

Contributed and Selected

UNITED STATES PHARMACOPCEIA.

NINTH REVISION.

ABSTRACT OF PROPOSED CHANGES WITH NEW STANDARDS AND DESCRIPTIONS.

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PART V—FIRST PROOF.

A fifth installment of the Abstract of proposed new descriptions and standards and of changes in descriptions and standards is herewith submitted.

This Abstract embraces most of the Biological Products and Volatile Oils. Where no reference is made to rubrics, tests or assays, it is understood that the material facts remain the same as in the *United States Pharmacopœia*, Eighth Revision.

Other Abstracts will be submitted later. Comments should be sent to the Chairman of the Revision Committee, Joseph P. Remington, Longport, New Jersey, before September 1, 1914.

BIOLOGICAL PRODUCTS.

Glandulæ Suprarenales Sicca.—The suprarenal glands of such animals as are used for food by man, cleaned, dried, freed from fat, and powdered, and yielding not less than 0.4 percent. nor more than 0.6 percent. of epinephrine. Added requirement: Not more than 7 percent. of moisture. *Assay*: Add 0.005 Gm. of finely powdered manganese dioxide and 10 Cc. of distilled water to 0.010 Gm. of Desiccated Suprarenal Glands; thoroughly shake the mixture several times during one hour and filter. Compare the color of the filtrate in a test-tube or in any convenient manner, with the color of standard solutions made as follows: Mix 1.85 Cc. of cobaltous chloride T. S. with 0.95 Cc. of gold chloride T. S. and 7.2 Cc. of distilled water; the color corresponds to 0.2 percent. of epinephrine in the filtrate obtained above; 2.95 Cc. of cobaltous chloride T. S. with 1.25 Cc. of gold chloride T. S. and 5.8 Cc. of distilled water corresponds in color to 0.4 percent. of epinephrine; 4.05 Cc. of cobaltous chloride T. S. with 1.35 Cc. of gold chloride T. S. and 4.6 Cc. of distilled water corresponds in color to 0.6 percent. of epinephrine; 5.15 Cc. of cobaltous chloride T. S. with 1.55 Cc. of gold chloride T. S. and 3 Cc. of distilled water corresponds in color to 0.8 percent. of epinephrine. The percentages of epinephrine indicated by the above color standards are based upon the maceration of 0.010 Gm. of the Desiccated Suprarenal Glands in 10 Cc. of distilled water as directed above and filtering. In samples containing more than 0.8 percent. of epinephrine, 0.005 Gm. of the Desiccated Suprarenal

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Glands may be taken, in which case the percentage stated above, as indicated by the color standards, should be doubled. The standard color solutions keep unchanged indefinitely if sealed in test-tubes. The former test with ferric chloride T. S. is omitted.

Note.—The test solutions required above are made as follows:

Cobaltous Chloride T. S.—Two Gm. of Cobaltous Chloride dissolved with the aid of 1 Cc. of hydrochloric acid in sufficient distilled water to measure 100 Cc.

Gold Chloride T. S.—An aqueous solution of Gold Chloride containing 0.1 Gm. of gold in each 100 Cc. of solution, determined by analysis.

Glandulae Thyroideae Sicca.—The Thyroid Glands of such animals as are used for food by man, freed from connective tissue and fat, dried and powdered and yielding not less than 0.17 percent. nor more than 0.23 percent. of iodine in thyroid combination. Iodine in inorganic or any other form of combination than that peculiar to the thyroid must be absent. Added requirement: Not more than 6 percent. of moisture. Ash changed from 6 percent. to 5 percent. Assay for iodine replacing former test for iodine compounds: Mix 1 Gm. of Desiccated Thyroid Glands in a nickel crucible of about 125 Cc. capacity, with 15 Gm. of a mixture composed of 138 parts by weight of anhydrous potassium carbonate, 106 parts of anhydrous sodium carbonate and 75 parts of potassium nitrate. Spread an additional 5 Gm. of this fusion mixture evenly over the surface. Heat the crucible over a Bunsen flame until no further carbonization is observed, cool it and dissolve the residue in about 150 Cc. of distilled water, warming to hasten solution. Transfer this solution to an Erlenmeyer flask of about 500 Cc. capacity, and add approximately 50 Cc. of a fresh solution of chlorinated soda. Now treat the mixture with enough phosphoric acid, diluted with an equal volume of distilled water, to produce an appreciable yellow tint of free chlorine, then add 10 Cc. more of the phosphoric acid diluted with an equal volume of distilled water and boil the contents of the flask for one-half hour or until the volume has been reduced to about 150 Cc. Cool the liquid, add 10 Cc. of an aqueous solution of potassium iodide (1 in 100) and titrate the liberated iodine with two-hundredth normal sodium thiosulphate V. S., starch paste being used as indicator just before the end of the reaction.

