evaporated, the residue is taken up in normal propanol and the active principle is crystallized and separated after addition of petroleum ether.—CHARLES G. KING and WM. A. WAUGH. U. S. pat. 2,233,417, March 4, 1941. (A. P.-C.)

Vitamin D Content of Some Indian Fish Oils. Cod liver oil imports to India have almost ceased. The vitamin D content of the body and liver oils of 10 Bengal fish, mainly sawfish and shark, is reported on. The oils were assayed by the "line test" technique based on the effect of substances to promote calcification in the bones of young rats on a rachitogenic diet. Most of the oils examined contained about the vitamin D content of "Finest Norwegian" cod liver oil. These oils are about 8 to 10 times as rich in vitamin A as is B. P. cod liver oil. The author suggests the Bengal fish oils be diluted with respect to their vitamin A content and then fortified with calciferol to yield a product comparable in strength to that ordinarily prescribed.—S. RANGANATHAN. *Indian J. Med. Research*, 29 (1941), 699-702. (W. T. S.)

Vitamin D—Photocolorimetric Determination of. The Tortelli-Jaffé reaction, which is the most specific one for vitamin D, yields an absorption spectrum with bands at 545 to 550 and 590 to 600 μ . The reaction obeys Beer's law, and may therefore be used for the quantitative photocolorimetric determination of vitamin D.—V. L. SOLIANIKOVA. *Biokhimiya*, 4 (1939), 483-491; through *Chimie & Industrie*, 44 (1940), 51. (A. P.-C.)

Vitamin D—Preparation and Stabilization of. A process of recovering a vitamin from materials containing a provitamin involves activation, extraction and distillation steps, and comprises adding a stabilizer consisting of sugar amines or aliphatic acid salts thereof, which stabilizer does not decompose in the presence of ultraviolet light and does not substantially absorb the same prior to the final distillation step.—HAROLD W. ELLEY and JAMES WADDELL, assignors to E. I. DU PONT DE NEMOURS & Co. U. S. pat. 2,234,554, March 11, 1941. (A. P.-C.)

Vitamin D—Spectroscopic Method for the Quantitative Estimation of. To 0.2 cc. of a solution of vitamin D containing oil in chloroform in a 1-cm. special spectroscopic cell are added 3.8 cc. of a saturated solution of antimony trichloride in freshly purified chloroform. The transmission curve (or absorption curve) in the visible region of the spectrum is then determined in the Hardy color analyzer in such a way that the maximum at 620 μ is reached exactly 3 min. after mixing. A second curve is then run, so that the maximum at about 500 to 520 μ is reached 10 min. after mixing. Finally a third curve is run 30 min. after mixing. In a second method a quantity of the fish liver oil in the neighborhood of 1 Gm. is accurately weighed and saponified in the usual manner. To remove the greater

portion of the sterols, the dried residue is then dissolved in about 5 cc. of pure methyl alcohol and the mixture is cooled to -10° to -15° C., then filtered at this temperature. The alcohol is completely removed from this filtrate, the residue is dissolved in about 5 cc. of freshly purified 1,4-dioxane (free from peroxides), and to it is added about 0.2 Gm. of maleic anhydride in about 2 cc. of dioxane. The maleic anhydride is more effective if it is freshly distilled in a moderate vacuum from a 1 to 1.5 mixture of maleic acid and phosphorus pentoxide. The dioxane mixture is heated on the water bath for 1 hr., then cooled, 10 cc. of a 0.5 N alcoholic potash are added and it is allowed to stand at room temperature for 5 to 10 min. Fifteen cubic centimeters of water are then added and the mixture is extracted several times with pure ether; the ether extracts are washed twice with water, dried over anhydrous sodium sulfate and filtered, and the ether is removed under reduced pressure. Finally, the vitamin D in the residue is estimated spectrophotometrically.—N. A. MILAS, R. HEGGIE and J. A. RAYNOLDS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 227-231. (E. G. V.)

Vitamin K Activity of Quinones—Additional Observations on. A naphthocopherol and its quinoid oxidation product have been assayed for minimum active dose.—E. FERNHOLZ, H. B. MACPHILLAMY and S. ANSBACHER. *J. Am. Chem. Soc.*, 62 (1940), 1619-1620. (E. B. S.)

