PHARMACEUTICAL ABSTRACTS

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ANALYTICAL

Albumin Tannate. The properties of various commercial albumin tannate preparations are compared with those of Tannalbin, Knoll. The moisture content, water solubility, ignition residue, solubility in pepsin-hydrochloric acid and the solubility in 1.5% sodium carbonate of the residue not digested by pepsin-hydrochloric acid as well as that of the substance *in toto* are reported. The tested preparations display a number of differences from Tannalbin. Different specimens of the latter product show some variability. Albumin tannate preparations are less soluble in pepsin-hydrochloric acid and in sodium carbonate solution and have lower tanning content than the Tannalbin. When made by the method of the Netherlands Phar., IV or V, albumin tannate is too soluble in pepsin-hydrochloric acid. A substance with the desired properties may be prepared by increasing either the temperature to which the solution of tanning mixed with albumin is heated or by increasing the duration and temperature of the process of hardening. Hardening of the dry material may be omitted if the solution is boiled for a couple of hours. Of eight foreign (non-Danish) commercial albumin tannate preparations tested only one had properties sufficiently similar to those of Tannalbin to serve as a substitute.—F. REIMERS. *Dansk. Tids. Farm.*, 9 (1935), 153. (C. S. L.)

Alkaloid-Containing Poisonous Drugs—Detection of, in Powdered Drugs. The following method is proposed: Moisten throughly a triturate of the finely powdered drug and 2.0 Gm. powdered slaked lime with 12 cc. water, extract the mixture in a small flask with 20 cc. of 90% alcohol for 1 hour with vigorous shaking and warming on a water-bath, separate the alcohol extract from the drug-lime-mixture by means of a small flat filter and wash the contents of the filter with 10 cc. alcohol. Evaporate the filtered extract and wash alcohol, take up the residue with 10 cc. water and 3 drops of dilute sulphuric acid in the cold, filter the solution of fat, resins and insoluble matter (calcium sulphate) through a small moistened filter and rinse the filter with some drops of water. Divide the filtrate on 3 watch glasses and 2 small porcelain dishes, evaporate each to dryness and carry out the following precipitations and color reactions for alkaloids.: (1) Dissolve two of the residues on the watch glasses in some drops of water and treat one with some drops of tannic acid solution (German Phar. VI, page 764) and the other with some drops of iodine-potassium iodide solution (see German Phar. VI under iodine solution page 765 and page 780). Gray-brown or red-brown amorphous precipitates indicate the presence of alkaloids. Take up the residue in the third watch glass with some drops of water and a drop of dilute sulphuric acid and add two drops of potassium-mercuric iodide solution. In this case the usual Mayer's reagent is not employed but Boehm's reagent, a highly concentrated solution of red mercuric iodide in concentrated potassium iodide solution in such proportions that the formula is $2KI.HgI_2$ and is prepared by dissolving 33.11 Gm. potassium iodide and 45.28 Gm. mercuric iodide in 33.11 Gm. water with some shaking (specific gravity 2.1694 at 15° C.). This reagent yields with bases containing trivalent amino-nitrogen white-yellow precipitates and with quaternary ammonium bases of pentavalent-nitrogen highly colored yellow precipitates. (2) the residues in the porcelain dishes are subjected to the following color reactions: (*a*) Take up the residues with 2–3 drops of concentrated sulphuric acid, which might not be colored characteristically. If it remains colorless, add to one dish some particles of fine sugar, whereby characteristic colors might appear. (*b*) Scatter a trace of finely powdered potassium dichromate which produces at the most a weak green color caused by the chromic sulphate formed in closing single small crystals but not colored streaks flowing from the crystals or odors like benzaldehyde, cumarin, spirea or the like. In cases of positive reactions a microscopic examination of the drug will give a clearer idea of the nature of the alkaloidal admixture.—H. KUNZ-KRAUSE. *Apoth. Ztg.*, 50 (1935), 862–864. (H. M. B.)

Alkaloids—Determination of, in Injectable Solutions. All of the common alkaloids used in solutions for injection were studied to determine the best methods of assay. Results were as follows: Adrenaline gives most accurate results when determined by the described Atherton-Seidele colorimetric method or by the photometric method; atropine by titration with periodic acid; caffeine by iodometric determination; cinchonine by gravimetric method; emetine by titration with acid; novacaine by retitration after titration with acid; pilocarpine by iodometric determination; quinine by colorimetric determination; sparteine by retitration; strychnine by use of periodic acid; yohimbine by retitration.—G. N. THOMIS and D. JATRIDES. *J. pharm. chim.*, 21 (1935), 585. (M. M. Z.)

Alkaloids—Rotatory Power of Various. Rotatory powers of the various alkaloids included in the French Codex (except quinine and its salts) were determined for the yellow sodium line and for the yellow, green, blue and violet mercury lines, as follows:

Alkaloid	Yellow Sodium α 5893	Yellow Hg. α 5780	Green Hg. α 5460	Blue Hg. α 4358	Violet Hg. α 4046	α5460/α5780
Cocaine-HCl	+ 72.30°	+ 75.53°	+ 84.11°	+144.48°	+168.46°	1.113
Codeine	−134.9°	−142.54°	−163.55°	−284.61°	−346.21°	1.147
Heroin-HCl	−148.35°	−156.37°	−174.91°	−308.49°	−361.11°	1.118
Picrotoxine	− 30.05°	− 31.37°	− 36.42°	− 63.52°	− 80.12°	1.161
Emetine-HCl	+ 53.85°	+ 57.04°	+ 60.49°	+ 85.69°		1.06
Pilocarpine-HCl	+ 89.09°	+ 94.63°	+108.61°	+181.53°	+230.97°	1.20
Pilocarpine-HNO_3	+ 81.46°	+ 84.96°	+ 97.21°	+168.68°	+211.16°	1.193
Scopolamine-HBr.$3H_2O$	+ 22°	+ 23.75°	+ 26.75°	+ 53.75°	+ 56.75°	1.126
Eserine (base)	− 78.97°	− 84.11°	− 94.13°	−133.01°	−161.37°	1.119
Eserine salicylate	− 78.87°	− 84.96°	− 99.26°	−179.35°	−251.92°	1.169

CH. LORMAND and P. GESTAU. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 3 pp. (A. P.-C.)

Anesthetics. With Special Reference to Cocaine and Novocaine in Illegal Trade. This paper is one of a series covering the detection of cocaine in combination with other anesthetics, in which combinations it often appears in illegal traffic. Several of these papers published in the *Pharm. Weekblad* in 1934 are abstracted in the YEAR BOOK of the AM. PHARM. ASSOC., 23 (1934). The paper outlines a general scheme for the examination of samples. (1) the numbing effect as well as (2) the taste (tasteless, bitter, sour) should be observed. (3) The general appearance is of importance. The following anesthetics are amorphous: Acoine-HCl; alypine; orthoform-N; propæsine and tutocaine. The following appear amorphous but are also hygroscopic: Allylcocaine-HCl (nycaine); psicaine-new (tartrate). The following are granular: Cocaine-HNO_3; psicaine; eucaine-A and B; butelline and subcutine. The following exhibit beautiful crystals: Cocaine-HCl (highly refractive); larocaine; novocaine; ecognine and benzoecognine. The following occur in finer crystals: Pantocaine; stovaine and tropacocaine and in very fine crystals: Alypine nitrate; holocaine; anæsthesine; psicaine-new-HCl; orthoform and cycloform. The following show a tendency to clump together: Allylcocaine nitrate; diocaine and percaine. (4) With reference to the reaction toward litmus paper the following show a neutral reaction: Alypine nitrate; butelline; diocaine; eucaine-A; eucaine-B; novocaine; nycaine; orthoform; orthoform new; percine; propæsine and tropacocaine and the following show a weakly acid reaction: Acoine; allylcocaine nitrate; alypine (very weak); anæsthesine; cocaine-HCl and cocaine-HNO_3; cycloform (very weak); larocaine; pantocaine; psicaine; psicaine-new-HCl; psicaine-new-tartrate; tutocaine; stovaine (weakly acid toward Congo paper); subcutine (strongly acid) and holocaine (alkaline). (5) The solubility in water is often an indication of identification. The following are easily soluble in water: Allycocaine nitrate; alypine-HCl; alypine nitrate; cocaine-HCl; larocaine; novocaine; nycaine; pantocaine; percaine; psicaine; psicaine-new-tartrate; psicaine-new-HCl; stovaine and tropacocaine. The following are fairly soluble in water: Acoine; butelline; cocaine-HNO_3; diocaine; eucaine-B; tutocaine. The following are poorly soluble in water: Anaesthesine; cycloform; eucaine-A; holocaine; orthoform; orthoform-new; propaesine and subcutine. (6) Eucaine-A is insoluble in alcohol and holocaine, psicaine, tutocaine, tropacocaine and novocaine are slightly soluble in alcohol. (7) The following are only slightly soluble in 0.5 N hydrochloric acid: Diocaine, holocaine, psicaine-new-HCl, tropacocaine and cocaine-HNO_3. (8) Reactions for acid radicles are as follows: Butelline gives a reaction for SO_4''; allylcocaine nitrate, alypine nitrate, cocaine nitrate and novocaine nitrate give reactions for NO_3'; psicaine and psicaine-new give reactions for tartaric acid; subcutine gives a reaction for sulfophenylic acid. The following give no reaction for acid radicles: Anaesthesine, cycloform, orthoform, orthoform-new and propaesine. The remaining anesthetics give reactions for Cl'. (9) With the reaction with potassium dichromate and strong hydrochloric acid, primary aromatic amines give a purple color; the benzoyl group gives a yellow resin and pantocaine gives a greenish resin accompanied by a considerable odor. (10) The lignin test (with paper) on primary aromatic amines shows the following reactions: without acid, very good—anæsthesine

and subcutine: without acid good—orthoform and orthoform-new: without acid fairly good—cycloform and propæsine: with acid very good—anæsthesine, subcutine, orthoform, orthoform-new, cycloform and propæsine; with acid fairly good—butelline, larocaine, novocaine and tutocaine. (11) The reaction of Denigès-Marquis: All phenols are colored at temperatures below 100°: Orthoform, orthoform-new, subcutine and ecgonine: The following phenol-ethers react: Acoine, diocaine, holocaine (after 24 hours), percaine, anæsthesine, cycloform and propæsine. Reactions on the benzoyl group (Denigès) are given by the following: Allylcocaine nitrate, alypine, benzoylecgonine, cocaine-HCl, eucaine-A, eucaine-B, nycaine, psicaine, psicaine-new-HCl, psicaine-new-tartrate, stovaine and tropacocaine. Alypine-nitrate and cocaine-nitrate do not give a reaction. (12) The following reactions are given with ferric chloride: Orthoform becomes violet-yellowish brown-dirty green; orthoform-new becomes green-violet-yellowish brown and subcutine becomes purple. Among the phenols and phenol-ethers, acoine becomes reddish brown, ecognine red, diocaine brown, holocaine brown and percaine yellowish brown. (13) Two tables of melting points for 38 anæsthetics are given, one alphabetical and one according to melting points. (14) Optical rotation is also of importance. Tables for $[\alpha]_D^{20^\circ}$ are given. The series of papers is to be continued.—C. OFFERHAUS and C. G. BAERT. *Pharm. Weekblad*, 72 (1935), 801. (E. H. W.)

Argyrol, Collargol, Electrargol and Protargol—Reactions for the Differentiation of. (1) Mix 2 cc. of a 0.5% solution of chromic acid with 7–8 cc. of the dilute solution of the silver preparation and add 0.5 cc. of a saturated sodium bicarbonate solution. Argyrol: first flocculation, later a clear yellow color. Collargol: a reddish precipitate. Electrargol like collargol. Protargol like argyrol, but more turbid. (2) Mix one cc. of 10% sodium thiosulphate solution with 9 cc. test solution and add phenophthalein. Argyrol: dark yellow color turning black. Collargol and electrargol: violet to black precipitate. Protargol: Pink to red color. (3) Mix 0.5% solution of chromic acid with nitric acid and add the silver preparation: Electrargol: the yellow liquid turns slowly to blue. The other compounds do not give that reaction. (4) Picric acid precipitates the latter three compounds.—CH. VAILLE. *Rev. centro estud. farm. bioquím.*, 25 (1935), 247. (A. E. M.)

Atropine—Study of Commercial Drugs Containing. Fifty-seven atropine-containing commercial preparations were assayed by observing the dilatation produced in the pupil of the white mouse. Most of the preparations were also assayed chemically. Comparison of the two methods indicated whether the preparations contained pure atropine (I), hyoscyamine (II) or a mixture thereof. II has 2.23 times the mydriatic effect of I in mice by subcutaneous injection. A 1% aqueous solution of I-sulphate kept for 2.5 months at room temperature in the dark lost 20% of its activity. A 1-mg. % aqueous solution kept in an incubator at 37°, was found to be inactive after 4 months.—H. A. OELKERS and E. VINCKE. *Arch. exptl. Path. Pharmakol.*, 178 (1935), 439; through *Squibb Abstr. Bull.*, 8 (1935), A-968.

Azulenogenic Sesquiterpenes—Color Reaction of. The greenish blue color reaction given by Bourbon geranium oil and imperfectly purified rhodinols with a chloroformic solution of bromine is due to an azulenogenic sesquiterpene which can be isolated from geranium oil, and which boils under 5 mm. pressure at 117° to 120°, has a refractive index at 20° of 1.5021, specific gravity at 15° of 0.9134, rotatory power (in a 1-dm. tube) of +11.40° (the sign of rotation was probably reversed by heating with boric acid to fix and separate the alcohols) and molecular refraction 66.2 (calculated 66.13). Azulenogenic sesquiterpenes are very widely distributed in nature; they are present in a large number of essential oils, in which they can readily be detected by the chloroformic bromine test.—SÉBASTIEN SABETAY and HERMINE SABETAY. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 3 pp. (A. P.-C.)

Bath Preparations XIV. Fifteen commercial preparations are tested and studied from the following points: (1) manufacturer, (2) how packed, (3) price, (4) physical properties, (5) reaction, (6) dry residue, (44–91.9%), (7) mineral matter (1.2–15.7%), (8) sulphur dioxide (4 positive), (9) Hirst-Prokter reaction (4 positive), (10) 5 volatile oil (0.2–2.5%, 2 negative), (11) appearance under the ultraviolet lamp, (12) appearance of the capillary streak in ordinary and ultraviolet light and (13) appearance and color of the aqueous solution (1:10). A second table lists a pine needle bath, a ladies' bath, rheumatism, morning, evening and children's bath, giving (1) appearance, (2) odor, (3) specific gravity, (4) dry residue, (5) mineral matter, (6) % volatile oil, (7) special properties and value and (8) chief constituent.—W. PEYER. *Apoth. Ztg.*, 50 (1935), 658–661, 675–678. (H. M. B.)

Bath Preparations—XV. Foot Baths. Twelve preparations were examined and reports made with regard to (1) manufacturer, (2) price, (3) physical properties and (4) qualitative and quantitative analysis.—W. PEYER and R. STADLER. *Apoth.-Ztg.*, 50 (1935), 924–927. (H. M. B.)

Bellier Number—Study of Its Use for the Detection of Peanut Oil in Olive Oils. The following definition is proposed for the Bellier number: the temperature of initial crystallization of the solid fatty acids of a fat or oil in alcoholic solution, when the solution is subjected to progressive cooling with constant agitation. A recommended technique for carrying out the test is described in minute detail, more particularly as regards the cooling; it is essentially as follows: to 1 cc. of well clarified oil or fat in a 26–27-mm. by 22-cm. test-tube add 5 cc. of alcoholic potash (75 Gm. per l.), saponify by careful heating using an air-cooled reflux condenser, cool to 30° to 35°, add 1.5 cc. of 1 + 2 acetic acid and 50 cc. of 70% alcohol, close the tube with a rubber stopper carrying a semi-precision thermometer graduated from 0° to 60° or 0° to 100° and note the temperature at which the solution becomes cloudy due to crystallization. The test may be repeated on the same portion of sample, but in order to obtain concordant results the temperature must be raised each time about 15° to 20° above the clouding point. The precautions which must be taken in the preparation of the reagents and in the manipulative operations are described. The Bellier number of peanut oil is generally given as 40° to 41° and that of pure olive oil as 11.8° to 14.5°. Examination of a number of oils of known purity gave values of 9.5° to 18°, the high values being obtained with Tunisian and Moroccan oils; oils obtained by extraction of press cake gave values of 9° to 16.5°. For the detection of peanut oil in doubtful cases, mix 9 parts of sample with 1 part of peanut oil (both by weight) and determine the Bellier number; with pure olive oil the value will not exceed 20° (except in extremely rare instances). By drawing a curve of the Bellier number of mixtures of peanut and olive oils, admixture of olive oil in peanut oil can also be detected and estimated quantitatively. If a mixture of 9 parts of the supernatant oil of canned fish and 1 part of peanut oil gives a Bellier number above 19.5°, the presence of peanut oil in the canning oil is indicated. As the Bellier number depends on the solid acids of oils, it might be deduced that its value was proportioned to the solid acids content of olive oils, with the proviso that the oils compared contain solid acids of the same type, of which there are two, acids separating as arborescent crystals and giving lead salts that are completely soluble in warm ether, and acids separating as glomerule-shaped crystals giving lead salts that are only partially soluble in warm ether. Some olive oils contain practically exclusively one or other type of acids, while others contain both. As the solid acids are insoluble in 70% alcohol, it is suggested that they might be separated by the technique of the preparation of the Bellier test followed by cooling overnight at 10° to 12° and then for 1 hour at 5°, centrifuging, filtering and determining the acids by weighing or acidimetric titration. The quantitative composition of a mixture of solid acids, the nature of which is known, could be obtained by determination of the Bellier number and comparing with curves prepared from the Bellier numbers of known mixtures of the same acids. Oils extracted from olive press cake, when subjected to the Bellier test and then allowed to stand over night at 20° to 25°, contain a suspended flocculent precipitate which gradually gathers at the top of the liquid. This test, though possibly not absolutely specific, is very characteristic and permits of detecting the addition of from 5 to 20% (according to the origin) of such extracted oil in pressed oil.—R. MARCILLE. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 13 pp. (A. P.-C.)

Benzene—Ultra-Violet Absorption Spectrum of. Utilization for the Detection and Determination of Small Amounts of Benzene in the Atmosphere. The benzene is absorbed by passing the dried atmosphere through 95% alcohol at −85°. The absorption spectrum curve is obtained by Fabre and Amy's technique of the imbricated spectra method. As little as 0.1 mg. benzene per liter of air can be determined with an accuracy of about 5% (in absence of homologs) on a 1-liter sample. The method is absolutely specific. The method is sensitive to about 1 part benzene in 100,000 parts of alcohol.—PIERRE LAURIAN. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 6 pp. (A. P.-C.)

Benzoic and Cinnamic Acids—Identification of, in Microsublimates of Balsams and Resins. The recognition is accomplished by the addition of 4–5 drops of dilute sulphuric acid and then some sodium amalgam to a portion of the sublimate, when benzoic acid is reduced to benzaldehyde. For cinnamic acid, a portion is treated with chromic acid or permanganate to oxidize it to benzaldehyde. The benzaldehyde is precipitated in a hanging drop slip containing *p*-nitrophenylhydra-

zine (freshly prepared saturated solution in 15% acetic acid). The melting point of the hydrazone is 190–192° C. The reaction is sensitive to 10 micrograms of benzoic acid and 20 micrograms of cinnamic acid.—R. FISCHER. *Pharm Zentralh.*, 76 (1935), 345. (E. V. S.)

Bismuth—Assay of National Formulary Preparations Containing. Report is made of a study undertaken to determine whether the method of assay used for the glycerite might be applied to determination of bismuth content of Solution of Bismuth, Elixir of Bismuth, Elixir of Pepsin and Bismuth and Elixir of Pepsin, Bismuth and Strychnine and also whether other methods could be used advantageously. Reference is made to the various methods which have been used for the quantitative determination of bismuth. In the present investigation a comparative experimental study of the official sulphide method, of the Mayer phosphate method and of the Schoeller and Waterhouse phosphate method was undertaken. Detailed procedures are given. Two samples of each preparation were assayed by each of two different people by the sulphide method and the Mayer phosphate method. The Schoeller-Waterhouse phosphate method was abandoned because it seemed to yield slightly high results. Either the sulphide method or the Mayer phosphate method yields accurate and comparable results when applied to glycerite, solution or elixirs of bismuth. Since the Mayer phosphate method is simple and accurate it might well replace the sulphide method now official for the glycerite and be made official for the solution and elixirs.—GLENN L. JENKINS and SYLVIA MILLETT. *J. Am. Pharm. Assoc.*, 24 (1935), 561. (Z. M. C.)

Borax-Glycerin-Water Solutions—Reactions of. When glycerin is added to a solution of borax a portion of the boric acid is converted into boroglycerin. The conversion is dependent upon the concentration of the glycerin. The reaction increases in acidity with an increase of free boroglycerin. With a certain concentration of glycerin the amount of free boroglycerin should be just sufficient that the acidity of the solution will neutralize the alkalinity of the borax. In order to determine which concentration of glycerin in combination with a concentration of borax will react neutral, the author made the following determinations:

Percentage of Glycerin in the Solution		Percentages of Borax (2%)	(6%)	(10%)
85.8	p_H	4.8	4.9	5.0
60	p_H	5.25	5.4	5.45
40	p_H	5.75	5.9	6.05
30	p_H	6.10	6.25	6.45
20	p_H	6.55	6.75	7.05
10	p_H	7.25	7.60	8.0
5	p_H	7.90	8.3	8.7
0	p_H	9.2		

These determinations, made at about 15°, show that a 20–25% solution of glycerin in borax solution reacts neutral.—P. VAN DER WIELEN. *Pharm. Weekblad*, 72 (1935), 877. (E. H. W).

