

PROPYLENE OXIDE

PRINCIPLE

Residual propylene oxide in starches treated with propylene oxide is removed by extraction at room temperature with a mixture of 2-propanol and water. The propylene oxide in the extract is determined by gas chromatography.

SCOPE

The method is applicable to propylene oxide-treated starches.

SPECIAL APPARATUS

1. Gas Chromatograph: Equipped with flame ionization detector or equivalent
2. Mechanical Shaker: Burrell wrist-action
3. Column: Stainless steel, 8 ft., 1/8 in., packed with 60-80 mesh Chromosorb7 W, coated with 20% by weight of Carbowax 400

REAGENTS

1. 2-Propanol Solution: Nanograde or Pesticide Grade. Mix 500 mL 2-propanol with 100 mL purified water.
2. Propylene Oxide, 99% Minimum

INSTRUMENT PARAMETERS

1. Column Temperature Program: Isothermal, 60 °C for 4 mins., then raise to 125 °C and hold 5 mins.
2. Injection Port and Detector Temperatures: 230 °C

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3. Gas Flows: Helium, 30 mL/min. Hydrogen and air, adjust for maximum sensitivity according to manufacturer's recommendations.

PROCEDURE

Standardization: Weigh 1.000 g propylene oxide into a 100 mL volumetric flask containing 50 mL of the 2-propanol solution. Dilute to volume with the same solution and mix thoroughly. Pipet a 1 mL aliquot into a 100 mL volumetric flask and dilute to volume with the 2-propanol solution. Prepare a second successive (5 to 50 mL) dilution. This second dilution contains 10 µg propylene oxide per mL. Inject a 5 µL sample of the second dilution into the gas chromatograph for standardization. Prepare fresh daily both the stock and dilute standards.

Sample Analysis: Weigh accurately 4 g of starch into a 1 oz. screw-cap bottle and add 10 mL of the alcohol-water solution. Place on a mechanical shaker and shake for one hr. Remove from shaker, allow to stand until the supernatant is clear and inject a 5 µL sample into the gas chromatograph (Note 1).

CALCULATIONS (Note 2)

Determine areas under the signal peaks corresponding to propylene oxide in the diluted standard and sample extract.

$$\text{Propylene} = \frac{(\text{Sample Signal Area})(\text{Propylene Oxide, Standard, } \mu\text{g/mL})(10 \text{ mL})}{(\text{Standard Signal Area})(\text{Sample Wt., g})}$$

NOTES AND PRECAUTIONS

1. Sample extracts should be analyzed on the day of preparation.
2. Detection limit based on a 4 g sample is about 0.5 to 1.0 ppm.