Vitamin K Preparations. In preparing a vitamin K preparation, a solution containing vitamin K is brought into contact with an activated carbon having an acid reaction, to adsorb the vitamin. Various examples are given, with operative details.—STEFAN ANSBACHER, ERHARD FERNHOLZ and MILDRED L. MOORE, assignors to E. R. SQUIBB & SONS. U. S. pat. 2,233,279, Feb. 25, 1941. (A. P.-C.)

Vitamin Distillates Resistant to Oxidation During Storage. High-vacuum distillation such as that of a fish oil is effected with an admixture of a natural oil containing a protective anti-oxidant substance, such as corn oil. Various operative details are described.—KENNETH C. D. HICKMAN and JAMES G. BAXTER, assignors to DISTILLATION PRODUCTS INC. U. S. pat. 2,221,692, Nov. 12, 1940. (A. P.-C.)

Vitamins—Continuous Extraction of, from Fish Livers and Fish Liver Oils. A vitamin-containing oil is saponified with an aqueous alcoholic solution of sodium hydroxide whereby a soap containing vitamins is obtained. The resulting soap solution is mixed with a water-miscible alcohol and the solution is substantially saturated with dichloroethylene. There is added to a quantity of dichloroethylene an amount of the alcohol which is equal to approximately 3% of the dichloroethylene. The soap solution is passed unidirectionally through the dichloroethylene which remains substantially stationary in a retarded flow attended by repeated contacts between the soap and the dichloroethylene through a determinate screw threadlike path, and with continuously increasing subdivision of the soap, thereby affording increased surface contact.—LOUIS FREDMAN and HARRY E. DUBIN, assignors to U. S. VITAMIN CORP. U. S. pats. 2,248,619 and 2,248,620, July 8, 1941. (A. P.-C.)

ANALYTICAL CHEMISTRY

Acidimetric Titrations—Use of the Photoelectric Cell in. Experiments in titrating various acids with different indicators show that the color change that takes place at the end point can be detected accurately by means of the photoelectric cell provided certain precautions are taken. Color-blind chemists should find this method of detecting the end-point very satisfactory. The Hellige cell was used. The

comparison solution should have the same concentration of indicator as the solution titrated and water should be added to it as the titration progresses. Good results were obtained with litmus, phenolphthalein and tropeoline 00. To titrate a mixture of a strong and a weak acid, it is recommended to titrate both with phenolphthalein as indicator and then, in another portion, titrate the strong acid with tropeoline 00. The results were accurate to within 0.5 mg.—R. MEURICE and A. DEVUYST. *Ann. Chim. Anal.*, 22 (1940), 61-67; through *Chimie & Industrie*, 44 (1940), 18.

(A. P.-C.)

Alkaline Peroxides—Determination of the Activity of, in the Regeneration of Air. An apparatus has been devised and is described which measures the oxygen evolved from a given amount of peroxide when a given volume of a mixture of gases which closely approximates exhaled air is passed through the sample.—M. SARTORI. *Ann. Chim. Applicata*, 29 (1939), 386-391; through *Chimie & Industrie*, 43 (1940), 655.

(A. P.-C.)

Analytical Balances—Simple Tests to Indicate the Condition of. Procedure are given for determining whether or not a balance is in satisfactory condition.—L. C. KREIDER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 117-118.

(E. G. V.)

Analytical Chemistry—Systematic Development of Atomic Groups in. Attention is drawn to the simultaneous presence of salt-forming groups: —OH, —SH, =NH, =NOH, —COOH, —SO₂H, —SO₃H and groups giving rise to complexes: =CO, —CS, —NH₂, =NOH, —NO, —NO₂. General rules regarding certain atomic groups and their use in internal complex salt formation are reported, based on Feigl's work. Among the reactions discussed is the detection of Ag⁺, Hg⁺⁺ and Cu⁺⁺ ions with 2-thiohydantoin and its condensation products. Lead ions do not react with the latter compounds, which give highly colored precipitates with the above mentioned ions. Reactions of dianiline-guanidines with Ag⁺, Pb⁺⁺ and Cu⁺⁺ salts are discussed. The selective affinity of gallocyanine for Sb⁺⁺⁺ in acid solution is pointed out. The reactions of carminic acid, hematoxylin, mercapto-benzthiazol and mercapto-benzimidazol with metallic ions are also included.—J. V. DUBSKY. *Mikrochemie*, 28 (1940), 145-172.

(R. H. B.)

