Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: LC
Detector: Refractive index
Column: 7.8-mm × 30-cm; 9-µm packing L19
Column temperature: 85°
Flow rate: 0.6 mL/min
Injection size: 20 µL
System suitability
Sample: *Standard solution*
Suitability requirements
Relative standard deviation: NMT 2.0% of replicate injections
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of tagatose ($C_6H_{12}O_6$) in the portion of Tagatose taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*
C_S = concentration of USP Tagatose RS in the *Standard solution* (mg/mL)
C_U = concentration of Tagatose in the *Sample solution* (mg/mL)

Acceptance criteria: NLT 98.0% on the dried basis

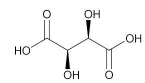
IMPURITIES
- **Limit of Lead**
Sample solution: 2.5 g of Tagatose dissolved in a mixture of 4 mL of sulfuric acid and 5 mL of hydrochloric acid. Dilute with water to 50 mL.
Standard stock solution A: Dissolve 1.60 g of lead nitrate in diluted nitric acid (10 mL of nitric acid diluted with 20 mL water, boiled to remove nitrous fumes, and cooled), and dilute with water to 1000 mL.
Standard stock solution B: *Standard stock solution A* and water (1:50). [Note—This solution contains the equivalent of 20 µg/mL of lead.]
Standard solutions: To a series of 100-mL volumetric flasks pipet 0, 1, 2, 3, 4, and 5 mL of *Standard stock solution B*, and dilute with water to about 50 mL. Add 8 mL of sulfuric acid and 10 mL of hydrochloric acid to each flask, shake to dissolve, and dilute with water to volume. [Note—These solutions contain 0, 0.2, 0.4, 0.6, 0.8, and 1.0 µg/mL of lead, respectively.]
Instrumental conditions
(See *Spectrophotometry and Light-Scattering* ⟨851⟩.)
Mode: Atomic absorption
Analytical wavelength: 283.3 nm
Analysis
Samples: *Standard solutions* and *Sample solution*
Concomitantly determine the absorbances of the *Standard solutions* and the *Sample solution*. Plot the absorbances of the *Standard solutions* versus the concentration of lead. Using this graph, determine the concentration of lead in the *Sample solution*.
Acceptance criteria: NMT 1 ppm

SPECIFIC TESTS
- **Microbial Enumeration Tests** ⟨**61**⟩ and **Tests for Specified Microorganisms** ⟨**62**⟩**:** It meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*. The total aerobic microbial count does not exceed 1000 cfu/g, and the total combined molds and yeasts count does not exceed 100 cfu/g.
- **Melting Range or Temperature,** *Class I* ⟨**741**⟩**:** 133°–144°
- **Optical Rotation,** *Specific Rotation* ⟨**781**⟩**:** –4° to –7°
Sample solution: 10 mg/mL
- **Loss on Drying** ⟨**731**⟩**:** Dry a sample at 102° for 2 h: it loses NMT 0.5% of its weight
- **Articles of Botanical Origin,** *Total Ash* ⟨**561**⟩**:** NMT 0.1%, determined on a 1.0-g specimen

ADDITIONAL REQUIREMENTS
- **Packaging and Storage:** Preserve in well-closed containers, and store at room temperature.
- **USP Reference Standards** ⟨**11**⟩
USP Tagatose RS

Talc—*see Talc General Monographs*

Tartaric Acid



$C_4H_6O_6$ 150.09
Butanedioic acid, 2,3-dihydroxy-;
Butanedioic acid, 2,3-dihydroxy-, [*R*-(*R**,*R**)]-;
Tartaric acid; L-(+)-Tartaric acid [526-83-0; 87-69-4].

DEFINITION
Tartaric Acid, dried over phosphorus pentoxide for 3 h, contains NLT 99.7% and NMT 100.5% of $C_4H_6O_6$.

IDENTIFICATION
- **A. Identification Tests—General,** *Tartrate* ⟨**191**⟩**:** It meets the requirements.
- **B. Infrared Absorption** ⟨**197K**⟩

ASSAY
- **Procedure**
Sample: 2 g of Tartaric Acid, previously dried, in a conical flask
Analysis: Dissolve the *Sample* in 40 mL of water, add phenolphthalein TS, and titrate with 1 N sodium hydroxide VS. Each mL of 1 N sodium hydroxide is equivalent to 75.04 mg of $C_4H_6O_6$.
Acceptance criteria: 99.7%–100.5%

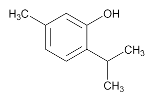
IMPURITIES
Inorganic Impurities
- **Residue on Ignition** ⟨**281**⟩**:** NMT 0.1%
- **Chloride and Sulfate,** *Sulfate* ⟨**221**⟩**:**
Sample solution: 10 mL of a 1-in-100 solution of Tartaric Acid
Analysis: Add to the *Sample solution* 3 drops of hydrochloric acid and 1 mL of barium chloride TS.
Acceptance criteria: No turbidity is produced.
- **Heavy Metals,** *Method II* ⟨**231**⟩**:** NMT 10 ppm

SPECIFIC TESTS
- **Optical Rotation,** *Specific Rotation* ⟨**781S**⟩**:** +12.0° to +13.0°
Sample solution: 200 mg/mL in water
- **Loss on Drying** ⟨**731**⟩**:** Dry over phosphorus pentoxide for 3 h: it loses NMT 0.5% of its weight.
- **Limit of Oxalate**
Sample solution: 10 mL of a 1-in-10 solution of Tartaric Acid
Analysis: Nearly neutralize the *Sample solution* with 6 N ammonium hydroxide, and add 10 mL of calcium sulfate TS.
Acceptance criteria: No turbidity is produced.