Serum Antidiphthericum.—Added description: "With sometimes a slight granular sediment." Definition and description otherwise as in U. S. P. VIII. The serum must come from healthy animals; must be sterile; must be free from toxin or bacterial products; and must not contain an excessive amount of preservative (0.5 percent. phenol, or 0.4 percent. cresol when such are used); and the total solids should not exceed 20 percent. Serum of a lower potency than 250 units per cubic centimeter is not to be sold. Only Serums may be sold as have been prepared and propagated in establishments licensed by the Secretary of the Treasury of the United States. The law requires that each container of Serum sold by licensed establishments shall bear upon the label, in addition to the name of the Serum, the name, address and license number of the manufacturer, and the date beyond which the contents cannot be expected to yield its specific results. The label should also contain the laboratory number of the Serum, the name and the percentage by volume of the antiseptic used (if such

be used) and the total number of antitoxic units claimed for the contents of the container.

Serum Antidiphthericum Purificatum.—A solution in physiological solution of sodium chloride of certain antitoxic proteins obtained from the blood serum or plasma of a horse, actively immunized against diphtheria toxin. After the serum or plasma from the immunized horse has been collected, the non-antitoxic proteins are removed by precipitation with ammonium sulphate and by dissolving the precipitate in saturated sodium chloride solution, the salts being then removed by dialysis. After dialysis is complete, sufficient sodium chloride is added to make an 0.8 percent. solution. Preserve in sealed glass containers in a dark place, at a temperature between 4.5° and 15° C. A transparent or slightly opalescent liquid, with sometimes a slight granular or ropy sediment, odorless, or having an odor due to the presence of the antiseptic used as a preservative. The liquid is sometimes more or less viscous. The serum must come from healthy animals; must be sterile; must be free from toxin or bacterial products and must not contain an excessive amount of preservative (0.5 percent. phenol or 0.4 percent. cresol, when such are used); and the total solids should not exceed 20 percent. Serum of a lower potency than 250 units per cubic centimeter is not to be sold. Other requirements as under Serum Antidiphthericum.

Serum Antidiphthericum Siccum.—Dried Diphtheria Antitoxin is obtained by the evaporation of either Antidiphtheric Serum or Purified Antidiphtheric Serum in a vacuum, over sulphuric acid, or by passing over it a current of warm air freed from bacteria. Preserve in amber-colored glass containers free from air in a dark place, at a temperature between 4.5° and 15° C. The Dried Serum occurs either in the form of orange or yellowish flakes or small lumps, or as a yellowish-white powder, without odor. The serum is readily soluble in nine parts of distilled water. The solution is opalescent and slightly viscous. For use the Serum should be dissolved under the most rigid asepsis, preferably in the original container. Dried Antidiphtheric Serum does not lose in potency, as does the liquid Serum. Other requirements as under Serum Antidiphthericum.

Serum Antitetanicum.—A fluid separated from the coagulated blood of a horse, *Equus Caballus Linne'*, highly actively immunized against tetanus toxin. Preserve in sealed glass containers in a dark place, at a temperature between 4.5° and 15° C. A yellowish or yellowish-brown transparent or slightly turbid liquid with sometimes a slight granular sediment, odorless, or having an odor due to the presence of the antiseptic used as a preservative. Antitetanic Serum gradually loses its potency, the loss being greater at higher than at lower temperatures. The Serum must come from healthy animals; must be sterile; must be free from toxin or bacterial products; and must not contain an excessive amount of preservative (0.5 percent. phenol, or 0.4 percent. cresol, when such are used); and the total solids should not exceed 20 percent. Only such Serums may be sold as have been prepared and propagated in establishments licensed by the Secretary of the Treasury of the United States. The law requires that each container of Serum sold by licensed establishments shall bear upon the label, in addition to the name of the Serum, the name, address, and license number of the manufacturer, and the date beyond which the contents cannot be expected to yield its

specific results. The label should also contain the laboratory number of the Serum, the name and the percentage by volume of the antiseptic used (if such be used) and the total number of antitoxic units claimed for the contents of the container. The standard of strength, expressed in units of antitoxic power, shall be that established by the United States Public Health and Marine Hospital Service.

