UNITED STATES PHARMACOPŒIA.

TENTH REVISION.

ABSTRACT OF PROPOSED CHANGES WITH NEW STANDARDS AND DESCRIPTIONS.*

PART III.

ORGANIC CHEMICALS.

(Part I of the Abstract of Proposed Changes also contained an abstract of many Organic Chemicals for the U. S. P. X.)

The Pharmacopœial Convention of 1920 recommended that abstracts of changes proposed for the U. S. P. X. and new standards and descriptions be published before final adoption, that those who are not members of the Revision Committee may have an opportunity for comment and criticism.

In compliance with this recommendation, the following abstracts are submitted. The nomenclature and the exact wording of the text do not necessarily represent that to be finally adopted and doses have not been appended. The official titles for trade-marked chemicals have not yet been announced, but trade-marked names will not be included even as synonyms.

Comments should be sent to the Chairman of the Revision Committee,

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Acetanninum (Acetyl Tannate—Dicetyl Tannin—Tannyl Acetate).—A product obtained by the acetylization of Tannic Acid.

Acetannin is a yellowish-white, or a grayish-white powder. It is odorless or has a slight acetous odor. It darkens in light.

Acetannin is only slightly soluble in water or alcohol, but soluble in ethyl acetate. It is soluble with gradual decomposition in aqueous solutions of alkali hydroxides and carbonates. It is also soluble in aqueous solutions of sodium borate and of sodium phosphate.

On warming 0.2 Gm. of Acetannin with 2 cc. of alcohol and 2 cc. of sulphuric acid, the odor of ethyl acetate is noticeable. A solution of Acetannin in sodium phosphate T. S. (1 in 100) after standing for several hours precipitates albumin from its solution, but this property is destroyed by the presence of an excess of alkali. Its solution in sodium borate does not precipitate albumin.

Ash: Not more than 0.3 per cent.

Dry about 1 Gm. of Acetannin, accurately weighed, for 3 hours at 100° C. The loss does not exceed 3 per cent.

Shake 1 Gm. of Acetannin with 50 cc. of water during 5 minutes and filter. 25 cc. of the filtrate require for neutralization not more than 1.2 cc. of tenth-normal sodium hydroxide, using 3 drops of phenolphthalein T. S. as indicator (*free acid*). To the remainder of the filtrate add 2 drops of ferric chloride T. S. A green but not a blue color is produced (*free tannic acid*).

Digest 1 Gm. of Acetannin with a solution of 2 Gm. of sodium bicarbonate in 200 cc. of water for 3 hours at about 40° C. Filter through a counterbalanced filter or a Gooch crucible, wash the insoluble residue with 4 portions of water, 10 cc. each, and dry to constant weight at 110° C. The weight of the residue does not exceed 0.15 Gm.

Digest 1 Gm. of Acetannin with 200 cc. of water for 2 hours, shaking at frequent intervals. Filter through a dry filter into a dry flask, rejecting the first 20 cc. of the filtrate. Evaporate 100 cc. of the subsequent filtrate to dryness on the water-bath and dry the residue for 3 hours at 100° C. The weight of the residue does not exceed 0.03 Gm. (*soluble substances*).

Preserve in well-closed containers, protected from light.

Acidum Acetylsalicylicum (Aspirin). $C_6H_4.O(CH_3CO).COOH$.—It contains not less than 99.5 per cent. of absolute acetylsalicylic acid.

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Acetylsalicylic Acid occurs in small, colorless crystals or needles, or as a white crystalline powder. It is odorless and stable in dry air. In contact with moisture, it gradually decomposes into salicylic and acetic acids.

One Gm. of Acetylsalicylic Acid dissolves in approximately 300 cc. of water, in 5 cc. of alcohol, in 17 cc. of chloroform and in 10 to 15 cc. of official ether at 25° C.; it is less soluble in absolute ether. It is also soluble with the decomposition in solutions of alkali hydroxides and carbonates.

Its melting point, when determined in a bath preheated to 120° C., is not below 132° C.

When the Acid is heated with water for several minutes, then cooled and a drop or two of ferric chloride T. S. added, a violet-red color is produced.

Boil about 0.5 Gm. of Acetylsalicylic Acid with 10 cc. of sodium hydroxide T. S. for a few minutes, cool and add 10 cc. of diluted sulphuric acid. A white precipitate of salicylic acid is produced and the odor of acetic acid is perceptible. Filter, add to the filtrate 3 cc. of alcohol and 3 cc. of sulphuric acid and warm. The odor of ethyl acetate becomes noticeable.

Ash: Not more than 0.05 per cent.

A solution of 0.5 Gm. of Acetylsalicylic Acid in 10 cc. of sulphuric acid is not darker than slightly yellow.

A solution of 0.5 Gm. of the acid in 10 cc. of warm sodium carbonate T. S. is clear.

Shake 1 Gm. of Acetylsalicylic Acid with 20 cc. of distilled water and 1 cc. of nitric acid for one minute and filter. Five-cc. portions of the filtrate yield no opalescence or turbidity at once on the addition of a few drops of silver nitrate T. S. (*chloride*) or barium chloride T. S. (*sulphate*).

Warm 1 Gm. of Acetylsalicylic Acid with 10 cc. of distilled water and 1 cc. of hydrochloric acid for two minutes, dilute with 40 cc. of water, cool and filter. The filtrate does not respond to the test for heavy metals. (*Part II, Test No.* —.)

Dissolve 1 Gm. of Acetylsalicylic Acid in 5 cc. of alcohol and allow the solution to evaporate spontaneously in a dish in a place protected from dust. A perfectly white crystalline residue is obtained. (Coloring matter, resinous substances.)

Dissolve 0.1 Gm. of Acetylsalicylic Acid in 1 cc. of alcohol, dilute the solution with 48 cc. of cold water and add at once 1 cc. of freshly prepared diluted ferric ammonium sulphate solution (made by adding 1 cc. of normal hydrochloric acid to 2 cc. of ferric ammonium sulphate T. S., and diluting with water to 100 cc.). The coloration produced, if any, in one-half minute, is not greater than that produced in a parallel test, using 1 cc. of solution containing 0.1 Gm. of Salicylic Acid in 1 liter of water.

Assay.—Weigh accurately about 1.5 Gm. of Acetylsalicylic Acid, previously dried for three hours over sulphuric acid, add 50 cc. of half-normal sodium hydroxide, boil gently for ten minutes, then titrate the excess of acidum hydroxide with half-normal sulphuric acid, using three drops of phenolphthalein T. S., as indicator. Each cc. of half-normal sodium hydroxide corresponds to 0.045027 Gm. of absolute acetylsalicylic acid.

Acidum Lacticum.—Test for glycerin is omitted.

Acidum Phenylethylbarbituricum (Luminal-Phenobarbital-Phenobarbiton). $CO(NH.CO)_2$-$C(C_2H_5)(C_6H_5)$.—Phenobarbital occurs in white, glistening, small crystals or as a white crystalline powder. It is odorless and is stable in the air.

One Gm. of Phenobarbital dissolves in about 1000 cc. of water, in 8 cc. of alcohol, in 40 cc. of chloroform, in 13 cc. of ether, in about 700 cc. of benzene at 25° C. It is soluble in alkali hydroxides and carbonates.

It melts between 172 and 174° C.

Its saturated aqueous solution is acid to litmus paper.

On boiling about 0.2 Gm. with 5 cc. of 25 per cent. sodium hydroxide, ammonia is evolved.

Shake about 0.3 Gm. of Phenobarbital for 2 minutes with 1 cc. of normal sodium hydroxide and 5 cc. of water, filter and divide this filtrate into 2 portions. To 1 portion add mercuric chloride T. S. A white precipitate is produced. To the other portion add silver nitrate T. S. a few drops at a time. A white precipitate is produced which at first redissolves but becomes insoluble when an excess of the silver nitrate has been added.

Ash from 0.5 Gm. is negligible.

A solution of 0.2 Gm. of Phenobarbital in 2 cc. of sulphuric acid is colorless or at most only faintly yellow (*readily carbonizable substances*).

