# The Analysis of Organophosphate Pesticides by LC/MS

Application

LC-MS

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## Abstract

**Organophosphate pesticides were readily analyzed using liquid chromatography-mass spectrometry with electrospray ion source. Sensitivity and selectivity were significantly better than using a diode-array UV detector.**

## Overview

Liquid chromatography-mass spectrometry (LC-MS) is rapidly becoming a routine technique for efficient trace analysis of polar pesticides in various types of samples. In comparison to existing methodologies, such as gas chromatography-mass spectrometry (GC-MS) and ultraviolet (UV) detection, LC-MS considerably simplifies cleanup procedures, reducing both time of analysis and method development time.[1]

LC-MS with an electrospray ion (ESI) source avoids the thermal degradation of labile pesticides encountered with GC and eliminates the need for preliminary derivatization to increase compound volatility.

Additionally, LC-MS provides unequivocal identification of each pesticide, even if the pesticide was not completely resolved from neighboring eluants. Traditional UV detection cannot provide the required specificity because many of the pesticides within the same class exhibit similar UV spectra.

## Sample case

A mixture of organophosphate pesticides and an internal standard were analyzed using an Agilent 1100 LC/MS with an ESI source (Table 1).

**Table 1. Mixture of Organophosphate Pesticides**

| Elution order | Compound | $[M+H]^+$ | Concentration µg/mL |
|---|---|---|---|
| 1 | Mevinphos isomer 1 | 225 | 0.2 |
| 2 | Dimethoate | 230 | 0.5 |
| 3 | Mevinphos isomer 2 | 225 | 0.5 |
| 4 | Dichlorvos | 221 | 0.5 |
| 5 | Azinphos methyl | 318 | 0.05 |
| 6 | Parathion methyl | 264 | 0.2 |
| 7 | Malathion | 331 | 0.5 |
| 8 | Diazinon | 305 | 0.2 |
| 9 | Triphenyl orthophosphate* | 327 | 1.0 |
| 10 | Parathion ethyl | 292 | 0.1 |
| 11 | Phorate | 261 | 0.1 |
| 12 | Reldan | 322 | 0.5 |
| 13 | Ronnel | 321 | 0.1 |
| 14 | Terbuphos | 289 | 0.2 |
| 15 | Dursban | 350 | 0.1 |
| 16 | Ethion | 385 | 0.2 |
| 17 | Temephos | 467 | 0.1 |