Bromides—Detection of Small Quantities of, in Sodium Chloride. Mix 10 cc. of water with one drop of a saturated solution of fuchsine and add drop by drop a solution of chlorine in water. Note how many drops are needed to destroy the red color. For the test use 10 cc. of a saturated solution of the sodium chloride. Add a drop of fuchsine solution and the double quantity of chlorine water then determined. A reddish tint turning violet indicates bromine. The reaction is sensitive to 0.1 mg. potassium bromide.—R. CASARES LÓPEZ. *Farm. Moderna*, 46 (1935), 55. (A. E. M.)

Bromine—Electrometric Determination of, in the Presence of Large Amounts of Chlorine. The method permits the determination of 0.24 mg. bromine in the presence of large amounts of chlorine. Chlorides are removed by means of acetone. The analysis of aqueous solutions is relatively simple, but it is applicable also to biological substrates after removal of proteins, this being effected by moist incineration. The reduction of silver chloride and bromide by means of sodium amalgam produces a rapid and complete conversion of the halogens to soluble sodium salts. The lower limit of bromine detected in tissue by this method was 5 mg. %.—G. E. VLADIMIROV and J. A. EPSTEIN. *Mikrochemie*, 18 (1935), 58. (L. L. M.)

Butterfat—Determination of, in food. A method was proposed for the isolation of fat. A weighed amount of paraffin equivalent to about one-third the weight of fat expected is added to the sample and the mixture hydrolyzed with hydrochloric acid. Filter cell and ice water are added and the mixture filtered through linen coated with filter cell in a Buchner funnel. The

precipitate is transferred to a beaker, dried, pulverized and treated with anhydrous sodium sulphate and petroleum ether and the extract filtered through asbestos in a Knorr tube. Four extractions are made and the combined filtrates evaporated to dryness and weighed. Correction is made for the added paraffin. Advantages cited are: the fat is extracted from the dried material with a single solvent, emulsions are avoided and the use of larger quantities of materials are possible. F. HILLIG. *J. Assoc. Official Agr. Chem.*, 18 (1935), 454. (G. S. W.)

Cæsium—New Spot Reaction for. Gold and platinum bromides yield with cæsium salts a black precipitate useful in the detection of cæsium by the spot method. Analysis of the black triple salt indicated the formula $Cs_2Au_2PtBr_{12}$. For the detection of small quantities of cæsium in the absence of rubidium the use of concentrated reagents is recommended. If rubidium is present the best results are obtained with a solution containing 3% gold and 1.5% platinum. A drop of the reagent is touched to a piece of paper and to the spot is added a drop of the test solution. In the presence of cæsium, a gray to black spot is produced. 0.25 γ per cu. mm. can be detected. With the exception of rubidium in concentrations above 2%, the chlorides of rubidium, potassium, sodium, lithium and ammonium do not interfere. Cæsium may be estimated quantitatively by the spot method with an accuracy of 5–10%.—E. S. BURKSER and M. L. KUTSCHMENT. *Mikrochemie*, 18 (1935), 18. (L. L. M.)

Caffeine—Determination of, in Decaffeinated Coffees. The following method is recommended: mix 50 Gm. of ground, roasted, decaffeinated coffee with glass wool cut into short lengths, introduce directly into a Soxhlet extractor between two wads of glass wool (avoid packing tightly), extract for 5 hours with slightly moist chloroform, adding during the first and third hours 3 cc. of 20% ammonia when the extractor has just been siphoned; evaporate to pasty consistency on the water-bath (using suction to remove the last trace of solvent); add successively 75 cc. water, 20 cc. of 10% sulphuric acid and 1.5 Gm. of paraffin having a melting-point of 60° to 62°, heat 20 minutes on the water-bath with occasional stirring, let stand over night in the refrigerator at 5°, filter in the refrigerator, make the filtrate alkaline with ammonia and evaporate to dryness on the water-bath; break up the residue with a nickel spatula, take up 4 times with anhydrous chloroform, filter, evaporate to dryness on the water-bath; dissolve in 30 cc. of boiling water, add 1% potassium permanganate solution to permanent violet coloration, let stand on the water-bath 15 minutes (adding permanganate if the color disappears), discharge the color by dropwise addition of 3% hydrogen peroxide containing 1% of acetic acid, filter if necessary, extract with 4 30-cc. portions of pure chloroform (recovered chloroform is not advisable), evaporate the chloroform, dry at 90° to 100° for 15 minutes and weigh. Duplicate determinations agree within 1 mg. (on a total weight of 12 to 20 mg.). The caffeine obtained is white, fairly pure, and melts at 220° to 225° (Maquenne block). If the technique is not strictly followed, there may remain small amounts of fat, which can be eliminated by another permanganate purification; this produces a loss of 1 to 1.5 mg. of caffeine, which should be corrected for.—A. FAURE and R. PALLU. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 4 pp. (A. P.-C.)

Carbon and Hydrogen—Preheater for the Microanalytic Determination of. A description of the apparatus with diagram.—W. F. BRUCE. *Mikrochemie*, 18 (1935), 103. (L. L. M.)

Carbon Dioxide—Determination of, in Confined Atmospheres. The principle of the method consists in shaking a given volume of the atmosphere with a hydro-alcoholic solution of rosaniline decolorized by hydrazine, and comparing the color with those obtained by similar treatment of samples of known carbon dioxide contents. To simplify the method from the standpoint of sanitary control, comparison may be made with the color obtained with an atmosphere having a carbon dioxide content that is at the limit of safety.—RENÉ DUBRISAY and LÉON GION. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 2 pp. (A. P.-C.)

Charcoal—Activated. Demand for a charcoal with greater adsorptive powers was responsible for the study reported. Adsorbent carbons may be produced in several ways, one of the most promising being that of carbonizing sulphite waste liquor from the pulping of wood for paper. Activation is usually resorted to, this usually involving some chemical treatment. A number of tests for purity, that is, freedom from toxic or inert substances are given. The German Phar. has a carbon with an adsorption capacity of 0.525 Gm. of methylene blue per Gm. of carbon. The Swiss Phar. has one with capacity of 0.24 Gm. of methylene blue per Gm. of carbon. Saturation of carbon is best obtained by reliance on the mass effect of an excess of dyestuff. The following is the method finally decided upon: "Dissolve 0.25 Gm. of methylthionine chloride (methyl-

ene blue) in enough distilled water to make 250 cc. of solution. Measure exactly 50 cc. of the solution, at 25° C., into each of two 100 cc. glass-stoppered flasks. To one flask add exactly 0.25 Gm. of Activated Charcoal, stopper the flask and shake vigorously for five minutes. Filter the contents of each flask through a filter which has not been previously moistened, rejecting the first 20 cc. of each filtrate. Measure exactly 25 cc. of each remaining filtrate into 250-cc. volumetric flasks. Add to each flask 50 cc. of a solution of sodium acetate (1 in 10) and mix thoroughly, then add from a burette 35 cc. of $N/10$ iodine, keeping the mixture in constant rotation. Stopper the flasks and allow them to stand for fifty minutes, shaking vigorously at intervals of ten minutes. Dilute each mixture to exactly 250 cc. with distilled water, mix thoroughly, allow to stand for ten minutes and filter each through a filter that has not been previously moistened, rejecting the first 30 cc. of each filtrate. Determine the excess of iodine in 100 cc. of each filtrate by titration with $N/10$ sodium thiosulphate. The difference between the two titrations, multiplied by 5, amounts to not less than 3.5." An important use of activated carbon is thought to be adsorption of gaseous products of fermentation or putrefaction and experiments led to adoption of the following: "Into each of two flasks place 185 cc. of distilled water and 5 cc. of glacial acetic acid and mix thoroughly. By means of a pipette add to each flask 10 cc. of a solution of 2.5 Gm. of crystallized sodium sulphide in 100 cc. of distilled water, placing the tip of the pipette at the bottom of the flask during delivery. Rotate the flask gently for thirty seconds, add to one of the flasks 1 Gm. of Activated Charcoal, stopper the flasks and shake them for five minutes. Filter the contents of each flask through a filter that has not been previously moistened, rejecting the first 20 cc. of each filtrate. Titrate 100 cc. of each subsequent filtrate with $N/10$ iodine, using starch T.S. as the indicator. The filtrate from the Activated Charcoal consumes at least 5 cc. less of $N/10$ iodine than the filtrate from the solution to which no Activated Charcoal was added." Response of charcoals to the proposed tests to be included in U. S. P. XI is given for sixteen different carbons.—JOSEPH ROSIN, GEO. D. BEAL and CHESTER R. SZALKOWSKI. *J. Am. Pharm. Assoc.*, 24 (1935), 630. (Z. M. C.)

Chaulmoogra Oil in the Pharmacopœias and in Commerce. The lower limit of acidity and the upper limit of optic rotation should be suppressed. A 10% solution of 10 Gm. oil in a total volume of 100 cc. in chloroform investigated with sodium light should give, at room temperature in a 10-cm. tube, a rotation of 5°12′.—A. and C. CHALMETA. *Farm. Moderna*, 46 (1935), 63 and 94. (A. E. M.)

Chloral Formamide—Melting Point of. The melting point of Chloral Formamide B. P. C. is given as between 114° and 115° C., which is the same as that in the B. P. 1914. However, as the result of several experiments, the authors recommend that the melting point should be recorded as 124° to 126°, with limits for pharmaceutical purposes between 122° and 126°. In determining the melting points, the values given are the mean temperature of melting, the transition range in most instances being one to two degrees. It is suggested that the recorded melting points represent the temperature of sintering possibly of impure material.—C. T. BENNETT and N. R. CAMPBELL. *Pharm. J.*, 134 (1935), 795. (W. B. B.)

Chloroform and Carbon Tetrachloride—Differentiation between. The author comments on the recent article of Rozeboom (*Pharm. Weekblad*, 72 (1935), 689) in which a reaction depending upon the solubility of papaverine hydrochloride in chloroform is used to differentiate between chloroform and carbon tetrachloride. Quinine sulphate may be employed in similar manner. The author suggests the two following reactions for distinguishing between chloroform and carbon tetrachloride: (*1*) *Positive for Chloroform.*—When boiled with Fehling's solution the sample with the chloroform will show reduction: that with the carbon tetrachloride will not. (*2*) *Positive for Carbon Tetrachloride.*—Heat 1 drop (no more!) in a test-tube lightly closed with a cork. The vapors will be oxidized by the air. Only carbon tetrachloride will liberate chlorine ($COCl_2$). After cooling shake with a few cc. of water and add potassium iodide-starch solution. A blue color will result.—N. SCHOORL. *Pharm. Weekblad*, 72 (1935), 751. (E. H. W.)

Chromatographic Adsorption Analysis in Pharmacy. Rum was colored with caramel and then allowed to run through a vertical column of a suitable adsorbent (aluminum oxide). Untreated rum left its coloring matter on the top layer of the aluminum oxide, the rest of the column remaining perfectly white. The rum colored with caramel produced more or less faded zones throughout the entire column. The filtrate was practically colorless. One-half per cent solutions of natural and synthetic balsam of Peru in alcohol and in petroleum ether were prepared. They

were treated as described above. The results show that the constituents in the natural and synthetic balsams are not the same, being retained differently by the aluminum oxide. Colorless constituents of preparations may in some cases be brought to view by examination of the aluminum oxide column under a quartz-analysis lamp. Official tincture of digitalis prepared with absolute alcohol was compared with a tincture prepared with diluted alcohol. Marked differences are apparent in the appearance of the aluminum oxide column. A review of the literature of adsorption analysis is given.—VALENTIN. *Pharm. Ztg.*, 80 (1935), 469. (G. E. C.)

Citronellal—Determination of, in Java Citronella Oil. Dutch Standard Method. The following method will be used as a standard for the determination of citronellal in Java citronella oil: Into a 150 cc. flask weigh accurately about 2 Gm. of Java citronella oil, add 10 cc. of 95% alcohol and 20 drops (1 cc.) of 0.1% alcoholic bromophenol blue solution and make neutral with $0.1N$ potash. To the neutralized liquid add from a burette or an automatic pipette 20 cc. of $0.5N$ alcoholic potash and immediately afterward from a measuring glass 20 cc. of a 5% alcoholic hydroxylamine hydrochloride solution. Shake, allow to stand at room temperature for an hour (under European conditions; at the much higher temperature in the Dutch East Indies a quarter of an hour is quite sufficient) and titrate the excess of potash with about $0.5N$ (alcoholic) hydrochloric acid, until the color of the indicator changes to greenish yellow, comparing the color with that of a blank determination made in the same way. The result is calculated from the following formula: per cent of citronellal $= \frac{(b-a) \times n \times 15.4}{g}$

where b = volume in cc. hydrochloric acid required for blank determination, a = volume in cc. of hydrochloric acid required for titration, n = normality of hydrochloric acid and g = weight, in grams, of oil taken.—ANON. *Perf. Ess. Oil Rec.*, 26 (1935), 253. (A. C. DeD.)

Cocaine—Separation and Detection of, in Mixtures of Cocaine and Procaine. Two methods are given for the separation and identification of small quantities of cocaine-HCl when a large amount of procaine-HCl is present. One method is given for the detection of small quantities of cocaine, free base, when a large amount of procaine, free base, is present. One method is given which is suitable for estimating within 4 mg. the amount of cocaine or cocaine-HCl present in a mixture of procaine, free base or salt, and cocaine, free base or salt. All of these methods are based on the fact that a buffer of proper acidity will cause the formation of a water-soluble salt of procaine and will not affect cocaine. All methods given separate the cocaine as the free base and do not affect its molecular structure, thus permitting the identification of pure cocaine and not its decomposition products.—C. H. RILEY. *Am. J. Pharm.*, 107 (1935), 270. (R. R. F.)

Cod Liver Oil—Determination of, in Emulsions. Weigh exactly 2 Gm. of emulsion in a 50-cc. beaker and add 8 cc. of 10% hydrochloric acid. Cover the beaker with a watch glass and heat for a half hour on a water-bath. Agitate the contents for a quarter hour, then transfer the liquid with 10 cc. of alcohol into a graduated vessel, shake vigorously for one minute and leave until it separates into two layers. Fill up nearly to the top graduation with petroleum ether, shake for a half minute and allow to stand for one hour. Withdraw 40 cc. of the solution with a pipette into a tared flask, distil the ether and heat for a half hour in an oven. Weigh, then heat again for a half hour and check the weight.—W. LEPPER. *Z. Analyt. Chem.*, 98 (1934), 164–166; through *Chimie et Industrie*, 33 (1935), 676. (W. A. P.)

Colorimetric Analysis and p_H Determinations—Applicability of the Color and Luminescence Comparator of Rojahn-Heinrici for. A discussion. Illustration.—R. SEIFERT. *Apoth. Ztg.*, 50 (1935), 590–593. (H. M. B.)

Colorimetric Methods of Analysis—Theory and Physico-Chemical Basis of. Assuming that color is numerically measurable, in order that a reaction may be used it is necessary to define the colorimetric function $I = f(C)$, which gives the relationship between the color intensity I and the initial concentration C of the substance to be determined. I is really a function of numerous variables (temperature, time, concentration of reagent, operating technique) to which suitably selected values must be assigned. The methods of selecting these values are discussed and exemplified by the case of the microdetermination of reducing sugars by means of phosphomolybdic reagent. The same procedure would be suitable in all cases, more particularly for the elaboration of methods for detecting toxic gases.—ISTIN. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 10 pp. (A. P.-C.)

Copper—Drop Method of Detection of. A few cc. of solution are heated for 1–1.5 minutes

with tin and hydrochloric acid, the solution is poured off, the tin washed and boiled for 1 minute with 1.5 cc. of 65% nitric acid, the solution diluted and 0.2 cc. of saturated aqueous sodium fluoride, 0.2 Gm. of zinc sulphate and 0.3 cc. of saturated aqueous $(NH_4)_2Hg(CNS)_4$ are added, when a lilac precipitate of $ZnHg(CNS)_4$, $CuHg(CNS)_4$ indicates $\nless 10^{-7}$ Gm. copper. Cobalt, lead, iron, tin, arsenic, antimony and bismuth do not interfere.—L. M. KULBERG. *J. Appl. Chem. Russ.*, 7 (1934), 1079; through *Squibb Abstr. Bull.*, 8 (1935), A-957.

Cresol Soap Solution—Determination of Cresol Content of. H.'s method previously proposed (*Apoth. Ztg.*, 49 (1934), 183) is not applicable to cresol soap solutions containing acids of castor oil and its derivatives since these are not separated from the phenolic constituents. The following procedure is recommended whereby the resin and fatty acids are separated by alkaline lead acetate solution: Shake a mixture of 30 Gm. lead acetate solution (20%), 50 Gm. sodium hydroxide and 20 Gm. water with 20 Gm. cresol soap solution for 1 minute. In 60 Gm. of the filtered liquid in a cassia flask dissolve 20 Gm. sodium nitrate, decompose this solution with 20 Gm. nitric acid and then proceed by the method previously reported above. For soap solutions containing chlorcresols the method previously described is modified: Before the decomposition of the alkaline solution by hydrochloric acid, add 5 cc. benzin in order to bring about an easy separation of the heavy halogen phenols (chlorcresol and chlorxylenol) from the salt solution. After subtracting the added volume of benzin from the volume observed the % cresol may be calculated in the usual manner.—K. HANDKE. *Apoth. Ztg.*, 50 (1935), 335. (H. M. B.)

Crystal Precipitation by Salting Out. II. The items discussed are: α-napthol, β-naphtol, resorcin, hydroquinone, pyrogallol, veronal, veronal-sodium, luminal, luminal-sodium, chloramine, 1,2,5-toluylendiamine hydrochloride, *p*-phenylenediamine, *p*-aminodiphenylamine, 1,2,5-diaminoanisole.—L. ROSENTHALER. *Mikrochemie*, 18 (1935), 50. (L. L. M.)

Diaminoacridine—Determination of, in Euflavine. The difference in basicity between diaminoacridine and diaminomethylacridine is used as the basis of the electrometric and colorimetric determinations of the former in the mixture. *Electrometric Method.*—The euflavine (containing diaminoacridine and diaminomethylacridine) is dissolved in 10 cc. of warm water with shaking. About 0.5–1.0 Gm. of the euflavine may be used. The solution is cooled and 0.5 *N* sodium hydroxide is added. The diaminoacridine then precipitates, apparently in crystalline form or as a reddish brown oily liquid which later becomes crystalline. Three of the preparations used could be titrated electrometrically after 15 minutes, having been stirred rapidly during this time. Glass electrodes were used in an arrangement described by G. Kilde (*Dansk Tidss. Farm.*, 9 (1935), 129). The method is accurate to about 1%. *Colorimetric Method.*—0.5 to 0.7 Gm. of euflavine is dissolved in 10 cc. of boiling water in a 250-cc. flask with careful shaking of the flask (so that the solution is not cooled too quickly); 90 cc. of isopropyl alcohol and 2 cc. of thymol blue solution are added and the solution titrated with 0.1*N* sodium hydroxide to a definite color change, *i. e.*, a murky brownish color. On account of the greenish fluorescence of euflavine solutions, the titrations must be made in strong electric light. The solution is then titrated back with 0.1*N* hydrochloric acid, 0.1 cc. being added at a time until the color no longer changes. About 0.2 to 0.5 cc. of hydrochloric acid is usually sufficient. If precipitation occurs on the addition of isopropyl alcohol, add 5 to 10 cc. of water and dissolve the precipitate by heating on a water-bath, and after the solution has cooled add a little isopropyl alcohol, since too high a concentration of water interferes with the end-point.—F. REIMERS. *Quart. J. Pharm. Pharmacol.*, 8 (1935), 218–230.

(S. W. G.)

2,4-Dinitrophenol—Detection and Determination of. Qualitative and quantitative tests were described. The sample is treated with water and 4% sodium hydroxide, filtered, the filtrate acidified with hydrochloric acid and extracted with chloroform. The chloroform is evaporated, 2 cc. of 10% sulphuric acid and 0.2 Gm. of powdered zinc are added to a portion of chloroform residue. After standing 10 minutes a pink color should appear. The solution is then filtered, 10 drops of 1% sodium nitrite added to the filtrate and the mixture allowed to stand in the dark for 5 minutes. Two cc. of saturated beta-naphthol in strong ammonia is added and after 2 minutes the mixture is extracted with ether. A pink or violet color in the ether layer should appear. Comparisons of crystals and melting points with authentic samples of 2,4-dinitrophenol are recommended. For quantitative determination the sample is macerated with 2% sodium hydroxide, acidified with hydrochloric acid and extracted with chloroform. The chloroform extracts are then extracted with 4% sodium hydroxide until the yellow color is removed. The alkaline solution is

made up to volume and an aliquot containing about 0.1 Gm. of dinitrophenol is made neutral with hydrochloric acid. Twenty cc. of $0.1N$ bromine and 5 cc. of concentrated hydrochloric acid are added to the solution in a glass stoppered bottle, the mixture shaken for 1 minute, cooled and 10 cc. of 15% potassium iodide added. After shaking, 1 cc. of chloroform is added and the mixture titrated with $0.1N$ sodium thiosulphate using starch indicator. Each cc. of $0.1N$ bromine corresponds to 0.0092 Gm. of dinitrophenol.—I. S. SHUPE. *J. Assoc. Official Agr. Chem.*, 18 (1935), 464. (G. S. W.)

Electric Micromuffle. A Pregl micromuffle was modified by replacing the wire gauze on the horizontal tube with an electric heater made by winding No. 18 Nichrome wire around a porcelain form. The advantages claimed are: An even heat is given over the whole surface so there is no spattering; no attention is required after turning on the current until the boat is removed; the heater may be slid off the tube and allowed to cool while the weighing is made.—C. J. RODDEN. *Mikrochemie*, 18 (1935), 97. (L. L. M.)

