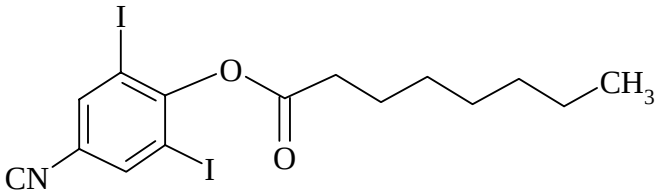
IOXYNIL OCTANOATE
86



ISO common name	Ioxynil octanoate
Chemical name	4-Cyano-2,6-di-iodophenyl octanoate (IUPAC and CA; *3861-47-0*)
Empirical formula	$C_{15}H_{17}I_2NO_2$
RMM	497.1
m.p.	59-61 °C
v.p.	3.7 mPa at 105 °C
Solubility	In water: practically insoluble; acetone: 100 g/l; chloroform: 650 g/l; xylene: 500 g/l
Stability	Subject to hydrolysis at alkaline pH
Formulations	Emulsifiable concentrates, usually as mixtures with bromoxynil and phenoxyalkanoic acid esters

IOXYNIL OCTANOATE TECHNICAL
*86/TC/M/-

1 Sampling. Take at least 100g. Use a sampling spear and homogenise either by grinding or melting.

2 Identity tests. The identity of the active ingredient is established by comparison with the equivalent authentic standard by at least two of the following techniques:

2.1 GLC. Use the GLC method below. The relative retention time obtained from the sample should not deviate by more than 1 % from that of the standard obtained under the same conditions.

2.2 Infra-red. Prepare potassium bromide discs from the sample and a pure standard, and scan from 4000 to 600 cm^{-1}. The two spectra should not be significantly different.

2.3 NMR. Use a solution in deutero-chloroform containing tetra-methylsilane as internal standard. The NMR spectrum should not be significantly different from that the pure standard obtained under the same conditions.

3 Ioxynil octanoate

OUTLINE OF METHOD The sample and internal standard are dissolved in acetone and the ioxynil octanoate content is determined by gas chromatography on an OV-101 column, using internal standardisation.

REAGENTS

Acetone GC grade

Ioxynil octanoate standard of known purity, at least 99% w/w

Diphenyl phthalate internal standard, must not show any interference at the retention time of ioxynil octanoate

Internal standard solution. Dissolve 0.8 ± 0.005 g diphenyl phthalate in acetone in a volumetric flask (250 ml), mix well and dilute to volume.

* CIPAC method 1993. Prepared by the Ioxynil Panel of PAC-UK. Chairman: G C Buddle. Based on method prepared by Rhône Poulenc Agriculture Ltd, UK.

Calibration solution. Weigh (to the nearest 0.1 mg) 200 mg (*s* mg) ioxynil octanoate reference standard into a stoppered measuring cylinder (50 ml). Add by pipette internal standard solution (25.0 ml) and dilute to 50 ml with acetone. Shake to dissolve and mix well. Prepare in duplicate.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector and an electronic integrator or data system

Column glass, 2 m × 3 (i.d.) mm, packed with 10% OV-101 on Chromosorb W(HP) 100-120 mesh, or equivalent

PROCEDURE

(a) Operating conditions (typical):

Oven temperature	240 °C
Injector temperature	250 °C
Detector temperature	250 °C
Carrier gas	nitrogen at 50 ml/min
Injection volume	2 µl
Retention times	ioxynil octanoate: about 14 min internal standard: about 12 min

(b) Equilibration of the system. Condition the column until a stable baseline is obtained and constant (within 1%) retention times and peak areas are obtained for subsequent injections of the calibration solution. Confirm the linearity of the detector over the range of concentrations to be studied using a range of standard weights (e.g. 0.5×, 1.0×, 2.5× expected weight). If non-linear, adjust concentrations used to give a linear response.

(c) Preparation of sample solution. Weigh (to the nearest 0.1 mg) enough sample to contain 200 mg (*w* mg) into a stoppered measuring cylinder (50 ml). Add by pipette internal standard solution (25.0 ml) and dilute to 50 ml with acetone. Shake to dissolve and mix well. Prepare in duplicate.

(d) Determination. Inject 2 µl aliquots of calibration and sample solution onto the column in the order: standard 1, sample 1, sample 1, standard 2, sample 2, etc. Average the response ratios from sample (R) and calibration (R') solutions for each sample and its bracketing standards. The ratios from paired injections should agree with ± 1%. If not, repeat the determination.

(e) Calculation

$$\text{Ioxynil octanoate content} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

where:

- R = average ioxynil octanoate to diphenyl phthalate peak area ratio for the sample solution
- R' = average ioxynil octanoate to diphenyl phthalate peak area ratio for the calibration solution
- s = mass of ioxynil octanoate in the calibration solution (mg).
- w = mass of sample taken (mg)
- P = purity of ioxynil octanoate standard (g/kg)

Repeatability r = 18.6 g/kg at 950 g/kg active ingredient content
Reproducibility R = 24.7 g/kg at 950 g/kg active ingredient content

IOXYNIL OCTANOATE EMULSIFIABLE CONCENTRATES
*__86__/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for ioxynil octanoate technical **86**/TC/M/2.1.
2.2 Infra-red. As for ioxynil octanoate technical **86**/TC/M/2.2 except that the ioxynil octanoate must be separated from co-formulants by an appropriate method.

3 Ioxynil octanoate. As for ioxynil octanoate technical **86**/TC/M/3.

Repeatability r = 6.8 g/kg at 280 g/kg active ingredient content
Reproducibility R = 6.8 g/kg at 280 g/kg active ingredient content

* CIPAC method 1993. Prepared by the Ioxynil Panel of PAC-UK. Chairman: G C Buddle. Based on method prepared by Rhône Poulenc Agriculture Ltd, UK.