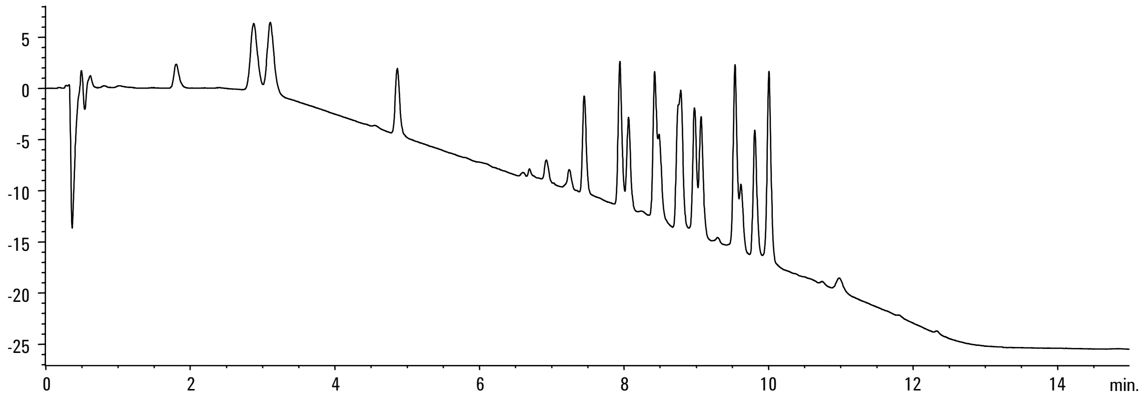
* Internal standard

## Method summary

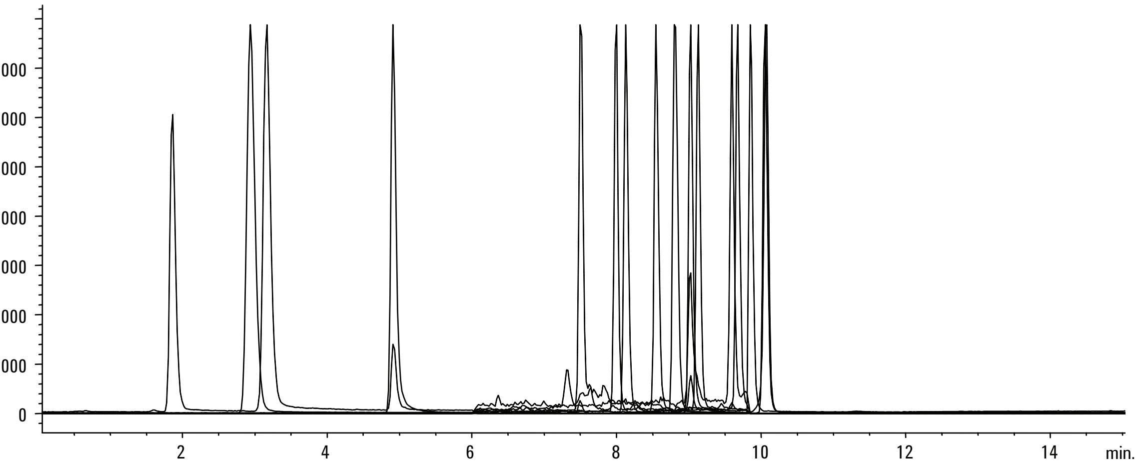
- Column 2.1 mm id × 5 cm long, filled with 3.5 µm particles, C18 chemistry
- 20 mM ammonium acetate vs. acetonitrile mobile phase gradient
  - 5 % to 95 % acetonitrile in 4 minutes
  - Hold 2 minutes
- Splitless 400 µL/min flow
- 3 µL injection volume
- Scan data 120 to 600 m/z
- SIM data as per Table 1. 95 msec dwell/ion in two groups

## Results

Simultaneous UV (220 nm) and MS detector outputs are compared in Figure 1. The MS plot is a composite of all the individual extracted ion chromatograms. Each was obtained at the $[M+H]^+$ value given in Table 1, and are separated and stacked in Figure 2 for easy comparison.



