PHARMACEUTICAL ABSTRACTS

EDITOR: A. G. DUMEZ, 32 S. Greene Street, Baltimore, Maryland.

ABSTRACTORS

WILLIAM B. BAKER
GERSTON BRUCH
ARTHUR H. BRYAN
HENRY M. BURLAGE
ALBERT H. CLARK
ZADA M. COOPER
GUSTAV E. CWALINA
AMELIA DEDOMINICIS
MELVIN F. W. DUNKER
GEORGE W. FIERO
PERRY A. FOOTE
RALPH R. FORAN
SAMUEL W. GOLDSTEIN
H. B. HAAG
G. W. HARGREAVES
WILLIAM H. HUNT
CASIMER T. ICHNIOWSKI
ROLAND E. KREMERS
CLIFFORD S. LEONARD
L. LAVAN MANCHEY
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A. PAPINEAU-COUTURE
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E. G. VANDEN BOSCHE
GLENN S. WEILAND
ANNA E. WHITE
ELMER H. WIRTH
THOMAS G. WRIGHT
MAX M. ZERVITZ

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BACTERIOLOGY

Acute Anterior Poliomyelitus—Tests for Immunity to. Antiviral antibody in 0.5 cc. of serum neutralizing at least 10 minimal infective doses of monkey passage virus is believed to indicate effective resistance to poliomyelitis. However, that resistance to poliomyelitis may be present without demonstrable amounts of this antibody in the blood. The antibody is absent in the sera of the majority of children over six months of age and present in the majority of adults. The number of minimal infective doses of virus for *Macacus rhesus* monkeys inoculated intracerebrally and neutralized by 0.5 cc. of serum in an exposure to two hours at 37° C. in a water-bath is the method of standardization. The spinal cords of monkeys developing the disease during the conduct of the test may be employed for the preparation of the Kolmer vaccine of attenuated virus for active immunization of human beings against the disease.—JOHN A. KOLMER and ANNA M. RULE. *J. Immunol.*, 29 (1935), 188. (A. H. B.)

Ammonium Chloride—Effect of, upon the Growth and Production of Acid by Aspergillus Niger. The authors found ammonium chloride to be an excellent sole source of nitrogen for the growth of, and production of, citric acid by *Aspergillus niger*. The optimum production of acid was obtained on the seventh day at a concentration of 0.188 Gm. of the salt per 100 cc. of medium. This is also the optimum concentration for the growth of yeast and for the minimum hydration of wheat gluten.—E. I. FULMER, L. M. CHRISTENSEN and H. SCHOPMEYER. *J. Am. Chem. Soc.*, 57 (1935), 1537. (E. B. S.)

Antigens—Favoring Effect of Certain Lipoidic Substances on the Immunizing Action of Some. It has been shown that with the horse the antitetanic immunity was increased when the antitoxin was injected, incorporated in wool fat. This principle has been used successfully with diphtheria, anthrax, yellow fever and typhus vaccinations. Using a mixture of 3 cc. of tetanic antitoxin, 3 cc. of oil of vaseline and 0.1 g. of cholesterol, it was found that the serum of horses injected twice at one-month intervals, had 7500 times the immunizing action of serum from horses injected only with the antitoxin.—GASTON RAMON. *Compt. rend.*, 201 (1935), 687. (G. W. H.)

Bactericidal Action of Antiseptics—Experimental Critique of the Allen Method of Evaluating the. Tubes containing 5 cc. of agar are allowed to solidify in an upright position so that the flat surface of the medium is at right angles to the walls of the tube. These tubes are inoculated with 0.1 cc. of a 24-hour old broth culture of the desired test organism, which has been transferred serially on the 5 preceding days through broth of the same composition as the agar medium. Before the agar is added, the agar tubes are incubated for 24 hours at 37.5° C.; 10 cc. of the germicidal agent to be tested are then introduced into the culture tubes slowly. The medicated cultures are held in a water-bath, and at 5-minute intervals transfers are made to 10 cc. of sterile broth by means of a 4-mm. nichrome loop. The flamed and cooled loop is passed through the germicidal solution to the surface of the agar without penetrating the medium and carefully withdrawn and the enoculum deposited in the receiving broth. The broth tubes are incubated for 48 hours at 37.5° C., and subjected to careful observation for evidence of growth. Allen's principle of determining bactericidal action of substances upon organisms growing on an agar surface was modified to incorporate the agar culture idea into a test sufficiently simple and accurate to make it practical. The presentation here of a further modification of the agar culture technic is based on limited observations. It does, however, appear to obviate some of the difficulties of Allen's methods.—KEITH H. LEWIS and LEO F. RETTGER. *Am. J. Pub. Health*, 25 (1935), 1125. (A. H. B.)

Biliary Acids—Comparative Action of, on Tetanic and Diphtheria Toxins. Special Properties of Lithocholic Acid. It is known that sodium glycocholate and taurocholate have a slight affinity for tetanic toxin, 1000 toxic doses (T. D.) of which is neutralized by 50 mg. of either salt after five hours' contact at 37–38°. Diphtheria toxin is not neutralized under these conditions. This is explained by the blocking of the COOH group in cholic acid because 1 mg. of sodium cholate neutralizes from 1–10,000 T. D. of tetanic toxin. This is the same for desoxy- and lithocholic acids. With diphtheria toxin 75 mg. of sodium cholate is necessary per 1000 T. D. and 5 mg. of the desoxycholate, a fact explained by the disappearance of the secondary alcoholic function in the desoxy. The sodium salt of the less oxygenated lithocholic acid required only 1 mg. to neutralize 1000 T. D. of diphtheria toxin. It is the most energetic detoxifying agent for diphtheria toxin and constitutes the first example of a cyclic molecule whose affinities are equivalent for the two experimental toxins.—LEON VELLUZ. *Compt. rend.*, 201 (1935), 432. (G. W. H.)

Biologicals—Preservatives for. No one preservative has yet been found that will maintain the sterility of serums, toxins, toxoids and bacterial vaccines. Organic mercury compounds are limited in their usefulness, *e. g.*, they are fairly satisfactory for diphtheria anatoxin but not so for serums. Phenol 0.5% is effective for antiserums.—COMMITTEE ON PRESERVATIVES IN BIOLOGICAL PRODUCTS. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 329; through *Squibb Abstract Bull.*, 8 (1935), A-1385.

Diphtheria Anatoxin. The recommendations are made: (1) That diphtheria toxin-antitoxin be continued or deleted as the individual manufacturer may determine, (2) That plain diphtheria toxoid be continued for the present; (3) That the dose of alum precipitated toxoid, be limited to a volume not exceeding 1 cc. In the preparation of alum precipitated toxoid, that the alum, bacillary protein or other irritating substances be reduced to a minimum; that the trade packages of alum precipitated toxoid be limited to a 1-dose package of 1 cc., and a 10-dose package containing one 10-cc. vial; and that instructions be given with the package to inject diphtheria toxoid alum precipitated either deep subcutaneously or intramuscularly.—COMMITTEE ON DIPHTHERIA TOXOID. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 326; through *Squibb Abstract Bull.*, 8 (1935), A-1371.

Diphtheria Immunization—Observations of, with Single Injection of Alum Toxoid. The author reports on immunization experiments with alum precipitated toxoid containing 9 Lf units per cc., prepared according to Havens' method. 489 Shick-positive persons received 1 cc. of the immunizing agent subcutaneously or intramuscularly. Local reactions included pain, 94%; swelling, 86%; induration, 79%; and abscess, 0.2%. Six per cent of these cases experienced general reactions. Characterized by fever, headache and malaise. Within 5 months following the inoculations re-Schick tests on 359 cases showed the presence of immunity in 225 or 62.7% of the cases. Previous reports have shown that the efficacy of the alum toxoid in inducing immunity to diphtheria ranged from 50.8–100%. The author suggests that this difference may be due to a difference in preparation, dosage, number of injections and cases and time interval between the immunization and re-Schick test. He concludes that the alum toxoid, given in 1 dose, does not give enough protection to all susceptible persons against diphtheria.—DANIEL G. LAI. *Chinese M. J.*, 49 (1935), 340; through *Squibb Abstract Bull.*, 8 (1935), A-799.

Diphtheria Toxoid—Purification of. The methods of purification described, which gave consistently good yields of diphtheria toxoid in a high stage of purity, consisted essentially of precipitation of toxoid by ferric chloride-citrate, acetone and ferric phosphate and removal from the precipitate of material soluble in the acid range of insolubility of the toxoid, *i. e.*, p_H 3.8–4.4. The degree of purification was of the same order for all three methods. The toxoids obtained were used on laboratory animals both as primary and secondary stimuli. The antigenic response, Lf per Lf, was of the order of that obtained with unpurified toxoids. Complete details are given.—PETER J. MOLONEY and MORTON D. ORR. *Biochem. J.*, 29 (1935), 1525; through *Squibb Abstract Bull.*, 8 (1935), A-1289.

Epithelioma of Pearce-Brown—Intracutaneous Vaccination Against. The successful intracutaneous vaccination of rabbits with an emulsion of Pearce-Brown epithelioma is described.—ALEXANDRE BSREDKA, ISRAEL MAGOT, PIERRE LAVAL and PIERRE BERNARD. *Compt. rend.*, 201 (1935), 690. (G. W. H.)

Germicidal Substances—Comparison of Resistance of Bacteria and Embryonic Tissue to. V. Iodine. The highest dilution showing no tissue growth is 1:1800, that inhibiting bacteria growth (*Staphylococcus aureus*) is 1:20,000. The corresponding values for phenol are 1:840 and 1:65. It is concluded that antiseptic power and low irritating qualities to tissues make iodine preferable to most other antiseptics.—A. J. SALLE and A. S. LAZARUS. *Proc. Soc. Exptl. Biol. Med.*, 32 (1935), 1481. (A. E. M.)

Methylene Blue Reduction Test—Standardization of the, by the Use of Methylene Blue Thiocyanate. It is recommended that methylene blue thiocyanate be substituted for methylene blue chloride in the standard tablets used in the methylene blue reduction test because of the reproducibility of the former dye, one part of dye to 300,000 parts of milk be adopted as the standard concentration in this test.—H. R. THORNTON and R. B. SANDIN. *Am. J. Pub. Health*, 25 (1935), 1114. (A. H. B.)

Poliomyelitis—Immunity and Vaccination in. Poliomyelitis is caused by a filterable virus and is a disease of world-wide distribution. It was found that 83% of the newborn infants have

specific antibodies in the blood, presumably passively transferred from the mothers; 80% of the children 1–4 years old have no antibody and are highly susceptible; about 50% of the children 5–14 years old and 75% of the adults have sufficient antibodies for immunity. It would appear that a temporary resistance to the disease might be conferred by injections of immune serum. The injection of antibodies is of very doubtful value once paresis is present. K. and his colleagues have succeeded in preparing an attenuated virus successful in the vaccination against the disease, by treating the virus with sodium ricinoleate. Over 125 children 8 mos. to 15 years old were successfully vaccinated with no ill effects aside from mild local reactions at the site of the subcutaneous injection. Three doses are given subcutaneously at weekly intervals in the following dosage: for 1–4 years, 0.25, 0.5 and 0.5 cc.; 5–15 years, 0.5, 0.5 and 1 cc.; and adults 0.5, 1 and 2 cc. In several children, antibodies have been found as early as four days after the first dose. It is not possible at present to state how long immunity will last in humans, but monkeys vaccinated 3 yrs. ago are still immune.—JOHN A. KOLMER. *Clin. Lab. Rev.*, 1 (1935), 47, 4; through *Squibb Abstract Bull.*, 8 (1935), A-860.

Scarlet Fever Immunization of Nurses. The Dick test is a reliable index of immunity to scarlet fever. Five injections of Dick toxin conferred an immunity of at least three years. The principal drawback to the toxin is the severity of the reactions that it occasionally produces.—GAYLORD W. ANDERSON and WARREN I. REINHARDT. *J. Inf. Diseases*, 57 (1935), 141.

(A. H. B.)

Scarlet Fever Toxin.—A purified and concentrated scarlatinal toxin containing 20,000,000 or more skin test doses per gram and of low nitrogen content has been prepared by a combination of fractional precipitation with ammonium sulphate, treatment with an aluminum hydroxide preparation, dialysis and evaporation.—GEORGE F. DICK and ALDEN K. BOOR. *J. Infect. Diseases*, 57 (1935), 164. (A. H. B.)

Smallpox—Duration and Degree of Immunity against, Conferred by Infantile Vaccination. Observations in 1102 cases indicated that $^1/_5$ of the children vaccinated against smallpox in their infancy completely lose immunity within 4 years, and about $^1/_2$ lose it in 7 years. P. advises vaccination within one year in the presence of smallpox epidemics and enforced revaccination at 4–5 years of age.—J. L. PINTO. *Indian Med. Gaz.* (Mar. 1935); through *Squibb Abstract Bull.*, 8 (1935), A-863.

Streptococcemia—Remarks on, with an Account of the New Results of Serum Therapy in This Infection. A discussion of the use of antistreptococcic serums and a report of the various maladies in which they have been employed. In 310 cases, 252 cures representing 81.30%, were reported.—HYACINTHE VINCENT. *Compt. rend.*, 201 (1935), 693. (G. W. H.)

Tubercle Bacilli—Demonstration of. The author reports from the Finsen Institute and the State Serum Institute in Denmark observations on 625 specimens (pus or tissues) from 589 patients suffering from extra-pulmonary tuberculosis. His opinion of the comparative value of the three main methods of demonstrating tubercle bacilli (guinea-pig inoculation, culture on media and direct microscopical examination) has changed since 1930, when he pointed out that animal inoculation was the most reliable method, as it had failed in only about 10% of the cases in which tubercle bacilli were demonstrable. In the same work he noted that culture on Petroff's media yielded positive results in 64%, and direct microscopical examination only in 36%. The comparative importance of these three methods has recently been changed by the introduction of new culture media—Löwenstein's, in particular. The author now finds that culture yields results as good as, or even better than, those obtained by animal inoculation. In the present study there is, however, a curious exception to this rule. Among 253 specimens of pus from tuberculous glands in the neck were 146 in which tubercle bacilli were found; and among them there were as many as seventy-three in which only the microscopical examination was positive. There were twenty-nine cases in which tubercle bacilli were found on microscopical examination and on animal inoculation or culture; and forty-four cases in which tubercle bacilli were found on culture alone or on animal inoculation alone. This comparative superiority of microscopical examination over the other two methods was not demonstrable in those groups represented by tuberculosis of the spine, various other bones, the hip and the knee.—E. HUSTED. *Acta. Chir. Scand.* (July 8, 1935), 589; through *Brit. Med. J.*, 3900 (1935), 650*D*. (W. H. H.)

Undulant Fever—Prevention of, by the Use of Polyvalent Vaccine. Since 1929 the authors have experimented clinically with a preventive vaccine against undulant fever. The authors

explain their method of vaccination. The polyvalent vaccine used during the past five years, and which gave excellent results, has the following composition: *Brucella melitensis*, human, *Br. melt.* ovin, *Br. melt.* caprin, *Br. abortus* bovin, *Br. ab.* porcin, of each 400 million germs per cc.—C. DUBOIS and N. SOLLIER. *Rev. Microb. appl. agr., hyg., ind.*, No. 6 (1935), 398; through *J. pharm. Belg.*, 17 (1935), 638, (S. W. G.)

Vaccine Virus—Purification of, and Subcutaneous Vaccination. Frozen crude vaccine is ground in a mortar and then extracted with sterile water. To the extract is added McIllvaine's phosphate–citric acid mixture, p_H 4.6, and kaolin. It is then centrifuged, the sediment washed with distilled water and again centrifuged. The sediment is eluted in *N*/25 ammonium hydroxide and the supernatant, containing the active virus, is neutralized with acetic acid. The virus may be further purified by dialysis. In spite of its low protein content, this purified vaccine produces typical pustules and does not differ antigenically from ordinary vaccine. Subcutaneous vaccination does away with formation of cutaneous pustules, and thus leaves no vaccination mark. Reactions are milder, and thus inconvenience in the vaccinated subject is reduced.—HIDETAKE YAOI. *Japan. J. Exptl. Med.*, 12 (1934), 123, 2; through *Squibb Abstract Bull.*, 8 (1935), A-1394.

Water—Bactericidal Power of, Subjected to the Combined Action of Metallic Silver and a Direct Current of Electricity. Tap water became bactericidal to typhoid, paratyphoid, colon and cholera bacilli and staphylococcus in 1–2 minutes after passing a direct current of 0.3 to 4 milliamps. and 1.5–4.5 volts through it by means of 16 sq. cm. silver electrodes. The bactericidal power lasts more than 3 months and is not destroyed by boiling. On dilution, the activated water confers its properties on the greater volume. This action is increased by increasing the current and the size of the electrodes, sporulating bacteria (*B. ephistiæ* and *B. cazabon*) as well as *Aspergillus niger* being killed in 2–3 hours in 250 cc. of tap water subjected to a current of 50 milliamps. and 10 volts for 3 minutes with 45 sq. cm. electrodes. The silver contained in this water does not give it any taste and does not appear harmful as 20 mice fed for 3 months with bread soaked in the activated water appeared well nourished.—NICOLAS METALNIKOFF. *Compt. rend.*, 201 (1935), 411. (G. W. H.)

Water—New Method for the Examination of, for the Presence of B. Coli. The two following culture media are prepared: (*a*) peptone 20 Gm., sodium chloride 10 Gm., lactose 40 Gm., distilled water 1 liter; (*b*) peptone 15 Gm., sodium chloride 7.5 Gm., lactose 30 Gm., distilled water 1 liter. The solutions are brought to p_H 7.4, and 10.5% and 7.5%, respectively, of tincture of litmus is added. Durham tubes are prepared containing 2, 6 and 10 cc. of (*b*) and large Durham tubes containing 10 and 20 cc. of (*a*), and are sterilized. One, 3, 5, 10 and 20 cc., respectively, of sample are added to the small tubes containing 2, 6 and 10 cc. and to the large tubes containing 10 and 20 cc. of culture medium. The tubes are incubated for 24 hours at 37° C., and those in which no acidity has developed are discarded; those in which acidity developed, but no gas was produced, are incubated for another 24 hours at 37° C. A sample from the tubes in which both acid and gas developed is inoculated into agar-lactose containing 1% litmus solution and 1% sodium taurocholate, and the plates are incubated for 24 hours at 44° C. If colonies develop and redden the medium, 1 or 2 colonies are transferred to gelatin tubes having the following composition: peptone 10, sodium chloride 5, glucose 5, gelatin 100, distilled water 1 liter, litmus solution 5%, p_H adjusted to 7.4. The tubes are incubated 4 days at 20° C., and the organisms which have reddened the gelatin without liquefying it and which have given rise to the formation of gas bubbles are examined microscopically to determine their morphological and tinctorial characteristics.—B. BABUDIERI. *Ann. igiene*, 44 (1934), 1025–1035; through *Chimie & Industrie*, 34 (1935), 312–322. (A. P.-C.)

BOTANY

Citrus Cultivation and Pests. A review of the literature on methods of spraying and fumigation is discussed.—ANON. *Perfumery Essent. Oil Record*, 26 (1935), 338. (A. C. DeD.)

Lavender—Growing, in America. Experimental work in the North Pacific Coast region of the United States over a period of ten years is reported. Plantings of local stock, later found to be hybrids, yielded a low quality oil. True lavender was found to be hardier than local hybrids. It was found that seeds of true lavender produced stronger plants if seeded in the open as opposed to cold frame or hot-bed seedlings. If planted in seed beds, which can be controlled, the seedlings should not be transplanted until stem and root are about lead pencil size. The plants responded

to either a stony, gravelly loam or a fine, sandy, clay loam with some gravel and stones.—L. J. WYCKOFF and A. F. SIEVERS. *Am. Perfumer*, 31 (1935), 67–71. (G. W. F.)

Selenium in Soils—Relation to Its Presence in Vegetation. Selenium occurs in soils to a varying extent over extremely wide areas. In certain limited areas it is present in quantities sufficient to produce toxic vegetation. The quantity of selenium in vegetation grown upon a soil depends upon the concentration, the plant species, composition of the soil, moisture, stage of development of the plant and the portion of the plant examined. The identification of the compounds of selenium, and the tracing of their developments in plants and metamorphoses which occur in the animal body are unknown problems.—HORACE G. BYERS and HENRY G. KNIGHT. *Ind. Eng. Chem.*, 27 (1935), 902. (E. G. V.)

Styrax in the Philippines. Several botanical expeditions to the Batanes and the Babuyanes, which form the northern portion of the Philippine Archipelago, yielded a new species of styrax manifestly allied to *Styrax japonicus* Sieb. and Zucc. and to *S. kotoensis* Hayata, of Formosa, and should be considered a northern type in the Philippine flora. It differs from *S. kotoensis* Hayata, the flowers of which have not yet been described, in its smaller leaves.—ELMER D. MERRILL and EDUARDO QUISUMBING. *Philippine J. Sci.*, 56 (1935), 313. (P. A. F.)

CHEMISTRY

GENERAL AND PHYSICAL

Chlorine Sterilizing Compounds—Study of. I. Relationship between p_H and Oxidation Potentials. There is a linear relationship between oxidation potential and p_H for chloramine-T, sodium hypochlorite and calcium hypochlorite. Between p_H 6.0 and 7.0 the time necessary to kill 99% of bacterial spores is a linear function of the oxidation potential. The bleaching of mercurochrome is also a linear function of oxidation potential and may serve as a suitable test for the germicidal efficiency of chlorine compounds. The undissociated hypochlorous acid seems to be the active germicide.—ARTHUR P. HELLWIG. *Iowa State College J. Sci.*, 9 (1934), 163; through *Chem. Abstracts*, 29 (1935), 5219.

Heavy Water—Mutual Solubility of, and Some Organic Liquids. The upper and lower consolute temperatures were determined for systems composed of heavy water with the following organic compounds; phenol, acetonitrile and triethylamine. Compared with ordinary water, the character of the mutual solubilities was the same but the upper consolute temperatures were raised and the lower consolute temperatures were lowered.—JEAN TIMMERMANS and GUSTAVE POPPE. *Compt. rend.*, 201 (1935), 524. (G. W. H.)

Microchemistry. A group of papers on the subject, including general discussions, catalytic reactions and recent advances.—A. A. BENEDETTI-PICHLER, *et al.* *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 207. (E. G. V.)

INORGANIC

Colloidal Silver Chloride—Preparation of. In general, colloidal silver chloride may be prepared as follows: silver nitrate and sodium chloride are each separately dissolved in a little water; a drop of dilute nitric acid is added to each solution. A solution of gum arabic previously heated to 100° for one hour is then added. The two solutions are then mixed by shaking vigorously, a milk-white mixture resulting. This may or may not be centrifuged and is then poured into an excess of strong alcohol. The white precipitate is then filtered off, washed with alcohol and dried at about 80° during which it is rubbed out in a mortar. The preparation must take place in a dark room or the powder will not remain white but will acquire a purplish color. The authors describe four modifications of this process, two of which yield colloidal silver chloride after the insoluble silver chloride is centrifuged out, and two yield a colloidal silver chloride from which no residue results upon centrifuging.—P. VAN DER WIELEN and S. WITTEBOON. *Pharm. Weekblad*, 72 (1935), 1037. (E. H. W.)

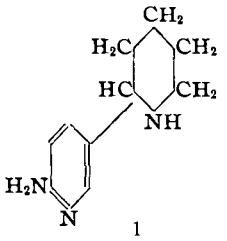
ORGANIC

Alkaloids

Aconitum Napellus—Chemical and Physiological Researches on the Alkaloids of. This article is a summary of a dissertation in which the object of the work was to find a method whereby

the quantity of aconitine in the mixture of alkaloids present in the *tubers* might be determined and to study the alkaloidal content and its variation at various vegetation periods. It is known that aconitine is more toxic than benzoylaconine and much more toxic than aconine. Therefore when two preparations showing identical alkaloidal content (total alkaloids by chemical assay) show variable results upon physiological testing, one preparation probably contains more aconitine and the other more aconine. Methods for the chemical determination of aconitine in the alkaloidal mixtures are discussed, the author devising one depending upon the determination of the benzoic acid formed upon the hydrolysis of aconitine. Tables of alkaloidal contents (both total, and aconitine) in various parts of the roots collected at about monthly intervals between April 1932 and January 1934 are given. Daughter tubers seem to be of greater therapeutic value than mother tubers. December seems to be the preferable month for collection. Several methods of physiological assay are mentioned, that of the M. L. D. on guinea pigs being discussed as to its relationship with chemical assay. In working with tinctures the author found that the chemical and physiological methods gave concordant results if a sufficient quantity of hydrochloric acid was added to the menstruum (before percolation) to give the tincture a p_H of not less than 5.2.—W. A. VAN BRONKHORST. *Pharm. Weekblad*, 72 (1935), 1056. (E. H. W.)

Amine Group—Introduction of, into a Series of Alkaloids. Alpha-aminoanabasine. The known methods for the preparation of amine compounds gave poor yields when carried out with pyridine derivatives or with alkaloids. The reaction of A. E. Tchitchibabine, whereby the amine group is introduced directly into the pyridine nucleus by the action of sodium amide, gives a better yield. The authors have studied this reaction to determine if it is general for alkaloids containing the pyridine nucleus. The authors have obtained a 30% yield in the preparation of the amino-derivatives of anabasine. The reaction is similar to that obtained with pyridine and nicotine. The physical and chemical properties of the derivatives obtained have been studied in addition to the chloroplatinate, picrate and hydrochloride. The amine group being in position alpha, the authors assigned to the products obtained one of the two following formulas:



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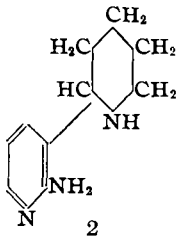

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KAZNELSOHN and I. KABATCHNICK. *Bull. Soc. Chim. France* (Mar. 1935); through *J. pharm. Belg.*, 17 (1935), 635. (S. W. G.)

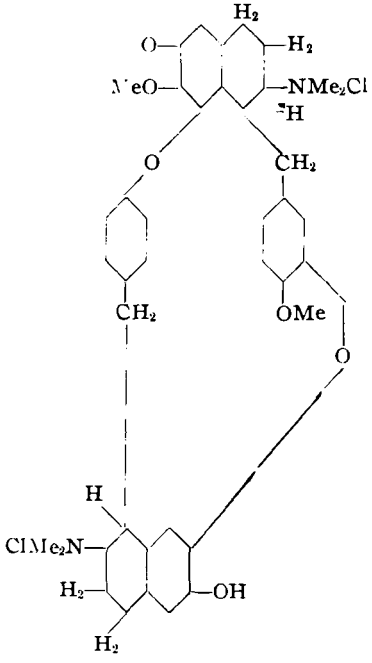
Anagyris Foetida—Alkaloids of, Part II. In addition to cytisine and anagyrine (previously reported), small amounts of *N*-methylcytisine and *d*-sparteine were found. The former (caulophylline) had been reported in *Caulophyllum thalictroides* and the latter in *Sophora pachycarpa* and *Thermopsis lanceolata*. No *l*-lupanine (tetrahydroanygyrine) was detected.—H. RAYMOND ING. *J. Chem. Soc.* (1935), 1053–1054. (G. W. F.)

