Quantitative determination of ethephon in soluble concentration (SL) by Ion chromatography

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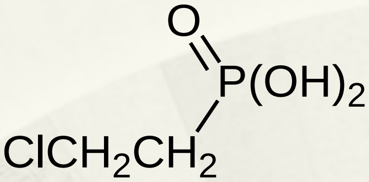
Abstract

- An ICS method for the determination of ethephon in an ethephon 40% SL has been developed and validated.
- The method was based on IC separation under the condition of gradient eluent with KOH solution by using IonPac AS11-HC analytical column and conductivity detector.
- The method validation consists of specificity, linearity, precision and accuracy.
- The result showed that the linearity ranged from 24.4- 98.8mg/L, the standard deviation was 0.19, the variation coefficient was 0.45% and the average recovery was 99.71% .

Introduction



Mol. wt. ： 144.5

Form ： colorless solid, M.p. 74-75° C

Solubility： In water 1 kg/l (23 ° C). 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 ： Stable in aqueous solutions having pH <5; at higher pH, decomposition occurs with the liberation of ethylene; DT502.4 d (pH 7, 25 ° C). Sensitive to u.v. irradiation. pKa pKa1 2.5, pKa2 7.2

- Ethephon is a broad spectrum of plant growth regulator. It is used to promote pre-harvest ripening in fruits and vegetables, to accelerate boll opening in cotton and to increase fruit setting, etc.
- The ethephon content was examined by chemistry titration in CIPAC method 373/SL/S/（2000）and Chinese national Standard GB 23554-2009. Ion chromatography was used for residue analysis of ethephon has been published. In my paper the Quantitative determination of ethephon in soluble concentration (SL) by ion chromatography will be introduced.

Analysis

REAGENTS

Water	18 MΩ.cm(25°C)；
ethephon analytical standard	100%

APPARATUS

- High Performance Ion Chromatograph System DIONEX ICS-3000 equipped with eluent generator (EG), RFIC eluent degasser, constant temperature compartment, an automated sample injector AS 3000, micro-membrane suppressor ASRS 300 4mm, Electrolytic Conductivity Detector with Chromatography workstation 6.80 Chromeleon software;
- Analytical column Pac AS11-HC 250 mm ×4 mm（i.d.）;
- Guard column： IonPac AG11-HC 50 mm×4 mm（i.d.）;
- Millipore filter： 0.45 µm.
- Analytical Balance
- Ultrasonic Bath
- Mili- Q water purification system
- Usual Laboratory apapratus

Chromatographic Condition

- Column : Pac AS11-HC 250 mm ×4 mm（i.d.） with Guard column IonPac AG11-HC 50 mm×4 mm（i.d.）
- Injection volume： 5 μL loop，25 μL；
- Flow Rate: 1.0 ml/min
- Retention time： 9.9 min。
- The typical ion chromatograms of ethephon standard and Ethephon 40%SL are as follows（figure 1、2）.

- Eluent：

	Retention (min)	Concentration of KOH (mol/L)
1	0	15
2	5	15
3	12	33
4	12.1	40
5	15	40
6	15.1	15
7	19	15