Enteric Coatings—I. Laboratory Method for the Study and Control of. A study made several years ago has been continued. The p_H of gastric and intestinal juices has been determined, normal range being p_H 1.6 to p_H 1.8, though usually showing much greater range. There is evidence that in human small intestines reactions may vary from distinctly acid to slightly alkaline. Temperature of the gastrointestinal tract has been measured with a recording thermometer. Gastric temperatures have been found between 97.5° and 102.2° F., the upper part of the intestine between 98° and 100.1° F. Ingestion of hot or cold drinks makes quick change of temperature. Motility or emptying rate of stomach has been studied. Normally, peristaltic activity begins soon after ingestion of a meal but its advent may be retarded or inhibited. Experimental work was carried out in an apparatus in which tablets rotate in buffers covering gastric and intestinal range. Though a mechanical test cannot duplicate conditions in stomachs, the procedure is valuable in a laboratory study. Hundreds of tablets have been studied. Coating surface of tablets immersed in buffers from p_H 1.2 to p_H 6.4, showed little change after eight hours; those immersed in buffers beginning at p_H 6.4 showed signs of attack and shriveling effect within five to fifteen minutes. In addition to requisite physical and chemical properties, and physiological inertness, a coating must resist a wide and variable acid range of the stomach and disintegrate at the acid reaction found in the small intestine. If a coating becomes decidedly alkaline before disintegration begins the tablet will probably pass through the small intestine without disintegrating.—MILTON WRUBLE. *J. Am. Pharm. Assoc.*, 24 (1935), 570. (Z. M. C.)

Essential Oils and Their Ethanol Solutions—Presence of Methanol and Formaldehyde in. A study of the methods which purport to or actually do permit of identifying or determining traces of methanol or formaldehyde in essential oils or in their solutions in ethanol, led to the conclusion that methods based on oxidation of methanol should be rejected. Ethanol that is absolutely free from methanol, and a large number of constituents of essential oils, when oxidized under analytical conditions, give rise to the formation of formaldehyde; the usual color reactions of formaldehyde, and especially the Schiff reaction, are not specific. Traces of methanol and formaldehyde are normal constituents of numerous essential oils; they exist from the time of distillation and the aero-oxidation which is more or less inevitable contributes to increase the methanol and formaldehyde contents. Commercial alcohols nearly always contain traces of methanol, and aero-oxidation of ethanol can give formaldehyde and possibly also methanol. Ethanol solutions of essential oils containing methyl esters undergo ethanolysis even at ordinary temperature, with liberation of methanol. Presence of traces of methanol or formaldehyde in essential oils or in their ethanol solutions cannot therefore be taken as proof of adulteration.—Y. R. NAVES. *Parfums France*, 13 (1935), 60–73, 91–104 (in French and English). (A. P.-C.)

Essential Oils—Determination of Ethyl and Methyl Alcohols in Natural. The authors find that ethyl alcohol is present in rose blossoms from the moment of plucking, in quite remarkable proportions. They show that the Thorpe method, which is customarily used for the determination of alcohol in essential oils, gives results which are clearly deficient. They describe and give results obtained by the Thorpe method and also the Zeisel method which they suggest as possessing great advantages of sensitiveness, accuracy, speed and simplicity. In conclusion the authors state that the results given by Thorpe's method are definitely below the true quantity of alcohol present in a synthetic oil. The same is true with regard to the free alcohol present in a natural essential oil. Thorpe's method requires 12 cc. of oil; that of Zeisel does not need so much

as 1 Gm. Zeisel's method is simpler and much more rapid, and is less susceptible to error than that of Thorpe.—R. GARNIER and L. PALFRAY. *Perf. Ess. Oil Rec.*, 26 (1935), 259.
(A. C. DeD.)

Extract and Fluidextract of Coca, B. P. C.—Assay of. It is suggested that the B. P. C. 1934 assay process given under "Ext. Cocae Liq." be modified to include the following: (1) That the extraction of the alkaloids from ammoniacal solution be made by successive portions of ether until complete. (2) That sufficient portion of dilute acid be used to completely extract the alkaloids. (3) That the final ethereal solution be washed with a little water in order to remove any traces of ammonium salts. (4) That the final residue be dehydrated with absolute alcohol. (5) That volatile bases be excluded by heating the alkaloidal residue at 80° C. for two hours.—W. A. N. MARKWELL. *Pharm. J.*, 134 (1935), 416. (W. B. B.)

Extracts—Determination of the Viscosity of Viscous. The pharmacopœial descriptions of the consistency of thick extracts are held to need revision. Relative or absolute values for limits of viscosity should be set. A simple, inexpensive viscosimeter is described consisting of a disk which sinks through the fluid placed in a glass cylinder, the disk bearing an index shaft which slides in a millimeter-graduated, glass tube. The sinking speed is determined with the stop-watch. The ordinary viscosimeters are described and discussed, both the nozzle-flow types such as the Ostwald pipette, and the Engler, the Redwood, the Saybolt and the Barbey instruments, and the torsion types such as Couette's, Searle's and Hoeppler's. The use of the sinking-disk viscosimeter is suggested for such extracts as *Ext. absinthii, belladonnæ, chamomillæ, filicis, gentianæ, glycyrrhizæ, hyoscyami, millefolii, secalis cornuti.* Figures are reported for various specimens of *Ext. glycyrrhizæ, gentianæ* and *menyanth.* For extracts made by the same general methods from the same drug a relationship of viscosity to content of dry residue can be seen, and a typical curve is given. The influence of temperature on the sinking speed is also shown in a typical curve. Here the viscosity coefficient is about three times greater at 15° C. than at 20° C., hence control of the temperature during viscosimetry is of good importance.—S. KJELLMARK. *Farm. Revy*, 34 (1935), 397, 413, 425. (C. S. L.)

Fats and Oils—Hydroxyl Number and Acetyl Value of. A report of a collaborative study of three methods was made. Values obtained by the André-Cook and the Roberts-Schuette methods were in good agreement. Low values were reported for the West-Hoaglund-Curtis method likely due to a fading of the end-point in titration.—W. L. ROBERTS. *J. Assoc. Official Agr. Chem.*, 18 (1935), 435. (G. S. W.)

Ferrous Iron—Determination of, in Presence of Organic Matter by Heisig's Method. Attention is drawn to the suitability of the iodate method (Heisig's Method) for the assay of the saccharated iron compounds of the B. P. and B. P. C. and of ferrous lactate. It is shown that ferrous iron may be titrated with accuracy by iodate in the presence of liquid glucose, acacia, tragacanth, sucrose, invert sugar in small amounts, levulose, dextrose, lactose, glycerin, lactic acid and citric acid. Invert sugar in great excess produces a small error. The method is unsatisfactory in the presence of licorice, marshmallow, quinine and aqueous extract of cochineal.—G. J. W. FERREY. *Pharm. J.*, 134 (1935), 784. (W. B. B.)

Glyceryl Trinitrate Tablets—Assay of. Anderson's method for the assay of glyceryl trinitrate tablets is based on the volatility of glyceryl trinitrate in steam, hydrolysis with sodium hydroxide solution, reduction of the nitrate formed and distillation of the ammonia produced into standard acid. The author suggests a method which is a modification of Anderson's method. The suggested modification is as follows: Place five tablets in a 500-cc. Kjeldahl flask, add 25 cc. saturated sodium sulphate solution, 75 cc. water and sufficient sulphuric acid to make just acid to litmus paper (usually 0.3 cc. of $N/1$ sulphuric acid required). Distil just to dryness, using a still head, into a flask containing 10 cc. of $N/10$ sodium hydroxide, keeping the outlet tube below the surface of the alkali. Wash down the condenser and outlet tube and evaporate the sodium hydroxide solution to dryness. Add 2 cc. of water, 0.3 Gm. ($\pm$0.01 Gm.) of reduced iron and 2 cc. of 50% v/v sulphuric acid, allow to stand for ten minutes and boil for two minutes. Transfer the acid solution to a steam distillation apparatus, make alkaline with 4 cc. of saturated hydroxide solution and distil the liberated ammonia into a flask containing 10 cc. of $N/10$ sulphuric acid until the distillate measures 500 cc. Take 100 cc. of the distillate, add 2 cc. of Nessler's reagent and compare the color produced with that produced by adding the same amounts of reagent to 100 cc. of a solution containing ammonium chloride equivalent to 0.1 mg. of nitrogen. The color of

the unknown should not vary more than 20% from that of the standard, and a control experiment must always be carried out, the 500-cc. distillate being concentrated to 100 cc. From the difference between the nitrogen content of the experimental solution and the control, the glyceryl trinitrate present can be calculated. Factor, nitrogen to glyceryl trinitrate 5.4.—W. SMITH. *Pharm. J.*, 134 (1935), 790. (W. B. B.)

Glyceryl Trinitrate Tablets—Assay of. The method adopted by the author for the assay of glyceryl trinitrate tablets is as follows: For tablets of B. P. strength, weigh accurately the equivalent of 1 mg. of glyceryl trinitrate, in the form of powdered tablets, into a stoppered cylinder containing exactly 5 cc. of glacial acetic acid. Shake continuously for one hour, filter and transfer 1 cc. to a small porcelain dish. To this promptly add about 2 cc. of phenoldisulphonic acid, stir well and allow to stand for fifteen minutes. Dilute with about 8 cc. of water, make alkaline cautiously with ammonia and transfer to a 25-cc. stoppered vessel. When cool adjust the volume to 20 cc. and the temperature to 20° C. and filter. Compare the color in suitable glass containers with that produced as follows: (1) Dilute 1 cc. of solution of glyceryl trinitrate of exactly 1% strength with 50 cc. of glacial acetic acid, mix thoroughly, transfer 1 cc. to a porcelain dish, add phenoldisulphonic acid and proceed as above. (2) Transfer 1 cc. of a 0.225% aqueous solution of silver nitrate B. P. to a porcelain dish, evaporate gently to dryness, add phenoldisulphonic acid and proceed as above. The color produced in all three cases should be equal to 7.0 Lovibond yellow units when viewed through a glass cell of 1 inch internal width.—H. O. MEEK. *Pharm. J.*, 134 (1935), 791. (W. B. B.)

Gold—Microchemical Determination of, in the Presence of Palladium and Tin. Large amounts of palladium and tin do not interfere with the determination of gold by a method previously reported by the author (*Mikrochemie*, 13 (1933), 165 and 17 (1935), 174).—J. DONAU. *Mikrochemie*, 18 (1935), 11. (L. L. M.)

Gold Sol—Preparation of, for Liquid Analysis. The stock solution contains gold bromide crystals, 5.0 Gm.; purified potassium bromate, 1.365 Gm.; and freshly distilled water, 38.7 Gm. This solution is stable for one year. For preparation of the gold sol, 1 cc. of the stock solution is first diluted with 14 cc. of distilled water and then further diluted with water to make one liter. For the reduction, 10 cc. of a 1% potassium oxalate solution is added, and the solution placed in daylight for about 20 minutes.—W. HERRMANN. *Ztschr. Immunitätsforsch.*, 84 (1935), 279; through *Pharm. Zentralh.*, 76 (1935), 394. (E. V. S.)

Heroin—Micro-Detection of. Among the various alkaloidal reagents the following give the most characteristic crystalline precipitates with heroin. (1) A saturated solution of mercuric diiodide in 10% hydrochloric acid; (2) gold chloride in concentrated hydrochloric acid and sodium picrate.—WILLIAMS and FULTON. *Freie Apoth. Stimmen.*, 17–18 (1934), 19; through *Pharm. Tijdschr. Nederland.-Indië*, 13 (1935), 56. (E. H. W.)

Indigocarmine—Use of, in Microvolumetric Analysis. One gram equivalent of indigocarmine corresponds to one-half gram mole. $\frac{C_{16}H_{18}N_2O_8S_2Na_2}{2} = 233.11$ Gm. The reagent is best suited for volumetric analysis in $0.001N$ solution. It is standardized for use in alkaline solution against $0.01N$ potassium ferricyanide in the presence of sodium carbonate; for use in acid solution, against potassium permanganate in the presence of sulphuric acid. 1 cc. of $0.001N$ indigocarmine solution corresponds to 0.3292 mg. potassium ferricyanide. 1 cc. of $0.001N$ potassium permanganate solution corresponds to 0.23311 mg. indigocarmine or to 0.21111 mg. indigodisulfonic acid. Small amounts of ferrous iron may be determined with fair accuracy by oxidizing the iron with excess standard potassium permanganate, then titrating the excess permanganate with indigocarmine solution.—I. M. KORENMAN. *Mikrochemie*, 18 (1935), 31. (L. L. M.)

Indophenine Reaction—Application of the, for the Identification of Some Organic Polyacids. A drop of the neutralized acid is evaporated to dryness, mixed with a trace of phosphorous trisulphide and heated with a few drops of a solution of isatin in sulphuric acid. A blue color is developed. The reaction is most sensitive with succinic and fumaric acid, but is produced also with maleic, malic, pyrotartaric, tartaric and citric acid.—JOSÉ VÁZQUEZ SÁNCHEZ. *Farm. Moderna*, 46 (1935), 58. (A. E. M.)

Iron—Cerimetric Titration of Small Amounts of, by Means of α,α'-Dipyridyl as Indicator. Dilute acid solutions of ferrous compounds give a sharp end-point (pink-colorless) when titrated with ceric sulphate. The indicator solution consists of 0.25 Gm. α,α'-dipyridyl dissolved in 50 cc.

water with 50 cc. of concentrated ammonia water added afterward. About 5 drops of this solution is used for 50 cc. of ferrous solution. The ammonia is added to speed the formation of the colored indicator complex which develops slowly in acid solution. With this indicator, ferrous solutions containing 0.1–10 mg. iron in 50 cc. $1N$ hydrochloric acid may be titrated by means of 0.002–$0.015N$ ceric sulphate in $1N$ sulphuric acid solution. The end-point appears within one drop and is far better than with a titanous solution. Ceric sulphate solution is quite stable when guarded against direct sunlight, even in extreme dilutions. For the reduction of ferric iron, the silver reductor of Walden, Hammett and Edmonds was found to present an improvement over the Jones reductor, *et al.*, provided the acid concentration is kept within 0.5–$1N$ hydrochloric acid.—C. J. VAN NIEUWENBURG and H. B. BLUMENDAL. *Mikrochemie*, 18 (1935), 39.

(L. L. M.)

Lactic Acid—Determination of. The author refers to the reports of Girault, who claims that the official method gives too high results, and of Bourdeau, who reports satisfactory results with the same method. The author's results indicate that the official method is a good one, but certain lactic acids yield results higher than 100% owing to the presence of esters which are hydrolyzed.—F. KAYSER. *J. pharm. chim.*, 21 (1935), 604. (M. M. Z.)

Medicinal Mixtures—Thermo-Analysis and Eutectics of. The author discusses the thermo-analysis and eutectic temperatures of mixtures of several organic medicinal chemicals. Cases where a molecular combination results between the two chemicals in the mixture are discussed along with cases where no reaction occurs. Combinations of antipyrine, veramon, compral, hypnal and trigemine are fully discussed as is also the caffeine-salicylic acid mixture (the sodium salicylate-caffeine mixture). The following eutectic temperatures of such combinations are given:

Combination	Eutectic Temp.	Composition
Bromural-pyramidon	78.0°	41 % bromural
Pyramidon-acetanilid	58.5°	41 % acetanilid
Pyramidon-phenacetin	78.5°	68 % pyramidon
Acetanilid-phenacetin	82.5°	58 % acetanilid
Salol-benzonaphthol	34.0°	86.9% salol
Bromural-salol	40.8°	3.5% bromural
Bromural-phenacetin	109.0°	53 % bromural
Veronal-salol	41.2°	1.5% veronal
Veronal-phenacetin	121.6°	26.2% veronal

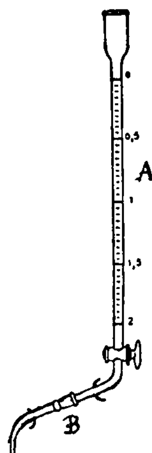
The liquification of powder mixtures is discussed and tables giving eutectic temperatures and compositions of mixtures of camphor with salol, naphthaline, β-naphthol, resorcin and ethylurethan; of salol with monobromcamphor, β-naphthol, thymol, guaiacol, naphthaline, antipyrine, urethan, menthol, chloral hydrate, methylacetanilid, sulfonal, phenacetin and terpin hydrate; of phenol with β-naphthol, naphthaline and acetanilid; and several others are quoted from the literature.—J. MEIJER. *Pharm. Weekblad*, 72 (1935), 922. (E. H. W.)

Mercurochrome—Determination of Mercury Content of. Assuming that the B. P. C. method of assay for the mercury content of mercurochrome is accurate, and adopting the limits laid down in the particular monograph, not one manufacturer's sample examined conformed to the required standard. The results obtained by the B. P. C. method of assay show an appreciable experimental variation. The alkaline-permanganate oxidation method for the assay of mercury appears to give more reliable and consistent results than the B. P. C. method.—R. F. CORRAN and F. E. RYMILL. *Pharm. J.*, 134 (1935), 783. (W. B. B.)

Methanol—New Method for the Determination of Small Quantities of, in Presence of Very Large Quantities of Ethanol and Its Homologues. The method, which is long and delicate, is essentially an accurate research method. It is based on the 4 following steps: (1) converting the primary alcohols into the corresponding iodides, (2) distillation of the alkyl halides, (3) treatment of the distillate with silver acetate to regenerate the alcohols with formation of silver iodide, which is weighed (giving the "iodization value," P) and finally (4) oxidizing the alcohols in the cold and determining the oxygen consumed (giving the "oxidation value," p). If X and Y are, respectively, the amounts of methanol and ethanol to be determined, $P = 234.8\ (X/32 + Y/46)$ and $p = 48X/32 + 32Y/46$, whence $X = 2 \times 32\ (p/32 - P/234.8)$ and $Y = 3 \times 36\ (P/234.8 -$

p/48). The method, which is described in detail, is essentially as follows: The methanol is concentrated by a suitable rectification collecting the head fraction containing all the methanol together with impurities such as acids, aldehydes, etc.; the latter are removed by adding a slight excess of silver nitrate, letting stand over night, neutralizing with 10% potassium carbonate and redistilling; further rectifications are carried out if necessary; iodization is carried out at about 105° C. with 3 cc. of hydriodic acid (specific gravity 1.7) and 0.5 cc. of sample, the vapors being washed by passing through a suspension of 10 mg. calcium carbonate in 5 cc. of water at 45° to 50° C. and collected under a solution of 0.5 Gm. silver acetate in 50 cc. of water, and a current of pure carbon dioxide being passed through the apparatus; iodization requires about one hour and a quarter from the time the distillate begins to collect in the silver acetate solution; not less than 2 hours after iodization is complete the solution in the receiver is redistilled, the distillate collected under 5 cc. of water and made to 50 cc., and p is determined in the distillate and P in the residue. To determine p, add 5 cc. of distillate to 10 cc. of solution containing 5 cc. of 66° Bé. sulphuric acid and 20 mg. of potassium dichromate in a 25-cc. glass-stoppered Erlenmeyer, stopper, let stand over night and titrate the excess of dichromate iodometrically; under these conditions methanol is oxidized according to equation $2CH_3OH + 3O_2 = 2CO_2 + 4H_2O$, and ethanol and higher homologues according to $2R.CH_2OH + 2O_2 = 2RCO_2H + 2H_2O$. To determine P add 1 cc. of nitric acid (specific gravity 1.38) and 50 cc. of water to the residue containing silver bromide, boil gently with successive additions of water to compensate for evaporation till the precipitate is a bright canary yellow, filter, wash, dry 1 hour at 130° to 150° C. and weigh. The limit of sensitiveness is about 0.05 mg. methanol in the 0.5 cc. taken for iodization, and the accuracy varies from about 0.5% for a methanol:ethanol ratio of 1:100 to about 5 to 10% for a ratio of 1:1000 to 1:2,000; this, however, requires the use of very pure reagents, and commercial hydriodic acid should be purified by treatment with red phosphorus and redistillation. The various steps of the method are discussed in detail to justify the technique adopted. Application of the method to a large number of wines, brandies and fermented fruit juices showed that methanol in amounts up to over 4,000 mg. per liter is a normal constituent of all natural alcoholic media.—M. Flanzy. *Ann. Fals.*, 28 (1935), 260–277; cf. *J. Am. Pharm. Assoc., Abstract Sect.*, 29 (1935), 125. (A. P.-C.)

Methyl Alcohol—Determination of, in Alcoholic Products. A method for the determination of methyl alcohol in the presence of ethyl alcohol was described. The method depends upon the formation of the iodides, their partial separation by fractionation, their conversion into salts by trimethylamine and the separation of the tetramethylammonium iodide formed from the methyl alcohol from absolute alcohol. The apparatus used is a modification of the Zeisel-Fanto apparatus for the determination of methoxy groups. Red phosphorus and iodine are used rather than the more expensive hydriodic acid. Most of the interfering substances may be removed by use of a distillate of the sample.—J. B. Wilson. *J. Assoc. Official Agr. Chem.*, 18 (1935), 477. (G. S. W.)



Microburette—New, with Removable Tip. The burette designed by Khouri and manufactured by Etablissements Prolabo, 12, rue Pelée, Paris, is made in two sections. The advantages claimed are: easy filling and cleaning and different tips giving the size of drops desired.—*J. pharm. chim.*, 21 (1935), 607. (S. W. G.)

