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PHARMACEUTICAL ABSTRACTS

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ABSTRACTORS

CONTENTS

BACTERIOLOGY (*Continued*)

Staphylococci—Bactericidal Power of Blood from Patients and Normal Controls for. The authors have examined the bactericidal power of bloods of 25 patients with staphylococcal lesions of different kinds (18 had osteomyelitis) and of 18 healthy individuals as controls. Thirty-one strains of staphylococci were used of which 17 were recovered from the patients included in this study while the remaining 14 were isolated from other patients. All but one strain produced *aureus* pigment, all but two were coagulase positive, all but two fermented mannitol and all produced a zone of clearing on a rabbit blood agar plate. The bactericidal power of the blood of both patients and controls was low, only very small inocula of bacteria being killed in most instances. No significant difference was observed between the bloods of the two groups and no correlation existed between the clinical condition of the patient and the bactericidal power of his blood. In some cases it appeared that the blood had a higher bactericidal power for heterologous than homologous organisms and a few strains appeared to be unusually susceptible to all bloods. Of these one was an exceptionally powerful toxin producer while two others did not fulfill all the usual criteria of pathogenic strains. These points are being further investigated.—W. W. SPINK and J. R. PAINE. *J. Immunol.*, 38 (1940), 383; through *Bull. Hyg.*, 15 (1940), 629. (T. C. G.)

Streptococcic Infections—Chemotherapy of. Sulfanilamide used in puerperal fever reduces the temperature and limits the infection promptly. In erysipelas it banishes the inflammation and edema but must be continued to complete cure, since there is a possibility of relapse. Sulfanilamide is also effective in both meningococcic and streptococcic meningitis. It gives better results in meningococcic infections if it is combined with serum. In gonococcic infections its action is very efficient for blenorrhagia, in acute urethritis and chronic urethritis and prostatitis. It should be given in courses with rest periods between but continued sufficiently long to prevent reinfection. Cases in women are slower to clear than in men. Benzylsulfanilamide is equal to, and in some cases superior to, sulfamido-crysoidin, and equal in activity to sulfanilamide; it is five times safer than sulfanilamide and ten times better than sulfamido-crysoidin. The relatively safe doses are: benzylsulfanilamide 10 Gm. per Kg. orally, sulfanilamide 40 mg. per 20 Gm. weight is the most that should be given. Sulfamido-crysoidin is even more toxic than sulfanilamide. For *p*(γ-phenyl-propyl-amino)-phenyl-sulfamide-α-γ-disulfonate of sodium the tolerance is 75 to 100 mg. per 20 Gm. Side effects are the production of sulfhemoglobinemia and metahemoglobinemia, with nausea, vomiting and irritation of urinary tract. None of these drugs has bactericidal action in the absence of oxygen, but may be bacteriostatic in anerobic cultures. They have no direct action on leucocytes or bacteria.—M. H. GIUNTI. *Rev. Col. Farm. Nac., Rosario*, 6 (1939), 152. (G. S. G.)

Streptococcus Infections—Chemotherapy for. A series of tests *in vivo* and *in vitro* were made to study the bacteriostatic properties of forms of sulfonamides. Prontosil and rubiazol were tested and also the various components of *p*-amino-phenyl-sulfamide. Some were inactive, some slightly active but none equalled the direct action of *p*-amino-phenyl-sulfamide itself for sterilizations of microörganisms.—M. H. GIUNTI. *Rev. Col. Farm. Nac., Rosario*, 6 (1939), 25. (G. S. G.)

Sulfanilamide. Antienzymatic Nature of Its Bacteriostatic Action. The free amine group of sulfanilamide when incompletely oxidized becomes poisonous for catalase, as a result of which the toxic hydrogen peroxide produced by many pathogenic organisms is permitted to accumulate in their cultures. In addition to catalase, other enzymes are adversely affected by the intermediate oxidation products of the sulfonamide compounds. As known so far, they are peroxidase, certain dehydrogenases and nitrotase. The nutrition of the bacteria is thus seriously affected and bacteriostasis results. The effect of such bacteriostasis against pneumococci *in vivo* is to bring about their "dissociation" into culture phases, whose principal metabolic functions, including hydrogen peroxide formation and virulence, are so critically suppressed that the organisms fall easy prey to the destructive action of the phagocytes.—R. R. MELLON, A. P. LOCKE and L. E. SHINN. *Am. J. Med. Sci.*, 199 (1940), 749–759. (B. H.)

Sulfanilamide Derivatives—Action of, in Streptococcal and Pneumococcal Infections in Mice. This paper describes the pharmacological examination of 17 sulfanilamide derivatives which were prepared with the following views: (*a*) to obtain a superior drug to sulfanilamide; (*b*) to elucidate the factors that govern the acetylation and hence the rate of excretion of these compounds; (*c*) to give an insight into the mechanism of their action. Some of the 17 compounds were substituted in the amino portion so that this radical could undergo neither oxidation nor acetylation. In the others the amido group was substituted with different types of acid groupings. 2-*N'*-Sulfanilamidothiazol showed a striking action in streptococcal and pneumococcal infections in mice, comparable to that of M. & B. 693 which suggest the need for a pyridine or probably a heterocyclic ring in the sulfanilamide part for strong activity against pneumococci. The Schiff base from sulfanilamide and vanillin in 10-mg. doses was less toxic than, and equal to, sulfanilamide in streptococcal infections. 4-Amino-5-(4'-sulfonamidophenylazo) uracil and its sulfur analog were less toxic and nearly as effective as sulfanilamide. The activity of the balance of the compounds ranged from half that of sulfanilamide to nil. 6-*N'*-Sulfanilamidoquinoline proved extremely toxic with a dose of 2.5 mg. killing one out of two mice in each experiment. The relationship of chemical constitution to therapeutic action as revealed by a study of these compounds is pointed out.—K. GANAPATHI. *Indian J. Med. Research*, 27 (1940), 971–978. (W. T. S.)

Sulfanilamide Derivatives. IV. N^1,N^4-Diacylsulfanilamides and N^1-Acylsulfanilamides. A large series of N'-acylsulfanilamides was prepared from aliphatic, aromatic, heterocyclic and carbocyclic acids. As intermediates, N^1,N^4 - diacylsulfanilamides were prepared in which the N^4-acyl group was usually acetyl. Salts of both series of compounds with various cations also were prepared. Preliminary pharmacological results indicated that N^1-dodecanoylsulfanilamide was effective in mice against infections by beta hemolytic streptococci and arrested the spread of tuberculosis infections in cavies.—M. L. CROSSLEY, E. H. NORTHEY and M. E. HULTQUIST. *J. Am. Chem. Soc.*, 61 (1939), 2950. (E. B. S.)

Sulfanilamide, Sulfapyridine, Sulfathiazole and Sulfamethylthiazole in Experimental Gas Gangrene in Guinea Pigs. The protection of the drugs in *Clostridium welchii* infections is rather insignificant. Sulfamethylthiazole and sulfathiazole had a slight therapeutic advantage over the other compounds. —ALFRED B. LONGACRE and EDITH HONOLD. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 9. (A. E. M.)

Sulfapyridine—Action of, upon Pulmonary Lesion of Experimental Pneumococcal Pneumonia. Whereas antipneumococcal serum acts immediately and

stops the spread of pneumococcic lesions by immobilizing the invading organisms through agglutination, sulfapyridine exerts the same effect more slowly through bacteriostasis. In both cases the final destruction of the pneumococci depends on phagocytosis. In animals treated with sulfapyridine, the phagocytic reaction may apparently occur in the absence of the circulating type specific antibodies.—W. BARRY WOOD, JR. *Proc. Soc. Exptl. Biol. Med.*, 45 (1940), 348. (A. E. M.)

Sulfonamides—Bacterial Endocarditis and. A case is recorded of recovery from bacterial endocarditis. Treatment was by sulfapyridine. The diagnostic and therapeutic difficulties are briefly discussed.—C. T. ANDREWS. *Brit. Med. J.*, 4122 (1940), 5. (W. H. H.)

Sweet Wine—Bacterial Spoilage in. The disease is characterized by an increase in acidity, decrease in reducing sugars, hot sharp flavor and the evolution of gas when working actively. It may be prevented by forcing fermentation with activated yeast, addition of 100 parts per million of sulfur dioxide to the crushed grapes, and liberal use of tannin for clarification. Wine contracting the disease in storage should be treated with sulfur dioxide, filtered and pasteurized at 70°.—D. G. QUINN. *J. Dept. Agric. Victoria*, 35 (1937), 335–337; through *J. Soc. Chem. Ind.*, 59 (1940), 312. (E. G. V.)

Testosterone and Androstanedione—Bacterial Hydrogenation of. It has been previously shown that putrefactive bacteria were responsible for the supposed enzymic hydrogenation of androstanedione and testosterone by an extract of stallion testicles. By the biochemical hydrogenation of testosterone by the bacteria growing in a bull testicle extract there has now been obtained, in addition to epietiocholanediol, isoandrostane-3,17-diol, probably because the bacterial mixture was different from that used in the earlier work. Androstane-17-ol-3-one is probably an intermediate product. This assumption is supported by the fact that the bacterial mixture used also hydrogenated saturated 3-oxo derivatives of the androstane series. Thus, androstanedione gave a mixture of androsterone and isoandrosterone. Like the hydrogenation by fermenting yeast, that effected by bacteria is strikingly specific; cholestenone is wholly unchanged under the conditions under which testosterone is smoothly hydrogenated.—L. MAMOLI and G. SCHRAMM. *Ber. deut. chem. Ges.*, 71 (1938), 2698–2701; through *Chimie & Industrie*, 42 (1939), 680. (A. P.-C.)

Tincture of Iodine and Alcohol as Disinfectants in the Operating Field. Tincture of iodine of the old French Pharmacopœia (1 Gm. iodine in 20 Gm. of ethyl alcohol) is preferred but the new formula (10 Gm. iodine, 4 Gm. KI, 126 Gm. ethyl alcohol 95% and 10 Gm. water) is hardly more efficient than pure ethyl alcohol.—E. MARQUIS. *Bull. acad. méd.*, 122 (1939), 305; through *Chem. Abstr.*, 34 (1940), 4526. (F. J. S.)

Tuberculosis—Immunization with BCG and Heat Killed Vaccines against Experimental. In this work 222 guinea pigs were used and were divided into groups in which BCG (subcutaneously or intradermally) or heat killed S or R variants of virulent human tubercle bacilli were given intramuscularly. The heat killed organisms were exposed to a temperature of 100° C., and the S and R variants were dissociated from the well-known strain H37. The dose of BCG was 0.03 mg., that of the heat killed organisms was 0.3 mg., repeated three times. The infecting dose, given on the 58th day after the first immunizing dose, consisted of 0.01 mg. of strain H63. Surviving animals were killed on the 461st day of the experiment. From 25% to 60% of the animals in the various groups died from intercurrent infection (usually due to *Pasteurella bronchiseptica*) before virulent inoculation was performed. All except two animals in the "heat killed" group died of tuberculosis after the first animal showed tuberculosis at necropsy, but 24 of 42 BCG treated animals showed no evidence of this disease. The difference is statistically significant and shows that BCG conferred a significant degree of protection. Heat killed S organisms gave slight protection and R none, but animals inoculated with these variants suffered from intercurrent infection more than the rest.—A. A. LIEBOW, C. G. BURN and W. B. SOPER. *Am. Rev. Tuberc.*, 41 (1940), 592; through *Bull. Hyg.*, 15 (1940), 638. (T. C. G.)

Typhus—Exanthematic Clinical Use of Weil-Felix Reaction in. The method used in cases suspected of typhus is to collect a drop of blood from a finger prick on a piece of filter paper. This is laid over a glass microscope slide and a drop of physiological serum is allowed to fall on it. After a few minutes the paper retaining the red cells is thrown aside and the glass slide containing the drop of serum with the absorbed agglutinins is reserved. Later a drop of rapid-acting antigen (Proteus X19) is mixed with this and the resultant agglutination (if positive) can be observed with the naked eye. It is better to wait at least an hour after obtaining blood before adding the physiological serum in order that there may be an absolute exclusion of red cells. Blood samples should be taken on the first examination of the patient. Confirmatory samples are taken again when the patient is hospitalized. It is important to test all suspects and even if the clinical test is negative it should be confirmed by the central laboratory. This technique is specific.—JORGE JOANNON INFANTE. *Bol. Ofic. Sanit. Panamericana*, 18 (1939), 1134. (G. S. G.)

Urinary Antiseptic. The use of phenothiazine and its oxidation products, diphenylene-*o*-sulfoxide, mono- and di-hydroxythiodiphenylimide, is claimed. —F. DE EDS, J. O. THOMAS and C. W. EDDY, assignors to U. S. SEC. OF AGRICULTURE. U. S. pat. 2,085,794; through *J. Soc. Chem. Ind.*, 59 (1940), 88. (E. G. V.)

Yaws and Syphilis—Simple Serologic Test for. A simple slide test using Kahn antigen is described. It has been compared with Kahn and Wassermann tests on 5390 samples. There is an agreement of 94.8% of all three tests, the Kahn agreeing in 98% and the Wassermann in 95%. This test can be recommended as routine serum diagnostic test for syphilis and yaws especially in provincial hospitals and field dispensaries in which experienced technicians and extensive apparatus are not available. Doubtful or weakly positive results should be checked with either Kahn or Wassermann whenever possible.—R. J. NAVARRO and F. GOMEZ. *J. Philippine Isls. Med. Assoc.*, 19 (1939), 607; through *Rev. Filipina Med. Farm.*, 30 (1939), 529. (G. S. G.)

BOTANY

Chenopodiaceæ—Meaning of Plant Names Associated with. In an article dealing with the meaning of plant names associated with the *Chenopodiaceæ*, the authors have this to say about *Chenopodium anthelminticum:* Chenopodium is from the Greek *chen*, meaning a goose, and *pous*, a foot, in reference to the shape of the leaves in some species; and the specie name *anthelminticum* refers, of course, to its use as a vermicide. This species is called "Jerusalem Oak" and more appropriately "stinking weed." The origin of the names of numerous other species of this family, consisting of some 75 genera, are given.—WILLARD N. CLUTE. *Am. Botanist*, 46 (1940), 113–119. (W. T. S.)

Hemicellulose Preparation—Influence of Alkaline Treatments and Pretreatments in. Using teak sawdust as the raw material, and from the evidence of important reductions in furfuraldehyde yield, it is shown that boiling alcoholic and aqueous NaOH solutions exert degradative effects on hemicelluloses. Pretreatment with boiling alcoholic NaOH may reduce the lignin content of subsequent hemicellulose preparations, but the disadvantages of the pretreatment outweigh its advantages. In view of the limited solvent action of cold 4% NaOH, it may not be possible at present to dispense with boiling extraction in all cases. The influence of concomitant substances on furfuraldehyde yield from hemicelluloses is noted.—ISAAC ARTHUR PREECE. *Biochem. J.*, 34 (1940), 251. (F. J. S.)

Photocalorimeter. The Quantum Efficiency of Photosynthesis in Algæ. A small calorimeter is described capable of measuring a rate of heat change as small as a millionth of a calorie per second. It is designed to admit light and thus give a measurement of heat changes accompanying photochemical reactions. The thermal efficiency of photosynthesis by green algæ has been measured. About four-fifths of the red light absorbed in living algæ is converted into heat, leading to a quantum yield of about 0.08 molecule per quantum absorbed.—J. L. MAGEE, T. W. DEWITT, E. C. SMITH and F. DANIELS. *J. Am. Chem. Soc.*, 61 (1939), 3529. (E. B. S.)

Photosynthesis—Quantum Efficiency of, in Chlorella. II. Quantum yields in photosynthesis by algæ have been measured under a variety of conditions, making use of the dropping mercury electrode for the rapid measurement of the dissolved oxygen. The results show that at 25°, 0.04–0.1 molecule of oxygen is evolved in photosynthesis by chlorella per quantum of red light absorbed. This efficiency is much lower than the efficiency 0.25 which has been hitherto accepted.—H. G. PETERING, B. M. DUGGAR and F. DANIELS. *J. Am. Chem. Soc.*, 61 (1939), 3525. (E. B. S.)

Pyrethrum. The Bureau of Agriculture of the Netherlands of East Indies has laid out experimental fields of pyrethrum on the Dieng plateau, near Wonosobo (Central Java). If these prove successful, commercial exploitation on a large scale will be carried out. At present Japan supplies three-quarters of the world supply of pyrethrum.—ANON. *Indian and Eastern Chemist*, 21 (1940), 23. (A. C. DeD.)

Thiamine Chloride, Ascorbic Acid and Phytohormones on Belladonna and Ricinus—Growth Effects of. Report is made of experimental work conducted to determine the specific effects of certain hormone and vitamin-like stimulants singly and in combination upon the entire growth cycle of *Atropa Belladonna* and *Ricinus communis*. Material used is listed and methods described and results shown by means of pictures of the plants. Findings are discussed. It was found that hormone and hormone plus vitamins B and C as dust treatment of seeds influence both germination and growth response, the effect, whether beneficial or injurious varying with concentration, species and age of plants. Using α-naphthalene acetic acid as a solution it was found that the toxic threshold varies with the type of plant. Simultaneous administrations of vitamins B_1 and C gave a combined effect on general growth, not merely additive. It also exhibited an antidoting property toward plants previously subjected to toxic doses of hormones such as α-naphthalene acetic acid. The antitoxic quality is of value in delaying and reducing the degree of epinastic responses of belladonnas about to flower. Photosensitizer in aqueous solution added to soil of plants containing vitamins B_1 and C increases top growth.—LOUIS C. ZOPF. *Jour. A. Ph. A.*, 29 (1940), 487. (Z. M. C.)

CHEMISTRY

GENERAL AND PHYSICAL

Acetophenone—Kinetics of the Thermal Decomposition of. The mechanisms involved in the thermal decomposition of aldehydes and ketones are varied, and the relations between them somewhat complex. In particular an interesting contrast in behavior has recently been found between benzaldehyde and acetaldehyde. An investigation of acetophenone has therefore been made for comparison with acetone. The thermal decomposition of acetophenone takes place predominantly by the step $C_6H_5COCH_3 \rightarrow C_6H_5CH_3 + CO$, the toluene undergoing a subsequent decomposition to give chiefly benzene, methane and carbon. It differs from that of acetone in yielding hardly any ketone. It is homogeneous and nearly of the first order, with no sharp falling off in rate at 20 mm. There is no retardation by nitric oxide or by greatly increased surface, nor can an increased rate of decomposition be induced by the presence of radicals from decomposing diethyl ether. This is taken as evidence for the absence of reaction chains. In this respect the behavior resembles that of acetone, but other differences in kinetics exist. These are briefly discussed.—C. N. HINSHELWOOD, F. R. S. and R. E. SMITH. *Proc. Roy. Soc.* (London) B., 129 (1940), S 65–S 66. (W. T. S.)

Blaugel—Adsorption Properties of, for Vapors. Blaugel (silica gel colored with a cobalt salt) is suitable as a drying agent for the storage of extracts and drugs, and has the additional advantage of adsorbing the vapors of volatile solvents. The maximum amount of water vapor adsorbed by blaugel at 20° is about 32.5% of its weight. The weights of various solvents adsorbed vary directly with their specific gravities, the average volume adsorbed being about 30.8 cc. per 100 Gm. The color change of the adsorbent is not uniform. Some particles turn red rapidly and it is somewhat difficult to set a point at which the gel should be regenerated by heating to 180–200°. One disadvantage of blaugel is that it loses its efficacy after the adsorption of a smaller per cent of water than other dehydrating agents. The rate of adsorption of water vapor and other vapors is tabulated and illustrated in graphs.—K. SEILER. *Pharm. Acta Helv.*, 14 (1939), 125–130. (M. F. W. D.)

Cation Permeability—Influence of the Lyotropic Series of Anions on. The influence of the lyotropic series of anions on sodium or potassium permeability in the cat erythrocyte has been studied. Anions at the dehydrating end of the series, *e. g.*, SO_4, retard potassium permeability and apparently accelerate sodium permeability. Anions at the hydrating end of the series, *e.g.*, CNS, have the reverse effects in the two instances. If the permeability to sodium is measured in isotonic solutions of the potassium salts of the homologous series of straight-chain fatty acids, a small decrease in permeability occurs with each step from acetate to valerate; on passing from valerate to hexoate permeability is completely inhibited. It is argued that the results show that the effects of anions on permeability phenomena are not predictable from a knowledge of their behavior in respect to the hydration of colloidal systems.—H. DAVSON. *Biochem. J.*, 34 (1940), 917. (F. J. S.)

Colloid Mills. A new kind of colloid mill has been developed, in which the grinding and/or dispersing action is produced additionally to rubbing and beating by the action of vibrations of a homorhythmic nature, of high frequency and large

amplitude, mechanically produced in the colloid mill itself, upon the material passed through the mill with periodic interruptions, so that an entirely new principle of colloid milling is involved.—HERMANN PLAUSON. British Patent Specification No. 525,323; through *Perfumer. Essent. Oil Record*, 31 (1940), 313. (A. C. DeD.)

Haschisch—Study of. A study of the adsorption of the resin of cannabis (in petroleum ether solution) by animal charcoal, as a function of resin concentration, of amount of charcoal and of time of contact. The quantity of resin adsorbed is proportional to the quantity of charcoal and to the time of contact, but inversely proportional to the concentration of the solution. The higher the dilution, the greater the adsorption; it is complete for 100 cc. of solution containing 0.5 Gm. of resin, left in contact with 1 Gm. of charcoal for 2 hours.—R. HISAR and S. EDESEN. *Kimya Annali*, 3 (1938), 167–193; through *Chimie & Industrie*, 42 (1939), 561. (A. P.-C.)

Insulin—Structure of. Using an electron density projection of the insulin structure from Crowfoot's *hk*.O x-ray data with a projection of Wrinch's "C_2 octahedron" superimposed, it is shown that there is no indication of a cage structure.—M. L. HUGGINS. *J. Am. Chem. Soc.*, 61 (1939), 2982. (E. B. S.)

Medicinal Carbons—Determination of Absorbent Power of. Absorbent power *in vitro* is not always an accurate criterion of therapeutic absorptive power. Tests were made on various activated carbons, animal and vegetable, with methylene blue, mercuric chloride, strychnine sulfate, antipyrin and sulfuretted hydrogen. Simplified methods were substituted for those in the Brazilian Pharmacopœia. Three techniques are recommended based on the absorption of methylene blue, strychnine sulfate and hydrogen sulfide.—C. H. LIBERALLI and OLAVO FONTURA. *Rev. quím. farm.*, 4 (1939), 101. (G. S. G.)

Oxidation-Reduction Potential—Improved Cell for Measurements of. An anerobic electrode vessel for oxidation-reduction potential measurements is described and three figures are given. It contains provision for the addition of several reagents successively without opening the apparatus and for removing any traces of oxygen which may be present. It is free from the risk of contamination of the solution with substances from the preceding experiments, which is usually associated with built-in salt bridges.—LEON GROTIUS ZERFAS and MALCOLM DIXON. *Biochem. J.*, 34 (1940), 365. (F. J. S.)

Pentothal Sodium as an Anesthetic in Encephalography. Encephalography can be one of the most drastic diagnostic procedures a patient has to undergo; serious symptoms and occasionally death can occur during this operation and after its completion. The development of barbiturates that are both transient and effective in their action has given a satisfactory intravenous anesthesia for encephalography. With pentothal sodium intravenous anesthesia, the one- to two-hour period of post-encephalographic somnolence is very helpful to the patient, as it is during this period that the headaches are the most severe. Other advantages are: The patient is quiet and in good condition throughout the procedure; undesirable reactions are avoided; danger of fire or explosion is eliminated; the drug provides smooth induction with the patients in sitting position; and it eliminates psychic shock. The average dose of pentothal sodium used in this procedure was reported to be 0.85 Gm. in 5% solution.—M. J. NICHOLSON and L. F. SISE. *New Engl. J. Med.*, 222 (1940), 994; through *Abbott Abstract Service*, (1941), No. 798. (F. J. S.)

Separation Operations. A group of symposium papers on the subject, including the following: Electrostatic Separation of Solids, Calculation of Particle Trajectories, Filtration's Future, Sedimentation in the Laboratory, Vacuum and Mechanical Crystallizers, "Krystal" Classifying Crystallizer, Adsorption as a Means of Separation, Flotation as Applied to Modern Manufacture, Froth Flotation Concentration, Separation and Fractionation of Colloidal Systems, Thickening Calcium Carbonate Slurries.—*Ind. Eng. Chem.*, 32 (1940), 599–667. (E. G. V.)

Viscosity. This is a comprehensive article dealing with the measurement of viscosity, classification of lubricating oils, and indices of viscosity. It discusses the advantages of various viscosimeters, those of Redwood, of Hoppler and of McConnel and Schneider. The practical value of the determination of the viscosity of oils is involved with temperature and film in lubricants, with facility of circulation in combustion oils and with absorption in illuminating oils. The index is the relation between variations of viscosity of the different oils and the determined changes in temperature.—RENATO DIAS DA SILVA. *Rev. quím. farm.*, 4 (1939), 78. (G. S. G.)

ORGANIC

Alkaloids

Alkaloids of the Morphine Group—Substitution in the Aromatic Nucleus of. Bromination of morphine and codeine and nitration of codeine produce substitution in the C_1 position. On the other hand, in the nitration of morphine by means of nitrous gases, the nitro group is fixed in C_2 position, *i. e.*, in *o*-position relatively to the hydroxyl. This would seem to be due to the formation, under the influence of the nitrous gases, of a quinone-monoxime having an *o*-quinonic structure, which is subsequently oxidized into an *o*-nitrophenol.—E. OCHIAI and T. NAKAMURA. *Ber. deut. chem. Ges.*, 72 (1939), 684–688; through *Chimie & Industrie*, 43 (1940), 320. (A. P.-C.)

Cinchona Bark Alkaloids—Bromometric Method for the Determination of. The alkaloids are extracted from an ether-chloroform solution into 1% hydrochloric acid and titrated with bromine water (standardized with pure quinine hydrochloride), using rhodamine as indicator. The result is multiplied by 1.6 because related alkaloids present consume less bromine than quinine.—O. IEFIMENKO. *Biokhimia*, 3 (1938), 792–795; through *Chimie & Industrie*, 43 (1940), 410. (A. P.-C.)

Duboisia Myoporoides—Minor Alkaloids of. III. Valeroidine. The hydrobromide of valeroidine (monoiso - valeryl - dihydroxytropane) previously stated to be sparingly soluble in cold chloroform is now shown to be very soluble. The free hydroxyl group has been acetylated and esterified with a second *iso*-valeryl group. The *p*-dihydroxytropane yielded a diacetyl derivative which was characterized as the hydrobromide. The same derivative has been prepared from the dihydroxytropane isolated from Peruvian coca leaves. Chlorination and oxidation of the alkaloid were carried out to determine the positions of the hydroxyl groups but the results were difficult to interpret. Treatment of the hydrobromide of the alkaloid with thionyl chloride produced demethylation to yield norvaleroidine.—WILLIAM F. MARTIN and WILLIAM MITCHELL. *J. Chem. Soc.*, (1940), 1155–1157. (W. T. S.)

Ergotocine—Hydrogenated. Dihydroergotocine, melting point 227–232°, specific rotation at 25°, −71°, obtained by hydrogenating ergotocine or an (organic) salt (maleate) thereof in an inert solvent (acetic acid) in presence of platinum or palladium,

is claimed.—M. S. KHARASCH, assignor to E. LILLY & Co. U. S. pat. 2,086,559; through *J. Soc. Chem. Ind.*, 59 (1940), 173. (E. G. V.)

