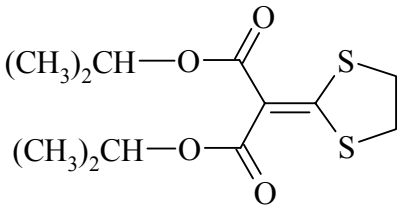
ISOPROTHIOLANE
456



ISO common name	Isoprothiolane
Chemical name	Di-isopropyl-1,3-dithiolane-2-ylidenemalonate (IUPAC,CA; *50512-35-1*)
Empirical Formula	$C_{12}H_{18}O_4S_2$
RMM	290.39
m.p.	54.0 - 54.5 °C
v.p.	8.8 mPa at 25 °C
Solubility	In water: 0.054 g/l; chloroform: 4130 g/l; acetone: 4060 g/l; benzene: 2770 g/l; xylene: 2260 g/l; methanol: 1510 g/l; ethanol: 760 g/l all at 25 °C
Description	White crystalline solid (technical: yellow solid)
Formulations	Wettable powders, emulsifiable concentrates and granules

ISOPROTHIOLANE TECHNICAL
*456/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests.

2.1 Infrared. Prepare 13 mm diameter KBr discs from the sample and from a standard isoprothiolane using 2 mg of material and about 200 mg of KBr. Scan the discs from 4600 to 400 cm^{-1}. The spectrum produced from the sample disc should not differ significantly from that of the standard.

2.2 GLC. Use the GLC method below. The relative retention time of isoprothiolane with respect to the internal standard for the sample solution should not deviate by more than 2% from that for the calibration solution.

3 Isoprothiolane

OUTLINE OF METHOD. The sample is dissolved in acetone with bis(2-ethylhexyl)adipate added as internal standard. The isoprothiolane content is determined by gas-chromatography with a flame-ionization detector.

* CIPAC method 1991. Prepared by the Japanese Committee (JAPAC). Chairman: T Suzuki. Based on a method supplied by Nihon Nohyaku Co., Ltd. Japan.

REAGENTS
Acetone
Isoprothiolane, pure
Bis(2-ethylhexyl)adipate, pure
Nitrogen, pure (carrier gas)
Hydrogen, pure
Air
Isoprothiolane calibration solution. Weigh (to the nearest 0.1 mg) about 500 mg of standard isoprothiolane into a 25 ml volumetric flask, dissolve in acetone and dilute to volume. Mix thoroughly.
Internal standard solution. Weigh (to the nearest 0.1 mg) about 1200 mg of bis(2-ethylhexyl)adipate into a 100 ml volumetric flask, dissolve in acetone and dilute to volume. Mix thoroughly. 10 ml of this solution contains (*A*) mg of bis(2-ethylhexyl)adipate.

APPARATUS
Gas chromatograph, suitable for on-column injection, equipped with a flame-ionization detector and injection port heating

Column, glass. 1.0 m × 3 mm (i.d.) packed with 5% Silicone SE-30 on Chromosorb W (AW-DMCS) 60-80 mesh
Automatic digital integrator
Microsyringe, 10 µl

PROCEDURE
(a) Operating conditions (typical):

Temperatures	
Column oven	210 °C
Injection port	230 °C
Detector	230 °C
Carrier gas flow rate	50 ml/min(N2)
Hydrogen flow rate	55 ml/min
Air flow rate	550 ml/min
Detector sensitivity	102 MΩ, 64 mV
Chart speed	5 mm/min
Retention times	isoprothiolane: about 2.5 min bis(2-ethylhexyl)adipate: about 5.9 min

(b) Preparation of calibration curve. Pipette 2, 4, 6, 8 ml of isoprothiolane calibration solution into separate 50 ml Erlenmeyer flasks (with stoppers), add 10 ml of the internal standard solution and dilute to 30 ml with acetone. Mix

thoroughly. Inject in duplicate 2 µl portions of the solution into the gas-chromatograph and record the chromatogram. Measure the peak area of the isoprothiolane and bis(2-ethylhexyl)adipate with an automatic digital integrator. Plot the average of the isoprothiolane/bis(2-ethylhexyl)adipate peak area ratio against the isoprothiolane/bis(2-ethylhexyl)adipate mass ratio and construct the calibration curve using the method of least squares.