Serum Antitetanicum Purificatum.—A solution in physiological solution of sodium chloride of certain antitoxic proteins obtained from the blood serum or plasma of a horse, *Equus Caballus* Linné, and actively immunized against tetanus toxin. After the Serum or plasma from the immunized horse has been collected, the non-antitoxic proteins are removed by precipitation with ammonium sulphate and dissolving the precipitate in saturated sodium chloride solution, the salts being then removed by dialysis. After dialysis is complete, sufficient sodium chloride is added to make an 0.8 percent. solution. It should be kept in sealed glass containers in a dark place, at a temperature between 4.5° and 15° C. A transparent or slightly opalescent liquid, with sometimes a slight granular or ropy sediment, odorless, or having an odor due to the presence of the antiseptic used as a preservative. The liquid is sometimes more or less viscous. The Serum must come from healthy animals; must be sterile; must be free from toxin or bacterial products; and must not contain an excessive amount of preservative (0.5 percent. phenol or 0.4 percent. cresol, when such are used), and the total solids should not exceed 20 percent. Other requirements as under Serum Antitetanicum.

Serum Antitetanicum Siccum.—Dried Tetanus Antitoxin is obtained by the evaporation of either Antitetanic Serum or Purified Antitetanic Serum in a vacuum, over sulphuric acid, or by passing over it a current of warm air freed from bacteria. It should be kept in amber-colored glass containers free from air in a dark place at a temperature between 4.5° and 15° C. The Dried Serum is either in the form of orange or yellowish flakes or small lumps, or a yellowish-white powder, without odor. The Serum is readily soluble in nine-parts of distilled water. The solution is opalescent and slightly viscous. For use the Serum should be dissolved under the most rigid asepsis, preferably in the original container. Dried Antitetanic Serum does not lose in potency, as the liquid Serum does. It is sometimes used as a dusting powder or for local application to infected wounds. Other requirements as under Serum Antitetanicum.

Virus Vaccinicum.—The pustules of vaccinia or cowpox removed, under aseptic conditions, from vaccinated animals of the bovine species. It should be kept in a dark place, at a temperature between 4.5° and 15° C. The vaccine pulp should be thoroughly rubbed up in a mortar or passed through a special grinder, strained to remove coarse particles, and made into a smooth emulsion with a glycerin solution. Vaccine Virus gradually loses in potency, the loss being more rapid at high temperatures than at low temperatures. Only such Vaccine Virus may be sold as has been prepared in establishments licensed by the Secretary of the Treasury of the United States. It is required by the law and the regulations now in force that the following requirements be observed. No Vaccine shall be used from any animal having a communicable disease, or suspected of having a com-

municable disease, other than vaccinia; animals used for propagating Vaccine Virus must be under daily veterinary examination for a period of not less than seven days before vaccination, and as soon as the vaccine pulp is removed a necropsy shall be made on each animal and permanent records kept of the same. Each and every lot of Vaccine Virus shall be examined to determine its freedom from pathogenic micro-organisms and a special examination must be made of each lot to determine the absence of tetanus spores or toxin, and permanent records must be kept of these examinations. The Virus must be marketed in sterile containers that comply with the requirements of the law and the regulations established by the United States Government mentioned above. Each package of Vaccine Virus shall bear upon the label the name, address and license number of the manufacturer and the date beyond which the contents cannot be expected to yield their specific results. The label should also contain the laboratory number of the Virus.

VOLATILE OILS.

The following article replaces the U. S. P. VIII text for Oil of Sweet Birch, Oil of Gaultheria and Methyl Salicylate.

Methylis Salicylas.—A product yielding, when assayed by the process given below, not less than 98 percent. of methyl salicylate ($\text{CH}_3\text{C}_7\text{H}_7\text{O}_2=152.06$). It is produced synthetically or obtained by distillation from *Betula lenta* Linné (Fam. Betulaceæ) or from *Gaultheria procumbens* Linné (Fam. Ericaceæ) and the source from which it is derived in every case must be stated on the label. Preserve it in well-stoppered, amber-colored bottles, in a cool place, protected from light. It is a colorless, yellowish or reddish liquid, having the characteristic odor and taste of Gaultheria. Specific gravity at 25° C.: Synthetic 1.180 to 1.185; when from Sweet Birch or Gaultheria 1.172 to 1.180. Boiling Point: 218° to 221° C. Synthetic Methyl Salicylate, or that from Sweet Birch, is optically inactive; when obtained from Gaultheria, it is slightly laevogyrate, not exceeding -1.5° in a 100 mm. tube at 25° C. Sparingly soluble in water; soluble in all proportions in alcohol and glacial acetic acid. It is soluble in 6 volumes of 70 percent. alcohol at 25° C. with not more than a slight cloudiness. The alcoholic solution is neutral or slightly acid to moistened litmus paper. A deep violet color will be produced by shaking a drop of Methyl Salicylate with about 5 Cc. of distilled water and adding a drop of ferric chloride T. S. Add 10 Cc. of potassium hydroxide T. S. to 1 Cc. of Methyl Salicylate, contained in a capacious test-tube, and agitate the mixture. A clear, colorless or faintly yellowish solution results, without the separation of any oily drops either on the surface or at the bottom of the liquid (other volatile oils or petroleum). It does not respond to the Volatile Oil Heavy Metals Test. *Assay for Methyl Salicylate*: Introduce about 2 Cc. of Methyl Salicylate into a tared flask, note the exact weight, add 50 Cc. of half-normal alcoholic potassium hydroxide V. S., connect the flask with a reflux condenser and heat the mixture on a water-bath during two hours. Then add a few drops of phenolphthalein T. S. and titrate the excess of alkali with half-normal hydrochloric acid V. S., noting the amount required. This shows, when calculated from the weight of the sample originally taken, not less than 98 percent. of methyl salicylate.

