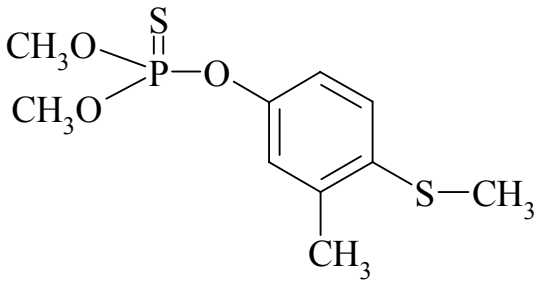
FENTHION
79



ISO common name	Fenthion
Chemical name	*O,O*-Dimethyl *O*-(3-methyl-4-methylthiophenyl) phosphorothioate (IUPAC); *O,O*-dimethyl *O*-[3-methyl-4-(methylthio)phenyl] phosphorothioate (CA; *55-38-9*)
Empirical formula	$C_{10}H_{15}O_3PS_2$
RMM	278.3
m.p.	Below -80 °C
v.p.	14×10^{-4} Pa at 25°C
Solubility	Water: 4.2 mg/l at 20 °C; soluble in acetone, toluene, methanol,
Stability	Stable under normal conditions. Subject to hydrolysis.
Description	Light yellow to reddish brown liquid
Formulations	Wettable powders, water dispersible granulate emulsifiable concentrate

FENTHION TECHNICAL

[*] **79**/TC/M2/-

1 Sampling. Take at least 100 g.

2 Identitiy tests

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

2.2 Infrared. Prepare KBr pellets of the sample and of pure fenthion. Scan the pellets from 4000 to 600 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard.

3 Fenthion

OUTLINE OF METHOD Fenthion is determined by capillary gas liquid chromatography using flame ionisation detection and internal standardisation.

REAGENTS

Acetone

Fenthion standard with known content

Di-2-ethylhexyl phthalate (DIOP), internal standard e. g. Merck, art.-no. 821874

Calibration solution. Weigh (to the nearest 0.1 mg) into a vial (50 ml) approximately 50 mg fenthion (*s* mg) and 50 mg DIOP (*r* mg), add acetone (40 ml) and dissolve. Prepare in duplicate (solutions C_1 and C_2). Solutions are stable for 24 h at room temperature.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector, a split injector and an autosampler

Column quartz, 25 m × 0.32 mm (i.d.), coated with Silicon SE 54, film thickness 0.17 µm

Burette: e.g. Metrohm Dosimat type 665

Microsyringe 1 µl

PROCEDURE

[*] CIPAC method 2005. Prepared by the the German PAC (DAPA). Chairman: R.Hänel. Based on a method supplied by Bayer CropScience GmbH, Germany.

(a) Operation conditions (typical):

Column	quartz, 25 m × 0.32 mm (i.d.), coated with Silicon SE 54, film thickness 0.17 µm
Injection system	
Injector	Split injection
Flow ratio	1 : 75
Temperatures	
Injector	240 °C
Detector	300 °C
Column	230 °C isothermic
Carrier gas	Helium
Injection volume	1.0 µl
Gas flow rates	
Helium	2.5 ml/min (at oven temperature)
Hydrogen	approx. 30 ml/min
Air	approx. 400 ml/min
Retention times	fenthion: about 1.2 min
	DIOP: about 6.2 min

(b) Sample preparation. Weigh (to the nearest 0.1 mg) into a vial (50 ml) sufficient sample to contain 50 mg fenthion (*w* mg) and 50 mg DIOP (*q* mg). Add acetone (40 ml) and dissolve. Prepare in duplicate (solutions S_1 and S_2). The solutions are stable for 24 hours.

*(c) Equilibration of the chromatographic system.*Inject the calibration solution and repeat the injections until retention times and calibration factors calculated from the peak areas vary by less than 1 % for successive injections.

(d) Determination. Inject 1 µl of the clear calibration and sample solutions in the following sequence: C_1, S_1, C_2, S_2, C_1 …….etc., bracketing the samples by the appropriate calibrations. Determine the peak areas and calculate the mean response factors from the calibration solution injections bracketing the injections of the sample solutions.

(e) Calculation

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

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

where:
f = response factor
H_s = peak area of fenthion in the calibration solution
I_r = peak area of DIOP in the calibration solution
H_w = peak area of fenthion in the sample solution
I_q = peak area of DIOP in the sample solution
s = mass of fenthion in the calibration solution (mg)
r = mass of DIOP in the calibration solution (mg)
q = mass of DIOP in the sample solution (mg)
w = mass of sample taken(mg)
P = purity of fenthion standard (g/kg)

Repeatability r = 10 g/kg at 951 g/kg active ingredient content
Reproducibility R = 44 g/kg at 951 g/kg active ingredient content

FENTHION WETTABLE POWDERS
*79/WP/M2/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 GLC. As for fenthion technical **79**/TC/M2/2.2.
2.2 Infrared. Disperse approximately 1 g of sample in dichloromethane (4 ml) and filter through a microfilter. Remove the solvent by a stream of nitrogen and transfer the residue to a small KBr plate. Scan the infrared spectrum from 4000 – 600 cm^{-1}. The spectrum produced from the sample should not differ significantly by from that of the standard.

