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 or equivalent

3. Column: or equivalent

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

Run analysis per manufacturers' instructions.

Column: Supel - Q PLOT, Fused silica capillary column (30m x 0.53mm id) p/n 2-5462

Oven temperature: 120°C; temp program at 4°C / min to 135°C; then Temp program at 20°C/min to 225°C; hold for 2 min.

PROPYLENE OXIDE — continued

Propylene oxide typically eludes at 5 min, depending on column flow rates.

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). Alternately, the sample may be centrifuged.

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.

METHOD HISTORY

Corn Starch, Propylene Oxide (C-50), Date of Acceptance 7-22-1977, Revised 12-08-2006.