Oleum Amygdalæ Amarae.—A volatile oil obtained by maceration and distillation from the ripe kernel of *Prunus Amygdalus Stokes var. amara* DeCandolle (Fam. Rosacæ), and from other kernels containing amygdalin, the source from which it is derived in every case to be stated on the label; yielding not less than 85 percent. of benzaldehyde and not less than 2 percent. nor more than 4 percent. of hydrocyanic acid. This Oil is intended for medicinal use; it is not to be used for flavoring foods. Specific gravity changed from "1.045 to 1.060" to "from 1.038 to 1.060" at 25° C. Added requirement: Refractive index: 1.5428 to 1.5439 at 20° C. "Optically inactive" changed to "optically inactive or dextrogyrate, not exceeding +0° 10' in a 100 mm. tube at 25° C." Soluble in "equal volume of 70 percent. alcohol" changed to "dissolves, forming a clear solution, in 2 volumes of 70 percent. alcohol." The test for presence of hydrocyanic acid and the flame test for chlorinated products omitted. Added test: Add 10 drops of the Oil to a little alcohol, introduce a small amount of zinc dust and 2 Cc. of acetic acid and boil the mixture for a short time; no odor of phenyl isonitrile should develop after rendering it strongly alkaline with potassium hydroxide T. S., adding a drop of chloroform and heating (nitro-benzene). *Assay for Benzaldehyde*: Dissolve 3 Gm. of phenylhydrazine (not darker in color than pale yellow) in 50 Cc. of alcohol and titrate 25 Cc. of the solution with half-normal hydrochloric acid V. S., using methyl orange T. S. as indicator, to a distinct change in color. To about 1 Gm. of Oil of Bitter Almond, accurately weighed, add 25 Cc. of the phenylhydrazine solution just prepared and titrate the mixture after thirty minutes with half-normal hydrochloric acid V. S. as just described. The difference between the number of cubic centimeters of half-normal hydrochloric acid V. S. required in the two titrations, multiplied by 0.053 will show the weight of benzaldehyde present. Always freshly prepare the phenylhydrazine solution when required for the assay. *Assay for Hydrocyanic Acid*: Dissolve 15 Gm. of crystallized magnesium sulphate in enough distilled water to measure 100 Cc., add 5 Cc. of this solution to 40 Cc. of distilled water, 5 Cc. of half-normal sodium hydroxide V. S. and two drops of potassium chromate T. S. and titrate the solution with tenth-normal silver nitrate V. S., to the production of a permanent reddish tint. Pour this mixture into a 100 Cc. flask containing about 1 Gm. of Oil of Bitter Almond, accurately weighed, and titrate again with tenth-normal silver nitrate V. S. until a red tint, which does not disappear on shaking, is reproduced.

Oleum Anisi.—A volatile oil distilled from the ripe fruit of *Pimpinella Anisum* Linné (Fam. Umbelliferæ), or from the ripe fruit of *Illicium anisatum* Linné (Fam. Magnoliaceæ), conforming in name to the plant from which it is derived. Specific gravity: Changed from "0.975 to 0.988" to "from 0.978 to 0.988 at 25° C." Added: Refractive index: 1.5440 to 1.5600 at 20° C. "Laevogyrate up to -2°" changed to "optical rotation varies from +1° to -2° in a 100 mm. tube at 25° C. (oil of fennel)." "Soluble in an equal volume of alcohol, forming a clear solution, also in 5 volumes of 90 percent. alcohol" changed to "soluble with not more than a slight cloudiness in 3 volumes of 90 percent. alcohol." Cool the oil in determining congealing point to 12° C. instead of 6° C. It does not respond to the Volatile Oil Heavy Metals Test as follows:

Volatile Oil Heavy Metals Test.—Shake 10 Cc. of the oil with an equal volume of distilled water and pass hydrogen sulphide through the mixture until it is saturated; no darkening in color will be produced in either the oil or the water (lead or copper):

Oleum Aurantii Corticis.—Obtained by expression from the fresh peel of *Citrus Aurantium sinensis* Galesio (Fam. Rutaceæ) and its varieties. Added: Refractive index: 1.4723 to 1.4737 at 20° C. Dextrogyrate—"not less than 95°" changed to "not less than 94°." Added test: Introduce 50 Cc. of Oil of Orange Peel into a 200 Cc. three-bulb Ladenburg flask of approximately the following dimensions: The lower or main bulb 6 cm. in diameter with the smaller condensing bulbs 3.5 cm., 3.0 cm. and 2.5 cm. in diameter, respectively; with the distance from the bottom of the flask to the side arm 20 cm. Distil the Oil at the rate of one drop per second until 5 Cc. has been obtained. The angle of rotation of the first 10 percent. of the distillate thus obtained is equal to or slightly greater than that of the original sample. The refractive index of this first 10 percent. of distillate is not less than 0.0008, nor more than 0.0015 lower than that of the original sample at 20° C. Nitrosolimonene test for added Oil of Turpentine omitted.

Oleum Cadinum.—Added specific gravity: 0.980 to 1.055 at 25° C. "Completely soluble in ether" changed to "Completely soluble in 3 volumes of ether, amyl alcohol, chloroform, glacial acetic acid, or oil of turpentine, but only partly soluble in petroleum benzin." Added tests: One part of the Oil of Cade shaken with 20 parts of warmed distilled water and filtered, yields a filtrate which gives with a few drops of ferric chloride solution (1 in 1000) a red coloration. Portions of this aqueous filtrate reduce silver nitrate in the cold and Fehling's solution on heating. An aqueous filtrate from a mixture with Oil of Cade (1 in 20) produces no red coloration on the addition of a few drops of aniline (wood tar products); another portion of the aqueous filtrate is not colored by the addition of potassium chromate T. S. (coal tar products). Agitate 1 Cc. of Oil of Cade with 15 Cc. of purified petroleum benzin, filter the benzin solution, add an equal volume of copper acetate solution (1 in 20), shake the mixture and then allow it to separate. On adding an equal volume of ether to the separated benzin solution, it produces no intensely green coloration and does not become colored more than from a light yellow to brown (rosin and rosin oil).

Oleum Cajuputi.—Distilled from the fresh leaves and twigs of several varieties of *Melaleuca Leucadendron* Linné, especially the var. *Cajuputi* Roxburgh and the var. *minor* Smith (Fam. Murtaceæ). Rubric and assay for cineol content omitted. "Colorless or greenish liquid" changed to "colorless or yellowish liquid." Specific gravity changed from "0.915 to 0.925" to "from 0.912 to 0.925 at 25° C." Laevogyrate—"not exceeding —2°" changed to "not exceeding —4° in a 100 mm. tube at 25° C." Added test: It does not respond to the Volatile Oil Heavy Metals Test.

Oleum Cari.—Yielding not less than 50 percent., by volume, of carvone, "Soluble in an equal volume of alcohol, also in from 3 to 10 volumes of 80 percent. alcohol" changed to "soluble in 8 volumes of 80 percent. alcohol. Assay for Carvone: Introduce 10 Cc. of the Oil into a 200 Cc. flask, with a

long, graduated neck, by means of a graduated pipette, add 50 Cc. of a saturated solution of sodium sulphite, which has been carefully neutralized by means of acetic acid containing a few drops of phenolphthalein T. S., heat the mixture in a bath containing boiling water and shake the flask repeatedly, neutralizing the mixture from time to time by the addition of a few drops of diluted acetic acid. When no coloration appears, upon the addition of a few more drops of phenolphthalein T. S. and heating for fifteen minutes, bring the residual oil into the neck of the flask by the further addition of the sodium sulphite solution and ascertain its volume. The difference in volume between this residue and the volume of the original oil (10 Cc. multiplied by 10), is equivalent to the percentage by volume of the carvone present.

