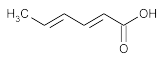
Sorbic Acid



$C_6H_8O_2$ 112.13
2,4-Hexadienoic acid, (*E*,*E*)-; 2,4-Hexadienoic acid; (*E*,*E*)-Sorbic acid; Sorbic acid [110-44-1].

DEFINITION
Sorbic Acid contains NLT 99.0% and NMT 101.0% of $C_6H_8O_2$, calculated on the anhydrous basis.

IDENTIFICATION
- **A. Infrared Absorption** ⟨197K⟩
- **B.** A 1-in-400,000 solution in isopropyl alcohol exhibits an absorbance maximum at 254 ± 2 nm.

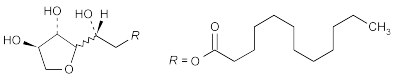
ASSAY
- **Procedure**
  Sample solution: Dissolve 250 mg of Sorbic Acid in a mixture of 50 mL of methanol and 25 mL of water that previously has been neutralized with 0.02 N sodium hydroxide. Add phenolphthalein TS.
  Analysis: Titrate with 0.1 N sodium hydroxide VS to the first pink color that persists for at least 30 s. Each mL of 0.1 N sodium hydroxide is equivalent to 11.21 mg of $C_6H_8O_2$.
  Acceptance criteria: 99.0%–101.0% on the anhydrous basis

IMPURITIES
Inorganic Impurities
- **Residue on Ignition** ⟨281⟩**:** NMT 0.2%
- **Heavy Metals,** *Method II* ⟨231⟩**:** 10 ppm

SPECIFIC TESTS
- **Melting Range or Temperature** ⟨741⟩**:** 132°–135°
- **Water Determination,** *Method I* ⟨921⟩**:** NMT 0.5%

ADDITIONAL REQUIREMENTS
- **Packaging and Storage:** Preserve in tight containers, protected from light, and avoid exposure to excessive heat.
- **USP Reference Standards** ⟨11⟩
  USP Sorbic Acid RS

Sorbitan Monolaurate



Sorbitan, esters, monododecanoate.
Sorbitan monolaurate [1338-39-2].

» Sorbitan Monolaurate is a partial ester of lauric acid with Sorbitol and its mono- and dianhydrides. It yields, upon saponification, not less than 55.0 percent and not more than 63.0 percent of fatty acids, and not less than 39.0 percent and not more than 45.0 percent of polyols (w/w).

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩**—**
USP Isosorbide RS
USP 1,4-Sorbitan RS
$C_6H_{12}O_5$ 164.16

Identification—
A: The residue of lauric acid obtained in the *Assay for fatty acids* has an acid value (see *Fats* ⟨401⟩) between 260 and 280, about 1 g of the residue, accurately weighed, being used, and an iodine value (see *Fats and Fixed Oils* ⟨401⟩) of not more than 5.

B: It responds to *Identification* test *B* under *Sorbitan Monooleate*.

Acid value ⟨401⟩: not more than 8.
Hydroxyl value ⟨401⟩: between 330 and 358.
Saponification value ⟨401⟩: between 158 and 170.
Water, *Method I* ⟨921⟩: not more than 1.5%.
Residue on ignition ⟨281⟩: not more than 0.5%.
Heavy metals, *Method II* ⟨231⟩: not more than 0.001%.

Assay for fatty acids—Transfer about 10 g of Sorbitan Monolaurate, accurately weighed, to a 500-mL conical flask, cautiously add 100 mL of alcohol and 3.0 g of potassium hydroxide, and mix. Proceed as directed in the *Assay for fatty acids* under *Sorbitan Monooleate*, beginning with "Connect a suitable condenser."

Assay for polyols—Proceed with Sorbitan Monolaurate as directed for *Assay for polyols* under *Sorbitan Monooleate*.

Sorbitan Monooleate

[Graphic formula same as for Sorbitan Monolaurate, except that *R* is $(C_{17}H_{33})COO$.]
Sorbitan, esters, mono(*Z*)-9-octadecenoate.
Sorbitan monooleate [1338-43-8].