Normalized UV Plot



MS selected ion mode (SIM), overlayed and normalized

**Figure 1. Comparison of UV and MS chromatograms.**

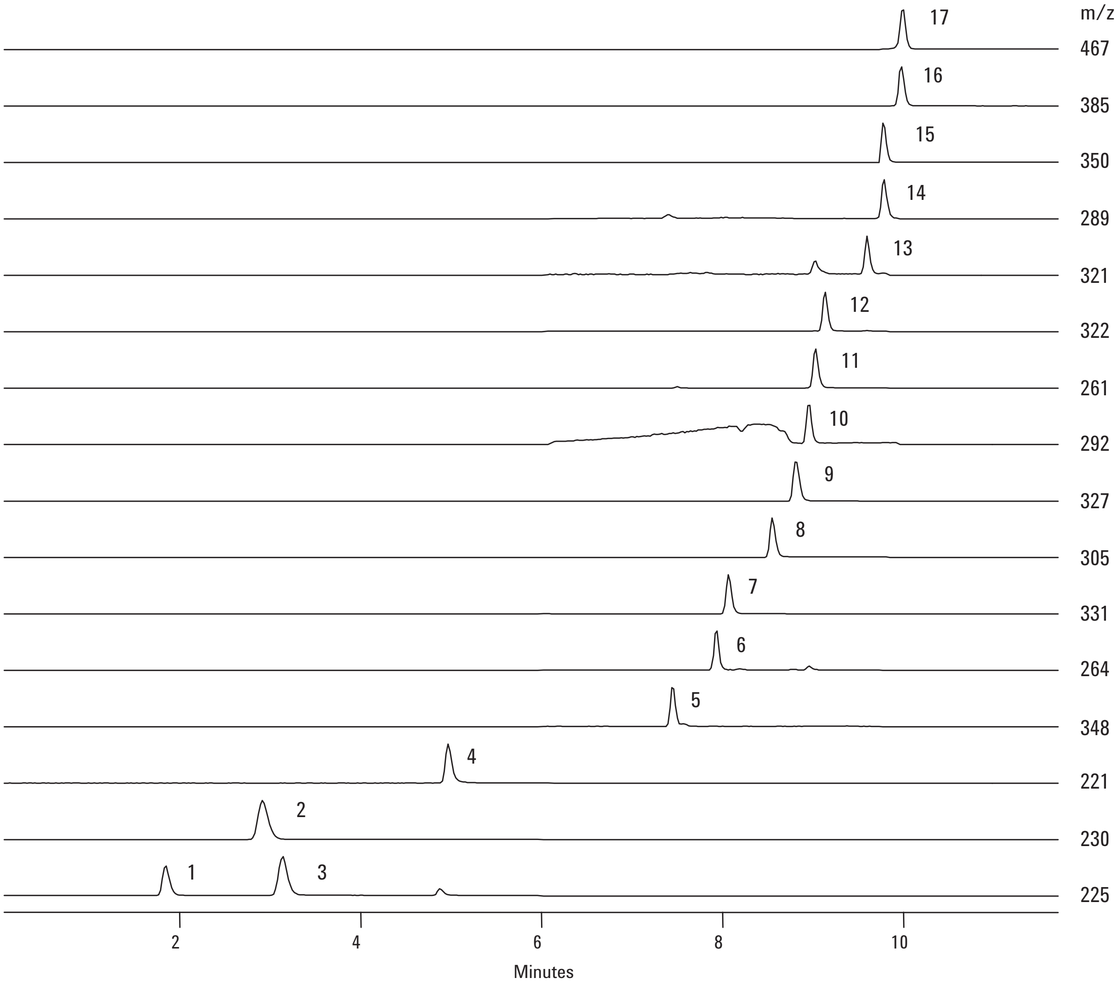


**Figure 2. Stacked normalized extracted ion chromatograms for compounds 1 through 17.**

Figures 3 through 6 show the resulting normalized mass ion spectra for each compound included in Table 1.

