# PHARMACEUTICAL ABSTRACTS

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## **CHEMISTRY**

# BIOCHEMISTRY (Continued)

Lead—Determination of, in Biological Materials. Comparative data for determinations of lead in biological material by means of a spectrograph, a dithizone and a chemical separatory (carbazide) method have been given. The specific field of usefulness of each method has been defined as follows: In dealing with the general run of biological samples in which the quantity of lead present exceeds 1 gamma, the spectrographic or dithizone method may be used with equal assurance of the reliability of the results. The spectrographic method is superior in dealing with quantities of lead less than 1 gamma. The chemical separatory (carbazide) method is chiefly useful in the case of large samples, and is satisfactory when the lead content of the samples is such that the loss inherent in the method (approximately 0.07 mg. per sample) is insignificant in either a chemical or a physiological sense.—J. Cholak, D. M. Hubbard, R. R. McNarry and R. V. Story. Ind. Eng. Chem., Anal. Ed., 9 (1937), 488.

Lead—Determination of. Photometric Dithizone Method as Applied to Certain Biological Materials. Directions for determining 0 to 100 gamma of lead by means of its dithizone compound are given. The final determination is based upon color measurements in a neutral wedge photometer. After removal of organic matter by ashing or otherwise, an aliquot part of the solution is treated with ammonium citrate and phenol red. By means of redistilled, concentrated ammonium hydroxide the solution is neutralized to  $p_{\rm H}$  7.5 after the addition of potassium cyanide solution. It is shaken with dithizone in chloroform and the latter is removed repeatedly until finally no more of the colored lead compound can be obtained. Then the aqueous phase containing most of the extraneous salts is discarded. The chloroform solution is shaken with 20 cc. of 0.1N nitric acid to decompose the complex lead salt and get the lead into an aqueous phase again. From this a second extraction with dithizone in chloroform is made under slightly different conditions. If bismuth is present it must be removed at  $p_{\rm H}$  2. This second solution of lead compound in chloroform is washed with dilute nitric acid and a third extraction made with dithizone in chloroform. In this way a lead compound of high purity is prepared. The chloroform used can be purified, and used again after recovery.—D. M. Hubbard. Ind. Eng. Chem., Anal. Ed., 9 (1937), 493–495.

Lipoid-Like Compounds in the Testes of Swine. Four new compounds were isolated from the lipoid fraction of swine testes. (1) Testalolone,  $C_{21}H_{32}O_5$ , a neutral, saturated compound, giving the characteristic sterol reaction with sulfuric acid, is physiologically inactive. (2) A "substance B,"  $C_{19}H_{88}O_5$ , which gives palmitic acid on saponification. (3) Testriol,  $C_{19}H_{40}O_3$ , a compound containing three hydroxyl groups. (4) A "substance D,"  $C_{24}H_{38}O$  or  $C_{24}H_{36}O$  or  $C_{34}H_{36}O$  or  $C_{34$ 

Monosaccharides, Disaccharides and Furfural—Determination of. Experiments are described with arabinose, d-glucose, fructose, cane sugar, lactose, maltose and furfuraldehyde. In all cases the aqueous solution was treated with Nessler's reagent and sodium carbonate. On boiling, mercury, equivalent to the reducing power of the sample, was precipitated and the analysis based upon the volume of iodine solution required to oxidize the precipitated mercury back to mercuric salt. The equivalent weights were: arabinose 0.5M, d-glucose 0.25M, fructose 0.2M, cane sugar 0.111M, lactose 0.2M and furfural 0.2M, where M is the molecular weight.—M. Goswami and B. C. Das-Purkayashta. J. Indian Chem. Soc., 13 (1936), 315-322; through Chimie & Industrie, 38 (1937), 33.

Morphine—Detection of, in Urine. Fifty cubic centimeters of urine are acidified with tartaric acid and evaporated to a syrup on a water-bath, 5 to 6 Gm. of clean dry sand is added and evaporation continued to dryness. The dry residue is extracted three times with 30 cc. of warm alcohol (95%); the liquid is filtered, and the solvent evaporated. The residue is dissolved in 25 cc. of water, transferred to a separator and neutralized with 25% sodium hydroxide solution. One cubic centimeter of phosphoric acid (85%) is added. The mixture is then shaken out with 10 cc. of amyl alcohol to remove colored impurities. The aqueous portion is separated, made alkaline with ammonia, and shaken out twice with 10 and 10 cc. of amyl alcohol. The amyl alcohol extract is then shaken out twice with 10 and 10 cc. of phosphoric acid, 3%. The acid solution is separated.

rated, made alkaline with ammonia, and shaken out with chloroform-alcohol mixture (9:1). The residue from the chloroform-alcohol extract after evaporating the solvent is tested for morphine with the Froehde, Froehde-Buckingham or Marquis reagent. It is claimed that this method will detect 0.005 mg. of morphine in 50 cc. of urine.—C. K. Liang. China Med. J., 211 (1937), page xx; through Brit. Med. J. Epit., 1 (1937), 92; through Quart. J. Pharm. Pharmacol., 10 (1937), 577. (S. W. G.)

Oxalic Acid—Direct Determination of, in Blood. Thomsen's criticism (Z. Physiol. Chem., 237 (1935), 199-213) of Izumi's (Japan. J. Med. Sci., 2 (1933), 195-204) cerium precipitation method for oxalates is not valid because of failure to use the special centrifuging tubes required. The effect of excess of cerium chloride can be overcome.—S. Suzuki. Hoppe-Seyler's Z. physiol. Chemie, 244 (1936), 235-237; through Chimie & Industrie, 38 (1937), 453. (A. P.-C.)

Oxydasis—Leucocytic Action of Irradiation with Ultraviolet Rays upon the Granulation of. A considerable increase of leucocytic oxydasis is produced in white rats and guinea pigs, by irradiation with ultraviolet rays.—P. CARNIELLI. *Biochim. terap. sper.*, 16 (1938), 1. (A. C. DeD.)

Parathormone—Action of. III. The hypothesis that parathyroid hormone acts directly on the kidneys to produce an increase of phosphate excretion is confirmed by the findings in six cases of acute nephritis to whom daily intramuscular injections of sixty units of the hormone were given. Such treatment produced a much smaller phosphate diuresis during the disease than after the recovery in these patients.—Hector K. Goadby. Biochem. J., 31 (1937), 1530; through Squibb Abstr. Bull., 10 (1937), A-1971. (F. J. S.)

pH of Human Blood Plasma in Respiratory and Cardiac Disease. The  $p_H$  of twelve healthy controls was found to vary from 7.39 to 7.43, with an average of 7.40. The p<sub>H</sub> of 150 patients varied from 7.31 to 7.58, with an average of 7.416. These  $p_{\rm H}$  values have a much wider range than the normals but the numbers investigated are not comparable. Little variation was noted in the few cases where repeated p<sub>H</sub> estimations were made. In mitral and aortic disease an increased acidity was found in the cyanotic group as compared with the non-cyanotic group. This difference was not observed in the cases of cardiovascular degeneration. The average  $p_{\rm H}$  values in pulmonary tuberculosis were somewhat higher than in healthy controls, but were lower than those of chronic pulmonary suppuration. In cases of pyrexia, irrespective of cause, there was a tendency toward alkalinity. While in a few cases wide variations in the  $p_H$  value of the blood were observed it appears that, unless there is a gross interference with the hemo-respiratory exchanges, the normal buffers of the blood are adequate to deal with changes in the acid-base equilibrium. The increase of acidity in mitral disease associated with cyanosis—a condition usually regarded as inimical to tuberculosis—is of possible significance.—C. Shiskin. Lancet, 233 (1937), 1191.W. H. H.)

Sugars in Champagne Production. A study was made of the rôle of a number of commercial sugars in the making of champagne. When used as the fermentable substance in the secondary fermentation, the commercial sugars tested were equally fermented, and it was impossible on a "blind" test to tell one lot from another. When these same sugars were used as the sweetening agents in making up the dosage, a difference could be detected in the finished champagne. There was no objectionable flavor in any case, but certain lots lacked the body and fullness of flavor found in a good commercial champagne. There was a considerable difference in the amount of sugar necessary to produce equal sweetness in the various lots.—H. E. GORESLINE and F. M. CHAMPLIN. Ind. Eng. Chem., 30 (1938), 112.

Sugars—Reducing, Application of the Reduction of Silver Nitrate by Cuprous Oxide to the Determination of. In neutral solution cuprous oxide forms a black bulky precipitate with silver nitrate; the solution is acidified with normal sulfuric acid, silver in "tree" form is soon precipitated according to the quantitative ratio Cu<sub>2</sub>O:2AgNO<sub>5</sub>:2Ag. This reaction can be used for determining reducing sugars by the Bertrand method after cuprous oxide has been precipitated. Filter the supernatant liquid in the reduction flask through porous porcelain, wash the cuprous oxide with boiled water, decant the washings upon the filter, then adjust for filtering by suction. Place most of 20 cc. of decinormal silver nitrate into the flask, the rest on the filter, similarly divide 5 cc. of normal sulfuric acid and agitate for a few minutes. When reduction to silver is complete, filter all of the silver nitrate solution and wash well 2 or 3 times. To the filtrate add 2 to 3 cc. of concentrated nitric acid, 2 cc. of 10% ferric alum solution, and titrate with decinormal ammonium thiocyanate solution. Each cc. of decinormal silver nitrate indicates 0.00636 Gm. of copper; the

relation of copper to sugar is given in Bertrand's table. The method cannot be used in the presence of notable amounts of chlorides.—V. HARLAY. J. pharm. chim., 23 (1936), 589-594; through Chimie & Industrie, 38 (1937), 449.

(A. P.-C.)

Urease—New Sources of, for Determination of Urea. The seed of the watermelon (Citrullus vulgaris) is shown to be a potent source of urease suitable for use in urea determinations. Watermelon urease is, for this purpose, distinctly superior to soy bean and jack bean preparations because it determines urea quantitatively even in such materials as whole blood and liver which give abnormally high values when the latter sources of urease are used. Using jack bean or soy bean with ox blood and liver it is shown that abnormal values are obtained even under standard experimental conditions while on increasing either the time of action or the concentration of the enzyme, the errors are considerably aggravated. Such effects are found to be negligible with watermelon seeds.—Manayath Damodaran and Palghat M. Sivaramakrishnan. Biochem. J., 31 (1937), 1041; through Squibb Abstr. Bull., 10 (1937), A-1907. (F. J. S.)

Ureometer—Simple. A description of a simple glass apparatus without stop-cocks, which is suitable for determining urea in 1.25 cc. of blood or in 0.5 cc. of urine by the sodium hypobromite method.—R. VLADESCO. Compt. rend. soc. biol., 123 (1936), 849-850; through Chimie & Industrie, 38 (1937), 451.

(A. P.-C.)

Urine—Preparation of Sediments as an Analytical Record for Microscopic Examination of. A detailed procedure with special emphasis on the treatment of samples to determine various cylinders, fatty constituents, filaments, epithelial cells and leucocytes in sediments is offered.—Paul Schugt. Apoth. Ztg., 52 (1937), 1488-1490. (H. M. B.)

Urine Coloration as Diagnostic Aid. Red urine with an acid reaction often indicates a feverish disease. In reddish urines it is advisable to look for pyrazolones, anthraquinones, trional or sulfonal. A brown or blackish brown acid urine can contain quinine, phenols, resorcinol, pyrogallol, thymol, naphthalene. In green or yellow acid urines it is advisable to look for bile colors, santonin, methylene blue, arbutin, anthraquinones. Red alkaline urine can indicate the presence of anthraquinones, phenolphthalein, santonin.—J. Gutschmidt. *Pharm.-Ztg.*, 82 (1937), No. 5, 61-62; through *Chimie & Industrie*, 38 (1937), 868. (A. P.-C.)

Vitamin Absorption and Liquid Paraffin. It has been shown that if rats are fed a preparation consisting of a solution or a suspension of vitamins A and D in liquid paraffin, and another batch of rats is fed with a similar preparation in which the liquid paraffin is replaced by a fixed vegetable oil, then the latter rats will thrive better than the first. The reason put forward is that the vegetable oil is absorbed through the intestinal wall, whereas the liquid paraffin is not absorbed. In the case, however, of therapeutic quantities of liquid paraffin taken by a patient, it is most unlikely that sufficient coating of the intestinal wall with liquid petrolatum will take place to prevent the absorption of the vitamins. It may be stated, for example, that a patient taking liquid paraffin regularly would not be able to absorb mineral salts and so would develop a state of deficiency of the ordinary inorganic salts which are necessary to health.—Anon. Pharm. J., 139 (1937), 668.

Vitamin-Antirachitic, from Bluefin Tuna Liver Oil. According to the previous method (cf. S. A. B., 9 (1936), 1153), the antirachitic vitamin was isolated as the 3,5-dinitrobenzoyl compound (I), m. p. 128-129°, from five Kg. Japanese bluefin tuna liver oil. I gave no m. p. depression with the 3,5-dinitrobenzoyl compound (II) of vitamin D<sub>8</sub>, and had the same empiric formula as II,  $C_{34}H_{46}O_6N_2$ . For I and II, respectively,  $\alpha_D^{20} = +98.5^{\circ}$  and  $+97.4^{\circ}$ . The vitamin obtained in oily form from I showed an antirachitic potency in rats of 35,000 international units. This value was somewhat lower than the 40,000 units obtained by Schenck (cf. S. A. B., 10 (1937), 718) for crystalline vitamin D<sub>3</sub>; the discrepancy may be due to contamination products in the oily vitamin preparation of B. and B. It is concluded that the antirachitic vitamin of bluefin tuna liver oil, like that of tuna fish- and halibut liver oils, is identical with vitamin  $D_3$ . The preliminary purification of the unsaponifiable portion was simplified by omission of the second distribution between benzine and 95% methyl alcohol. The product, however, was not sufficiently pure when this was done, so that it was found necessary to put repeated extraction of the benzine solution residue with cold 99% methyl alcohol in place of the second distribution.—HANS BROCKMANN and Anneliese Busse. Z. physiol. Chem., 249 (1937), 176; through Squibb Abstr. Bull., 10 (1937), A-1943. (F. J. S.)

Vitamin A and B Contents of Cod Liver Oils. Of 42 commercial samples of cod liver oil the iodine numbers varied from 85.4 to 149.5; iodine number alone is not characteristic of the quality of cod liver oil. Vitamin A varied from zero to 6800 I. U. per Gm. vitamin D from zero to 187.5 I. U. per Gm. Cod liver oil for pharmaceutical purposes should contain at least 800.0 I. U. vitamin A and at least 85.0 I. U. vitamin D per cc. and should have an iodine number of 140-160. Cod liver oils for feeding purposes should at least contain 40 I. U. vitamin D per cc.—Jenö Becker. Mezőgazdasági Kutatások, 10 (1937), 247-254; through Chem. Abstr., 32 (1938), 1863. (F. J. S.)

Vitamin  $B_1$ —Determination of, in Human Urine. For the determination of vitamin  $B_1$  in urine, the vitamin  $B_1$  is absorbed on frankonite and the adsorbate subjected to the fluorometric thiochrome test of Karrer and Kuble (*Helv. Chim. Acta.*, 20 (1937), 369). Amounts of 3-5 $\gamma$  of vitamin  $B_1$  per 100 cc. of urine can be determined quantitatively with sufficient certainty. In a determined case, under normal diet,  $97\gamma$  of vitamin  $B_1$  was eliminated in 24 hours. In oral charging with large amounts of vitamin  $B_1$  (3 × 20 and 3 × 40 mg.) only about 3-5% was eliminated in the urine; the greater the charging the slighter the percentage elimination. A slight temporary retention of vitamin  $B_1$  in the body was established. Digestive ferments do not destroy vitamin  $B_1$ —Walter Karrer. *Helv. Chim. Acta.*, 20 (1937), 1147. (G. W. H.)

Vitamin B<sub>1</sub>—Difficulties in the Use of Brachycardia Method of Assaying. The heart rate observed by electrocardiograph showed in the different parts of the same reading variations up to 100 beats per minute. The results obtained by the method are absolutely inconsistent.—ELIZABETH CHANT ROBERTSON and M. ELIZABETH DOYLE. Proc. Soc. Exptl. Biol. Med., 37 (1937), 139.

(A. E. M.)

Vitamin B<sub>1</sub> Tests—Use of Yeast or Other Fungi for. R. points out that although vitamin B<sub>1</sub> has a great influence on the growth of certain yeasts, these fungi cannot be used for assaying the vitamin because there is another fraction not possessing high vitamin B<sub>1</sub> activity which is twice as potent as B<sub>1</sub> in stimulating yeast growth (cf. S. A. B., 3 (1930), 724).—ROGER J. WILLIAMS. Science, 86 (1937), 349; through Squibb Abstr. Bull., 10 (1937), A-1985. (F. J. S.)

Vitamin C Content—True, Chemical Determination of. Certain plant tissues contain an agent, oxidase, which can transmute ascorbic acid into an oxidized form. Determination of the reducing capacity before and after the action of pumpkin extract makes it possible to determine whether the reducing action is due to ascorbic acid or not. The usual titration of human urine with dichlorophenolindophenol almost regularly yields too high values; the true quantity of reduced ascorbic acid is 5 to 20 mg. per liter. Preliminary removal of reducing substances with lead or mercuric acetate gave low values for vitamin C. On the basis of results the authors do not recommend the iodine or methylene blue titration methods, or the tungstic acid, phosphotungstic acid and potassium ferricyanide colorimetric methods.—P. E. SIMOLA, S. JALAS and EVA YLINEN. Suomen Kem. (B), 9 (1936), 23-24; through Chimie & Industrie, 38 (1937), 452. (A. P.-C.)

Vitamin C—Determination of. It is extremely difficult to determine ascorbic acid in urine by Bezssonoff's method (Bull. soc. chim. biol., 16 (1934), 1160-1175) because of interference by uric acid. The following modification is proposed: To 5 cc. of the liquid add 2.5 cc. of a solution containing 100 Gm. of neutral lead acetate and 120 cc. of acetic acid per liter; after 2 minutes add 2.5 cc. of 2.5% sulfuric acid by volume, let stand 1 minute, filter and examine the filtrate in the Pulfrich-Zeiss colorimeter.—N. Bezssonoff and Mme. V. Woloszyn. Compt. rend. soc. biol., 124 (1937), 353-355; through Chimie & Industrie, 38 (1937), 869. (A. P.-C.)

Vitamin C—Excretion of, in Sweat. Miners working in the Witwatersrand gold mines at a temperature of 96-97° F. excreted in the sweat 0.5-1.1 mg. of vitamin C per 100 cc. or about 2 mg. per hour. This may account for the relative frequency of scurvy or subscurvy among these miners.—R. E. Bernstein. Nature, 140 (1937), 684-685; through Chem. Abstr., 32 (1938), 626.

(F. J. S.)

Vitamin C—Phenomena Which Accompany the Oxidation of. The spontaneous oxidation of ascorbic acid by atmospheric oxygen under various conditions was studied. There is some evidence of the formation of a compound,  $(C_6H_7O_6)_2$ , intermediate between ascorbic acid and dehydroascorbic acid, although the compound was not isolated.—N. Bezssonoff and Mme. L. Woloszyn. Compl. rend. soc. biol., 122 (1936), 941-944; through Chimie & Industrie, 38 (1937), 527.

(A. P.-C.)

Vitamin C—Requirements of Adults. Four schizophrenic but physically healthy adult patients were selected for these experiments, after ascertaining that their gastric juices contained

free hydrochloric acid. They remained for about six months on a varied diet calculated to provide an average of only 2 mg. ascorbic acid daily. During three-week periods a fixed amount of synthetic ascorbic acid was given daily, the amount being increased in successive periods. At the end of each period both arms of each patient were subjected to an overpressure in the veins of 35 mm. and the petechiæ resulting from capillary fragility counted. On the following day the extra petechiæ caused by 50-mm. overpressure were counted. The "petechial index" (the number caused by 50 mm. plus twice the number caused by 35-mm. overpressure) was plotted against the dose of ascorbic acid. The index rose during the smaller dosage-levels, while gingivitis appeared, then the index began to fall again. The level at which the index returned to the figure normal for the individual when receiving a mixed diet generous in ascorbic acid was taken as the minimum requirement of ascorbic acid for that individual. The values varied from 24 to 35 mg, per day or from 0.39 to 0.48 mg. per day per Kg. of body weight for the four test patients. In planning a dietary, and still more in therapeutic treatment, the amount of ascorbic acid given should obviously be more than this indispensable minimum. Children living in a district within the Polar Circle were examined by the capillary test and 6 out of 27 gave an index indicating that their diet contained less than the indispensable minimum of ascorbic acid.—G. F. Gothlin, E. Frisell and N. Rund-QUIST. Acta med. Scand., 92 (1937), 1; through Quart. J. Pharm. Pharmacol., 10 (1937), 573. (S. W. G.)

Vitamin C—Reversible Oxidation of, Demonstrated by Experiments with Guinea Pigs. Guinea pigs on ascorbutic diet made nearly normal gains in weight when given lemon juice or ascorbic acid solution which had been treated with enough dichlorophenolindophenol or iodine to convert the ascorbic acid to a reversibly oxidized form.—N. Bezssonoff and Mme. M. Woloszyn. Compt. rend. soc. biol., 122 (1936), 944-946; through Chimie & Industrie, 38 (1937), 527-528.

(A. P.-C.)

Vitamin C—Stabilization of, by Adrenalin. Vitamin C is protected from autoxidation by adrenalin. dl-Adrenalin is half as effective as l-adrenalin. The inhibition of oxidation increases with the concentration of adrenalin.—M. Yamamoto. Hoppe-Seyler's Z. physiol. Chemie, 243 (1936), 266–269; through Chimie & Industrie, 38 (1937), 936–937. (A. P.-C.)

Vitamin C in Vegetables. Ascorbic acid occurs exclusively in the reduced state in fresh vegetables, but during the process of extraction for titrimetric estimation, oxidation may occur. Although the dehydroascorbic acid formed can be reduced again by hydrogen sulfide, this must be done promptly if further decomposition is to be avoided. The oxidation is catalyzed by copper and by an oxidase present in most vegetables. The effect of copper can be inhibited by 2% of metaphosphoric acid in the extracting solution. The activity of the enzyme is maximal at  $p_{\rm H}$  5.5, but is completely inhibited by strong acid. If therefore an acid ionized sufficiently to inhibit this enzyme action were used for the extraction, the necessity for reduction should be avoided. This expectation was entirely confirmed. With string bean, cabbage, spinach, pea and carrot the ascorbic acid determined by direct titration increased regularly to a maximum as the pn of the extracting solution increased to about  $p_H 1$  irrespective of the nature of the acid used. Five per cent sulfuric acid plus 2\% metaphosphoric acid (\rho\_H 0.8) gave the highest values, which could not be increased by reduction. If hydrogen sulfide reduction is used, the time of treatment should not exceed thirty minutes to avoid reduction of other substances, although this interference is also minimized by the use of a strongly ionized acid.—G. L. MACK and D. K. TRESSLER. J. Biol. Chem., 118 (1937), 735; through Quart. J. Pharm. Pharmacol., 10 (1937), 573. (S. W. G.)

Vitamins—Knowledge of. A continuation of a review dealing especially with vitamin D.—A. Richard Bliss, Jr. Drug and Cosmetic Ind., 41 (1937), 636-638, 651. (H. M. B.)

Vitamins—Knowledge of. A discussion dealing especially with vitamin E.—Anon. Drug and Cosmetic Ind., 41 (1937), 762-763. (H. M. B.)

Vitamins—Knowledge of. The conclusion of a series of articles dealing specifically with vitamins F, H, K and P.—A. RICHARD BLISS, JR. Drug and Cosmetic Ind., 42 (1938), 41–42.

(H. M. B.)

### ANALYTICAL

Allyl Compounds—Brometric Determination of. Allyl compounds such as diallylacetic acid and a series of allylbarbituric acid derivatives can be determined brometrically only if care is taken that not more than 10% hydrochloric acid is present during the entire titration. In the

determination of diallylacetic acid, 0.05-0.06 Gm. of the material is dissolved in 10 cc. of methyl or ethyl alcohol, mixed with 15 cc. of 20% hydrochloric acid. Five-tenths gram potassium bromide is then added and N/10 potassium bromate to a weak yellow color. After the addition of a little potassium iodide and starch solution it is back titrated with N/10 thiosulfate. The barbituric acid derivatives (diallyl-, allylisopropyl- and allylphenylbarbituric acid) can only be titrated after they have been hydrolyzed to acetylcarbamide derivatives with alkali. A quantity of material equivalent to 10-15 cc. of bromate solution (e. g., 0.05-0.07 Gm. of the diallyl compound) is boiled for 20 minutes under a reflux condenser with 5-6 cc. of 10% sodium hydroxide. After cooling 25 cc. of 20% hydrochloric acid is added, and after cooling again 0.5 Gm. of potassium bromide is added and the titration carried out as before.—F. Wessel and M. Keszler. Ber. ungar. pharm. Ges. (1937), 244; through Pharm. Weekblad, 74 (1937), 962. (E. H. W.)

Aneurin (Vitamin B<sub>1</sub>)-Chemical Determination of, by the Thiochrome Reaction. The uncertainties and labor involved in animal tests for vitamin B<sub>1</sub> make a chemical test essential for any rapid development of the physiology and pathology connected with this vitamin. The method of Kinnersley and Peters applies only to fairly pure samples, involving loss in purification. Investigation of the oxidation of vitamin B<sub>1</sub> to thiochrome showed that this reaction may be used for the quantitative reaction of vitamin B<sub>1</sub> because of the high fluorescence of thiochrome. A solution containing 1 to  $20\gamma$  of vitamin  $B_1$  in 0.2 cc. is diluted to 2 cc. with methanol. To this is added 1 cc. of 30% sodium hydroxide and 0.1 cc. of potassium ferricyanide. After about 2 minutes 13 cc. of isobutanol is added; after shaking and centrifuging, 10 cc. of the isobutanol solution is transferred to a photoelectric fluorometer standardized for thiochrome by using quinine in sulfuric acid. The fluorescence of thiochrome in isobutanol is much brighter than in water. In the presence of methanol, 20 times the required amount of potassium ferricyanide can be used without affecting the thiochrome. The purification of aneurin is accomplished by absorption on Fuller's earth, using the international adsorption method, followed by elution by the alkaline oxidizing solution, for aneurin is unstable in alkaline solution but thiochrome is stable. The experimental error using yeast to which aneurin was added was 17%, but this error was much less than that involved in animal experiments.—B. C. P. Jansen. Rec. trav. chim. Pays-Bas, 55 (1936), 1046–1052; through Chimie & Industrie, 38 (1937), 452.

Antimony Electrode—Applicability of, to Determination of Hydrogen-Ion Concentration, Especially That of Blood. The potential- $p_{\rm H}$  curve varies according to the nature of the buffer, the kind of antimony electrode (stick or plated) and the form of electrode vessel. It is impossible to obtain a general empirical calibration curve of the potential against the  $p_{\rm H}$  for different experimental conditions; hence the antimony electrode is not suitable for general  $p_{\rm H}$  determination, especially for blood plasma.—H. Yoshimura. Japan J. Med. Sci. (Biophysics), 4 (1936), 131–141; through Chimie & Industrie, 38 (1937), 453. (A. P.-C.)

