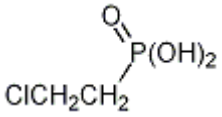
Ethephon

HPIC method

CIPAC Full Scale Collaborative Trial

5315/m

ETHEPHON



Common name	ethephon
Chemical name	2-chloroethylphosphonic acid
CAS No.	16672-87-0
Empirical formula	$C_2H_6ClO_3P$
RMM	144.5
m.p.	74–75 ℃
pKa	pKa1 2.5, pKa2 7.2
v.p.	<0.01 mPa (20 ℃)
Solubility	In water 800 g/l (pH 4). Readily soluble in methanol, ethanol, isopropanol, acetone, diethyl ether and other polar organic solvents. Sparingly soluble in non-polar organic solvents such as benzene and toluene. Insoluble in kerosene and diesel oil.
Stability	Stability Stable in aqueous solutions having pH <5; at higher pH, decomposition occurs with the liberation of ethylene; DT_{50} 2.4 d (pH 7, 25 ℃). Sensitive to uv irradiation
Description	White crystalline powder
Formulation	Soluble Concentrate (SL)

ETHEPHON TECHNICAL
373/TC/M/-

1 Sampling. Take at least 100 g.

2. Identity test

2.1 HPIC. Use the reversed phase HPIC method below. The relative retention time of the ethephon peak in the sample solution should not deviate by more than 1.5% from that of the calibration solution.

2.2 NMR. Use a solution in deuterium oxide containing sodium 3-trimethylsilyl propionate as internal standard. The 1H spectrum of ethephon displays the following characteristics:

a single peak at δ 4.79 ppm J(O–H) = 7.5 Hz, corresponding to P-(OH)2
a split triplet at δ 2.34 ppm J(H–H) = 7.5 Hz and J(H–C–H) = 18 Hz, corresponding to P-CH2
a split triplet at δ 3.79 ppm J(H–H) = 7.5 Hz and J(H–C–C–H) = 14 Hz, corresponding to CH2Cl.

3 Ethephon

OUTLINE OF METHOD
Ethephon is determined by high performance ion chromatography using electrolytic conductivity detector and external standardisation.

REAGENTS
Ethephon reference standard of known content
Water, HPLC grade
Sodium Carbonate, Analytical reagent or Guaranteed Reagent
Sodium Hydroxy Carbonate, Analytical reagent or Guaranteed Reagent
Calibration solution. Prepare calibration solution in duplicate. Weigh (to the nearest 0.1 mg) sufficient reference standard (w mg) to contain about 120 mg (ideally between 112 and 130 mg) of ethephon into a volumetric flask (100 ml). Add ultrapure water to the mark and mix thoroughly. Transfer 5.00 ml of the above solution into a 50 ml volumetric flask, add ultrapure water to the mark and mix thoroughly. (Calibration solutions C_A and C_B).

APPARATUS
High performance ion chromatograph, equipped with an electrolytic conductivity detector and an injection system capable of injecting 25 µl.
Ion chromatographic column, Dionex IonPac AS23, 250 x 4.0 mm (i.d.), or equivalent with the same selectivity.
Ion chromatographic guard cartridges, Dionex IonPac AG23, 50 x 4.0 mm (i.d.), or equivalent.
Electronic integrator or data system
Analytical Balance
Ultrasonic bath

PROCEDURE

(a) Liquid Chromatographic conditions **(typical)**

Mobile phase:	7.2 mM Na_2CO_3 + 9.0 mM $NaHCO_3$
Flow rate:	1.0 ml/min
Current of inhibitor:	70 mA
Temperature of detector cell:	35℃
Temperature of column:	30℃
Mode of injection:	Push Full
Volume of Injection:	25μl
Frequency of data sampling:	5.0 Hz
Run time:	13 min
Retention time:	9.5 min

(b) ***Equilibration of the system.*** Pump sufficient mobile phase through the column to equilibrate the system. Inject 25 μl portion of calibration solution C_A until the response obtained from two consecutive injections deviate by less than 1.5%. Then inject 25 μl portion of calibration solution C_B. The response factor for this solution should not deviate by more than 1.5% from that for calibration solution C_A, otherwise prepare new calibration solutions.

(c) ***Preparation of ethephon sample.*** Prepare sample solutions in duplicate for each sample. Heat the sample in 95 ℃ water bath until the sample becomes a transparent liquid and mix well. Before the liquid solidifies, weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 120 mg (ideally between 112 and 130 mg) of ethephon into a volumetric flask (100 ml). Add ultrapure water to the mark and mix thoroughly. Transfer 5.00 ml of the above solution into a 50 ml volumetric flask, add ultrapure water to the mark and mix thoroughly. (Sample solutions S_1 and S_2). Filter through 0.2 μm filter before use.

(d) ***Determination.*** Inject in duplicate 25 μl portions of each sample solution bracketing them by injections of the calibration solutions as follows: calibration solution C_A, sample solution S_1, sample solution S_1, calibration solution C_B, sample solution S_2, sample solution S_2, calibration solution C_A, and so on. Measure the relevant peak areas.