Boil 2 Gm. of Phenobarbital with 10 cc. of alcohol under a reflux condenser during 3 minutes. A clear and complete solution results (phenylbarbituric acid).

Albutanninum (Albumin Tannate—Tannalbumin).—A compound of albumen and tannic acid.

Albutannin is a yellowish-white, odorless powder.

It is almost insoluble in water, alcohol, chloroform, or ether. It is decomposed by aqueous solutions of alkali hydroxides or carbonates.

Ash: Not more than 0.3 per cent.

Dry about 1 Gm. of Albutannin for 3 hours at 100° C. The loss does not exceed 6 per cent.

Shake 1 Gm. of Albutannin with 50 cc. of water for 5 minutes and filter. 25 cc. of the filtrate require for neutralization not more than 1 cc. of tenth-normal sodium hydroxide using 3 drops phenolphthalein T. S. indicator (*free acid*).

Digest 1 Gm. of Albutannin with 200 cc. of water for 2 hours, shaking at frequent intervals. Filter through a dry filter into a dry flask, rejecting the first 20 cc. of the filtrate. Evaporate 100 cc. of the subsequent filtrate to dryness on the water-bath and dry the residue for 3 hours at 100° C. The weight of the residue does not exceed 0.06 Gm. (*soluble substances*).

Place 1.5 Gm. of Albutannin in a 500-cc. glass-stoppered flask. Dissolve 2.5 Gm. of sodium bicarbonate in 250 cc. of water, warm the solution to 40° C. and gradually add it with agitation to the albutannin. Then add a filtered aqueous solution of 0.25 Gm. of pepsin in 5 cc. of water. Place the flask in a suitable bath, maintain at 40° C. for 2 hours, inverting the flask every ten minutes. Allow to remain in the bath for one-half hour longer without inverting. Filter through a counterbalanced filter, wash the undissolved residue with 4 portions of cold water, 10 cc. each, and dry to constant weight at 110° C. The weight of the residue does not exceed 0.45 Gm.

Place 1.5 Gm. of Albutannin in a 500-cc. glass-stoppered flask. Mix 20 cc. of normal hydrochloric acid with 230 cc. of water, warm the mixture to a temperature of 40° C. and add it gradually, with agitation, to the albutannin. Then add a filtered aqueous solution of 0.25 Gm. pepsin in 5 cc. of water. Place the flask in a suitable bath, maintain at 40° C. for 2 hours, inverting the flask during the digestion every 10 minutes. Allow to remain in the bath for another half-hour without inverting. Filter through a counterbalanced filter, wash the undissolved residue with 4 portions of cold water, 10 cc., each and dry at 110° C. to constant weight. The weight of the residue is not more than 0.37 Gm.

Preserve protected from light.

Alcohol.—Amount of solids changed from limit of 0.002 Gm. in 50 cc. to 0.001 Gm. in 40 cc.

Methyl Alcohol test altered—Dilute the alcohol with water to contain about 5 per cent. by volume of ethyl alcohol. To 5 cc. of this diluted alcohol contained in a test tube add 0.5 cc. of phosphoric acid and 2 cc. of a 3 per cent. aqueous solution of potassium permanganate and allow the mixture to stand for 10 minutes. Add 1 cc. of an aqueous 10 per cent. solution of oxalic acid and let stand until the solution is clear brown. Now add 1 cc. of sulphuric acid, cool to about 25° C., add 5 cc. of fuchsin sulphurous acid T. S., mix well and allow to stand for 10 minutes. At the end of this time the solution, when observed against a white background, may have a reddish or pale green color, but not a distinct blue or violet color (*methyl alcohol*).

Alcohol Dehydratum.—No change.

Alcohol Dilutum.—Formula by weight is omitted.

Amidopyrina (Pyramidonum—Dimethylaminoantipyrine—amidopyrine). $C_3ON_3(CH_3)_4.C_6H_5$.

Pyramidon occurs as colorless or white, small crystals, or as a crystalline powder. It is odorless, and stable in the air.

One Gm. of Pyramidon dissolves in 18 cc. of water, in 1.5 cc. of alcohol, in 12 cc. of benzene, in 1 cc. of chloroform, in 13 cc. of ether, at 25° C.

It melts between 107° and 109° C.

Its aqueous solution (1 in 25) is slightly alkaline to litmus paper.

To 5 cc. of an aqueous solution of Pyramidon (1 in 25) add 3 drops of diluted hydrochloric acid and 1 cc. of ferric chloride T. S. A bluish-violet color is produced. On the subsequent addition of a few cc. of diluted sulphuric acid, the color changes to violet-red.

Addition of 5 drops of silver nitrate T. S. to 5 cc. of an aqueous solution of Pyramidon (1 in 25) produces an intensely violet color (oxidizing substances yield the same color). On allowing the mixture to stand, a grayish-black precipitate of metallic silver is deposited.

To a solution of about 0.02 Gm. of Pyramidon in 5 cc. of water, add 2 drops of sulphuric acid and 2 drops of a 10 per cent. solution of sodium nitrite. A transient blue color is produced which disappears quickly, the liquid remaining colorless (the appearance of a green color indicates antipyrine).

An aqueous solution of Pyramidon (1 in 25) when added to a freshly prepared solution of potassium ferricyanide containing a little ferric chloride produces at once a dark blue color or precipitate (difference from *antipyrine*).

Ash not more than 0.1 per cent.

A solution of 0.1 Gm. of Pyramidon in 1 cc. of sulphuric acid is colorless (*readily carbonizable substances*).

Its aqueous solution (1 in 25) does not respond to the test for heavy metals (page —).

The addition of a few drops of silver nitrate T. S. to 5 cc. of an aqueous solution of Pyramidon (1 in 25) previously acidulated with a few drops of diluted nitric acid, produces no turbidity at once (*chloride*).

Argento-Proteinum Fortis (Strong Silver-Protein).—Silver oxide rendered colloidal by the presence of or combination with proteins. It contains from 7.5 to 8.5 per cent. of silver (Ag).

Strong Silver-Protein is a brown, odorless powder. It is usually somewhat hygroscopic.

It is freely soluble in water, but almost insoluble in alcohol, chloroform or ether.

Heat about 0.2 Gm. of Strong Silver-Protein in a porcelain crucible until all the carbonaceous matter is burned off, warm the residue with 1 cc. nitric acid, dilute with 10 cc. of water and add hydrochloric acid. A white precipitate is produced which dissolves in ammonia water.

The addition of 2 cc. of a 1 per cent. aqueous solution of sodium chloride to 10 cc. of an aqueous solution (1 in 100) of Strong Silver-Protein causes no turbidity.

The addition of 2 cc. of diluted hydrochloric or diluted sulphuric acid, or of lead acetate, zinc sulphate, or silver nitrate T. S. to 5 cc. of an aqueous solution (1 in 100) of Strong Silver-Protein usually produces no immediate precipitation.

Ferric chloride T. S. added to its aqueous solution (1 in 100) discharges the dark color and a precipitate is gradually produced. With mercuric chloride T. S. a white precipitate is formed and the supernatant liquid becomes colorless or nearly so.

Dissolve 0.5 Gm. of Strong Silver-Protein in 3 cc. of water. Pour this solution drop by drop with stirring into 17 cc. of alcohol. Filter, and refilter if necessary until a clear filtrate is obtained, keeping the funnel covered during the filtration. Dilute 10 cc. of the filtrate with an equal volume of water, add 2 drops of diluted nitric acid and 5 drops of a 1 per cent. aqueous solution of sodium chloride and mix well. A distinct turbidity is produced (difference from mild *silver-protein*).

Assay.—Weigh accurately about 2 Gm. of Strong Silver-Protein and ignite it in a porcelain crucible until all the carbon is burned off. Transfer as much as possible of the residue into a beaker, add to the crucible 5 cc. of nitric acid, warm to dissolve any adhering silver and transfer the solution into the beaker with the aid of a little water. Cover the beaker and heat on the water-bath until all the metallic silver is dissolved, adding a little more nitric acid if necessary. Filter into a flask, wash well the insoluble residue with water, cool and dilute with water, if necessary, to about 50–75 cc., add 2 cc. of ferric ammonium sulphate T. S. and titrate with tenth-normal ammonium sulphocyanate. Each cc. of tenth-normal ammonium sulphocyanate corresponds to 0.01079 Gm. of silver.

