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GC-MS Application Data Sheet No. 001 (Environment)

Analysis of Acephate in Water Using GC-MS

Acephate (Fig.1) is an organophosphorus insecticide developed by Chevron Chemical in the United States. It has strong permeability into crops, and has been used mainly for pest control of vegetables. Acephate penetrates into plants from their roots, and its effects extend throughout the plants. Acephate is converted into methamidophos through metabolism of plants and animals, or in the environment. However, even after changing into methamidophos, its insecticidal function remains. In other words, it is a persistent agricultural chemical with aftereffects.

Acephate and methamidophos are both highly soluble in water. This means that when analyzing these agricultural chemicals in water, the styrene-diphenyl copolymer column used for solid phase extraction of residual agricultural chemicals in water can only achieve low recovery. For this reason, activated carbon columns are used for the analysis of these substances. In this report, extraction was conducted using porous diatomaceous earth, as shown in Fig.2. This enabled a recovery close to 100%.

As acephate and methamidophos are both highly polar substances, in GC/MS analysis, their chromatogram peaks tend to show excessive tailing and they are subject to matrix effects at the injection port. To solve these problems, the injection port of the GCMS-QP2010 is designed to avoid uneven temperature distribution due to the column oven. In addition, availability of three regression modes of calibration curve -- linear, first order and second order -- allows quantitative processing to be performed according to the characteristics of the agricultural chemicals (Fig. 4 & 5). The lower limit of determination (10σ) and lower limit of detection (3σ) were 0.323µg/mL and 0.097µg/mL respectively

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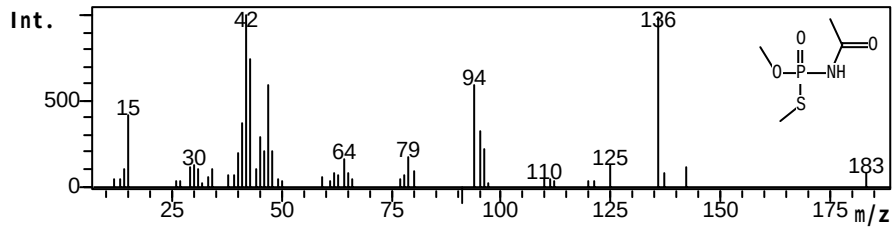


Fig. 1 Mass Spectrum of Acephate

CAS Number: 30560-19-1, Molecular weight: 183.2, Molecular formula: $C_4H_{10}NO_3PS$, Compound name: Acephate Phosphoramidothioic acid, acetyl-, O,S-dimethyl ester; Acetamidophos O,S-Dimethyl acetylphosphoramidothioate