Cactus—Alkaloids of. The first alkaloid obtained from *Cactus Anhalonium* (Lewinii) was anhalonine $C_{12}H_{15}O_3N$. In the same plant, the following alkaloids were found to be present: Mescaline, lophophorine which are of an oily nature, anhalonidine, pellotine, anhalamine, anhalinine and anhalidine, which are of a crystalline nature. Anhalonidin, pellotin, anhalonin and lophophorin all have an asymmetric carbon atom. Anhalonine and lophophorine are optical active; anhalonidine and pellotine are of a racemic nature. Anhalonine was produced synthetically, the constitution of which corresponds to that of the natural product. The melting point and the specific rotation (α) D = $-56.3°$ were the same as that of the natural product. The synthetic *l*-anhalonine was converted to *n*-methyl-*l*-anhalonine; the picrate of this base is identical with the picrate obtained from the natural lophophorine.—E. SPÄTH and F. KESZTLER. *Ber.*, 68 (1935), 1663. (G. B.)

Cinchona Alkaloids. III. Chlorodihydro-Bases. Using methods of Comstock and Koenigs or of Hesse, the following chloro-derivatives were obtained: α-Chlorodihydroquinine—

sinters at 203°; m. p. 210°; froths at 215°; $(\alpha)_D^{20°}$ −251.0 (c = 0.5). α′-Chlorodihydroquinine—sinters at 183°; m. p. 194°; froths at 225°; $(\alpha)_D^{22°}$ −168.1 (c = 0.5). α-Chlorodihydrocinchonine—sinters at 233°; froths at 236°; $(\alpha)_D^{20°}$ +226.0 (c = 0.5). α′-Chlorodihydrocinchonine—sinters at 220°; froths at 223°; $(\alpha)_D^{22°}$ +176.0 (c = 0.5). α-Chlorodihydrocinchonidine—sinters at 229°; froths at 231°; $(\alpha)_D^{23°}$ −135.6 (c = 0.5). α′-Chlorodihydrocinchonidine—sinters at 244°; froths at 246°; $(\alpha)_D^{23°}$ −62.5 (c = 0.5). α-Chlorodihydroquinidine—sinters at 198°; m. p. 206°; froths at 225°; $(\alpha)_D^{24°}$ +276.3 (c = 0.5). α′-Chlorodihydroquinidine—sinters at 195°; m. p. 200°; froths at 229°; $(\alpha)_D^{22°}$ +240.7 (c = 0.5). In doses of 5 mg. per 20 g. of bird, the two chlorodihydroquinines were about as active as quinine as anti-malarial agents. The author concludes that chlorine addition takes place on the alpha position in the side chain.—JOHN A. GOODSON. *J. Chem. Soc.* (1935), 1094–1097. (G. W. F.)

Curare Alkaloids. Tartaric acid solution of tubocurare was deprived of curarine by extracting with ether and chloroform. The aqueous liquid, after precipitation with lead acetate was treated with phosphotungstic acid. The precipitate was decomposed with baryta solution. After treatment with sulfuric acid, filtering and excess acid removed with barium chloride, the filtrate yielded tubocurarine salt by addition of mercuric chloride. Removal of mercuric chloride and keeping the crude alkaloid, in aqueous solution, at 0° yielded crystalline tubocurarine (hitherto only isolated in an amorphous state): m. p. 274–5°, efferv.; $(\alpha)^{20°}_{5461}$ +235 (c = 0.97) (hydrated salt); $(\alpha)_{5461}$ +264.8 (anhydrous salt); $(\alpha)_{5461}$ +295 (ion). Tubocurarine was found to be closely related structurally to the alkaloid curarine. Tubocurarine chloride was considered:



HAROLD KING. *J. Chem. Soc.* (1935), 1381–1389. (G. W. F.)

Efiri—Alkaloids of. The liana "Efiri," which is a plant from the Congo identified by Staner as *Triclisia Gilletti*, contains alkaloids. The author records that at the Institute of Pharmacy of Louvain University they have been able to isolate two. One is crystalline, and has the formula $C_{38}H_{44}O_7N_2$. The second has not yet been obtained in the crystalline state.—E. DELVAUX. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Ephedrine—Standards for. The following standards were studied and recommended for ephedrine base: (1) Total alkalinity: A solution of 1.0–1.5 Gm. alkaloid dissolved in 10 cc. neutralized alcohol is made up to 100 cc. with water and 25-cc. aliquots are titrated with 0.1N hydrochloric acid using bromthymol blue as indicators; (2) Specific rotation: 1 Gm. ephedrine,

7 cc. water and 1 cc. concentrated hydrochloric acid are shaken in a glass-stoppered cylinder, the volume made up to 10 cc. with water, the mixture shaken and then read in a polariscope at 25°. One gram ephedrine = 0.22 Gm. ephedrine-hydrochloride, the reading being unaffected by a reasonable excess of hydrochloric acid. When water is present the reading should be divided by a factor which is % of ephedrine in the sample divided by 100; (3) Congealing temperature: fit a 4-in. test-tube containing 3 Gm. ephedrine with a stopper through which has been inserted an Anschutz thermometer. Melt the ephedrine by placing the tube in a water-bath, then cool the tube and contents to 30°. Dry the outside of the tube and cover the lower part with several layers of paper or other insulating material. Seed with a small crystal of ephedrine and stir the supercooled liquid ephedrine with the thermometer. The highest temperature obtained is the congealing point. The results of these 3 tests made by 7 laboratories are tabulated.—*Am. Drug Manufacturers Assoc., Proceedings*, (1935), 251; through *Squibb Abstract Bull.*, 8 (1935), A-1372

Ergometrinine—Another Alkaloid of Ergot. The discovery of another alkaloid of ergot, named ergometrinine, has been reported. It is stated that during the process of manufacture of ergometrine this new alkaloid, which is isomeric with ergometrine and convertible into it, has been isolated. Whether the new alkaloid has the same clinical action as ergometrine or is a relatively inert isomeride remains a question for clinical investigation. Ergometrinine has the formula $C_{19}H_{23}O_2N_3$. It is fairly soluble in chloroform, and has $[\alpha]^{20°}_{5461} = +520°$ (in chloroform $c = 1$). It decomposes at about 195°. It forms a crystalline nitrate $B.HNO_3$, hydrobromide, B.HBr and an acid sulphate $B.H_2SO_4$. The salts are easily soluble in water and give dextrorotatory solutions.—ANON. *Pharm. J.*, 135 (1935), 212. (W. B. B.)

Ergostetrine—Some Properties of. The method of separating this "X-alkaloid" is described. A 10-Kg. lot yielded 983 mg. of crystals. Repeated recrystallizations gave white radiating doubly refractive needles up to $1^1/_4$ inches in length. They melted at 160° to 162.5° C. with decomposition. Specific rotation (0.2% solution in ethyl alcohol) was $[\alpha]^{25}_D = -45°$ to $-55°$ ($\pm 10°$ because of weak solution). Aqueous solutions were alkaline and dextrorotatory; chloroform and benzol solutions were lævo-rotatory. Solutions of base or salts are fluorescent. The base is only slightly soluble in water, chloroform and benzol, more soluble in ether, ethyl and methyl alcohol; the salts are very soluble in water, slightly in organic solvents. It is present in ergot in such small quantities that experimental work is difficult. Analysis showed the ratio of C, H, O and N to each other similar to other alkaloids of ergot but in a smaller molecule. Crystals form with solvent of crystallization which is expelled with such difficulty that analytical results become doubtful. The author believes that ergostetrine, ergometrine, ergotocin and ergobasine are the same alkaloid. They were independently obtained by different investigators. Pharmacological and clinical activity, optical activity, decomposition point, solubility and color reactions all point to the same alkaloid. Empirical formulas point the same way. Ergostetrine gives the usual color reactions of ergot alkaloids. Some of these are discussed. There seems no possibility of assay without resorting to chemical separation of the alkaloids except as explained in an earlier report. Simple extracts showing satisfactory color value would contain all the ergostetrine plus variable amounts of other alkaloids because of its greater stability and solubility in the usual solvents. It is intensely active in producing cyanosis of the combs of cockerels. Using birds standardized with U. S. Phar. standard ergotoxine ethanesulphonate the base was judged to be approximately 160% ($\pm 20\%$) as potent as the standard ergotoxine salt. The cyanosis developed more quickly and a deeper blue-black was produced. Difference is not well defined. Limitations of the cockscomb assay are the same as those for the colorimetric method. Ergostetrine stimulates the rabbit uterus into strong contractions, mainly through sympathetic endings. The Broom-Clark method can measure ergotoxine type of activity but not ergostetrine unless there has been chemical separation. Variation in sensitivity to ergostetrine was to be expected. The rabbit uterus provides a good method for different lots of ergostetrine in terms of a lot preserved *in vacuo* as standard because of constant responses in proportion to dosage. Guinea pig uteri are serviceable for assay of ergostetrine in terms of a lot preserved as standard but presence of other alkaloids makes the method worthless for assay purposes unless constituents are separated. Intravenously ergostetrine usually produced pressor effect but relatively large doses did not give reversal of the effect of epinephrine. The chief characteristic of action on pregnant cats was that it acts more promptly and powerfully. Clinical results now available have caused obstetricians to speak of ergostetrine as the true active principle. It seems to account for most of the traditional

oxytocic activity of whole ergot.—MARVIN R. THOMPSON. *J. Am. Pharm. Assoc.*, 24 (1935), 748. (Z. M. C.)

Senecio—Alkaloids of. The alkaloids, platyphylline and seneciphylline, were isolated by the authors from the plant *Senecio platyphyllus* in an earlier investigation. The empirical formula $C_{17}H_{25}NO_5$ was chosen because of the following reasons: the nature of platyphyllin was that of an amine-ester; when heated with alcohol-alkali it was converted to an acid $C_{10}H_{14}O_4$ and an alkamine: platynecin. From a gold chloride solution platynecin crystallized out and was further analyzed. The empirical formula $C_{17}H_{25}NO_5$ was used as a base in giving to platynecins, the formula $C_7H_{15}NO_2$. The acid was crystallized out, analyzed, and assumed the formula $C_8H_{15}NO_2$. According to this, then, the empirical formula $C_{17}H_{25}NO_5$ should be changed to $C_{18}H_{25}NO_5$ which will correspond with the rest of the alkaloids derived from senecio which have a C_{18} group. The properties of platynecine are analogous to that of retronecine, senecifolinine and heliotridine, *e. g.:* it is slightly soluble in water practically insoluble in organic solvents, optically active and a tertiary base. With concentrated sulphuric acid at 100° it gives off one molecule of water, the remaining substance is a methyl-iodide of an oily nature with the formula $C_8H_{13}NO$. The anhydro-platynecine does not react further if hydrogen is used as a catalyst. No carbonyl groups were found to exist in the compound. The attempt to reduce platynecine electrochemically into platynecane ($C_8H_{15}N$) was not successful; the compound obtained was anhydro-platynecine $C_8H_{13}NO$. Another attempt to reduce platynecine was made by using thionylchloride, the result being anhydroplatynecine. Finally platyphyllin ($C_{18}H_{25}NO_5$) was treated with concentrated sulphuric acid; the result was anhydro-platynecine ($C_8H_{13}NO$) —A. ORECHOFF and R. KONOWALOWA. *Ber.*, 68 (1935), 1886. (G. B.)

Strychnine and Brucine. Neobrucine, benzene and methyl sulphate were refluxed, dissolved in hot water, basified and mixed with sodium iodide to form neobrucinide dimethiodide: m. p. 298° (decomp.). This proved to be identical with supposed methoxymethyldihydrobrucidine dimetho-salts-B previously reported. Likewise, dimetho-salts-of-dihydrobrucidine were found identical with dimetho-salts-B of methoxymethyltetrahydrobrucidine. In a similar manner methoxybenzylhydroneobrucidine yielded N(a)-Methyl-N(b)-benzylneobrucidinium di-iodide (m. p. 246–248°). When the dichloride (m. p. 90–92°) was heated, neobrucidine (m. p. 198–199°) was produced.—O. ACHMATOWICZ and ROBERT ROBINSON. *J. Chem. Soc.* (1935), 1291–1295. (G. F. W.)

Totaquina—Philippine. A brief review of the history of cinchona and its alkaloids, especially quinine, is given. Cinchona cultivation has been found practicable in the Philippines by the Bureau of Forestry. There are now 38,000 trees on the experimental plantation. The species include *C. ledgeriana, C. hybrida, C. succirubra.* An analysis of the bark from five-year old trees gave 9.6%, 4.74%, 4.56%, respectively. Three methods for inexpensive extraction of totaquina (total alkaloids) are given. The most efficient is as follows: Finely powdered bark is macerated with dilute hydrochloric acid. The mixture is then poured into a percolator and extraction completed with water as the solvent. The acid percolate is treated with a sufficient amount of sodium hydroxide solution to precipitate the coloring matter as flakes. Remove the precipitate by filtration. Make the acid filtrate strongly alkaline with sodium hydroxide. Allow to stand over night. Filter off the alkaloids and wash with water. A further quantity of alkaloids can be salted out with sodium chloride. The combined alkaloids are taken up by dilute sulphuric acid, decolorized by animal charcoal, and precipitated by pouring into an excess of sodium hydroxide solution. After washing, drying, and alcoholic extraction, the product, totaquina, represented a 9% yield. Details of 21 cases of malaria in which it was used successfully are reported. In a few cases it was necessary to supplement the treatment with atabrine or plasmochin. Figures are presented showing that by growing cinchona in the Philippines and producing totaquina therefrom, the cost of malarial therapy can be brought within the reach of millions of natives.—JOAQUIN MARANON, AMANDO PEREZ and PAUL F. RUSSELL. *Philippine J. Sci.*, 56 (1935), 229. (P. A. F.)

Essential Oils and Related Products

Aromatics—New Methods in the Field of. A review dealing with aromatic aldehydes.—A. LEWINSON. *Riechstoff-Ind. Kosmetik*, 10 (1935), 133–136. (H. M. B.)

Aromatics—New Procedures in the Chemistry of. A review dealing with aldehydes. - A. LEWINSON. *Riechstoff-Ind. Kosmetik*, 10 (1935), 160–163. (H. M. B.)

Camphor—A New Source of. The plant *Ocinum canum* var. *camphoratum* produces camphor even when the drug is only a year old. From 1000 Kg. of fresh material, 7.67 Kg. of ethereal oil extract was obtained. The camphor yield obtained from this ethereal oil, amounted to a maximum of 54% at 0°. The following constants were observed: Melting point: 175°, boiling point 204° density: 0.993, $(\alpha)_D = +8°$, solubility 1:1 in 90% alcohol. The liquid remaining after the separation of camphor is of a yellowish color; this color disappears when the solution is distilled with lime water. An additional yield ranging from 43–53% of camphor can be obtained from this ethereal oil. The solution thus obtained resembles oil of turpentine.—A. I. ROTERMEL. *Sowjet-Pharmaz.*, 5. NI 5.50–54, 1934. Nordkankas. Inst. Speztechkultur; through *Chem. Zentralb.*, 106 (1935), 1088. (G. B.)

Camphor and Camphor Oil from Mauritius. Mauritius camphor trees yield an oil from which no solid camphor can be separated. In 1927 seeds of *Cinnamomum camphora* from trees known to yield solid camphor were imported from Formosa and planted at Curepipe (altitude 1850 ft.). Leaves and twigs removed by pruning in 1931 were distilled; of 160 samples, 17 gave oil only having the following characteristics: d_{20} 0.8831, n_{20} 1.4669, α 14°34′. Oil obtained from the distillation of 785 kilos of twigs and leaves had the following characteristics after removing camphor by cooling for several hours at 0° C.: $d_{15.5}^{15.5}$ 0.9189, α_D^{16} 33.22°, n_D^{20} 1.4754; a further 26% of solid camphor was obtained from the oil by fractional distillation and cooling of the appropriate fraction. The light oil (fraction boiling up to 195° C.) contained 2.0% cineole (equivalent to 0.5% on the decamphorated oil). No safrole could be detected in the higher-boiling fractions.—ANON. *Bull. Imp. Inst.*, 33 (1935), 142–146. (A. P.-C.)

Carvone Series—Researches in. *d*-Carvomenthol was prepared in four ways: deamination of *d*-carvomenthylamine-HCl, hydrogenation of *d*-dihydrocarveol, hydrogenation of *d-cis*, carveol, from *l*-carvomenthone. Constants: b. p. 101.8–102°/14 mm., 104–105°/17 mm.- $n_D^{12°}$ 1.4650, $n_D^{17°}$ 1.4629, $n_D^{20°}$ 1.4617; $d_4^{13°}$ 0.9056, $\alpha_D^{13°}$ +25.08, $(\alpha)_D^{13°}$ +27.69, $(R_L)_D$ 47.62. *l-neo*-Carvomenthol was likewise prepared in four ways: from *l-neo*-carvomenthylamine-HCl, *l-neo*-dihydrocarveol, *d-trans*-carveol and *l*-carvomenthone. Constants: b. p. 95–96°/11.5 mm. $n_D^{18°}$ 1.4652, $\alpha_D^{18°}$ −35.0. *l-iso*-Carvomenthol was prepared by two methods: hydrogenation of *d-trans*-carveol, and deamination of *l-iso*-carvomenthylamine. Constants: b. p. 106°/17 mm., $n_D^{14°}$ 1.4683, $n_D^{20°}$ 1.4662, $d_{20°}^{20°}$ 0.9133, $\alpha_D^{16°}$ −16.14, $(\alpha)_D^{16°}$ −17.72, $(R_L)_D$ 47.54. *l-neo-iso*-Carvomenthol was prepared in small quantities from the mother liquid from hydrogenation of *d-cis*-carveol. Constants: $n_D^{16°}$ 1.5689, $d_{20°}^{20°}$ 0.9137, $\alpha_D^{17°}$ −31.70, $(\alpha)_D^{17°}$ −34.7, $(R_L)_D$ 47.66. *l-iso*-Carvomenthone was prepared from *l-iso*-carvomenthol using Beckman's reagent $n_D^{18.5°}$ 1.4562, $d_4^{20°}$ 0.9042, $\alpha_D^{20°}$ −51.12, $(\alpha)_D^{20°}$ −56.5.—ROBERT G. JOHNSTON and JOHN READ. *J. Chem. Soc.* (1935), 1138–1143. (G. W. F.)

Methyl Ionones—Constitution of the. By condensation of citral with methyl ethyl ketone and cyclization of the mixture of *n*-methyl-pseudoionone and iso-methyl-pseudoionone arise four isomers: (I) alpha-*n*-methyl-ionone, b. (9 mm.) 125–126° C. (semicarbazone m. 146°), (II) beta-*n*-methyl-ionone, b. (9 mm.) 133° (semicarbazone, m. 140°), (III) alpha-*iso*-methyl-ionone, b. (9 mm.) 121–122° (semicarbazone, m. 202°) and (IV) beta-*iso*-methyl-ionone, b. (9 mm.) 124–125° (semicarbazone, m. 182°). (III) and (IV) are valuable in perfumery and known as Iraldeine Gamma and Iraldeine Delta.—H. KOSTER. *Riechstoff-Ind. Kosmetik*, 10 (1935), 159. (H. M. B.)

Oil of Cloves and Oil of Patchouli in the Belgian Congo. The analyses of these two oils, obtained from plants grown at the botanic garden at Eala, shows that the oil of cloves differs in its constants from that of commerce. The optical rotation is dextrorotatory (+0° 58′), density is high (1.0771), refractive index 1.4980, and eugenol content 86%. It is thought that the yield of oil could be increased. The yield of oil of patchouli is so low that cultivation would be uneconomical, even though the oil is of good quality.—P. DENIS. *Pharm. J.*, 135 (1935), 231. (W. B. B.)

Oil of Hyssop. The specific gravity of oil of hyssop frequently exceeds 0.945 and can even reach 0.969. The ester value is generally low (1.4–13.0) but can at times be as high as 33.0. The refractive index generally ranges from 1.476 to 1.482, but sometimes reaches 1.490, exceeding the maximum of 1.486 given by Gildemeister and Hoffmann. The free alcohols, determined by

cold formylation and expressed as $C_{10}H_{18}O$, range from 10.4% to 15.1%. Contrary to Gildemeister and Hoffmann, it is very seldom that the oil is soluble in 70% alcohol.—ETABLISSEMENTS ANTOINE CHIRIS. *Parfums France*, 13 (1935), 239. (A. P.-C.)

Oil of Myrtle Sicilian. Analysis of oils of myrtle produced in the Avola district during the past 10 years had the following characteristics: specific gravity at 15° 0.8792–0.8912, optical rotation 24°30′–28°48′. refractive index at 20° 1.466–1.469, acid value up to 2.8, ester value 42.7–59.5, esters as $C_{12}H_{20}O_2$ 14.95–20.8%, ester value after acetylation 88.3–105.7, soluble in 0.2–1.5 volumes and more of 90% alcohol, except in a few rare instances where the more dilute solutions are opalescent. These characteristics differ considerably from those published by Pellini and Morani (*Ann. chim. applicata*, 7 (1923)).—ETABLISSEMENTS ANTOINE CHIRIS. *Parfums France*, 13 (1935), 239. (A. P.-C.)

Oleoresin of Excoecaria Africana from Kenya. Two samples of oleo-resin extracted with petroleum ether from the wood of *Excoecaria africana* Muell. Arg. (= *Spirotachy africana* Sond.) had: $d_{16.5}^{15.5}$ 1.068, 1.112; α_D^{24} −4.2°, −; n_D^{20} 1.5243, 1.5440; acid value 51.8, 61.0; saponification value 83.2, 88.3; on steam distillation they yielded 7.6% and 19.7%, respectively, of very viscous volatile oil, the viscosity of which increased markedly on keeping. The reddish brown brittle resin remaining after removal of the volatile oil softened at about 40° C. and was liquid at about 50° C. The oleoresin has the properties of a liquid oleoresin or balsam, and rather resembles Canada balsam. Outlet as a substitute for Canada balsam would be very small; it might possibly be used as a fixative of a new character for use in perfumery.—ANON. *Bull. Imp. Inst.*, 33 (1935), 136–140. (A. P.-C.)

Otto of Rose. A review of the new enactments published by the Bulgarian Government for the regulation of the rose otto industry is given.—ANON. *Perfumery Essent. Oil Record*, 26 (1935), 336. (A. C. DeD.)

Palmarosa Oil from Seychelles. Oil obtained in 0.3% yield by distillation of whole plants of palmarosa, grown in Seychelles from seed received from India, had constants falling within the range of figures recorded for Indian palmarosa oil.—ANON. *Bull. Imp. Inst.*, 33 (1935), 140–142. (A. P.-C.)

Perfume Chemistry—Recent Procedures in. The continuation and completion of a review on aromatic alcohols.—A. LEWINSON. *Riechstoff-Ind. Kosmetik*, 10 (1935), 120–123. (H. M. B.)

Perfumery Prime Materials. An alphabetical list of perfumery prime materials including their origin, characters, grades and uses is given.—ANON. *Perfumery Essent. Oil Record, Special Number* (1935), 15. (A. C. DeD.)

Perfumery Products—Notes on New. Coumarin is well known as one of the most important of the synthetic aromatics used in perfumery. It is a valuable fixative, especially for face-powder perfumes, to which it gives necessary sweetness. Synthetic irone suggests itself for use as an alternative to orris oil in high-class perfumery, or as an adjunct thereto when the exact note of orris is required. It may also be used in compositions of a varied type, in order to enrich and to lift up their odors.—ANON. *Pharm. J.*, 135 (1935), 213. (W. B. B.)

Siam Wood Oil. This oil is obtained in Tongkin by steam distillation from the conifer, *Fokienia Hodginsii* and is of value because it possesses a characteristic mild balsamic odor similar to apoponax and auracaria oil. The work of Glichtisch (*Parfums France*, 8 (1930), 157) and Ruzicka is reviewed, and its use in perfumery and soap is discussed.—A. M. BURGER. *Riechstoff-Ind. Kosmetik*, 10 (1935), 139. (H. M. B.)

Soap Perfumes—Suggestions for. Supplemental Formulæ for 21 Odors. Suggestions and formulæ for 21 floral odors for soap are given.—ANON. *Perfumery Essent. Oil Record, Special Number* (1935), 3. (A. C. DeD.)

Terpene Compounds—Formation of, in Plants. A review.—L. S. MALOWAN. *Riechstoff-Ind. Kosmetik*, 10 (1935), 118–120. (H. M. B.)

Fatty Oils, Fats and Waxes

Laurus Nobilis—Oil Content of the Cotyledons of. The method used in the paper was as follows: The weighed cotyledon was rubbed with sand and extracted in a Soxhlet apparatus with petroleum ether, the solvent distilled off and the residue dried to constant weight at 100°. The residue consisted practically entirely of fatty oil since the volatile oil content of the cotyle-

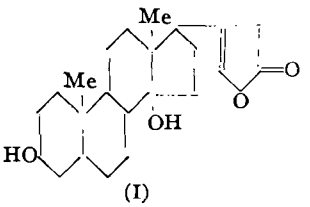
don is small and since it is volatile at 100°. Both cotyledons were treated in the same manner and the results of 50 pairs of cotyledons tabulated. The average weight of a cotyledon was 0.249 Gm., the average weight of residue was 0.058 Gm., representing 23.32% of oil. As was found for almonds in an earlier paper, the oil content of the two adhering cotyledons was the same in many cases, and where there was a difference, it did not exceed 2% as a rule.—L. ROSENTHALER. *Pharm. Acta Helv.*, 10 (1935), 135. (M. F. W. D.)

Sunflower Seed Oil—Change in the Acidity and Iodine Number of, on Heating the Pulp Prior to Extraction. When sunflower seed pulp is heated prior to extraction of the oil the chemical constants of the latter undergo the following changes: the acidity decreases, being 1.5 to 12.0 units lower on leaving the press than in the crude pulp; the iodine number increases, being 7.7 to 10.4 units higher on leaving the press than in the crude pulp.—I. KOLPAKOV. *Masloboino Zhirovoĕ Dĕlo*, 10 (1934), No. 11, 32–34; through *Chimie & Industrie*, 34 (1935), 641–642. (A. P.-C.)

Glycosides, Ferments and Carbohydrates

Chamomile Flowers—III Investigation of the Glycosides of. The article continues some previous work on the glycosides of chamomile obtained from various sources. A table showing the optical rotation and reducing sugar content of the original extracts, the change produced after treatment with invertase and after emulsin is included in the article. The author could not confirm earlier conclusions as to the constancy of the glycosidal content. The glycoside content was found to be quite variable, chamomile from the same source varying greatly from year to year.—C. BÉGUIN. *Pharm. Acta Helv.*, 10 (1935), 147. (M. F. W. D.)