Preserve in well-closed containers, protected from light.

Argento-Proteinum Mite. (Mild Silver-Protein).—Silver oxide rendered colloidal by the presence of or combination with proteins. It contains from 19 to 25 per cent. of Silver (Ag).

Mild Silver-Protein occurs in dark brown or almost black shining scales or granules. It is odorless, and is frequently hygroscopic.

It is freely soluble in water, but almost insoluble in alcohol, chloroform or ether.

Heat about 0.1 Gm. of Mild Silver-Protein in a porcelain crucible until all the carbonaceous matter is burned off, warm the residue with one cc. nitric acid, dilute with 10 cc. of water and add hydrochloric acid. A white precipitate is produced which dissolves in ammonia water.

The addition of 2 cc. of a 1 per cent. aqueous solution of sodium chloride to 10 cc. of an aqueous solution (1 in 100) of Mild Silver-Protein causes no turbidity.

Diluted hydrochloric or sulphuric acid or solutions of heavy metals usually produce an immediate precipitation in its aqueous solutions.

Ferric chloride T. S. added to its aqueous solution (1 in 100) discharges the dark color and a precipitate is gradually produced. With mercuric chloride T. S. a white precipitate is formed and the supernatant liquid becomes colorless or nearly so.

Dissolve 0.5 Gm. of Mild Silver-Protein in 3 cc. of water. Pour this solution drop by drop with stirring into 17 cc. of alcohol. Filter and refilter if necessary until a clear filtrate is obtained, keeping the funnel covered during the filtration. Dilute 10 cc. of the filtrate with an equal volume of water, add 2 drops of diluted nitric acid and 5 drops of a 1 per cent. aqueous solution of sodium chloride and mix well. No turbidity is produced within one minute (difference from strong silver-protein).

Assay.—Proceed as directed under Argento-Proteinum Fortis, using about 1 Gm. for the assay.

Preserve in well-closed containers, protected from light.

Arsphenaminum (Arsphenolamine Hydrochloride—Arsenobenzol—Salvarsan—3-diamino, 4-dihydroxy Arsenobenzene Hydrochloride). $HCl.NH_2.OH.C_6H_3As:AsC_6H_3.OH.NH_2.HCl + 2H_2O$.

Arsphenamine contains from 30 to 32 per cent. of arsenic (As), and it complies with the toxicity requirements, control and labeling regulation of the United States Public Health Service.

Arsphenamine is prepared for sale in sealed containers of colorless glass, from which the air has been excluded either by production of a vacuum or by displacement with a non-oxidizing gas.

Arsphenamine is a light yellow powder. It is odorless or has a slight odor, and is hygroscopic. In dry state or in solution it is oxidized by exposure to the air, becoming darker and more toxic.

It is soluble in water, alcohol or glycerin, but only very slightly soluble in chloroform or ether.

Its aqueous solution (1 in 100) is acid to litmus (difference from *Neoarsphenamine*).

Add sodium hydroxide T. S. drop by drop to an aqueous solution of Arsphenamine (1 in 100). A precipitate is formed (difference from *Neoarsphenamine*) which readily dissolves in an excess of the sodium hydroxide. Sodium carbonate T. S. or solution of Sodium bicarbonate also produce a precipitate but it is insoluble in an excess of the reagent. It is also precipitated by mercuric potassium iodide T. S. (difference from *neoarsphenamine*).

An aqueous solution of Arsphenamine (1 in 100) is unaffected by the addition of diluted hydrochloric acid, diluted nitric acid, or by acetic acid even after heating (difference from *neoarsphenamine*). With an excess of hydrochloric acid a precipitate is formed. Diluted sulphuric acid or solutions of alkali sulphates immediately produce a precipitate.

The addition of 2 drops of freshly prepared ferric chloride T. S. to 5 cc. of the aqueous solution (1 in 1000) produces a brownish, violet tinted color, rapidly changing to deep red.

On adding 3 cc. of silver nitrate T. S. to 5 cc. of an aqueous solution of Arsphenamine (1 in 100) a red color isp roduced, but no precipitate is formed even after standing at the ordinary room temperature for 10 minutes (difference from *neoarsphenamine*). When 6 cc. of silver nitrate T. S. is added to the same volume of solution a precipitate is immediately formed which blackens on heating. On now adding 5 cc. of nitric acid and again heating the precipitate becomes white and dissolves in ammonia water.

The solution resulting from the assay yields with hydrogen sulphide a yellow precipitate soluble in ammonium carbonate T. S.

Assay.—Weigh accurately about 0.2 Gm. of Arsphenamine and transfer it into a glass-stoppered 200–250 cc. Erlenmeyer flask. Add 1 Gm. of finely powdered potassium permanganate and 5 cc. of diluted sulphuric acid and allow to stand for 10 minutes, rotating the contents of the flask during this time to insure thorough mixing. Now add 10 cc. of concentrated sulphuric acid in portions of about 2 cc., rotating the flask after each addition. When the reaction has ceased, add sufficient hydrogen dioxide solution to dissolve completely the brown precipitate (about 5 to 7 cc.). Towards the end, the hydrogen dioxide is added drop by drop to avoid any great

excess. Dilute with 25 cc. of distilled water and boil gently over an asbestos wire gauze for 15–20 minutes, or until the excess of hydrogen dioxide is expelled. Dilute with 50 cc. of distilled water and add tenth-normal potassium permanganate until the liquid is faintly pink, then discharge the pink color by addition of a drop of tenth-normal oxalic acid. Cool the solution, add 2.5 Gm. of potassium iodide, stopper the flask tightly and allow to stand in a cool, dark place for 1 hour. Then titrate the liberated iodine with tenth-normal sodium thiosulphate without the use of starch indicator. Make a blank test with the same quantities of the reagents and correct the assay for the volume of tenth-normal sodium thiosulphate used in the blank. Each cc. of tenth-normal sodium thiosulphate corresponds to 0.003748 Gm. As.

Benzocainum (Anasthesin—Ethylaminobenzoate). $C_6H_4NH_2COO(C_2H_5)$.—Benzocaine occurs as small white or colorless crystals, or as a white crystalline powder. It is odorless, and stable in the air.

One Gm. of Benzocaine dissolves in about 2500 cc. of water, in 5 cc. of alcohol, in 2 cc. of chloroform, in 4 cc. of ether, in 30 to 50 cc. of almond oil or olive oil, at 25° C. It is soluble in diluted acids.

It melts between 88° and 90° C.

When boiled with alkali hydroxides, alcohol is liberated.

Dissolve about 0.02 Gm. of Benzocaine in 10 cc. of water with the aid of a few drops of diluted hydrochloric acid, add to the solution 5 drops of 10 per cent. sodium nitrite solution, followed by 2 cc. of a solution of 0.1 Gm. of betanaphthol in 5 cc. of sodium hydroxide T. S. An orange-red precipitate is produced.

Its aqueous solution (1 in 50) prepared with the aid of a slight excess of diluted hydrochloric acid yields a precipitate with iodine T. S. and instantly reduces potassium permanganate T. S.

Ash: Not more than 0.1 per cent.

A solution of 0.1 Gm. of Benzocaine in 1 cc. of sulphuric acid is colorless (*readily carbonizable substances*).

Dissolve 1 Gm. of Benzocaine in 10 cc. of neutral alcohol. A clear solution results. Dilute this solution with 10 cc. of water, add 2 drops of phenolphthalein T. S. and 1 drop of tenth-normal sodium hydroxide. A red color is produced (*free acid*).

The addition of a few drops of silver nitrate T. S. to a solution of 0.2 Gm. of Benzocaine in 5 cc. of alcohol, previously acidulated with a few drops of diluted nitric acid, produces no immediate turbidity (*chloride*).

An aqueous solution of Benzocaine (1 in 20) prepared with the aid of just sufficient hydrochloric acid, does not respond to the test for heavy metals (page —).