* CIPAC method 2005. Prepared by the the German PAC (DAPA). Chairman: R.Hänel. Based on a method supplied by Bayer CropScience GmbH, Germany.

3 Fenthion As for fenthion technical **79**/TC/M2/3, except:
(b) Sample preparation. Weigh (to the nearest 0.1 mg) into a vial (50 ml) sufficient sample to contain about 50 mg fenthion (*w* mg) and 50 mg DIOP (*q* mg). Add acetone (40 ml) and homogenise. Extract for 10 minutes in an ultrasonic bath and clear the solution by centrifugation. Prepare in duplicate (solutions S_1 and S_2). The solutions are stable for 24 hours.

Repeatability r = 8.9 g/kg at 375 g/kg active ingredient content
Reproducibility R = 18 g/kg at 375 g/kg active ingredient content

4 Suspensibility (draft method)

REAGENTS AND APPARATUS as for **79**/TC/M2/3 and MT 184.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 184.

(b) Determination of fenthion in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension transfer/add (to) the remaining 25 ml to a 500 ml stoppered flask. Add 300 ml acetone containing an appropriate amount of dioctyl phthalate (internal standard) corresponding to the expected content of fenthion. Mix and clarify by centrifugation. Determine the mass of fenthion (*Q* g) by 79/TC/M2/3.

(c) Calculation

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

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

FENTHION GRANULES
[†]**79**/WP/m/-

1 Sampling. Take at least 1 kg.

2 Identity tests
2.1 GLC. As for fenthion technical **79**/TC/M2/2.1.
2.2 Infrared. Disperse approximately 5 g of sample in dichloromethane (5 ml) and treat in an ultrasonic bath for 5 min. Filter through a microfilter. Remove the solvent by a stream of nitrogen and transfer the residue to a small KBr plate. Scan the infrared spectrum from 4000 – 600 cm^{-1}. The spectrum produced from the sample should not differ significantly by from that of the standard.

3 Fenthion As for fenthion wettable powders **79**/WP/M2/3.

Repeatability r = 5.4 g/kg at 53.4 g/kg active ingredient content
Reproducibility R = 6.7 g/kg at 53.4 g/kg active ingredient content

FENTHION EMULSIFIABLE CONCENTRATES
[*]**79**/EC/M2/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for fenthion technical **79**/TC/M2/2.1.
2.2 Infrared. Dry the sample using a stream of nitrogen. Transfer the residue to a small KBr plate and scan the infrared spectrum from 4000 – 600 cm^{-1}. The spectrum produced from the sample should not differ significantly by from that of the standard.

3 Fenthion . As for fenthion technical 79/WP/M2/3, except:
(b) Sample preparation. Weigh (to the nearest 0.1 mg) into a vial (50 ml) sufficient sample to contain about 50 mg fenthion (*w* mg) and 50 mg DIOP

† Tentative CIPAC method 2005. Prepared by the the German PAC (DAPA). Chairman: R.Hänel. Based on a method supplied by Bayer CropScience GmbH, Germany.
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(*q* mg). Add acetone (40 ml) and homogenise. Prepare in duplicate (solutions S_1 and S_2). The solutions are stable for 24 hours.

Repeatability r = 7.4 g/kg at 496 g/kg active ingredient content
Reproducibility R = 29 g/kg at 496 g/kg active ingredient content

FENTHION OIL IN WATER EMULSIONS
[*]**79**/EW/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for fenthion technical **79**/TC/M2/2.1.
2.2 Infrared. Dry approximately 2 g of the sample in a petry dish for ca 2 h in an oven at 60°C. Two phases are formed. Transfer the clear phase to a small KBr plate and scan the infrared spectrum from 4000 – 600 cm^{-1}. The spectrum produced from the sample should not differ significantly by from that of the standard, apart from additional bands of non volatile components, which can be observed in the spectrum produced from the sample.

3 Fenthion. As for fenthion technical **79**/TC/M2/3.

Repeatability r = 1.4 g/kg at 97.3 g/kg active ingredient content
Reproducibility R = 7.5 g/kg at 97.3 g/kg active ingredient content

[*] CIPAC method 2005. Prepared by the the German PAC (DAPA). Chairman: R.Hänel. Based on a method supplied by Bayer CropScience GmbH, Germany.