Magnolia Fuscata Alkaloids. From the leaves of *Magnolia fuscata* there were isolated two new alkaloids—magnoline, $C_{18}H_{21}NO_3$, and magnolamine, $C_{20}H_{23}NO_4$. Both contain a phenolic hydroxyl and a methoxyl group; the nitrogen is combined with the methyl group and seems to be tertiary. Magnolamine is more soluble than magnoline in the ordinary organic solvents.—N. F. PROSKOURNINA and A. P. OREKHOV. *J. Obchtch. Khim.*, 9 (1939), 126–128; through *Chimie & Industrie*, 43 (1940), 410. (A. P.-C.)

Morphine—Electrophotometric Microdetermination of. The morphine is converted into apomorphine by treating the dry morphine residue with 15 drops of sulfuric acid, heating 2 minutes on a boiling water bath, cooling, adding 5 cc. of saturated sodium acetate solution and 2 drops of 4% mercuric chloride solution, boiling and making to 10 cc. The apomorphine is then determined photometrically. The method can determine 0.02 to 0.2 mg. of morphine with a maximum error of 5% for 0.2 mg. and 1% for 0.16 mg.—R. CAHEN and H. FEUER. *Compt. rend. acad. sci.*, 208 (1939), 1907–1910; through *Chimie & Industrie*, 43 (1940), 411. (A. P.-C.)

Opium Alkaloids—Chromatographic Study of. The study deals with morphine, codeine, narcotine and papaverine demonstrating the position of each.—J. R. LEVI and F. CASTELLI. *Arq. Biol.* (S. Paolo), 23 (1939), 263; through *An. Farm. Bioq., Sup.*, 11 (1940), 6. (G. S. G.)

Papaverine—Attempted Synthesis of. An attempt to synthesize papaverine by condensing ω-bromo-6-cyano-3:4-dimethoxystyrene with the Grignard compound of veratryl bromide was not successful due to a failure to obtain the latter compound. Certain pertinent intermediates are described for the first time. Malonic acid condensation of 6-nitroveratraldehyde gave 6-nitro-3:4-dimethoxycinnamic acid (I). Reduction of I by ferrous sulfate and ammonia gave the corresponding amino acid (II). Treatment of II with concentrated HCl and sodium acetate yielded white needles of 6,7-dimethoxycarbostyril, m. p. 229° C. Diazotization of II followed by treatment with NaCN in the presence of $CuSO_4$ yielded the corresponding cyano-acid. Keeping the cyano-acid over bromine in a desiccator for three days followed by refluxing with sodium acetate and subsequent ether extraction gave α,β-dibromo-6-carboxy-3:4-dimethoxyphenylpropionic acid in the water layer and *cis*-ω-bromo-6-cyano-3:4-dimethoxystyrene and the ether layer. It was assumed that this was the *cis* form since the above procedure yielded the *trans* cyano-acid.—J. F. KEFFORD. *J. Chem. Soc.*, (1940), 1209. (W. T. S.)

Salsola Richteri Alkaloids. IV. Optically active salsolidine can exist under two reversible forms. By steam distillation in vacuum it is obtained in a low-melting form (41° to 46° C.), which is converted into a higher melting form (71° to 73° C.) by crystallization from water and drying. From the mother liquor produced in the purification of *l*-salsolidine there was also separated *dl*-salsolidine. The latter fixes carbon dioxide more easily than the optically active forms of the alkaloids.—N. F. PROSKOURNINA and A. P. OREKHOV. *J. Obchtch. Khim.*, 9 (1939), 415–418; through *Chimie & Industrie*, 43 (1940), 411. (A. P.-C.)

Essential Oils and Related Products

Bitter Almond Oil. A Second Addendum to the British Pharmacopœia 1932 was published on June 14th and became official on that day. It contains a monograph on *Oleum Amygdalæ Volatile Purificatum* (Purified Volatile Oil of Bitter Almond). This permits the oil to be made from bitter almonds, peach kernels or apricot kernels, and the benzaldehyde content must be 95% minimum. Characters and tests for identity and purity are described. It is directed that the oil should be kept in a well-closed container, protected from light and stored in a cool place. Appendix XI C amplifies the Pharmacopœia appendix by including a method of determination of aldehyde in the oil.—ANON. *Perfumer. Essent. Oil Record*, 31 (1940), 183. (A. C. DeD.)

Essential Oils and Allied Articles—Industrial Uses of. The many uses of essential oils and their isolates and allied synthetics are listed.—ANON. *Perfumer. Essent. Oil Record*, 31 (1940), 364. (A. C. DeD.)

Ethereal Oils—Refining and Improving the Odor and Taste of, by Fermentation. A process for improving crude terpenes, camphor and peppermint oil or mixtures containing such crude ethereal oils, involves the steps of emulsifying the substances to be improved with a fermenting culture medium for yeast and bacterial acid formers, subjecting the substances to a biological fermentation by yeast or bacterial acid formers, and separating the substances thus improved from the culture medium after the fermentation. A catalyst such as tin or zinc and an oxidizing agent such as ozonized air may be used to facilitate the process, as may also ultraviolet rays. A p_H of 4 to 7 is suitable.—WILLY EKHARD. U. S. pat. 2,184,637, Dec. 26, 1939. (A. P.-C.)

Oil of Petitgrain. The distillation of, factors influencing yields and quantity of Paraguayian oil, handling, constitution and constants are discussed.—ERNEST S. GUENTHER. *Drug and Cosmetic Ind.*, 48 (1941), 38–39. (H. M. B.)

Oils of Lippia Adoensis Hochst. Steam distillation of both fresh and dried flowers of *Lippia adoensis* from different regions in French West Africa yielded 10 to 21 Gm. per Kg of essential oil and 4.2 to 8.2 Gm. per Kg. of *l*-camphor (by cooling to −10° to −15° C.). The decamphorated oil had an optical rotation (in 1 dm. tube) of −31.5° to −46°. Oil obtained from flowers grown at Ziguinchor yielded no camphor on cooling. Drying the flowers (even rapidly) before distilling produces a loss of 30% to 40% of oil and of camphor. Leaves placed in hermetically sealed metal boxes and shipped to France yielded on steam distillation 7.2 Gm. of essential oil and 3.5 Gm. of *l*-camphor per Kg.—E. LAFFITTE and J. RABATÉ. *Chimie & Industrie*, 43 (1940), 365–366. (A. P.-C.)

Oleum Juniperi—New Hydrocarbon from. Previous work by other investigators had indicated the presence of a new hydrocarbon in oil of juniper and the presence of a diuretic hydrocarbon. In the present paper, these two hydrocarbons are shown to be identical and the name junene is suggested. Junene has the molecular formula, $C_{10}H_{16}$, is a volatile colorless liquid, which is rapidly resinified on exposure to air, becoming acid in reaction. It has a strong characteristic odor resembling that of oil of Bergamot. It can be distilled undecomposed in a stream of carbon dioxide. A solution of junene in acetic anhydride, when treated with sulfuric acid, gives a red color. The physical constants of junene indicated a similarity to the hydrocarbon chamene isolated from the oil of the leaves of *Chamæcyparis obtusa*. Chamene gave the same red color with acetic anhydride and sulfuric acid. However, further comparison of derivatives indicated that these two hydrocarbons are not identical. Junene decolorizes solutions of bromine. From the molecular refraction, two double bonds are indicated. Hydrogenation yielded dihydrojunene, $C_{10}H_{18}$, which has a faint odor entirely different from that of junene.

The presence of the second double bond could not be shown by saturation reactions. The experimental details of the isolation of junene, its properties, hydrogenation and its chemical behavior are given. Physiological tests showed it to be diuretic.—P. CASPARIS and W. FREUND. *Pharm. Acta Helv.*, 14 (1939), 1–8. (M. F. W. D.)

Glycosides, Ferments and Carbohydrates

Arginase and Canavanase—Comparative Study of. A comparative study of the enzymes arginase and canavanase from both animal and plant sources has been made. In their kinetics the two enzymes show only such differences (p_H-optimum, optimum substrate concentration and Michaelis constant) as are to be expected from the nature of the substrates. In other respects there is a close parallelism between arginase and canavanase actions. In tissues and blood of different animals they are either present together or not at all and in every case the action on canavanine is slower than that on arginine. By no procedure has it been possible to reverse this quantitative relationship and obtain a preparation in which canavanase activity is stronger than arginase activity. In any particular preparation, factors affecting arginase influence canavanase in the same direction and often to the same extent, *e. g.*, the loss of activity on autolysis and the increase in activity in the presence of cobalt ions affect both enzymes equally. In the presence of cobalt the p_H-optima for the two reactions also become identical at p_H 7.5. The balance of evidence is thus in favor of the two enzymes being identical.—M. DAMODARAN and K. G. A. NARAYANAN. *Biochem. J.*, 34 (1940), 1449. (F. J. S.)

Enzymes—Effect of X-Rays on. The following conclusions are given: (1) The effects of X-rays on crystalline carboxypeptidase and on partly purified polyphenoloxidase have been examined. (2) The percentage inactivation of each of these enzymes is a function of the concentration of the enzyme for a given dose of radiation. It can be shown for carboxypeptidase that a definite amount of radiation energy absorbed corresponds to a constant amount of enzyme inactivated. In consequence the inactivating dose of X-rays decreases with decreasing concentration of the enzyme for a given percentage inactivation to levels far below those normally used in radiotherapy. (3) X-rays have a qualitatively uniform effect on carboxypeptidase over the whole range from very small doses to doses 1000 times as large. (4) Inactivation of carboxypeptidase does not take place when the enzyme is acting on its substrate during irradiation, whereas the enzyme irradiated without its substrate is inactivated to 85%. (5) The biological significance of the experimental results is discussed.—WALTER M. DALE. *Biochem. J.*, 34 (1940), 1367. (F. J. S.)

Fructose—Determination of, in Blood and Urine in Presence of Glucose. For the estimation of fructose, blood is deproteinized with cadmium sulfate and sodium hydroxide and urine is suitably diluted, if necessary treated with lead acetate and trisodium phosphate; 4 cc. of the solution are mixed with 2 cc. of 37% hydrochloric acid and 0.4 cc. of 10% diphenylamine in absolute ethanol; the mixture is immersed in boiling water for 15 minutes and cooled in tap water for 3 minutes; 5 cc. of *n*-propanol are added and shaken; the blue color is read on the Pulfrich photometer with Filter S61. Total sugar is determined by standard methods and glucose is calculated by difference.—R. W. MARTIN. *Hoppe-Seyler's Z. phyisol. Chem.*, 259 (1939), 62–74; through *Chimie & Industrie*, 42 (1939), 965. (A. P.-C.)

Glucose to Gluconic Acid—Fermentation of Concentrated Solutions of. A difficulty encountered in previously described processes for the fermentation of concentrated solutions of glucose—namely, the inhibition of the fermentation by precipitation of calcium gluconate or through injury to the fermenting organism by exposure to free gluconic acid—has been overcome by the addition of boron compounds during the fermentation to prevent the precipitation of calcium gluconate. This use of boron compounds, such as boric acid or borax, with an excess of calcium carbonate permits the continuous neutralization of the gluconic acid formed. With an improved nutrient medium and an organism able to tolerate the amount of boron necessary, it is possible to ferment 20, 25 and 30% solutions of glucose in half the time previously reported. Under these conditions and with a process made semi-continuous by re-use of the mycelium, it is possible to ferment 25% solutions of glucose to gluconic acid every 24 hours. This method is applicable to gluconic acid fermentations by certain bacteria as well as by fungi.—A. J. MOYER, E. J. UMBERGER and J. J. STUBBS. *Ind. Eng. Chem.*, 32 (1940), 1379–1383. (E. G. V.)

Glucosides—Hydrogen Ester Salts of. Water-soluble hydrogen ester salts of glucosides such as digitoxin, *g*-strophanthin and oleandrin are obtained by the action of phosphorus oxychloride on the glucoside (suitably in pyridine solution). Several examples with details are given.—MAX BOCKMÜHL and GUSTAV EHRHART, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,189,723, Feb. 6, 1940. (A. P.-C.)

Ketogluconic Acids from Glucose. The bacterial fermentation of glucose to 5-ketogluconic acid and to 2-ketogluconic acid has been studied in rotary drum fermenters and in vertical vat fermenters. Approximately 90% yields of 5-ketogluconic acid have been obtained from 10% glucose solutions in 33 hours, using *Acetobacter suboxydans* as the bacterial agent. Approximately 82% yields of 2-ketogluconic acid have been obtained from 10% glucose solutions in 25 hours, using an unnamed bacterium as the fermentative agent.—J. J. STUBBS, L. B. LOCKWOOD, E. T. ROE, B. TABENKIN and G. E. WARD. *Ind. Eng. Chem.*, 32 (1940), 1626–1631. (E. G. V.)

Mucin—Chemical Determination of the, Content of Gastric Juice, Saliva and Sputum. A procedure is given for determining mucin in gastric juice, saliva and sputum, which is based on the tendency of mucin to react with iodine. The sample is digested with sodium hydroxide and when the mucin is all in solution, trichloroacetic acid is added and the solution filtered. Acetone is added to the filtrate, which precipitates the mucin. The centrifuged precipitate is dissolved in sodium hydroxide and carefully neutralized to p_H 6.8 in the presence of neutral red and methylene blue. After this, fiftieth-normal hydrochloric acid is added until the color of the solution turns from green to violet. Then two-hundredth-normal iodine solution is added in slight excess and after 15 minutes the excess iodine is determined by titrating with two-hundredth-normal thiosulfate solution, with more methylene blue added until the color changes from greenish to clear blue. The method was standardized with the aid of the nitrogen content of mucin from gastric juice which had been purified by electrodialysis. Tables are given for determining the mucin content from the results of the final titration. The mucin content of 40 samples of gastric juices was found to vary from 0.065% to 0.32%. In the saliva the mucin was found to vary from 0.050% to 0.30%.—J. GLASS. *Mikrochemie (Mikrochim. Acta)*, 26 (1939), 95–112; through *Chimie & Industrie*, 42 (1939), 794. (A. P.-C.)

Papain as a Precipitant of Gums. Experimental work is reported on a continuation of a study of

means of identifying and differentiating gums. Papain was found to be a precipitant for several gums. The reason for the action was not established but possible explanations are discussed.—GEORGE E. EWE. *Jour. A. Ph. A.*, 30 (1941), 19. (Z. M. C.)

Phenothiazine and Sulfanilamide Derivatives—Inhibition of Enzymes by. Phenothiazine was prepared as an insecticide by Smith, *et al.* (*J. Econ. Entomol.*, 28 (1935), 727) and its anthelmintic properties first described by Harwood, *et al.* (*Vet. Med.*, 34 (1939), 440). The actions of phenothiazine, sulfanilamide and certain of their hydroxy derivatives on mammalian enzyme systems have been studied with a view toward explaining the pharmacological action of these substances. Mammalian catalase and cytochrome oxidase are strongly inhibited by the hydroxy derivatives of phenothiazine and by *p*-hydroxylaminobenzenesulfonamide. Phenothiazine, sulfanilamide and sulfapyridine have little or no effect. Cytochrome-c is irreversibly reduced by the hydroxysulfanilamide, as indicated by spectroscopic observation. The inhibitory activity apparently depends on the presence of the hydroxyl group. The relation of these findings to the vermicidal and bactericidal action of the compounds is discussed. Phenothiazine and thionol have no effect on *Ascaris lumbricoides in vitro*.—H. B. COLLIER. *Can. J. Research B*, 18 (1940), 345–350. (W. T. S.)

Strophanthus Glucoside. For the preparation of *k*-strophanthoside, and extract rich in the glucoside is made by treating the seeds of *Strophanthus kombé* with ethanol or with a mixture of ethanol and chloroform, the solution thus obtained being evaporated and the residue treated with an acetylating agent such as acetic anhydride to form a heptaacetyl compound of the glucoside which is then separated by fractional crystallization and saponified by treatment with a non-aqueous solution of an alcoholate such as a solution of barium methylate in methanol, followed by crystallization of the glucoside.—ARTHUR STOLL and JANY RENZ, assignors to CHEMISCHE FABRIK VORM. SANDOZ. U. S. pat. 2,179,204, Nov. 7, 1939. (A. P.-C.)

Sugars and Other Glucids—Microbiological Methods for the Detection of. Two or more different yeasts or bacteria are allowed to act on a solution of the sugar, and the actions of the microorganisms are compared. From the known properties of each of the microörganisms, and more particularly from their ability to ferment various sugars, the presence or absence of any given sugar may be deduced.—A. CASTELLANI DI CHISIMAJO. *Ann. Fac. Méd. Chir. Perugia*, 37 (1938), 1–17; through *Chimie & Industrie*, 43 (1940), 642. (A. P.-C.)

Sugars in Plant Materials—Shaffer-Somogyi Reagent for the Determination of. The Shaffer-Somogyi method (*J. Biol. Chem.*, 100 (1933), 695) offers possibilities for determining sugars in plant materials because it is economical, fast and utilizable on material having a low sugar content. The results from the analyses of 27 materials with the Shaffer-Somogyi and Quisumbing-Thomas methods show fairly close agreement between the fermentable sugar values as determined by the former method and the reducing sugar values as determined by the latter method. The sucrose values obtained with the two methods show close agreement.—T. A. PICKETT. *J. Assoc. Official Agr. Chem.*, 23 (1940), 431–437. (A. P.-C.)

Sugars in Wheat Gliadin. The sugars in wheat gliadin were proved to consist of glucose, fructose and rhamnose by the spectrographic method for oricin reaction. The ratio of sugars differed with varieties and cultural conditions. The ratio of sugars was 1:1:3 for *Triticum monococcum;* 1:1:2 for *T. diococcum* and *T. spelta* and 2:2:1 for the Japanese variety.—KINSUKE KONDO and UITIRO SARATA. *J. Agr. Chem. Soc. Japan*, 16 (1940), 163; through *Chem. Abstr.*, 34 (1940), 4764. (F. J. S.)

Tetra-acetyl-aldehydo-phenylglucosides—Preparation of Some. Certain phenolic glucosides were needed to comparatively study the fate of phenols and glucosides in the animal body. In a note, the author describes a method for preparing these compounds by melting together equal weights of β-glucose penta-acetate and hydroxybenzaldehydes in the presence of a catalyst (anhydrous $ZnCl_2$ or *p*-toluene sulfonic acid). *o*-, *m*- and *p*-Hydroxybenzaldehydes were condensed with the β-glucose penta-acetate in this manner and the properties of the resulting glucosides described.—R. TECWYN WILLIAMS. *J. Chem. Soc.*, (1940), 1402–1403. (W. T. S.)

Trehalose—Mechanism of Fermentation of, by Fusarium Lini Bolley. Unlike living yeast, living *F. lini* Bolley ferments trehalose more rapidly than glucose. Growth of the organism is more rapid and more abundant on trehalose than on glucose. Since the rate of trehalose dissimilation is more rapid than that of glucose the occurrence of the latter as an intermediary is excluded, for, in such a case, the dissimilation rate of glucose would be the controlling step. The indications point toward a direct fermentation of the disaccharide. It was not determined whether there was a trehalase active or present in *F. lini* Bolley.—RITA C. O'CONNOR. *Biochem. J.*, 34 (1940), 1008. (F. J. S.)

Vanguerin, a New Saponin from Vangueria Tomentosa. From the dried-root bark of *Vangueria tomentosa* (a drug possessing a certain anthelmintic activity) a compound was isolated that is difficulty soluble in cold alcohol, more soluble in hot alcohol, very soluble in pyridine and especially in alcoholic potash. It is called vanguerin, $C_{41}H_{64}O_{11}$, and seems to possess a glucosidic structure. By the action of 45% hydrochloric in hydroalcoholic solution it is converted into its genin and a mixture of sugars. Vanguerigenin, $C_{30}H_{46}O_3$, has a triterpenic structure.—K. W. MERZ and H. TSCHUBEL. *Ber. deut. chem. Ges.*, 72 (1939), 1017–1020; through *Chimie & Industrie*, 43 (1940), 410. (A. P.-C.)

Other Plant Principles

Baeckeol—Synthesis of. Ramage and Stowe (*J. Chem. Soc.*, (1940), 425) reported a synthesis of methylphlor*iso*butyrophenone dimethyl ether and showed it to be identical with baeckeol, a phenol obtained from the volatile oils of certain *Myrtaceæ* species. Some earlier synthetic work on the constituents of the anthelmintic drug, kousso, led to a preferable synthesis of this phenol if it should be needed in quantity. Phlor*iso*butyrophenone was refluxed with MeI, Me_2CO and anhydrous K_2CO_3 for two hours. The liquid was allowed to cool, filtered and the filtrate evaporated to dryness. The residue was then recrystallized from MeOH. A mixed melting point of the resulting crystals showed it to be identical with the natural product and with that of Ramage and Stowe.—A. R. HEMS and A. R. TODD. *J. Chem. Soc.*, (1940), 1208. (W. T. S.)

Cannabinol—Synthesis of. From experiences gained from model experiments, it has been possible to synthesize cannabinol (6″-hydroxy-2:2:5′-trimethyl-4″-*n*-amyldibenzopyran) by two routes. Acetylation of 6″-hydroxy-2:2:5′-trimethyl-4″-amyl-3′:4′:5′:6′-tetrahydrodibenzopyran gave the acetate which on heating with palladized charcoal followed by hydrolysis produced a resin resembling cannabinol. This resin yielded a *p*-nitrobenzoate whose melting point at 163–164° C. was undepressed on

admixture with the *p*-nitrobenzoate of authentic cannabinol. Method II. A solution of 5-acetoxy-5'-methyl-7-*n*-amyl-3:4-benzocumarin (0.5 Gm.) in dry anisole (20 cc.) was added to a solution of CH_3-MgI in anisole. The mixture was heated, cooled, decomposed with ice and dilute H_2SO_4, the anisole was removed with steam, the residue extracted with ether, dried and finally distilled gave a resin whose acetate was identical with cannabinol acetate.—R. GHOSH, A. R. TODD and S. WILKINSON. *J. Chem. Soc.*, (1940), 1393–1396. (W. T. S.)

Carotene—Effect of Certain Carbohydrates on the Determination of. In samples containing large amounts of carbohydrate carotene may be determined by extraction with ethanol; if alcoholic potassium hydroxide is used, the material should be subsequently boiled with water to dissolve the resins before extraction of the carotene by fat solvents.—E. J. LEASE and J. H. MITCHELL. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 337–338. (E. G. V.)

Condurite—Obtaining, from Condurango Bark and Its Determination. The method is based on that of Kern and Monsen and comprises hydrogenation of the condurite in presence of a catalyst formed of palladium deposited on animal charcoal. The dihydrocondurite obtained crystallizes easily and its separation offers no difficulty.—W. KERN and W. FRICKE. *Pharm. Zentralhalle*, 80 (1939), 349–352; through *Chimie & Industrie*, 43 (1940), 410. (A. P.-C.)

Curare—Active Principles of. The action of curare depends on two alkaloids present in *Strychnos lethalis* Bard. Rods. and in samples of the poison examined. From their ultimate analysis and molecular weights these new compounds have the formulas $C_{22}H_{27}O_4N$ (strychnolethaline) and $C_{25}H_{31}O_7N$ (curalethaline). They were isolated in the form of silicotungstates. They are readily identified by their fluorescence spectra.—P. DE BERREDO-CARNEIRO. *Bull. soc. chim. biol.*, 21 (1939), 282–293; through *Chimie & Industrie*, 42 (1939), 1025. (A. P.-C.)

Curare—Composition and Preparation of. The preparation of curare is carried out in three stages: exhaustion of the bark with cold water, rapid boiling and concentrating with mild heat. The fresh bark is treated with cold water and extracted for 15 to 20 minutes. This process is repeated eight times with fresh water. The liquid is filtered and brought to a boil. The different fractions obtained are combined and heated gently. The concentration requires 2 to 3 hours, producing a brown-black liquid, reddish by transparence. The curare, nambikwara, is of vegetable nature. One plant is used, a *Strychnos*, a bush about 1 to 2 m. high with branching twigs. Other curares are more complex, with cobra venom or other plant or animal factors added.—J. VELLARD. *Compt. rend.*, 208 (1939), 2104–2106; through *Chimie & Industrie*, 43 (1940), 411. (A. P.-C.)

Drugs—Chemical Characterization of. X. Quinones and substances easily converted to quinones occur widely in the plant kingdom. Ten Gm. of the drug to be tested are heated with 100 Gm. water in a porcelain dish until the volume is reduced to half. The filtrate is treated with an excess of lead acetate. Both the precipitate and filtrate are heated for one-half hour under a reflux condenser with dilute sulfuric acid. The lead sulfate is filtered off and the filtrate distilled after the addition of potassium dichromate. A few cc. of the distillate when treated with dilute sulfuric acid and starch paste give a blue color in the presence of quinones. In those cases in which a blue color was obtained, the remainder of the distillate was extracted with ether and the ether dried. The crystalline deposit was observed under the microscope. Both the lead acetate precipitates and filtrates from 10 drugs were tested for quinones and the results tabulated.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 93–94. (M. F. W. D.)

Drugs—Chemical Characterization of. XI. Thirty-three drugs were tested for volatile bases after steam distillation from an alkaline suspension. The distillate was collected in hydrochloric acid. The distillate was then concentrated to 10 cc. and one portion tested with iodine test solution. In strongly positive tests, Mayer's reagent was used on the other portion. The results of these somewhat crude tests were tabulated.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 123–124. (M. F. W. D.)

Drugs—Chemical Characterization of. XII. The Presence of Phenol-Like Substances in Leaves and Herbs. The test for phenol-like substances was carried out as follows: the aqueous percolate from 100 Gm. of drug was treated with an excess of lead acetate and the filtrate from the lead precipitate then treated with dilute sulfuric acid. The filtrate after the removal of the lead sulfate was then shaken out with ether. The ether solution was washed free of acid with aqueous potassium carbonate solution and then dried over anhydrous sodium sulfate. The residue after the removal of the ether was taken up in 15 cc. water, divided into three portions and tested with ferric chloride, Millon's reagent and an alkaline solution of diazobenzenesulfonic acid. The results for 12 official (Swiss Pharm.) leaves and 6 herbs were tabulated. All of them showed phenolic bodies.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 217–218. (M. F. W. D.)

Insecticide Analysis. Official Methods for Determination of Pyrethrins and Rotenone. A critical review is given of the methods adopted by the A. O. A. C. for determination of pyrethrins-I and -II in pyrethrum powders and commercial pyrethrum extracts in mineral oil, and determination of rotenone in derris and cubé powders.—J. J. T. GRAHAM. *Soap*, 16 (1940), 99, 101, 103; through *J. Soc. Chem. Ind.*, 59 (1940), 306. (E. G. V.)

Isoeugenol and Similar Compounds. An improved method for the manufacture of isoeugenol and other propenyl benzene derivatives is given.—STAFFORD ALLEN AND SONS, LTD. and T. F. WEST. British pat. 525,705; through *Perfumer. Essent. Oil Record*, 31 (1940), 339. (A. C. DeD.)

Lindera Strychnifolia—Constituents of the Root of. III. A check of a similar investigation carried out in 1925 showed that the previous results require correction. Linderal was shown to be identical with *l*-borneal, and the former name should therefore be discarded. In the more recent investigation linderane was found in quantities 5 times greater than previously. Its constitution could not be established. The amount of linderene ($C_{15}H_{18}O$ or $C_{16}H_{20}O$) in the root of *Lindera strychnifolia* is 2.5 times greater than that previously found.—H. KONDO and K. TAKEDA. *J. Pharm. Soc. Japan*, 59 (1939), 162–169; through *Chimie & Industrie*, 43 (1940), 585. (A. P.-C.)