Oleum Caryophylli.—Distilled from the flower-buds of *Eugenia Aromatica* (Linné) O. Kuntze *Jambosa Caryophyllus* (Sprengel) Niedenzu (Fam. Myrtaceæ). Rubric changed from "80 percent." to "82 percent." of eugenol. Specific gravity: Changed from "1.040 to 1.060" to "from 1.038 to 1.060 at 25° C." Added test: Slightly laevogyrate, not exceeding $-1^{\circ} 10'$ in a 100 mm. tube at 25° C. Tests with potassium hydroxide solution or ammonia water and with ferric chloride T. S. omitted. In the assay the mixture of 50 Cc. of potassium hydroxide T. S. and 10 Cc. of oil, after shaking it for five minutes, is heated on a water-bath for ten minutes to complete the reaction.

Oleum Chenopodii.—Distilled from *Chenopodium ambrosioides anthelminticum* Linné (Fam. Chenopodiaceæ). Added tests: Specific gravity: 0.955 to 0.980 at 25° C. It is laevogyrate, varying between -4° and -10° in a 100 mm. tube at 25° C. It is soluble in 8 volumes of 70 percent. alcohol.

Oleum Cinnamomi.—Distilled from the young twigs of *Cinnamomum Cassia* (Nees) Blume (Fam. Lauraceæ), rectified by steam distillation. Rubric changed from "75 percent." to "80 percent." of cinnamic aldehyde. Specific gravity changed from "1.045 to 1.055" to "from 1.045 to 1.063 at 25° C." "Almost optically inactive" changed to "optical rotation varies from $+1^{\circ}$ to -1° in a 100 mm. tube at 25° C." Hydrogen sulphide test replaced by "It does not respond to the Volatile Oil Heavy Metals Test." Lead acetate test for rosin replaced by the following: Shake 2 Cc. of the Oil in a test-tube with from 5 to 10 Cc. of purified petroleum benzin and decant the latter. This liquid is colorless and does not give a green color upon shaking with an equal volume of copper acetate solution (1 in 1000) (rosin). *Assay for Cinnamic Aldehyde*: Assay as for carvone under *Oleum Cari*.

Oleum Coriandri.—Specific gravity: Changed from "0.863 to 0.878" to "from 0.863 to 0.875 at 25° C." Optical rotation changed from " $+7^{\circ}$ to $+14^{\circ}$ " to "from $+8^{\circ}$ to $+13^{\circ}$ in a 100 mm. tube at 25° C." It is soluble in 3 volumes of 70 percent. alcohol. Solubility in 80 and 90 percent. alcohol omitted.

Oleum Cubebæ.—Optical rotation changed from " -25° to -40° " to "from -20° to -40° in a 100 mm. tube at 25° C."

Oleum Eucalypti.—Distilled from the fresh leaves of *Eucalyptus Globulus* Labillardière (Fam. Myrtaceæ) or from other species of *Eucalyptus*. Rubric changed from "50 percent." to "not less than 70 percent." of eucalyptol. "Soluble in all proportions in alcohol; also soluble in 3 volumes of 70 percent. alcohol"

changed to "soluble in 4 volumes of 70 percent. alcohol." Optical rotation omitted. *Assay for Eucalyptol* (replacing former assay): Introduce 10 Cc. of the Oil into a 100 Cc. flask with a long, graduated neck (cassia flask) by means of a graduated pipette, add enough of an aqueous solution of resorcinol (1 in 2) to fill the flask about four-fifths full. Shake the mixture thoroughly for five minutes and then bring the residual oil into the neck of the flask by the further addition of the same strength resorcinol solution, rotating or gently tapping the flask, if necessary, to cause the oil to rise to the surface. When the resorcinol solution has become clear (usually after standing for several hours) ascertain the volume of the residual oil. The difference in volume between this residue and the volume of the original Oil (10 Cc.) multiplied by 10, is equivalent to the percentage, by volume, of the eucalyptol present. When Oils are rich in eucalyptol, dilute the 10 Cc. taken for the assay with an equal volume of oil of turpentine, before applying the test, to avoid crystallization in the resorcinol solution.

Oleum Fœniculi.—Distilled from the ripe fruit collected from cultivated varieties of *Fœniculum vulgare* Miller (Fam. Umbelliferæ). Added test: Optical rotation varies from $+12^{\circ}$ to $+24^{\circ}$ in a 100 mm. tube at 25° C. "Soluble in an equal volume of alcohol; also soluble in 10 volumes or less of 80 percent. alcohol" changed to "soluble in 8 volumes of 80 percent. alcohol and 1 volume of 90 percent. alcohol." Congealing point changed from "not below 5° C." to "not below 3° C." Proceed as directed under *Oleum Anisi*, cooling the Oil to 0° C.