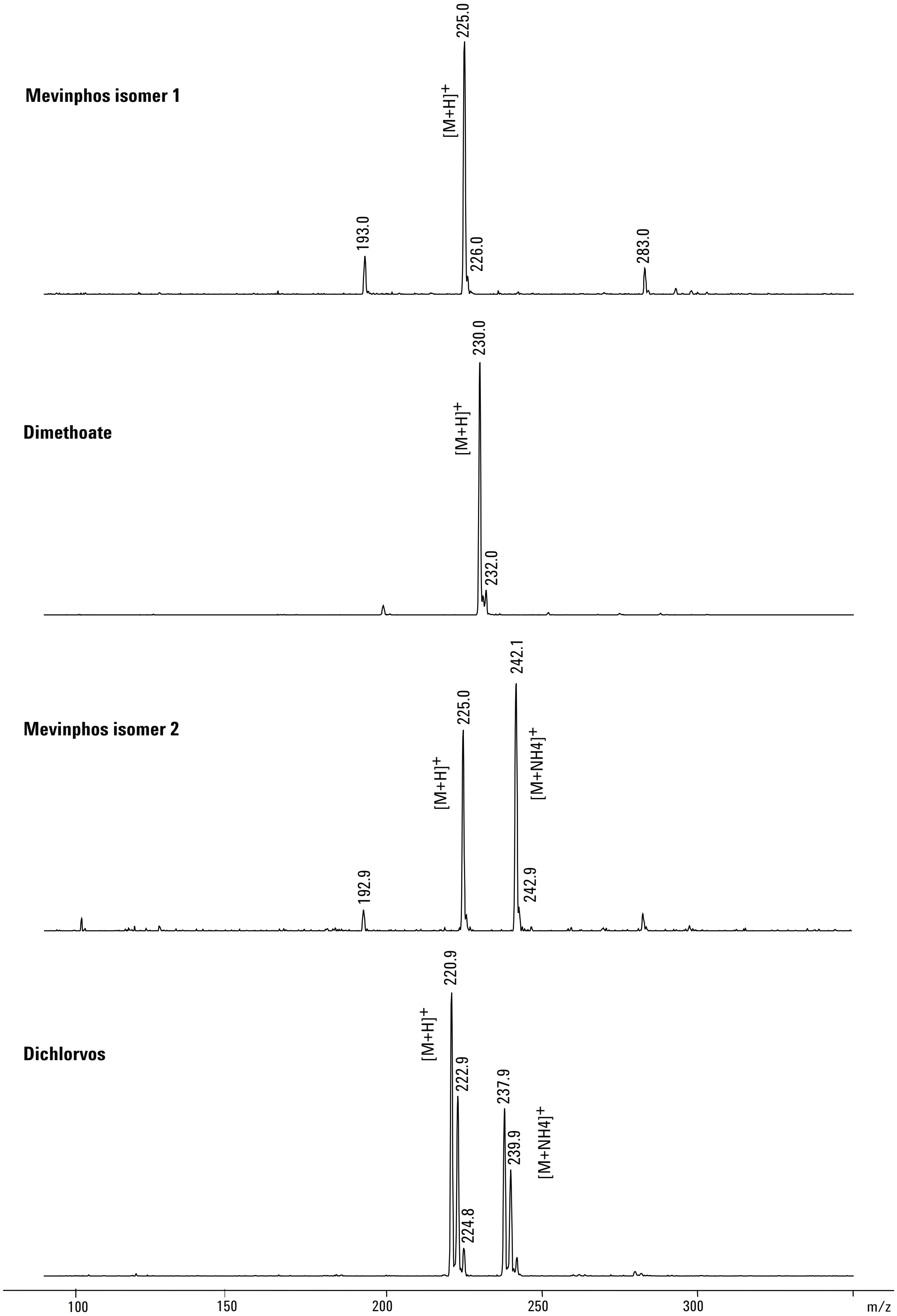


Figure 3. Stacked normalized ion mass spectra for compounds 1 through 4.

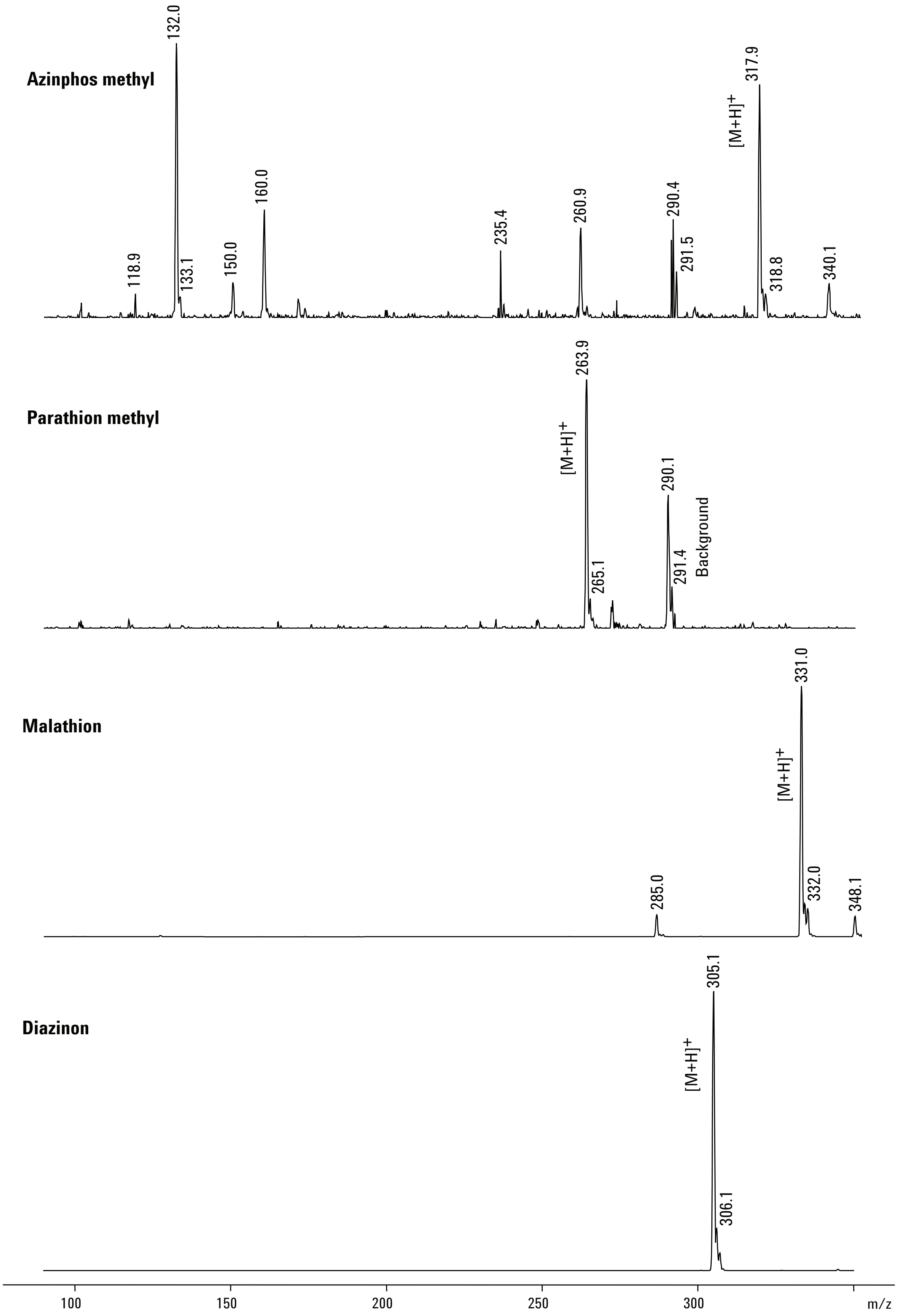


Figure 4. Stacked normalized ion mass spectra for compounds 5 through 8.

