

Analysis of Pesticides by UHPLC/SQ MS

Environmental



LC/MS provides an excellent alternative to conventional LC with UV, PDA or Fluorescence detection or GC/MS to measure polar pesticides in food matrices and in the environment. Many of these pesticides are not very volatile and have to be derivatized prior to detection. Also, many of these pesticides are labile compounds and may get degraded easily in GC. We present a UHPLC/SQ MS separation and detection method for a mixture of organophosphorus pesticides, carbamates, triazines and chlorinated pesticides.

Experimental Conditions

Target Analytes: Acephate, oxamyl, mevinphos, pyrazon, dimethoate, aldicarb, simazine, dichlorvos, prometon, carbofuran, atrazine, chlorotoluron, carbaryl, prometryn, methiocarb, azinophos methyl, malathion, diazinon, ethion

Sample Preparation Conditions

A mixture of 20 pesticides (organophosphorous pesticides, carbamates, chlorinated pesticides and triazines) at a concentration of 1 mg/mL each in acetonitrile was obtained from SPEX Certiprep group (Metuchen, NJ). The stock solution was diluted to 10 µg/mL in acetonitrile and injected on column.

Liquid Chromatography Conditions

Pump Type: PerkinElmer® Flexar™ FX-15
 Column: PerkinElmer Brownlee™ Supra™ Aqueous C18 column (2.1 mm x 50 mm, 1.9 µm)
 Mobile Phase: A: water containing 0.1% formic acid
 B: acetonitrile containing 0.1% formic acid
 Flow Rate: 0.4 mL/min
 Injection Volume: 2 µL in partial fill mode

Gradient:	Time (min)	%A	%B
	0	90	10
	3	80	20
	3.5	70	30
	5.4	35	65
	5	10	90

Mass Spectrometer Conditions

Ionization: Ultraspray™ ESI – Positive mode
 Scan Range: 60-410 m/z
 Scan Rate: 1000 u/sec
 Capillary Exit Voltage: 50 V

Results

The separation of the 20 pesticides was accomplished within 13 min (Figure 1). Extracted ion chromatograms allowed good selectivity and accurate quantitation even at low concentration levels (Figure 2).

The soft ionization provided by the Flexar SQ 300 MS combined with the low capillary exit voltage values used, allowed for excellent sensitivity while at the same time obtaining molecular ion information (Figures 3 and 4).

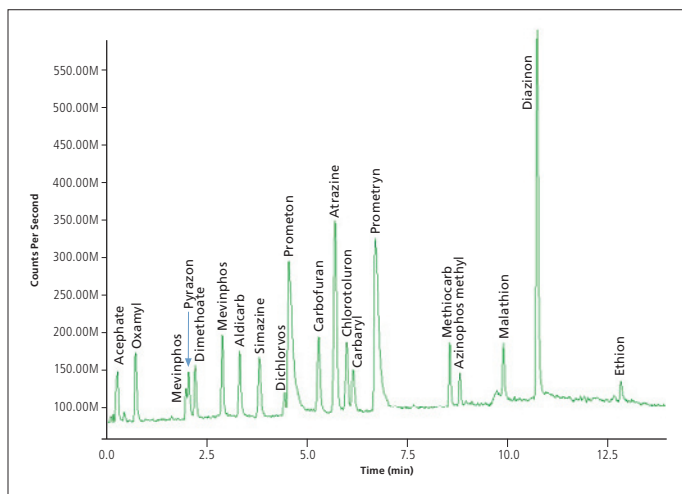


Figure 1. TIC of pesticides by UHPLC/SQ MS.

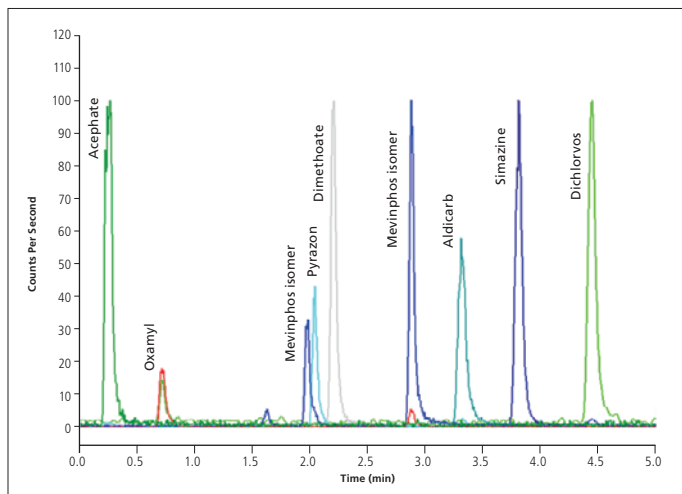


Figure 2. Extracted ion chromatogram (EIC) from 0-5 min.

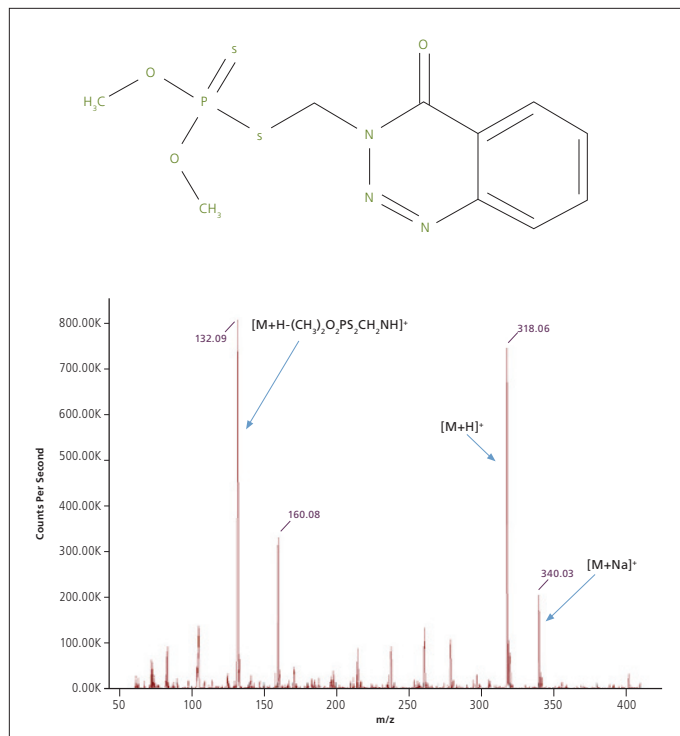


Figure 3. Molecular structure and mass spectrum of azinophos methyl.

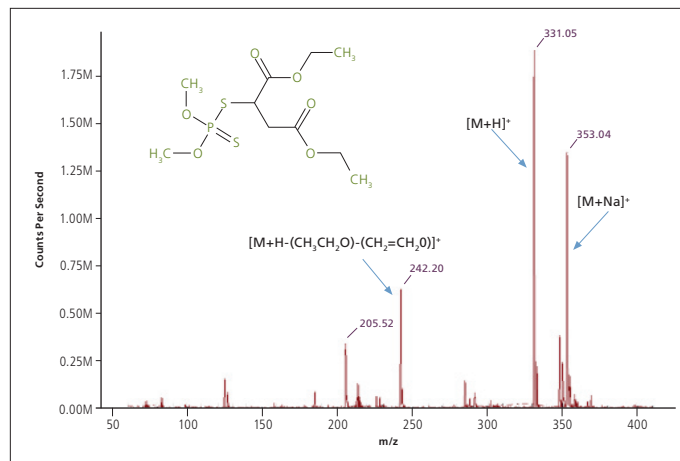


Figure 4. Mass spectrum of malathion.