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PHARMACEUTICAL ABSTRACTS

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CONTENTS

Bacteriology (*Continued*) 2
Chemistry:
General and Physical 6
Inorganic 7
Organic:
Alkaloids 8
Essential Oils and Related Products 9
Glycosides, Ferments and Carbohydrates 9
Other Plant Principles 10
Fixed Oils, Fats and Waxes 12
Unclassified 13
Biochemistry 15
Analytical 23
Pharmacognosy:
Vegetable Drugs 30
Pharmacy:
Galenical 32

BACTERIOLOGY (*Continued*)

Erythroblastosis Virus. The purification of chicken erythroblastosis virus (precipitation with ascorbic acid, absorption with aluminum hydroxide) are reported. Some atypical forms following injections with leukemic plasma from which the proteins have been absorbed, are described. A cause of neoformation in the inoculation point following an injection of active material in an animal immune is mentioned. Tests of infection of erythroblastic virus from a chicken to a pheasant are reported.—E. MORELLI and A. VERCELLONE. *Biochim. terap. sper.*, 26 (1939), 241. (A. C. DeD.)

Food Microörganisms—Stasis and Death of, in Syrups. Types of preservative used in fruit syrups are discussed. A mixture of α- and β-(β-trichloroisopropyl) esters of glycerol (preparation from chlorotone described) is recommended as a basis for preservatives.—G. ISSOGLIO. *Atti X Congr. Internaz. Chim.*, IV (1938), 524–531; through *J. Soc. Chem. Ind.*, 59 (1940), 638. (E. G. V.)

Fungi in Cutaneous Eruptions—Incidence of. The authors found that of 476 patients with cutaneous eruptions 37% were due to fungi. Culture and microscopic examination were employed. The most commonly found genera were *Microsporon, monilia* and *Trichophyton*. Most of the conditions due to fungi were found during warm weather. The head showed the greatest number of lesions with fungi (29%) with the upper and lower extremities next (26% each) and the body the least (19%). It is believed that diagnosis and treatment of cutaneous infections is greatly facilitated by a study of mycology. —J. G. DOWNING, B. MERRILL and D. L. BELDING. *New Eng. J. Med.*, 222 (1940), 263; through *Am. J. Pharm.*, 113 (1941), 80. (A. C. DeD.)

Germicides—Consideration for Evaluation of. It has been shown that with the four main types of antiseptics, mercurials, phenols, halogens and oxidizers, a decrease in pH always results in an increase in germicidal effect, an increase which is independent of the germicidal action of the agent used to lower the pH. So, merely stating the concentration of a given chemical does not indicate its performance. A 5 per cent phenol does not effect spore bearing organisms but with hydrochloric acid added to lower its pH it becomes a better sporicide than 20 per cent formaldehyde solution. Tincture of Iodine, 7 per cent, will not kill spores of *B. subtilis* in two hours, but an acidified 5 per cent iodine solution kills in 10 minutes. Other examples are cited. Besides germicidal action, "affinity" to organic matter is important. It has been shown that common germicides except iodine do not kill germs at body temperature in the presence of 50 per cent horse serum. This fact is significant when antiseptics are to be used in deep wounds or body cavities where there is always organic matter. An investigation of the superiority of iodine in the presence of organic matter was made by studying the chemical interaction between iodine serum and acidifying agents. Acidified solutions of iodine, titrated after reacting with definite quantities of serum, retained more available iodine than ordinary iodine solutions of the same content. The same thing seems to be true with other halogens. Various acidifying agents had the same effect.—PAUL GOEDRICH. *Jour. A. Ph. A.*, 30 (1941), 88. (Z. M. C.)

Gonococcal Infection—Improved Culture Medium for the Diagnosis of. The composition of the medium is as follows: (*A*) Difco proteose peptone No. 3, 40 Gm.; dextrose 1 Gm.; sodium chloride 10 Gm.; disodium phosphate 10 Gm.; agar 30 Gm.; distilled water 1000 cc.; sterilize at 15 lbs. for 20 minutes; final pH 7.4. (*B*) Bacto hemoglobin 2 Gm.; distilled water 100 cc. Sterilize as above. Mix equal amounts of *A* and *B* (cooled to 60° C.) and dispense in Petri dishes. The exudate for culture was mixed with 1 cc. of beef infusion broth and kept at 4° C. for not more than 6 hours and approximately 0.1 cc. of the broth is then spread on the culture medium. Plates are incubated in closed jars from which 12 per cent of the air has been removed and replaced by 10 per cent carbon dioxide. Colonies are picked by the oxydase method and the gonococci identified in the first instance by morphology plus their inability to grow on plain agar. In 2050 examinations using smears and cultures simultaneously, 284 were positive by one or both methods, by the cultural in 241 and by the smear in 127. The authors state that by the use of this medium (which may be obtained in dehydrated form) twice as many positive results are obtained as with stained smears alone.—S. E. SULKIN and E. GOTTLIEB. *Am. J. Syphilis*, 25 (1941), 22; through *Bull. Hyg.*, 16 (1941), 268. (T. C. G.)

Hydrogen Ion Concentration—Significance of, in the Evaluation of the Bactericidal Efficiency of Surface Tension Depressants. The hydrogen ion concentration exerts a most profound influence on the efficiency of surface tension depressants as bactericidal or bacteriostatic agents. The authors in this preliminary report give figures illustrating the extent of this effect with one of the very common surface active substances.—L. GERSHENFELD and D. PERSTEIN. *Am. J. Pharm.*, 113 (1941), 89. (A. C. DeD.)

Hydrogen Peroxide—Action of, on Bacterial Suspensions in Initial Autolysis. In the case of protein synthesis in suspensions of *Eberthella typhi* and *Escherichia coli* it was shown that the addition of hydrogen peroxide water increases the fraction formed at 0° and it seems that enzymatic rather than physical or chemical factors interfere in its production. The importance of hydrogen ion concentration, of temperature and of various precipitating agents was established.—M. CALCINAI. *Biochim. terap. sper.*, 26 (1939), 262. (A. C. DeD.)

Influenza Virus—Inactivation of, by Soaps. It has long been recognized that soaps are destructive to certain bacteria and their toxins. In the present work an attempt was made to determine whether or not certain soaps or fatty acids had a similar effect upon the virus of epidemic influenza. The PR 8 strain of the virus was mixed with various soaps and after staining for 90 minutes or 24 hours, the mixtures were instilled into the nostrils of mice. These animals were observed for 10 days and autopsies made to determine the cause of death. Of all the soaps or fatty acids tested only oleic, linoleic, linolenic, ricinoleic, chaulmoogric, lauric, erucic and lauryl sulfuric were effective in destroying the virus. An analysis of the structure, surface tension depression, length of carbon chain, etc., failed to reveal any factor which appeared to govern the viricidal properties of the substances studied. Virus which had been inactivated by oleic acid was found to be capable of immunizing mice to subsequent lethal doses of the virus. Apparently virus inactived by soaps is a better antigen than virus inactivated by formalin or heat, hence the promise of an effective vaccine for human immunizations.—C. C. STOCK and T. FRANCIS, JR. *J. Exp. Med.*, 71 (1940), 661. (T. C. G.)

Kline Test. The article compares the value of reactions involved in the several tests for syphilis, Kahn, Kline, Meinicke and Hinton. Materials such as pipettes and glass plates for use in the Kline precipitation test are described, and also the preparation of the 0.85% sodium chloride in double distilled water at pH 6, and of the specific cholesterol anti-

gen. Plates demonstrating negative, doubtful, positive and strong positive reactions are printed also. The advantages of the Kline test are its simple technique and rapid execution. It is particularly valuable as an exclusion test.—OSVALDO A. FONIO. *Trib. Farm., Tucuman*, 5 (1940), 81. (G. S. G.)

Lactic Ferments—Storage of, for Therapeutic Purposes. *Bacillus acidophilus* has been conserved for 5 years in a medium of liver highly peptonized with lactic serum together with soluble starch.—C. A. SAGASTUME and V. RIVERA. *Rev. facultad cienc. quím. Univ. nac. La Plata*, 14 (1939), 111–114; through *J. Soc. Chem. Ind.*, 59 (1940), 492. (E. G. V.)

Mercury Compounds—Antiseptic Organic. *2,206,804*—Antiseptics highly effective against microörganisms and relatively non-irritating to living tissues comprise furan mercurial compounds of the general formulas O.C*R*:CH.CH:CHg*X*, O.C*R*:CH.C(Hg*X*):C*R*′ and O.C*R*:CH.CH:CHg-C:CH.CH.C*R*′O (where *R* and *R*′ represent hydrogen, chlorine, bromine, iodine, nitro or alkyl groups containing not more than 6 carbon atoms, and *X* is a hydroxyl or a negative organic or inorganic acid radical). 2-Furylmercuric hydroxide melts with decomposition at 101° C. and may be caused to react with various acids to form salts. *2,206,805*—Furylmercuric chloride is used as an antiseptic for contact with human tissue.—ROBERT R. BURTNER, assignor to G. D. SEARLE & Co. U. S. pats. 2,206,804 and 2,206,805, July 2, 1940. (A. P.-C.)

Nicotinic Acid—Function of, in the Metabolism of the Colon-Typhoid Group. The authors have previously shown that nicotinic acid does not act as a "growth factor" for *Bact. paratyphosum* A and *Bact. dysenteriae* Shiga, but activates the fermentation of glucose, since growth but not fermentation occurs in the absence of nicotinic acid. The findings are now extended to other substrates. Glucose and all the other fermentable sugars, with the exception of maltose, actually inhibit growth to some extent in the absence of nicotinic acid. Sucrose, which is not fermented, causes no such inhibition. Nicotinic acid in the absence of a fermentable sugar or acid (lactic or acetic) also diminishes growth. Fermentation occurs when a fermentable substrate and nicotinic acid are both present, glucose yielding lactic and acetic acids and carbon dioxide. Lactic acid in the absence of nicotinic acid is oxidized to give a small amount of acetic acid only, but in the presence of nicotinic acid the oxidation is completed and carbon dioxide only formed. The intensity of fermentation parallels the density of growth. Using lactate as a hydrogen donator in the Thunberg tube, washed suspensions of bacteria grown in the presence of nicotinic acid (0.005 gamma per cc.) reduce methylene blue promptly. Organisms grown in the absence of nicotinic acid reduce methylene blue in a substrate containing nicotinic acid after a delay, suggesting that nicotinic acid is converted into a codehydrase before it becomes active.—I. J. KLIGLER and N. GROSSOWICZ. *Nature*, 146 (1940), 652; through *Bull. Hyg.*, 16 (1941), 196. (T. C. G.)

Nitrogenous Organic Matter in Sea Water—Decomposition and Regeneration of. The source of the water is important in determining the nature of the decomposition cycle. Oxidation of ammonia to nitrite is retarded in water from the deep sea (1200 m.). Inconclusive results were obtained from efforts to sterilize both the water and the original organic matter, but it is evidently easier to eliminate organisms responsible for the oxidation processes than those which may participate in the formation of ammonia. The speed of the whole decomposition cycle was more than doubled by an increase of 6° or 8° in temperature. Growth of diatoms is possible at any stage in the cycle of decomposition.—T. VON BRAND and N. W. RAKESTRAW. *Biol. Bull.*, 79 (1940), 231. (A. C. DeD.)

Paper—Significance of Bacterial Control in. Pathogenic types of organisms are not present in paper or paperboard owing primarily to the high drying temperature during manufacture, and no single case of a communicable disease has been traced in the U. S. A. to contamination of milk and other foods by paper packings.—C. M. BAKER. *Paper Trade J.*, 110 (1940), TAPPI Sect., 217–219; through *J. Soc. Chem. Ind.*, 59 (1940), 596. (E. G. V.)

Paraffin Wax—Germicidal Properties of. Tests show that the hot wax has a decided germicidal action, and that bacterial cells cannot penetrate (cold) films of the wax. Paraffination of paperboard milk containers contaminated with *Esch. coli* and an aerobic sporeforming bacterium has definite germicidal effect and real sanitary value.—F. W. TANNER and H. F. LEWIS, *Oil and Soap*, 17 (1940), 26–30; through *J. Soc. Chem. Ind.*, 59 (1940), 500. (E. G. V.)

Pertussis Antigens—Immunization by the Intranasal Route with. Mice immunized with various antigens, administered intranasally under anesthesia, were tested by a 100 per cent killing dose of living phase I *H. pertussis*, given in the same manner. The survival rate after immunization with commercial soluble antigens derived from *H. pertussis* varied from 17 to 42 per cent, whereas four different brands of vaccine consisting of whole bacilli each protected 80–90 per cent of the test mice. The immunity, in part at any rate, appears to be general and not local, for it begins to decrease at about the same time as the agglutination titer in the blood of the mice. There is, however, no necessary association between agglutination titer and protective power of the serum. Active immunization of human subjects by this route has been tried. No untoward reactions were observed. The serum from persons so immunized appeared in two cases to protect two children who had been intimately exposed to whooping-cough.—R. P. DOW. *Can. Pub. Health J.*, 31 (1940), 370; through *Bull. Hyg.*, 16 (1941), 199. (T. C. G.)

Phenol—Influence of, on Bacterial Growth. The influence of phenol on the death rate of cells of *Bact. lactis aerogenes* is quantitatively smaller than the influence on the division rate. It depends upon the stage of growth at which the phenol is added. Viable counts confirm that the stationary phase is not due to a simple balancing of death rate and division rate, but depends upon an actual cessation of division.—E. A. POOLE and C. N. HINSHELWOOD. *J. Chem. Soc.*, (1940), 1573–1574. (W. T. S.)

Phenols—Sterilizing Action of. The effect of phenol (I), *o*-cresol, guaiacol (II), thymol (III), picric acid (IV), pyrocatechol (V), resorcinol (VI), quinol (VII), pyrogallol (VIII) and phloroglucinol (IX) on *Staph. pyogenes*, *Proteus vulgare*, *B. typhosus* and *Vib. choleroe* is determined. At the same concentration IV has the greatest effect and next in order come II, VII, V and VIII. 0.1*N* I is effective as a sterilizer, but 0.01*N* I has no action. The actions of II and VI are very weak and in 0.001*N* solution they promote bacterial growth. IX has no bactericidal action and promotes growth. Calcium, sodium and ammonium salts of III and VII have relatively strong action, while the salts of other phenols are inactive and act as promoters. *p*-Compounds are the most, and *m*-compounds the least active, while the activity of di-, tri- and monohydric phenols decreases in this order.—S. TET-

SUMOTO. *J. Agr. Chem. Soc. Japan*, 16 (1940), 299–305; through *J. Soc. Chem. Ind.*, 59 (1940), 645. (E. G. V.)

Pneumococci Types I, II and III—Immunity of the Experimental Infection of, in Animals Treated with 2(*p*-Aminophenylsulfamide)pyridine. The author confirms the statement of Whitby that active immunity arises in mice infected with pneumococci and treated with 2(*p*-aminophenylsulfamide)-pyridine.—C. CALLERIO. *Biochim. terap. sper.*, 26 (1939), 338. (A. C. DeD.)

Poliomyelitis—Treatment of, with Potassium Chlorate. Since 1937 a number of European workers have recommended the use of potassium chlorate for the treatment of poliomyelitis. The dosage recommended has varied from 0.1 Gm. per Kg. body weight as the daily total given by mouth in divided doses every two hours, to 10/250 or even 14/250, and at the same time 5 drops of a 2 per cent solution of salt were instilled into the nostrils four times daily. The results of the author's experiences are summed up as follows: Of 289 cases, 97 were given the chlorate treatment and 6 of them ended fatally (6.18%), 7 (7.22%) remained permanently invalided, 5 (5.16%) had slight residual pareses and 79 (81.44%) cleared up completely. Of 192 who did not receive this treatment 3 (1.56%) died, 10 (5.21%) were invalids, 22 (11.46%) had slight residual pareses and 57 (81.77%) cleared up. In other words the proportion clearing up was practically the same in each group, while the fatalities were four times as great among those receiving the chlorate treatment.—O. GSELL. *Schweiz. med. Wochschr.*, 70 (1940). 803; through *Bull. Hyg.*, 16 (1941), 72. (T. C. G.)

Poliomyelitis Virus—Isolation of, from Human Stools. The authors collected fecal specimens from human cases of poliomyelitis and after suitable treatment injected them intraperitoneally into monkeys. By this method virus was detected in 10 stools from 8 individuals. The positive stools were distributed among 56 specimens collected from 53 persons during the first 4 weeks of illness. The virus is relatively stable in stools since a number of specimens were sent through the mails over long distances during the hot summer months.—J. D. TRASK, J. R. PAUL and A. J. VIGNEC. *J. Exp. Med.*, 71 (1940), 751. (T. C. G.)

Poliomyelitis Virus—Isolation of, from Sewage. These workers obtained samples of sewage from Charleston, S. C., Detroit, Mich., and Buffalo, N. Y., where epidemics of poliomyelitis were in progress at the time. After suitable treatment of the specimens they were injected into monkeys and positive isolation of the virus was indicated by the development of poliomyelitis in the experimental animals. In two of the three cities mentioned above the virus was isolated from the sewage. The virus appeared to be found in higher concentrations in sewage collected near isolation hospitals. The authors do not commit themselves to the possible implications of their findings but it would appear that the possibility is now definitely established that poliomyelitis may be transmitted by the gastro-intestinal route as well as by the respiratory route.—J. R. PAUL, J. D. TRASK and S. GARD. *J. Exp. Med.*, 71 (1940), 765. (T. C. G.)

Procaine Solutions—Preparation of Sterile. Solutions of procaine hydrochloride cannot be autoclaved without serious decomposition and wholesale demands show that it is mainly required to be in rubber-capped bottles, which do not easily lend themselves to heat sterilization, especially in large scale work, and the tendency of the solutions to darken if contaminated with iron or copper, or on account of oxidation, is well known. By including 0.025% of sulfur dioxide in solutions of procaine hydrochloride and preventing further oxidation by displacing the air in the containers with carbon dioxide, it is possible to obtain a solution which remains water-white for a period of eighteen months or more. From bacteriological investigations the following observations have been made: (1) Solutions of procaine hydrochloride have (*a*) some bactericidal action on *B. typhosus* and *Streptococcus haemolyticus* Richards, the action increasing with increase in strength, (*b*) together with 0.5% of chlorbutol, a distinct lethal action on *B. typhosus* and *S. haemolyticus* Richards after three hours' exposure; (2) 4% solutions, with 0.5% of chlorbutol, show complete destruction of the same organisms after one hour's exposure; (3) the presence of 0.025% of sulfur dioxide assists in killing the organisms, probably due to increased acidity; (4) the combined bactericidal action of procaine hydrochloride, chlorbutol and sulfur dioxide is definitely bactericidal to *B. typhosus*, *S. haemolyticus* Richards and *Staphylococcus aureus* but has no apparent lethal action on spore-bearing *B. subtilis*. The authors summarize as follows: Sterile solutions of procaine hydrochloride can be prepared without the use of bacteria-proof filters or heat sterilization, provided aseptic methods are used; solutions prepared with an antiseptic present would not be vulnerable to contaminations likely to occur in a laboratory devoted to the production of sterile preparations; solutions prepared with both an antiseptic present and an oxidation-preventing substance such as sulfur dioxide are even less vulnerable to contamination; the antiseptic properties of the solutions tend to increase with increase of percentage strength.—A. M. BRIGGS and D. E. CALLOW. *Chemist and Druggist*, 134 (1941), 252. (A. C. DeD.)

Sterilizing Process. The inventions provide a process of sterilizing normally dry colloid material having useful viscosity and gelling characteristics and water-absorption capacity (such as foods, dentifrices, bandages, etc.) without substantial loss in these properties. *2,189,947*—The material is subjected to a temperature of about 43° to 45° C. for at least one hour and is then subjected in a closed chamber to a vacuum of 27 to 28 inches whereby moisture and removable gas are taken from the heated material. Substantially undiluted ether in gas form is admitted into the chamber containing the evacuated material in amount at least 12 oz. per 35 cu. ft. of chamber volume, and the material is exposed to the gas for at least 2 hours, whereby substantially all the microörganic life in the material is killed. *2,189,948*—Pancreatin is treated by this process to reduce its bacterial and mold count. *2,189,949* relates to a similar sterilization of colloidal materials such as gums, gelatin, agar agar, etc.—CARROLL L. GRIFFITH and LLOYD A. HALL, assignors to GRIFFITH LABS., INC. U. S. pats. 2,189,947 to 2,189,949, Feb. 13, 1940. (A. P.-C.)

Sulfanilamide. This is a comprehensive discussion of the drug, from the preliminary history of its development from the study of azo dyes. Its chemical composition and antistreptococcic activity are described in detail, methods of experimental study are sketched, and its activity in certain animals and against specific organisms is tabulated. Its pharmacology is detailed as to absorption and distribution in various tissues, and any pathological effect is noted. Its mode of action is still subject to investigation, since its antibacterial activity is not of itself but due to transformations which it undergoes in the body.—A. D. MARENZI. *Anales farm. bioquím* (Buenos Aires). *Suplemento*, 11 (1940), 65. (G. S. G.)

Sulfanilaminoquinolines—Chemotherapeutic Action of. Seven isomeric sulfanilaminoquinolines (2, 3, 4, 5, 6, 7, 8, positions on the quinoline), pre-

pared by Jensen and Lundquist (*Dansk Tids. Farm.*, 14 (1940), 208), were tested as to action on pneumococci *in vitro*, in comparison with sulfanilamide and sulfapyridine. Also tested was *p*-aminobenzenesulfonacetamide, on pneumococci and gonococci. Serum bouillon was used with pneumococcus Type I. Ascites agar was used with the gonococcus. The sulfanilaminoquinolines all had a marked bacteriostatic action on pneumococcus Type I at the same concentration as was effective with sulfapyridine. The acetamide derivative was not effective against either organism at a concentration of 1:2500, while at a much lower concentration, sulfapyridine checked growth (pneumococci at 1:80,000). Here sulfanilamide was bacteriostatic at 1:16,000.—K. SCHMITH. *Dansk Tids. Farm.*, 14 (1940), 215. (C. S. L.)

Surgical Dressings—Sterility Tests on. The following procedures for testing the sterilizer are given: 1. Earth packet method. Wrap about 0.5 Gm. of earth in a piece of filter paper, to form a packet about 1 cm. long and 0.25–0.5 cm. in diameter. Insert into a dressing which is marked and place among other dressings undergoing sterilization. After sterilization, cautiously unwrap the marked dressing until the little earth packet is just uncovered and transfer to broth by means of sterile forceps. Accidental contamination is too rare to cause any trouble. 2. Glass tube method. Place some earth in a piece of glass tube 2–4 cm. long and about 0.5 cm. in diameter. Plug both ends with cotton and insert the tube into a marked dressing The tube can be recovered without sterile precautions, and the actual sterility test made later by removing the plugs and shaking the earth into broth. This method is especially suitable for large dressings, in which an earth packet may take some time to find, during which contamination may occur. The earth contains spores of high thermal endurance so that if the earth is sterilized, the surrounding dressing is so too. The nature and position of the test dressing must be similar to the others about which the information is needed. The following summary is given: (1) The proportion of laboratory infections in testing dressings is very much greater than in testing other materials. (2) Experiments have shown the main factors responsible for this condition to be the area exposed, and the manipulative difficulties. The quantitative contributions of the several factors have been investigated. (3) A suitable method of sterility testing is described.—R. M. SAVAGE. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 237–251. (S. W. G.)

Toxins of Hemolytic Streptocci. The acid precipitable fraction (A. P. F.) and the acid soluble fraction (A. S. F.) were separated from the culture supernatant fluid of hemolytic streptococci by means of acetic acid. A. P. F. of the culture supernatant fluid possessed almost the same toxicity, skin reactivity and chemical properties as the bacterial protein described in a previous report. It is presumed, therefore, that a portion of bacterial protein might be dissolved and removed into culture media. For the purpose of obtaining a carbohydrate fraction, four fractions with various degrees of purification were isolated from A. S. F. by digestion with saliva and precipitation with alcohol. But by this method the minimum lethal dose for mice was never brought below 1 mg., nor could the carbohydrate and protein contained in them be separated from each other. Accordingly, it appears that the lethal toxin of hemolytic streptococcus may be somewhat different from those of other kinds of bacteria and may not exist in the carbohydrate fraction. Each fraction isolated from A. S. F. of the culture supernatant fluid by ammonium sulfate showed a toxicity for mice, which decreased in the order of the progess of saturation. The fraction precipitated by half saturation with ammonium sulfate has acted as a strongest lethal toxin for mice among those which have been hitherto isolated. Its minimum lethal dose for a mouse weighing 10 Gm. is 0.1 mg. and it is confirmed that it belongs to a protein on account of the content of nitrogen of 14.18% and other chemical properties characteristic of protein. With the exception of A. P. F., all fractions mentioned showed very slight intracutaneous reactions for rabbits, and the fractions which had a strong toxicity for mice did not always show a strong skin reaction. It is possible to say, therefore, that the lethal toxin and the skin activity have no direct connection between them.—T. SUGANUMA. *Tôhoku J. Exp. Med.*, 39 (1940), 180. (A. C. DeD.)

Tubercle Bacilli—Fluoroscopic Method for the Detection of. For employing the fluorescent lamp, preparations are made in the following way: A smear is made and fixed in the flame then stained for 15 minutes in carbol-auramin, after which it is washed in running water, decolorized in acid alcohol for 3 minutes and again washed. Carbol-auramin is made by mixing auramin "Bayer" Hollborn 1 Gm., liquefied phenol 5 Gm., distilled water a liter and thoroughly shaking. 823 specimens of varied origin were examined by the auramin fluorescence, by Ziehl-Neelsen staining, by culture and guinea-pig inoculation. By the first method 128 specimens were positive, by the second method 56 were positive, 90 by the third method and 140 by the fourth method. Twelve of the auramin positive were not confirmed by culture or animal inoculation. The author states that those slides which are only slightly positive should be examined again, for the fluoroscopic method does not distinguish *Myco. tuberculosis* from acid-fast saprophytes.—A. MOCHTAR. *Geneesk. Tijds. Nederlandsch-Indie.*, 80 (1940), 2432; through *Bull. Hyg.*, 16 (1941), 144. (T. C. G.)

Vitamin B_1 and B_6—Effect of Parenteral Administration of, on a Coccidium Infection. Vitamin B_1 and B_6 have an inhibiting influence on the development of *Eimeria nieschulzi* in rats on a special ration, when administered by a parenteral route, one at least as striking as when the vitamins are fed.—E. R. BECKER. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 494. (A. E. M.)

Wartime Storage of Antitoxins. Two methods are suggested for eliminating the need for glass containers and the use of refrigeration in storing antitoxic serum while preserving the potency and maintaining the sterility of the product. These are: (1) Reduction of the serum to powder by desiccation (removal of water by distillation at an effective working temperature of 36° C); (2) drying an intermediate precipitate produced during the manufacture of concentrated serum. Both methods yield a dry powder which may be stored in any type of container. The potency of the product is maintained. Cold storage accommodation is unnecessary and the powder is self-sterile. The desiccation method requires special apparatus for drying but this does not involve a large capital outlay. The material contains so much preservative that complete asepsis is not essential. The powder may be accurately assayed and normally, for issue, the required weight is dissolved in the required volume of sterile water. Sodium chloride and preservative are added as necessary, the pH is adjusted, the solution is filtered through a Seitz filter and tested for potency and sterility. In an emergency the Seitz filtrate can be distributed immediately; in a more serious emergency a solution could be made up with aseptic precautions and used as such at once. Advantage has been taken of subsidiary uses of the dried powder. It is noted that the ammonium sulfate product may be produced without special plant. Before issue it must be dialyzed, the pH adjusted, sodium chloride

and preservative added and assay completed before subjecting it to final dilutions. The method is limited to such antibodies as are normally precipitated with ammonium sulfate.—G. E. SHAW and H. G. HIND. *Chemist and Druggist*, 134 (1941), 252. (A. C. DeD.)