Pinus Pinea L.—Essence of. The essence contains principally two hydrocarbons, lemonene 83.6% and pinene 10.4%. The sesquiterpene fraction 2% of the whole boils at 266° to 270° but gives no additional crystalline products with hydrochloric or hydrobromic acids. The essence of *Pinus pinea* cultivated in Portugal in the highland of Coimbra is practically the same as that of Spain and Italy. Slight physico-chemical differences may be attributed to race or climate or may even be due to a seasonal variation. Frequently samples from the same tree differ in different seasons.—ALOISIO FERNANDES COSTA. *Noticias farm.*, 6 (1939), 39. (G. S. G.)

Purgative Drugs—Constituents of. During the distillation of a fluidextract of black alder, yellow

crystals were deposited in the condenser. The same crystals can be obtained by steam-distilling black alder bark and extracting the distillate with ether. This volatile substance seems to be the aglucone of a glucoside which has been broken down by an enzyme present in the bark. Further investigations are required to establish the composition of the crystals and of the glucoside from which they are derived. It should be noted that cascara sagrada bark also gives a yellow substance under the same conditions. —L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 122–123; through *Chimie & Industrie*, 43 (1940), 585. (A. P.-C.)

Purgatives—Studies on the Composition of. VII. A material volatile with steam was removed from the extract of frangula. The material was yellow, crystalline, easily soluble in ether and chloroform, less soluble in benzene and petroleum ether and slightly soluble in hot water. Preliminary studies indicate that it occurs in frangula bark as a glycoside, has acid character and that it is not hydroxymethylanthraquinone.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 112. (M. F. W. D.)

Rhus Toxicodendron—Effects of Some Adsorbents, Precipitants and Oxidants upon the Resin of. The layman treats ivy poisoning with the fresh juice of some plants, particularly that of *Impatiens biflora*. The active substance in *Rhus toxicodendron* is phenolic. A qualitative study of the effect of adsorbents, precipitants and oxidants upon the physiologically active milky exudate is reported. Calamine and aluminum oxide were very poor. Magnesium oxide was very active but the adsorbate of the ivy phenol was a very active vesicant. Lead acetate and basic lead acetate were the best precipitants found but the precipitates were also very active. Oxidants such as ferric chloride and cupric acetate gave precipitates which were very active. The only successful method studied rendered the phenols inactive by oxidation with alkaline peroxide. —OLE GISVOLD. *Jour. A. Ph. A.*, 30 (1914), 17. (Z. M. C.)

Rotenone in Derris Root—Determination of. The analysis of finely powdered derris root by the ether-extraction method has been compared with the method of analysis proposed by Jones and Graham. The pure rotenone content by the former method was equal to the crude rotenone of the latter. In samples with a fineness of 80% to 90% through a 200-mesh sieve the crude rotenone content by the Jones-Graham method was generally higher. For complete chloroform extraction of samples in which the ratio of rotenone to total ether extract exceeds 40%, a greater fineness than the one given in the Jones-Graham method will be required. Heating derris powder at 60° and 80° C. for definite periods considerably lowered the rotenone and total ether extract contents.—T. M. MEIJER and D. R. KOOLHAAS. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 205–209. (E. G. V.)

Vaccinium Uliginosum L.—Constituents of the Fruit of. The alcohol extract gave quercetin-3-monogalactoside, $C_{21}H_{20}O_{12}.1.5H_2O$, which melts at 235° to 237° C., has a rotatory power at 24° C. of 51.6° (in a 1:1 alcohol-pyridine mixture) and gives a tetramethyl derivative melting at 217° to 219° C.—R. KAWAGUCHI, K. W. KIM and K. MATSUSHITA. *J. Pharm. Soc. Japan*, 59 (1939), 50–51; through *Chimie & Industrie*, 42 (1939), 1226–1027. (A. P.-C.)

Viburnum Stem and Root Barks—Comparative Study of the Total Volatile Acids of. Literature indicates no good reason for changes at various times from stem to root bark so comparative study was undertaken. Details of experimental work are reported and the following conclusions reached: Root bark of *Viburnum prunifolium* contains up to 20% more total volatile acid following hydrolysis than does the stem bark. The proportion and kinds of acids present in the mixture are about the same in the root as compared to stem bark. The greater amount present in the root is due to more acid of all kinds. One sample of rossed stem bark had more total acidity than any sample of root bark but the proportion of lower acids was greater than in unrossed samples. Such treatment may not be desirable. If the amounts and proportions of the mixture of volatile acids present are a measure of activity the difference between stem and root bark of viburnum does not justify making only the root bark official.—IRVIN W. GROTE and CHARLES COLBURN. *Jour. A. Ph. A.*, 29 (1940), 485. (Z. M. C.)

Fixed Oils, Fats and Waxes

Almond Oil. The annual report of the City of Salford analyst for 1939 states that eight samples of oil expresses from different types of bitter almonds were examined to ascertain what variations might be expected.—ANON. *Perfumer. Essent. Oil Record*, 31 (1940), 318. (A. C. DeD.)

Burbot Liver Oil—Chemistry of. The liver of the burbot, a fresh water cod, is much larger than that of any other fish in the same waters and contains from 10% to 56% of oil. A study has been made of this oil and an analysis is reported. The claim that burbot liver oil is superior to cod liver oil therapeutically is being studied and report will be made later.—R. T. LAKEY, F. W. MITTLESTADT and M. G. DENAVARRE. *Jour. A. Ph. A.*, 30 (1941), 18. (Z. M. C.)

Carnauba Wax—Fatty Acids of. Some references are made to the literature dealing with this wax and with separation of homologs of fatty acids. Preliminary experiments showed that about half of the methyl esters of the acids of carnauba wax would not distil without decomposition. So the work reported has reference to the distillable portion. Following is an outline of the procedure: (1) Saponification of the crude wax. (2) Separation of the soap from the non-soap portion. (3) Preparation of the methyl esters from the acids obtained from the soaps. (4) Distillation of the crude methyl esters into distillable and non-distillable fractions. (5) Fractionation of the distillable portion into like-boiling fractions. (6) Division into ten-degree distilling fractions. (7) Division into five-degree distilling fractions. (8) Saponification of the methyl esters into the corresponding acids. (9) Repeated recrystallization, determinations of neutral equivalents and solidification points until definite results were obtained from Schuette and Vogel's curves. Details of experimental work are reported and apparatus is illustrated. Finally a summary is given which indicates what was found in each fraction.—ROY A. BOWERS and A. H. UHL. *Jour. A. Ph. A.*, 30 (1941), 10. (Z. M. C.)

Castor Oil—Composition and Analysis of Dehydrated. Contrary to the common belief that dehydrated castor oil consists mostly of the triglyceride of 9,11-linoleic acid, it is found to contain more of the 9,12-linoleic acid than of the former. The diene values of commercial oils, as well as samples made by various methods in the laboratory, range from 15 to 22, indicating a content of 17.3 to 25.4% of the 9,11 isomer in the glycerides. From these diene values it is calculated that only one-fourth to one-third of the dehydration reaction leads to the formation of conjugated double bonds. Analytical data are listed for a number of commercial dehydrated castor oils, including both raw oils and oils processed to higher viscosities. The latter have lower acetyl values than raw oils; however, it is shown that the designation "completely dehydrated" for the bodied oils, as opposed to the raw oils, is not

justified. An analysis of the distilled fatty acids, which are found to contain a somewhat greater proportion of conjugated double bonds than the oils, is included. The application of the customary analytical methods to dehydrated castor oil is discussed and some changes are recommended.—G. W. PRIEST and J. D. VON MIKUSCH. *Ind. Eng. Chem.*, 32 (1940), 1314–1319. (E. G. V.)

Cetyl Palmitate—Occurrence of, in Corals. Corals of coastal waters contain from 2% to 7% of organic matter, the composition of which is not well known. Acetone extraction of crushed staghorn corals, *Madrepora cervicornis*, gave a 0.25% yield of cetyl palmitate which was identified by saponification to cetyl alcohol and palmitic aid.—DAVID LESTER and WERNER BERGMANN. *J. Org. Chem.*, 6 (1941), 120–122. (W. T. S.)

Chaulmoogra Oil—Chemistry of. The chemical composition of chaulmoogra oil and its ethyl esters is still imperfectly known. But chaulmoogric and hydnocarpic acids are relatively well known and it is to their optic activity that the antileprous potency is attributed, at least empirically. The active acids of chaulmoogra oil are: chaulmoogric, gorlic, hydnocarpic, alepric, aleprilic and alepresticic. Chaulmoogric and hydnocarpic compose 30% to 40% each of the oil, gorlic constitutes 15% and the others make up the remainder. All except gorlic acid are solid at room temperature, soluble in 80% alcohol and 80% acetone and in petroleum ether, from which they may be recrystallized. Gorlic acid is liquid and less stable than its ethyl and methyl esters. All are dextrorotary. Chaulmoogric and gorlic acids each have 18 carbon atoms, but gorlic forms a dehydrochaulmoogric acid, while the others are lesser homologues, having 16, 14, 12 and 10 carbon atoms, respectively.—ALUISIO MARQUES LEAL. *Notic. Farm.* 6 (1939), 50. (G. S. G.)

Drugs—Variation Statistics of. By the same method described in an earlier publication (*Pharm. Acta Helv.*, 13 (1938), no. 7/8), the fat content of Trinité cacao beans was determined. Three beans contained 40–45% fat, 42 contained 45–50% fat, 44 contained 50–55%, 10 contained 55–60% and 1 contained over 60% fat. The results are tabulated. —L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 95–97. (M. F. W. D.)

Fish Oils Containing Vitamins—Vacuum Distillation of. A process of molecular distillation of fish oils to obtain concentrated fat-soluble vitamins involves segregating components distilling up to 170° C., separating a fraction at about 170° to 195° C. and returning it to the fresh incoming oil to be distilled, and separating a fraction high in vitamin content at about 195° to 230° C.—KENNETH C. D. HICKMAN, assignor to DISTILLATION PRODUCTS, INC. U. S. pat. 2,180,356, Nov. 21, 1939. (A. P.-C.)

Germ Oil of Theobroma Cacao L.—Vitamin Content of the. Experiments with rats and comparison with the action of pure β-carotene showed that cacao germ oil possesses vitamin A activity equal to 825 to 1400 International Units per 100 Gm.—K. H. WAGNER and L. SEBER. *Bull. officiel l'office intern. cacao chocolat*, 9 (1939), 147, 149. (A. P.-C.)

Grapefruit Seed Oil. The commercial preparation of grapefruit seed oil as practised in Florida is described and physical characteristics of the crude oil obtained are given. The crude oil has an extremely bitter taste which is probably due to limonin ($C_{26}H_{30}O_8$). The oil can be easily refined, and the refined oil has a bland taste. Physical characteristics of refined and "wintered" refined oils are given.—A. W. NOLTE and H. W. VON LOESECKE. *Ind. Eng. Chem.*, 32 (1940), 1244–1246. (E. G. V.)

Indian Fish Oils. The Fanur Research Station (Madras) reports the discovery of four more native fish oils which possess 3 to 19 times the vitamin potency of cod liver oil. This, and the confirmation that the Malabar sardine contains appreciable vitamin A, will stimulate the application of practical methods for increasing the catch of these fish.—*Indian Med. Gaz.*, 75 (1940), 490. (W. T. S.)

Mineral Oil Mixture Suitable for Medicinal Use. A mineral oil composition which is of good flavor contains a mineral oil together with a small proportion of the mineral oil-soluble constituents derived from a dehydrated, low moisture-containing, salted, macerated olive paste and is substantially free from fibers of the olive paste.—SIDNEY MUSHER, assignor to MUSHER FOUNDATION, INC. U. S. pat. 2,192,866, March 5, 1940. (A. P.-C.)

Mustard Oils. The patentees have found that mustard oils can be obtained by the treatment of chlorolefines in which a chlorine atom is situated in *alpha*-position to an olefine double bond in the liquid phase, preferably in aqueous solution, with thiocyanic acid salts. The reaction proceeds with the formation of mustard oils wherein the thiocyano group is attached in the *alpha*-position with reference to the olefinic double linkage.—I. G. FARBENINDUSTRIE A.-G. British Patent Specification No. 525,136; through *Perfumer. Essent. Oil Record*, 31 (1940), 311. (A. C. DeD.)

Oil of Hydnocarpus Wightiana. Oil of hydnocarpus is used as a substitute for chaulmoogra oil in the treatment of leprosy. Determination of the characteristics of 3 samples from Mauritius showed that they met Brit. Pharmacopœial requirements, except as regards solubility in 90% alcohol, which is explainable by the absence of fixed and volatile fatty acids.—R. LINCOLN. *Rev. Agr. Maurice*, (1939), No. 105, 81; through *Chimie & Industrie*, 43 (1940), 841. (A. P.-C.)

Phosphorus and Iodine Contents of British Columbia Fish Oils. The amounts of phosphorus, iodine and lecithin in the oils from ten species of fish inhabiting the waters adjacent to British Columbia have been determined. To determine phosphorus, a modification of the procedure used to determine total phosphorus in eggs, as described in *Methods of Analyses of Off. Agri. Chem.* was used. Iodine was determined by the method of Mathews, Curtis and Brode (*Ind. Eng. Chem. Anal. Ed.*, 10 (1938), 612). The amounts of phosphorus were generally too low to retard oxidation but the iodine content was generally high, averaging 3200 parts per million.—R. D. HEDDLE and J. S. BRAWN. *Can. J. Research B*, 18 (1940), 386–387. (W. T. S.)

Sunflower Seed—Formation of Oil in, Studied during the Different Stages of the Process. Contrary to the generally admitted theory according to which sunflower seed oil is formed at the expense of the glucids and is distributed exclusively in the protoplasma cells, it was found that the aleurone cells also contain some. In order to obtain a maximum oil yield it is therefore necessary to destroy the latter as well as the protoplasma. On the other hand, the formation of oil in sunflower seed is closely related to the formation of proteins, there even being an inverse relation between the contents of these two constituents.—O. I. SHCHEPKINA. *Uch. Sapiski Biol. Inst. Rostov. Univ.*, 1 (1938), 37–75; through *Chimie & Industrie*, 43 (1940), 680. (A. P.-C.)

Tocopherol Content of Oils—Proposed Modification of Emmerie's Iron-Dipyridyl Method for Determining. Emmerie's method for determining tocopherol is superior to most procedures but interfering substances are a problem. The authors have suggested a modification of this method for use with wheat germ oil. A petrol ether solution of the oil is treated with 85% sulfuric acid, centrifuged and the

supernatant layer washed with dilute alkali. This removes carotenoids and other interfering substances without affecting tocopherol. The tocopherol was then determined colorimetrically with iron-dipyridyl. —W. E. PARKER and W. D. MCFARLANE. *Can. J. Research B*, 18 (1940), 405–409. (W. T. S.)

Unclassified

Absolute Alcohol. Absolute alcohol is a valuable and necessary material in various scientific and industrial uses; and in recent years it has become of increasing importance abroad in admixture with other materials for motor fuels. Interest has also been evinced in this country along these lines. It is produced by an azeotropic distillation with a water insoluble compound, such as benzene, but the high heat cost, the complications and expenses of the necessary equipment and other factors are major disadvantages. A new method based on the use of ethyl ether as the entraining agent has been developed and operated for months in a pilot plant and is now being expanded to industrial scale operations. By its use the steam consumption may be reduced considerably; and the equipment may be greatly simplified because there is no ternary constant-boiling mixture of ether, water and alcohol as there is with all other materials which have been used heretofore. A table shows the relative costs of operation, compared with other methods, as well as the number of plates required in the various distilling columns required by each.—D. F. OTHMER and T. O. WENTWORTH. *Ind. Eng. Chem.*, 32 (1940), 1538–1539. (E. G. V.)

Acetyl-*p*-Phenetidine—Mercury and Bromine Derivatives of. Fusion of 1 molecule of acetyl-*p*-phenetidine with 3 and 4 molecules, respectively, of mercuric acetate gives tris(acetoxymercuri)- and tetrakis(acetoxymercuri) - acetyl - *p* - phenetidine, which both form colloidal solutions in water, probably because of their high molecular weight. Treated with bromine in alkaline solution in the proportion of 3 bromine atoms to each mercury atom, the corresponding tri- and tetra-bromo derivatives of acetyl-*p*-phenetidine are formed. The tris and tetrakis acetoxymercuri derivatives decompose on heating to 155° and 170° C., respectively, while the tri- and tetra-bromo derivatives melt at 165° and 268° C., respectively.—M. RAGNO. *Ann. chim. applicata*, 29 (1939), 148–151; through *Chimie & Industrie*, 43 (1940), 148. (A. P.-C.)

Adermin—Constitution of. Experiments on the synthesis and oxidation of adermin showed it to be identical with 3-hydroxy-4,5-di(hydroxymethyl)-2-methylpyridine.—R. KUHN, G. WENDT and K. WESTPHAL. *Ber. deut. chem. Ges.*, 72 (1939), 310–311; through *Chimie & Industrie*, 42 (1939), 1031. (A. P.-C.)

Adermin—Oxidative Degradation of. Adermin (vitamin B_6) contains a phenolic hydroxyl and two alcoholic hydroxyl groups, and a methyl group attached to a carbon atom. The experiments described showed that it is identical with methylhydroxymethyl-hydroxymethyl-3-hydroxypyridine. The two hydroxymethyl groups are in adjacent positions.—R. KUHN and G. WENDT. *Ber. deut. chem. Ges.*, 72 (1939), 305–309; through *Chimie & Industrie*, 42 (1939), 1030. (A. P.-C.)

Adermin—Synthesis of. 2-Methyl-3-methoxypyridine-4,5-dicarboxylic acid (obtained from 3-methyl-4-methoxy-isoquinoline by oxidation) is converted into 2-methyl-3-methoxy-4,5-dicyanopyridine, then by catalytic hydrogenation into 2-methyl-3-methoxy-4,5-bis(aminomethyl) pyridine. Treatment of the latter with nitrite yields a product that crystallizes as colorless needles melting at 20° C., the hydrochloride of which does not lower the melting point of the methyl ester of adermin. Treatment of the methyl ester with hydrobromic acid followed by substitution of a hydroxyl for the bromine, gives 2-methyl-3-hydroxy-4,5-bis(hydroxymethyl) pyridine, which is synthetic adermin.—R. KUHN, K. WESTPHAL, G. WENDT and O. WESTPHAL. *Naturwissenschaften*, 27 (1939), 469–470; through *Chemie & Industrie*, 43 (1940), 498. (A. P.-C.)

Aerogel Catalysts. A comparison is made of the activities of aerogels, xerogels and precipitates as catalysts for the dehydration of alcohols, the esterification of acetic acid with ethanol and the dehydration and decarboxylation of aliphatic acids. The aerogels were often, but not always, the most active catalysts. The advantages of the aerogel form are found to be of smaller magnitude than was predicted from earlier work.—K. KEARBY and S. SWANN. *Ind. Eng. Chem.*, 32 (1940), 1607–1614. (E. G. V.)

***o*-Alkylsulfonylamides.** The reactions of the sodium mercaptides from ethyl, *n*-propyl and *n*-butyl mercaptans with seven α-haloamides have been carried out and the twenty-one α-(alkylthio)-amides oxidized to the corresponding sulfones, which are under examination as hypnotics. The metathesis reaction of the α-bromoamides was shown to be normal by the synthesis of the β-isomer from the reaction of *n*-butyl mercaptan with α-methylacrylamide. The preparation of α-bromoamides, including two not previously reported, has been described.—A. POMERANTZ and R. CONNER. *J. Am. Chem. Soc.*, 61 (1939), 3386. (E. B. S.)

Amino Alcohols of Tetrahydronaphthalene as Possible Analgesics. The phenanthrene nucleus is not entirely essential to morphine-like action since amino alcohols of the dibenzopyran and the carbazole series have proved effective. A series of amino alcohols of tetrahydronaphthalene have now been prepared in view of the promising results obtained with the 1,2- and 3,4-amino alcohols derived from phenanthrene. Complete laboratory directions are given for preparing the amino alcohol, 1-hydroxy-2-[(1,2,3,4-tetrahydro-2-isoquinolyl)-methyl]-1,2,3,4-tetrahydronaphthalene (I) along with various derivatives of it carrying a hydroxyl or acetoxyl, or methoxyl group in the naphthalene portion or in the isoquinoline or in both. Catalytic reduction of the corresponding ketone gave I. The amino ketones were obtained according to the directions of Mannich *et al.* (*Arch. Pharm.*, 275 (1937), 54) by the condensation of α-tetralone and its proper derivative with formaldehyde and tetrahydroisoquinoline or 6-methoxytetrahydroisoquinoline. The next lower homolog of I was prepared by refluxing 2-bromo-1-hydroxy-1,2,3,4-tetrahydronaphthalene with tetrahydroisoquinoline in benzene for twelve hours.—ERICH MOSETTIG and EVERETTE L. MAY. *J. Org. Chem.*, 5 (1940), 528–543. (W. T. S.)

Aminobenzoic Acid Esters of Substituted Monoalkylamino Alcohols—Preparation of. A series of new β-alkylamino-α,α-dimethylethanols has been prepared. From these alcohols, as well as two β-alkylamino-α,α-diethylethanols, by combination with *p*-nitrobenzoyl chloride and reduction, there has been prepared a series of new esters. A pharmacological study of one ester shows it to have interesting surface anesthetic properties.—S. D. GOLDBERG, W. F. RINGK and P. E. SPOERRI. *J. Am. Chem. Soc.*, 61 (1939), 3562. (E. B. S.)

Arachidonic Acid—Constitution of. Oxalic, succinic, acetic, valeric with probably some hexoic and small amounts of glutaric and formic acids have been identified among the oxidation products of arachidonic acid with alkaline permanganate. A formula is suggested in which the ten terminal atoms are similar in structure to those of linoleic acid.—DORIS ELAINE DOLBY, LESLIE CHARLES ALFRED NUNN

and IDA SMEDLEY-MACLEAN. *Biochem. J.*, 34 (1940), 1422. (F. J. S.)

Arsonophenylimidazoles—Synthesis of. Chemotherapeutic tests of some 2-alkyl-4(or 5)-arsonophenylimidazoles showed that unsubstituted arsonophenylimidazole and its 2-methyl, 2-ethyl and 2-propyl derivatives possess considerable therapeutic activity against nagana and syphilis.—R. WEIDENHAGEN and H. RIENACKER. *Ber. deut. chem. Ges.*, 72 (1939), 57–67; through *Chimie & Industrie*, 24 (1939), 1034. (A. P.-C.)

Bee Venom. V. Simple Chemical Separation of the Two Constituents. Saturating concentrated bee venom solution at 0° C. with ammonia gas precipitates constituent II, which is a weak base that gives the reactions of proteins. Attempts to purify the convulsant constituent I (weak acid having a high phosphorus content) in the filtrate by precipitating the compounds which accompany it in the form of brucine salts, have so far been unsuccessful. G. HAHN and MARIE E. FERNHOLZ. *Ber. deut. chem. Ges.*, 72 (1939), 1281–1290; through *Chimie & Industrie*, 43 (1940), 495–496. (A. P.-C.)

Benzimidazoles as Possible Local Anesthetics. In view of the local anesthetic activity of 2-alkylaminoethyl benzimidazoles, a study has been made of the conditions necessary for the formation of this base and certain of its derivatives prepared. 2-(α-Chloroethyl) benzimidazole (I) was prepared by refluxing *o*-phenylenediamine with α-chloropropionic acid followed by neutralization with $NaHCO_3$. Condensation of I with a series of six secondary amines and three primary amines, above methylamine, yielded the corresponding 2-(α-dialkylaminoethyl)-benzimidazoles and 2-(α-alkylaminoethyl)-benzimidazoles, respectively. The dialkyl derivatives formed dihydrochlorides which in 2% aqueous solution were quite acidic. The products from primary amines above methylamine formed monohydrochlorides, aqueous solutions of which had a p_H of 6–7. With ammonia or methylamine, two moles of I reacted with one mole of the amine to give disubstituted derivatives. These form dihydrochlorides on the ring nitrogens, and gave aqueous solutions which were weakly acidic.—C. H. ROEDER and A. R. DAY. *J. Org. Chem.*, 6 (1941), 25–35. (W. T. S.)

Benzothiazole Derivatives. III. Derivatives of 2-chlorobenzothiazole and some 2-alkyl- and 2-arylaminobenzothiazoles were synthesized. The latter were obtained by reaction of 2-chlorobenzothiazole and its derivatives with primary alkyl- and arylamines. The products obtained possessed no therapeutic activity against the malaria of birds.—N. S. DROZDOV and V. I. STAVROVSKAÏA. *J. Obchtch. Khim.*, 9 (1939), 409–414; through *Chimie & Industrie*, 42 (1939), 1034. (A. P.-C.)

Buna Rubbers. Processing and handling techniques are given for Buna 85 (a butadiene polymer), Buna S (a polymer of butadiene and styrene) and Perbunan (polymer of butadiene and acrylic nitrile).—A. KOCH. *Ind. Eng. Chem.*, 32 (1940), 464–467. (E. G. V.)

Carbinolamines Derived from Naphthalene and Quinoline—Chemical Constitution and Antiplasmodial Action of. In a series of 1:2-carbinolamines prepared as possible antiplasmodial agents from naphthalene and quinoline and their methoxy derivatives, several have shown activity when tested on bird malaria due to *Plasmodium relictum*. Dibutyl-, diamyl- and dihexyl-aminoethyl-6-methoxy-4-quinolylcarbinols were obtained by condensing the appropriate dialkyl amines with 6-methoxy-4-quinolyl halogenomethyl ketones and then hydrogenating to the corresponding carbinol. These are some of the simplest compounds showing antiplasmodial activity. Under the same conditions, corresponding naphthyl, methoxynaphthyl and quinolyl derivatives showed no activity. Nor were the higher and lower homologs of the aminomethyl-6-methoxy-4-quinolylcarbinols active.—HAROLD KING and THOMAS S. WORK. *J. Chem. Soc.*, (1940), 1307–1315. (W. T. S.)

Carbocyclic Alcohol Esters of N-Alkylpiperidinecarboxylic Acids. Preparation and properties are described of a large number of such esters which are nonirritating to delicate mucous membranes and which inhibit the development of staphylococci, coli and gonococci. The N-methyl-3-piperidinecarboxylic acid thymol ester is especially effective.—OTTO DALMER and CLAUS DIEHL, assignors to MERCK & CO., INC. U. S. pats. 2,182,791 and 2,182,793, Dec. 12, 1939. (A. P.-C.)

Cashew Nut Shell Liquid. Manufacturing operations and chemical composition are given.—ANON. *Ind. Eng. Chem.*, 32 (1940), 1306–1310. (E. G. V.)

4,4′-Diaminodiphenylsulfone, 4-Amino-4′-Hydroxydiphenylsulfone and Related Compounds—Chemistry and Chemotherapy of. Chemical properties and methods of preparation of 4,4′-diaminodiphenylsulfone, 4,4′-diaminodiphenylsulfide, 4-amino-4′-hydroxydiphenylsulfone and their acetyl derivatives are described. The diaminodiphenylsulfone is superior, while the aminohydroxydiphenylsulfone and the acetyl derivatives are about equal to sulfanilamide in their therapeutic effect.—G. W. RAIZISS, L. W. CLEMENCE, M. SEVERAC and J. C. MOETSCH. *J. Am. Chem. Soc.*, 61 (1939), 2763. (E. B. S.)