» Sorbitan Monooleate is a partial oleate ester of Sorbitol and its mono- and dianhydrides. It yields, upon saponification, not less than 72.0 percent and not more than 78.0 percent of fatty acids, and not less than 25.0 percent and not more than 31.0 percent of polyols (w/w).

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩**—**
USP Isosorbide RS
USP 1,4-Sorbitan RS
$C_6H_{12}O_5$ 164.16

Identification—
A: The residue of oleic acid obtained in the *Assay for fatty acids* has an acid value (see *Fats and Fixed Oils* ⟨401⟩) between 192 and 204, about 1 g of the residue, accurately weighed, being used, and an iodine value (see *Fats and Fixed Oils* ⟨401⟩) between 75 and 95.

B: *Standard preparation*—Transfer 33 mg of USP Isosorbide RS, 25 mg of USP 1,4-Sorbitan RS, and 25 mg of sorbitol to a 1-mL volumetric flask, dilute with water to volume, and mix to dissolve.

Test preparation—Transfer 500 mg of the polyols obtained in the *Assay for polyols* to a 2-mL volumetric flask, dilute with water to volume, and mix to dissolve.

Procedure—Apply 2 µL each of the *Standard preparation* and of the *Test preparation* to a suitable thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel. Allow the spots to dry, and develop the chromatogram in a solvent system consisting of a mixture of acetone and glacial acetic acid (50:1) until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and allow the solvent to evaporate. Spray evenly with a mixture of equal volumes of sulfuric acid and water until the surface is uniformly wet (do not overspray), and immediately place the sprayed plate on a 200° hot plate *in a*

well-ventilated hood. Char until white fumes of sulfur trioxide cease, and cool: the R_F values of the spots obtained from the *Test preparation* correspond to those of the spots obtained from the *Standard preparation*.

Acid value ⟨401⟩: not more than 8.

Hydroxyl value ⟨401⟩: between 190 and 215.

Iodine value ⟨401⟩: between 62 and 76.

Saponification value ⟨401⟩: between 145 and 160.

Water, *Method I* ⟨921⟩: not more than 1.0%.

Residue on ignition ⟨281⟩: not more than 0.5%.

Heavy metals, *Method II* ⟨231⟩: 0.001%.

Assay for fatty acids—Transfer about 10 g of Sorbitan Monooleate, accurately weighed, to a 500-mL conical flask, cautiously add 100 mL of alcohol and 3.5 g of potassium hydroxide, then add a few glass beads, and mix. Connect a suitable condenser to the flask, reflux the mixture on a hot plate for 2 hours, add about 100 mL of water, and heat on a steam bath to evaporate the alcohol, adding water occasionally to replace the alcohol. Continue the evaporation until the odor of alcohol can no longer be detected, and transfer the saponification mixture, with the aid of about 100 mL of hot water, to a 500-mL separator. Using extreme caution, neutralize to litmus with a mixture of equal volumes of sulfuric acid and water, noting the volume used, and add a 10% excess of the dilute acid. Allow the solution to cool. If salts appear, add sufficient water to produce a clear solution. Cautiously add 100 mL of solvent hexane, shake thoroughly, and withdraw the lower layer into a second 500-mL separator. Similarly extract with 2 more 100-mL portions of solvent hexane. Extract the combined hexane layers with 50-mL portions of water until neutral to litmus paper, and combine the extracts with the original aqueous phase, for the *Assay for polyols*. Evaporate the solvent hexane in a tared beaker on a steam bath nearly to dryness, dry in vacuum at 60° for 1 hour, cool in a desiccator, and weigh the fatty acids.

Assay for polyols—Neutralize the aqueous solution of polyols retained from the *Assay for fatty acids* with potassium hydroxide solution (1 in 10) to a pH of 7, using a suitable pH meter. Evaporate on a steam bath to a moist residue, extract the polyols from the salts with three 150-mL portions of dehydrated alcohol, boiling the salt residue for 3 minutes, and crushing it, as necessary, with the flattened end of a stirring rod, during each extraction, filtering each extract, while hot, through a medium-porosity, sintered-glass funnel, provided with a sheet of retentive filter paper on which a layer of purified siliceous earth has been superimposed, and receiving the filtrates in a 1-liter suction flask. Transfer the clear alcoholic polyols solution to a tared beaker, evaporate the alcohol on a steam bath, dry in vacuum at 60° for 1 hour, cool in a desiccator, and weigh the polyols.