Berberin-Determination of, in the Homeopathic Mother Tincture of Hydrastis Canaden-The 7th contribution offers the following method: Treat 10 Gm. of the tincture in a small dish with 5 cc. distilled water and remove the alcohol by heating on a water-bath. Transfer the aqueous solution to a separatory funnel (200 cc.) rinsing the dish with  $2 \times 5$  cc. water, followed by 3 cc. ammonia and about 10 cc. ether. After adding 50 cc. ether to the separatory funnel shake for 3 minutes, allow to settle and carefully remove the water layer to a beaker and shake the ether solution with 20 cc. water. Combine the aqueous extracts and set aside. Filter the ether solution into a small wide-necked flask and rinse the separatory funnel and filter with  $2 \times 10$  cc. ether. Treat the combined filtrates as described in the homeopathic pharmacopæia under the evaluation of hydrastis mother tinctures. Acidify to litmus the aqueous solution above (free of hydrastin) with dilute hydrochloric acid, add 10 cc. sulfurous acid and 5 cc. potassium iodide solution (1:10), stir well, allow the berberine iodide to settle and after one-half hour collect the precipitate in a tared crucible and weigh. The sulfurous acid is added to prevent the formation of periodides; the precipitate obtained should be bright yellow.—WALTHER AWE. Apoth. Ztg., 52 (H. M. B.) (1937), 1359-1360.

Calomel Powder—Determination of the Degree of Fineness of. The degree of fineness of calomel is determined in Andreasen's apparatus (Angew. Chem., 48 (1935), 283), by a sedimentation pipette method. The powder is rubbed up in a small quantity of cyclohexane (technical grade), it is transferred to the lower cylinder of the apparatus, brought to constant temperature in a bath, filled to the mark and shaken. At various time intervals test portions are drawn into the evacu-

ated pipette. The pipetted material is transferred onto a tared Jena glass filter disk with ether, washed three times with ether, dried and weighed. The viscosity of the cyclohexane is determined in a Raaschou viscosimeter. The particle size of the calomel (length in  $\mu$ ) is determined from Stokes law. Various commercial and laboratory preparations are tested. They are also examined microscopically. All the commercial products, designated either "pharmacopæial grade" (pharmacopæware) or "vapor paratum," were truly monodisperse powders consisting of tiny crystal asters, prepared by the condensation of calomel vapor. Polydisperse products, showing crystal fragments, and made by mechanical pulverization of sublimed calomel, were not found on the Danish market. Claims that the Danish preparations labeled "vapore paratum" are finer grained than the items labeled "pharmacopoeware" were not supported. Preparations made by precipitation were finer in particle size than the ordinary sublimed products.—A. D. Hörlutck. Dansk Tids. Farm., 11 (1937), 331.

Carbohydrates-Microchemical Analysis of, in Plant Substances. A complete scheme of analysis is described and the results of numerous experiments are tabulated. The carbohydrate complexes are divided into six groups: (I) carbohydrates soluble in hot ethyl alcohol, (II) carbohydrates soluble in cold water but insoluble in alcohol, (III) carbohydrates soluble in water at 45-47°, (IV) carbohydrates which are hydrolyzed by diastase, (V) carbohydrates soluble in hot water and (VI) carbohydrates which undergo scission by treatment with 2\% sulfuric acid. The systematic procedure covers (1) the defatting of the material, (2) the extraction of Group I, (3) extraction of Group II, (4) extraction of Group III, (5) hydrolysis of the starches, (6) extraction of Group V, (7) extraction of Group VI, (8) dissolving of the cellulose. Then, after the separation into groups, the following determinations are made—in Group I, (a) determination of the invert sugar, (b) determination of fructose in the presence of glucose. In Group I, maltose, fructosides, galactosides and trehalose are also determined. In Group II, pectin substances, dextrin, gum and  $\beta$ -amylene are determined. In Group III inulin is determined. In Group IV, starches are determined. In Group V, xylan or araban and protopectin are determined. In Group VI, mannan + galactan, pentosans and  $\alpha$ -amylan are determined. In Group VII, cellulose is determined.—S. M. Strepkov. Z. anal. Chem., 111 (1937), 57-94; through Chem. Abstr., 32 (1938), 882. (F. J. S.)

Carbon and Hydrogen—Microdetermination of. The usual Pregl pressure regulator is used, having the U-tube filled with Ascarite and Drierite. The Pregl combustion tube, filled with silver gauze, is heated with an electric heater. The substance is weighed in an ordinary platinum boat and sprinkled with fine, previously heated copper oxide. Another, somewhat smaller, platinum boat is inserted into the former, so that the handles are at opposite poles. By conducting combustion slowly, accurate results have been obtained. The sample is weighed in a regular capillary. The tip is broken off and the capillary is placed in a somewhat larger capillary about 1 cm. long which is closed at one end. The larger capillary is filled with copper oxide, or if the substance is a halide, with precipitated and previously dried silver. These two capillaries are placed in a suitable long platinum vessel.—A. Elek. Ind. Eng. Chem., Anal. Ed., 30 (1938), 50–51.

Chlorine and Bromine—Microanalytic Determination of, in Organic Substances. Substance incinerated and treated with sodium carbonate and sulfite and acidulated with nitric acid to determine chlorine content. Bromine determined by sodium carbonate and sulfite, but not acidulated. Both titrated with N/100 mercuric nitrate.—M. Jurecek. Coll. Trav. Chim. Tchecoslov., 7 (1935), 316; through Rev. sud-americana endocrinol. inmunol., químioterap., 20 (1937), 538. (G. S. G.)

Chlorophyll—Gravimetric Determination of. Chlorophyll contains 2.7% of magnesium. The sample is triturated with sand and 30% acetone. This extract is filtered off and rejected. The chlorophyll is then extracted with pure acetone. By shaking with ether, the chlorophyll is obtained in ethereal solution. This is evaporated to dryness and ashed. The ash is digested with acid and the magnesium in it is precipitated as magnesium ammonium phosphate. The precipitate is washed with dilute ammonia solution, dissolved in acid and the solution treated with ammonium molybdate reagent. The yellow precipitate is filtered off and weighed.—F. Rogoziński. Bull. inter. acad. polon. sci., Classe sci. math. nat., 1937A, 483–489 (in French); through Chem. Abstr., 32 (1938), 882.

Cleome Pentaphylla L.—Chemical Examination of. II. Constituents of the Oil from the Seeds. The oil obtained by the extraction of the seeds with benzene shows the following chemical and physical constants: Sp. gr. (20° C.) 0.9268,  $n_{25}$  1.4653, solidification point  $-12^{\circ}$  C., acid value 36.5, saponification value 194, iodine value 122.6, acetyl value 33.5, Hehner value 91.5, unsaponifiable matter 2.08%. The oil was saponified and the mixed fatty acids showed the following properties: semi-solid, liquefying point 33–35° C., sp. gr. (40° C.) 0.8873, neutralization value 188, mean molecular weight 298, iodine value 126.5. These acids were resolved into saturated acids (22.4%), iodine value 1.7, mean molecular weight 259.8 and unsaturated acids (77.6%), iodine value 139.5, mean molecular weight 280.3. The former were shown to consist of oleic acid (32.02%) and linolic acid (38.97%). The latter acids consisted of palmitic acid (9.57%), stearic acid (9.5%) and arachidic acid (0.44%); unsaponifiable matter consisted of a phytosterol, m. p. 131–132° C.—RAM. NATH MISRA and SIKHIBHUSHAN DUTT. Proc. Natl. Inst. Sci. India, 3 (1937), 325–329. (H. M. B.)

Cocaine—Indirect Method of Estimating, in Mixtures of Cocaine and Novocaine. Cocaine (I), when found admixed with novocaine, can be rapidly determined in the following manner: the mixture (2–10 mg.) is dissolved in water and treated with slight excess of sodium nitrate in presence of dilute sulfuric acid and the reaction continued for three minutes. The solution is then treated with 5 cc. of 10% sodium hydroxide solution and the resulting mixture treated with 1 cc. of 1%  $\beta$ -naphthol solution. The color is matched against a suitable standard solution of novocaine in a colorimeter. I does not interfere in the reaction and the amount of I is obtained by difference (maximum error = 1%). A table of melting points of cocaine-novocaine (HCl) mixtures is given.—S. N. Chakravarti and M. B. Roy. Current Sci., 6 (1937), 219–220; through Chem. Abstr., 32 (1938), 1866. (F. J. S.)

Cocaine Cuprocyanide—Composition of, Obtained with Cherry-Laurel Water. On treating an aqueous solution of cocaine hydrochloride with cherry-laurel water, there is formed a light, complex precipitate, the composition of which corresponds to the formula: CuCH.3(HCN.-C11H21NO4).5HCN. The precipitate therefore contains less cocaine and more cyanogen than the normal cuprocyanide, CuCN.4(HCN.C17H21NO4).4HCN. Cherry-laurel water contains about 0.007 Gm. of copper per Gm. of cyanogen. Excess of hydrocyanic acid decreases the proportion of cocaine precipitated, as part of the base must be dissolved, probably as the cyanide.—P. Mesnard. Bull. trav. soc. pharm. Bordeaux, 74 (1936), 127–131; through Chimie & Industrie, 38 (1937), 523.

Contaminants—Atmospheric, Determination of. I. Organic Halogen Compounds. Organic halogen compound vapors and dusts in the atmosphere are determined by absorption of the vapors of volatile liquids in ammonium acetate in a special absorber, burning the ammonium acetate in a modified sulfur lamp apparatus, absorbing the combustion products in dilute sodium hydroxide and determining the halogen present, after neutralization, by a Mohr titration. The method was shown to be satisfactory for eight chlorine compounds of industrial importance. It was shown theoretically and experimentally that the volume of air that can be sampled successfully is inversely proportional to the vapor pressure of the solvent. The apparatus used is described in detail, with diagrams.—H. B. Elkins, A. K. Hobb and J. E. Fuller. J. Ind. Hyg. Toxicol., 19 (1937), 474; through Squibb Abstr. Bull., 10 (1937), A-2271. (F. J. S.)

Copper and Urobilin—New Color Reaction between. A 1:1000 solution of urobilin gives with a neutral solution of copper (1:1,000,000) a rose or, with slightly more copper (1:100,000), a purple tint. The test is less sensitive if the solution is acid, and if this is due to mineral acid a little sodium acetate should be added after neutralization. The test is unaffected by the presence of sulfates or chloride of aluminum, barium, calcium, cadmium, cobalt, stannous tin, ferrous iron (though this intensifies the color), beryllium, lithium, magnesium, manganese, nickel, gold, platinum, strontium or zinc, silver nitrate, sodium arsenate, arsenite, molybdate or tungstate, or ammonium vanadate. A zinc salt produces a green fluorescence. The method is suitable also for the colorimetric determination of copper.—G. Bertrand and L. De Saint-Rat. Compt. rend. acad. sci., 203 (1936), 140–143; through Chimie & Industrie, 38 (1937), 444. (A. P.-C.)

Cysteine and Nitroprusside—Colored Reaction between. When a cold saturated aqueous solution of cysteine hydrochloride is treated with an excess of a methanol solution of sodium nitroprusside and a methanol solution of potassium hydroxide is added with continual cooling, there is obtained an abundant, violet red, microcrystalline precipitate, similar in appearance to that

obtained by the reaction of sulfides on alkali nitroprussides. The —SH group of the cysteine probably reacts with the NO group of the nitroprusside to produce a complex. The reaction is very sensitive (up to a dilution of 1:60,000 of cysteine), and lends itself to colorimetric estimation. Cysteine does not contain any free —SH group and therefore does not give the reaction, contrary to reduced glutathion.—G. SCAGLIARINI. Alti V congr. naz. chim. pura applicata Sardegna, 1 (1936), 546-547; through Chimie & Industrie, 38 (1937), 453. (A. P.-C.)

Derris Root—Colorimetric Determination of the Quality of. The authors have studied the various methods for the colorimetric determination of rotenone in derris root and arrive at the following conclusions: (1) The reaction of Danckwort was really discovered in 1899 by Sillevoldt; (2) rotenone gives a permanent color reaction with ordinary sulfuric acid; (3) the color reaction follows the law of Beer in concentrations suitable for colorimetry; (4) the color reaction of rotenone with NO-free sulfuric acid is more sensitive and more specific than the diphenylamine reaction; (5) the color reaction of rotenone-containing substances, as known at present is not specific for rotenone and therefore cannot be used to determine rotenone in derris root.—Arie Goudsward and J. Ch. Timmers. *Pharm. Weekblad*, 74 (1937), 630. (E. H. W.)

2,6-Dichlorophenolindophenol—Standardization of. The desired quantity of dye (35 to 70 mg, per 100 cc. of solution) is placed in a small beaker and successive portions of hot water are added. After each addition of water the solution is decanted through a filter into a volumetric flask, and when all the dye has been dissolved the filter is washed with small portions of hot water until the washings are colorless or nearly so. After cooling to room temperature, the solution is made up to volume. Fifteen cubic centimeters of the dye solution are pipetted into a 50-cc. Erlenmeyer flask, 0.5 to 1.0 Gm. of potassium iodide and 0.5 to 1.0 cc. of dilute sulfuric acid (1 to 4) are added, and after shaking to facilitate the oxidation of the potassium iodide, the liberated iodine is titrated with 0.01N sodium thiosulfate using the usual starch indicator. It has been established that 1 cc. of 0.01N iodine solution is equivalent to 0.88 mg. of ascorbic acid; consequently, 1 cc. of 0.01N sodium thiosulfate solution should also be equivalent to 0.88 of ascorbic acid. The curve obtained for the titrations against sodium thiosulfate is comparable with that in which ascorbic acid was used, but is somewhat lower than that obtained with lemon juice. This leads to the conclusion that lemon juice contains small amounts of substances other than ascorbic acid which are oxidized by iodine but not by the dye.—M. H. MENAKER and N. B. GUERRANT. Ind. Eng. Chem., Anal. Ed., 30 (1938), 25-26. (E. G. V.)

Dioxane as a Reagent for Detection and Determination of Small Amounts of Iodide. Its Application to the Detection of Iodine in Iodized Salt. A Correction. The statement in the previous article (Chem. Abstr., 31, 39531) that "no reference could be found to the reaction of dioxane with iodides" is in error. That the method previously described is feasible for small quantities of iodine from 0.1 to 5 mg. has been shown by additional experiments with a photoelectric colorimeter which proves conclusively that the color produced follows the Lambert-Beer law.—Abraham Saifer and James Hughes. J. Biol. Chem., 121 (1937), 801-802; through Chem. Abstr., 32 (1938), 613. (F. J. S.)

Drugs—Absorption of, in Mother's Milk. The author reports on the quantitative analysis of mother's milk in cases where the drugs have been administered by mouth. The following drugs were used: iodides, bromides, arsenic and quinine. Results of analyses of samples taken at various periods after the administration of the drugs are given and methods of analysis are discussed.—Th. A. G. Haanappel. *Pharm. Weekblad*, 74 (1937), 871. (E. H. W.)

Drugs, Nostroms and Cosmetic Preparations—Examination of. The 32nd of a series of articles dealing with the examination of 61 preparations and items.—C. Griebel. Apoth. Ztg., 52 (1937), 1218–1221. (H. M. B.)

Ergot—Nucleic Acid of. The authors have separated a nucleic acid from ergot, of which the phosphorous content varied between 8.30 and 8.46%; the nitrogen content between 14.63 and 15.47% (the ratio of P:N was from 1:1.75 to 1:1.84). In determining the constitution of the acid the authors employed a special method to accurately determine purines and guanine and at the same time to find the ratio between guanine and adenine. Comparative studies of the nucleic acid from ergot and that from yeast showed the two acids to be chemically identical; the formula having been previously stated by Levene. Upon hydrolysis of the nucleic acid the authors obtained guanine, adenine, cytosine, uracyl and a compound having the properties of pentose. The greater

quantity of the latter compound was combined with purines.—M. GATTY-KOSTYAL and J. TESARZ. Wiadomości Farm. (1936), 213, 229; through Pharm. Weekblad, 74 (1937), 61. (E. H. W.)

Esters—Rapid Saponification of, by Potassium Hydroxide in Diethylene Glycol. In a 10- or 25-cc. distilling flask are placed 3 cc. of diethylene glycol and 0.5 Gm. of potassium hydroxide pellets. Following the addition of 10 drops of water, the mixture is heated over a small flame until the potassium hydroxide has dissolved. The mixture is cooled if the ester to be tested is volatile. The ester is added, 1 cc. if a liquid, 1 Gm. if a solid, or double these amounts if the substance is known to be of high molecular weight. The neck of the flask is now closed with a cork stopper carrying a thermometer of suitable temperature range. The side arm is fitted with a cork stopper of suitable size to connect the flask to a small water-cooled condenser. The flask is heated over a small flame, during which time the contents of the flask are mixed by shaking. When only one liquid phase, or one liquid and one solid phase, remains in the flask, the flask is connected to the condenser and the alcohol is carefully distilled. When done carefully, this procedure generally yields sufficient alcohol to prepare at least two solid derivatives such as 3,5dinitrobenzoate, the nitrophenyl urethane, or others. The residue left in the distilling flask is either a solution or a suspension of the potassium salt of the acid portion of the ester in diethylene glycol. Derivatives of this salt may be prepared as follows: About 10 cc. of water and 10 cc. of ethyl alcohol are added to the diethylene glycol solution; then a drop of the phenolphthalein solution and 6N sulfuric acid, drop by drop, are added until just acid. The resulting solution is set aside for a few minutes to allow as complete precipitation of the potassium sulfate as possible. This precipitate is separated by filtration and the clear filtrate is divided into two equal parts. One portion may be treated with p-nitrobenzyl bromide, and the other with p-phenylphenacyl bromide or other suitable reagent. This procedure will generally yield two solid derivatives with melting points of use in identifying the acid constituent of the ester. A procedure is described for determination of the saponification equivalent.—C. E. REDEMANN and H. J. LUCAS. Ind. Eng. Chem., Anal. Ed., 9 (1937), 521. (E. G. V.)

Fluorine-Titration of, in Aqueous Solutions. Fluorine may be titrated quantitatively with thorium nitrate (0.1N) in an aqueous solution, instead of the 48% ethyl alcohol solution, closely controlling the  $p_{\rm H}$  of the solution to be titrated. A  $p_{\rm H}$  of 2.9 to 3.1 was found to be suitable and is obtained by the use of monochloracetic acid, half neutralized by sodium hydroxide. Prepare the buffer by dissolving 9.448 Gm. of monochloracetic acid and 2.000 Gm. of sodium hydroxide in 100 cc. of water. Take an aliquot of 50 or 100 cc., depending upon the amount of fluorine present. If 50 cc. are taken, dilute to 100 cc. with water. Add 8 drops of sodium alizarin sulfonate indicator. Adjust the acidity back and forth with 2% sodium hydroxide and 1 to 200 hydrochloric acid, finally leaving the solution just acid and the pink color discharged. Add 1 cc. of the buffer solution and titrate with 0.1N thorium nitrate to a permanent pink. A blank determination should be made. Standardize the thorium nitrate by titration of the fluorine obtained by distillation of 100% natural cryolite, fluorspar or sodium fluoride. The end-point is more definite in an aqueous solution than in a 48% ethyl alcohol solution. When using a 48% alcoholic solution and 0.1N thorium nitrate, a fluorine content of approximately 20 mg. can be titrated to a fairly definite end-point; if a greater amount of fluorine is present, the end-point is indefinite. However, in an aqueous solution (using 0.1N thorium nitrate) as much as 50 mg. of fluorine may be titrated to a definite end-point.—R. J. ROWLEY and H. V. CHURCHILL. Ind. Eng. Chem., Anal. Ed., 9 (1937), 551. (E. G. V.)

Fractionating Columns—Designs for Laboratory. Columns for liquid partition take-off, vapor partition take-off, and moderately low temperatures, also pressure and low temperature columns, are described.—J. H. Simons. *Ind. Eng. Chem., Anal. Ed.*, 30 (1938), 29-31.

(E. G. V.)

Furfuraldehyde—Gravimetric Determination of. There has been a tendency to use thio-barbituric acid or diphenylthiobarbituric acid in place of phloroglucinol for the determination of pentosans in plant materials. The suitability of the three reagents for the determination of pentosans in wood has been re-investigated. It was found that whereas furfuraldehyde phloroglucide, resulting from the reaction with phloroglucinol of the furfuraldehyde produced on distilling the wood with 12% hydrochloric acid, could be collected and washed with comparative ease, furfurylidenemalonylthiocarbamide resulting from the use of thiobarbituric acid was much more troublesome. The furfurylidenemalonylthiocarbamide is slightly soluble in 12% hydrochloric

acid, but it also appears to adsorb the reagent, so that these compensating errors probably account for the high results obtained in furfuraldehyde estimations with the reagent. The claim that thiobarbituric acid avoids the errors due to precipitation of hydroxymethylfurfuraldehyde with phloroglucinol is of no account with many woods, since little or no hydroxymethylfurfuraldehyde is formed. Certain soft woods, on the other hand, do yield appreciable quantities of this substance, but in such cases the furfurylidenemalonylthiocarbamide precipitate is readily peptized during washing and cannot be collected quantitatively. It is concluded that thiobarbituric acid presents no real advantages over phloroglucinol, but some results obtained with diphenylthiobarbituric acid suggest that it may prove to be a more suitable reagent.—W. G. CAMPBELL and L. A. SMITH. Biochem. J., 31 (1937), 535; through Quart. J. Pharm. Pharmacol., 10 (1937), 576. (S. W. G.)

Fusel Oil-Determination of, in Spirits. Since the usual method of Röse (shaking out with chloroform and measuring the loss in volume) does not give good results, the author has devised a method of procedure using the refractometer. He shakes out the fusel oil with a highly refractive liquid which, after the addition of the slightly refractive amyl alcohol, shows a diminution in refraction. The liquid under investigation is freed of esters in the usual manner (boiling with alkali), distilled and the distillate adjusted to 30 volumes per cent (15°). Twenty cubic centimeters of the distillate and 22 cc. of water are mixed in a stoppered vessel of about 80 cc. volume and 20 Gm. of pure ammonium sulfate is dissolved in this mixture by shaking and warming. It is then cooled to 15°. If the fusel oil content is high droplets will separate. Three cubic centimeters of  $\alpha$ -chloronaphthalene are then added and the flask, which is protected from the warmth of the hand by a layer of felt, is shaken vigorously (about 200 times) until solution is effected. After a short time it is centrifuged. A blank consisting of 30% pure alcohol is treated similarly. The chlornaphthalene solution is then transferred to a refractometer by means of a pipette. The value  $n(blank)^{-n}Cln + fusel$  is practically independent of the temperature. In the Abbé refractometer reading a diminution of 1 in the fourth place is equivalent to a fusel oil content of 0.00625 volume per cent.—W. Leithe. Z. Untersuch Lebensm., 72 (1936), 351; through Pharm. Weekblad, 74 (1937), 713. (E. H. W.)

Graphite—Purification of, for Spectrochemical Analysis. Cut the graphite into the desired form for use as electrodes. Heat in a silica dish over an oxy-gas burner to redness. Cool and place in a flask fitted with a reflux air condenser. Cover the electrodes with 1 to 1 sulfuric acid and boil on a hot plate for at least 24 hours. Wash by decantation with distilled water until the water is no longer acid to litmus, then boil 15 minutes in a fresh portion of water. Again wash by decantation and repeat the boiling. Continue the alternate washing and boiling until acid is no longer extracted. Usually four such operations will suffice. Transfer to the silica dish and heat to bright redness, allowing the flame to play directly on the electrodes. Cool and store in capped bottles until used.—A. H. Stand and A. E. Ruehle. Ind. Eng. Chem., Anal. Ed., 30 (1938), 59.

Hexamine and Ammonia-Some Identity Reactions of. Five tests for hexamine are described, based on the formation of: (1) A compound with antipyrine. (2) A yellow bromine compound, C<sub>6</sub>H<sub>12</sub>N<sub>4</sub>Br<sub>2</sub>. A washed particle of this dibromo-derivative liberates iodine from potassium iodide, whereas tribromo derivatives of phenol, orcine and aniline do not. (3) Crystallized phenates. Mix a particle of hexamine with about twice its weight of phenol, stir with a few drops of alcohol to dissolve, and evaporate on a slide; long needles are formed. To detect ammonia in a gaseous mixture containing primary amines by this method, spread on a slide a drop of reagent containing 1 Gm. of pure phenol and 3 cc. of 40% formalin per 100 cc. of 85 to 90% alcohol; then expose the layer to the gas mixture at the top of the flask containing it; crystals form notably at the margin if ammonia is present; the amines form liquid imines with formaldehyde, but may prevent crystallization. To avoid this allow the gas mixture first to bubble through ethanol. (4) When a particle of hexamine is put into piperonal at its boiling point (263° C.), an intensely black liquid is formed even at a dilution of 0.04%; at a dilution of 0.004% a dark color still prevails after boiling for 1 minute. Addition to piperonal at its boiling point of glucose, lactose, sucrose, starch, tartaric acid, aniline or antipyrine in 2% concentrations produces only a yellow tint. No color changes take place with citric or oxalic acids, benzyl alcohol, phenol, guaiacol, quinol or camphor. With ammonium salts, a dark color is produced in 2\% concentration. To remove ammonia in a mixture of hexamine and ammonium salt, evaporate the aqueous solution gently with a little sodium carbonate; add piperonal to the residue, and boil. (5) When dry hexamine is heated slowly in a test-tube with 2 to 3 parts of absolute phenol, a clear yellow resin is formed; after removal of the condensed phenol, add 1 cc. of sodium hydroxide and a few drops of sodium hypochlorite; this dissolves the resin with a strong, brownish green color. A cold mixture of hexamine, phenol and hypochlorite solution produces no color (proving the absence of aminophenol or aniline)—G. BOUILLOUX. J. pharm. chim., 24 (1936), 58-64; through Chimie & Industrie, 38 (1937), 448. (A. P.-C.)

Hormones. A continuation of the review on hormones (*Pharm. Ztg.*, 82 (1937), 516-518). The thyroid gland is discussed from the standpoint of its discovery, anatomy, physiological activities, relationship to the other hormones and vitamins, and the chemistry of its active principle—thyroxin.—J. Martinius. *Pharm. Ztg.*, 82 (1937), 829-831. (N. L.)

Hydrocyanic Acid—Methods of Estimating. Titration of hydrocyanic acid with silver nitrate by the Liebig method is affected by the alkalinity of the solution. The preferred form of the Dènigés method is the use of 5 cc. of ammonium hydroxide solution in a total volume of 100 cc. However, the use of ammonium hydroxide delays the end-point; this is obviated by neutralizing with sodium bicarbonate. Conditions for successful iodometry of cyanides are: sufficient dilution (0.025 Gm. hydrocyanic acid in 350 cc.), and presence of sodium bicarbonate (5 Gm. per 350 cc.). Under these conditions starch is an excellent indicator. Fresh and dilute solutions of cyanogen iodide do not affect starch even in acid solution. The ferric thiocyanate reaction is excellent for the colorimetric determination of cyanides in acid (hydrochloric) media if the ferric and chloride concentrations are both held constant. Cyanides can also be titrated after transformation into thiocyanates if the presence of thiosulfates is avoided. In the determination of hydrocyanic acid in flaxseed by a modification of the Kohn-Abrest method (Ann. fals., 28 (1935), 547-561) 25 Gm. of seed are finely ground in a steel mortar and macerated 45 minutes at 45° C. with 500 cc. of water, followed with two distillations, one with 5 cc. and the other with 50 cc. of hydrochloric acid; the expelled hydrocyanic acid is absorbed in sodium hydroxide solution and titrated. The hydrocyanic acid content of Uruguayan flaxseed varies greatly (14.9 to 28.4 mg. per 100 Gm.) according to variety and date of planting.—J. F. SAREDO. Ph. Org. Assoc. Estud. Quim. Farm. (Montevideo), 8 (1936), No. 2, 16-42; through Chimie & Industrie, 38 (1937), 450. (A. P.-C.)