Dibenzopyran Derivatives, Including an Isomer of Cannabinol—Synthesis of. Instead of relying on degradative experiments, the authors undertook the following syntheses in an effort to arrive at the structure of cannabinol. 3-N-Nitrosoacetamide-4-cyanotoluene (I) was obtained in a 90% yield by passing nitrous fumes through 3-acetamido-4-cyanotoluene. Condensation of I with quinol dimethyl ether gave a 41% yield of 2′-cyano-2:5-dimethoxy-5′-methyldiphenyl (II). Treatment of II with HBr yielded 6-hydroxy-5′-methyl-3:4-benzocumarin (III). The acetate of III on treatment with methylmagnesium iodide produced 5″-hydroxy-2:2:5′-trimethyl dibenzopyran. In a similar manner 5″-hydroxy-2:2:5′-trimethyl-4″-N-amyl dibenzopyran was prepared and this compound is an isomer of cannabinol. This isomer, like cannabinol, was inactive in the Gayer test on rabbits in a dose of 5 mg./Kg. Attempts to condense I with orcinol dimethyl ether gave 2-cyano-2′:6′-dimethoxy-4′:5-dimethylazobenzene. The synthesis was likewise unsuccessful with ethyl veratrate.—R. GHOSH, D. C. S. PASCALL and A. R. TODD. *J. Chem. Soc.*, (1940), 1118–1121. (W. T. S.)

Diethylenediamine—Process for the Manufacture of. A mixture of ethylenediamine with an ethylene glycol ester of the type $CH_2OH.CH_2R$ (*R* being a mineral or organic acid radical) is melted at a temperature of between 200° and 300° C.—SCOTT & BROWNE, Spolka Akcyjna, Towarzystwo Przemyslowo-Handlowe Dla Wyrobow Chemiczno-Farmaceutycznych. Belg. pat. 435,180, July 31, 1939. (A. P.-C.)

***d*-ψ-Ephedrine and *l*-Ephedrine—Acetylation of.** Schmidt (*Arch. Pharm.*, 252 (1914), 111) confirmed his previous findings that vigorous acetylation of the hydrochloride of either *l*-ephedrine or *d*-ψ-ephedrine yielded N-acetyl-*d*-ψ-ephedrine. Contrary to Schmidt's report it has now been found that both of these bases on gentle acetylation yield acetyl-derivatives which may be converted to the corresponding nitroso compounds, proving them to be O-acetyl derivatives. Hydrolysis of nitrosoacetyl-*d*-

ψ-ephedrine yielded nitroso-*d*-ψ-ephedrine which was also obtained directly from the alkaloid. *l*-Ephedrine and *d*-ψ-ephedrine are known to be mutually convertible on heating with HCl. In view of the commercial importance of the conversion of *d*-ψ-ephedrine into its more useful isomer, it must be stressed that the reverse hydrolysis is easier to effect. This is illustrated by the fact that acetyl-*d*-ψ-ephedrine readily yields *d*-ψ-ephedrine on either acid or alkaline hydrolysis, but the acetyl-*l*-ephedrine gives *l*-ephedrine only on hydrolysis with an alkali, and even with quite dilute HCl the product consists of a mixture.—WILLIAM MITCHELL. *J. Chem. Soc.*, (1940), 1153–1155. (W. T. S.)

Ergot Extracts—Obtaining. Ergot is extracted with a low boiling, non-aqueous solvent (liquid sulfur dioxide, ammonia, acetone) and the crude alkaloids so obtained are separated in fractions soluble and insoluble in water. The soluble fraction produces uterine contractions with marked tetany and is 10–20 times as active as crude ergot extracts. The insoluble fraction produces violent contractions with slight tetany and a greatly elevated base line, *e. g.*, the sulfur dioxide extract (1) is triturated with 95% ethyl alcohol (3), shaken with water (50) and filtered. The residue is shaken with water (40 parts) and the filtrate added to the first filtrate. The combined filtrates are clarified by shaking with a little sodium chloride and calcium carbonate. The aqueous extract may be evaporated to dryness in a vacuum.—M. S. KHARASCH and R. R. LEGAULT, assignors, to E. LILLY AND CO., U. S. pat. 2,082,343; through *J. Soc. Chem. Ind.*, 58 (1939), 1295. (E. G. V.)

Estrogens Related to Triphenylethylene—Synthesis of. The marked estrogenic activity of triphenylchloroethylene has led to a synthesis of related compounds. α,α-Di-*p*-bromophenyl-β-phenylethylene (I) and the corresponding di-iodo-compound were prepared by refluxing in sulfuric acid the appropriate di-*p*-halogeno-phenylbenzylcarbinol which in turn was obtained from benzylmagnesium chloride and *p*-*p*'-dibromobenzophenone. α,α-Di-*p*-chlorophenyl-β-phenylbromoethylene and the corresponding dibromo- and di-iodo-derivatives were obtained by adding a slight excess of bromine to I and its bromine or iodine analog as the case may be. The di-iodo-derivative induced some estrogenic activity when injected into mice. α,α-Di-*p*-methoxyphenyl-β-phenylbromoethylene was even more active than triphenylchloroethylene in this respect. Reduction of anisil in the presence of acetic acid yielded 4:4'-dimethoxystilbendiol diacetate.—A. SCHONBERG, J. M. ROBSON, WADIE TADROS and H. A. FAHIM. *J. Chem. Soc.*, (1940), 1327–1329. (W. T. S.)

Glycol Derivatives—Manufacture of. An invention relating to the manufacture of glycol derivatives, particularly glycol ethers, is described. It has been proposed to use tertiary amines as catalysts for promoting the reaction between alkylene oxides such as ethylene oxide with hydroxy compounds and particularly tertiary aliphatic amines such as tributylamine and heterocyclic bases such as pyridine have been referred to.—H. M. STANLEY and P. EAGLESFIELD. British Patent Specification No. 530,230; through *Perfumer. Essent. Oil Record*, 32 (1941), 20. (A. C. DeD.)

Hydrocarbons—Aromatic, Oxidation of. All benzoic acid and anthraquinone and its derivatives now made in the United States are produced from phthalic anhydride which, in turn, is made by the vapor-phase catalytic oxidation of naphthalene. In addition to these uses, phthalic anhydride is of great importance in its own right, especially in the synthesis of synthetic resins. The present production capacity of phthalic anhydride equipment in the United States is estimated at 70 to 75 million pounds per year. There is no potential shortage of domestic naphthalene for producing phthalic anhydride in much larger quantities than is indicated for future consumption. If the catalytic oxidation of naphthalene to phthalic anhydride were not known, phthalic anhydride could not occupy its present outstanding importance as an intermediate. By the old process using sulfuric acid to oxidize naphthalene, the present installed capacity would require an estimated annual consumption of 700,000 to 750,000 tons of 100% sulfuric acid. The cost of contact sulfuric acid plant for this purpose is estimated at close to $8,000,000 and the acid cost alone would probably amount to 25% of the present sales price of phthalic anhydride.—C. R. DOWNS. *Ind. Eng. Chem.*, 32 (1940), 1294–1296. (E. G. V.)

Iodohippuric Acids—Synthesis of. II. The authors now report the synthesis of 2,3,5- and 3,4,5-triiodohippuric acid prepared with a view toward studying the efficacy of certain iodohippuric acids as contrast agents for clinical radiography. 2-Amino-3,5-diiodobenzoic acid (I) was prepared from anthranilic acid and iodine monochloride by a previously reported procedure. 2,3,5-Triiodobenzoic acid (II) was prepared from I by the method of Wheeler and Johns (*Am. Chem. J.*, 43 (1910), 405). Two and five-tenths grams of II were refluxed with 5 cc. of thionyl chloride to give 85.4% yield of the corresponding acid chloride. Shaking this acid chloride with glycine, previously dissolved in 3% sodium hydroxide solution, gave a 32% yield of 2,3,5-triiodohippuric acid as white platelets, m. p. 255.5–257° C. In a similar manner the isomeric 3,4,5-triiodohippuric acid was obtained in a 90% yield from *p*-aminobenzoic acid.—CARL J. KLEMME and JAMES H. HUNTER. *J. Org. Chem.*, 5 (1940), 508–511. (W. T. S.)

Isoergosterone—Hydrogenation of. Dehydrogenation of ergosterol according to Dimroth-Wetter or Oppenhauer gives an ergosterone to which is assigned a 4,17-diene structure and which is isomerized by alcoholic hydrochloric acid to an isoergosterone which, from its spectrum, must have two conjugated double bonds adjacent to the carbonyl group and is presumably the 4,6-diene. Windaus and Buchholz reported recently that hydrogenation of ergosterone yields a mixture of allo- and epiallo-ergosterols with relatively little ergosterol and very little epiergosterol. The author likewise has hydrogenated isoergosterone according to Meerwein-Ponndorf and obtained no ergosterol or epiergosterol but an alcohol with an absorption maximum at 240 mμ which he designates as 4,6,22-ergostatriene-3-ol, and which is precipitated by digitonin. There is also formed the epi- derivative, not precipitated by digitonin, which has not as yet been further characterized.—R. GÜNTZEL. *Ber. deut. chem. Ges.*, 72 (1939), 1317–1318; through *Chimie & Industrie*, 43 (1940), 320. (A. P.-C.)

3-Ketopolyhydrocyclopentanophenanthrenes. Therapeutic compounds forming freely water-soluble salts are produced by treating a hydroxy ketone such as $\Delta^{4,6}$-androsten-17-ol-3-one or the like with esterifying reagents such as succinic anhydride, maleic anhydride, phthalic anhydride, chlorosulfonic acid or phosphorus oxychloride. Absolute pyridine, quinoline or a dialkyl-aniline may be used (although not necessarily) as a condensation agent in the esterification reaction.—MAX HARTMANN and ALBERT WETTSTEIN, assignors to SOC. POUR L'INDUSTRIE CHIMIQUE À BÂLE. U. S. pat. 2,182,920, Dec. 12, 1939. (A. P.-C.)

Maleic Anhydride—Reactions of, with Abietic Acid and Rosin. Contrast is made of results where rosin is used instead of pure precipitated abietic acid previously discussed in the study of the structure of abietic acid, and of the addition products

formed by abietic acid with maleic anhydride. Although the existing theoretical data on the reaction of resinic acids with maleic anhydride which exist in abundance are valuable, commercial operations have to be carried out with rosin. Much of the theoretical work previously done was performed on freshly prepared abietic acid on which no oxidation had taken place if it could possibly be prevented. On the other hand, industry has to use rosin that may or may not have undergone a certain degree of oxidation.—A. G. HOVEY and T. S. HODGINS. *Ind. Eng. Chem.*, 32 (1940), 272–279. (E. G. V.)

Mercurimercapto Compounds—Stable Germicidal Heteroatomic. By refluxing a mercaptopyridinecarboxylic acid in a solvent such as ethanol with an ethyl-, propyl-, butyl- or like mercuric chloride, products are obtained having the general formula $N:CH.C(CO_2M):CH.CH:CSHgR$, where R is an alkyl radical and M is hydrogen, alkali metal, alkaline earth metal, ammonium, alkylammonium or alkanolammonium. They may be used as antiseptics in aqueous solution and are stable toward free oxygen even at elevated temperatures and in the presence of metal ion catalysts.—RUSSEL J. FOSBINDER and LEWIS A. WALTER, assignors to MALTBIE CHEMICAL CO. U. S. pat. 2,176,457, Oct. 17, 1939. (A. P.-C.)

Mercury Compounds—Organic. By effecting reaction between a soluble mercury salt such as mercuric acetate, nitrate, chloride or sulfate and an unsaturated hydrocarbon containing an ethylene linkage with a halogenated aldehydic carbonyl compound, disinfectant and fungicidal compounds are produced.—KARL GÖRNITZ, WILLY HARNACK and OTTO WURM, assignors to SCHERING A. G. U. S. pat. 2,178,099, Oct. 31, 1939. (A. P.-C.)

Monocrotaline—Structure of. III. Monocrotalic Acid. Monocrotalic acid, obtained by hydrogenolysis of monocrotaline, has been shown to be monobasic with the carboxy group tertiary, and to contain a lactone grouping. It decomposes when heated to lose water and carbon dioxide with formation of α,β,γ-trimethylangelicalactone. The discussion of the possible structure for monocrotalic acid is given with the conclusion that it is optically active α-carboxy-α,β-dimethyl-γ-hydroxy-γ-valerolactone.—R. ADAMS, E. F. ROGERS and R. S. LONG. *J. Am. Chem. Soc.*, 61 (1939), 2822. (E. B. S.)

Monocrotaline—Structure of. II. Monocrotic Acid Obtained by Alkaline Hydrolysis of the Alkaloid. Monocrotic acid is optically inactive, monobasic and forms a monomethyl ester with diazomethane. The methyl ester gives a dinitrophenylhydrazone indicating the presence of a ketone group. The acid is oxidized by sodium hypobromite to α,α'-dimethylsuccinic acid. It is concluded that monocrotic acid is α,β-dimethyllevulinic acid.—R. ADAMS, E. F. ROGERS and F. J. SPRULES. *J. Am. Chem. Soc.*, 61 (1939), 2819. (E. B. S.)

Phenanthrenes—Biochemical Hydrogenation of. A process for the conversion of a keto compound of the 10,13-dimethylpolyhydrocyclopentanophenanthrene series into a corresponding hydroxyl compound of the same series involves subjecting the keto compound to the action of a reducing yeast-containing fermenting solution such as one of top yeast. Details are given for the production of testosterone from $\Delta^{4,5}$-androstene-3,17-dione, $\Delta^{4,5}$-androstenediol from dehydroandrosterone, $\Delta^{4,5}$-androstene-17-ol-3-one and isoandrostanediol from $\Delta^{4,5}$-androstene-3,17-diol, isoandrostanediol from androstane-3,17-dione, 3-hydroxy-12-ketocholaic acid from 3,17-diketocholaic acid, and cholestanol from cholestanone. The fermentation may be promoted by the use of substances such as primary or secondary sodium phosphates, calcium carbonate or nitrogen-containing compounds.—LUIGI MAMOLI, assignor to SCHERING CORP. U. S. pat. 2,186,906, Jan. 9, 1940. (A. P.-C.)

Phenolic Compounds from Petroleum Sources. Petroleum acids are an excellent source for the recovery of certain individual phenols—E. FIELD, F. H. DEMPSTER, and G. E. TILSON. *Ind. Eng. Chem.*, 32 (1940), 489–496. (E. G. V.)

Polycyclic Aromatic Hydrocarbons. XXV. 1- and 2-Alkyl Derivatives of 3:4-Benzphenanthrene. 1- and 2-Alkyl derivatives of 3:4-benzphenanthrene have been obtained by reduction of the ketones arising by the action of alkylmagnesium halides on the corresponding 3:4-benzphenanthramides. 1-*iso*-Propyl-3:4-benzphenanthrene has been obtained from methyl 3:4-benz-1-phenanthroate by treatment with methylmagnesium iodide and subsequent dehydration and hydrogenation.—JAMES L. EVERETT and C. L. HEWETT. *J. Chem. Soc.*, (1940), 1159–1162. (W. T. S.)

Polyhydrocyclopentanophenanthrene—Derivatives of. Products such as progesterone and testosterone are obtained by transforming an acid such as acetylbisnorlithocholic acid or 3-acetoxyetiocholanylcarboxylic acid, by means of the Curtius degradation method, into the corresponding amine, oxidizing the 3-hydroxyl group into the keto group and producing a double bond in the associated ring by treatment with a halogenating agent such as bromine, and subsequently dehalogenating with silver acetate. Various details of procedure are given.—MAX BOCKMÜHL, GUSTAV EHRHART, HEINRICH RUSCHIG and WALTER AUMÜLLER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,188,870, Jan. 30, 1940. (A. P.-C.)

Progesterone—Process for the Preparation of. Cholestenone is oxidized in sulfuric acid at a concentration of more than 50%.—CHINOIN, GUOGYSZER AS VETYESZETI TERMEKEK GYARA R. T. Belg. pat. 434,745, July 31, 1939. (A. P.-C.)

Propenyl Benzene Derivatives. The improved process for manufacture of propenyl benzene derivatives is effected more easily by heating the allyl compound with caustic alkali and using a glycol or polyglycol or a glycol ether (preferably in the presence of a hydroxyamine) as diluent for the melt. The reaction is endothermic, and the melt is heated to about 160–180° until the temperature remains constant or falls slightly owing to the absorption of heat. After a few minutes at this temperature, excellent yields of the propenyl compounds are obtained.—*Chemist and Druggist*, 133 (1940), 282. (A. C. DeD.)

Pseudo-Eleostearic Acid. Linolenic acid is partially converted by alcoholic alkalies into pseudo-eleostearic acid, 10,12,14-octadecatrienoic acid, m. p. 79°, methyl ester, m. p. 41°. Pseudoeleostearic acid forms mixed maleic anhydride addition products and mixed bromides, among them a tetrabromide, m. p. 104.5°, and a hexabromide, m. p. 152.5°.—J. P. KASS and G. O. BURR. *J. Am. Chem. Soc.*, 61 (1939), 3292. (E. B. S.)

5:6:3′:2′-Pyridoquinoline Derivatives as Possible Antimalarials. In order that they might be tested for antimalarial activity, derivatives of 5:6:3′:2′-pyridoquinoline, carrying basic side chains, have been prepared by condensation of the corresponding chloro-derivative with the appropriate amine. In this way, compounds such as 2-β-diethylaminoethylamino-4-methyl-5:6:3′:2′-pyridoquinoline and 4-γ-diethylaminopropylamino-2-methyl-5:6:3′:2′-pyridoquinoline have been obtained. Compounds have also been synthesized lacking the methyl group by the condensation of *p*-aminoacetanilide with ethyl oxaloacetate to yield ethyl 6-acetamido-4-hy-

droxyquinoline-2-carboxylate, which by hydrolysis and decarboxylation yields 6-amino-4-hydroxyquinoline, whence 4-hydroxy- and 4-chloro-5:6:3′:2′-pyridoquinoline have been prepared. The latter compound reacts with suitable amines to yield compounds of the desired type. The nitration of ethyl 4-hydroxyquinoline-2-carboxylate is shown to occur in the 6-position. It is proved that the pyridoquinoline formed by the Skraup reaction from 6-amino-4-hydroxyquinoline has the angular, and not the linear, structure.—WILLIAM O. KERMACK and ALICE P. WEATHERHEAD. *J. Chem. Soc.*, (1940), 1164–1169. (W. T. S.)

Quaterphenyl—Some Dihydroxy-Derivatives, as Possible Estrogens. 4:4‴-Dihydroxyquaterphenyl (HO—C_6H_4—C_6H_4—C_6H_4—C_6H_4—OH) were prepared by treating the corresponding dimethoxy derivative in acetic acid with HI, followed by hydrolysis with HCl. It was also obtained in smaller yields by heating 4′-iodo-4-nitrodiphenyl with copper bronze to give 4-4‴-dinitroquaterphenyl which was reduced to the corresponding diamine which in turn was tetrazotized and boiled with water to give 4:4‴-dihydroxyquaterphenyl. The compound was devoid of estrogenic activity. Attempts to oxidize 4:4‴-dihydroxyquaterphenyl to the quinone destroyed the terminal benzene nuclei, giving diphenyl-4:4′-dicarboxylic acid. Similarly, oxidation of the dinitro derivative caused rupture of the inner benzene nuclei producing 4′-nitrodiphenyl-4′-carboxylic acid. The isomeric 2:2‴- and 2:4‴-dihydroxyquaterphenyls were also prepared from the corresponding dimethoxy-compounds and their properties studied.—JOHN HARLEY-MASON and FREDERICK G. MANN. *J. Chem. Soc.*, (1940), 1379–1385. (W. T. S.)

Ricinus Communis. I. Oxidation of Ricinoleic Acid. Ricinoleic acid and its ethyl ester with theoretical iodine numbers have been prepared. The oxidation of ethyl ricinoleate in dry acetone yields the liquid acids, caproic, heptylic and caprylic; and the solid acids, β-hydroxypelargonic acid, azelaic, suberic and an acid of m. p. 96°. The oxidation of ricinoleic acid in alkaline solution of permanganate at 0° gave two trihydroxystearic acids already known, m. p. 110° and 141°, respectively, in approximately equal amounts. The oxidation of ricinelaidic acid, on the other hand, gave a large percentage of the acid, m. p. 110°, and a small amount of the acid, m. p. 141°. The oxidation of both the trihydroxystearic acids obtained from ricinoleic and ricinelaidic acids, respectively, with periodic acid, resulted in the same products, β-hydroxypelargonic aldehyde and aldehydoazelaic acid.—S. E. BRADY. *J. Am. Chem. Soc.*, 61 (1939), 3464. (E. B. S.)

Snake Venoms and Proteins—Formation of Thiol Groups in the Hydrolysis of. The proteins and venoms studied (ovalbumin, casein, edestin, venoms of *Naja tripudians*, of *Crotalus atrox*, of *Bothrops jararaca* and *alternata*), after hydrolysis by formic and hydrochloric acids in an atmosphere of carbon dioxide, strongly reduce phosphotungstic acid. Only *Naja tripudians* venom, however, gives a strong sodium nitroprusside reaction due to thiol groups; in the other cases the reaction was relatively weak or very weak and in no way corresponded to the phosphotungstic acid reaction. It can therefore be considered that the reduction of the latter is due to a nonthiol compound formed during hydrolysis.—F. MICHEEL. *Ber. deut. chem. Ges.*, 72 (1939), 397–400; through *Chimie & Industrie*, 42 (1939), 1024. (A. P.-C.)

Spirochaeticide. A preparation of a dry salt (hydrochloride) of 3-amino-4-hydroxyphenylarsine oxide (60 mg.), sufficient of a base to neutralize the hydrochloric acid (sodium carbonate, 13 mg.), and sodium chloride (60 mg.) so that a non-hemolyzing solution suitable for intravenous injection is obtained on dissolving in water (15 cc.) is claimed. The use of the hydrochloride in stable solid form in human therapy is claimed. Toxicity and medical data are given.—A. B. SCOTT, O. M. BRUHZIT and A. L. TATUM. U. S. pat. 2,092,028 and 2,092,036; through *J. Soc. Chem. Ind.*, 59 (1940), 173. (E. G. V.)

Steroids—High Vacuum Distillation of. Sterols can be separated from the natural oils in which they occur by distillation in the molecular still at 100–220° C. The steroids which are not oil-soluble are preferably separated by crystallization. If it is desired to distil them, they are best handled as solids in the molecular pot still or in stills of the diffusion type. Even the sterols which occur naturally in oil are only partially oil-soluble. They separate as crystals from the enriched distillates, and after separation of the crystals these distillates can be returned to the molecular still to give new distillates, from which a further crop of crystals can be extracted. The antirachitic materials formed by the irradiation of sterols are more soluble in oil. They can be separated from the parent sterol by a combination of distillation and crystallization, the antirachitic material remaining in the soluble portion of the distillate. The molecular still is useful for purifying steroids and waxes isolated in biological research. Small quantities are generally all that are available, and these are handled in the pot still with a detachable head. Although distillation effects concentration of the steroids, it is seldom able to separate them in absolute purity. It must be assisted by saponification and crystallization.—K. C. D. HICKMAN. *Ind. Eng. Chem.*, 32 (1940), 1451–1453. (E. G. V.)

Steroids—Investigations of. *III.* Partial Oxidation of 3,5,6-Triols and Oxidation with Permanganate of 5,6-Unsaturated Steroids. One of the authors (M. E.) has previously reported on the transformation of pregnane-20-one-3(β),5,6(*trans*)-triol into pregnane-3,20-dione-5,6(*trans*)-diol 6-monoacetate. A detailed report is now made of some experiments designed to produce the two possible oxidation products of 3,5,6-triols, namely, the corresponding 3-one-5,6-diols or 6-one-3,5-diols.—MAXMILIAN EHRENSTEIN and MARGUERITE TWADDELL DECKER. *J. Org. Chem.*, 5 (1940), 544–560. (W. T. S.)

Sterols. LXXIV. Acetic Acid Derivatives of Estrone and α-Estradiol. The 3-oxyacetic acid derivatives of estratriene-1,3,5-one-17-ol-3 and of estratriene-1,3,5-diol-17(α),3 were prepared by the reaction of these substances with ethylchloroacetate in the presence of an excess of an ethanolic solution of sodium ethylate. Estratriene-1,3,5-one-17-ol-3 was also caused to react in a similar way with α-chloropropionic acid to yield estratriene-1,3,5-one-17-oxymethylacetic acid-3. The acidic substances were characterized further by the formation of the methyl esters.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 2974. (E. B. S.)

Sterols—Molecular Rearrangements in. IV. The Structure of *i*-Cholestanone. Further evidence has been given to show that Windaus' method of removing hydrogen chloride from α-3-chlorocholestanone-6 gives *i*-cholestanone and not the unsaturated ketone heterocholestanone. The true heterocholestanone, Δ^4-cholestanone-6, has been obtained and characterized. A new dicarboxylic acid, m. p. 233°, has been prepared from *i*-cholestanone. This acid has been shown to be an isomer of another new acid, m. p. 231°, obtained from β-3-chlorocholestanone-6. A third isomeric acid, m. p. 265°,

has been prepared from α-3-chlorocholestanone-6. A detailed discussion of the stereochemical relationships of these compounds has been given.—K. LADENBURG, P. N. CHAKRAVORTY and E. S. WALLIS. *J. Am. Chem. Soc.*, 61 (1939), 3483. (E. B. S.)

Sterols—New Epimerization Process of. A novel process for the conversion of sterols into their epimeric forms, in which the hydroxyl group assumes the alternative steric position to the ring plane, has been discovered, involving heating under reflux with aluminium *iso*propoxide in xylene solution. By this means *epi*cholesterol and *epi*lumisterol have now been obtained directly from the normal sterols, but attempts similarly to epimerize ergosterol, or to prepare *epi*ergosterol by other methods, have not been successful.—J. BARNETT, I. M. HEILBRON, E. R. H. JONES and K. J. VERRILL. *J. Chem. Soc.*, (1940), 1390–1393. (W. T. S.)

Sterols. LXXVI. Oxidation and Reduction Products of Equilenin. Equilenin derivatives upon mild oxidation with chromic anhydride yield 11-keto derivatives. Catalytic hydrogenation of equilenin derivatives in acidic medium gives 5,7,9-estratrienol-17(α).—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 3314. (E. B. S.)

Sterols. LXXIX. Oxidation Products of Dihydrosarsasapogenin. The chromic anhydride oxidation products of dihydrosarsasapogenin have been studied, and the relation of the products to sarsasapogenin is given in structural sequence.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 3477. (E. B. S.)

Sterols. LXXII. Oxidation Products of Sarsasapogenin. C_{19} Dibasic Acid. The acetates of sarsasapogenin, tetrahydrosarsasapogenin and sarsasapogenoic acid upon oxidation with chromic anhydride yield a C_{19} dibasic acid, probably 3-hydroxy-*etio*-bilianic acid.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 2722. (E. B. S.)

Sterols. LXXX. Reactions of Chlorogenin. Chlorogenone prepared by the oxidation of chlorogenin appears to be identical with the 3,6-diketone of diosgenin. Chlorogenin was reduced catalytically to give dihydrochlorogenin, mild oxidation of which gave an acid, $C_{27}H_{40}O_5$, which formed a disemicarbazone and a methyl exter. Chlorogenin formed a monobromo derivative with bromine in acetic acid. Reduction of this gave chlorogenin. The lactone diacetate, dihydroxy lactone, lactone dibenzoate and diketo lactone were prepared from chlorogenin.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 3479. (E. B. S.)