Arsenate of Thallium and Silver—Potentiometric Study of the Formation of a Double. The AsO₄ ion can be determined as the double arsenate of silver and thallium, Ag₂TlAsO₄ by potentiometric titration of a solution of thallium and silver with the solution to be tested in presence of alcohol. The quantity of thallium present must be in a ratio higher than 1Tl:2Ag. The sensitivity of the method is increased by increasing the excess of thallium and the concentration of alcohol.—G. SPACU and C. DRAG-LESCU. *Bull. Sect. Sci. Acad. Roumaine*, 22 (1939), 172-179; through *Chimie & Industrie*, 44 (1940), 18.

(A. P.-C.)

Bismuth—Volumetric Determination of, and of the Acidity of Solutions of Bismuth Salts. It is difficult to neutralize free acid in a solution of bismuth salt because during the neutralization fresh hydrogen ions are formed by hydrolysis. If, however, a bismuth nitrate solution containing only a slight excess of nitric acid is treated with excess sodium thiosulfate, the bismuth ions are converted into Bi(S₂O₃)₃ anions which are not hydrolyzable. Free nitric acid can then be neutralized by titrating with potassium hydroxide to a methyl red end-point. Too much nitric acid should not be present at the start or there is likely to be decomposition of the thiosulfate and precipitation of some bismuth sulfide. If now the neutralized solution containing potassium nitrate is titrated with potassium hy-

droxide to a phenolphthalein end-point, the following reaction takes place: 2Bi(S₂O₃)₃ + 5OH + NO₃ → BiO·NO₂·BiO₂H → 6S₂O₃ + 2H₂O and this titration will serve for the determination of the bismuth. To apply this method of analysis to a solid sub-nitrate, oxide or sulfide, dissolve the sample in as little nitric acid as possible, add a few drops of methyl red indicator solution and titrate with potassium hydroxide until the indicator begins to show a yellow color. Now add about 10 Gm. of sodium thiosulfate crystals and add a little dilute nitric acid very carefully until the precipitate of bismuth salt dissolves. When the precipitate shows a very slow rate of solution, add a few drops of the acid at a time and wait for some time before adding fresh acid, and in this way use very little excess acid after a permanent pink color of the methyl red is reached. Then titrate back carefully with potassium hydroxide solution. Now add 10 drops of phenolphthalein indicator solution and titrate slowly and carefully, particularly toward the last. The results obtained with a sample of 0.365 Gm. of bismuth oxide were within 0.5% of the truth.—L. MALAPRADE. *Ann. Chim. Anal.*, 22 (1940), 5-8; through *Chimie & Industrie*, 44 (1940), 105.

(A. P.-C.)

Boric Acid—Determination of, in Burov's Solution. The following procedure is proposed: Pipette 25 cc. of sample (sp. gr. 1.022 at 25° C.) into 75 cc. water in a 500-cc. Erlenmeyer flask. Add 3 cc. of phenolphthalein as indicator. Titrate to an end-point immediately with 0.5 N NaOH. Cool to 25° C. and retitrate to a faint pink end-point with 0.5 N NaOH (a blank may also be used). Disregard burette readings on the first three end-points since they are not titrations of the acid but of the aluminum acetate present. Add either 10 Gm. mannitol or 150-cc. glycerin and swirl the flask until dissolved. Titrate with 0.5 N NaOH comparing with a blank. A blank must be run on the glycerin or mannitol and subtracted from the boric acid titration. Calculations are: with glycerin and a 25-cc. sample, $\frac{\text{cc. alkali} \times N \times 0.2717}{\text{sp. gr. of the soln.}} = \% \text{ H}_3\text{BO}_3$;

with mannitol and a 25-cc. sample, $\frac{\text{cc. alkali} \times N \times 0.2688}{\text{sp. gr. of the soln.}} = \% \text{ H}_3\text{BO}_3$.—MAX R. COLLINS. *Bull. Natl. Formulary Committee*, 10 (1942), 42-44. (H. M. B.)

Boron Determination in Volatile Organic Compounds. The Parr oxygen bomb is satisfactory for the combustion of organic boron compounds in preparation for the determination of boron. It provides a means for decomposing organic boron compounds without the use of large amounts of reagents, and gives equal or greater accuracy than some methods heretofore proposed.—W. M. BURKE. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 50-51.

(E. G. V.)