Digitalis Glucosides. The existence of two anhydrodigitoxigenins is reported. Upon treatment with diluted acids, digitoxigenin yields two crystalline anhydrides. No indication of a third isomeride was observed; this is in accordance with Tschesche's formula for digitoxigenin (I). Both anhydrides gave the Legal reaction and therefore retain the double bond in the lactone group ($\Delta^{20:21}$) and both retain the secondary alcoholic group as indicated by formation of an acetate and upon oxidation yielding a ketone. The two isomerides, obtained by fractional crystallization had the following characteristics: *alpha-Anhydrodigotoxigenin:* m. p. 234°; $(\alpha)^{20°}_{Hg}$ gr + 43.7; $(\alpha)^{20°}_{D}$ +39.0 (in methyl alcohol, c = 0.43); *beta-Anhydrodigotoxigenin:* m. p. 202°; $(\alpha)^{20°}_{Hg}$ gr −17.3°; $(\alpha)^{20°}_{D}$ −13.3 (in methyl alcohol, c = 2.05).



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SYDNEY SMITH. *J. Chem. Soc.* (1935), 1050–1051. (G. W. F.)

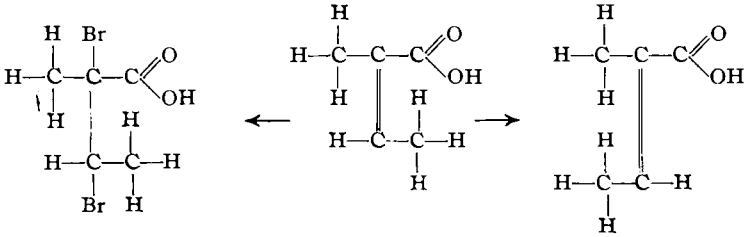
Digoxigenin—Constitution of. This aglucone of digoxin is thought to have the same skeletal structure as digitoxigenin. Of the five oxygens present, one is a tertiary alcohol group on C 14, two are in a lactone group on C 17 and two are secondary alcohol groups. On biogenic grounds, one is on C 3, and the other remains unaccounted for.—SYDNEY SMITH. *J. Chem. Soc.* (1935), 1305–1309. (G. W. F.)

Ouabain and *k*-Strophanthin—Stability of Aqueous Solutions of. Preliminary experiments with ordinary unbuffered solution of ouabain and *k*-strophanthin in distilled water (the initial of p_H of the solutions lying between 5.5 and 6.0) indicated that after autoclaving for one hour, the ouabain solution retained its full activity, while in the case of *k*-strophanthin there was an appreciable reduction. When the p_H was altered to 2.0 and 9.0, ouabain showed on heating some reduction in activity while *k*-strophanthin lost all activity at p_H 2.0 and an appreciable amount at p_H 9.0. The exact effect of alteration of p_H was then traced, solutions being prepared in the buffer solutions of the Pharmacopœia. Tables showing the results of these experiments are given, in which it is shown that ouabain is much more stable in aqueous solution than *k*-strophanthin, the stability zone of the latter being between p_H 6.0 and 7.0 while that of ouabain is between p_H 5.0 and 8.0. Glass containers for solutions of *k*-strophanthin should comply with the tests for limit of alkalinity.—H. BERRY. *Pharm. J.*, 135 (1935), 10. (W. B. B.)

Toad Poisons—Note on the X-ray Crystallography of, Bufagin and Cinobufagin, and of Strophanthidin. The pharmacological action of the toad poisons is very similar to that of the heart poisons and suggests that these compounds, although of animal and plant origin, respectively, are closely related structurally. The X-ray investigations did not prove very illuminating concerning the structures of cinobufagin and strophanthidin. Work on derivatives of the compounds might be more helpful. Results obtained for bufagin strongly support the suggestion that this compound is closely related to the cardiac aglucones.—DOROTHY CROWFOOT. *J. Soc. Chem. Ind.*, 54 (1935), 568. (E. G. V.)

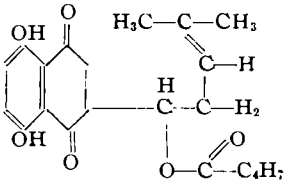
Other Plant Principles

Alkannet—Analysis of. The authors state that the root of *Alkanna tinctoria* was analyzed for many years for a coloring material obtained from the root. The root was first extracted with ether, then finally reacted with a solution of copper acetate. The formula $C_{21}H_{20}O_6Cu$ or $C_{20}H_{20}O_6Cu$ was given to this compound. It was then washed with water and then extracted with benzol, to remove traces of wax-like substances. To this copper compound ether and then 25% of hydrochloric acid was added and a free coloring material was taken up. This ethereal extraction was of an oily reddish nature which did not crystallize out. On examination it was found that it had no methoxy group. During the extraction process of the oily coloring material and subsequent purification with potassium hydroxide a sharp unpleasant odor developed. To determine the source of this odor, the mixture was acidified, separated and then neutralized with neutral sodium carbonate. After distillation with 50% sulphuric acid a product was obtained which because of its composition and behavior proved to be angelic acid (I). By reacting it with bromine, α,β-dibrom-α-methyl-butyric acid (II) formed which had a melting point of 86°. If this compound was heated in a closed tube to 300°, tiglic acid (III) was the result.



II I III

Angelic acid can in turn be esterified (IV). To which of the organic radicles, the hydroxyl group is attached, has not been determined yet.

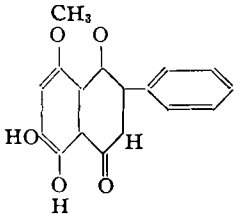


IV

K. BRAND and A. LOHMAN. *Ber.*, 68 (1935), 1487. (G. B.)

Alpinia Japonica Miq. Constituents of. II. Izalpinin, a New Flavone. Ether extracted from the seeds of **A. japonica** Miq. (Japanese drug "Izu-sy-ukusya) 0.39% of a crystalline mixture. The substance less soluble in toluene, "alpinon," constitutes 3/4 of the mixture; it is to be described subsequently. The more soluble compound was called izalpinin: $C_{16}H_{12}O_5$; yellow needles, m. p. 195°; sl. soluble in cold ether, ethanol, chloroform, acetone and toluene, more readily in hot solvents; freely soluble in pyridine, α of solution $\pm 0°$; dirty green color with alcoholic ferric chloride; yellow solution in alkali; sulphuric acid solution yellow with green fluorescence; dibenzoate, colorless, m. p. 189° ex. hot toluene; diacetate, colorless, m. p. 170–71° ex. methanol; dimethyl ether, yellowish needles, m. p. 193.5° ex. methanol. Isalpinin contains 2 phenolic

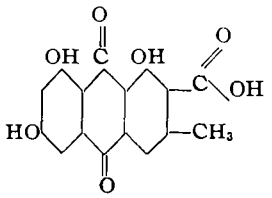
hydroxyls and 1 methoxy. Hydriodic acid removed methyl to give *nor*-izalpinin; m. p. 217° ex. 70% acetic acid; reddish brown crystals resembling glass wool. Fusion of izalpinin with potassium hydroxide yielded chiefly acetic acid (anilide, m. p. 114°), benzoic acid (m. p. 121° with anthentic sample) and an unidentified poly-phenol. These findings indicated a flavone structure. Of the 4 such structures possible for *nor*-izalpinin, 2 have been assigned to baicalein and *nor*-wogonin, which differ from *nor*-izalpinin. A third is not appropriate because izalpinin contains 1 difficultly methylated —OH, which is therefore probably adjacent to a CO group. The most probable methyl-ether of the remaining flavone structure is



Y. KIMURA and M. HOSHI. *J. Pharm. Soc. Japan*, 54 (1934), 135–137. (R. E. K.)

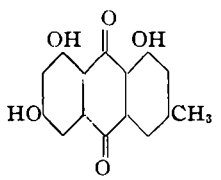
Aperta Ruao—Pharmaceutical Study of. Velloso is described. Analysis of the leaves shows the presence of an essential oil, a resinous substance, a crystalline principle quite similar to artantic acid and an amorphous bitter substance which is probably maticine. The leaves have astringent menostatic and antiblennorhagic properties.—OSWALDO DE ALMEIDA COSTA. *Rev. flora med.*, 1, 415–422 (1935), Piper Aduncum.; through *Chem. Abstracts*, 29 (1935), 5224.

Endocrocin. A New Oxy-anthraquinone Derivative. The oxy-anthraquinone which is found in a large variety of lichens play an important part in the changes of color which occur during their lifetime. Its presence can easily be detected by the color it imparts to the lichen which is either, yellow, orange, red or brown. The addition of a solution of an alkali to colored lichens will give a purplish red to violet blue color. A japanese leaf lichen *Nephromopsis endocrocea* contains an orange-yellow coloring substance which turns, purplish-red with a solution of an alkali. This proves to be an oxy-anthraquinone derivative. By extraction with acetone the color was isolated and named Endocrocin. It melts at a temperature of 318° and has the formula $C_{16}H_{10}O_7$ (I).



I

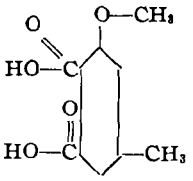
Two of the groups are ketonic, two are of a carboxylic type and the rest are of a phenolic type. If endocrocin is decarboxylized it will change to a frangula-emodin (II)



II

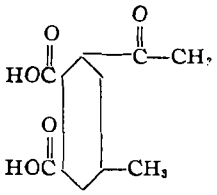
type of a compound. According to the spectroscopic examination, Endocrocin is quite similar to emodin. In order to ascertain the presence of carboxyl groups, the methyl group of the side chain was oxidized to a carboxyl group. The tri-acetyl-endocrocin obtained was heated on the water-bath with chromic acid and a solution of acetic anhydride so as to eliminate the formation of any other compound. In comparison to this, trimethyl ether-endocrocin-methylester yielded with the same reagents a combination compound ($C_{12}H_{12}O_7$) which corresponds to a tricarbonic acid with a methyl-ether and methyl-ester group. These methoxyl and carbo-methoxyl groups were evi-

dently in the starting product. The authors found that emodin-tri-methyl ether (II) could be oxidized to dicarbonic acid $C_{10}H_{10}O_5$ which was identical with methyl-ether-*y*-cochinic acid (III).



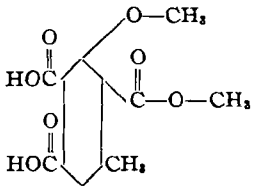
III

The oxidation product of the methylated endocrocin has as a nucleous a methyl-ether-*y*-cochinic acid which has the constitution of either formula IV



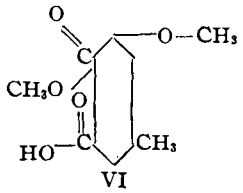
IV

or V.



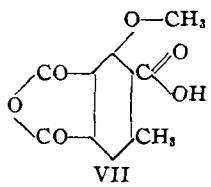
V

The only difference between methyl ether—cochinillic acid tri-methyl ester VI



VI

and formula IV is in the position of the methyl group. Although the constitution of tricarbonic acid VII,



VII

methyl-ether-cochinillic acid and methyl-ether-cochinillic acid tri-methyl ester is nearly similar, their melting point differs considerably.—YASUHIKO ASAHINA and FUKUZITO FUZIKAWA. *Ber.*, 68 (1935), 1558. (G. B.)

Ergot of Rye—Nucleic Acid from. The sclerotium of *Claviceps purpurea* is a raw material rich in phosphorus compounds. The authors have been able to isolate sufficient quantities of nucleic acid to be able to determine the chemical structure and have elaborated methods for

estimation of the purine bases occurring in small quantities in nucleic acid.—M. GATTY-KOSTYAL and J. TESARZ. *Pharm. J.*, 135 (1935), 231. (W. B. B.)

Holarrhaena Antidysenterica—Resinols of the Latex of. No alkaloids were found in the latex. The alcohol soluble portion of the latex, treated with acetone, left a residue—lettoresinol-A ($C_{26}H_{50}O_5$), m. p. 227–228°, neutral, dextrorotatory, very inert. From the warm acetone solution, lettoresinol-B ($C_{32}H_{56}O_2$), m. p. 136–137°, separated. It was neutral, dextrorotatory, gave a diacetyl compound and a mixture of oxidation products with chromic acid.—J. C. CHOWDHURY and D. H. PEACOCK. *J. Chem. Soc.* (1935), 1129–1131. (G. W. F.)

Ramaline Scopulorum—Constituents of. The substance, previously called scopuloric acid, obtained from the acetone extract, was proved to be identical with stictic acid (sinters at 258°, m. p. 270°).—FRANCIS H. CURD and ALEXANDER ROBERTSON. *J. Chem. Soc.* (1935), 1379–1381. (G. W. F.)

Red Saunders—Colorless Principles of. The scope of this experiment was to analyze the active principles of red saunders, namely: Santalin (santalic acid) the principle which imparts color to the drug, and pterocarpin and homopterocarpin which are the colorless principles. The first problem was to determine the molecular weights of pterocarpin and homo-pterocarpin, and second to ascertain whether there is any relationship between the formula of santalin and the colorless principles which are found in the drug. Subsequently the coarsely rasped powder was extracted with ether completely. To this mixture ethyl acetate was added and gentle heat applied. On cooling this mixture a large quantity of crystals separated out. They were dried and washed with cold methanol. A hot solution of carbon tetrachloride was then used in order to dissolve out the pterocarpin and homo-pterocarpin compounds. The remaining santalin crystals were further washed with hot carbon tetrachloride. The crystals were of a yellowish color, having the shape of leaflets and melting at 218°. On analysis it was found to have the formula $C_{13}H_{10}O_5$. The carbon tetrachloride mixture which was obtained during the washing of the santalin crystals was cooled off; yellow prismatic crystals of pterocarpin separated out which melt at 161°. In order to obtain colorless crystals of pterocarpin, the crystals were dissolved in alcohol to which animal charcoal was added. Colorless needles separated out which melted at 165°. Its formula was found to be $C_1H_{174}O_5$. On further concentration of the mother liquor colorless needle-like crystals of homo-pterocarpine separated out. These crystals melted at a temperature of 87–88°. The formula of this last compound was $C_{17}H_{16}O_4$.—H. RANDNITZ and G. PERLMANN. *Ber.*, 68 (1935), 1862. (G. B.)

Sanguisorba Officinalis, L.—II. Constituents of the roots of. Abe and Kodake preferred the formula $C_{23}H_{44}O_3$ for the hydroxy-acid sanguisorbigenin (I). The present authors also changed their earlier findings and advocated $C_{30}H_{46}O_3$ or $C_{29}H_{44}O_3$. I was prepared by 40 hours hydrolysis of the saponin sanguisorbin with 5% alcoholic hydrochloric acid: m. p. 272–273°; m. wt. 441 (av., Rast), 443 (av., titration). It yielded 3 acetates: (*a*) m. p. 314–317°, (*b*) 259.5° and (*c*) 212°; (*a*) and (*b*) showed theoretical analyses, m. wt. and acetyl no.; (*c*) contains 1 H_2O; saponified to I, m. p. 278–80°. Dimethyl sulphate and diazomethane converted I into the methyl-ester: m. p. 166.5°. (Abe and Kodake found m. p. 201–203°); acetyl methyl ester, m. p. 242–244°, saponified to methyl-ester m. p. 216° (identical with methyl-ester of A. and K.?). "Decarboxy-genin" was obtained by eliminating CO_2 from I: $C_{29}H_{46}O$; m. p. 205.5°; no alcohol-insoluble digitonide; acetate, m. p. 185.5, yielding substance m. p. 216° after saponification. Dehydrogenation, 5 Gm. sanguisorbigenin heated 50 hrs. under carbon dioxide with 7.3 Gm. selenium, yielded an ether-soluble hydrocarbon, probably sapotalin: b. p. 130–145° at 13 mm.; picrate, $C_{19}H_{17}O_7N_3$, m. p. 129°. The authors concluded that sanguisorbigenin may have a skeleton similar to such sapogenins as hederagenin, oleanolic and ursolic acids.—K. FUJII and H. SHIMAD. *J. Pharm. Soc. Japan*, 54 (1934), 129–132. (R. E. K.)

Unclassified

Alcoholic Fermentation—Chemistry of. A review of what is known at present about the chemical processes involved in alcoholic fermentation. A list of most of the compounds involved in fermentation is given. The field of fermentation is without boundary; progress in its exploration has always meant progress in the field of general metabolism, both of animals and plants, and vice versa.—LEONOR MICHAELIS. *Ind. Eng. Chem.*, 27 (1935), 1037. (E. G. V.)

Alkylresorcinolcarboxylic Acids—Germicidal Properties and Mercuration of Some.

Ethyl, *n*-propyl, *n*-butyl and *n*-hexylresorcinolcarboxylic acids were prepared and mercurated. The phenol coefficients of the acids were determined, using *B. typhosus*, and were found to be much less than the corresponding alkylresorcinols. The acids were readily mercurated, but the resulting mercury derivatives, though readily soluble in dilute alkali, were quite unstable, and their germicidal activity could not be determined.—S. C. OVERBAUGH and R. B. SANDIN. *J. Am. Chem. Soc.*, 57 (1935), 1658. (E. B. S.)

Antimalarials—Attempts to Find New. Numerous derivatives of 8-methylquinoline were prepared; although inactive to bird malaria, they were found to possess local anesthetic activity: 6-methoxy-8-methylquinoline, an oil forming hydrobromide (m. p. 268°) and picrate (m. p. 232–233°); 5-bromo-6-methoxy-8-methylquinoline (m. p. 116–117°), hydrobromide (m. p. 230°); 4-bromo-6-nitro-*m*-cresol (m. p. 146°); 4-bromo-6-nitro-*m*-tolyl methyl ether (m. p. 110–111°); 4-bromo-5-methoxy-*o*-toluidine (m. p. 78–79°); 4:6-dibromo-*m*-tolyl methyl ether (m. p. 73–74°); 6-bromo-4-nitro-*m*-tolyl methyl ether (m. p. 113–115°), reducing to form 2-bromo-5-methoxy-*p*-toluidine (m. p. 71–73°); 5-bromo-6-methoxy-8-methylquinoline (m. p. 116–117°); 7-bromo-6-methoxy-8-methylquinoline (m. p. 134–135°); 3-bromo-6-nitro-8-methylquinoline (m. p. 188–189°); 8-quinolylmethyl alcohol (m. p. 75–76°); 5-nitro-8-quinolylmethyl alcohol (m. p. 148–149°); 5-nitro-8-piperidinomethylquinoline, an oil, forming hydrobromide (m. p. 248–249°); beta-(*o*-nitrobenzylethylamino)triethylamine, an oil, forming picrate (m. p. 167–168°); beta-(*o*-aminobenzylethylamino)triethylamine, forming picrate (m. p. 134°); beta-(benzylethylamino) triethylamine, forming picrate (m. p. 150–152°); beta-(*p*-nitrobenzylmethylamino)-triethylamine, forming picrate (m. p. 195–197°); beta-diethylamino ethylpropylamine (b. p. 184–200°) forming monopicrate (m. p. 133–135°); beta-diethylaminoethyl-*n*-butylamine (b. p. 207–212°) forming dipicrate (m. p. 234°); beta-diethylaminoethylisobutylamine (b. p. 194–200°) forming dipicrate (m. p. 141°); beta-diethylaminodiethylamine forming monopicrate (m. p. 139–140°) and dipicrate (m. p. 150–151°); beta-diethylaminoethylmethylamine (b. p. 157–160°); beta-diethylaminoethylamine (b. p. 145–155°); beta-piperidinoethyl chloride hydrochloride (m. p. 228–230°); beta-piperidinoethylpropylamine (b. p. 220–230°), dipicrate (m. p. 169°); beta-piperidino-*n*-butylamine (b. p. 230–240°), dipicrate (m. p. 191–192°); beta-piperidinoisobutylamine (b. p. 230–240°), dipicrate (m. p. 167–168°); beta-piperidinoethylmethylamine (b. p. 190–200°), dipicrate (m. p. 174°); beta-piperidinodiethylamine (b. p. 200–210°), dipicrate (m. p. 154°); *s.*-dimethylethylenediamine (b. p. 150–160°), picrate (m. p. 160°); 8-(beta-diethylaminoethylmethylaminomethyl)quinoline, an oil, forming trihydrobromide (m. p. 215–216°); 8-(beta-diethylaminodiethylaminomethyl)quinoline trihydrobromide (m. p. 218–219°), picrate (m. p. 131–132°); 8-(beta-diethylaminoethylpropylaminomethyl)quinoline monopicrate (m. p. 113–115°); dipicrate (m. p. 163–164°); 8-(beta-diethylaminoethyl-*n*-butylaminomethyl)quinoline dipicrate (m. p. 178–180°); 8-(beta-diethylaminoethylisobutylaminomethyl)quinoline dipicrate (m. p. 169–171°); 8-(beta-piperidinoethylpropylaminomethyl)quinoline trihydrobromide (m. p. 210°); 8-(beta-piperidinoethyl-*n*-butylaminomethyl)quinoline trihydrobromide (m. p. 211–212°); 8-(beta-piperidinoethylisobutylaminomethyl)quinoline dipicrate (m. p. 210–211°); 8-(beta-piperidinoethylmethylaminomethyl)quinoline dipicrate (m. p. 205–206°); 8-(beta-piperidinodiethylaminomethyl)quinoline trihydrobromide (m. p. 222°); *s.-bis*-(8-quinolylmethyl)dimethylethylenediamine dihydrobromide (m. p. 232°); *bis*-(8-quinolylmethyl)-beta-diethylaminoethylamine (m. p. 97–98), picrate (m. p. 191°); 8-(beta-diethylaminoethylaminomethyl)quinoline trihydrobromide (m. p. 223–224°); *l*-beta-*bis*-(8'-quinolylmethyl)aminoethylpiperidine (m. p. 97–98°); picrate (m. p. 228–229°); 1:4-*bis*-(8'quinolylmethyl)piperazine (m. p. 153–154°), picrate (m. p. 265–267°).—WM. O. KERMACK and THOS. W. WIGHT. *J. Chem. Soc.* (1935), 1421–1426.

(G. W. F.)

Antimalarials—Attempts to Find New. Synthesis of ω-substituted derivatives of 8-methylquinoline is reported. 8-Piperidinomethylquinoline picrate (m. p. 179°) was prepared by allowing a mixture of glycerin solution of boric acid, *o*-aminobenzylpiperidine, ferrous sulphate, and concentrated sulphuric acid to simmer for 20 hours. The solution was then steam distilled, basified, and re-steam distilled, extracted with ether, washed with water, dried, and ether evaporated. The oily residue did not crystallize but yielded a crystalline picrate. It (m. p. 183°) was also formed by refluxing 8-bromomethylquinoline and piperidine in benzene solution. This was extracted with dilute hydrochloric acid, the extract basified, extracted with ether and ether removed. 6-Piperidinomethylquinoline (m. p. 195–196° decomp.) was prepared from *p*-amino-

benzylpiperidine and *p*-nitrobenzylpiperidine by the first method. 8-Diethylaminomethylquinoline hydrobromide (m. p. 238–239°) was prepared by refluxing a benzene solution of 8-bromomethylquinoline and diethylamine in presence of potassium carbonate. Di(quinolyl-8-methyl)ethylamine hydrobromide (m. p. 202°) was prepared by refluxing 8-bromomethylquinoline, ethylamine and alcohol, diluting with water, made strongly alkaline, extracted with ether, washing the extract with water, drying and evaporating ether. 8-Anilinomethylquinoline (m. p. 71–73°) was prepared by heating 8-bromomethylquinoline with aniline, the cake formed dissolved in water, basified with sodium carbonate, steam distilled, allowed to stand over night, and crystallized from alcohol. N-Di(quinolyl-8-methyl)-*p*-aminoacetanilide (m. p. 236–237°) was prepared by refluxing a concentrated alcoholic solution of *p*-aminoacetanilide with 8-bromomethylquinoline, filtering, dissolving in hot hydrochloric acid and basifying, which precipitated the product.—WM. O. KERMACK, WM. MUIR and THOS. W. WIGHT. *J. Chem. Soc.* (1935), 1143–1145. (G. W. F.)

Antimalarials—Chemistry of the New. A review.—KONRAD FUNKE. *Pharm. Monatsh.* 16 (1935), 129–131. (H. M. B.)

Arsenated Phenoxyethanols. A series of arsenated phenoxyethanols were prepared and their physical properties listed. No report on their trypanocidal action is given.—M. R. STEVINSON and C. S. HAMILTON. *J. Am. Chem. Soc.*, 57 (1935), 1600. (E. B. S.)

5-Benzyl-8-hydroxyquinoline—New Synthesis of, and the Antiseptic Activity of Its Sulphate. 5-Benzyl-8-hydroxyquinoline was synthesized from *o*-amino-*p*-benzylphenol by a modified Skraup reaction yielding 86% of the theoretical. The antiseptic action of its sulphate was tested on *B. coli*. It was found to be more germicidal than phenol and as effective as 8-hydroxyquinoline sulphate, and much less irritating to mucous membranes.—L. MCMASTER and W. M. BRUNER. *J. Am. Chem. Soc.*, 57 (1935), 1697. (E. B. S.)

Ergosterol—Chemistry of Some of the Irradiation Products of. A discussion of the various structural formulæ assigned to lumisterol, tachysterol and calciferol.—I. M. HEILBRON and F. S. SPRING. *J. Soc. Chem. Ind.*, 54 (1935), 795. (E. G. V.)

Gutta-Percha and Balata Hydrochlorides. Reduction of gutta-percha hydrochloride with zinc dust and alcohol in ethylene dichloride solution, precipitation with alcohol, and extraction with ether resulted in α-monocyclogutta-percha: softens at 70°, m. p. 85–90°, decomposes at 120°; the ether-insoluble portion was β-monocyclogutta-percha: softens at 65–68°, m. p. 84–86°, decomposes at 120°. Likewise α-monocyclobalata (m. p. 100–105°, decomp. 120–125°) and β-monocyclobalata (softens at 85–90°, m. p. *ca.* 100°, decomp. 115–120°). All were unsaturated compounds.—THOMAS HARDIE AND JOHN A. MAIR. *J. Chem. Soc.* (1935), 1239–1241. (G. W. F.)

Homoapofenchocamphoric Acid—Synthesis of. Ethyl-1-1-dimethyl glutarate (b. p. 110–115°/9 mm.) prepared from *iso*-laurolonic acid, was condensed with ethyl oxalate in the presence of potassium ethoxide. The product, when distilled under reduced pressure, yielded ethyl γ-methylbutane-α-α-γ-tricarboxylate (b. p. 145–148°/10 mm.). A sodio-derivative, prepared with alcoholic sodium ethoxide, on digestion with ethyl bromoacetate gave a good yield of ethyl β-methylpentane-β-δ-δ-ε-tetracarboxylate (b. p. 175–176°/9 mm.). Upon the hydrolysis and elimination of carbon dioxide, β-methylpentane-β-δ-ε-tricarboxylic acid (m. p. 185–187°) was obtained. By the alcohol-vapor method, it yielded an ethyl ester (b. p. 159°/8 mm.). This was cyclized by means of sodium, producing ethyl 2:2-dimethylcyclopentan-1-one-4:5-dicarboxylate (b. p. 147–149°/9 mm.). This was hydrolyzed by 8% sulphuric acid to 2:2-dimethylcyclopentan-1-one-4-carboxylic acid (m. p. 93°). The ethyl ester (m. p. 113°/9 mm.), on treatment with ethyl bromoacetate and zinc, gave a product (hydroxy-ester) which could not be obtained pure. It was converted into a mixture of unsaturated esters (b. p. 126–127°/4 mm.) by means of phosphoryl chloride. Reduction of this mixture by hydrogen and platinum oxide gave a fully saturated ester which on hydrolysis with alcoholic potash gave homoapofenchocamphoric acid (m. p. 246°).—J. C. BARDHAN, S. K. BANERJI and M. K. BOSE. *J. Chem. Soc.* (1935), 1127–1129. (G. W. F.)