Sterols. LXXIII. Reactions of Digitogenin and Gitogenin. Gitogenin and digitogenin resemble sarsasapogenin and tigogenin in their behavior toward catalytic hydrogenation, bromination and oxidation with selenium dioxide. They are relatively inactive toward isomerization with hydrochloric acid and Clemmensen reduction. The C_{22} lactones of digitogenin and gitogenin have been prepared.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 2724. (E. B. S.)

Sterols. LXXVII. The Oxidation of Pregnanetriol-3,4,20(α) and of Coprostanediol-3,4. Pregnanetriol-3,4,20(α) was oxidized to the 3,4-dicarboxylic acid of pregnanone-20, which was different from the dicarboxylic acid of pregnanone-20 formed by the direct oxidation of pregnanedione. The latter acid must therefore be a 2,3-derivative. Coprostanediol-3,4 was oxidized to a coprostanedicarboxylic acid. This acid was different from the dicarboxylic acid obtained by direct oxidation of coprostanone. The greater part of the oxidative fission of 3-substituted derivatives of the coprostane configuration (except those of the bile acid series) takes place at the 2,3-bond rather than at the 3,4-bond as has been generally assumed.—R. E. MARKER, E. L. WITTLE, L. PLAMBECK, JR., E. ROHRMANN, J. KRUEGER and P. R. ULSHAFER. *J. Am. Chem. Soc.*, 61 (1939), 3317. (E. B. S.)

Sterols. LXXI. Urane Derivatives. Reaction of urane derivatives have been observed, and the results are given. Uranedione on partial oxidation with chromic anhydride yielded uraneol-3(β)-one-11. Uranediol on oxidation with aluminum isopropylate gave uraneol-11-one-3. Mild catalytic hydrogenation of uranedione-3,11 gave uraneol-3(β)-one-11. Similar results were obtained on mild reduction with aluminum isopropylate. Mild Clemmensen reduction of uranedione-3,11 gave uranone-11. Vigorous reduction gave urane. Mild Clemmensen reduction of uranetrione gave uranedione-11,20. Uranedione has been prepared from bromouranedione.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 2719. (E. B. S.)

Terpenes and Terpene Ketones—Catalytic Disproportion and Dehydrogenation of. A study has been made of the action of palladium and platinum catalysts on the following compounds of the terpene and sesquiterpene groups: limonene, pinene, pinane, cadinene, *iso*cadinene, selinene, pulegone, menthone, carvone, dihydrocarvone, carvomenthone, camphor. The results throughout were in harmony with the known structures. The experiments under mild conditions gave clear evidence of the skeleton structure and the number of double bonds. For instance, all the unsaturated substances underwent disproportionation into aromatic and saturated compounds at comparatively low temperatures (140° to 205°), the proportions formed being those predictable from the number of double bonds in the original terpene. Carvone was isomerized almost quantitatively to carvacrol. Pinene was converted into *p*-cymene and pinane, the *cyclo*butane ring being broken. Selinene was the least easily disproportionated hydrocarbon, owing to the presence of a quaternary carbon atom. Nevertheless it gave eudalene with surprising ease at 205°. All the compounds studied, whether unsaturated or saturated (with the exception of camphor, which was completely resistant), gave their aromatic counterparts with elimination of hydrogen at higher temperatures. The yields of aromatic material were in general considerably better than those obtainable by other methods. Under vigorous dehydrogenating conditions, cadinene underwent elimination of the *iso*propyl group with the formation of 1:6-dimethylnaphthalene.—R. P. LINSTEAD, K. O. A. MICHAELIS and S. L. S. THOMAS. *J. Chem. Soc.*, (1940), 1139–1147. (W. T. S.)

Tetrahydrodibenzopyran Derivatives—Synthesis of, in an Attempt to Elucidate the Cannabinol Structure. A general method for the synthesis of 2:2-dimethyl-3′:4′:5′:6′-tetrahydrodibenzopyrans by the action of excess of methylmagnesium iodide on 3:4-*cyclo*hexenocoumarins is described. The compounds prepared include 6″-hydroxy-2:2:5′-trimethyl-4″-*n*-amyl-3′:4′:5′:6′-tetrahydrodibenzopyran which may be a tetrahydrocannabinol. Several of the synthetic substances have been tested on rabbits by the Gayer hashish test, but all were inactive at a dosage of 5 mg./Kg. None of the compounds prepared gave a positive Beam test (red-violet color with alcoholic potash). Although hydroxy-3:4-*cyclo*hexenocoumarins could be prepared from quinol and from resorcinol derivatives, no such compounds could be synthesized from derivatives of catechol.—R. GHOSH, A. R. TODD and S. WILKINSON. *J. Chem. Soc.*, (1940), 1121–1125. (W. T. S.)

Theobromine Alkaline Earth Metal Gluconates. By reaction or quicklime, theobromine and cal-

cium gluconate, etc., a double salt of theobromine and calcium with gluconic acid is obtained, which is suitable for use in the treatment of cardiovascular diseases.—RUSSEL J. FOSBINDER, assignor to MALTBIE CHEMICAL CO. U. S. pat. 2,182,314, Dec. 5, 1939. (A. P.-C.)

Thiophen Sulfur—Oxidation of, by Calcium Hypochlorite Solutions. Factors influencing the proportion of thiophen sulfur converted into sulfate as a result of the oxidative action of calcium hypochlorite solutions are discussed. The range of p_H values found most effective for the oxidation of thiophen sulfur by this method lies in the neighborhood of 8 to 7. Such average p_H values for the hypochlorite solution will result from the use of an initial p_H 8 or, for long periods of treatment, p_H 9. The use of other acidifying agents for the adjustment of the initial p_H gave essentially the same results as acetic acid.—E. G. R. ARDAGH, W. H. BOWMAN and A. S. WEATHERBURN. *J. Soc. Chem. Ind.*, 59 (1940), 27–28. (E. G. V.)

Trialkylacetamides. Tertiary saturated alkyl-substituted acetic acid amides with from 12 to 17 carbon atoms, made according to known processes from tertiary nitriles of the general formula $RR'R''CCN$ (in which R, R' and R'' indicate the same or different saturated alkyl groups such that in the case of the saturated trialkylacetamides with 12 carbon atoms R, R' and R'' constitute alkyl with at least 3 carbon atoms) exhibit good spasmolytic action. Details are given of the production of a number of these compounds.—KARL JUNKMANN and HANS-GEORG ALLARDT, assignors to SCHERING A.-G. U. S. pat. 2,186,976, Jan. 16, 1940. (A. P.-C.)

5:6:4′- and 5:8:4′-Trihydroxyflavones—Derivatives of. A Note on the Structure of Ginkgetin.—WILSON BAKER and W. H. C. SIMMONDS. *J. Chem. Soc.*, (1940), 1370–1374. (W. T. S.)

Vitamin B_6 as a β-Hydroxypyridine Derivative. The dicarboxylanhydride, $C_8H_5O_4N$, resulting from the oxidative degradation of the methyl ester of adermin is identical with the anhydride of 3-methoxypyridine-4,5-dicarboxylic acid. Adermin is therefore a derivative of β-hydroxypyridine. As to the position of the two hydroxymethyl groups in vitamin B_6, partial oxidation tests of the methyl ester of adermin show that neither of them could be in α-position with respect to the nitrogen atom.—R. KUHN, H. ANDERSAC, K. WESTPHAL and G. WENDT. *Ber. deut. chem. Ges.*, 72 (1939), 309–310; through *Chimie & Industrie*, 42 (1939), 1031. (A. P.-C.)

Vitamin E—Chemistry of. XVIII. Condensation of Phenols and Hydroquinones with Allylic Alcohols, Allylic Halides and Conjugated Dienes. Allylic alcohols can be condensed directly with phenols and hydroquinones, thus avoiding the necessity of converting the alcohols into halides. Although dienes also can be condensed with phenols and hydroquinones, the dienes are not always intermediates in the condensations when allylic alcohols and halides are used, for the products are not always the same. A mechanism is proposed for these condensations, which, though not proved, correlates the facts well. Allylic chlorides condense well with phenols and hydroquinones as the bromides do. An improved procedure is given for the preparation of α-tocopherol from trimethylhydroquinone and phytol.—L. E. SMITH, H. E. UNGNADE, J. R. STEVENS and C. C. CHRISTMAN. *J. Am. Chem. Soc.*, 61 (1939), 2615. (E. B. S.)

BIOCHEMISTRY

Amines—Production of, by Bacteria. II. The Production of Tyramine by Streptococcus Faecalis. The following conclusions are given: (1) Washed suspensions of stock *Strep. faecalis* decarboylate *l*(—)-tyrosine to form tyramine. (2) The decarboxylase involved is optimally active at p_H 5.0. (3) Cultures grown at 27° have the same activity as those grown at 37°. (4) The activity of the washed suspension varies with the age of the culture, maximum activity occurring between the 16th and 18th hours when growth takes place in 2% glucose broth (tryptic digest of casein) at 27°. (5) The activity of the washed suspension depends upon the p_H of the growth medium. Cultures grown in non-carbohydrate media at p_H 7 have little activity, this being increased 30–40 fold by growth at p_H 5. In carbohydrate media the activity obtained is explained by the fall in p_H during growth due to fermentation acids. (6) The decarboxylation is quantitative and the tyramine has been isolated and identified as the dibenzoyl derivative. (7) Of seven strains of *Strep. faecalis* investigated, six decarboxylated tyrosine. These active strains appear to be the more rapid acid-producing strains; five strains also formed ornithine by liberation of $(NH_4)_2CO_3$ from arginine. (8) In no case was any other amino acid decarboxylated to form an amine under the experimental conditions described. The tyrosine decarboxylase is thus strictly specific.—E. F. GALE. *Biochem. J.*, 34 (1940), 846. (F. J. S.)

Antimalarial Synthetics—Detection of, in Urine. The following identification tests are recommended. To 10 cc. of urine acidified with 0.2 cc. of acetic acid, add 2 cc. of Tanret's reagent and let stand for ten minutes. A definite precipitate forms. If the urine contains albumin or pseudo-albumin the reaction does not occur, and a sample should be treated as follows: Alkalinize 100 cc. of urine with 5 cc. of potassium hydroxide solution, shake for five minutes with 50 cc. of ether and repeat twice with 30-cc. portions of ether. If an emulsion forms, shake with 5 cc. of alcohol. Add 10 cc. of 2% acetic acid solution to the combined ether extracts and mix. To 5 cc. of the aqueous acetate solution (colored faintly yellow if "praequine" or "rhodoquine" is present) add 1 cc. of Tanret's reagent and let stand for three minutes. With 0.01 mg. of the synthetic compound a precipitate is formed. Different procedures for quantitative determination are reviewed and discussed.—P. DUBOST and A. ALLINNE. *Bull. sci. pharmacol.*, 46 (1939), 367–375. (S. W. G.)

Arakawa's Reaction and Urine Diastase Content of Lactating Mothers. It is a general rule that the diastase content in lactating mothers' urine is large in Arakawa-positive cases and small in Arakawa-negative cases except some cases with an intake of vitamin B preparations. As to the disease of infants, it seems that the diastase content in lactating mothers' urine is small in the case of avitaminosis B and hereditary syphilis.—S. SATO. *Tôhoku J. Exp. Med.*, 37 (1940), 393. (A. C. DeD.)

Arakawa's Reaction and Vitamin C of Human Milk. The author tried a series of experiments to test the accuracy of Harris and Ray's method for vitamin C determination, but was unable to obtain a definite conclusion. The question whether the difference of the vitamin C-like substance between Arakawa-positive and -negative milk is the difference of vitamin C itself or vitamin C plus some other substance remains unsolved. The author believes that it is not altogether satisfactory to rely upon animal experimentation for the purpose of assaying the vitamin C which may remain undetermined by Harris and Ray's method.—S. ISONO. *Tôhoku J. Exp. Med.*, 36 (1939), 590. (A. C. DeD.)

***l*-Ascorbic Acid.** 2-Keto-*l*-gulonic acid and its readily hydrolyzable derivatives such as diacetone-2-keto-*l*-gulonic acid are converted into *l*-ascorbic acid

by the combined action of a solvent capable of reacting with the hydroxyl groups of the 2-keto-*l*-gulonic acid, such as glacial acetic acid, and a sufficient amount of concentrated hydrochloric or sulfuric acid to make a solution of at least twice normal strength. Various details and modifications of procedure are described.—RICHARD PASTERNACK and GORDON O. GRAGWALL, assignors to CHARLES PFIZER & Co. U. S. pat. 2,185,383, Jan. 2, 1940. (A. P.-C.)

Ascorbic Acid Content of Blood and Urine of Filipinos. After examining 50 people of different ages results showed that the blood ascorbic acid of Filipinos varies from 0.393 to 275 mg. % with an average of 1.007 mg. %; 24-hour excretion varied from 11.51 to 19.7 mg. Three hours after a test dose of 100 mg. of ascorbic acid there was found no increase of it in the urine in a large majority of the cases. This is an indication of deficiency or unsaturation of vitamin C in the system of many Filipinos.—ISABELO CONCEPCION. *Nat. Res. Council Philippines Bull.*, 23 (1939), 142. (P. A. F.)

***l*-Ascorbic Acid—Process for the Manufacture of.** 2,179,977—2-Keto-*l*-gulonic acid or a derivative such as the methyl ester is heated in a solution of acid reaction (suitably in a mixture of ethanol and chloroform with hydrochloric acid). 2,179,978—2-Keto-*l*-gulonic acid or a derivative such as the methyl ester is heated with an alkali salt of a weak acid such as sodium bicarbonate or acetate in substantially anhydrous alcoholic solution such as methanol.—FRANZ ELGER, assignor to HOFFMANN-LA ROCHE, INC. U. S. pats. 2,179,977 and 2,179,978, Nov. 14, 1939. (A. P.-C.)

Barbital—Microchemical Determination of, in Blood and in Cerebrospinal Fluid. The material to be tested is treated with an acid buffer solution to prevent separation of albumin from blood, and the barbital is extracted with ether. The extract is dried, purified by treatment with charcoal and magnesia, filtered, the filtrate evaporated and the residue subjected to sublimation. The sublimate is weighed. Very carefully purified ether must be used. A separation of barbital from succinic acid with the aid of adsorption agents is described.—R. FISCHER. *Mikrochemie (Mikrochim. Acta)*, 26 (1939), 255–263; through *Chimie & Industrie*, 42 (1939), 964. (A. P.-C.)

Barbitals—Colorimetric Cobalt Reaction of W. Parri for the Identification of, in the Urine and in Other Organic Liquids. The authors have simplified the colorimetric cobalt reaction of W. Parri for the identification of barbitals in urine.—M. A. MACINE and E. PECCIARINI. *Biochim. terap. sper.*, 26 (1939) 119. (A. C. DeD.)

Blood—Normal p_H Value of, among Filipinos. Using the Beckman's glass electrode p_H meter, a series of 85 specimens of whole blood from normal adult Filipinos of both sexes and of different constitution and nutrition, was determined. The surprising rapidity of result and ease of technique is remarkable in comparison with other methods. The precautions against undue gas evolution from the specimens and the use of properly standardized potassium oxalate solution, were taken into consideration. The figures indicate higher values than those obtained by Austin, Cullen, Van Slike and others, and oscillate from 7.3 to 7.55, the majority falling within the margin 7.4–7.45. These figures, though still within normal limits, show a marked leaning to the alkaline side.—JESUS P. CELIS, CARLOS J. ZIALCITA and ANDRES R. CRUZ. *Bull. San Juan de Dios Hosp.* (Manila), 13 (1939), 427; through *Proc. Fifth Sci. Convention Nat. Res. Council Philippines Bull.*, 23 (1939), 137. (P. A. F.)

Blood Phosphorus—Estimation of, by the Method of Fiske and Subbarow. The blood has its albumen removed by trichloracetic acid. A sulfo-molybdic reagent is added and then a solution of aminonaphthosulfonic acid. The blue color resulting (due to the formation of molybdenum oxide) is analyzed spectrophotometrically, and the amount of phosphorus is calculated in milligrams per cent. By a modification of this method both the total phosphorus and the acid-soluble phosphorus are estimated.—ALUISIO MARQUES LEAL. *Noticias farm.*, 5 (1939), 456. (G. S. G.)

Blood Test—Mendinger's Reaction for. There is a need for a specific chemical reaction for blood. Color reactions due to the oxidation action of hemoglobin are not specific and the same reaction can be produced by certain minerals and organic substances. Its chief value is negative. Mendinger's reagent is an acidified leuco-malachite green. It is colorless but on contact with blood it becomes bluish green. No other substance, mineral or organic, produces this color reaction. The reagent is a mixture of benzaldehyde 10 Gm., dimethylaniline 25 Gm. and HCl 12 Gm. This is heated, evaporated with ether, taken up with NaOH and distilled. The distillate is washed with absolute alcohol and heated. On cooling it exhibits a clear green color. This is the leuco-base. The color is removed by filtering *in vacuo* and washing the precipitate with absolute alcohol and drying. This precipitate is colorless, odorless, insoluble in water or cold alcohol but soluble in warm alcohol or acidified water. The final reagent is made by mixing 1 Gm. of this leuco-base of malachite green with 100 cc. glacial acetic acid and 150 cc. distilled water. It should be kept in an amber flask, with a ground-glass stopper, or in amber ampuls. If it is put in clear glass it should be kept in the dark. It is good indefinitely as long as it remains colorless. For use, the suspected stain is moistened with a filter paper soaked in the reagent, or the reagent is sprayed on the stain. The characteristic blue-green color surrounding the stain indicates blood.—JULIO VALLADARES MARQUEZ. *Escuela Farm.*, 2 (1939), No. 20, 13. (G. S. G.)

Blood Ultrafiltrates—Some Nitrogen and Sulfur Partitions on. In order to obviate difficulties in routine clinical methods of blood and tissue deproteinization the authors made use of a purely physical method of removing the proteins by ultrafiltration at a slightly reduced pressure through collodion of the oxalated-fluorided blood. Nitrogen partitions carried out on these filtrates, after the proper correction for protein volume, have yielded results comparable to the usual methods with the further advantages of allowing the authors to determine the single amino acid, cystine, to bring about the elimination of artifacts introduced by disintegration of red corpuscles such as undetermined nitrogen and non-sugar reducing substances. By ultrafiltration of both laked and unlaked samples they were able to verify the results of Folin and Svedberg with their unlaked filtrates. By use of these filtrates it was possible to carry out experimental work on the cystine content of blood under various conditions, which has heretofore been impossible. By application of the Stufenphotometer to the microdetermination of cystine it was found that normal blood contains about 1.0 mg. % which rises slightly after feeding and markedly after administration of cystine and methionine either orally, subcutaneously or intravenously. Analyses of the blood of a cystinuric failed to show abnormal values as might be expected. Detailed studies on the cystine content and sulfur partition values on rabbits and rats under various conditions showed the further possibility of a close metabolic relationship between cystine and methionine, which has been promulgated on the anomalous behavior of these two amino acids in cystinuria, and

the recently established fact that methionine is the essential sulfur-containing amino acid while cystine is definitely non-essential. Sulfur partitions were carried out by photometer.—H. B. LEWIS and B. H. BROWN. *Proc. Fifth Sci. Convention Nat. Res. Council Philippines Bull.*, 23 (1939), 156. (P. A. F.)

Calcium Intake—Effect of the Level of, on the Biological Value of a Protein. The biological values of the nitrogen of two diets, both containing egg white as a source of protein but one having a calcium content of 0.86% and the other of 0.07%, were measured on rats by the balance sheet method of Mitchell. No differences were found in the biological values and true digestibilities of the protein of the two diets. The availability of dietary nitrogen is apparently not decreased when the diet is deficient in calcium.—K. M. HENRY, S. K. KON and S. Y. THOMPSON. *Biochem. J.*, 34 (1940), 998. (F. J. S.)

Capillary Fragility and Ascorbic Acid Studies. In a study on a group of 150 children, in which titrations of vitamin C content in the blood plasma and capillary fragility tests were made, there appeared to be no correlation between abnormal capillary fragility and avitaminosis C.—H. G. RAPAPORT, S. H. MILLER and A. SICULAR. *J. Pediatrics*, 16 (1940), 624; through *Chem. Abstr.*, 34 (1940), 4768. (F. J. S.)

Carotenoids and Vitamin A—Chemical Determination of, in Serum. In the determination of serum vitamin A by the Carr and Price reaction (*Biochem. J.*, 31 (1926), 497–501), cold saponification yields accurate results even with hemolyzed serum. Serum adsorption-analysis of the carotenoids is of value in the determination of the correction factor for the Carr-Price reaction. Lycopene could not be detected in serum, even when the diet was rich in that carotenoid.—H. WILLSTAEDT and T. K. WITH. *Z. Vitaminforsch.*, 9 (1939), 212–223; through *Chimie & Industrie*, 43 (1940), 198. (A. P.-C.)

Cobalt in Foods—Determination of. The determination of small amounts of cobalt in foods and other organic materials is a subject which has attracted attention in recent years, particularly in view of its bearing on "bush sickness" and similar diseases. Methods described in the literature on the subject have, however, usually been concerned with the analysis of the materials that yield a low ash, and consequently separation of the cobalt from the solution of the ash has not been necessary. A method is now described for the separation of cobalt from the ash of food materials, the cobalt being determined colorimetrically with nitroso-R-salt. Recovery of cobalt added to foods is satisfactory, and the normal cobalt content of a number of food materials has been determined. A qualitative test is described depending on the adsorption of the red color which cobalt gives with nitroso-R-salt.—N. D. SYLVESTER and L. H. LAMPITT. *J. Soc. Chem. Ind.*, 59 (1940), 57–60. (E. G. V.)

Detoxication—Studies in. V. The Preparation of *d*-Glucurone from Ammonium Menthylglucuronate. A new method is described for the preparation of glucurone from ammonium menthylglucuronate isolated from the urine of rabbits fed with *dl*-menthol. A yield of about 39–40 Gm. of glucurone per 100 Gm. *dl*-menthol fed was obtained.—R. TECWYN WILLIAMS. *Biochem. J.*, 34 (1940), 272. (F. J. S.)

Diazo Reaction of Blood Serum—New Argument in Favor of Attributing, to Histidine. By new refinements of technique it was shown that the Pauly diazo reaction of serums is due almost entirely to histidine. A method of estimating the histidine before and after transformation into imidazolelactic acid is described.—A. SCHWARTZ. *Compt. rend. soc. biol.*, 131 (1939), 519–522; through *Chimie & Industrie*, 42 (1939), 794. (A. P.-C.)

α-Estradiol and Estrone—Isolation of, from Horse Testes. Using 28 Kg. of horse testes α-estradiol has been isolated (0.21 mg./Kg.) as its di-α-naphthoate and estrone (0.36 mg./Kg.) as its 3:5-dinitrobenzoate. The concentrations of estrone and of α-estradiol in horse testes are much higher than the values reported for any other tissue to date.—D. BEALL. *Biochem. J.*, 34 (1940), 1293. (F. J. S.)

Estrogens—Determination of, in the Urine, in Cases of Uterine Hemorrhage. Fourteen cases of uterine hemorrhage are described. Six cases were diagnosed as *metropathia hemorrhagica* and among these the outputs of estrogens were uniformally low. A. P. L. treatment elevated the urinary levels of estrogens and checked the hemorrhage. The treatment is discussed.—HAZEL LIN. *Chinese Med. J.*, 57 (1940), 216–230. (W. T. S.)

Fumarate and Malate—Determination of, in Animal Tissues. A method for the determination of fumarate and *l*(—)malate is described. Fumarate is reduced to succinate in the presence of zinc and phosphoric acid and the succinate formed is determined manometrically with succinic dehydrogenase. The concentration of *l*(—)malate is calculated from the equilibrium constant. At p_H 7.4 the ratio *l*(—)malate/fumarate was found to be 2.65 at 50°, 3.17 at 40°, 3.54 at 30° and 4.57 at 20°. A figure is given of the succinic acid extractor.—HANS ADOLF KREBS, DAVID HENRY SMYTH and EARL ALISON EVANS, JR. *Biochem. J.*, 34 (1940), 1041. (F. J. S.)

Gelatin with Vitamin A, Etc.—Composition of. In making an "activated" gelatin product, in finely divided form, a polyphase dispersion is produced in which liquid droplets of gelatin containing a vitamin A concentrate or the like dispersed therein, constitute the dispersed phase and a vegetable oil, such as castor oil, constitutes the continuous phase, the droplets are caused to solidify (as by cooling to about 8° C.), oil is removed by use of an oil solvent such as "industrial methylated spirit," and at least part of the water is removed, as by the use of alcohol.—STANTON REYNOLDS, assignor to ATLANTIC COAST FISHERIES CO. U. S. pat. 2,183,084, Dec. 12, 1939. (A. P.-C.)

Glucose—Renal Threshold for. The study of the renal threshold in diabetics and normals with healthy renal function shows frequent deviations from the accepted "normal" figure of some 170 mg. per 100 cc. The conception of a "normal" threshold is false. There is an average renal threshold, just as there is an average blood pressure, and the many deviations from it should be looked upon as physiological—a matter of individual idiosyncrasy, and of no pathological significance. High and low thresholds are frequent and usually persist at the same level throughout life. A few factors outlined above affect the threshold temporarily or even permanently. The stigma of something abnormal (and hence pathological) that has been imputed to a high or low renal threshold should be removed.—R. D. LAWRENCE. *Brit. Med. J.*, 4140 (1940), 766. (W. H. H.)

Glutathione—Colorimetric Determination of Reduced. Deproteinize with 5% metaphosphoric acid; to 1 cc. of filtrate add 4 cc. of saturated sodium chloride, 0.5 cc. of a freshly prepared 2% sodium nitroprusside solution and 0.5 cc. of normal ammonia. The red color developed in the presence of glutathione is examined in the Pulfrich photometer with a 10-mm. cell and filter S53. The temperature must be taken into consideration. The glutathione content is calculated as follows: mg. % = $(E - E_0)f_{t.v}$, where E and E_0 are the extinction coefficients of the unknown and blank,

respectively, v is the dilution and f_t the temperature factor which is tabulated for various temperatures.—A. FUJITA and I. NUMATA. *Biochem. Z.*, 300 (1939), 246–256; through *Chimie & Industrie*, 42 (1939), 636. (A. P.-C.)

Glutathione—Iodometric Determination of, in Tissues. II. For the determination of glutathione in both the reduced and oxidized forms, the latter is reduced by means of hydrogen sulfide, the ascorbic acid is removed by oxidizing with oxidase and total glutathione is titrated with potassium iodate in presence of potassium iodide.—A. FUJITA and I. NUMATA. *Biochem. Z.*, 299 (1938), 262–273; through *Chimie & Industrie*, 42 (1939), 31. (A. P.-C.)

Glycuronic Acid and Its Conjugated Compounds—Quantitative Estimation of, by Means of the Naphthoresorcinol Test of Tollens. The naphthoresorcinol test of Tollens is suitable under certain conditions for the quantitative determination of glycuronic acid in pure solution. The modification used by Maughan, *et al.* (1938), for the estimation of pregnandiol-glycuronic acid was tested on solutions of glycuronic acid, glycurone, benzoylglycuronic and bornylglycuronic acids with satisfactory results. It was found, however, that if the sample examined contains more than 0.05 mg. glycuronic acid variable results are obtained. Single estimations cannot be relied on and series of estimations at varying dilutions are therefore necessary; only the series which shows proportionality between the concentration of glycuronic acid and the intensity of color is used. The method can be applied to blood and urine but account must be taken of interfering substances. Errors can be avoided by serial estimations, choosing only the series which shows proportionality. In urines which contain interfering substances the estimation can only be carried out if the concentration of glycuronic acid is sufficiently high to allow a considerable dilution.—W. MOZOLOWSKI. *Biochem. J.*, 34 (1940), 823. (F. J. S.)

