

PROPYLENE OXIDE

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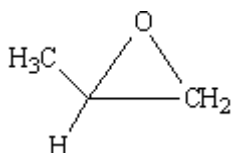
DEFINITION

Chemical names Propylene oxide, methyl oxirane, 1,2-epoxypropane

C.A.S. number 75-56-9

Chemical formula C_3H_6O

Structural formula



Formula weight 58.08

Assay Not less than 99%

DESCRIPTION A colourless liquid with a sweetish odour

FUNCTIONAL USES Antimicrobial preservative

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) 1 g of the sample is soluble in 1.7 ml of water. Miscible in ethanol and in ether

Infrared absorption Infrared spectrum is recommended

PURITY

Water (Vol. 4) Not more than 0.1% (Karl Fischer Method)

Distillation range 32 - 37° (at 760 mm Hg)
See description under TESTS

Insoluble matter (Vol. 4) Substantially free from suspended matter

Non-volatile residue (Vol. 4) Not more than 0.1%

Total acidity Not more than 0.05% (as acetic acid)
See description under TESTS

Total chlorides Not more than 0.1%
See description under TESTS

Aldehydes

Passes test
See description under TESTS

TESTS

PURITY TESTS

Distillation range

Measure a 100-ml sample in a 100-ml graduated cylinder at 10 to 15° and transfer directly to the distillation flask. Connect the flask to the condenser. Place the graduated cylinder used to measure the sample, without rinsing or drying, at the outlet of the condenser tube in such a position that the condenser tube extends into it for at least 2.5 cm but not below the 100-ml mark. Immerse the receiving cylinder up to the 100- ml mark in a transparent bath maintained between 10 and 15°. Cover the top of the graduated cylinder with a piece of blotting paper cut so as to fit tightly over the end of the condenser tube. Apply heat slowly with a micro-Bunsen burner and regulate (a good needle valve is best) so that the first drop of condensate falls from the condenser in not less than 5 or more than 10 min. Adjust the receiving cylinder so that the end of the condenser tube touches its side. Regulate the heat so that the distillation proceeds at a uniform rate of 4 to 5 ml per min. Maintain this rate and continue the distillation to dryness. Record the temperature when the first drop of distillation falls from the end of the condenser. Take readings when the level reaches 5 ml, 10 ml, and each additional 10 ml, including 90 ml, and at 95 ml. Take the final reading when the liquid disappears from the bottom of the flask. The boiling points observed may be corrected for barometric pressure by adding 0.044° to the observed boiling point for each mm Hg pressure under 760 mm or by subtracting an equal amount for each mm Hg pressure over 760 mm. The range of difference between the initial boiling point and the dry point should be 32-37°.

Total acidity

Measure 50 ml of the sample in a chilled 100-ml graduate cylinder and transfer to a cooled 250-ml conical flask. Add 1 ml of phenolphthalein TS and titrate with 0.01 N sodium hydroxide to a faint pink colour which persists for at least 15 sec. The proportion, expressed as acetic acid, should not exceed 0.05%.

$$\% \text{ acetic acid} = \frac{\text{ml of } 0.01 \text{ N NaOH} \times 0.060}{\text{ml of sample} \times \text{specific gravity}} \times 100$$

Total chlorides

Method A:

Connect a weighed stainless steel sample cylinder (30-ml capacity) to the sample container and flush the cylinder for a few sec with liquid ethylene oxide. Close the valve on the cylinder and allow it to fill to between 22 and 25 g of the sample. Weight the cylinder and contents to the nearest 0.1 g. Connect the cylinder to the burner assembly as shown in Figure 1. Pour 50±0.5 ml of freshly prepared 0.02 N sodium arsenite into the gas absorber. Connect the absorber to the air condenser. Connect the vacuum line to the gas absorber and apply a light vacuum to the system. Light the pilot burner and burn the sample in the following manner: allow a small flow of oxygen to the ring around the burner (Figure 1, valve A). Allow a small flow of hydrogen to the burner (Figure 1, valve B) and light it with a flint lighter.

Adjust the flame to a height of about 2.5 cm. Regulate the oxygen flow to the burner tip so as to obtain a flame cone about 1.3 cm high (Figure 1, valve C). Set the chimney in place secure the clamps. Place a beaker of hot water (50°) around the cylinder (replenish with warm water during the run) Open the Hoke valve very slowly and allow the sample to raise the flame height to about 6.5-7.5 cm. Adjust the oxygen flow to the burner tip to obtain a cone in the flame about 1.3 cm high.