Wassermann Reagin—Relation of Antibodies in Syphilitic Serum to the. While it was originally believed that the Wassermann reaction was a specific antigen-antibody reaction, it was subsequently shown that similar results could be obtained with non-specific antigens, *i. e.*, alcoholic extracts of mammalian organs. This finding led to the assumption that the so-called Wassermann reagin present in syphilitic serum was not a true antibody against *T. pallidum*. In the present work the authors used a cultured strain of *T. pallidum* (Reiter strain) which though differing morphologically from the organisms found in human and rabbit infections, appears to be closely related serologically. Syphilitic serum absorbed with the Reiter strain no longer gave positive Wassermann or flocculation tests. Syphilitic human and rabbit sera agglutinated the Reiter strain to a high titer. Absorption of syphilitic sera with Wassermann and flocculation antigens did not change the reactivity of the sera for the Reiter strain. It was concluded that the Reiter strain contained antigens also present in mammalian tissue extracts. The findings support the thesis that the primary serologic change in syphilis is the development of antibodies to *T. pallidum*. The fact that the Reiter strain contains antigenic factors which react with syphilitic serum, some of which are not present in alcoholic beef heart extracts, suggests that many cases of syphilis which are negative to the Wassermann and flocculation tests now in use may be seropositive when tested with spirochete suspensions. This has been found true in a small number of cases already studied. Conversely, in conditions other than syphilis the serum may conceivably give false positive Wassermann or flocculation reactions with the usual tissue extracts, and yet be negative when tested with a spirochetal suspension. Thus a suspension of spirochetes may prove more valuable in the diagnosis of syphilis than the tissue extracts now used.—H. EAGLE and R. B. HOGAN. *J. Exp. Med.*, 71 (1940), 216. (T. C. G.)

Water Supplies—Bacteriological Examination of. The production of acid and gas within 48 hours in MacConkey's medium at 44° proved a very reliable test for *B. coli* in waters.—C. RAVEN, D. PEDEN and H. D. WRIGHT. *J. Path. Bact.*, 50 (1940), 287–294; through *J. Soc. Chem. Ind.*, 59 (1940), 646. (E. G. V.)

Whooping-Cough—Prophylactic Inoculation against. The views on and the results of active immunization against whooping-cough were gathered from the author's experiences in a London Whooping-cough clinic. Pertussis vaccines should be made only from strains which are in Phase I and which are either newly isolated, or have been maintained in Phase I by growth on Bordet-Gengou medium. Also vaccines are best made from cultures grown on media containing human blood. No chemically altered vaccine is as good as the simple suspension of killed bacteria. The author is not in favor of the very large dose and believes that effective prophylaxis consists in a primary stimulus followed by a secondary stimulus after an appropriate interval. He therefore gives a course of three injections, each of 4000 million organisms, at intervals of 3 to 7 days, following this up with a final injection of the same strength after the lapse of a month. Employing this method in a test group of 513 children with 46 known exposures and 45 suspected exposures, not a single case of whooping-cough occurred. The earliest age at which immunization can be attempted successfully is between 6 and 12 months. Children of this age may safely be given the full scale of dosage and from this period until 7 or 8 years of age they are easily immunized. Also immunization may be undertaken safely during epidemic periods, though this is not the best time to choose. Active artificial immunity is generally considered to be life-long but in the face of exaggerated exposure immunity can only be relative. Whooping-cough in a vaccinated child is usually mild and atypical.—I. H. MACLEAN. *Proc. Roy. Soc. Med.*, 33 (1940), 425; through *Bull. Hyg.*, 16 (1941), 69. (T. C. G.)

CHEMISTRY

GENERAL AND PHYSICAL

Absorbent Charcoal—Sedimentation Analysis of. The degree of fineness of powders of absorbant charcoal was determined by sedimentation in Andreasen's apparatus (*Dansk.Tids.Farm.*, 4 (1930), 261) in two solutions, sodium linoleate solution and sodium metaphosphate solution. Using the soap solution absorption is great; while with the phosphate solution absorption is minimal. Various commercial charcoals were studied. Particle size was determined with the aid of Andreasen's formula.—L. M. PAULSSEN. *Dansk Tids. Farm.*, 14 (1940), 201. (C. S. L.)

Anesthetics—Physicochemical Properties of Local. IV. Influence of Acid in Novocaine Salt on Passage through Cellophane. The following conclusions are given: (1) Starting with different equivalent salt solutions, the base passes through the membrane at different rates. The differences in pH of the solutions did not seem to affect the rates of passage. (2) The following list of salts is arranged in order of decreasing rates of passage: hydrochloride, salicylate, phenylpropionate, phenylacetate, isobutyrate, benzoate, tartrate, gluconate and citrate. (3) The rates of passage bear no apparent relationship to the molecular weights of the salts. (4) Three groups of salts may be formed according to experimental results: (*a*) hydrochloride; (*b*) benzoate, phenylacetate, phenylpropionate, isobutyrate; (*c*) tartrate, citrate, gluconate.—J. REGNIER, A. QUEVAUVILLER and A. FIEYRE. *Bull. sci. pharmacol.*, 47 (1940), 69–72. (S. W. G.)

Chemical Reactivity and Light Absorption. IV. Total absorption of light by a mixture of the reacting substances has been observed to be greater than the absorption of each substance taken separately in reactions studied. The increase in light absorption in each case varies with the concentration of the reducing agent. The dark reaction velocity also decreases with the decrease of concentration of the reducing agent. The chemical reactivity and the increase in the light absorption by a mixture are almost equally affected by a change in concentration of the reducing agent. The increased light absorption appears to be due to the activation of the molecules in the presence of the sensitizing agents. The activation of the molecules is associated with the loosening of the binding forces and the consequent increased light absorption.—N. R. DHAR, A. K. BHATTACHARYA and S. P. AGARWAL. *J. Indian Chem. Soc.*, 17 (1940), 675. (F. J. S.)

Colloids—Electrodeposition of Nickel on Iron and the Effect of, on the Nature of Deposit. The action of different inorganic colloids, both of elements and salts, on the electrodeposition of nickel on iron has been found to have a very good effect in so far as they give hard lustrous deposits with minimum number of pits. Prussian blue sol. comes next in order. The formation of pits has been discussed.—V. S. PURI and F. R. M. ALVI. *J. Indian Chem. Soc.*, 17 (1940), 699. (F. J. S.)

Distillation Apparatus—Recent. A pipe still and fractionating column are briefly described.—C. H. BORRMANN. *Oel Kohle, Petroleum,* 35 (1939), 784–785; through *J. Soc. Chem. Ind.*, 59 (1940), 332. (E. G. V.)

Drying Psychrometry. The value of the concept of the "drying potential" and the use of psychrometric charts in connection with air drying are stressed.—ANON. *Chem. Met. Eng.*, 47 (1940), 332; through *J. Soc. Chem. Ind.*, 59 (1940), 577. (E. G. V.)

Electro-organic Chemical Preparations. II. The list given previously is extended to the end of 1939.—S. SWANN, JR. *Trans. Electrochem. Soc.*, 77 (1940), Preprint 26, 315–354; through *J. Soc. Chem. Ind.*, 59 (1940), 427. (E. G. V.)

Evaporation from Earthen Jugs. A theory of the evaporation of water from earthen jugs, based on the theory of the wet- and dry-bulb hygrometer, has been developed. The agreement between theory and experimental data is good.—H. GUPTA and A. CHANDRA. *Indian J. Physics,* 13 (1939), 305–308; through *J. Soc. Chem. Ind.*, 59 (1940), 416. (E. G. V.)

Hydrogen Overpotential at Mercury Surface—Decay of. The decay of hydrogen overpotential was noted against colored electrode during the electrolyte reduction of maleic and fumaric acids in an aerobic condition. A tentative scheme as to the various stages that might occur during the discharge of H ions has been suggested and discussed.—S. C. GANGULI. *J. Indian Chem. Soc.*, 17 (1940), 691. (F. J. S.)

Luximeter. A photoelectric instrument for measuring light transmission through a liquid is described. It can be used for determining the degree of pasteurization of milk, the blue color of the sample being α, the amount of phosphatase present.—W. L. CARSON. *Gen. Elec. Rev.*, 43 (1940), 91–92; through *J. Soc. Chem. Ind.*, 59 (1940), 542. (E. G. V.)

pH—Role of, in Pectic Material. A review of published work, stressing the importance of pH in the preparation and application of the pectins. Maximum gel formation takes place on reduction of the negative charge on the colloidal pectin particles by hydrogen ions and in presence of a high concentration of sugar.—M. DERIBERE and M. DE BUCCAR. *Bull. assoc. chim. sucr.*, 57 (1940), 37–44; through *J. Soc. Chem. Ind.*, 59 (1940), 486. (E. G. V.)

Resin Solutions—Physical Chemistry of. III. Viscosity of Shellac Solutions in Mixed Solvents. The viscosity of shellac dissolved in a solvent mixture composed of two non-solvents, acetone and water, has been studied over a wide range of concentration and temperature. The viscosity-solvent composition curves show well-defined minimum at a definite ratio of water to acetone in the solvent mixture for shellac concentration of 25% and higher; the lower the temperature, the more sharp and well-defined are the minima. For lower concentrations of shellac below 20% the viscosity continually rises with increase in the proportion of water. The striking difference in properties in shellac solutions in aqueous acetone below and above 20% of shellac has also been noted in the case of some other properties, *e. g.*, gelation capacity, temperature coefficient of relative viscosity, etc., for which either solvation or micelle formation has been suggested as an explanation. The solvent power as determined by precipitation with an inert solvent like benzene, or by cooling, always shows a maximum at about 80% water content. Thus the idea that viscosity is a criterion of solvent power is shown to be of restricted validity.—SANTI RANJAN PALIT. *J. Indian Chem. Soc.*, 17 (1940), 663. (F. J. S.)

Sol Particles—Studies on the Cataphoretic Speed of, as Dependent on the Redox Potential of the Liquid Medium. Mobility of colloidal particles of platinum and gold in very dilute aqueous solutions of a few redox systems has been studied. Results indicate that the change in the cataphoretic speed and hence the electrokinetic potential of the gold and platinum particles are not probably due to any specific ion adsorption but are due to more general factors, the predominant one being the electron activity of the medium which is responsible for the oxidation-reduction potential of the system.—J. C. GHOSH and N. G. BASAK. *J. Indian Chem. Soc.*, 17 (1940), 721. (F. J. S.)

Surface Active Agents—Laboratory Uses for. Surface active agents may be used to aid quantitative separations by centrifuging, prevent creeping of precipitates, stabilizing suspensions, etc.—C. M. ALTER and D. S. THOMAS. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 525.

Sugar Charcoal—Adsorption of Mono- and Polybasic Acids by. The adsorption of mono-, di- and tri-carboxy acids and bi-salts of dibasic acids by sugar charcoal activated at 800–850° has been studied. Adsorption of solutes, particularly monocarboxy acids, from solution by sugar charcoal follows the ordinary laws of adsorption and is mainly a surface phenomenon as log x and log C curves are straight lines.—KESHO DASS JAIN and J. B. JHA. *J. Indian Chem. Soc.*, 17 (1940), 685. (F. J. S.)

Vapor Pressures of Aqueous Solutions. A new method has been devised to determine the vapor pressure of liquids and solutions. Compared to the other methods, this method has been found to be simple, rapid and to yield accurate and reproducible results. Observed values of vapor pressures of aqueous solutions of $ZnCl_2$, $Ca(CNS)_2$, $Zn(CNS)_2$ and H_3PO_4 are recorded. From thermodynamical considerations osmotic pressures of these solutions have been calculated. Experimental results suggest a strong affinity between water and these salts which is in agreement with the properties of these substances.—G. S. KASBEKAR. *J. Indian Chem. Soc.*, 17 (1940), 657. (F. J. S.)

INORGANIC

Barium—Modification of the Iodate Method for Determining. In the method described for the determination of barium, the iodate is precipitated adding a measured excess of potassium iodate; the whole is transferred to a graduated flask and the excess of potassium iodate is determined in an aliquot part by sodium thiosulfate, allowance being made for the volume of the precipitate. The error introduced by washing the precipitate owing to its appreciable solubility is thus avoided.—F. C. GUTHRIE. *J. Soc. Chem. Ind.*, 59 (1940), 98. (E. G. V.)

Iodic Acid—New Monograph for. This monograph includes a description, residue on ignition, Cl^-, Br^-, I^-, SO_4^{--}, heavy metals and Fe tests.—GLENN L. JENKINS and W. THOMAS SPAIN. *Bull. Natl. Formulary Committee,* 9 (1941), 153–154. (H. M. B.)

Magnesium and Alkalies in Lime—Limit for. The following altered procedure for testing these impurities is recommended: "Dissolve 0.5 Gm. of lime in 30 cc. of H_2O and 15 cc. of diluted hydrochloric acid. Neutralize the solution with ammonia T.S., heat to boiling and add ammonium oxalate T.S. to precipitate the calcium completely. Heat the mixture on a steam bath for one hour, cool, dilute to 100 cc. with water, mix well and filter. To 50 cc. of the filtrate add 0.5 cc. H_2SO_4, evaporate

to dryness and ignite to constant weight. The weight of the residue does not exceed 9 mg.—JOSEPH ROSIN. *Bull. Natl. Formulary Committee*, 9 (1941), 155–156. (H. M. B.)

Monobasic Potassium Phosphate—New Monograph for. This new monograph for the Natl. Formulary includes a test for Ca, loss on drying over H_2SO_4, loss on ignition, pH values, Cl^-, N compounds, SO_4^{--}, heavy metals, Fe and N compounds.—GLENN L. JENKINS and W. THOMAS SPAIN. *Bull. Natl. Formulary Committee*, 9 (1941), 154–155. (H. M. B.)

Phosphate—Detection and Elimination of, in Qualitative Analysis. A revised method for the detection and elimination of phosphate, based on Curtman's zirconyl chloride method, depends upon the fact that zirconium (with hafnium) forms a phosphate which is insoluble in strongly acid solution, and hence phosphate can be removed effectively from acid solution by the addition of soluble zirconium salts. Semiquantitative data are given to show that the method of detection is sensitive, and the method of elimination is completely reliable from a quantitative standpoint. Experiments are cited which show that nearly all the disadvantages of the present methods have been overcome.—F. K. PITTMAN. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 514–515. (E. G. V.)

Phosphoric Acids—Viscosity of Strong. A nomograph is presented giving relationships between temperature, phosphorous pentoxide and refractive index.—D. S. DAVIS. *Chem. Met. Eng.*, 47 (1940), 155; through *J. Soc. Chem. Ind.*, 59 (1940), 441. (E. G. V.)

Potassium Determination—Modified Silver Cobaltinitrite Method for. A procedure for the determination of potassium as potassium silver cobaltinitrite has been described. In the temperature range studied, it appears that 20° is the most favorable for precipitation, since at that point the nitrite to potassium ratio remains constant over a wide range of potassium concentrations. Water as a wash reagent has been replaced by a solution of water, alcohol and ether. The method appears to be accurate within ±2 per cent.—JOHN E. HARRIS. *J. Biol. Chem.*, 136 (1940), 619. (F. J. S.)

Selenium—Titrimetric Determination of Quadrivalent, with Permanganate. Excellent results were obtained by adding selenite solution containing 0.04–0.120 Gm. of selenium to an excess of potassium permanganate in 23–25% sodium hydroxide solution. In ten to fifteen minutes all the selenite is oxidized to sexivalent selenium and the permanganate is reduced to manganate. After diluting with water, adding sulfuric acid, an excess of oxalic acid and some manganous salt (in the order given) all permanganate and manganate are reduced to manganous and the excess oxalic acid can be titrated with potassium permanganate at a temperature above 50°. A blank should be run under the same conditions.—H. STAMM and M. GOEHRING. *Z. anal. Chem.*, 120 (1940), 230–232. (S. W. G.)

Sulfurous Acid—Sharpening Test for. The test specified for meat inspection and in the German pharmacopœia is based on heating with phosphoric acid and testing the vapors with moist potassium iodate-starch paper. The test is much more sensitive if the potassium iodate is moistened with normal sulfuric acid instead of with water. Moistened with acid, the paper showed a blue color after exposure for forty-five seconds; while the water-moistened paper required exposure for about an hour for the development of the blue color. When water is used, colorless hydriodic acid is formed which, in the presence of sufficient acid, will react with iodate to liberate iodine.—B. STEMPEL *Z. anal. Chem.*, 120 (1940), 311–313. (S. W. G.)

ORGANIC

Alkaloids

Aconite Alkaloids. III. The Oxidation of Aconitine and Derivatives with Nitric Acid and Chromic Acid. A study of the oxidation of aconitine and a number of its derivatives with nitric acid and chromic acid has shown that a complicated series of steps is involved which may be intercepted at different stages or in different orders. The product of vigorous action of nitric acid on aconitine, oxonitine, oxoaconitine, aconitoline, etc., is a neutral N-nitrosonitro derivative containing only three methoxyl groups with the formula $C_{31}H_{35}O_{18}N_3$ which represents a stage beyond all of the others. On acid hydrolysis this yields the corresponding secondary base $C_{31}H_{36}O_{12}N_2$. Gentler action of nitric acid on oxonitine and oxoaconitine gives intermediate nitro derivatives with loss of one methoxyl group, respectively, $C_{32}H_{36}O_{13}N_2$ (or possibly $C_{33}H_{38}O_{13}N_2$) and $C_{33}H_{36}O_{13}N_2$. The formula of Lawson's aconitoline, obtained from aconitine with chromic acid, has been revised to $C_{33}H_{41}O_{10}N$ and apparently results from oxidation of a secondary OH group to CO with simultaneous loss of methyl alcohol. On saponification it yields the tertiary base, $C_{24}H_{35}O_8N$, obtained by Schulze from aconine which in turn readily gives a methiodide, in contrast to aconitoline itself. The production and interpretation of other substances from aconitine and related substances are discussed.—WALTER A. JACOBS and LYMAN C. CRAIG. *J. Biol. Chem.*, 136 (1940), 323. (F. J. S.)

Caffeine in Santa Catarina (Brazilian) Maté Plant. Samples from a number of localities contain 0.51–1.73% of caffeine.—A. A. ADDOR. *Rev. Aliment. Chim. Ind.*, 4 (1940), 3; through *J. Soc. Chem. Ind.*, 59 (1940), 762. (E. G. V.)

Erythrina Alkaloids. VII. Isolation and Characterization of the New Alkaloids, Erythraline and Erythratine. Erythramine and two new alkaloids, named erythraline and erythratine, have been isolated from the seeds of *Erythrina glauca* Willd. Erythraline was also isolated from the seeds of *Erythrina fusca* Lour., *Erythrina Folkersii* Kruk. and Mold., *Erythrina variegata* L. var. *orientalis* (l.) Merr., *Erythrina velutina* Willd., and *Erythrina macrophylla* DC. Hypaphorine was isolated from all the above mentioned species and all for the first time, with the exception of *Erythrina variegata* var. *orientalis* from which it had been previously isolated by others. Erythraline and hypaphorine undoubtedly exist also in *Erythrina velutina* forma *aurantiaca* (Ridl.) Kruk. and *Erythrina Grisebachii* Urb. The crystalline erythraline and erythratine bases, their hydrobromides and hydriodides, have been described. Many microanalyses on several samples and from different species have shown that erythraline has the empirical composition $C_{18}H_{19}NO_3$, and erythratine has the empirical composition $C_{18}H_{21}NO_4$. Erythraline and erythramine were of comparable activity (7–8 mg./Kg.) in causing a curare-like paralysis in frogs, whereas erythratine had one-tenth of this activity.—KARL FOLKERS and F. KONIUSZY. *J. Am. Chem. Soc.*, 62 (1940), 436–441. (E. B. S.)

Fluidextract of Ergot—Quantitative Determination of, with the Aid of Chromatography. A solution of ergometrine and ergotoxine in 50% alcohol was chromographically passed through a column of Al_2O_3 with ether as developing fluid. The alkaloids passed through quantitatively and could be separated from the thus-obtained fluid by shaking with two different buffer solutions. Conducting similar chromatography of fluidextract of ergot the ballast substances mostly remained in the column and the obtained liquid was but faintly colored and con-

tained the alkaloids of the galenical in sufficient purity for the separation by the cited method. Assay method: 5 cc. of fluidextract of ergot is chromatographed in a column (11 cm. high, 1.2 cm. dia.) of aluminium oxide, Merck. Development is with 50 cc. of ether. The approximate 40 cc. of fluid obtained is first shaken 6 times with 15-cc. portions of phosphate buffer, pH 6.8, and the aqueous layers separated, combined and made to 100 cc. This contains the ergometrine group of alkaloids. The ergotoxine group are then separated by again shaking the ether from the column three times with 15-cc. portions of 1% tartaric acid solution, and the combined extracts here are made to 50 cc. To 1-cc. aliquots of each of these solutions, 2 cc. of *p*-dimethylaminobenzaldehyde reagent is used to develop the color, and this is measured in the step photometer, reading the concentration of alkaloid from a standard curve. The authors consider the method superior to their previously described perforation extraction method for ergot alkaloids (*Dansk Tids. Farm.*, 12 (1938), 279).—P. F. JÖRGENSEN and M. TÖNNESEN. *Dansk Tids. Farm.*, 14 (1940), 134. (C. S. L.)

Iodo-Cuprous Reagent for Alkaloids: Precipitation Reactions and Color Reactions. A study was made of the application of Grichard, Rivat and Scatchard's reagent (*Ann. chim.*, 15 (1921), 14) which gives with yperite a yellowish white precipitate of di-iodomethyl sulfide. Numerous alkaloid salts, aconitine, brucine, cinchonidine, codeine and nicotine give more or less marked yellowish-white or brown precipitates. Colchicine, ephedrine, eserine and veratrine precipitate after careful addition of dilute hydrochloric acid. The purine group of alkaloids (uric acid, caffeine, theobromine), the chief glucosides, the barbiturates, picrotoxine, adrenaline, do not give any precipitate, either from simple aqueous or from hydrochloric acid solution. While for most alkaloids Bouchardat's iodine-iodide reagent is more sensitive, for quinine and sparteine the iodo-cupric reagent is more sensitive. The precipitates obtained with sparteine, quinine and cocaine contain copper. The eserine precipitate dissolves in ammonia to an unstable reddish violet which gradually turns brown. The violet coloration given by ephedrine with copper is not interfered with by the iodine or sodium iodide of the reagent. This very stable color disappears on acidification. Adrenaline under the same conditions gives a reddish color that also disappears on acidification.—M. PÉRONNET and J. GUÉNIN. *J. pharm. chim.*, [9], 1 (1940), 142–147. (A. P.-C.)

Lupine Studies. XV. The Alkaloids of Lupinus Sericeus Pursh. *Lupinus sericeus* Pursh. collected in Colorado contained when dried 3.68% of alkaloids consisting of sphatulatine, previously described, and nonalupine, a new lupine alkaloid. A reinvestigation of the chemistry of spathulatine has confirmed the empirical formula previously assigned to this base. Nonalupine is not basic enough to form salts with strong acids.—J. F. COUCH. *J. Am. Chem. Soc.*, 62 (1940), 554–556. (E. B. S.)

Essential Oils and Related Products

Citrus Oils—Studies on Palestine. A modification of the Wilson method of essential oil determination is given.—ANON. *Perfumer. Essent. Oil Record*, 32 (1941), 139. (A. C. DeD.)

Cupweed from Provence—Concrete Oil of. Fresh flowers of *Helicrysum angustifolium*, collected in Esterel, yielded by extraction with light petroleum 1% of a waxy, yellow oil (drop point, Ubbelohode, 47–48°, acid value 73, ester value 136) yielding by distillation in water vapor under reduced pressure 4.9% of a thick oil which has d_{15}^{15} 0.9239, n_D^{20} 1.5005–1.5046, acid value 14, ester value 28.1, formaldehyde 1.25%; on oximination, 1 Gm. required 0.5 cc. of 0.5*N* potassium hydroxide. This oil contained octoic acid, acetic acid, eugenol and a sesquiterpene, $C_{15}H_{24}$, having a boiling point of 123–127°/13 mm., d_{15}^{15} 0.9068, n_D^{20} 1.5026, α (1 dm.) + 10°, iodine value 275. Extraction of the concrete with ethyl alcohol gave an absolute oil in 85% yield which yielded 6.35% of oil on distillation with water vapor under reduced pressure.—S. SABETAY. *Ann. chim. anal.*, 22 (1940), 89–91; through *J. Soc. Chem. Ind.*, 59 (1940), 494. (E. G. V.)

Essential Oils—Storing. Essential oils are classified into seven groups on the basis of composition and properties. Methods of storage for each group are offered.—JOSEPH KALISH. *Drug and Cosmetic Ind.*, 48 (1941), 412–413, 419. (H. M. B.)

Macauba Oil. Kernels of *Acrocomia totai* and *A. aculeata*, Lodd (*A. sclerocarpa*), contain 65 and 55–60% of oil, respectively. A typical sample of the former has melting point 20.5°, solidification point 18.5°, saponification value 237, index of refraction 38.7, iodine value 29.6; the corresponding figures for the latter are 25.6°, 21.3°, 240, 37.4 and 20.8.—M. SILVA. *Industria y quím.*, 3 (1940), 39; through *J. Soc. Chem. Ind.*, 59 (1940), 746. (E. G. V.)

Perfumery Materials and Essential Oils—Fluorescence (Luminescence) of, in Filtered Ultraviolet Light. After detailed consideration of the published data it is concluded that the majority of perfumery materials show little or only a feeble blue or violet fluorescence in ultraviolet light and the results recorded vary with different observers. The results obtained by the use of the analytical quartz lamp are therefore unreliable in assessing the purity of these materials.—A. MULLER. *Deut. Parfüm.-Ztg.*, 26 (1940), 37–40; through *J. Soc. Chem. Ind.*, 59 (1940), 566. (E. G. V.)

Wild-Rose Seeds and Wild-Rose Seed Oil—Analysis of. Russian wild-rose (*Rosa canina*) seeds contained 7.54% of water and (calculated on dry basis) fat 9.44, ash 1.94, nitrogen 1.45, carbohydrates 45.05% (comprising glucose 1.96, sucrose 1.01, maltose 0.70, starch 0.0, hemicellulose 8.15, cellulose 32.23) and ethereal oil 0.2–0.3%. The ether-extracted oil possessed a vanilla-like odor and had index of refraction 1.492, density at 20° 0.9269, viscosity (Ostwald at 20°) 51.68, acid value 2.84, saponification value 191.5, iodine value (Hubl-Waller) 154.9, thiocyanogen value 94.9, Reichert-Meissl value 1.45, Polenske value 0.40, unsaponifiable matter 1.65%. The fatty acids consisted of solid acids 4.52, oleic 29.32, linoleic 56.71 and linolenic acid 9.45%. Films of the raw oil on glass showed "crawling" and did not dry in 3 weeks; after heating to 250° it became dust dry in 14 days.—V. A. RUSCH and G. A. IVANOVA. *Compt. rend. acad. sci. U. R. S. S.*, 26 (1940), 259; through *J. Soc. Chem. Ind.*, 59 (1940), 680. (E. G. V.)

