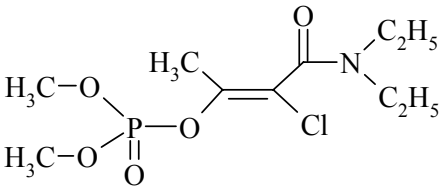
PHOSPHAMIDON
110



ISO common name	Phosphamidon
Chemical name	2-Chloro-2-(diethylcarbamoyl)-1-methylvinyl dimethyl phosphate (IUPAC); 2-chloro-3-(diethylamino)-1-methyl-3-oxo-1-propenyl dimethyl phosphate (CA; *13171-21-6 E + Z* isomers; *23783-98-4*, *Z* isomer and *297-99-4*, *E* isomer)
Empirical formula	$C_{10}H_{19}ClNO_5P$
RMM	299.7
b.p.	162 °C at 200 Pa
v.p.	3.3×10^{-3} Pa at 20 °C
n_D^{25}	1.4721
Solubility	Miscible with water and common organic solvents; moderately soluble in hexane: 32 g/kg
Description	Yellow liquid
Stability	Very stable in neutral and acidic media. Rapidly hydrolysed by alkalics.
Formulations	Soluble liquids

PHOSPHAMIDON TECHNICAL
*110/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity test

2.1 HPLC. Use the HPLC method below. The retention time of phosphamidon for the sample solution should not deviate by more than 2% from that for the calibration solution.

3 Phosphamidon

OUTLINE OF METHOD Phosphamidon is separated from impurities by reversed phase liquid chromatography, using UV detection at 218 nm and external standardization.

REAGENTS

Acetonitrile HPLC grade
Methanol HPLC grade
Buffer solution pH 6. Dissolve 0.071 mol of potassium dihydrogenphosphate and 0.010 mol of disodium hydrogenphosphate in 1 l of water.
Mobile phase buffer solution-water-acetonitrile-methanol, 170 + 170 + 135 + 25 (v/v). Filter through a 0.45 µm filter and degas before use.
Phosphamidon standard of known purity
Calibration solutions. Weigh (to the nearest 0.01 mg) into each of two volumetric flasks (50 ml) 50 to 60 mg phosphamidon standard (*s* mg) and dissolve in mobile phase. Dilute to volume with mobile phase, and mix well. Phosphamidon is sensitive to hydrolysis. Avoid any contact with water. Check the standard substance by recording a chromatogram of the freshly prepared calibration solution. The hydrolysis product of phosphamidon elutes before deschlorophosphamidon. Its peak area should not exceed 0.5% of the area of the phosphamidon peak. Otherwise take a new standard substance. The stability of the calibration solution is limited. After 24 h at room temperature usually more than 0.5% degradation can be observed. Do not use calibration solutions for longer periods than 24 h and prepare the calibration and sample solutions at the same time.

* CIPAC method 1990. Prepared by the German and Swiss Committees. Based on a method supplied by Ciba-Geigy, Switzerland.

APPARATUS

Liquid chromatograph fitted with an UV spectrophotometric detector capable of operating at 218 nm, a pulse free pump and a loop injection valve.
Column stainless steel, 250 × 4.6 (i.d.) mm, packed with Nucleosil C_{18} (10 µm)
Electronic integrator

PROCEDURE

(a) Operating conditions (typical):

Flow rate	2 ml/min
Column temperature	ambient
Detector wavelength setting	218 nm
Injection volume	10 µl
Retention times	deschlorophosphamidon: 3.6 min phosphamidon: 5.95 min. Depending on the quality of the column, the phosphamidon peak may split. γ-chlorophosphamidon: 11.1 min

(b) Preparation of sample. Weigh (to the nearest 0.01 mg) into a volumetric flask (50 ml) enough sample to contain 55 to 65 mg (*w* mg) of pure phosphamidon. Dissolve in mobile phase. Dilute to volume with mobile phase, and mix well.
(c) Determination. Inject 10 µl portions of the first calibration solution until the peak areas of phosphamidon varies by less than 1% for successive injections. Inject the second calibration solution. The response factor (*f*) of that solution (mean of two injections) should not deviate by more than 0.5% from that of the first one. Otherwise prepare new calibration solutions. Carry out injections of 10 µl of the calibration solutions (C) and the sample solutions (S) in the following sequence: CA, CA, S_1, S_1, S_2, S_2, CB, CB, S_3, S_3 .....
If the *E* and *Z* isomers of phosphamidon are separated on the column used, the sum of the areas of both peaks should be taken for the calculations.
(d) Calculation. Calculate the mean value of each pair of response factors (*f*) bracketing the injections of the two sample solutions

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

$$\text{Phosphamidon content (A)} = \frac{H_w}{w \times f} \text{ g/kg}$$

where:

f = mean of response factor
H_s = peak area of phosphamidon in the calibration solution (or sum of the areas the *E* and *Z* phosphamidon peaks)
H_w = peak area of phosphamidon in the sample solution (or sum of areas of the *E* and *Z* phosphamidon peaks)
s = mass of phosphamidon in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of phosphamidon standard (g/kg)

4 Deschlorophosphamidon and γ-chlorophosphamidon

OUTLINE OF METHOD Deschlorophosphamidon (2-diethylcarbamoyl-1-methylvinyl dimethyl phosphate) and γ-chlorophosphamidon (2-chloro-2-diethylcarbamoyl-1-chloromethylvinyl dimethyl phosphate) are determined together with phosphamidon using a fixed response factor relative to the phosphamidon factor.

REAGENTS, APPARATUS and PROCEDURE As for phosphamidon **110**/TC/M/3 except:

(d) Calculation

$$\text{Deschlorophosphamidon content (B)} = \frac{H_{wd} \times 0.73}{w \times f} \text{ g/kg}$$

$$\text{γ-Chlorophosphamidon content (C)} = \frac{H_{wc} \times 0.83}{w \times f} \text{ g/kg}$$

where:

f = mean response factor of phosphamidon
H_{wd} = peak area of deschlorophosphamidon in the sample solution
H_{wc} = peak area of γ-chlorophosphamidon in the sample solution
w = mass of sample taken (mg)

5 Total active substances

Content of total active substances = A +B + C g/kg

Repeatability r = 16.2 g/kg at 937 g/kg active ingredient content
Reproducibility R = 24.9 g/kg at 937 g/kg active ingredient content

Based on a study with 9 participants and 36 values.

PHOSPHAMIDON SOLUBLE LIQUIDS
*110/SL/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests. As for phosphamidon technical **110**/TC/M/2.

3 Phosphamidon. As for phosphamidon technical **110**/TC/M/3.

4 Deschlorophosphamidon and γ-chlorophosphamidon. As for phosphamidon technical **110**/TC/M/4.

5 Total active substances. As for phosphamidon technical **110**/TC/M/5.

Repeatability r = 6.1, 4.5, 2.1 and 2.0 g/kg at active ingredient contents of 474, 203, 179 and 105 g/kg respectively.
Reproducibility R = 14.7, 8.0, 3.8 and 3.2 g/kg at active ingredient contents of 474, 203, 179 and 105 g/kg respectively.

Based on a study with 9 participants and 36 values for each level.

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