Sorbitan Monopalmitate

[Graphic formula same as for Sorbitan Monolaurate, except that *R* is $(C_{15}H_{31})COO$.]
Sorbitan, esters, monohexadecanoate.
Sorbitan monopalmitate [26266-57-9].

» Sorbitan Monopalmitate is a partial ester of palmitic acid with Sorbitol and its mono- and dianhydrides. It yields, upon saponification, not less than 63.0 percent and not more than 71.0 percent of fatty acids, and not less than 32.0 percent and not more than 38.0 percent of polyols (w/w).

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩**—**
USP Isosorbide RS
USP 1,4-Sorbitan RS
$C_6H_{12}O_5$ 164.16

Identification—

A: The residue of palmitic acid obtained in the *Assay for fatty acids* has an acid value (see *Fats and Fixed Oils* ⟨401⟩) between 210 and 225, about 1 g of the residue, accurately weighed, being used, and an iodine value (see *Fats and Fixed Oils* ⟨401⟩) of not more than 4.

B: It responds to *Identification* test *B* under *Sorbitan Monooleate*.

Acid value ⟨401⟩: not more than 8.

Hydroxyl value ⟨401⟩: between 275 and 305.

Saponification value ⟨401⟩: between 140 and 150.

Water, *Method I* ⟨921⟩: not more than 1.5%.

Residue on ignition ⟨281⟩: not more than 0.5%.

Heavy metals, *Method II* ⟨231⟩: not more than 0.001%.

Assay for fatty acids—Transfer about 10 g of Sorbitan Monopalmitate, accurately weighed, to a 500-mL conical flask, cautiously add 100 mL of alcohol and 3.0 g of potassium hydroxide, and mix. Proceed as directed in the *Assay for fatty acids* under *Sorbitan Monooleate*, beginning with "Connect a suitable condenser."

Assay for polyols—Proceed with Sorbitan Monopalmitate as directed for *Assay for polyols* under *Sorbitan Monooleate*.

Sorbitan Monostearate

[Graphic formula same as for Sorbitan Monolaurate, except that *R* is $(C_{17}H_{35})COO$.]
Sorbitan, esters, monooctadecanoate.
Sorbitan monostearate [1338-41-6].

» Sorbitan Monostearate is a partial ester of Stearic Acid with Sorbitol and its mono- and dianhydrides. It yields, upon saponification, not less than 68.0 percent and not more than 76.0 percent of fatty acids, and not less than 27.0 percent and not more than 34.0 percent of polyols (w/w).

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩**—**
USP Isosorbide RS
USP 1,4-Sorbitan RS
$C_6H_{12}O_5$ 164.16

Identification—

A: The residue of stearic acid obtained in the *Assay for fatty acids* has an acid value (see *Fats* ⟨401⟩) between 200 and 215, about 1 g of the residue, accurately weighed, being used, and an iodine value (see *Fats and Fixed Oils* ⟨401⟩) of not more than 4.

B: It responds to *Identification* test *B* under *Sorbitan Monooleate*.

Acid value ⟨401⟩: not more than 10.

Hydroxyl value ⟨401⟩: between 235 and 260.

Saponification value ⟨401⟩: between 147 and 157.

Water, *Method I* ⟨921⟩: not more than 1.5%.

Residue on ignition ⟨281⟩: not more than 0.5%.

Heavy metals, *Method II* ⟨231⟩: not more than 0.001%.

Assay for fatty acids—Transfer about 10 g of Sorbitan Monostearate, accurately weighed, to a 500-mL conical flask, cautiously add 100 mL of alcohol and 3.0 g of potassium hydroxide, and mix. Proceed as directed in the *Assay for fatty acids* under *Sorbitan Monooleate*, beginning with "Connect a suitable condenser."

Assay for polyols—Proceed with Sorbitan Monostearate as directed for *Assay for polyols* under *Sorbitan Monooleate*.