Glycosides, Ferments and Carbohydrates

Akebia Quinata—Constituents of the Branches of. Previous investigations showed that the branches of *Akebia quinata* Decne yielded a saponin, akebin, of the composition $(C_{36}H_{56}O_{20})_3$. On hydrolysis this compound gave a sapogenin, $C_{31}H_{50}O_4$, called akebigenin. The present authors show that akebigenin has the same composition as hederagenin ($C_{30}H_{48}O_4$ or $C_{31}H_{50}O_4$) and that akebigenin is not a single compound but on recrystallization from alcohol yields both hederagenin and oleanolic acid. The nature of these two products was proved by their identity with the same compounds from other sources.—RITTI KAWAGUCHI and K. W. KIM. *J. Pharm. Soc. Japan*, 60 (1940), 585–591 (in English, 237). (N. L.)

Carbonic Anhydrase—Relation of Zinc to. The report that carbonic anhydrase contains at least

0.3 per cent of zinc and that all of the zinc in the red blood cells is bound into this enzyme has been confirmed. The highly sensitive zinc reagent, dithizone, produced little significant inhibition of carbonic anhydrase. KCNS was markedly inhibitory. An equivalent amount of zinc ion was unable to neutralize this inhibitory property of thiocyanate. Zinc ions alone, in relatively large amounts, produced a 50 to 60 per cent inhibition. Under the experimental conditions employed there is but little significant decrease in the carbonic anhydrase to hemoglobin ratio in zinc-deficient rats.—E. HOVE, C. A. ELVEHJEM and E. B. HART. *J. Biol. Chem.*, 136 (1940), 425. (F. J. S.)

Enzymes—Assay of. The difficulties (from the twofold standpoint of chemical analysis and regulatory enforcement) of the determination of the activity of papain and of fat-splitting enzymes are briefly discussed.—A. K. BALLS. *J. Assoc. Official Agr. Chem.*, 23 (1940), 446–447. (A. P.-C.)

Glycerol—Determination of. A method for the determination of glycerol, in the presence of dextrose, which is applicable to a fermented medium. The procedure involves the determination of the sugar by a copper titration method, followed by the oxidation of another sample with ceric sulfate under standard conditions. Both the sugar and the glycerol are oxidized, but from a correction for the ceric sulfate used by the sugar, the glycerol is calculated by means of suitable equations or read from a graph.—E. I. FULMER, R. J. HICKEY and L. A. UNDERKOFLER. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 729–730. (E. G. V.)

Glycerol—Determination of Pure. The heat of combustion, Q, of the sample is determined. Then % glycerol is given by $(100Q/4315)-1.13A$, where A is the % content of non-volatile organic substances.—B. RAVITSCH. *J. Applied Chem. Russ.*, 12 (1939), 1571-1574; through *J. Soc. Chem. Ind.*, 59 (1940), 512. (E. G. V.)

Horsechestnuts as a Source of Medicinal Saponin. Owing to difficulty of obtaining supply of saponin-containing drugs in Denmark, horsechestnut seed preparations were studied. A purified preparation of the saponin was made by boiling the shelled and pulverized defatted nuts in 93% alcohol. After separation, the saponin was precipitated with ether. It was purified by boiling in absolute alcohol and precipitation; after cooling, it was treated with 96%, then with 93% alcohol. Taken up again, it was treated with active charcoal and on precipitation with ether a colorless saponin separated. This had a hemolytic index of 37,000. The hemolytic indices of the powdered horsechesnut preparations were 5820 and 5550 indicating a content of about 15% saponin. Tinctures were made with dilute spirit and these contained practically all the saponin of the seed. They were of about the same hemolytic index as Tincture of Quillaga Bark and could be used for the preparation of a Liquor Carbon. Detergens. Three fluidextracts were made using 20, 40 and 60% spirit. All gave good extraction of the saponin, best with the 40 and 60% spirit. The first fraction of percolate (800 Gm.) contained most of the saponin. The first after-percolate (1 kilo) had about $^1/_{10}$ the activity of the above, and further percolates had little activity. A decotion (1–20) extracted about 30% of the saponin; an infusion, about 40%. If infusion was done at 45° C. for $^1/_2$ hour, 60% of the saponin was extracted. Attempting to make a preparation analogous to Decoctum Chinae cum Senegæ, if horsechestnut powder and cinchona bark were boiled together a far lower extraction of saponin occurred (hemolytic index less than 6). Tannic acid precipitated this saponin.—C. J. T. MADSEN. *Dansk Tids. Farm.*, 14 (1940), 225. (C. S. L.)

Mannitol and Sorbitol in Pharmacy. Considerable interest is being shown mannitol and sorbitol not only as glycerin substitutes, but also inasmuch as they seem to possess some points of superiority for certain specific uses. Derivatives of these polyhydric alcohols are also used as vasodilators, emulsifying agents, absorption bases and in the preparation of organo-metallic medicinals. The pharmaceutical chemist will find much of interest in this article from the standpoint of both improved and new product possibilities.—H. C. Speel. *Am. J. Pharm.*, 113 (1941), 134. (A. C. DeD.)

γ-Sugars—Structure of. V. The following summary is given: (1) The absence of mutarotation in 6-methylfructose, 3,4,6-trimethylfructose, 1,3,4,6-tetramethylfructose and 5-methylglucose precludes a cyclic structure being adopted for these substances. (2) Studies of the action of perbenzoic acid and of ozone on 6-methylfructose, 3,4,6-trimethylfructose, 1,3,4,6-tetramethylfructose and their methylfructosides show no evidence of the presence of an olefinic linkage and exclude an enediol structure for these substances. (3) A keto-alcohol structure is assigned to γ-fructose and its non-glycosidic derivatives, an aldehyde-alcohol structure to γ-glucose and its non-glycosidic derivatives, and a furanose ring structure to γ-glycosides. Thus a rational explanation of the striking difference in stability of γ-sugars and their glycosides is provided. (4) Kinetic studies of the hydrolysis of α-methylglucofuranoside and of α-methylglucopyranoside by 0.1*N* hydrochloric acid lead to values for the activation energies of 16,055 and 17,590 calories per Gm. molecule, respectively, from which, using the kinetic equation $\log_e K = \log_e PZ - E/RT$, the values of the term $\log_e PZ$ are calculated to be 19.57 and 16.78, respectively. (5) Interpreting the factor P as a measure of orientation effect an explanation is put forward for the more rapid hydrolysis of glycofuranosides than glycopyranosides by dilute acid.—F. HARTLEY and W. H. LINNELL. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 332–343. (S. W. G.)

Other Plant Principles

Cannabidiol—Structure of, Product Isolated from the Marihuana Extract of Minnesota Wild Hemp. I. A new compound, cannabidiol, present in the purified red oil of *Cannabis sativa* has been isolated through the *bis*-3,5-dinitrobenzoate. This diester is a crystalline, easily purified compound. Ammonolysis of it gives cannabidiol which has the formula $C_{21}H_{30}O_2$ or $C_{21}H_{32}O_2$, the former probably being the correct one. Cannabidiol is oxidized to n-caproic acid, methylated with difficulty to a dimethyl ether and converted to a *bis*-*m*-nitrobenzenesulfonate. It is concluded that this substance is closely related to cannabinol in structure. Half the molecule is probably a dihydroxy n-amylphenyl, the other half probably an unsaturated alicyclic nucleus.—ROGER ADAMS, M. HUNT and J. H. CLARK. *J. Am. Chem. Soc.*, 62 (1940), 196–200. (E. B. S.)

Carotene—Adsorption Method for the Determination of Pure. The modified A. O. A. C. method, in which the carotene in petroleum benzin solution is purified by shaking with an adsorbent to remove impurities, is shortened by adsorbing the xanthophyll and impurities directly, instead of first washing with methanol and then using the adsorbent. Comparisons on 19 samples showed that the shorter adsorption method gives the same results for pure carotene as does the longer modified A.O.A.C. method.—G. S. FRAPS, A. R. KEMMERER and S. M. GREENBERG. *J. Assoc. Official Agr. Chem.*, 23 (1940), 659–662. (A. P.-C.)

Carotene—Determination of, in Presence of Lycopene. Lycopene-absorbing reagent is pre-

pared as follows: heat 100 Gm. of magnesium carbonate in an electric furnace at 200° C. for 1 hour; test the reagent for adsorption of carotene; if it does not adsorb carotene, test with lycopene solution; if 3 to 5 Gm. of the reagent extracts all the red pigment from 50 cc. of the lycopene solution, the reagent is ready for use. If the reagent adsorbs carotene, add small quantities of water (3 cc. at a time) and test again; repeat this treatment until the reagent does not adsorb carotene; then test with lycopene solution as before. As this lycopene reagent is unstable, it must be kept in tightly closed containers to prevent change in its moisture content and it must be tested each time it is used. The lycopene reagent removed much more impurity than the xanthophyll reagent (*J. Assoc. Official Agr. Chem.*, 22 (1939), 190) from crude carotene solutions derived from watermelons, dried apricots, red peppers and tomatoes, and 2% to 3% more impurities from crude carotene solutions from alfalfa. It is thus possible to prepare at least two adsorbents with different selective powers for purifying carotene solutions: the xanthophyll reagent adsorbs xanthophyll but no lycopene or carotene; the lycopene reagent adsorbs xanthophyll and lycopene but not carotene.—G. S. FRAPS, A. R. KEMMERER and S. M. GREENBERG. *J. Assoc. Official Agr. Chem.*, 23 (1940), 422–425. (A. P.-C.)

Frangula Emodin. Frangula emodin was obtained from Fluidextract of Frangula by chromatography. The red color given by this emodin in alkali was not found constant; it faded on standing. However the yellow color given in acid solutions was stable, and a standard curve for estimation in the step photometer using filter S 50, was constructed. If frangula emodin was boiled 7 hours in alcohol solution with 2% HCl or an equivalent strength of H_2SO_4, colorimetry results became too high, hence it was concluded that this method was not satisfactory for determination of the glucoside-bound fraction. The emodin, a phenol with 3 hydroxyl groups, has the characteristics of a weak acid and the colors developed in acid and alkaline solution are those of the non-ionized and ionized salts. They made possible an approximate determination of dissociation constant. The first acid dissociation constant was approximately $10^{-10.8}$.—P. F. JÖRGENSEN. *Dansk Tids. Farm.*, 14 (1940), 169. (C. S. L.)

Helenium—Constituents of Certain Species of. III. The Ester Nature of Tenulin. Evidence has been presented to show that tenulin contains a double bond, an acetoxyl group and a hydroxyl and a carbonyl group, both of which are sterically hindered. A summary of the reactions of tenulin which have been used in this discussion and which have been hitherto unrecorded is shown diagrammatically in a chart. Two hitherto unexamined species of *Helenium, i. e., H. quadridentatum* and *H. montanum*, have been investigated for bitter and sternutative substances. *H. quadridentatum* contains helenalin, while *H. montanum* contains tenulin.—E. P. CLARK. *J. Am. Chem. Soc.*, 62 (1940), 597–600. (E. B. S.)

Momordica Cochinchinensis—Sterol of the Seeds of. Extraction of the seeds of *Momordica cochinchinensis* with ether, followed by saponification of the extractive with alcoholic potassium hydroxide, yields 0.15% of an unsaponifiable oily substance, which, after standing, formed needles, melting at 132–152°. The impure crystals were converted into a meta-dinitrobenzoate which after recrystallization from chloroform-methanol or ethyl acetate melted at 195–199°. After purification by chromatographic methods, the sterol melted at 156.5–163.5°; analysis indicated a formula of $C_{28}H_{46}O$; $[\alpha]_D^{23} = +5.81$. It gave an acetate and a benzoate, melting 174.5–176.5° and 196–198°, respectively. The authors indicate that the sterol, $C_{28}H_{46}O$, found in the seeds of *Momordica cochinchinensis* is identical with cucurbitasterin, $C_{28}H_{46}O$, which had been previously isolated from several plants of the *Cucurbitaceae* family.—SATORU KUWADA and SIZUO YOSIKU. *J. Pharm. Soc. Japan*, 60 (1940), 581–585 (in German, 232). (N. L.)

Nobiletin—Synthesis of. The author investigated the chemical constitution of nobiletin, a new flavone from *Citrus nobilis* Lour and found it to be identical with 5,6,7,8,3′,4′-hexamethoxyflavone prepared synthetically. 2-Hydroxy-3,4,6-trimethoxyacetophenone was converted into 2,5-dihydroxy-3,4,6-trimethoxyacetophenone by oxidation with potassium persulfate in aqueous sodium hydroxide. Partial methylation using methyl sulfate and potassium carbonate in acetone gave 2-hydroxy-3,4,5,6-tetramethoxyacetophenone, which formed 2-veratroyloxy-3,4,5,6-tetramethoxyacetophenone when treated with veratroyl chloride in the presence of pyridine. The ester was then treated with sodamide and toluene to form 2-hydroxy-3,4,5,6-tetramethoxy-ω-veratrolacetophenone which on treatment with sulfuric acid cyclized to give 5,6,7,8,3′,4′-hexamethoxyflavone.—ZEN'ICHI HORII. *J. Pharm. Soc. Japan*, 60 (1940), 614–616 (in English, 246–248). (N. L.)

Osage Orange Pigments. III. Fractionation and Oxidation. It is shown that the two pigments of the fruit of the osage orange (*Maclura pomifera* Raf.) are present in approximately equal amounts. Osajin dimethyl ether has been prepared. Anisic (*p*-methoxybenzoic) acid has been obtained from the oxidation of osajin dimethyl ether with alkaline hydrogen peroxide. Veratric (3,4-dimethoxybenzoic) acid has been obtained from the oxidation of pomiferin trimethyl ether with alkaline hydrogen peroxide.—M. L. WOLFROM and A. S. GREGORY. *J. Am. Chem. Soc.*, 62 (1940), 651–652. (E. B. S.)

Pyrethrum Grown in India—Pyrethrin Content of. Flowers of *Chrysanthemum cinerariaefolium* or extracts of them are widely used as household insecticides, livestock sprays and horticultural dusts and sprays. The control of disease due to insects is an acute problem in India and attempts are being made to cultivate the plant there. Chemical assay depends on estimation of pyrethrin I and pyrethrin II which are esters of the ketone-alcohol pyrethrolone. Pyrethrin content was determined by the method of Gnadinger and Corl with slight modifications. Flowers from different parts of India were tested and they showed a high percentage of pyrethrin as compared to imported sample. Experimental details are given and results are tabulated.—J. K. LAHIRI, S. GHOSH and R. N. CHOPRA. *Jour. A. Ph. A.*, 30 (1941), 72. (Z. M. C.)

Rapanone—Anthelmintic Principle of Rapanea Maximowiczii. Rapanone, the orange colored crystalline substance from the bark and woody portion of *Rapanea maximowiczii* Koidz, a shrub of the *Myrsinaceae*, possesses strong anthelmintic properties. Rapanone, $C_{19}H_{30}O_4$, resembles embelin, $C_{20}H_{32}O_4$, in its compostion but differs in that the former yields myristic acid on permanganate oxidation. The authors synthesized 3,6-dihydroxy-2-tridecylbenzoquinone and found this to be identical with natural rapanone. The ethyl ester of 3,4,5-trimethoxybenzoylacetate was condensed with *n*-undecyl iodide in the presence of sodium ethylate to give ethyl α-(trimethoxybenzoyl)-myristicate, m. 49–50°, which on treatment with ethanolic potassium hydroxide formed 3,4,5-trimethoxytridecanophenone $(CH_3O)_3C_6H_2.CO.CH_2.C_{11}H_{23}$, m. 61–62°. Reduction of this ketone with sodium and isoamyl alcohol gave 3,5-dimethoxy-1-tridecylbenzene $(CH_3O)_2C_6H_3.CH_2.C_{12}H_{25}$, m. 41.5–42.5°, which in oxidation with sodium dichromate in glacial

acetic acid gave 1-tridecyl-3-methoxybenzoquinone (CH_3O).$C_6H_2O_2$.$C_{13}H_{27}$, m. 82–83.5°. Treatment with methylamine gave 1-tridecyl-3,6-di(methylamino)-benzoquinone, m. 141–142°, which on boiling with a mixture of sulfuric and acetic acids gave 1-tridecyl-3,6-dihydroxybenzoquinone, m. 139–140°, which was found to be identical with rapanone from natural sources. Rapanone forms a dibenzoyl ester, which melts at 88–90° and a leucotetraacetate, melting at 117–118°.—MITIZO ASANO and KAZUTAKA YAMAGUTI. *J. Pharm. Soc. Japan*, 60 (1940), 585–591 (in English, 246–248). (N. L.)

Zyzyphus Vulgaris—Betulinic Acid of. The authors obtained betulonic acid by the chromic acid oxidation of betulinic acid, which was isolated from the seeds of *Zyzyphus spinosi*. Betulonic acid, $C_{30}H_{46}O_3$, forms needles, m. 253°; it is readily soluble in organic solvents and yields a semicarbazone, $C_{31}H_{49}O_3N_2$, consisting of colorless needles, m. 282–283°. On catalytic reduction, it forms dihydrobetulonic acid, $C_{30}H_{48}O_3$, colorless needles, m. 256–257°. Both dihydrobetulin and dihydrobetulinic acid, on oxidation by chromic acid, give dihydrobetulonic acid.—RIITI KAWAGUCHI and K. W. KIM. *J. Pharm. Soc. Japan*, 60 (1940), *Transactions* (in English, 235–236). (N. L.)

Fixed Oils, Fats and Waxes

Ceylon Estate Copra—Analyses of. Analyses of copra averaged water 6.8, oil 63.7%. The extracted oil has iodine valence 8.16, saponification value 259.1 and free fatty acid (lauric) less than 0.1%.—R. CHILD. *Trop. Agr.*, 88 (1937), 137–149; through *J. Soc. Chem. Ind.*, 59 (1940), 372. (E. G. V.)

Chaulmoogra Oil—Purification and Esterification of. The oil from *Hydnocarpus wightiana* and *H. anthelmintica* is purified for injections by washing with sodium hydroxide, treatment with steam, and filtration; the product contains 0.2% of fatty acids. Ethyl chaulmoograte is obtained by boiling the oil with an equal volume of 95% ethyl alcohol and 4–12% of sulfuric acid for 8 hours. The crude ester is washed 4 times with hot water and distilled at 15–20 mm. pressure; the distillate (0.25–3% of fatty acids) is washed with sodium hydroxide until neutral, filtered and distilled in steam until the sharp odor disappears (2 hours). Up to 20% of the purified oil may be added to the ester.—H. I. COLE and H. CARDOSO. *Int. J. Leprosy*, 4 (1936), 455–466; through *J. Soc. Chem. Ind.*, 59 (1940), 403. (E. G. V.)

Fat Analysis—International Standard Methods of. Standard methods of German experts are given in detail for the determination of density, refraction, titer, water and volatile substances, impurities, ash, acidity, saponification value, unsaponifiable matter, oxidized acids, iodine value, polybromide value, OH value, preparation of insoluble fatty acids, sampling of hard soaps and their analysis for water, ethyl alcohol-insoluble foreign matter, total fatty acids, total alkali and free alkali.—H. P. KAUFMANN. *Fette u. Seifen*, 46 (1939), 499–517; through *J. Soc. Chem. Ind.*, 59 (1940), 372. (E. G. V.)

Fat Study—Dilatometer Measurement Method as a Useful Tool in. Readings obtained with the Salway dilatometer are readily reproducible and serve to differentiate unsaturated from saturated fats and to distinguish between these and hydrogenated fats. The more saturated fats and hydrogenated fats (containing *iso*oleic acid) show greater dilatations than those of the more unsaturated type. The dilatation curve of a hydrogenated fat showed a break at approximately the Wiley melting point of the fat.—C. A. COFFEY and H. T. SPANNUTH. *Oil and Soap*, 17 (1940), 41–42; through *J. Soc. Chem. Ind.*, 59 (1940), 462. (E. G. V.)

Fats—Thermostability of. The liberation of acid from beef and pork fat, coconut, palm, sesame, arachis, and olive oils, and triolein at 60–120° during 7 days is independent of the original free acid, but depends only on temperature and time. Each of the vegetable oils has a characteristic temperature below which no decomposition occurs. In the case of beef fat, the rate of decomposition is approximately constant from 60° to 90°, and in that of triolein from 75° to 105°. The Kreis test shows that aldehydes accumulate in the initial stages, but later disappear.—E. GLIMM, H. WITTMEYER and W. JAHN-HELD. *Z. Untersuch Lebensm.*, 78 (1939), 285–293; through *J. Soc. Chem. Ind.*, 59 (1940) 461. (E. G. V.)

Fatty Acids and Glycerols of Solid Seed Fats. IX. Mimusops Heckelii (Baku) Kernel Fat. *Minusops Heckelii* (Baku) fat contains as component acids: palmitic 4.4, stearic 36.0, arachidic 0.5, hexadecenoic 0.3, oleic 58.5 and linoleic 0.3% (wt.). The component glycerides of the fat, determined by study of three fractions of differing solubility obtained by crystallization from acetone, are approximately stearodiolein 41–47, oleodistearin 32–26, palmitodiolein 14–6, triolein 10–12, oleopalmitostearin 2–8 and fully-saturated (palmitodistearin) 1% (mol.).—D. ATHERTON and M. L. MEARA. *J. Soc. Chem. Ind.*, 59 (1940), 95–96. (E. G. V.)

Fusel Oil Values—Correlation of, by the Allen-Marquardt and Acetyl Chloride Methods. The Allen-Marquardt method (adopted as official by the A. O. A. C.) and a modification of the Schicktanz-Etienne acetyl chloride method (*Ind. Eng. Chem., Anal. Ed.*, 11 (1939), 390) (technique described in detail) were compared on a number of samples of whisky, Scotch and brandy. In the majority of instances the results obtained by the two methods on whisky were in good agreement; usually when check results were not obtained, the values by the official method were high, possibly because the fusel oil in these samples contained a large proportion of normal propyl and isobutyl alcohols, which contribute to the value by the official method but are removed during the dealcoholization in the acetyl chloride method. Likewise, the excellent agreement obtained in the majority of cases can be attributed to the fact that the fusel oil consists mainly of the higher boiling amyl alcohols and only a small percentage to the lower boiling alcohols such as normal propyl and isobutyl. In every case the acetyl chloride method gave much lower results on Scotch than the official method, the constancy of the variation making it possible to correlate the evaluation by means of a correction factor. As in the case of whisky, the difference between the results by the two methods on brandy was not constant and cannot be correlated by means of a correction factor. It would seem that the fusel oil (or more correctly the amyl alcohol) value obtained by the acetyl chloride method gives a true picture of the concentration of those alcohols which are responsible for the characteristic and essential taste and bouquet of distilled spirits, but this would require confirmation.—S. T. SCHICKTANZ, A. D. ETIENNE and J. L. YOUNG. *J. Assoc. Official Agr. Chem.*, 23 (1940), 368–373. (A. P.-C.)

Malt Extract with Cod Liver Oil—Oil Contents of. As extract of malt with cod liver oil is a B. P. article and this is required to be supplied for N. H. I. prescriptions, it seems reasonable that the official article should be supplied when extract of malt with cod liver oil is asked for under this or a similar name, except in the case of branded articles. Analytical results of samples sold in the retail trade showed

that the composition of the majority approximated to that stated in the B. P.; nineteen contained 10–11% by weight of oil, five 9–10%, four contained over 11% and two less than 9%, the extreme figures being 11.81% and 8.43%. It has been established that the low result obtained with the Gunn and Venables method is due to the almost quantitative solubility of the fatty acids in the alcohol used, the figure for extracted oil being that of neutral oil only. Determination of the fatty acid content of all the samples of extract of malt with cod liver oil was made. The significant fact emerging from the results of analysis is the fact that the acidity of the oil in extract of malt with cod liver oil increases, in some cases to an extraordinary degree. In the thirty samples examined the free acidity of the extracted oil, calculated as clupanodonic acid, may be grouped as follows: less than 2.5%, 6; 2.5–5.0%, 9; 5.0–7.5%, 5; 7.5–10.0%, 3; above 10.0%, 7. With development of free fatty acid an unpleasant after-taste in the throat was noticeable, being most pronounced in the worst samples. The authors find that a surprisingly large proportion of these samples have developed excessive acidity. They recommend that extract of malt with cod liver oil sold under this or a similar name without qualification should be that of the B. P., and suggest tentatively a limit of 5% acidity in the oil.—D. C. GARRATT and J. E. WOODHEAD. *Chemist and Druggist*, 134 (1941), 267. (A. C. DeD.)

Mineral Oil—Detection of, in Crude and Refined, Pressed Olive Oil. The olive oil, saponified with 15% potassium hydroxide in ethyl alcohol and heated on a water bath, yields clear solutions if greater than 20% of mineral oil (I) is present; if less than 20% of I is present, it is detected by the formation of a turbidity on addition of 70% ethyl alcohol at higher than 20°. Adulteration of almond oil with I is similarly detected.—S. ANSELMI. *R. Ist. San. Pubbl.*, 2 (1939), 979–983; through *J. Soc. Chem. Ind.*, 59 (1940), 463. (E. G. V.)

Olive Oil—Analysis of. The oil is treated with acetic anhydride-sulfuric acid; a red color (due to resinous matter) indicates addition of olive-husk oil (I). I is also indicated by the Bellier test for arachis oil, while refined I is detected by a positive test for carbon disulfide (used as solvent) by the copper xanthate reaction.—S. FACHINI and G. B. MARTINENGHI. *Olii min. grassi*, 19 (1939), 86–88; through *J. Soc. Chem. Ind.*, 59 (1940), 374. (E. G. V.)

Olive Oil and Fluorescence. Genuine fresh olive oils give a yellow-orange fluorescence in ultraviolet light. Refined husk oils and genuine oils that have been stored for a long time give a blue fluorescence.—G. LUCENTE. *Atti X Congr. Internaz. Chim.*, IV (1938), 594–599; through *J. Soc. Chem. Ind.*, 59 (1940), 545. (E. G. V.)

Olive Oil—Susceptibility of, to Autoxidation. The antioxidants naturally present in cod liver oil and olive oil (I) are removed by adsorption on carbon or activated silicon dioxide. Curves are given for the oxygen uptake (Barcroft-Warburg technique) of I at 40°, 60°, 70° and 90°; that at 90° indicates the occurrence of an induction period.—W. CIUSA. *Ann. chim. applicata*, 30 (1940), 141–146; through *J. Soc. Chem. Ind.*, 59 (1940), 463. (E. G. V.)

Unclassified

Aliphatic Acids—Color Reactions of Some. It has been found that alkali citrates heated in acetic anhydride give deep red colored solutions. Probably an alkali acetate and a mixed anhydride of acetylated citric and acetic acid is the first phase; then the alkali acetate brings about the condensation of the mixed anhydride into the colored compound. The same color reaction occurs when alkali salts of any organic acid are added instead of the alkali acetate. Shades of color depend upon temperature, heating time, quantity of added base and upon presence of other solvents mixed with acetic anhydride. Experimental work includes malonic acid, aconitic acid, citric acid, cetyl citric acid, tartaric acid, acetonedicarbonic acid, ascorbic acid, *d*-isoascorbic acid, glucono-*d*-lactone, glucoheptonic lactone and hydroxy-dimethylbutyrolactone.—GEORGE ROEDER. *Jour. A. Ph. A.*, 30 (1941), 74. (Z. M. C.)

