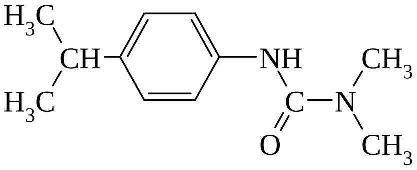
ISOPROTURON
336



ISO common name	Isoproturon
Chemical name	3-(4-Isopropylphenyl)-1,1-dimethylurea (IUPAC); *N*,*N*-dimethyl-*N'*-[4-(1-methyl-ethyl)phenyl]urea (CA; *34123-59-6*)
Empirical formula	$C_{12}H_{18}N_2O$
RMM	206.3
m.p.	158 °C
v.p.	3.3×10^{-6} Pa at 20 °C
d^{20}	1.16
Solubility	In water: 55 mg/l; benzene: 5 g/l; dichloromethane: 63 g/l; hexane: 0.1 g/l; methanol: 56 g/l, all at 20 °C.
Description	Creamy-white powder
Stability	Stable to light and acids hydrolysed by strong alkalis on heating. A slow exothermic decomposition occurs at temperatures above 230 °C.
Formulations	Wettable powders and suspension concentrates

ISOPROTURON TECHNICAL

[*]**336**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of isoproturon with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

2.2 Infrared. Prepare 13 mm diameter KBr discs using 1 mg of sample and about 250 mg of KBr and also using standard isoproturon. Scan the discs from 4000-700 cm^{-1}. The spectrum produced from the sample disc should not differ significantly from that from the standard.

2.3 Melting point

REAGENTS

Isoproturon pure

APPARATUS As for MT 2, CIPAC F, *p 5*

PROCEDURE Determine the melting point of the sample and an intimate mixture of equal parts of the sample and isoproturon by MT 2. The melting point of the sample should not be depressed by the admixture.

3 Isoproturon

OUTLINE OF METHOD Isoproturon is determined by normal phase high performance liquid chromatography, using acetanilide as internal standard.

REAGENTS

Chloroform HPLC grade, stabilized with 1% ethanol. If the available chloroform contains less than 1% ethanol, anhydrous ethanol must be added accordingly. The resulting ethanol content of the mobile phase is critical.
Dichloromethane
Ethanol anhydrous HPLC grade
n-Heptane HPLC grade

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[*] CIPAC method 1988. Prepared by the French committee (CFAPP).
Chairman: B Declercq. Based on a method supplied by Ciba-Geigy.

Mobile phase *n*-heptane + chloroform (with 1% ethanol) + ethanol, 70 + 15 + 1 v/v. The ethanol should be measured accurately.
Isoproturon standard of known purity
Acetanilide internal standard
Calibration solution. Weigh (to the nearest 0.1 mg) into a weighing boat 0.24 to 0.26 g of pure isoproturon (*s* mg) and 0.12 to 0.13 g of acetanilide (*r* mg). Transfer the contents of the boat into a 250 ml volumetric flask with dichloromethane. Dissolve, make up to the mark with dichloromethane, and homogenize.

APPARATUS

Liquid chromatography equipped with a constant flow pump permitting solvent delivery at 3 ml/min at about 20 MPa, and a 10 µl injection loop.
Detector UV-spectrophotometer or a fixed-wavelength UV detector at 254 nm
Integrator
Chromatographic column stainless steel, 250 × 4 (i.d.) mm packed with LiChrosorb Si 100, 5 µm. If the recommended packing material is not available it may be substituted by non-spherical porous silicagel with an average particle size of 5 µm, a pore-diameter of 10 nm and a surface area of 400 m^2/g.
Filtration device e.g. a glass syringe fitted with a membrane filtration unit (Millex-SR, 0.5 µm from Millipore, Minisart SRP 15, 0.2 µm from Sartorius or equivalent) compatible with organic solvents
Ultrasonic bath or a reciprocating mechanical shaker

PROCEDURE

(a) *Operating conditions (typical):*

Flow rate	3.0 ml/min (resulting pressure 14 to 20 MPa)
Temperature of column	Room temperature (20-25 °C). Since the retention time can be influenced by changes in temperature, temperature fluctuations of more than 2 °C should be avoided during the complete sequence of analyses.
Injection volume	10 µl. The injected volume may be increased to 20 µl in order to get peaks with a suitable size, within the linear range of the detector.
Detector wavelength	254 nm
Detector sensitivity	Settings should be chosen such that peaks are within 60-90% of the recorder scale.

