DIMETHOATE
59

See CIPAC H, *p. 153*.

DIMETHOATE TECHNICAL
*59/TC/M3/-

HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC METHOD

1 Sampling. Dimethoate technical may undergo spontaneous zone purification in storage. Take at least 100 g and store at a maximum temperature of 20 °C. When taking subsamples heat the sample gently in a water bath at 60 °C until it melts. Homogenize by stirring and remove portions for all the analyses at the same time.

2 Identity tests

2.1 TLC and Infrared. - CIPAC H, *p. 154*

2.2 HPLC. Use the HPLC method below. the retention time of dimethoate for the sample solution should not deviate by more than 1 % from that for the calibration solution (successively injected).

3 Dimethoate

OUTLINE OF METHOD The active ingredient is chromatographed by HPLC on a reversed phase column (C_8, acetonitrile/water) and quantitatively determined by UV detection at 210 nm with external calibration.

REAGENTS

Acetonitrile HPLC grade
Glacial acetic acid
Water HPLC *grade*
Dimethoate calibration standard of known purity; CIPAC PP 59
Mobile phase acetonitrile-water-glacial acetic acid, 400 + 600 + 1 (v+v+v)
Calibration solution. Weigh (to the nearest 0.01 mg) about 20 - 30 mg of dimethoate (*s* mg) into a 50 ml volumetric flask and dissolve quantitatively in acetonitrile (35 ml). Add 10 ml of water and, after temperature equalization, make up to volume with acetonitrile. Adjust the amount of dimethoate (*s* mg) such that the peak height is not more than 80 % of full scale. In case of doubt, check the linearity of the detector. Prepare two calibration solutions of similar concentration. Do not use the same calibration solutions for periods longer than 48 hours.

* CIPAC method 1992. Prepared by the German Committee (DAPA), Chairman: Dr W Dobrat. Based on a method supplied by BASF (FRG)

APPARATUS

HPLC system consisting of:
- high precision HPLC pump
- injection valve with 10 µl loop, e. g. Rheodyne 7010
- stainless steel column 125 × 4.6 mm, packed with NUCLEOSIL®5 C_8 of Macherey and Nagel or equivalent (RP-8, 5 µm)
- variable wavelength UV detector with linear response at high absorbance values, e. g. Perkin Elmer LC 75
- data system with recorder for peak evaluation

PROCEDURE

(a) Preparation of sample solution. Weigh (to the nearest 0.01 mg) into a 50 ml volumetric flask enough sample (*w* mg) to contain about 25 mg of pure dimethoate. Dissolve in acetonitrile (35 ml). Add water and after temperature equalizations make up to volume with acetonitrile. The concentration of dimethoate should be similar to its concentration in the calibration solutions. Prepare two solutions for each sample and analyze them immediately.

(b) Chromatographic conditions (typical):

Column	stainless steel column 125 x 4.6 i.d. mm
Stationary phase	Nucleosil/5 C8 (5 m) from Macherey & Nagel
Mobile phase	acetonitrile/water/glacial acetic acid, 400:600:1 (v+v+v)
Injection volume	10 µl
Flow rate	1.5 ml/min
Detector wavelength	210 nm
Detector sensitivity	0.5 absorbence units full scale
Chart speed	1 cm/min
Retention time of dimethoate	about 2 min
Temperature	ambient

(*c) Determination*. Inject 10 µl portions of both calibration solutions. Inject each calibration solution at least two times and calculate the average peak area to mass ratio. The individual values should not deviate from the mean by more than ± 0.7 %, otherwise repeat the calibration. Then inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution I, sample solution 1, sample solution 1, calibration solution II, sample solution 2, sample solution 2, calibration solution I,

and so on for other sample solutions. Measure the relevant peak areas. Calculate the mean value of each pair of calibration factors *(d)* bracketing two sample injections and use this value for evaluating the two bracketed sample runs.

(d) Calculation
Calibration factor:

$$f = \frac{H_s}{s}$$

where:

H_s = area of the dimethoate peak in the calibration solution
s = mass of dimethoate standard taken (mg)

$$\text{Dimethoate content} = \frac{H_w \times P}{f \times w} \text{ g/kg}$$

where:

H_w = area of the dimethoate peak in the sample solution
w = mass of the sample taken (mg)
P = Purity of dimethoate standard (g/kg)

The content of dimethoate is the mean value of the results of the sample solutions.

Repeatability r = 30 at 955 g/kg active ingredient content
Reproducibility R = 32 at 955 g/kg active ingredient content

Based on a study with 20 participants and 80 values.

DIMETHOATE EMULSIFIABLE CONCENTRATES
[*]**59**/EC/(M3)

1 Sampling. Take at least 1 l.

2 Identity tests. As for **59**/TC/M3/2
3 Dimethoate. As for **59**/TC/M3/3 except:
(a) Preparation of sample solutions. Weigh (to the nearest 0.1 mg) into a 50 ml volumetric flask enough sample (*w* mg) to contain about 25 mg of pure dimethoate.

[*] CIPAC method 1992. Prepared by the German Committee (DAPA), Chairman: Dr W Dobrat. Based on a method supplied by BASF (FRG)

Dissolve in acetonitrile (35 ml) and proceed as described for **59**/TC/M3/3(a). The concentration of dimethoate should be similar to its concentration in the calibration solutions. Prepare two solutions for each sample.

Repeatability r	=	7 at 463 g/kg active ingredient content
	=	11 at 381 g/kg active ingredient content
	=	9 at 215 g/kg active ingredient content
Reproducibility R	=	20 at 463 g/kg active ingredient content
	=	17 at 381 g/kg active ingredient content
	=	13 at 215 g/kg active ingredient content

Based on results of a study with 20 participants and 80 values.