Heat a crucible to redness and introduce, in small portions, an intimate mixture of 0.2 Gm. of Benzocaine, about 0.5 Gm. of potassium nitrate, and about 0.3 Gm. of anhydrous sodium carbonate. Maintain a red heat until the reaction ceases, then boil the cooled residue for five minutes with 10 cc. of diluted sulphuric acid, filter, and wash the undissolved residue with 10 cc. of distilled water. Evaporate the filtrate and washings until sulphuric acid vapors begin to evolve. The residue, dissolved in 5 cc. of distilled water, meets the requirements of the test for arsenic (page —).

Caffeina Citrata.—The rubric has been changed from "Citrated Caffeine contains, when dried to constant weight at 80° C., not less than 48 per cent. of anhydrous caffeine ($C_8H_{10}O_2N_4$ = 194.12)" to "A mixture of Caffeine and Citric Acid. It contains, when dried to constant weight at 80° C., not less than 48 per cent. of anhydrous Caffeine."

The formula for its preparation is omitted.

Solubility statement changed to "One Gm. of Citrated Caffeine dissolves in about 4 cc. of warm water. On diluting the solution with an equal volume of water, a portion of the Caffeine gradually separates, which redissolves on the further addition of water."

Added identity test, "Add 1 cc. of mercuric sulphate T. S. to 5 cc. of an aqueous solution of Citrated Caffeine (1 in 100), heat the mixture to boiling and add 1 cc. of potassium permanganate T. S. A white precipitate is formed."

Calcii Iodobehenas (Calioben).—Calioben consists principally of Calcium Monoiodobehenate $(C_{21}H_{42}ICO_2)_2Ca$. It contains, when dried to constant weight at 100° C., not less than 22.5 per cent. of Iodine (I).

Calioben is a white or yellowish powder, unctuous to the touch. It is odorless or has a slight fat-like odor.

It is insoluble in water, very slightly soluble in alcohol or ether, but is freely soluble in chloroform.

When strongly heated, it is decomposed with the evolution of white vapors, having an odor of burning fat and violet vapors of iodine.

Heat about 0.2 Gm. of Calioben with 2 cc. of sulphuric acid in a water-bath during 5 to 10 minutes, cool, add 2 cc. of chloroform and shake gently. The chloroform, upon separation, is colored violet.

Add 10 cc. of diluted hydrochloric acid to 1 Gm. of Calioben. No effervescence occurs (*carbonate*). Add 40 cc. of water and boil gently. A layer of fatty acid separates on the surface, which is soluble in ether or chloroform. Render the aqueous layer slightly alkaline with ammonia and add an excess of ammonium oxalate T. S. (about 5 cc.), a white precipitate of calcium oxalate is produced. Allow to stand for four hours, filter, evaporate the filtrate to dryness and ignite. The residue does not exceed 0.003 Gm. (*magnesium and alkali salts*).

Dried to constant weight at 100°, Calioben loses not more than 2 per cent. of its weight.

One Gm. of Calioben dissolves in 10 cc. of warm chloroform with not more than an opalescence.

Triturate 1 Gm. of Calioben with 10 cc. of water. The mixture is neutral to litmus paper. Add 15 cc. more water, mix well during 5 minutes and filter. Evaporate 10 cc. of the filtrate to dryness on the water-bath and dry to constant weight at 120° C. The weight of the residue does not exceed 0.001 Gm. (*soluble salts*). Other 5-cc. portions of the filtrate tested for halogens and sulphate as described under turbidimetric tests (see page —) show no greater turbidity than 0.1 cc. of fiftieth-normal hydrochloric acid or sulphuric acid.

Weigh accurately 1 Gm. of Calioben previously dried to constant weight at 100°, add 40 cc. of tenth-normal hydrochloric acid and boil gently in a flask until thoroughly decomposed. Cool and titrate the excess of acid with tenth-normal sodium hydroxide, using methyl orange T. S. as indicator. Not less than 20 cc. nor more than 20.6 cc. of the tenth-normal is consumed.

Assay.—Mix about 0.5 Gm. of Calioben, previously dried to constant weight at 100° C. and accurately weighed, with about 3 Gm. of anhydrous potassium carbonate. Place the mixture in a platinum crucible, cover with about 1 Gm. more of anhydrous potassium carbonate and heat moderately, gradually increasing the heat, but not exceeding a dull redness, until completely carbonized. Extract the residue with boiling distilled water and wash it on the filter with boiling water until the washings produce no opalescence with silver nitrate T. S. Heat the combined washings, which measure about 150 cc. on a water-bath and add an aqueous solution of potassium permanganate (1 in 20) in small portions, until the hot liquid remains permanently pink. Add just enough alcohol to remove the pink tint, cool to room temperature, dilute to 200 cc., mix well and filter through a dry filter, rejecting the first 50 cc. of filtrate. To 100 cc. of the subsequent clear filtrate add about 1 Gm. of potassium iodide (free from iodate) and an excess of diluted sulphuric acid, and titrate the liberated iodine with tenth-normal sodium thiosulphate, adding starch T. S. near the end of the titration. Each cc. of tenth-normal sodium thiosulphate used corresponds to 0.002115 Gm. of iodine.

Preserve in well-closed containers protected from light.

Carbo Ligni.—Ash: "Not more than 7 per cent." instead of 7.5.

Carbonei Tetrachloridum (Carbon Tetrachloride). CCl_4.—Carbon Tetrachloride is a clear, colorless, mobile liquid. It has a characteristic, ethereal odor, resembling that of chloroform.

It is not inflammable. In presence of moisture it is slowly decomposed by light.

Carbon Tetrachloride dissolves in 2000 times its volume of water. It is miscible with alcohol, chloroform, ether, benzene petrolatum, benzin, and is soluble in most of the fixed and volatile oils.

Specific gravity: From 1.588 to 1.590 at 25° C.

It boils between 76° and 77° C., but volatilizes at lower temperatures.

Evaporate 50 cc. of Carbon Tetrachloride in a dish on a water-bath to about 1 cc. and then allow to evaporate spontaneously to dryness. The residue, if any, is odorless. Now dry at 100° C. and weigh. The weight of the residue does not exceed 0.001 Gm.

Shake 15 cc. of Carbon Tetrachloride with 25 cc. of water during 5 minutes and allow

the liquids to separate completely. The aqueous layer is neutral to litmus, and 10-cc. portions are not affected by a few drops of silver nitrate T. S. (*chloride*), nor colored blue by the addition of a few drops each of potassium iodide T. S. and starch T. S. (*free chlorine*).

Warm 10 cc. of Carbon Tetrachloride with 10 cc. of 25 per cent. potassium hydroxide solution for 5 minutes at about 60° C. with frequent agitation. No yellow or brown color develops in either liquid (*aldehyde*).

Transfer 20 cc. of Carbon Tetrachloride into a glass-stoppered cylinder previously rinsed with sulphuric acid. Add 5 cc. of colorless sulphuric acid, shake vigorously for five minutes, then allow the liquids to separate completely. The sulphuric acid layer is colorless or nearly so (*readily carbonizable substances.*)

Mix 10 cc. of Carbon Tetrachloride with an equal volume of a 10 per cent solution of potassium hydroxide in dehydrated alcohol and allow the mixture to stand for one hour. Add 5 cc. of acetic acid and follow with one cc. of copper sulphate T. S. No yellow precipitate is formed within two hours (*carbon disulphide*).

Preserve in well-closed containers protected from light.

Carbromalum (Adalin Bromdiethylacetylcarbamide). $C(C_2H_5)_2Br.CONH.CONH_2$.

Carbromal is a white, crystalline odorless powder.

One Gm. of Carbromal dissolves in about 3000 cc. of water, in 18 cc. of alcohol, in 3 cc. of chloroform, in 14 cc. of ether, at 25° C. It is very soluble in boiling alcohol. It dissolves in sulphuric, nitric, and in concentrated hydrochloric acids, from which it is precipitated by dilution with water. It is also soluble in solutions of alkali hydroxides.

It melts between 116° and 117° C.

On boiling about 0.2 Gm. of Carbromal with 5 cc. of 10 per cent. aqueous sodium hydroxide solution, ammonia is evolved.