Myrtenol—Synthesis of. In an earlier paper it was pointed out that by the action of selenium oxide on pinene there is obtained verbenal and verbenone. A more complete study led the authors to change their opinion; the principal product of the reaction is really myrtenol and myrtenal. Until recently these products were rare and expensive; their presence is described in essence of myrte (myrtenol d.), in the false oil of camphor wood (myrtenal d.); the

levorotatory and inactive varieties are unknown. The method of synthesis used by the authors gives a good yield (35% of myrtenol and 10.6% of myrtenal) and has been carried out on the dextro-pinene extract of the essence of maritime pine, on the levo-pinene extract of the oil of Alep and on an inactive pinene obtained by the mixture of the two preceding varieties. Thus they are able to obtain the different optical varieties.—G. DUPONT and W. ZACHAREWICZ. *Bull. Soc. Chim. France* (Mar. 1935); through *J. pharm. Belg.*, 17 (1935), 670. (S. W. G.)

Thiophen—Removal of, from Benzene by the Action of Acidified Hypochlorite Solutions. A mixture of 500 cc. of benzene solution of 5.82×10^{-2} thiophen molarity, 299 cc. of high test hypochlorite solution (Mathieson) of 0.4869 hypochlorite ion molarity, and 78 cc. of 5.96 *M* acetic acid was thoroughly agitated at room temperature for 15 minutes. The amounts of reagents correspond to a thiophen:hypochlorite ion:acetic acid molar ratio of 1:5:16, which was the established minimum. The aqueous layer with water washings was retained for further examination. As the benzene layer was now colored greenish yellow, washings with caustic soda solution followed. This alkaline washing removed this coloration, which was no doubt due to dissolved chlorine. After drying over calcium chloride the benzene was distilled using a six-bulb column to insure retention of chlorinated derivatives in the residue. The distilled product was bright and clear, free from thiophen as shown by the isatin test, and free from sulphur as indicated by the lamp method of analysis.—E. G. R. ARDAGH and WM. H. BOWMAN. *J. Soc. Chem. Ind.*, 54 (1935), 267T. (E. G. V.)

BIOCHEMISTRY

Adrenaline Stabilization—Perfusion of the Adrenal Gland with Reference to the Mechanism of. The perfusion of the adrenal gland confirmed the secretion of a stable sympathomimetic substance, which may be stabilized adrenaline, since adrenaline added to the perfusate is likewise stabilized. Stabilization is effected by means of an oxidation reduction system which tends to reduce the 3, 4-quinone of adrenaline and thereby inhibit pigment formation. The reduction of 2,6-dichlorophenolindophenol indicates that this reducing agent is ascorbic acid. It is also significant that there is no diminution in the pressor activity of the perfusate until its ascorbic acid is oxidized.—ROBERT D. H. HEARD and ARNOLD D. WELCH. *Biochem. J.*, 29 (1935), 998. (J. C. B.)

Alcohol—Microdetermination of 0.1–0.5 mg. of, in Blood and Tissues. The alcohol is distilled in a micro-apparatus, the distillate is collected and is oxidized by sulpho-chromic mixture in a ground-glass apparatus having a capacity of 14 cc. After the addition of Mohr's salt, the excess is titrated with potassium permanganate.—L. NICLOUX, LE BRETON and A. DONTCHEFF. *Bull. Soc. Chim. Biol.* (1934), 16; through *J. pharm. Belg.*, 17 (1935), 638. (S. W. G.)

Arsenic and Selenium—Biological Methylation of Compounds of. A review, with bibliography.—FREDERICK CHALLENGER. *J. Soc. Chem. Ind.*, 54 (1935), 657. (E. G. V.)

Ascorbic Acid—Catalytic Oxidation of. Copper and iron catalyze this reaction. Ordinary laboratory distilled water from metal stills contains sufficient copper and iron to cause the irreversible oxidation of dissolved ascorbic acid. In the very early stages of the process dehydroascorbic acid is formed in quantities almost equivalent to the amounts of ascorbic acid oxidized, but as the reaction proceeds the quantities of dehydrogenated acid present diminish. This catalytic action is barely perceptible when the vitamin is dissolved in water which has been redistilled several times from glass apparatus and used immediately or in water distilled from and received in quartz. Saturating glass or quartz distilled water with oxygen does not increase significantly the disappearance of ascorbic acid. The addition of aqueous extracts of liver, kidney, muscle spleen, large and small intestine, plasma, intact or hemolyzed erythrocytes and 0.1*N* sodium chloride inhibits the oxidation of ascorbic acid in ordinary distilled water or water to which iron or copper has been added. Leucocytes have no influence on this reaction. Although hemolyzed erythrocytes inhibit this oxidation reaction, when first added this solution produces a slight but immediate disappearance of ascorbic acid, which may be due to the liberated oxyhemoglobin since the addition of carbon monoxide will eliminate this effect.—ALEXANDER E. KELLIE and SYLVESTER S. ZILVA. *Biochem. J.*, 29 (1935), 1028. (J. C. B.)

Biochemical Analysis—Clinical Value of Recent Methods of. Inspection, reaction, protein, reduction—sums up the minimum examination required in the routine qualitative chemical testing of urine. Some of the recently introduced drugs have definitely complicated the in-

spection. Thus amidopyrine (pyramidon, dimethylaminophenazone) as such, or in combination or in mixture with a number of other drugs sold under different proprietary names is excreted as rubazonic acid, which is red, and as a glycuronate which may cause a slight reduction. Both acid and alkaline urines have a pink or red color which may be mistaken for that of blood. They are easily differentiated by means of the direct-vision spectroscope, which shows no definite band, but an ill-defined absorption of the green-blue. An excess of either acid or alkali changes the color to yellow. The red pigment is very readily extracted by amyl alcohol. The urinary antiseptics Pyridium (phenyl azo-alpha-alpha-diamino pyridine hydrochloride) and Neotropin (2-butyloxy-2′.6′-diamino 5.5′-azo-pyridine) are further examples. They make the urine deep orange or orange-red, and spectroscopically there is a very intense general absorption of about half the green and of the blue-violet, which may catch the unwary when testing for urobilin. Amyl alcohol partially extracts the dye. Excess alkali causes no significant color change, but excess acid makes the color deeper and more red, and shifts the absorption obviously toward the red end of the spectrum. Methods used in testing for reducing substances, hematuria, bilirubin and in blood and gastric analyses are also discussed.—G. A. HARRISON. *Pharm. J.*, 135 (1935), 183. (W. B. B.)

Blood Phosphorus—Researches on Acid-Soluble, Mineral and Ultrafilterable. The ratio between the acid-soluble, mineral and ultrafilterable phosphorus in normal serum and plasma of dog and man was determined, as well as their variations in experimental acidosis and alkalosis with the dog. In normal serum and plasma 60–80% of the acid-soluble phosphorus was found to be ultrafilterable and consisting entirely of orthophosphates. In experimental acidosis, the proportion of acid-soluble phosphorus and orthophosphates increased while in the alkalosis it diminished. The increase might be explained by migration of the phosphorus derivatives of the corpuscles into the serum, by a migration of the tissular phosphorus into the blood or by a transformation of organic combinations of total acid-soluble phosphorus into mineral phosphorus. The decrease might be explained either by a synthesis of phosphorus organic compounds, by a migration of the seric phosphorus to the tissues, or by renal elimination. It is possible that several of these factors intervene at the same time.—ISABELA POTOP. *Compt. rend.*, 201 (1935), 490. (G. W. H.)

Calciferol—Molecular Shape of, and Related Substances. Molecular dimensions are given for cholesterol, ergosterol, dihydroergosterol, ergostenol (α and β) lumisterol, calciferol, calciferol-pyrocalciferol, and the suprasterols (I and II). With complicated formula the X-ray method does not lead directly to a formula, but may serve to discriminate between formulæ.—J. D. BERNAL and D. CROWFOOT. *J. Soc. Chem. Ind.*, 54 (1935), 701. (E. G. V.)

Calciferol (Vitamin D)—Constitution of. A Review and a Suggestion. The photochemical changes induced by irradiation of ergosterol seem to follow the sequence: ergosterol ⟶ lumisterol ⟶ tachysterol ⟶ calciferol. Suggestions with regard to the constitution of calciferol are discussed. The assumption of the presence in calciferol of a tricyclic ring system, embodying a combination of a ten-, six-, and five-membered ring, to which is joined a nine-membered side-chain, offers such a unique and specific arrangement of carbon atoms that it would not be surprising to find it associated with the equally specific antirachitic activity of calciferol. The formulæ based on this ring system are in agreement with the results of uni-molecular surface film measurements and fulfil the demands of X-ray crystallography.—O. ROSENHEIM and H. KING. *J. Soc. Chem. Ind.*, 54 (1935), 699. (E. G. V.)

Chlorine in Blood—Determination of. The present methods are critically reviewed. The following method is proposed: *Reagents.*—(*a*) Solution of potassium ferrocyanide: 150 Gm. of anhydrous salt or 172 Gm. of the trihydrate, and enough water to make a liter of solution. (*b*) Solution of zinc acetate: 112 Gm. of anhydrous salt or 134 Gm. of the dihydrate, and enough water to make a liter of solution. (*c*) *N*/50 ammonium thiocyanate. (*d*) *N*/50 silver nitrate. (*e*) Official nitric acid. (*f*) Toluene. (g) Saturated solution of iron ammonium alum. *Method.*—Collect 6–7 cc. of blood and mix with 2–3 cg. of sodium citrate. Measure 2 cc. of the total blood into a conical centrifuge tube, using an Ostwald-Meillère pipette. Centrifuge for 2 minutes and decant the plasma with an Ambard siphon. Wash three times by centrifuging with serum treated with 5.6% glucose (2 minutes of centrifuging with about 1.5 cc. of the treated serum). *Chlorine in the Plasma of a Liter of Blood.*—The plasma and the washings from the 2 cc. of blood are placed in a 50-cc. volumetric flask. Add to the diluted plasma 0.4 cc. of potassium ferrocyanide solution, then 0.8 cc. of zinc acetate solution. Make up to 50 cc. and filter. Transfer an

aliquot portion (25 cc.) to an Erlenmeyer flask, add an excess (N) of $N/50$ silver nitrate solution (let N equal 5 cc.), 1 cc. of nitric acid and 5 cc. of toluene. Close the flask with a rubber stopper and shake vigorously for about half a minute, then add 1 cc. of iron alum solution and titrate with ammonium thiocyanate using a microburette. If n_1 equals the quantity of thiocyanate, the weight of plasmatic chlorine in a liter of blood will be $(N - n_1)$ 0.71 Gm. *Chlorine in the Corpuscles of a Liter of Blood.*—The corpuscles from the 2 cc. of total blood in the conical tube are transferred to a 50-cc. volumetric flask. Wash the tube carefully with water, using a drawn-out rod and add the washings to the corpuscles. Add 0.7 cc. of potassium ferrocyanide solution and 1.4 cc. of zinc acetate solution. Make up to 50 cc., filter, take an aliquot part (25 cc.) and proceed as above. If n_2 equals the quantity of ammonium thiocyanate, the quantity of chlorine in the corpuscles from 1 liter of blood will be $(N - n_2)$ 0.71 Gm. *Plasmatic Chlorine.*—Measure 1 cc. of plasma into a 50-cc. volumetric flask, dilute with about 10 cc. of distilled water and add 0.3 cc. of potassium ferrocyanide solution and 0.6 cc. of zinc acetate solution. Make up to 50 cc. and proceed as above. If n_3 equals the quantity of thiocyanate, the chlorine in 1 liter of plasma will be $(N - n_3)$ $0.71 \times 2 = (N - n_3) \times 1.42$ Gm. *Total Blood Chlorine.*—Measure 1 cc. of blood into a 50-cc. volumetric flask, dilute, add 0.5 cc. of potassium ferrocyanide solution and 1 cc. of zinc acetate solution, then proceed as above. If n_4 equals the quantity of thiocyanate, the chlorine in 1 liter of blood will be $(N - n_4)$ 1.42. Calculation of the plasmatic volume:

$$\frac{\text{Cl in plasma of 1 liter of total blood}}{\text{Cl from 1 liter of plasma}} = \frac{(N - n_1)}{2(N - n_3)}$$

Corpuscular volume:

$$1 - \frac{(N - n_1)}{2(N - n_3)} = \frac{N - 2n_3 + n_1}{2(N - n_3)}$$

Chlorine in 1 liter of corpuscles:

$$(N - n_2)0.71 \quad : \quad \frac{N - 2n_3 + n_1}{2\,(N - n_3)} \qquad \frac{1.42\,(N - n_2)N - n_2)}{N - 2n_3 + n_1}$$

The method is claimed to be rapid and precise.—C. VAILLE and P. HAUTEVILLE. *J. pharm. chim.*, 22 (1935), 61–67. (S. W. G.)

Choline. A New Vitamin. B. reported on the discovery of a new vitamin, choline, which is essential for liver function and which may play an important rôle in controlling diabetes. The best sources of choline are meat, egg yolk and yeast. Lack of this vitamin causes the condition of fatty liver in which the liver fails to make sugar, handle bile, etc. Dogs with no pancreas lived only a few months even with the injection of insulin but with the addition of minced pancreas, which contains choline, in the diet, the dogs lived for years. Diabetes is a liver-disorder rather than a disorder of the pancreas. Diabetes may be caused by injury or disease of the liver which becomes over-active; by deficiency of insulin resulting in an over-active liver; or over-active pituitary, thyroid and adrenal glands alone or in combination.—C. H. BEST, *Am. Canadian Med. Assoc. meeting* (June 1935); through *Squibb Abstract Bulletin*, 8 (1935), A-878.

Corpus Luteum Hormones—Nomenclature of. A. *et al.* have agreed to use the name progesterone for the pure progestational hormone isolated from the corpus luteum, instead of the previous names of progestin or luteosterone. The hormone exists in 2 forms and the higher melting form, m. p. 128° (uncor.) will be called α-progesterone and the other form, m. p. 121° (uncor.) will be called β-progesterone.—W. M. ALLEN, A. BUTENANDT, G. W. CORNER and K. H. SLOTTA, *Science*, 82 (1935), 153, 2120; through *Squibb Abstract Bull.*, 8 (1935), A-1368.

Cortin—Chemical Nature of. Crystals will separate from partially purified cortin treated with a small volume of water and ether. Purification gives a crystalline compound, which is oxidized to an acid without loss of carbon with ammoniacal silver. Oxidation of the acid with chromic acid results in the loss of 2 atoms of carbon and the formation of a ketone. The most probable structure is a trihydroxy aldehyde. Two hydroxyl groups are in positions α and β to the aldehyde. The compound is therefore a derivative of glyceraldehyde. With hot sodium hydroxide the aldehyde is converted into an acid with intermolecular rearrangement. Absence of a specific absorption spectrum shows the absence of the benzene ring. Distillation of the ketone with zinc dust gives a hydrocarbon of high molecular weight, apparently three 6-membered rings fully

saturated. Oxidation with silver of solutions from which the crystals separate gives products similar to the acid and ketone, except that they are more unsaturated. The specific rotation of the crystalline material is from 65° to 110°. The specific rotation of products from the mother liquor of the crystals is as high as 180°.—EDWARD C. KENDALL, HAROLD L. MASON, BERNARD F. MCKENZIE and CHARLES S. MYERS.—*Am. Soc. Biological Chemists, meeting* (April 10–13, 1935); through *J. Biol. Chem.*, 109, L (1935), No. 2; through *Squibb Abstract Bull.*, 8 (1935), A-786.

Creatinic Compounds—Colloidogenic Properties of. When a urine with high sugar content is sufficiently diluted to bring the proportion to 2 Gm. per liter, it is observed that if the dilution is made with water the reduction of Fehling's solution gives a red anhydrous oxide; if with normal urine, the precipitate is yellow hydrated oxide. The author shows that in the presence of creatinic compounds a colloid state is produced, which influences the detection of sugar.—G. DENIGES. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Estrin—Determination of Urinary. Olive oil, chloroform, ethyl acetate and benzene did not give complete recovery or comparable percentages of estrone in untreated pregnancy urine. However, extraction with these solvents after 5–10 min. boiling with 15% by volume of concentrated hydrochloric acid gave much more uniform results. The amounts of estrone found in untreated urines was called "free" estrone, and that found after boiling with acid was called "total" estrone, by the authors. Increase of acid and time of boiling did not increase the "yields." The ratios of "free" to "total" estrin in both pregnant and non-pregnant urines were not consistent, although in pregnant urines the increase of total estrone was uniform. Benzene was found to be the best solvent. The procedure for the determination of estrone in non-pregnant urines is as follows: From a 24-hr. volume of urine 200–800 cc., depending on the probable amount of estrone, is boiled for 10 min. with 15 volumes per cent concentrated hydrochloric acid. The material is extracted with benzene for 24 hours (diagram of app.). Most of the benzene can be recovered. Six cc. of olive oil are added and the benzene is evaporated off. The potency of the olive oil preparation is determined by $\frac{(24 \text{ hrs. volume} + \text{cc. extracted}) \times 6}{R}$ = r. u. in 24 hrs. volume where R is the smallest amount that will produce estrus in 50% of the test rats.—GEORGE VAN S. SMITH and O. WATKINS SMITH. *Am. J. Physiol.*, 112 (1935), 340, No. 2; through *Squibb Abstract Bull.*, 8 (1935), A-883.

Ethyl, Propyl, and Isopropyl Bromides—Micro-Detection of, in Tissues of Animals Anesthetized by These Substances. A colorimetric method of Hahn previously described (*Compt. rend.*, 197 (1933), 245; 201 (1935), 296) was used to detect ethyl, propyl and isopropyl bromides in tissues of anesthetized rats. Figures given are in thousandths of a milligram per Gm. of fresh tissue. *Ethyl Bromide.*—With rapid anesthesia, the blood contained more than the brain, and the base of the brain and cerebellum more than both hemispheres; while the reverse was true with progressive anesthesia. Respiration was stopped when the blood concentration reached (400). The right hemisphere generally fixed more (219) than the left (194). The slight affinity for the lungs was shown by the figure obtained in one case of rapid anesthesia; blood (320), brain (230), liver (120) and lungs (80). *Propyl.*—The lower concentration in the brain (170) showed it to be more active than ethyl bromide. *Isopropyl.*—This was more active than its isomer showing a concentration in the brain of (126). With both propyl compounds the concentration in the base of the brain and cerebellum was always more than that of the two hemispheres.—TIFFENEAU and BROWN. *Compt. rend.*, 201 (1935), 353. (G. W. H.)

Foodstuffs—The Physiological Aspects of Additions to. The Addition of Substances to Foodstuffs Affecting Their Nutritive Value. The change from whole meal to white flour was one of the most serious in dietary history. The addition of iodine or iodides to certain foods, the addition of concentrated forms of vitamin A and D to margarine, the irradiation of milk with ultraviolet light and the addition of viosterol or calciferol to milk are all justifiable. But the haphazard addition of copper and iron salts to milk, causing undesirable changes in the milk, and the flood of irradiated foods, are not so desirable. Sooner or later we shall have to face the position and do something to overcome the difficulties which at present make it advisable to supplement the nutritive value of our foods by artificial means.—J. C. DRUMMOND. **Substances Other Than Food Constituents Which May Be Present in Food.** Foreign substances present in food may consist of coloring matter, synthetic organic essences and flavoring materials, bleaching agents and improvers, special chemicals such as sodium nitrate in meat or magnesium chloride in processed

cheese and metallic contamination. Though the natural source cannot be controlled, in the case of fungicide sprays and material and equipment used in manufacture control is possible.—E. B. HUGHES. **Pharmacological Action of Adulterants and Impurities in Small Quantities.** A table is given for arsenic, antimony, tin, lead, zinc, copper and aluminum, showing the toxic dosage, the quantity which may be present in the foodstuffs, and quantities which have been recorded in certain cases of food poisoning by impurities. The pharmacological action of various preservatives, sulphites, benzoates, nitrates, sodium phosphate and of coloring matters is discussed.—A. ST. GEORGE HUGGETT. **Metabolic Alterations Due to Additions of Materials with Special Reference to Vitamins and Synthetic Foodstuffs.** The following factors must be considered in order that the diet may be satisfactory from the point of view of the general metabolism of the body: foods should be palatable and digestible; the energy requirements should be satisfied; the protein supply must be satisfactory; vitamins A, B_1, B_2, etc., C, D and E must be supplied; mineral salts are required; water is required. Of the substances added to our food many are beneficial, some have no influence and others may be harmful.—A. WORMALL. *J. Soc. Chem. Ind.*, 54 (1935), 744. (E. G. V.)

Fructose—a New Method for the Determination of, in Blood. *Reagents and Apparatus.*—10% zinc sulphate solution, 0.5*N* sodium hydroxide, 1% acetic acid by volume, 1% stock fructose (Merck or B. D. H. glucose-free) in a saturated solution of benzoic acid, absolute alcohol, 2.25% alcoholic sodium tauroglycocholate (B. D. H.), concentrated hydrochloric acid (B. D. H. "Analar"), Monax boiling-tubes 25 x 150 mm. *Method.*—Into a boiling-tube graduated at 40 cc., place 5 cc. of whole blood. Add about 30 cc. of water, followed by 5 cc. of 10% zinc sulphate and 5 cc. of 0.5*N* sodium hydroxide. Mix well after each addition. Dilute to 40 cc., mix and heat in a water-bath at 80–85° for five minutes, noting that the entire blood mixture is immersed. Cool thoroughly in running water and filter through No. 41 Whatman paper, stirring gently the remaining gelatinous precipitate on the filter paper to free the last few cc. of filtrate. Transfer 20 cc. of the clear filtrate to a Monax boiling-tube, add 3 drops (0.15 cc.) of 1% acetic acid and a few silica chips to prevent bumping and carefully boil down to about 1.5 cc. When the fluid decreases in volume avoid excessive heat so as not to char the solution. Prepare the standard by adding to a Monax tube 2.5 cc. of 0.01% fructose solution, and carefully evaporating to 1.5 cc. To the standard and unknown add 3 cc. of 2.25% alcoholic sodium tauroglycocholate, 7–10 cc. of absolute alcohol, a few silica chips and evaporate to dryness on a water-bath. Allow the tubes to remain in the water-bath one minute after the last drops of alcohol have disappeared to ensure complete removal of the alcohol. When cool add 10 cc. of concentrated hydrochloric acid to both tubes, mix, stopper securely and heat in a water-bath at 40° for one-half hour with occasional agitation. A reddish purple color, proportional to the fructose content, develops slowly and reaches a maximum in thirty minutes. Cool the tubes in running water, filter the colored solutions through No. 50 Whatman paper and compare in a colorimeter. *Calculation:* $\frac{\text{Standard}}{\text{Unknown}} \times$ *strength of standard* (0.25 mg.) $\times \frac{100}{2.5}$ = mg. of fructose per 100 cc. of blood. The method is sufficiently delicate to permit the estimation of fructose contents of the order of 5–20 mg. per 100 cc. of blood with an error of approximately ±1 mg.—LESLIE D. SCOTT. *Biochem. J.*, 29 (1935), 1012. (J. C. B.)

Gastric Juice—Proteolytic Ferments Contained in. Qualitative and Quantitative Determination of. The authors have attempted to use the interferometric method for the determination of the fermentative activity of the gastric juice, or fæcal matter, basing their work on the possibility of determining specifically the enzymes and measuring their hydrolytic power. In this paper their actual technic is described and the results obtained with the use of gastric juice. It is possible to determine the several enzymes present in normal gastric juice, and, in case of partial or total deficiency, this method permits diagnosis of a pathological state of the digestive function.—A. and R. SARTORY and J. MEYER. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Hormones and Their Use in Cosmetics. A review of the present state of the hormone question and of the use that can be made of this knowledge in dermatology.—R. BRUGNENS. *Parfums France*, 13 (1935), 218–226. (A. P.-C.)

Hormones of the Pituitary Gland—Purifications of the Pressor and Oxytocic. Chemistry of the Products. The authors describe an extension of their method for the separating and purifying of the oxytocic and pressor fraction of the posterior lobe of the pituitary gland. Both con-

stituents appear to be polypeptides containing tyrosine, cystine, arginine and probably other amino acids.—R. L. STEHLE and A. M. FRASER. *J. Pharmacol.*, 55 (1935), 136. (H. B. H.)

Indoxylometer—Use of, in the Laboratory. The urine is defecated with one-tenth of its volume of lead subacetate solution. Five parts of the filtered liquid is mixed with one part of a 5% thymol solution in alcohol and 10 parts hydrochloric acid containing 13 Gm. iron perchlorate per liter. Chloroform is added after 30 minutes to make 22 volumes. The color is taken up by the chloroform on shaking. Comparison with a set of colored glass disks in the indoxylometer gives an estimation of the indoxyl. Serum is deproteinized with trichloroacetic acid. The clinical significance of indoxyl is discussed.—A. GRIGAUT. *La Farmacia Moderna*, 46 (1935), 420. (A. E. M.)

Insulin, Crystalline. A quantity of insulin-hydrochloride having an ash content of only 0.02% was prepared as follows: The 1st crystallization of crystalline insulin was conducted in a phosphate buffer solution containing zinc chloride. The crystals were removed, washed and dried and then recrystallized from ammonium acetate at room temperature without acetone or the further addition of metal. The final product had an ash content of 0.7% and contained 25 units per mg. 0.5 Gm. of the crystals was placed in each of two 250-cc. centrifuge tubes and to each tube was added 10 cc. 0.1*N* hydrochloric acid. As soon as the crystals had dissolved, 200 cc. of acetone was added to each. The insulin readily precipitated. The tubes were placed in a refrigerator, for 2 hrs. The mixtures were then centrifuged, the acetone decanted and the precipitations combined by immediately dissolving them in 500 cc. water containing 2 cc. *N* hydrochloric acid. The solution was then transferred to a liter beaker and electrodialyzed, with vigorous stirring for 1.25 hrs., during which time, 25 liters acid water, p_H 2.3, circulated through the thimbles. The solution was then filtered into a 600-cc. beaker and very slowly neutralized with ammonium hydroxide until maximum precipitation was reached. The mixture was placed in the refrigerator over night. The supernatant liquid was then decanted and the precipitate centrifuged in a 250-cc. tube. The clear solutions were combined and the p_H found to be 6.2. The precipitated insulin was then dissolved in 15 cc. 0.1*N* hydrochloric acid treated with 100 cc. acetone divided into two parts and each part precipitated by 150 cc. acetone. The mixtures were then placed in the refrigerator over night. After centrifuging, the supernatant liquids were discarded and the precipitates dried *in vacuo*. The test for zinc in this insulin was faintly positive and physiological assay showed 23 units per mg. Different samples of this insulin (ash content 0.02%) were crystallized from an ammonium acetate buffer solution by means of zinc, cobalt or cadmium. Ash estimations made on different samples of the crystalline products showed constant values, for the respective metals. Further, the average ash content of each insulin salt was proportional to the atomic weight of the metal which it contained. The fact strongly indicates that crystalline insulin contains the metals as chemically combined constituents and not as impurities. The % ash values are discussed in respect to certain observations regarding the chemistry of insulin.—DAVID A. SCOTT and ALBERT M. FISHER. *Biochem. J.*, 29 (1935), 1048, No. 5; through *Squibb Abstract Bull.*, 8 (1935), A-851.