Hygroscopic Substances—Drying and Weighing, in Microanalysis. Methods for drying and weighing hygroscopic substances are described, stressing the necessity for determining the relative hygroscopicity of every sample submitted for analysis. A microbalance, capable of giving readings constant to  $\pm$  0.002 mg. over a period of several minutes, is essential for this determination. A new type of pig is described, with a special technic which makes it possible to weigh the sample and pig to  $\pm$  0.002 mg. Used with a copper boat, the special pig has also proved useful for the determination of nitrogen by Dumas method. A revised method is given for handling substances which must never come in contact with damp air. Two samples are dried in the combustion tube: one of about 3 mg., the other of 15 to 20 mg. The water is determined on the larger sample, while the actual combustion is made on the smaller sample.—D. F. HAYMAN. Ind. Eng. Chem., Anal. Ed., 30 (1938), 55–56. (E. G. V.)

Indicators—Mixed, Application of, in the Acidimetric Titration of Dilute and Colored Solutions. The use of fluorescein (I) as an auxiliary indicator is based on the complete masking of the green fluorescence of I by the red ions of methyl red and methyl orange. Thus, the exact neutralization point is indicated sharply by the reappearance of the green fluorescence in the titration of an acid solution with sodium hydroxide and its disappearance in the titration of an alkaline solution with an acid. Excellent results were obtained by working in diffused and artificial light in the titration of highly diluted and colored solutions, such as pitch, tar, contact mixtures and caramel.—R. K. Burshtein. Zavodskaya Lab., 6 (1937), 825–826; through Chem. Abstr., 32 (1938), 73. (F. J. S.)

Indigofera Enneaphylla L.—Chemical Examination of, and the Isolation of Its Active Principle. The plant yields 43.2% moisture, a gray-colored ash (15.27%), 39% of which is water-soluble and this portion showed the presence of K, SO<sub>4</sub> and Cl ions and the water-insoluble portion, Al, Fe, Ca, Mg and CO<sub>5</sub> ions, 9% insoluble in a mixture of nitric acid and hydrochloric acid (chiefly silica). The plant yielded the following extracts upon successive treatment: (1) Alcohol.—7.21%, deep green, sticky, peculiar odor. (2) Methyl Alcohol.—6.12%, less sticky, similar odor to (1). (3)

Acetone.—1.31%, dry yellow-green, faint odor. (4) Benzene.—7.38%, thick viscid, dark green, strong odor. (5) Chloroform.—5.15% light green dry, faint odor. (6) Carbon Tetrachloride.— 5.56%, dry yellow-green, faint smell. (7) Water.—4.65%, viscid green, unpleasant smell. The active principle of the plant was found to occur in the 90% alcohol extract as a mixture of two unsaturated hydrocarbons of high molecular weights: Indigoferin, C<sub>70</sub>H<sub>140</sub> (A), soluble in hot acetone and Enneaphyllin, C<sub>90</sub>H<sub>164</sub> (B), insoluble in hot and cold acetone. Details for obtaining the alcohol extract (0.15-0.20%) which also contained potassium sulfate are given. A has the following properties: white, melting at 77° C., slightly soluble in hot alcohol, ethyl acetate, pyridine, soluble in hot methyl alcohol and acetone crystallizing as needles, easily soluble in benzene, toluene, xylene, fairly soluble in hot chloroform, carbon tetrachloride, glacial acetic acid, very sparingly soluble in hot petroleum ether, ether, carbon disulfide, dissolves in molten naphthalene, insoluble in water, one double bond, iodine value (Wiji) 24.80, dibrom-derivative, C<sub>70</sub>H<sub>140</sub>Br<sub>2</sub>, melting at 81° C., soluble in hot benzene, alcohol soluble in chloroform and carbon tetrachloride, insoluble in acetone and water. Bafter the removal of A from nitrobenzene solution is crystallized from benzene and has the following properties: rod-shaped crystals, melting at 98° C., soluble in hot alcohol, methyl alcohol, ethyl acetate, very sparingly soluble in petroleum ether and carbon disulfide, fairly soluble in chloroform and carbon tetrachloride, easily soluble in warm benzene, toluene and xylene, insoluble in acetone, pyridine, glacial acetic acid and water, dissolves in molten naphthalene, one double bond, iodine value 22.44, dibrom-derivative, C<sub>90</sub>H<sub>154</sub>Br<sub>2</sub>, melting at 88° C., fairly soluble in hot benzene, slightly soluble in hot alcohol and methyl alcohol, soluble in carbon tetrachloride and chloroform, insoluble in acetone and water.-SATYENDRA NATH and SHIKHIVHUSHAN DUTT. Proc. Natl. Inst. India, 3 (1937), 371-376.

H. M. B.)

Iodides—Electrometric Titration of. Analysis of Drug Preparations Containing Iodine. Small quantities of iodides may be determined electrometrically, using the silver electrode. At the start of the titration the potential changes are slight, but when iodide begins to precipitate the rate of change of potential increases. At the equivalent point the change of potential is great and permits very accurate determination of this point. From studies of the titer of 25-cc. quantities of 0.04, 0.004 and 0.0004 molar potassium iodide, using, respectively, 0.1, 0.01 and 0.001 molar silver nitrate, it is evident that 1.66 mg. potassium iodide may be determined with an accuracy of 3%, while ten times greater quantities of potassium iodide or more may be determined with great accuracy. The results are not affected by the presence of chlorides, phosphates, carbonates, oxalates, urates or hippurates provided the  $p_{\rm H}$  does not exceed 8 or 9. Small quantities of iodides in urine may be determined electrometrically with 0.01N silver nitrate if the urine is acidified to the reddening point of methyl red. In colloidal preparations the iodine behaves as iodide practically completely and is excreted as iodide in the urine. One preparation, "Mecojod, Medicinalco," in which the iodine is largely bound in organic form and not as ionic iodine, does not give good results by the electrometric method. The iodine of "Iodferpon," "Iodalferraton" and "Iodoferral" is present as iodide. Studying the time of appearance of iodide in the urine after human ingestion of "Iodoferral," it is found that claims of the makers that the iodine in this preparation is not absorbed until it reaches the stomach are not supported. It behaves like other iodides as regards absorption.—J. K. GJALDBAEK. Dansk Tids. Farm., 11 (1937), 315.

Iodine—Volumetric Determination of, Using Elek-Hill Microbomb. A sample of from 3 to 6 mg., accurately weighed, is ignited in the bomb as described by Elek and Hill. After cooling, the bomb contents are dissolved in 15 to 20 cc. of hot water in a 125-cc. Erlenmeyer flask. When solution is complete, the bomb is removed from the flask and carefully rinsed. To the fluid sufficient boiled-out Alundum, grain size 16, is added to cover about two-thirds of the bottom of the flask, and the liquid is kept in moderate ebullition for 30 minutes, the whole of the free surface being covered with breaking bubbles. The use of Alundum instead of glass beads completely prevents bumping, even when this alkaline solution becomes very concentrated. After cooling under the tap, the solution is made just acid to methyl orange with 3N sulfuric acid, run in from a 50-cc. burette. About 10 cc. are required. The acid solution is filtered, with suction, into a ground-glass-stoppered 125-cc. Erlenmeyer flask where it is treated with 1 cc. of saturated bromine water to insure complete oxidation of the iodine to iodate. Iodine is almost quantitatively converted to iodate in the bomb, but the oxidation with bromine is necessary to make certain the

completeness of the conversion. After the solution has stood, stoppered, for 5 minutes, the excess of bromine is destroyed by the addition of 2 to 3 drops of formic acid with thorough shaking to remove the last traces of the bromine from the vapor phase as well as from the solution. During this treatment a moderate pressure is built up in the flask, necessitating care in subsequently opening it. If, after 2 minutes, the solution is not completely decolorized, another drop of formic acid should be added with shaking. To the colorless solution 0.2 to 0.3 Gm. of potassium iodide is added, the flask is stoppered and shaken to dissolve the salt, and, after 5 minutes, the liberated iodine is titrated with 0.01N sodium thiosulfate (prescribed instead of 0.02N, as recommended by several authors, because of the greater accuracy it permits, in addition to its more general usefulness in the microanalytical laboratory), which is standardized daily against 0.01N potassium biiodate —A. Elek and R. A. Harte. Ind. Eng. Chem., Anal. Ed., 9 (1937), 502. (E. G. V.)

Kreis Test—Improved. A new Kreis test is described in which an organic acid (trichloracetic) dissolved in an organic solvent (amyl acetate) is substituted for the mineral acids used in previous Kreis tests. The phloroglucinol used is dissolved separately in amyl acetate. These modifications have the effect of making the reaction proceed in one phase with the result that the color can be measured directly either on the Lovibond tintometer or the Zeiss tintometer. It has also been found that atmospheric oxygen is an important factor in the formation of the Kreis colored substance. The reaction in the new test cannot be allowed to proceed to completion because of the formation of a secondary yellow color and therefore the conditions of concentration of the reactants, temperature and time taken for the test have been stipulated. The test is superior to preceding Kreis test both in accuracy and sensitivity, and this is most marked in the stage of early oxidative spoilage in fats.—W. P. Walters, M. M. Muers and E. B. Anderson. Chemistry and Industry, 56 (1937), 1055. (E. G. V.)

Lead—Determination of, in Maple Products. A modified partial digestion method for the determination of lead in maple products is suggested and described in detail. With the proper apparatus and assistance about twenty-five samples can be run daily; a simple sample can be reported in about two hours. Tin, manganese and reasonable amounts of zinc do not interfere with the lead recovery as indicated from the analysis of syrups containing added known quantities of lead plus normal amounts of these contaminants. During the 1937 season in Vermont and New York, state-controlled laboratories determined the lead content of about one thousand syrups by this method with no apparent difficulty. Analysis of thirty samples of maple sirup by this method showed 0.001-0.055 grain of lead per pound. (One sample, not commercial grade, gave 0.156 grain per pound.)—J. L. Perlman. J. Assoc. Official Agr. Chem., 20 (1937), 622-627; through Chem. Abstr., 32 (1938), 258. (F. J. S.)

Lead—Determination of, in the Presence of Other Metals. To the solution containing lead which is one to two normal in nitric acid, add an equal volume of a saturated solution of thiourea in normal nitric acid. This causes precipitation of  $2Pb(NO_3)_2.11CS(NH_2)_2$ . The corresponding complexes of other ions are more soluble in dilute nitric acid and are not precipitated by this treatment. Cool in ice water, filter and wash with an acid solution of the reagent. Dissolve the washed precipitate in hot water and determine the lead as chromate, anthranilate or picrolinate in the usual way.—C. Mahr and Bertha Ohle. Z. anorg. Chem., 234 (1937), 224–228; through Chem. Abstr., 32 (1938), 878. (F. J. S.)

Lochnera Rosea—Chemical Examination of. Petroleum-ether extract of leaves of "chichirika" found. brownish syrupy alkaloid, a black resin, with bitter nauseous fatty taste, and amorphous white tasteless substance reacting as higher solid alcohol.—Lualhati Aldaba and Luz Oliveros-Belardo. Rev. Filipina de Med y. Farm., 28 (1937), 308. (G. S. G.)

Lovibond Tintometer—Some Colorimetric Determinations with the. The author discusses the Lovibond tintometer and its value in correcting errors in colorimetric determinations due to the variation in the eyesight of individuals. Three illustrative experiments are described: (1) the determination of the color series of a solution of potassium bichromate in water; (2) the determination of tincture of crocus and (3) the determination of vitamin A in cod liver oil.—J. VAN As. Pharm. Weekblad, 74 (1937), 695. (E. H. W.)

Magnesium—Determination of. After the precipitate of MgNH<sub>4</sub>PO<sub>4.6</sub>H<sub>2</sub>O is washed with dilute ammonia solution, it can be washed with acetone and weighed with its water of crystallization. To determine very small quantities of magnesium it is recommended to precipitate as Mg-NH<sub>4</sub>PO<sub>4.6</sub>H<sub>2</sub>O, dissolve the precipitate in acid, precipitate the phosphorus with ammonium molyb-

date and weigh the yellow precipitate. On the assumption that the precipitate is ammonium phosphomolybdate and that one mg. of magnesium corresponds to 89 mg. of precipitate, fairly accurate results were obtained.—F. ROGOZINSKI. Bull. intern. acad. polon. sci., Classe sci. math. nat. 1937A, 477–482 (in French); through Chem. Abstr., 32 (1937), 878. (F. J. S.)

Magnesium—Separation of, as Oxalate. Concentrate the neutral solution containing magnesium, ammonium and the alkali metals to a volume of 5 cc. or, preferably, evaporate it to dryness and dissolve the residue in 5 cc. of water. If less than 40 mg. of magnesium as metal is present, add 85 cc. of glacial acetic acid, to this solution, and with constant stirring add slowly from a pipette 10 cc. of saturated ammonium oxalate solution. If a larger amount of magnesium is present, add 70 cc. of glacial acetic acid to the sample solution, and then slowly add 25 cc. of a solution made up of 1 Gm. of ammonium oxalate dissolved in a mixture of 15 cc. of acetic acid and 10 cc. of water. If both magnesium and sulfate are present in large amounts, first add the 25 cc. of special oxalate reagent, which should be warm in this case, and then add the 70 cc. of acetic acid. After precipitation allow the covered vessel to stand on the steam-bath until the magnesium oxalate precipitate settles in a flocculent mass, leaving the supernatant liquid clear. This should require from 30 to 60 minutes. Decant the supernatant liquid through retentive quantitative paper, such as Munktell's No. OO, and wash the precipitate once or twice by decantation using hot 85% acetic acid. Finally, transfer the precipitate to the filter and wash with hot acid solution which is preferably delivered from an all glass wash bottle. For ignition, place the paper and precipitate in a weighed platinum crucible, heat gently at first to expel the acetic acid and water, then char and burn off the paper in the usual way. Finally, ignite in a covered platinium crucible at the highest temperature obtainable with a Meker burner or blast lamp. Cool in a desiccator and weigh as magnesium oxide. If a platinum crucible is not available, ignition may be made in porcelain, but it is difficult in this case to ignite the magnesia at a temperature high enough to render it sufficiently nonhygroscopic for accurate weighing and to burn off all carbonaceous matter. If a porcelain crucible is used it is therefore best to check the result by dissolving the magnesia in a slight excess of dilute sulfuric acid in the crucible, followed by evaporation to dryness, ignition at 400° to 500° C. and the final weighing as magnesium sulfate. For preparation of the filtrate from the magnesium determination for the preparation of the alkalies, add 25 to 30 cc. of nitric acid (concentrated) and evaporate to dryness. Add 2 cc. of nitric and 5 cc. of perchloric acid to the residue and again evaporate to dryness. Finally fume off all excess perchloric acid. The residue may then be used for the systematic separation of the alkalies or for their direct or indirect determination by various procedures.—P. J. ELVING and E. R. CALEY. Ind. Eng. Chem., Anal. Ed., 9 (1937), 558.

(E. G. V.)

Medicinal Plants-Indian, Chemical Analysis of. The Active Principle and Other Constituents of Fumaria Officinalis Bedd. This plant known in India as "Shaheterah" or "Pitpapra" (Hindustani) and "Pipara" (Bengali) has been regarded for a long time as a diuretic and an alterative, known to remove hepatic obstructions, an aperient, a laxative and is beneficial in dyspepsia and scrofulous infections. The present work shows the drug to contain 1% inorganic salts consisting of a mixture of potassium nitrate (70.90%) and potassium chloride (29.1%), penta-triacontane, C<sub>35</sub>H<sub>72</sub> (0.5%), an alkaloidal principle (0.13%) identical with protopine, tannins, phlobaphene and sugars. The diuretic properties of the drug are due to the potassium compounds and the protopine is responsible for the other physiological properties. Successive extractions with the selective solvents yielded the following extracts: (1) Benzene. -3.3%, a waxy green mass mostly chlorophyll and a low melting crystalline material. (2) Chloroform.—4.5%, yellowgreen sticky mass smelling of sugars and yielding alkaloids. (3) Alcohol.—13.1%, a yellow semisolid mass with some brown crystalline matter yielding a yellow precipitate with lead acetate, a green color with alcoholic ferric chloride, reduced Fehling's solution. (4) Water.—5.1%, brown crystalline residue containing organic salts, nitrates and chlorides, reduced Fehling's solution and gave faint precipitates with alkaloidal reagents. A hot alcoholic extract of the drug upon cooling yielded a crystalline mass containing potassium nitrate and chloride; the benzene washing of the cooled alcohol extract yielded penta-triacontane, melting at 75-76° C., soluble in benzene, less so in petroleum ether, alcohol, methyl alcohol and glacial acetic acid, insoluble in water, b<sub>16</sub> 329-331°. The purified extract treated with acetic acid yielded protopine acetate, C₂₀H₁₂O₅N.CH₃C₀₀H, melting at 267°; the free alkaloid melting at 206-207°. Its reactions toward reagents are described. The hydrochloride, melting at 274° is formed as fine crystals and the picrate melts at 249° with decomposition.—RADHA RAMAN AGARWAL. *Proc. Natl. Inst. Sci. India*, 3 (1937), 319–323.

(H. M. B.)

Mercurous and Mercuric Chlorides-Action of Ammonia on, and Their Determination. The results of qualitative tests indicate that the reaction between mercurous chloride and ammonium hydroxide is best expressed by the equation:  $2Hg_2Cl_2 + 4NH_4OH = Hg + Hg_2O + Hg_2O$  $NH_2Cl + 2H_2O + 3NH_4C1$ . The following procedure is recommended for the assay of calomel: Treat 0.3 to 0.4 Gm. of sample with 25 to 40 cc. of thrice normal ammonium hydroxide, heat 5 to 10 minutes with frequent shaking, filter, wash free from chlorides and make the filtrate barely acid with nitric acid; titrate with decinormal mercurous nitrate solution to the decolorization of ferric thiocyanate. Correct for the volume of solution required to decolorize the amount of ferric thiocyanate used as indicator. For the assay of corrosive sublimate first prepare 100 to 150 cc. of a decinormal solution of the sample. To 20 cc. of this solution in a 100-cc. measuring flask add 25 cc. of thrice normal ammonium hydroxide, shake, and make to volume; filter, discard the first 20 cc. of filtrate, neutralize a 50-cc. aliquot with nitric acid and titrate with mercurous nitrate. The method depends on the titration of chlorine and is based on the fact that three chlorine ions go into the filtrate when mercurous chloride is treated with ammonia and one chlorine ion remains in solution when mercuric chloride is treated similarly. The titration can also be applied to the analysis of any soluble chloride.—M. CHTCHIGO. Ann. chim. anal., 18 (1936), 149-151; through Chimie & Industrie, 38 (1937), 445-446. (A. P.-C.)

Methylene Blue—Precipitation of, by Cuprohydrocyanic Reagent. Cuprohydrocyanic reagent, prepared by mixing equal volumes of a 5% copper sulfate solution and a 5% potassium cyanide solution, neutralizing with sulfuric acid and filtering, gives with aqueous solutions of methylene blue a voluminous violet-blue precipitate, which first crystallizes as fine needles, but dries to a hard mass which does not show the presence of crystals. Its composition corresponds to the formula 2CuCN.13 (HCN.C<sub>16</sub>H<sub>18</sub>N<sub>3</sub>SC1)5HCN.—P. MESNARD. Bull. trav. soc. pharm. Bordeaux, 74 (1936), 161–164; through Chimie & Industrie, 38 (1937), 449. (A. P.-C.)

α-Methylpyrrylketone—Spectrophotometric Determination of. In hopes that a spectrometric method might prove satisfactory for the determination of the active agent in valerian, α-methylpyrrylketone, the ultraviolet absorption of synthetic α-methylpyrrylketone was studied. Synthesis was effected by Grignard reaction from pyrryl and methyl magnesium iodide, followed by condensation with acetyl chloride. The pure product melted at 90° C. Ether solutions of this have absorption maximum at 2810 Å., while aqueous solutions have the maximum at 2880 Å. In both solvents the absorption coefficient,  $\log k = 3.24$ . The partition of the ketone between water and ether was followed with the aid of the spectrometric method. The partition coefficient was found to be 0.13. The partition coefficient between water and hexane is 5.5. Ether is thus an excellent extraction solvent for the ketone. However, on studying the ether extract from aqueous preparations of Rhizoma Valerianæ, Dan. Phar., no characteristic spectrum of α-methylpyrrylketone was observed. Possibly the ketone could be identified in the fresh drug in this way, but the method apparently will not serve to control the official drug.—S. A. Schou and M. Tönnensen. Dansk Tids. Farm., 11 (1937), 344.

Microgram Samples—Qualitative Analysis of. A general working technic applicable to the qualitative analysis of 1-microgram solid samples is described. The chemical work is performed in cones of 0.5 cubic mm. capacity which are prepared from capillary tubing of approximately 0.5-mm. bore. Most of the manipulations are carried out by observation with the low-powered microscope. The transfer of solutions is performed with the use of micrurgical pipettes operated by a hypodermic syringe. A mechanical stage and a manipulator with rack and pinion motions in the three directions are used to bring the tools into proper positions under the microscope. Working procedures are described in the analysis of 0.01 cubic mm. of a solution containing 0.1 microgram of antimony and 0.01 microgram of bismuth. The separations obtained have the same sharpness as in the analysis of large quantities. Confirmatory tests of sufficient sensitivity are already available.—A. A. Benedetti-Pichler. Ind. Eng. Chem., Anal. Ed., 9 (1937), 483. (E. G. V.)

Milk—Evaporated, Lead Content of. Analyses of canned evaporated milk for lead by the photometric and diphenylthiocarbazon methods gave average values of 0.076 and 0.11 parts per million for a number of brands of various ages and from widely separated localities. It is concluded that contamination from the lead-tin alloy solder used in sealing the can is of negligible hy-

gienic importance.—L. T. FAIRHALL. J. Ind. Hyg. Toxicol., 19 (1937), 491; through Squibb Abstr. Bull., 10 (1937), A-2280. (F. J. S.)

Minerals and Intestinal Flora. Experimental diets on rats demonstrate necessity of calcium and phosphorus in diet to maintain typical aciduric organisms of intestine, and keep under control concentration of colon bacilli and anterococci.—Editorial. J. Am. Med. Assoc., 109 (1937), 1638. (M. R. T.)

Moisture—Modified Distillation Method for. Prepare and weigh the sample for distillation in the usual manner. Boil continuously for 1 hour at the rate of approximately 5 cc. of reflux per minute and for 3 hours at double that rate. Without interrupting the boiling, now add through the top of the condenser 2 drops of 95 per cent ethanol. After the violent ebullition and the refluxing have ceased, continue boiling for 5 minutes. Should drops of water still remain in the inner wall of the condenser tube, prolong the boiling and give a second treatment with ethanol, using 3 drops. After the violent action has stopped, maintain the boiling for 5 minutes, then remove the source of heat, allow the apparatus to cool, and read the volume of aqueous distillate. The introduction of a very small quantity of ethanol removes water adhering to the inner wall of the condenser tube, thus increasing the accuracy of the method. The ethanol should be added only after dehydration of the sample is complete. Ethanol should not be used as an indicator to show when dehydration of the sample is complete.—H. N. Calderwood and R. Piechowski. Ind. Eng. Chem., Anal. Ed., 9 (1937), 520. (E. G. V.)

Morphine—Quantitative Determination of. VI. Determination in Opium by Continuous Extraction with Isopropyl Alcohol and Benzol. A preliminary report on studies of a method of determining morphine in opium intended to avoid difficulties of the League of Nations lime method. The method involves continuous extraction in a glass extraction apparatus (depicted in the article) of a type described by Kutscher and Steudel, using a mixture of 70% isopropyl alcohol and 30% benzol by volume. No emulsion formation occurs. Preliminary tests showed that morphine may be extracted and separated quantitatively from a mixture with narcotine, papaverine and codeine. The method for opium is: 2 Gm. of opium are kneaded into an homogenous mass with 0.80 Gm. of calcium hydroxide and 5 cc. of water. After addition of 10 cc. of water, the mixture is let stand 15 minutes with frequent stirring. It is filtered through paper or through a glass filter, into the extraction apparatus. The precipitate and filter are washed with about 30 cc. of water, added in 3 or 4 portions. To the combined filtrates is added 1 Gm. of solid ammonium chloride, then they are extracted with a mixture of 24 cc. of benzol and 56 cc. of isopropyl alcohol for 3 hours. The solution thus obtained is evaporated to dryness and the residue twice extracted with 10-cc. portions of benzol. These extracts are transferred to a separatory funnel. After adding 5 cc. of normal sodium hydroxide the liquid is shaken vigorously and allowed to stand and separate. The aqueous layer is run into a flask and the benzol layer shaken up with another portion of 5 cc. of the sodium hydroxide and three times with 5 cc. of water. The combined aqueous extracts to which are added 1 Gm. of solid ammonium chloride (corresponding to a buffer mixture of equimolar quantities of ammonia and ammonium chloride,  $p_{\rm H}$  about 9.25) and a little ether, are shaken up and let stand some hours, or over night. The morphine crystals separate, and are washed, dried, dissolved in methyl alcohol and titrated as in the League of Nations method. The percentage of morphine is calculated by the following formula:  $(a \times 0.02852 + 0.004) \times 50$ , where a is the number of cc. of 0.1N acid used. The figure 0.004 results from the circumstance that at the  $p_{\rm H}$  of the precipitating liquid, 0.16 mg. of morphine is dissolved per cc. and the volume of the precipitating liquid is 25 cc. The advantages of the method are: (a) by the extraction with water and calcium hydroxide in the manner prescribed, morphine is extracted quantitatively so that no determination of the extract content of the opium sample is needed, (b) the morphine precipitated is considerably purer than that obtained by the lime method, (c) the precipitation takes place at a well-defined pn value and in a liquid containing no colloidal substances tending to restrain precipitation. Hence the correction for non-precipitated morphine can be made accurately. The method is tested on 5 samples of opium and compared with the results obtained by the League of Nations method, with fairly good agreement. In test of the purity of the precipitated morphine the Zeisel determination of methoxyl is made. The morphine isolated by the new method, while not absolutely pure, is considerably purer than that obtained by the lime method. In continuation of the studies, effort is being made to obtain absolutely pure morphine, and

also to permit an approximate determination of the secondary alkaloids.—H. BAGGESGAARD-RASMUSSEN and J. C. JESPERSEN. Dansk Tids. Farm., 11 (1937), 278. (C. S. L.)

Morphine and Heroin—Determination of. For the colorimetric determination of morphine and heroin separately hydrogen iodate seems the most suitable reagent; it reacts with morphine but not heroin. Use of two parts of hydrogen iodate to one of morphine produces a maximum color in ten minutes; the stronger the acid and the higher the temperature, the faster the reaction occurs. There is no reaction in alkaline media. Heroin can be quantitatively reduced to morphine by heating with 2.75% sulfuric acid at 100° for 50 minutes in a closed tube and then determined by this method. Experiments were conducted to determine the amounts of morphine and heroin adsorbed by charcoal.—R. Ito. Manshu. Ig. Z., 24 (1936), 1–44; through Chem. Abstr., 32 (1938), 726. (F. J. S.)

