

Polyethylene Oxide

» Polyethylene Oxide is a nonionic homopolymer of ethylene oxide, represented by the formula:



in which n represents the average number of oxyethylene groups. It is a white to off-white powder obtainable in several grades, varying in viscosity profile in an aqueous isopropyl alcohol solution. It may contain a suitable antioxidant.

Packaging and storage—Preserve in tight, light-resistant containers. No storage requirements specified.

Labeling—The labeling indicates its viscosity profile in aqueous isopropyl alcohol solution. Label it to indicate the name and quantity of any added antioxidant.

USP Reference standards <11>—*USP Polyethylene Oxide RS*.

Identification—

A: *Infrared Absorption* <197K>—

Test specimen: previously dried in vacuum at room temperature to constant weight.

B: The aqueous isopropyl alcohol solution viscosity, determined using a suitable viscometer with appropriate validation at 25° and in a concentration as directed in the labeling, falls within the viscosity range indicated by the labeling.

Loss on drying <731>—Dry about 4 g of it at 105° for 45 minutes: it loses not more than 1.0% of its weight.

Silicon dioxide and nonsilicon dioxide residue on ignition—

Weigh accurately about 1 g into a previously ignited, tared 50-mL platinum crucible. Add 4 drops of sulfuric acid. Heat carefully on a hot plate until the specimen is thoroughly charred and fumes no longer are evolved. Ignite the crucible at $700 \pm 25^\circ$ (see *Residue on Ignition* <281>) to constant weight. Wet the residue carefully with 1 mL of water, and slowly add 20 drops of hydrofluoric acid. [*Caution—Hydrofluoric acid is an extremely hazardous chemical. When handling it wear a face shield, arm protection, and rubber gloves, and perform the operation in a hood.*] Evaporate slowly on a hot plate to dryness, then ignite at $700 \pm 25^\circ$ for 10 minutes, cool to room temperature in a desiccator, and weigh accurately. Repeat the addition of hydrofluoric acid, evaporation, and ignition, to constant weight. Calculate the percentage of silicon dioxide from the difference between the net weights before and after the hydrofluoric acid treatment: it is not more than 3.0%. Calculate the percentage of nonsilicon dioxide residue on ignition from the final net weight: it is not more than 2.0%.

Heavy metals, Method II <231>: 0.001%.

Limit of free ethylene oxide—

Standard stock solution—[*Caution—Ethylene oxide is toxic and flammable. Prepare solutions of it in a well-ventilated fume hood.*] Using the special handling described in the following, complete the preparation. Ethylene oxide is a gas at room temperature. It is usually stored in a lecture-type gas cylinder or small metal pressure bomb. Chill the cylinder in a refrigerator before use. Transfer about 5 mL of the liquid ethylene oxide to a cold, 10-mL serum vial. Seal the vial, and store in a refrigerator. Transfer about 40 g of acetone, accurately weighed, to a tared 50-mL serum vial that is capable of being tightly sealed with a polytef-lined septum and a metallic crimp cap. Seal the vial, and accurately weigh it. Using a gas-tight gas chromatographic syringe that has been chilled in a refrigerator,

transfer about 60 μL of the liquefied ethylene oxide to the same vial. Weigh the vial, and determine the amount added by weight difference. This *Standard stock solution* contains about 1 μg of ethylene oxide per μL . [NOTE—This solution may be kept for 1 week in the crimp-sealed serum vial, stored in a freezer.]

Standard preparations—To four separate, tared 50-mL serum vials that are capable of being tightly sealed with polytef-lined septa and metallic crimp caps, transfer 1.0 g of the Polyethylene Oxide under test. Seal the vials. To the separate vials, transfer 2.0, 4.0, 6.0, and 8.0 μL of the *Standard stock solution*, and mix. These vials contain about 2, 4, 6, and 8 μg of ethylene oxide, respectively, from the *Standard stock solution*. Heat the vials at 100° for 30 minutes, and cool to room temperature.

Test preparation—Transfer about 1 g of the Polyethylene Oxide under test, accurately weighed, to a tared 50-mL serum vial that is capable of being tightly sealed with a polytef-lined septum and a metallic crimp cap. Weigh the vial, and determine the amount of Polyethylene Oxide added by weight difference. Seal the vial, heat at 100° for 30 minutes, and cool to room temperature.

Chromatographic system (see *Chromatography* <621>)—The gas chromatograph is equipped with a flame-ionization detector, a 0.53-mm \times 10-m capillary column bonded with a 20- μm layer of phase G45, and a split injection system. The carrier gas is helium, flowing at the rate of about 15 mL per minute. The makeup gas is also helium, with a split flow rate of about 15 mL per minute. The injection port and detector temperatures are maintained at about 200° and 250°, respectively. The column temperature is maintained at about 70° for 5 minutes after injection, then programmed to increase at a rate of 10° per minute to about 200° and to maintain this temperature for 5 minutes. Inject 300 μL of the gaseous headspace from the vial of *Standard preparation* that contains about 6 μg of ethylene oxide from the *Standard stock solution* into the gas chromatograph, and record the peak responses as directed for *Procedure*: the relative standard deviation for replicate injections is not more than 5%. [NOTE—Multiple vials are prepared for replicate injections.]

Procedure—[NOTE—A headspace apparatus that automatically transfers the measured amount of gaseous headspace may be used to perform the injections.] Using a gas-tight syringe, separately inject equal volumes (about 300 μL) of the gaseous headspace of each of the *Standard preparations* and the *Test preparation* into the gas chromatograph, record the chromatograms, and measure the areas of the peak responses. Determine by a retention time comparison whether ethylene oxide is detected in the *Test preparation*. Plot the responses of the *Test preparation* and the *Standard preparations* versus the content, in μg , of ethylene oxide in each vial, as furnished by the *Standard stock solution*; draw the straight line best fitting the five points; and calculate the correlation coefficient for the line. [NOTE—The content of ethylene oxide, as furnished by the *Standard stock solution*, is 0 μg in the *Test preparation*.] A suitable system is one that yields a line having a correlation coefficient of not less than 0.99. Extrapolate the line until it intercepts the content axis on the negative side. From the intercept, determine the total amount, T_U , in μg , of ethylene oxide in the *Test preparation*. Calculate the percentage of ethylene oxide in the portion of Polyethylene Oxide taken by the formula:

$$100(T_U / W)$$

in which W is the weight, in μg , of Polyethylene Oxide taken to prepare the *Test preparation*: the limit is 0.001%.

Organic volatile impurities, Method I <467>: meets the requirements.

(Official until July 1, 2008)