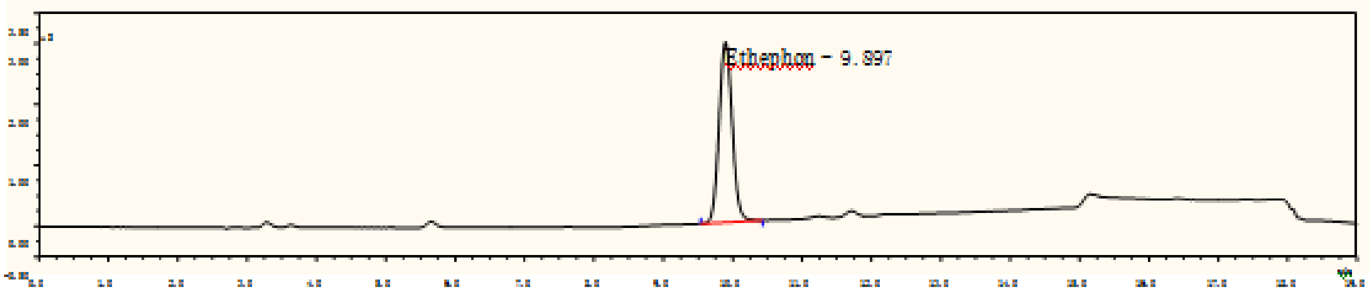


Figure 1 ion chromatogram of ethephon standard

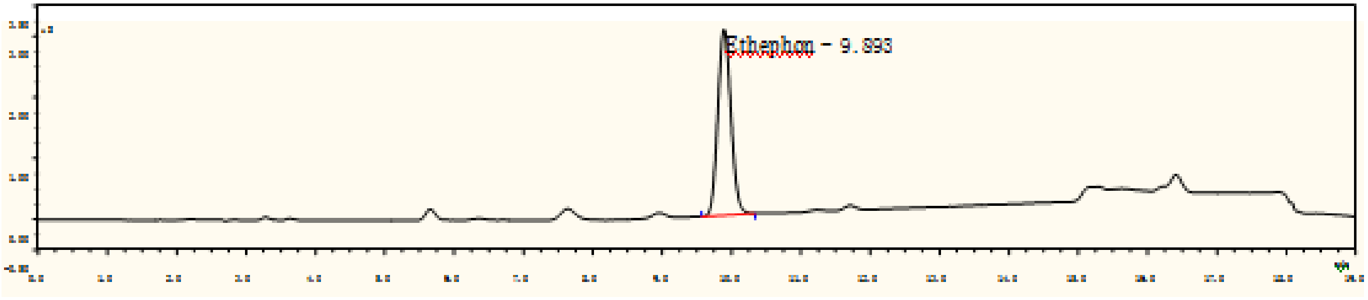


Figure 2 ion chromatogram of Ethephon 40% SL

Preparation of Calibration solution

0.05g of ethephon analytical standard was weighed accurately (0.01mg) into a 100ml volumetric flask, then 90mL water was added. The flask was placed in ultrasonic bath for 5 minutes to dissolve. The solution was equilibrated to room temperature and the flask was filled right up the mark with water and mixed well. Pipette 10mL of the solution into a 100mL flask. Dilute to volume with water and mix well. Filter with 0.45μm filter membrane.

Preparation of sample solution

Weight 0.12g (nearest 0.01mg) ethephon 40% soluble concentration into 100ml volumetric flask. Dissolved in water by shaking and make up to volume with water. Pipette 10mL of the solution into a 100mL flask. Dilute to volume with water and mix well. Filter with 0.45µm filter membrane.

Determination

- Make injections of calibration solutions until the system is stable (the relative variation of the response value of adjacent injections not varying by more than 1.5%).
- Then each measured solution is injected in the order of calibration solution, sample solution, sample solution, calibration solution.

Calculation

- The content of ethephon is determined in accordance with the following equation:

$$\%\ of\ ethephon = \frac{A_2 \times m_1 \times p}{A_1 \times m_2}$$

- where：
- A_1—— the mean peak area of ethephon in the calibration solution
- A_2—— the mean peak area of ethephon in the sample solution
- m_1—— mass of ethephon in the calibration solution (g)
- m_2—— mass of sample in sample solution (g)
- —— purity of ethephon standard (%)

Specificity

In ion chromatography, because the end-use products usually consist of co-formulant ext , some Common anions may interfere with the target ion peaks. To ensure a good separation, a solution which consists of Cl, F^- , SO_4^{2-} , PO_4^{3-} , $ClO2^-$, $ClO3^-$, Br^- , $BrO3^-$,$NO2^-$, $NO3^-$ was injected. The results showed that no interference between 6.7 min to 10.6 min where the peak of ethephon stayed.(9.9mim)