ADDITIONAL REQUIREMENTS
- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards** ⟨**11**⟩
USP Tartaric Acid RS

Thimerosal—*see Thimerosal General Monographs*

Thymol



$C_{10}H_{14}O$ 150.22
Phenol, 5-methyl-2-(1-methylethyl)-.
Thymol.
p-Cymen-3-ol [89-83-8].

» Thymol contains not less than 99.0 percent and not more than 101.0 percent of $C_{10}H_{14}O$.

Packaging and storage—Preserve in tight, light-resistant containers.

USP Reference standards ⟨11⟩—
USP Thymol RS

Identification—

A: *Infrared Absorption* ⟨197K⟩.

B: It meets the requirements under *Melting range.*

Melting range ⟨741⟩: between 48° and 51°; but when melted, Thymol remains liquid at a considerably lower temperature.

Limit of nonvolatile residue—Volatilize about 2 g, accurately weighed, on a steam bath, and dry at 105° to constant weight: not more than 0.05% of residue remains.

Assay—Transfer about 100 mg of Thymol, accurately weighed, to a 250-mL iodine flask, and dissolve in 25 mL of 1 N sodium hydroxide. Add 20 mL of hot dilute hydrochloric acid (1 in 2), and immediately titrate with 0.1 N bromine VS to within 1 to 2 mL of the calculated endpoint. Warm the solution to between 70° and 80°, add 2 drops of methyl orange TS, and continue the titration slowly, swirling vigorously after each addition. When the color of the methyl orange is bleached, add 2 drops of 0.1 N bromine VS, shake for 10 seconds, add 1 drop of methyl orange TS, and shake vigorously. If the solution is red, continue the titration, dropwise and with shaking, until the color is discharged. Repeat the alternate addition of the titrant and the methyl orange TS until the red color is discharged after the addition of the TS. Each mL of 0.1 N bromine is equivalent to 3.755 mg of $C_{10}H_{14}O$.

Titanium Dioxide—*see Titanium Dioxide General Monographs*

Tocopherols Excipient

» Tocopherols Excipient is a vegetable oil solution containing not less than 50.0 percent of total tocopherols, of which not less than 80.0 percent consists of varying amounts of beta, gamma, and delta tocopherols.

Packaging and storage—Preserve in tight containers, protected from light. Protect with a blanket of an inert gas.

Labeling—Label it to indicate the content, in mg per g, of total tocopherols and of the sum of beta, gamma, and delta tocopherols.

USP Reference standards ⟨11⟩—
USP Alpha Tocopherol RS

Identification—

A: Dissolve about 50 mg in 10 mL of dehydrated alcohol, add, with swirling, 2 mL of nitric acid, and heat at about 75° for 15 minutes: a bright red or orange color develops.

B: The retention time of the third major peak (i.e., the peak occurring just before that of the internal standard) in the chromatogram of the *Assay preparation* corresponds to that of the *Standard preparation*, both relative to that of the internal standard, as obtained in the *Assay*.

Acidity—Dissolve 1.0 g in 25 mL of a mixture of equal volumes of alcohol and ether (which has been neutralized to phenolphthalein with 0.1 N sodium hydroxide), add 0.5 mL of phenolphthalein TS, and titrate with 0.10 N sodium hydroxide until the solution remains faintly pink after being shaken for 30 seconds: not more than 1.0 mL of 0.10 N sodium hydroxide is required.

Assay —

Internal standard solution—Transfer about 600 mg of hexadecyl hexadecanoate, accurately weighed, to a 200-mL volumetric flask, dissolve in a diluting solution containing 2 volumes of pyridine and 1 volume of propionic anhydride, dilute with the diluting solution to volume, and mix.

Standard preparations—[NOTE—Use low-actinic glassware.] Transfer 12-, 25-, 37-, and 50-mg portions of USP Alpha Tocopherol RS, accurately weighed, to separate 50-mL conical flasks having 19/38 standard-taper ground-glass necks. Pipet 25 mL of the *Internal standard solution* into each flask, mix, and reflux for 10 minutes under water-cooled condensers.

Assay preparation—[NOTE—Use low-actinic glassware.] Transfer about 60 mg of Tocopherols Excipient, accurately weighed, to a 50-mL conical flask similar to the flasks used in preparing the *Standard preparations*, add 10.0 mL of *Internal standard solution*, mix, and reflux for 10 minutes under a water-cooled condenser.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector and contains a 4-mm × 2-m borosilicate glass column packed with 2% to 5% liquid phase G2 on 80- to 100-mesh support S1AB utilizing either a glass-lined sample introduction system or on-column injection. The column is maintained isothermally at a temperature between 245° and 265°, and the injection port and detector block temperatures are maintained at about 10° higher than the column temperature. The flow rate of dry carrier gas is adjusted to obtain a hexadecyl hexadecanoate peak 30 to 32 minutes after sample introduction. [NOTE—Cure and condition the column as necessary.]

System suitability—Chromatograph a sufficient number of injections of the *Assay preparation*, as directed under *Calibration*, to ensure that the resolution, *R*, between the major peaks occurring at retention times of approximately 0.50 (delta tocopheryl propionate) and 0.63 (beta plus gamma tocopheryl propionates), relative to hexadecyl hexadecanoate at 1.00, is not less than 2.5.

Calibration—Chromatograph 2- to 5-µL portions of each *Standard preparation*, and record the peak areas as directed for *Procedure*. Calculate the relative response factor, *F* , for each concentration of the *Standard preparation* taken by the formula:

$$(A_S / A_D)(C_D / C_S)$$

in which C_D and C_S are the concentrations, in mg per mL, of hexadecyl hexadecanoate and of USP Alpha Tocopherol RS, respectively, in the *Standard preparation.* Successively chromatograph a sufficient number of portions of each *Standard preparation* to ensure that the factor, *F*, is constant within a range of