N'garo—Investigations on. N'garo is the native name for Cissus populnea (Ampelidaceæ). No alkaloids, heterosides or lactones can be found. It contains large quantities of tannin and mucilage. This justifies its use in the treatment of diarrheas. The roots are the most suitable parts of the plant. A dry powder obtained by precipitating an aqueous extract with ethyl alcohol is recommended as a galenic preparation.—L. VIGNOLI and J. BALANSARD. Bull. sci. pharmacol., 44 (1937), 503-507; through Chem. Abstr., 32 (1938), 1048. (F. J. S.)

Nicotic Acid—Derivatives of. In the research of new cardiac remedies the author has ottained several derivatives from nicotic acid and studied their properties: The chlormethylate of the nicotic diethylamide, colorless needles, f. p. 163–165°; the bromomethylate of the nicotic diethylamide, colorless crystals, f. p. 164–166°; the iodomethylate of the nicotic diethylamide, clear yellow needles, f. p. 157–158°; the iodomethylate of the pyridine-γ-carboxylic diethylamide, yellow needles, f. p. 138–139°; the ethyl-(diethylamine)-nicotic ethylamide, yellowish viscous liquid, f. p. 193–194° under 10 mm.; the diodomethylate of the ethyl-(diethylamino)-nicotic ethylamide, yellow needles, f. p. 203–205°. All these compounds are totally deprived of the cardiac tonic characteristics of nicotic diethylamide.—E. Gryszkiewicz-Trochimowski. Arch. chem. farm., 3 (1937), 211.

Nicotinic Acid Diethylamide and Phthalic Acid-bis-Diethylamide—Quantitative Determination of. The diethylamide of nicotinic acid has appeared in the pharmaceutical market under the names "Coramin, Ciba," "Nicordamin, Leo," "Tonocard, Astra" and others. The double diethylamide of phthalic acid ("Neospiran, Grünau") is also offered. Analysis methods are described in which the compounds are hydrolyzed and the liberated diethylamine is determined. The nicotinic acid compound is best hydrolyzed with 5N sodium hydroxide, the phthalic acid compound best with dilute hydrochloride acid. Method for Nicotinic Acid Diethylamides.—The specimen (0.400 Gm.) is mixed with 100 cc. water in a liter flask. After adding 100 cc. of the concentrated sodium hydroxide solution and a little pumice the flask is connected to a Kjeldahl distillation apparatus with receiver containing 25 cc. of 0.1N hydroxide acid. The flask is heated gently to boiling without distillation for 20 minutes, then 100 cc. distilled into the receiver. Twentyfive cubic centimeters of water are added through a small separatory funnel and about 25-cc. further distillate collected. The residual acid in the receiver is titrated with 0.1N sodium hydroxide (methyl red indicator) and a blank deducted, determined by distillation of 100 cc. of water and 100 cc. of the concentrated sodium hydroxide solution. One cc. 0.1N acid = 0.0178 Gm. of nicotinic acid diethylamide. Method for Phthalic Acid-bis-Diethylamide.—The specimen (0.300 Gm.) is boiled thirty minutes with 20 cc. 2N hydrochloric acid with refluxing. After cooling, the solution is washed with 80 cc. of water into a liter flask. Pumice and 100 cc. of concentrated sodium hydroxide are added and distillation immediately made (100 + 25 cc. as in the previous assav method) into the receiver containing 25 cc. 0.1N hydrochloric acid. The blank is determined by distilling 80 cc. water, 20 cc. 2N hydrochloric acid and 100 cc. concentrated sodium hydroxide. One cc. 0.1N acid = 0.1381 Gm. of phthalic acid-bis-diethylamide. In these distillations the tube of the condenser should dip a few mm. below the surface of the acid in the receiver. Tests of the methods in repeated analyses of five commercial specimens of the nicotinic acid compound and of two specimens of the phthalic acid compound are tabulated.—K. A. JACKEROTT and F. REIMERS. Dansk Tids. Farm., 11 (1937), 306.

Nitrogen—Determination of, in Complex Nitrogenous Substances. The use of selenium with yellow mercuric oxide as catalyst for the digestion of milk, whey, lymph and other protein-containing substances takes ten to fifteen minutes as compared to several hours when other meth-

ods are used. Mix 2 cc. of a liquid or 0.1-0.5 Gm. of a solid with 10 cc. concentrated sulfuric acid in a Kjeldahl flask, 5 Gm. potassium bisulfate, 0.5 Gm. copper sulfate, 0.05 Gm. selenium and 0.1 Gm. mercuric oxide. Heat the mixture gently for five minutes and then vigorously for five to ten minutes until a clear blue solution is obtained. After cooling and dilution with 25 cc. water, wash the solution into a 250-cc. flask and make alkaline with 25% the amount of alkali usually added after addition of sodium thiosulfate in the customary manner.—H. C. Goswam and M. R. Ray. Science and Culture, 3 (1937), 180; through Chem. Abstr., 32 (1938), 79. (F. J. S.)

Oakmoss Products—Commercial Composition of. Conclusion of an article with 19 references.—Alexander St. Pfau. Riechstoff-Ind. Kosmetik, 12 (1937), 208-209. (H. M. B.)

Orange Flower Water. The author recommends three assay standards: (a) determine free acidity in 50 cc. with cold decinormal potassium hydroxide, using phenolphthalein as indicator; (b) determine esters in the same sample by refluxing with excess of potassium hydroxide and titrating the excess; (c) determine bromine absorption by the bromoacidimetric method described in Pharm. Acta Helv., 6 (1931), 179–181. In 13 commercial samples (a) varied from 0 to 0.80 cc. potassium hydroxide, (b) from 0.60 to 2.05 cc. and (c) from 0.50 to 3.25 cc. Two samples were prepared by shaking, respectively, orange flower oil and methyl anthranilate with water; this method would insure a colorless preparation, as demanded by the Pharm. Helv. V.—L. ROSENTHALER. Pharm. Acta Helv., 11 (1936), 111–114; through Chimie & Industrie, 38 (1937), 525. (A. P.-C.)

Pai-Mien-Yao—Composition of. The narcotic pai-mien-yao contains 18.7 to 86.8% of heroin and 0.2 to 3.45% of morphine hydrochloride.—R. Ito. Manshu. Ig. Z., 24 (1936), 523-533; through Chem. Abstr., 32 (1938), 726. (F. J. S.)

Phosgene—Action of, on Hexamethylenetetramine. In reacting the formine and phosgene, independently of quantities of phosgene and temperature, there is formed but one compound in which two molecules of formine are bound with one molecule of phosgene, having the formula  $2C_0H_{12}N_4C1_2CO$ . Not one of these components can be separated; therefore it is not a simple product of addition. The compound crystallizes in methyl alcohol, f. p. 196–197°. It is very soluble in water and decomposes with heat. Total decomposition with acids gives  $CH_2O$ ,  $NH_3$  and  $CO_2$ . Applying the theory of constitution of formine the author gives the formula of this compound as a carbonyl-chloro-diformine. Since the reaction of the aqueous solution of this compound is acid it can be then titrated as an acid. With silver nitrate it yields chlorine, which proves, that it possesses in the molecule, chlorine and hydrogen ions. The hydrochloric acid dried with the carbonyl-chloro-diformine in ether yielded a dichlorhydrate having the formula:  $2C_0H_{12}N_4C1_2$ . CO.2HCl.—M. Dominikiewicz. Arch. Chem. Farm., 3 (1937), 248. (A. C. DeD.)

Picric Acid—Methods for Determining. Of the methods examined the best results were obtained in the determination of picric acid in waste waters contaminated with mineral acids and dinitrophenol by neutralizing the mixture with sodium hydroxide with the addition of ammonium hydroxide and determining the picric acid by either of the two methods of Ugnyachev and Rikhter (Chem. Abstr., 30, 16949).—N. P. Agafoshin. Zavodskaya Lab., 6 (1937), 1016–1018; through Chem. Abstr., 32 (1938), 458. (F. J. S.)

Podophyllum Resin—Test for Insoluble Matter in. The author refers to the test by which the insoluble matter is determined when 0.5 Gm. of podophyllum resin is shaken for half an hour with a mixture of 15 cc. of dilute solution of ammonia and 15 cc. of water. A table is given which shows the results obtained in respect to three samples of peltatum resin A, B and C, and one of emodi, D. The proportional figures represent the cc. of dilute solution of ammonia and of water, respectively, other conditions being as prescribed.

	15:15	20:15	25:15
A	13.2	9.2	8.0
В	10.6	8.8	8.0
С	12.2	9.6	8.6
D	50.4	$\boldsymbol{49.2}$	45.1

All the samples of *peltatum* resin failed to pass the B. P. test, but by increasing the volume of dilute ammonia solution from 15 to 20 cc. they all passed under the limit of 10% insoluble.—D. B. DOTT. *Pharm. J.*, 139 (1937), 469. (W. B. B.)

Poisons—Organic, Isolation of. The standard method for the isolation of organic poisons from viscera, stomach contents, etc., is described as a slow, laborious and often difficult method.

A new method is described, in which the minced material is treated with trichloracetic acid, and from the filtrate alkaloids present may be removed by adsorption on kaolin. The improved method is said to take less time than the older process and, from the results of experiments, yields satisfactory results. The new method, using the isolation of strychnine as an example, is described as follows: From a large excess of proteins, fats, etc., a dilute solution of strychnine hydrochloride, containing 50 mg. of the hydrochloride (equivalent to 41 mg. of strychnine) was mixed thoroughly with 400 Gm. of minced meat. After standing over night, this was ground with 100 cc. of 10% trichloracetic acid solution, and refiltered. The filtrates were mixed and shaken with 10 Gm. of kaolin which had been washed with alcohol, chloroform and finally with ether. After an hour the kaolin was filtered off and the adsorbed strychnine determined. Good results were obtained with strychnine, and equally good quantitative results were obtained using morphine and quinine; good qualitative results were obtained with nicotine and aconitine; with atropine the yield was poor; barbitone was recovered when the kaolin was replaced by animal charcoal. The method is being tested further and extended to other organic poisons.—Anon. Pharm. J., 139 (1937), 470.

(W. B. B.)

Potassium—Determination of, Note on, by the Method of Shohl and Bennett. The original method of Shohl and Bennett determined 0.1-4.0 mg. of potassium in biological materials with chloroplatinate. By some refinements in technic and careful control of certain variables H. uses this method for the determination of amounts of potassium as small as 0.04 mg.—E. R. HARTZLER. J. Biol. Chem., 122 (1937), 19; through Squibb Abstr. Bull., 11 (1938), A-56.

F. J. S.)

Potassium—Gravimetric Microdetermination of, in Presence of Sodium. Emich's adaptation of the method proposed by Fresenius in 1876 for determination of potassium as potassium chloroplatinate is shown to be capable of giving very accurate results in determining 0.5 mg. of potassium in the presence of four times as much sodium. In the presence of considerable sodium, however, the method of Smith and Gring forms a more satisfactory basis for the microchemical determination. In this method, the potassium is first precipitated as perchlorate and, after removal of excess perchloric acid, the precipitate is converted into potassium chloroplatinate by means of a very little chloroplatinic acid.—P. Wenger, Ch. Cimerman and C. J. Rzymowska. Mikrochem., 20 (1936), 1–10; through Chimie & Industrie, 38 (1937), 446. (A. P.-C.)

Potassium Iodate Volumetric Solutions—Report on the Stability of. Collaborative study showed that a solution of potassium iodate undergoes substantially no change in strength over a period of twenty months.—S. M. Berman. J. Assoc. Official Agr. Chem., 20 (1937), 590-592; through Chem. Abstr., 32 (1938), 75. (F. J. S.)

Potassium Permanganate—Arsenious Oxide in the Standardization of. Values obtained in the standardization of 0.1N potassium permanganate solutions by National Bureau of Standard's arsenious oxide No. 83, using potassium iodide or potassium iodate as a catalyst (Lang's procedure), have been compared to those obtained with sodium oxalate by the method of Fowler and Bright. The normalities found agree to within one part in 3000, which demonstrates the suitability of arsenious oxide as a direct primary standard in permanganimetry. Accurately weigh approximately 0.25 Gm. of the dried oxide and transfer to a 400-cc. beaker. Add 10 cc. of a cool 20% solution of sodium hydroxide, free from oxidizing or reducing substances. Let stand for 8 to 10 minutes, stirring occasionally. When solution is complete, add 100 cc. of water, 10 cc. of hydrochloric acid (specific gravity 1.18), and one drop of 0.0025M potassium iodate or potassium iodide. Titrate with permanganate solution until a faint pink color persists for 30 seconds. Add the last one to 1.5 cc. dropwise, allowing each drop to become decolorized before the next is introduced. Determine the volume of permanganate required to duplicate the pink color of the endpoint. This is done by adding permanganate to a solution containing the same amounts of alkali, acid and catalyst as were used in the test. The correction should not amount to much more than 0.03 cc. The end-point can also be taken with ferrous phenanthroline indicator. In this case, add one drop of a 0.025M solution of the indicator as the end-point is approached. Then add permanganate slowly until the pink color of the indicator changes to a very faint blue. The blank correction should average about 0.02 cc.-H. A. BRIGHT. Ind. Eng. Chem., Anal. Ed., 9 (1937), (E. G. V.) 577.

Precipitates—Naturally and Artificially Colored. A distinction is made between precipitates possessing color as an inherent property and those having acquired color by adsorption of a

dyestuff. In general the eye detects a little of a colored precipitate more readily than it does one that is not colored. Tartaric acid plus mercuric acetate gives a colored precipitate in the presence of Violet 5BO; theophyllin with the same mercury salt gives a colored precipitate in the presence of gentian violet; tartar emetic plus silver nitrate gives a colored precipitate in the presence of aniline blue or water blue. The characteristic precipitates obtained with the following organic compounds in the presence of a little of a 1% solution of Orange II in decinormal hydrochloric acid are described: aconitine, alypine, antipyrine, apomorphine, atropine, berberine, brucine, quinidine, quinine, cinchonine, cinchonidine, diocaine, emetine, ephedrine, ephetonine, eucain, heroine, hydratinine, hyoscyamine, codeine, cotarnine, morphine, novocain, pentocain, percain, scopolamine, stovain, strychnine; the color of all these precipitates varies from yellow to orange red. Under the same conditions colored crystals are obtained with inorganic compounds of calcium, barium, strontium, magnesium, zinc, thallium, cobalt, nickel and cadmium. Barbiturates yield colored crystals with several dyestuffs, particularly rhodamin, the color of the precipitates being generally deep red.—L. Rosenthaler. Mikrochem., 20 (1936), 85-90; through Chimie & Industrie, 38 (1937), 450.

Qualitative Separations on a Micro Scale. Analysis of the Alkali Groups. A procedure is proposed for the qualitative analysis of the alkali group from 1-mg. solid samples. The scheme provides for the detection and estimation of 10 micro Gm. of any alkali metal in mixtures containing up to 500 micro Gm. of other alkalies. Sulfate ion is removed as barium sulfate. Chloroplatinic acid is used for separation of the potassium and sodium subgroups. The analysis of the sodium subgroup follows the scheme of Noyes and Bray. In the analysis of the potassium subgroup the scheme of these authors is modified by using the reagents in different order. The work is carried out in microcentrifuge cones of clear fused quartz and of Pyrex glass.—A. A. Benedetti-Pichler and J. T. Bryant. Ind. Eng. Chem., Anal. Ed., 36 (1938), 107-110. (E. G. V.)

Riboflavin—Estimation of. Reflux a weighed sample of the given product three times with methanol acidulated with acetic acid, in the presence of carbon dioxide and in the absence of light, washing the residue with fresh methanol between extractions. Add a volume of acetone equal to the combined methanol extracts and refrigerate over night at -17.78° C. (0° F.) to precipitate flocculent impurities. Filter and condense filtrate in vacuo, adding enough water to insure removal of organic solvents and to leave the riboflavin in aqueous solution. Make this final solution up to a definite volume and assay. If any turbidity is present, it must be removed by filtration, or if of fatty origin it may be removed by ether extraction. This method is also applicable to liquid samples, but no refluxing is required. The methanol carries the riboflavin into solution and the acetone precipitates out extraneous matter from the methanol. In the authors' method sodium fluorescein in an aqueous solution of 0.001% is used for making up the fluorescent standards, and the fluorescence of the unknown aqueous solution, purified so as to contain only riboflavin as the fluorescing agent, is compared with the sodium fluorescein standards under the ultraviolet light. The fluorescein standards are calibrated by means of a solution of riboflavin. The fluorescein standards are made up in terms of 0.1 to 1.0 gamma of riboflavin per cc. Higher concentrations are difficult to match, owing to the high intensity of the fluorescence of the green component of the sodium fluorescein. The purified unknown solution is diluted so that it falls into the range of 0.1 to 1.0 gamma per cc. and the various dilutions are multiplied by the dilution factor to obtain the flavin concentration. In making the assay, various dilutions of the unknown sample are made up to 50 cc., placed in French square bottles of 60 cc. (2-ounce) capacity, and compared under ultraviolet light with the fluorescein standards contained in similar bottles.—S. M. Weisberg and I. LEVIN. Ind. Eng. Chem., Anal. Ed., 9 (1937), 523.

Senega—Detection of Preparations of, in Mixtures. If a preparation of senega root is present in a pharmaceutical mixture, the following method may be used for qualitatively detecting the senega: To 2 cc. of the solution to be tested add 2 cc. of a 10% aqueous solution of sodium nitrite, and 3 drops of concentrated sulfuric acid. At the end of 30 seconds add 20 cc. of N/1 sodium carbonate. In the presence of senega a reddish color will be obtained, the tint varying from deep claret according to the amount of senega present. It is not suggested that this test is specific for senega, since several phenols give colored reactions with the reagents used, but these are not likely to be present in such preparations as cough mixtures, etc., in which it may be desired to test for the presence of senega.—J. RAE. *Pharm. J.*, 139 (1937), 466. (W. B. B.)

Silica—Gravimetric Determination of Small Amounts of, as Pyramidone Silicomolybdate. In acid solutions silicic acid reacts with ammonium molybdate to give a precipitate containing 3 mols. of ammonia, 1 of silica and 12 of MoO<sub>3</sub>. The formation of this yellow compound has been the basis of several methods for determining small quantities of dissolved silicic acid. The silicomolybdic acid forms insoluble salts with organic bases as well as with ammonia and typical compounds are formed with coniine, pyridine, pyramidone, etc. The salt with pyramidone yields a weighable precipitate with very small quantities of silica. If phosphoric acid is present, it must be removed, preferably by the basic acetate procedure after the addition of sufficient ferric chloride. To determine the silica content of animal tissue, fuse 0.1 Gm. of the dry powder in a platinum crucible with 0.25 Gm. of sodium carbonate; extract the cooled melt with water and make acid to methyl orange (outside indicator) with normal sulfuric acid; make to 100 cc., add 10 cc. each of 1% ferric chloride in fiftieth-normal hydrochloric acid and 1.5% sodium acetate in thirty-fifth normal sodium hydroxide; transfer to a 500-cc. Erlenmeyer flask and rapidly bring to a boil; filter and to the measured filtrate add 5 cc. of 5% ammonium molybdate solution and 2.5 cc. of ten times normal sulfuric acid; heat for 5 minutes in a hot water-bath and precipitate with ice-cold decinormal hydrochloric acid; dry at 60° to 70° C. for 30 minutes and weigh. The results obtained from precipitates containing 0.25 to 0.30 mg, of silicate indicate that the molecular weight of the precipitate is 2680. The molecular weight of the compound (C<sub>18</sub>H<sub>17</sub>N<sub>3</sub>O)<sub>3</sub>H<sub>8</sub>Si(Mo<sub>2</sub>O<sub>7</sub>)6.6H<sub>2</sub>O is 2661. The determination of the nitrogen and the silica indicates that this formula is correct.— E. J. King and J. L. Watson. Mikrochem., 20 (1936), 49-56; through Chimie & Industrie, 38 (1937), 446.

Soma-Homa—Holy Plant of the Indians and Persians. The author discusses the mythology of Soma, one of the principal gods of the ancient Indians. A plant often mentioned in ancient writings in connection with sacrifices to this god has also been named Soma and is probably the same as the Haoma in Persian mythology. Various conflicting statements appear in the literature as to the identity of this plant. The author received a cutting of Sarcostemma acidum from Malabar. This plant is used by the Nambudiri Indians in Soma sacrifices at the present time. The cutting was planted and yielded after cultivation, about 100 plants which were used in a preliminary phytochemical investigation. Sarcostemma acidum is a hanging plant with a fleshy green leafless stem not over 3 or 4 mm. thick. The stem contains a white latex having a strongly acid, slightly bitter but not astringent taste. The stems were comminuted and pressed and the juice as well as an alcoholic extract of the marc after expression were investigated. The acid reaction seems to be due to malic acid; citric, oxalic and tartaric acid were not found. The carbohydrate consists of small quantities or reducing sugar and saccharose. Tannins appear to be present by the ferric chloride test but could not be detected by the ammoniacal silver nitrate test. The tannin question needs further investigation and clarification. A phytosterin with a melting point of about 142° and a glucoside precipitated with basic lead acetate were found. A small amount of alkaloid (300 Gm. of material yielded less than 1 mg.) which gave precipitates with picric acid, potassium-mercuric iodide and potassium iodide was also found.—L. VAN ITALLIE. Pharm. Weekblad, (E. H. W.) 74 (1937), 5.

Starch—Direct Determination of. Th. v. Fellenberg in proving the applicability of his method for the determination of starch in table mustard made several improvements. The revised method is described.—Th. v. Fellenberg. Mitt. Lebensm. Hyg., 28 (1937), 111-115; through Chem. Abstr., 32 (1938), 259. (F. J. S.)

Sulfates—New Modification of Andrews' Method for the Determination of, in Water. The L. W. Andrews' method for the determination of sulfates in water was verified and the following conclusions obtained: The variation in the analysis arises in the principle of the solubility of barium chromate and its instability; it may also be caused by a great amount of alkalinity of the barium chromate. The error proving the solubility of the barium chromate can be seen by the diminution of the dissociation of the barium chromate by the addition of an excess of potassium bichromate. The harmful influence of the instability of the barium chromate can be eliminated by the use of standard solutions of barium chloride and potassium bichromate. The organic substances do not have a harmful action in the determination of sulfates, if they do not exceed 8 mg. of oxygen, on the contrary the water must be purified, preferably by active charcoal. A new modification of Andrews' method has been presented for the determination of sulfates by

means of weight. Two hours are required to make four determinations.—W. SKORECKI. Arch. Chem. Farm., 3 (1937), 218. (A. C. DeD.)

Sulfur—Determination of, in Drugs. Free or loosely bound sulfur can be determined in drug mixtures by a modification of the method of Schulek (cf. Chem. Abstr., 17, 3465; 19, 1829). To a portion of the mixture containing about 1-25 mg. sulfur in a fifty-cc. flask add 0.2 Gm. of potassium cyanide and dissolve in 10-20 drops water. Add five cc. pure acetone and boil the mixture with some pumice under a reflux condenser for 15-20 minutes. Dissolve the residue in water (after previously removing acetone on the water-bath) and fill up to 60 cc. Traces of fat or dyes can be removed by following treatment. Boil the residue with fifteen cc. water, shake with one Gm. magnesium oxide, filter. Now add one Gm. of boric acid and some pumice to the filtrate and remove hydrogen cyanide by boiling for ten minutes. Pour the remaining 20-25 cc. into a 100-cc. Erlenmeyer flask with glass stopper, add 5 cc. 20% phosphoric acid and freshly made bromine water until the liquid remains yellow. Remove excess bromine with one or two cc. of a five per cent phenol solution, add one Gm. KI and titrate with thiosulfate. Sulfur organic combination can be determined by the distillation method of Dorza (Chem. Abstr., 28, 1813) or that of Schulekand Szlatinay (Chem. Abstr., 31, 55101) or by a new method devised especially for metansulfonic acids as follows: Place the substance in a Jena-glass centrifuging tube, dissolve it in 30 cc. water, add 5 cc. 10% barium chloride, two cc. hydrogen peroxide and 5 cc. 10% hydrochloric acid. Let stand twelve hours, centrifuge, wash the precipitate by centrifugation, add two cc. concentrated sulfuric acid and some perhydrol, boil until it dissolves, dilute, filter the precipitate, wash out, ignite and weigh. Sulfonic acid sulfur can be determined by ignition in a specially constructed Berthelot bomb (cf. Dorzsa, Chem. Abstr., 28, 1813 and Clauder, Chem. Abstr., 29, 3555), in which halogen also can be determined. The bomb is made of V2A steel. Add paraffin oil to the drug sufficient to make up the weight to 0.7-0.8 Gm., press a platinum wire into the mixture and add 10 cc. of a 3% fresh hydrogen peroxide solution to the bomb. Ignite in an oxygen atmosphere of twenty to twenty-five atmosphere pressure. Let out gases slowly after five or six minutes through a hydrogen peroxide containing solution of barium chloride to absorb possible traces of sulfur dioxide. Wash out the bomb and precipitate sulfates according to Winkler. In analyzing nitrogen containing substances, remove the formed nitric acid but repeat evaporation of the solution with hydrochloric acid. For the determination of total sulfur decomposition in a Kjeldahl flask with concentrated nitric acid is proposed (Schulek and Szlatinay, Chem. Abstr., 31, 5510).—Elmer Schulek and Otto Clauder. Magyar Gyógyszerésztud. Társaság Értesítője, 13 (1937), 795-811; through Chem. Abstr., 32 (1938), 1401. (F. J. S.)

Sulfur-Organic, Microdetermination of. For precipitation a thin-walled crucible (preferably with black interior glazing) of about 15 cc. capacity and a small porcelain filter stick are used. The crucible should be cleaned of precipitate from previous analyses and rinsed with distilled water, the exterior wiped with clean cloth. The filter stick may be cleaned by brushing off any adhering precipitate and then washed by sucking water through it in both directions to rid the porous filter surface of precipitate as much as possible. Reverse washing is facilitated by use of a suitable "adapter." The crucible containing the filter stick should be dried at 150° to 200° C. for about 10 minutes. The crucible is then placed on a piece of clean metal, covered and allowed to cool. A metal cooling block, with a glass cover for protection against dust, allows more secure handling of the crucible. After drying, the crucible should be handled with suitable (curved) metal forceps until after weighing. After about 15 to 20 minutes, when the crucible has cooled to room temperature, it is placed on the balance for about 5 to 10 minutes and then weighed to onehundredth mg. The filter stick is then removed from the crucible and kept in the balance until needed for filtration. Combustion is carried out according to Pregl. Then the combustion tube is either clamped in a vertical position over the crucible, or held horizontally, and 1 to 2 cc. of water acidulated with hydrochloric acid (1 to 300) are blown into the wide end from a wash bottle fitted with a fine nozzle. For convenience a length of thin rubber tubing may be attached to the mouthpiece of the wash bottle. The combustion tube is rotated in such a manner as to wash the entire inside surface, and the wash liquid is then transferred to the crucible by holding the tube vertical with the capillary end in the crucible. Care must be taken not to lose wash liquid outside the crucible. Since the liquid flows out through the capillary rather slowly, expulsion may be hastened by blowing into the combustion tube through an air filter to which a length of flexible rubber tubing is attached for more convenient operation. This process is repeated four to five

times to ensure complete rinsing. The amount of wash liquid employed should be so adjusted that the crucible will not become much more than two-thirds filled. For checking the completeness of the rinsing, another larger washing of the combustion tube may be collected in the protect-to the crucible during subsequent filtration. It has been found, however, in several hundred sulfur determinations, that complete removal of the sulfuric acid produced in the combustion can easily be affected with three to four washings of 1 to 2 cc. as described. For precipitation of the barium sulfate the crucible is placed on a suitable steam-bath, and about 0.5 cc. of 10% barium chloride solution is added dropwise. Any combustion residue in the boat is extracted with small portions of wash liquid and filtered into the crucible. After about 15 minutes the solution is allowed to cool and filtration is carried out as described below. In case of very low percentages of sulfur, the volume should be reduced before filtration. For filtration the filter stick is attached to a suitable suction device, and sufficient suction is applied so that the liquid filters at the rate of 1 to 2 drops per second. The precipitate is then washed in such a manner that the whole inside surface of the crucible is thoroughly moistened and rinsed 3 to 4 times with small portions of acidulated water, making a total wash volume of about 3 cc. The filtrate should be collected and examined in a separate vessel of the suction device, and must be perfectly clear. Traces of barium sulfate may pass through a filter used for the first time; in this case the filtrate is poured back into the crucible and refiltered before washing. The filter is then detached and left in the crucible, which is wiped outside with a clean moist cloth and placed in the oven. With moist precipitate in the crucible, drying should be started below 100° C. to allow the moisture to be driven off slowly without spattering. When thoroughly dried, the crucible is allowed to cool and is weighed as before. Many trials have shown that another washing with 2 to 3 cc. as before should not cause a decrease of weight of more than 0.01 to 0.02 mg.—A. J. BAILEY. Ind. Eng. Chem., Anal. Ed., 9 (1937), 491.

