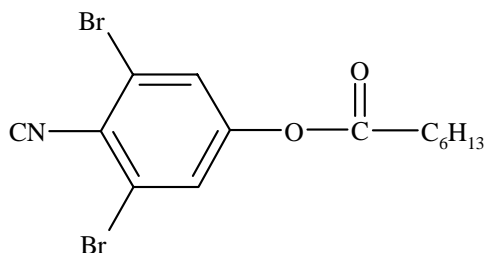


BROMOXYNIL HEPTANOATE
87.407



<i>ISO common name</i>	Bromoxynil heptanoate
<i>Chemical name</i>	4-Cyano-2,6-dibromophenyl heptanoate (IUPAC and CA RN; 56634-95-8)
<i>Empirical formula</i>	$C_{14}H_{15}Br_2NO_2$
<i>RMM</i>	389.1
<i>m.p.</i>	38 - 44°C
<i>v.p.</i>	1.06×10^{-6} Pa at 20 °C
<i>Solubility</i>	In water: 0.08 mg/l; <i>n</i> -heptane, methanol: 500g/l; toluene, ethyl acetate: 800 g/l
<i>Description</i>	A buff to brown crystalline mass
<i>Stability</i>	Stable in air up to its melting point. Hydrolysed at strongly alkaline pH
<i>Formulations</i>	Emulsifiable concentrates, often co-formulated with other herbicides

BROMOXYNIL HEPTANOATE TECHNICAL

* 87.407/TC(M)/-

1 Sampling. Take at least 100 g. Ensure that any sample taken is well mixed. Given the low melting point of the material, it is preferable to sample from a molten bulk.

2 Identity tests

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

2.2 Infra-red. Prepare potassium bromide discs from the sample and from pure bromoxynil heptanoate using 1.5 mg of the pesticide and 300 mg potassium bromide. Scan the discs from 4000 to 450 cm^{-1} . The two spectra should not be significantly different.

3 Bromoxynil heptanoate

OUTLINE OF METHOD Bromoxynil heptanoate is determined by gas chromatography on an OV-101 column, using internal standardisation.

REAGENTS

Acetone GC grade

Bromoxynil heptanoate standard of known purity, at least 990 g/kg

Diphenyl phthalate internal standard, must not show any interference at the retention time of bromoxynil heptanoate

Internal standard solution. Dissolve 1 g diphenyl phthalate in acetone (100 ml).

Calibration solution. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) 100 mg (*s* mg) bromoxynil heptanoate standard. Add by pipette internal standard solution (10.0 ml), dilute to the mark with acetone and mix well (solutions C_A and C_B).

APPARATUS

* Provisional CIPAC method 1997. Prepared by the Ioxynil Panel of PAC-UK. Chairman: P T Patel. Based on method prepared by Rhône Poulenc Agriculture Ltd, UK.

Gas chromatograph fitted with a flame ionisation detector

Column glass, 2 m × 3 mm (i.d.), packed with 10% OV-101 on Chromosorb W HP 100-120 mesh or equivalent. The top of the column should be packed with silanised glass wool, but it is important that the needle penetrates the column packing to a depth of at least 1 cm.

Electronic integrator or data system

PROCEDURE

(a) *Operating conditions* (typical):

<i>Column temperature</i>	230 °C
<i>Injector temperature</i>	250 °C
<i>Detector temperature</i>	280 °C
<i>Carrier gas</i>	nitrogen at a rate to give a retention time of about 12 min
<i>Injection mode</i>	on-column (i.e. into the glass wool)
<i>Injection volume</i>	1 µl
<i>Retention times</i>	bromoxynil heptanoate: about 12 min diphenyl phthalate: about 27 min

(b) *Linearity check.* Check the linearity of the flame ionisation detector over the appropriate concentration range for the analyte and the internal standard before conducting any analysis.

(c) *Preparation of sample solution.* Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) sufficient sample to contain about 100 mg (*w* mg) bromoxynil heptanoate. Add by pipette to each volumetric flask internal standard solution (10.0 ml) and dilute to volume with acetone. Mix well (solutions S_A and S_B).

(d) *Determination.* Inject 1 µl portions of the two calibration solutions until the consecutive response ratios agree within 1 %. Then inject aliquots of the samples and one calibration solution in the order: C_A , S_{A1} , S_{A2} , C_A , S_{B1} , S_{B2} , C_Aetc. Average the response ratios for each sample solution (R) and its bracketing injections of calibration solution (R').

(e) *Calculation*

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

where:

R = average bromoxynil heptanoate to diphenyl phthalate peak area ratio for the sample solution

R' = average bromoxynil heptanoate to diphenyl phthalate peak area ratio for the calibration solution

s = mass of bromoxynil heptanoate in the calibration solution (mg).

w = mass of sample taken (mg)

P = purity of bromoxynil heptanoate standard (g/kg)

The bromoxynil heptanoate content is the mean of the results obtained from the two sample solutions.

Repeatability r = 20 g/kg at 869 g/kg active ingredient content

= 17 g/kg at 415 g/kg active ingredient content

Reproducibility R = 32 g/kg at 869 g/kg active ingredient content

= 25 g/kg at 415 g/kg active ingredient content

BROMOXYNIL HEPTANOATE EMULSIFIABLE CONCENTRATES

* 87.407/EC/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 GLC. As for bromoxynil heptanoate technical 87.407/TC/M/2.1.

3 Bromoxynil heptanoate. As for bromoxynil heptanoate technical 87.407/TC/M/3.

Repeatability r = 11 g/kg at 340 g/kg active ingredient content

= 5.6 g/kg at 167 g/kg active ingredient content

Reproducibility R = 21 g/kg at 340 g/kg active ingredient content

= 11 g/kg at 167 g/kg active ingredient content

* Provisional CIPAC method 1997. Prepared by the Bromoxynil Panel of PAC-UK. Chairman: P T Patel. Based on a method supplied by Rhône Poulenc Agriculture Ltd, UK.