Oleum Hedeomæ.—Distilled from the flowering plant of *Hedeoma pulegioides* (Linné) Persoon (Fam. Labiataë). Optical rotation changed from " $+18^{\circ}$ to $+22^{\circ}$ " to "from $+17^{\circ}$ to $+28^{\circ}$ in a 100 mm. tube at 25° C." Added to solubility requirement, "forming a solution showing not more than a slightly acid reaction with litmus."

Oleum Juniperi.—Distilled from the ripe fruit of *Juniperis communis* Linné (Fam. Pinaceæ). Specific gravity changed from "0.860 to 0.880" to "from 0.854 to 0.879 at 25° C." Added tests: The optical rotation varies from 0° to -15° , in a 100 mm. tube at 25° C. It is soluble in 4 volumes of alcohol with not more than a slight cloudiness.

Oleum Lavandulæ.—Distilled from the fresh flowering tops of *Lavandula vera* DeCandolle (*Lavandula officinalis* Chaix, *Lavandula spica* Linné) (Fam. Labiataë). Specific gravity changed from "0.875 to 0.910" to "from 0.875 to 0.888 at 25° C." Added tests: The optical rotation varies from -1° to -10° in a 100 mm. tube at 25° C. Shake 20 Cc. of the Oil with 40 Cc. of 5 percent. alcohol and when the mixture has cleared withdraw 30 Cc. of the alcoholic solution. Neutralize this with half-normal potassium hydroxide V. S., using phenolphthalein T. S. as indicator, then add 5 Cc. of half-normal potassium hydroxide V. S., and heat the mixture on a water-bath under a reflux condenser during one hour. Not less than 4.7 Cc. of half-normal hydrochloric acid V. S. is required for neutralization after saponification.

Oleum Limonis.—Obtained by expression from the fresh, ripe peel of *Citrus medica* Linné variety *Limonum* (Risso) Hooker filius (Fam. Rutaceæ). Added test: Refractive index: 1.4744 to 1.4755 at 20° C. Optical rotation changed from "not less than $+58^{\circ}$ " to "from $+57^{\circ}$ to $+64^{\circ}$ in a 100 mm. tube at 25° C." Added test: The angle of rotation of the first 10 percent. of the Oil, obtained by

distillation, as described under *Oleum Aurantii Corticis*, is not more than 5° less than that of the original Oil. The refractive index of this first 10 percent. of distillate is not less than 0.0020 nor more than 0.0027 lower than that of the original oil at 20° C. *Assay for Citral*: (Replacing former assay.) Introduce about 15 Cc. of Oil of Lemon into a tared 300 Cc. flask, by means of a pipette, and note the exact weight; add 10 Cc. of a solution of phenylhydrazine (not darker in color than pale yellow) in alcohol (1 in 10), and allow it to stand for one-half hour at room temperature. Then add a few drops of methyl-orange T. S. and neutralize the liquid exactly by the cautious addition of half-normal hydrochloric acid V. S. If difficulty is experienced in detecting the end point of the reaction, carry the titration until the solution is distinctly acid, transfer it to a separatory funnel and draw off the alcoholic portion. Now wash the Oil with distilled water, adding the washings to the alcoholic solution, and titrate the latter with half-normal potassium hydroxide V. S. Carry out a blank test identical with the foregoing except that the Oil of Lemon is omitted, and note the amount of half-normal hydrochloric acid V. S. consumed. Subtract the number of cubic centimeters of half-normal potassium hydroxide V. S. from the number of cubic centimeters of half-normal hydrochloric acid V. S. used in the original test and this result from the corresponding number of cubic centimeters required in the blank test; each cubic centimeter of this difference corresponds to 0.076 Gm. of aldehydes, calculated as citral.

Oleum Mentha Piperita.—Distilled from the flowering plant of *Mentha piperita* Linné (Fam. Labiatae), rectified by steam distillation. Ester content in rubric changed from "6 percent." to "not less than 5 percent." Laevogyrate changed from "—20° to —33°" to "from —20° to —35° in a 100 mm. tube at 25° C." Solubility in alcohol and reaction to litmus omitted, and "no separation of oil globules" added to solubility in 70 percent. alcohol statement. Modified test: Distil about 1 Cc. from 25 Cc. of the Oil and pour the distillate on an aqueous solution of mercuric chloride (1 in 25); a white film does not form at the zone of contact within one minute (dimethyl sulphide—found in non-rectified oils). *Assay for Esters and Total Menthol*: Ten Cc. of the original oil is taken for the menthol assay instead of the washed residual oil from the menthyl acetate assay; otherwise the assay remains unchanged.