Integration	Electronic. If an electronic integrator is not available the peak areas may be measured by peak height × retention time (expressed in mm measured on the recorder chart). The chart speed and the recorder sensitivity should be adjusted so that the peak height and the "retention time" are not less than 100 mm.
Run time	20 min
Retention times	isoproturon: about 7.5 min acetanilide: about 12 min

(b) Sample preparation. Weigh (to the nearest 0.1 mg) into a weighing boat enough sample to contain 0.24 to 0.26 g of pure isoproturon (*w* mg) together with 0.12 to 0.13 g of acetanilide (*q* mg). Transfer the contents of the boat to a 250 ml volumetric flask with dichloromethane. Dissolve, make up to the mark with dichloromethane, and homogenize.

c) Determination. Inject 10 µl aliquots of the calibration solution until the response factor varies by less than 1% for successive injections. Then make duplicate injections (10 µl) of the sample solution, followed by another injection of the calibration solution. Measure the peak areas and calculate the response factors for the pair of calibration injections which bracket the sample injections.

Continue in this way for other sample and calibration solutions.

(d) Calculation

$$\text{Response factor } f = \frac{I_r \times s \times P}{H_s \times r}$$

where:

H_s = peak area of isoproturon in the calibration solution
I_r = peak area of the internal standard in the calibration solution
P = purity of the standard isoproturon (g/kg)
r = mass of the internal standard in the calibration solution (mg)
s = mass of isoproturon in the calibration solution (mg)

Average the response factors of the calibration injections preceding and following the two sample injections.

$$\text{Isoproturon content } = \frac{f \times H_w \times q}{I_q \times w} \text{ g/kg}$$

where:
H_w = peak area of isoproturon in the sample solution
I_q = peak area of the internal standard in the sample solution
q = mass of the internal standard in the sample solution (mg)
w = mass of isoproturon in the sample solution (mg)

Repeatability r = 12.2 g/kg at 980 g/kg active ingredient content

Reproducibility R = 14.4 g/kg at 980 g/kg active ingredient content

ISOPROTURON WETTABLE POWDERS

[*]**336**/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 HPLC As for **336**/TC/M/2.1.
2.2 Infrared. Extract the sample with dichloromethane. Filter and evaporate the solvent with a stream of clean, dry air. Continue according to **336**/TC/M/2.2

3 Isoproturon. As for **336**/TC/M/3 except:
(b) Sample preparation. Weigh (to the nearest 0.1 mg) into a weighing boat enough sample to contain 0.24 to 0.26 g (*w* mg) of pure isoproturon together with 0.12 to 0.13 g of acetanilide (*q* mg). Transfer the contents of the weighing boat into a volumetric flask (250 ml) with about 200 ml dichloromethane. Place the flask in an ultrasonic bath for 2 min or shake the flask in a mechanical shaker for 30 min. Make up to the mark with dichloromethane. Homogenize and allow to settle for at least 1 h. Withdraw about 5 ml of the supernatant and filter through the membrane filter unit.

Repeatability r = 6.5 g/kg at 500 g/kg active ingredient content

Reproducibility R = 12.4 g/kg at 500 g/kg active ingredient content

4 Suspensibility (Draft method)
(a) Preparation of suspension MT 15.1 (*i*)
(*b) Determination of sedimentation* MT 15.1 (*ii*)

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[*] CIPAC method 1988. Prepared by the French committee (CFAPP).
Chairman: B Declercq. Based on a method supplied by Ciba-Geigy.

(c) Determination of isoproturon in the bottom 25 ml of suspension. After removal of the top 225 of suspension wash the slurry remaining in the cylinder into a tared Petri dish or other suitable receptacle, and dry under an infrared lamp to constant weight, taking care that no sputtering and overheating occur. Brush the residue into an agate mortar, grind and mix thoroughly. Weigh a portion of the residue sufficient to contain not more than 0.3 g of pure isoproturon and determine the content of active ingredient by **336**/WP/M/3.

(d) Calculation of suspensibility

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

where:

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

Q = mass of isoproturon in the bottom 25 ml of suspension (g)