Microchemical Laboratory Technique—Improvements in. The items discussed are: (*a*) some devices for working with the filter-stick of Emich, *i. e.*, frame, forceps and wash bottle; (*b*) sharpening block for glass knives; (*c*) an improvement in the determination of residues in the micromuffle.—H. K. Alber. *Mikrochemie*, 18 (1935), 92. (L. L. M.)

Microdistillation—Apparatus for. The apparatus was designed for use with vacuum. 0.05 cc. of liquid may be distilled. No special technique is required for operation and the unit need not be constructed for each distillation. Specifications and illustrations are given.—L. V. Peakes, Jr. *Mikrochemie*, 18 (1935), 100. (L. L. M.)

Microiodometric Determinations. The investigation showed that the microiodometric determination of sulphites and tin salts by iodine may be replaced by the use of iodate. The following advantages are claimed: In the preparation of the volumetric solution no special pre-

caution need be taken to prevent loss of material through volatilization. Volumetric solutions of potassium iodate do not require restandardization as frequently as do diluted solutions of iodine. *Determination of Sulphite.*—To 2–5 cc. of the diluted sulphite solution are added about 0.5 cc. of sulphuric acid (1:5) and several drops of starch solution. To the mixture is added 0.01*N* potassium iodate solution to the appearance of a weak permanent color. *Determination of Stannous Chloride.*—To 2–5 cc. of the stannous chloride solution, acidified with hydrochloric or sulphuric acid, is added the starch solution and then 0.01*N* potassium iodate solution to the appearance of a weak blue color. $2KIO_3 = 6SnCl_2$.—I. M. KORENMAN. *Mikrochemie,* 17 (1935), 361.
(L. L. M.)

Micropolarographic Investigations. A description with illustrations is given of an apparatus by the use of which the polarographic electrolysis of quantities of solution as low as 0.005 cc. is made possible. The use of the apparatus and of micropipettes is discussed.—V. MAJER. *Mikrochemie,* 18 (1935), 74. (L. L. M.)

Microshaker Useful in Microtitrations. The apparatus is made from an electromagnetic loud-speaker.—K. SCHWARZ. *Mikrochemie,* 18 (1935), 106. (L. L. M.)

Microsublimation—Is It Useful in the Investigation of Powdered Drug Mixtures? The method employed was that of Tunmann. Since little could be found in the literature, 19 drugs were first investigated individually as to sublimation products. Of them, 13 were found to give characteristic results. Using these drugs, mixtures first of only two drugs were investigated, then mixtures of three or more components. The results were tabulated. Of 92 drug mixtures investigated, 61 could be fully identified by microsublimation, in 25 mixtures not all of the drugs could be identified and in only 6 mixtures could none be identified.—F. WIESMANN. *Pharm. Acta Helv.,* 10 (1935), 125. (M. F. W. D.)

Mineral Oils—Contribution to the Study of Official. A comprehensive study and survey of the physical and chemical properties of mineral oils obtained from French, Belgian, German and American refineries. The result of this study is a proposal of a monograph on mineral oils for the French Codex now in preparation.—F. GRÉGOIRE. *Bull. sci. pharmacol.,* 42 (1935), 152, 217.
(C. T. I.)

Mineral Pigments—Systematic Method for the Identification of. II. Blue and Green Pigments. The blue pigments were differentiated: (1) By their behavior toward sodium hydroxide in the warm. Prussian blue gives a red-brown precipitate, other pigments remain unaffected. (2) With warmed diluted hydrochloric acid, ultramarine liberates hydrogen sulphide; mineral blue (blue basic carbonate of copper), carbon dioxide; Bremen blue dissolves without evolution of gas; cobalt blue and smalt blue remain unchanged. In the last two cases cobalt may be detected. Cobalt blue is insoluble in boiling hydrochloric acid (1:1), whereas smalt blue is partly soluble leaving a residue of silicic acid. The green pigments are warmed with diluted hydrochloric acid. Mineral green dissolves with evolution of carbon dioxide; Paris green evolves vapors of acetic acid; Bremen green, Scheele's green and cobalt green dissolve completely without evolution of gas. The last three pigments are differentiated by tests for copper, for copper and arsenic and for cobalt, respectively. Partial solubility in diluted hydrochloric acid suggests further test for glauconite or for mixed chromium pigments. Finally, if the pigment is practically insoluble in diluted hydrochloric acid, it is dissolved in concentrated hydrochloric acid and tested for chromium (chromium green). In all cases special microchemical reactions are given for the elements mentioned.—S. AUGUSTI. *Mikrochemie,* 17, (1935), 344. (L. L. M.)

Morphine—Colorimetric Assay of. The method is based upon the blue color formed by morphine on reduction with silicomolybdic acid. The method consists of dissolving 0.2–0.10 mg. of morphine or an equivalent amount in liquid form in 5 cc. of 1% hydrochloric acid, then 2 cc. of silicomolybdic acid, 5 cc. of ammonia water (5%) and water to make 25 cc. added in order given. After standing for five minutes, it is standardized against the standard prepared by using 5 cc. of a 1:1000 solution of morphine in 1% hydrochloric acid in the given test. The silicomolybdic acid is prepared according to a previous article (*Biochem. Ztschr.,* 27 (1933), 260).—R. HOFMANN and N. POPOVICI. *Pharm. Zentralh.,* 76 (1935), 346. (E. V. S.)

Morphine—Determination of, in Tincture and Extract of Opium. Mannich's method (*Arch. Pharm.,* 273 (1935), 112) is modified: *Tincture of Opium.*—Evaporate 30 Gm. of the tincture in a porcelain dish on a water-bath to 10 Gm., triturate with 1.5 Gm. calcium hydroxide and add water to 31.5 Gm. After standing for $^1/_2$ hour with frequent stirring, filter through a plaited

filter (10 cm.). To 20 Gm. of the filtrate (= 20 Gm. of tincture) in a 100-cc. Erlenmeyer flask, add 5 Gm. water, 38 Gm. methyl alcohol and 7 Gm. alkaline potassium oxalate solution (18.4 Gm. neutral potassium oxalate, 10 Gm. 0.1N potassium hydroxide, water *q. s.* 100.0 Gm.) and warm on a water-bath to 50° C. After cooling filter through a plaited filter (8 cm.), keep covered, triturate 56 Gm. of the filtrate (= 16 Gm. of tincture) with a solution of dinitrochlorbenzene (0.6 Gm. in 10 Gm.) (= 12.5 cc.) and then add 10 Gm. water. Collect the crystals which separate over night in a Gooch crucible, wash with 6 cc. methyl alcohol and 4 cc. ether in small portions and weigh after drying at 100° C. $\frac{100\ (\text{morphine ether} + 0.005) \times 0.632}{16} = \%$ morphine (Factor 0.632 is obtained by $\frac{285.2\ (\text{morphine})}{451.19\ (\text{morphine ether})}$. *Extract of Opium.*—Triturate 1.5 Gm. of the extract with 1.5 Gm. potassium hydroxide and 10 Gm. water and add water to 31.5 Gm. Proceed as under the assay of the tincture in which a 56 Gm. aliquot is equal to 0.8 Gm. of the extract. $\frac{100\ (\text{morphine ether} + 0.005).\ 0.632}{0.8} = \%$ morphine. Results obtained are compared with those by the German Phar. VI method and volumetric method.—K. HANDKE. *Apoth. Ztg.*, 50 (1935), 5101. (H. M. B.)

Morphine—Micromethod of Determination of, in Opium Preparations. Mix 0.05 Gm. of dried and ground opium with 0.02 Gm. calcium hydroxide and add gradually 10 cc. of water. Mix thoroughly for 30 minutes, leave standing for 15 minutes and filter through a dried filter. Take 5 cc. and evaporate to dryness. Add 0.2 cc. of an ammonium chloride solution (10%) and 3 cc. of purified ether. Decant the ether after 24 hours through a small filter and wash two times with 3 cc. ether. Finally wash the filter with 10 cc. ether. Dry residue and filter at moderate heat and dissolve in hot methyl alcohol, washing it through the filter, using 3, 3.2 and 2 cc. Add to the filtrate one cc. of 0.1N phosphoric acid and bring with water to a volume of 50 cc. Ten cc. are mixed with 10 cc. of a saturated sodium carbonate solution and 2 cc. of a solution of sodium phosphotungstic-molybdate (Folin and Denis). The blue color is compared colorimetrically with a standard morphine solution containing 0.1 mg. base per cc. The method can be applied to opium extract, tincture and morphine solutions.—LUIS DE PRADO. *Anales de Farm. Bioquim.*, 6 (1935), 12. (A. E. M.)

Nicotine—Nephelometric Determination of Small Quantities of. Advantages of the nephelometric method over the author's colorimetric method are: (1) smaller quantities may be used for the determination without decreasing the accuracy; (2) less time is required; (3) economy of materials, which is of particular importance in forensic investigations. *Method.*—Tobacco was steam distilled as described in *Bul. Cultivărei si Fermentărei Tutunului*, 42 (1932). One hundred cc. of distillate were collected, of which 25 cc. were reserved for gravimetric analysis. In each of 5 test-tubes was placed 5 cc. of 0.5% hydrochloric acid. The contents of the first tube were mixed with 5 cc. of distillate, and 5 cc. of the diluted distillate were transferred to the second tube. Continuing in this manner, five different dilutions were prepared which differed from each other in nicotine concentration by 50%. To each tube was added 1 cc. of silicomolybdate reagent and the resulting turbidity was compared with that produced by a 0.0015% nicotine solution. That dilution was then chosen which produced a turbidity greater than that of the standard nicotine solution. An additional 100 cc. of this dilution were prepared by neutralizing the required volume of distillate with 0.1N hydrochloric acid, using methyl red as indicator. Ten cc. of 5% hydrochloric acid were added and the whole diluted to 100 cc. with water. From this solution different dilutions of 10 cc. each were prepared with 0.5% hydrochloric acid; each dilution was mixed with 2 cc. of reagent and the mixtures were compared nephelometrically.

$$\%\ \text{nicotine} = \frac{100{,}000\ \text{V. f. F.}}{\text{V}_1.\text{V}_2.\text{E}}$$

where V = cc. of distillate; f = observed nephelometer reading (standard compared with test sample); V_1 = cc. of diluted distillate prepared; V_2 = cc. of diluted distillate used in the determination; F = titer of the standard solution in Gm.; E = weight of standard.—R. HOFMANN. *Mikrochemie*, 18 (1935), 24. (L. L. M.)

Nitrogen—Volumetric Determination of Residual, without Distillation. In each of two or three small Wassermann tubes are placed 3 cc. of sulphuric acid (0.1% by volume). 0.04–0.05 cc. of blood (serum) are added from a capillary pipette, the pipette being washed by drawing acid into the tube two or three times. To each tube is added 1 cc. of phosphomolybdic acid solution (5 Gm. anhydrous sodium sulphate and 8.3 Gm. phosphomolybdic acid in 200 cc. of water plus 20 cc. of about $5N$ sodium hydroxide, then boiled $^1/_2$ hour over a direct flame; after cooling, 10.6 cc. of concentrated sulphuric acid are added and made up to 1,000 cc.). After mixing, the tubes are placed for 5 minutes in a water-bath at 60° C., then cooled to room temperature and filtered through a 5 cm. Schleicher and Schüll 595 filter. Three cc. of filtrate are placed in a 10 cc. incinerating flask together with 0.5 cc. of sulphuric acid (25% by volume) and ashed over a microburner until acid vapors are no longer emitted and the residue remains colorless upon cooling. The cooled residue is diluted with a little absolutely ammonia-free water, and 1 cc. of indicator (15 mg. methyl red, Kahlbaum, dissolved in 10 cc. $1N$ sodium hydroxide and diluted to 1,000 cc. with water). A solution of 27% purest sodium hydroxide is added dropwise from a capillary to the appearance of the indicator change. The neutral mixture is transferred quantitatively with a little wash water to a Hagedorn-Jensen flask containing 5 cc. of hypobromite buffer mixture (*a*) 85.5 Gm. boric acid, 14.6 Gm. sodium hydroxide dissolved in 800 cc. water, the solution being boiled 30 minutes to expel ammonia, then diluted to 1,000 cc. (*b*) 20 Gm. of potassium bromide dissolved in 100 cc. $1N$ sulphuric acid in a 1-l. volumetric flask, afterward dissolving in the mixture 8 Gm. bromine, then diluting to the mark. To 4–5 cc. of "*b*" is added dropwise sodium hydroxide until the brown color is changed to yellow, then 10 cc. of buffer solution "*a*" and finally enough water to make 100 cc. After the addition of several granules of iodate-free potassium iodide and 3 cc. of hydrochloric acid (fuming acid diluted with an equal volume of water), the mixture is titrated with $0.0025N$ sodium thiosulphate delivered from a Pregl semi-microburet, using starch as indicator. 1 cc. of $0.0025N$ thiosulphate corresponds to 11.66 mg. nitrogen.—F. RAPPAPORT and R. PISTINER. *Mikrochemie*, 18 (1935), 43. (L. L. M.)

Oil of Hypericum—Examination of. Fifteen samples of oil are examined by means of the color comparator of Rojahn-Heinrici (*Pharm. Ztg.*, 78 (1933), 504) and the quartz lamp. It is observed that the oil from *Hypericum perforatum* L. shows a yellow-red fluorescence and the capillary streaks prepared from the oil of *Hypericum verum* fluoresces red-violet under the quartz lamp.—F. SONNTAG. *Apoth. Ztg.*, 50 (1934), 399–401. (H. M. B.)

Oil of Peppermint—Determination of. B. proposes the following procedure: Weigh the oil, acetylate and saponify as directed in the German Phar. VI, then add 1 cc. phenolphthalein solution and 1.5–2 cc. of official methylene blue solution diluted 1:10 and titrate with $0.5N$ hydrochloric acid until the color change is red-violet to green.—G. BAUMGARTEN. *Apoth. Ztg.*, 50 (1935), 364. (H. M. B.)

Oil of Peppermint—Determination of Menthone in. Menthone will form a ketoxime with hydroxylamine hydrochloride which reaction releases free hydrochloric acid, thus, 1 molecule of menthone will liberate 1 molecule of free hydrochloric acid which may be titrated (1 cc. $N/2$ KOH is equivalent to 0.077 Gm. menthone). The reagents used are a 50% solution of hydroxylamine hydrochloride in alcohol, half normal alcoholic potash and methyl orange Poirier No. 3 as indicator. Two–3 Gm. of peppermint oil are weighed out; two drops of helianthine and 15–20 cc. of the solution of hydroxylamine hydrochloride added. The liquid is colored red. Half normal alcoholic potash is then carefully added, care being taken that the solution does not become alkaline. The solution is then repeatedly shaken, after which small quantities of the hydroxylamine solution are repeatedly added until the red color disappears permanently. The solution is then carefully neutralized with the half normal alkali and the menthone content calculated from the number of cc. used.—GASTON PARRAUD. *Bull. sci. pharm.*, (1935), 337; through *Pharm. Weekblad*, 72 (1935), 878. (E. H. W.)

Oil of Turpentine—Detection of Pine Oil in. H. finds that the following modification of Wolff's test (*Farbenztg.*, 17, No. 2) is satisfactory: Mix equal parts of a solution of 0.5 Gm. calcium ferricyanide in 250 Gm. water and a solution of 0.2 Gm. ferric chloride in 250 Gm. water and add to 8 cc. of this mixture in a test-tube 5–8 drops of the oil and shake vigorously for 15 seconds. If *pure pine oil* is present a light blue color is noticeable in the border zone becoming quickly stronger and after 2–5 minutes strong blue color. The iron solution becomes immediately green-yellow to yellow-green to green to blue-green and after 5 minutes blue. After 15 minutes there is a

deep dark blue turbidity in the border zone and the iron solution is deep blue and somewhat turbid; after 1/2 to 1 hour a precipitate of Berlin Blue is formed which settles to the bottom. With *Pure oil of turpentine* the border zone after shaking and after 2–3 minutes remains colorless and the lower liquid is pure yellow to yellow-green (5–10 minutes) and then green (slowly); in 3–5 minutes a light blue color appears in the border zone. Old and fresh oils react alike. With pine oil additions up to 30% the test is as with pure pine oil. Additions of 10–20% may be easily recognized especially by comparison of the iron solutions, since with pure oil this solution becomes yellow-green in 10 minutes and is, by only a small addition of pine oil, soon colored blue-green or blue; with 10% pine oil a green color and after 5 minutes a blue-green color. The pure oils in the course of an hour or more never give more than a green color to the iron solution. Oils containing pine oil color the iron solution in this time deep blue and also become turbid. Two tables are given: (1) six oils from various sources are compared as to the color produced with potassium hydroxide and (2) the appearance of the border zone and ferric chloride solution after 1, 2, 3 and 5 minutes and 10–15 minutes with shaking with six samples of oils using the Berlin Blue test just described.—K. HÖLL. *Apoth. Ztg.*, 50 (1935), 748–750. (H. M. B.)

Opium, Concentrated, D. A. B. VI—Preparation of, in the Drug Store and Critical Evaluation of the D. A. B. VI Method of Preparation and Assay. The author discusses in great detail the preparation and evaluation of concentrated opium. The pharmacopœial directions are reviewed and criticisms, suggestions and explanations made. The D. A. B. VI method is discussed under the following headings: I. Extraction of all the strongly basic alkaloids which are precipitated from aqueous solution by ammonia; II. Extraction of the alkaloids not precipitated by ammonia but extractable with ether; III. Extraction of the weak alkaloids precipitated by sodium acetate; IV. Extraction of the non-precipitable alkaloids by shaking out with chloroform-phenol after alkalization with sodium bicarbonate; V. Conversion of the free alkaloids into hydrochlorides. Methods for the estimation of the morphine content of concentrated opium are also reviewed and compared with the official calcium method.—F. HAGELSTEIN *Pharm. Ztg.*, 80 (1935), 544. (G. E. C.)

Peppermint Oils—Detection of Japanese Mint Oil in. In the course of an investigation with large quantities of low-boiling fractions of Japanese oil, furfuraldehyde to the extent of 0.018% was detected. This proportion was found to be sufficient to give a typical aniline acetate color reaction. When the test was applied to American oil, a slight color developed, but to a much smaller degree. The details of the test are as follows: The oil (0.1 cc.), measured from a 1 cc. pipette (graduated in 1/100th cc.), is mixed in a test-tube with 5.0 cc. of a 2% solution of freshly redistilled aniline in glacial acetic acid, added from a burette. The reaction mixture is examined in a 1-cm. cell of a Lovibond tintometer (B. D. H. pattern) after an interval of 10 minutes. The reaction mixture must be protected from bright light. Many samples of American oils, Italian oils, French oils, English oils and Japanese oils were subjected to the tests. The results obtained are tabulated.—ANON. *Perf. Ess. Oil Rec.*, 26 (1935), 247. (A. C. DeD.)

Pepsin—Action of, Especially in Pepsin Wine. Methods of evaluation are reviewed especially that of Utkin (*Biochemische Zeitsch.*, (1934), 271). Good results are also obtained by Brandrup's method (*Apoth. Ztg.*, 43 (1928), No. 97). Commercial pepsin wines were tested and it was found that they did not correspond to the standards of the German Phar. VI or contained no pepsin at all.—H. ESCHENBRENNER. *Apoth. Ztg.*, 50 (1935), 795–797. (H. M. B.)

p_H—Conception, Value, and Measurement of. A review.—A. KUFFERATH. *Apoth. Ztg.*, 50 (1935), 348–350. (H. M. B.)

Phenylethylbarbituric Acid—Solubility of, in Ether. One gram of phenylethylbarbituric acid is soluble in 18.6 Gm. of anæsthetic ether and 20.2 Gm. of pure ether (treated with sodium). One gram of the barbiturate will dissolve in 17.4 Gm. and 15.35 Gm. of ether containing respectively, 1% and 2% alcohol.—M. ARQUET. *Bull. sci. pharmacol.*, 42 (1935), 200. (C. T. I.)

Platinum Metals—Microscopic Identification of. The microbehavior of the elements of the platinum group toward many reagents was observed. The group as a whole, with the possible exception of ruthenium and rhodium, shows a predominating tendency to form salts of definite crystalline form which are possibly, in many cases, complex compounds of the Werner type. The triad made up of osmium, iridium and platinum forms an isomorphous series as is apparent in the isomorphous crystals formed with the same reagent in many of the tests. A method for the microscopical analysis of the group was developed. In the analytical method gold

was separated from the rest of the group by extraction with ethyl acetate, and osmium by distillation of the volatile tetroxide. Both separations are rapid and are advantageous in that interferences in testing directly for the presence of the remaining elements will therefore be lessened by the absence of these two. Numerous tables giving a description of the results of the analyses are shown.—W. F. WHITMORE and H. SCHNEIDER. *Mikrochemie*, 17 (1935), 279. (L. L. M.)

Polarization in the Apothecaries' Laboratory. The application of polarization in the examination of medicaments with direct and specific action, and in urine analysis is discussed.—ENGELESLEBEN. *Apoth. Ztg.*, 50 (1935), 538–540. (H. M. B.)

Potassium Bromate—Note on Standard Solutions of. It was found that potassium bromate may be readily purified by recrystallization. The salt is quite stable if kept in brown stoppered bottles and may be weighed directly to make a standard solution.—M. L. YAKOWITZ. *J. Assoc. Official Agr. Chem.*, 18 (1935), 505. (G. S. W.)