Bromine Addition Number—Determination of Weigh 0.100 to 1.000 Gm. of sample, depending on the degree of saturation, into a 25-cc. Erlenmeyer flask and to it add 5 cc. of chloroform. Volatile samples are best measured (0.1 to 1.0 cc.) into 5 cc. of chloroform, and the weight of the sample calculated from its specific gravity. Titrate the sample directly (no indicator) with bromine reagent (bromine, 2% by volume, in C.P. glacial acetic acid, keep in a dark, glass-stoppered bottle), adding it until a distinct orange-yellow color is obtained which persists for about 15 sec. With a little experience it is possible to add the bromine solution rapidly at first and in 0.05-cc. portions toward the end of the titration. The end-point is obscure with some dark samples. In these cases, add the bromine reagent slowly, especially when approaching the end-point, and add with a glass rod one drop of the mixture to about 1 cc. of starch-iodide solution in a spot plate cavity. The appearance of a blue color

marks the end-point. This latter procedure is recommended only for dark samples with which the end-point cannot be otherwise ascertained. Determine the titer of the bromine reagent by introducing 5 cc. of the bromine solution into 25 cc. of 10% potassium iodide solution and 5 cc. of C.P. chloroform in a 250-cc. iodometric flask, and titrating promptly with 0.1 *N* thiosulfate in the usual manner, using starch as indicator toward the end of the titration. Calculations: 1 cc. of 0.1 *N* thiosulfate is equivalent to 7.992 mg. of bromine: (mg. of Br per cc. of Br reagent \times cc. of Br reagent consumed \times 100)/wt. of sample in mg. = bromine addition number.—K. UHRIG and H. LEVIN. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 90-92. (E. G. V.)

Cadmium—Rapid and Accurate Determination of. A solution containing cadmium-zinc salts, and freed from lead, copper and iron, is reduced to a volume of 50 cc., if previous manipulations have resulted in excess volume. The original sample is so taken as to give 20 to 50 mg. of cadmium in the solution. It may be necessary to take aliquot portions of the original solution to get this correct cadmium content. For every milligram of cadmium believed present, 1.5 cc. of a brucine sulfate solution are added, followed immediately by 1.5 cc. of a 10% potassium iodide solution. The solution is well stirred and allowed to stand for 10 min., then filtered rapidly through a Büchner funnel using suction. The precipitate is first washed with a 50-50 mixture of the brucine and iodide solutions, then with an organic solvent such as a 1 to 4 mixture of ethanol and toluene until free from potassium iodide. It is then dissolved by heating in water and the iodine content of the organic iodide is determined in any convenient manner. The cadmium content of the sample may be determined by comparison with the iodine value of standards previously determined.—T. L. THOMPSON. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 164-165. (E. G. V.)

Calomel—Determination of, in Pharmaceuticals. Several methods were suggested for the determination of calomel in Compound Cathartic Tablets and Pills and other preparations containing calomel. Method I consists of oxidation of a given sample, using potassium chlorate, to remove all organic material. The mercury was then precipitated by saturating the solution with hydrogen sulfide; the precipitate thus obtained was filtered off, washed, dried and weighed as mercuric sulfide. The amount of calomel in the sample is given by, $HgS \times 1.0146 = \text{calomel}$. The various laboratories objected to this method chiefly because of the time required and also because of the extreme caution which must be exercised in carrying out the potassium chlorate oxidation in the presence of organic material. Method II is a volumetric procedure based on the residual titration of excess *N*/10 iodine solution with *N*/10 sodium thiosulfate using starch as the indicator. Both procedures were found to give good results, but the latter method was favored by several laboratories.—REPORT OF THE SUBCOMMITTEE ON ALKALOID AND DRUG STANDARDS. *Proceedings, American Drug Manufacturers Association, Thirtieth Annual Meeting, May (1941), 176-178.* (N. L.)