Mix 0.1 Gm. of Carbromal with 0.5 Gm. of anhydrous Sodium Carbonate and ignite gently until completely decomposed. Dissolve the residue in 5 cc. of hot water, cool, acidulate with acetic acid and filter. Add to the filtrate 2 cc. of chloroform and introduce chlorine water drop by drop. On shaking, the chloroform acquires a red-brown color.

Ash: From 0.5 Gm. is negligible.

A solution of 0.2 Gm. of Carbromal in 2 cc. of sulphuric acid is colorless or at most faintly brown (*readily carbonizable substances*).

Shake 1 Gm. of Carbromal with 20 cc. of water during 5 minutes and filter. The filtrate is neutral to litmus paper and 5-cc. portions tested for halogen and sulphate as described under the turbidimetric test (see page —) show no greater turbidity than 0.1 cc. of fiftieth-normal hydrochloric acid or sulphuric acid.

Chloramina (Chloramine T. Sodium paratoluenesulphonochloramide). $C_6H_4(CH_3)$-$(SO_2NNaCl) + 3H_2O$.

It contains from 11.5 to 13 per cent. active chlorine.

Chloramine T. occurs as white or faintly yellow crystals or crystalline powder. It has a slight odor of chlorine and slowly decomposes on exposure to air, losing chlorine.

One Gm. of Chloramine T. dissolves in about 7 cc. of water at 25° C. and in about 2 cc. of boiling water. It is decomposed by alcohol, and it is insoluble in benzene, chloroform or ether.

At about 95° to 100° C. it loses all of its water of crystallization.

An aqueous solution of Chloramine T. (1 in 20) is alkaline to litmus or phenolphthalein T. S.

The addition of potassium iodide T. S. to an aqueous solution of Chloramine T. (1 in 20) causes the liberation of iodine, but no bromine is liberated from alkali bromides unless the mixture is acidulated with an acid (*difference from dichloramine T.*).

Acids produce in an aqueous solution of Chloramine T. (1 in 20) a white turbidity or precipitate which redissolves in an excess of alkali hydroxide solutions. With strong mineral acids chlorine is simultaneously liberated.

On adding 2 cc. of sulphuric acid to 0.1 Gm. of Chloramine T. chlorine is evolved but no appreciable darkening occurs (*readily carbonizable substances*).

Assay.—Weigh accurately about 0.5 Gm. of Chloramine T., dissolve it in 50 cc. of water, add 5 cc. of potassium iodide T. S. and 5 cc. of acetic acid, allow the mixture to stand for ten

minutes, then titrate the liberated iodine with tenth-normal sodium thiosulphate, using starch T. S. as indicator. Each cc. of tenth-normal sodium thiosulphate corresponds to 0.001773 Gm. of chlorine.

Preserve in well-closed containers, protected from light.

Chlorcosanum.—A liquid, chlorinated paraffin.

Chlorcosane is a light yellow to light amber-colored, thick, oily liquid. It is odorless, and stable in the air.

It is insoluble in water, slightly soluble in alcohol, but miscible with benzene, carbon tetrachloride, chloroform or ether.

Specific gravity: 1.00 to 1.07 at 25° C.

Boil 5 drops of Chlorcosane with 10 cc. of alcoholic potassium hydroxide T. S. under a reflux condenser for 30 minutes, cool, dilute with 10 cc. of distilled water and acidulate with diluted nitric acid. The liquid becomes turbid from the separation of small, oily drops. Shake the mixture with an equal volume of ether, allow the liquids to separate, draw off the clear aqueous layer and add to it a few cc. of silver nitrate T. S. A white precipitate is formed.

Ash: Not more than 0.1 per cent.

"Shake about 5 Gm. of Chlorcosane with 25 cc. of warm distilled water for five minutes and filter through a filter paper moistened with distilled water. The filtrate is neutral to litmus paper (*acids or alkalies*) and on adding a few drops each of potassium iodide T. S. and of starch T. S. to 5 cc. of the filtrate, no blue color is produced (*free chlorine*). Another 5-cc. portion of the filtrate shows no more chloride than 0.05 cc. of fiftieth-normal hydrochloric acid (*chloride*)."

Dissolve 10 Gm. of Chlorcosane in 10 cc. of carbon tetrachloride in a 50-cc. volumetric flask, dissolve in this solution about 0.5 Gm. of powdered Dichloramine T. accurately weighed and allow the mixture to stand at 40° C. for 4 hours, protected from light. Cool and dilute with sufficient carbon tetrachloride to make the volume 50 cc. Transfer 10 cc. of this dilution into a glass-stoppered flask, add 10 cc. of glacial acetic acid and 5 cc. of potassium iodide T. S. and tightly stopper the flask. Allow to stand for 10 minutes, then add 25 cc. of distilled water and titrate the liberated iodine with tenth-normal sodium thiosulphate, using starch T. S. as indicator. Run a parallel blank with the same quantity of the same Dichloramine T. and the other reagents, but using sufficient carbon tetrachloride to make the volumes 50 cc. The difference between the volumes of tenth-normal sodium thiosulphate consumed in the blank and in the test is not greater than 0.6 cc. (*completeness of chlorination*).

Cocainæ Hydrochloridum.—Statement of origin is omitted.

Melting point.—"Not below 183° C." instead of "between 183° and 191° C."

Ash changes.—"Ash from 0.1 Gm. is negligible."

Added test.—"A solution of 0.5 Gm. of Cocaine Hydrochloridein, 10 cc. of distilled water requires not more than 0.5 cc. of fiftieth-normal sodium hydroxide to neutralize, using one drop of methyl red as indicator."

Codeinæ Sulphas.—Statement of origin is omitted.

Added test.—"A solution of 0.5 Gm. of Codeine Sulphate in 15 cc. of distilled water requires not more than 0.3 cc. of fiftieth-normal sulphuric acid for neutralization, using 1 drop of methyl red T. S. as indicator."

Creosoti Carbonas.—Specific gravity—"not below 1.150" instead of "1.145 to 1.170."

Test for Creosote altered.—After treating with Hydrochloric Acid and allowing to separate into two layers:

"Separate the creosote layer and wash it by shaking at first with 15 cc. of 20 per cent. aqueous solution of sodium chloride, then with 10 cc. of water and distil. Eighty-five per cent. of the creosote thus obtained, allowance being made for adhering moisture, distils between 200 and 220° C. The distillate, after separation of adhering water, does not respond to the test for *hydrocarbons* and *'coal tar creosote'* described under *Creosotum.*"

Test for acid and free creosote changed.—"A one to five solution of the creosote in alcohol being used instead of a saturated solution, as in the present U. S. P."

Dextrosum (*d*-Glucose—Grape Sugar—Starch Sugar). $C_6H_{12}O_6$.—Dextrose is a white, crystalline powder or granular. It is odorless and has a sweet taste.

One Gm. of Dextrose dissolves in 1 cc. of water, and in about 40 cc. of alcohol at 25° C. It is more soluble in boiling water or in boiling alcohol.

Its aqueous solution (1 to 20) is neutral to litmus paper and is dextrorotatory.

The specific rotation $[\alpha]_D$ of Dextrose, determined at 25° C. in an aqueous solution containing in 100 cc., 10 Gm. of Dextrose, previously dried to constant weight at 105° C., and using a 200-mm. tube, is from + 52.3° to 52.8° (page —). The solution must be allowed to stand 24 hours before observing the rotation.

Add a few drops of the aqueous solution (1 in 20) to 5 cc. of hot alkaline cupric tartrate T. S. A copious red precipitate of cuprous oxide is formed (*difference from cane sugar.*)

Dried to constant weight at 105° C., it loses not more than 1 per cent. of its weight.

Ash: Not more than 0.1 per cent.

One Gm. of finely powdered Dextrose dissolves completely on boiling with 20 cc. of alcohol under a reflux condenser (*dextrin, milk sugar*).

On adding 1 drop of iodine T. S. to a solution of 1 Gm. of Dextrose in 10 cc. of distilled water, the liquid is colored yellow (*soluble starch, sulphite*).

The aqueous solution (1 in 20) does not respond to the test for heavy metals (page —).

