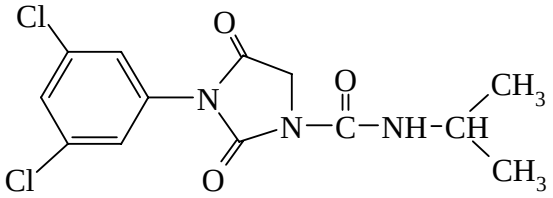
IPRODIONE
278



ISO common name	Iprodione
Chemical name	3-(3,5-Dichlorophenyl)-*N*-isopropyl-2,4-dioxo-imidazolidine-1-carboxamide (IUPAC); 3-(3,5-di-chloro-phenyl)-*N*-(1-methylethyl)-2,4-dioxo- 1-imidazolidine-carboxamide (CA; *36734-19-7*)
Empirical formula	$C_{13}H_{13}Cl_2N_3O_3$
RMM	330.2
m.p.	136 °C
v.p.	5×10^{-7} Pa at 25 °C
Solubility	In water: 13 mg/l; acetone, acetophenone and anisole: 300 g/l; dichloromethane, dimethyl-formamide and 1-methyl-2-pyrrolidone: 500 g/l; all at 20 °C
Description	White crystalline powder
Formulations	Wettable powders and suspension concentrates

IPRODIONE TECHNICAL
*__278__/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 iprodione 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 potassium bromide discs from the sample and from pure iprodione using 1.5 mg of material and 300 mg of potassium bromide. Scan the discs from 4000 to 450 cm^{-1}. The spectrum produced from the sample disc should not differ significantly from that from the standard.

3 Iprodione

OUTLINE OF METHOD Iprodione is determined by reversed phase high performance liquid chromatography (HPLC), using the internal standard method.

REAGENTS

Acetic acid HPLC grade
Acetonitrile HPLC grade
Methanol HPLC grade
Sodium acetate anhydrous
Water HPLC grade
Iprodione standard of known purity
Aqueous buffer solution, pH 4.5. Dissolve sodium acetate (about 3 g) in water (3 l). Using a pH meter, adjust to pH = 4.5 with acetic acid (about 2.7 ml).
Solvent mixture. Transfer, using a measuring cylinder, aqueous buffer, pH = 4.5 (400 ml) to a volumetric flask (1000 ml). Dilute to the mark with acetonitrile and mix well.

* CIPAC method 1993. Prepared by PAC-F. Chairman: B Declercq. Based on a method supplied by Rhône Poulenc Agro, France.

Mobile phase aqueous buffer (pH = 4.5)-methanol-acetonitrile, 450 + 330 + 220 (v/v)

Propiophenone internal standard

Internal standard solution. Weigh (to the nearest 0.1 mg) propiophenone (about 3 g) into a volumetric flask (250 ml). Dissolve in and dilute to the mark with acetonitrile.

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 0.09 g to 0.11 g (*s* g) of iprodione standard into Erlenmeyer flasks (125 ml). Add by pipette to each flask, internal standard solution (20.0 ml) and acetonitrile (about 30 ml). Dissolve by sonication for 5 minutes or by mechanical shaking for 30 minutes and allow to cool to room temperature. Transfer by pipette 10.0 ml of each solution into Erlenmeyer flasks (125 ml). Add to each flask solvent mixture (about 40 ml) and mix well (Solutions C_1 and C_2). Filter through a 0.5 µm filter before HPLC analysis.

APPARATUS

Liquid chromatograph equipped with a UV detector capable of measuring at 220 nm, a constant flow pump (at 1.5 ml/min), a loop injection valve (10 µl), a column heating compartment (at 40 °C) and an electronic integrator

Liquid chromatographic column stainless steel, 250 × 4.6 (i.d.) mm, packed with Nucleosil C18, 5 µm

Sample filtration device glass syringe fitted with a membrane filtration unit compatible with organic solvents, e.g. Millex-SR, 0.5 µm (Millipore ref. SLSRNO25NB) or equivalent

Ultrasonic bath or *mechanical shaker*

pH meter

PROCEDURE

(a) Operating conditions (typical):

Flow rate	1.5 ml/min
Temperature of column	40 °C
Injection volume	10 µl
Detector wavelength	220 nm

Retention times	propiophenone: about 6.5 min; iprodione: about 19 min
Chromatographic run duration	20 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain 0.09 to 0.11 g (*w* g) of iprodione into an Erlenmeyer flask (100 ml). Add by pipette internal standard solution (20 ml) and acetonitrile (about 30 ml). Dissolve by sonication for 5 min or by mechanical shaking for 30 min (Solution S) and allow to cool to room temperature. Transfer by pipette 10.0 ml of this solution into an Erlenmeyer flask (125 ml). Add solvent mixture (about 40 ml) and mix well. Filter through a 0.5 µm filter before HPLC analysis.