Tin—Iodometric Determination of. To overcome the oxidation of Sn<sup>++</sup> it is proposed to dissolve the sample under a Göckel valve containing sodium bicarbonate solution so that when the solution cools an atmosphere of carbon dioxide is provided. Then to the Sn<sup>++</sup> solution a known quantity of potassium borate together with an excess of potassium iodide is added. In this way the Sn<sup>++</sup> solution comes in contact with a known quantity of iodine and the excess can be determined by titration with sodium thiosulfate.—M. Hegedüs. Z. anal. Chem., 110 (1937), 338-348; through Chem. Abstr., 32 (1938), 78. (F. J. S.)

Vacuum Distillation—Fractionating Device for. The device described for removing fractions of distillate, without interrupting the course of a distillation under reduced pressure, is a modification of that designed by Bogert.—E. NOONAN. Ind. Eng. Chem., Anal. Ed., 30 (1938), 34.

Veronal and Luminal—Extraction and Detection of. For detection of veronal and luminal the colorimetric method of Zwikker (*Pharm. Weekblad*, 68 (1931), 975-983) was chosen from a number tested, and for determination the method of Vieböck and Fuchs (*Pharm. Monatsh.*, 15 (1934), 39-40). For extraction from ground flesh, the method of Wasicky (*Pharm. Ztg.*, 16 (1934), 20) gave the highest yields, but the product was impure. For extraction from urine, the method of Straub and Mihalovits (*Pharm. Zentralhalle*, 75 (1934), 226-288) was the most satisfactory.—J. H. Krepelka and V. Švarc. *Coll. Trav. Chim. Tchécoslov.*, 8 (1936), 191-206; through *Chimie & Industrie*, 38 (1937), 524-525. (A. P.-C.)

Volatile Oil Drugs—Determination of the Volatile Oil Content of. Two requirements are necessary for an ideal method for the determination of volatile oils in drugs: (1) that the method must be simple enough to be carried out with the customary apparatus found in the pharmacy and (2) that it must give reliable results. The author discusses the method of Mijnhardt (Pharm. Abstracts, 2 (1936), 456) and points out several possible sources of error in this method. The following new method is suggested: A quantity of material containing between 100 and 200 mg. of volatile oil is ground to a powder in a hand mill and then placed in an Erlenmeyer flask containing 50 cc. of petroleum ether. (If the quantity of drug exceeds 20 Gm., a 500-cc. flask with 200 cc. of petroleum ether should be used.) The flask is then heated for one hour on the water-bath under a reflux condenser. After cooling the contents are filtered through a small filter (7 cm. in diameter) placed in a large funnel (to prevent creeping of the volatile oil). The filtrate is collected in a 300-cc. Erlenmeyer flask, the contents of the original flask and filter being washed with 15 cc.

of petroleum ether. The filter with its contents is returned to the original flask and the process repeated three times. (A total of 4 filters is thus employed.) The united filtrates and washings are freed from a greater portion of the solvent by distillation on the water-bath at a temperature not exceeding 50° C., the distillation being continued until the volume of the residue is about 10 cc. This residue is transferred quantitatively with the aid of 15 cc. of petroleum ether and 425 cc. of water to a 1-liter flask. After the addition of a few pieces of pumice stone and 60 Gm. of sodium chloride, the contents are distilled by boiling vigorously over a Bunsen burner, until bumping due to the crystallization of the salt commences, care being taken that the temperature of the distillate does not rise above 20° (condenser 40 cm.; water as cold as possible). The tube connecting the flask and the condenser, and the condenser itself, are washed three times with 5 cc. of petroleum ether, the washings being added to the distillate. 120 Gm. of sodium chloride are then dissolved in the distillate and the whole allowed to stand until the petroleum ether layer has completely separated. As much as possible of the liquid is transferred to a 100-cc. separatory funnel, and the aqueous solution allowed to run off. This is repeated until all of the distillate has been collected in the separatory funnel. The empty flask is washed, the washings being transferred to the separatory funnel and the water separated. The united aqueous liquids are shaken out with 15 cc. of petroleum ether, and as before, the petroleum ether is added to the separatory funnel, the remaining aqueous portion being placed in a flask with 1 Gm. of norite (the norite must be free from petroleum ether-soluble substances). After vigorous agitation the norite is separated on a suction filter. The contents of the separatory funnel are treated with 5 Gm. of dried sodium sulfate, shaken for a few minutes and filtered through a small filter (placed in a large funnel) into a dried and tared 100-cc, wide-mouthed Erlenmeyer flask, into which 1 Gm, of liquid paraffin (carefully weighed) has previously been introduced. The filter with the norite is then placed in the separatory funnel and vigorously shaken with 5 cc. of petroleum ether which is then filtered into the tared flask. This treatment with petroleum ether is then repeated three times after which the solvent is distilled from the flask on the water-bath at a temperature not exceeding 50° C., until the residue has a volume of about 10 cc. The residue is allowed to cool to room temperature, the flask removed from the distillation set-up and the volatile oil brought to constant weight with a current of dry air. The flask is closed with a two-holed stopper from which one tube (the inlet) leads to a wash bottle containing strong sulfuric acid and the other to an inverted 10-liter flask containing water. The dry air is passed at the rate of 5 cc. per second and after the volume of the residue has reached about 2 cc. the first weighing is made. Following this about 1/2 liter of air is passed through the flask and another weighing made. This is repeated until the loss in weight does not exceed 5 mg., when the end-point is assumed to be reached. Comparative data are given for various drugs using this method and the method of the Codex Medicamentorum, as well as the Dutch Spice method. Data on the following drugs are also given: clove, cascarilla, cinnamon, orange peel, chamomillæ vulgaris, lavender, boldo, buchu, jaborandi, rosemary, anise, star anise, caraway, cumin, fennel, juniper, parsley fruit, menthæ crisp., thyme, sassafras, galanga and nutmeg.—H. J. VAN GIFFEN. Pharm. Weekblad, 74 (1937), 812. (E. H. W.)

Warfare Chemicals—Objective Methods for the Determination of. The author suggests the following analytical scheme: I. Orientation (reactions not specific)— (a) with benzaldehydamine; (b) with sudan; (c) with sodium sulfide. II. Orientation (specific reactions on determined elements or radicles)— (a) halogens; (b) sulfur; (c) arsenic; (d) iron; (e) nitrogen; (f) cyanogen group. III. Boiling-point determinations. IV. Specific reactions on determined warfare chemicals: vesicants (mustard gas, lewisite); suffocants (phosgene, diphosgene, chlorpicrine) sternutatories (adamsite, Clark II); lacrimogenics (chloracetophenone). Methods are discussed in detail.—D. H. Wester. Pharm. Weekblad, 74 (1937), 742. (E. H. W.)

## PHARMACOGNOSY

# VEGETABLE DRUGS

Aphrodisiacs—Some. The author has selected for description, the following 8 drugs which are rather widely used by the laity: abacate, Laurus persea L. or Persea gratissima Gaertn., active as grown naturally but practically valueless as cultivated; abacatirana, Nectandra Amazonica Mart., has little aphrodisiac action and is dangerous; abio, Lucuma dissepala (Krause) Ducke, L. parviflora (Benth.) Miq., and L. macrocarpa Hub. depends for its activity on its con-

tent of myristicin; ayry, Toxophoenix aculeatissima Schott., is used in breeding horses; mais, Stigmata maidis, is very active and also dangerous; para nut, Bertholettia excelsa H. B. K. and B. nobilis Miers, is very weak requiring large amounts for aphrodisiac action; taperebá-assu, Poupartia Amazonica Ducke and closely related species, contains an alkaloid similar in action to yohimbine. Many drugs are put on the market as patent medicines for which unwarranted claims are made but which act chiefly by suggestion rather than by any direct action.—
F. W. Freise. Scientia Pharm., 8 (1937), 146. (M. F. W. D.)

Austrian Drugs—Standardization of. The article is the report of the committee for furthering the cultivation of drugs in Austria. The report describes the methods by which the following determinations are to be made: Volatile oil content, bitterness, color, moisture in drugs, viscosity (by the capillary viscosimeter) and the volume-weight (weight of 100 cc. of dry drug). Some pieces of apparatus are illustrated. A table is included giving the average values of the constants for marshmallow leaves, wormwood leaves, mullein leaves, digitalis leaves, peppermint leaves, fennel seed, wormwood plant and peppermint plant.—Anon. Scientia Pharm., 8 (1937), 122; also Pharm. Monatsh., 18 (1937), 167-172. (M. F. W. D.)

Derris and Lonchocarpus Powders—Differentiation between. A large number of samples of powdered derris root and powdered lonchocarpus root were examined microscopically with the object in view of finding a definite way to differentiate between these two powders. A brief description of the general characteristics of these two powders is given. The woody elements of both powders, examined in chloral hydrate do not offer a distinct means of differentiation. The starch, however, does offer an explicit way of distinguishing between these two species of plants. An accurate description of the starches together with drawings is included. Besides the difference in shape, the greater size of the lonchocarpus starch is evident. A graph of the grain size and occurrence is given. The starch offers a ready means for differentiating between these two powders and also a means by which the addition of one to the other may be detected.—A. DIAKONOFF. Pharm. Weekblad, 74 (1937), 901.

Drugs—Obsolete, Pharmacognosy of. An address before the Austrian Pharmaceutical Society in the form of a historical review.—J. A. Hefliger. *Pharm. Monatsh.*, 18 (1937), 193-199. (H. M. B.)

Fennel. H. Kutter describes two insects found in the grain of fennel, Systole albipernnis and Tetrastichus rapo, which deteriorate the fennel.—Anon. J. suisse pharm., 11 (1916); through Farm. ital., 14 (1936), 396. (A. C. DeD.)

Folia Orthosiphonis and Rhizoma Curcumæ Javanicæ—Requirements for. From the viewpoint of regulation the author suggests the following standards for these Netherlands-Indian drugs: Folia Orthosiphonis Javanici or Javanese Orthosiphonis leaves (koemis koetjing); the unground leaves of Orthosiphon stamineus Benth. (synonym Orthosiphon grandiflorus Bold.); color: light green (petioles and veins purple); odor: aromatic, characteristic; taste: saline, somewhat bitter and astringent; unavoidable impurities: not more than 2% of stem fragments more than 1 mm. thick; moisture: not more than 13%; ash: 8-12% (with high potassium content); acid-insoluble ash (sand): not more than 2%; aqueous extract: not less than 40% (calculated on the dried drug). Rhizoma Curcumæ Javanicæ or Javanese Curcuma Root (temoe lawak); the rhizome of Curcuma xanthorrhiza, Roxb., in thin, unground disks; color: orange-yellow to orange-brown; odor: aromatic, characteristic; taste: spicy and somewhat bitter; moisture: not more than 12%; ash: 3-7%; acid-insoluble ash (sand): not more than 1%; volatile oil content: not less than 5%.—P. A. Rowaan. Pharm. Weekblad, 74 (1937), 910. (E. H. W.)

Majoran—an Adulteration of. The author describes in detail a sample of drug bought under the name of majoran. While it resembled majoran (Origanum majorana L.) in many characteristics, the leaves were somewhat smaller and of a different appearance. The specimen was also examined carefully under the microscope and drawings accompanied by descriptions are included. By comparison with descriptions in several botanical texts, the drug was found to be identical with Organum hirtum.—M. Fichter. Pharm. Acta Helv., 12 (1937), 363. (M. F. W. D.)

Manihot Utilissima and Mercurialis Annua or Perrenis—Therapeutically Utilizable Constituents of. Of the above-mentioned *Euphorbia* representatives in the Brazilian flora, one of the most important is the manioc tuber, the meal of which serves as human food as well as a source of starch, while *Mercurialis annua*, though in reality a troublesome weed, yields preparations employed to some extent medicinally, like the expressed juice of the manioc, for the same purpose

as the latter, hence may have similar or identical constituents. The juice of M. utilissima presents a milky, faintly opalescent liquid smelling distinctly like bitter almonds, and forming on shaking a strongly foaming product with  $d_4^{20}$  1.015 and viscosity ( $\eta$ ) 3.65 centipoises, in which the following constituents were established: water 91, essential oil 0.13, gum-like substances 2.3, saponins 1.14, glucoside 1.66, inorganic salts, traces of fat and dyes 3.80%. The essential oil is faintly yellow with celery-like odor, scratchy-burning taste, with  $d_4^{20}$  0.898-0.911,  $n_D^{20}$  1.4447; it contains sulfur in organic combination and operates as a moderate diuretic. The saponin in a dilution of 1:110,000 effects complete hemolysis. The glucoside is split by a ferment into dglucose, hydrocyanic acid and a substance of undetermined constitution, the analysis of which points to the formula  $C_{1\delta}H_{10}O_6$ . This substance melts at 144-146°, sublimes undecomposed at 238°, is easily soluble in water and yields with ferric chloride an evanescent blue. The expressed juice of the Mercurialis tubers is given as an empiric medicine in thin coffee, maté or sweetened brandy. It presents a reddish liquid (from unpeeled tubers), yellowish white (from peeled tubers) milky, and smells somewhat like rutabaga juice, tastes scratchy,  $d_4^{20}$  1.015–1.022, viscosity ( $\eta$ ) 3.55 centipoises, and consists of water 85.5, essential oil 0.08, fatty oil 0.12, gummy substances 2.8, saponins 2.25, glucoside 1.35, tannins 3.35, sugar 1.49, mineral substances 2.95% in addition to traces of an alkaloid and reddish yellow dyestuff. The faintly yellow essential oil is odorless, scratchy to the taste, contains sulfur and acts as a diuretic. 90% of the saponin is neutral in character; the glucoside like that from manioc splits off hydrocyanic acid, in addition to d-glucose and a third substance as a colorless bitter-tasting amorphous product little soluble in cold water, quite soluble in hot water; it melts at 172° and sublimes 226-228°; yields in aqueous solution a permanently faint blue color. Its composition is identical with that from manioc, and it is a diuretic. The sugar is maltose. Among the diseases in which these plant juices find therapeutic applications are ascites, eczema, ascarides, scabies, Sycosis staphylogenes, etc.—Friedrich W. FREISE. Süddeut. Apoth.-Ztg., 77 (1937), 1007-1008; through Chem. Abstr., 32 (1938), 2288. (F. J. S.)

Root—Famous. In Italy licorice root is used only for medicinal purposes, in the form of extract or powder. The licorice is Glycyrrhiza glabra of Linnæus, belonging to Leguminosæ family. The official part is the root. It has a sweetish taste; but becomes bitter after chewing. The root should be collected in the autumn or in spring. The drying of the drug should be in the shade. It is grown in clay regions, woody-land of the southern part of Italy especially Sicily and Calabria. The principal component of the drug is glycyrrhizin which is an amorphous substance, yellow in color when powdered. Licorice has been recommended for bronchial catarrh and inflammatory fevers.—E. Flaccomio. Farm. ital., 14 (1936), 408. (A. C. DeD.)

Sterculia Lurida—Fruit and Oil of. Sterculia lurida F. Muell, is a tree, indigenous to Australia but it grows well in Palermo and the authors obtained their material from the botanic gardens there. The fruit is a large woody follicle from 12 to 16 cm. long and about 11 cm. in circumference. It weighs from 35 to 45 Gm. and contains 35 to 50 seeds. Each seed is in a hairy cell, is of ovid shape and pale yellow color with an average weight of 0.24 Gm. The seeds contain 24% of oil, 15% of starch and 3.3% of ash. The oil obtained by extraction with carbon tetrachloride is of an orange-yellow color which is bleached to lemon-yellow by fuller's earth. The expressed oil has a fresh odor and pleasant taste but the extracted oil is tasteless and flat. It is liquid at ordinary temperatures but solidifies on cooling to 3° to 8° C. and melts again at 10° to 14° C., sp. gr., 0.952; n<sup>40° C</sup>., 1.4640; acid number, 15; saponification number, 209; ester number, 194; acetyl value, 38; iodine value, 112. The solid fatty acids had a m. p. 53° C. and consisted of 75% of stearic acid and 25% of palmitic acid; the liquid fatty acids melted at 4° C. and were a mixture of oleic acid with other acids of higher molecular weight and greater degree of unsaturation.—E. Corsini and R. Indovina. Ann. chim. appl. Roma, 27 (1937), 263; through Quart. J. Pharm. Pharmacol., 10 (1937), 565.

Sunflower Seeds and Castor Beans—Thermal Effect on the Drying of, under Laboratory Conditions. A charge of 4.5 Kg. of the seeds and beans was heated, with mechanical stirring, in a wire-gauze drum set in a tile oven at 55–350° for 2–30 minutes. Before drying, the moisture content of seeds was 16.1% and of beans 14.3%. The hygroscopically stable moisture at 75% of the relative air moisture was 9.2 and 6%, respectively. The rate of drying increases with higher temperatures. Thus, the moisture content of seeds was reduced 6% at 150° in five minutes (0.33 calories per Kg. per second) and 5.5% (to 10.6%) at 250° in one minute (0.78 calories per Kg.

per second). Analogous results were obtained in drying of castor beans. The quality of seeds and beans and that of the pressed oils (iodine value, density, clarity, taste, etc.) are not affected by drying up to 350° for definite periods of time. The optimum duration of heating with a loss of moisture by 6-7% (from 16 to 9-10%) is at 350° for two minutes (0.8 calories per Kg. per second), at 250° for three minutes (0.5 calories per Kg. per second) and at 150° for five minutes. The work is being continued.—F. Gogolev. Masloboino Zhirovoe Delo, 13 (1937), 18-20; through Chem. Abstr., 32 (1938), 821. (F. J. S.)

Vegetable Drugs—Short Method for the Determination of Volatile Oil in. The author has devised a shorter method than the one originally devised by him (Pharm. Weekblad, 74 (1937), 812) which required about  $6^{1}/_{2}$  hours. The shorter method requires about three hours but gives lower results than the longer method and higher results than the (Netherlands) Spice method. The shorter method is carried out as follows: A quantity of powdered drug containing from 100 to 200 mg. of volatile oil is introduced into a liter flask, 50 cc. of petroleum ether (b. p. not higher than 35° C.) are added and heated 45 minutes at 60° C. on a water-bath under a reflux condenser. If the drug runs large in volume, enough additional petroleum ether should be added to cover the drug completely. After cooling, 35 Gm. of sodium chloride and 365 cc. of water and a few pieces of pumice stone are added and the whole distilled until 300 cc. of aqueous distillate has passed over. A 40-cm. condenser is employed and the flask is shaken after about 1/4 of the distillate has passed over to wash down any material adhering to the walls. After the distillation the condenser is washed with small portions (about 10 cc.) of petroleum ether which are added to the distillate. About 110 Gm. of salt are added to the distillate and the whole shaken vigorously. The petroleum-ether solution is separated by means of a separatory funnel, dried with 2 Gm. of sodium sulfate and filtered into a dry, tared, wide-mouth Erlenmeyer flask containing 1 Gm. (carefully weighed) of liquid paraffin. The brine is washed twice with 10-cc. portions of petroleum ether, the washings being dried and added to the filtrate. The flask is now placed on a water-bath and the solvent distilled off until a few cc. remain. (The temperature of the water-bath should not exceed 60° C.) The remainder of the solvent is then removed with a current of dry air, the residue being weighed at intervals. When the loss is not over 5 mg, the end-point is assumed to have been reached. The treatment with dry air is carried out as described in the author's previous paper. Comparative results are given for caraway, anise, juniper, buchu, thyme, cinnamon, galangal and Chamomillæ vulgaris using the Spice method, the author's original method and his shortened method.—H. J. van Giffen. Pharm. Weekblad, 74 (1937), 954. (E. H. W.)

# **PHARMACY**

#### GALENICAL

Cardiazole Ampuls. Cardiazole solutions, prepared according to different methods, were examined chemically and pharmacologically for their respective activities. Chemically, no change was observed in the solutions investigated. Pharmacologically, cardiazole solutions were evaluated on rats in pernocton anesthesia (4th degree anesthesia via Schön). As the test, the minimum waking action (1st degree via v. Nyary) was employed. Cardiazole solutions sterilized 15 minutes at 120° or two hours at 120° in air, carbon dioxide or oxygen atmosphere, or degerminated three times 30 minutes at 100° in carbon dioxide atmosphere, suffer no loss of their original activity after six months' storage. Degermination for 15 minutes at 120° in nitrogen atmosphere leads to no loss, as probably is also the case on heating two hours at 120°. Twenty-four references.—W. Lühr and H. G. Rietschel. Pharm. Zentralhalle, 79 (1938), 1, 19; through Chem. Abstr., 32 (1938), 2688. (F. J. S.)

Cocaine Solutions—Stability of, Influence of Sterilization on. With the surface sensibility of the human cornea as reference value, it was found that boiling at  $100^{\circ}$  did not affect the anesthetic activity of an aqueous solution of cocaine. Change in  $p_{\rm H}$  due to boiling in a glass container producing no alkali is of no significance in drop anesthesia. Increase in  $p_{\rm H}$  to 7.39 does not increase the anesthetic property of cocaine applied locally.—W. Nordlöw. Acta Ophthalmol., 15 (1937), 84-95; through Chem. Abstr., 32 (1938), 728. (F. J. S.)

Digitalis Pills—Stability of. A comparison is made of the stability of digitalis pills made by the Swedish Medical Control Board's 1930 formula (binders: glycerol and sugar syrup) with the 1933 formula (binders: cocoa butter and almond oil). The loss in frog dose over 12 months

was about 60% in the case of the formula made with glycerol, whereas the cocoa butter formula showed no loss in this time. The practical keeping qualities of digitalis pills made with fat are limited by the keeping qualities of the fat.—K. Edner, P. Johansson and G. Aberg. Farm. Revy, 36 (1937), 825. (C. S. L.)

Enteric Coating—New, and a Laboratory Method for Its Control. Some of the previous reports on enteric coating are briefly discussed, together with a discussion of what is required of such a coating. Attention is directed to the fact that most workers have assumed that intestinal contents are always alkaline. Physiological aspects of the problem seem to indicate that an "efficient enteric coating should be stable for nearly six hours and should then begin to disintegrate quickly regardless of the  $p_{\rm H}$  of the body fluid" in which it happens to be. The authors reached the following conclusions: Efficient enteric coated tablets of the type based upon the principle of timed disintegration should begin to break up in vivo after six hours, and should be completely disintegrated within eight hours. The disintegration time of the coating herein described may easily be controlled because of the moisture-absorbing and swelling properties of its vegetable components. A laboratory method of control for enteric coated tablets may be established by correlating the disintegration time in vitro and that in vivo of enteric coated barium sulfate tablets found to be efficient radiographically.—A. G. Worton, G. F. Kempf, P. L. Burrin and F. E. Bibbins. J. Am. Pharm. Assoc., 27 (1938), 21.

Extracts, Concentrated, and Their Preparations. Methods for the preparation of concentrated extracts are of two general types. The first involves the extraction of the drug with an unlimited quantity of menstruum and subsequent evaporation of this to the required concentration. The second consists of extraction with a limited volume of menstruum, so that a concentrated product is obtained without evaporation. A number of methods involving concentration which include maceration, percolation, reserved macerate process, reserved percolate process, methods employing small volumes of solvent, repercolation and continuous extraction are given.—
Colin Gunn. Retail Chemist (April 1938), 40. (A. C. DeD.)

Infusions—Preservation of Concentrated and Fresh. I. Application of Heat and Alcohol. The authors summarize their findings as follows: 1. The fact is confirmed that preserved or concentrated infusions are not as good as freshly made infusions. 2. If it is necessary to store infusions, then in descending order or preference the following are recommended. (a) Fresh infusions rendered sterile by boiling or autoclaving, and stored and drawn off aseptically. (b) Concentrated infusions made with water, rendered sterile by boiling or autoclaving and stored and drawn off aseptically. (c) Concentrated infusions preserved with the minimum quantity of alcohol, as follows: For concentrated infusions of clove and senna alcohol (10%), for all the other official concentrated infusions alcohol (15%). 3. It is shown that fresh infusions of quassia and calumba should be boiled after preparation. The same procedure could with advantage be applied to fresh infusion of senna. 4. It is suggested that the resistant organisms in calumba be destroyed either by boiling the aqueous extracts during preparation of the concentrated infusion, or by boiling the finished product.—K. Bullock and C. J. L. Elsdon. Quart. J. Pharm. Pharmacol., 10 (1937), 413-438. (S. W. G.)

Liquor Derris—Stability of. Liquor Derris, made according to the Danish Apothecaries Control Laboratory formula, was found very stable. Tests made at room or ice box temperature, showed no greater loss than 2-3% in rotenone content in about 300 days, and this is within the error of analysis. Passing air through the liquor for 30 hours, 6% loss was seen.—A. Lannung. Arch. Pharm. og Chemi, 45 (1938), 5.

Pharmaceutical Solutions—Sterilization of, by Chemical Methods. The following esters: methyl ester of p-oxybenzoic acid (Nipagin M), propyl ester of p-oxybenzoic acid (Nipagin M), propyl ester of p-oxybenzoic acid (Nipagin (Nipagin M)), propyl ester of p-oxybenzoic acid (Nipagin (Nip-Nip)) are the ones most frequently used, although some studies have been made with chloretone, cardiazol and zephirol. However, the esters of p-oxybenzoic acid, chloretone and cardiazol while active against living bacteria cannot be practically used against spore-bearing organisms. Their chief application is as preservatives. A 0.2% solution of zephirol is effective in any except grossly contaminated preparations although its toxicity for humans or its effect on pharmaceuticals is not known. However, the sterilization of pharmaceutical solutions by chemical means has not progressed far enough to supplant thermal methods.—J. Thomann. Schweiz. Apoth.-Ztg., 76 (1938), 121.