Dissolve 1.5 Gm. of Dextrose in 5 cc. of distilled water, add 5 cc. of diluted sulphuric acid and 1 cc. of bromine water and heat for 5 minutes on a water-bath. Add 0.5 Gm. of potassium iodine, follow with 5 drops of stannous chloride T. S., cool and subject to the arsenic test (page —). The stain produced, if any, is not greater than that produced in a test made with the same quantities of reagents to which 2 cc. of the standard arsenic solution (page —) has been added.

Two-Gm. portions of Dextrose show no more chloride than corresponds to 0.5 cc. of fiftieth-normal hydrochloric acid, and no more sulphate than corresponds to 0.5 cc. of fiftieth-normal sulphuric acid.

Diacetylmorphinæ Hydrochloridum.—Solubility changed to—"One Gm. of Diacetylmorphine Hydrochloride dissolves in 2 cc. of water, in 13 cc. of alcohol, and in 2 cc. of chloroform at 25° C. It is almost insoluble in ether."

Test added.—"A solution of 0.5 Gm. of Diacetylmorphine Hydrochloride in 15 cc. of distilled water requires not more than 0.7 cc. of fiftieth-normal sodium hydroxide for neutralization, using one drop of methyl red T. S. as indicator (*free acid*)."

Dichloramina (Dichloramine-T. Paratoluenesulfonedichloramide). $C_6H_4(CH_3)(SO_2NCl_2)$.

It contains from 28 to 30 per cent. active chlorine.

Dichloramine T. occurs as pale yellow crystals or yellowish crystalline powder. It has an odor of chlorine, and gradually decomposes on exposure to air, losing chlorine.

Dichloramine T. is almost insoluble in water. It is soluble in eucalyptol and in chlorinated paraffin hydrocarbons, also in glacial acetic acid. One Gm. dissolves in about 1 cc. of benzene or chloroform and in about 2.5 cc. of carbon tetrachloride. It is decomposed by alcohol.

It melts at about 80° C.

On adding about 0.1 Gm. of Dichloramine T. to 5 cc. of an aqueous solution of sodium bromide (1 in 10) bromine is liberated (difference from *chloramine T.*).

Strong mineral acids added to Dichloramine T. liberate chlorine.

Add 1 cc. of sulphuric acid to 0.1 Gm. of Dichloramine T. Chlorine is evolved but no appreciable darkening occurs (*readily carbonizable substances*).

Assay.—Weigh accurately about 0.1 Gm. of Dichloramine T. in a dry glass-stoppered bottle, dissolve it in 20 cc. of glacial acetic acid, then add 10 cc. of potassium iodide T. S., dilute with 50 cc. of water, allow to stand for ten minutes and titrate the liberated iodine with tenth-normal sodium thiosulphate. Each cc. of tenth-normal sodium thiosulphate corresponds to 0.001873 Gm. of available chlorine.

Preserve in well-closed containers protected from light.

Epinephrina (Lævomethylaminoethanol Catechol). $C_9H_{13}O_3N$.—Epinephrine occurs as a white or light brownish, microcrystalline, odorless powder, gradually darkening on exposure to the air.

It is very slightly soluble in water or alcohol, insoluble in ether, chloroform, acetone and fixed or volatile oils.

Epinephrine combines with acids, forming salts which are readily soluble in water, and from these solutions the base may be precipitated by ammonia or alkali carbonates.

The acid solution is not affected by picric acid, tannic acid, phosphomolybdic acid, mercuric potassium iodide, or platinic chloride.

Its saturated aqueous solution is slightly alkaline to litmus paper.

A dilute (1:1000) slightly acid solution of Epinephrine gives an emerald-green color with ferric chloride T. S., turning to cherry-red and finally to brown on standing. Other oxidizing agents produce red, pink or violet colors which change to brown. Fixed alkali hydroxides cause the solution to darken on standing, but do not precipitate the Epinephrine.

Ash is negligible from 0.1 Gm.

Preserve in well-closed containers, protected from light.

Eugenol.—Statement of origin is omitted.

Fel Bovis.—No change.

Hypophysis Sicca.—No change.

Liquor Epinephrini Hydrochloridi.—A solution of epinephrine in water and hydrochloric acid, containing in each 1000 cc. not less than 0.95 Gm. nor more than 1.05 Gm. of epinephrine and 0.005 per cent. of HCl.

(The formula for the preparation of this solution and assay will be supplied by the Subcommittee on Liquors and Bio-assays, respectively.)

Solution of Epinephrine Chloride is a nearly colorless, slightly acid liquid, gradually turning dark on exposure to air and light. When the solution has become brown in color and contains a precipitate it must be rejected.

The addition of one drop of ferric chloride T. S. to 10 cc. of the solution produces an emerald-green color which soon turns to a cherry-red and finally to a brown. Other oxidizing agents produce red to pink or violet colors which change on standing. Fixed alkali hydroxides darken it, but do not precipitate the epinephrine.

Preserve in small, well-filled, amber-colored bottles.

Maltum.—Rubric changed to "The grain of one or more varieties of *Hordeum sativum* Jessen (Fam. *Gramineæ*) partially germinated artificially, and containing amylolytic enzymes. It converts not less than 5 times its weight of starch into sugars."

Assay changed to "Mix a quantity of potato starch, purified as directed under Pancreatinum, equivalent to 5 Gm. of dried starch, in a beaker with 10 cc. of cold, distilled water. Add 140 cc. of boiling distilled water and heat on a water-bath with constant stirring for two minutes, or until a translucent, uniform paste is obtained. Cool to 40° C. in a suitable bath previously adjusted to this temperature. Add 20 cc. of an infusion of Malt freshly prepared as described above and previously heated to 40° C. Mix well and maintain the same temperature for exactly thirty minutes, stirring frequently; a thin, nearly clear liquid is produced. Add at once 0.1 cc. of this liquid to a previously made mixture of 0.2 cc. of tenth-normal iodine and 60 cc. distilled water. No blue or reddish color is produced."

Mel.—Statement of origin changed to "A saccharin substance gathered mostly from the nectar of flowers, and deposited in the honey comb by the bee, *Apis Mellifera* Linné (Fam. *Apidæ*).

Optical activity statement changed to "Honey is lævorotatory at 20° C."

Tests for chloride and sulphate changed.—"Ten-cc. portions of an aqueous filtered solution of Honey (1 in 10) show no more chloride than 0.2 cc. of fiftieth-normal hydrochloric acid and no more sulphate than 0.2 fiftieth-normal sulphuric acid (see turbidimetric tests)."

Neoarsphenaminum (Novarsenobenzol, Neosalvarsan).—A product obtained by the action of sodium methanol sulphoxylate on arsphenamine, and consisting partially of sodium 3-diamino 4-dihydroxy arsenobenzene methanol sulphoxylate ($NH_2OH.C_6H_3As:SC6H_3.OH.NH.(CH_2O)OSNa$. It contains from 18 to 20 per cent. of arsenic (As), and it complies with the toxicity requirements, control and labeling regulations of the United States Public Health Service.

Neoarsphenamine is prepared for sale in sealed containers of colorless glass, from which the air has been excluded either by production of a vacuum or by displacement with a non-oxidizing gas.

Neoarsphenamine is a yellow powder. It is odorless or has a slight odor. In dry state or in solution it is readily oxidized by exposure to the air, becoming darker or more toxic. Higher temperatures accelerate the oxidation.

It is very soluble in water, soluble in glycerin, slightly soluble in alcohol and almost insoluble in chloroform or ether.

Its aqueous solution (1 in 20) is neutral to litmus paper (difference from arsphenamine).

An aqueous solution of Neoarsphenamine (1 in 100) is precipitated by diluted mineral acids (difference from arsphenamine) but yields no precipitate with solutions of alkali hydroxides or carbonates, and is not precipitated by mercuric potassium iodide T. S. (difference from arsphenamine).

The addition of two drops freshly prepared ferric chloride T. S. to 5 cc. of the aqueous solution (1 in 1000) produces a purple or purplish-red color changing to dark red.

On adding 3 cc. of silver nitrate T. S. to 5 cc. of the aqueous solution (1 in 100) a brown precipitate is immediately formed, rapidly becoming black (difference from arsphenamine).

