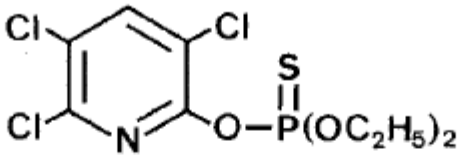
CHLORPYRIFOS

221.b



ISO common name	Chlorpyrifos
Chemical name	*O,O*-diethyl *O*-(3,5,6-trichloro-2-pyridyl) phosphorothioate (IUPAC, CA; *2921-88-2*)
Other names	chlorpyriphos, chlorpyriphos-ethyl.
Empirical formula	$C_9H_{11}Cl_3NO_3PS$
RMM	350·6
m.p.	42·5 to 43°C
v.p.	2·49 mPa ($1·87 \times 10^{-5}$ mm Hg) at 25°C
Solubility	At 35°C, 2 mg/l water, 790 g/kg octanes, 430 g/kg methanol. Readily soluble in most other organic solvents.
Description	Colourless crystals with a mild mercaptan odour.
Stability	Stable under normal storage conditions.
Formulations	As dustable and wettable powders, emulsifiable concentrates and granules.

CHLORPYRIFOS TECHNICAL

***221.b**/TC/M/–

1 Sampling. Take at least 50 g.

2 Identity tests. Use the HPLC method 3 below. The identity is confirmed if the difference between the retention times of chlorpyrifos and internal standard for the sample solution does not deviate by more than 10 seconds from that for the standard solution.

3 Chlorpyrifos

OUTLINE OF METHOD Extract the active ingredient with acetonitrile containing 1,4-dibromonaphthalene as internal standard and separate chlorpyrifos on a Zorbax ODS column by reverse phase HPLC adding a small amount of acetic acid to the mobile phase to suppress a non-reproducible ionization of 3,5,6-trichloro-2-pyridinol, which might otherwise interfere.

* AOAC–CIPAC method 1983.

REAGENTS

Acetonitrile, HPLC grade
Water, HPLC grade
Acetic acid glacial, HPLC grade
Eluant: acetonitrile, water, acetic acid (82 + 17·5 + 0·5). Mix 820 ml acetonitrile, 175 ml water and 5 ml glacial acetic acid and degas.
1,4-dibromonaphthalene internal standard, m.p. 81–83°C.
Solution: (37·5 mg/25 ml). Weigh 1·5 ± 0·1 g 1,4-dibromonaphthalene into 1 liter volumetric flask, dissolve and dilute to volume with acetonitrile and mix.
Chlorpyrifos, standard purity better than 99·7%.
Calibration solution: weigh (to the nearest 0·1 mg) about 80 mg of chlorpyrifos standard (s g) into a 50 ml glass-stoppered conical flask. Add by pipette 25 ml of the internal standard solution (Note 1) and mix thoroughly.

APPARATUS

Liquid chromatograph Perkin-Elmer modular apparatus LC 55 or LC 75 or equivalent with a variable wavelength detector, an Altex 100 pump or equivalent, a Micromeritics Model 725 sample-injector or equivalent, a column heating unit LC-22 temperature controller and LC-23 column heater or equivalent. For manual injections, a Rheodyne Model 7125 is recommended.
Liquid chromatographic column Zorbax® ODS, 4·6 mm × 25 cm; 2 μm column filter

PROCEDURE

(*a*) *Operating conditions (typical)*
Eluant flow rate: 2 ml/min (about 7·4 MPa)
Wavelength: 300 nm
Absorbance range: 0–1·0 absorbance unit full scale
Injection volume: 10 μl
Column temperature: ambient (if a temperature control is available, 30°C is recommended)

(*b*) *Sample preparation.* Weigh (to the nearest 0·1 mg) sufficient sample to contain about 80 mg (w g) into a 50 ml glass-stoppered conical flask. Add by pipette 25 ml of the internal standard solution (Note 1) and mix thoroughly.

(*c*) *Determination.* Inject 10 μl chlorpyrifos calibration solution into the column and adjust the attenuation to give the largest possible on scale peaks (about 1·0 absorbance unit full scale). Repeat injections until the ratio of the chlorpyrifos peak height (or area) to the internal standard peak height (or area) varies ≤ 0·5% (R'). Without changing the conditions, inject 10 μl aliquots of the sample solution until the ratio of the chlorpyrifos peak height (or area) to the internal standard peak height (or area) varies ≤0·5%. Average the last two ratios for the sample solution (R).

(*d*) *Calculation*

$$\text{Chlorpyrifos content} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

where: R = average peak height (or area) ratios of chlorpyrifos and 1,4-dibromonaphthalene for the sample
R' = average peak height (or area) ratios of chlorpyrifos and 1,4-dibromonaphthalene for the calibration solution
w = mass of the sample (in mg)
s = mass of pure chlorpyrifos (in mg) in the calibration solution
P = purity (g/kg) of the pure chlorpyrifos

Note 1 The same pipette must be used for measuring the internal standard for both sample and calibration solutions.

CHLORPYRIFOS DUSTABLE POWDER

***221.b**/DP/M/–

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **221.b**/TC/M/2.

3 Chlorpyrifos. As for the technical **221.b**/TC/M/3, but add at:

APPARATUS

Millipore filtration apparatus with filter, 1·0 μm porosity
Wrist-action shaker

PROCEDURE

(*b*) *Sample preparation.* Weigh (to the nearest 0·1 mg) sufficient sample to contain about 80 mg (w g) into a 50 ml glass-stoppered flask. Add by pipette 25 ml of the internal standard solution using the same pipette as used for the calibration chlorpyrifos solution. Place on a wrist-action shaker for 5 min. Filter the sample through a 1 μm filter before injection.

CHLORPYRIFOS WETTABLE POWDER

***221.b**/WP/M/–

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **221.b**/TC/M/2.

3 Chlorpyrifos. As for the dustable powder **221.b**/DP/M/3.

* AOAC–CIPAC method 1983.

CHLORPYRIFOS EMULSIFIABLE CONCENTRATE

***221.b**/EC/M/–

1 Sampling. Take at least 500 ml.

2 Identity tests. As for the technical **221.b**/TC/M/2.

3 Chlorpyrifos. As for the technical **221.b**/TC/M/3.

CHLORPYRIFOS GRANULES

***221.b**/GR/M/–

1 Sampling. Take at least 500 g.

2 Identity tests. As for the technical **221.b**/TC/M/2.

3 Chlorpyrifos. As for the technical **221.b**/TC/M/3, except:

APPARATUS

As for the dustable powder **221.b**/DP/M/3

PROCEDURE

(*b*) *Sample preparation*. Weigh (to the nearest mg) sufficient sample to contain about 160 mg (*w* g) into a 100 ml glass-stoppered flask. Add by pipette 50 ml of the internal standard solution. Place on a wrist-action shaker for 5 min. Filter the sample through a 1 μm filter before injection.

* AOAC–CIPAC method 1983.