Carbon and Hydrogen—A Simplified Method for the Determination of, in Organic Compounds. I. A method for the microdetermination of carbon and hydrogen in organic substances is described, which differs from the Pregl apparatus in the following manner: (1) the absorption tubes are shorter than the Pregl type, enabling them to be weighed rapidly by vertical suspension on the balance pans; (2) the combustion tube contains a 35-mm. copper gauze roll, the combustion of the substance being achieved mostly by the action of the oxygen current. Satis-

factory results were obtained using benzil, benzoic acid and succinic anhydride as test substances. II. The preceding method is extended to include substances containing nitrogen, halogens and sulfur. The nitrogen oxides were trapped in a U-tube placed between the two usual ones for water and carbon dioxide, and containing aminoazobenzene, boric acid and potassium dichromate. This tube changes color when exhausted. When halogen and sulfur compounds are to be determined, the filling in the combustion tube is packed with silver wool, cupric oxide, lead chromate and reduced silver in the order named. If nitrogen, halogens and sulfur are to be determined simultaneously, the above plus the aminoazobenzene tube is employed.—ANNA DOMBROWSKI. *Mikrochemie*, 28 (1940), 125-135, 136-140. (R. H. B.)

Carbon and Hydrogen—Wet Combustion Method for Determining. Solubility and volatility are the dominant factors in a wet combustion. Wet combustion with iodic acid is best adapted to materials known to be readily oxidized by this reagent, though any organic substance can be oxidized if it is retained in the oxidizing medium for a sufficient time. Modifications of methods for determining carbon dioxide and unreduced iodate are given.—B. E. CHRISTENSEN and R. WONG. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 444-446. (E. G. V.)

Carbon Monoxide—Detection of Very Small Quantities of, in Gas Mixtures. A method for the determination of small amounts of carbon monoxide by means of palladous chloride colorimetry is described. It is shown from a number of tests with palladium chloride paper in mixtures of city gas and air, of hydrogen, carbon dioxide, carbon monoxide and air, and in flue gases, that a proper gradation of blackening can be obtained with palladium chloride papers without addition of gold chloride. In the presence of the latter the color tends to be purple and inconclusive below carbon monoxide concentrations of 0.1%. It is furthermore pointed out that the blackening is more intensive if hydrogen is present in the gas mixture, least intensive in dilute city gas. The palladium chloride paper was exposed to the gases for 20 min., then washed out in dilute hydrochloric acid to remove excess chemical, dried and compared with standards made up under similar conditions. The lowest concentration used was 0.0125% carbon monoxide.—P. J. G. NELL. *Helv. Chim. Acta*, 23 (1940), No. 2, 18-21; through *Chimie & Industrie*, 44 (1940), 29. (A. P.-C.)

Celandine—Identity of the Substance Responsible for the Formation of Microchemical Crystals on Treatment with Potassium Hydroxide. In a previous paper, the author has shown that powdered celandine gave characteristic crystals when treated with a few drops of 20% potassium hydroxide solution, a reaction which may be used to identify the powdered drug. Preliminary investigation showed that neither the individual pure alkaloids of celandine nor a combination of them gave the reaction. The substance responsible for the test was shown to be water soluble and was obtained in 0.6 Gm. yield from 200 Gm. of drug as follows. The powdered drug was exhausted with wet ether and the solvent removed. The fatty mass was extracted several times with hot water. Extraction of the water with ether removed a phenolic substance. Concentration of the water solution gave a mass of fine yellow crystals. The crystals were purified as the silver salt. Analyses on the silver salt indicated a composition in agreement with that calculated for chelidonic acid, a substance reported in celandine a hundred years ago. A sample of the purified material was decomposed with alkali and the acetone identified. The oxalic acid obtained was in good agreement with that calculated and the presence of

chelidonic acid thus confirmed.—EGIL RAMSTAD. *Pharm. Acta Helv.*, 16 (1941), 40. (M. F. W. D.)

Ceric Sulfate—Use of, in Pharmaceutical Analysis. The use of ceric sulfate (with ferroin indicator) in place of potassium permanganate in determinations of the iron content of reduced iron, ferrous sulfate, dry ferrous chloride, ferrous lactate, ferrous tartrate, Blaud pills and syrup of ferrous iodide, and for determination of the nitrite content of sodium nitrite, was studied. Three or more determinations on each type of chemical were made and averaged. In some cases the results were compared with results obtained by methods of the Swed. Phar. X. It was noted that ferrous lactate or tartrate, Blaud pill and syrup of ferrous iodide, or chloride, could not be determined directly by permanganate or dichromate titrations but could be analyzed by ceric sulfate titration. Sixteen literature references were cited. *Method for reduced iron:* 0.500 Gm. of specimen and 2.5 Gm. of fine powdered mercuric chloride were mixed by stirring in a 100-cc. volumetric flask. Fifty cubic centimeters of boiling water were added and the flask warmed 30 min. on the water bath with frequent stirring. Then it was filled to the mark with freshly boiled, cooled water and cooled to 20° C. for the final adjustment of volume. It was shaken and the mixture was allowed to settle. The clear supernatant solution was filtered through a dry filter discarding the first 15 cc. To 25 cc. of filtrate were added 150 cc. of water, 10 cc. of concentrated H₂SO₄, 5 cc. of concentrated H₃PO₄ and a drop of ferroin solution. The solution was titrated to faint blue color with N/10 ceric sulfate solution.—T. CARLSSON. *Farm. Revy*, 40 (1941), 441, 457, 469, 477. (C. S. L.)