Pituitary and Preparations Made from It. The author reviews the evidence for the various principles found in the pituitary gland. It has been shown that the bovine pituitary is rich in vitamin C. The following hormones of the anterior pituitary are described and their isolation, properties and actions given: the somatotropic or growth hormone, the gonadotropic hormones, the hormones occurring during pregnancy, the thyrotropic hormone, the parathyrotropic hormone, the lactogenic hormone, the cortical hormone acting on the adrenal cortex, a pancreatic hormone, some principle having a profound influence on metabolism, especially principles affecting fat metabolism. The posterior lobe has been shown to elaborate at least three principles: vasopressin, oxytocin and an antidiuretic principle. The separation and properties of the principles are described. A blood-pressure-lowering principle depressant has been found in the urine. The middle portion of the gland has yielded a fraction called the melanophore hormone or intermedin. In preparing the various principles, the glands of all animals do not serve alike. For example the pituitary of the sheep, hog and horse contains more gonadotropic hormone than the bovine pituitary while the latter contains more growth hormone and the hog gland more thyrotropic hormone. A wide variety of preparations is available on the market, many of which the author enumerates and describes. The methods of standardizing preparations for their content of growth-promoting hormones, the gonadotropic hormones, the thyrotropic hormones, prolactin, oxytocin, vasopressin are briefly described. Twenty-five references.—R. WASICKY. Scientia Pharm., 9 (1938), 23–28; 32–36.

Tropacocaine Solutions—Stability of. A freshly prepared 5% solution of tropacocaine having a  $p_{\rm H}$  of 5.2 was divided into various groups and the  $p_{\rm H}$  of each group was varied by the addition of diluted hydrochloric acid. Three ampuls (brown ampuls of Jena glass were used) of each group were then heated for varying periods in an autoclave at 120°. The results of the experiment show that aqueous solutions (containing free hydrochloric acid) of tropacocaine hydrochloride having a  $p_{\rm H}$  of 3.3 to 5.6, when sterilized for 20 minutes at 120°, were most stable.—H. Lausten Hansen. Dansk Tids. Farm., 6 (1937), 109–117; through Pharm. Ztg., 82 (1937), 832.

# PHARMACOPŒIAS AND FORMULARIES

French Codex—New. The new French Codex (Codex Medicamentarius Gallicus VI) which came into force at the beginning of April is the result of work commenced in 1922 by the Codex Commission. The book, which is published in two volumes, contains 1299 items. The first volume consists mainly of matters relating to the practice of pharmacy and sales of poisons, together with general analytical and technical data of use in pharmacy. Much space has been devoted to a complete list of preparations and drugs which appear in the present and all the preceding editions of the publication, and the dates at which the various items have been added are also given. The second volume, which is the larger portion, contains the monographs of the Codex. A departure from the usual style of compiling a pharmacopæia is made by the inclusion of colored illustrations of medicinal plants, which will serve a useful purpose in their identification and for students of pharmacognosy. The main titles used in the monographs are given in French, while subheadings appear in Latin. Synonyms and some of the trade names under which the preparations are sold are also included. General information which might be expected to be found in an official work of reference, such as solubility figures, incompatibilities and amounts of water of crystallization in different substances, is set out in many monographs. A complete list of the medicaments which have been added and a summary of the monographs on all the biological products appearing in the new Codex are given.—Anon. Chemist and Druggist, 128 (1938), 583, 622. (A. C. DeD.)

Pharmacopæia—Polish, Becomes Official. A new National Polish Pharmacopæia Second Edition, to take the place of the Austrian, German and Russian official standards still in force in this country, has been released, and it became official on December 1, 1937. The last previous edition of the Polish Pharmacopæia occurred in 1817. During the intervening time, when Poland was under control of the interior nations, and since its post-war independence no other edition has appeared.—Anon. Drug Trade News, 12 (1937), 38; through Squibb Abstr. Bull., 10 (1937), A-2247. (F. J. S.)

Pharmacopæia of the U. S. A. (U. S. P. XI) and Its Value in Maintaining Drug Standards for the People and the Medical Profession as a Whole. The topics discussed are: General description; clinically tested drugs; new drugs included; changes; deletions; retentions; nar-

cotics; interesting points; biological assay; omissions.—E. Rost. Deut. med. Wochschr., 64 (1938), 165; through Squibb Abstr. Bull., 11 (1938), A-301. (F. J. S.)

Pharmacopæias—Unification of Chemical Monographs in the. A report on the discussions held by the International Union of Chemistry pertaining to pharmacopæial monographs.—C. LORMAND. J. pharm. Belg., 19 (1937), 837-839. (S. W. G.)

#### Non-Official Formulæ

Cream Manufacture—Advanced Methods in. A discussion of new emulsifying agents. Chemical emulsification with ammonia is preferred to sodium or potassium carbonates because ammonium stearate possesses a great affinity for water. Glycol stearate is discussed in detail. Formulæ are included. Face Cream.—(1) White liquid petrolatum 100, white viscous petrolatum 50, glycol stearate 30, anhydrous lanolin 20, white wax 50, stearin 30 Gm. (2) Warm water 350, borax 4, sodium benzoate 4 Gm. Emulsified Oil.—(1) Glycol stearate 30, white liquid petrolatum 220, white viscous petrolatum 50, anhydrous lanolin 30 Gm. (2) Warm water 100, sodium benzoate 2, borax 3 Gm. In both formulæ, mix ingredients of (1), warm solution (2), add and boil for 15 minutes.—Fred Winter. Am. Perfumer, 35 (1937), 53. (G. W. F.)

Hydrogenated Oils in Cosmetics. A discussion of the chemistry of hydrogenation of oils, the methods of hydrogenation, and the use of hydrogenated oils in cosmetics. Formulæ are included as follows: Vegetable Oil Cleansing Cream.—Hydrogenated oil 11.0%, beeswax 5.0, stearic acid 0.5, sesame seed oil 60, avacado oil 7, antioxidant 0.1, distilled water 15.3, borax 0.5, perfume 0.6. Vegetable Oil Tissue Cream.—Hydrogenated oil 25.6%, beeswax 9.7, anhydrous lanolin 8.5, spermaceti 2.5, sesame oil 36.5, antioxidant 0.1, distilled water 15.8, borax 0.5, oleic acid 0.3, perfume 0.5. Brushless Shaving Cream.—Stearic acid 15.6%, anhydrous lanolin 1.7, hydrogenated oil 5.7, sesame seed oil 7.6, triethanolamine 0.7, distilled water 68.3, perfume 0.4. Absorption Base Tissue Cream.—Absorption base 7.5%, water 37.5, hydrogenated oil 7.5, mineral oil 12.5, petrolatum 5.5, cetyl alcohol 0.5, water 28.5, perfume 0.5. Camphor Cerate.—Camphor liniment 10%, beeswax 35, white petrolatum 15, hydrogenated oil 40. Basilicon Cerate.—Rosin 35%, beeswax 15, hydrogenated oil 50. Turpentine Ointment.—Oil of turpentine 12.5%, methyl salicylate 12.5, hydrous lanolin 37.5, hydrogenated oil 37.5.—R. F. Eaton. Am. Perfumer, 35 (1937), 33–35. (G. W. F.)

Perfumes—Toilet Soap. Formulæ are given for nine different odors. About 350 Gm. of the compound are required for 100 Kg. of the soap. A white soap odor consists of: methylacetophenone 20, p-cresyl acetate 10, buxine 20, amyl salicylate 50, jasmine for soap 350, benzyl acetate 200 and terpineol 350 (parts by weight).—Monsoin. D. P. Z., 23 (1938), 142; through Am. Perfumer, 36 (1938), 64. (G. W. F.)

Tooth Powder-Oxygenated. The chief ingredients of a tooth powder are stated to be light precipitated chalk, magnesium carbonate, kaolin and, in the cheaper products, ground chalk and infusorial earth; soap powder may also be added. The chief advantage of the powders over the pastes is that such ingredients as peroxide compounds, which are unstable in the latter preparations, may be added. The following oxygen-containing compounds used in oral hygiene are discussed: sodium perborate, sodium persulfate, magnesium peroxide, hydrogen peroxide-urea compounds and sodium perpyrophosphate. The last two compounds are considered the best for powders. Suitable polishing agents for these preparations are extra light precipitated calcium carbonate, medium heavy precipitated calcium carbonate, magnesium carbonate, kaolin, bentonite, magnesium phosphate, titanium dioxide, silica gel, tricalcium phosphate and calcium sulfate. Oil flavors not affected by oxygen to the extent of 1% may be added. The following formulæ are offered: (1) Magnesium peroxide (30%) 25.0, precipitated chalk 50.0, precipitated magnesium carbonate 15.0, bentonite 10.0. (2) Magnesium peroxide (30%) 25.0, silica gel 10.0, precipitated chalk 50.0, soap powder 10.0, milk sugar 5.0. (3) Perpyrophosphate 20.0, precipitated chalk 60.0, colloidal clay 20.0. (4) Perpyrophosphate 20.0, silver p-hydroxybenzoate 0.5, precipitated chalk 60.0, magnesium carbonate 10.0, milk sugar 5.0, saccharin 0.2, soap powder 4.3. (5) Combined Tooth Powder.—Sodium perpyrophosphate 5.0, magnesium peroxide (25%) 5.0, precipitated chalk 70.0, milk sugar 5.0, tricalcium phosphate 10.0, soap powder 5.0.—Hugo Janistyn. Drug and Cosmetic Ind., 41 (1937), 626-627, 635. (H. M. B.)

#### DISPENSING

Ascorbic Acid—Syrup of. Three formulæ for a syrup of ascorbic acid were studied as to stability in comparison with an 0.5% aqueous solution of ascorbic acid. Formula 1: Acid ascorbic, 0.50 Gm., acid citric, 0.20 Gm., Tinctura aurantii dulcis, 5.0 Gm., syrupus sacchari, q. s., 100 Gm. Formula 2 (issued by the Swedish Medical Control Board, 1937): Acid ascorbic, 0.50 Gm., acid citric, 0.10 Gm., sodium citrate, 0.90 Gm., Tinctura aurantii dulcis, 5.0 Gm., syrupus sacchari, q. s. 100 Gm. Formula 3: Same as Formula 2 with addition of 0.1 Gm. cystine. By iodine titer, the keeping qualities were studied over a 6-week period. In a week the simple, aqueous solution lost about 25%, in 14 days about 40% and in 6 weeks was practically without potency. The syrups were much more stable, after 6 weeks showing a loss of titer corresponding to about 4% decrease in ascorbic acid content. Tinctura aurantii dulcis, Pharm. Helv., is the most convenient of the official tinctures to mix with sugar syrup. The iodine uptake of this tincture and of the sugar syrup were separately determined as controls—J. Häkanson and T. Ahlm. Farm. Revy, 37 (1938), 37.

Benzyl Benzoate Liniment—New Formula for. A stable, clear benzyl benzoate liniment for treatment of scabies can be made with the aid of propyl alcohol and either Spir. sapon. kalin., Dan. Disp., or Sapo fuscus. A satisfactory formula is: Benzyl benzoate, 50 parts, propyl alcohol, 25 parts, Spir. sapon kalin., 65 parts.—O. M. Olsen. Dansk Tids. Farm., 12 (1938), 33. (C. S. L.)

Calcium Phosphate with Frangula—Granules of. A formula is cited for preparing granules of calcium phosphate (25%) with frangula: Calcii phosphas præcipitatus, 250 Gm., Saccharum pulveratum, 450 Gm., Pasta cacao deoleata, 300 Gm., Extractum fluidum frangulæ, 2.5 Gm., Spiritus dilutus, q. s. Makes 1000-Gm. granules.—Anon. Arch. Pharm. Chemi, 44 (1937), 727. (C. S. L.)

Castor Oil—Sulfonated. Sulfonated castor oil containing sodium salts might cause turbidity in liquid potassium soap preparations. It is of little value in preventing the precipitation of calcium soaps, and when used in greater amounts has a deleterious effect on the potassium soap as a detergent.—Janistyn. Seifensieder Ztg., 64 (1937), 8; through Am. Perfumer, 36 (1938), 70. (G. W. F.)

Cod Liver Oil Ointments—Experiments with. M. discusses the effect of external cod liver oil ointment which is due not only to the action of vitamin A but also to the action of unsaturated acids, iodine and phosphorus occurring in natural cod liver oil. The most favorable concentration in an ointment is 33.3° of a cod liver oil containing 1200 international units. Adeps lanæ containing a small amount of vaseline is recommended as the ointment base. Unguentum molle is not useful because of its water content. The addition of essential oils must be avoided as they exert an irritating effect on larger wounds. The addition of 2% of Balsam of Peru is recommended. For the preparation of the ointment the molten and almost cooled adeps is mixed with the balsam and vaseline and then the cod liver oil is added without too long stirring. A method for the standardization of cod liver oil, based on its vitamin A content, is given in the original paper.—V. Meckelbach. Veröff. Gebiete des Heers-Sanitätswesens, No. 103; through Squibb Abstr. Bull., 11 (1938), A-382.

(F. J. S.)

Effervescent Salts. A formula is cited for a stable preparation of effervescent "fruit salts," of the type of Eno's or Wex's "fruit salts." The formula is: Coarsely powdered (granular) tartaric acid, 450 Gm., mixed by stirring in a large glass vessel with anhydrous sodium carbonate, 50 Gm., then there is stirred in coarsely powdered (granular) sodium bicarbonate, 500 Gm. The mixing must be done in a dry room. The sodium carbonate is dried to constant weight at 100-120° C. It is present in the formula for the purpose of taking up any traces of moisture from the other ingredients, and so to stabilize the mixture.—E. V. Christensen. Arch. Pharm. Chemi, 45 (1938), 16, 33.

Extractum Hyoscyami Siccum—Preparation of, with Use of the Percolator and Evacolator. Two 250-Gm. samples of Folia Hyoscyami were treated in an evacolator with 450 Gm. diluted alcohol and in a percolator with 600 Gm. of dilute alcohol, respectively, and were further treated according to the extraction directions of the German Pharmacopæia VI. The evacolator process yielded 350 Gm. extraction fluid and 29 Gm. extract (0.289%), while the percolator process yielded 250 Gm. extraction fluid and 47 Gm. extract (0.2516%). Belladonna extracts prepared by the two methods did not show such marked differences. From the results with belladonna and

hyoseyamus it appears that a German Pharmacopæia VI extract cannot be prepared from all German Pharmacopæia VI drugs, but only from those possessing an alkaloid content over the minimum demanded by the German Pharmacopæia VI. The percolation method is generally easier and more economical than the German Pharmacopæia VI method utilizing 8 parts diluted alcohol for each part drug. The percolator apparatus is more stable than the evacolator.—W. Brandrup. Pharm. Ztg., 82 (1937), 1214; through Squibb Abstr. Bull., 11 (1938), A-245.

(F. J. S.)

Eye Salves—Alkaline. The following formula is recommended as an eye ointment to combat the effects of war gases: sodium biborate (impalpable powder), 1 part; purified sodium bicarbonate, 2 parts; distilled water and lanolin, of each 10 parts; and white vaseline q. s., 100 parts. The amount of water will not dissolve all of the borax and bicarbonate. However, some will dissolve and this will be readily available, the undissolved material being later taken up by the tears. Ointments containing saturated solutions of borax and bicarbonate always separate crystals with changes in temperature or on long standing. This crystallization can be retained within reasonable limits when some undissolved salt is present. Complete elimination of the water is undesirable since the fat-coated crystals would be too slowly available.—Gemeinhardt. Gasschutz u. Luftschutz, No. 10 (1937); through Schweiz. Apoth.-Ztg., 76 (1938), 49.

(M. F. W. D.)

Grapefruit, Citrus Grandus, C. Decumana and Related Species as a Pharmaceutical Flavoring Agent and Vehicle. The author sees the possibility of a new vehicle, because of the abundant supply, the low cost and the pleasant taste and aroma. The literature reveals no previous work and the subject will be presented in a series of papers. The present report covers bibliography, chemical and physical properties of constituents and botanical classification. Names of varieties and where grown, gross anatomy, methods of obtaining the oil and some of its constants are given. Analysis of the oil shows limonene, 90%, waxy materials (non-volatile), 7.5%, oxygenated volatile constituents and sesquiterpenes 2 to 3 %. There are octyl and decyl aldehydes; octylic and decylic acids, probably as esters; geraneol and octyl alcohol, free and as acetates; other constituents in small quantity. The relatively high percentage of aldehydes necessitates protection from oxidation and, to avoid as much as possible undesirable nonvolatile material, use of cold pressed oils is advisable. The glucoside margingen is a crystalline bitter substance which diminishes in quantity as fruit matures. Pectin is present as calcium pectinate. An analysis of grapefruit juice shows citric, tartaric, malic and oxalic acids. Vitamin C content is well preserved in fruit packed in tin cans because oxygen disappears because of slow evolution of hydrogen from action of acid on metal, giving a reducing medium. Cold storage of fruit is effective: below 40° F. flavor improves, fruit becomes sweeter, acid content less. When there is arsenical spraying of trees arsenic is found in rind, rag and juice. It is negligible from the health viewpoint but reduces acidity, decreases soluble solids and sometimes hastens maturation. High temperatures give juice a cooked flavor. Heating lowers vitamin C content. No process for storage of juice has been found which will yield juices exactly similar to fresh fruit in "bouquet" and aroma. For pharmaceutical use juice should be prepared from fresh mature fruit or concentrates no more than a few months old. Only glass or stainless steel equipment should be used. Much has been written about vitamin content of the Genus Citrus. Reference is made to some of this work.—David J. Mason. J. Am. Pharm. Assoc., 27 (1938), 42. (Z. M. C.)

Ipecac Extracts—Preparation of. Extracts prepared by the method of Pharm. Hung. IV contained 1.33% of active matter; those according to Pharm. Helv. 5, 1.77%; others made by the author's method (boiling the drug for 10 minutes in a solution containing 3 drops of 10% hydrochloric acid solution to each Gm. of the drug), 1.39%; and by this method with boiling for 15 minutes, 2.43%. For determination of the alkaloids in the solution and in the original drug the gravimetric method is more reliable than the volumetric.—L. DAVID. Pharm. Ztg., 81 (1936), 806–810; through Chimie & Industrie, 38 (1937), 101. (A. P.-C.)

Orange Flower Water. The composition and analysis is given.—T. FRERES. Perfumery Essent. Oil Rec., 29 (1938), 296. (A. C. DeD.)

Physostigmine Salicylate—Stable Non-irritating Solution of. Solutions of 1% eserine in a phosphate buffer solution at  $p_{\rm H}$  6.2, to which sodium formaldehyde sulfoxylate (concentration of 1:5000) has been added, have been kept for six months without developing a pink color. This solution is non-irritating to the conjunctiva, and has been effective in producing miosis and in

reducing the tension in cases of glaucoma which would not respond to treatment with solutions of pilocarpine.—Avery M. Hicks. Am. J. Ophthalmol., 20 (1937), 1040-1042; through Chem. Abstr., 32 (1938), 2287. (F. J. S.)

Soap Solutions—Turbidity in. Turbidity is due to either free fatty acid or an acid soap. The greater the dilution, then turbidity is due to fatty acid. In more concentrated solutions the turbidity is due to acid soap.—P. Exwall. Kolloid Ztg., 77 (1936), 320; through Am. Perfumer, 36 (1938), 70. (G. W. F.)

Solution of Ferric Chloride Easily Prepared. The authors submit a formula which involves oxidation of the ferrous chloride with solution of hydrogen peroxide. The method was tested and results are tabulated. Following is the formula: Solution of ferrous chloride 100 Gm., hydrogen peroxide 125 Gm. not less than 3% strength, to make 100 Gm. Procedure: Weigh 100 Gm. of the solution of ferrous chloride in a suitable, tared dish. Add the hydrogen peroxide from a burette slowly and with gentle stirring. Place on a water-bath and concentrate to 100 Gm. The solution of ferrous chloride is prepared by the procedure given in the U. S. P. IX for the ferric chloride solution. If this is done with care and carefully oxidized, according to the procedure just outlined, a product of U. S. P. quality will be the result.—C. O. Lee, F. J. Leblanc and H. Bang. J. Am. Pharm. Assoc., 27 (1938), 40.

Tablets—the Exactness of the Dosage of. In commenting upon the article appearing earlier by Spengler and Schenker, the author recommends the use of 30 tablets as a control on the weight. The pharmacopæial limit of 10% variation is not to be the guide line but rather the extreme permissible limits of variation. A table illustrating the variations obtained using 30 tablets as a control is shown. Seven kinds of tablets were studied.—E. Höst-Madsen. Pharm. Acta Helv., 13 (1938), 18. (M. F. W. D.)

Tragacanth Powder—Value of Compound, as Suspending Agent. The following summary is given: The addition of mucilage of acacia in any proportion to mucilage of tragacanth has been shown to result in a dehydration of the gel masses of tragacanth and their deposition as white floccules, the viscosity of the mixture being lower than that of either constituent mucilage. A minimum viscosity was attained in a mixture consisting of 80% mucilage of tragacanth and 20% mucilage of acacia. Similar results were obtained when these mixtures were diluted with 7 volumes of water and the viscosities and suspending powers measured; although a minimum value was found at a different concentration. The presence of the relatively small quantity of acacia in compound tragacanth powder has been shown to produce a considerable reduction in the viscosity and suspending power of the tragacanth constituent either in the presence or absence of electrolytes in solution. No similar reduction is brought about by the starch or sucrose present, and it is suggested that the acacia be omitted from the preparation, the starch and sucrose being retained.—J. M. Rowson. Ouart. J. Pharm. Pharmacol., 10 (1937), 404-412. (S. W. G.)

# PHARMACEUTICAL HISTORY

Apothecary Emblems. The article presents the need for a uniform international emblem for pharmacy. Many of the symbols that have been used through the ages up to modern times are described. Twelve plates accompany the article. Ten references are given.—J. A. Häfliger Pharm. Acta Helv., 13 (1938), 3-17. (M. F. W. D.)

Gildemeister, Eduard. Obituary.—Perfumery Essent. Oil Rec., 29 (1938), 219.

(A. C. DeD.)

Karrer, Paul. A brief biographical sketch.—B. L. VANZETTI. Biochim. terap. sper., 16 (1938), 52. (A. C. DeD.)

Nobel Prize—History of a. A review of the work of Albert v. Szent-györgyi, the Hungarian researcher, on the oxidation of cells which merited a Nobel prize for 1937.—B. v. ISSEKUTZ. Pharm. Monatsh., 18 (1937), 222-223. (H. M. B.)

Trommsdorf, Johann Bartholomaus. Pharmacist, Teacher, Scientist. A sketch of the life of the first of a group of German scientists who insisted that pharmacists must be scientific men.—Curt P. Wimmer. J. Am. Pharm. Assoc., 27 (1938), 56. (Z. M. C.)

United States Pharmacopæia—President of the First Convention Called to Formulate the. Samuel Latham Mitchill, August 20, 1764—September 7, 1831; was a physician, chemist, author, senator, representative and promoter of the sciences. In a splendid historical paper the author presents a biographical sketch of Dr. Mitchill covering his early life environment and edu-

cation, his education and experience abroad and the beginning of his professional career at home, and his devotion to the practical side of life. Other phases of his work are indicated by the following sub-titles: The Medical Repository; the first medical scientific journal in the United States; hospital experience, prescription writing and pharmacopæial work; American activities presaging a national pharmacopæia; assembling of delegates and consummation of the United States Pharmacopæia; reception of the first edition of the United States Pharmacopæia, some subsequent events connected therewith and its revision in 1830; comments on the two 1830 revisions of the 1820 United States Pharmacopæia.—Lyman F. Kebler. J. Am. Pharm. Assoc., 26 (1937), 908.

#### MISCELLANEOUS

Acetylsalicylic Acid Products—Water-Soluble Storage-Stable. Acetylsalicylic acid or one of its metal salts is mixed with an amino acid or a salt of an amino acid.—CLEMMY O. MILLER and ARTHUR E. SIEHRS. U. S. pat. 2,101,867, Dec. 14, 1937. (A. P.-C.)

Bath Salts and Fluids. A review of modern bath preparations, including practical suggestions as to compounding and manufacture.—S. P. Jannaway. *Perfumery Essent. Oil Rec.*, 29 (1938), 80. (A. C. DeD.)

Cleaning Agents. For general toilet use, especially with warm water, tallow soaps are most satisfactory, containing 70-80% tallow, 20-30% coconut oil in the original soap fat with no blender added, no more than 0.05% free alkali, and 15% moisture when milled, 35% moisture when unmilled. Vegetable-oil (corn, cottonseed or olive) soaps are preferable with cold or tepid water. Hard-water soaps are usually a straight coconut oil product, working only fairly well. As a softener, zeolite is easier to handle than lime soda. For surgical soap, J. recommends a clear, odorless amber jelly made from purest potash, refined corn oil, and a small quantity of finest quality coconut oil, and containing no other ingredient, the free alkali not over 0.5%. Soaps for general laundering should be commercially neutral tallow soaps containing no excess caustics or unsaponified fat. They should have a titer of about 40.0° C. (separated fatty acids will solidify at this point), and a free alkali content of less than 0.5%. They may also contain cottonseed or corn oil, but not coconut oil or rosin. Soaps for washings at low temperature should be made from blended olive, cottonseed or corn oil, having a titer of about 20° and a free alkali content of less than 0.5%. For washing dishes and glassware, trisodium phosphate is most economical. Scouring bars and powders should contain 88-94% ground feldspar, about 2% soap, not over 1% alkali salt, not over 0.1% free alkali and not over 4% moisture. Walls, woodwork and painted floors can safely be treated with an entirely neutral soap made from highgrade corn oil with potash.-NEAL R. JOHNSON. Hospitals, 12 (1938), 105; through Chem. Abstr., 32 (1938), 1968. (F. J. S.)

Cosmetic Preparations—Dry. Improved applications of lysalbinic or protoalbinic acids of derivatives are described.—Anon. Perfumery Essent. Oil Rec., 29 (1938), 135. (A. C. DeD.)

Cosmetic for Use as a Photographic Make-Up. A cosmetic preparation adapted for use on humans to be photographed on panchromatic type emulsions contains white and colored pigments such as zinc oxide and titanium dioxide and various colored pigments in quantity and proportion sufficient to impart to the cosmetic a high reflection coefficient to wave-lengths of about 6200 to 6500 Angstrom units and lower coefficients to shorter wave-lengths of light, the reflection coefficient to wave-lengths within the range 4000 to 4500 Angstrom units being 45 to 75% of that to wave-lengths within the range 6200 to 6500 Angstrom units.—Max Firestein and Steven Ferentzy, assignors to Max Factor and Co. U. S. pat. 2,099,010, Nov. 16, 1937. (A. P.-C.)

Cosmetics—Acid. Since the  $p_{\rm H}$  of the skin is said to range from 3 to 5 on the surface, various cosmetics are discussed as to their effect on the acidity. Vanishing creams are usually acid, face powders containing carbonates are not satisfactory nor should alkaline emulsifying agents be employed. Absorption bases, glyceryl monostearate and sulfonated alcohols are valuable because they are not alkaline. Lemon cream is discussed.—H. S. REDGROVE. Am. Perfumer, 35 (1937), 31-32, 83-84. (G. W. F.)