(c) Preparation of sample solution. Grind about 10 g of the technical material with a mortar and pestle. Weigh (to the nearest 0.1 mg) into 50 ml Erlenmeyer flasks (with stoppers) enough sample to contain 100 mg of the iosprothiolane (*w* mg). Add 10 ml of internal standard solution with a pipette, dissolve and dilute to 30 ml with acetone. Mix thoroughly.

(d) Determination. Inject in duplicate 2 µl portions of the solution into the gas-chromatograph, and record the chromatogram. Measure the peak area of the iosprothiolane and bis(2-ethylhexyl)adipate with an automatic digital integrator and calculate isoprothiolane/bis(2-ethylhexyl)adipate peak area ratio. Convert the average of the peak area ratio to the isoprothiolane/bis(2-ethylhexyl)adipate mass ratio (*B*) using the calibration curve.

(e) Calculation. Calculate the isoprothiolane content using the following formula and report the values in g/kg to one decimal place.

$$\text{Isoprothiolane content} = \frac{A \times B \times P}{w} \quad \text{g/kg}$$

where:

A = mass of the internal standard added to the sample solution (mg)
B = isoprothiolane/bis(2-ethylhexyl)adipate mass ratio obtained from the calibration curve
P = purity of isoprothiolane standard (g/kg)
w = mass of sample taken (mg)

Repeatability r = 3.1 g/kg at 976 g/kg active ingredient content

Reproducibility R = 31.3 g/kg at 976 g/kg active ingredient content

ISOPROTHIOLANE WETTABLE POWDERS
*456/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests. As for **456**/TC/M/2.

3 Isoprothiolane. As for **456**/TC/M/3 except:
(c) Preparation of sample solution. Weigh (to the nearest 0.1 mg) into 50 ml Erlenmeyer flasks (with stoppers) enough sample to contain 100 mg of isoprothiolane (*w* mg). Add 10 ml of internal standard solution with a pipette and dilute to 30 ml with acetone. Dissolve ultrasonically and mix thoroughly.

Repeatability r = 1.8 g/kg at 411 g/kg active ingredient content

Reproducibility R = 14.6 g/kg at 411 g/kg active ingredient content

4 Suspensibility

APPARATUS AND REAGENTS As for **456**/TC/M/3 together with:
Separating funnel 250 ml
Round-bottomed flask 250 ml
Chloroform

PROCEDURE
(a) Preparation of suspension MT 15.1(*i*).
(b) Determination of sedimentation MT 15.1(*ii*).
(c) Determination of isoprothiolane in the bottom 25 ml of suspension. After removal of the top 225 ml suspension, wash the bottom 25.0 ml of suspension from the suspensibility cylinder into a 250 ml separating funnel using the minimum amount of water. Add 25 ml of chloroform and shake for 1 min. Allow to stand so that the layers separate. (If an emulsion is formed, add a small quantity of potassium chloride to the flask and shake again.). Run off the organic layer into a 250 ml round-bottomed flask. Repeat the extraction a further three times, using 25 ml of chloroform and collecting the chloroform layers together in the round-bottomed flask. Evaporate the chloroform using a rotary evaporator. Add 10 ml of internal standard solution with a pipette, dissolve and dilute to 30 ml with acetone. Determine the isoprothiolane content as in **456**/TC/M/3.

* CIPAC method 1991. Prepared by the Japanese Committee (JAPAC). Chairman: T Suzuki. Based on a method supplied by Nihon Nohyaku Co., Ltd. Japan.

(d) Calculation

$$\text{Suspensibility} = \frac{111(c - Q)}{c}\%$$

where:

c = mass of isoprothiolane in the sample taken for the preparation of the suspension (g)

Q = mass of isoprothiolane in the 25 ml remaining in the suspensibility cylinder (g)

ISOPROTHIOLANE EMULIFIABLE CONCENTRATES
*456/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests. As for **456**/TC/M/2.

3 Isoprothiolane. As for **456**/TC/M/3 except:
(c) Preparation of sample solution. Weigh (to the nearest 0.1 mg) into 50 ml Erlenmeyer flasks (with stoppers) enough sample to contain 100 mg of isoprothiolane (*w* mg). Add 10 ml of internal standard solution with a pipette, dissolve and dilute to 30 ml with acetone. Mix thoroughly.

Repeatability r = 3.1 g/kg at 412 g/kg active ingredient content

Reproducibility R = 13.7 g/kg at 412 g/kg active ingredient content

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