Chemical Glassware—Comparative Tests of. Four brands of glassware were tested: (1) Tamworth-Glasbake (McKee Glass Co.); (2) Kimble No. N5la; (3) Pyrex (Corning 774); and (4) Vycor (Corning 790). (4) had the lowest coefficient of linear expansion, then came (3), (1) and (2); resistance to thermal shock was in the same order with (4) showing the greatest resistance. The comparative resistances to water and acid reagents is in the order (4), (2), (3) and (1), while the resistance toward alkaline reagents in (4), (2), (1) and (3), and the order of resistance to nearly neutral reagents is (4), (2), (3) and (1). The resistance to mechanical shock is in the order (1), (3) and (2). Compositions of the various glasses are given.—E. WICHERS, A. N. FINN and W. S. CLABAUGH. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 419-422. (E. G. V.)

Chloroform—Rapid Determination of, in Air. The chloroform contained in a given volume of air is burned in contact with incandescent platinum in a normal combustion furnace. The halogen is collected in sodium arsenite and carbonate solution and titrated electrometrically by means of silver nitrate.—K. BEYER. *Biochem. Z.*, 302 (1939), 287-293; through *Chimie & Industrie*, 43 (1940), 905. (A. P.-C.)

Cobalt—Determination of, as Trioxalatocobaltiate. The basis of the procedure is the measurement of the absorption at 605 m μ due to the trioxalatocobaltiate ion, Co(C₂O₄)₃, which is produced by oxidation of a cobaltous solution by lead dioxide in a weakly acid solution of potassium oxalate.—G. H. CARTLEDGE and P. M. NICHOLS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 20-21. (E. G. V.)

Colorimeter—Rapid-Operating. The instrument, designed to remedy the defects of the Stammer instrument, comprises a series of wooden strips colored in tones of yellow, yellowish brown and brown characteristic of beet juices; the Stammer color values corresponding with the color tones are also marked on the strips.—H. ERCHLER. *Deut.*

Zuckerind., 65 (1940), 118; through *J. Soc. Chem. Ind.*, 60 (1941), 47. (E. G. V.)

Copper—Determination of Electrolytic. The microcerimetric method described is based on the finding that electrolytic copper can be stoichiometrically oxidized with excess ceric sulfate in 2 N sulfuric acid when the proper amount of chloride ion is present. The excess ceric sulfate can then be titrated with Mohr salt, using *o*-phenanthroline ferrous complex as an indicator. A fine-grained adherent copper deposit may be obtained by electrolysis at 2.0 volts from a 2 N sulfuric acid bath when the bath is 0.8 N to 1.0 N in nitric acid. This concentration of nitric acid prevents interference of other metals when electrolysis is conducted at this voltage.—L. H. BRADFORD and P. L. KIRK. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 64. (E. G. V.)

Copper—Estimation of, in the Presence of Iron.—P. L. KAPUR and BADAR-UD-DIN. *J. Indian Chem. Soc.*, 18 (1941), 585. (F. J. S.)

Copper in Plant Materials—Determination of. A method for the determination of copper in plant materials is proposed which involves the use of the dropping mercury electrode. Copper is determined in the presence of all the constituents ordinarily present in plant ash except those that are removed by addition of a slight excess of ammonium hydroxide. No interference is offered by any of the cations or anions likely to be found in plant ash even when present in comparatively large quantities. Copper added to plant materials as copper sulfate could not be recovered by any of the dry-ashing methods used. A wet-ashing procedure was adopted involving final solution in sulfuric acid. Limits of the method using a 1-Gm. sample of plant material are from 0.2% or greater to 0.0002% of copper in the plant.—J. F. REDD and R. W. CUMMINGS. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 124-127. (E. G. V.)

