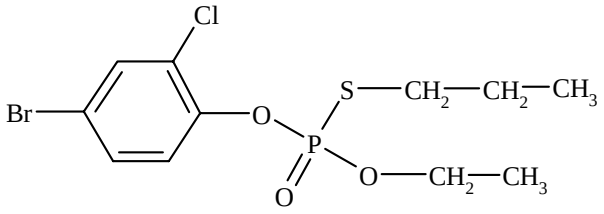
PROFENOFOS
461



ISO common name	Profenofos
Chemical name	*O*-(4-Bromo-2-chlorophenyl) *O*-ethyl *S*-*n*-propyl phosphorothioate (IUPAC and CA; *41198-08-7*)
Empirical formula	$C_{11}H_{15}BrClO_3PS$
RMM	373.6
b.p.	100 °C at 1.8 Pa
v.p.	1.2×10^{-4} Pa at 25 °C
Solubility	In water: 28 mg/l at 22 °C
Formulations	Emulsifiable concentrates and ultra-low volume liquids

PROFENOFOS TECHNICAL

[*] **461**/TC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 GLC. Use the GLC method below. The retention time of profenofos in the sample solution should not deviate by more than 0.2 min from that of the calibration solution.

2.2 Infrared. Prepare films between sodium chloride discs from the sample and pure profenofos. Scan the discs from 4000 to 600 cm^{-1}. The spectrum obtained from the sample should not differ significantly from that of the standard.

3 Profenofos

OUTLINE OF METHOD Profenofos is separated from other components by gas chromatography on a packed column with flame ionisation detection and internal standardisation.

REAGENTS

Acetone

Profenofos of known purity; better than 980 g/kg

Di-(2-ethylhexyl) adipate internal standard

Internal standard solution. Dissolve di-(2-ethylhexyl) adipate (980 to 1020 mg) in acetone (500 ml).

Calibration solution. Weigh (to the nearest 0.1 mg) profenofos standard (90 to 100 mg, *s* mg) into a glass-stoppered conical flask (50 ml). Add by pipette internal standard solution (25.0 ml) and mix well. Prepare in duplicate.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector

Column glass, 1.8 m × 2 mm (i.d.) packed with 3 % OV-210 on Supelcoport, 80-100 mesh

Electronic integrator or data system

Microsyringe 10 µl

[*] CIPAC method 1996. Prepared by the Swiss Committee (PAC-CH). Chairman: H-P Bosshardt. Based on a method supplied by Ciba, Switzerland.

PROCEDURE

(a) Operating conditions (typical):

Column	glass, 1.8 m × 2 mm (i.d.)
Packing	3 % silicone OV-210 on Supelcoport 80-100 mesh
Column temperature	180 °C
Injector temperature	240 °C
Detector temperature	280 °C
Carrier gas	helium, 35 ml/min
Injection volume	1 µl
Retention times	profenofos: 6.5 min
	internal standard: 11.4 min

(b) Linearity check. Check the linearity of the detector response by injecting 1 µl of solutions with profenofos concentrations 0.5, 1 and 2 times that of the concentration of the calibration solution. Be sure that the concentrations of the solutions are in the linear range of the detector, otherwise alter the weighings or the dilutions accordingly. Inject each solution at least twice and determine the response factor (*f*). The single values should differ by less than 0.5 % from the mean value, otherwise repeat the calibration.

(c) Preparation of sample. Weigh (to the nearest 0.1 mg) into a glass stoppered conical flask (50 ml) enough sample to contain 90 to 100 mg profenofos (*w* mg). Add by pipette internal standard solution (25.0 ml) and mix well. Prepare in duplicate.

(d) Determination. Inject each sample solution in duplicate into the gas chromatograph and bracket a series of sample solutions by duplicate injections of the calibration solutions as follows: calibration solution 1 (double injection), sample solution 1 (double injection), sample solution 2 (double injection), calibration solution 2 (double injection), and so on. Measure the relevant peak areas.
Average the response factors of each double injection. Calculate the mean value of each pair of response factors bracketing the injections of two samples and use this value for calculating the profenofos contents of the bracketed sample injections.