Potassium Chlorate and Sodium Chlorate—Determination of Chlorate in. Most pharmacopœias utilize the method of Mohr for the determination of chlorate in potassium chlorate. This method depends upon the liberation of an equivalent quantity of iodine from potassium iodide in the presence of acid, the iodine resulting from the reaction being titrated with thiosulphate. While this method gives satisfactory results if the directions are carefully followed (strong acid must be used) the authors suggest the following method: 0.8 Gm. of potassium chlorate is dissolved in 100 cc. of distilled water. Ten cc. of this solution is mixed with 15 cc. of commercial sulphurous acid (about 6% and free from sulphuric acid and chloride). This solution is boiled for ten minutes, small quantities of distilled water being added from time to time to keep the volume constant. After the sulphur dioxide is evolved the chloride content is determined by the Volhard method using potassium thiocyanate. In this method the potassium chlorate is reduced to potassium chloride while the sulphurous acid is oxidized to sulphuric acid. The principal advantage of the method is the getting away from working with strong acids. Mixtures of chlorate and chloride may be determined by running a chloride determination first, then one after the reaction is complete and determining the chlorate by difference. The method works equally well with sodium chlorate.—A. ENSINK and J. J. HOFMAN. *Pharm. Weekblad*, 72 (1935), 950. (E. H. W.)

Pyramidon—Color Reactions of. It is well known that Pyramidon—in contradistinction to antipyrine—gives a purple color with a variety of mild oxidizing agents. Wagenaar (cf. *J. Am. Pharm. Assoc., Abstract Sect.*, 24 (1935), 171), has suggested potassium persulphate as particularly well adapted to this reaction. The author has comparatively studied the effect of a 5% potassium persulphate solution and a $0.1N$ solution of iodine as oxidants of pyramidon in solutions of various concentrations. He finds: (1) that iodine is much more sensitive, one drop in 5 cc. giving a perceptible purple with 0.01% of pyramidon while potassium persulphate gives no reaction with this concentration. (2) In pyramidon solutions of the same concentration the purple color appears more rapidly with iodine than with potassium persulphate. (3) The purple color obtained with iodine is much more lasting than that obtained with potassium persulphate, the color of the latter changing rather quickly to reddish brown.—N. SCHOORL. *Pharm. Weekblad*, 72 (1935), 669. (E. H. W.)

Quinine Iodobismuthate—New Method for the Quantitative Determination of. The following method is recommended: Dissolve 1 Gm. of quinine iodobismuthate, accurately weighed, in 10 cc. of acetone, add to the solution of 0.9 Gm. of silver nitrate dissolved in 20 cc. of water. Remove the acetone by heating on a water-bath, and add 50 cc. of 95% alcohol. Allow the precipitate to settle completely by setting the mixture aside for several hours, then decant onto a tared Gooch crucible. Wash the precipitate with three 20-cc. portions of 95% alcohol, heating each time on a water-bath and taking care that the lukewarm liquid does not carry any of the precipitate onto the filter. Combine the washings with the filtrate (1st filtrate). Remove the alcohol from the Gooch crucible by drawing through it a current of air, and remove the alcohol from the precipitate in the beaker by adding 10–15 cc. of water and heating on a water-bath almost to dryness. Wash the precipitate with four 10-cc. portions of nitric acid (1:1), each time heating carefully, with agitation, on a water-bath, finally decant the boiling acid liquid onto a Gooch crucible and, after cooling, apply suction. After the fourth washing transfer the entire precipitate to the filter using cold water acidified with several drops of nitric acid, and combine the wash water and the acid liquid (2nd filtrate). Wash the precipitate twice with alcohol and then dry

in an oven first at 100° C. and then at 130° C. to constant weight. The percentage of iodine may be obtained from p′ × 54.05, where p′ equals weight of silver iodide. Evaporate the 1st filtrate to a small volume on a water-bath, removing all the alcohol. Take up the warm residue with 20–25 cc. of water and 4 cc. of *N* sulphuric acid, transfer to a hard glass tube marked at 50 cc. and make up to the mark with the washings from the beaker, using hot water. Filter and take a polarimetric reading using a 2-dm. tube. The percentage of quinine may be obtained from −1.68°:15.5 = p″: x, where p″ is the reading observed expressed in degrees and hundredths of a degree. Quantitatively transfer the 2nd filtrate into a 600-cc. beaker, using water, then add in small portions, with continuous shaking, a cold saturated solution of ammonium carbonate until a persistent precipitate forms (the beaker should be covered with a watch glass to prevent loss), then add a slight excess of the carbonate solution, boil and then collect the precipitate on an ashless filter, washing 4 or 5 times with hot water. Dry and ignite the precipitate in a small tared crucible. The percentage of bismuth may be obtained from p‴ × 89.7, where p‴ is the weight of Bi_2O_3. The results obtained with the above method check those obtained by the method now in use.—LORENZO BRACALONI. *J. pharm. chim.*, 22 (1935), 49–52. (S. W. G.)

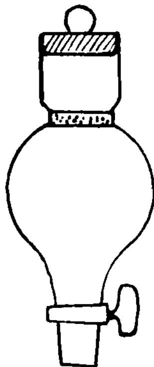
Remedies, Nostrums and Cosmetic Agents—Results of the Examination of. The tests on 47 products are reported.—C. GRIEBEL. *Apoth. Ztg.*, 50 (1935), 848–850. (H. M. B.)

Santonin—Artemisias of Afghanistan Producing. Samples of Artemisia species were subjected to microchemical tests to determine the presence of santonin. Five tests described are: (1) A 0.5-Gm. sample is extracted for 5 minutes with 5 cc. of rectified boiling alcohol. To the extract a small pellet of potassium hydroxide is added and the mixture is shaken and heated cautiously. A carmine color indicates the presence of santonin. (2) A small sample of finely powdered drug is extracted with benzene and then heated over an alcohol flame to volatilize the solvent. One–2 drops of sodium methylate are added—a red-orange color indicates santonin. (3) A microscopic examination for santonin crystals. (4) A 0.5-Gm. sample is shaken with 5 cc. of chloroform, benzene, carbon tetrachloride and alcohol, respectively, for 10 minutes. Each filtrate is then evaporated to dryness. If santonin is present, a deep orange color will be produced after adding 2–3 drops of sodium methylate along the edges of the extracts. (5) Same as (4) except potassium methylate is used in place of sodium methylate. A procedure for a chemical analysis and determination of santonin in Artemisia is as follows: 13 Gm. of coarsely powdered drug is extracted with 65 cc. of water for 15 minutes on a water-bath and then heated with careful stirring for 15 minutes more after adding 25 cc. 4*N* hydrochloric acid. The mixture is cooled and while still tepid is poured into a separatory funnel. After complete cooling, the sample is treated with 13 Gm. of tragacanth and 130 cc. chloroform and shaken for $^1/_2$ hour vigorously. This is set aside for $^1/_2$ hour and shaken at frequent intervals. The chloroform extract (101.5 cc. ≡ Gm. of drug) is separated, filtered and concentrated to 5 cc. The concentrate is heated with 100 cc. of 5% baryta until all chloroform has been removed and a green-yellow resinous precipitate separates. After 5–10 minutes' additional heating the mixture is filtered and the precipitate washed with 100 cc. warm water. The filtrate is made acid to Congo red with dilute hydrochloric acid. This solution is heated at 60–70° for several minutes and further acidified with hydrochloric acid and reheated for 10 minutes more on a water-bath. The product while still warm is poured into a separatory funnel and extracted with 25, 15 and 10 cc. portions of chloroform. The combined extracts are distilled and the dry residue is dissolved in 7.5 Gm. of absolute alcohol and 42.5 cc. of 60–70° water. The solution is refluxed on a water-bath for 15 minutes and then filtered. The flask is washed with 2–5 cc. portions of 15% alcohol (w/w). The cooled filtrate is refluxed with 0.08–0.09 Gm. of infusorial earth for 5–10 minutes. The mixture is filtered and the residue washed with 10 cc. of 15% alcohol. The filtrate is set aside for crystallization and the product, after 24 hours, is recrystallized from 15% alcohol and dried at 100–105°. To the weight of santonin obtained is added 0.046 Gm. for solubility correction. The article includes a short history of investigation on Indian Artemisias.—N. A. QAZILBASH. *Bull. sci. pharmacol.*, 42 (1935), 129. (C. T. I.)

Saponification Number—Determination of, in Oils. Hard fat, soya bean oil, peanut oil, fatty acids, tallow, cotton oil, palm kernel oil and coconut oil were completely saponified with potassium hydroxide in alcoholic solution after 15 minutes' boiling. Saponification of 89–99.5% and 99–100% were obtained after 5 and 10 minutes' boiling, respectively. It is therefore recommended that the boiling time of the saponification mixture in the determination of the saponifica-

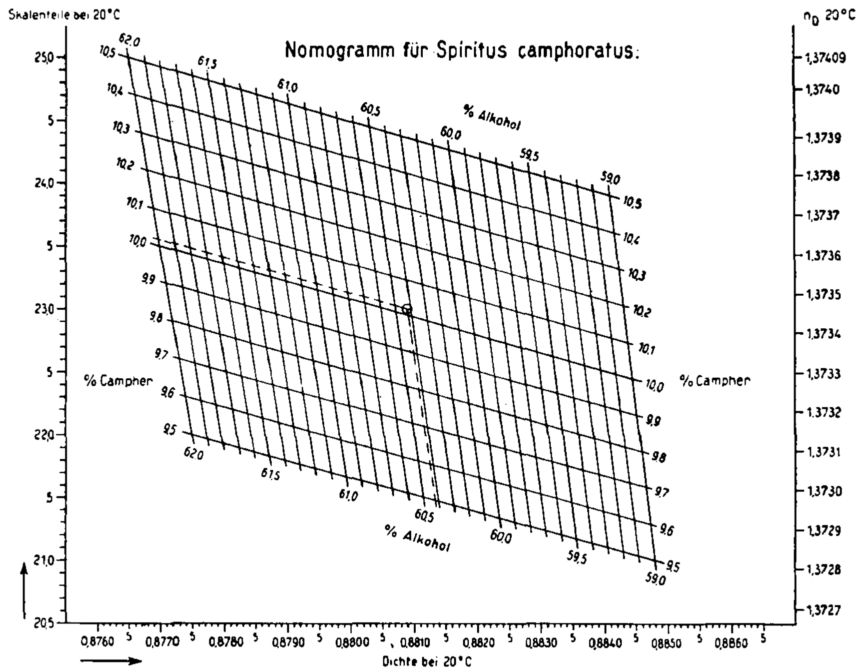
tion number be 15 minutes.—J. HETZER. *Fettchem. Umschau*, 42 (1935), 87; through *Squibb Abstr. Bull.*, 8 (1935), A-938.

Separatory Funnel. A new form of separatory funnel in which a glass filter plate is molded below the stopper. The apparatus is used in such cases where ether Soxhlet extractions remove fat when the ethereal solution will pass through while the filter will retain the fat.—HANS BARSCH. *Pharm. Zentralh.*, 76 (1935), 379. (E. V. S.)



Solution of Aluminum Acetate, G. P. VI. It is concluded that a large number of the solutions of aluminum acetate on the market have not been prepared according to the official directions; that in properly prepared solutions there is but little change to the basic acetate and gel formation is not observed; turbidites and precipitates are unavoidable; it should be dispensed as a clear colorless liquid by filtering with or without the use of a filtering agent; specific gravity decreases with age with but a slight decrease in the basic acetate content. Boric acid was found in only one product. The aluminum content may be determined by the method of Matthes and Schütz (*Pharm. Ztg.*, 43 (1928), 353) or that of Holdermann (*Pharm. Ztg.*, 45 (1930), 1332). Coagulation tests were also carried out. Calcium was determined by the following procedure: Weigh accurately 5 Gm. of the acetate solution, add 1 Gm. of ammonium chloride (instead of 2 Gm. as directed by the Pharmacopœia) and proceed as directed in the German Phar. under the determination of aluminum. Wash the aluminum hydroxide precipitate with 50 Gm. of hot water 5 times by decantation. Heat the united filtrates to boiling and precipitate with boiling ammonium oxalate solution in excess (50 Gm.). After several hours filter off the supernatant liquid and wash the precipitate by repeated decantations with hot water containing ammonium oxalate free from chlorides; dry, and ignite to calcium oxide.—A. HANNER and E. DICKE. *Apoth. Ztg.*, 50 (1935), 713–715. (H. M. B.)

Spirit of Camphor—Refracto-Densimetric Determination of. By means of the illustrated chart the amount of camphor and the percentage of alcohol in the sample may be easily and ac-



curately determined by carrying out a density and a refractive index determination at 20° C.—E. WEBER. *Apoth. Ztg.*, 50 (1935), 642–644. (H. M. B.)

Spirit of Ethyl Nitrite—Preparation and Testing of. H. points out that the directions of the German Phar. for preparing this spirit are deficient for the preparation of a strong product (2.4+ %). Attention is called to the fact that the reducing action of the alcohol on the nitric acid takes place in the warm and that it is of advantage to use a long condenser with an adapter dipping into the alcohol and to use absolute alcohol. The following determination for the minimum content is given: Place 10 cc. of 0.1N silver nitrate in a medicine bottle, rinsed with distilled water, and introduce 10 Gm. of the spirit, 20 Gm. potassium chlorate solution (5%) and 5 Gm. nitric acid (25%). Close the container and allow to stand with frequent vigorous stirring for $^1/_4$ hour. In the filtrate hydrochloric acid does not produce a turbidity or precipitate. This corresponds to a minimum density of 0.835 or 1.88 Gm. of ethyl nitrite in 100 cc. of spirit; a maximum density permitted should be 0.845 equal to 1.9 Gm. of ethyl nitrite.—G. HAMANN. *Apoth. Ztg.*, 50 (1935), 922–923. (H. M. B.)

Strychnine and Quinine—Quantitative Determination of, in Mixtures. The distribution ratios of strychnine and quinine between aqueous hydrochloric acid and chloroform and hydrochloric acid-sodium chloride solutions and chloroform are reported and this information is applied in a study of the methods for separating these alkaloids which have been published by Evers (*Pharm. J.*, 109 (1922), 90), a modification by Evers and Haddock (*Quart. J. Pharm. Pharmacol.*, 4 (1931), 314) adopted by the Brit. Phar., and the method of the Dan. Phar., 1933. The first of these methods employs 2N hydrochloric acid, the two latter methods use 1N hydrochloric acid and saturated, aqueous sodium chloride, mixed 1:1. The distribution coefficient of strychnine between chloroform and 2N hydrochloric acid is approximately 1.0, while that of strychnine between chloroform and the acid-salt mixture is about 3.2 at room temperature. Using ordinary commercial chloroform no constant distribution ratios can be obtained with quinine. This is shown to be due to the content of alcohol present as a stabilizer in such chloroform. Using alcohol-free chloroform constant values are obtained. The distribution of quinine between chloroform and 2N acid is approximately 1.6×10^{-3}, while its distribution between chloroform and the acid-salt mixture is about 3.4×10^{-2}. This information is applied in the assay methods above cited. Thus, assaying Easton tablets by the Dan. Phar. method, after 5 extractions of the aqueous solution of the tablets with one-half volume of chloroform, about 1% of strychnine will remain in the aqueous layer. In the after-extraction of the chloroform extract with acid-salt solution (2 × 5 cc.) about 2.6% of the strychnine enters the aqueous phase. In a single extraction of this with 10 cc. chloroform about 80% is reëxtracted, leaving 0.6% of the strychnine content of the assay specimen. Thus the loss of the alkaloid in process is about 1.0% plus 0.6% or 1.6%. If the process of extracting with anhydrous ether described in the Dan. Phar. is conducted the loss may be brought under 1%. As to quinine, with the use of alcohol-free chloroform in Ever's method about one third as much chloroform is held back in the chloroform layer as when using chloroform which contains alcohol. Using the commercial chloroform in the Dan. Phar. method about 0.028 Gm. of quinine base remains in the strychnine, but the ether extraction removes this. Using alcohol-free chloroform the amount of quinine remaining in the strychnine is lessened. A little strychnine tends to be removed suspended in the wash-ether, this is collected on a filter and returned by dissolving in 10 cc. chloroform. Titrations in alcoholic solution using a methyl red-methylene blue indicator mixture are recommended both for total alkaloid and for the strychnine assay.—F. HALSTROM. *Dansk Tidsskr. Farm.*, 9 (1935), 181. (C. S. L.)

Strychnine Salts—Micromethod for the Identification of. The strychnine salts are heated with a 4% sodium glycerophosphate solution which results in a characteristic crystalline precipitate. By this method small quantities of strychnine sulphate, strychnine nitrate, strychnine phosphate and strychnine glycerophosphate may be identified in mixtures with other alkaloids.—V. KLOBUSITZKY. *Freie Apoth. Stimmen.* (Aug. 17, 1934), 18; through *Pharm. Tijdschr. Nederland.-Indië*, 13 (1935), 56. (E. H. W.)

Sweet Lupines—Chemical Method for Rapid Determination of Alkaloid Content of, for Breeding. Dissolve 0.8 Gm. of potassium iodide and 0.4 Gm. iodine and dilute to 1,000 cc. with distilled water. This solution gives a color reaction with alkaloids when these are present in about 0.1% concentration. For further separation for solutions containing under 0.1% alkaloids, use a stronger solution made up of 1.2 Gm. potassium iodide and 0.6 Gm. iodine and diluted to 1,000 cc. with distilled water.—E. KUNZ and J. HOREL. *Sbornik Ceskoslov. Akad. Zemedelske*, 10 (1935), 95; through *Chem. Abstr.*, 29 (1935), 4520.

Syrup of Ferrous Iodide. The syrup prepared according to the official process does not always meet the requirements of the D A. B. When 42 parts of iodine are used in place of the 41 directed a satisfactory preparation is obtained. Inability to prepare an unobjectionable syrup is attributed to failure to comply rigidly with the D. A. B. VI directions to add the iodine very slowly with constant cooling to the iron-water mixture. The following method employed by the author yields a preparation with the required iodine content: The iron-water mixture is placed in a strong, glass-stoppered flask which is cooled by immersion in ice water. For the preparation of 250 Gm. of syrup the iodine is introduced in 10–12 portions, the flask being kept tightly stoppered between additions.—P. HORKHEIMER. *Pharm. Ztg.*, 80 (1935), 441. (G. E. C.)

Tartaric Acid—New Reaction of. A solution containing 2 Gm. of resorcin and 10 Gm. of potassium bromide dissolved in 100 cc. of distilled water is prepared, and 1 cc. of concentrated sulphuric acid is added. One-tenth cc. of the above reagent, 2 cc. of sulphuric acid and 0.1 cc. of tartaric acid solution are mixed in a tube. The mixture is then placed in a boiling water-bath, and at the end of one minute a pale blue color appears, which becomes deepest at the end of five minutes. If 1 cc. of distilled water is added, the color changes to red, and further dilution only changes the depth of the red color. If the solution is neutralized, the color changes to violet. This reaction is specific for resorcin, as other phenols do not react in this manner. The reaction can be used to identify tartaric acid even in the presence of bromides, nitrites, bromates, and in iron compounds.—MAURICE PESEZ. *J. pharm. chim.*, 21 (1935), 542. (M. M. Z.)

Tollens' Reaction—Use of, in the Analysis of Medicinal Products. Tollens reaction can be applied to a large number of substances. Guaiacol can be determined by it in mixtures with other substances. The optimal quantity is about 0.05 Gm.—R. SAN MARTÍN CASAMADA. *Farm. Moderna*, 46 (1935), 89. (A. E. M.)

Ultraviolet Absorption—New Apparatus for Measuring. The apparatus depends upon the use of a set of 25 quartz plates 0.5 mm. thick, and the comparison of the absorption by the quartz plates and the sample. A table is given showing the values in wave-lengths (A.) and densities for the series of plates. The use of the apparatus is explained in detail and an illustration is given.—R. FABRE and L. AMY. *J. pharm. chim.*, 22 (1935), 5–15. (S. W. G.)

Variation Statistics of Drugs—Contribution to. Three tables accompany the article: one showing the fixed oil content of sweet almonds, one of the fixed oil content of bitter almonds and one giving the amygdalin content of bitter almonds. As a rule, the smaller cotyledons have a higher percentage of oil. In the case of amygdalin content, both cotyledons generally contain about the same amount of glycoside, and the percentage of amygdalin in a single cotyledon does not vary far from the average value. In many cases the fatty oil content of the two cotyledons of bitter almonds varies more than the amygdalin content. The average values for the oil content of the two cotyledons of both sweet and bitter almonds differ by very little.—L. ROSENTHALER. *Scientia Pharm.*, 6 (1935), 79. (M. F. W. D.)

War Gases—Detection of. A discussion of the importance of detecting the presence and nature of war gases, of methods of investigation in the laboratory, of sources of error and of the practical realization of detection in actual warfare.—LUCIEN LEROUX. *14me Congrès de Chimie Industrielle, Paris* (Oct. 21–27, 1934), 7 pp. (A. P.-C.)

Yohimbine—Composition of Commercial. The authors found that a number of commercial preparations of yohimbine consisted principally of a base which possessed the melting point and optical rotation of isoyohimbine.—J. P. WIBAUT and A. J. P. VAN GASTEL. *Rec. trav. chim.* (1935), 85; through *Pharm. Weekblad*, 72 (1935), 879. (E. H. W.)