(c) Determination. Inject 10 µl of the calibration solutions C_1 and C_2 until the consecutive response ratios agree within 1%. Inject 10 µl of the calibration solutions and the sample solutions in the following sequence: C_1, S_1, S_1, C_2, S_2, S_2, C_1... Determine the peak areas.

(d) Calculation. Calculate the response factors (f_1, f_2) for the pair of calibration injections which bracket the sample injections and obtain the mean response factor *f*.

$$f = \frac{I_r \times s \times P}{H_s}$$

where:

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

Calculate the iprodione content for each sample injection.

$$\text{Content of iprodione} = \frac{f \times H_w}{I_q \times w} \text{ g/kg}$$

where:

f = mean response factor
H_w = area of the iprodione peak in the sample solution
I_q = area of the internal standard peak in the sample solution
w = mass of sample taken (g)

Take the mean of the four values as the iprodione content in the sample.

Repeatability r = 20 g/kg at 950 g/kg active ingredient content
Reproducibility R = 36 g/kg at 950 g/kg active ingredient content

IPRODIONE WETTABLE POWDERS

***278**/WP/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests

2.1. HPLC. As for iprodione technical **278**/TC/M/2.1.

2.2 Infrared. Extract the sample with dichloromethane, filter and evaporate the solvent. Proceed as for iprodione technical **278**/TC/M/2.2.

3 Iprodione. As for iprodione technical **278**/TC/M/3 except:

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into an Erlenmeyer flask (100 ml) enough sample to contain 0.09 to 0.11 g (*w* g) of iprodione. Add by pipette internal standard solution (10 ml) and acetonitrile (about 20 ml). Place the flask in an ultrasonic bath for 5 min or shake mechanically for 30 min and allow to cool to room temperature. Transfer by pipette 10.0 ml of this solution into an Erlenmeyer flask (125 ml). Add solvent mixture (about 40 ml) and mix well. Allow to settle insoluble material, withdraw a few ml of the supernatant solution and filter through a membrane filtration unit. Use the filtrate for the determination.

* CIPAC method 1993. Prepared by PAC-F. Chairman: B Declercq. Based on a method supplied by Rhône Poulenc Agro, France.

Repeatability r = 12.7 g/kg at 500 g/kg active ingredient content
Reproducibility R = 16.7 g/kg at 500 g/kg active ingredient content

4 Suspensibility (Draft method)
REAGENTS AND APPARATUS As for MT 15 and **278**/TC/M/3.

PROCEDURE

(a) Preparation of suspension. MT 15.1(*i*).

(b) Determination of sedimentation. MT 15.1(*ii)*.

(c) Determination of iprodione in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension, transfer the 25 ml remaining in the cylinder to a tared evaporating dish. Rinse the cylinder with water (3 × 10 ml) and add the rinsings to the dish. Evaporate to dryness at 60 °C. Dissolve the residue in acetonitrile and transfer quantitatively to an Erlenmeyer flask (100 ml). Determine the mass of iprodione (*Q*) according to **278**/WP/M/3 starting at: 'Add by pipette internal standard solution.....'

(d) Calculation

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

where:
c = mass of iprodione in the sample taken for the preparation of the suspension (g)
Q = mass of iprodione in the bottom 25 ml of suspension (g)

IPRODIONE SUSPENSION CONCENTRATES
*278/SC/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests
2.1. HPLC. As for iprodione technical **278**/TC/M/2.1.
2.2 Infrared. Extract the sample with dichloromethane, filter and evaporate the solvent. Proceed as for iprodione technical **278**/TC/M/2.2.

3 Iprodione As for iprodione wettable powders **278**/WP/M/3.

Repeatability r = 6.4 g/kg at 420 g/kg active ingredient content
Reproducibility R = 25.2 g/kg at 420 g/kg active ingredient content

4 Suspensibility (Draft method)
REAGENTS AND APPARATUS As for MT 15 and **278**/TC/M/3.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 161.
Proceed as for iprodione wettable powders **278**/WP/M/4*(c)*.

* CIPAC method 1993. Prepared by PAC-F. Chairman: B Declercq. Based on a method supplied by Rhône Poulenc Agro, France.