(e) Calculation

$$f = \frac{I_r \times s \times P}{H_s} 1$$

$$\text{Content of profenofos} = \frac{f \times H_w}{I_q \times w} \text{ 2g/kg}$$

where:

f = response factor
H_s = area of profenofos peak in the calibration solution
H_w = area of profenofos peak in the sample solution
I_r = area of internal standard peak in the calibration solution
I_q = area of internal standard peak in the sample solution
s = mass of profenofos in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of profenofos reference substance (g/kg)

Repeatability r = 10 g/kg at 967 g/kg active ingredient content
Reproducibility R = 35 g/kg at 967 g/kg active ingredient content

4 4-Bromo-2-chlorophenol (Draft method)

OUTLINE OF METHOD The impurity 4-bromo-2-chlorophenol is determined by capillary gas chromatography using flame ionisation detection and internal standardisation.

REAGENTS

Dichloromethane
4-Bromo-2-chlorophenol standard with known content
Lindane internal standard
Internal standard solution. Weigh into a volumetric flask (100 ml) 0.45 to 0.55 g lindane. Dissolve in and make up to the mark with dichloromethane.
Calibration solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (250 ml) 50 to 75 mg (*s* mg) of 4-bromo-2-chlorophenol. Dissolve in and fill to the mark with, dichloromethane. Transfer by pipette 5.0 ml of this solution to a volumetric flask, add by pipette internal standard solution (5.0 ml) and dilute to the mark with dichloromethane. Mix well.

APPARATUS

Capillary gas chromatograph capable of operating in the range 50 to 300 °C, equipped with a flame ionisation detector and an on-column injection system

Column fused silica, 15 m × 0.32 (i.d.) mm and 0.25 µm film thickness coated with OV 1701
Electronic integrator or *data system*

PROCEDURE

(a) Gas chromatographic conditions (typical):

Column	fused silica, 15 m × 0.32 (i.d.) mm, film thickness 0.25 µm, coated with OV 1701
Injection technique	On-column
Column temperature	3 min at 50 °C; → 160 °C at 10 °C/min; → 280 °C at 4 °C/min; hold at 280 °C for 10 min
Detector	flame ionisation
Detector temperature	280 °C
Carrier gas	hydrogen at 25 cm/min (determined with dichloromethane)
Make up gas	helium
Injection volume	1 µl
Run time	50 min
Retention times	4-bromo-2-chlorophenol: 10.5 min lindane: 17.8 min profenofos: 25.5 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 90 to 100 mg (*w* mg) profenofos. Add by pipette internal standard solution (5.0 ml) and dilute to the mark with dichloromethane. Mix well.

(c) Determination. Inject 1 ml portions of the calibration solution until the internal standard to the 4-bromo-2-chlorophenol peak area ratio for two consecutive runs differs by less than 2 %. Then inject each sample solution in duplicate into the gas chromatograph and bracket a series of sample solutions by injections of the calibration solutions as follows: calibration solution, sample solution 1 (double injection), calibration solution, sample solution 2 (double injection), calibration solution, and so on. Measure the relevant peak areas.
Calculate the mean response factor of the two calibration solutions preceding and following the duplicate sample solution injections and use this value for calculating the 4-bromo-2-chlorophenol contents of the bracketed sample injections.

(d) Calculation

$$f = \frac{I_r \times s \times P}{H_s \times 50} 3$$

$$\text{Content of 4-bromo-2-chlorophenol} = \frac{f \times H_w}{I_q \times w} 4\text{g/kg}$$

where:

f = response factor
H_s = area of 4-bromo-2-chlorophenol peak in the calibration solution
H_w = area of 4-bromo-2-chlorophenol peak in the sample solution
I_r = area of internal standard peak in the calibration solution
I_q = area of internal standard peak in the sample solution
s = mass of 4-bromo-2-chlorophenol in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of 4-bromo-2-chlorophenol reference substance (g/kg)

PROFENOFOS EMULSIFIABLE CONCENTRATES

[*] **461**/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 GLC. As for profenofos technical **461**/TC/M/2.1.

2.2 Infrared. Prepare a solution or dispersion of 2 g test material in 50 ml hexane- toluene, 90 + 10 (v/v). After centrifugation transfer the clear supernatant liquid to a silica gel-60 column (30 g, 25 × 2.5 cm). Wash with 200 ml hexane + toluene (90 + 10). Discard this fraction. Then elute with acetone (150 ml). Evaporate the solvent with a stream of clean, dry air and proceed as for profenofos technical **461**/TC/M/2.2.

3 Profenofos. As for profenofos technical **461**/TC/M/3. except:

Repeatability r = 8 g/kg at 448 g/kg active ingredient content
9 g/kg at 352 g/kg active ingredient content
Reproducibility R = 15 g/kg at 448 g/kg active ingredient content

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23 g/kg at 352 g/kg active ingredient content