Hair. The physiology of the hair, factors affecting its growth, graying and baldness are discussed. Twenty-five references.—M. A. LESSER. *Drug and Cosmetic Ind.*, 47 (1940), 645–648. (H. M. B.)

Hair Keratin—Analysis of. I. A Method for the Quantitative Removal of Cystine from Keratin Hydrolysates. The authors summarize their work as follows: (1) Cuprous oxide can be used to remove cystine quantitatively from protein hydrolysates. (2) Since the cuprous oxide procedure to remove cystine does not require the use of alkalis or inorganic salts, the remainder of the hydrolysate is in a suitable condition for the application of other isolation procedures. (3) Cuprous oxide simultaneously removes most of the chloride ion and all of the colloidal coloring matter from the hydrolysate. (4) Pure crystalline cystine (or cysteine hydrochloride) may be readily obtained in excellent yield from the cuprous mercaptide precipitated. (5) The yield of pure cystine isolated from a human hair hydrolysate has been considerably increased above the usual amount obtained by fractional crystallization of neutralized hydrolysates.—COLIN CAMERON LUCAS and JAMES MACDONALD RICHARDSON BEVERIDGE. *Biochem. J.*, 34 (1940), 1356. (F. J. S.)

Hevea Brasiliensis—Proteins of. I. Analysis of a Product Isolated from Dried Latex. A protein has been isolated from dried *Hevea* latex and analyzed for amide-N, tyrosine, tryptophan, cystine, methionine, the base-amino acids and the dicarboxylic acids. The dicarboxylic acids were estimated in 4.0 Gm. of protein; experiments on casein having shown that, under such conditions, approximately 90% of the glutamic acid and aspartic acid were recovered as crystalline derivatives. It was also found possible to estimate arginine and lysine in the filtrate from the calcium salts of the dicarboxylic acids. The protein is, in many respects, similar to those found in the leaves of other plants.—G. R. TRISTRAM. *Biochem. J.*, 34 (1940), 301. (F. J. S.)

Histamine, Tyramine and Tryptamine Values—Colorimetric Determination of, in Blood Serum. Evaluation of Aminemia. The methods are discussed and the following modification of the procedure for the determination of histamine is given: Decompose 2 cc. of the aqueous solution of histamine hydrochloride (or other imidazol derivatives, as in blood serum) with 1 cc. of concentrated solution of sodium carbonate, then add 0.5 cc. of freshly prepared diazo reagent. A cherry-red color is produced; and immediately after add 1.5 cc. of 95% ethyl alcohol. The intensity of the color in the alcohol layer may be read in a Pulfrich photometer, using 5-cc. tubes with a depth of 1 cm. and filter S50. The following procedure is given for tryptamine: At least 30 cc. of serum are extracted with alcohol-acetone in acid medium (p_H 1–2) by heating on a water bath not quite to boiling and filtering while hot. Let stand over night and filter again. Remove the solvent, take up the residue in alcohol-acetone and evaporate to dryness with a mixture of mercurous oxide and acetic acid. Extract the residue with diluted alcohol-acetone, neutralize and clarify. Evaporate again to dryness, dissolve the residue in phosphoric acid, add a drop of alcoholic solution of *m*-nitrobenzaldehyde and heat on a water bath to develop the blue color. Compare the color with that developed by a standard solution of tryptamine hydrochloride. The clinical interpretations are discussed.—A. LESURE. *J. pharm. chim.*, 1 (1940), 55–69. (S. W. G.)

Hormone Extraction. Pictures showing processes in the preparation of sex hormones.—ANON. *Chemist and Druggist*, 134 (1941), 69. (A. C. DeD.)

Iodides—Detection of, in Urine by the Orthotolidine Test. The orthotolidine reagent has been established as a rapid presumptive test for the presence of iodine in the urine by reason of the purple-black color reaction. By use of this test it has been determined that following a dose of two to five grains of potassium iodide, the medicine is excreted gradually in the urine for periods ranging from 36 to 60 hours. The amount of iodine furnished by four grams of iodized table salt daily, or one grain of thyroid medicine daily, is not sufficient to cause any color change with orthotolidine. There are very few other medicines in common use that give the same color reaction in the urine. The test is of value as a rapid presumptive test for the detection of iodides in urine in cases where there is a suspected attempted suicide by swallowing tincture of iodine.—CHARLES C. GILL. *Military Surgeon*, 88 (1941), 184. (F. J. S.)

Iron and Copper—Factors Influencing the Absorption of, from the Alimentary Tract. The author summarizes the work as follows: (1) The dialysis of ferric iron, but not of ferrous ion, is inhibited by phosphatides and phosphoproteins. (2) Ferric iron may be reduced by certain foodstuffs. (3) Ferrous iron is oxidized readily to the ferric state in the presence of egg yolk, presumably by a process of autoxidation. (4) Ferric iron does not appear to be capable of absorption from the alimentary tract. Iron, to be absorbed, must be in the ferrous state. (5) Reduction of ferric iron can occur in the stomach, this being effected by the diet. (6) The absorption of both iron and copper from the alimentary tract is influenced by the gastric acidity and the calcium content of the diet. (7) The relation of these factors to human nutrition has been dis-

cussed.—S. L. TOMPSETT. *Biochem. J.*, 34 (1940), 961. (F. J. S.)

Iron in Blood—Colorimetric Determination of Total. The reaction between iron and potassium ferrocyanide producing ferric ferrocyanide is used in the determination of iron in blood. Estimation may be made on 0.02 cc. of blood from a finger prick. Add 1 cc. water, 1 cc. nitro-perchloric mixture (100 cc. HNO_3 and 40 cc. perchloric acid) and a few beads. Heat in a pyrex tube 15 to 20 minutes but not to dryness. Add 2 or 3 cc. of water and 0.1 cc. H_2SO_4, make up to 5 cc. with a 0.15% solution of potassium ferrocyanide. Let stand until color develops, transfer to a larger tube and make up to 25 cc. with water. Examine in Leif Photometer, using a filter of 620 Å. and 20 mm. thick.—EUGENIO E. VONESCH. *An. Farm. Bioq.*, 10 (1939), 124. (G. S. G.)

Isoandrosterone—Isolation of, from the Urine in a Case of Virilism. Isoandrosterone, androsterone and etiocholanol-3(α)-one-17 have been isolated from the urine of a woman suffering from virilism. No dehydroisoandrosterone has been found.—H. HIRSCHMANN. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 51. (A. E. M.)

Lactic Acid Content in Human Milk and Arakawa's Reaction. Lactic acid content in human milk is large in Arakawa-negative milk and small in Arakawa-positive milk.—S. SATO. *Tôhoku J. Exp. Med.*, 38 (1940), 237. (A. C. DeD.)

Lactose—Estimation of, in Milk. A test for estimating lactose in milk was needed to detect diluted buffalo's milk being sold as cow's milk. By removing casein with acetic acid and dialyzed iron, and employing methylene blue as an internal indicator, a previously reported method based on the use of Fehling's solution was made more rapidly and accurately provided a correction factor was used. After describing their new procedure, the authors compared it to other methods and outlined the conditions under which this was done. Under certain conditions and with the use of matching standards the amount of reduced copper could be estimated colorimetrically. Polarimetry of the clear milk filtrate gave high results.—N. K. GHOSH and B. K. DATTA ROY. *Indian J. Med. Research*, 27 (1940), 953–962. (W. T. S.)

Methemoglobin—Determination of, in Blood with the Photoelectric Colorimeter. The extinction of methemoglobin at the wave length of 5930 Å. was determined at different p_H values. A method was worked out depending upon conversion of methemoglobin to cyanmethemoglobin and determining the extinction in the band between 6000 and 6600 Å. It is confirmed that up to 8% of the total hemoglobin in the blood of normal persons may be methemoglobin.—R. HAVEMANN, F. JUNG and B. VON ISSEKUTZ. *Biochem. Z.*, 301 (1939), 116–124; through *Chimie & Industrie*, 42 (1939), 963. (A. P.-C.)

Nicotinic Acid—Estimation of, in Animal Tissues Blood and Certain Foodstuffs. I. Method. The procedure is discussed for the estimation of nicotinic acid in biological material by means of the cyanogen-*p*-aminoacetophenone method of Harris & Raymond. A quantitative study has been made of the various factors influencing the accuracy of the results: *e. g.*, alternative methods of extraction, rate of hydrolysis, removal of interfering substances by means of ethyl alcohol, effects of changes in p_H and of the final acidification. The specificity of the color reaction is discussed. As little as 1–2μg. of nicotinic acid can be detected in 1 Gm. of material and in control tests quantitative recovery of added nicotinic acid was effected. This method is more sensitive than those in which metol and aniline are used. In certain animal tissues nicotinic acid seems to be present bound to substances which are easily extracted by boiling water but which are partly precipitated by ethyl alcohol. In some cereals only a small fraction of the chromogen behaves like the nicotinic acid of animal tissues, *i. e.*, is readily extracted by boiling water. It is believed that only this fraction (or a portion of it) is the "true" or "active" nicotinic acid; otherwise yellow maize which is known to be deficient would have a high concentration of nicotinic acid.—E. KODICEK. *Biochem. J.*, 34 (1940), 712. (F. J. S.)

Nitrite—Manometric Estimation of, in Solution and in Tissue. Manometric methods for the estimation of nitrite are described. The methods are based on the reaction of NH_2SO_3H with nitrite in the Van Slyke or Warburg apparatus. The quantitative evolution of nitrogen from nitrite and NH_2SO_3H is not affected by the presence of NaCl, $NaNO_3$, sucrose, glucose and muscle extractives. Nitrite in muscle can be estimated by the manometric analysis of a H_2O extract of the tissue. With certain limitations nitrite in muscle can be estimated directly by the action of NH_2SO_3H on tissue contained in the Warburg apparatus. On mixing muscle and $NaNO_2$ there is both a rapid disappearance of a small amount of nitrite (of the order of 10^{-5} Gm. $NaNO_2$/Gm. tissue) and a slow disappearance of nitrite.—J. BROOKS and J. PACE. *Biochem. J.*, 34 (1940), 260. (F. J. S.)

Nutrition—Links in. Vitamin studies are reviewed.—A. L. BACHARACH. *Chemistry and Industry*, 59 (1940), 308. (E. G. V.)

Optimal and Marginal Diets—Composition of. A survey of the state of knowledge to-day.—A. L. BACHARACH and J. C. DRUMMOND. *Chemistry and Industry*, 59 (1940), 37–40. (E. G. V.)

Phosphorus—Estimation of. Certain defects in the King-Fiske & Subbarow method of phosphorus determination, which become serious when the method of analysis depends on the determination of absolute color density, are described. A method of phosphorus determination is proposed in which these adverse features are eliminated. A procedure for the estimation of phosphorus in cloudy or highly colored solutions is described.—RUSSELL JAMES LAURENCE ALLEN. *Biochem. J.*, 34 (1940), 858. (F. J. S.)

Placental Blood—Use of, in Transfusion. Because of emergency needs it is advantageous to have a stock of blood conserved for transfusion. Placental blood offers a source of supply which can be collected aseptically typed and preserved for use. It may be preserved with different anticoagulants. Sodium citrate 2.5% is most frequently used, though the Moscow Institute recommends a mixture of sodium chloride, sodium citrate, potassium chloride and magnesium sulfate in distilled water. Sodium citrate solution, 15 cc. to 100 cc. of blood, is slightly more likely to produce hemolysis after being kept than is the Moscow solution, which is used in equal parts with the blood. Citrated blood is kept in 200-cc. quantities in 300-cc. Erlenmeyer flasks in a refrigerator. Observations on 100 cases prove its efficacy even when preserved 50 days, though the usual time limit is 10 to 16 days.—HUGO MORGANTINI. *Col. Farm. Nac., Rosario*, 6 (1939), 14. (G. S. G.)

Plasma (Dried) for Transfusion. The method of withdrawing, storage and delivery of plasma is described. Details of the continuous-feed plasma drier are given for the preparation of dried plasma. The production of dried plasma is cheap and larger quantities may be prepared by reduplication or enlargement of the apparatus. The ideal plasma for administration is Group AB (I) plasma, but the

plasma of any group may be given to any patient up to 500 cc. Group AB (I) plasma can be administered in any amount; and it may be prepared artificially by mixing Group A (II) and Group (III) bloods and withdrawing the resultant plasma. The dried plasma may be carried in ampuls and administered by any intravenous saline apparatus. It can be stored at room temperature and will apparently remain effective indefinitely. The rationale for the administration of plasma in wound shock, postoperative shock, incipient pulmonary edema, burns, nephritic edema and malnutritional edemas is discussed. Twenty grams of dried plasma, dissolved in 250 cc. of distilled water or 500 cc. of 5% glucose in distilled water, is equivalent in plasma protein value to one pint of citrated blood. The authors' experience of its use is not great enough to assess its value, but its anti-shock property appears to be comparable to that of whole blood. It seems to be ideal for use in emergency, where no supply of blood is easily available and in war surgery.—F. R. EDWARDS, J. KAY and T. B. DAVIE. *Brit. Med. J.*, 4131 (1940), 377. (W. H. H.)

Plasma—Iron of. The "Barkan Effect" observed in plasma and serum has been shown to be due to the reduction of the plasma iron to the ferrous state with a consequent change from a non-ultrafiltrable to an ultrafiltrable condition.—S. L. TOMPSETT. *Biochem. J.*, 34 (1940), 959. (F. J. S.)

Protein Sugar—Determination of, and of Its Constituents. After hydrolysis with dilute sulfuric acid part of the hydrolyzate is dealbuminized with phosphotungstic acid and the remainder with mercuric nitrate. Both solutions are titrated with Bertrand's alkaline copper solution. Titration of the first portion gives total protein sugar (mannose, galactose and glucosamine), while the second gives only mannose and galactose.—H. BIERRY, B. GOUZON and COLETTE MAGNAN. *Compt. rend. soc. biol.*, 130 (1939), 1454–1456; through *Chimie & Industrie*, 42 (1939), 794. (A. P.-C.)

Prothrombin—Stability of, in the Presence of Thrombin. Thrombin and citrated prothrombin can co-exist with no more destruction of either than can readily be attributed to the natural instability of both preparations. Thrombin itself is neither an activator nor a destroyer of prothrombin. The lytic factor is an independent entity which seems to be an enzyme, probably serum tryptase.—JOHN H. FERGUSON. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 80. (A. E. M.)

Riboflavin Requirement of Growing Rats—Relation of Dietary Fat to. Increasing the fat level in a riboflavin-low ration has a corresponding deleterious effect on the growth of young rats. Administration of adequate amounts of riboflavin corrects completely this deficiency.—G. J. MANNERING, M. A. LIPTON and C. A. ELVEHJEM. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 100. (A. E. M.)

Seromucoid and the Bound Carbohydrate of the Serum Proteins. Seromucoid, the carbohydrate-rich, non-coagulable protein of serum has been reinvestigated and an improved method for its isolation worked out, leading to a main fraction of uniform properties. This contains 10.7% of carbohydrate, consisting of *N*-acetylglucosamine, galactose and mannose in equimolecular proportions, and also approximately 3% tyrosine, 1% tryptophan and 2.3% cystine. The specific rotation ($[\alpha]_{5461} - 54°$) is lower than that of serum albumin. Methods are described for the determination of non-amino sugars and glucosamine in proteins. The ratio of hexose/glucosamine was found to be 2/1 for all fractions examined.—C. RIMINGTON. *Biochem. J.*, 34 (1940), 931. (F. J. S.)

Serum Albumin. II. Identification of More than One Albumin in Horse and Human Serum by Electrophoretic Mobility in Acid Solution. Although crystalline serum albumin has been demonstrated to consist of more than one chemical individual, it has been reported to migrate with uniform velocity both during ultracentrifugation and electrophoresis at neutral reactions. At p_H 4.0, where a fraction of the horse serum albumin crystallizes as a sulfate, the protein exhibits two boundaries in the Tiselius electrophoresis apparatus. In normal human sera, the faster moving component constitutes nearly two-thirds of the albumin, but in certain pathological conditions in man, both serum and urinary albumins show a greater diminution of the faster component, leaving the slower component preponderant.—J. A. LUETSCHER, JR. *J. Am. Chem. Soc.*, 61 (1939), 2888. (E. B. S.)

Serum Albumin. I. The Preparation and Properties of Crystalline Horse Serum Albumin of Constant Solubility. Carbohydrate-free crystalline serum albumin has been separated into two fractions by crystallizing the sulfates from water. The solubility in salt solution of the protein regenerated from the insoluble albumin sulfate was found to be essentially independent of the amount of protein in the system, and of the volume of the solvent with which it was equilibrated, suggesting that this albumin component had been separated as a chemical individual. The percentage of nitrogen in this serum albumin was found to be higher, tyrosine lower and tryptophan the same as in unfractionated carbohydrate-free crystalline serum albumin.—T. L. MCMEEKIN. *J. Am. Chem. Soc.*, 61 (1939), 2884. (E. B. S.)

Sodium—Determination of, in Biological Fluids. The color developed with uranyl zinc sodium acetate by sulfosalicylic acid and sodium acetate has been studied. While the color does not follow Beer's law exactly, it has been found to be reproducible to time and temperature. A new method, consisting of precipitation of uranyl zinc sodium acetate from alcoholic solution, removal of the excess of precipitant by washing with ethyl acetate in acetic acid and with ether, removal of phosphate as uranyl phosphate and photoelectric measurement of the color developed by sulfosalicylic acid and sodium acetate, has been developed for the determination of sodium in biological fluids. Recovery of sodium from known amounts of sodium chloride, both in simple aqueous solution and added to blood sera, has indicated that the various steps, as well as the assembled procedure, have a maximum error of less than 1%.—M. C. DARNELL, JR., and B. S. WALKER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 242–244. (E. G. V.)

Sodium-Potassium—Antagonism of, in Plasma. Injection of hypertonic solutions of various sodium salts provokes an initial lowering of potassium in the plasma which in most cases recovers and increases above initial value. A study of various organs indicates that liver and muscles have the largest influence on these variations. A possible explanation is that the passage of sodium salts through the tissues blocks off a large quantity of potassium which eventually levels rapidly. Tests were made on normal animals, ones totally eviscerated and ones with kidneys and spleen ligated off.—C. CARDINI and J. L. MOGLIA. *An. Farm. Bioq.*, 10 (1939), 130. (G. S. G.)

Sterol Metabolism of Microörganisms. I. Yeast. The production of sterols by yeast under a variety of conditions has been studied. Of the various substrates examined, sodium acetate led to a slight increase in sterol; ascorbic acid gave a definite increase in one case and no effect in others; hydrolyzed yeast glycogen was not effective; the role of ethyl alcohol as a sterol precursor is open to doubt; a mixture of amino acids, tyrosine and tyrosol, respectively, in-

hibited sterol formation. It is established that sterol production (*a*) is a feature of aerobic metabolism, (*b*) arises early in the course of fermentation, (*c*) is accompanied by a corresponding increase in total lipin and (*d*) develops at a greater rate than growth, and is not directly correlated with it. Most of the new sterol formed is esterified.—WILLIAM HARVEY MAGUIGAN and ERNEST WALKER. *Biochem. J.*, 34 (1940), 804. (F. J. S.)

Sterols. LXXXI. Conversion of Sarsasapogenin to Pregnanediol-3(α),20(α). A brief outline of the method used in converting sarsasapogenin to pregnanediol-3(α),20(α) is given, and a more complete description of the process is promised in a later paper.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 3592. (E. B. S.)

Sterols. LXXVIII. Sterols from Sow Pregnancy Urine. Sow pregnancy urine differs from other pregnancy urines studied in that the pregnanediols appear to be absent. Pregnanol-3(α)-one-20, *allo*-pregnanol-3(β)-one-20, cholesterol and the urinary hydrocarbon were isolated. Evidence of the presence of androsterone was obtained.—R. E. MARKER and E. ROHRMANN. *J. Am. Chem. Soc.*, 61 (1939), 3476. (E. B. S.)

Sulfanilamide and Sulfapyridine—Oxidation of, by Hydrogen Peroxide. Pigments have been obtained by the oxidation of sulfanilamide and sulfapyridine which are absorbed by the blood fat of the cell envelope and this alters the color of the blood without affecting the oxygen capacity or the spectrum. These pigments are the probable cause of the cyanosis when both methemoglobin and sulfhemoglobin are absent.—G. V. JAMES, *Biochem. J.*, 34 (1940), 636. (F. J. S.)

Sulfanilamide—Isolation of Some Oxidation Products of, from Urine. *p*-*N*-Acetylhydroxylaminobenzenesulfonamide, *p*-hydroxylaminobenzenesulfonic acid and *p*-aminophenol have been isolated from the urine of patients treated with sulfanilamide. They appear to be excreted in conjugation with sulfates and glycuronates. The *p*-aminophenol undergoes further change to give rise to a pigment which is also excreted. It is suggested that these compounds may be the cause of some of the toxic effects noted during therapy with sulfanilamide.—G. V. JAMES. *Biochem. J.*, 34 (1940), 640. (F. J. S.)

Vitamin A and D Preparations, D.A.K. Danish Apothecaries Society Control Laboratory formulas, preparation methods and standards are cited for a vitamin A concentrate (100,000 I. U. vitamin A per Gm.), a vitamin D concentrate (20,000 I. U. vitamin D per Gm.) and for peanut oil mixtures thereof called: Adetamin (7000 A units and 500 D units per Gm.), Adetamin Forte (42,000 A units and 3000 D units per Gm.) and Adetamin oil (700 A units and 50 D units per Gm).—ANON. *Arch. Pharm. Chemi*, 47 (1940), 683. (C. S. L.)

Vitamin A—Determination of, in the Form of the Free Alcohol. In blood much of the vitamin A is in a nonesterified form and Chevallier's photochemical method (*Z. Vitaminforsch.*, 7 (1938), 10-16) is not suitable for its estimation. A possible modification consists in subjecting vitamin A in the form of free alcohol to continuous ultraviolet irradiation until the absorption curves become stabilized.—A. CHEVALLIER, P. DUBOULOZ and R. MATHERON. *Compt. rend. soc. biol.*, 131 (1939), 372-373; through *Chimie & Industrie*, 42 (1939), 963. (A. P.-C.)

Vitamin B and G Concentrate from Yeast. A process for the preparation of a vitamin B and G concentrate from yeast comprises plasmolyzing yeast, adjusting the p_H of the mass to 5.0 to 6.5, coagulating protein material by adding methanol, ethanol or ethyl acetate to a concentration of 50% to 60% of the mass whereby filterability thereof is increased, filtering and concentrating the filtrate so obtained at a temperature not exceeding 75° to 80° C.—ROBERT F. LIGHT and CHARLES N. FREY, assignors to STANDARD BRANDS, INC. U. S. pat. 2,184,748, Dec. 26, 1939. (A. P.-C.)

Vitamin B_1 Complex of Liver. The Identity of the Liver Filtrate Factor with Pantothenic Acid. The liver filtrate factor has been shown to be identical with pantothenic acid. Evidence is presented that the factor in liver extracts which is neither adsorbed by fuller's earth nor extracted from acid aqueous solution by amyl alcohol is a biological entity (factor β) and that a third unidentified factor (γ) is present in acid-autoclaved whole extract of liver. A synthesis of γ:δ-dihydroxyvaleryl-β-alanine methyl ester is described.—B. LYTHGOE, T. F. MACRAE, R. H. STANLEY, A. R. TODD and C. ELIZABETH WORK. *Biochem. J.*, 34 (1940), 1335. (F. J. S.)

Vitamin B_1—Determination of, in Urine, Gastric and Duodenal Fluids, Bile, Milk and Blood Serum. Vitamin B_1 was always present in normal urine, but in widely varying proportion. Bile and gastric juices contained only traces, even after intravenous injection of large doses. After addition of small quantities of blood to gastric juice vitamin B_1 disappears from the latter, in spite of the fact that the vitamin is resistant to changes in p_H. Addition of blood has the same effect on vitamin C.—L. BENACCHIO. *Diagnostici tec. lab.*, 10 (1939), 81-93; through *Chimie & Industrie*, 42 (1939), 452. (A. P.-C.)

Vitamin B_1—Distribution of, in Some Plant Families. The author gives the following conclusions: (1) The vitamin B_1 content of different parts of a number of plants from certain important botanical families has been determined by the thiochrome technique. The results obtained compare favorably with published figures where these are available. (2) In general, the vitamin B_1 content is highest in seeds. In most leaves the concentration of vitamin B_1 amounts to a constant value of about 25 I. U. per 100 Gm., regardless of the botanical family. (3) The possibility is tentatively suggested that variation in vitamin B_1 content of fruits of the *Rosaceæ* is due to the degree of ripeness.—MAGNUS PYKE. *Biochem. J.*, 34 (1940), 330. (F. J. S.)

Vitamin B_1—Effect of a High-Fat Diet on the Excretion of Bisulfite-Binding Substances in the Urine of Rats Deficient in. The ingestion of a high-fat diet by rats deprived of vitamin B_1 diminished the excretion of bisulfite-binding substances in their urine and alleviated the severity of the bradycardia. The aneurin-sparing action of fat, therefore, is not limited to its effect in protecting such animals against loss of weight or the development of the better known symptoms of deficiency, but shows itself likewise in the absence of these two characteristic signs of avitaminosis. The significance of these results for vitamin assays, and for the assessment of deficiency, is discussed.—G. G. BANERJI. *Biochem. J.*, 34 (1940), 1329. (F. J. S.)

Vitamin B_1—Estimation of, in Urine. The following conclusions are given: (1) A rapid and simple method of estimating vitamin B_1 as thiochrome is applied to urine. The powdered Decalso used is a more specific adsorbent of the vitamin than are activated earths and gives lower "blanks" and more consistent yields. Fluorescence is measured with the Pulfrich photometer. (2) The method does not estimate low dietary excretions accurately, but is suitable for measuring the response to test-doses, such response being the best test of nutritional level. (3) Measurements of urinary excretion at short intervals after ingestion of 5 mg. of vitamin show that the time lag before excretion depends mainly

on the time required for the stomach to empty and that the shortest suitable period of urine collection in saturation tests is five hours. The kidney can concentrate vitamin B_1 from plasma.—MAURICE JOWETT. *Biochem. J.*, 34 (1940), 1348. (F. J. S.)

Vitamin B_1—Experiments on the Biogenesis of. Yeast fermentation of *dl*-α-amino-β-(4-methylthiazole-5)-propionic acid leads to 4-methyl-5-(β-hydroxyethyl)-thiazole and the *d*(−)-form of the amino acid. The initial stages of a synthesis of the α-aminopropionic acid analogous to aneurin are described.—C. R. HARINGTON and R. C. G. MOGGRIDGE. *Biochem. J.*, 34 (1940), 685. (F. J. S.)

Vitamin B_1—Preparation of. 2-Methyl-4-amino-5-formaminomethylpyrimidine is treated with γ,γ-diaceto-γ-mercaptopropyl alcohol and glacial acetic containing hydrochloric acid (suitably by heating at 100° to 110° C. for 2 hours). Details of making the initial reacting materials are given and a crystalline compound melting at 245° C. is obtained.—TAIZO MATUKAWA and MASAKI OTA, assignors to K. K. TAKEDA TYOBEI SYOTEN. U. S. pat. 2,184,720, Dec. 26, 1939. (A. P.-C.)