Zinc—Estimation of, in the Presence of Iron, Aluminum, Uranium, Beryllium and Titanium. III. About 1–7 mg. of potassium zinc sulphate, $K_2SO_4.ZnSO_4.6H_2O$ (double recrystallized), corresponding to 0.15–1 mg. of metallic zinc, were weighed into a microbeaker and dissolved in about 1.5 cc. of water. The necessary amount of any of the foreign elements (Fe, Al, U, Be and Ti), in the form of solution of one of their suitable salts, was added to this solution. Iron must be present in the ferric state, or converted into the latter by oxidation with a few drops of bromine water. 0.4–1 cc. of a 5% sodium tartrate solution was then added to the zinc salt solution and ammonia vapor was blown over its surface until the latter smelt of ammonia. The zinc was now precipitated by adding, drop by drop, to the mixture a solution of quinaldinate (equivalent to 1 Gm. quinaldinic acid per 100 cc. of solution), rotating the beaker occasionally during the addi-

tion of the reagent. 0.2–0.25 cc. of the reagent in excess of the theoretically required amount (0.2–1 cc.) was added in each case. The alkaline tartrate prevents the precipitation of iron, aluminum, etc., by quinaldinic acid. But as zinc quinaldinate is appreciably soluble in alkalies, the excess ammonia was removed by blowing air through a capillary over the surface of the mixture at a temperature of 60° C. The microbeaker was gently warmed on its stand in the water-bath. Temperatures higher than 60° C. must be avoided to prevent reduction of ferric to ferrous iron which at once forms ferrous quinaldinate that precipitates along with or is absorbed by the zinc quinaldinate. As soon as the ammonia was removed, the solution was rapidly cooled, and filtered at once through Emich's asbestos-packed filterstick. The zinc precipitate was washed with hot water and then dried in the Benedetti-Pichler drying apparatus in a current of air at 125° C. Weighings were made in a Kuhlman's balance. The zinc precipitate contains 15.29% zinc according to the formula $Zn(C_{10}H_6NO_2)_2.H_2O$.—P. RÂY and M. K. BOSE. *Mikrochemie*, 18 (1935), 89. (L. L. M.)

Zinc—Microdetermination of, by Means of Anthranilic Acid. A comparative study of the techniques of Pregl and Emich. An excess of 0.3–0.33 cc. of reagent should be used in the assay, the exact amount to be determined by a preliminary test. The use of reagent in excess of this amount affords results about 1% too high.—C. CIMERMAN and P. WENGER. *Mikrochemie*, 18 (1935), 53. (L. L. M.)

TOXICOLOGICAL CHEMISTRY

Coniine and Nicotine—Microchemical Detection of. Coniine may be detected in the urine in cases of poisoning from the alkaloid by the following procedure: About 100 cc. of urine, alkalinized with potassium hydroxide, are extracted twice with 50-cc. portions of ether. The combined ether extracts are shaken 4 times with 1 cc. of 1% hydrochloric acid. The separated aqueous liquid is warmed to expel the ether, transferred to a 50-cc. Erlenmeyer flask and alkalinized with potassium hydroxide. A 1-cm. glass tube, 10 cm. in length and widened at one end to a diameter of 2 cm., is now inserted into the neck of the flask through a tight-fitting stopper. The widened end of the tube is covered with a short microscope slide on the under side of which has been placed a drop of reagent. The latter consists of a saturated solution of picrolonic acid in 20% alcohol. The Ernlenmeyer flask is then placed on a hot-plate at 150–160° C. and the slide is weighted with a 20 Gm. weight. The vapors of the alkaloid form a precipitate in the drop of reagent which is seen to possess crystalline form when viewed under the microscope. The micromelting point of coniine picrolonate thus obtained, after careful washings with water, is 192–194° C. Nicotine picrate melts at 218° C. By this method 50γ of coniine may be detected in 50 Gm. of urine, corresponding to a dilution of 1:1,000,000. This alkaloid may also be detected in tissues by the same method after first extracting the alkalinized tissue with ether. Coniine may be identified also by the micromelting point of coniine hydrochloride sublimate, but the determination cannot be made with quantities less than 200γ. The micromelting point is 218° C. Nicotine may be identified by the use of the apparatus described. In this case the reagent consists of a saturated solution of picric acid.—R. FISCHER and W. PAULUS. *Mikrochemie*, 17 (1935), 356. (L. L. M.)

Ethyl Bromide—Detection of Very Small Quantities of, in Blood and Brain Tissue. Minute quantities of ethyl bromide can be detected in small samples of blood or brain tissue by distilling and passing the distillate over a red hot quartz tube which converts the ethyl bromide into hydrogen bromide. After careful neutralization of the distillate, the bromine is determined colorimetrically by treatment with chloramine and fluorescein which is converted into eosin by the bromine liberated. Experimentally the method was shown to be accurate quantitatively within 5%.—FRIEDRICH L. HAHN. *Compt. rend.*, 201 (1935), 269. (G. W. H.)

PHARMACOGNOSY

VEGETABLE DRUGS

"Chih-Mu"—Anatomy of the Drug. The drug "Chih-Mu" is the dried rhizome of *Anemarrhena asphodeloides* Bunge (*Liliaceæ*). This plant is a native of Northern China and was introduced into Japan between 1715 and 1735. Microscopic studies of this drug may be summarized as follows: *A. asphodeloides* belongs to the monocotyledons in spite of the fact that the vascular bundles are parallel and that there are seldom any lignified cells forming a sheath around

the vascular bundles. The endodermis is generally not well defined. Long, thin bundles of prismatic crystals which are surrounded by a cork-like membrane occur in the intercellular spaces. The oil cells which are occasionally found in the cork layer contain volatile oil and their walls consist of pectinous substances. Starch grains are found only in the tip of the rhizome. The original article contains 11 figures illustrating the anatomy of this drug. An explanatory key is given in the original abstract.—N. FUJITA and M. FUJITA. *J. Pharm. Soc. of Japan*, 55 (1935), 71–72. (R. E. K.)

Compositæ Leaves—V. Pharmacognostic Study of. The article consists of a table containing anatomical characteristics of the following classes of Liguliforæ: Cichorieae-Scolyminae, Cichorieæ-Cichorinæ, Cichorieæ-Leontodontinæ, Cichorieæ-Crepidinæ. The species included are: Scolymus (2), Hymenonema (1), Cichorium (2), Lapsana (2), Scorzonera (1), Zacyntha (1), Rhagadiolus (2), Hypochoeris (3), Arnopogon (1), Leontodon (3), Picris (2), Tragopogon (6), Podospermum (2), Scorzonera (7), Andryala (1), Chondrilla (2), Taraxacum (3), Launaea (2), Microrhynchus (2), Sonchus (5), Lactuca (14), Picridium (1), Crepis (2), Prenanthes (3), Hieracium (8). The table includes a description of the cell wall, cuticle, epidermal cells, stomata, mesophyll and epidermal hairs.—W. HIMMELBAUR and T. KALTSCHMID. *Scientia Pharm.*, 6 (1935), 69. (M. F. W. D.)

Karkade. There has come onto the market in comparatively recent times in Switzerland a drug under the name of karkade. The drug is an annual shrub *Hibiscus Sabdariffa*, native to tropical America and cultivated in the East Indies, Java, Sumatra, Ceylon, tropical Africa and the West Indies. The leaves may be used as a salad, medicinally for various conditions of inflammation, or as a coagulation medium for rubber. The seeds when roasted can be used as food. The calyx, which becomes fleshy when the fruit is ripe, is used in many ways for food and drinks. It has gentle laxative properties. The paper gives gross and microscopic description of the calyx and epicalyx and of the epidermal hairs. Microphotographs accompany the description. A short chemical investigation is made and indicates the presence of oxalic, malic, citric and tartaric acids which provide the taste.—K. LEUPIN. *Pharm. Acta Helv.*, 10 (1935), 138. (M. F. W. D.)

Microscopic Mounts—Permanent Aqueous. Specimens which must be kept in aqueous mountings may be preserved by sealing the edge of the cover slip with a melted wax made as follows: Heat anhydrous wool fat with not more than 20% rosin, until the constituents are blended. The mixture is firm at ordinary temperatures but becomes liquid on heating. The slide and cover slip should be dry when the wax is applied, and the wax must come on top of the slip all the way around.—H. R. SMITH. *Ind. Eng. Chem., Anal. Edit.*, 7 (1935), 286. (E. G. V.)

Myrrh from Kenya. A sample from the Mandera district, a sample from the Wajin district and a 5-cwt. trial consignment all met the requirements of the B. P.—ANON. *Bull. Imp. Inst.*, 33 (1935), 134–136. (A. P.-C.)

Sandalwoods—Structure of Some. A discussion of a paper which appeared in the "Bulletin of Miscellaneous Information" (Kew), No. 4, 1935, by Dr. C. R. Metcalfe, describing the structure of the following Santalaceæ: *Santalum album*, East Indian sandalwood, *S. freycinetianum* Gaud., Hawaiian sandalwood, *S. austro-caledonicum* Vieill., New Caledonian sandalwood, *S. Yasi* Seem., Fiji sandalwood, *Eucarya spicata* and *E. acuminata* Sprague et Summerhayes, which yield Australian sandalwood, and *Exocarpus latifloius* R. Br., also from Australia is given. The author states that no reliable macroscopic characters have been found whereby it is possible to distinguish the wood of *Santalum album* from the various local sandalwoods belonging to the same genus. The microscopical differences between the genera Santalum and Eucarya, do not appear to be more clearly defined than those existing between the various species within the genus Santalum. The wood structure of all the Santalaceæ examined is remarkably similar. *S. album* is easy to identify on account of its taller rays, and the structure of *Eucayra spicata* cannot readily be confused with that of *S. album*. A table is given which shows the more important measurable characters by which the species may be distinguished. A short discussion of each species is also included.—ANON. *Perf. Ess. Oil Rec.*, 26 (1935), 244. (A. C. DeD.)

Scrophularia Species—Pharmacognosy of the Roots and Rhizomes of Two. The Chinese drug "Hsiian-shen" is said to be derived from **Scrophularia Oldhami** Oliv. Comparison of the fresh roots with those obtained in commerce corroborated this statement. The fresh root reaches 10 cm. in length, 2 cm. in thickness and is somewhat spindle shaped. The drug is dark or blackish

brown, dark colored within, tastes sweet at first and then rather bitter. The epidermis of the thick tubers changes into a metadermis. The outer layers contain thickened, mottled, stone cells, either singly or in clusters. The radially elongated woody portions consist of both parenchyma and scattered lignified tubules, frequently accompanied by wood fibers. There is no core at the center of the root. Starch and crystals are entirely absent, inulin occurs in the parenchyma cells. The underground portions of **S. Patriniana** Wydl. (—**S. Duplicato-Serrata** Makino) consist largely of rhizomes which have no stone cells in the outer bark and a core of parenchyma tissue at the center of the rhizome. The anatomical details as well as external appearances of the two drugs are portrayed in 13 figures.—T. MUNESADA. *J. Pharm. Soc. Japan,* 54 (1934), 41–48.

Star Anise—Poisonous Species of. H. finds that commercial samples of genuine star anise (*Illicium verum* Hook, fil) contain also the fruits of the poisonous Japanese star anise (Shikimi, *Illicium anisatum* L., *Illicium religiosum* Sieb. and Zucc.) and that this adulterant may be detected by cooking cross sections through the columella or the fruit stalks in chloral hydrate and adding phloroglucinhydrochloric acid. In the genuine anise appear red-colored astrosclereids; these are lacking in the poisonous fruits. In this manner 20–50% admixtures may be detected.—HAHMANN. *Apoth. Ztg.*, 50 (1935), 335. (H. M. B.)

Viscum Album L. A review of the history, botanical, pharmacognostical, chemical and pharmacological studies of this ancient drug.—R. KRESS. *Apoth. Ztg.*, 50 (1935), 453–455. (H. M. B.)

ANIMAL DRUGS

Endocrine Glands—Microscopy of Powdered Desiccated. A study has been made of the microscopy of certain powdered desiccated glands with a view of providing standards for them. An abstract of a paper presented before Section N3, American Association for the Advancement of Science, at the Minneapolis meeting, June 27, 1935, is given. The descriptive microscopical standards are given for thyroid, suprarenal, whole pituitary, anterior pituitary, posterior pituitary, ovary, ovarian residue and corpus luteum.—HEBER W. YOUNGKEN. *J. Am. Pharm. Assoc.*, 24 (1935), 576. (Z. M. C.)

PHARMACY

GALENICAL

Atropine Eye Ointments—Deterioration of, on Storage. A thorough investigation of eye ointments containing atropine was made with a view to determining the effect of storage on their alkaloidal content. Results of the investigation are given in table form. Atropine Eye Ointment, B. P. C. 1923, was found to lose strength when stored in collapsible tubes, although the loss was only 4.8% compared with 16.3% in the case of the material in capsules. The rate of deterioration diminishes very considerably after about a month. Yellow Eye Ointment with Atropine, B. P. C. 1923, showed the most marked loss of atropine. The strength of the material in glycero-gelatin capsules falls more quickly than the portions stored in collapsible tubes and jars. The recorded alkaloidal deficiency is startling, the capsules losing 24.5% in twenty days and 89.5% in 189 days, the corresponding figures for the ointment stored in tubes being 15.2% and 86.4%. The deterioration of Eye Ointment of Atropine with Mercuric Oxide, B. P. 1932, is not affected by the capsule material. In the first month after manufacture the loss of atropine amounts to 20.5%, and during the full period of observation the loss was 32.8%. Iodoform and Atropine Eye Ointment, B. P. C. 1934, maintained its alkaloidal strength best, the loss after storage for 189 days being 4.2%. The mode of packing did not influence the results.—N. L. ALLPORT. *Pharm. J.*, 135 (1935), 4. (W. B. B.)

Cinchona and Belladonna—Percolation of. Rate of Alkaloidal Extraction and Effect of Degree of Comminution. It was found that moderately fine powder shows most rapid extraction of total solids, and also gives quicker extraction of alkaloids of cinchona. Also, in the case of cinchona, the inert material is extracted more rapidly than the alkaloids. After extraction of 50-cc. fractions of percolates from belladonna root it was apparent that an optimum degree of comminution exists for the extraction of total solids of belladonna root, namely, a 44–85 powder.—A. W. BULL. *Pharm. J.*, 134 (1935), 792. (W. B. B.)

Crude Drug Extraction. In a continuation of a previous work C. discusses the extraction of crude drugs and states that the following factors determine the design of equipment to be used: (1) rate of penetration which is a function of the porosity of the drug and the size of the particles involved. This is accomplished by soaking or maceration, mixing, pressure and vacuum, (2) rate of solution which is influenced by mixing, heat, vacuum and pressure, (3) rate of diffusion which depends upon the structure of the cell wall, temperature and pressure of the menstruum, centrifugal force and pressure, (4) rate of separation is accelerated by pressure and centrifuging; pressure by means of steam or air or by presses appears to be the most efficient in accomplishing this end and (5) rate of concentration. The Stokes vacuum process and the Scott process of extraction are discussed.—*Drug and Cosmetic Ind.*, 37 (1935), 36–38. (H. M. B.)

Digitalis Tincture—Preparation of. Relative Merits of Maceration and Percolation. The full activity of digitalis leaf is readily extracted by a maceration process, a period of two days with occasional shaking being as effective as the official percolation process, although the latter gives a higher total solids figure. As percolation can yield varying results in the hands of different workers, particularly on a small scale, it is suggested that the official process should be changed to the simpler process of maceration which would facilitate the preparation of small quantities of the tincture.—H. BERRY and H. DAVIS. *Pharm. J.*, 135 (1935), 7. (W. B. B.)

Dilutions—Preparation of, according to the Homeopathic Pharmacopeia. The author laments the fact that the prescribed methods are not accurate for the preparation of high dilutions. Preference is given to the so-called one glass method for preparing high dilutions of liquids.—H. NEUHEBAUER. *Pharm. Zentralh.*, 76 (1935), 405. (E. V. S.)

Distilled Water, Sterile—Apparatus for the Preparation of. Since the full significance of the *Aqua destillata sterillisata* of the Swiss Phar. V is not appreciated by the pharmacists, the justification for this preparation is set forth and its importance stressed. Three types of small scale distilled water apparatus previously described in detail are compared from the standpoint of ease of operation and cleansing, degree of efficiency, quality of the product based on the Swiss Phar. V requirements and economy of operation. It is shown that the Kontadest-Apparatus (Büchi, *Schweiz. Apoth.-Ztg.*, 72 (1934), 61) yields a distilled water meeting the pharmacopœial requirements at the lowest cost per liter.—BÜCHI. *Schweiz. Apoth.-Ztg.*, 73 (1935), 397, 417. (M. F. W. D.)

Dried Extracts of the New Swiss Pharmacopœia—Hygroscopicity of. The new pharmacopœia has substituted dry extracts for the classical soft extracts to a greater degree than before. The following is the degree of hygroscopicity of the dry extracts as given by the pharmacopœia: only slightly hygroscopic: extract of aloes; somewhat hygroscopic: extracts of opium, cinchona; *Rhamnus purshiana*, hyoscyamus, *Rhamnus cathartica*, and valerian; hygroscopic, extracts of belladonna, digitalis, gentian, rhubarb, strychnine, cola, ipecac, ergot and oxgall. Since the purpose of the paper is to show the result of exposure of the extracts to the air under conditions prevailing in the pharmacy, the only factors taken into account were those of time of exposure and moisture absorbed. The sample of extract was transferred to a tared bottle and weighed accurately. The bottle was then opened for one minute, closed and reweighed. The process was repeated leaving the bottle open for periods of 3 to 10 minutes in the author's pharmacy. A similar series was carried out in a laboratory very close to the seashore, the bottle being opened for periods of 2, 5, 15 and 60 minutes, and lastly for 24 hours. The results were tabulated. The amounts of moisture found to be absorbed do not correspond so well to the terms applied by the pharmacopœia. Of practical importance to the pharmacist is the fact that the absorption of moisture during the first minute is quite rapid for extracts of digitalis and opium. Another point of interest is that all the extracts continue to absorb moisture for a long time, so that a preparation will be cut down in potency by dilution and probable alteration as a result of moisture.—C. BÉGUIN. *Pharm. Acta Helv.*, 10 (1935), 131. (M. F. W. D.)

Drug Extraction. III. Function of Preliminary Maceration in Relation to the Percolation of Belladonna Root. A historical review shows by tabulation the changes made by the U. S. P. since 1840. Various suggestions made by investigators since 1833 are briefly reviewed. Comparative percolations were carried out, using the U. S. P. process somewhat modified and with varying amounts of liquid used for moisteneing. Tables show the amount of alkaloid obtained, amount of extractive and per cent of total alkaloid. Rate of extraction was found to be equally rapid whether moistened with 25 cc. or not. Increase in moistening liquid reduced yield in first

percolate but in all cases it was all contained in the first 280 cc. of percolate. Another experiment varied the time. Again, alkaloid, total extraction and per cent of alkaloid are shown and results indicate that maceration before or after packing is of no appreciable value in promoting rapid extraction. Preliminary maceration was varied and results indicate no particular advantage. In discussing results the author compares them with the findings of other investigators. In the case of powdered drugs that swell only slightly in the menstruum used, apparently the preliminary maceration serves no useful purpose. As to quantity of liquid used, similar results have been found with other drugs. Previous workers have not explained why increase in liquid decreases rate of extraction. Several factors seem to have a bearing. When no liquid is used, all of the reserve percolate must have traversed the entire column of drug. When moistened before packing, most of the first percolate has not traversed the entire column of drug. With 90 cc. of moistening liquid and 80 cc. of reserve percolate, more than 10 cc. of the moistening liquid remains and it has traversed the greatest distance. With 25 cc. of moistening liquid, extraction was as good as when the drug was packed dry, indicating that small quantities may become rather fully saturated. When more liquid is used than can become saturated, the reserve is less concentrated. Maceration after liquid begins to drop is of little benefit in percolation of belladonna, casting doubt on the wisdom of the 48-hour maceration period of U. S. P. X for fluidextracts. Saving of time is important. Perhaps the U. S. Pharmacopœial Revision Committee might introduce a type process for percolation without maceration either before or after packing. The process could be specified for drugs that can be extracted as well without as with maceration.—WILLIAM J. HUSA and S. B. YATES. *J. Am. Pharm. Assoc.*, 24 (1935), 538. (Z. M. C.)

Drug Extraction. IV. Effect of Variation in Solvents on the Extraction of Jalap. Jalap was selected as a typical resin-containing drug and the effect of solvents studied in relation to swelling, penetration, inhibition and extraction. Thin strips of jalap tissue were measured before and after addition of solvents. Swelling equilibrium was attained during the first minute in water but not for 40 or more minutes in alcohol. Testing penetration on blocks, it was found to be rapid during the first hour. Between three and nine hours there was a sharp drop in weight probably due to loss of soluble constituents, followed by an increase. Swelling of blocks in water reached a maximum of 31% in nine hours. Alcohol caused little swelling and glycerin caused slight shrinkage. Extraction of resin was as complete in 15 minutes as in 24 hours but total extraction increased with time. Alcohol 4 volumes, water one volume extracted less resin than more alcoholic mixtures. Alcohol of U. S. P. strength and absolute alcohol seem to be best solvents for extraction of jalap resin; with more water more inert extractive is obtained.—WILLIAM J. HUSA and PAUL FEHDER. *J. Am. Pharm. Assoc.*, 24 (1935), 619. (Z. M. C.)

Emulsions. Experiments described in this paper indicate that, as a general rule, the homogenization of an emulsion containing more than 74% by volume of disperse phase leads to a partial breakdown, and accordingly emulsions which are to receive this treatment should not contain a larger percentage of disperse phase than 74. On the other hand, if the emulsion is prepared by agitation alone, then the percentage of disperse phase should be at least 74 by volume, otherwise it is very probable that creaming would occur. Very stable non-creaming emulsions are possible if the continuous phase can be induced to set to a jelly-like form by the addition of a substance such as gelatin.—J. B. PARKE. *Pharm. J.*, 135 (1935), 8. (W. B. B.)