Cosmetics—Artificially Fortifying, with Vitamins. A mouth wash was fortified with vitamin C (concentrate of dog-rose fruits) and a cold cream with carotene (from carrots). Pure alcohol, benzoic acid, glycerol, peppermint oil and coloring matter do not affect vitamin C. Impure glycerol caused 100% decomposition in the test period of eleven days. Benzoic acid exerts

a strong preservative action on the vitamin potency, while peppermint oil has a somewhat similar action. Air oxygen, daylight, elevated temperature (40° and higher) and alkalies destroy vitamin C and carotene. Wax, paraffin, stearic acid, petrolatum, borax, ceresin and zinc oxide in the cold cream do not decompose carotene. Biological tests showed that under normal conditions the two preparations retained their potencies for several months. Clinical tests indicate that the use of the fortified mouth wash has a prophylactic and even curative (paradentosis) action. Carotene in cold cream has a beneficial effect on the follicular condition of the skin.—A. I. Naĭmark. Masloboĭno Zhirovoe Delo, 13, No. 5 (1937), 30-31; through Chem. Abstr., 32 (1938), 1397.

Cosmetics—Protective. A cosmetic effective in thin films to retard ultraviolet absorption contains not over 5% of an amino or substituted amino-o-benzoic acid.—Samuel Isermann, Ernst Ohlsson and John W. Orelup. U. S. pat. 2,102,712, Dec. 21, 1937. (A. P.-C.)

Cosmetics for the Skin. Shaving Soaps and Creams. Shaving soaps consist of not more than 0.03% of free alkali, and are characterized by their white or yellowish white coloration, light weight and firmness. The various types of soaps used in the preparation of shaving soaps are discussed and a table showing the per cent hydrolysis of some potassium and sodium soaps is also given. Soaps prepared from the higher molecular weight esters of fatty oils show a greater degree of hydrolysis than the lower ones.—H. Janistyn. Seifensieder Ztg., 64; Der Parfümeur, 11 (1937), 557. (N. L.)

Cream Manufacture. A brief discussion of the materials used in face creams including cocoa butter, castor oil, marrow fat, oil of spermaceti, carnauba wax and Japan wax, beeswax, natural yellow wax, white wax, spermaceti, anhydrous lanolin, free fatty acids, stearine and "artificial petrolatum (a mixture of (1) 20 Gm. of ceresin in 80 of liquid petrolatum, or (2) 30 Gm. in 70, or (3) 27 Gm. and 3 Gm. of white wax in 70 Gm. of liquid petrolatum, or (4) 40 Gm. and 10 Gm. lanolin in 50 Gm. of liquid petrolatum)."—Fred Winter. Am. Perfumer, 35 (1937), 47-48, 95.

(G. W. F.)

Deodorant—Skin, Semi-Solid. A process is described consisting of heating together a large percentage of ethyl, propyl or isopropyl alcohol (or a mixture), aluminium and/or zinc chloride, a soap of aluminium or zinc or a fatty acid or ester, and candelilla or carnauba wax.—Anon. Perfumery Essent. Oil Rec., 29 (1938), 132. (A. C. DeD.)

Drinks—Natural Fruit. The manufacture, coloring and defects of fruit beverages are described.—S. V. Poultney. Food, 6 (1937), 263-264; through J. Soc. Chem. Ind., 56 (1937), B., 615. (E. G. V.)

Hair Restorers. A discussion of metallic hair dyes, their claims and actual results. The metals commonly employed are lead, silver and copper. Other metals sometimes found are nickel, iron and bismuth.—F. E. Wall. Am. Perfumer, 35 (1937), 34–38. (G. W. F.)

Hair—Treating Medium. A preparation for nourishing hair consists of a clear aqueous solution of lecithin, a sulfonated oil or fat, for example, highly sulfonated olive oil, and an additional dispersing agent, for example, sulfonated high-molecular alcohols or their esters, or acidyl derivatives of taurine, as alkali salts or albumin condensation products (prepared by interaction of albumin decomposition products with sebacyl chloride in alkaline solution).—G. F. Stroher. Brit. pat. 473,638; through J. Soc. Chem. Ind., 57 (1938), 105. (E. G. V.)

Insects—Protection from, Preparations for. The persistence of insect-repellent mixture bases on coumarin (I) or o-diethylphthalate (II) is increased by dissolving them with a water-soluble alkaline-earth salt in an organic solvent containing less than 15% of water. Among examples of such mixtures are I (10) and calcium chloride (10) in 96% ethyl alcohol (80), or II (15), magnesium bromide (6), calcium bromide (8), and water (10) in  $\beta$ -propyl alcohol (120 parts). Similar preparations of stiffer consistency are also claimed.—A. CARPMAEL. Brit. pat. 473,592; through J. Soc. Chem. Ind., 57 (1938), 112. (E. G. V.)

Lipstick Colors—Preparation of. It was found that Rouge Sombre is alizarin precipitated on Al-Ca and Lake Rouge Geranium is eosin precipitated on aluminum. The two lakes contain 80-83% aluminum hydroxide. The prospects of manufacturing lipstick pigments from domestic dyes are tentatively discussed.—N. Kirzner. Masloboino Zhirovoe Delo, 13, No. 5 (1937), 31-32; through Chem. Abstr., 32 (1938), 1397. (F. J. S.)

Musts—Relation between Density and Alcohol Content of, and Its Application to the Improvement of Musts. It is confirmed that the customary method of calculating the per cent

of ethyl alcohol of the wine from the density of the must, and vice versa, gives inaccurate results when applied to musts of high density. Both in laboratory tests and in the vat the yield of ethyl alcohol from sugar in Palatinate wines is 46.4-47.1%, that is, consistently greater than the 45% assumed by von der Heide. A method of calculating the per cent of sugar in the must from the per cent of ethyl alcohol of the wine is outlined.—F. Trauth and K. Bassler. Z. Unters. Lebensm., 72 (1936), 476-498; through J. Soc. Chem. Ind., 56 (1937), B., 606. (E. G. V.)

Powder Colors. Colors used in tinting powders from pink to blue are considered. Formulas for different shades are given. For Rachel II concentrate: 7 Kg. light ocher, 3 Kg. burnt sienna and 1 Kg. geranium lake. Other formulas are given including lake mixtures for rouge manufacture.—H. Janistyn. Seifens. Ztg., 47 (1936), 966; through Am. Perfumer, 35 (1937), No. 3, 44.

Radio-Activity—Maintaining, in Creams. An invention is described consisting of immersing in the product in which a constant degree of radio-activity is to be maintained, a device containing a predetermined quantity of substances capable of setting up radio-active emanations, the quantity being so regulated as to maintain the previously radio-activated product in radio-active equilibrium.—Anon. Perfumery Essent. Oil Rec., 29 (1938), 129. (A. C. DeD.)

Sesquiterpene Alcohols and Their Acetates as Valuable Aromatics for Modern Perfumes. A discussion.—Anon. Riechstoff-Ind. Kosmetik, 12 (1937), 220-221. (H. M. B.)

Skin Foods—Preparation of. Milk freed from butter fat and sterilized is emulsified with a fat, for example, a mixture of wool fat and arachis oil, and then dehydrated and dried. Before dehydration the emulsion may be fermented with an organism, for example, B. bulgaricus, which converts lactose into lactic acid.—R. Hellerud. Brit. pat. 473,592; through J. Soc. Chem. Ind., 57 (1938), J., 111. (E. G. V.)

Sun Tan Preparations. Sun-protecting preparations are sold in the form of fatty creams, oils, milks, clear lotions, glycerin salves, vanishing creams or powders. The work of various writers is reviewed. New materials suggested as sun filters are:  $\beta$ -methyl- $\alpha$ -naphtho-coumarin,  $\beta$ -methyl escualtin,  $\beta$ -methyl umbelliferone, and coumarin derivatives. Formulas for several products are given.—J. Augustin. Riechstoff-Ind. Kosmetik, 11 (1936), 218; through Am. Perfumer, 35 (1937), 44. (G. W. F.)

Suppositories—New Mixtures for. It is reported that a Russian chemist has succeeded in establishing the relation existing between the sliding resistance of suppositories and the point of fusion of the materials from which they are made. According to his researches this relation may be expressed as  $P=mt \div nt^2$ , where P is the resistance, m and n are constants which for fats are equal to 32.75 and 1.82, respectively, and for waxes to 50.37 and 2.07, respectively. From this formula he has found that by making a mixture of a vegetable oil with a melting point of 32° and a paraffin wax with a melting point of 56° it is possible to obtain a product the mechanical properties of which correspond to 35 and 80% of those of cocoa butter. Products of this type will, it is felt, be able successfully to replace cocoa butter for many chemical and cosmetic preparations.—Anon. Mfg. Chemist., 8 (1937), 349; through Squibb Abstr. Bull., 10 (1937), A-2252.

Tannin (in Wines). White wines contain only small amounts of tannin (I) and as a result are subject to bacterial diseases. Flavor of red wines are largely due to I (chiefly oenotannin). In fining, the gelatin: I ratio is approximately one to one. Maximum I content for red wine is 0.3%. In industrially useful proportions I does not inhibit growth of wild yeasts, but considerably retards that of tourne.—M. V. Cruess. Wines and Vines, 16 (1935), 5-7; through J. Soc. Chem. Ind., 56 (1937), 383.

(E. G. V.)

Tetrahydronaphthalene and Ethylene Dichloride-Carbon Tetrachloride Mixtures—Comparison of, against Clothes-Moth Larvæ. Tetrahydronaphthalene was more effective than ethylene dichloride-carbon tetrachloride (3:1) when compared on an equal volume basis.—W. Colman. J. Econ. Entomol., 29 (1936), 629; through J. Soc. Chem. Ind., 57 (1938), 109.

(E, G, V.)

Thiocyanates—Aliphatic, Insecticidal Activity of. III. Red Spiders and Mites. OC<sub>4</sub>H<sub>9</sub>.(CH<sub>2</sub>)<sub>2</sub>.O.(CH<sub>2</sub>)<sub>2</sub>.CNS emulsion gives good control of Tetranychus telarius and Paratetranychus pilosus, and may be used in combination with sulfur or calcium oxide-sulfur in double-purpose sprays.—D. F. Murphy. J. Econ. Entomol., 29 (1936), 611; through J. Soc. Chem. Ind., 57 (1938), 93. (E. G. V.)

Wine Clarifications—Difficult. Wines obtained from incompletely matured grapes from which juice is extracted by a continuous pressing process are difficult to clarify. Such wines contain large amounts of malic acid, pectic and protein matter and even cellulosic compounds. For good clarification the  $p_{\rm H}$  must be increased to approximately 4 (with calcium carbonate), and much gelatin is necessary. The latter must be added before the tannin.—I. C. ROMEU. Bol. inst. investigaciones agron., 1, No. 2 (1935); through J. Soc. Chem. Ind., 56 (1937), B., 607.

(E. G. V.)

Wines—Filtration of. The efficacy of various types of filter is discussed. Cellulose has both an absorptive and a screening effect, but with very turbid wines it is unsatisfactory after repeated use. Screening filters (asbestos, etc.) are most suitable for very turbid wines, and adsorption filters (kieselguhr etc.) for less turbid. Addition of kaolin or kieselguhr prevents choking of the filters, especially in presence of mucoids, and increases the rate of filtration.—Robereau-Gayon. *Progrès agr. vit.*. 103 (1935), 355; through *J. Soc. Chem. Ind.*, 56 (1937), B., 607. (E. G. V.)

## PHARMACOLOGY, TOXICOLOGY AND THERAPEUTICS

## PHARMACOLOGY

Acaprine—Studies on. I. The authors study the action of acaprine on the glycemic rate, on the volume of the spleen and on the blood count, and show that when this substance is administered subcutaneously, it has the same effects as adrenalin administered by intravenous injections.—F. Lenzi and S. Lenzi. *Biochim. terap. sper.*, 16 (1938), 31. (A. C. DeD.)

Acaprine—Studies on. II. The authors find the acaprine given subcutaneously, stimulates hydrochloric secretion.—F. Lenzi and S. Lenzi. *Biochim. terap. sper.*, 16 (1938), 35.

(A. C. DeD.)

Adrenaline and Acetylcholine—Obtaining Active Perfusates Reacting Like. A frog is perfused repeatedly with the same lot of Ringer solution. If the perfusion is done at room temperature while both vagi are weakly excited electrically, the perfusate gives an adrenaline-like physiological reaction. If the frog is eserinized and perfused at 5° to 8° C. while the vagi are very strongly stimulated the perfusate has an acetylcholine-like reaction.—N. Gavrilescu and N. Ionescu. Compt. rend. soc. biol., 123 (1936), 840-841; through Chimie & Industrie, 38 (1937), 934.

Alkaloids—Influence of, on the Fermentative Power and Multiplication of Yeast. Investigation of the stimulatory (at low concentration) and inhibitory (at higher concentration) action of quinine, papaverine, caffeine, cinchonine and pilocarpine on the growth of yeast for twenty-four hours and forty-eight hours shows that the toxicity of these alkaloids (reckoned in terms of a 25% inhibition) decreases in the order given. At higher concentrations the fermentative power is inhibited to the same extent as the multiplication.—C. Enders and F. M. Wieninger. Biochem. Z., 293 (1937), 22; through Physiol. Abstr., 22 (1937), 1097. (F. J. S.)

Alloxan—Hypoglucemic Action of. Intravenous injections of alloxan causes in normal rabbits hypoglucemia to the convulsive stage. Convulsions are promptly relieved by intravenous doses of glucose. The effect persists for at least 24 hours; its mechanism is not understood.—Henry R. Jacobs. *Proc. soc. exptl. biol. med.*, 37 (1937), 407. (A. E. M.)

Antimalarial Drugs—Experimental Study of Pharmacologic Action of, upon the Heart. Quinine, atebrine (acridine derivative) or plasmochin (quinoline derivative) given to man in therapeutic doses has little effect on the heart rhythm, producing only slight negative chronotropic and dromotropic action. The effect of these drugs on the dog heart when injected intravenously and on the isolated auricle of the turtle was also studied.—J. M. Hoyos and I. L. PORTILLO. Anales inst. biol. (Mex.), 8 (1937), 353-373; through Chem. Abstr., 32 (1938), 989. (F. J. S.)

Apomorphine and Morphine—Apparent Synergism of. Although a true synergism between morphine (I) and apomorphine (II) in regard to narcotic effects does not exist, a case of apparent synergism of I and II was noted in a 35-year old man. The patient, weighing 210 pounds, was an habitual alcoholic, to whom 35 mg. I sulfate was usually given during the periods of excitement after drinking. When he was given 30 mg. I sulfate and 6 mg. II hydrochloride subcutaneously at the same time, he vomited three times in thirty minutes, and developed cyano-

sis and respiratory embarrassment during the next half hour. He recovered under stimulants and gas administration. The response is interpreted as an abnormal depressant reaction to II occurring in a morphinized patient. In such cases I may synergize with the depressant symptoms due to II, and energetic treatment of the collapse is indicated. Since danger of such occurrences is constant though small, it is recommended that II not be given to morphinized patients under any circumstances. In studies on mice by M. and E., II neither effectively synergized with nor antagonized I effects.—G. R. MAXWELL and G. A. EMERSON. West Virginia M. J., 34 (1938), 25; through Squibb Abstr. Bull., 11 (1938), A-75. (F. J. S.)

Atropine—Effect of, on the Gastric Contents of Man. One hundred and seventy patients were examined for change in fasting contents of the stomach and one hundred and five for change in test meal, after 1.75 gr. of atropine had been allowed to act, respectively, 0.5 and 1.5 hours. The volume of the fasting contents was reduced 17.4 cc., the volume of the test meal was increased 41.5 cc.—Samuel L. Immerman. J. Lab. Clin. Med., 23 (1937), 256; through Squibb Abstr. Bull., 11 (1938), A-173. (F. J. S.)

Diarylethanolamines—Some, Chemical and Pharmacodynamic Study of. Diphenylhydroxyethylamine and its p-dimethoxy derivative lower the blood pressure in dogs. The stilbenediamine prepared from benzoin by means of nitric acid exerts, in strong doses, a hypotensive action preceded by a short hypertensive period. Diphenylaminopropane, prepared by reduction of dibenzylketone oxime is also hypotensive.—A. Lespagnol, G. Bizard and J. Turlur. Bull. sci. pharmacol., 43 (1936), 555-571; through Chimie & Industrie, 38 (1937), 935. (A. P.-C.)

Diethylaniline and Diethylaniline Oxide—Degradation of, in the Animal Body. In dogs and rabbits subcutaneously injected diethylaniline is converted into p-hydroxy-diethylaniline. No unchanged diethylaniline is excreted in the urine. Diethylaniline is more toxic than dimethylaniline. Methemoglobinemia may be produced in cats.—F. Horn. Hoppe-Seyler's Z., 249 (1937), 82; through Physiol. Abstr., 22 (1937), 1074. (F. J. S.)

Digitaloids of Magnolia Cortex and Fluctuations in Potency of This Cortex-Variations in Sensitivity of Esculenta to. On the seventh of each month of the year, the cortices of young magnolia twigs were carefully collected, selected to ensure uniformity as to age and development. After drying for forty-eight hours at 65° C., part was biologically assayed immediately, the remainder preserved in a paraffined glass-stoppered bottle and at regular intervals of one or two months reassayed. For the bioassays, an alcoholic Soxhlet extract was diluted before the injection with water to bring the alcohol concentration to 25%. Tabulated results of these tests indicate the variation in frog sensitiveness for one year. Evaluation of the two unknown variables; sensitivity, E, and potency, G, suggested the following equation:  $C_{pq} = G_p.E_q$  wherein C =observed Gm. of frog killed by one Gm. of drug; p =number of the month in which the drug was gathered; q = number of the month in which the drug was assayed; G = Gm. of toxic principles per Gm. of drug; E = Gm of frog killed by one Gm. of drug. Graphical representation of values of E for the various months exhibited convergent tendencies within the limits of experimental accuracy. It was assumed that the drug was homogeneous in active principle throughout the year and did not change during storage. Results of these experiments indicate that at the beginning of the year frogs have little sensitivity to magnolia cardiac poisons with but a slight rise by the end of April. From then, their capability of resistance declines abruptly, reaching bottom around the beginning of August after an almost perpendicular fall. Less precipitously, it rises by the middle of October to its maximum where it remains throughout the winter. Correspondence with the spawning time of the animal is suggested. Other workers have measured the variation in sensitivity of Rana temporaria to digitalis, squill and convallaria throughout the year. The general agreement of these three curves and their disagreement with curves derived by the authors may be due either to a different frog reaction to magnolia or to a fundamental difference between esculenta and temporaria. -R. JARETZKY and W. LIER. Arch. Pharm., 275 (1937), 599. (L. L. M.)

Ergot and Pituitary—Use and Abuse of, Valuable as Oxytocics. Undesirable consequences from improper use. Ergot: oxytocic activity due to alkaloids, especially ergonovine. Official preparation, fluidextract. Assayed by cockscomb, rabbit uterus and rabbit intestine, and colorimetrically. Alkaloidal content varies in various samples. Taken orally, 2 to 4 cc. daily not harmful but prolonged use produces ergotism, with dry gangrene of extremities. Crystalline ergonovine may be administered orally or parenterally. No cases of ergotism yet reported clini-

cally, but found in experimental animals from prolonged use of ergonovine. Characteristic response to ergot in post-partum period; promotes more rapid uterine involution and controls postpartum bleeding or urinary output. Therefore, indicated in toxemias of late pregnancy. Fluidextract may affect blood pressure. Solution Posterior Pituitary: used first as oxytocic, later fractionated into oxytocic and pressor principles. Solution Posterior Pituitary U. S. P. contains both principles. Standardized oxytocic, 1 cc. equals 8 to 12 international units of 0.5 mg. powder. Pressor principle adjusted to oxytocic activity. Pharmacologic activity: inactive orally; intramuscular or subcutaneous administration produces prompt uterine contractions. Intravenous Produces rise of blood pressure and decrease in urinary output in toxemias of use inadvisable. pregnancy, therefore contraindicated. In non-pregnant patients, ergot or pituitary should not be given for uterine bleeding. Oxytocic drugs will not terminate normal gestation in first trimester of pregnancy, uterus only susceptible near end of term. But in induced or incomplete abortion ergot and pituitary may be used to hasten or complete process. Medicinal induction of labor by use of castor oil, quinine and pituitary. Oxytocic drugs given only in last stages of labor as they interfere with uterine contractions in earlier stage and may cause asphyxia. Ergonovine best because of rapid and sustained action. Pituitary diluted with inactive thymus retains all undesirable and dangerous action of pituitary.—M. EDWARD DAVIS. J. Am. Med. Assoc., 109 (1937), 1631. (M. R. T.)

Gasoline and Petroleum Ether—Action of, on the Respiratory Centers of the Frog. Both products increase the frequency and amplitude of the respiratory movements of the isolated frog head. In the frog in vivo there is observed an increase in the frequency of pulmonary respiratory movements and a decrease or suppression of the respiratory movements of the pharynx. In both cases, after this period, the respiratory movements cease completely. The action of petroleum ether is slightly slower than that of gasoline.—V. A. GLADYCHEVSKAYA. Hig. Truda, 14 (1936), No. 3, 33–37; through Chimie & Industrie, 38 (1937), 476. (A. P.-C.)

Guaiacolsulfonic Organic Derivatives—Biological Properties of. The salts of mono-, diand tri-ethylamine of guaiacolsulfonic acid are very soluble in water, slightly toxic, and adapt themselves perfectly to the parenteral application, being administered in larger doses than those of guaiacolic preparations previously applied. The salt of diethylamine (Tussinon) has been administered already per os. The guaiacolsulfonate of ethylene diamine, although relatively less soluble can also be introduced in the organism in doses sufficiently high and can be administered better per os, than parenterally. The salts of piperidine, ephedrine and tyramine show a decreased pharmacological activity of the organic bases when united with guaiacolsulfonic acid. It is certain that in several diseases such as broncho-pneumonia, asthma and others, the administration of a remedy such as the guaiacolsulfonate of ephedrine gave the best results. This salt facilitates the ventilation of the lungs, increasing the blood pressure compensates the reducing action of guaiacolsulfonic acid. Further, the aerated action of the lungs by ephedrine is increased by the analogous action of guaiacolsulfonic action.—S. Otolski. Arch. Chem. Farm., 3 (1937), 260.

(A. C. DeD.)

Guanidinoglyoxaline—Behavior of, in the Animal Body. 4- (or 5-) Guanidinoglyoxaline was prepared by Hunter's method and was found to be toxic to dogs, doses of 40 mg. per Kg. producing tonic contractions, opisthotones and death within five hours. With sublethal doses there is a temporary fall in blood pressure, and about 33% of the dose may be recovered from the urine. After administration of glyoxaline, about 8% may be recovered from the urine, while carnosine, even after 30 Gm. administered, does not occur in the urine at all.—M. Mohr. Hoppe-Seyler's Z., 248 (1937), 57; through Physiol. Abstr., 22 (1937), 1074. (F. J. S.)

Insulin—Cutaneous Absorption of. Cutaneous absorption of insulin occurs in rabbits and in patients independent of abrasions produced on the skin. This absorption, however, is very inconstant.—RALPH H. MAJOR and MAHLON DELP. *Proc. Soc. Exptl. Biol. Med.*, 37 (1937), 338.

(A. E. M.)

Lactoflavin—Biological Assay of, with Chicks. Chicks needed both flavin and filtrate factor for normal growth. A basal diet was devised which contained filtrate factor in the form of a rice-bran extract adsorbed repeatedly with fuller's earth; with this it was possible to assay lactoflavin by observing growth responses. Maximal growth of chicks required the addition of about 0.60 mg, lactoflavin to 100 Gm, of diet. Green foods and milk were better sources of flavin

than of filtrate factor, while cereals furnished appreciable amounts of filtrate factor and very little flavin.—T. H. Jukes. J. Nutrition, 14 (1937), 223; through Physiol. Abstr., 22 (1937), 1053.

(F. J. S.)

Laxative Action of Active Principles Extracted from Some Commonly Used Plants. Tests have shown that agar-agar (I), elder (II), chicory (III), aloes (IV) and senna (V) contain principally laxative and purgative principles to the exclusion of deleterious secondary principles. I has a slight nutritive value and maintains the tone of the gastrointestinal tract. II contains 0.32% of sambunigrin and has a diaphoretic and diuretic action. The desiccation of the root of III augments the laxative principles and the sugar content which maintains muscle tone and temperature in the patient. The purgative action of IV depends on the amount of free hydroxyanthraquinone existing in the plant and the ease of decomposition of the corresponding glucosides. V is similar in purgative action to IV but is less irritating. Methods are given for the extraction of the active principles of the drugs in the form of a dry powder which is compressed into pastilles with an inert filler. These preparations act uniquely on the intestine by exciting peristals without affecting the functions of the stomach and duodenum. They are painless in use and the intestine does not become habituated to their use.—A. Caravaggi and A. Manfredi. Boll. chim. farm., 76 (1937), 117; through Squibb Abstr. Bull., 10 (1937), A-2288. (F. J. S.)

Lycopodium—Alkaloids of, Contribution to the Study of the Toxicity of. The alkaloids of lycopodium may be classified in two groups based upon their pharmacological action. Group I alkaloids, annotine, clavatine, complanatine and inundatine, cause, in warm-blooded animals especially cats and rabbits, a marked excitability within the first moments of intoxication, the animals returning to normal within 8-10 hours. A lethal dose of these drugs causes death within one to two and a half hours accompanied by tonic and clonic convulsions and asphyxia. Group II alkaloids, selagine and piliganine, act more rapidly than Group I, causing death with violent convulsions within one hour after administration. A toxic dose of these drugs causes extreme restlessness and intoxication with a return to normal in five to seven hours. Group II alkaloids are also distinguished from those of Group I, in that the former produced a marked contraction of the pupil thirty to fifty minutes after injection. The lethal dose of the Group I alkaloids was 0.05-0.1 Gm. per Kg. cat, and of Group II was 0.005 Gm. per Kg. cat. Throughout the experiment cats, rats, rabbits and frogs were injected subcutaneously with 1-2% aqueous solutions of the sulfites of these alkaloids.—P. Oficjalski. Bull sci. pharmacol., 44 (1937), 470; through Squibb Abstr. Bull., 11 (1938), A-90.

Morphine Sulfate—Intravenous Use of, for Analgesia. The intravenous route of administering morphine sulfate is preferable to the subcutaneous, especially in regional anesthesia (10 mg.), peroral endoscopy (2.5-15 mg.), analgesia and preoperative medication, because of its immediate effect, administration at the moment needed and accurate regulation of the dose. An ampul containing 10-15 mg. in 1.5 to 2 cc. of sterile water is injected via a 20- or 22-gage intravenous needle so that only \(^{1}/\_{24}\) grain of the morphine is given at first and the rest 20-30 seconds later. As much of the drug is injected slowly as is needed to produce the desired effect.—C. J. Betlach. Proc. Staff Meetings Mayo Clinic, 12 (1937), 733; through Squibb Abstr. Bull., 11 (1938), A-32.

Narcotics—Action of, on Enzymes and Cells. There are two important rival theories which attempt to explain the action of aliphatic narcotics on living cells: (a) The properties of the cell surface are determined by its lipide content, and narcotics change these properties by dissolving in this surface; (b) narcotics are adsorbed on the cell surface, thus covering it with an inert layer. The adsorption hypothesis is supported by the following data: (1) Resemblance between the action of narcotics on living cells and on enzymes. The concentration-action curves obtained were of an adsorption, linear or intermediate type. The linear curve is usually adduced as proof for theory (a). But it was shown that the action of narcotics in lowering the air-water surface tension follows, over the range of concentration of physiological interest, a nearly linear relation exactly similar to that obtained with living cells. It is suggested that if more than half the total adsorption possible is required in order to produce full inhibition of an action then an exponential concentration-action curve is obtained. If inhibition is produced when the surfaces are less than half saturated, then the curve approximates a linear form, although it is really a portion of an exponential curve. (2) Pharmacological activity of narcotics increases with the increase in the length of the carbon chain, thus showing a marked parallelism between equinarcotic and iso-

capillary concentrations. This rule, though, often breaks down when different series of compounds are compared. However, the same divergence is obtained in the action of narcotics on enzymes. (3) Warburg showed that narcotics interfered in a similar manner with the action of cyanides on cells and on the inorganic catalyst, blood charcoal. (4) Schurmeyer showed that alcohol inhibited the action of purified invertase only after globulin was added, suggesting that inhibition depends on the active group of the enzyme being fixed to particles of colloidal dimensions. Quantitative data support the adsorption hypothesis but do not entirely exclude theory (a).—A. J. Clark. Trans. Faraday Soc., 33 (1937), 1057-1061; through Chem. Abstr., 32 (1938), 991.

