

Polyisobutylene

Butyl Rubber

Homopolymer of 2-methyl-1-propene

[9003-27-4]

Definition Polyisobutylene is a polymer of isobutylene. It may contain not more than 2% of total polymer unit derived from isoprene.

Description Polyisobutylene occurs as a colorless to light yellow elastic rubbery semi-solid or viscous substance. It is odorless or has a slight, characteristic odor, and is tasteless.

Identification Dissolve about 1 g of Polyisobutylene in 5 ml of *n*-hexane, and proceed as directed under the Thin Film Method in Infrared Spectrophotometry. Absorptions are observed at about 1,393 cm^{-1} , 1,370 cm^{-1} , 1,230 cm^{-1} , 950 cm^{-1} , and 920 cm^{-1} .

Purity (1) Clarity of solution Slightly turbid.

Weigh 0.50 g of Polyisobutylene, add 50 ml of *n*-hexane, and dissolve while heating in a water bath at 80 °C.

(2) Heavy metals Not more than 10 $\mu\text{g/g}$ as Pb (2.0 g, Method 2, Control solution Lead Standard Solution 2.0 ml).

(3) Arsenic Not more than 4.0 $\mu\text{g/g}$ as As_2O_3 (0.50 g, Method 3, Apparatus B).

(4) Chlorinated compounds Not more than 0.028% as Cl.

Test Solution Weigh 0.5 g of Polyisobutylene and 0.7 g of calcium carbonate, transfer into a porcelain crucible, mix with a small amount of water, dry at 100 °C, and heat at about 600 °C for 10 minutes. After cooling, dissolve the residue with 20 ml of diluted nitric acid (1 : 10), filter, wash the insoluble residue with about 15 ml of water, combine the filtrate and the washings, and add water to make 50 ml.

Control Solution Dissolve 0.7 g of calcium carbonate in 20 ml of diluted nitric acid (1 : 10), filter if necessary, and add 0.40 ml of 0.01 mol/l hydrochloric acid and water to make 50 ml.

Add 0.5 ml of silver nitrate solution (1 : 50) to the test solution and the control solution, shake well, and allow to stand for 5 minutes. The test solution is not more turbid than the control solution.

(5) Total unsaturated substances Not more than 2.0%.

Weigh accurately 0.5 g of Polyisobutylene, transfer into a 500-ml flask containing 100 ml of carbon tetrachloride, stopper tightly the flask, and allow to stand overnight

to dissolve completely. Add 5 ml of a trichloroacetic acid - carbon tetrachloride solution (1 : 5), and add 20 ml of iodine - carbon tetrachloride TS and 20 ml of a solution of mercuric acetate in diluted acetic acid (3 : 100). Stopper tightly the flask and mix thoroughly by shaking vigorously. Allow to stand in a dark place for exactly 30 minutes, add 75 ml of potassium iodide solution (3 : 40), and shake vigorously for 2 minutes. Remove the stopper, wash the liquid on the wall into the flask with distilled water, and immediately titrate with 0.1 mol/l sodium thiosulfate (indicator: starch TS). Separately,

perform a blank test in the same manner, and make any necessary correction.

Calculate the total amount of unsaturated substances by the formula

$$\text{Total amount of unsaturated substances} = \frac{1.87 \times (a - b) \times 0.1}{\text{Weight (g) of the sample}} (\%),$$

where a = volume (ml) of 0.1 mol/l sodium thiosulfate consumed in the blank test,

b = volume (ml) of 0.1 mol/l sodium thiosulfate consumed in the test.

(6) Low molecular weight polymer Not more than 1.2%.

Weigh accurately about 10 g of Polyisobutylene, add 40 ml of benzene, and dissolve while heating with a reflux condenser on a water bath and shaking occasionally. After cooling, add 40 ml of methanol, shake well, and allow to stand in a cold place for 1 hour. Transfer the supernatant to a flask, evaporate under reduced pressure at about 50 °C, and dry in a vacuum desiccator for 20 hours. Weigh the residue.

Residue on Ignition Not more than 0.20%