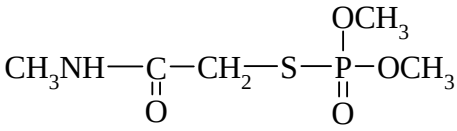
OMETHOATE
202



ISO common name	Omethoate
Chemical name	*O,O*-Dimethyl *S*-methylcarbamoylmethyl phosphorothioate (IUPAC); O,O-dimethyl *S* -[2-(methylamino)-2-oxoethyl]phosphorothioate (CA; *1113-02-6*)
Empirical formula	$C_5H_{12}NO_4PS$
RMM	213.2
v.p.	3.3×10^{-3} Pa at 20 °C
d_4^{20}	1.32
n_D^{20}	1.4987
Solubility	Miscible with water, dichloromethane and 2-propanol at 20 °C; solubility in *n*-hexane: < 1 g/l at 20 °C; toluene: 50 - 100 g/l at 20 °C
Description	Colourless-to-yellow oil
Stability	Halve life at pH 4: 120 d; pH 7: 17 d; pH 9: 28 h, at 22 °C
Formulation	Water soluble liquid

OMETHOATE TECHNICAL
[*]**202**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 Infrared. Fill a 0.015 mm KBr infrared absorption cell with the neat liquid sample. Scan the transmission spectrum from 4000 to 400 cm^{-1}. The spectrum should not differ significantly from that of an authentic sample.

[*] CIPAC-method 1992. Prepared by the German PAC (DAPA)
Chairman: W Dobrat. Based on a method supplied by Bayer AG, (FRG).

2.2 HPLC. Use the HPLC method below. The retention times of omethoate obtained from sample solution and calibration solution must not deviate by more than 2%.

2.3 TLC. Carry out a thin layer chromatographic identity test by comparing the sample with the standard using the ^following conditions:

TLC plate	Coated with silica gel 60 F_{254} 0.25 mm (e.g. Merck, Darmstadt, FRG, Art.-No. 7515)
Solvent	chloroform/methanol 95 + 5 (v/v)
Sample solution	Dissolve a sample amount containing about 60 mg omethoate in approximately 5 ml methanol in a 10 ml volumetric flask and make up to volume with methanol. Apply the solution immediately.
Reference solution I	Dissolve 60 mg of omethoate authentic substance in approx. 5 ml methanol in a 10 ml volumetric flask and make up to volume with methanol. Apply the solution immediately.
Reference solution II	Mix 5 ml of the sample solution with 5 ml of the reference solution I. Apply the solution immediately.
Loading	10 µl of reference solution I, II and of the sample solution
Travelling distance	15 cm
Visualization	Spray the plate with a 0.5% aqueous solution of palladium-II-chloride
Rf value	omethoate, approximately 0.45

3 Omethoate

OUTLINE OF METHOD A solution of the sample is separated by reversed phase high performance liquid chromatography. The content of active ingredient is determined by external standardization.

REAGENTS

Methanol HPLC quality
Water HPLC quality
Eluent water/methanol 90 + 10 (v/v), premixed
Omethoate reference substance of known purity
Calibration solution Weigh (to the nearest 0.1 mg) about 0.1 g of pure omethoate (*s* g) into a 25 ml volumetric flask, dissolve in 20 ml of eluent and make up to volume with eluent and homogenize.

APPARATUS

Liquid chromatograph equipped with a spectrophotometric detector (wavelength: 220 nm) and a loop-injection valve (5 µl).
Liquid chromatographic column stainless steel, 150 × 3.9 (i.d.) mm, packed with Resolve C 18, 5 µm (Waters)

PROCEDURE

(a) Operating conditions (typical):

Eluent flow rate	1.0 ml/min
Column temperature	40 °C
Injection volume	5 µl
Detector wavelength	220 nm
Retention time	about 4 min
Run time	15 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) sufficient sample (*w* g) to contain 0.1 g of omethoate into a 25 ml volumetric flask, dissolve in 20 ml of eluent and make up to volume with eluent.
(*c) Determination*. Inject 5 µl of two calibration solutions (different weights of pure omethoate) alternately until the calibration factor varies by less than 1% for the two last injections. Inject 5 µl of the sample solution. Repeat the calibration after injection of two samples using the calibration solutions alternately. Calculate the average calibration factor with the factors of the calibration solution preceding and following the samples.
(d) Calculation

$$\text{calibration factor} \quad f = \frac{s \times P}{H_s}$$

$$\text{Omethoate content} \quad = \frac{H_w \times f}{w}$$

where:

H_w = peak area of omethoate in sample solution
w = mass of sample taken (g)
s = mass of pure omethoate in calibration solution (g)
P = purity of omethoate reference substance (g/kg)
H_s = peak area of omethoate in calibration solution

Repeatability r	=	13 g/kg	at	967 g/kg	active	ingredient	content
Reproducibility R	=	14 g/kg	at	967 g/kg	active	ingredient	content

OMETHOATE SOLUBLE CONCENTRATES
*202/SL/M/-

1 Sampling. Take at least 500 ml.

2 Identity test. As for omethoate technical **202**/TC/M/2.

3 Omethoate. As for omethoate technical **202**/TC/M/3.

Repeatability r	=	10.8 g/kg	at	800 g/kg	active	ingredient	content
	=	6.3 g/kg	at	50 g/kg	active	ingredient	content
	=	5.0 g/kg	at	500 g/kg	active	ingredient	content
	=	2.3 g/kg	at	25 g/kg	active	ingredient	content
Reproducibility R	=	13.9 g/kg	at	800 g/kg	active	ingredient	content
	=	11.3 g/kg	at	50 g/kg	active	ingredient	content
	=	7.1 g/kg	at	500 g/kg	active	ingredient	content
	=	5.0 g/kg	at	25 g/kg	active	ingredient	content

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