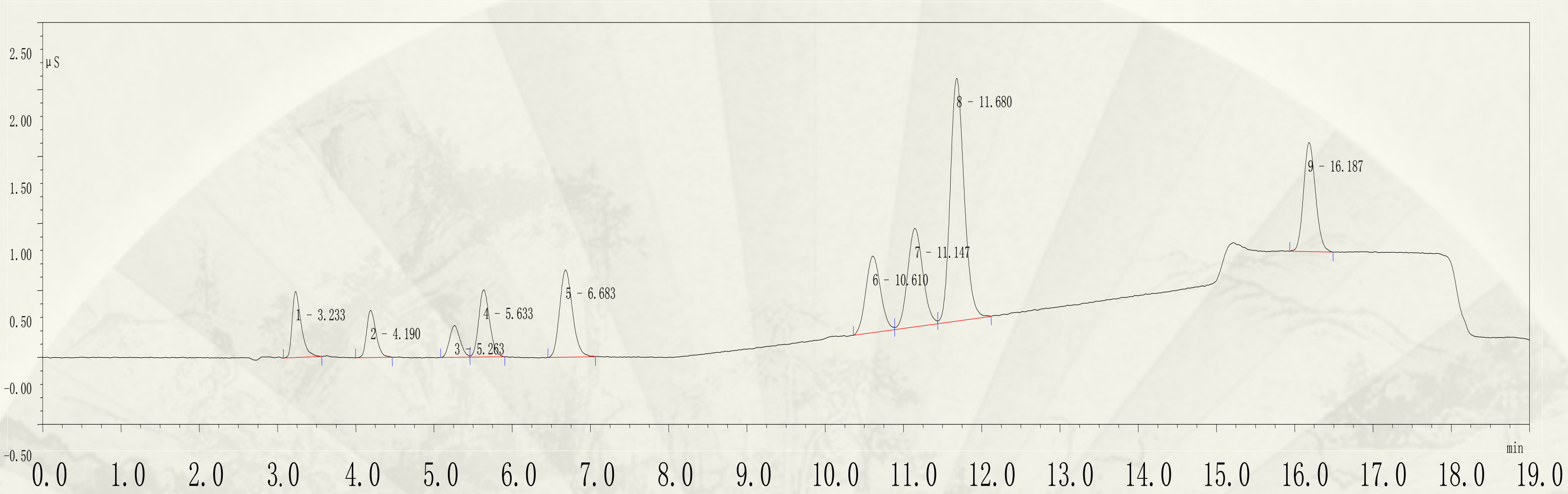


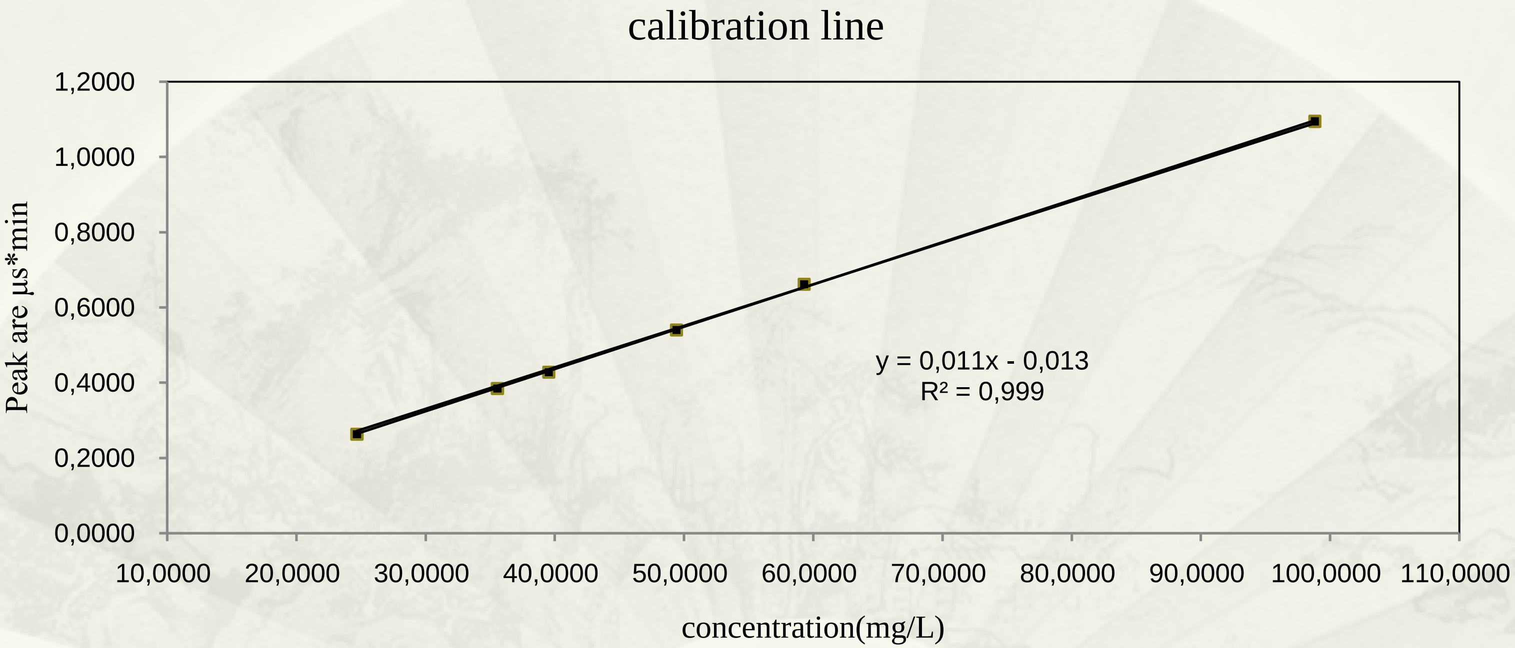
Figure 3 The ion chromatogram of the mixture of the ions

Linearity

Linearity was demonstrated through the analysis of replicate standards at six levels ranging from 25 to 98 mg/L. Make duplicate injections for each concentration, then calculated the mean value. Calibration data and corresponding calibration curve were as follows:

number	1	2	3	4	5	6
ncentration (mg/L)	24.7000	35.5680	39.5200	49.4000	59.2800	98.8000
Peak area µ S*min)	0.2640	0.3855	0.42850	0.5410	0.6625	1.0945

Linearity


calibration line
1,2000
1,0000
0,8000
0,6000
0,4000
0,2000
0,0000
Peak are µs*min
y = 0,011x - 0,013
R² = 0,999
10,0000
20,0000
30,0000
40,0000
50,0000
60,0000
70,0000
80,0000
90,0000
100,0000
110,0000
concentration(mg/L)

Precision

Table 2 The results of the precision

Preparation No.	ethphon ， %(m/m)
1	41.94
2	41.97
3	42.23
4	41.79
5	42.22
$\bar{x}$	42.03
S	0.19
RSD	0.45
Modified Horwitz Limit	1.54

Accuracy (% recovery)

Table3 the results of the accuracy

mber	Theoretical value(mg)	Actual value(mg)	Recovery(%)	Average recovery(%)
1	1.13545	1.13530	99.99	99.71
2	1.04273	1.03775	99.52	
3	1.03441	1.02535	99.12	
4	1.15416	1.15680	100.23	
5	1.04261	1.04040	99.79	
6	1.04454	1.04050	99.61	

Conclusions

The results showed that the method has a high precision, accuracy and a good linearity within wide range of concentration. Comparing to chemistry titration the main advantage of this method is that it can be used to identify the active ingredient by retention time. In addition, the method is simple, fast and effective.

Thank you!