Oleum Mentha Viridis.—Distilled from the flowering plant of *Mentha spicata* Linné (*Mentha viridis* Linné) (Fam. Labiatae). Added rubric: Yielding not less than 40 percent., by volume, of carvone. Laevogyrate changed from "—35° to —48°" to "from —35° to —50° in a 100 mm. tube at 25° C." *Assay for Carvone*: Assay as directed for carvone under *Oleum Cari*.

Oleum Myristica.—Specific gravity changed from "0.884 to 0.924" to "from 0.859 to 0.924 at 25° C." Added test: Optical rotation varies from +14° to +30° in a 100 mm. tube at 25° C. Modified test: Evaporate 3 Gm. of the Oil on a water-bath; not more than 0.06 Gm. of residue should remain.

Oleum Picis Liquida.—Specific gravity changed from "about 0.892" to "from 1.012 to 1.065 at 25° C." It is soluble in alcohol, the solution showing an acid reaction with litmus.

Oleum Pimenta.—Specific gravity changed from "1.028 to 1.048" to "from

1.018 to 1.048 at 25° C." ~~Added test:~~ Its optical rotation varies from 0° to -4° in a 100 mm. tube at 25° C. "Miscible in all proportions in 90 percent. alcohol" changed to "soluble in an equal volume of 90 percent. alcohol." *Assay for Eugenol:* Assay as directed under *Oleum Caryophylli*.

Oleum Pini Pumilionis.—A volatile oil distilled with steam from the fresh leaves of *Pinus montana* Miller (*Pinus Pumilio* Haenke) (Fam. Pinaceæ). It is a colorless or faintly yellowish colored oil having a pleasant, aromatic odor, and a bitter and pungent taste. Specific gravity: 0.853 to 0.869 at 25° C. No portion of the Oil distils below 170° C.

Oleum Rosmarini.—Solubility in 90 percent. alcohol omitted. *Assay for Ester and Total Borneol:* Assay as directed under *Oleum Menthæ Piperitæ*, the Oil of Rosemary factors remaining unchanged.

Oleum Santali.—Optical rotation changed from "-16° to -20°" to "from -15° to -20° in a 100 mm. tube at 25° C." Soluble in 5 volumes of 70 percent. alcohol, forming a solution having a slightly acid reaction with litmus. Solubility in alcohol omitted. *Assay for Santalol:* Assay as directed under *Oleum Menthæ Piperitæ*, using the factors under Oil of Santal, but changing "11.026" to "10.926."

Oleum Sassafras.—Distilled from the root of *Sassafras variifolium* (Salisbury) O. Kuntze (Fam. Lauraceæ). Specific gravity changed from "1.065 to 1.075" to "from 1.065 to 1.077 at 25° C." Optical rotation changed from "not more than +4°" to "from +3° to +4° in a 100 mm. tube at 25° C." Added test: Soluble in 2 volumes of 90 percent. alcohol, forming a solution neutral to litmus.

Oleum Sinapis Volatile.—A product yielding, when assayed by the process given below, not less than 92 percent. of allyl iso-thiocyanate ($\text{CSNC}_3\text{H}_5 = 99.12$). It is produced synthetically or obtained from the seed of *Brassica nigra* (Linné) Koch (Fam. Cruciferæ) (freed from fatty oil) by maceration with water and subsequent distillation, and must conform in name to the source from which it is derived. Added test: Optically inactive. Modified tests: The Oil completely distils between 148° and 154° C., and both the first and last portions have nearly the same specific gravity as the original Oil (alcohol, chloroform, petroleum, and fatty oils). The addition of a drop of ferric chloride T. S. to the Oil, diluted with several volumes of alcohol, does not produce a blue coloration (phenols). *Assay for Allyl Iso-thiocyanate:* Connect the flask with a reflux condenser when heating the mixture as directed in the assay for one hour—otherwise the process remains unchanged.

Oleum Terebinthinæ Rectificatum.—The freshly distilled Oil is to be dried by shaking it with anhydrous calcium chloride, and filtering. Specific gravity changed from "0.860 to 0.865" to "from 0.856 to 0.865 at 25° C." "No weighable residue on evaporation of 10 Cc." changed to "not more than 0.010 Gm. of residue."

Oleum Thymi.—Distilled from the flowering plant of *Thymus vulgaris* Linné (Fam. Labiatae). Specific gravity changed from "0.900 to 0.930" to "from 0.894 to 0.929 at 25° C." Optical rotation—"not more than -3°" omitted. "Soluble in half its volume of alcohol and in 1 volume of 80 percent. alcohol" omitted; "soluble in 2 volumes of 80 percent. alcohol" retained. The test with ferric chloride to show a greenish-brown color omitted. *Assay for Phenols:* The assay is to be conducted in a flask with a long, graduated neck instead of in a burette.