1-Alkyl-5-Methylbarbituric Acid Compounds. Barbituric acid derivatives generally suitable for therapeutic use are prepared, having the general formula $CO.NX.CO.C(CH_3)R'CO.N$-alkyl (where R' is a cyclopentyl, cyclohexyl or cyclohexylalkyl group, alkyl is a saturated or unsaturated alkyl group and X is hydrogen or an alkali or alkaline earth metal). The compounds are whitish crystalline products, insoluble in water in the form of the free acid but soluble in the form of the alkali metal salts. Production of various of these compounds is described.—WALTER KROPP and LUDWIG TAUB, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,206,779, July 2, 1940. (A. P.-C.)

Amines and Their Salts—Manufacture of. In the preparation of β-(4-hydroxy-4-methoxyphenyl)-isopropylamine, an aryl ether of 4:3:1—OH.-C_6H_3(OMe).CHO is first condensed with an α-halogenopropionic ester. 4-Benzyloxy-3-methoxyphenylacetone, melting point 58° (from vanillin benzyl ether and ethyl α-brom propionate), β-(4-benzyloxy-3-methoxyphenyl)isopropylamine, melting point 57–58°, and β-(4-hydroxy-3-methoxyphenyl)isopropylamine hydrochloride, melting 254–255° (decomposition), are described. The products are useful medicinally.—R. ROBINSON, A. LOWE and IMPERIAL CHEM. INDUSTRIES, LTD. Brit. pat. 519,894; through *J. Soc. Chem. Ind.*, 59 (1940), 515. (E. G. V.)

***p*-Aminophenylsulfonacetamide and Related Compounds.** Reaction of *p*-aminobenzenesulfinic acid with chloracetamide in alcohol or water as solvent gave *p*-aminophenylsulfonacetamide, $NH_2C_6H_4SO_2.CH_2CONH_2$. Unlike acetylsulfanilamide or Albucid (N-(*p*-aminobenzenesulfonyl)-acetamide), the compound had practically no chemotherapeutic activity. The m. p. was 225° C., a white crystalline powder, difficultly soluble in cold water, alcohol or benzene, soluble in HCl solution, although the amino group had no acid character. Using α-brompropionic acid amide in place of chloracetamide in a similar reaction, *p*-aminophenylsulfon-α-methylacetamide was obtained; m. p. 179–180° C., soluble in water. With α-chlorphenylacetamide there was obtained *p*-aminophenylsulfon-α-phenylacetamide; decomp. at 300° C., very insoluble in all ordinary solvents. When *p*-aminophenylmercaptan was reacted with chloracetamide in alkaline solution, S-(*p*-aminophenyl)-thioglycollic acid amide was obtained; m. p. 112–113° C., white crystalline powder, difficultly soluble in water, very soluble in alcohol. When *p*-nitrobenzenesulfonamide was boiled 1/2 hour in acetic anhydride, acetyl-*p*-nitrobenzenesulfonamide was formed; m. p. 192–193° C., soluble in alcohol. On catalytic hydrogenation (Raney nickel catalyst), this was converted to N-(*p*-aminobenzenesulfonyl)-acetamide; m. p. 182° C., easily soluble in hot water and alcohol. Similiarly heating the corresponding *m*-nitro compound with acetic anhydride gave acetyl-*m*-nitrobenzenesulfonamide; m. p. 190–191° C. Catalytic hydrogenation of this gave N-(*m*-aminobenzenesulfonyl)-acetamide; m. p. 156–157° C., soluble like the para deriv-

ative.—K. A. JENSEN and F. LUNDQUIST. *Dansk Tids. Farm.*, 14 (1940), 129. (C. S. L.)

Ascorbic Acid Series—Synthesis of Compounds of the. A process of synthesizing compounds of the ascorbic acid series, such as glucoascorbic acid, involves acting upon a solution of a sugar such as glucose with a slightly acidic cupric salt of an organic acid, such as the acetate in the presence of a material such as calcium carbonate for regulating the pH, adding a copper precipitant such as oxalic acid, removing the precipitate formed, treating the solution with a cyanide such as sodium cyanide, recovering the resulting imino compound in crystalline form and hydrolyzing it to produce the corresponding ascorbic acid. Various similar reactions are described or mentioned.—IRWIN STONE, assignor to WALLERSTEIN CO. U. S. pat. 2,206,374, July 2, 1940. (A. P.-C.)

Barbituric Acid—Ultraviolet Absorption Spectra of, and Its 1-Methyl and 1,3-Dimethyl Derivatives. The following summary is given: (1) The ultraviolet absorption spectra of 1-methylbarbituric acid, 1,3-dimethylbarbituric acid and barbituric acid itself have been examined in acid and alkaline solution and at varying dilutions in water. (2) The similarities shown in the spectra of all three compounds, and in particular the almost identical value of $\epsilon_{max.}$ at *ca.* 2600 Å. in alkaline solution, suggest that barbituric acid in aqueous solution undergoes only one enolization, involving the active methylene group in position 5.—R. E. STUCKEY. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 312–317. (S. W. G.)

Barbituric Acids. Hypnotic compounds of the general formula $CH_2(CH_2)_nCH{:}CYCH.CR.CO.NR'.CX.NM.CO$ (where n represents one of the numerals 1 and 2, R represents a member of the class consisting of alkyl, alkenyl, monocyclic aryl and monocyclic aralkyl, R' represents a member of the class consisting of hydrogen, alkyl and alkenyl, X represents a member of the class consisting of oxygen and sulfur, M is hydrogen, an alkali metal, an alkaline earth metal or an organic ammonium radical, and Y represents a halogen) are prepared by treating a substituted malonic acid derivative of the general formula $CH_2(CH_2)_nCH{:}CYCHCR(COZ)_2$ (where n, R and Y have the designated meaning and Z is an alkoxy group) with a urea of the formula $R'NHCXNH_2$, in which R' and X have the designated meanings. Details are given of the production of a number of such compounds.—WALTER G. CHRISTIANSEN, assignor to E. R. SQUIBB AND SONS. U. S. pat. 2,187,728, Jan. 23, 1940. (A. P.-C.)

Butyrolactones and Primary Amines of the Aromatic and Heterocyclic Series—Condensation Products of. New compounds, useful, *e. g.*, in combating malaria parasites, are obtainable by condensation of butyrolactones which contain a keto group only attached to the lactone ring at the carbon atom standing in alpha-position to the carbonyl group of the lactone ring, with primary amines of the aromatic or heterocyclic series while splitting off water. Thus, compounds of the type of Schiff's bases are obtained having the general formula $RR'C{:}NR''$, where R stands for an alkyl, aryl or aralkyl radical (such as methyl, ethyl, propyl, butyl, phenyl and benzyl), R' stands for the butyrolactone radical or for a substituted butyrolactone radical (such as the beta-methyl, beta-ethyl and beta-hydroxymethylbutyrolactone radical, which butyrolactone radical is bound through its alpha-carbon atom), and R'' stands for a radical of the aromatic, particularly the benzene and naphthalene, series or the heterocyclic series (for instance, the phenyl, naphthyl, pyrazolyl, pyridyl, quinolyl, isoquinolyl and acridyl radical, and such radicals as are substituted by halogen, alkyl, hydroxyl, alkoxy, amino, acylamino, alkylamino, alkylaminoalkylamino, alkylaminoalkoxy, carboxyl, etherified and esterifies carboxyl and similar groups). The new products when heated with dilute inorganic acids are split into one molecule of the amine used for the condensation, one molecule of carbon dioxide and one molecule of a keto alcohol. Numerous examples with details are given.—HANS ANDERSAG, assignor to WINTHROP CHEMICAL CO. U. S. pat. 2,187,847, Jan. 23, 1940. (A. P.-C.)

Catalyst for Hydrogenation of Organic Compounds. A double copper-ammonium chromate is prepared, preferably on an inert carrier, the product is heated until spontaneous decomposition starts (450°), and when reaction is complete soluble impurities are removed by extraction with aqueous ammonia, leaving copper chromate on the carrier.—P. L. SALZBURG, assignor to E. I. DU PONT DE NEMOURS AND CO. U. S. pat. 2,089,433; through *J. Soc. Chem. Ind.*, 59 (1940), 443. (E. G. V.)

Emulsion Polymerization. A review.—H. BARRON. *Brit. Plastics*, 11 (1940), 464–467; through *J. Soc. Chem. Ind.*, 59 (1940), 466. (E. G. V.)

Heterocyclic Compounds—Preparation of Therapeutically Useful. *p*-Aminobenzenesulfonamido-derivatives of glyoxaline (I) are prepared by interaction of an amino-derivative of I and a *p*-acylaminobenzenesulfonyl halide and hydrolysis of the product. Alkyl, aryl and aralkyl derivatives are prepared by treatment before or after hydrolysis with alkyl, etc., halides or sulfates. Thep reparation of 2-*p*-aminobenzenesulfonamido-, melting point 259° and 2-*p*-aminobenzenesulfonmethylamido-3-methyl-glyoxaline, melting point 227–228°, is described.—A. J. EWINS, and J. N. ASHLEY. Brit. pat. 521,821; through *J. Soc. Chem. Ind.*, 59 (1940), 642. (E. G. V.)

High Molecular Weight Primary Amines—Preparation and Properties of. The boiling of C_{6-18} primary amines (prepared by hydrogenation of the corresponding fatty acid nitriles) are given and solubility phenomena of binary mixtures of the individual amines with water have been studied. Uses of these amines as wetting and flotation agents, sterilizing media, and for the flocculation of suspended particles in turbid waters are mentioned.—A. W. RALSTON. *Oil and Soap*, 17 (1940), 89–91; through *J. Soc. Chem. Ind.*, 59 (1940), 513. (E. G. V.)

Lævo-Piperitone. Some physical constants of piperitone, together with those of menthones and methols prepared during work on the hydrogenation of the pure ketone, are included in tabular form.—ANON. *Perfumer. Essent. Oil Record*, 32 (1941), 172. (A. C. DeD.)

2 - Methyl - 1,4 - Naphthohydroquinone Diphosphoric Acid Ester—Sodium Salt of. It has been found that this antihemorrhagic salt is effective in doses of 0.6 to 0.8 gamma per chick administered subcutaneously, while for oral effectiveness the dose is below 2 gamma. The lethal dose in mice both by subcutaneous and intravenous administration is approximately 450 mg./Kg. All preliminary tests indicate an enormous margin of safety and absence of serious side reactions in any but the highest doses.—R. H. K. FOSTER, J. LEE and U. V. SOLMSSEN. *J. Amer. Chem. Soc.*, 62 (1940), 453–454. (E. B. S.)

Plastics—Modern Developments in. Developments in plastics are discussed. Plastic containers in color, texture, presentation and cheapness offer many advantages, particularly where mass produc-

tion is practicable.—V. E. YARSLEY. *Chemist and Druggist*, 134 (1941), 374. (A. C. DeD.)

Steroids—Manufacture of Carbonyl Compounds of. Steroids containing $CH_2.CO$ interact with nitrous acid, alkyl nitrites or aromatic NO-compounds and the resultant oximes, oximinio-compounds or anils are converted into α-dicarbonyl or α-ketocarboxylic derivatives. In the examples, Δ^5-pregnen-3-ol-20-one (I) (3.15) is added to ethyl alcohol containing sodium (0.23) followed by $C_5H_{11}\cdot O.NO$ and the mixture kept for some days; the precipitated sodium salt is decomposed with acetic acid, an aqueous solution of sodium nitrite (0.7 part) is added, and the whole poured into water, from which ether extracts Δ^5-20-glyoxylpregnen-3-ol (II), crystallized in two forms, melting point 140° and 172°. II is also formed from I by interaction with p-$NO\text{-}C_6H_4.NMe_2$.—*Soc. Chem. Ind. in Basle.* Brit. pat. 521,109; through *J. Soc. Chem. Ind.*, 59 (1940), 641. (E. G. V.)

Sulfanilamide Derivatives of Heterocyclic Amines. I. Quinoline Derivatives. By reaction of p-acetaminobenzenesulfonchloride with seven isomeric aminoquinolines, followed by hydrolysis of the condensation products, seven sulfanilaminoquinolines: $NH_2C_6H_4SO_2NH.C_9H_6N$, were obtained. All had similiar potency of action on pneumococci to that of sulfapyridine. The amino groups were in the 2,3,4,5,6,7 and 8 positions on the quinoline, respectively. Melting points of the sulfanilaminoquinolines: 2, 198° C.; 3, 185° C.; 4, 248° C.; 5, 230° C.; 6, 298° C.; 7, 206° C.; 8, 195° C.; of 6-methoxy-8-sulfanilaminoquinoline, 195–196° C. Preparation method: 0.1 mole of the aminoquinoline was dissolved in 25 cc. dry pyridine, and, portionwise, 0.1 mole of pure, dry, p-acetaminobenzenesulfonchloride was added. The solution was heated on the steam bath $^1/_2$ hour, then poured into water. The precipitated acetyl compound could be used directly, or recrystallized from alcohol. It was hydrolyzed usually by boiling 1 hour in 2*N* NaOH, but, as the sodium salts of the 8-aminoquinoline and the 6-methoxy-8-aminoquinoline derivatives were very insoluble, here hydrolysis was conducted in 4*N* HCl. In either case, on neutralization, the sulfanyl derivative was precipitated, and purified by recrystallization from alcohol, alcohol and water (1:1), or acetone.—K. A. JENSEN and F. LUNDQUIST. *Dansk Tids. Farm.*, 14 (1940), 208. (C. S. L.)

Sulfonamide Compounds—Manufacture of. Readily soluble aromatic polyhydroxy-alkylaminosulfonamides are obtained by heating approximately equimolecular quantities of an aromatic aminosulfonamide, *e. g.*, sulfanilamide (I), and an aldose having an ether linking between two carbons other than $carbon_1$ and $carbon_4$, or a polyose, derived from such aldoses, preferably in a water-miscible solvent, *e. g.*, a lower aliphatic alcohol, or a dioxan, which may contain a little water, filtering the solution, and recovering the products by evaporation of the solvent. Alternatively, the reaction may be carried out under pressure or in presence of a small amount of an organic, alcohol-miscible solvent, *e. g.*, glycerol, of boiling point greater than the reaction temperature. The preparation of compounds from I and galactose, lactose and xylose is described.—F. MEYER. Brit. pat. 519,661; through *J. Soc. Chem. Ind.*, 59 (1940), 566. (E. G. V.)

Vitamin K-Active Derivatives of 2-Methyl-1,4-Naphthohydroquinone. The preparation of esters of acetic, propionic, butyric, benzoic, iso-butyric, valeric and iso-valeric acids and a dimethoxy ether with 2-methyl-1,4-naphthohydroquinone is described. The various derivatives were found to have different vitamin K potencies. A discussion of the relationship between structure and vitamin K activity is presented.—S. ANSBACHER, E. FERNHOLZ and M. A. DOLLIVER. *J. Am. Chem. Soc.*, 62 (1940), 155–158. (E. B. S.)

BIOCHEMISTRY

Acetone Bodies in Blood and Urine—Modified Salicylaldehyde Method for the Determination of. A modified salicylaldehyde method for the determination of acetone in distillates from urine or blood filtrate is described. The reaction takes place in a concentrated mixture of the reacting substances without application of heat and is complete in 20 minutes. The precipitate which forms is dissolved either in water or alcohol. The range and sensitivity of the method are somewhat increased over that of previous salicylaldehyde methods. Either a visual or photoelectric colorimeter can be used. An almost exact proportionality exists between acetone concentration and scale reading in a visual colorimeter. Artificial color standards for visual colorimetry are also described. Procedures for the oxidation of β-hydroxybutyric acid and the distillation of acetone have been slightly modified. The specificity of the reaction is briefly discussed.—JEANETTE ALLEN BEHRE. *J. Biol. Chem.*, 136 (1940), 25. (F. J. S.)

"Acid" Phosphatase Activity of Blood Serum—Estimation of. Optimal conditions of hydrolysis in the estimation of serum "acid" phosphatases were determined. The King and Armstrong method for "alkaline" phosphatases was adapted to the estimation of serum "acid" phosphatases.—ETHEL BENEDICT GUTMAN and ALEXANDER B. GUTMAN. *J. Biol. Chem.*, 136 (1940), 201. (F. J. S.)

Alanine—Improved Method for the Resolution of Synthetic. An improved method of resolution of synthetic alanine into the active components is given. The yields are much higher than any previously reported and the time and labor of preparation are also considerably cut down. Racemization studies show that active alanine is perfectly stable to boiling 20 per cent hydrochloric acid, whereas benzoylation does attack the asymmetric center. The specific rotation of the benzoylalanine is a function of the concentration.—EUGENE PACSU and JAMES W. MULLEN, 2ND. *J. Biol. Chem.*, 136 (1940), 335. (F. J. S.)

Alkapton—Micromethod for the Determination of, in Urine. Alkapton in alkaline solution reduces silver nitrate to silver oxide, which is redissolved in nitric acid and titrated with ammonium thiocyanate using ferric ammonium alum as indicator. To 1 cc. of urine in a centrifuge tube add 5 cc. of a solution made up of 50 cc. of 20% silver nitrate solution, 40 cc. of concentrated ammonia water and 10 cc. of oxalic acid; after 5 minutes add 5 drops of 10% calcium chloride solution; in the presence of alkapton silver oxide is formed, which is carried down by the calcium oxalate also formed; centrifuge the solution, carefully remove the liquid with a pipette, redissolve the silver oxide and titrate the solution with decinormal ammonium thiocyanate solution. Analyses of aliquot portions of a solution indicate that results to within 0.5% are obtained on a 1 to 2-cc. sample of urine.—V. FIGURA. *Atti R. Acad. Lincei*, 29 (1939), 329–332; through *Chimie & Industrie*, 43 (1940), 17. (A. P.-C.)

Aminemia—Estimation of. Colorimetric Determination of Histamine, Tyramine and Tryptamine Values of Blood Serum. Maciag and Schoental's modification of the Pauly test for histamine (imidazole bodies), Lesure and Thomas' method for tyramine, and their method for tryptamine were used. In each case they give an "index" or "value,"

not a measure of the pure compound. For the first two, determinations on close to 500 blood serums gave: in normal subjects, 2 to 6 mg. of tyramine and 0 to 2 mg. of histamine per liter, in pathological cases 10 to 20 mg. of tyramine and 4 to 8 mg. of histamine per liter. The tryptamine value for normal subjects varied from 0 to trace, *i. e.*, the value was apparently smaller than the limit of sensitivity of the method. In various bacterial cultures, the tyramine and histamine values were higher than in pathological cases.—ANDRÉ LESURE. *J. pharm. chim.*, [9], 1 (1940), 55–69. (A. P.-C.)

Aneurin—Thiochrome Method for Estimation of. The examination of thiochrome methods put forward for the assay of aneurin is recorded, and these methods have been suitably modified and combined to provide increased accuracy in respect of foodstuffs. The method thus evolved is described and then applied to the measurement of the aneurin content of a large number of cereals.—R. G. BOOTH. *J. Soc. Chem. Ind.*, 59 (1940), 181–184. (E. G. V.)

Ascorbic Acid from Sorbose. An aqueous solution of sorbose is treated with oxygen at pH 6 to 11, and the oxidation product is treated with concentrated hydrochloric acid at room temperature to 80° C. – OTTO DALMER and KURT HEYNS, assignors to MERCK & CO. U. S. pat. 2,189,778, Feb. 13, 1940. (A. P.-C.)

Ascorbic Acid—Oxidation of, by Hemoglobin. Ascorbic acid added to blood is partially oxidized during defecation and passes into the filtrate. Oxidation is not due to oxyhemoglobin proper; on addition of the defecating agent it is converted into hematin and it is the liberated oxygen that oxidizes the ascorbic acid. If the oxygen is removed from the oxyhemoglobin by means of nitrogen, hydrogen or especially carbon monoxide, the vitamin is not oxidized, which permits of determining ascorbic acid in media rich in hemoglobin.—A. FUJITA, T. EBIHARA and I. NUMATA. *Biochem. Z.*, 301 (1939), 245–252; through *Chimie & Industrie*, 43 (1940), 758. (A. P.-C.)

Bile Salts—Composition and Analysis of. Methods of analysis of commercial bile salts were investigated. The following procedure for the determination of total mixed acids is given: Heat 0.5 Gm. of sample with 30 cc. of 15% sodium hydroxide solution under a reflux condenser for twelve hours. If excessive frothing occurs add about one cc. of alcohol or ether. Add 30 cc. of water and filter through a No. 54 Whatman filter paper into a 250-cc. separator and wash the flask and filter paper thoroughly with hot water. Acidify the contents of the separator with diluted sulfuric acid, cool and extract with four separate quantities of 50 cc. of ether. Bulk the ethereal extracts and wash with two separate portions of 10 cc. of water. Filter into a tared flask and wash the filter paper with ether. Evaporate off the ether, dry the residue at 100° and weigh. The acid value and specific rotation of the acids should be determined. The approximate composition of the salts may be calculated from a determination of total sulfur and the total mixed acids. Both sodium tauroglycocholate and sodium glycocholate should contain not less than 70% of total mixed acids after hydrolysis. The acid value of these acids should not be greater than 145. Sodium glycocholate should contain not more than 0.8% of total sulfur.—N. EVERS and W. SMITH. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 213–218. (S. W. G.)

Bilirubin—Method for the Estimation of, in Whole Blood and in Plasma and Serum Containing Hemoglobin. Saturate a 5-cc. sample of whole blood plasma or serum with carbon monoxide and add 0.5 cc. of twentieth-molar ascorbic acid; dilute the mixture to 50 cc. with purified acetone and filter; measure the absorption of the filtrate with a Pulfrich photometer, using Filter S 57; mix 20 cc. of the filtrate with 2 cc. of fresh diazo reagent (10 cc. of a solution containing 5 Gm. of sulfanilic acid, 50 cc. of 37% hydrochloric acid per liter and 0.1 cc. of 0.5% sodium nitrite); after 10 minutes add 1 cc. of 37% hydrochloric acid and acetone to 25 cc.; read the solution again in the photometer. The average error is 0.037 mg. per 100 cc. Precipitation of the hemoglobin as the carbonyl compound and with acetone causes less adsorption on the precipitate than the oxygenated compound precipitated with ethanol. The ascorbic acid probably reacts with excess diazo compound.—M. ENGEL. *Hoppe-Seyler's Z. physiol. Chem.*, 259 (1939), 75–82; through *Chimie & Industrie*, 42 (1939), 965. (A. P.-C.)

Bilirubin—Preparation of. Bile is treated with an alkaline-earth hydroxide. The precipitated salts are dried and treated with acetic acid and a chlorinated aromatic hydrocarbon (phenyl chloride); bile acids remain undissolved. Evaporation (vacuum) gives crude bilirubin, which is purified by ethyl alcohol.—ARMOUR and Co. Brit. pat. 518,-637; through *J. Soc. Chem. Ind.*, 59 (1940), 495. (E. G. V.)

Bilirubin—Stabilization and Determination of, in Duodenal Fluid. The addition of 7.5 mg. of ascorbic acid per 100 cc. of alkaline solution of bilirubin prevents destruction by the alkali and stabilizes the color, thus facilitating the colorimetric or photometric determination. Formaldehyde, sodium hyposulfite and sodium thiosulfate, used in the same manner, exert no protective action.—M. G. BARAC. *Bull. soc. chim. biol.*, 21 (1939), 1163–1170; through *Chimie & Industrie*, 43 (1940), 461. (A. P.-C.)

Blood—Determination of the pH of the. After having determined the pH value in veinous peripheric blood and in myeloid blood, and other hematological data, the authors admit that a certain relation exists between the pH value and the eosinophile level in bone marrow.—A. BARASCIUTTI and M. FRANCESON. *Biochem. terap. sper.*, 28 (1940), 207. (A. C. DeD.)

Blood—Fresh and Stored, Transfusion of. A study has been made of the effect of fresh and stored blood in cases in which the clinicians in charge considered that blood transfusion was indicated. The six clinical observers attached to the four blood depots who were engaged in this investigation are unanimously agreed from a consideration of the clinical results that stored blood is as good as fresh blood in the treatment of acute hemorrhage. They are not so certain that stored blood is as valuable as fresh blood in the treatment of non-acute hemorrhage but at the same time the available data present no evidence that stored blood is in fact of less value to such cases. Analysis of the figures regarding gain in hemoglobin shows that in many cases stored blood will certainly give a rise in hemoglobin as great as that produced by fresh blood, but on the average in both the acute and non-acute hemorrhage cases the gain was greater with fresh than with stored blood. (In the miscellaneous cases no difference in the average gain was noted.) More extensive figures than those available here will be necessary before any final conclusion can be reached. Mild reactions, excluding rises in temperature, occurred more commonly following stored than following fresh blood. The numbers of severe rigors observed after the use of warm, fresh and stored blood were practically identical (4.7% and 5.4%). In a total of 153 cases analyzed in this investigation no severe hemolytic reactions were found and no fatal

result attributable to transfusion occurred. With regard to rise in temperature following transfusion, cases of non-acute hemorrhage are the most informative and these reveal no material difference between fresh and stored blood administered either warm or cold. From a study of the occurrence of reactions it is considered that there is an advantage in warming the blood to approximately 37° C. before administration. Analysis of the figures available reveals no association between either the amount of blood given and the risk of reactions or the rate of administration and the risk of reactions. The experience is, however, insufficient to give a decisive answer.—H. F. BREWER, M. MAIZELS, J. O. OLIVER and J. VAUGHAN. *Brit. Med. J.*, 4149 (1940), 48. (W. H. H.)

Blood in Urine—Color Reaction for the Detection of. The reaction is based on the liberation of oxygen by hemoglobin in contact with hydrogen peroxide and on the formation of the formate of dimethylamino-*p*-quinone di-imine by oxidation of *p*-phenylenediamine and dimethyl amine in acid medium. In a test-tube pour 1 cc. of alcoholic 1% *p*-phenylenediamine, 1 cc. of 1.6% alcoholic solution of dimethyl amine, 5 cc. of 27% formic acid and 3 drops of 6% hydrogen peroxide; add 3 cc. of urine, shake and let stand; a green color indicates the presence of blood. The color is produced with 3 drops of a solution of 0.5 cc. of blood in 2000 cc. of physiological salt solution.—V. INDACOCHEA ZARAUZ. *Prim. Congr. Peruano Quim. (Actas y Trab.)* (1938), 729–733; through *Chimie & Industrie*, 43 (1940), 642. (A. P.-C.)

Blood Iodine—Nature of. II. Nature of the Plasma Iodine. The plasma iodine is divided between the albumin and the globulin fractions of the plasma in proportion to the relative amounts of albumin and globulin present. There is no chemical evidence for the presence of a preponderant amount of thyroglobulin in the circulating blood.—SOLOMON SILVER. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 213. (A. E. M.)

Blood Plasma—Problems Connected with the Storage, Drying and Filtering of. The authors described how plasma is transferred from its original container to a second container. Methods of drying were described.—J. P. TODD, G. R. MILNE and G. RATTRAY. *Chemist and Druggist*, 134 (1941), 336. (A. C. DeD.)

Blood Sugar Determination—Applicability of the Huzita-Iwatake Method for. The author uses smaller amounts of the reagents and a thousandth-normal sodium thiosulfate solution for the titration. The filter paper, in which blood is absorbed, should be boiled with the ferricyanide reagent, washed several times with boiling water and dried shortly before using. By this modified procedure accurate determinations of sugar can be made on quantities of 5 to 50 mg. The procedure was used for determining the sugar content of the body fluids from beetles.—W. SZEKESSY. *Biochem. Z.*, 303 (1940), 364–367; through *Chimie & Industrie*, 43 (1940), 812. (A. P.-C.)