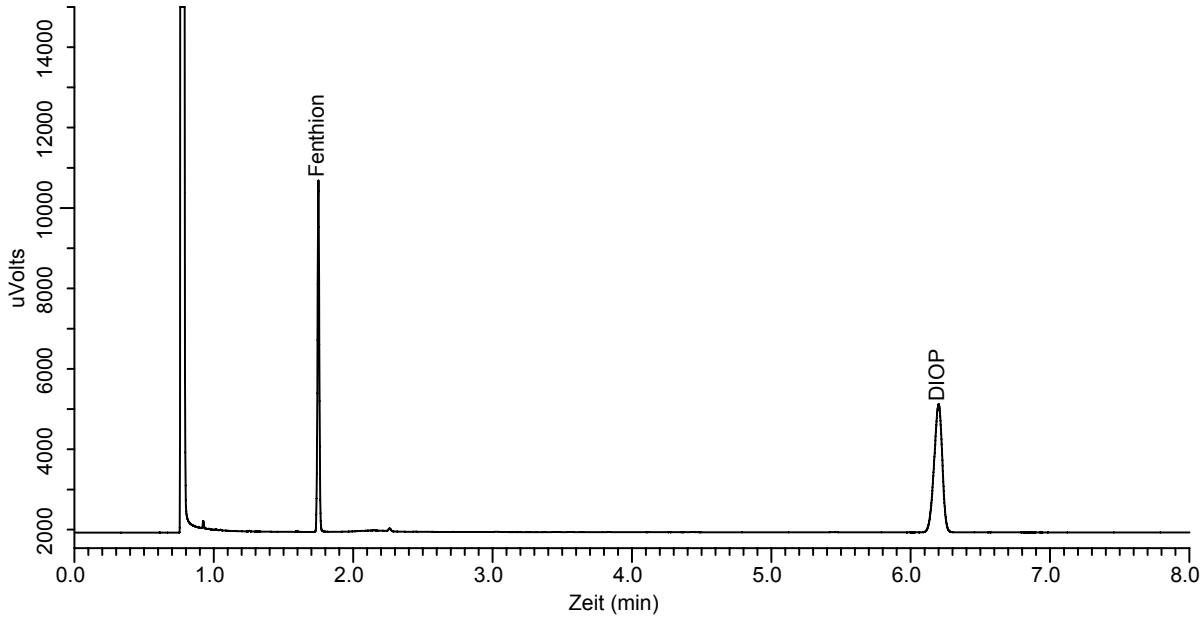

Fig. 32 Gaschromatogram of fenthion

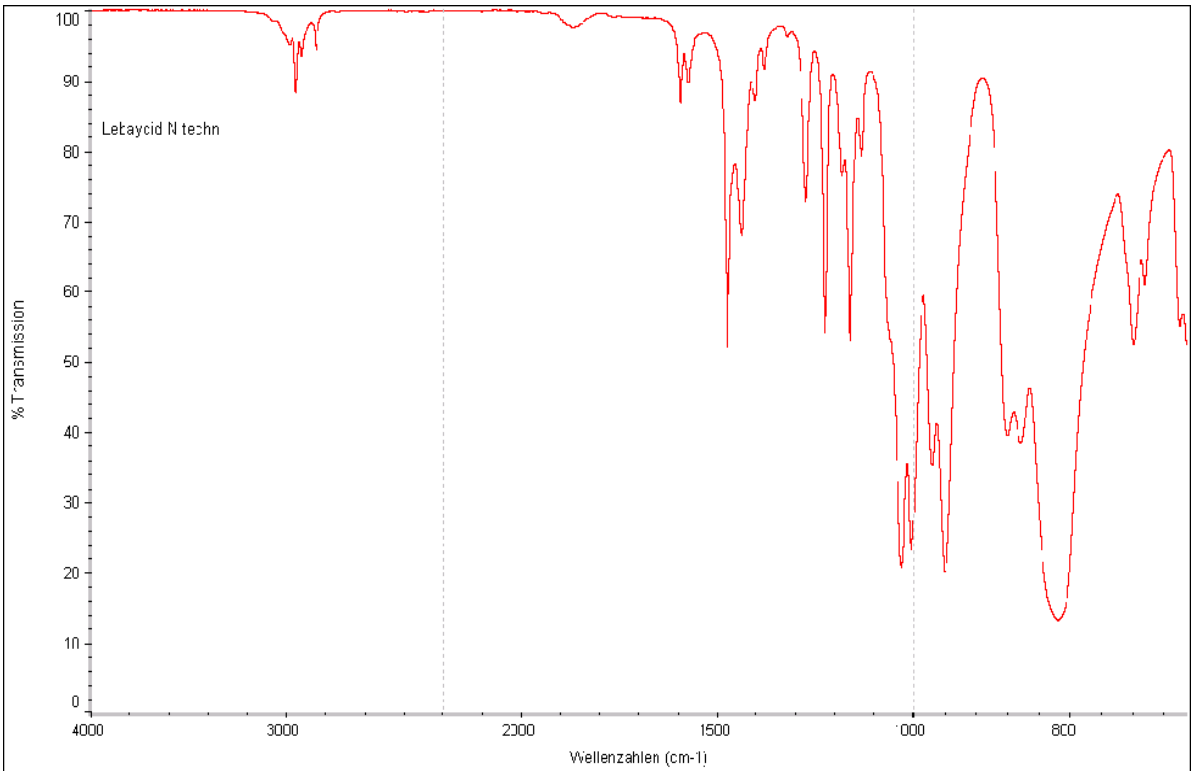

Fig. 33 Infrared spectrum of fenthion