Emulsions—Pharmaceutical. An extensive review of the theories of emulsion formation is offered. Three series of experiments were conducted (1) Cod liver oil emulsion of the German Phar. VI in which the proportions of gum, tragacanth, lime, calcium hypophosphite and benzaldehyde remain unchanged and the cod liver oil (200–300 parts), cinnamon water (255–383 parts) and glycerin (38–57 parts) were altered; these varied emulsions were prepared according to the pharmacopœial directions and after five hours were pressed twice through a small hand homogenizer and again in 18 hours; they were then observed at the end of 8 and 14 days and 7 weeks. Those containing more than 250 parts of oil, 320 parts of cinnamon water and 47 parts of glycerin separated after 7 weeks. (2) Cod liver oil emulsions were made according to the following directions: Allow all of the tragacanth, which should be of the best quality to swell with 200 Gm. water in a wide-necked flask for 2 days at room temperature (15–20° C.), then pour through a single layer of gauze; rub all portions remaining behind in a mortar with some water, pour through the gauze, wash with water and add to the freshly prepared gum-mucilage. Dissolve the hypophosphite in 150 Gm. water, mix in a large flask with the lime water and the gum-tragacanth mixture, bring

to 590 Gm. with water and shake for 1/2 hour. In 50 Gm. of the cod liver oil, dissolve the vanillin by gentle warming on a water-bath to 40° C., add peppermint oil and benzaldehyde and shake with the previous mixture. Add the remainder of the oil in six portions with shaking. After standing for 3 hours add nipagin dissolved in a mixture of alcohol and tinctures, and mix vigorously. Ten emulsions were prepared with oil varying in amounts from 200–300 parts, lime water 25–38, salts 4.5–6.7, water 255–395, aromatic nipagin 6.0–9.0. These were treated after 5 and 18 hours as under (1). In every case an emulsion was prepared which settled but slightly after six weeks. (3) Saponin, tylose (6–7 Gm. tylose S 400 to 300–350 Gm. water) and pectin emulsions were also studied.—W. KERN, A. BÜCHNER, W. LEOPOLD and H. MOMSEN. *Apoth. Ztg.*, 50 (1935), 691–697. (H. M. B.)

Ergot—Effect of Hot Solvents on. Note on Effect of Storage on Activity of Ergot. The effect of hot solvents on original ergot has been investigated. Ether, dichlorethylene, trichlorethylene and benzene extract the major portion of the alkaloids; light petroleum does not extract the alkaloids. In the case of dichlorethylene and benzene, quantitative recovery of the alkaloids has been made, proving that the alkaloids are extracted and not destroyed by the solvents. *Ergota Præparata* has been found to retain its alkaloidal strength over a period of eighteen months.—R. F. CORRAN and F. E. RYMILL. *Pharm. J.*, 134 (1935), 782. (W. B. B.)

Extract of Belladonna—Preparation of Dry. The following directions are offered for the preparation of this extract: (1) *By Maceration.*—Extract 500 Gm. of the leaves with 4,000 Gm. of diluted alcohol, express (yield about 3,800 Gm.), evaporate the liquid extract to 1,200 Gm., add 1,200 Gm. water to precipitate the chlorophyll; evaporate 5,000 Gm. of the extract in a vacuum and dilute the dry extract if necessary by dextrin. Yield of alkaloids by this method is not very great. (2) *By Percolation.*—Moisten 500 Gm. of coarsely powdered leaves with 200 Gm. diluted alcohol, percolate with diluted alcohol (as directed in the Swiss Phar.) (800 cc.) and 1,000 Gm. water, collecting 1,000 Gm. of percolate, evaporate in a vacuum to 300 Gm. and add 300 Gm. of water to precipitate the chlorophyll and proceed as in (1).—W. BRANDRUP. *Apoth. Ztg.*, 50 (1935), 921–922. (H. M. B.)

Extract of Ipecac—Preparation of Aqueous. A comprehensive review of the work performed shows that the alkaloidal content of the finished preparation depends upon the following factors: degree of fineness of the drug, the concentration of the infusion and the use of an acid to aid in the extraction of the alkaloids. The acids used are either hydrochloric or citric acids. The results and findings of the various authors are tabulated in fifteen tables.—F. GSTIRNER. *Pharm. Zentralh.*, 76 (1935), 421, 437. (E. V. S.)

Fluidextract of Ergot. Fluidextract of *Secalis cornuti* is a pharmacopœial product frequently employed therapeutically. Of the various methods employed in the preparation of this fluidextract the least logical is that given in the Russian Phar. The most rational method of preparation of this fluidextract is that given by the American Phar., since this method gives a maximal extraction of the alkaloids. One of the favorable factors of stability of alkaloids and fluidextracts is a proper concentration of the hydrogen ions; a medium of a p_H of 3–4 is most satisfactory. The fluidextract must be kept at a temperature of 15–20° C. in one-ounce bottles, of a brown color, tightly covered with oiled paper to prevent the entrance of air. When the stopper is removed for a sufficient length of time the alkaloid content is rapidly decreased. The fluidextract loses 50–60% of its alkaloids after having been kept under the usual conditions of a drug store over 6 months. Fluidextracts are unstable products under usual conditions.—G. Y. TROPP. *Soviets. Pharm.*, 3 (1935), 23. (A. S.)

Gauze Containing Yatren—Sterilization of. This type of gauze may be sterilized in an autoclave at 120° C. for 15 minutes. Laboratory tests show that Yatren when heated to 130° C. for a long time shows no appreciable change; at 135° C. sintering begins with no sharp melting point; at 140° C. an orange to gray-yellow color appears; at 170° C. the preparation becomes darker changing gradually at 200° C. to a dark gray-brown color with no splitting off of iodine. The following method is offered for the determination of the Yatren content: Boil 2–3 Gm. of gauze with 100 cc. water and 10 cc. of normal alkali, filter and boil twice with water. Add sufficient 1% potassium permanganate so that an excess remains after 15 minutes' boiling. The excess is then reduced by some drops of alcohol. Pour the cooled liquid into a 250-cc. graduated flask, make to mark and filter through a double filter discarding the first portions of the filtrate. Acidify 200 cc. of the clear filtrate with dilute sulphuric acid, add 10 cc. potassium iodide solution and ti-

trate the iodine with 0.1N sodium thiosulphate. 1 cc. 0.1N $Na_2S_2O_3$ = $\frac{0.0127}{6}$ Gm. iodine.—*Muntsch. Apoth. Ztg.*, 50 (1935), 574–575. (H. M. B.)

Glass—Color of, Used to Protect from Light. Various types and colors of colored glass containers are tested by filling with a potassium iodide solution acidified with sulphuric acid which is then exposed one-half hour to ultraviolet light from a quartz lamp or one hour to direct sunlight, and the freed iodine is titrated. The results are tabulated. The red and brown glasses protect the solution best.—A. JERMSTAD and O. OSTBY. *Arch. Pharm. og Chemi*, 42 (1935), 463. (C. S. L.)

Glycols—Some Properties of, with Special Reference to the Use of Propylene Glycol as a Solvent in Pharmaceutical Preparations. The glycols, as a class of solvents, have developed rapidly during the last few years, but for pharmaceutical purposes have been almost completely ignored. The reason for this is due to the probable high toxicity of these compounds. It has been shown that diethylene dioxide is definitely toxic and ethylene glycol very probably gives rise in the human body to diethylene dioxide. Propylene glycol is less toxic than ethylene glycol, is probably innocuous and was therefore chosen for experiments demonstrating the following: suitability of propylene glycol in other solvents, solubility of alkaloids, solubility of inorganic substances. Some dyes (Bordeaux B, brilliant green, methylene blue, orange G, tartrazine, trypan blue) were found to be reasonably soluble. Simple syrup and a number of concentrated solutions for syrups were miscible in all proportions. Arachis, linseed, olive and persic oils were immiscible; castor oil, especially at very high temperatures, was partly miscible. Volatile oils and some tinctures were partly immiscible in varying degrees, depending on the oil.—C. L. M. BROWN. *Pharm. J.*, 134 (1935), 794. (W. B. B.)

Homeopathic Pharmacy—Regarding the Progress of. A review which includes a comparative discussion of some of the methods employed. The original references are given.—W. PEYER. *Pharm. Zentralh.*, 76 (1935), 407. (E. V. S.)

Hydrogen Peroxide Solution—Stability of. Experiments show that the distilled water used in making the dilute solution of hydrogen peroxide should not be prepared in metallic apparatus. Hydrogen peroxide solutions prepared with such distilled water deteriorate rapidly. The distilled water required in the preparation should be made by use of glass apparatus. The kind of glass used in preserving the solution has very little influence on the stability.—P. HORKHEIMER. *Pharm. Ztg.*, 80 (1935), 507. (G. E. C.)

Solutions—Sterile, Preparation of. II. A complete investigation of the Tyndallization process for preparing sterile solutions has been made and the results show that the principles underlying the process are seriously at fault. It is suggested that the Tyndallization process be considerably modified if it is to be retained as an official process. The germicidal action of some common medicaments on *S. aureus* has been investigated and it is evident that many medicaments used in the preparation of solutions for parenteral injections possess some germicidal activity at atmospheric temperatures. An investigation of the effect of some common preservatives on *S. aureus* has been made. Merthiolate is shown to be the most powerful germicide, a solution 1–100,000 being sufficient to kill the organism within thirty minutes.—H. DAVIS. *Pharm. J.*, 134 (1935), 788. (W. B. B.)

Sterilization—Remarks on the Swiss Phar. V. Chapter on. The Swiss Phar. V specifies under the various solutions for injection the method of steiilizing the filled ampuls. However, while requiring the ampuls to be sterile before filling, it does not specify the method. No sterilization is required for ampuls of adrenaline hydrochloride solution.—J. B. L. *Schweiz. Apoth.-Ztg.*, 73 (1935), 474. (M. F. W. D.)

Sterilization in the Swiss Phar. V—Note on an Article on. The article is comment upon one appearing earlier in the journal and points out a misinterpretation of the pharmacopœial directions. The Swiss Phar. V makes a provision for the sterilization of porcelain and glass apparatus in general, under which ampuls are expected to fall. The neglect to specify sterilization for ampuls of solution of adrenaline hydrochloride is not important, since the solution is directed to be prepared under aseptic conditions and, as has been proven, is sterile.—J. THOMANN. *Schweiz. Apoth.-Ztg.*, 73 (1935), 534. (M. F. W. D.)

Sterilization Methods in Pharmaceutical Instruction and Operations—Physiological-Chemical Investigations and. A review of sterilization methods of the Swiss Phar., the work of

Konrich with regard to killing of organisms with heat under pressure, that of Eschenbrenner concerning the use of the esters of *p*-hydroxybenzoic acid, ultrafiltration and Katadyn methods. A table gives the most satisfactory of the Swiss methods for the sterilization of 64 substances and their preparations.—W. KERN. *Apoth. Ztg.*, 50 (1935), 916–921. (H. M. B.)

Tablet Granules—Method for the Preparation of. A granulation technique is described which is stated to yield superior granules for tabletting. This depends on shaking rather than pressing the dampened clumps through a coarse sieve. Working directions for the preparation of *Tablettæ Bromisovali* are cited as an example. The method is described in greater detail in *Farm. Tidende*, 45 (1935), 201. The mean error of distribution of the active principle in a batch of tablets is found to be less than that of tablets made by the press granulation method.—K. NIELSEN. *Dansk. Tidsskr. Farm.*, 9 (1935), 174. (C. S. L.)

Tablets—Hints and Formulæ for Making, on a Small Scale. The chief troubles experienced in the preparation of compressed tablets are "capping," "sticking" and "picking." Capping is the term applied when the upper surface of the tablet splits off. The cause is usually that of excess powder in the granulation and may sometimes necessitate regranulation. Picking is the adherence of granules to the face of the punch and occurs from a granulation which is not quite dry, or from a scratched punch. The top punch is more usually affected, and the face should be smoothed with a portion of well-used, fine emery cloth and a trace of oil. Sticking is the effect produced when the bottom punch binds in the die, and may be caused by a slightly damp granulation or excess of powder. This sometimes is caused by substances such as calcium lactate, and may be overcome by using 4% of talc as a lubricant and placing one or two drops of liquid paraffin in the die, working the machine for a minute and removing excess of grease before compression. The degree of depression will vary with different types of tablets. Tablets such as potassium chlorate, soda-mint and formamint which are required to be dissolved in the mouth are usually compressed as hard as possible. Precautions must be taken with granulations of deliquescent or hygroscopic ingredients, such as thyroid, and there should be no delay between drying and compression. In regard to shape and size of the tablet, the thickness should be at least $^1/_3$ of the diameter. Formulæ are given for the manufacturing of the following tablets: aspirin, phenacetin and caffeine, saccharin, soda-mint, calcium lactate and aloes, nux vomica and belladonna.—H. DAVIS and F. H. GILLETT. *Australasian J. Pharm.*, 16 (1935), 380. (T. G. W.)

Tinctures of B. P. C. 1934—Alcohol Content and Specific Gravities of. In the B. P. C. 1934, limits are given for the alcoholic content of tinctures. As these limits may be used as legal standards it is desirable that they should be in accordance with samples prepared on a manufacturing scale. Figures which are recorded in a table show that the alcoholic content of the majority of the tinctures is well within the limits allowed by the B. P. C., and in most cases approaches the higher limit. Specific gravities of these tinctures are also recorded in the table.—C. T. BENNETT and F. C. L. BATEMAN. *Pharm. J.*, 135 (1935), 796. (W. B. B.)

Wax-Paraffin Ampuls—Use of, for Silver Nitrate Solution Used in Prevention of Ophthalmia Neomatorum. The ampul is lined with paraffin by dipping the spindles in a mixture containing 71% beeswax, 8% paraffin oil and 21% of a 56° C. melting point paraffin. Silver nitrate in this paraffin lined ampul will probably retain the characteristics of a fresh solution in the new ampuls for from 10 to 12 times as long at the relatively high temperature of 37.5° C. as does the silver nitrate solution in the old type of ampul and will be distributed with an expiration date of 1 year, instead of the present 6 months' dating.—W. E. BUNNEY. *Am. J. Pub. Health*, 25 (1935), 813. (A. H. B.)

Zephirol-Bayer—Use of, in Pharmaceutical Practice. Zephirol is an aqueous solution of a mixed high-molecular alkyl-dimethylbenzyl ammonium chloride. Several investigations were carried out to determine the usefulness of Zephirol pharmaceutically. Immersion in a 1% aqueous solution sterilized spore-free apparatus such as filter plates, etc., on standing with them for one hour; however, longer periods of immersion rendered the filter plate hard and brittle. More concentrated solutions produce this result more rapidly. Spore-bearing material resisted a 1% solution at room temperature, but on heating to 100° C. for 30 minutes was rendered sterile. Immersion for 15 to 30 minutes in a 10% solution of Zephirol, containing 1% of soda to prevent rusting, was sufficient to sterilize surgical instruments. The 10% solution had a greater sterilizing capacity than Sapo formaldehydratus of the Swiss Phar. V.—J. THOMANN. *Pharm. Acta Helv.*, 10 (1935), 117. (M. F. W. D.)

Pharmacopœias and Formularies

Codex and Public Pharmacy. The public pharmacist may rightly claim some credit for the acceptance of the Codex as an authoritative work of reference, for he was one of the first to adopt the Codex standards in contracts for unofficial preparations. In the formulary section the use of "g" for gram is a serious mistake, and the pharmacopœial recommendation of "G" received a warm welcome from all pharmacists who have to dispense in both systems of weights. The emulsion of petrolatum and agar is a good example of a vicious circle. The medical man who accepts the Codex as a standard wants to prescribe it because it is in the Codex, while the Codex apparently includes it because doctors ask for a formula combining the effects of agar and paraffin.—W. A. Knight. *Pharm. J.*, 134 (1935), 88. (W. B. B.)

Dutch Pharmacopœias. This splendid article is of considerable historical interest. The national pharmacopœias of the world are listed in chronological order as to their appearance, and dates of the latest edition of each are given. The history of the various Dutch pharmacopœias is then discussed. The first official pharmacopœia to appear in Holland was the Pharmacopœia Amstelodamensis appearing in Amsterdam in 1636. Other important municipal pharmacopœias are: Pharmacopœia Leidensis 1638; Pharmacopœia Ultrajectina 1656; Pharmacopœia Hagiensis 1659; Pharmacopœia Leovardiensis 1687; Pharmacopœia Harlemensis 1693; Pharmacopœia Dordracena 1709; Pharmacopœia Roterodamensis 1709; Pharmacopœia Almeriana 1723 and Pharmacopœia Groningana 1729. Many of the formulas appearing in the old Dutch pharmacopœias are discussed both comparatively and in connection with the period at which they appeared. The article concludes with a table of 114 pharmacopœias published in Holland between the Amsterdam Pharmacopœia of 1636 and the Pharmacopœia Batava of 1805.—W. F. Daems. *Pharm. Weekblad*, 72 (1935), 1078. (E. H. W.)

Liver Extracts. The proposed U. S. P. monograph on liver extracts, Circular 465 U. S. P. Revision Committee, was considered and the unanimous opinion arrived at that liver extract should not be included in U. S. P. XI until such time as a satisfactory animal or chemical assay is available.—Subcommittee on Digestive Ferments and Glandular Products.—Am. Drug Manufacturers Assoc., Proceedings (1935) 197; through *Squibb Abstract Bull.*, 8 (1935), A-1376.

Swiss Pharmacopœia—Remarks on the New. The author makes some comments on various preparations and tests of the Swiss Phar. V. The points commented upon are: the definition of unsaponifiable matter, purity test for salicylic acid, identity test for tartaric acid, identity test under agar, test under solution of aluminum aceto-tartaric acid, identity reaction of antipyrine, lack of assay for orange flower water, identity test for codeine phosphate, identity test under compresses of iodine, assay of solution of formaldehyde, identity test for mercuric oxycyanide, requirements under white precipitate of mercury, the title of solution of potassium arsenite, detection of benzonaphthol and the assay of rhubarb.—L. Rosenthaler. *Schweiz. Apoth.-Ztg.*, 73 (1935), 469. (M. F. W. D.)

Surgical Dressings—Criticisms of Some Codex Standards for. In the new Codex, general instruction regarding sterilization has been included under unmedicated gauze (and under the ribbon gauze) for some reason and not in the other monographs. This instruction could well be dispensed with, as most dressings can be sterilized by the same means. Similarly, the sentence "Aseptic absorbent gauze is absorbent gauze in a sterile condition" is a curious inclusion, the exact purpose of which is not clear. If the monograph is intended to describe sterile, in addition to the ordinary gauze, the phrasing requires separation as formerly. A fuller description of a container which could maintain the sterile condition would be illuminating. Euflavine, mercuric chloride and carbolic gauzes are worthy of corresponding definite assay processes and standards in common with the other medicated dressings. Some attempt has been made to describe the *Sterilization of Dressings* in a very sketchy manner. The article also briefly comments on the following topics: *Battista, Cellulosum Ligni, Charta Oleata, Corchorus, Emplastra, Gossypia, Jaconettum, Lana, Ligamenta, Linteæ, Stupæ, Telæ.*—J. Bain. *Pharm. J.*, 134 (1935), 87. (W. B. B.)

Non-Official Formulæ

Adepdelen. (A. Erdmann, Berlin-Schöneberg) is a scouring and degreasing agent prepared from urea borate, sodium sulphate, magnesium sulphate and other corrigents.—*Pharm. Zentralh.*, 76 (1935), 553. (E. V. S.)

Brushless Shaving Creams. A good cream of this type must (1) spread easily, (2) soften the beard, (3) remain soft on the skin and in the tube, (4) promote sliding of the razor on the skin, (5) be removed easily by water from the razor and the face, (6) not irritate the skin and (7) be pleasantly perfumed. Essentially these products are modified day creams of the oil-in-water types of emulsions and small amounts of superfatty bodies. Stearate creams are preferred in which stearin is saponified by potassium hydroxide, potash, ammonia or triethanolamine or their mixtures up to 20–40%. Less than 14% stearin produces a cream without body; over 20% one with too much body. The purposes of the various ingredients are discussed. The following formulas are offered: (1) Cefatin 20%, stearin 5%, glycerin 5%, water 70 parts, liquid petrolatum 1 part. For strong beards the addition of 0.5% triethanolamine worked in while hot is suggested. (2) Stearic acid 17%, glycerin 10%, potassium hydroxide (or potassium hydroxide solution, 50° B, 2%), mineral oil 2.5%, water 69.5%. The quality of the product may be varied by the addition of traces or small amounts of lanolin, cocoa-butter, magnesium stearate, zinc stearate, zinc oxide, talc, colloid-kaolin suspension, (3) (an American product) stearic acid 20%, cetyl alcohol 1.1%, mineral oil 2%, ethylene glycol (or preferably diglycol, triglycol or carbitol) 1.5%, triethanolamine 1.65%, borax 1.85%, water 71.4%, perfume 0.5%. Melt the fats and add with stirring to the boiling mixture of other ingredients. After cooling to 40° C. add the glycol and the perfume, (4) stearic acid 20%, glycerin 5%, white mineral oil 5%, ammonia (26%) 2.2%, zinc oxide 1.5%, phenol 0.05%, perfume 0.75% and remainder water.—JOSEF AUGUSTIN. *Reichstoff-Ind. Kosmetik,* 10 (1935), 116–118. (H. M. B.)