Vitamin B_1. Preparation—Assay of a Variety of, by the Fluorophotometric Method. The cost of biological assays and the time required are disadvantagous. Chemical methods cannot replace biological ones but are useful for preliminary testing so as to reduce the amound of biological testing to a minimum. A fluorophotometric method which can be used on complex mixtures has been devised. The method is described as well as the fluorophotometer used. The method has been applied to a considerable number of raw materials, mixtures and finished products. Results of these are tabulated along with bioassay figures.—J. W. COLE, W. S. JONES and W. G. CHRISTIANSEN. *Jour. A. Ph. A.*, 29 (1940), 434. (Z. M. C.)

Vitamin B_6—Synthesis of. II. Variations and improvements in the synthesis of vitamin B_6 have been made. The compounds 2-methyl-3-hydroxy-4-ethoxymethyl-5-hydroxymethylpyridine and 2-methyl-3-amino-4-ethoxymethyl-5-aminomethylpyridine have been hydrolyzed directly to the corresponding hydroxy derivatives by heating with dilute hydrochloric acid at 150–175° under pressure. Derivatives of some of the intermediates and a new inner ether of vitamin B_6 have been described.—S. A. HARRIS and K. FOLKERS. *J. Am. Chem. Soc.*, 61 (1939), 3307. (E. B. S.)

Vitamin C in Certain Fruits. Juices of oranges and lemons were studied under different conditions: exposed to air, in the daylight, in vacuum, in darkness and exposed to carbonic acid. The best results for preservation of the vitamin C content were obtained from pressed juice kept at room temperature in daylight with carbonic acid.—CARLOS A. SAGASTUME, *et al. Rev. facultad cienc. quím.*, 14 (1939), 193. (G. S. G.)

Vitamin C—Relation of Urinary Output of, to Saturation in Bengali Boys. The average daily urinary output of free and total ascorbic acid of 17 Bengali schoolboys has been determined. Excepting five boys who took fruits during the experiments, all had a daily excretion of less than 13 mg. of free ascorbic acid and in one case less than 1.5 mg. Over 1000 mg. of ascorbic acid were required to produce saturation in the twelve individuals not taking fruit. In no cases were symptoms of scurvy observed. There is no proportional relationship between the amount of vitamin C excreted and the degree of saturation unless the excretion is some 30–40 mg. per day. The excretion of dehydro- and combined ascorbic acid was compared to that of free ascorbic acid under both normal conditions and during periods of excess intake of vitamin C. Excretion of the former has no relationship to the latter, but when all three appear in the urine the ratio of dehydro- to free acid and the ratio of combined to free acid lies in a certain range which is almost similar in both cases.—N. M. BASU and G. K. RAY. *Indian J. Med. Research*, 27 (1940), 917–930. (W. T. S.)

Vitamin D Concentration—Process of. Material containing either natural or synthetic vitamin D is dissolved in an organic solvent such as benzene or ether and the solution is passed through a mass of activated tricalcium phosphate which adsorbs a major portion of the vitamin D; the phosphate is washed with an organic solvent and the vitamin D is separated by distilling off the solvent.—SIDNEY E. MILLER, assignor to GENERAL MILLS, INC. U. S. pat. 2,179,560, Nov. 14, 1939. (A. P.-C.)

Vitamin K—Chemistry of. A review covering its discovery, structure, assay and clinical use.—C. R. ADDINALL. *Chemistry and Industry*, 59 (1940), 233. (E. G. V.)

Vitamin Problems. This is a review article concerning various open questions about vitamins such as the value of the citrin content of natural vitamin C concentrates as compared to the therapeutic value of synthetic ascorbic acid, the comparative biological value of vitamin K and synthetic substitutes, the human daily requirements of various accessory factors, the possibility of standardizing vitamin preparations by other than biological methods and the lack of such a chemical or physical method for vitamin D.—E. EHRENBERG. *Farm. Revy*, 39 (1940), 581. (C. S. L.)

Whey as a Source of Vitamins and Vitamin Products. Whey or milk serum following removal of the fat and casein may serve as the starting material for the further isolation of lactalbumin, milk sugar, milk phosphates, mixed sterols, cholesterol, fat-soluble vitamin K (prothrombin) and water-soluble vitamins, particularly commercial quantities of lactoflavin (riboflavin). The presence of an ostrogenic substance has been demonstrated in fractions derived from whey. The multiplicity of vitamins found in the whey fraction after removal of protein and milk is adequate in water-soluble factors to support growth, reproduction and lactation. Eight successive generations of white rats have been maintained with a normal life cycle on a restricted experimental diet in which the whey vitamin fraction supplemented with rice polish served as the sole source of all vitamins except the fat-soluble factors carried by a small percentage of cod liver oil.—G. C. SUPPLEE. *Ind. Eng. Chem.*, 32 (1940), 238–240. (E. G. V.)

Wines—Tannin Materials in. The role of polyphenols and tannin materials in wine making, and methods of determining them, are reported.—NÈGRE. *Compt. rend. acad. agric. France*, 25 (1939), 647–652; through *J. Soc. Chem. Ind.*, 59 (1940), 236. (E. G. V.)

Yeast Ribonucleic Acid—Constitution of. Guanine Uridylic Acid. Evidence of the existence of guanine uridylic acid is presented.—J. M. GULLAND. *Chemistry and Industry*, 59 (1940), 321–324. (E. G. V.)

Zinc and Copper Contents of the Organs and Tissues of Chinese Subjects. An investigation into the distribution of zinc and copper in the human body, involving over 300 zinc and 200 copper analyses of 13 different organs and tissues from 26 Chinese subjects of both sexes, ranging in age from 156 days to 60 years and dying from various causes, has shown that both metals are always present. Comparison with the average amounts of these metals present in the dry matter of blood shows that

zinc is concentrated in all organs and tissues examined, but that copper is concentrated only in the liver, brain, kidney and heart in order of descending magnitude. Cerebrum and cerebellum contain less zinc and, with the exception of liver, more copper than any organ or tissue examined. Cerebellum contains more zinc and copper than cerebrum. The increased concentration of zinc and copper in the liver and kidney observed in cases of malnutrition is thought to be due to the relatively greater withdrawal of the fats and carbohydrates from these organs, than of the proteins with which the metals remain in association.—WILLIAM GEORGE ELFORD EGGLETON. *Biochem. J.*, 34 (1940), 991. (F. J. S.)

ANALYTICAL CHEMISTRY

Acetylsalicylic Acid Preparations—Characterization of. Iodometric Determination of Salicylic Acid. Determinations of melting points are unsuitable for detecting small differences in chemical composition of acetylsalicylic acid (I) preparations. The double alkalimetric titration and determinations of free and total acetic acid are not sufficiently accurate. The accuracy of the iodometric determination of the salicylic acid (II) content of I permits the detection of impurities due to acetic acid or II by-products in I preparations. In this method the concentration of the excess of sodium hydroxide is without effect on the iodine consumption only within certain limits. Iodine excess and time of keeping are not critical. The factor 6 iodine to 1 molecule of II has been confirmed.—D. KRUGER. *Z. anal. Chem.*, 117 (1939), 318–325; through *J. Soc. Chem. Ind.*, 59 (1940), 85. (E. G. V.)

Alcohols—Determination of, in Dilute Aqueous Solutions. The method of Fischer and Schmidt (*Chem. Abstr.*, 20 (1926), 2802) depends on the esterification of the alcohol with a saturated solution of potassium nitrite in the presence of carbon dioxide and acetic acid. By heating to about 60° the ester is volatilized and after absorbing the nitrous acid present the action of the ester on potassium iodide solution causes the liberation of an equivalent quantity of iodine which is titrated with thiosulfate. Modifications in the procedure are suggested. Nitrogen is used in place of carbon dioxide and, for the absorption, the bulb tube is replaced by a gas-washing flask containing a fine-grained plate of sintered glass and a high column of liquid. Potassium hydroxide is used for absorbing the nitrous acid instead of sodium bicarbonate solution. A suitable apparatus is illustrated, and results obtained with various alcohols show that the method is accurate to within about 1% of the theoretical.—R. SKRABAL. *Z. anal. Chem.*, 119 (1940), 222–226. (S. W. G.)

Aluminum Sulfate—Determination of Free Acid in, When Iron and Ammonium Salts Are Present. To 1 Gm. of the salt add 40 cc. of carbon dioxide-free water. If the salt dissolves with difficulty add a carefully measured volume of standardized 0.2*N* sulfuric acid (about 4 cc.). To the cold solution add 1 Gm. of potassium persulfate to oxidize ferrous iron. In another flask dissolve 10 Gm. of monohydrated potassium fluoride in 100 cc. of water. To this add 0.5 cc. of phenol red in ethanol, add 0.2*N* sulfuric acid until a distinct yellow color is obtained and then just enough 0.2*N* sodium hydroxide to give a carmine color. Pour this cold, neutralized potassium fluoride solution into the cold aluminum sulfate solution and titrate with 0.2*N* sodium hydroxide to a distinct red color (light red if ammonium alum is present) which persists for at least twenty seconds. If more than 0.3% of sulfuric acid is found present, repeat the analysis but, before adding the potassium fluoride solution, add sufficient sodium hydroxide so that in the final titration only 0.2–0.3 cc. of the sodium hydroxide solution will be needed.—T. EDER. *Z. anal. Chem.*, 119 (1940), 409–412. (S. W. G.)

Amines—Some Derivatives of Indane Group as Reagents for. II. Detection of Primary Di- and Polyamines with Bindone. The tabulated results obtained by testing fifty organic compounds show that aliphatic and aromatic di- and polyamines give with bindone (anhydrobisindanedione) in glacial acetic acid the same reaction as do the monoamines. With the aromatic diamines, the sensitivity of the test is influenced by the position of the amino groups; the closer the groups, the more sensitive the test. 1,8-Naphthylendiamine gives no test but hexamethylenetetramine gives a green coloration.—G. VANAGS. *Z. anal. Chem.*, 119 (1940), 413–418. (S. W. G.)

2-Aminoethanol—Identification of. Mix equimolar proportions of oxalic acid and 2-aminoethanol (for example, dissolve 0.090 Gm. of oxalic acid in water and add it to the aqueous solution containing 0.061 Gm. of the 2-aminoethanol). Evaporate the solution to dryness and heat the residue at 110° C. for 5 minutes. Recrystallize from 70% alcohol. The resulting elongated hexagonal plates melt with decomposition at 199–200° C., uncorrected. The salt is very soluble in water and insoluble in absolute alcohol or glacial acetic acid. Analysis for $C_6H_{16}O_6N_2$: calculated, N 13.20, oxalic acid 42.5; found, N 12.65, oxalic acid 44.0. If heated past the melting point of about 222° C., brisk boiling occurs with formation in good yield of N,N-bis-(2-hydroxyethyl)-oxamide, m. p. 168° C. uncorrected.—B. KEISER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 284. (E. G. V.)

Aqueous Solutions—Continuous Extraction of, by Acetone-Petroleum Ether. The extractor, useful for mixed solvents, is described.—M. WAYMAN and G. F. WRIGHT. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 91. (E. G. V.)

Aromatic Materials—Fluorescence of, in Filtered Ultraviolet Light. Out of 252 aromatic substances which were examined, 160 (about 63%) show a more or less violet fluorescence; 37 (about 15%) a radiant violet; 13 (about 5%) a bright white and blue; 22 (about 9%) a light greenish; 10 (about 4%) a bright yellow-greenish coloring under the influence of filtered (nickel oxide filter) ultraviolet light. The frequency of diffusion of the various fluorescences is shown in a table.—W. E. BAIER. *Perfumer. Essent. Oil Record*, 31 (1940), 307. (A. C. DeD.)

Arsenic—Determination of Trivalent, Alone or in Presence of Antimony, with Silver Nitrate. Arsenite is oxidized to arsenate by means of 30% hydrogen peroxide in a buffered solution, the arsenate is precipitated with silver nitrate solution, the mixture is filtered and the silver arsenate precipitate is dissolved in nitric acid. The nitric acid solution is titrated with thiocyanate using ferric alum indicator. The presence of antimony does not interfere although more hydrogen peroxide is needed. To 10–20 cc. of solution containing trivalent arsenic and antimony add 3–15 drops of perhydrol, 5 cc. of 20% ammonium nitrate and a drop of methyl red indicator solution. Neutralize the acid solution with 10% ammonia solution until the color of the solution is yellow, then make acid with acetic acid and add 10 cc. of 20% ammonium acetate and 20–25 cc. of water. Heat the solution until it begins to boil at 103–105°, remove the flame and stir until a temperature of 93° is reached, then add 3–4 cc. more of 0.1*N* silver nitrate than was found necessary in a preliminary trial. Filter after twelve hours, wash three times with 0.001*N* silver nitrate and once with water. Dissolve the precipitate in 2*N* nitric acid and titrate the solution by the Volhard method. A black residue

may remain on the paper after all the silver arsenate has been dissolved from it. The black residue should be disregarded. Excellent results were obtained in determining 10–50 mg. of arsenic but the values were low when less than 0.2 as much arsenic as antimony was present. The antimony can be determined in another portion of the solution by the bromate method.—E. CHIRNOAGA. *Z. anal. Chem.*, 120 (1940), 9–15. (S. W. G.)

Arsenic—Titrimetric Determination of Very Small Quantities of, in the Presence of Antimony. The method of Gangl and Vazquez Sanchez depends on the formation of an arsenic mirror by the Marsh test, solution of the mirror in iodine chloride and titration with potassium iodate in the presence of strong hydrochloric acid and hydrocyanic acid. The iodine chloride oxidizes the arsenic mirror to arsenic acid with liberation of an equivalent quantity of iodine which in turn is oxidized to iodine cyanide by the potassium iodate. The conclusion drawn by Gangl and Vazquez Sanchez that granulated zinc is less satisfactory than powdered zinc appears erroneus. Moreover, the transformation of the arsine into arsenic is not quite complete and if antimony is present it is determined together with the arsenic. It is true that 0.005 to 0.100 mg. of arsenic can be determined with a negative error of not over 5%. If tin and hydrochloric acid are used for the reduction, only about one-quarter of the time is required for an analysis, the loss of arsenic is no greater and no antimony is determined with the arsenic because tin and hydrochloric acid do not reduce trivalent antimony solutions to silbine.—J. BODNAR, E. SZEP and W. CIELESZKY. *Z. Anal. Chem.*, 115 (1939), 412–420; through *Chimie & Industrie*, 42 (1939), 795. (A. P.-C.)

Ascorbic Acid—An Iodate Assay for Relatively Pure. A method for the assay of relatively pure ascorbic acid, involving oxidation with potassium iodate volumetric solution, has been developed. Comparison of the results obtained by this method with those obtained by applying the U. S. P. XI Second Supplement iodine oxidation procedure indicates that the former is capable of greater precision than the official method.—W. BANDARUK. *Am. J. Pharm.*, 113 (1941), 18. (A. C. DeD.)

Ascorbic Acid—Phosphotungstic, Arsenotungstic and Arsenotungstomolybdic Reagents in Photometric Determination of. The reagents are: (1) Sodium tungstate 20 Gm., sodium molybdate 2 Gm., arsenic acid 50 Gm., water 150 Gm., (2) Sodium tungstate 25 Gm., arsenic acid 20 Gm., water 250 Gm., (3) Reagent of Folin-Marenzi. Mix 1 cc. of a solution containing about 0.2 mg. of ascorbic acid with 1 cc. of the reagent, add, after a few minutes, 3 cc. of a 20% sodium carbonate solution (in case of reagent No. 1 use 5 cc.) and 15 cc. water. Allow 15 minutes to develop the blue color and fill up to a volume of 100 cc. The sensitivity of the test is 0.005 mg. The reaction follows the law of Lambert-Beer. For the photometric reading a filter of 620 Å. is used. The coefficients of specific extinction are 7.95 for reagent 1, 8.02 for 2 and 8.0 for 3. The coefficient for molecular extinction is 11.31. The method can be applied to tablets and injectable solutions.—EUGENIO E. VONESCH and ALFREDO L. REMEZZANO. *Rev. farm. (Buenos Aires)*, 82 (1940), 431. (A. E. M.)

Ash in Organic Compounds. In the determination, samples of 100 to 150 mg. are weighed in the platinum boat on a microbalance with the cylinder also included in the weighing. The empty weight of the boat with the cylinder must be taken as accurately as possible, but a sample of this size need only be weighed to the nearest 0.1 mg. The boat is then inserted in the cylinder and the cylinder placed in the end of the quartz tube with the tube extending out of the furnace. A turn of a knob engages the tube carrier to the moving screw and will continue to pull the tube into the furnace until the end of the tube is completely in the furnace. Then the engaging pin runs off the end of the screw and the tube moves no farther. At this point the combustion has required 20 minutes and is complete. The cylinder and boat are removed, cooled for 5 minutes and weighed as accurately as possible.—A. R. NORTON, G. L. ROYER and R. KOEGEL. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 121–123. (E. G. V.)

Assay of Elixir of Terpin Hydrate and Codeine. The proposed standard is: Each 100 cc. of the elixir contains not less than 0.1820 Gm. and not more than 0.218 Gm. anhydrous codeine. The following assay is proposed: Measure accurately 100 cc. of the elixir, transfer into a 300-cc. separator and dilute with 50 cc. distilled water. Make the mixture alkaline with ammonia T.S. and extract with 25-cc. portions of chloroform until extraction is complete. Transfer the chloroformic solution into a separator and extract with 20-cc. portions of 2% sulfuric acid until extraction is complete. Discard the chloroformic solution. Place the acid solution in a separator, make alkaline with ammonia T.S., again extract with chloroform and again repeat the extraction with 2% sulfuric acid. Again extract the acid solution (after making alkaline with ammonia T.S.) with chloroform and place the combined chloroformic extracts in a wide-mouthed Erlenmeyer flask. Evaporate nearly to dryness, add 10 cc. 0.1*N* sulfuric acid and 20 cc. water and warm to drive off the excess chloroform. Titrate the excess acid with 0.02*N* sodium hydroxide using methyl red T.S. as indicator. Each cc. 0.1*N* sulfuric acid = 0.02992 Gm. anhydrous codeine.—WALTER D. DEMBECK. *Bull. Natl. Formulary Committee*, 9 (1941), 119. (H. M. B.)

Barbituric Acid Spectrum—Effect of p_H and Irradiation on. The ultraviolet absorption of barbituric acid in aqueous solution at various p_H's changed from a maximum molecular extinction of 31,000 at p_H 7.0 to 25,000 at p_H 11.0 and to a minimum of 6800 at p_H 3, the lowest p_H investigated. The marked change in absorption with change in p_H is consistent with the theory that tautomerism of the amide-imidol type is responsible for the change, since three groups in this molecule can undergo such isomerization. The sharp peak at p_H 7.0 indicates the additive absorption of several groups with similar absorbing characteristics. Ultraviolet irradiation resulted in a rapid progressive decrease in extinction at the 2600° Å. maximum, followed by increased absorption in the short-wave and the long-wave region of the spectrum.—JOHN R. LOOFBOUROW and MIRIAM M. STIMSON. *J. Chem. Soc.*, (1940), 1275–1277. (W. T. S.)

Benedict's Solution—Use of Monohydrated Sodium Carbonate in. Due to the hygroscopic character of anhydrous sodium carbonate which is in the present formula for the solution, it is recommended that 117 Gm. of sodium carbonate monohydrated be used in the formula instead.—EDWARD M. GERSTENZANG. *Bull. Natl. Formulary Committee*, 9 (1941), 99. (H. M. B.)

Bismuth—Determination of, as Bismuth-Chromium Thiocyanate. Potassium-chromium thiocyanate ($K_3[Cr(SCN)_6]$) is an excellent precipitant for bismuth ions. Other ions of the hydrogen sulfide group give precipitates but the presence of cations of Groups III and IV causes no interference. The reagent, however, does not keep very well and develops, on standing, an onion-like odor and then gives impure precipitates which may weigh as much as 3% too much. Very old preparations must be recrystallized from ethanol and in all cases the 4% reagent should be filtered through charcoal. To

precipitate 0.1 Gm. of bismuth dissolve 1 Gm. of potassium-chromium thiocyanate in 25 cc. of water, add 4–5 Gm. of animal charcoal, stir well and filter after 2–3 minutes. Allow the filtrate to drop into the bismuth solution which is at 25–30° and is 0.3–1.0*N* in nitric acid. Inoculate the solution with a very tiny crystal of solid potassium-chromium thiocyanate. After about 10 minutes filter off the dark red precipitate, wash it with cold water, dry at 120–130° and weigh. The precipitate contains 34.3% of bismuth.—C. MAHR. *Z. anal. Chem.*, 120 (1940), 6–9. (S. W. G.)

Caffeine Sodium Benzoate—Refractometric Method for the Determination of, in Solutions. The n_D^{20} and d_{20}^{20} of 1–30% (vol.) aqueous solutions of caffeine sodium benzoate were determined. A linear relationship was found between concentrations of the solutions and n_D^{20} and d_{20}^{20}. By the use of simple equations of these relationships or of graphs of the concentrations of caffeine sodium benzoate solutions can be determined from the values of n_D^{20} (with a 0.05% accuracy) and of d_{20}^{20} (with a 0.5% accuracy). However, n_D^{20} depends only on the total concentration of caffeine and sodium benzoate in the solution and it changes very little from variations of the caffeine contents between 30% and 45%. The d_{20}^{20} of solutions containing a large concentration of sodium benzoate (with a constant total concentration) is slightly higher, but this higher value is not sufficiently large for the determination of a smaller content of caffeine in the product.—V. L. PAVLOV and A. M. KLESHNYA. *Farmatsiya*, (1939), No. 1, 7–12; through *Chem. Abstr.*, 34 (1940), 4861. (F. J. S.)

Calcium—Estimation of Submicroquantities of. A method is described for the titrimetric estimation of submicroquantities of calcium (0.004 to 0.0400 mg.) with the use of the ordinary 5- to 10-cc. microburette. Calcium is precipitated as the oxalate in a specially designed centrifuge tube. The calcium oxalate is converted to the carbonate at 475° to 525° C. The carbonate is dissolved in an excess of 0.01*N* hydrochloric acid by heating. The excess hydrochloric acid is determined iodometrically by the addition of an excess amount of potassium iodate and potassium iodide, which releases an amount of iodine equivalent to the excess acid. The released iodine is determined by titrating with approximately 0.0007*N* sodium thiosulfate, using starch as an indicator. This procedure retains the theoretical and practical advantages of the methods where calcium oxalate is converted to calcium carbonate and combines with it the sensitivity of iodometric titrations. All solutions are standardized against the relatively stable 0.01*N* hydrochloric acid used.—A. E. SOBEL and I. A. KAYE. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 118–120. (E. G. V.)

Carbon Content of Organic Materials—Determination of. An apparatus which embodies the Pettenkofer principle has been devised.—B. E. CHRISTENSEN, R. WING and J. F. FACER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 364–365. (E. G. V.)

Carbonic Acid—Determination of. II. The apparatus used for the determination of carbon dioxide either gravimetrically or titrimetrically has been made much smaller so that not more than ten minutes is required to remove all carbon dioxide after the decomposition of the sample with diluted sulfuric acid is complete. By inserting tubes containing sulfuric acid with chromium trioxide and chromium trioxide alone, the analysis can be carried out without interference by hydrogen sulfide or sulfur dioxide. In the titrimetric method, the carbon dioxide is absorbed in a measured volume of standard sodium hydroxide solution and the absorption is aided by forcing the gas to pass through a plate of sintered glass which breaks it into a spray. After the absorption, the carbon dioxide is precipitated as barium carbonate and the excess alkali hydroxide is titrated with 0.1*N* hydrochloric acid to a bromothymol end-point. If less than 1% carbon dioxide is present, it is recommended to weigh the barium carbonate precipitate after drying at 110°. Particular precautions are necessary in filtering, and a blank test is desirable. The results are very good.—F. RICHTER. *Z. anal. Chem.*, 119 (1940), 335–351. (S. W. G.)

Cerate Oxidimetry. Eighteen solutions of the nitrato and perchlorato cerate ions in various concentrations of nitric and perchloric acid were prepared and their stability under various conditions and for extended time intervals was determined. A small but detectable photochemical decomposition was observed in the case of solutions stored in diffused daylight. Nitric and perchloric acid solutions of hexanitrato ammonium cerate between 1 and 3 molar in nitric and perchloric acids were found to show the highest stability. Perchloric acid solutions of perchlorato ceric acid required standardization at frequent intervals. The stability of all solutions increased with time, showing the influence of the accumulation of cerous ions in lowering the oxidation potentials, 5% to 10% increasing the stability two- and threefold. Five per cent to 10% of other rare earths of the cerium group present as impurities did not affect the stability adversely. A 0.1*N* solution of commercial hexanitrato ammonium cerate in 1.0*N* perchloric acid was sufficiently stable in ordinary light not to require restandardization for a period of 20 days.—G. F. SMITH and C. A. GETZ. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 339–340. (E. G. V.)

Choline—Study of Some Microchemical Reactions of. Because several organic substances give similar crystals it is necessary to find a specific reaction for the identification of choline. The following method has been found satisfactory. To a small drop of a solution of 1:1000 of choline hydrochloride placed on a microscope slide, add a drop of 2% phosphotungstic acid. This forms a precipitate which, under the high power of the microscope, shows colorless hexagonal planes. With phosphomolybdic acid in 2% solution added to a 1:1000 solution of choline hydrochloride definite crystals appear.—ARDOINO MARTINI. *Rev. Col. Farm. Nac., Rosario*, 6 (1939), 77. (G. S. G.)

Chromatographic Adsorptionanalysis—Use of, in Pharmacy. II. The chromatographic adsorption of *Extractum Æsculi Hippocastani Fluidum* (Swiss Pharm.) and of various samples of *Oleum Hyperici* (unofficial) was studied with the idea of separating some of the constituents. In the case of the fluidextract, the saponins passed through the aluminum oxide column. The bitter principle remained in the column. The tannin was adsorbed in the top zone and the quercitin in the second or third zone. The *Oleum Hyperici* in ether solution was passed through the column of aluminum oxide also. The eight samples of oil used showed some slight differences in their chromatographs. A red to violet zone appeared at the top and was generally followed by a green zone of chlorophyll and a narrow yellow zone which probably comes from the vehicle. A partial separation of the hypericumired and alkannin was indicated. The chromatograph may be used to indicate adulterants.—MARGUERITE FICHTER. *Pharm. Acta Helv.*, 14 (1939), 23–28. (M. F. W. D.)

Cobalt and Nickel—Electrolytic Determination of. Good results were obtained in determining nickel or cobalt in solutions containing 0.01–0.2 Gm. of cobaltous thiocyanate, 30 cc. of concentrated ammonia solution and 2.5 Gm. of ammonium sulfate with 0.5–

1.8 amperes.—P. CSOKAN. *Z. anal. Chem.*, 119 (1940), 418–421. (S. W. G.)

Copper—Colorimetric Estimation of Minute Amounts of, in Drugs. Two procedures for the determination of copper by means of dithizone are outlined, one involving operations in aqueous solution and the other in butyl alcohol solution. The first is considerably shorter and in general more convenient than the second. Both are rather involved.—G. VASTAGH. *Pharm. Zentralhalle*, 80 (1939), 273–281; through *Chimie & Industrie*, 42 (1939), 1023. (A. P.-C.)

