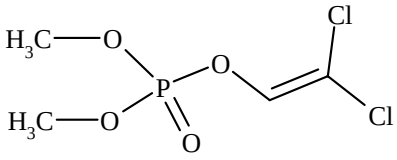
DICHLORVOS
11



ISO common name	Dichlorvos
Chemical name	2,2-Dichlorovinyl dimethyl phosphate (IUPAC); 2,2-dichloroethenyl dimethyl phosphate (CA; *62-73-7*)
Empirical formula	$C_4H_7Cl_2O_4P$
RMM	221.0
b.p	74 °C at 133 Pa
v.p.	1.6 × Pa at 20 °C
Solubility	In water: 10 g/l; readily soluble in most organic solvents
Description	Colourless to yellowish liquid with mild aromatic odour
Stability	Slowly hydrolysed in the presence of water; decomposes very rapidly in the presence of alkali; more slow decomposition under acidic conditions
Formulations	Emulsifiable concentrates, water miscible solutions, aerosols, oil soluble concentrates, baits and slow release formulations

DICHLORVOS TECHNICAL

*__11__/TC/(M)/-

1 Sampling. Take at least 100 g. Dichlorvos is very hygroscopic and rather volatile. Fill sample containers completely and seal with airtight caps.

2 Identity tests

2.1 GLC. Use the method outlined below. The relative retention time of dichlorvos 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 films between sodium chloride discs from the sample and pure dichlorvos. 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 Dichlorvos

OUTLINE OF METHOD The sample is dissolved in acetone and the dichlorvos content is determined by gas liquid chromatography using flame ionisation detection and internal standardisation.

REAGENTS

Dichlorvos analytical standard of known purity (purity at least 990 g/kg)

Diethyl pimelate internal standard, purity at least 990 g/kg, free from components which co-elute with dichlorvos under the conditions given

Acetone

Calibration solutions. Weigh (to the nearest 0.1 mg) into separate volumetric flasks (50 ml) 200 and 250 mg (*s* mg) dichlorvos reference standard. Into each flask weigh (to the nearest 0.1 mg) diethyl pimelate (about 100 mg, *r* mg), dilute to volume with acetone and mix thoroughly.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector

Column glass, 1.5 m × 4 mm (i.d.) packed with 3% OV-225 on Gas Chrom Q, 100 - 120 mesh or equivalent high performance support

Electronic integrator or data system

Microsyringe 10 µl

*Provisional CIPAC method 1981. Prepared by the Dichlorvos Panel of PAC-UK.

PROCEDURE

(a) Operating conditions (typical):

Column	
dimensions	1.5 m × 4 mm (i.d.)
packing	3 % silicone OV-225 on Gas Chrom Q, 100-120 mesh
Detector system	flame ionisation detector
Temperatures	
injection port	160 °C
column oven	140 °C
detector	250 °C
Flow rates	
nitrogen (carrier)	40 ml/min
hydrogen)	as recommended for the detector
air)	
Injection volume	2 to 6 µl
Number of theoretical plates	1200 (dichlorvos)
Retention times	dichlorvos: about 5 min
	internal standard: about 9.5 min

(b) Preparation of sample solution. Weigh (to the nearest 0.1 mg) in triplicate into separate volumetric flasks (50 ml) enough sample to contain 230 ± 10 mg dichlorvos (*w* mg). Weigh (to the nearest 0.1 mg) into each flask about 100 mg of diethyl pimelate (*q* mg). Dilute to volume with acetone and mix thoroughly.

(c) System equilibration. Inject 2 to 6 µl of the calibration solution and adjust the instrument controls so that the maximum of the larger peak (either dichlorvos or diethyl pimelate) gives a recorder reflection of 80 to 90 % full scale. Note the volume injected (*y* µl). Then inject in duplicate *y* µl portions of the calibration solutions. Measure the peak areas and calculate the response factors (f_i).

$$f_i = \frac{I_r \times s}{H_s \times r} 1$$

where:

H_s = area of dichlorvos peak in the calibration solution
I_r = area of internal standard peak in the calibration solution
r = mass of internal standard in the calibration solution
s = mass of dichlorvos in the calibration solution (mg)

Repeat the injections until the values of f_i obtained do not differ from each other by more than ± 0.5% relative to the mean. If consistent and unacceptable differences between calibration solutions are obtained, the solution should be prepared again.

(d) Determination. Inject in duplicate *y* µl portions of the sample solution. Bracket each group of four sample injections with calibration solution injections e.g., calibration solution A_1, sample solution S_1, sample solution S_1, sample solution S_2, sample solution S_2, calibration solution A_2. Determine the response factors f_{i1} and f_{i2} of each of the calibration solutions bracketing the four sample injections. Calculate the mean response factor *f*. If the factors f_{i1} and f_{i2} differ by more than 0.5 % of the mean *f*, repeat both calibration and sample injections and if the response factor *f* differs from f_i determined at system equilibration by more than 2% relative, stabilise the operating conditions and re-calibrate.

(d) Calculation

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

where:

f = average response factor
H_w = area of dichlorvos peak in the sample solution
I_q = area of internal standard peak in the sample solution
q = mass of internal standard in the sample solution (mg)
w = mass of sample taken (mg)
P = purity of dichlorvos reference substance (g/kg)

Repeatability r not determined
Reproducibility R not determined

DICHLORVOS EMULSIFIABLE CONCENTRATES
*__11__/EC/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for dichlorvos technical **11**/TC/(M)/2.1.
2.2 Infrared. Not available.

*Provisinal CIPAC method 1981. Prepared by the Dichlorvos Panel of PAC-UK.

3 Dichlorvos. As for dichlorvos technical **11**/TC(M)/3 except:
(b) Preparation of sample solution. Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (100 ml) enough sample to contain about 450 mg dichlorvos (*w* mg). Weigh (to the nearest 0.1 mg) into each flask about 200 mg of diethyl pimelate (*q* mg). Dilute to volume with acetone and mix thoroughly.

Repeatability r not available
Reproducibility R not available

DICHLORVOS WATER SOLUBLE CONCENTRATES
[*]**11**/SL/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for dichlorvos technical **11**/TC/(M)/2.1.
2.2 Infrared. Not available.

3 Dichlorvos. As for dichlorvos technical **11**/TC(M)/3 except:
(b) Preparation of sample solution. Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (100 ml) enough sample to contain about 450 mg dichlorvos (*w* mg). Weigh (to the nearest 0.1 mg) into each flask about 200 mg of diethyl pimelate (*q* mg). Dilute to volume with acetone and mix thoroughly.

Repeatability r not available
Reproducibility R not available

[*]Provisional CIPAC method 1981. Prepared by the Dichlorvos Panel of PAC-UK.

DICHLORVOS HOT FOGGING CONCENTRATES
*11/HN/(M)/-

1 Sampling. Take at least 500 ml.

2 Identity tests
2.1 GLC. As for dichlorvos technical **11**/TC/(M)/2.1.
2.2 Infrared. Not available.

3 Dichlorvos. As for dichlorvos technical **11**/TC(M)/3 except:
(b) Preparation of sample solution. Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (100 ml) enough sample to contain about 450 mg dichlorvos (*w* mg). Weigh (to the nearest 0.1 mg) into each flask about 200 mg of diethyl pimelate (*q* mg). Dilute to volume with acetone and mix thoroughly.

Repeatability r not available
Reproducibility R not available

* Provisional CIPAC method 1981. Prepared by the Dichlorvos Panel of PAC-UK.