Blood Sugar—Photometric Determination of, by the Folin and Wu Method. The errors due to fading of colors and the relation between the concentration of glucose and the intensity of color are discussed. The Folin and Wu technique was modified by heating the solution in boiling water after the addition of alkaline copper and again after the addition of tungstophosphomolybdate solution. This stabilized the color and allowed the direct reading in a photometer without a standard solution for comparison.—M. FIORENTINO and G. GIANNATTASIO. *Diagnostica tec. lab.*, 10 (1939), 401–412; through *Chimie & Industrie*, 43 (1940), 17. (A. P.-C.)

Chlorine in Blood—Microdetermination of. The method is based on the destruction of the chlorine-albumin complex by means of nitric acid and potassium permanganate. To 0.2 to 0.5 cc. of plasma. erythrocytes or total blood in a test-tube add 2 cc, of silver nitrate (4.791 Gm. per liter) and after 5 minutes 1 cc. of nitric acid, boil, add potassium permanganate solution drop by drop, add a small quantity of anhydrous glucose and heat to complete decolorization; add 0.1 cc. of 20% ferric alum solution and titrate to a persistent pink with ammonium thiocyanate solution standardized against silver nitrate so that 1 cc. = 1 mg. Cl.—M. T. A. TAVERA. *Prim. Congr. Peruano Quim. (Actas y Trab.)*, (1938), 691–693; through *Chimie & Industrie*, 43 (1940), 552. (A. P.-C.)

Cholesterol Determinations—Studies on the Source of Error in Photometric. It is pointed out that the extraction of the plasma cholesterol must be made with no less than a 10-fold volume of 3:1 alcohol-ether mixture. The saponification of cholesterol esters cannot be done even with strong aqueous solution of potassium or sodium hydroxide but proceeds to completion with 5% sodium ethylate. The Liebermann-Burchard color reaction follows Lambert-Beer's law.—B. C. NOYONS and M. K. POLANS. *Biochem. Z.*, 303 (1940), 415–424; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Cholesterol—Production of. Animal tissue containing cholesterol such as minced beef spinal cord, is treated with slaked lime or other alkaline earth metal oxide or hydroxide (suitably with heating for about 3 hours at 100° to 125° C.), and the reaction mixture is treated with a solvent for cholesterol, such as ethylene dichloride or *o*-dichlorobenzene. Various examples with details are given.—JULES D. PORSCHE and FRED J. SOLMS, assignors to ARMOUR & Co. U. S. pat. 2,191,260, Feb. 20, 1940. (A. P.-C.)

Cholesterol—Technic for the Determination of, by Chromate Oxidation. For the determination of sterol in animal tissue it is best to precipitate it with digitonin. Various attempts were made to measure the reducing power of the precipitate. It was found possible to completely oxidize to carbon dioxide and water by means of potassium dichromate and sulfuric acid in presence of silver nitrate as catalyst. The excess of dichromate is then determined iodometrically. One cc. of normal potassium dichromate = 0.3938 mg. of cholesterol or 0.254 mg. of the complex digitonin-cholesterol compound.—F. KAYSER and C. MATHIEU. *Bull. Soc. Chim. France*, 6 (1939), 715–717; through *Chimie & Industrie*, 42 (1939), 1032. (A. P.-C.)

Chlorine Content of Mother's Urine and Arakawa's Reaction. Urine of Arakawa-negative mothers is essentially lower in chlorine content than that of Arakawa-positive mothers. Chlorine content in the urine of Arakawa-negative mothers shows an average figure of −34% deviation as compared with that of Arakawa-positive mothers. Viewed from the chlorine content of milk (Nozaki and Ishii), blood (Ishii) and urine (author), it is not difficult to presume that there is a tendency to abnormal chlorine retention in Arakawa-negative mothers.—H. UMEMURA. *Tôhoku J. Exptl. Med.*, 39 (1940), 103. (A. C. DeD.)

Cholesterol-Fed Rats—Middle and Old Age in. Rats have been fed diets containing 1% cholesterol from weaning throughout life. Their growth, health and longevity have not differed significantly from those of control animals receiving the same diet without cholesterol.—RUTH OKEY. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 466. (A. E. M.)

Copper—Determination of, in Blood Serum. A colorimetric method is described consisting in con-

verting the copper into the diethyldithiocarbamate (by means of an alcohol solution of the corresponding sodium salt) and measuring the color produced by means of a photometer. To ensure total ionization of the copper, it is necessary to add 6 times normal hydrochloric acid (2 cc.) to the serum (4 cc.) before shaking it with trichloroacetic acid.—H. G. SCHMIDT. *Biochem. Z.*, 302 (1939), 256–261; through *Chimie & Industrie*, 43 (1940), 812. (A. P.-C.)

Coproporphyrin—New Method of Determining. The ultraviolet spectrum of coproporphyrin in solution in 5% hydrochloric acid shows a very sharp absorption maximum at 400 Å. The width of this band is proportional to the concentration of the product, which permits of preparing a scale of standard spectra; and by comparing the spectrum of the unknown its coproporphyrin content can be obtained.—GENIVIÈVE GLOTZ. *Compt. rend. soc. biol.*, 132 (1939), 194–195; through *Chimie & Industrie*, 43 (1940), 461. (A. P.-C.)

Creatinine and Creatine—Determination of. I. A study of the conditions required to obtain satisfactory results by Jaffe's reaction with solutions of pure creatinine: the chemicals used must be exceptionally pure and commercial creatinine must be subjected to thorough purification in order to calibrate the colorimeter. A method of absorption of pure creatinine by means of clay was developed for urine; the results are lower than by direct determination. Precipitation by phosphotungstic acid gives even lower results; this acid eliminates practically nothing but the creatinine, and the other chromogens of urine remain practically unprecipitated. Langley and Evans' 3,5-dinitrobenzoic acid method is not suitable for clinical use.—E. C. NOYONS. *Chem. Weekblad*, 36 (1939), 63–68; through *Chimie & Industrie*, 42 (1939), 964. (A. P.-C.)

Creatinine—New Colorimetric Microdetermination of, and Its Application to Plasma and Serum. Mix 5 cc. of plasma or serum with 5 cc. of water, add 5 cc. of 20% trichloroacetic acid solution and filter. Take 10 cc. of filtrate, neutralize with sodium hydroxide, add 10 cc. of thirtieth-molar phosphate buffer of pH 6.9 and pass the mixture through a column containing a mixture of 10 Gm. alumina, 0.5 Gm. of medicinal charcoal and 0.5 Gm. of talc. This adsorbs arginine and creatinine. Wash with the phosphate buffer, then elute with 5 cc. of aqueous pyridine solution, which elutes only creatinine. To the eluate add 1 cc. of 10% sodium hydroxide solution and 0.1 Gm. of yellow mercuric oxide and heat at 100° C. for 5 minutes, thus oxidizing the creatinine to oxalic acid and methylguanidine. Dilute to 6 cc., filter, take 4 cc., add 1 cc. of 0.02% α-naphthol solution, cool in an ice bath for 5 minutes and add 0.2 cc. of a hypobromite solution obtained by adding 2 Gm. of bromine to 100 cc. of 5% aqueous sodium hydroxide. Compare the red color with a similarly treated creatinine standard. The limit of sensitivity is 1 in 1,000,000.—A. RIEGERT. *Compt. rend. soc. biol.*, 132 (1939), 535–537; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Daylight—Reproduction of. A light source, consisting of an incandescent lamp with a ground-glass screen fitted with 3 dichromate filters (violet-blue, green-yellow, orange-red), is described.—R. TOUSSAINT. *Atti X Congr. Internaz. Chim.*, IV (1938), 689–693; through *J. Soc. Chem. Ind.*, 59 (1940), 542. (E. G. V.)

Fat-Soluble Vitamin Concentrates. By a process which may involve the use of palmitoyl chloride or a similar vitamin A esterifying agent, there is produced a concentrate of vitamin A which is obtainable from naturally occurring animal oils and fats and which contain the vitamin A substantially entirely in ester form and in the same chemical state in which it occurs in the natural oils; the concentrate having a vitamin concentration much higher than that of the raw oil and being substantially free from other constituents of the oil which are separable by subjecting the raw oil to high vacuum distillation at a pressure below 0.1 mm.—KENNETH C. D. HICKMAN, assignor to DISTILLATION PRODUCTS, INC. U. S. pat. 2,205,925, June 25, 1940. (A. P.-C.)

Flavin-Adenine Dinucleotide—Synthesis of, from Riboflavin by Human Blood Cells in Vitro and in Vivo. Human blood cells can synthesize flavin-adenine dinucleotide from riboflavin *in vitro* and *in vivo*. After the ingestion of approximately 200 mg. of riboflavin by mouth, approximately 25 per cent was recovered in the urine as riboflavin and 0.14 per cent in the cells as flavin-adenine dinucleotide. The increase in the dinucleotide concentration in the blood cells was approximately 30 per cent. The concentration of flavin-adenine dinucleotide in urine and saliva after the ingestion of riboflavin is less than 0.005 γ per 0.5 cc.—J. RAYMOND KLEIN and HENRY I. KOHN. *J. Biol. Chem.*, 136 (1940), 177. (F. J. S.)

Folliculin—Determination of, in Urine. A method for the extraction of folliculin from urine and its determination by ultraviolet absorption is described. The peak of the absorption curve for solutions in absolute alcohol is at 280 $\mu\mu$ for pH 6 and at 300$\mu\mu$ for pH 9.5. The folliculin content of human pregnancy urine fluctuates within wide limits from day to day.—A. CHEVALLIER and S. MANUEL. *Compt. rend. soc. biol.*, 132 (1940), 521–523; through *Chimie & Industrie*, 43 (1940), 723. (A. P.-C.)

Glucuronic Acids—Estimating Ether-Linked Conjugated, in Blood of Mammals. The blood is deproteinized by ultrafiltration under pressure and the ultrafiltrate is analyzed in the same manner as in urine (*Compt. rend. soc. biol.*, 131 (1939), 1277–1280).—R. CRISMER. *Compt. rend. soc. biol.*, 132 (1939), 482–484; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Glucoronic Acids—Photometric Estimation of Conjugated, in Urine. The method of Florkin and Crismer (*Compt. rend. soc. biol.*, 131 (1939), 1277–1280) determines only glucuronic acid conjugated with an ether linkage. Glucuronic acid esters are hydrolyzed and the acid is eliminated in the preliminary steps.—R. CRISMER. *Compt. rend. soc. biol.*, 132 (1939), 481–482; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Glutathione—Determination of, in Tissues. It was shown that in certain tissues (liver, spleen) heating at 52° to 55° C. for 30 minutes, after protein precipitation, led to the complete oxidation of the ascorbic acid present. This was due to a reaction involving the presence of ferrous iron. To other tissues iron had to be added in order to oxidize the ascorbic acid by this method. The glutathione, not oxidized by this procedure, was determined by iodometric titration before and after oxidation of the ascorbic acid.—B. I. GOLDSTEIN, M. I. KOLOMIDTS and P. G. KNICHENKO. *Biokhimiya*, 13 (1939), 119–136; through *Chimie & Industrie*, 42 (1939), 964. (A. P.-C.)

Gold—Microdetermination of, in Biological Fluids. An accurate and specific microcolorimetric method for the determination of gold by the use of the Evelyn photoelectric colorimeter has been described. The method is based upon the production of a stable red color by the reaction between auric chloride and *o*-dianisidine in an acid medium in the presence of potassium fluoride. The method is applied to the determination of gold in urine in the

range of 5 to 300γ, and to blood plasma in the range of 5 to 30γ.—WALTER D. BLOCK and OLIVER H. BUCHANAN. *J. Biol. Chem.*, 136 (1940), 379. (F. J. S.)

Histidine—Determination of, in Urine. Ordinary bone charcoal oxidizes histidine; if the charcoal is treated with hydrogen sulfide under water for 3 to 4 days this action is prevented and the "inactivated" charcoal is useful in the estimation of histidine. To estimate histidine in urine, shake a 5 to 10-cc. sample with 5 Gm. of moist or 0.5 Gm. of dry "inactivated" charcoal for 10 minutes and filter through kieselguhr; elute the histidine from the charcoal with three 25-cc. portions of 12% acetic acid, evaporate the eluate to 3 to 4 cc. and transfer to a graduated tube; add 1% bromine in 33% acetic acid in small portions (first addition 1 cc.) until excess of bromine is present; add 2 cc. of a mixture of 2 parts of concentrated ammonia and 1 part of saturated ammonium oxalate solution; heat the solution for one and a half minutes at 100° C., cool at once, dilute to 15 or 30 cc. and read in the Pulfrich photometer with Filter S 50. Adults excrete 100 to 200 mg. of histidine per day, children down to 40 mg.—F. NIENDORF. *Hoppe-Seyler's Z. physiol. Chem.*, 259 (1939), 194–200; through *Chimie & Industrie*, 42 (1939), 965. (A. P.-C.)

7(β)-Hydroxycholesterol—Isolation of, from the Serum of Pregnant Mares. A sterol not hitherto encountered in biological material, 7(β)-hydroxycholesterol, has been isolated from the unsaponifiable matter of pregnant mare serum. Its identity was established by comparison with the synthetic compound which Barr, Heilbron, Parry and Spring prepared by oxidation of cholesterol acid phthalate with permanganate.—O. WINTERSTEINER and JOHANA R. RITZMANN. *J. Biol. Chem.*, 136 (1940), 697. (F. J. S.)

Ketones of the Sterol Group—Polarographic Estimation of Biologically Important. Testosterone and its propionate, progesterone and desoxycorticosterone which contain a keto group in conjugation with a double bond give a polarographic wave between 1.6 and 1.8 v. in 90% alcohol and decinormal lithium chloride. Androsterone and its derivatives give no wave. The height of the wave is proportional to the concentration of sterone and is very nearly the same for equimolecular solutions of different sterones. The polarographic method for estimation of the reactive sterones has advantages over colorimetric and spectrographic methods, which are demonstrated particularly with a testis extract.—J. EISENBRAND and H. PICHER. *Hoppe-Seyler's Z. physiol. Chem.*, 260 (1939), 83–99; through *Chimie & Industrie*, 42 (1939), 1033. (A. P.-C.)

Ketonic Compounds—Determination of, in Muscle. The following conclusions are given. The determination of ketonic compounds (aceton plus acetylacetic acid and beta-hydroxybutyric acid) in muscle and in tissues was found to be quite difficult because the organic tissues did not readily liberate the acetone they contained and adsorbed more or less of the acetone added. A preliminary digestion with pepsin in hydrochloric acid medium is recommended to free the ketonic compounds. A simple trichloracetic defecation then permits the application of Engfeldt's method which gives satisfactory results.—R. LECOQ. *Bull. sci. pharmacol.*, 47 (1940), 87–94. (S. W. G.)

Lactic Acid in Blood—Determination of. A modification of the Mendel-Goldscheider method for the determination of lactic acid in blood has been developed after a critical review of the methods for lactic acid determinations and a study of the method for blood precipitation, the relation of sulfuric acid and veratrole concentration to the final color, the proportionality of color to concentration of lactic acid and the adaption of the Evelyn photoelectric colorimeter.—S. ELGART and J. S. HARRIS. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 758–762. (E. G. V.)

Lactoflavin—Concentrating. An inorganic adsorbate containing lactoflavin is eluted with a solvent such as a mixture of 4 parts of acetone and 1 part of water, by volume.—STEFAN ANSBACHER, GEO. E. FLANIGAN and GEO. C. SUPPLES, assignors to THE BORDEN CO. U. S. pat. 2,186, 314, Jan. 9, 1940. (A. P.-C.)

Male Sexual Hormone—Isolation of. An oily starting material containing a male sex hormone is subjected to condensation with a compound (such as hydroxylamine hydrochloride) having a free amino group and capable of reacting with the carbonyl group of a ketone with separation of water; the resulting condensate is isolated, and is decomposed to split off the hormone.—ADOLF BUTENANDT, assignor to SCHERING CORP. U. S. pat. 2,188,881, Jan. 30, 1940. (A. P.-C.)

Mercury in Urine—Determination of. The photometric mixed color method has been developed for the determination of mercury in urine by the use of di-β-naphthylthiocarbazone, an analog of dithizone. Small samples (50 cc.) are prepared for analysis by oxidizing the organic matter with potassium permanganate in the presence of sulfuric acid. The mercury is extracted in two steps, the first to remove copper (the only interfering element found present in urine) and the second to separate the mercury as a pure complex of di-β-naphthylthiocarbazone for final photometric estimation. The method is very sensitive. An accuracy of ±0.2γ has been obtained for 5γ or less of mercury. Amounts exceeding 50γ can be determined with an error not greater than ±2%.—D. M. HUBBARD. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 768–771. (E. G. V.)

Nitrogen—Determination of, as Ammonia in Monosubstituted Ureas, Urethans, Allophanates and Semicarbazones. The use of a benzyl alcohol solution of potassium hydroxide for determining the nitrogen of amides and nitriles requires in some cases an excessive period of heating, *e. g.*, 4 hours for a semicarbazone. The use of a higher-boiling solvent, *e. g.*, glycerol, is therefore advantageous. The time necessary is thus reduced by more than half with no loss of accuracy. Results with 24 substances were within 3% of theoretical.—S. ROVIRA. *Compt. rend. acad. sci.*, 209 (1939), 754–757; through *Chimie & Industrie*, 43 (1940), 551. (A. P.-C.)

Nitrogen—Iodometric Estimation of Small Quantities of, without Distillation. Methods for the estimation without distillation of nitrogen in samples of 0.5 to 0.005 mg. of nitrogen as protein have been described based on the reaction of ammonia with hypobromite.—MILTON LEVY and ALBERT H. PALMER. *J. Biol. Chem.*, 136 (1940), 57. (F. J. S.)

Phenols—Determination of, in Blood and Urine by Means of the Pulfrich Photometer. The most suitable filter is S 47. The specific extinction coefficient is determined for 1 mg. of phenol by means of a standard phenol solution. For concentrations of 0.019 to 0.050 mg., the reaction is strictly proportional. The mean specific extinction coefficient is 20.31 for free phenols and 16.54 for total phenols. Two titration curves are constructed.—A. D. MARENZI. *Anales farm. bioquím.*, 10 (1939), 82–87; through *Chimie & Industrie*, 43 (1940), 812. (A. P.-C.)

Phosphatase in Yeast—Estimation of. A procedure is presented for the determination of phosphatase in yeast based on King's application of the

use of Folin and Ciocalteu's phenol determination to measure the phenol liberated from disodium phenyl phosphate. The method is preferable to those now in use because it is more rapid and less yeast is required for each determination. It is also preferable to the phosphate method because yeasts almost always contain inorganic phosphate which appears in the controls whereas none of the yeasts investigated contained phenol. The effect of composition of the medium in which the yeast grows on the phosphatase content of the yeast is being investigated.—JAMES J. RAE and EDNA V. EASTCOTT. *J. Biol. Chem.*, 136 (1940), 443. (F. J. S.)

Potassium—Microcolorimetric Method for the Determination of, in Biological Materials. A microcolorimetric method for potassium determinations in biological materials is described. The material is ashed in specially made nickel centrifuge ashing tubes in the presence of HgO at a temperature of 465°. The potassium in the soluble ash is determined by an application of Shohl and Bennett's colorimetric chloroplatinate method. Details are given for whole blood, serum, urine and feces.—PETER WALDEMAR SALIT. *J. Biol. Chem.*, 136 (1940), 191. (F. J. S.)

Preservatives in Foods—Determination of. The following method (technique described in detail) for the identification of saccharin was studied: make the aqueous extract of the product acid with acetic acid, add a slight excess (5 cc.) of 20% lead acetate solution, extract with a mixture of equal volumes of ether and petroleum ether, after removing the solvent add 5 cc. of phenol-sulfuric acid (equal weights), heat for 2 hours at 135° to 140° C., cool, pour into 250 cc. of water, make alkaline with sodium hydroxide and dilute to 500 cc. A magenta or reddish purple color develops if saccharin is present; a yellow, buff or pale salmon shade is not significant. The test is positive with much less than 1.2 mg. of saccharin. Vanillin interferes and must be removed, suitably by extracting the residue with carbon tetrachloride.—WM. F. REINDOLLAR. *J. Assoc. Official Agr. Chem.*, 23 (1940), 288–289. (A. P.-C.)

Protamine Insulin—Soluble. A product which is entirely water-soluble is obtained by combining protamine and insulin in the proportions of at least 2.5 mg. of protamine to 100 units of insulin in an aqueous medium at a pH below 3.5 and heating to about 50° to 55° C. for about 2 to 3 hours. Various operative details are described.—MELVILLE SAHYUN, assignor to FREDERICK STEARNS & CO. U. S. pat. 2,190,137, Feb. 13, 1940. (A. P.-C.)

Radio-Iron—Secretion of Orally Administered, in the Milk of Cows. In two cows studied, 1.5% and 2.5% of the radio-iron, administered orally, was secreted, respectively in the milk during a 78-hour period.—L. A. ERF. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 284. (A. E. M.)

Sex and Gonadotropic Hormones—Effects of, on Red Cell Counts in Rats. Pregnant mare serum hormone, when injected into normal and hypophysectomized males, causes an elevation in red cell count. Injections of testosterone into castrated females or hypophysectomized males likewise increased the red cell count. The count of normal females, treated with pregnant mare serum was lowered, whereas that of castrate or hypophysectomized females, treated similarly, remained unchanged. Estradiol lowers the cell count in normal females. It seems possible that the gonadal secretions are responsible, to some extent, for the sex differences in normal cell count found in many species of animals.—ERWIN P. VOLLMER, ALBERT S. GORDON, IRVING LEVENSTEIN AND H. A. CHARIPPER. *Proc. Soc. Exptl. Biol. Med.*, 46 (1941), 409. (A. E. M.)

Silica—Photometric Determination of, in Biological Materials. The dried material to be analyzed is fused with sodium and potassium carbonates; the melt is dissolved in hydrochloric acid; the iron present in the solution is converted into a water-soluble complex by means of potassium cyanide and citric acid; phosphates are precipitated quantitatively by means of magnesia mixture in ammoniacal solution and the precipitate is separated by centrifuging. In the clear solution the silica is converted into a silico-molybdic complex and determined photometrically by reducing the complex by means of hydroquinone and sodium sulfite.—J. BODNAR and T. TOROK. *Hoppe-Seyler's Z. physiol. Chem.*, 261 (1939), 257–268; through *Chimie & & Industrie*, 43 (1940), 812. (A. P.-C.)

Skatole Red from Urine—Formation and Constitution of. Urochrome, freed from indoxyl sulfate and glucuronates, with 2% sulfuric acid at 100° C. yields indoxyl, removed by ether. Evaporation of the mother liquor yields a substance (melting-point 85° C.) which polymerizes easily, gives a red color (skatole red) with warm hydrochloric acid in air, and with ferric chloride an intense blue. Skatole red is probably indirubin.—M. RANGIER and P. M. DE TRAVERSE. *Bull. soc. chim. biol.*, 21 (1939), 1318–1326; through *Chimie & Industrie*, 43 (1940), 812. (A. P.-C.)

Sterol Derivatives of the Character of Sexual Hormones. Various details of procedure are described by which, *e. g.*, the hydroxybisnorallocholanic acid which is prepared from stigmasterol is transformed, in the form of its ester, into the corresponding keto acid by oxidation with chromic acid. By a catalytic reduction in an acid solution this keto acid is transformed again into the alcohol acid with the production of the epi configuration of the hydroxyl. By causing Grignard reagent, such as phenyl magnesium bromide, to act upon the ester of the hydroxy acid there is obtained the tertiary alcohol which by heating under reduced pressure or with glacial acetic acid or acetic anhydride is transformed into the ethylene derivative belonging thereto. By an oxidizing agent, such as ozone, the ethylene derivative may be transformed into the ketone belonging thereto, an isomeric product of the allopregnan-3-ol-20-one to be found in the female germ gland. By causing the Grignard reagent to act again on the keto compound obtained, by a separation of water and an oxidation there is produced the 3-epiacetoxyetioallocholanone, which by saponification is transformed into the corresponding keto alcohol.—MAX BOCKMÜHL, GUSTAV EHRHART and HEINRICH RUSCHIG, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,188,330, Jan. 30, 1940. (A. P.-C.)

Sulfamide Derivatives—Detection of, in Blood and Urine by the Paracresol-Tyrosinase Reagent. In sulfamide derivatives used in pharmacology the amino group combined with the phenyl radical is the only group which can react with the *p*-cresol-tyrosinase reagent. Tests showed that the only source of error lies in the action of reducing substances which may be present (vitamin C in urine). If the reaction is carried out on urines of patients treated for gonorrheal metritis, when cure has been obtained the color reaction is generally red (or brownish red); if the treatment has failed, the color varies from brownish yellow to brownish red. There consequently seems to be a correlation between the presence of the free amino group of the sulfamide molecule in the urine and the curing of the patients. Its absence can be due either to its modification before elimination through the kidneys, or to a too great dilution of the drug in the urine, or to its non-utilization by the bacteria for some as yet unknown reason.—F. WYSS-CHODAT and R. PAILLARD.

Arch. sci. phys. nat., 21 (1939), 61–67; through *Chimie & Industrie*, 42 (1939), 966. (A. P.-C.)

Thiamine and Certain of Its Metabolic Products —Method for the Determination of, in Urine. The authors summarize as follows: A method is described for the rapid determination of the true vitamin B_1 content of urine. The content of thiamine breakdown products in the urine which are still active in the fermentation test is also determined by this method. The method depends on the oxidative inactivation of the thiamine molecule by alkaline ferricyanide and the determination of vitamin B_1 by fermentation before and after oxidation. The efficiency of the inactivation is determined by the proportion of a superimposed quantity of thiamine which is inactivated in a parallel test. Comparison of the results with the results of rat growth tests shows a satisfactory agreement.—ALFRED S. SCHULTZ, LAWRENCE ATKIN and CHARLES N. FREY. *J. Biol. Chem.*, 136 (1940), 713. (F. J. S.)

Tocopherol (Vitamin E)—Colorimetric Determination of. IV. Determination in Oils after Saponification. Most favorable conditions, *e. g.*, 2*N*-potassium hydroxide-methyl alcohol at 72–74° for 10 minutes, for saponification of oils, *e. g.*, wheat germ and olive, and determination of tocopherol content, are described.—A. EMMERIE. *Rec. trav. chim.*, 59 (1940), 246–248; through *J. Soc. Chem. Ind.*, 59 (1940), 375. (E. G. V.)

3,5,6-Trihydroxy-Androstane and -Pregnane Compounds. Details are given of the production of physiologically active or intermediate compounds such as water-soluble 3,5,6-trihydroxy-androstane or -pregnane compounds having in the 17- or 20-position a =CO or CHOH group.—KARL MIESCHER and WERNER H. FISCHER, assignor to SOCIÉTÉ POUR L'INDUSTRIE CHIMIQUE Á BÂLE. U. S. pat. 2,191,576, Feb. 27, 1940. (A. P.-C.)