Copper, Lead and Zinc—Determination of Small Quantities of, by Means of Dithizone, Particularly in Biological Substances. II. Ashing. Biochemical material and natural substances can be ashed dry in a quartz crucible for the analysis of copper, lead and zinc. The ash is treated with nitric acid, ashed again at 500° to 600° C., again treated with nitric acid and the acid is driven off. The final ash is dissolved in hot dilute hydrochloric acid. The determinations are carried out by the method described in *Biochem. Z.*, 297 (1938), 324–331.—J. SCHWAIBOLD and A. LESMÜLLER. *Biochem. Z.*, 300 (1939), 331–337; through *Chimie & Industrie*, 42 (1939), 963. (A. P.-C.)

Copper—Microdetermination of, in Biological Material. The diethyldithiocarbamate method for the estimation of copper in biological material is improved in simplicity, speed, accuracy and sensitivity. Rapid wet digestion, with a combination of sulfuric, perchloric and nitric acids in 8 × 1 inch Pyrex test-tubes, is employed for the destruction of organic matter, followed by deionization of iron with citrate or pyrophosphate in strongly alkaline medium. Subsequent color formation and extraction with amyl alcohol are effected in the combustion tube itself. With quantities such as 5 cc. of blood containing 3μg. of copper, the final reading is either colorimetric or photometric, but with limited material from small laboratory animals, involving the determination of real amounts of copper as low as 0.3μg., photometric finish with a low wave-length light filter is essential. About 30 determinations per day can be completed by a single analyst.—ALFRED EDEN and HENRY HAMILTON GREEN. *Biochem. J.*, 34 (1940), 1202. (F. J. S.)

Cresols—Critical Study of Methods for the Analysis of Industrial. Commercial cresol preparations often contain a number of constituents and none of the 17 methods studied applies to the complete analysis of some samples. This is shown by results obtained in the analysis of synthetic mixtures as well as of commercial products. For determining *m*-cresol, Raschig's method (*Z. angew. Chem.*, (1900), 760) is very satisfactory. For determining *p*-cresol, *o*-cresol and phenol the method of Dawson and Mountfort is to be recommended. Potter and Williams' method (*J. Soc. Chem. Ind.*, 51 (1932), 59–60T) gives good results with synthetic mixtures but is not altogether satisfactory for commercial products.—MELLE. SANSIN. *Ann. chim. anal.*, 21 (1939), 89–93; through *Chimie & Industrie*, 42 (1939), 961. (A. P.-C.)

Datura Alba Nees—Main Constituents of. *Datura alba* Nees is common in the Philippines and has many medicinal uses. Therefore this detailed study was made on the alkaloids in seeds, pericarps, leaves and stems. A modified method of extracting and purifying the alkaloids was introduced avoiding heat as much as possible. The seeds contain mainly hyoscine with a trace of hyoscyamine. Only hyoscine was found in pericarps, leaves and trunk. Fixed oil was 11.96% and had a dull yellow color, repulsive smell and disagreeable taste. Its physical constants were: specific gravity 0.9255, refractive index 1.4730, optic rotation none, coefficient of viscosity 0.09949 poise, relative viscosity 11.90, kinematic viscosity 0.1075, surface tension 30.024 dynes/cm., congealing point −14.3° C. Chemical constants were: acid value 46.31, saponification value 189, iodine number 84.65, acetyl number 42.28. The plant may be a good source of hyoscine if a simple inexpensive means of isolation can be devised.—CHEOA-SAKUL PRADISTH and ALFREDO C. SANTOS. *Rev. Filipino Med. Farm.*, 31 (1940), 1. (G. S. G.)

Drug Ash—Composition of. VI. The detection of tin in the presence of molybdenum can be carried but with a 0.2% solution of 4-methyl-1,2-dimercaptobenzene or 4-chloro-1,2-dimercaptobenzene in sodium hydroxide solution. A red color is obtained when tin is present. The test is not applicable if only traces of tin are present. Thioglycolic acid and its methyl derivative give a green color with molybdenum salts which may be concentrated by shaking into benzene. Boric acid may be detected by conversion to magnesium boride which when treated with dilute acids gives boron hydride which imparts a green color to the flame. This test can be made sensitive to 0.5 gamma of boric acid. The Beilstein halogen test may be reversed and used to test for copper, being sensitive to 1 part in 200,000. Manganese interferes in the Feigl test for silicic acid. This can be avoided by the use of hydrogen peroxide to first oxidize the manganese. Tests for uranium and for nickel in the presence of cobalt are also given. The ash of 12 drugs was analyzed and the elemental analysis tabulated.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 89–93. (M. F. W. D.)

Elixir of Cinchona Alkaloids—Assay of. Each 100 cc. should contain not less than 0.2940 Gm. and not more than 0.3593 Gm. of anhydrous quinine, cinchonidine, and cinchonine with a tolerance of ±10%. Using 100 cc. of the elixir, the assay process is the same as the one proposed for elixir of iron, quinine and strychine phosphates (*Bull. Natl. Formulary Comm.*, 9 (1941), 98) except for the calculations which are as follows: For 100 cc. of product, 0.2 Gm. × 0.8286 = 0.16572 Gm. anhydrous quinine; 0.1 Gm. × 0.7946 Gm. = 0.07946 Gm. anhydrous cinchonidine; 0.1 Gm. × 0.8144 Gm. = 0.08144 Gm. anhydrous cinchonine. Total anhydrous quinine, cinchonidine and cinchonine = 0.32662 Gm.—WALTER D. DEMBECK. *Bull. Natl. Formulary Committee*, 9 (1941), 98–99. (H. M. B.)

Elixir of Iron, Quinine and Strychnine—Assay of. Each 100 cc. of the elixir contains not less than 0.6008 Gm. and not more than 0.7343 Gm. total anhydrous quinine and strychnine with a tolerance of ± 10%. Using 100 cc. of the elixir the assay procedure is the same as for the elixir of iron, quinine and strychnine phosphates (*Bull. Natl. Formulary Committee*, 9 (1941), 98) except for the calculation which follows: 0.8 Gm. × 0.8173 = 0.65384 Gm. quinine and 0.0175 Gm. × 0.7803 = 0.01365 Gm. strychnine equivalent to 0.6675 Gm. total anhydrous quinine and strychnine.—WALTER D. DEMBECK. *Bull. Natl. Formulary Committee*, 9 (1941), 107. (H. M. B.)

Ferric Sulfate Reagent—New Monograph for.—JOSEPH ROSIN. *Bull. Natl. Formulary Committee*, 9 (1940), 57–58. (H. M. B.)

Ferrous and Ferric Iron—Colorimetric Determination of, in Presence of Aluminum, Manganese, Zinc, Mercury, Copper, Phosphoric Acid or Organic Substances, Especially in Drug Preparations. The method is based on the use of α,α′-dipyridyl which forms with ferrous iron a red complex cation which is very stable and resistant to dilute acids and alkalies and alkali sulfides. To determine ferric iron, it must first be reduced to the ferrous state. Certain other metals (zinc, mercury, copper) also form complexes with dipyridyl, but these complexes are

either colorless or less stable, so that their formation can easily be prevented.—E. SCHULEK and I. FLODERER. *Z. anal. Chem.*, 117 (1939), 176–195; through *Chimie & Industrie*, 43 (1940), 318. (A. P.-C.)

Flammable Liquids, Gases and Solids—Properties of. The following are given for some 365 substances: name, synonym, flash point, explosive limits, self-ignition temperature, specific gravity, vapor density, melting point, boiling point and suitable extinguishing agents.—ANON. *Ind. Eng. Chem.*, 32 (1940), 880–884. (E. G. V.)

Forensic Chemical Identifications by Micromelting Point. A new apparatus for micromelting point determinations is described, with improvements of the method of heating the warm slide and for corrections of the observed temperature. Micromethods of preparation of nitrobenzylchloride derivatives of various barbiturates are described whereby one may start with 0.5 mg. of the barbiturate and by using the above-mentioned micromelting point apparatus to determine the m. p. of the derivative, thus identifying the barbiturate. (*Cf.* also F. Reimers, *Arch. Pharm. Chemi*, 47 (1940), 489, for discussion of Halström's technique.)—F. HALSTRÖM. *Om Stofidentifikation i den forensiske Kemi*, Dissert. Copenhagen, (1940); through *Arch. Pharm. Chemi*, 47 (1940), 632. (C. S. L.)

Fruit Juices—Determination of Ascorbic Acid in. Known methods of determining ascorbic acid are compared with the polarographic method. The iodometric method, applied to untreated juices, gives high values; the iodate is preferred to the iodine method. The specificity of the dichlorophenolindophenol and methylene blue methods is discussed. Iodometric methods are inferior to the polarographic method.—D. COZZI. *Ann. chim. applicata*, 29 (1939), 434–442; through *J. Soc. Chem. Ind.*, 59 (1940), 167. (E. G. V.)

Glucosamine and Chondrosamine—Isolation of Small Quantities of. A method is described for the isolation of small quantities (10–30 mg.) of glucosamine and chondrosamine. The method depends on the sparing solubility in water of the Schiff's bases, 2-hydroxynaphthylidene-glucosamine or -chondrosamine. The presence of sugars and amino acids does not appreciably decrease the yield of Schiff's base.—Z. E. JOLLES and W. T. MORGAN. *Biochem. J.*, 34 (1940), 1183. (F. J. S.)

Hydrochloric Acid in Diluted Hydrocyanic Acid—Correction in the Test for. Evidence is presented to show that the National Formulary statement "from the number of cc. of 0.1*N* silver nitrate consumed in this titration, subtract the number of cc. of 0.1*N* silver nitrate consumed in the assay of the same quantity of diluted hydrocyanic acid as is present in this aliquot" should be changed "from the number of cc. of 0.1*N* silver nitrate consumed in this titration, substract twice the number of cc. of 0.1*N* silver nitrate. . . . "—E. DOWZARD. *Bull. Natl. Formulary Committee*, 9 (1940), 42–43. (H. M. B.)

Iodine—Estimation of, in Fucus Vesiculosus. For the accurate determination of iodine in bladder rack the procedure of Leipert (*Biochem. Z.*, 262 (1933), 99–118) is especially suited, for which a simple apparatus is described. For the current control of the drug in the pharmaceutical laboratory a rapid incineration method with colorimetric iodine estimation is recommended.—R. SEIFERT. *Süddeut. Apoth.-Ztg.*, 79 (1939), 555–558; through *Chimie & Industrie*, 43 (1940), 231. (A. P.-C.)

Iodine—New Standard (Sodium Acetomercurithymol Sulfonate) for the Determination of. Sodium acetomercurithymol sulfonate, which is readily obtained in a very pure state, reacts with iodine which displaced the acetomercuric group. The end-point is observed with the help of starch. The error is ± 0.1%. This reagent possesses the advantage over arsenic trioxide of having 10 times the molecular weight of the latter, thereby reducing the errors of analysis.—C. V. BORDEIANY, I. N. PETRESCU and L. STAICOVICI. *Bul. Soc. Stiinte Farm. Romania*, 4 (1939), 473–485; through *Chimie & Industrie*, 43 (1940), 458. (A. P.-C.)

Lactic Acid—Composition of. Production of a Highly Concentrated Acid. Water-white, viscous lactic acid of 105% purity (due to the presence of lactyllactic acid and a small amount of lactide) can be obtained from solutions of lactic acid by distillation in the presence of liquids which with water form binary mixtures boiling at temperatures below that of the acid.—P. D. WATSON. *Ind. Eng. Chem.*, 32 (1940), 399–401. (E. G. V.)

Lead and Thallium—Estimation of Traces of, in Pharmaceutical Chemicals. A dithizone limit test for lead and thallium in some 50 pharmaceutical chemicals is described. The test does not require special apparatus and is specific, rapid (a single test can usually be carried out in 15 minutes), sensitive and sufficiently accurate to take the place of the official lead tests and to be useful as a supplement to the present unsatisfactory official heavy metals test. It is possible with the dithizone test to detect and estimate less than one part of lead and thallium in one million parts of any official chemical; a table is given which includes analytical data for at least one representative of each type of official chemical. Small quantities of lead in iron or bismuth salts, gelatin or cerium oxalate cannot be detected by the present official lead tests or the official heavy metals test; the proposed dithizone limit test is as sensitive in these cases as in others.—K. BAMBACH. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 63–66. (E. G. V.)

Lead—Determination of Minute Amounts of, in Urine. A sample (100 cc.) of urine was evaporated with 100 cc. 2*N* H_2SO_4 in a 350-cc. Kjeldahl flask and digested with the addition of four drops of perhydrol until clear. The digest was diluted with water, 5 cc. 20% sodium citrate were added and the solution was made alkaline to litmus with ammonia (sp. gr. 0.880). After the addition of 5 cc. 10% KCN, lead was removed by shaking twice with ether and approximately 20 mg. sodium diethyldithiocarbamate. The ethereal extract was washed with water, the solvent evaporated and the residue digested with 1 cc. H_2SO_4. Three to four drops of perhydrol were added and heating was continued for 5 minutes after clearing. The acid was distilled with 4 cc. water and 0.2 cc. CH_3COOH; 3 cc. ammonia (sp. gr. 0.880) were added to make the reaction alkaline. This solution was washed into a 50-cc. separating funnel (with very short stem) with 5 cc. 1% KCN and 5 cc. CCl_4 were added. After vigorous shaking with a slight excess of dithizone solution (2–3 drops) the aqueous phase was drawn off with a Pasteur pipette and the remaining dithizone washed out with 1% KCN. The colored CCl_4 layer was run off and compared with a standard (lead acetate) in a Klett microcolorimeter, using photometric technique. The method is economical in both time and reagents; and 50–100-cc. samples of urine are adequate.—JAMES EDWARD KENCH. *Biochem. J.*, 34 (1940), 1245. (F. J. S.)

Lead Limit Test by the Dithizone Method. The following procedure is proposed: Add 3 cc. potassium cyanide solution to the sample prepared as specified in the monograph. Extract with two 10-cc. portions of the stronger dithizone solutions followed with one 10-cc. portion of chloroform. Combine the chloroformic extracts in another separator and wash with 10 cc. distilled water to which a few

drops of potassium cyanide solution have been added. Withdraw this chloroformic layer into a separator and wash the aqueous layer with 10 cc. chloroform. Combine this chloroform with the first chloroformic extracts and extract with two 25-cc. portions of nitric acid solution. Discard the chloroform from the second extraction and transfer the aqueous layer to that of the first extraction, rinsing the separator with 4 cc. distilled water and add it to the combined aqueous layers. Wash the aqueous solution with 10 cc. chloroform. Discard the chloroform layer and add 10 cc. of ammonia-cyanide solution to the aqueous solution. Extract with successive 20-cc. portions of standard dithizone solution, shaking each portion with the aqueous layer for two minutes. Allow the aqueous layer to become clear and shake down the chloroform suspended by adhesion on top of the aqueous layer. Collect each extraction in test-tubes of 25-cc. capacity and of the same diameter and shape. The end-point to the number of extractions is that point at which the color of the chloroform layer, when viewed through the top of the tube and above a white background, has changed to purple. Record the number of extractions.—EMERSON C. BEELER. *Bull. Natl. Formulary Committee*, 9 (1941), 124–130. (H. M. B.)

Lead—Rapid Titrimetric Determination of. The lead is precipitated as lead chromate, filtered off, the excess of chromate is reduced to trivalent chromium with a measured volume of standard ferrous solution and the excess ferrous iron is titrated with potassium permanganate. For the determination of lead in an alloy, treat 5 Gm. of borings with 20 cc. of water, 15 drops of 13.5*N* hydrofluoric acid and 75 cc. of 6*N* nitric acid. Transfer the solution to a 500-cc. volumetric flask, make up to the mark and mix. Take 100 cc. of the solution, add 50 cc. of water heat to boiling and to the boiling solution add very slowly, while stirring, 50 cc. of a standardized solution containing 18 Gm. of ammonium chromate per liter. About 10 minutes should be required for adding the reagent. After standing for a while, filter off the lead chromate precipitate and wash it with hot water. To the filtrate add 50 cc. of standardized approximately 0.2*N* acid ferrous sulfate solution and titrate the excess with 0.2*N* permanganate.—A. GRUPP. *Z. anal. Chem.*, 119 (1940), 333–335. (S. W. G.)

Manganese—Detection of, in Organic Tissues. The method is based on the following reaction: addition of potassium permanganate to a boiling, slightly acid solution of manganous sulfate produces a brown precipitate of manganous acid and of manganous manganites. Presence of metallic ions in the solutions, more particularly of zinc, calcium or barium, facilitates the formation of these complexes. A permanent pinkish coloration in the liquid indicates the end-point of the reaction. Liver from calf fetuses contained about 3 mg. of manganese per 100 Gm. at 4 to 5 months and 7 to 8 mg. at 6 to 9 months.—G. GRUZEWSKA and G. ROUSSEL. *Bull. soc. chim. biol.*, 21 (1939), 730–736; through *Chimie & Industrie*, 42 (1939), 963. (A. P.-C.)

Melting Point—Microdetermination of the, as a Pharmacopœial Procedure. The recommended method depends upon the light refraction of the melted substance and a comparison of its refraction with glass powders of different refractive indices. The value of euctectic temperatures is also emphasized. Nine references.—L. KOFLER. *Scientia Pharm.*, 11 (1940), 41–42. (H. M. B.)

Mercury—Modification of Rauscher's Method for Determining. The sample is weighed directly into a tared centrifuge tube of 15-cc. capacity, and the digestions of all subsequent manipulations are carried out therein instead of in a Pyrex test-tube as provided for by Rauscher. At the end of the digestion period, the tube and its contents are centrifuged for 5 minutes at about 2800 r. p. m. In this way all the floating particles become combined with the main globule in the bottom of the tube. The supernatant liquid is then sucked off, water is added to wash the globule of mercury and the tube and contents are centrifuged again. The washing operation may be repeated as many times as desired. Solution of the mercury and its determination by titration are carried out in general as indicated in the original method. In order to complete the reaction in the same centrifuge tube, however, 0.1 to 0.2*N* thiocyanate solution is used to avoid the large volume necessitated by the weaker solutions used by Rauscher. Accuracy in titration is achieved by the use of microburette graduated in 0.02 cc.—A. SHUKIS, JR., and R. C. TALLMAN. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 123. (E. G. V.)

Mercury-Sealed Vessels for the Storage of Solutions. Various combinations of mercury-sealed vessels are described for use in protecting the contents and preventing contamination.—F. E. HOLMES. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 425–427. (E. G. V.)

Micro-Kjeldahl Apparatus. The apparatus which is described (figure) is suitable for the estimation of amounts of nitrogen as small as 50μg.—HANS HOCH. *Biochem. J.*, 34 (1940), 1209. (F. J. S.)

Molybdate—New Adsorption Indicators for Titrimetric Determination of, with Lead Nitrate. The lead molybdate formed when a soluble lead salt is added to a solution of molybdate is colloidal at first and flocculates completely only when the equivalent point is reached. In the colloidal state the precipitate adsorbs molybdate as long as this ion is in excess, but as soon as the end-point is reached it adsorbs excess lead ion which changes the electric charge on the precipitate, making it positively charged and as a result negatively charged particles of a dyestuff are adsorbed. Alizarin red, phenol red and eosin A have already been recommended for the molybdate titration. Twenty-two other dyes of the phthalein, azo- and anthraquinones classes were tested and five of them proved suitable to act as inticators: erythrosin AGZ, benzoechtscharlach 4BA, siriusrosa, echtrot A and siriusviolett 3B. With the last dye, the precipitate becomes colored only after the end-point is reached; while in the other cases the precipitate changes color. The best conditions for using the indicators are described.—H. HENKEL. *Z. anal. Chem.*, 119 (1940), 326–333. (S. W. G.)

Nitrite Nitrogen Standards. Sodium nitrite of a sufficiently high degree of purity to serve as a primary standard in the nitrite nitrogen determination is readily obtainable. Its use obviates the delay attendant upon the use of silver nitrite and permits the preparation of all three dilutions of the reagent in a short period. As sodium nitrite is relatively inexpensive and the solution is readily prepared, it may be made fresh when needed, thus eliminating any question of deterioration arising from the use of an old solution.—W. F. REINDOLLAR. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 337–338. (E. G. V.)

***m*-Nitrobenzazide as a Reagent for Phenols.** On boiling *m*-nitrobenzazide decomposes into nitrogen gas and *m*-nitrophenyl isocyanate, which reacts with phenols with formation of *m*-nitrophenyl urethanes. The latter can be identified by microscopic examination of the crystals and determination of their melting point.—P. P. T. SAH and T. WO. *Rec. Trav. Chim. Pays-Bas*, 58 (1939), 1013–1017; through *Chimie & Industrie*, 43 (1940), 469. (A. P.-C.)

o-Nitrophenol in p-Nitrophenol and o-Aminophenol in p-Aminophenol—Estimation of, by Fluorescence Analysis. A method is reported for the estimation of 1% or less of *o*-nitrophenol in *p*-nitrophenol, or of *o*-aminophenol in *p*-aminophenol. The nitrophenol is reduced in aminophenol and the latter is made to react with benzoic acid. A fluorescence is produced as a by-product of the formation of phenylbenzoxazole from *o*-aminophenol, which is not produced by *m*- or *p*-aminophenol. By matching the fluorescence against standards, as little as 0.05% of the ortho compounds can be detected.—W. SEAMAN, A. R. NORTON and O. E. SUNDBERG. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 403–405. (E. G. V.)

Organic Chemical Drugs—Microchemical Identification Methods for. This is a review article with 140 references. Methods considered include color reactions, crystal precipitation methods, use of micromelting point apparatus, microrefraction determination, determinations of crystal optics, polarization, crystal angles, optical axis, etc., microsublimation, organic spot analyses and microchemical fluorescence methods. Comparison is made of the older technique of micromelting-point determination described by Kofler (*Mikroscopische Methoden in der Microchemie*, Vienna-Leipzig, 1936) and a new modification described by Halström (*Om Stofidentifikation i den forensiske Kemi*, Dissert., Copenhagen, 1940), with mention of apparatus described by Weygand and Grüntzig (*Microch.*, 9 (1931), 38), Dunbar (*Ind. Eng. Chem., Anal. Ed.*, 11 (1939), 516) and Fuchs (*Microch. Akta*, 2 (1937), 317). Fischer's microsublimation apparatus (*Microch.*, 15 (1934), 247) is described. Choice of methods of separation and identification for drugs is considered, also identification *via* micropreparation of derivatives.—F. REIMERS. *Arch. Pharm. Chemi*, 47 (1940), 489. (C. S. L.)

Pectin—New Monograph for. After considerable experimental work which is reported, the following procedure for determining the limit of lead in pectin is offered: Add 4 Gm. pectin to 20 cc. nitric acid in a 250-cc. Erlenmeyer flask and heat the contents gently while shaking with a swirling motion until solution is affected. Add 20 cc. nitric acid solution and 4 Gm. citric acid. Cool in an ice bath and cautiously add 40 cc. stronger ammonia water. Transfer to a separator, rinsing the flask with 5 cc. of distilled water. Adjust to room temperature and apply the N. F. test for lead. Prepare a control standard using 20 cc. of standard lead solution. The number of extractions should not exceed those required for the control standard.—EMERSON C. BEELER. *Bull. Natl. Formulary Committee*, 9 (1941), 119–124. (H. M. B.)

Pentamethylenetetrazol Preparations (Cardiazol, Tetracor)—Determination of. Pentamethylenetetrazol is supplied in a 10% solution (ampuls and drops) and in tablets. The liquid preparations are easily determined by carefully removing the solvent and weighing the residue. The tablets are first extracted with alcohol and the solution so obtained, evaporated. The residue may be identified as the difficultly soluble double salt, melting at 176°, formed with mercuric chloride. The solutions of pentamethylene tetrazol are concentrated to a small volume on a water bath (to about 0.6 cc.) and then dried to constant weight in a vacuum desiccator over sulfuric acid. The mercury in the purified double salt is determined by reduction with formaldehyde in alkali, the liberated mercury dissolved in an excess of standard iodine solution and the excess iodine determined with standard thiosulfate solution. The tablets are first powdered in a mortar and then extracted with 50 cc. absolute alcohol. The alcohol is then carefully evaporated and the residue treated as described for the liquid preparations. Any similar compound may be tested by the above procedure.—GYULA V. MIKO. *Pharm. Acta Helv.*, 14 (1939), 31–34. (M. F. W. D.)

Perforation Apparatus—Simple, for Use with Both Light and Heavy Solvents. A process termed perforation may be used for the extraction of difficulty soluble materials with immiscible solvents or for the extraction of materials from solutions which easily emulsify. Many types of apparatus devised for this purpose are fragile, expensive or require numerous parts. A simple apparatus has been devised which may be used for the extraction with both heavy solvents such as chloroform and light solvents such as ether. The apparatus is simple of construction, compact and easy to use. A diagram of the essential parts of the apparatus and the details of its use are given.—J. PRITZKER and R. JUNGKUNZ. *Pharm. Acta Helv.*, 14 (1939), 223–225. (M. F. W. D.)

Phenols and Cresols—Determination of, in Air. Phenol and cresols are determined colorimetrically as azo dyes, obtained by coupling the compound to be determined with a diazo solution prepared with *p*-nitroaniline. The sensitiveness of the method is 5 Gm. for phenol, *m*-cresol and *o*-cresol and 15 Gm. for *p*-cresol.—V. P. MAIEVSKAIA, *Zavodskaya Lab.*, 8 (1939), 812–815; through *Chimie & Industrie*, 43 (1940), 299. (A. P.-C.)

Phytochemical Notes. Suggestion for the Improvement of the Dragendorff Methods. Experiments indicate that the successive use of 1% sodium carbonate and 1% sodium hydroxide in the examination of the petroleum ether extracts obtained from plant materials by the Dragendorff method of extraction with selective solvents is an improvement.—PAUL J. JANNKE. *Pharm. Arch.*, 12 (1941), 6–8. (H. M. B.)

Piperazine—Determination of. Piperazine is sometimes present in effervescent powders containing citric or tartaric acid, sodium bicarbonate and sugar. To determine piperazine dissolve the sample in a little water, make acid to Congo red with dilute hydrochloric acid, add a large excess of 10% aqueous solution of silicotungstic acid, let stand 7 to 8 hours, filter, wash the precipitate (which has the composition $12WO_3.SiO_2.2C_4H_{10}N_2.4H_2O$) with dilute hydrochloric acid, dry at 105° C. and ignite to constant weight; weight of ignited precipitate × 0.6046 = weight of piperazine. On samples containing 0.05 to 0.23 Gm. of piperazine the results were all within 1% of theoretical.—A. CASTIGLIONI. *Z. anal. Chem.*, 117 (1939), 25–26; through *Chimie & Industrie*, 43 (1940), 411. (A. P.-C.)

Purshia Tridentata (Pursh) DC.—Phytochemical and Histological Study of. Investigation of the plant was undertaken to isolate and identify constituents responsible for purported curative action obtained by Indians who have used it in decoctions, infusions and poultices. Constituents found were a catechol or catecholphloroglucin tannin, a bitter substance, starch, pectin, mucilage, sterols, an aromatic ester, a wax, an ester-resin, small quantities of resin-acids and galactose. Purported curative action obtained by Indians was probably due to bacteriostatic action of the tannins and the demulcent effect of the mucilage and pectin.—CHARLES V. NETZ, CHARLES H. ROGERS and GLENN L. JENKINS. *Jour. A. Ph. A.*, 29 (1940), 490. (Z. M. C.)