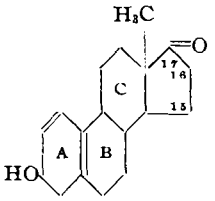
Insulin, Crystalline. From samples of insulin hydrochloride containing only 0.02% ash, crystals of zinc, cobalt and cadmium insulinate were obtained by crystallizing from ammonium acetate buffer solutions. The average ash content of each insulin salt was proportional to the atomic weight of the metal present indicating that the metals were in chemical combination and not impurities.—DAVID A. SCOTT and ALBERT M. FISHER. *Biochem. J.*, 29 (1935), 1048. (J. C. B.)

Insulin, Crystalline—Chemical Investigation of. The behavior of cystine and the free amino nitrogen of insulin under the action of various reagents which inactivate the hormone, either reversibly or irreversibly, has been investigated. The following reagents have been employed: *N*/30 sodium hydroxide, formaldehyde, benzaldehyde, *o*-chlorobenzaldehyde, acetic anhydride, methyl iodide, hydriodic acid, iodine, hydrochloric acid and methyl alcohol, diazomethane and nitrous acid. It was further found jointly with L. Hellerman (Department of Physiological Chemistry) that insulin was inactivated when treated with benzoquinone (in *M*/15 Na_2HPO_4 solution in a nitrogen atmosphere). In acid solution no inactivation occurs. Hydroquinone does not influence the activity under the same conditions. Unlike cysteine and glutathione, the thiol compounds, thiolhistidine and ergothionine do not readily inactivate insulin; thiolsalicylic acid under the same conditions renders the hormone inactive. The inactivation of insulin by the different re-

agents may be explained as the result of either destruction or modification of certain component groupings in the molecule.—H. JENSEN, E. A. EVANS, JR., W. D. PENNINGTON and ELLEN D. SCHOCK.—*Am. Soc. Biological Chemists, meeting* (April 10–13, 1935); through *J. Biol. Chem.*, 109 (1935), 44; through *Squibb Abstract Bull.*, 8 (1935), A-805.

Orthophenylphenol—Detection and Estimation of. The following procedure is suitable for estimating orthophenylphenol recovered from body eliminations, notably urine: In a 500-cc. Erlenmeyer flask containing a little water dissolve 200 mg. of orthophenylphenol by means of a few drops of 10% sodium hydroxide solution, place on a steam-bath and add 200 cc. of water, 2 Gm. of anhydrous sodium carbonate and 0.2 Gm. of sodium bicarbonate; heat for some minutes, then add 0.2*N* iodized potassium iodide solution in about 10-cc. portions, until after heating 15 to 20 minutes the reaction mixture retains a permanent yellow coloration due to excess iodine. Now add a small crystal of sodium sulphite to discharge the yellow coloration, remove from bath, allow the purplish red precipitate to settle, then decant the supernatant liquid into a tared Gooch crucible, add hot water to the flask, mix thoroughly and transfer the entire precipitate to the crucible, washing with 200 cc. of hot water. During the operation of washing the precipitate on the filter should remain somewhat moist. Dry in an oven at 100° C. to constant weight. The yield should be 0.3459 Gm. of the iodine derivative.—WILLIAM O. EMERY and HENRY C. FULLER. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 248. (E. G. V.)

Sex Hormones. II. Equilin and Equilenin. The absorption curve of equilenin, a sex hormone of mare's urine, (I) was measured in the ultraviolet. I showed 4 absorption maxima, namely, 270, 282, 325 and 340 mμ (alcohol and ether solution) indicating a certain similarity to the curve of dehydrogenated neoergosterol. A study was made of the behavior of equilin I and II toward several color reactions. I showed a yellow color in daylight and a yellow fluorescence in daylight or ultraviolet light. II exhibited a yellow color in daylight, a slightly greenish fluorescence in daylight and a very meager fluorescence in ultraviolet light. In the presence of palladium and hydrogen dehydrogenation as well as hydrogenation occurred. The latter was considerable when palladium was made to act in the absence of hydrogen. The reaction product formed a picrate, showed almost the same absorption curve in the ultraviolet as did I and seemed very similar to I in all its properties, without, however, being completely identical with it. This slight difference may be due to impurities or isomerism. It was concluded from the ready dehydrogenation of II, that the double bond must be present in one of the rings without a quaternary Me-group adjacent to the Ph-nucleus. It is probable that II has the structure



Follicular hormone, I and II do not appear to carry the CO-group on C_{16} but either on C_{15} or C_1 (the position of the side chain in sterols).—WILHELM DIRSCHERL and FRITZ HANUSCH. *J. physiol. Chem.*, 233 (1935), 13; through *Squibb Abstract Bulletin*, 8 (1935), A-802.

Urea—Colorimetric Determination of, in Blood, Spinal Fluid and Tissues. The urea is destroyed with nitrous acid and the excess of the latter estimated by the diazo-reaction with sulphanilic acid and phenol. Mix one cc. of blood or serum with water, add one cc. of a 10% solution of sodium tungstate and two drops of sulphuric acid and bring to a volume of 10 cc. Heat to 100° and filter. Mix 5 cc. of the filtrate with one cc. of a solution of sodium nitrite (2:10,000) and sufficient water to make 10 cc. Cover with a layer of liquid petrolatum. Add 30 drops of sulphuric acid, shake and heat to 65° for 25 minutes. Add one cc. of a solution containing one Gm. sulphanilic acid and 1.5 Gm. phenol in 200 cc. of 2.5 % sulphuric acid, shake, add 4 cc. of ammonia solution and compare with a standard after 5 minutes. The latter is made up by using a urea solution 1:10,000 in varying quantities, usually from 1 to 5 cc. with intermediates to obtain a complete match. The method is suitable for the determination of up to one Gm. of urea per 1000 cc. if 1 cc. is used. For higher concentration use a correspondingly smaller quantity. Amino-acids and

ammonia do not interfere with the accuracy.—JUAN A. SÁNCHEZ. *Semana méd. (Buenos Aires)*, 42 (1935), 503. (A. E. M.)

Urea—Determination of, in Blood. The method is based on the conversion of the urea to ammonia by urease, carrying over the ammonia formed by the water vapor and acidimetric titration.—G. L. PESKETT. *Brit. J. exper. Pathol.* (1934); through *J. pharm. Belg.*, 17 (1935), 671. (S. W. G.)

Urine of Cows—Œstrogenic Activity of, during Pregnancy. Since the discovery by Ascheim and Zondek in 1927 that œstrin is excreted in the urine of women during pregnancy, its presence has been detected in the urine of several other species of mammals, in some of which it may be excreted in relatively large quantities. Samples of urine were collected approximately every two weeks from three cows. Twelve samples in all were examined. Spayed mice were used throughout for biological assays. The amount of œstrin excreted in the urine of cows during pregnancy was found to be less than fifty international units per liter during the first twenty-one weeks of gestation. Œstrin can be readily detected in the urine at the twenty-third week, when the concentration is about 100 units per liter. At the thirtieth week 700 units were obtained, at the thirty-second about 9000, at the thirty-fourth 4000 and at the thirty-seventh 17,000 units per liter. The variations in the amount present in the last weeks of pregnancy are probably due to variations in the concentration of the urine.—M. M. O. BARRIE, J. B. E. PATTERSON and S. W. F. UNDERHILL. *Pharm. J.*, 135 (1935), 799. (W. B. B.)

Urine Sediments—Microscopy of. Directions for the preparation of the urine sediment and of the necessary solutions and explanation of technique are given. The sediment may be obtained in various ways: by the use of a sedimentation cone, separatory funnel, sedimentation burette, glass suction filter or centrifuge. The author recommends the use of a sedimentation pipette described by him in *Pharm. Ztg.*, No. 39 (1934). The magnification of the microphotographs shown varies from 80 to 400. Figures 2 to 5 show unorganized elements; 6 to 15 show organized elements. Figures 16 to 20 show contaminations of the sediment. The photographs show crystals of sodium urate, uric acid, calcium oxalate, ammonium and magnesium phosphates, and calcium phosphate, granulated cylinders, hyalin cylinders mixed with red blood corpuscles, and epithelial cells of various kinds.—K. BECHER. *Pharm. Ztg.*, 80 (1935), 418. (G. E. C.)

Vitamin A—Determination of, Content of Blood. The new color reaction obtained on warming vitamin A-containing material with a chloroform solution of catechol or guaiacol and antimony chloride has been employed for direct determination of vitamin A in blood. A dilute solution of potassium permanganate or the "grey solution" of the Leitz absolute colorimeter may be used as standards for comparison.—EUGENE ROSENTHAL and CATHERINE SZILARD. *Biochem. J.*, 29 (1935), 1039, No. 5; through *Squibb Abstract Bull.*, 8 (1935), A-910.

Vitamin A—Difference in Content of, in Cow and Bull Livers. The author has determined the vitamin A and the carotin (provitamin A) content of the livers of cows and bulls including calves and mature animals. He found very little reserve vitamin in calves and as far as the vitamin is concerned very little difference between the sexes. Older animals showed a much greater reserve, cows yielding 4 to 5 times the amount yielded by the bulls.—F. ENDER. *Zeitschr. f. Vitaminforschung* (1934), 247; through *Pharm. Weekblad*, 72 (1935), 607. (E. H. W.)

Vitamin B_1—Studies of Crystalline. IX. Action of Concentrated Hydrochloric Acid. Treatment of crystalline vitamin B_1 with concentrated hydrochloric acid converted the vitamin $\left[C_{12}H_{15}N_3S\begin{Bmatrix}-NH_2\\-OH\end{Bmatrix}\right]^{++}Cl_2^-$ to a compound having similar properties but having the formula $\left[C_{12}H_{15}N_3S\begin{Bmatrix}-OH\\-Cl\end{Bmatrix}\right]^{++}Cl_2^-$. Thus the $-NH_2$ group was hydrolyzed and an OH was replaced by Cl. This is presented as proof of the existence of an aliphatic hydroxyl group in the vitamin. The new product did not show physiological activity.—E. R. BUCHMAN and R. R. WILLIAMS. *J. Am. Chem. Soc.*, 57 (1935), 1751. (E. B. S.)

Vitamin E—Absorption Spectrum and Other Properties of. A band at 2940 Å. has previously been detected in the ultraviolet absorption spectra of vitamin E concentrates from wheat germ and cottonseed oils. The presence of this band in the spectrum of material prepared in an exactly similar manner from palm oil but devoid of vitamin E activity is further evidence that the band is the property of some other constituent of wheat germ and cottonseed oils, and not the

property of the vitamin. Vitamin E is destroyed by methylation with dimethyl sulphate or with methyl iodide and silver oxide. Hydrogenation over nickel at 250 to 280 atmospheres and 230° suffices neither to saturate the concentrate nor to destroy the vitamin activity. These and previous results suggest that vitamin E contains a hydroxyl group and a double bond. The double bond adds halogens easily and hydrogen with difficulty if at all.—H. S. OLCOTT. *Am. Soc. Biological Chemists, meeting* (Apr. 10–13, 1935); through *Squibb Abstr. Bull.*, 8 (1935), A-944.

Vitamin G—Preparation of Crystalline. Vitamin G can be easily prepared in crystalline form by the following method. An extract of liver is shaken with fuller's earth and the activated clay eluted with diluted sodium hydroxide. The solution is neutralized with hydrochloric acid and again adsorbed with Fuller's earth. The Fuller's earth is again eluted with acid and concentrated *in vacuo*. It is centrifuged occasionally during the concentration and when quite concentrated, the vitamin G drops out as a very fine crystalline mass. It is recrystallized a few times from hot water and dehydrated with acetone. It is a dark yellow non-hygroscopic powder. 0.1 Mg. enabled rats on a vitamin G-free diet to gain about 26 Gm., and 0.3 mg., about 30 Gm. in 9 days.—SAMUEL LEPKOVSKY, WILLIAM POPPER, JR. and HERBERT M. EVANS. *Am. Soc. Biological Chemists, meeting* (Apr. 10–13, 1935); through *J. Biol. Chem.*, 109 (1935), liv, No. 2; through *Squibb Abstract Bull.*, 8 (1935), A-870.

Vitamins, Antineuritic. In the isolation of water-soluble antineuritic vitamins from materials such as yeast, wheat germ or rice polish concentrate, impurities present are oxidized (suitably by the action of hydrogen dioxide, nitric acid, potassium permanganate or potassium dichromate) in the presence of an organic solvent for the vitamins such as alcohol-carbon tetrachloride or alcohol-chloroform and the vitamin solution is separated from precipitated impurities.—R. J. BLOCK and G. R. COWGILL (to Simon J. Dannenberg). U. S. Pat. 2,002,519 (May 28, 1935); through *Chem. Abstr.*, 29 (1935), 4524.

ANALYTICAL

Adrenaline and Ephedrine—Study of Reactions of. The author gives the following reactions. With iron perchlorate, adrenaline gives an emerald-green coloration changing to brown on addition of ammonia, and to rose on addition of an acetic solution of mercuric oxide. Ephedrine, in basic medium, gives a violet coloration with copper sulphate. Adrenaline, in basic medium, gives a rose coloration with sulphanilic acid and sodium nitrate in the cold; while ephedrine gives no reaction in the cold or on heating. With a sulphuric solution of selenous acid, adrenaline readily forms a red coloration on heating; while with ephedrine an olive-like color passing slowly to red is observed.—M. EKKERT. *Ber. ungar. pharm. gesell.* (1934), 277; through *J. pharm. chim.*, 22 (1935), 168. (S. W. G.)

Alcohol Contents of Some Preparations of B. P. 1932. Tables of some B. P. 1932 preparations containing alcohol. The figures are given together with their equivalents expressed as percentages of proof spirit.—H. D. R. MATTHEWS. *Pharm. J.*, 135 (1935), 278. (W. B. B.)

Almonds—Commercial Ground, and Their Adulteration. This paper is devoted to the detection of apricot-kernel oil in almond oil or ground apricot-kernels in ground almonds. Physical constants of the two oils are so nearly alike that no amount of one oil can be detected in the other by the use of any of the ordinary physical constants. The author of this paper has adapted the lime water emulsion test of de Negri and Fabris to this problem. This test consists in shaking the oil with lime water. Apricot-kernel oil gives a permanent emulsion while most vegetable oils remain clear. The method recommended is to shake vigorously 4 cc. of oil with 4 cc. of lime water and let stand over night. Pure almond oil separates in two or three layers while apricot-kernel oil gives a permanent emulsion. Ten to fifteen % of the latter can be detected in almond oil in this way. In some cases 4 cc. of oil and 5 cc. of lime water were used but the sensitivity of the test was somewhat reduced. A modification of the Bieber Test is also described.—G. N. GRINLING. *Analyst*, 60 (1935), 461–463. (A. H. C.)

Amidopyrine and Dinitrophenol (2,4)—Identification of. The tentative microscopic methods for identification of amidopyrine and dinitrophenol (*J. Assoc. Official Agr. Chem.*, 18 (1935), 86) were studied collaboratively and found to be satisfactory.—I. S. SHUPE. *J. Assoc. Official Agr. Chem.*, 18 (1935), 523. (G. S. W.)

Amines—New Aliphatic. Aliphatic amines are valuable for soaps, cosmetic creams, soluble oils and many other emulsions. The physical constants and solvent properties of some 12 amines

are given, including ethylene diamine, morpholine and their derivatives.—A. L. WILSON. *Ind. Eng. Chem.*, 27 (1935), 867. (E. G. V.)

Ammoniacal and Urea Nitrogen—Determination of. Ammonia is determined by distillation at 40° C. with an alcoholic solution of an alkali under reduced pressure together with aeration. The urease reaction and distillation are carried out at 40° C., because this temperature is near the optimum point for the enzyme action. Free ammonia or ammonium salts present in the sample can be liberated at this temperature by alkali without any danger of decomposing the urea. The urea is determined in such samples by difference (total ammoniacal and urea nitrogen minus that for free ammonia or ammonium salts). Complete recovery of the ammonia can be accomplished by this method in 10 to 15 minutes.—J. Y. YEE and R. O. E. DAVIS. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 259. (E. G. V.)

Arsenates—Volumetric Determination of. The general method depends upon the reduction of the arsenate to arsenite by potassium iodide in the presence of hydrochloric acid as represented by the equation:

$$As_2O_5 + 4HI = As_2O_3 + 2H_2O + 4I$$

and the final titration of the arsenite. The liberated iodine is completely removed with thiosulphate, using starch indicator. The authors used the same method replacing the hydrochloric acid with sulphuric acid, and they studied the influence of the acid concentration, the iodide concentration and the period of boiling on the reduction of the arsenates. Good results are obtained if the solutions are expressed in moles of As_2O_5 and are between 0.0165 and 0.000989 M, and 2 cc. of sulphuric acid (D. = 1.85) and 0.8 Gm. of potassium iodide are added. The mixture should be boiled for about 10 minutes.—M. F. TABOURY and H. AUDIDIER. *Bull. Soc. Chim. France* (Nov. 1934); through *J. Pharm. Belg.*, 17 (1935), 551. (S. W. G.)

Ascorbic Acid—Colorimetric Method for the Determination of. A colorimetric method has been developed for the determination of ascorbic acid which is based upon the formation of furfural from ascorbic acid when the latter is boiled with hydrochloric acid and the development of a color by the reaction of aniline with the furfural. Acetic acid is used as a buffer against excess aniline and the color is stabilized by the use of an antioxidant, stannous chloride. Ascorbic acid exists in foods in a reduced form and reversibly oxidized form. Only the reduced form of ascorbic acid gives furfural under the conditions of this procedure, but the oxidized form may be readily determined, by boiling with hydrochloric acid containing stannous chloride. A comparison of the ascorbic acid content of foods determined by this method and Tillmans' 2,5-dichlorophenol-indophenol titration procedure has been made. Good agreement between the two methods was obtained upon fresh foods, but foods that have aged showed a higher value by the colorimetric method because Tillmans' method does not measure the reversibly oxidized ascorbic acid. A study of the antiscorbutic value of reversibly oxidized ascorbic acid is being made.—JOSEPH ROE. *Am. Soc. Biol. Chem.*, 109 (1935), 75, No. 2; through *Squibb Abstract Bull.*, 8 (1935), A-832.

Ascorbic Acid—Interfering Action of Glutathione in the Silver Nitrate Test For. Glutathione interferes markedly with the silver nitrate test for ascorbic acid, which explains to a great extent the differences reported by many investigators who tried to use this reduction as a criterion for the presence of ascorbic acid in any organ or tissue extract. Cystine does not interfere to the same extent as glutathione. Adrenaline, glycogen, lactose, gelatin and starch do not interfere in the silver nitrate test with 0.15 mg. ascorbic acid. A description is given of how to use the mercuric acetate and lead acetate methods to remove the greater part of the interfering substances before applying the silver nitrate test to liver extracts. A method is also discussed for preparing gold solutions with ascorbic acid in the absence of heat.—JOSEPH L. SVIRBELY. *Biochem. J.*, 29 (1935), 1547; through *Squibb Abstract Bull.*, 8 (1935), A-1281.

Barbiturates—Alkalimetric Determination of. The following method is proposed: Dissolve about 0.2 Gm. of the sample, accurately weighed, in 30 cc. of neutralized acetone, add 4–5 drops of a 1:1000 solution of thymol blue in 95% alcohol and titrate with a methyl alcoholic solution of potassium hydroxide (about 0.1*N*) to the appearance of a definite blue (passing from yellow to green and then to blue). The alkali solution is previously titrated with 10 cc. of 0.1*N* sulphuric acid, using the same indicator. Results obtained with Veronal, Soneryl, Gardenal, Rutonal and Dial are tabulated.—C. MORIN. *J. pharm. chim.*, 22 (1935), 59–61. (S. W. G.)

Barbituric Acid Derivatives—Quantitative Analysis of. To a solution of 0.2–0.3 Gm. of the

substance to be analyzed in 20–25 cc. of acetone, or 30–35 cc. of ethyl alcohol, 15–20 cc. of a normal solution of sodium hydroxide and 20–30 cc. of water are added. This clear solution is titrated with a 0.1*N* solution of silver nitrate until a turbidity is obtained. As this method is simple and accurate to 0.1%, its use is adapted to pharmaceutical laboratories.—K. KALINOWSKI. *Pharm. J.*, 135 (1935), 231. (W. B. B.)

Benzyl Benzoate—Determination of, in Alcoholic Solutions. A method for the determination of benzyl alcohol and ethyl alcohol in the same solution was suggested. Sufficient 95% ethyl alcohol is added to a measured volume of sample so that the total amount of alcohol is about 50 cc. There is added 5 cc. of 1:1 aqueous solution of sodium hydroxide and the mixture refluxed for one hour. After cooling, 60 cc. of water is added, and the mixture distilled until about 15 cc. remains. After cooling, 50 cc. of water is added to the mixture in the distilling flask and the contents again distilled until 15 cc. remains. This procedure is again repeated. When slightly less than 200 cc. of distillate has been collected, distillation is stopped and the distillate made up to 200 cc. with water and the specific gravity (20/20) and the refractometer reading at 20° C. are determined. The values are substituted in the following equations: $5.18\,X - 7.918\,Y = 7400\,A - 7375.65$ and $5.18\,X + 1.624\,Y = B - 11.33$ where X = grams per 100 cc. of benzyl alcohol, Y = per cent by volume of ethyl alcohol, A = specific gravity (20/20) of mixture and B = refractometer reading at 20° C. The amount of benzyl benzoate is calculated from the amount of benzyl alcohol.—J. CALLAWAY, JR. *J. Assoc. Official Agr. Chem.*, 18 (1935), 535. (G. S. W.)

Capsicum—Examination of Extractives of. An investigation was carried out by the author in connection with the preparation of oleoresin of capsicum of the B. P. C. When extracted with ether, the total extractive of capsicum consisted of:

Fraction "A"	Very pungent	Soluble in ether and alcohol.....	7.8%
Fraction "B"	Non-pungent	Soluble in ether, insoluble in alcohol.....	9.8%
Fraction "C"	Non-pungent	Insoluble in ether, soluble in alcohol.....	7.3%
Fraction "D"	Non-pungent	Insoluble in ether and alcohol, soluble in water....	7.5%

H. BERRY. *Pharm. J.*, 135 (1935), 12. (W. B. B.)

Cassia Oil—Determination of Petroleum in. The method for the detection and approximate determination of light petroleum consists in steam-distilling a small quantity of the oil and collecting an oily distillate measuring 10% of the volume of the oil taken. When petroleum is present, this distillate on standing a short time, separates into two distinct layers, and the volume of the top (petroleum) layer may be read off. The identity of this layer may be confirmed by determination of its gravity and refractive index.—W. H. SIMMONS. *Perfumery Essent. Oil Record*, 26 (1935), 408. (A. C. DeD.)

Chloral—Color Reaction of, and Identification of Syrup of Chloral. The reaction suggested for tartaric acid (*Soc. pharm.* Paris, Jan. 1935) gave negative results with other organic acids and antipyrine, ethanol, ethanal, glycerol, formaldehyde, acrolein and chloroform. Chloral gave a positive reaction, even without the preliminary heating required for tartaric acid. The following test for chloral is offered: Place in a tube 2 cc. concentrated sulphuric acid (d. 1.84), 0.1 cc. of bromine-resorcin reagent (resorcin 2, potassium bromide 10, water 100, sulphuric acid 1 cc.), 0.1 cc. of the chloral solution. Heat to 100° over a small flame or in a boiling water-bath. At first a rose-yellow, then a violet and finally at 100° a deep blue coloration appears. If the chloral solution is sufficiently concentrated, a white turbidity may appear at first owing to the formation of parachloral, but this clears up at 100°. Addition of 2 cc. of water changes the blue to reddish orange. The color may be extracted by shaking with organic solvents. Addition of sodium hydroxide changes the orange color to violet and then reddish. The sodium derivative is insoluble in organic solvents. *Application to Syrup of Chloral.*—Add 5 cc. of water and 2 cc. of ether to 1 cc. of syrup. Shake, allow to separate, then transfer the ethereal layer to a tube. Evaporate the ether on a water-bath and test the residue for chloral as above.—M. PESEZ. *J. pharm. chim.*, 22 (1935), 68–69. (S. W. G.)

Chlorine, Active—Determination of, in Chlorinated Lime, Labarraque's and Dakin's Solutions. The author contends that the active chlorine content is not only the chlorine present as hypochlorite, but all the chlorine liberated when the sample is treated with an acid. If enough chloride is present, the active chlorine will be twice the amount present as hypochlorite. Corre-

sponding changes are recommended in the monographs in the Belg. Phar. CARL STAINIER. *J. pharm. Belg.*, 17 (1935), 775–777. (S. W. G.)

Chloroform Liniment and Spirit of Chloroform—Assays for. Three methods of chloroform assay were compared: 1. Proposed U. S. P. XI method reported in *J. Am. Pharm. Assoc.*, 22 (1933), 540; *S. A. B.*, 6 (1933), A-193; 2. The Warren method, *J. Am. Pharm. Assoc.*, 16 (1927), 1029; and 3. The A. O. A. C. pressure method recorded in the *Methods of Analysis, A. O. A. C.*, 3rd Ed., page 481. The results indicated the 1st method to be both cumbersome and inaccurate. The Warren method was preferred because of the accurate results and ease and rapidity of the assay. The A. O. A. C. method gave slightly more accurate results but since only 2 firms had submitted results on it and the time necessary for checking it further was not available at the time, it was recommended that the Warren method be adopted.—SUB-COMMITTEE ON ANALYTICAL ASSAY METHODS. *Am. Drug. Manufacturers Assoc., Proceedings*, (1935), 168; through *Squibb Abstract Bull.*, 8 (1935), A-1365.

Citric Acid—A New Method of Identification of. *Reagents.*—Saturated bromine water or a solution containing 0.5 Gm. of potassium bromate and 1.5 Gm. of potassium bromide in 30 cc. of distilled water, saturated solution of potassium permanganate (1 Gm. in 15 cc. of water), a 1:20 solution of the phenol employed in alcohol (90°). *Method.*—Place in a tube 0.1 cc. (2 drops) of citric solution, 0.05 cc. (1 drop) of bromine water or bromide-bromate solution, 2–3 drops of sulphuric acid and 0.05 cc. (1 drop) of saturated permanganate solution. Heat to boiling over a small flame. The liquid is decolorized and with concentrations greater than 2% a precipitate forms. Add 2 cc. of sulphuric acid and heat gently until clear. Allow to cool for a short time and add 0.1 cc. of an alcoholic solution of codeine or a phenol. If no coloration appears in several seconds place in a boiling water-bath for 1–2 minutes. The following reactions are reported: With codeine in the cold, a rose color; heated, a blue-violet. With resorcin in the cold, rose; heated, a light green fluorescence. With thymol, no color in the cold; a blood red when heated. With β-naphthol in the cold, faint green; heated, an emerald green. With salicylic acid, no color in the cold; heated, violet-red. The following preparation, depending upon the formation of glyoxal from the citric acid, gives reactions with the opium alkaloids similar to those with the Denigès methylglyoxalic reagent. Solution (1:10) of citric acid, 10 cc.; saturated bromine water, 30 cc.; sulphuric acid, 1 cc.; 4% solution of potassium permanganate. Mix, heat to decolorization of the liquid, add 5 cc. of sulphuric acid and boil for several seconds. Add sample and observe color reaction.—M. PESEZ. *J. pharm. chim.*, 22 (1935), 160–163. (S. W. G.)