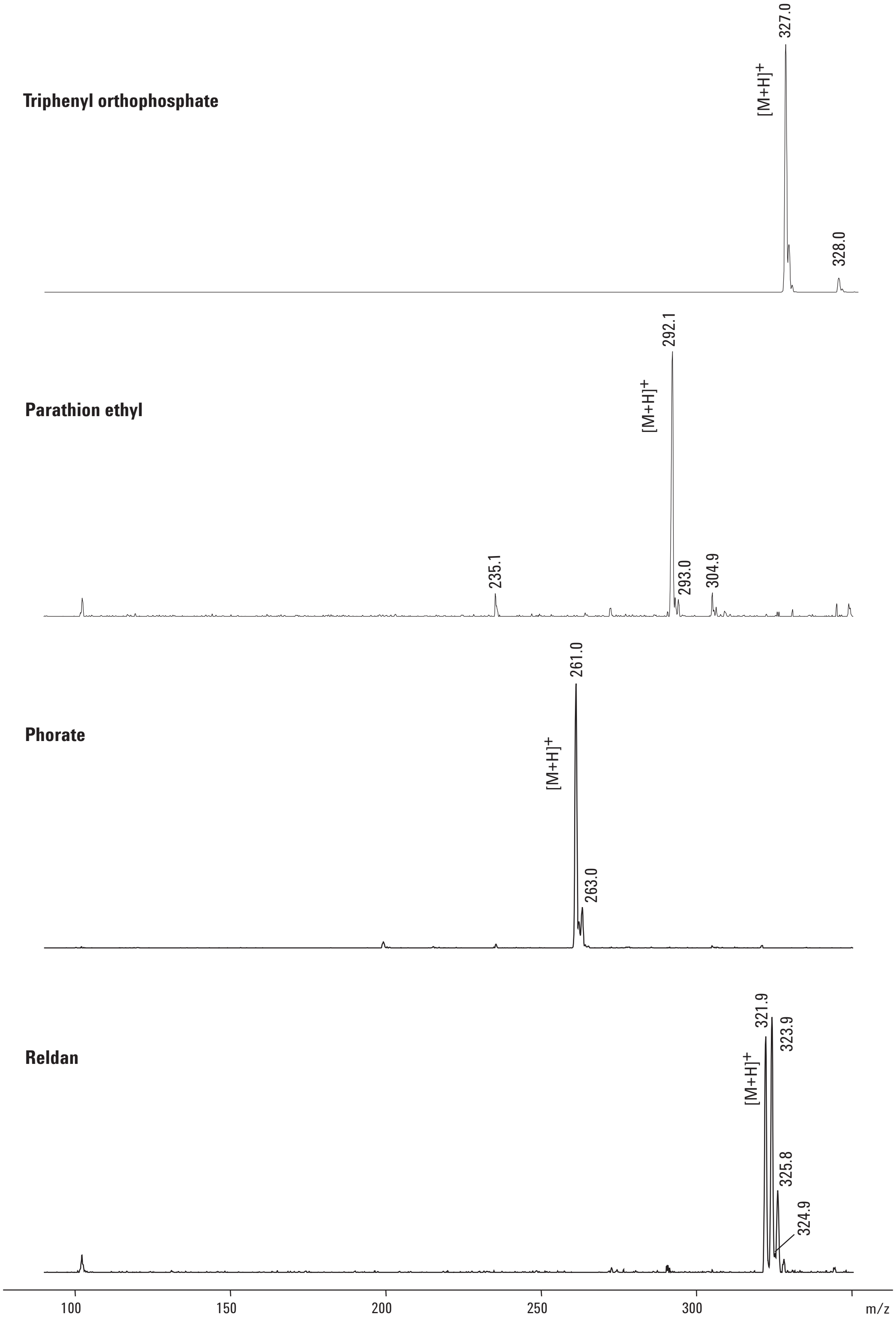


**Figure 5. Stacked normalized ion mass spectra for compounds 9 through 12.**

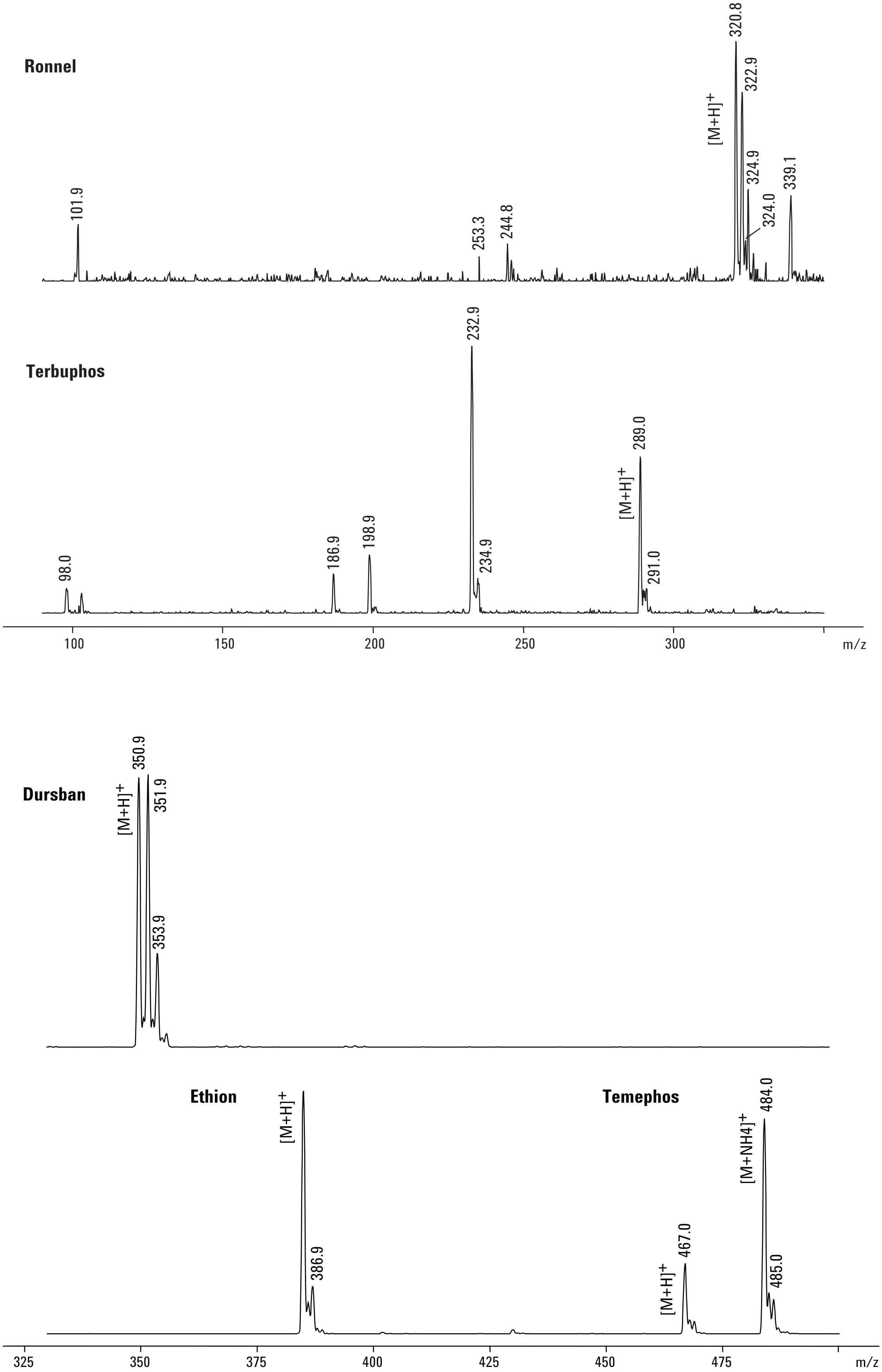


Figure 6. Stacked normalized ion mass spectra for compounds 13 through 17.

## Conclusions

When determining organophosphate pesticides using LC-MS with an ESI source:

- All the tested organophosphate pesticides ionized well and gave definite $[M+H]^+$ ions
- Sensitivity and selectivity are significantly better than using diode-array UV detector
- Overall chromatography and analysis is simple and straightforward
- Positive identification and quantification are performed using integrated software

For more information, contact your local Agilent sales representative, or visit www.agilent.com.

## Reference

1. Elbert Hogendoorn and Peit van Zoonen, *Journal of Chromatography A*, 892 (2000) 435-453.

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