To 10 cc. of an aqueous solution of Neoarsphenamine (1 in 100) add 10 cc. of diluted hydrochloric acid and heat. The odor of sulphur dioxide is perceptible.

The solution resulting from the assay yields with hydrogen sulphide a yellow precipitate soluble in ammonium carbonate T. S.

Add 5 cc. of distilled water to 0.5 Gm. of Neoarsphenamine in a test tube and gently agitate the mixture. A complete solution results in 5 minutes.

Assay.—Proceed as directed under *Arsphenaminum.*

Preserve in a cool place, preferably at a temperature not above 10° C.

Oleum Olivæ.—Test for peanut oil added—"Saponify 10 Gm. of Olive Oil by heating it under a reflux condenser with a solution of 4 Gm. of potassium hydroxide in 80 cc. of alcohol for one hour." Neutralize exactly with diluted acetic acid using phenolphthalein as indicator, and wash into 120 cc. of boiling lead acetate T. S. Boil the mixture for a minute, then cool by immersing the flask in cold water, occasionally rotating the contents to cause the precipitate to adhere to the sides of the flask. Decant the liquid and wash the precipitate with cold water to remove excess of lead acetate, then wash with 90 per cent. alcohol (by volume). Add 100 cc. of ether, cork well and allow to stand until the precipitate is disintegrated. Connect with a reflux condenser, boil for 5 minutes, then cool to about 15° C. and allow to stand over night. Filter and thoroughly wash the precipitate of lead soaps with ether. Wash the precipitate into a 500-cc. separator by means of a jet of ether, alternating at the end, if a little of the precipitate adheres to the filter paper, with diluted hydrochloric acid. Add enough diluted hydrochloric acid to make the total volume about 100 cc. and also enough ether to make its total volume about 100 cc. and shake vigorously for several minutes. Allow the layers to separate, run off the acid layer, and wash the ether once by shaking with 50 cc. of diluted hydrochloric acid and then with several portions of water until the water washings are no longer acid to methyl orange. Transfer the ether solution into a dry flask, evaporate off the ether, add a little dehydrated alcohol and evaporate on a steam-bath. Dissolve the dry fatty acids by warming with 50 cc. of 90 per cent. alcohol (by volume), slowly cool the solution to 15° C., shaking frequently to aid crystallization, and allow to stand at 15° C. for 30 minutes. No crystals separate (*peanut oil*).

Pancreatinum.—Added to rubric—"It converts not less than 25 times its weight of casein into proteoses."

Assay with milk omitted.

Assay for Casein Digesting Power added.—"*Assay for Casein Digestive Power.*

"Place 0.1 Gm. of finely powdered casein in a 50-cc. volumetric flask, add 30 cc. of distilled water and shake well to bring the casein to suspension. Add exactly 1 cc. of tenth-normal sodium hydroxide and heat the mixture at 40° C. until the casein is completely dissolved, which should not require more than 30 minutes. Cool, add sufficient water to make 50 cc. and mix well.

"Dissolve 0.1 Gm. of Pancreatin in 500 cc. of distilled water. Mix 1 cc. of glacial acetic acid with 9 cc. of water and 10 cc. of alcohol.

"Place 3 cc. of the casein solution in a test tube, add to it 2 cc. of the well-shaken Pancreatin solution and 3 cc. of water and mix by gentle agitation.

"Immediately immerse the test tube in a water-bath of 40° C. and keep it at this temperature for one hour. Then remove from the bath and add 3 drops of the acetic acid. No precipitate is produced."

Phenolsulphonephthaleinum (Sulphenthal—Phenol Red) $(C_6H_4OH)_2CO.C_6H_4SO_2$.—Sulphenthal is a crystalline powder, varying in color from bright to dark red. It is stable in the air.

One Gm. of Sulphenthal dissolves in about 1300 cc. of water, 350 cc. of alcohol, 500 cc. of acetone. It is almost insoluble in chloroform or ether.

It is readily soluble in solutions of alkali hydroxides and carbonates as well as in ammonia water or in solutions of ammonium carbonate with a color varying from deep red in concentrated to violet-tinted red in dilute solutions. The red color of the solution is changed to orange or yellow by a slight excess of acid. Its solution in alkali hydroxide is decolorized by warming with zinc dust.

Dried to constant weight at 110° C. Sulphenthal loses no more than 1 per cent. of its weight.

Ash: Not more than 0.2 per cent.

Fill a 100-cc. glass-stoppered flask with thoroughly boiled and cooled water to within a few cc. of the stopper. Add 1 cc. of an alcoholic solution of Sulphenthal (1 in 1000) and 0.5 cc. of fiftieth-normal sodium hydroxide, stopper immediately and mix well. A strong red color is produced (*sensitiveness*).

To about 1 Gm. of Sulphenthal accurately weighed, add a filtered solution of 0.5 Gm. of sodium bicarbonate in 20 cc. water. Rotate frequently during one hour, dilute to 100 cc. and let stand over night. Filter on counterbalanced filters or a Gooch crucible, wash at first with 25 cc. of 1 per cent. solution of sodium bicarbonate, then with 25 cc. of distilled water, and dry to constant weight at 110°. The weight of the insoluble residue does not exceed 0.2 per cent.

Heat a crucible to redness and introduce, in small portions, an intimate mixture of 0.2 Gm. of Sulphenthal, about 0.5 Gm. of potassium nitrate, and about 0.3 Gm. of anhydrous sodium carbonate. Maintain a red heat until the reaction ceases, then boil the cooled residue for five minutes with 10 cc. of diluted sulphuric acid, filter, and wash the undissolved residue with 10 cc. of distilled water. Evaporate the filtrate and washings until sulphuric acid vapors begin to evolve. The residue, dissolved in 5 cc. of distilled water, meets the requirements of the test for arsenic (page —).

Physostigminæ Salicylas.—The definition "The salicylate ($C_{15}H_{21}O_2N_3.C_7H_6O_3$) of an alkaloid obtained from physostigma" is omitted but the formula follows the title.

Quinidinæ Sulphas (Quinidine Sulphate). $(C_{20}H_{24}O_2N_2)_2.H_2SO_4 + 2H_2O$.—Quinidine Sulphate occurs in fine, needle-like, white crystals, frequently cohering in masses. It is odorless and has a very bitter taste. It darkens on exposure to light.

One Gm. of Quinidine Sulphate dissolves in about 90 cc. of water, 10 cc. of alcohol, at 25° C., in about 15 cc. of boiling water. It is soluble in chloroform but almost insoluble in ether.

The aqueous solution of Quinidine Sulphate (1 in 100) is neutral or slightly alkaline to litmus paper and is dextrorotatory. (Quinine Sulphate is lævorotatory.)

An aqueous solution of Quinidine Sulphate when acidulated with diluted sulphuric acid develops a vivid blue fluorescence. Add 1 or 2 drops of bromine T. S. to 5 cc. of an aqueous solution of the salt (1 in 1000) and follow with 1 cc. of ammonia water. The liquid acquires an emerald-green color.

To 5 cc. of a solution of Quinidine Sulphate (1 in 100) add 1 cc. of silver nitrate T. S. and stir the mixture with a glass rod. A white precipitate forms after a short interval (difference from *many other alkaloids*).

Barium Chloride produces in an aqueous solution of the salt a white precipitate which is insoluble in hydrochloric acid.

Dried to constant weight at 100° C., Quinidine Sulphate loses not more than 5 per cent. of its weight.

Ash not more than 0.1 per cent.

A solution of 0.1 Gm. of the salt in 2 cc. of sulphuric acid is not darker than light yellow (*readily carbonizable substances*).

One Gm. of Quinidine Sulphate dissolves in 5 cc. of a warm mixture of two volumes of chloroform and one volume of dehydrated alcohol with not more than a slight turbidity (*ammonium or other inorganic salts*).

Dissolve 0.5 Gm. of Quinidine Sulphate in 15 cc. of boiling water and add a solution of 0.5 Gm. of potassium iodide in 5 cc. of water which has previously been neutralized to litmus paper with tenth-normal sulphuric acid if necessary. Mix well, cool the mixture to 15° C. and keep it at this temperature for one hour with frequent agitation. A white precipitate is formed. Filter and add 2 drops of ammonia water to the filtrate. No turbidity is produced within one minute (*other cinchona alkaloids*).