Cocoa—The Determination of Total Alkaloids in, and of Cocoa-Matter in Flour Confectionery. Based upon the well-known methods of Kunze and of Wadsworth, the authors of this paper have worked out methods for the determination of total alkaloids in cocoa that are applicable in the presence of other material and when the proportion of cocoa is very small such as in chocolate cake and like confections. The essential features of the method are: (1) Extraction of the material (after admixture with magnesium oxide) with two or three portions of alcohol, 80% by volume, refluxing the mixture well to insure complete extraction of alkaloids. (2) Evaporation of the alcohol and clarification by a special zinc ferrocyanide-zinc acetate solution. (3) Filtering and extraction of the filtrate with chloroform. (4) Evaporation of the chloroform and determination to total nitrogen. Certain correction factors are necessary and a special factor for the nitrogen equivalent of the alkaloids is given. For the numerous details of the method the original paper must be consulted.—D. D. MOIR and E. HINKS. *Analyst*, 60 (1935), 439–447. (A. H. C.)

Colorimetry, Titrimetric. Colorimetry at a constant depth (10 cm.) of solution can be carried out by comparing the color intensity developed in the unknown solution with that obtained by allowing a standard solution to run from a burette into a colorimeter glass containing distilled water and the same reagents as the solution under examination. The colorimetric glasses, 15 cm. high, had a 50 cc. mark at 10 cm. from the bottom. They were placed on a glass plate mounted over a white, matt card. The formulæ for the solutions to be used are given in the paper, *e. g.*, "Determination of Lead with Sulphide." 1 cc. 10% acetic acid, 1 drop 10% sodium sulphide in glycerin, 1 Gm. ammonium chloride to intensify the color. Quantities Pb (divalent)...0.08–0.20 Gm. Disturbing elements—Cu, Zn, Hg, Bi, Cd, Ni, Co, Sn, Sb, As (trivalent), Fe (removed with sodium pyrophosphate).—P. KARSTEN and D. VAN OS. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Concentration Cell—Use of, in Quantitative Analysis. I. Estimation of Small Amounts

of Chloride in Salts. The procedure, which is described in detail along with a theoretical discussion and equations, is used in estimating traces of chlorides as impurities in other salt solutions. It consists in measuring the e. m. f. between two silver-silver chloride electrodes, one of which is dipping into a solution containing the unknown amount of chloride whereas the other dips into a solution containing the unknown amount of chloride plus a definite known amount of chloride which is added. The method is comparable in accuracy to the nephelometric procedure, and possesses the advantage that foreign salts do not cause difficulties. Traces of chloride of as low a concentration as 0.00035 Gm. of chloride per liter of solution may be measured. The procedure is also rapid.—N. H. FURMAN and G. W. LOW, Jr. *J. Am. Chem. Soc.*, 57 (1935), 1585. (E. B. S.)

Conductometric Method for Determination of Gases and Weak Acids—Limits to the Use of. The conductometric method was used for the quantitative analysis of derivatives of barbituric acid (veronal and luminal), salicylic acid, coumarin, urotropin, papaverine. The accuracy is 0.2 to 0.5%, and substances insoluble in water but soluble in alcohol may be titrated in the latter solvent. For weak acids the best results were given by dissolving them in alkalis and titrating the excess of alkali with a solution of hydrochloric acid.—K. HRYNAKOWSKI and F. MODRZEJEWSKI. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Digitalis—Assay of. Ten laboratories coöperated in assaying 2 samples of tincture of digitalis by the 1 hr., min. systolic dose, frog method and by the over night M. L. D., frog method. More uniform results were obtained than previously but were subject to several different mathematical interpretations. A tolerance of 80–130% of standard activity for both powdered and tinctured digitalis was recommended to the U. S. P. Revision Committee. It was furthermore suggested that unavoidable variations in assay be recognized in the U. S. P. monographs; that the limits of tolerance be as narrow as permissible; that factors limiting accuracy of assay be considered; and that the statement of standard strength for powdered digitalis and tincture of digitalis be stated in either one of 2 specified forms: 1. "Powdered digitalis shall be of such strength that 0.1 Gm. shall represent not less than 1.0 U. S. P. digitalis unit, and not more than 1.1 U. S. P. digitalis unit. 2. In recognition of the fact that the assay results cannot be considered accurate except between limits of 20% above and 20% below the actual potency, the product assayed shall be deemed to be within the U. S. P. limits if a product of true U. S. P. potency could have given the same assay results;" or the following qualifying paragraph beginning after No. 1: "Due to the limitations of the assay process, a product assaying 20% above or 20% below the stated limits shall be considered as complying with the required standard."—SUB-COMMITTEE ON DIGITALIS. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 292; through *Squibb Abstract Bull.*, 8 (1935), A-1370.

Digitalis Preparations—Comparative Titration of Commercial, by the Chemical Colorimetric Method and the Pigeon and Cat Methods. The method of Knudson and Dresbach was used. The solution is decolorized with lead acetate and the latter removed with disodium phosphate. The color developed with an alkaline solution of picric acid gives a means of colorimetric comparison. The results agree fairly well with those obtained by the method of Hatcher-Magnus on the cat and the pigeon method. Six out of eight commercial preparations showed deviations from 28% to 45% from the statements on the label.—ROGELIO E. CARRATALÁ. *Semana méd.* (*Buenos Aires*), 42 (1935), 154. (A. E. M.)

Ergometrine—Spectrographic Absorption of, in Relation to the B. P. Color Test. A presentation of results of spectrographic and chemical observations on specimens of ergot alkaloids prepared in accordance with Dudley and Moir's method. It was noticed that aqueous solutions of ergometrine showed a pronounced blue fluorescence when observed in daylight, and in filtered ultraviolet radiation the fluorescence was considerably more brilliant than that exhibited by ergotoxine. The blue color produced in the *p*-dimethylamino-benzaldehyde test was examined visually by means of Bellingham and Stanley's latest type spectrometer fitted with a polarizing photometer. Using the reagent proposed by Allport and Cocking, the blue colors produced by ergometrine and by ergotoxine were the same, and exhibited a maximum absorption of 550 mμ. A solution of ergotoxine ethanesulphonate was submitted to the color test described in the B. P., and it was confirmed that the blue color produced exhibited maximum absorption at 580 mμ. Ergometrine yielded a spectroscopically identical color.—N. L. ALLPORT and S. K. CREWS. *Pharm. J.*, 135 (1935), 8. (W. B. B.)

Ergot—Assay of. The proposed revisions of the U. S. P. monographs for ergot and fluidextract of ergot include the adoption of ergotoxine ethanesulphonate as a standard. Considerable

variation in results was obtained when the suitability of this standard was tested, indicating the desirability of further study. The Contact Committee last October adopted the sub-committee's recommendations: 1. Adopt ergotoxine ethanesulphonate as a standard for the interim revision; 2. Retain a minimum standard only for fluidextract of ergot; 3. Add a requirement to the assay procedure providing for elimination of cocks that are not sufficiently sensitive as to show definite differences in the amount of bluing with dose variations of 20%; 4. Use at least 7 cocks to confirm the results of an assay. The U. S. P. Interim Revision Announcement No. 3 of January 1935, altered these recommendations and left out No. 2, whereupon the Contact Committee in March asked that recommendation No. 2 be adopted and that the monograph of the U. S. P. Interim Revision becoming effective May 1, 1935, be considered tentative. Other points raised in connection with ergot assay before decision on the final form of the U. S. P. XI monographs for ergot and fluidextract of ergot are the photometric method of recording and comparing the bluing of the cock's comb, the value of standard powdered ergot as a reference standard instead of ergotoxine ethansulphonate, and the use of a modified Broom-Clark assay based upon the use of powdered ergot as the reference standard.—SUB-COMMITTEE ON ERGOT. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 297; through *Squibb Abstract Bull.*, 8 (1935), A-1373.

Ergot—Determination of, in Fluidextracts. A study made on the tentative colorimetric method (*J. Assoc. Official Agr. Chem.*, 18 (1935), 88) by eight collaborators. With two exceptions the results obtained were in good agreement.—C. K. GLYCART. *J. Assoc. Official Agr. Chem.*, 18 (1935), 540. (G. S. W.)

Ergot and Ergot Extracts—Detection of Alkaloids in. *Drng.*—Treat 0.2 Gm. of the powder in a test-tube or a small flask with 20 cc. of ether containing acetaldehyde (2–3 drops per 100 cc.) (I), 1 cc. water and 1–2 drops of ammonia (II) or sodium carbonate solution (10%) (III). Shake the mixture vigorously for 10 minutes and allow the ether layer to separate completely. Draw off carefully 10 cc. of the ether layer (= 0.1 Gm. of the drug) and shake strongly for $^1/_2$ minute with 2 cc. acetic acid (30%). After clearing separate the acid alkaloid solution and underlay carefully with 3–3.5 cc. sulphuric acid (80%). After a short time a blue-violet ring should appear at the zone of contact. If alkaloids are absent this zone is colorless or not more than pale yellow. Mix the layers and in a few minutes if alkaloids are present the mixture is dark to pale blue-violet; if absent colorless or not more than pale yellow. *Fluidextract.*—Add to 0.4 Gm. of the fluidextract 10 cc. water and 20 cc. of (I) and make alkaline with 1–2 drops of (II) or (III). Shake the mixture for some minutes vigorously, allow the ether layer to separate completely, draw off 10 cc. and proceed as above. *Extract.*—Dissolve, with trituration, 0.2 Gm. of the extract in 10 cc. water, make alkaline with 1–2 drops of (II) or (III). Shake vigorously for some minutes and treat the ether layer as above. The test will detect 0.05% alkaloids in the drug and extract and 0.025% in the fluidextract.—DRAGUTIN BARKOVIC. *Pharm. Monatshefte*, 16 (1935), 154–155. (H. M. B.)

Fats—Microchemical Analysis of. The microchemical examination of seeds is based on their coloration in presence of a suitable dyestuff, both natural and synthetic dyestuffs being very soluble in fats and oils. An alcoholic tincture of alcannine is used. Place a thin section of the seed, prepared with a razor blade, on two drops of tincture of alcannine on a microscope slide, cover with a cover glass, exert light pressure to force the oil droplets out of the fibres, and examine the color under the microscope. In carrying out the test it is important to take the properties of the oil into consideration; *e. g.*, non-drying, semi-drying and drying oils behave quite differently, especially as regards the permanence of the color. The concentration of the dyestuff depends on the nature of the oil; *e. g.*, for linseed oil use a 3.5% tincture, for oil of mustard a 33%, and for castor oil a 10% tincture.—V. GLEIZIN. *Masloboino Zhirovoé Délo*, 10 (1934), Nos. 9–10, 64–65; through *Chimie & Industrie*, 34 (1935), 641. (A. P.-C.)

Fine Powders—Influence of Particle Size, Shape, Aggregation, and Hardness in the Abrasiveness of. An apparatus is described by which the abrasion of very fine powders used in dentifrices can be quantitatively measured and compared. The abrasiveness of calcite increases regularly with the weight-average particle diameters. Aragonite needles are less abrasive than calcite spheres or rhoms of the same volume, despite the greater hardness of the former.—M. L. SMITH. *J. Soc. Chem. Ind.*, 54 (1935), 269T. (E. G. V.)

Fumigants—Determination of. I. Residual Hydrogen Cyanide in Stored Products (CaCaO, Wheat, Tobacco, etc.). The steam-distillation method for the determination of hydro-

gen cyanide in stored products is compared to the æration method, which is studied in detail and is standardized. The acidity of the solution for extracting the cyanide should lie between p_H 1.0 and 2.0. Subdivision of the material is accompanied by a decrease in cyanide recovery. The proportion of product to water and cyanide, and the rate of aeration, are the main factors determining the percentage recovery. The primary mechanism of the retention of cyanide appears to be adsorption.—O. F. LUBATTI. *J. Soc. Chem. Ind.*, 54 (1935), 275T. (E. G. V.)

Furfural Test for Mint Oils—Application of, to Other Essential Oils. A number of common essential oils have been examined by the furfural test (*Perfumery Essent. Oil Record*, 26 (1935), 247) which was used for the detection of Japanese mint oil in peppermint oils. The results obtained are tabulated.—ANON. *Perfumery Essent. Oil Record*, 26 (1935), 347. (A. C. DeD.)

Gums—Identification of, in Mixtures. A procedure for preparation of samples of gums in mixtures for testing was reported. Twenty cc. of the sample is boiled with 20 cc. of water, 2 cc. of 10% acetic acid and 3 teaspoonfuls of kieselguhr are added and the boiling continued for 2 minutes. The mixture is filtered hot, 4 volumes of 95% alcohol added to the filtrate and the mixture centrifuged. After the gum has settled the supernatant liquid is decanted off and 30 cc. of 80% alcohol added to the gum. After centrifuging the alcohol is decanted off and the gum is removed to a tared beaker with the aid of water, evaporated to dryness on the steam-bath and weighed. A 0.2% solution is made for the precipitation tests, described in *J. Assoc. Official Agr. Chem.*, 17 (1934), 468. Collaborators with one exception were able to identify Irish Moss in a mixture but all failed to identify agar.—J. H. CANNON. *J. Assoc. Official Agr. Chem.*, 18 (1935), 552. (G. S. W.)

Haddock Liver Oil—Norwegian. The constants for this oil have been determined on two samples together with the results of vitamin A assay. Also the vitamin A content of a large number of oils as shown by the antimony chloride reaction mainly, is given. These latter results vary widely from a blue value of less than 100 up to 3500.—T. THORBJARNARSON. *Analyst*, 60 (1935), 525–528. (A. H. C.)

Halides—Identification of, in the Presence of Thiocyanates. To 10 cc. of neutral solution add 1 cc. of carbon tetrachloride and 0.5 Gm. of potassium persulphate in the order named and shake. A violet color indicates the iodide ion. Shake thoroughly in a separatory funnel to remove as much of the iodine as possible, and after removing the carbon tetrachloride destroy the remainder of the iodide and the thiocyanate ions, if present, before testing for the bromide ion. This is accomplished by neutralizing the solution with 2*N* sodium carbonate and adding an additional 15 cc. of the sodium carbonate. Evaporate to dryness. Test to determine the completeness of removal of the iodide ion by dissolving the residue in 10 cc. of water, adding 1 cc. of carbon tetrachloride, acidifying with dilute nitric acid, and adding a few crystals of potassium persulphate. Warm gently. If iodine is liberated repeat the addition of the persulphate and the extraction until the carbon tetrachloride is no longer colored. When a test for an iodide is no longer obtained, add 1 cc. of carbon tetrachloride and 4 cc. of concentrated nitric acid to the cool solution contained in a casserole. The mixture must be allowed to stand with occasional agitation in order to allow the orange color of the oxidation products of the thiocyanate to disappear. If as little as 0.5 mg. of bromide ion is present a yellowish color due to bromine will remain. The color of the carbon tetrachloride solution should be determined by daylight. If less than 0.5 mg. of bromide ion is present in 10 cc. of solution, it will be necessary to run a blank and compare the color of the carbon tetrachloride used to make the extractions. The bromide ion, if present, is removed before testing for the chloride ion by adding a slight excess of potassium permanganate and boiling 1 minute. The sulphate ion formed by the decomposition of potassium persulphate must be removed by the addition of a slight excess of barium nitrate; filter and add silver nitrate. A curdy white precipitate proves the presence of the chloride ion.—G. B. HEISIG and L. K. HEISIG. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 249. (E. G. V.)

Homatropine, Hyoscyamine and Scopolamine—Identification of. Tentative microscopic methods for identification of homatropine, hyoscyamine and scopolamine (*J. Assoc. Official Agr. Chem.*, 18 (1935), 85), were studied by collaborators and found to be satisfactory.—C. K. GLYCART. *J. Assoc. Official Agr. Chem.*, 18 (1935), 521. (G. S. W.)

Hydrogen Electrode—a New Type of, for p_H Determination. The electrode is so constructed that the whole surface of the platinum is intermittently exposed to hydrogen and liquid, but in such a way that the platinum always makes good electrical contact with the liquid. The

hydrogen entering through a limb at the lower part of the vessel passes through a jet, B, and impinges on the pointed end of the wire. B opens into a small bulb to which is also connected an open tube, G, dipping into the liquid. Thus the bubbles of hydrogen draw a certain amount of liquid through G, expelling it at an upper opening F, thereby causing a slight circulation. The electrode is very economical with hydrogen. Two smaller electrodes are described for use with 5 and 1 cc. of liquid, respectively.—H. C. LOCKWOOD. *J. Soc. Chem. Ind.*, 54 (1935), 295T.

(E. G. V.)

Hydrogen-Ion Concentration—Determination of. Nine laboratories coöperated in testing the uniformity of hydrogen-ion determinations. As regards the method, a potentiometer using a glass electrode gave the most consistent results, the hydrogen electrode followed closely and the quinhydrone electrode and the colorimetric procedure were about equal. Buffered and unbuffered aqueous acid and alcoholic solutions gave the most concordant results. All 4 methods were reliable with acid solutions. The quinhydrone electrode gave widely varying results with alkaline solutions the colorimetric method fairly uniform ones but not as high as the potentiometric procedures.—SUB-COMMITTEE ON HYDROGEN-ION CONCENTRATION. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 306; through *Squibb Abstract Bull.*, 8 (1935), A-1375.

Hypophosphites—Determination of. The tentative method for determination of hypophosphites by oxidation with nitric acid and subsequent precipitation of the phosphate (*J. Assoc. Official Agr. Chem.*, 18 (1935), 87), was studied by collaborators and found to be accurate.—H. R. BOND. *J. Assoc. Official Agr. Chem.*, 18 (1935), 525. (G. S. W.)

Iodine and Potassium Iodide—Determination of, in Ointments. The following method was suggested. One gram of ointment is added on a nickel crucible containing 2 Gm. of anhydrous potassium carbonate and an additional 2 Gm. of carbonate added. The mixture is heated to a dull redness until completely carbonized. The residue is extracted with boiling water and washed on a filter with water until iodine free. The combined filtrate and washings are heated on a water-bath, aqueous potassium permanganate is added until faintly pink, alcohol is added until the color is removed and the solution cooled and made up to 100 cc. The solution is filtered, rejecting the first 25 cc. and a 50-cc. aliquot acidified with dilute sulphuric acid. Ten cc. of 15% potassium iodide is added and the solution titrated with 0.1*N* sodium thiosulphate. Each cc. of thiosulphate solution corresponds to 0.002115 Gm. of iodine.—W. F. REINDOLLAR. *J. Assoc. Official Agr. Chem.*, 18 (1935), 546. (G. S. W.)

Iodine-Iodide Solutions—Simplified Assay for, Official. Reference is made to the long use of these preparations and their recognition by the Pharmacopœia. Attention is directed to undesirable features of the assays: (1) use of two 5-cc. portions though some bottles of the Tincture of Iodine on the market do not contain 10 cc.; (2) assay for potassium iodide is a total solids determination since presence of potassium iodide sufficient to give the qualitative test would meet requirements; (3) determination of potassium iodide is time consuming; (4) sodium thiosulphate requires frequent standardization. Among other methods suggested have been determination of iodine colorimetrically or by conversion to iodide with zinc and precipitation with silver nitrate; determination of iodide argentimetrically or by difference after getting free and total iodine; and modifications of iodine cyanide method. The author suggests the following method believed to be free from objections against the official: "Five cc. of the solution is measured into an iodine flask and the free halogen is titrated with 0.1*N* alkaline potassium arsenite solution using starch indicator. Fifty cc. of concentrated hydrochloric acid and 5 cc. of chloroform are then added, the mixture is well cooled and then titrated with *M*/20 potassium iodate solution until the chloroform layer is colorless. The iodine may be calculated from the number of cc. of 0.1*N* potassium arsenite required; the potassium iodide from the difference, in cc., between the amounts of potassium iodate and potassium arsenite solutions used. The reactions may be represented by the following equations:

$$As_2O_3 + 2I_2 + 5H_2O \longrightarrow 2H_3AsO_4 + 4HI$$
$$KIO_3 + 2KI + 6HCl \longrightarrow 3KCl + 3ICl + 3H_2O$$

Experimental.—The volumetric reagents were prepared from C. P. chemicals. The arsenite and thiosulphate solutions were standardized with Baker's reagent iodine, the iodate solution with Bureau of Standards arsenic trioxide. The 0.1*N* potassium arsenite solution was made to contain a definite excess of potassium bicarbonate as recommended by Gooch to neutralize the hydriodic

acid formed." Results of a comparative study are tabulated. Slight modifications will make the suggested method applicable to Churchill's Tincture or to other similar preparations. The following advantages are claimed: it employs for the estimation of iodine, a stable solution prepared directly from arsenic trioxide—a primary standard; it substitutes volumetric for gravimetric determination of potassium iodide which takes less time, and it employs a reagent that is specific for the iodide ion.—WILLIAM F. REINDOLLAR. *J. Am. Pharm. Assoc.*, 24 (1935), 756.

(Z. M. C.)

Iodine Number—Rapid Determination of, by the Hübl Method. The determination of the Hübl iodine number can be appreciably hastened without affecting its accuracy by holding the sample in contact with the Hübl reagent for 20 min. at 60°. This is applicable to oils having iodine values of 15 to 133. The time required for the preparation of the reagent may be shortened by heating for 2 hrs. at 80° to 90° and then cooling as usual to atmospheric temperature; this produces a stable solution and dispenses with the necessity of keeping for a prolonged period at atmospheric temperature before use. For the determination of the iodine number of oleic acid by this method, the air should be first swept out of the reaction flask with carbon dioxide. In this case agreement between duplicate determinations is somewhat poorer on account of the variation of the iodine number of oleic acid.—A. KLYUCHEVICH and A. VISHNEVSKAYA. *Masloboino Zhirovoé Délo*, 10 (1934), No. 11, 50–52; through *Chimie & Industrie*, 34 (1935), 642. ((A. P.-C.)

Iodoform—Oxidation of Solutions of. The decomposition of solutions of iodoform in organic solvents is due to the combined action of light and oxygen. The authors measured the decomposition of the iodoform by titrating the liberated iodine with $N/100$ or $N/200$ thiosulphate away from direct light. The formation of hydriodic acid which catalyzed the decomposition was shown. The influence of the concentrations of iodoform, iodine and acid, an well as the action caused by the nature of the solvent was studied. The decomposition occurs in benzene solution without addition of iodine or acid, even in the dark. The presence of 1:1,000,000 of phenol, hydroquinone, aniline, pyrrol; 1:100,000 of vanillin, carbazol; 1:10,000 of pyridine, azobenzene and coniferine prevented the oxidation of a 1% solution of iodoform at 10°. The oxidation of carbon tetrachloride was also studied along similar lines.—RENÉ DUBRISAY and GUY EMSCHWILLER. *Bull. Soc. Chim. France* (Jan. 1935); through *J. pharm. Belg.*, 17 (1935), 571. (S. W. G.)

Iron and Quinine Citrate and Quinine Salts—Assay of Quinine in. The authors have found that for scale preparations, no matter which solvent is used, an assay by weight always gives a higher figure than one based on the titratable value of the quinine, when the process outlined in the B. Phar. is followed, and that the ratio between the two results is not constant, and further, that the same observation applies to quinine salts, depending upon the details of technique employed. A table gives a list of comparative assays on a number of samples of a scale preparation. It is pointed out that evaluation of quinine in iron and quinine citrate by weight differs from evaluation by titration, and this difference is due, in part, to the loss of basicity in the quinine which is effected during the manufacture of the scales. Loss of basicity in quinine may be brought about in the process of assay unless special precautions are observed. A suggestion is made to standardize the upper limit for the assay of iron and quinine citrate by weight, and the lower limit by titration.—J. S. TOAL and A. J. JONES. *Pharm. J.*, 135 (1935), 795. (W. B. B.)

Lead—Determination of. A Modification of the Fischer-Leopoldi Method. A colorimetric method based on the use of dithizone (diphenylthiocarbazone) for the microdetermination of lead is described. The lead is extracted from solution by means of a chloroform solution of dithizone, the intensity of the resultant red color of the lead dithizone compound in chloroform solution is measured, and the corresponding quantity of lead is read from a curve constructed from measurements made on known amounts of lead. The method is applicable to the estimation of lead in spray residues and biological materials and is sensitive to approximately 0.001 mg. in these materials. Slight modifications of the method permit determination of lead in the presence of the interfering elements, bismuth and stannous tin. The method has the advantage of being both extremely sensitive and relatively rapid. With spray residues as many as 36 determinations may be made in one day, and with biological materials approximately 20, depending upon the type of material and method of preparing samples for analysis.—O. B. WINTER, HELEN M. ROBINSON, FRANCES W. LAMB and E. J. MILLER. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 265. (E. G. V.)

Linoleic and Linolenic Acids—Identification of. No better method of characterization of linoleic or linolenic acids has been found than Hazura's original process of oxidation with alkaline

permanganate although the isolation of crystalline tetra- or hexa-hydroxystearic acids is poor. Of the alternative methods, the only promise has been shown in the oxidation of linoleic acid by perbenzoic acid. Together with a discussion of the hydroxystearic acids from linoleic and linolenic acids, "β"-linoleic acid, "elaidinized" "α"-linoleic acid, etc., this paper forms an essential preliminary to the further investigation of the unsaturated C_{18}-acids of butter fat described in another paper (*J.*, 29 (1935), 1564; *S. A. B.*, 8 (1935), 1284).—THOMAS G. GREEN and THOMAS P. HILDITCH. *Biochem. J.*, 29 (1935), 1552; through *Squibb Abstract Bull.*, 8 (1935), A-1293.