4-Nitro-4'-Amino-Diphenylsulfoxide—Preparation of, and Action on Experimental Infections in Mice. 4-Nitro-4'-amino-diphenylsulfoxide has the highest antigonococcic action of this type of compound. It is prepared by the action of 30% hydrogen peroxide on a cold acetic acid solution of nitro-amino-diphenylsulfide or better its acetyl derivative. In contrast to the corresponding sulfone and sulfide, it is soluble in dilute mineral acids and this property is used in its purification. It is a lemon-yellow body slightly soluble in cold alcohol, benzene and ether, very soluble in acetone, almost insoluble in water and melts at 132°. Administered orally to mice, it is well tolerated in doses of 10 mg. per 20 Gm. of body weight. On infected animals it causes in single doses of 5 mg. the indefinite survival of all the animals.—Constantin, Andre Ray and Guy Richard. Compt. rend., 205 (1937), 1018. (G. W. H.)

Phosphorus—Radioactive, Circulation of Phosphorus in the Body Revealed by Application of, as Indicator. Radioactive phosphorus (as phosphate) was injected subcutaneously in rabbits. Within twenty-seven days 45% was excreted in the urine and 11.5% in the feces. A phosphorous atom spends approximately thirty days in the body. The ratio of active phosphorus: normal phosphorus is highest in the kidney, liver and muscle, and lowest in the bones.—L. A. Hahn, G. C. Hevesy and E. C. Lundsgaard. Biochem. J., 31 (1937), 1705; through Physiol. Abstr., 22 (1937), 1073. (F. J. S.)

Piperazine—Iodine Derivative of, Some Pharmacological Studies on. The commercial compound "Iodazine" containing 59.538% iodine was strongly dissociable in aqueous solution. It was not highly toxic to guinea pigs or rabbits. The various physiological and therapeutic effects were discussed.—Renzo Benigni. Arch. farm. sper., 64 (1937), 193-213; through Chem. Abstr., 32 (1938), 993. (F. J. S.)

Piperidomethylbenzodioxane (F. 933), Diethylmethylbenzodioxane (F. 883), Phenoxy-1-dimethylamino-2-ethane (L. 407) and  $\alpha$ -Methoxyphenoxy-1-ethanol-amino-2-ethane (L. 416)—Effects of, on the Isolated Uterus of the Guinea Pig and Rabbit. In small doses these four substances induce or increase the rhythmic movements and the tonus of the isolated guinea pig and rabbit uterus. In strong doses they produce intense contraction of the uterine musculature. The normally inhibiting effect of adrenaline on the guinea-pig uterus is never reversed by these compounds, but the substance F. 933 decreases the inhibiting action of adrenaline. In the rabbit, on the other hand, all four compounds reverse the normally stimulant action of adrenaline on the uterus into an inhibitory action; F. 933 exerts the strongest action, F. 883 and L. 416 have equal activities and L. 407 is least active.—J. DAELS. Compt. rend. soc. biol., 123 (1936), 989–991; through Chimie & Industrie, 38 (1937), 935.

Prolactin—Test for. The smear from the pigeon's crop constitutes a sensitive test for prolactin. The cells show typical modifications long before the macroscopic appearance of hypertrophy.—J. R. Valle. Compt. rend. soc. biol. Paris, 126 (1937), 134; through Physiol. Abstr., 22 (1937), 1086. (F. J. S.)

Testosterol and the Androsterol Series—Some Esters of. The esters between the propionate and the valerianates are the most active sexual hormones according to tests with rats Esterification with palmitic and stearic acids lower considerably the activity of testosterone. The presence of palmitic acid as impurity has no effect on the potency of the above esters.—L. RUZICKA and A. WETTSTEIN. Helv. Chim. Acta, 19 (1936), 1141-1146; through Chimie & Industrie, 38 (1937), 937. (A. P.-C.)

Tetramethylammonium Glycerophosphate—Sodium, Pharmacology of. The substance tested is a double salt of glycerophosphoric acid with sodium and tetramethylammonium. It destroys the life of unicellular organisms, inhibits alcohol fermentation and oxidase action and has a curare-like action in the frog. It destroys the response of the isolated gastrocnemius muscle

to stimuli and produces hypertonus of the muscles of the leech in vitro. Tonus and contraction of the isolated mammalian intestine are enhanced. Other effects are inhibition of the isolated frog heart, peripheral vasoconstriction, stimulation of respiration and increase in blood pressure. Conclusions as to possible therapeutic uses could not be drawn.—P. Pratesi and L. Donatelli. Arch. fisiol., 37 (1937), 422-448; through Chem. Abstr., 32 (1938), 996. (F. J. S.)

Tikitiki (Rice Bran)—Standardization of Extract of. Biological assays for vitamin B<sub>t</sub> using rats and pigeons are described. A good extract should contain at least 33 U. S. P. units of vitamin B<sub>1</sub> per Gm.—A. J. Hermano. Rev. filipina med. farm., 28 (1937), 343-346; through Chem. Abstr., 32 (1938), 724. (F. J. S.)

Ultraviolet Rays—Action of, upon the Behavior of Each of the Substances of the Bones and of the Organism in Toto of the Guinea Pig. The author has determined the water, calcium, phosphorous and magnesium contents of the organism in toto and the bones of guinea pigs submitted to forty days of irradiations with ultraviolet rays. The analysis gives a decrease in the percentage contents of the water, an increase of the calcium and phosphorus, and a more marked increase of the magnesium both in the organism analyzed in toto and in the bones (femurs). The percentage is nearly doubled in the case of magnesium.—L. Comi. Biochim. terap. sper., 16 (1938), 20.

Vitamins—Accuracy of Biological Determinations of. The importance of variation of response of different animals to the same dose of vitamin is stressed. The variation in the response of animals of different litters is even greater than the response of individuals. Evidence of fluctuations in the average response of a whole stock of animals over a long period of time shows that it is essential that the standard of reference should always be tested with the vitamin source of unknown potency. Statistical methods for calculating the accuracy of vitamin D determinations are given.—K. H. Coward. *Proc. 5th Intern. Congr. Tech. Chem. Agric. Ind., Holland*, 1 (1937), 39; through Squibb Abstr. Bull., 11 (1938), A-183. (F. J. S.)

Zinc—Action of, on the Effect of Adrenaline Given Subcutaneously. With small amounts of zinc the total activity of adrenaline appears to be definitely diminished. Since adrenaline is oxidized rapidly in the body, it seems probable that, under these conditions, it has been absorbed more slowly and therefore a greater proportion oxidized in a given interval of time.—Elinor Kohn and H. A. Bulger. *Proc. Soc. Exptl. Biol. Med.*, 37 (1937), 421. (A. E. M.)

## Toxicology

Antidote Box—Poison. A wooden box 9 x 13 x 9", canvas-covered, was equipped with various poison antidotes, stomach tube, boric acid ointment, material for demulcent drinks, emetics, a hyperdermic case containing cardiac and respiratory stimulants and morphine, and a list of common poisons with their antidotes and directions for use. The list as given by B. includes antidotes for mineral acids, caustic alkalies, alkaloids, arsenic, mercuric chloride, cocaine, copper sulfate, cyanides, iodine, lysol, morphine, oxalic acid, phenol, silver nitrate, sodium fluoride and strychnine.—James D. Blackwood, Jr. U. S. Naval Med. Bull., 35 (1937), 489; through Squibb Abstr. Bull., 10 (1937), A-2021. (F. J. S.)

Barbituric Poisoning. Two cases of severe poisoning are reported, one (fatal) due to 120 (estimated) grains of barbital, the other (recovered) due to 120 grains of sodium 5-ethyl-5-isoamyl barbiturate (Sodium Amytal). Symptomatology, pathology and diagnosis of acute barbiturate poisoning are discussed with references, as is also the use of therapeutic agents. Prompt removal of unabsorbed barbiturates, e. g., stomach lavage, and administration of antagonistic drugs, quantity guided by the depth of respiratory depression and degree of vascular collapse, permit a more favorable prognosis.—F. L. McDaniel and Robert A. Bell. U. S. Naval Med. Bull., 36 (1938), 32; through Squibb Abstr. Bull., 11 (1938), A-220. (F. J. S.)

Gas-Attack Protection—Task of the Hospital Pharmacist in Connection with. The author discusses the task of the hospital pharmacist which he divides into three parts: (1) pharmaceutical provision for the treatment of gas poisoning; (2) the protection of hospitals against gas attacks and (3) the functioning of the medical service, such as first aid stations, etc. Under the first heading he discusses a number of specific gases and the treatment of poisoning from them. These gases are classified into six groups as follows: (a) the poisonous gases including hydrocyanic acid, carbon monoxide and nitrous vapors; (b) the asphyxiating gases including phosgene, diphosgene, chlorine and chlorpicrine; (c) the irritating gases including bromacetone, brommethyl-

ethylketone, brombenzylcyanide and chloracetophenone; (d) the blistering gases including mustard gas, lewisite, ethyldichlorarsine and dibromdiethylsulfide; (e) the tickling gases (a new group having properties of both irritating and blistering gases) and (f) fire bombs containing phosphorus and thermite. The treatment of the particular gas poisoning is discussed under each group and remedies (both simples and formulæ) are also discussed.—M. J. Schulte. Pharm. Weekblad, 74 (1937), 25.

(E. H. W.)

Hydrocarbons—Chlorinated, Toxic Action of. Risks of Poisoning in Their Manufacture and Use. The chlorine-derivatives from ethane are more toxic than those from methane; those derived from ethylene are intermediate in their toxicity. This property decreases with increasing chlorine content. Masks should be used in closed spaces containing carbon tetrachloride or trichloroethylene.—R. Freitag. Rayon Text. Month., 18 (1937), 543; through J. Soc. Chem. Ind., 57 (1938), 109. (E. G. V.)

Ketene as a Noxious Gas. Death occurred after fifty to two hundred and fifty minutes in all mice, rats and guinea pigs exposed five minutes, respectively, to 100, 200-300 and 200-300

parts per million ketene (CH<sub>2</sub>=CO). The lower range of toxicity was not determined. Autopsies showed respiratory failure with intense pulmonary edema as the direct cause of death. Administration of oxygen after a lethal dose of ketene had no beneficial effect. When air containing ketene was passed through dry soda lime it lost its toxic power. The present results compared with previous data on other poison gases give the following ascending order of toxicity: benzene, hydrogen cyanide, chlorine, sulfur dioxide, arsine ketene, phosgene. Caution is urged in industrial use of ketene. Gas masks with the canister containing dry soda lime should prove effective against even high concentrations.—G. R. CAMBRON and A. NEUBERGER. J. Path. Bact., 45 (1937), 653; through Squibb Abstr. Bull., 10 (1937), A-2278. (F. J. S.)

Lead Poisoning of Telephone and Telegraph Workers. In a large number of telephone workers there were observed slight but quite perceptible signs of saturnism; lead in the urine (0.008 to 0.088 mg. per liter), gastro-intestinal pains, excessive gastric acidity, anemia, considerable decrease in the hemoglobin and erythrocite contents, hemolysis.—P. I. MYTNIK and D. A. SHEVELUKHIN. Hig. Truda, 14 (1936), No. 3, 38-40; through Chimie & Industrie, 38 (1937), 473.

(A. P.-C.)

Mercurial Poisoning—Acute, Sodium Formaldehyde Sulfoxylate as an Antidote for Case report of a patient admitted to the hospital in an almost moribund condition caused by mercuric chloride poisoning, via the vagina, the mercuric chloride having been introduced about an hour before. Treatment included restoratives, douche, local application of sodium formal-dehyde sulfoxylate solution and administration of 10 Gm. of the latter in 250 cc. of distilled water (80 cc. were given intravenously, the rest per rectum by the drip method). On the following day 250 cc. of the solution were given intravenously for two days, and about 100 cc. on the following day. Sodium bicarbonate, morphine and atropine, forced fluids, and intravenous saline and dextrose were also used. The patient recovered. Three references.—Spencer T. Trice. Texas State J. Med., 33 (1937), 584; through Squibb Abstr. Bull., 11 (1938), A-142. (F. J. S.)

Mustard Gas—Neutralization of, in Streets and Public Squares. The value of a number of substances used for the neutralization of mustard gas is discussed. Among these are chlorinated lime, chloramine, caporit and potassium permanganate. The author concludes that chlorinated lime should receive the first consideration as a neutralizing agent for mustard gas. The objection often mentioned, that chlorinated lime deteriorates rapidly is off-set by the much higher prices of other agents.—H. J. van Giffen and W. A. van Bronkhorst. *Pharm. Weekblad*, 74 (1937), 102. (E. H. W.)

Nicotine Thiocyanate. A Contact Insecticide. Nicotine thiocyanate was prepared by adding 100 parts of cold anhydrous nicotine to 46.5 parts of dry ammonium thiocyanate in a round-bottom flask provided with bubbling and vacuum connections. The flask was immediately evacuated and kept at  $16^{\circ}$  C. by cooling with running water. A stream of nitrogen gas, passed through the reacting mixture, kept it stirred and assisted in removing the evolved ammonia. The temperature of the reaction was slowly increased to  $60^{\circ}$  C. in order to maintain a viscosity sufficiently low to permit free bubbling of the nitrogen. The tendency of the compound to decompose increased rapidly with the elevation in temperature. The final detectable traces of ammonia

and the slight excess of nicotine were removed by allowing the compound to remain for several weeks in shallow dishes over sulfuric acid in an evacuated desiccator. The compound is very effective as an aphidicide and, in sufficient concentrations with a suitable spreader, controls red spider. Practical spreaders include sodium oleate, diglycol oleate, triethanolamine oleate, rosin, sulfonated alcohol, naphthalene sodium sulfonate. The compound is decomposed by the more positive ions in solution, and is then capable of serious foliage injury.—J. S. McHargue and R. K. Calfee. Ind. Eng. Chem., 29 (1937), 1232. (E. G. V.)

Organic Poisons—a Rapid Method for Isolation of. It is shown that in the extraction of alkaloidal poisons from viscera the laborious and time-consuming Stas-Otto process may be replaced by treatment of the minced material with trichloracetic acid. This at once yields a waterclear filtrate free from protein and fat, and containing the whole of the alkaloid originally present. From this filtrate the alkaloid can be conveniently removed by adsorption on kaolin, from which after neutralization it is eluted by hot chloroform. After removal of the alkaloids veronal can be adsorbed on charcoal and eluted with ether. The method is being tested further and extended to other organic poisons.—G. P. Stewart, S. K. Chatterji and Sydney Smith. Brit. Med. J., 4007 (1937), 790. (W. H. H.)

Plasmocide—Working Conditions in the Production of. In the production of plasmocide the workers are exposed to the pathological effects of a number of toxic substances: hemotoxic compounds, forming methemoglobin-acetanisidide and methemoglobin-nitroacetanisidide; compounds producing skin diseases, and possibly cancerigenic (quinoleic derivatives); narcotics (dichloroethane, ether); arsine, etc. The equipment should therefore be made as gas tight as possible, and certain solvents which are not absolutely essential (e. g., ether, dichloroethane) might advantageously be replaced by less harmful ones.—E. V. Klenova. Hig. Truda, 14 (1936), No. 3, 81–85; through Chimie & Industrie, 38 (1937), 473. (A. P.-C.)

Protoplasma—Mechanism of the Toxic Action of Various Substances toward. A study of the variation of the adsorbent properties of protoplasma under the influence of various narcotics does not confirm Warburg's theory according to which the cause of narcotic action resides in the suppression of contact catalysis resulting from the desorption from the surface of the cells of substances that react under the conditions of structural catalysis. It is to be supposed rather that the blocking of the substrate by adsorption of narcotics directly produces a modification of the state of the molecules of living matter which cause a depression of the vital functions, followed by death.—D. N. Nasonov and V. I. Aleksandrov. Biologych. Zhur., 6 (1937), 117–164; through Chimie & Industrie, 38 (1937), 987.

Pyrethrum Sprays—Dosage-Mortality Curve of, on the House Fly (Musca Domestica). Toxicity data are recorded.—D. Hoyer, S. Z. Von Schmidt and A. Weed. *J. Econ. Entomol.*, 29 (1936), 598; through *J. Soc. Chem. Ind.*, 57 (1938), 109. (E. G. V.)

Trichloroethylene—Poisoning by. A review of a number of cases of poisoning. The symptoms in most cases were headache, conjunctivitis, stomach pains and vomiting. The toxicity is greater than would be expected.—E. Holstein. Zentr. Gewerbehyg. Unfallverhüt., 24 (1937), 49-54; through Chem. Abstr., 32 (1938), 647. (F. J. S.)

Trinitrotoluene—Sanitary and Toxicological Features of the Production of. Experiments on rabbits and observations on men working in the industry confirmed that trinitrotoluene is a hemolytic poison, but nothing indicated that it had an irritating effect on the skin nor confirmed the pretended unfavorable influence on its action of compounds such as tetranitromethane and diphenylnitromethane. Sanitary measures to be taken in the production of trinitrotoluene consist mainly in perfecting the technological process, particularly in making the washing equipment absolutely gas tight. Careful selection of workmen from the standpoint of health is also important.—Z. I. LAGOVIER. Hig. Truda, 14 (1936), No. 5, 39–40; through Chimie & Industrie, 38 (1937), 685–686.

## THERAPEUTICS

Agua Copelina. Pharmacodynamic Study of Argentine Mineral Waters. Has inhibitory action on bacteria. No anaphylactic activity on isolated tissue (guinea-pig uterus and rabbit intestine) but experiment inconclusive as were tests with toxins. Favors germination of seeds and growth of plants. Favors elimination of uric acid, and cholesterin, being beneficial in uri-

cemias.—Irene Pisarro. Rev. sud-americana endocrinol. inmunol. químioterap., 20 (1937), 501. (G. S. G.)

Amyl Salicylate. While not possessing antiseptic action, amyl acetate has been found valuable as an analgesic in treatment of burns.—Anon. *Brit. Med. J.* (1937), 380; through *Am. Perfumer*, 36 (1938), 64. (G. W. F.)

Arthritis—Chronic, Logical Treatment of. Moderate to excellent improvement was obtained in about 50% of sixty cases of arthritis treated only with the calcium double salt of benzoic and benzyl succinic acids (Subenon). The preparation appeared to check the progress of the disease and alleviate the symptoms. Best results were obtained when therapy was started in the early stages of the disease, when treatment was given for three months or more, and when use of the preparation was accompanied by control of intestinal toxicity, diet and foci of infection, and by decreasing nerve tension and increasing peripheral circulation. The beneficial results of the drug may be due to its cholagogic, detoxifying and intestinal-antiseptic action.—J. K. Leir. Clin. Med. Surg., 44 (1937), 498; through Squibb Abstr. Bull., 10 (1937), A-2218. (F. J. S.)

Benzodioxanes—Therapeutic Use of. Diethylaminomethylbenzodioxane, 883F (Fourneau), and piperidinomethylbenzodioxane, 933F, were investigated. The former is a sedative used in angina pectoris where it excels over the therapeutics when given in doses of 0.05 Gm. four times daily by mouth. 933F is efficacious as an analgesic in certain painful affections. Special indications are Raynaud's disease and sclerodermia. Its hypnotic and antirheumatic actions are not yet sufficiently investigated.—J. Sterne. Ann med., 42 (1937), 541-560; through Chem. Abstr., 32 (1938), 1403. (F. J. S.)

Cancer—Struggle against. A review of the second volume of the Act of the International Congress for the scientific and social struggle against cancer is given.—E. Morelli. Biochim. terap. sper., 16 (1938), 37. (A. C. DeD.)

Chemotherapeutic Agents—Three, Relative Toxicities and Therapeutic Values of, of the Sulfonamide Type. Prontosil is more efficacious by oral application than by injection whereas the reverse is found with sulfanilamide. The latter given orally is 1.8 times more effective in low-grade infection and 1.4 times in high-grade infections. The therapeutic margin of safety, however, is more in favor of prontosil. Disulon (amino-benzolsulfonamide-benzol-sulfonamide) is of lower toxicity, better tolerance and of greater protective efficiency than prontylin.—O. W. BARLOW. Proc. Soc. Exptl. Biol. Med., 37 (1937), 315. (A. E. M.)

Creosote Burns. Use a bland ointment containing one part gum tragacanth and three parts lanolin to protect the skin against chemical injuries from contact with creosote. This ointment may be made up at any drug store and can be spread on the hands or other parts of the body likely to be exposed. This ointment is soluble in water and can be washed off easily whenever desired. Another preventive ointment has been suggested as follows: calcium carbonate, 145; pine tar, zinc stearate and petrolatum, 285 Gm.—M. M. Wells. Natl. Safety News, 36 (1937), 70; through Squibb Abstr. Bull., 11 (1938), A-16. (F. J. S.)

Cyanide Poisoning. Successful Treatment of Two Cases with Intravenous Sodium Nitrite and Sodium Thiosulfate. Two cases of cyanide poisoning are reported, one from inhalation of hydrocyanic acid, the other following the ingestion of potassium cyanide. Both cases were treated with sodium nitrite and sodium thiosulfate intravenously with complete recovery. This method of therapy has been successful in animal experiments. The recovery in these two cases and in three cases reported in the literature indicates a similar clinical efficacy.—Alfred P. Ingegno and Saverio Franco. Ind. Med., 6 (1937), 573; through Squibb Abstr. Bull., 11 (1938), A-143. (F. J. S.)

Doryl—Control of Post-Operative Urinary Retention with. The effect of Doryl in the treatment of fifty-five cases of post-operative urinary retention is recorded. In a small series of experiments it was found to cause a considerable but short-lived rise of intravesical pressure. Doryl is useful in the relief of post-operative retention of urine. It has less effect in cases with mechanical obstruction, but it is still worthy of trial before resorting to catheterization. Minor side-effects make its use inadvisable for very ill or shocked patients. In two cases, in which the condition of the bladder resembled that seen with certain cord lesions, administration of doryl twice daily for three weeks caused no harm. It is therefore suggested that doryl should be given an extensive trial in the treatment of urinary retention associated with spinal cord injuries or tumors or diseases.—R. Officer and J. C. Stewart. Lancet, 233 (1937), 850. (W. H. H.)

Insulin Tannate. Tannic acid added to insulin prolongs its hypoglycemic action.—F. Lun. Compt. rend. soc. biol. Paris, 125 (1937), 1088; through Physiol. Abstr., 22 (1937), 1081. (F. J. S.)

Iodine—Effect of, Air-Borne from Brittany to Central Europe. It is considered that during 1933 and 1934 approximately 6500 Kg. of iodine escaped annually into the atmosphere from the Breton kelp-burning regions. Numerous analyses of the atmosphere at various localities near these regions indicate that sufficient of this iodine reaches Germany and other Central European countries to have a significant biological effect on the inhabitants.—H. CAUER. Biochem. Z., 292 (1937), 116; through Physiol. Abstr., 22 (1937), 1080. (F. J. S.)

Magnesium Trisilicate. Medicinal magnesium trisilicate is defined as a "compound represented by the formula H4Mg2Si4O10 and giving the pure diffraction radiograph of the natural mineral sepolite No. 1." The ratio MgO: SiO<sub>2</sub> = 1:2.24 (gravimetric). It is shown that many of the marketed brands fail entirely to conform to these basal requirements. The following therapeutic applications are discussed: (a) The continuous control of gastric acidity in hyperacid states; (b) the production of gastric anacidity without systemic alkalosis; (c) the utilization of the general adsorbent properties of the silicate in hypo-acid conditions. It is demonstrated that: (a) Artificial sepolite No. 1 does not cause alkalosis. (b) The extent and speed of interaction with acids diminish rapidly at hypochlorhydric strengths. Within certain limits of dosage the neutralizing effect is automatically adjusted to the demands of the gastric juice. (c) Within a certain range a useful amount of the silicate can be given in hypochlorhydric states without its neutralizing the gastric contents to such an extent as to destroy peptic activity. An explanation of the variability of its mildly laxative action is put forward. Schemes of dosage appropriate to hyperchlorhydric and hypochlorhydric conditions are discussed.-N. MUTCH. Brit. Med. J., (W. H. H.) 4006 (1937), 735.

Morphine—Effect of, in Rickets. Young growing white rats rendered rachitic were used. One-third of these animals reacted abnormally to morphine. The analgesic effect of morphine was decreased and the excitatory effect increased, as in morphine addiction, which is therefore regarded as due to alteration of calcium metabolism.—C. Amsler. Arch. exptl. Path. Pharmakol., 185 (1937), 263; through Physiol. Abstr., 22 (1937), 1058. (F. J. S.)

Nicotinic Acid—Treatment of Human Pellagra with. Improvement in four patients with pellagra following administration of nicotinic acid was as satisfactory as that following administration of liver filtrate except for an increase in time required for complete disappearance of dermatitis.—Paul J. Fouts, O. M. Helmer, S. Lepkovsky and T. H. Jukes. *Proc. Soc. Exptl. Biol. Med.*, 17 (1937), 405. (A. E. M.)

Peptic Ulcer—Etiology and Medical Treatment of. Severe ulcers sometimes require surgical intervention while many heal spontaneously. Due chiefly to faulty cellular equilibrium, in regard to diet, activity of gastric secretions and bacterial invasion. Can be treated by alkaline powders. Psychologic factor is most difficult to combat, and is most important. Dietary taboos are condiments, alcoholic drinks, tobacco, extremely hot or extremely cold foods. Foci of infection should be extirpated, such as tonsils or teeth. Give sedative for pain. Medical therapy should continue six to twelve months. Patient should be warned against permitting recurrence.—Howard Hartman. Reforma Medica, 23 (1937), 829. (G. S. G.)

Prontosil—Use of, in the Treatment of Gonorrhea. Treatment with Prontosil Soluble and sulfanilamide, combined with irrigations, proved efficient in ninety out of one hundred cases of acute and chronic gonorrhea. In favorable cases of acute gonorrhea cure is obtainable in about fifteen days. Bad health, giving rise to poor resistance in the patient, is an adverse factor to cure by any form of treatment, including treatment with prontosil. The dosage of prontosil should be adjusted to the needs of the individual patient, and other forms of treatment used as the occasion demands. The shorter the time between infection and start of treatment the greater the success of prontosil therapy. Cases which failed to report, or had been unable to get efficient treatment for 15-20 days after infection, did not react as well to prontosil as cases which were treated within 5 days of infection. It has been noted that cases which do not react to prontosil therapy within 18 days are not likely to react at all. It appears that these patients reach a stage where no further advance is made, and their powers of reaction to other forms of treatment is adversely affected. The dosage, therefore, should be limited to 40 cc. of the prontosil soluble together with 13 oz. of colsulanyde over a period of 15 days.—T. F. CREAN. Lancet, 233 (1937), (W. H. H.) 895.