(e) ***Calculation.*** Determine the peak area of ethephon, calculate the mean value of response factors from the calibration solutions bracketing the injections of the sample solutions, and use this value for calculating the ethephon content of the bracketed sample. The ethephon content is the mean value of two sample solutions.

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

$$\text{Ethephon content} = \frac{f \times H_w}{w} \quad \text{g/kg}$$

where:

f_i = individual response factor
f = mean response factor of bracketing calibration injections
H_s = peak area of ethephon in the calibration solution
H_w = peak area of ethephon in the sample solution

s = mass of ethephon reference standard in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of ethephon reference standard (g/kg)

ETHEPHON TECHNICAL CONCENTRATE

373/TK/M/-

1 Sampling. Take at least 600 ml.

2. Identity test

2.1 HPIC. As for ethephon technical 373 /TC/M/2.1.

2.2 NMR. As for 373/TC/M/2.2

3 Ethephon

As for ethephon technical 373/TC/M/3, except that the clause to melt the sample is omitted in (*c)Preparation of ethephon sampl*e..

ETHEPHON SOLUBLE CONCENTRATE

373/SL/M/-

1 Sampling. Take at least 600 ml.

2. Identity test

2.1 HPIC. As for ethephon technical 373/TC/M/2.1.

2.2 NMR. As for 373/TC/M/2.2

3 Ethephon

As for ethephon technical 373/TC/M/3, except that the clause to melt the sample is omitted in *(c)Preparation of ethephon sampl*e.