Cupric Ion—A New Microchemical Reaction Specific for the. On a white porcelain plate place 1 drop of alcoholic sodium sulfate solution, 2 drops of a concentrated solution of β -naphthylamine in 96% alcohol, and 1 drop of the solution to be analyzed. In presence of copper there is immediately formed a reddish-brown precipitate having a composition corresponding to the formula Cu(C₁₀H₇NN₂)₂·2SO₄.—P. G. SPACU. *Bull. Sect. Sci. Acad. Roumaine*, 22 (1939), 162-164; through *Chimie & Industrie*, 44 (1940), 18-19. (A. P.-C.)

D. A. K. Monographs—New. The Danish Apothecaries Society Control Laboratory has issued monographs citing standards and tests for betaine HCl, hexynal (ethylcyclohexenyl barbituric acid), enhexynal (methylcyclohexenyl-N-methyl barbituric acid), hexylresorcinol, phenantoin (5,5-diphenylhydantoin), phentiazine (thiodiphenylamine), stilbestrol dipropionate, sulfathiazole, vasimid (2-benzyl-4, 5-imidazole HCl) and formulas and directions for making, nicotinamide solution for injection, nalactol (a powder of lactic acid and sodium lactate), suppositories of aminophylline with benzocaine, enhexynal tablets, hexenal tablets, Nicotinamide tablets, phenantoin tablets, sulfa-pyridine tablets, sulfathiazole tablets.—ANON. *Arch. Pharm. Chemi*, 48 (1941), 301. (C. S. L.)

Drop Reactions—Interferences Occurring with Selected. A systematic study of interferences occurring with selected drop reactions has been made. The investigation dealt with tests for 38 elements. Special attention is given to the types of interferences encountered and the term "positive interferences" is applied to interferences giving false tests. Interferences which inhibit a true positive test are designated by the term "negative interference." The data obtained in this investigation are tabulated and applied to a study of the

two-group sodium peroxide-sodium carbonate system of analysis. The effect of this separation on the elimination of interferences is noted.—L. SMITH and P. W. WEST. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 271-275. (E. G. V.)

Ethylene Glycol Determination in and Removal from Alkyl Ethers of Diethylene Glycol. Samples of commercial diethylene glycol monoethyl and mono-methyl ethers have been shown to contain 28% and 8.5% of ethylene glycol, respectively, by either of two independent methods, ditrityl ether formation or lead tetraacetate oxidation. A solvent partition method was devised for removing this glycol, since fractionation is relatively ineffective.—M. K. SEIKEL. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 388-389. (E. G. V.)

Extract of Licorice—Analysis of Dried. Lehman's method (*Deutsche Apoth. Zeit.*, 88 (1934), 1425) for the determination of the glycyrrhizic acid content of dried extract of licorice was studied. Results, using three strengths of alcohol for extraction, determinations of the glycyrrhizic acid and of the alcohol insoluble residue, showed that as the alcohol content of the menstruum decreased the yield of glycyrrhizic acid increased, while the amount of alcohol-insoluble residue decreased. Two methods of determining alcohol-insoluble residue were compared: Lehman's (*loc. cit.*) and Madsen's (*Arch. Pharm. Chemi.*, 47 (1940), 130). A table gave results for 6 preparations of the extract both with and without drying to constant weight. Determinations included glycyrrhizic acid content, alcohol-insoluble residue, ash content and water-insoluble residue. The range of variation in glycyrrhizic acid content was small, 19.6-22.0%. The amount of alcohol-insoluble residue was far larger by Madsen's method than by Lehman's method, and quite variable in different preparations (range, Madsen, 19.7-38.8%). Water content of the extracts varied between 13.05-16.25%; ash, 6.9-8.9%. No extract gave positive test for copper. Water-insoluble residue varied between 0-11.6%.—N. THORN. *Farm. Revy*, 40 (1941), 509, 525, 541. (C. S. L.)

Ferric and Ferrous Iron. Using precipitated copper as a reducing agent, determinations of ferrous and ferric iron are carried out in less than 10 min., with a minimum of equipment and skill, with common stable reagents, in the presence of large amounts of cupric, ferrous, manganous, zinc, nickel and chromic ions, and with a precision and accuracy of around 3 parts per thousand.—J. O. PERCIVAL. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 71-72. (E. G. V.)