Urinary Stones—Analysis of. For determining the composition of urinary stones three types of tests are proposed. I. The appearance and character on pulverization of the stone. II. Burning the powdered stone on platinum foil. III. Chemical analyses. Test I gives rough indication: (*A*) Mulberry colored stones give on crushing a crystalline white or grayish powder which scrapes in the mortar—suspect calcium oxalate. (*B*) Brick red, yellowish red or gray-white stones giving a soft powder usually of grayish red or brick-red color—suspect uric acid and urates. (*C*) White or gray-white stones giving a white chalky powder—suspect phosphates. (*D*) Stones composed of large white or colorless crystals giving a soft, often moist, powder—suspect ammonium magnesium phosphate. (*E*) Hard white stones giving a chalky powder—suspect carbonate or phosphate. (*F*) Bone white, facetted stones giving soft, greasy, scaly crystals on crushing—suspect cystine. Test II, on Pt foil: (*A*) Powder burns with flame. 1. Fibrin gives a yellow flame, smelling like burnt feathers. 2. Cystine gives a bluish flame, smelling like burning rubber. (*B*) Ashed without flame: Uric acid, ammonium urate, xanthine. Sodium or calcium urate leave a white residue. (*C*) Gives more or less residue on burning: 1. Shines with sparkles of light—Calcium oxalate. 2. Does not shine—phosphates, calcium carbonate, mixed stones. III. Chemical tests. (*A*) On burnable powders: 1. Fibrin, insoluble in ether or water, soluble in 10% NaOH; positive biuret reaction; precipitated from alkaline solution by acetic acid with evolution of H_2S (Pb acetate paper). 2. Cystine. Lead test: Add 10% NaOH to a little Pb acetate in water, drop in powdered stone, heat on boiling water bath 15 minutes, gives precipitation of black PbS. Crystallized from ammonia solution cystine gives characteristic crystals, thin six-angled tables. Uric acid crystals are insoluble in dilute HCl, while cystine crystals are soluble. 3. Uric acid, ammonium urate and xanthine give murexide test. Test material for evolution of NH_3. Fibrin may interfere with the murexide test. (*B*) Tests to be made on unburnable powders: 1. If, under the microscope, gas is developed with 10% HCl; carbonate. 2. Ignite dry powder on Pt foil, under microscope add 1 drop of 10% acetic acid. If CO_2 given off, but not before ignition; oxalate. 3. Dissolve ignited residue in acetic acid, place in small test-tube, add ammonium oxalate, precipitation indicate calcium. 4. To the acetic acid solution add molybdenum reagent (150 Gm. ammonium molybdate dissolved in a liter of distilled water with the aid of a little ammonia. Add to a liter of dilute HNO_3). A yellow precipitate indicates phosphorus. 5. Test above solution for evolution of NH_3 on adding 10% NaOH. If positive, indicates ammonium phosphate, which forms if the stone has stood in infected urine. If no ammonia but positive phosphorus the stone contained tertiary calcium phosphate or magnesium phosphate (may be primary, or a result of standing in infected urine).—G. HAMMARSTEN. *Nord. Med. Ark.*, 7 (1940), 1329; through *Farm Revy*, 39 (1940), 778. (C. S. L.)

Urine—Millon Reaction of, and the Influence of the Addition of Tyrosine. The intensity of the Millon reaction, in man and rabbit, is increased by the administration of tyrosine *dl*-tyrosine being much more active in this respect than *l*-tyrosine. Part of the so-called "Millon" bodies is soluble in ether, this solubility being greater with the racemic amino-acid than with the natural product.—K. FELIX and I. MARCO. *Kolloid Z.*, 89 (1939), 293–297; through *Chimie & Industrie*, 43 (1940), 552. (A. P.-C.)

Vitamin A Content of Shanghai Foods. I. Provitamin A Contents of Some Green Vegetables. A preliminary report is given of the vitamin A contents of some thirty vegetables marketed in Shanghai (seventeen of these were estimated in terms of Moore's yellow units and the remainder in terms of their carotenoid contents). The importance of taking seasonal variations into account when evaluating the provitamin A content of vegetables and the need for further work on the botanical identification of our common plant foods are stressed. Further work on the carotenoid contents of these and other Shanghai foods is in progress.—PETER G. MAR. *J. Chinese Chem. Soc.*, 6 (1938), Nos. 1 and 2, 71–77. (F. J. S.)

Vitamin A—Correlation between International Unit and Cod Liver Oil Unit of. III. The International Unit (determined spectroscopically) is about 5 times the cod liver oil unit. Some fresh, unsaponified liver oils have a relatively low cod liver oil value, which becomes normal after hydrolysis.—Z. NAKAMIYA. *Sci. Papers Inst. Phys. Chem. Research Tokyo*, 35 (1939), 875–884; through *Chimie & Industrie*, 43 (1940), 588. (A. P.-C.)

Vitamin B Complex—Biological Methods for the Determination of. The use of sulfite for destruction of vitamin B_1 in the preparation of B_1-free diets eliminates the uncertainties of the autoclaving process for B_1 destruction and gives a diet extremely low in vitamin B_1. Owing to the limitations of the rat-curative and chick-prophylactic types of assay, it is recommended that a collaborative study be made of rat-growth types of method.—O. L. KLINE. *J. Assoc. Official Agr. Chem.*, 23 (1940), 653–654. (A. P.-C.)

Vitamin B_1 Content of Rice—Effect of the Method of Preparation on the. Samples of Shanghai rice contain from 34 to 180γ of vitamin B_1; and the yellow rice contains only a very slight amount. During washing and cooking both polished and un-

polished rice lose much vitamin B_1. The loss with parboiled rice is small. The diet of Shanghai factory workers can be much improved by not washing the rice or by using partially polished rice or parboiled rice. The latter is specially recommended since it involves a minimum change in food habits.—E. F. YANG. *J. Chinese Chem. Soc.*, 6 (1938), Nos. 1 and 2, 55–65 (F. J. S.)

Vitamin B_6-Borate Complex—Formation of a. Vitamin B_6 has been shown to undergo complex formation with boric acid. Boric acid, with a coordination number of 4, is linked to two molecules of the vitamin through the oxygen atoms in the 3 and 4 positions. The complex has the physiological activity of vitamin B_6, and is thermostable in neutral solution.—JOHN V. SCUDI, W. A. BASTEDO and T. J. WEBB. *J. Biol. Chem.*, 136 (1940), 399. (F. J. S.)

Vitamin C—Detection of a Reversibly Half-Oxidized Form of, in Tissues. Monodehydroascorbic acid reduces dichlorophenolindophenol but does not decolorize methylene blue. This suggests a means of detection. Diketogulonic acid, a degradation product of ascorbic acid, reduces methylene blue more readily than it reduces dichlorophenolindophenol.—N. BEZSSONOFF and M. WOLOSZYN. *Compt. rend. soc. biol.*, 132 (1939), 538–540; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Vitamin C Determination—New Biochemical Method of. The amount of vitamin C in the organs of guinea pigs varies as the logarithm of the daily dose of ascorbic acid administered. The following equation was deduced to calculate the quantity of ascorbic acid present in a given substance, if the amount of ascorbic acid existing in the organs of the guinea pig after 18 days of dosage is known: $y = 68.44 \log (x + 1) - 4.90$ (where x = daily dose of ascorbic acid administered, y = amount of ascorbic acid in organs). Results obtained with such experiments are given in a graph.—L. RANDOIN and C. P. LEBLOND. *Bull. soc. chim. biol.*, 21 (1939), 604–608; through *Chimie & Industrie*, 42 (1939), 1032. (A. P.-C.)

Vitamin C—Determination of, in Urine. The urine is acidified with metaphosphoric acid and phosphotungstic acid is added, which removes reducing substances other than ascorbic acid and decolorizes the urine. The vitamin is then determined by titrating the defecated liquid with dichlorophenolindophenol solution.—T. NAGAYAMA, T. TOMOI and T. SACARA. *Biochem. Z.*, 303 (1940), 354–363; through *Chimie & Industrie*, 43 (1940), 812. (A. P.-C.)

Vitamin C Needs of Native Mine Laborers. Two groups, each of 950 mine laborers, chosen so as to be as comparable as possible in respect to tribe, age, physical condition and occupation, were subjected to an experimental test. One group remained on the usual mine diet which was estimated to contain from about 12 to 25 mg. of ascorbic acid, while to the other an additional daily ration of 40 mg. of ascorbic acid was administered in the form of a standardized orange juice concentrate. The experiment was continued for seven months and the behavior of the two groups was studied in various ways. Twelve cases of scurvy occurred in the untreated group and one mild case in the group receiving the additional ration of ascorbic acid. No significant difference could be detected in the behavior of the two groups. Numerous tests showed that the plasma ascorbic acid values were extremely low, even in apparently healthy individuals engaged in hard work. These low values were not significantly raised by the additional daily ration of ascorbic acid. Among other conclusions it may be mentioned that there was no evidence to suggest that natives engaged on the more strenuous types of work developed scurvy either more often or more quickly than those whose metabolic resources were less severely taxed.—F. W. FOX, L. F. DANGERFIELD, S. F. GOTTLICH and E. JOKL. *Brit. Med. J.*, 4152 (1940), 143. (W. H. H.)

Vitamin C Reserves—Rapid Test for. Observations are recorded, before and after a test dose of ascorbic acid, on the variation in concentration of vitamin C in the urine of an adult male whose daily intake of vitamin C was approximately 73 mg. A simplified test, based on the estimation of vitamin C in a single specimen of urine following a test dose of ascorbic acid is described for assessing the past vitamin C intake. The test clearly differentiated two groups of boys whose daily vitamin C intakes were approximately 35 mg. and 63 mg.—J. PEMBERTON. *Brit. Med. J.*, 4154 (1940), 217. (W. H. H.)

Vitamin D Carriers—Biological Methods for Determination of. The A. O. A. C. method for vitamin D assay by preventive biological test was subjected to a critical collaborative test. The data obtained from the ashing of individual bones of chicks provided evidence that the provision for exclusion of chicks weighing 100 Gm. or less is not serving the purpose intended. Cf. following abstract.—CHESTER D. TOLLE. *J. Assoc. Official Agr. Chem.*, 23 (1940), 648–652. (A. P.-C.)

Vitamins D_2 and D_3—Spectrophotometric Determination of. The following conclusions are given: (1) A new reagent for the determination of vitamins D_2 and D_3, consisting of a solution of antimony trichloride and acetyl chloride in chloroform, has been described. (2) The limits of concentration of antimony trichloride and acetyl chloride, within which the sensitivity of the reagent is constant, have been determined. (3) The reagent produces a yellowish pink color with vitamins D_2 and D_3 which reaches its maximum intensity within 30 seconds and is stable for from 4 to 5 minutes. (4) The absorption curves of the reaction product of the reagent with vitamins D_2 and D_3 have been determined in a Bausch and Lomb spectrophotometer. The two curves are identical, having a maximum at 500 mμ. (5) The $E_{1\text{ cm.}}^{1\%}$ values at 500 mμ for vitamins D_2 and D_3 are identical and are approximately 1800 which is about 3 times the value given by the reagent proposed by Brockmann and Chen. (6) The optical density, as determined by the difference in absorption at 500 and 550 mμ is directly proportional to the vitamin concentration. (7) The lower limit of the amount of vitamin that can be accurately determined by the method described is approximately 0.2γ.—CYRIL H. NIELD, WALTER C. RUSSELL and A. ZIMMERLI. *J. Biol. Chem.*, 136 (1940), 73. (F. J. S.)

Vitamin E—Crystallized Derivatives of. A raw concentrate of vitamin E is treated with cyanuric acid; the solution thus obtained is dried; the dry product is dissolved in an organic solvent such as ether, resulting solution is brought into contact with an adsorbing material such as alumina and the adsorbed substance is eluted by another organic solvent such as a mixture of 4 parts of methanol and 1 part of ether, from which a crystalline product is then produced. Various details of procedure are given.—CARL L. LAUTENSCHLÄGER and FRITZ LINDNER, assignors to WINTHROP CHEMICAL CO. U. S. pat. 2,188,878, Jan. 30, 1940. (A. P.-C.)

Vitaminizing Coffee. Freshly ground coffee is directed in a stream through a closed passage, and there is directed across and through the flow of ground coffee a foglike mist formed by the atomization of a vitamin-containing solution with an inert gas thereby guarding against oxidation of the freshly ground coffee and accomplishing a vitamin film

coating subsequently preserving the freshness of the coffee.—FRED GEITZ, JR. U. S. pat. 2,206,319, July 2, 1940. (A. P.-C.)

Vitamins—Concentrating, from Fish Press Water. Coagulable protein matter is precipitated from fish press water by an alum to inhibit enzymolysis and simultaneously precipitate the proteolytic enzymes; the supernatant liquid is filtered and contains substantially all of the vitamin content of the original press water, and is treated for vitamin concentration, as by evaporation or adsorption.—SVEN H. LASSEN, assignor to PHILIP R. PARK, INC. U. S. pat. 2,188,008, Jan. 23, 1940. (A. P.-C.)

Water—Semimicromethod for the Determination of, in Blood, Serums, Plasma and Cells. Capillary blood (for arterial blood) was collected in capillary tubes 7 cm. long graduated into 50-cu. mm. sections; venous blood was collected in tuberculin syringes. The blood was deposited in a neutral glass ampul and kept 24 hours at 105° C. Another ampul received the blood for water determination in plasma or serum; after sealing, it was centrifuged at 3000 r. p. m. for 10 minutes for serum, for 60 min. for plasma and cells. The plasma and serum were collected in another ampul, weighed and dried. A semimicrobalance of rapid weighings sensitive to 0.00004 Gm. must be used. The amount of blood necessary for each determination is 40 to 60 mg. for total blood, 70 to 90 mg. for serum, 110 to 140 mg. for cells. For plasma or blood the ampul must contain 0.0001 Gm. of polyanetholesulfonic acid (anticlotting agent). The method gives figures within ±0.1%.—E. SARTORI. *Arch. Ist. biochim. ital.*, 11 (1939), 3–22; through *Chimie & Industrie*, 43 (1940), 17. (A. P.-C.)

***l*-Xylose—Metabolism of.** The feeding of *l*-xylose to rats increased significantly the non-fermentable reducing substances of the liver, kidney and blood but failed to increase the glycogen content of the tissues. Significant decreases occurred in liver and blood lactic acid, muscle glycogen and in the fermentable reducing substances of the liver after its administration. *l*-Xylose is very poorly absorbed from the gastro-intestinal tract. Its coefficient of absorption, 0.007, is the lowest recorded for any carbohydrate.—HARDY W. LARSON, N. R. BLATHERWICK, PHOEBE J. BRADSHAW, MARY E. EWING and SUSAN D. SAWYER. *J. Biol. Chem.*, 136 (1940), 1. (F. J. S.)

ANALYTICAL

Absorption Spectrophotometry in Pharmaceutical Analysis. II. The following summary is given: (1) The paper continues the work and gives corrected data for diethylstilbestrol and its dipropionate ($E^{1\%}_{1\,cm.}$ being 600 ± 20 for the former: and the readings of the absorption spectrum of the latter showed a flat peak between 230 and 240 mμ). Figures are given for testosterone and testosterone propionate. (2) The difficulties of dealing with oil solutions are indicated. (3) Figures are given for a range of halibut liver oil samples with particular reference to spectrophotometric criteria of purity. (4) Absorption curves and extinction values have been obtained for a number of alkaloidal and other important drugs, including the cocaine group, solanaceous alkaloids, barbiturates, ephedra alkaloids, morphine and related compounds, emetine, strychnine and plant hormones. The influence of solvents and pH on these have been investigated so that the data obtained may be adopted for analytical purposes. Many of the data have been of a primary nature preparatory to applying them to the examination of drugs and galenicals, which work it is proposed to carry out in the future.—W. F. ELVIDGE. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 219–236. (S. W. G.)

Alcohols—Identification of. The optical crystallographic data for fifteen esters of carbanilic acid have been determined and compared with similar data for diphenyl urea. The optical properties provide a means of identifying the urethans even when they are mixed with diphenyl urea. A method for confirming the identity of an alcohol is outlined.—B. T. DEWY and N. F. WITT. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 459–460. (E. G. V.)

Aliphatic Amides—Primary, Identification of, as Oxalates. Primary aliphatic amides easily form stable oxalates with oxalic acid, and the derivatives are titratable with a base or potassium permanganate.—C. A. MACKENZIE and W. T. RAWLES. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 737–738. (E. G. V.)

Allyl Alcohol—Determination of, in Air. Pass a definite volume of air containing the allyl alcohol through 3 Petri tubes each containing 15 cc. of water, at the rate of 5 liters per hour. The allyl alcohol is usually absorbed completely in the first tube. Transfer the solution to an Erlenmeyer flask, add a definite volume (10 to 25 cc.) of bromide-bromate solution, then 40 cc. of 10% sulfuric acid solution, and allow the stoppered mixture to stand for 15 minutes. Add 3 cc. of 1% potassium iodide solution, stopper, and after 15 minutes titrate with hundredth-normal sodium thiosulfate. For 2 mg. of allyl alcohol the errors did not exceed 2% and for 0.1 mg. the error reached as high as 10%. The presence of methanol in the air in amounts equal to that of the allyl alcohol did not affect the determinations, while an equal amount of acetone increased the results slightly. Formaldehyde also increased the results slightly, but the errors were not higher than the experimental deviations.—N. B. BARANOV. *Zavodskaya Lab.*, 8 (1939), 931–932; through *Chimie & Industrie*, 43 (1940), 653. (A. P.-C.)

Arsenate and Phosphate Ions—Two New Gravimetric Methods for Determining. Accurate results in the determination of arsenic and phosphorus can be obtained by precipitation as silver thallium arsenate and silver thallium phosphate. The recommended reagents are 4% thallyl acetate and 0.1*N* silver nitrate. At least four parts of univalent thallium salt must be present for each part of phosphorus or arsenic and the silver nitrate should be added dropwise from a buret. To 50 cc. of solution containing 0.05–0.1 Gm. of disodium hydrogen arsenate heptahydrate add 5–10 cc. of thallyl acetate solution and 5–10 cc. of silver nitrate solution while stirring. Filter and wash the precipitate first with water, then three times with alcohol and 5–6 times with ether. Dry for fifteen minutes in a desiccator over calcium chloride. The phosphate precipitate is slightly soluble in water and for its precipitation take 25 cc. of solution containing 0.05–0.1 Gm. of disodium hydrogen phosphate with twelve molecules of water of hydration, add 6–12 cc. of thallyl acetate solution and 10–20 cc. of silver nitrate solution. Filter and wash the precipitate first with a mixture of three parts alcohol and 1 part water, then twice with pure alcohol and finally with ether. Dry in a vacuum for twenty minutes. The precipitates contain 6.02% arsenic and 27.86% phosphorus, respectively. If the original solution is acidic, neutralize to methyl orange, make slightly acidic with 2*N* nitric acid, boil a few minutes, neutralize with ammonia and heat to remove the excess.—G. SPACU and L. DIMA. *Z. Anal. Chem.*, 120 (1940), 317–322. (S. W. G.)

Arsenic—Determination of Traces. A collaborative study was made of the Cassil-Wichmann method for the determination of 30 to 375γ of arsenic and its combination with the sulfuric-nitric-perchloric acid method of sample preparation. Some of the recoveries were not so good as those shown in the de-

velopment of the method, but in general they were a definite improvement over the empirical procedures previously used for small quantities of arsenic. The method can be improved in the following respects: (1) omission of the potassium iodide reagent, there being sufficient potassium iodide in the standard iodine solutions; (2) preparing the starch indicator from a good grade of soluble starch instead of potato starch; (3) specifying granulated zinc of 20-mesh (instead of 20- to 30-mesh); (4) adding stannous chloride to the evolution solution only after the final dilution to 80 to 90 cc.; (5) ensuring tightness of the ground glass joints either by grinding them with fine emery dust or by sealing with a little stopcock grease; if an aliquot greater than 50 cc. is used, the quantities of acid and potassium iodide should be correspondingly increased. Reasons for these modifications are given.—C. C. CASSIL. *J. Assoc. Official Agr. Chem.*, 23 (1940), 297–301. (A. P.-C.)

Artemisia Filifolia Torrey—Preliminary Study of. This plant is widely distributed in western Nebraska and it is thought had not previously been investigated. Data on moisture, volatile material and ash are reported. Flowers and leaves were extracted with selective solvents by a modification of Dragendorff's method. Fresh plants of 1938 yielded 0.16 per cent of volatile oil and the 1939 yielded 0.13 per cent. Physical and chemical constants and properties reported include specific gravity, specific optical rotation, refractive index, congealing point, solubilities, behavior with potassium permanganate, bromine and nitrogen oxychloride, qualitative elementary analysis, quantitative determination of aldehyde-ketones and phenols; acid, saponification, ester, acetyl and methoxy values. Additional investigation will be necessary before a complete report can be made.—HOWARD HOPKINS and JOSEPH B. BURT. *Jour. A. Ph. A.*, 30 (1941), 77. (Z. M. C.)

Cadmium Tetrapyridinethiocyanate—Use of, in Gravimetric Analysis. To a neutral solution containing cadmium add 1 Gm. of ammonium thiocyanate and precipitate the cadmium as cadmium tetrapyridinethiocyanate ($CdPy_4(CNS)_2$) by adding 10–15 cc. of pyridine to the boiling solution. Cool, filter through a Gooch crucible, wash as recommended by Spacu and Dick (*Chem. Abstr.*, 22 (1927) 2448), dry in a vacuum desiccator and weigh. The precipitate contains 20.63% of cadmium. If only 1 cc. of pyridine is added the precipitated compound is the dipyridene thiocyanate.—G. VORNWEG. *Z. anal. Chem.*, 120 (1940), 243. (S. W. G.)

Bile Salts. The salts of ox bile are a mixture of the sodium salts of taurocholic and glycocholic acids with small proportions of the sodium salts of tauro- and glyco-deoxycholic acids and tauro- and glycocholeic acids. The authors have evolved the following method for estimating: Heat 0.5 Gm. with 30 cc. of a 15% solution of sodium hydroxide under a reflux condenser for twelve hours. Add 30 cc. of water and filter through a No. 54 Whatman filter paper into a 250-cc. separator and wash the flask and filter paper thoroughly with hot water. Acidify the contents of the separator with diluted H_2SO_4, cool and extract with four separate quantities of 50 cc. of ether. Bulk the ethereal extracts and wash with two separate quantities of 10 cc. of water. Filter into a tared flask and wash the filter paper with ether. Evaporate off the ether, dry at 100° and weigh the total mixed acids left. The total sulfur content can be used to calculate the amount of sodium taurocholate present; from this figure and the acid value of the total mixed acids the approximate composition of the salts can be calculated. The acid value of pure cholic acid is 137, while that of any fatty acids likely to be present is about 280. Both sodium tauro-glycocholate and sodium glycocholate should contain not less than 70% of total mixed acids after hydrolysis. The acid value of these acids should not be greater than 145. Sodium glycocholate should contain not more than 0.8% of total sulfur.—N. EVERS and W. SMITH. *Pharm. J.* 144 (1940), 375. (W. B. B.)

Carbonyl Compounds—Identification of, with *p*-Carboxyphenylhydrazine. The carboxyphenylhydrazones of 48 carbonyl compounds were prepared and characterized by the melting points and the molecular weights. *Preparation method:* To 50 cc. of a 4% solution of *p*-carboxyphenylhydrazine hydrochloride was added 0.5–2 Gm. of the carbonyl compound dissolved in 25 cc. of alcohol or water. After 1–3 hours, heating on the water bath the hydrazones precipitated. They decompose during determination of fusion point. An electric melting point block is described. Usually reaction was smooth and normal but occasionally the mol. wt. found did not correspond to that of the expected substance. Thus *o*-acylbenzoic acids condensed to form substituted phthalazones—4. Gamma aliphatic ketonic acids condensed to form phenylhydrazones which by boiling with glacial acetic acid or by heating to 150° C. for several hours, formed substituted pyridazinones—6. Ketones containing a double bond conjugated with the CO group, condensed to form hydrazones which immediately or after boiling with glacial acetic acid formed substituted Δ^2-pyrazolines. Sixteen of the carbonyl compounds were also condensed with *o*-carboxyphenylhydrazine, and the melting points and mol. wts. of these hydrazones are also tabulated. The ortho compound was less useful than the para for identification reactions, for it readily lost a mole of water forming a substance incapable of condensation with the carbonyl group. The claim of Bilz, Maue and Sieden (*Ber.*, 35 (1902), 2004) that it is difficult to condense *p*-sulfoxyphenylhydrazine with carbonyl compounds was confirmed.—S. VEIBEL, A. BLAABERG, and H. H. STEVNS. *Dansk Tids. Farm.*, 14 (1940), 184. (C. S. L.)

Copper—Gravimetric Determination of, as Cuprous Thiocyanate. A laboratory report showed that students obtained high values in determining copper as cuprous thiocyanate by the gravimetric method of Rivot (*Z. anal. Chem.*, 58 (1919), 124). The trouble was traced to the presence of an impurity in the supposedly pure ammonium thiocyanate used; the impurity yielded a precipitate of higher molecular weight. Correct results were obtained with most of eleven other samples of ammonium thiocyanate purchased although all but two gave slightly high results. The precipitates of pure cuprous thiocyanate dissolved completely in concentrated ammonia solutions but the impure precipitates left a slight residue which may have been cuprous sulfide. It is recommended to test each lot of ammonium thiocyanate with a copper solution of known content and then, if the values obtained in determining 150–250 mg. of copper are in error by more than 0.1%, recrystallize the reagent. In ordinary work the positive error is likely to compensate a slight error arising from the fact that a little of the precipitate is usually lost by its sticking to the beaker in which the precipitation was carried out.—J. BODNAR and V. TOLNAY. *Z. anal. Chem.*, 120 (1940), 336–341. (S. W. G.)

Cordials and Liqueurs—Analysis of. A collaborative study was made of the determination of alcohol by volume, total solids, benzaldehyde, volatile esters (as ethyl acetate) and γ-undecalactone in imitation apricot and peach cordials. Excellent agreement was obtained on alcohol and total solids. Most of the collaborators obtained better results for benzaldehyde than they had in a similar study the previous year, indicating improvement with increased

familiarity with the method. The method for γ-undecalactone is suitable as a qualitative test but cannot be depended upon for quantitative data in its present form.—JOHN B. WILSON. *J. Assoc. Official Agr. Chem.*, 23 (1940), 198–201. (A. P.-C.)

Cyanide—Determination of Small Amounts of, in Water. Where the cyanide content is greater than 0.1 p. p. m. the ferric cyanate method is recommended, and where less than 0.1 p. p. m. the phenolphthalein method of Childs and Ball. A Prussian-blue method is described in which 3–4 drops of a 3% ferrous sulfate solution and 1 drop of 1% aqueous ferric chloride are added to distillates in 50-cc. Nessler tubes from 1 liter of acidified sample, the whole is mixed, and 10% aqueous sodium hydroxide added dropwise until no further precipitate appears. Then 10% sulfuric acid is added until the precipitate is dissolved, the solution set aside for several hours and the blue color which develops is matched against sodium cyanide standards. Complex cyanides (*e. g.*, ferricyanide, cyanate, etc.) are not determined. In the ferric cyanate method, a 500-cc. sample acidified with 0.5 Gm. of tartaric acid is distilled and 50 cc. of distillate are collected. The distillate, 0.2 cc. of 10% aqueous sodium hydroxide, and 0.5 cc. of yellow ammonium sulfate solution are evaporated to dryness, and the residue is dissolved in 10 cc. of distilled water and 1 cc. of 5% hydrochloric acid and heated just to boiling. After several hours the sulfur coagulates and is then filtered off, washed into a 50-cc. Nessler tube and made up to 40 cc. Then 1 cc. of 10% aqueous ferric chloride is added, the volume adjusted to 50 cc. and the solution mixed and matched against standards immediately. Standards containing 0.02–2 milligrams of cyanide in 50 cc. are prepared from potassium cyanate stock solution by adding 1 cc. of 10% aqueous ferric chloride after acidifying with 1 cc. of 5*N* hydrochloric acid. This method is not recommended for less than 0.05 miligrams of cyanide (*i. e.*, 1 p. p. m. in sample) as the color fades quickly.—J. E. FASKEN. *J. Am. Water Works Assoc.*, 32 (1940), 487–493; through *J. Soc. Chem. Ind.*, 59 (1940), 502. (E. G. V.)