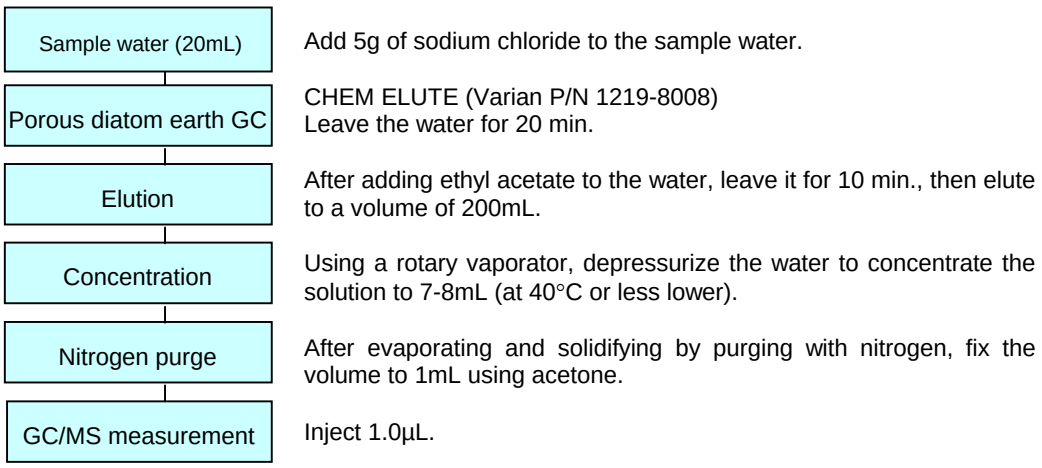


Fig.2 Flow Diagram of Sample Pretreatment

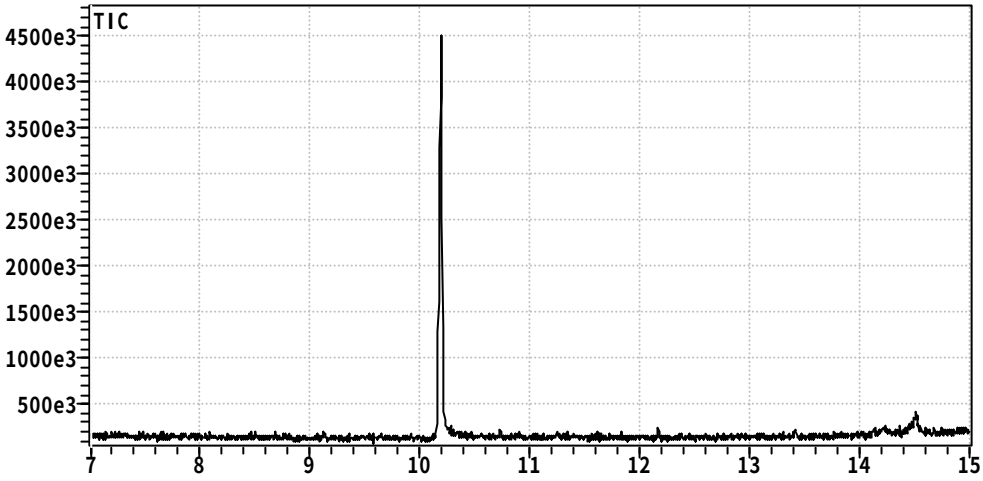
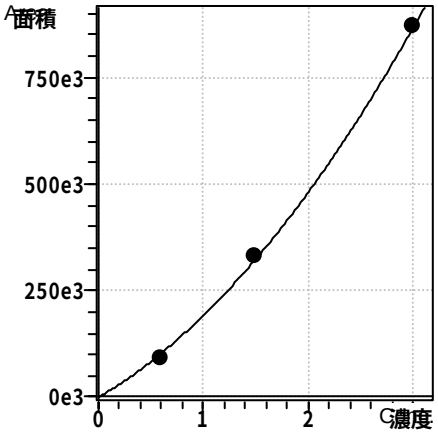


Fig. 3 Total Ion Chromatogram



Conc. (μg/mL)	Area
0.6	92,898
1.5	332,517
3.0	869,466

Fig. 4 Calibration Curve of Total Ion Chromatogram

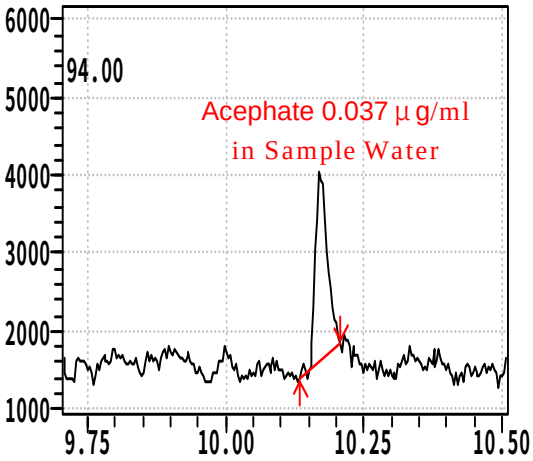


Fig.5 SIM Chromatogram of Real Sample

Table 1 Analytical conditions for GC-MS

Equipment	: **AOC-20i+GCMS-QP2010**
Column	: DB-5 (30m x 0.25mm i.d. df=0.25μm)
Column Time program	: 50°C (2min)-15°C/min-250°C(3min)
Carrier Gas	: He 100kPa
Injection Temp.	: 250°C
Injection Method	: Splitless (Sampling Time 2min)
Injection Volume	: 1μL
Interface Temp.	: 280°C
Ionsource Temp.	: 200°C
Scan	: m/z 40-500 (interval 0.5sec)
SIM	: 94,136(interval 0.2sec)

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