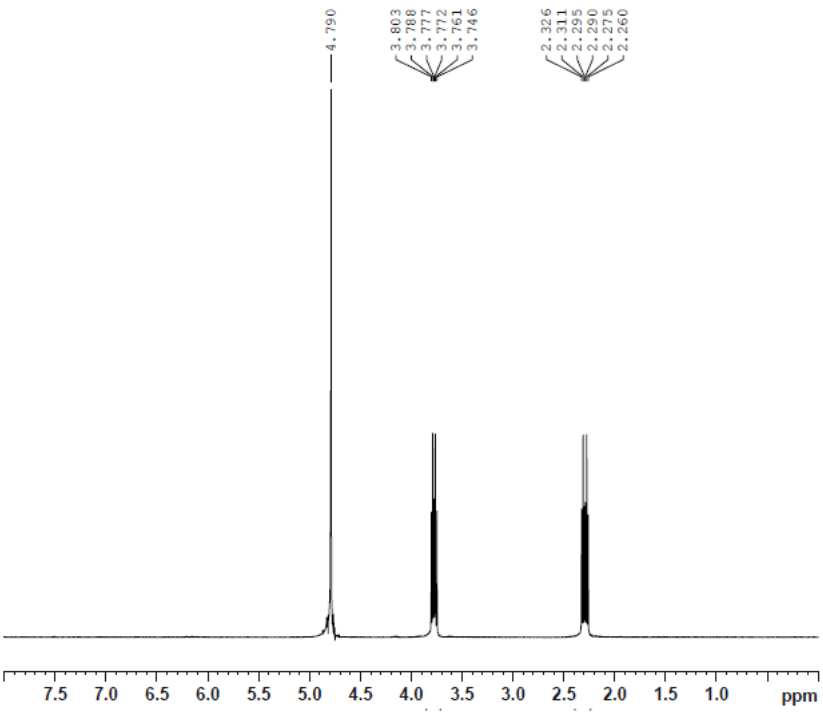


Fig.1 NMR spectrum of ethephon

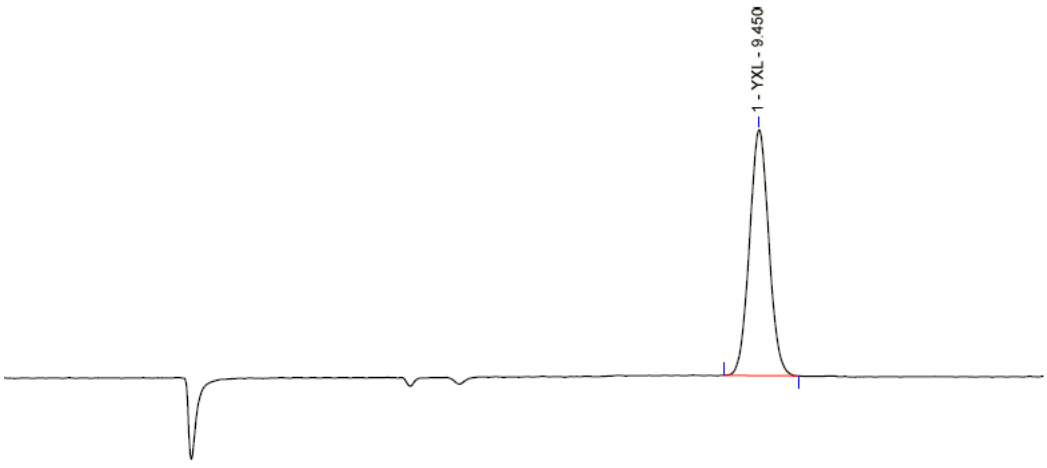


Fig.2 HPIC chromatogram of ethephon standard

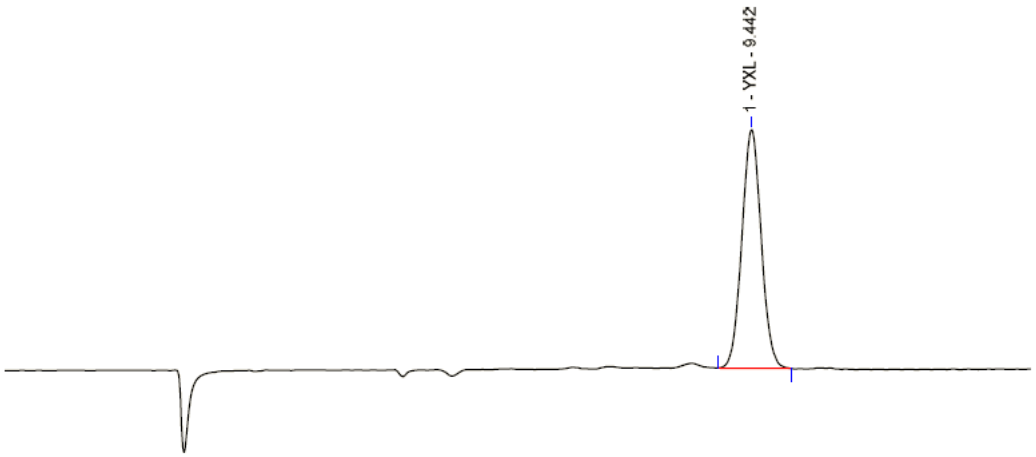


Fig.3 HPIC chromatogram of ethephon TC

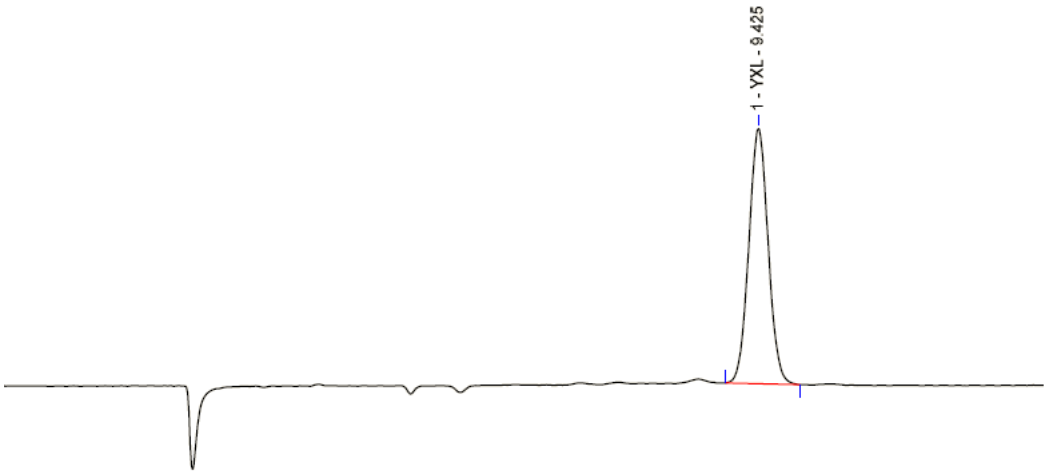


Fig.4 HPIC chromatogram of ethephon TK

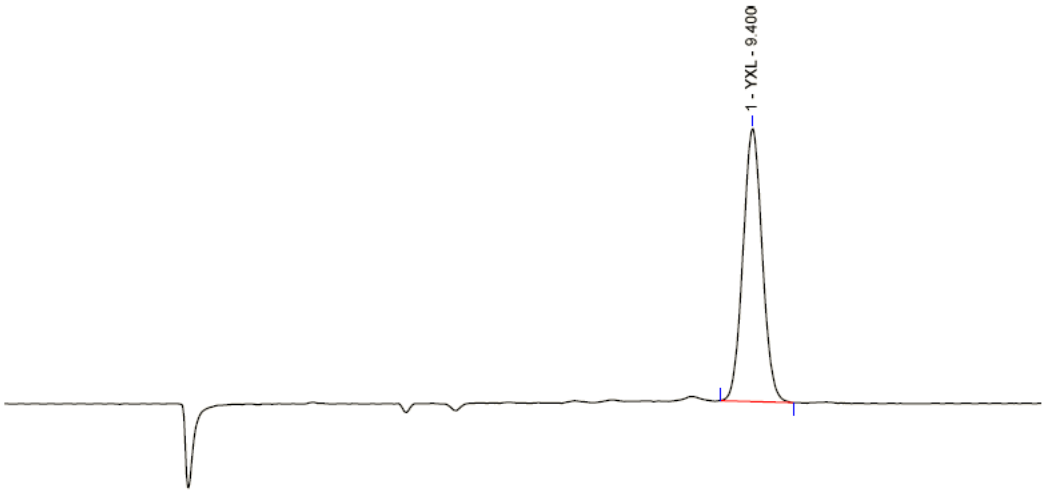


Fig.5 HPIC chromatogram of ethephon SL