Preserve in well-closed containers protected from light.

Quininæ Ethylcarbonas (Quinine Ethylcarbonate). Euquinine. $C_{20}H_{23}O_2N_2.CO_2.C_2H_5$.

Quinine Ethyl Carbonate occurs in white, fine, soft needles, usually matted together in fleecy masses. It is odorless and possesses no bitter taste, but when masticated it slowly develops a slightly bitter taste. It darkens on exposure to light.

Quinine Ethyl Carbonate is only slightly soluble in water. 1 Gm. dissolves in 2 cc. alcohol, 1 cc. of chloroform, about 10 cc. ether, at 25° C. It is readily soluble in diluted acids.

It melts between 89° and 91° C.

Its saturated aqueous solution is slightly alkaline to litmus paper.

Its solution in diluted sulphuric acid exhibits a blue fluorescence.

To 5 cc. of an aqueous solution of Quinine Ethyl Carbonate (1 in 1000) made with the aid of a slight excess of diluted sulphuric acid, add 2 or 3 drops of bromine T. S. and follow with one cc. of ammonia water. The liquid acquires a green color.

To about 0.2 Gm. of Quinine Ethyl Carbonate add 2 cc. of sodium hydroxide T. S. and 5 cc. of iodine T. S. and gently warm the mixture. The odor of iodoform is evolved.

When about 1 Gm. of Quinine Ethyl Carbonate is digested with 10 cc. of a 5 per cent. solution of potassium hydroxide in dehydrated alcohol a white precipitate is gradually formed which effervesces with acids.

Ash not more than 0.2 per cent.

Dried over sulphuric acid for 24 hours the loss in weight does not exceed 2 per cent.

Dissolve 0.3 Gm. of Quinine Ethyl Carbonate in 10 cc. of diluted nitric acid and 20 cc. water. Ten-cc. portions of this solution are not rendered turbid at once by the addition of a few drops of silver nitrate T. S. (*chloride*) or of barium chloride T. S. (*sulphate*).

Preserve protected from light.

Pyroxylinum.—No change.

Quininæ Hydrobromidum.—The statement of origin is omitted.

Morphine test omitted.

Test for ammonium salts added. "One Gm. of Quinine Hydrobromide dissolves in 5 cc. of a mixture of 2 volumes of chloroform and 1 volume of dehydrated alcohol with not more than a slight turbidity (*ammonium or other inorganic salts*)."

Test for sulphate replaced by turbidimetric test.

Test for other Cinchona alkaloids changed.—"Dissolve about 3 Gm. of Quinine Hydrobromide in 80 cc. warm water in a separator, add 10 cc. ammonia water, shake out the mixture successively with 30 and 20 cc. of chloroform, and evaporate the chloroform solution to dryness on the water-bath. Dissolve 1.5 Gm. of this residue in 25 cc. of alcohol, dilute the solution with 50 cc. of hot water, add normal sulphuric acid (about 5 cc.) until the solution is acid, using 2 drops of methyl red T. S. as indicator, then neutralize the excess of acid with normal sodium hydroxide. Evaporate the liquid to dryness on a water-bath, powder the residue, mix it in a test tube with 20 cc. of distilled water and complete the test for other cinchona alkaloids as described under Quinine Sulphas."

Quininæ Tannas.—Test for uncombined Quinine changed.—One Gm. of salt is directed and the residue should not exceed 1 per cent.

Sapo.—No change in description or tests but the synonym changed to "White Olive Oil-Castile Soap."

Sulphonmethanum.—No change.

Terebenum.—The definition "A liquid consisting of dipentene and other hydrocarbons, obtained by the action of concentrated sulphuric acid on oil of turpentine" is replaced by "A mixture of terpene hydrocarbons, chiefly dipentene and terpinene, obtained from Oil of Turpentine."

Thyroidum.—No change.

Thyroxinum (Thyroxin).—Triiodo oxyindol beta-propionic acid $(C_8H_5ONI_3).C_2H_4COOH$.

The active principle of the thyroid gland. It contains not less than 63 per cent. of iodine.

Thyroxin occurs as white or slightly yellow, needle-like, odorless crystals or powder.

Thyroxin is insoluble in water and practically insoluble in alcohol or other usual organic solvents, but in the presence of mineral acids it dissolves in alcohol. It is soluble in solutions of alkali hydroxides and when the alkaline solutions are saturated with sodium chloride the sodium salt of thyroxin separates.

Ignite a few milligrams of Thyroxin with about 0.1 Gm. of sodium carbonate, dissolve the

residue in 2 cc. of water, acidulate with acetic acid, add 1 cc. of chloroform and a few drops of chlorine water. The chloroform acquires a violet color.

Ash from 0.01 Gm. is negligible.

Dry about 0.02 Gm. of Thyroxin over sulphuric acid for 24 hours. The loss in weight is negligible.

Shake 0.01 Gm. of Thyroxin with 10 cc. of water during 5 minutes and filter. Acidulate the filtrate with one drop of diluted nitric acid and add 3 drops of tenth-normal silver nitrate. The turbidity so produced is not greater than that produced in a control test by one drop of fiftieth-normal hydrochloric acid (*soluble iodides*).

Assay.—Weigh accurately about 0.02 Gm. of Thyroxin previously dried over sulphuric acid for 24 hours. Mix it with about 0.5 Gm. of anhydrous potassium carbonate in a small platinum crucible, cover the mixture with an additional 1 Gm. of anhydrous potassium carbonate and heat gradually until it is completely decomposed. Treat with water and transfer into 100-cc. graduated flask. Heat the solution on the water-bath and add potassium permanganate solution (1 in 20) drop by drop until the liquid remains pink. Then add drop by drop just enough alcohol to discharge the pink color, cool, dilute to the mark with boiled out, distilled water. Mix well and filter through a dry filter into a dry flask, rejecting the first 20 cc. of the filtrate. To 50 cc. of the filtrate add about 0.5 Gm. potassium iodide and 30 cc. of diluted sulphuric acid and titrate the liberated iodine with two-hundredth normal sodium thiosulphate using starch T. S. as indicator towards the end. Correct for any iodine liberated in a blank made with the same quantities of water, sulphuric acid, and potassium iodide. Each cc. of two-hundredth normal sodium thiosulphate corresponds to 0.01058 milligram of iodine.

CORRESPONDENCE

CORRESPONDENCE RELATING TO RESOLUTIONS OF THE HOUSE OF DELEGATES A. PH. A.

Secretary William B. Day has received replies to his communications from the Departments of the Federal Government. The following refer to Resolutions Nos. 16 and 24, which read:

16. *Resolved*, That the House of Delegates of the A. Ph. A. request the heads of those departments of the Federal Government having jurisdiction and supervision of matters relating to pharmaceutical practice to employ one or more registered pharmacists in each and every such department.

24. WHEREAS, It has come to our ears that the Medical Department of the Army, the U. S. Public Health Service and The General Supply Committee of the Treasury Department have eliminated the metric system from their specifications for the purchase of medical supplies, and

WHEREAS, There exists good reason to believe that the Navy Department and the Veterans Bureau are considering doing likewise,

Be It Resolved, That this House of Delegates of the American Pharmaceutical Association considers the elimination of the metric system for the purchase of supplies as a distinct backward step. This House of Delegates also wishes to express the sincere hope that the use of the metric system will be continued in the hospitals and other institutions under the supervision of the three departments mentioned above.

Be It Furthermore Resolved, That the Navy Department and the Veterans Bureau be requested to continue the use of the metric system for the purchase of medical supplies.

Resolved, That the Secretary of the House of Delegates is hereby directed to send copies of these resolutions to each of the respective departments mentioned.

ACTING SURGEON GENERAL M. J. WHITE.—I am in receipt of your letter of November 6, 1923, quoting a resolution of the House of Delegates of the American Pharmaceutical Association relative to the employment of registered pharmacists in the departments of the Federal Government having jurisdiction and supervision of matters relating to pharmaceutical practice, and bearing upon the use of other standard measures than those of the metric system in the purchase of medical supplies.