Readjust the vacuum as necessary to ensure a slight vacuum in the system at all times. Allow the sample to burn until the flame gives evidence that the sample is almost burned out (20 to 25 min) then adjust the oxygen flow to the burner to keep the flame cone at about 1.3 cm. Adjust the Hoke valve on the sample cylinder during the run so that when the run is finished the valve is completely open. Shut off the oxygen flow to the tip of the burner when the sample is burner out, then shut off the hydrogen flow. Allow several min for the system to be purged with the oxygen flow from the ring around the burner. Then release the vacuum and shut of the oxygen flow to the ring. Allow the condenser and glass to cool to room temperature and then rinse with a minimum amount of water into the gas scrubber. Transfer the contents of the gas-washing bottle to a 250-ml beaker and chill to about 5° in a refrigerator or in crushed ice. Insert a magnetic agitator and the freshly washed silver chloride electrodes into the beaker. Acidify the solution with nitric acid using methyl red TS as indicator. Titrate the solution potentiometrically with 0.02 N silver nitrate solution. Prepare a blank solution of the reagents and titrate. The proportion of total chlorides should not exceed 0.1%.

$$\% \text{ total chlorides} = \frac{(V_1 - V_2) \times N \times 0.0355}{\text{weight of sample}} \times 100$$

where

V_1 = ml of silver nitrate required by the sample

V_2 = ml of silver nitrate required by the blank

N = normality of the silver nitrate

Method B (Alternative method):

Cool a 50-ml graduated cylinder by immersing in ice water or by placing in a refrigerator at 0° for about 15 min. Pour 50 ml of sodium ethoxide TS and 70 ml of methyl alcohol into a 250-ml conical flask and place in ice water or in refrigerator at 0° for 30 min. Pour as rapidly as possible 30 ml of the sample into the cooled cylinder and transfer the contents to the flask containing the cooled sodium ethoxide. Attach a water-cooled reflux condenser and gently boil the mixture for 1 h. Cool and transfer the mixture to a 600-ml beaker rinsing the flask with 100 ml of water. Make the liquid just acid with 1.5 N nitric acid (usually about 130 ml) using litmus paper as indicator and add 10 ml more. Keep the solution cool (below 30°) and stir constantly during the neutralization. Titrate the chloride present electrometrically using 0.025 N silver nitrate. Prepare a blank in the same way but omitting the sample.

$$\% \text{ total chlorine} = \frac{(V - V_b) \times N \times 35.5}{10V_s \times 0.89}$$

where

V = volume of silver nitrate used in titrating sample

V_b = volume of silver nitrate used in titrating blank
 N = normality of silver nitrate
 V_s = volume of sample.

Aldehydes

Measure 20 ml of the sample into a glass-stoppered conical flask containing 5 ml of chilled sodium bisulfite TS and 25 ml of water. Place the flask and its contents in a refrigerator for exactly 15 min. Prepare a reagent blank along with each set of samples and treat it in the same manner as the samples. Titrate the excess bisulfite solution in the blank and sample flasks with 0.1 N iodine solution until persistence of the yellow brown colour due to the liberated iodine. The proportion, expressed as propionic aldehyde, should not exceed 0.1%. Calculate the ion % propionic aldehyde from:

$$\frac{(V_b - V_s) \times N \times 0.029}{(\text{ml of sample}) \times \text{specific gravity}} \times 100$$

where

V_s = ml of iodine solution required by the sample

V_b = ml of iodine solution required by the blank

N = normality of iodine solution.

METHOD OF ASSAY

Introduce 0.8000-1.3000 g of propylene oxide into a tared thin-walled glass bulb (15 mm in diameter with a neck 30 mm long) which is packed in dry ice. Seal the bulb with the flame from an oxygen-gas torch, remove the bulb from the dry ice, wash with acetone, dry in a 60° oven, cool and weigh. From a pipette introduce 25 ml of pyridinium chloride-chloroform TS into a pressure bottle. Insert the bulb into the pressure bottle, seal the latter, and place it in an ice bath for 30 min. Remove the bottle from the ice bath, put it into a canvas bag and shake to break the bulb. After the bulb is broken, allow the pressure bottle to warm to room temperature and then put it into a steam bath for 20 min. Remove the bottle from the steam bath and cool. After the bottle is cool remove the canvas bag, wash the stopper and neck of the bottle with methanol, add 10 drops of phenolphthalein TS and titrate with N sodium hydroxide in methanol (which should be freshly standardized before using). Two blanks should be run with the sample (A N aqueous solution of sodium hydroxide may be used if methanol is added to the chloroform solution in the pressure bottle.) Calculate the % propylene oxide from:

$$\frac{\text{ml of } N \text{ NaOH} \times 0.0581}{(\text{weight of sample})} \times 100$$