Extractum Filicis—Crude Filicin Content of. Methods of determination of crude filicin in Extractum Filicis were studied as regards best quanity of extract, quantity and concentration of BaOH, acidity for precipitation after extraction, etc. Three Gm. of extract were found the best quantity to avoid emulsion formation, less gave low results. Use of 100 cc. BaOH water and 60 minutes, standing, with tapping off of 85 cc. of the aqueous layer were found advantageous. The quantity of crude filicin obtained depended on the time from the beginning of shaking until its precipitation with HCl. Modified Method proposed: The extract is well stirred several minutes, then 3 Gm. weighed out and dissolved in 40 cc. ether, placed in a separatory funnel, and 100 cc. of 3% BaOH solution added. Shake vigorously for 5 minutes, allow to stand 60 minutes until the layer is clear. Tap off 85 cc. of the aqueous layer and add 20 cc. of dilute HCl. The precipitated crude filicin is shaken portionwise with ether: 25 + 15 + 10 + 10 cc. The ether is collected in a tared flask and distilled on the water bath. After drying the flask 3 hours in a drying oven, the residue corresponds to 75% of the total content of crude filicin in the sample.—H. I. TOFT. *Dansk Tids. Farm.*, 14 (1940), 241. (C. S. L.)

Ferric Iron—New Method for the Volumetric Determination of. The volumetric methods for the determination of ferric iron which involve reduction followed by titration with a standard oxidizing agent have several obvious advantages compared with a direct titration process. In the direct methods at present in use unstable solutions (titanous or stannous chloride) are used, or else special precautions are needed. In the method described in the present paper the ferric iron is titrated directly with mercurous nitrate solution in the presence of ammonium thiocyanate. The method is shown to be accurate both for direct determination of ferric iron and for total iron following permanganate determination of ferrous iron. Hydrogen chloride interferes with the reaction when present in concentrations greater than 0.1*N*.—F. R. BRADBURY and E. G. EDWARDS. *J. Soc. Chem. Ind.*, 59 (1940), 96–98. (E. G. V.)

Flavors and Non-Alcoholic Beverages—Analysis of. A collaborative study of the previously described method for the determination of β-ionone (*J. Assoc. Official Agr. Chem.*, 22 (1939), 378–388) was made on a stock alcoholic β-ionone solution and on two imitation raspberry flavors. Excellent results were obtained with the pure alcoholic solution. On the imitation flavors, the results were encouraging in view of lack of previous experience of the collaborators with the method. In most cases the precipitates obtained were well crystallized and satisfactory for microscopic identification. In one instance the precipitate was oily instead of crystallized; but imperfect crystallization does not result in low recovery.—JOHN B. WILSON. *J. Assoc. Official Agr. Chem.*, 23 (1940), 572–576. (A. P.-C.)

Fluorine—Determination of Traces. Attempts by various investigators to adapt the Willard and Winter thorium nitrate titration method to the determination of micro quantities (less than 50γ) of fluorine have so far not proved satisfactory. A photometric study of the rate of development of the end-point showed that it is apt to be sluggish, and that the titration with very dilute (0.0004 times normal) thorium nitrate does not involve an end-point in the usual sense of an abrupt color change after the equivalence point is reached, but rather an equilibrium condition between fluorine and thorium and indicator and thorium. If the conditions are rigidly standardized and titrations carried to the same end-point, accurate results can be obtained, but it is sometimes difficult to reproduce the end-point even when a reference titration vial is used. Dahle's "back titration" method (*J. Assoc. Official Agr. Chem.*, 21 (1938), 459–467) is the best modification of the thorium nitrate method because it effectively eliminates salt or neutralization errors; it is believed that the only factor limiting the accuracy of this method is the "distillation blank," which it is impossible to eliminate completely. This "blank," which varies from 2 to 7γ of fluorine per 150 cc. of distillate, can be due to fluorine from glass only in very slight degree, the greater part coming from some other source, possibly chlorine from the perchloric acid developed during the distillation which alters or partially bleaches the color of the end-point lake. The determination of fluorine by means of its bleaching effect on the reddish purple aluminum-aluminon (aurintricarboxylic acid) lake in conjunction with the back-titration method will be studied.—P. A. CLIFFORD. *J. Assoc. Official Agr. Chem.*, 23 (1940), 303–307. (A. P.-C.)

Fluorine—Thorium Nitrate Titration of Micro Quantities of, in Aqueous and Alcoholic Systems. Micro quantities of fluorine cannot be determined accurately in aqueous systems by application of the normality value of thorium nitrate solution. Were normality value applied for the 2 to 50γ range in aqueous system adjusted with hydrochloric acid, the means apparent incidence would be 1.5 times the true value; similar application of normality value for γ range in the buffered aqueous systems

would give a medial apparent indication 2.05 times the true medial. For such γ range, titration value of thorium nitrate solution must be determined empirically against corresponding quantities of fluorine from a material of known assay and for the specific solvent, definite volume and identical quantity of indicator. When titrations are made in alcoholic solution, however, application of stoichiometrical value of the standard thorium nitrate will give accurate values in a solution of adjusted pH and without inclusion of a buffer solution.—J. W. HAMMOND and W. H. MACINTIRE. *J. Assoc. Official Agr. Chem.*, 23 (1940), 398–404. (A. P.-C.)

Formaldehyde—Determination of, in the Presence of Acetaldehyde. The formation of hexamethylenetetramine by the reaction of formaldehyde and ammonium salt has been used for the determination of formaldehyde. Under the same conditions acetaldehyde reacts much more slowly, so that it is possible to carry out the procedure in the presence of acetaldehyde provided certain precautions are taken. To the solution to be analyzed add an excess of 5% ammonium sulfate solution, shake and allow to stand exactly 15 minutes. Then add 2–3 drops of a solution of rosolic acid in ethanol and sufficient 0.5*N* sodium hydroxide to give a distinctly basic reaction. Titrate the excess base with 0.5*N* hydrochloric acid. Three moles of formaldehyde are equivalent to 1 mole of sulfuric acid.—A. CASTIGLIONI. *Z. anal. Chem.*, 119 (1940), 287–290. (S. W. G.)

Glass Electrode—Laboratory Note on. The adherence of films to the electrode may cause errors. Sometimes the film can be removed by repeated washing with water and thorough wiping with a piece of silk. The pH will gradually approach the true reading as the film is removed. At other times chromic acid solution may be required to clean the electrode surface.—G. E. SHAW. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 271–273. (S. W. G.)

Hippuric Acid—Colorimetric Determination of. The method, specially suited for determining hippuric acid in the presence of benzoic acid, depends on the formation of the red cochineal-like substance by the action of sodium hypobromite on hippuric acid; a similar reaction does not take place with benzoic acid. To 5 cc. of a solution to be tested, on a boiling water bath, add 2 cc. of sodium hypobromite solution recently prepared by adding 20 cc. of water and 1 cc. of bromine to 10 cc. of a 30% solution of sodium hydroxide. Stir the mixture for 30 seconds and then add 1 cc. of chloroform or 1.5 cc. of ether which dissolves the red substance. Remove the colored solution and determine the hippuric acid by the depth of color. The method is sensitive to 0.5 Gm. per liter. The sodium hypobromite can be replaced by sodium hypochlorite but the color is more yellow and the test is less sensitive.—G. DENIGÈS. *Compt. rend. acad. sci. U. R. S. S.*, 209 (1939), 972–974; through *Chimie & Industrie*, 43 (1940), 724. (A. P.-C.)

Histamine Phosphate—Colorimetric Assay Method for Solution of. A colorimetric method for the assay of Solution of Histamine Phosphate, U. S. P. XI, based on a comparison of the colored product formed upon diazotization of histamine with either of two standards, an Artificial Color Standard or a Standard Histamine Phosphate Solution, is described. When used to assay a solution of histamine phosphate of the same concentration as that of the U. S. P. XI solution, the method showed an average accuracy of 0.3%.—F. A. FUHRMAN. *Pharm. Arch.*, 12 (1941), 1. (A. C. DeD.)

Hydroxyl Compounds—Identification of, with Nitrobenzazide. Sah had used phenyl benzazide for preparation of phenylcarbaminic ethers. In a similar reaction the authors tried *m*-nitrobenzazide, obtaining *m*-nitrophenylcarbaminic ethers of hydroxy compounds, useful for identifications. Usually xylene was used for reaction solvent; with tertiary alcohols, toluene was used. The reaction products could be recrystallized from ligroin or petrol ether, if they melted below about 60° C. With higher melting ethers, toluene or alcohol could be used in recrystallization. Mol. wts. were determined by titration with $TiCl_3$. The m. ps. and mol. wts. of 33 ethers made from alcohols, carbinols, glycols, etc., are tabulated. *Preparation method:* 2 Gm. of the *m*-nitrobenzazide were mixed in a small flask with the equivalent amount of the OH compound. Ten cc. of xylene were poured on and the mixture refluxed 1–3 hours (if in toluene, 4–6 hours). Then the reaction mixture was set in the ice box over night, the remainder of the reaction product, not thus crystallized, was separated from the solvent by evaporation of the latter at room temperature, or by vacuum distillation.—S. VEIBEL and H. LILLELUND. *Dansk Tids. Farm.*, 14 (1940), 236. (C. S. L.)

Hydroxyl Groups—Determination of, with Grignard Reagent. The apparatus and procedure described give accurate results rather quickly and show considerable improvement over conventional apparatus. Each hydroxyl-containing substance that was completely soluble in the Grignard reagent gave a methane evolution corresponding to some whole number of hydroxyl groups in its structure.—W. FUCHS, N. H. ISHLER and A. G. SANDHOFF. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 507–509. (E. G. V.)

Iodine in Thyroid. The uncertain end-point and the blank are eliminated and accurate results obtained by the U. S. P. XI assay for iodine in thyroid when the pH is adjusted to about 2.5 to 2.7 and the temperature to about 33° C. before titration.—R. S. BURNETT and R. F. WARKOW. *Ind. Eng. Chem., Anal. Ed.*, 12 (1940), 734–735. (E. G. V.)

Ion Exchange—Preparation and Inorganic Chromatographic Application of Paper Capable of. Schwab, *et al.*, have designated as chromatography a separation of ions such as occurs when a solution is filtered through a column of aluminum oxide. At different places segregation of certain ions is found in definite zones whereas some ions such as sodium will pass through the column without being absorbed to any extent. Organic chromatography depends upon a stepwise adsorption of the molecules present in a solution, but inorganic chromatography depends upon an exchange of ions of the solution with ions present as impurity in the aluminum oxide. Earlier the author proposed the use of a porous paper suitably impregnated with a substance capable of ion exchange. The paper was cut into strips, folded and by means of a piece of apparatus similar to a filter press was introduced into the solution in a direction perpendicular to the folds. This method was later given up because it was found that better results would be obtained if the solution were sucked up into the paper by capillarity. The impregnation of such paper is described and some analytical uses for technical purposes are discussed. A simple way to impregnate the paper consists in causing a precipitate of aluminum hydroxide to form in the pores of the paper; hydrated oxides of chromium and silicon behave in a similar manner. The paper is dipped in a solution of sodium aluminate, dried and then moistened with a solution of sodium bicarbonate. The paper is then washed for several days and then dried. A little of the solution to be tested is placed on the paper which is then developed by a suitable reagent. Tests with solutions of copper-cadmium-nickel; copper-cadmium; and copper-nickel are described. Estimations of the quantities

of these metals present are approximately correct.—H. FLOOD. *Z. anal. Chem.*, 120 (1940), 327–35. (S. W. G.)

Lead—Determination of Traces of. The rapid colorimetric dithizone method of Perlman (*Ind. Eng. Chem., Anal. Ed.*, 10 (1938), 134–135) for the determination of lead in maple products has the following drawbacks: (1) the use of a rather small (15-Gm.) sample; (2) the somewhat awkward way of taking the aliquot and the difficulty sometimes experienced in withdrawing a clear 11-cc. aliquot after the preliminary dithizone shake-out because of emulsion formation; (3) the necessity for working up another sample for a repeat determination if the range is exceeded, as 10 of the 11 cc. of "strip acid" is used in the color development. To overcome these objections the method was modified as follows: (1) 50 Gm. were used; (2) preliminary acid treatment was with 10 cc. of concentrated hydrochloric acid added directly in the centrifuge bottle; (3) the concentration of the ammonia-cyanide-citrate reagents was doubled; (4) aliquots were changed to allow the use of ordinary transfer pipettes (10/20 x 10/25). Collaborative study of the modified method gave recoveries sufficiently close to theoretical in most cases to demonstrate the general reliability of the Perlman technique, but the suggested modifications apparently do more harm than good because of zinc interference and the appearance of "off" shades in the color development.—P. A. CLIFFORD. *J. Assoc. Official Agr. Chem.*, 23 (1940), 307–310. (A. P.-C.)

Mercury—Determination of, in Mercurochrome. The following procedure is recommended: Weigh 0.5 to 1 Gm. of the mercurochrome in a 250-cc. conical flask and dissolve in 20 cc. of distilled water. Add 5 Gm. of potassium hydroxide pellets and 2 Gm. of zinc filings and boil the mixture under a reflux condenser for at least fifteen minutes; remove the flame and wash down the condenser with 50 cc. of distilled water. Filter off the amalgam by decantation, wash it well with distilled water and then dissolve the amalgam in a mixture of 20 cc. of nitric acid and 20 cc. of distilled water. After gently boiling off the nitrous fumes, cool the solution, add a slight excess of permanganate, decolorize with a drop of solution of hydrogen peroxide and titrate with 0.1*N* ammonium thiocyanate. The results are in agreement with those given by the B. P. C. assay method and by methods involving the destruction of the mercurochrome by oxidation.—G. J. W. FERREY. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 210–212. (S. W. G.)

Mercury—Determination of Traces of. In the removal of mercury from a slightly acid solution by replacement precipitation with zinc (Winkler, *J. Assoc. Official Agr. Chem.*, 21 (1938), 220–228), by preparing a smooth coat (filter strata) of zinc on a No. 3 porosity fritted glass filter crucible (Ace) it was found possible to remove mercury completely from the pure solutions (slightly acid with hydrochloric acid) using only 0.8 Gm. of zinc. Results obtained showed the procedure to be wrothy of further study. Isolation and preparation of the mercury solution can be effected more satisfactorily than by the present A. O. A. C. method by transferring the mercury from the dithizone solvent phase to the aqueous phase by shaking with aqueous sodium thiosulfate solution, destroying the thiosulfate with permanganate and the excess of permanganate with hydroxylamine hydrochloride as described in *J. Assoc. Official Agr. Chem.*, 21 (1938), 220–228. A study of the actual determination has resulted in increased sensitivity of the titration procedure (details of the technique to be published later) and in a clearer diagnosis of some of the difficulties encountered in the photometric determination. In some cases (*e. g.*, when a No. 61 filter centered at 610$\gamma\gamma$ is used) there occurs a rise in the photometric reading of the dithizone solution containing mercury shortly after it is placed on the photometer; but with a No. 49 filter there appears to be a drop in the reading; there appears to be a shift in the mercury-dithizone equilibrium, probably from keto to enol form under the influence of a strong light.—W. O. WINKLER. *J. Assoc. Official Agr. Chem.*, 23 (1940), 310–313. (A. P.-C.)

Microchemical Methods. II. Micromelting Points of Barbiturates. Using the Kofler micromelting point apparatus (Supplement to *Angew. Chem.*, No. 36, 1940), the melting points and appearance under the microscope during heating, and during solidification of the drop, were determined and tabulated for barbituric acid and 29 different barbiturates. The observations were of use for extending the value of the micromelting point determination for identification purposes. The polymorphic compounds showed characteristic behavior during both the melting and the after-cooling; examples are depicted in illustration. Such large differences were observed in the crystal optics of sublimed crystals as to afford distinctive identification of different derivatives with the same melting points. Impurities not only altered the melting temperature and melting interval but also affected the sublimation forms and the conversions of crystal form. Sublimation in drop form was obtained at lower temperatures with impure preparations than with pure substances. Hence considerable index of the state of purity was obtained by sublimation.—F. REIMERS. *Dansk Tids. Farm.*, 14 (1940), 145. (C. S. L.)

Microchemical Methods. III. Determination of Mixtures of *p*-Hydroxybenzoic Acids by Microrefraction. Applying Kofler's method of microoptical refraction determination in his micromelting point apparatus (Supplement to *Angew. Chem.*, No. 36, 1940), the optical refraction of mixtures of ethyl and propyl *p*-hydroxybenzoic acids was studied. These were such mixtures as would be obtained by extraction of foods or medicines containing these substances used as preservatives. The temperature intervals in which the mixtures in various proportions had the same refraction exponent as glass were recorded. The esters, freed of free *p*-hydroxybenzoic acid, obtained from a commercial preservative, Nipakombin A, were analyzed with the aid of this information, and were found to consist of 30% ethyl ester and 70% propyl ester. Accuracy of determination is highest with any mixture containing either the methyl or the benzyl ester, for these have higher coefficient of refraction than do the ethyl or propyl esters.—F. REIMERS. *Dansk Tids. Farm.*, 14 (1940), 219. (C. S. L.)

Molybdenum—Method for Determining very Small Quantities of, Applicable to Biological Materials. Ter Meulen studied the determination of molybdenum in vegetable products but used samples weighing 3 to 5 kilos. His method of basing the determination upon the color of colloidal molybdenum sulfide can serve to detect in 5 cc. of solution as little as 0.2 mg. of molybdenum per liter. Antimony tin and copper interfere. A procedure is described in detail for determining as little as 0.002 mg. of molybdenum, working on 100 Gm. of material. With 14 to 15 Gm. of ash as little as 0.001 to 0.002 mg. of molybdenum can be determined. Special pains should be taken during the calcination of the sample. Then, on treating the ash with hydrochloric acid and removing the silica, it is necessary to remember that some of the molybdenum is contained in the insoluble residue and it can be recovered by fusion with sodium bicarbonate. The sulfide test does not work well if much salt is present

and it is convenient to concentrate the molybdenum by forming a complex with cupferron and extracting it with chloroform. To remove the tin, careful neutralization with ammonia succeeds provided a little ferric iron is present. Copper is removed by precipitation with sodium sulfide. The final determination of molybdenum is made with the aid of a photoelectric colorimeter in a volume of less than 9 cc.—D. BERTRAND. *Bull. soc. chim. France*, 6 (1939), 1676–1689; through *Chimie & Industrie*, 43 (1940), 642. (A. P.-C.)

Neocupferron (Ammonium Salt of α-Nitroso-naphthylhydroxylamine)—New Organic Reagent in Chemical Analysis. A simple and inexpensive way of preparing neocupferron from α-nitronaphthalene, ammonia and hydrogen sulfide (these form α-naphthylhydroxylamine), ethyl nitrite and hydrochloric acid is described. The new reagent can be used to advantage for determining traces of iron and copper in mineral waters. In many respects the new reagent is like cupferron, but sometimes it succeeds where the older reagent does not. Typical results are given for the determination of about 5 mg. of iron per liter.—O. BAUDISCH and S. HOLMES. *Z. anal. Chem.*, 119 (1940), 241–245. (S. W. G.)

Organic Compounds—Detection of. Contrary to methanol, ethanol reacts at atmospheric temperature with 65% nitric acid with evolution of nitrous fumes, which permits of detecting it in presence of 9 parts of methanol. The reaction of glycerol with bichromate and nitric acid is not specific, and is also given by other alcohols, sugars, etc. Phenols can be identified by converting into sulfophthaleines by heating with *o*-sulfobenzoic anhydride. Hydrocyanic acid can be identified by the reaction $Hg + 2HCN = Hg(CN)_2 + 2H$, or by converting it into nitroprusside. Theophylline in ammoniacal solution reacts with thallium acetate to give light brown spherical crystals. The reaction of certain acids (ascorbic, etc.) and of *o*-dihydroxylated compounds with ferrous iron (blue color) should preferably be carried out after addition of calcium carbonate. The oxidizing action of ferric iron or organic acids can be established by detection of the resultant ferrous iron by means of dimethylglyoxime, dipyridyl or *o*-phenanthroline.—L. ROSENTHALER. *Pharm. Acta Helv.*, 14 (1939), 218–221; through *Chimie & Industrie*, 43 (1940), 641. (A. P.-C.)

Organic Solvents in Flavors—Determination of. In the application to flavoring extracts of the previously described method for the determination of isopropyl alcohol (*J. Assoc. Official Agr. Chem.*, 23 (1939), 594–596), as the latter is insoluble in the saturated sodium chloride generally used for salting out the flavoring ingredients, the sample is mixed with 10% sodium chloride solution and shaken with petroleum benzin; the separated alcohol layer is distilled and treated as previously described.—R. D. STANLEY. *J. Assoc. Official Agr. Chem.*, 23 (1940), 576–577. (A. P.-C.)

Phenarsazine Chloride—New Color Reaction for. Cândéa and Macovski's test gives with phenarsazine chloride (Adamsite) or oxide a fugitive red color. A modification of the test, dispensing with silver persulfate and using a larger amount of silver nitrate, gives a stable, greenish yellow color. The reagent giving best results is a mixture of equal volumes of 10% silver nitrate and glacial acetic acid. The limit of sensitivity lies between 0.02 and 0.04 mg. The reaction was tried on a score of arsenic compounds (substituted or unsubstituted mono- or diphenylchloroarsine, substituted or unsubstituted phenylarsine oxides, substituted or unsubstituted phenylarsinic acids, substituted or unsubstituted acridarsines, substituted or unsubstituted mono- or di-methylchloroarsines and -ethylchloroarsines, sodium cacodylate, arrhenal, β-chlorovinylchloroarsines). No color reaction was obtained in any case; the chloro-derivatives gave a curdled precipitate of silver chloride. Only *p*-nitrophenarsazine chloride gave a reaction similar to that of Adamsite; it dissolves to a yellow solution in acetic acid, but the color does not increase by heating even in presence of silver nitrate. The color is not due to diphenylamine, which is always present as an impurity in commercial Adamsite. For the detection of Adamsite in water, to 5 cc. of water add 0.25 Gm. of silver nitrate, shake to dissolve the salt, add 5 cc. of acetic acid and heat 10 minutes on the boiling water bath; the reaction is still perceptible at a dilution of 1:125,000.—J. DELGA. *J. pharm. chim.*, [9], 1 (1940), 73–76. (A. P.-C.)

Phenols—Extraction of, from Aqueous Solution. Phenols (*e. g.*, in waste liquors) are extracted by esters (the boiling point of which permits their subsequent distillation from the phenols) and, if desired, auxiliary solvents such as benzene or toluene. —I. G. FARBENIND. A.-G. Brit. pat. 520,198; through *J. Soc. Chem. Ind.*, 59 (1940), 574. (E. G. V.)

Potassium Permanganate Solutions—Standardization of. The method of preparing standard potassium permanganate involving acidifying an unboiled and unfiltered solution yields an unstable solution. Solutions prepared by the official A. O. A. C. method showed no significant change in strength after 4 months.—GEO. M. JOHNSON. *J. Assoc. Official Agr. Chem.*, 23 (1940), 543–546. (A. P.-C.)

Riboflavin—Assay of. A brief review of the literature on riboflavin assay is given. A colorimetric procedure (technique described in detail) which is a combination of the methods of Kharit and Khaustov (*Biochem. J.*, 29 (1935), 34) and of Emmerie, *et al.* (*Acta Brevia Neerland. Physiol. Pharmacol., Microbiol.*, 5 (1935), 77) was developed, and worked fairly well and gave results that appeared to be quite reliable. It consists essentially in extracting yeast or dried skim milk at 35° to 40° C. with 40% methanol containing 4% of acetic acid, filtering, oxidizing at room temperature with permanganate, destroying the excess of permanganate with hydrogen peroxide, and estimating riboflavin colorimetrically against 0.02% potassium chromate solution, or (if proper equipment and standards are available) photometrically of fluorometrically. A collaborative study of the method gave results that were high and that varied widely even in the hands of the same operator. The photoelectric photometer did not give much better results. It seemed to be the concensus of opinion of the collaborators that fluorometric procedures are likely to give better results than colorimetric. The results are considered to be promising and to indicate that a reliable chemical method for riboflavin assay is not an impossibility.—A. R. KEMMERER. *J. Assoc. Official Agr. Chem.*, 23 (1940), 346–351. (A. P.-C.)

Silicic Acid—Precipitation of, with Gelatins. The results of more than 200 experiments show that in many cases colloidal precipitates are favored by the addition of gelatin, glue or similar substances. Purified gelatin was not much better than the common product. Experiments with albumin, egg albumin, casein, hide powder, plant albumin, eaton (hydrolyzate of blood albumin), wheat gluten, hemoglobin, glutanic acid, betaine, molasses, vinasse, milk tragacanth, Irish moss, gum arabic, psyllium seed, coconut oil soap, agar agar, Nekal and castor bases showed no such effect in the cases of the last fourteen substances, and plant albumin was the only substance that was nearly as effective as gelatin. Molybdic, tungstic and titanic acids are also precipitated more readily in the presence of gelatin.

In the analysis of a silicate, such as feldspar, it is not necessary to evaporate the hydrochloric acid solution of the melt obtained by fusing 1 Gm. of the sample with soda and then go through the tedious filtrations and reëvaporation, but it is sufficient to add concentrated hydrochloric acid until about 20% hydrochloric acid is present, boil at least for five minutes, add more hydrochloric acid to replace that lost by evaporation, cool to 70° and add dropwise about 0.1 Gm. of gelatin or fish glue in 2% aqueous solution. The effect of the gelatin is probably a result of partly dehydrated silicic acid precipitating some of the gelatin upon it and thus causing enlargement of the particles so that filtration is easier. Excellent results were obtained in determining silicon in reduced iron. Gelatin also aids in the precipitation of calcium fluoride.—L. WEISS and H. SIEGER. *Z. anal. Chem.*, 119 (1940), 245–280. (S. W. G.)

Standard Solutions—Standardization of. A brief discussion of the reports of the Associate Referees on the standardization of sulfuric acid and potassium permanganate solution.—R. L. VANDAVEER. *J. Assoc. Official Agr. Chem.*, 23 (1940), 540–542. (A. P.-C.)