Luminal and Veronal—Volumetric Method for the Determination of, and Their Sodium Salts. Luminal and veronal are monobasic towards alkalies and dibasic towards silver nitrate with which they form insoluble salts. If to a solution of luminal in two equivalents of caustic soda are added two equivalents of silver nitrate, the silver salt is precipitated and the aqueous solution is neutral: $C_{12}H_{11}O_3N_2Na + NaOH + 2AgNO_3 = C_{12}H_{10}O_3N_2Ag_2 + 2NaNO_3 + H_2O$; whence the following method: Dissolve 0.2 Gm. of sodium luminal in 25 cc. of water, titrate with decinormal sulphuric acid in presence of methyl orange (7.5 to 7.8 cc.), boil to drive off carbon dioxide, add a definite amount of carbon dioxide-free caustic soda (14 to 14.5 cc. of decinormal soda for 7.5 cc. of decinormal acid), shake to complete solution, add about 25 cc. decinormal silver nitrate and 0.5 to 1 Gm. of sodium salicylate to avoid precipitation of silver oxide and titrate with decinormal caustic soda in presence of phenolphthalein.—F. VIEBÖCK and K. FUCHS. *Pharm. Ber.*, 10 (1935), No. 1, 5–6; through *Chimie & Industrie*, 34 (1935), 627. (A. P.-C)

Magnesium—Micro-Determination of. *Solutions*—(1) Dissolve 1 Gm. of 8-hydroxyquinoline in 89 cc. of absolute alcohol and add 10 cc. of ammonia (sp. gr. 0.880) and 1 cc. of hydrochloric acid (sp. gr. 1.180). Two-tenths cc. of this solution will suffice for the estimation of 100 γ of magnesium. (2) Dissolve 2.7 Gm. of potassium bromate and 25 Gm. of potassium bromide in 500 cc. of water and dilute 1:10. This solution must be standardized against the thiosulphate solution. (3) Prepare an $N/40$ sodium thiosulphate solution. This solution may be standardized against an $N/200$ potassium iodate solution. The actual normality of the thiosulphate is not determined but it is standardized in terms of length of fluid column in a graduated Conway burette, corresponding to either 1 mg. or 1 γ of magnesium. *Method.*—To 3 cc. or less of the test solution contained in a 4 cc. centrifuge tube, add a moderate excess of 8-hydroxyquinoline reagent and dilute to approximately 3.5 cc. with water. Stopper the tube with a rubber stopper fitted with a capillary tube and immerse it three-fourths of its length in a beaker (100 cc.) of water. Gradually heat the bath to 90–95° C. and maintain at this temperature for a few minutes. Cool and centrifuge for 10 minutes to separate the precipitated magnesium 8-hydroxyquinoline complex. Syphon off the supernatant liquid leaving only about 0.1 cc. Dilute to about 3.5 cc. with approximately N ammonium hydroxide agitate the sediment to free it of excess reagent and again centrifuge and remove the supernatant fluid. To the washed precipitate add a convenient volume of the bromide-bromate solution (2.0 cc. per each 100 γ of magnesium present), dilute to approximately 2.5 cc. with distilled water and add 1.0 cc. of $4N$ hydrochloric acid to hydrolyze the magnesium 8-hydroxyquinoline complex and liberate free bromine. Immediately stopper the centrifuge tube and disturb the sediment by inverting and gently tapping the tube for a moment. Allow the tube to stand at room temperature for three minutes during which time the 8-hydroxyquinoline is converted quantitatively to 5:7-dibromo-8-hydroxyquinoline. Add a crystal of potassium iodide to the solution and titrate the liberated iodine with the standard sodium thiosulphate solution using a microburette as described by Conway (1934) and a drop of starch solution as the indicator. It is important that a blank be run simultaneously. The method permits the determination of quantities of magnesium down to 20 γ in 3 cc. or less of standard solutions or solutions made from ashed residues, with an error not exceeding 10%, and in quantities of 50 γ of magnesium in 3.5 cc. the presence of four times as much calcium will not increase the error.—GEORGE CRUESS-CALLAGHAN. *Biochem. J.*, 29 (1935), 1081. (J. C. B.)

Manganese—Titrimetric Determination of. Manganese may be determined titrimetrically, using a solution of permanganate standardized with oxalic acid and without using zinc or sodium acetate. The titration is carried out in neutral solution in the presence of large quantities of sodium or potassium sulphate and the results are a few thousandths of the theoretical values. Errors of 0.8% were obtained in the presence of iron salts, and sodium chloride.—J. LEROIDE and A. BRUILTET. *Bull. soc. chim., mem.*, 2 (1935), 740, No. 5; through *Squibb Abstract Bull.*, 8 (1935), A-855.

Medicinal Charcoal—Determination of the Absorption Power of. The history of the use of charcoal medicinally is cited and the various methods for estimating charcoal which are official in pharmacopœias are briefly reviewed and commented upon. Two samples of charcoal run by the method of the Austrian Phar. showed that the chief difficulty came in recognizing the end-point. This fact made it seem more correct to determine the difference between the color remaining in the solution after treatment and a total lack of color. A color scale using various concentrations of methylene blue showed that one part in 8 million of water is sufficient to impart a noticeable color and at the same time, by a comparison of the solution after charcoal treatment with this scale, offers a means of estimating the percentage absorption of the methylene blue. The method is as follows: 100 mg. of dried charcoal is shaken for 5 minutes with 35 cc. of 0.15% methylene blue solution and filtered, the first 10 cc. being discarded. The clear filtrate must not be darker than a methylene blue solution containing 0.25 cc. to 0.50 cc. of 0.15% solution of methylene blue in 1 L. water, the comparison being made in similar tubes with equal depths of liquid. If the percentage of water in the charcoal is known, a correction may be made without drying the charcoal. By an *in vivo* test using strychnine in a dose of 5 mg./Kg. of rabbit (a dose found to be lethal), it was established that a three-fold dose of a poor charcoal was not sufficient to prevent an acute poisoning while one-third as much charcoal showing a titre 3 times as strong evidenced no trace of symptoms. What has been said about the absorption capacities of charcoal cannot be directly applied to other absorbents.—E. STARKENSTEIN and H. LANGECKER. *Scientia Pharm.*, 6 (1935), 89. (M. F. W. D.)

Mercury—Determination of, in Ointments. The tentative method for determination of mercury in mild mercurial ointments by digestion with dilute nitric acid and extraction of the ointment base with ether, followed by titration of the aqueous extract with ammonium thiocyanate. (*J. Assoc. Official Agr. Chem.*, 18 (1935), 85) was studied by collaborators and found to be accurate.—W. C. CAVETT. *J. Assoc. Official Agr. Chem.*, 18 (1935), 520. (G. S. W.)

Methyl Alcohol—Determination of, in the Air. The author of this paper reviews the literature on the harmful effects of methyl alcohol. He points out that methyl alcohol vapor can be readily removed from the air by bubbling the latter through water. In the solution thus obtained the methyl alcohol can be oxidized to formaldehyde and determined colorimetrically by the use of Schiff's reagent. He points out the necessity of having a small amount of ethyl alcohol present when the oxidation takes place otherwise the methyl alcohol is not completely oxidized to formaldehyde. After considering all factors carefully the following method for the determination of the methyl alcohol was adopted. Ten cc. of the methyl alcohol solution is measured into a 25-cc. graduated flask and 1 cc. dilute ethyl alcohol (5%), 5 cc. of potassium permanganate (1%), and 1 cc. of dilute phosphoric acid (25 cc. of 85% phosphoric acid with water to make 100 cc.), added. The oxidation is allowed to proceed at room temperature for 1 hour with occasional gentle shaking. One cc. of oxalic acid solution (5%) is then added and the flask is again shaken. When the solution has become colorless or nearly so, 2 cc. of dilute sulphuric acid (30 cc. concentrated acid with water to make 100 cc.) are added, followed by 5 cc. of Schiff's reagent and the solution mixed by inverting the flask two or three times. After standing for three hours the solution is compared in a colorimeter with a standard methyl alcohol solution prepared at the same time and in the same way. The author points out that the intensity and the character of the color varies out of proportion with the amount of formaldehyde in the solution and therefore the standard methyl alcohol solution used for comparison must be adjusted to approximately the same concentration as the unknown. Under these conditions the error is less than 5%.—C. M. JEPHCOTT. *Analyst*, 60 (1935), 588–592. (A. H. C.)

Moisture—Determination of, in Cereal Products by Distillation with Tetrachloroethane. The purpose of this investigation was principally to devise a method for determining moisture in baked cereal products of low moisture content and also unbaked materials, the results of which could be correlated with baking losses. The results of the investigation led to certain conclusions which may be summarized as follows: 1. A sufficient sample of the material must be taken to yield from 2 to 5 cc. water. 2. The sample should be so finely powdered that the distillation is complete in less than 10 minutes when carbohydrate material is present. 3. Sufficient liquid must be used to allow enough remaining in the flask after the distillation is stopped to keep the mixture liquid. Usually about 150 cc. to 30 Gm. of sample or 300 cc. to 100 Gm. of sample. 4. The distillation technique is as follows: The sample and liquid are thoroughly mixed in a suit-

able boiling-flask connected by an ordinary cork or ground-glass connection to a still-head having a wide bore at its lower end to prevent splashing at the rate of distillation required. This is connected with a vertical condenser having a burette, partly filled with tetrachloroethane, fixed at the outlet. The tetrachloroethane is distilled at the rate of 20 to 25 cc. per minute. The water collects at the top of the distillate, and the level of liquid in the burette is maintained by a suitable adjustment of the burette-tap. When almost all the moisture has been distilled, the distillate, which is milky at first, suddenly clears. The distillation is continued for about a minute longer, when the cooling water of the condenser is turned off. Heating is continued until the tip of the condenser becomes hot, when the gas is removed. The volume of water in the burette is measured, after cooling, by taking the readings of the top of the top meniscus and the bottom of the water and tetrachloroethane junction. Modifications of this technique were used and a special form of continuous still-head has been devised and is illustrated in the original paper.—J. M. TUCKER and T. E. BURKE. *Analyst*, 60 (1935), 663–667. (A. H. C.)

Morphine—Determination of, in Syrups. A collaborative study of the tentative method (*J. Assoc. Official Agr. Chem.*, 17 (1934), 446) showed fair results were obtainable. Larger amounts of solvent were recommended.—E. A. EATON. *J. Assoc. Official Agr. Chem.*, 18 (1935), 539. (G. S. W.)

Nitrates—Determination of. Various methods have been studied. The method of Ulsch which reduces the nitrate to ammonia which is removed and determined is used, except that the reduction is made with Devarda's alloy in alkaline medium. Ulsch's method gives satisfactory results if the reduction and distillation are carried out very slowly. Better results are obtained with the modified method.—R. MEURICE and P. MARTENS. *Ann. chim. anal. chim. appl.*, (Mar. 15, 1935); through *J. pharm. Belg.*, 17 (1935), 551. (S. W. G.)

Nitrites—Determination of, in Tablets. A collaborative study of the tentative method using potassium chlorate followed by silver nitrate to precipitate the chloride formed (*J. Assoc. Official Agr. Chem.*, 18 (1935), 89) indicated that the method is satisfactory.—F. C. SINTON. *J. Assoc. Official Agr. Chem.*, 18 (1935), 544. (G. S. W.)

Nitrites—Volumetric Determination of, by Means of Ceric Sulphate Solution. To the knowledge already possessed on this subject the authors of this paper have added a method for the direct titration of nitrite with ceric sulphate using erioglucine as an internal indicator and have also modified the method so that it may be used for the determination of potassium by the titration of the potassium-sodium-cobaltinitrite precipitated in the separation or detection of potassium. The ceric sulphate solution is prepared either from technical ceric oxide and sulphuric acid or from ceric ammonium nitrate by repeated evaporation with an excess of sulphuric acid. The ceric sulphate solution thus obtained is standardized against pure ferrous ammonium sulphate using the indicator mentioned above. A $N/10$, $N/50$ or $N/100$ solution is used depending upon circumstances. To the nitrite solution there was added an excess of standard ceric sulphate solution containing a small amount of $4/N$ sulphuric acid, the tip of the pipette being kept below the surface of the liquid. The excess of ceric sulphate was determined by titration with standard ferrous ammonium sulphate. Results obtained were checked against the standard permanganate titration and excellent agreement shown. In the determination of potassium the precipitated potassium-sodium-cobaltinitrite was dissolved by the aid of heat in an excess of $N/100$ ceric sulphate and acidulated with $4/N$ sulphuric acid. After cooling 5 cc., $N/50$ ferrous ammonium sulphate solution was then added and the excess of this titrated with ceric sulphate using the above-named indicator. Excellent results are reported.—H. BENNETT and H. F. HARWOOD. *Analyst*, 60 (1935), 677–680. (A. H. C.)

Nitrogen Bases—Sensitive Reaction for the Detection of. *Procedure.*—To 10 cc. of a very dilute solution of the alkaloidal base add 0.5 cc. of 15% copper sulphate and a trace of potassium cyanide (or 1–2 drops of a 1% solution). The coloration appears immediately without heating. A turbidity may form if the solution of the base is too concentrated. An excess of potassium cyanide is undesirable as it hastens the decoloration. The reaction is not specific for the cyanide ion. Morphine produces a yellow-green coloration, but the result is doubtful with concentrations below 1:15,000–1:20,000. Pyramidon produces a violet, which is definite with concentrations below 1:700,000. Apomorphine produces a red, which changes to rose, then to brown, then to gray and finally to a stable green. The color may be observed with concentrations about 1:1,200,000, and it may be removed by shaking out with organic solvents. Adrenaline produces a red colora-

tion, which is still perceptible with concentrations of 1:5,500,000.—H. WACHSMUTH. *J. pharm. Belg.*, 17 (1935), 795–798. (S. W. G.)

Oils, Medicinal and Edible—Surface Tension of. The authors find that the surface tension of the oils vary to a limited extent inversely with the degree of acidity. Washing the oil restores the normal surface tension. The surface tension at 25° for triolein and oleic acid are reported as 35.25 dynes/cm. and 34.60 dynes/cm., respectively.—E. CANALS and E. FLOUS. *J. pharm. chim.*, 22 (1935), 151–154. (S. W. G.)

Ointments, B. P. C.—Analysis of Some Complex. Methods for analyses for the following B. P. C. ointments are given: Compound Benzoic Acid Ointment, Methyl Salicylate Ointments, Resorcin Ointment and Compound Resorcin Ointment.—D. C. GARRATT. *Pharm. J.*, 135 (1935), 11. (W. B. B.)

Oleic, Linoleic and Linolenic Acids in Mixtures—Determination of. A certain number of oils contain, side by side, saturated glycerides, oleic acid esters, linoleic and linolenic acid esters. There is no method which permits exact determination of these. The author has prepared mixtures of the three acids and estimated them by different methods. Kauffmann's method gave the best results.—E. DELVAUX. *Pharm. J.*, 135 (1935), 251. (W. B. B.)

Oxidation-Reduction Indicators—I. Diphenylbenzidine Sulphonic Acid. A 0.1% solution of sodium diphenylbenzidine sulfonate in water is recommended; this can be kept for several months. Diphenylbenzidine sulfonic acid strongly resembles diphenylamine sulphonic acid in most of its properties. When small amounts are oxidized in 0.5 to 1.0 *N* sulphuric acid solution, the pale yellow becomes first deep yellow, then green and finally violet. Upon reduction, the color changes are reversed. It works perfectly in the presence of tungstate, and its color development is extremely sensitive to the catalytic effect of traces of ferrous iron, when dichromate is used as the oxidant. In such a case, back-titration with ferrous solution, after treatment with an excess of oxidant, is more reliable; the error involved by even 10 minutes contact with a reasonable excess of dichromate is negligible. For accurate microanalysis, the indicator corrections should be determined experimentally, but they are approximately equal to the volumes of indicator used, in terms of a 0.001*N* solution.—L. A. SARVER and WM. VON FISCHER. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 271. (E. G. V.)

Oxydimorphine—Determination of. Introduce 5 cc. of the sample into a 15-cc. centrifuge tube which has been treated with a boiling sulpho-chromic mixture and then rinsed with distilled water. Add 5 drops of a 5% solution of silicotungstic acid and about 0.03 Gm. of pure potassium bicarbonate. Allow the tube to stand in an ice-water mixture for at least 30 minutes. Then add 1 drop of a gelatinous barium sulphate suspension (about 10%). Mix, then centrifuge for 3 minutes at 3000 revolutions. Add another drop of the suspension and centrifuge again. Remove the limpid supernatant liquid, dry the inside of the tube and introduce a roll of dried filter paper. Cool the tube again and add 2 cc. (exactly measured) of freshly prepared aceto-sulphuric reagent. After several minutes, stir the mixture with a small rounded rod. Let the contents of the tube come to room temperature, and after 30 minutes compare the tint in a Baudouin and Besnard colorimeter or compare with results obtained under the same conditions with known solutions.—B. DREVON. *J. pharm. chim.*, 22 (1935), 97–106. (S. W. G.)

Oxygen—Examination of Therapeutic. To the methods given in the British and U. S. Pharmacopœias for the determination of the purity of oxygen, the author of this paper adds a method of his own for determining carbon monoxide. For the details of this method the original article should be consulted. The methods for determining the total oxygen content is given by these two pharmacopœias is criticized by the author and he proposes another method. This method consists in substituting for the usual pyrogallic acid solution a copper-ammonia solution as originally proposed by Hempel. A full description of the method and apparatus employed by the author is given.—ROBERT C. FREDERICK. *Analyst*, 60 (1935), 581–587. (A. H. C.)

Pancreatin—Assay of. Results of seven laboratories indicated the method of preparing washed starch described in U. S. P. X to be unnecessary. The proposed modified assay method is as follows: Determine % of water in potato starch. Mix an amount of starch equivalent to 3.75 g. dry starch with 10 cc. cold distilled water. Add suspension with stirring to 75 cc. 50–60° water, wash the remainder in with 10 cc. water and boil for 5 minutes with good stirring. Make up to 100 cc. with water and cool to and maintain at water-bath temperature of 40°. Add a suspension of 0.15 Gm. pancreatin in 5 cc. water to starch paste, pouring back and forth for at least 30 sec.,

maintain at 40° for 5 minutes and add 0.1 cc. to a mixture of 0.2 cc. 0.1*N* iodine and 60 cc. distilled water at about 23–25°. No blue, red or violet color is produced.—SUB-COMMITTEE ON DIGESTIVE FERMENTS. *Am. Drug Manufacturers Assoc., Proceedings,* (1935), 180; through *Squibb Abstract Bull.*, 8 (1935), A-1380.

Phosphorus—Colorimetric Methods for the Determination of. The methods are applicable in the presence of silicates, arsenates, nitrates and ferric iron. The first method using molybdenum blue reagent (available from chemical houses) follows: Take 0.5 to 15 cc. of the standard or unknown solution (containing 0.01 to 0.3 mg. of phosphorus pentoxide) in a 50-cc. volumetric flask (with a mark at 30 cc.); add 5 drops of α-dinitrophenol (saturated aqueous solution) and neutralize drop by drop with 2% sodium bicarbonate when acid, or with 1*N* sulphuric acid when alkaline, to a very faint yellow; then add separately 5 cc. of 1*N* sulphuric acid and 5 cc. of 8% sodium bisulphite; make up to about 30 cc. with distilled water and shake well; allow to stand over night or heat on a steam-bath for at least 1 hour; add 5 cc. of ten times diluted molybdenum blue reagent and continue to heat on the steam-bath at least 30 minutes more (the velocity of color development is a function of the temperature: At 20° to 30° C. the maximum color is attained in 3 days; at 50° C. in 10 hours; at 70° C. in 3 hours; at 95° to 100° C. in 30 minutes; and by direct boiling 4 to 5 minutes); cool to room temperature; make up to 50 cc. with distilled water; shake thoroughly and measure the color in an ordinary or photoelectric colorimeter. The blue color of the solutions remains stable for 2 or 3 days, provided they are stored in the dark in well stoppered flasks. If the reagents as prepared are pure, no blue color will appear in the phosphorus-free check; it remains slightly yellowish. A faster method follows: Prepare the aliquot for analysis as in the molybdenum blue reagent method up to the addition of the molybdenum blue reagent. Cool to room temperature, add to each flask 5 cc. of the molybdenum trioxide reagent and mix well; prepare standard and unknown solutions exactly in this manner; then add rapidly in turn to each flask, while shaking, 5 cc. of fresh stannous chloride solution (add 0.16 Gm. of stannous chloride to 200 cc. of 1% gum arabic solution); make up to 50 cc. with distilled water and shake thoroughly; after 20 minutes make comparisons. The determinations should not stand longer than 6 hours; after this time the solution becomes slightly turbid. Standards and unknowns must be prepared rapidly and simultaneously. Other precautions are the same as for the molybdenum blue method. The detection of phosphorus by a drop method may be carried out as follows: To a few drops of the unknown solution in a small test-tube add 1 drop of 1*N* sulphuric acid and 1 drop of 8% sodium bisulphite, and boil gently in an open flame for about one-half minute. Add to the boiling solution one drop of the molybdenum blue reagent diluted 10 times and boil another half minute, or cool it and add one drop of molybdenum oxide reagent and one drop of stannous chloride solution. In this way it is possible to get a preliminary estimate of the approximate concentration of phosphorus in solution.—CH. ZINZADZE. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 227. (E. G. V.)

Pilocarpine and Its Salts—Determination of. The present methods are reviewed. The recommended procedure depends upon the reaction of sodium hydroxide with the alkaloid (an inner ester) forming the sodium salt of pilocarpinic acid. *Method.*—Dissolve at least 0.05 Gm. of pilocarpine in a mixture of 2 volumes of 96% alcohol and 1 volume of distilled water, add exactly 10 cc. of 0.1*N* sodium hydroxide and 3 drops of phenolphthalein. Heat the mixture in a long-necked flask for 5 minutes in a boiling water-bath. To the cooled mixture add 5 cc. of alcohol and titrate the excess alkali with 0.1*N* sulphuric acid. The same method may be applied to the salts of pilocarpine after neutralizing the acid in the salt with alkali. This also allows a check on the purity of the salt; since the same quantity of alkali should be required to neutralize the saline acidity as is needed to saponify the inner ester. 1 cc. 0.1*N* sodium hydroxide = 0.0208 Gm. of pilocarpine.—JUAN-A. SANCHEZ. *Ann. chim. anal. chim. appl.*, (Apr. 15, 1935); through *J. pharm. Belg.*, 17 (1935), 552. (S. W. G.)

Podophyllin—Solubility of. On the basis of solubility tests made by 7 laboratories on five different samples of podophyllum resin from four different sources, it is recommended that the next edition of the U. S. P. require that the resin be soluble in chloroform to the extent of not less than 60% and in ether to the extent of not less than 65%. The quantity of solvents used should be considerably increased above that now required in the U. S. P. and a longer time for complete solubility set. More than 3 successive 5 cc. portions of solvent should be employed in washing the insoluble residue.—SUB-COMMITTEE ON ALKALOID AND DRUG STANDARDS. *Am. Drug*

Manufacturers Assoc., Proceedings, (1935), 163; through *Squibb Abstract Bull.*, 8 (1935), A-1384.

Potassium—Determination of Small Amounts of, by Means of Sodium Cobaltinitrite. Experimental investigation of the precipitation of potassium as sodium-potassium cobaltinitrite indicates that the precipitate obtained varies in composition with respect to potassium content, depending upon the conditions of precipitation. Consistent results may be obtained by using recrystallized reagent.—H. W. LOHSE. *Ind. Eng. Chem., Anal. Ed.*, 7 (1935), 272. (E. G. V.)

Quinidine Thiocyanate. Gravimetric and Volumetric Determination of Quinidine. *Gravimetric.*—Concentrate the quinidine salt solution on a water-bath to about 20 cc., transfer the hot solution to a precipitation flask. If the solution is acid to litmus, neutralize with sodium hydroxide. Add 3 Gm. of potassium thiocyanate, mix carefully, test the reaction and bring back to neutral if necessary. Allow to cool, shaking from time to time. After 2 hours, collect the precipitate on a tared double filter, using the filtrate to aid in removal of the precipitate from the flask. Wash twice with 2 cc. of distilled water and then with 16 cc. of distilled water saturated with quinidine thiocyanate, using 2-cc. portions. Dry in an oven at 105–110° to constant weight. If p is the weight found, the quantity of quinidine in the solution is 0.846 p. *Volumetric.*—Concentrate the aqueous solution of quinidine nitrate or basic sulphate to about 20 cc. and transfer to a flask marked at 50 cc. Add 20 cc. of $N/10$ potassium thiocyanate, see that the solution is neutral to litmus, then make up to the mark with distilled water. After shaking 4 or 5 times during 20 minutes, filter through paper, reject the first part of the filtrate and take a 25-cc. aliquot. Titrate the excess thiocyanate by the Charpentier-Volhard method after adding 10 cc. of $N/10$ silver nitrate. If n is the number of cc. of $N/10$ potassium thiocyanate added in the final titration, 32.4 mg. $\times$ 2 $\times$ n represents the amount of quinidine in the sample.—R. MONNET. *J. pharm. chim.*, 22 (1935), 112–119. (S. W. G.)

Quinine Suppositories—Analysis of. This paper discusses the analysis of quinine suppositories. Methods for the detection of carnauba wax, paraffin and other adulterants of cacao butter are discussed. A direct gravimetric determination of quinine in suppositories as well as a volumetric method for this determination are considered, tables of data and directions for procedure being given.—J. TAVERNIER. *Pharm. Tijdschr. Nederland.-Indie*, 13 (1935), 61. (E. H. W.)

Raman Effect—Analytical Applications of. An explanation of the nature of the Raman effect, of the technique of its spectrographic investigation, of its application to the identification of pure compounds, to the qualitative study of mixtures (and more particularly of mixtures of isomers) and of the possibility of using it as a quantitative analytical method.—LÉON PIAUX. *Chimie & Industrie*, 34 (1935), 507–516. (A. P.-C.)

Resins—Behavior of Certain, with Respect to Solvents. The author studied the properties of solutions of copal, dammar, mastic, rosin, resin of guaiac, resin of jalap and resin of podophyllum in ether, alcohol, benzin, benzol, chloroform, carbon tetrachloride and carbon disulphide. Tables of specific gravities and concentrations are given. The author concludes: (1) that the specific gravities of the resin solutions decline with liquids heavier than water and rise with liquids having a density less than one; (2) that this change in specific gravity does not depend on the amount of dissolved resin as much as upon the nature of the resin; (3) that there is no direct relation between solubility and influence of specific gravity on the one hand and absorptive ability of the resins on the other; (4) that in certain preparations, *e. g.*, adhesive solution of mastic, the specific gravity offers a means of control; and (5) that the various solvents serve, in the purification of the resins mentioned from a standpoint of the solubility or insolubility of the resins in them.—L. M. VAN BERG. *Pharm. Weekblad*, 72 (1935), 1027. (E. H. W.)

Rhubarb—Evaluation of. The article represents a criticism of the method adopted by the Swiss Phar. V for the estimation of rhubarb. The method consists essentially of treating a weighed sample of powdered rhubarb with 0.1N sodium hydroxide, heating, diluting and back-titrating the excess of alkali with 0.1N hydrochloric acid, the results being calculated as chrysophanic acid. The assay depends on the principle that the free and the split oxyanthracene derivatives are easily titrated as dibasic acids without the use of an indicator by virtue of their own color change. Such, however, is not the case. A sample of pure chrysophanol could not be easily dissolved in 0.1N unless heated for some time, and the end-point of the titration was not sharp. The conclusion drawn is that the method for the determination of rhubarb as adopted by the Swiss

Phar. V is based on false assumptions and is totally unnecessary.—L. ROSENTHALER. *Scientia Pharm.*, 6 (1935), 93. (M. F. W. D.)