Fluorine—Determination of Dietary. In determining fluorine in the presence of large amounts of organic matter, a closed system of ashing prevents the loss of fluorine by volatilization. An improved still allows separation of fluosilicic acid in a single distillation. The perchloric acid carried over may be 0.00015 *N* and does not interfere with the titration. The interference of sodium ion may be due to the formation of $\text{ThF}_4 \cdot \text{NaF}$, precipitation of the lake and effect of change in ionic strength on alizarin. At *pH* greater than 3.5 there may be error due to free alizarin in the red form. At *pH* less than 3.5 the thorium lake does not show its full color; therefore the thorium must be standardized with fluoride at exactly the same *pH* at which the titration is made. The back titration on a blank distillate after adjusting *pH* with the glass electrode allows this to be accomplished, as well as correcting for fluorine in reagents. From 2.5 to 10 mgm. of fluorine added to the sample may be recovered with an error of 5%. The greater percentage inaccuracy with smaller quantities may be due to fluorine in the reagents and to the uncertainty of the end-point. It appears that the color of the

thorium lake is not entirely suppressed by adding the equivalent of fluoride. The method is considered valuable in determining 1 mgm. of fluorine in foodstuffs (error, 7%).—J. F. McCLENDON and W. C. FOSTER. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 280-283. (E. G. V.)

Formaldehyde—Determination of, with 5,5-Dimethylcyclohexanedione-1,3. Very accurate results can be obtained in the determination of formaldehyde with dimethylcyclohexanedione if the precipitation is carried out in a sodium acetate-hydrochloric acid buffer solution at *pH* 4.6. In such a solution containing a 10% excess of reagent, only 0.05 mg. of formaldehyde remains unprecipitated in 300 cc. of solution and 1 mg. each of acetaldehyde and propionaldehyde do not precipitate in this same solution. Twelve hours or longer should be allowed for the precipitation to take place, during which time the solution should be shaken occasionally. After being filtered through a sintered-glass filtering crucible and washed with distilled water, the precipitate may be dried to constant weight in several hours at 60° C. If an acid or alkaline solution of formaldehyde is to be analyzed, the sample is first neutralized to the purple color of bromphenol blue or to the yellow color of nitrazine indicator. The resulting solution is buffered at *pH* 4.6 and the precipitation carried out as before.—J. H. YOE and L. C. REID. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 238-240. (E. G. V.)

Furfural As a Solvent. Solubilities of various salts and organic acids in furfural are tabulated.—F. TRIMBLE. *Ind. Eng. Chem.*, 33 (1941), 660-662. (E. G. V.)

Gaseous Hydrocarbons—Microanalysis of. A procedure has been developed applying Pregl's microanalytical method for the determination of carbon and hydrogen to the analysis of gaseous hydrocarbons obtained in very small quantities. By introducing about 2 cc. of the gas into the combustion tube by means of a burette inserted into the Pregl train and slightly modifying the well-known procedure, extremely good results are obtained. As in the ordinary microdetermination of carbon and hydrogen in organic substances, the water and carbon dioxide are weighed. From these the weights of carbon and hydrogen are calculated and the sum of the two is obviously equal to the weight of the sample taken. This weight makes it possible not only to determine the percentage composition but to calculate the molecular weight of the hydrocarbon from the relation of the weight of the sample to the volume of gas taken.—L. MARION and ARCHER LEDINGHAM. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 269-271. (E. G. V.)

Infrared Spectroscopy—Application of, to Industrial Research. Apparatus, sample preparation and methods are described.—N. WRIGHT. *Ind. Eng. Chem., Anal. Ed.*, 13 (1941), 1-8. (E. G. V.)

Iodide in the Presence of Interfering Substances—Determination of. Determination of iodides in heterogeneous mixtures has always presented difficulty. Pills of ferrous iodide were chosen for the study. The work presented included an investigation of the efficiency of methods that have been proposed and development of an accurate quantitative procedure as applied to ferrous iodide pills. Methods studied included those of Fullerton, Watkins and Graham, Sheringa, Kolthoff and a modification. The method worked out is described and the apparatus is illustrated. It is based on the release of iodine with ferric chloride in a specially designed distillation apparatus. A modification of Kolthoff's method gave satisfactory recoveries of iodine but consumed considerable time.—KIMIO F. SHIMIZU and EDGAR A. KELLY. *J. A. Ph. A.*, 31 (1942), 103. (Z. M. C.)