Burns—Preparations for. (1) Picric acid 1%, boric acid 2%, lanolin, absorption base 34%, cetyl alcohol 3%, spermaceti 2%, water 58%. Dissolve the acids in water; melt the other ingredients at 40° C. Heat the water solution to this temperature and carefully mix in small amounts. (2) Tannic acid 4.8%, lanolin, hydrous 27%, thymol 0.2%, cod liver oil 20%, short fibre soft amber petrolatum 47%, eucalyptus oil 1%. Dissolve the thymol in the eucalyptus oil, add a small amount of the cod liver oil and rub up the acid in this mixture. Melt the lanolin and petrolatum and mix until it thickens, then stir in the remainder of the cod liver oil and finally the thymol solution. (3) Tannic acid 5%, oil of cade 5%, cod liver oil 15%, lanolin absorption base 40%, water 34.8%, phenol 0.2%. Dissolve the acid in water and warm to 45° C. Warm the base to 40° C., add phenol and the oils. Slowly stir in the tannic acid solution. (4) Linseed oil, refined 45%, cottonseed oil, refined 42%, cod liver oil 10%, phenol 1%, camphor 2%. Mix the last three ingredients together and add to a mixture of the first two. (5) *An emulsified lotion:* lecithin 1%, cetyl alcohol 2%, cholesterin 1%, triethanolamine stearate 7%, methyl para-hydroxybenzoate 0.5%, mineral oil 18.5%, water 70%. Heat the oil and melt in the lecithin, cetyl alcohol and cholesterin. Put the stearate in water and heat with stirring until melted, pour in the oil solution slowly and stir constantly for an hour. (6) Tannic acid 5%, glycerin 5%, water 90%. Dissolve the acid in the water and add the glycerin. (7) Picric acid 2%, alcohol 10%, water 88%. Dissolve the acid in the alcohol and add the solution to the water.—ANON. *Drug and Cosmetic Ind.,* 37 (1935), 45–46. (H. M. B.)

Chamomile Cosmetics. Formulas for 18 preparations are offered in which the substance is used as the extract, oil, powder or water.—ANON. *Drug and Cosmetic Ind.,* 37 (1935), 187–190. (H. M. B.)

Colloid Mills and Cosmetics. Factors affecting the use of these mills in making creams are (1) the speed of the mill and the extent to which air bubbles are eliminated and (2) the rate at which the emulsion formed is passed through the mill. Six mills were studied and in a table various characteristics and their applicability in the manufacture of creams, emulsions, mucilages, etc., are discussed.—THORPE W. DEAKERS. *Drug and Cosmetic Ind.,* 37 (1935), 41–43. (H. M. B.)

Creams—Claims for. The following table is offered:

Type of Product	Described as	Advertising Claims	Adapted to
1. Cleansing Cream	Liquefying Cream, Cold Cream	Cleanses, penetrates, liquefies instantly, mild, stimulates, softens, refines the skin	All types of skin, especially oily skins

2. Lubricating Cream	Nourishing Cream, Tissue Cream, Tissue Builder, Skin Food, Nutrient Cream, Emollient Cream, Wrinkle Cream, Facial Cream, Massage Cream, Night Cream, Gland Cream	Nourishes, beautifies, softens, whitens, restores relaxed tissues, prevents a crepey throat, banishes lines, tones muscles, replaces natural oils, penetrates	Normal, dry, older and wrinkled skins
3. Foundation Cream (Vanishing Cream)	Finishing Cream, Make-up Foundation	Finishes, protects from dust and wind, acts as a powder base	All but very young skins
4. Stimulating Cream	Circulation Cream, Circulation Ointment	Refines, stimulates, brings natural color	Oily, sallow, lustreless, coarse and older skins
5. Eye Cream	Eye Wrinkle Cream, Eye Wrinkle Paste, Eye Tissue Cream	Prevents crow's feet, wrinkles, laugh wrinkles	Skins with incipient wrinkles and crow's feet; thin and dry skins
6. Hand Cream		Feeds skin, makes it soft, white, protects from chapping and roughness, soothes, penetrates, dries rapidly, non-sticky	Especially for dry, rough or red hands; foot massage

Primary Composition	Typical Formula	
1. Water-in-oil emulsion; low-viscosity oil; low melting emulsion containing oil, wax, water	Beeswax 15 Petrolatum 10 Mineral Oil 54 Water 20.3 Borax 0.7	
2. Water-in-oil emulsion; vegetable oils, cholesterin, lecithin, cetyl alcohols, etc.	Beeswax 15 Cetyl alcohol 5 Vegetable Oil 20 Mineral Oil 32 Cholesterin 2 Water 25 Borax 1 Preservative ..	
3. Oil-in-water emulsion; stearic acid, alkali, glycerin, water; cetyl alcohol, butyl stearate, cocoa-butter, oil	Stearic Acid 24 Potassium Hydroxide 1 Glycerin 5 Butyl stearate 6 Water 64	
4. Oil-in-water, water-in-oil emulsions with mineral or vegetable astringents	Paraffine 5 Lanolin 2 Mineral Oil 15	Cetyl alcohol 4 Alcohol 6-Water 65 Tannic acid 3
5. Extra-effective lubricating cream with higher proportion vegetable oils, etc.	Beeswax 15 Cetyl alcohol 10 Vegetable oil 45 Cholesterin 4	Water 25 Borax 1 Preservative ..

6. Like foundation cream with skin softeners; also mucilages.

Stearic acid	18.8	Glycerin	12
Triethanolamine	1.8	Alcohol	6.5
Cholesterin	2	Water	59.7

Drug and Cosmetic Ind., 37 (1935), 34–35. (H. M. B.)

Depilatory Developments. An account of recent progress in the United Kingdom. The use of the product of passing hydrogen sulphide into milk of lime is recommended.—HENRY LEE-CHARLTON. *Am. Perfumer*, 30 (1935), 278–279, 310. (G. W. F.)

Face Powder—Types of. The following table is offered—see pages 329 and 330.

Hormone and Vitamin Cosmetic Creams. A brief discussion of the efficiency of cold creams as vehicles for the application or administration of hormones and vitamins.—R. M. GATTEFOSSE. *Parfumerie Moderne*, 29 (1935), 141, 143. (A. P.-C.)

Liquid Shampoo. A shampoo should cleanse the hair thoroughly but instead of leaving the hair in a dry brittle condition it should impart lustre without greasiness. This is accomplished by removing the natural oil and depositing oils from the shampoo in minute traces on the hair shafts which makes the hair soft and lustrous. These preparations are essentially solutions of potassium soap to which alcohol and glycerin are sometimes added. The soap is made by the cold or hot method with preference to the former. A typical soap formula is given: Coconut oil 15%, palm oil 5, caustic potash (90%) 3, caustic soda (90%) 1, alcohol 7, water 69. Dissolve the alkalis in $^1/_3$ the water, using stoneware. Liquefy the oils at 120° F., vessel glass-lined or stainless steel. Agitate slowly and run in the alkali, add alcohol and mix for $^1/_2$ hour, allow to stand over night and stir in the remainder of the water, age and filter. If made by the hot process follow the same procedure except after the addition of the alkali, the temperature is increased and maintained for $^1/_2$ hour or until saponification is complete. The following formula yields a clear amber product which does not become turbid upon standing: Coconut oil (best Cochin grade) 14%, caustic potash (90%) 6, caustic soda 0.5, oleic acid 10, glycerin 12, perfume oil 0.5, water 57. Dissolve the potash in $^1/_4$ of the water using a steel kettle. Melt the coconut oil, and at 130° F. stir in the potash solution with constant mixing. Increase the temperature to 180° C. and stir for an hour and allow to stand over night, then bring to a boil $^1/_2$ the remaining water and the glycerin. Add the coconut soap in small portions with constant slow stirring until dissolved; heat the remaining water to boiling, add the sodium hydroxide and stir this solution into the soap; heat the oleic acid to 160° F. and add to the soap solution with agitation. Mix until tests show complete saponification. The finished soap may be run through a homogenizer or colloid mill and chilled to 32° F., producing a product which will not become cloudy after bottling. *Olive Oil Shampoo.*—Olive oil 8%, palm oil 4, coconut oil (Cochin) 8, caustic potash (90%) 5, alcohol 8, water 58, glycerin 6, oleic acid 2.5, perfume 0.5. Warm the oils to 120° F. and dissolve the potash in $^1/_4$ the water and stir into the oils, heat the mixture to 180° stirring constantly; stir in the acid, cool to 110°, stir in the alcohol and perfume, allow to stand over night and add the remainder of the water mixed with the glycerin. Age or chill and filter. Equipment necessary for this type of manufacture is mentioned.—ANON. *Drug and Cosmetic Ind.*, 37 (1935), 39–40, 43–44.
(H. M. B.)

Shaving Soap. The following formulas and procedures for modern shaving soaps are offered: (1) *Shaving Cream.*—Triple-pressed stearic acid 26%, coconut oil (Cochin) 8%, cetyl alcohol 5%, sodium hydroxide (85%) 0.75%, potassium hydroxide (85%) 9%, glycerin 9%, boric acid 1%, water 40.75%, oil of lavender 0.5%. Weigh out the stearic acid and divide into 2 equal parts; put one part into a steam-jacketed kettle, add the coconut oil and heat to 180° F.; put the other portion of the acid and the alcohol into another kettle and melt. Dissolve the boric acid in water and set aside; dissolve the alkalis in the remainder of the water. To the stearic acid and coconut oil melts add the alkalis slowly, mixing and maintaining the temperature for 20 minutes, add the glycerin and boric acid solution, mix for 10 minutes, add the remainder of the stearic acid and cetyl alcohol slowly, remove heat and mix for one hour, add the perfume oil, age for one month with occasional stirring. By reducing the amount of cetyl alcohol, anhydrous lanolin, lecithin or cholesterin may be added. The final product dissolved in warm alcohol should not show more than a pale pink to phenolphthalein. (2) Beef tallow 6%, triple-pressed stearic acid 25%, coconut oil (Cochin) 10%, lanolin 2%, cholesterin 0.25%, glycerin 7%, boric acid

Product	Purpose	Properties	Primary Composition	Formulas	Light	Medium	Heavy
1. Face Powder	Cosmetic, to improve texture, color and finish of the skin. Cover minor imperfections	Color, to match and flatter skin, is of paramount importance. Perfume must be attractive. Should have covering power, be adherent, apply smoothly, hold odor, not clog pores	May consist wholly or in part of powder bases. Pigments give covering power, slip given by talc, powder bases, etc. Metallic soaps increase adhesion, pptd. chalk acts as binder and absorber of perfume. Materials must be finely dispersed and color well mixed. Light, heavy and medium refer to covering power and type of skin	Zinc oxide	22.00	...	3.00
				Titanium Oxide	...	4.00	20.00
				Kaolin	...	20.00	...
				Zinc stearate	6.00	...	4.00
				Talc	65.00	66.00	66.00
				Magnesium stearate	...	3.00	...
				Precipitated chalk	6.00	...	6.00
				Mag. carbonate	...	6.00	...
				Perfume	1.00	1.00	1.00
				Color 9.5			
2. Compact Powder	Same as Face Powder	Color, perfume, covering power, adhesion, slip are important. Cake should be smooth, reasonably hard, non-crumbling. Should rub off onto puff smoothly and easily	Suitable face powder formula with addition of binder. May be molded or pressed. Finished cake should be smooth with satin finish. Gum or resin (1%) in water as binder	Talc	37.00	54.00	54.00
				Kaolin	25.00	26.00	29.00
				Starch	33.00	...	...
				Zinc oxide	5.00	9.00	...
				Liquid binder	...	10.00	10.00
				Titanium oxide	...	...	7.00
				Cetyl alcohol	...	1.00	...
				Color and perfume			

(*Continued from page 329*)

Product	Purpose	Properties	Primary Composition	Formulas	Light	Medium	Heavy
3. Cream Powder	Same as Face Powder	Color, perfume, covering power. Foundation cream should be non-greasy and leave no shine; should leave a thin film of fine powder which adheres well. Should benefit the skin	Covering agt. (white pigment) mixed with colored pigment is dispersed in foundation cream, which is non-greasy, leaving smooth finish on the skin; creams may be made for dry, normal, oily skins. Emulsion is liquid cream with finely divided pigment; formulas are for dry, oily skin and emulsion, respectively	Zinc oxide	3	5.0	3.0
				Titanium oxide	3	...	...
				Oil-in-water absorption base	10	...	...
				Stearic acid	3	22.8	2.0
				Potassium hydroxide	0.1	0.9	0.2
				Glycerin	4	10.0	3.0
				Spermaceti	5	...	3.0
				Water	71.9	61.3	82.8
				Pigment color and perfume			
4. Liquid Powder	Same as Face Powder	Color, perfume, covering power. Emulsion (thin cream) or suspension; should suspend easily after settling	Suspension type contains about 80% water, glycerin (emollient), alcohol (astringent) and pigment. Color must be non-bleeding and should be mixed with pigment. Grind fine (colloid mill) to facilitate suspension	Zinc oxide	6	..	8
				Titanium oxide	..	5	..
				Precipitated chalk	8	8	6
				Kaolin	3	5	5
				Zinc stearate	2	..	..
				Glycerin	3	3	5
				Alcohol	5	..	..
				Water	73	79	76
				Color and perfume			

1.5%, sodium hydroxide (85%) 8%, potassium hydroxide (85%) 0.75%, water 39%, menthol 0.1%, perfume 0.4%. The procedure is the same as in (1), the lanolin and cholesterin being melted with the second portion of the stearic acid and the menthol added with the perfume.—ANON. *Drug and Cosmetic Ind.*, 37 (1935), 181–182. (H. M. B.)

Tooth Paste. The following parts of a tooth paste are discussed: (1) polishing agents as chalk, tricalcium phosphate, calcium sulphate, magnesia and magnesium carbonate, (2) vehicles including glycerin, alcohol and water, (3) binders to prevent separation and give colloidal protection such as tragacanth, starch as the glycerite, Irish Moss as the mucilage, acacia and bentonite. Tragacanth is to be preferred and is prepared (*a*) glycerite of the gum (10%), glycerin 78% and water 12%. Heat until clear on the water-bath, or (*b*) by the cold method mixing the gum with glycerin and allowing to stand and (*c*) allowing the gum to swell with water and stirring until a smooth paste is obtained, (4) 1% mineral oil is added, (5) sweeteners such as saccharine, (6) potassium chlorate (10% or more), sodium bicarbonate, camphor, silica gel, cuttlefish bone, kaolin, talc, etc., are sometimes used, (7) color of the paste should be creamy white; erythrosine is used to give a pink paste and (8) flavors such as the oils of peppermint, spearmint, cassia, cloves, pimenta, anise, cardamom and eucalyptus, menthol and thymol. Necessary equipment for preparing is a mixer, sifter and ointment mill. The following formulas are offered: (1) precipitated chalk 40%, tricalcium phosphate 6%, white soap powder 2%, mucilage of tragacanth 5%, soluble saccharine 0.1%, mineral oil 1%, glycerin 15%, water 30.4% and flavor 0.5%. Soak the gum in $^1/_3$ of the water and place in a mixer with the remainder of the water and glycerin. Stir and add the sifted powders containing the saccharine and add the flavor and mineral oil. Mix until smooth, mill through an ointment mill, allow to stand and then tube in the usual way. (2) Precipitated chalk 42%, neutral soap powder 10%, benzoic acid 1%, saccharine 1%, glycerin 25%, alcohol (specially denatured) 20% and flavor 1%. Dissolve the saccharine and flavor in $^1/_2$ of the alcohol and the benzoic acid in the rest of the alcohol. In the mixing machine place the glycerin and add the alcohol solution of the acid passing through a strainer. Now add the other alcohol solution, agitate well; add $^1/_2$ of the chalk and mix, add the soap, mix and then add the rest of the chalk. Mix and mill. (3) Acid Dental Cream: tricalcium phosphate 23%, calcium sulphate 35%, glycerin 25%, water 10%, glycerite of tragacanth 4%, mineral oil 1%, acid 0.1%, sodium lauryl sulfonate 0.5% and flavor 1.4%. Prepare the glycerite by the heat method, place with the glycerin in a mixer and mix, add the calcium salts, mix, add the sodium salt and the flavor containing the saccharine, then the mineral oil. All powders should be free from carbonates. (4) Milk of Magnesia tooth paste: Milk of magnesia 24%, precipitated chalk 32%, white powdered soap 2%, glycerite of tragacanth 10%, glycerin 10%, water 20%, mineral oil 1%, saccharine (soluble) 0.1% and flavor 0.9%. Mix the glycerite, glycerin and water, add $^1/_2$ the chalk, mix, add the milk, mix, then the soap and the remainder of the chalk and the flavor containing the saccharine, mix well and mill. Time can be saved in making the milk of magnesia by using pulverized magnesium oxide, add water, allow to stand and pass through a colloid mill. Creams may be tested by placing in freezing mixtures, in an oven at 100–110° F. in a window. After three weeks the preparation should remain creamy, uniform and soft.—J. W. WILLIAMS. *Drug and Cosmetic Ind.*, 37 (1935), 31–32, 44. (H. M. B.)

DISPENSING

Fluidextract of Ergot in a Mixture. Considerable doubt exists as to the best means of dispensing Liquid Extract of Ergot of the B. P., 1932, and as to the value of the preparation when dispensed in the form of a mixture. In January 1933, Dr. J. H. Burn made the statement that "Extractum Ergotæ Liquidum must be given alone, and it cannot be included in general prescriptions if the desired therapeutic result is to be obtained." Dr. B. L. Stanton submitted the following formula for trial as a Quinine, Ergot and Strychnine Mixture: Ext. Ergot, Liq. B. P. '32 M xv, Quinini. Dihydrochlor. gr. v, Tr. Nuc. Vom. M xv, Tr. Aurant. M xv, Glycerin, ad Z ii. The object of the present work was to determine whether this mixture would keep for a sufficient length of time, without loss of activity due to alteration of the ergotoxine, for the mixture to be of practical value. A period of fourteen days was taken as a reasonable length of time, as normally such a mixture would be used in that time. A sample of mixture made according to Dr. Stanton's formula was prepared and after storage under ordinary conditions of light and temperature has remained clear after 15 months. A sample of Liquid Extract of Ergot was obtained and assayed

by the B. P. method. From the extract a sample of Quinine, Ergot and Strychnine Mixture was prepared according to the above formula and assayed by the same method as the extract. After 14 days, the Liquid Extract of Ergot and the mixture (without quinine) were again assayed. A loss of 12.9% of the original amount of ergotoxine present had occurred in both cases. The mixture suffered no greater loss in alkaloidal content than the Liquid Extract of Ergot.—E. E. NYE. *Australasian J. Pharm.*, 16 (1935), 385. (T. G. W.)

Medicines for Injection—Physiological and Physico-Chemical Considerations in the Preparation of. A lecture, concerned chiefly with the question to what degree the pharmacist can and should take into account physicochemical and physiological considerations in preparing medicines which are to be injected. In particular the question of making the hydrogen ion concentration of the preparation approach that of the blood is discussed. The question of isotonicity and that of sterility are also considered. A signature label is proposed for use by the pharmacist who attempts to employ these considerations: "In the preparation of this medicine injection account has been taken of p_H—Osmotic pressure—Sterility, in so far as the properties of the preparation permit."—S. A. SCHOU. *Arch. Pharm. og Chemi*, 42 (1935), 401. (C. S. L.)

Solution of Methenamine and Ammonium Chloride. A formula is cited for preparation of a solution recommended by Prof. E. Warburg for acidifying the urine, called: *Mixt. Methenamini-ammoni chloridi.* This consists of ammonium chloride, 80 Gm., methenamine, 12 Gm., *Tinctura Aurantii dulcis*, 10 Gm. and distilled water, 300 Gm.—ANON. *Arch. Pharm. og Chemi*, 42 (1935), 450. (C. S. L.)

PHARMACEUTICAL HISTORY

Apothecaries in Landau (Pfalz)—History of the. Historical.—HAGEN. *Apoth. Ztg.*, 50 (1935), 789–790. (H. M. B.)

Apothecaries of the City of Königsberg—History of the. The Adler Apothecary. A historical account of an apothecary dating from 1739–1831. C. f. *Apoth. Ztg.*, 44 (1929), No. 101; (1931), Nos. 44–47; (1934), Nos. 16, 71.—G. E. DANN. *Apoth. Ztg.*, 50 (1935), 407–410. (H. M. B.)

"Apothecary of the Golden Eagle" in Landburg on the Warther—History of the. G. WARTENBERG. *Apoth. Ztg.*, 50 (1935), 927–929. (H. M. B.)

Badianus Manuscript. An Aztec Pharmacopœia. Only one Aztec written medical text has come down to us. It is known as the Badianus Manuscript and is the earliest pharmacopœia written on this side of the Atlantic. The original is in the Vatican Library where its identity is obscured by the title "Codex Barberini Latin 244." Its precise title is "A book of Indian Medical Herbs composed by a certain Indian physician of the College of Santa Cruz, who is not theoretically learned, but is taught only by experience. In the year of our Lord Saviour 1552." The book was discovered about six years ago by Thorndike and Clark. Clark brought back a photographic copy. From photostats of this the present study was made. It is the work of two Indians, the original text being in Aztec. Badianus made the Latin translation. Ailments are grouped according to the region in the body, beginning with the head. There are remedies for mange, scabs, falling hair, cataract, cold in the head, quinsy, fever, fatigue and many others. Knowledge of medicinal plants and treatment of diseases was considered equal to that of Europe. The Franciscan friars included Mexican medicine in the curriculum of the College of Santa Cruz. The spreading of Aztec medical knowledge was by the writings of several physicians as well as by travelers and merchants.—EMILY W. EMMART. *J. Am. Pharm. Assoc.*, 24 (1935), 771. (Z. M. C.)

Cosmetics—History of, in Recent Times. A continuation dealing with the development of waters, toilet vinegars and pomades.—A. HAUENSTEIN. *Riechstoff-Ind. Kosmetik*, 10 (1935), 124–127. (H. M. B.)

Digitalis—Early History of. Since 1775, when Dr. Withering cured Dr. Crawley, the Dean of Brazen Nose College of Oxford, suffering from a dropsical condition, with a preparation of foxglove, many practitioners and investigators have worked with the drug. Digitalis, now a recognized medicine for over one hundred and seventy years, has grown in stature as the years went by until it has now attained recognition as the greatest of all heart healers.—EDWARD PODOLSKY. *Am. J. Pharm.*, 107 (1935), 352. (R. R. F.)