Sulfanilamide Derivatives—Quantitative Determination of. Electrometric titration curves were obtained for sulfanilamide, sulfapyridine, sulfathiazol and N-(*p*-aminobenzenesulfonyl)-acetamide (Albucid). All were ampholytes. Direct titration of Albucid could be done using 0.1*N* NaOH and phenolphthalein indicator. The acid dissociation constant of sulfapyridine was found to be $10^{-8.7}$, of sulfathiazole, $10^{-7.6}$. All four drugs could well be determined bromometrically. *Method:* 300 mg. of the drug are dissolved in 5 cc. dilute HCl and diluted with water to 100 cc. A 20-cc. aliquot is placed in a 500-cc. iodine flask and neutralized to phenolphthalein with normal NaOH. Then 30 cc. water and 20 cc. 0.1*N* $KBrO_3$ and 10 Gm. KBr and 5 cc. concentrated HCl are added and the flask stoppered after moistening the stopper with 10% KI solution. After allowing the proper bromination time (see below), 10 cc. of 10% KI solution and 250 cc. of water are added, and at the usual interval back-titration is done with 0.1*N* $Na_2S_2O_3$. Bromination times: for sulfanilamide, 5 minutes; for Albucid, 5 minutes; for sulfapyridine, 10 seconds (longer gives high results); for sulfathiazol, 2 hours. Factors: 1 cc. $KBrO_3$ = 0.004302 Gm. sulfanilamide, 0.005352 Gm. Albucid, 0.006228 Gm. sulfapyridine, 0.005352 Gm. sulfathiazole. The sulfathiazole forms a tribrom derivative and uses 6 atoms of Br, the others use 4 atoms per mole. Method for determination in blood by colorimetry: 2 cc. of blood is precipitated with 18 cc. of 4% *p*-toluenesulfonic acid, which was found better than trichloracetic acid. The blood is centrifuged 3 minutes at 3000 r. p. m. The supernatant fluid is poured off and filtered. To determine free sulfanilamide derivative: 5 cc. of clear filtrate, representing $^1/_2$ cc. of blood is neutralized to phenolphthalein, then 1 drop of 4% *p*-toluenesulfonic acid added to discharge the color. One cc. of *p*-dimethylaminobenzaldehyde reagent (3 Gm. of *p*-dimethylaminobenzaldehyde dissolved in 100 cc. of a solution of 7 cc. concentrated H_2SO_4 in 100 cc. water) is added and water to 10 cc. volume. The extinction is determined in a cell of 1.0 cm. depth in the Pulfrich photometer with filter S 45. The compensating cell contains reagent and water (1 + 9). Total (free + combined) sulfanilamide derivative is determined on another 5-cc. aliquot of the original filtrate. This is heated 2 hours on the steam bath, replacing evaporated water. After cooling, treatment is the same as above. Both *E* values are corrected by subtracting 0.04 for each cm. depth of cell. Standard curves are given for conversion of *E* values to mg. % in the diluted blood. This value is multiplied by 20 to give mg. per 100 cc. blood. Determination in urine can be done directly, without foreprecipitation. Here the urine is diluted 1 to 100. Only for determination of 5 mg. % or less in deeply colored urine is it necessary to determine the *E* of the urine's own color for correction.—I. S. NIELSEN and C. G. WOLFFBRANDT. *Dansk Tids. Farm.*, 14 (1940), 113. (C. S. L.)

Sulfuric Acid—Standardization of. Standardization of decinormal sulfuric acid by means of borax gave results that are believed to be correct and that agreed with those of Marshall's formula (*J. Soc. Chem. Ind.*, 18 (1899), 1091) for calculating % strength from specific gravity, based on the data of S. U. Pickering (*J. Chem. Soc., Trans.*, 57 (1890), 64–184).—W. H. KING. *J. Assoc. Official Agr. Chem.*, 23 (1940), 542–543. (A. P.-C.)

Thallium—Gravimetric Method for Determining. A finely crystalline precipitate of thallium cobaltic ammono chloride ($Tl[Co(NH_3)_6]Cl_6$) is obtained when a solution of cobaltic ammono chloride ($[Co(NH_3)_6]Cl_3$) is added to one of potassium thallium chloride dihydrate, $K_3TlCl_6.2H_2O$. The precipitate contains 35.35% of thallium. It is practically insoluble in dilute hydrochloric acid as well as in alcohol and ether. The results of twenty-six tabulated analyses of thallium carbonate, thallium chloride and thallium iodide showed an average deviation of 0.03% and greatest deviation of 0.1%. If the solution contains thallous salt add 0.5–1.0 Gm. of potassium chlorate, heat to boiling and add 4–6 cc. of concentrated hydrochloric acid. Boil a few minutes to clear the solution, then dilute with water to 100–400 cc., heat to boiling and add a slight excess of cobaltic ammono chloride solution. Cool, transfer the precipitate to a weighed filtering crucible with 2% hydrochloric acid and, after washing well with the 2% acid, wash 6–7 times with 96% alcohol and 4–5 times with ether. Dry 15–20 minutes in a vacuum desiccator and weigh. The procedure can be carried out in the presence of other elements except mercury, bismuth and lead. If silver is present, the solution must be freed from silver chloride by filtration. The quantity of hydrochloric acid can vary but an excess of chloride ions is necessary and the solution should not be over 2.4*N* in hydrochloric acid. The method is inexpensive and the reagent can be prepared as follows: To a concentrated solution of 100 Gm. cobaltous chloride hexahydrate and 30 Gm. of ammonium chloride add a solution of pure silver chloride in 20% aqueous ammonia, allow to stand for twenty-four hours at 40° or two days at room temperature and filter. Extract the filtered precipitate with water at 25°, heat on a water bath to 80° and add concentrated hydrochloric acid until a permanent precipitate is produced. Cool and recrystallize from water.—G. SPACU and A. POP. *Z. anal. Chem.*, 120 (1940), 322–326. (S. W. G.)

Theophylline Sodium Acetate—Assay of. The following procedure is recommended: Weigh out 0.5 Gm. of theophylline sodium acetate into a flat-bottomed porcelain dish (diameter 2.5 in.; height 0.5 in.); add about 3 cc. of 2 *N* sulfuric acid and 5 cc. of water and evaporate to dryness on a water bath. Repeat the evaporations with 5 cc. and 3 cc. of water. Transfer the residue to a beaker or flask by means of about 100 cc. of water; heat to dissolve, and to remove carbon dioxide, cool to about 40°, add 1 cc. of bromocresol purple indicator and *N* sodium hydroxide in very slight excess. Titrate with 0.1*N* sulfuric acid to the full yellow end-point. Add 20 cc. of 0.1*N* silver nitrate and titrate with 0.1*N* sodium hydroxide just to the full blue end-point. The method will determine theophylline accurately in the presence of caffeine. Theobro-

mine must be absent.—G. J. W. FERREY. *Quart. J. Pharm. Pharmacol.*, 13 (1940), 274–276. (S. W. G.)

Tin—Electrolytic Determination of, in Foil and in Tin-Plated Objects. The usual method for determining tin electrolytically calls for preliminary precipitation of iron and lead by alkaline sulfide, but this sulfide precipitation is unnecessary when, as is often the case, very little lead is present. In such instances take 100–200 sq. cm. of foil (0.2 Gm.), cut into small pieces, and heat slowly to boiling with 10% hydrochloric acid. Pour off the solution through a filter into a 250-cc. volumetric flask and repeat the treatment with hydrochloric acid, and, in some cases, repeat the treatment again. Wash with water, dilute to the mark and mix. To 125 cc. of the solution in a narrow beaker, add about 200 cc. of water, 16 Gm. of ammonium oxalate dissolved in hot water, and follow this with 30 cc. of concentrated hydrochloric acid. Electrolyze with a gauze cathode for six hours at 40–50° with a current of 1 ampere. Wash the deposited tin with water and finally alcohol, dry at 80° and weigh. Good results were obtained in the analysis of samples containing 0.25% of lead or less and about 1.7% of tin.—A. FOSCHINI. *Z. anal. Chem.*, 119 (1940), 281–286. (S. W. G.)

Titanium and Copper—Determination of. Basis and Application of Absolute Colorimetry. XIX. The hydrogen peroxide method for determining titanium was tested again. For determining 15–120 micrograms of titanium an "absolute layer depth" of 45.5 mm. was calculated and should be used for determining 0.015–0.120 mg. of titanium in 100 cc. of solution. For higher titanium concentrations a depth of 46.1 mm. should be used. The determination of copper by the ammonia and hydrogen sulfide methods was tested and a procedure given for determining copper in light metals and in baths used for the electrolytic plating of nickel. In the ammonia method the addition of a little sulfur dioxide to the ammoniacal tartrate solution often proved helpful. However, this seems to be ineffective in preventing the formation of colloidal manganese dioxide monohydrate when only copper and manganese are present, but when aluminum is also present as much as 5 mg. of manganese and 250 mg. of aluminum do no harm when sulfur dioxide is added together with sufficient tartaric acid. For some reason the extinction of solutions containing manganese but no aluminum is less than that of the same copper solutions without manganese. The sulfide method can be used for as much as 0.6 mg. of copper in a solution containing 6 cc. of 10% hydrochloric, 9 cc. of *M* sodium tartrate solution, 10 cc. of cold, saturated ammonium chloride (to increase the extinction), 2 cc. of 1% gelatin solution, 4 cc. of freshly saturated, perfectly clear hydrogen sulfide solution and water to make 100 cc. After thirty minutes this is measured under mercury light and filter QF 436 a. The determination of copper in the presence of considerable nickel is possible if a suitable quantity of sodium hyposulfite is added to reduce the cupric complex. Enough hyposulfite to reduce the copper to the free metal should be avoided.—A. THIEL and H. HEINRICH. *Z. anal. Chem.*, 120 (1940), 305–311. (S. W. G.)

Vanillin in Vanilla Extracts—Proposed Modification of the Official Colorimetric Method for Determining. In determining vanillin in concentrated vanilla extracts containing added vanillin by the official A. O. A. C. colorimetric method (originally developed by Folin and Dennis, *J. Ind. Eng. Chem.*, 4 (1912), 670) it was found impossible to recover a considerable portion of the vanillin actually added. As the vanillin is not lost, it would seem to be either precipitated by the lead solutions or occluded by or adsorbed on the precipitated lead resinate. It can be removed practically quantitatively by washing the lead precipitate, but this requires so much time and such a large volume of water that the method is impracticable. Solution of the problem lies in precipitation in a much more dilute solution to preclude, as far as possible, precipitation of the vanillin, as follows: transfer to a 100-cc. volumetric flask a quantity of the sample containing 8 to 12 mg. of vanillin, make to 100 cc. with water; transfer 5 cc. to a 50-cc. volumetric flask, add 0.2 cc. of lead solution (50 Gm. each of basic and neutral lead acetate per liter); into another 50-cc. volumetric flask pipette 5 cc. of standard vanillin solution (1 cc. = 0.1 mg. vanillin); into each flask pipet 5 cc. of the reagent, mix, after 5 minutes dilute to 50 cc. with saturated sodium carbonate solution, mix thoroughly, allow to stand at least 10 minutes so that the precipitate formed may separate completely, filter through dry filter papers, and compare the blue color of the clear solutions in a colorimeter. The method gave results about 60% higher than the official method on 3 samples of compound vanilla extracts.—H. J. LYNCH and NEULON DEAHL. *J. Assoc. Official Agr. Chem.*, 23 (1940), 429–431. (A. P.-C.)

Zinc—Electrolytic Determination of, on Brass-Gauze Electrodes. I. Deposition of Zinc from Solutions Buffered with Sodium Acetates. The deposition potential of zinc upon brass cathodes was —0.82 volt at pH 3.9–6.0. With zinc cathodes, the potential increased with rising pH. For complete deposition the pH should not be less than 6. To the solution containing 0.15–0.2 Gm. of zinc add sodium hydroxide solution until neutral. Add a solution of 5 cc. of 2 *N* acetic acid and 95 cc. of 0.2 *N* sodium acetate as buffer. Add 2–3 Gm. of sodium sulfate to 200 cc. of electrolyte to increase the conductivity. Using a brass-gauze electrode of about 55 sq. cm. surface, electrolyze at 20° with 0.7 ampere at about 6 volts between the terminals; all the zinc should be deposited in thirty minutes. Without turning off the current, remove the electrode from the bath, wash with water and ethanol, disconnect dry and weigh. This procedure was tested with chloride, acetate and sulfate solutions. **II. Separation of Zinc from Copper in Buffered Solutions.** The results of twenty-one determinations with solutions of known copper-zinc content show that in 0.2*N* solutions of sulfuric acid, it is possible to deposit all copper and no zinc and then, by buffering, all the zinc can be deposited as described above.—M. KARSCHULIN and S. BAN. *Z. anal. Chem.*, 120 (1940), 244–247; 248–252. (S. W. G.)

PHARMACOGNOSY

VEGETABLE DRUGS

Alstonia Constricta. A review of the botanical origin, chemical investigation, biological and pharmacological tests is given.—H. FINNEMORE. *Australasian J. Pharm.*, 22 (1941), 281. (A. C. DeD.)

Boerhaavia Diffusa Linn. and the White and Red Flowered "Varieties" of Trianthema Portulacastrum Linn.—Comparative Study of. Three drugs, namely: *Boerhaavia diffusa* Linn., and the white and red flowered "varieties" of *T. portulacastrum* Linn. are sold indiscriminately in the Indian market for the Ayurvedic drug, Punarnava, which is used as a specific for dropsy, beriberi, ascites, etc. The first-named drug belongs to the family, *Nyctaginaceæ*, and the latter two to the family, *Ficoideæ*. It is significant that comparative chemical study of authentic samples of the three plants has shown them to contain much the same active constituents (potassium nitrate and the

alkaloid punarnavine) in spite of their botanical differences.—R. N. CHOPRA, N. R. CHATTERJEE and S. GHOSH. *Indian J. Med. Research*, 28 (1940), 475–480. (W. T. S.)

Cinchona Barks—Capillary and Luminescence Analysis of. By comparison of analytical results it is shown that different sorts of cinchona bark can be definitely differentiated by capillary-luminescence analysis. Samples of bark of *Chinæ succirubræ* (from 3 sources), *Calisayæ regiæ* (from 2 sources), *Ledgerianæ*, ruber verus, flavus Barth. and Fuscus Loxa were ground to 484 mesh per sq. cm., dried at 105%° C., and macerated with 68.8% alcohol for days at room temperature to make a 1:10 tincture according to the Pharm. Asutria VIII. Strips of Durieux filter papers Nos. 120 and 121 (for luminescence analysis) and off Schleicher and Schüll No. 604 (for capillary analysis), 25 x 2 cm., were suspended from glass rods at 18° to 20° C., relative humidity 70% to 75%, for 24 hours in vessels 3 cm. x 6.5 cm. containing 5 cc. of tincture, so that the strips touched the bottom without touching the sides. The strips were then dried at room temperature in free air in a darkened room. The more alkaloids are present in the bark, the wider are the fluorescent bands obtained after capillary analysis of alcoholic and acid macerations. The dissimilarity of barks having the same name is shown not only in the luminescent part of the test strips but also in the portions of the strips containing non-alkaloidal components. More differentiations were revealed by ultraviolet light of uniform nature, unadmixed with visible light.—E. SVAGR and E. KINSKI. *Coll. Trav. Chim. Tchèques*, 11 (1939), 256–265; through *Chimie & Industrie*, 43 (1940), 585. (A. P.-C.)

Cocositol—New Source of. Cocositol, $C_6H_{12}O_6$, a polyhydric alcohol isomeric with inositol, has previously been obtained from the leaves of two plants of the family *Arecaceæ*. Manske now reports finding this alcohol in the leaves of *Calycanthus floridus* and *C. glaucus* in yields of 0.5%.—RICHARD H. F. MANSKE. *Can. J. Research B*, 19 (1941), 34–37. (W. T. S.)

Frangula and Purshiana Extracts—Fluorescence Analysis Studies of. The reaction for identity of frangula and purshiana extracts of the Swiss Pharm. V, indicated only that one was dealing with emodin containing preparations but did not differentiate them. Microscopic examination of the powdered barks in filtered ultraviolet light showed that with certain reagents, characteristic fluorescence colors could be obtained for the two drugs. In the present study, 0.05 Gm. of the dry extracts of the two drugs or 4–5 drops of the fluidextract were treated with 10 cc. of various solvents and the solution or suspension observed in ultraviolet light. The chloroform extract followed by treatment with barium hydroxide was found satisfactory. The method was modified by absorbing the chloroform extract in No. 604 paper (Schleicher and Schüll), drying and observing under the ultraviolet light. The capillary pictures in daylight showed no essential differences between frangula and purshiana but under ultraviolet light the two drugs could readily be distinguished. The difference can be enhanced by spraying the strips with 10 per cent barium hydroxide solution.—I. STEINER and K. LEUPIN. *Pharm. Acta Helv.*, 15 (1940), 145–149. (M. F. W. D.)

Fraxinus Species—Constituents of the Bark of. The author investigated the constituents of *Fraxinus Sambucia* Koidz, *F. pubinervis* Blume and *F. japonica* Blume and found that aesuletin was common to the various species of *Fraxinus*. Also isolated from *F. pubinervis* was fraxetin. Both fraxetin and aesculetin are hydroxycoumarins.—HARUYA SIMADA. *J. Pharm. Soc. Japan*, 60 (1940), 508–510 (in English, 200–201). (N. L.)

Japanese Alpinias—Pharmacognosy of Seeds of the. The Japanese drug Izu-Syuku-Sya is the seed of *Alpinia japonica* Miq. and has been used in medicine as an aromatic and stomachic. There are two other varieties of *Alpinia* which yield this drug, *Alpinia chinensis* Roscoe and *Alpinia speciosa* K. Schumann. A detailed account of the pharmacognosy and histology of the seeds of five varieties of *Alpinia* is presented. The author also gives in table form, the water, ash, ethereal oil and fat content of the seeds of a number of species of the *Alpinia* group.—YUSIRO KIMUA. *J. Pharm. Soc. Japan*, 60 (1940), (*Transactions*, in English, 113–117). (N. L.)

Medicinal Plants. A reminder that a country in time of war is dependent upon other countries for all kinds of vegetable materia medica. Why not grow plants in the home garden for their utility as well as for the brightness of their flowers?—ANON. *Chemistry and Industry*, 59 (1940), 614. (E. G. V.)

Mexican Drugs—Notes on. I. Zapote borracho (Lucuma Salicifolia, Kunth, Sapotac). This fruit, Zapote borracho, "drunk zapote," was found to contain neither glucosides nor alkaloids; there was tannin, starch, carbohydrates. It possesses no intoxicating properties. Probably its common name "drunk zapote" has reference to the musty alcoholic smell of the overripe fruit.—MARCEL BACHSTEZ and ALTAGRACIA ARAGON. *Jour. A. Ph. A.*, 30 (1941), 218. (Z. M. C.)

Poppies. Poppies are raised for commercial use in the Balkan States, Turkey and India. The chief product is the fruit, a capsule from which opium is extracted. Oil pressed from the seeds and flour made from the seeds are also important articles of commerce.—JULIO ENRIQUE CASTANEDA DE RANERO. *Escuela Farm.*, 3 (May–June 1940), 13. (G. S. G.)

Quina do Campo (Strychnos Pseudo-Quina, Saint Hilaire). The bark of this plant is frequently found substituted for quinine in commercial samples. Used in place of quinine or to adulterate, it has a much less bitter taste, but also lacks the therapeutic value of quinine. Its pharmacognosy is given in detail. Chemical analysis reveals no alkaloids or glucosides. It is recommended that it be deleted from the Pharmacopœia of Brazil and its sale prohibited as a substitute for true quinine or for any therapeutic use.—HENRIQUE LUIZ LACOMBE. *Trib. Farm., Parana*, 8 (1940), 97. (G. S. G.)

"Rasaut" and "Hing"—Chemical Analysis of. "Rasaut," a native drug occurring as a brown, semisolid extract, contains berberine as its active constituent. It is used in eye inflammations and as a bitter. An analysis of 15 samples from the Punjab market showed half of them to be grossly adulterated. "Hing," a native drug of the *Ferula* genus, is sold in India as a household remedy and a condiment. Of 13 samples examined, only 2 contained over 50% of alcohol-soluble resin indicating adulteration.—K. S. GREWAL and B. D. KOCHHAR. *Indian J. Med. Research*, 28 (1940), 463–468. (W. T. S.)

Resins—Test for. The purgative action of the convolvulaceous drugs (ipomoea, jalap and scammony) is regarded by the author as being due to the resins which they contain. Neither the Pharmacopœia nor the Codex includes tests of identity for the resins, but under jalapin the Codex describes certain reactions. The reduction of ammoniacal silver nitrate, without previous hydrolysis, and of Fehlings solution, after mineral acid hydrolysis, are typical reactions. These reactions may be regarded as general tests for reducing sugars, and it was considered desirable to find a more specific reaction.

From a consideration of the suggested chemical structure of the resins it was thought that the production of methyl-pentose as a hydrolysis product might serve as a basis of an identity test. Experiments were performed and the action of concentrated hydrochloric acid on an acetone solution of the resin adopted. A positive reaction was also obtained with two mils of an acetone tincture (10 per cent w/v) of jalap tuber, ipomoea root and turpeth root. With kaladana seed a positive reaction was not obtained, the color produced being deep orange-red without the characteristic purplish shade exhibited by the other drugs.—E. J. SCHORN. *Chemist and Druggist*, 134 (1941), 336. (A. C. DeD.)

Sarsaparillas. Pharmacognostic study of several sarsaparillas of Vera Cruz, Jamaica and Honduras compared to that of Parana. Photomicrographs of its structure are given. Chemically the existence of saponins or saponisides varies in these several samples.—NARCISO SOARES DA CUNHA. *Trib. Farm., Parana*, 8 (1940), 105. (G. S. G.)

Zea Mays Stigmas—Constituents of the Petroleum Extract of. Reference is made to other investigations devoted to the constituents of certain corn products. Investigation of the petroleum extract of *Zea mays* stigmas has led to the isolation of a saturated hydrocarbon, m. p. 65°, and a phytosterol mixture from which stigmasterol has been separated, and in which ergosterol has been detected by color reactions. The presence of a water-soluble acid claimed by other workers has not been confirmed.—A. ZAKI and G. SOLIMAN. *J. Chem. Soc.*, (1940), 1545–1547. (W. T. S.)

PHARMACY

GALENICAL

Calcium Gluconate Solutions—Stabilization of, by Camphorsulfonic Acid. Supersaturated (*e. g.*, 10%) solutions of calcium gluconate can be prepared by addition of 1% of camphorsulfonic acid.—G. LUSIGNANI. *Boll. chim.-farm.*, 79 (1940), 137–138; through *J. Soc. Chem. Ind.*, 59 (1940), 565. (E. G. V.)

Elixir of Thiamine Hydrochloride and Its Stability in Combinations. The pharmacy of thiamine hydrochloride is reviewed. Experiments were conducted to ascertain if the prescribing of sodium bromide, phenobarbital and phenobarbital sodium in elixir of thiamine hydrochloride proposed for N. F. VII was rational. Samples were prepared containing 1 dram and one and a half drams of sodium bromide per fluidounce of the elixir and 24 grains of phenobarbital and 24 grains of phenobarbital sodium per fluidounce. The samples were observed over a period of approximately sixty days and the pH determined at intervals and any precipitations noted. Sodium bromide apparently had no effect on the stability of the elixir. Combinations with phenobarbital and its soluble form are not stable as there is a tendency for the hypnotic to crystallize out even upon the addition of alcohol and a precipitate may be formed by a possible decomposition of the vitamin due to pH changes. A table shows that the proposed elixir may be made by the pharmacist at a price considerably lower than ten advertised products, thus lowering the cost of medication of this type.—HENRY M. BURLAGE. *Carolina J. Pharm.*, 22 (1941), 130–132, 138–139. (H. M. B.)

Ether—Effect of Alkalinity or Acidity on the Stability of. U. S. P. anesthetic ether must be free from aldehyde and peroxide but experiments have been made also to see whether alkalinity or acidity had any bearing on stability. It was determined that faintly alklaine ethers stored in copper-free containers are superior in stability to the regular U. S. P. ether or those which are faintly acid. Ethers made alkaline with ammonia are superior to those made alkaline with other substances. Ammoniated ethers are more stable in copper-lined cans under adverse storage conditions than in other types of containers studied. The protective effect of tinned-iron containers is more easily demonstrated than that of copper-lined containers because of the difficulty of causing an appreciable aldehyde development even under very bad storage conditions when ether is stored in copper.—A. W. BERRY and E. S. HERLONG. *Jour. A. Ph. A.*, 30 (1941), 73. (Z. M. C.)

Ferrous Sulfate Solutions—Effect of Certain Agents on the Stability of. The results indicate that hypophosphorous and citric acids are inefficient in stabilizing the ferrous sulfate solutions in the concentrations used. Technical dextrose and technical dextrose with hypophosphorous acid seemed to be quite efficient in stabilizing ferrous sulfate solutions that were exposed to air at room temperature over a period of six months. These results are in general agreement with the results of H. W. Tomski and L. J. Waller.—C. L. HUYCK. *Am. J. Pharm.*, 113 (1941), 189. (A. C. DeD.)

Galenical Preparations—Study of, VIII. Deterioration of Galenical Preparations. *Spiritus sinapis* prepared with isopropyl alcohol should form the allyl thiocarbamic acid esters. Over a period of one year, there was a 22 per cent reduction in the amount of mustard oil present. *Tinctura Sabadillæ acetosa* is made with a menstruum of acetic acid, alcohol and water and in the course of 225 days the amount of acetic acid fell from 3.87 per cent to 1.54 per cent. Sucrose syrups hydrolyze especially in the presence of acids. A study of syrup of citric acid indicated a rapid and extensive inversion of the sucrose in a period of 200 days as measured in terms of optical rotation. Syrup of calcium lactophosphate underwent a slower inversion during the course of 260 days and even simple syrup showed a surprisingly large amount of inversion. Eight plasters which had been kept for 10 years under the usual drug store conditions were investigated and reported upon.—L. ROSENTHALER. *Pharm. Acta Helv.*, 15 (1940), 210–213. (M. F. W. D.)

Hexylresorcinol—Dispensing. Improved composition of tablets of hexylresorcinol was sought. The presence of alum was found to prevent discoloration during preparation. Alcohol-benzol mixture was found a good granulating fluid. Tablets of 0.1 Gm. hexylresorcinol were made as follows: I. Chocolate Coated: For 100 tablets, the hexylresorcinol, 10 Gm., was mixed with 1 Gm. of dry aluminium potassium sulfate and 3 Gm. of powdered glucose, moistened with 5–7 Gm. of a mixture of 10 Gm. absolute alcohol and 90 Gm. rectified benzol and granulated through sieve 15. After drying below 50° C., the granules were mixed with 4 Gm. powdered agar and 2 Gm. talcum. Tablets of 20 Cg. weight were punched with 6.5-mm. punch and coated with cacoa. These opened up in 10 minutes in warm water. II. Or the granules as made above were mixed only with talcum after drying and 16 Cg. tablets punched with 9-mm. punch. These dissolved in about 5 minutes in stirred, warm water.—E. P. NIELSEN. *Arch. Pharm. Chemi*, 47 (1940) 753. (C. S. L.)

Magnesium Hydroxide—Production of Medicinal Preparations of. Claim is made for dry preparations containing (*a*) magnesium hydroxide 5–45, magnesium citrate 5–25, sodium citrate 2–15, effervescent salt 20–60 and sugar 30–70 %; or (*b*) magnesium hydroxide 5–50, magnesium citrate 5–45, sodium citrate 2–15, magnesium sulfate ($7H_2O$) 1–7 and sugar 30–70%. In all cases the magnesium hydroxide is suspended in a solution of sugar and the mixture evaporated to dryness. Effervescent salt consists of sodium bicarbonate and tartaric or citric acid.—C. H. PHILLIPS CHEM. CO. Brit. pat. 524,756; through *J. Soc. Chem. Ind.*, 59 (1940), 897. (E. G. V.)