Santalols in Sandalwood Oils. Determination of Free Alcohols. The method used by the authors is as follows: Heat 0.5 to 3 Gm. of the substance with 5 cc. (accurately measured) of an acetylant mixture consisting of 1 part of acetic anhydride with 2 parts of anhydrous pyridine (previously refluxed with baryta and distilled) in a flask of rather more than 100 cc. capacity fitted with a reflux air-condenser. Immerse in a boiling water-bath for $^1/_2$ to 1 hr., $^1/_2$ hr. sufficing in most cases. Then run in 50 cc. water through the condenser and continue heating for 15 min., shaking from time to time, in order to decompose excess acetic anhydride. Cool, transfer to a 250-cc. beaker, wash out condenser and flask and add the washings, and titrate with $N/2$ alcoholic potash using phenolphthalein as indicator (= n cc.); the end-point is very sharp. A blank determination is carried out in identical conditions (= N cc.). Allowance must be made for the acidity of the substance under test, the acid value (AV) being previously determined. Then if p grams of sample were taken, the percentage of alcohol of molecular weight M present is $\frac{\left(N-n+\frac{p \times AV)}{28}\right) M}{20p}$. In these conditions primary alcohols, primary amines and phenols are acetylated quantitatively in $^1/_2$ to 1 hr., secondary alcohols almost quantitatively in 1 hr., while tertiary alcohols and aldehydes scarcely react at all. The method can therefore be used in place of phthalization for the determination of free primary and secondary alcohols in the presence of tertiary alcohols and aldehydes in essential oils, and requires only 1 to 2 hrs. in place of 18 hrs. Some of the results obtained with various substances and essential oils are given. The authors find this method rapid and satisfactory for free primary and secondary alcohols comparable with those of phthalic esterification. This method applied to sandalwood oils gives results below those of the classical method of acetylation.—DELABY, SABETAY and BREUGNOT. *Perfumery Essent. Oil Record*, 26 (1935), 334. (A. C. DeD.)

Santonin—Determination of, in Mixtures and in Santonica. The tentative method for determination of santonin in mixtures by precipitation with dinitrophenylhydrazine (*J. Assoc. Official Agr. Chem.*, 18 (1935), 87) was found by collaborators to give accurate results. An attempt made to modify the method for determination of santonin in santonica was not entirely satisfactory.—H. J. FISHER. *J. Assoc. Official Agr. Chem.*, 18 (1935), 526. (G. S. W.)

Silver-Silver Bromide Electrode Suitable for Measurements in Very Dilute Solutions. The preparation and use of the electrode are given. Measurements of the cell H_2 (1 atm.)/$HBr_{(m)}$ /AgBr/Ag have been made at concentrations from 0.0001 to 0.02*M* in quartz and Pyrex glass cells of very simple design. The value of the molal electrode potential of the electrode was found to be 0.0711 volt.—ALBERT S. KESTON. *J. Am. Chem. Soc.*, 57 (1935), 1671. (E. B. S.)

Strychnine—Separation of, from Cinchona Alkaloids. The authors have evolved a method for detecting minute traces of nux vomica alkaloids in the presence of large quantities of cinchona alkaloids and numerous other herbaceous ingredients. Briefly, the method is as follows: The combined alkaloids are obtained and purified in the usual manner by extraction from alkaline solution, then by acid and in turn again from alkaline solution. This purified residue is obtained in final form in a 100-cc. pyrex beaker and brought to dryness on the steam-bath. To the residue is added 3 cc. of distilled water, the mixture is heated over a small opening in steam-bath with constant stirring with glass rod and for at least 10 minutes, replacing with a small amount of additional water that which has evaporated. This heating will soften the alkaloidal residue and allow the water to take up the strychnine and just so much of the cinchona alkaloids. In this manner the ratio of strychnine to cinchona alkaloids is boosted to nearly equality of each. Cool and filter through a very small paper filter placed in a 1-inch glass funnel, having the filter paper when folded cut down to a length of not over one-half inch. When inserted in funnel, wet with water and pour through this the alkaloidal solution allowing the filtrate to go into a 50-cc. pyrex beaker. Wash filter paper with 2 cc. of distilled water and evaporate the solution to dryness in the steam-bath by inserting into opening of the bath using beaker tongs. After evaporation the residue will usually be pure white and crystalline. If any strychnine is present a small amount of this crystalline material will readily show up by any of the principal color tests for strychnine. It was found that the best qualitative test is still that of rubbing up a small amount of the above residue with concentrated sulphuric acid and adding and dragging several small crystals of potassium dichro-

mate through the acid by aid of a small stirring rod.—G. E. MALLORY and PETER VALAER. *Am. J. Pharm.*, 107 (1935), 349. (R. R. F.)

Sulphur—Micro-Volumetric Determination of, in Organic Compounds Containing Halogen and Nitrogen. Further improvements on the Cowie and Gibson method as modified by Viebock are described and much data are given to show the accuracy of the method.—D. T. GIBSON and T. H. CAULFIELD. *Analyst*, 60 (1935), 522–525. (A. H. C.)

Tetrachlorethylene—Determination of. The tentative method for determination of tetrachlorethylene by treatment with metallic sodium followed by acidification with nitric acid and precipitation of silver chloride (*J. Assoc. Official Agr. Chem.*, 18 (1935), 84), was studied by collaborators and found to be accurate.—G. M. JOHNSON. *J. Assoc. Official Agr. Chem.*, 18 (1935), 519. (G. S. W.)

Thyroid—Determination of Iodine in. A method was recommended for incorporation in U. S. P. XI which agrees in principle with the present official method but contains changes which aim to clarify the directions and eliminate uncertainties, *e. g.*, using fusion mixture of carbonates of sodium and potassium with potassium nitrate; heating crucible to a dull red in about 10 minutes and maintaining thus for 10 minutes; leaching out fused flux with hot distilled water; oxidizing iodine to iodic acid with sodium hypochlorite solution at 15–20° and acidifying sufficiently to neutralize the alkaline solution and give a proper value for the final titration; boiling solution for no less than 1.5 hours; adding enough pure arrowroot starch solution to obtain a sharp and true endpoint; control testing with each set of determinations; etc.—GEORGE D. BEAL and CHESTER R. SZALKOWSKI. *American Drug Manufacturers Assoc., Proceedings*, (1935), 208; through *Squibb Abstract Bull.*, 8 (1935), A–1392.

Thyroid—Standardization and Dosage of. Some confusion has arisen as a result of the various attempts which have been made to compare the dose of Thyroideum Siccum, with those hitherto prescribed. Data are given in a table in which batches of ox thyroid glands are referred to, both fresh and frozen, processed on a manufacturing scale, the batch size varying between 250 and 750 lb. of wet glands. It is suggested that the table embodies all the information required to make a comparison between the more common official and non-official thyroid preparations. The moisture contents of the thyroid preparations in the table seem to be fairly constant, only three samples showing a variation of the order of 100%, due in part, to the degree of trimming. The percentage of the total iodine of the dried, defatted gland varies very considerably, from 0.559% to 0.134%—a variation of approximately 300%. An equally wide variation is also found in the figures for percentage of thyroxine iodine, the limits in this case being 0.200% and 0.51%. Results of analyses for thyroxine iodine are shown in a second table, a comparison being made between the direct thyroxine iodine method and the B. Phar. method.—R. F. CORRAN. *Pharm. J.*, 135 (1935), 781. (W. B. B.)

Thyroid Assay—Study of the U. S. P. Difference in interpretation in a case in Federal Court has been indirectly responsible for a critical study of determination of iodine content. Search for sources of error in the method and better directions in order to avoid pitfalls constituted the task. The method recommended for U. S. P. XI agrees with U. S. P. X in principle but directions are clarified: "*Procedure.*—Thoroughly mix 1 Gm. of thyroid, finely powdered and accurately weighed, with 15 Gm. of an intimate mixture of 138 parts by weight of anhydrous potassium carbonate, 106 parts of anhydrous sodium carbonate and 75 parts of powdered potassium nitrate in a nickel crucible of about 125 cc. capacity, and spread an additional 5 Gm. of this mixture evenly over the surface. Heat the crucible with the flame of a Bunsen burner at such a rate as to attain a dull red color in ten minutes, and continue the heating at the same temperature for an additional ten-minute period. At the end of this time the carbonaceous material is completely oxidized and the mixture has just begun to melt around the wall of the crucible. Cool the fusion mixture and place the crucible and contents in a 400-cc. beaker. Add 150 cc. of hot distilled water and stir until the contents of the crucible are completely dissolved. Transfer the solution to a 500-cc. Erlenmeyer flask and rinse the beaker and crucible with four 10-cc. portions of hot water adding the rinsings to the solution in the flask. Cool the solution to 15° C. and add 50 cc. of freshly prepared chlorinated soda T.S. Cautiously add 60 cc. of diluted phosphoric acid (made by mixing equal volumes of phosphoric acid and distilled water), place the flask on a hot plate and boil the solution until a strip of filter paper moistened with starch-potassium iodide T.S. does not become blue when held in the vapor in the mouth of the flask. The final volume of solution in the flask

must be about 175 cc., and distilled water must be added, if necessary, during the boiling to maintain this volume. Cool the solution to about 25° C. and add 10 cc. of a freshly prepared aqueous solution of potassium iodide (1 in 100). Titrate the liberated iodine with *N*/200 sodium thiosulphate, add 3 cc. of starch T.S. as indicator shortly before the end of the titration. Conduct a blank determination with the same quantities of the same reagents and subtract the volume of sodium thiosulphate consumed from that consumed by the thyroid. Each cc. of the corrected volume of *N*/200 sodium thiosulphate is equivalent to 0.0001058 Gm. of iodine." Compressed thyroid can be assayed by the same method with a few modifications. The procedure is given though it is not included in U. S. P. XI: "*Procedure.*—Select 50 perfect tablets, or enough more than this number to make at least 3.5 Gm. of sample, weigh accurately and calculate the average weight per tablet. Finely powder the tablets in a clean, dry mortar. Mix 1.5 Gm. of the powdered tablets, accurately weighed, with 15 Gm. of an intimate mixture of 138 parts by weight of anhydrous potassium carbonate, 106 parts of anhydrous sodium carbonate and 75 parts of powdered potassium nitrate in a nickel crucible of about 125 cc. capacity, and spread an additional 5 Gm. of this mixture evenly over the surface. Heat the crucible with the flame of a Bunsen burner at such a rate as to attain a dull red color in ten minutes, and continue the heating at the same temperature for an additional fifteen-minute period. Proceed as directed under the assay of thyroid, beginning with 'Cool the fusion mixture.' One cc. of *N*/200 sodium thiosulphate is equivalent to 0.0001058 Gm. of iodine. Divide the weight of iodine found by the number of tablets represented by the weight of sample taken to find the weight of iodine per tablet." Results of sixteen assays are tabulated.—GEORGE D. BEAL and CHESTER R. SZALKOWSKI. *J. Am. Pharm. Assoc.*, 24 (1935), 742. (Z. M. C.)

Thyroid Tablets—Assay of. A considerable difference of opinion was expressed by 8 laboratories relative to the applicability of the U. S. P. method *per se* to the assaying of thyroid tablets. In view of the fact that thyroid tablets are to be included in U. S. P. XI, it is recommended that a statement of assay for all thyroid tablets be temporarily omitted from the U. S. P. for at least a year or until adequate studies can be made. If the present method is extended to tablets, then only plain tablets should be included in the U. S. P. and sufficiently wide tolerances be given to allow for variation dependent on assays. All coated tablets should be excluded from the U. S. P. until adequate data on assays of them are available.—SUB-COMMITTEE ON DIGESTIVE FERMENTS AND GLANDULAR PRODUCTS. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 190; through *Squibb Abstract Bull.*, 8 (1935), A-1392.

Tinctures—Modern Viewpoints and Methods for the Examination of. A review with 34 references.—A. MAYRHOFER. *Pharm. Monatsh.*, 16 (1935), 109–118. (H. M. B.)

Trypsin—Smith-Sorensen Method of Assay of. It could not be concluded from the examination of 2 samples that the Smith-Sorensen method is to be preferred to the present U. S. P. method and its recognized lack of preciseness; the reported potency of the samples showed too great a divergence.—SUB-COMMITTEE ON DIGESTIVE FERMENTS AND GLANDULAR PRODUCTS. *Am. Drug Manufacturers Assoc., Proceedings*, (1935), 184; through *Squibb Abstract Bull.*, 8 (1935), A-1392.

Turtle Oil—Physical and Chemical Characteristics of. Because of the growing use of turtle oil in cosmetic preparations such as skin foods and the like, a more thorough knowledge of the characteristics of a typical turtle oil is desirable The greater portion of these oils come either from West Africa or Egypt and are free from odor, are semisolid, and of a light yellow to golden-yellow color. A sample of refined West African oil was prepared from four separate pounds of the commercial article and was found to have the following characteristics: Melting point (incipient fusion) 24.6° C., melting point (final fusion) 25.6° C., solidifying-point 22.5° C., titre 25.5° C., sp. gr. at 40°/40° C. 0.9112, refractive index, n_D^{40} 1.4599, saponification value 209, unsaponifiable matter (S. P. A. method) 0.6%, iodine value of the unsaponifiable matter (Bolton and Williams method) 92.5, iodine value 64.6, acid value 2.0, percentage of insoluble bromides on the free acids (Gemmel's method) 5.0, melting point of the bromides darken above 200° C., Reichert-Meissl value 0.2, Polenske value 1.7, Kirschner value 0.06, acetyl value 3.5. Since the oil was said to possess a high vitamin activity an examination of its ultraviolet absorption spectrum was made. From the results obtained insignificant amounts of vitamin A, ergosterol or other pro-vitamin D are present, and the absence of vitamin E is indicated.—WALTER LEE. *Analyst*, 60 (1935), 650–653.

(A. H. C.)

Vegetable Oils—Color and Spectral Transmittance of. The spectral transmittance and colorimetric properties of 125 vegetable oils including 111 cottonseed, 3 peanut, 5 soybean, 2 sesame, 1 rape, 1 corn and 2 olive oils, are given in tabular and graphical form. The oils in a $5^1/_4$ in. thickness, may be classified into 2 general spectral types, one containing principally green materials from chlorophyll, the other containing several yellow or amber-colored materials of unknown identity. The spread of lightness over the group of vegetable oils is greater than can be conveniently matched in the present method of color grading. An improved color comparator is desirable, providing a more suitable arrangement for equalizing brightnesses of the sample and comparison fields. The independent variation in relative and abstract concentrations of the several pigments present in the oils, makes it impossible to represent all oils accurately on any one-dimensional chromaticity scale. For accurate color grading in the Lovibond system, this situation demands the introduction into trade practice of a two-dimensional scale established by the independent variation of both yellow and red. It is shown that the best yellow numeral varies approximately from 20 for very light cottonseed oils to 80 or above for the prime yellow oils. An improved one-dimensional Lovibond scale would be provided by the fixation of yellow in some definite relation to red. Some abridged color grading methods are discussed.—HARRY J. MCNICHOLAS. *J. Res. Natl. Bur. Standards*, 15 (1935), 99; through *Squibb Abstract Bull.*, 8 (1935), A-1302.

Veronal—Microscopic Determination of, by Means of Ammoniacal Silver Nitrate. Veronal, when warmed with the reagent, gives a microscopic octahedral crystalline precipitate; while dial forms crystals which appear in the form of needles deposited in masses or in a double fan. It is possible to obtain these crystals with 0.0005 Gm. of veronal.—C. STRYZYZOWKI and L. DÉVERIN. *Chim. Acta*, 16 (1933), 1288; through *J. pharm. Belg.*, 17 (1935), 609. (S. W. G.)

Vitamin A—Assay of. A series of 13 halibut liver oils were assayed for vitamin A spectrographically using the Hilger vitameter A. There was some divergence in the values obtained by two groups of laboratories although agreement was fairly good within each group. It was proposed to test the value of the vitameter for the vitamin A of cod liver oil and cod liver oil concentrates of lesser potency than halibut liver oils. It should then be possible to correlate these data with the physical measurements of the U. S. P. reference cod liver oil and thus predict fairly closely the vitamin A potency in terms of biological units. Discussion brought out the fact that whereas earlier makes of the vitameter did not give very accurate results the present model has been greatly improved and will undergo further changes as required. Certain details of operation which are essential for the success of assay with the vitameter were described. The Report was referred to the Executive Committee.—SUB-COMMITTEE ON VITAMIN ASSAYS. *Am. Drug. Manufacturers Assoc., Proceedings*, (1935), 262; through *Squibb Abstract Bull.*, 8 (1935), A-1396.

Vitamin B—Potentiometric Titration of Solutions of. Potentiometric titrations were made on solutions of crystalline vitamin B_1-2HCl. There was a marked difference in behavior of the solutions with a hydrogen and a glass electrode. It seems likely that the vitamin is reduced with the former so that the results with the latter are more reliable; they are also more reproducible. The vitamin does not poison the hydrogen electrode nor does it appear to be strongly adsorbed. The vitamin contains 2 weakly basic groups of pk 3.4 and 4.8, and a pseudo-acid group at p_H 9. The dissociation constant of the acid form of the vitamin has the value pk 6.8 in water at 0°.—RICHARD C. G. MOGGRIDGE and ALEXANDER G. OGSTON. *Biochem J.*, 29 (1935); through *Squibb Abstract Bull.*, 8 (1935), A-824.

Vitamin C—Assay of. It has been said that trichloroacetic acid (I) which is ordinarily used to extract ascorbic acid (II) from tissues prior to its determination by 2,6-dichlorophenolindophenol, (III) stabilizes II. F. & I. have observed, however, that II is readily oxidized by I, whether in the pure II solution or in the deproteinized tissue filtrate, and that it should therefore be replaced by metaphosphoric acid (IV) in whose presence II is very stable. I is furthermore undesirable because it exerts an appreciable decolorizing effect on III, with resulting error in the determination; IV has no such effect. To illustrate, the iodine consumption of II in I decreases with the concentration of I and the temperature; iodine consumption in 2% IV on the other hand, remains practically unchanged after five hours. The oxygen consumption in I is considerably greater than that in distilled water; in IV there is practically none. One cc. 5–10% I decolorizes III in 5.5–7.5 min., whereas 1–5 cc. 2% IV decolorizes III in 30 to more than 60 min. Experimental errors with Tillmans' procedure of vitamin C determination range from 20–50%, whereas those with Harris and Ray's method in which titration is carried out as rapidly as possible and the

total volumes of the added acid vitamin solution are smaller than those of the III, are about 5%.—AKIJI FUJITA and DANZO IWATAKE. *Biochem. Z.*, 277 (1935), 293; through *Squibb Abstract Bull.*, 8 (1935), A-825.

Vitamin C—Colorimetric Determination of. A simple quantitative colorimetric method requiring no special chemicals is described which is specific and determines vitamin C in amounts as small as 3γ. The reagents used are: 1. 2 g. sodium tungstate added to 10 cc. *N* sulphuric acid, and dissolved by stirring in a boiling water-bath, then cooled by flowing water and filtered. The filtrate is light greenish yellow and if not clear must be made so by heating. This reagent should be prepared fresh daily; 4 days after preparation it is no longer of use. 2. Two *N* sodium hydroxide. Five cc. of this solution should correspond to 10 cc. of the sulphuric acid used in reagent 1. In carrying out the test, 4 cc. of the vitamin C solution in 2% metaphosphoric acid are treated with 1 cc. of reagent 1 and after mixing, 0.4 cc. of reagent 2 is added and the whole thoroughly mixed. The liquid is sky blue in color and is compared colorimetrically with a similarly prepared standard solution. The color is quite different from that of methylene blue or copper sulphate; it resembles cobalt or night blue more. The color obtained with 4.61 mg. ascorbic acid has been found to correspond approximately to that of 1 mg. night blue. It is important to follow the order of procedure given above. When very acid or alkaline plant material is used, the extract should be neutralized and then brought up to a 2% metaphosphoric acid content. The concentration of ascorbic acid in the solution measured must be less than 10 mg./100 cc. Concentrations below 0.5 mg./100 cc. do not give very good results and must be concentrated as the previous ones diluted. The ratio of the concentrations of the standard solution to the test solution must lie within 1:2 or 2:1. Organs which have formerly given negative results for vitamin C show low values with the described method, *e. g.*, liver, lungs, kidneys, adrenals and heart. A substance other than vitamin C may be responsible.—AKIJI FUJITA, DANZO IWATAKE and TADAO MIYATA. *Biocehm. Z.*, 277 (1935), 296; through *Squibb Abstract Bull.*, 8 (1935), A-825.

Vitamin D—Evaluation of, in Fatty Oils by Spectrographic Methods. The authors review in detail the methods in use and those which have been proposed for the determination of the activity of vitamin D containing preparations, enumerating their disadvantages. The method proposed in this paper depends on the quantitative evaluation of the ultraviolet absorption spectra of fatty oils containing vitamin D. The principle stated by Lambert-Beer that a beam of light falling normally on the surface of an absorbing medium will be weakened by passage through the medium, the extent of the weakening being proportional to the thickness of the medium and the concentration of the absorbing material, is utilized. A detailed description of the apparatus necessary accompanies the article. The spectra as obtained from both the preparation and a control on the solvent medium are photographed and then compared. A plate of such photographs is shown. The two vitamin D products investigated in this article are: Calciferol (British Drug Houses Ltd.) and crystalline "D-Vitamin" (E. Merck, Darmstadt). On plotting curves from the spectrographs, it is found that vitamin D shows a characteristic maximum at $\gamma = 265 m\mu$. The method of plotting these curves, several of which are shown, is given in detail.—L. FUCHS and Z. BECK. *Pharm. Presse*, 40 (1935), 411. (M. F. W. D.)

Yohimbine—A New Specific Reaction for. The reaction as applied to yohimbine in tablets is as follows: Dissolve a tablet in 5 cc. of boiling water, add 1 cc. of ammonia water and 2 cc. of chloroform. Shake without emulsifying and separate the chloroform layer. The chloroform is washed with distilled water and, after separation, is evaporated on a water-bath. To the residue is added several drops of water, then 3 cc. of sulphuric acid, to which has been added 4 drops of 20% chloral solution, and the mixture is placed on a water-bath. In about 1 minute a definite blue coloration should appear.—M. PESEZ. *J. pharm. chim.*, 22 (1935), 164–165. (S. W. G.)

PHARMACOGNOSY

VEGETABLE DRUGS

Aconitum Napellus—Histological Study of. A study of the histology of the tuberous roots of *Aconitum napellus* L. subspecies *neomontanum* Wulf was made.—H. W. YOUNGKEN. *J. Assoc. Official Agr. Chem.*, 18 (1935), 515. (G. S. W.)

British Pharmaceutical Codex—Systematic List of Vegetable Drugs. A list of the genera and species of vegetable drugs in the B. P. C. 1934 is given. Certain abbreviations are employed

in the list to indicate that the plants are mentioned only in particular connections.—T. A. SPRAGUE. *Pharm. J.*, 135 (1935), 214. (W. B. B.)

Cinnamon and Cassia—Microscopic Analysis of. The authors have examined *Cinnamonum Zeylanicaum, Cinnamonum Cassia* (*C. aromaticum* or *Chinense, Cassia lignea*), *Cinnamomum Burani* (*Cassia vera*). They conclude that to determine the type of *Cinnamonum* or indicate its purity it is necessary to examine it under the microscope and use reagents. Starch is not characteristic, except for Chinese cinnamon. The crystals observed under polarized light differentiate the three types. In the cellulose residue after treatment, the tissues which permit identification of the three types are suber and sclerenchyma.—V. ESTIENNE and G. SPRUMONT. *Pharm. J.*, 135 (1935), 231. (W. B. B.)

Drugs—Structural Standards for Crude. Structural standards for crude drugs are valuable criteria for controlling purity and uniformity of drugs. Methods of analysis are available for determining the quantities laid down as standards, whether for unground drugs or for powders. It is suggested that the errors of analysis of powders amount to 5%, due to the method, and to about 15% for natural variation of vegetable substances. The standards at present in use are of various types, some applicable to unground drugs, some to powders, and others to all material whether unground or in powder. There is sometimes a wide variation in standards established for the same drug by different countries, and this would seem to call for some international discussion with a view to arriving at uniform requirements.—T. E. WALLIS. *Pharm. J.*, 135 (1935), 207. (W. B. B.)

Ginger—Curing of. Of the two methods of curing ginger—(1) sulphur curing, (2) ordinary curing—the former, which had a number of advantages over the ordinary curing, has now to be discontinued owing to the regulations introduced in most importing countries prohibiting the sale of ginger containing sulphur dioxide. An investigation was made by the Department of Agriculture, Ceylon, to discover whether equally good samples of ginger could be prepared without the use of sulphur.—ANON. *Tropical Agriculture*, 5 (1935), 125; through *Pharm. J.*, 135 (1935), 157.

Plants—Medicinal, Aromatic and Similar. The International Federation is studying the possibility of publishing a "Code International," the aim of which is to provide data for the standardization of medicinal, aromatic and similar plants, and the Federation aims at the establishment of agreement among the different nations, so that a uniform quality may be available in all parts of the European Continent.—T. E. WALLIS. *Pharm. J.*, 135 (1935), 274. (W. B. B.)

Trixis Divaricata Spreng. Var. Discolor Griseb—Pharmacognosy of. The botanical description is given. The drug contains resins, saponins and an alkaloid. It causes in animals sensory and motor paralysis. Medicinally it is used as a diaphoretic and diuretic in chronic arthritis.—LUIS FLORIANI. *Rev. farm.* (*Buenos Aires*), 77 (1935), 223. (A. E. M.)

PHARMACY

GALENICAL

Antipyrine Tablets. The following formula is suggested for making antipyrine tablets: Antipyrinum (Sieve No. 10), 150 Gm.; amylum marantæ, 40 Gm.; agar pulvaratum, 8 Gm. Mix this together with mucilago cydoniæ, 6 Gm.; spiritus concentratus, 4 Gm. Rub through a No. 5 sieve. After careful drying, mix the granules and add talcum, 42 Gm.—ANON. *Pharm. J.*, 135 (1935), 213. (W. B. B.)

Aromatics and Volatile Oils—Pharmaceutical Action of. The action and use of fenchone, benzyl and fenchyl alcohols, anisic acid, anethol, anise oil, menthol, mustard oil, angelica root oil, apiol, ambra, anthranilic acid and its derivatives, Atlas cedar oil and amyl isovalerianate in pharmaceuticals are discussed.—J. AUGUSTIN. *Riechstoff-Ind. Kosmetik*, 10 (1935), 157–158. (H. M. B.)

Coffiplex Tablets. A formula for Coffiplex Tablets, which produces white tablets with no tendency to split or stick to the punches of the tablet machine is as follows: Caffeinum 50 Gm., Acetanilidum 150 Gm., Phenacetinum 150 Gm., Agar pulveratum 5 Gm. To be mixed and passed through a No. 15 sieve. Add sol. gelatinæ (3 Gm.–40 Gm.) 3 Gm., and finally acid stearinicum 2 Gm. dissolved on the water-bath, and Spiritus alcoholisatus 10 Gm. To be rubbed through a No. 5 sieve.—ANON. *Pharm. J.*, 135 (1935), 213. (W. B. B.)