Application Note: 434

Analysis of Triazine Pesticides in Drinking Water Using LC-MS/MS (EPA Method 536.0)

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Key Words

- Drinking Water Analysis
- Herbicides
- Hypersil GOLD Columns
- Triazines
- TSQ Quantum Access

Introduction

The US EPA has recently issued a draft form of a proposed method for the analysis of triazine compounds in drinking water.[1] This method uses a simple method to directly analyze triazine compounds using LC-MS/MS without requiring any solid phase extraction (SPE) or other lengthy sample preparation steps. This application note demonstrates the analysis of these compounds over the concentration range 0.25 – 5.0 ng/mL (ppb) using the Thermo Scientific TSQ Quantum Access™ triple stage quadrupole mass spectrometer and the Thermo Scientific Accela™ HPLC system.

Experimental Conditions

The following triazine and triazine degradates were analyzed: Atrazine, Atrazine-desethyl, Atrazine-desisopropyl, Cyanazine, Propazine, and Simazine, purchased from Sigma-Aldrich, St. Louis, MO, and Ultra Scientific, North Kingstown, RI. The following internal standards were used: Atrazine-d_5, Atrazine-desethyl-d_7, Atrazine-desisopropyl-d_5, Cyanazine-d_5, Propazine-d_{14}, and Simazine-d_{10}, purchased from C/D/N Isotopes, Inc., Pointe-Claire, Quebec, Canada. Standards and internal standard stocks were prepared in solutions of methanol and diluted to their appropriate concentrations prior to analysis.

Sample Preparation

While no SPE was required for this method, samples were treated as per the EPA's draft method. The method calls for the addition of ammonium acetate at 20 mM for pH adjustment and dechlorination and sodium omadine at 64 mg/L to prevent microbial degradation, both purchased from Sigma-Aldrich, St. Louis, MO. All samples were prepared in reagent water. All samples were spiked with the internal standard solution, resulting in a final concentration of 5 ng/mL (ppb) for each internal standard. Calibration standards were prepared at the following levels: 0.25, 0.5, 1, 2, 2.5 and 5 ng/mL.

HPLC Conditions

Column:	Thermo Scientific Hypersil GOLD™ 100 x 2.1 mm, 3 µm		
Solvent A:	5 mM Ammonium Acetate		
Solvent B:	Methanol		
Flow Rate:	400 µL/min		
Injection Volume:	100 µL		
HPLC Gradient:	Time	%A	%B
	0:00	98	2
	10:00	98	2
	20:00	10	90
	25:00	10	90
	25:06	98	2
	30:00	98	2

Mass Spectrometer Conditions

Ionization Source:	Positive Electrospray
Sheath Gas:	30 arbitrary units
Auxiliary Gas:	10 arbitrary units
ESI Voltage:	3.5 kV
Ion Transfer Tube Temperature:	350 °C
Collision Gas:	1.5 mTorr
Q1/Q3 Peak Resolution:	0.7 Da
Scan Width:	0.01 Da

MS Parameters

Compound	Precursor Mass	Product Mass	Collision Energy	Tube Lens
Atrazine-desisopropyl	174	132	17	90
Atrazine-desethyl	188	146	16	95
Simazine	202	124	17	80
Atrazine	216	174	16	85
Propazine	230	124	17	80
Cyanazine	241	214	15	100
Atrazine-desisopropyl-d_5	179	137	17	85
Atrazine-desethyl-d_7	195	147	17	95
Simazine-d_{10}	212	137	19	95
Atrazine-d_5	221	179	17	95
Propazine-d_{14}	244	196	18	95
Cyanazine-d_5	246	219	16	100

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Results and Discussion

The triazine compounds eluted from the LC column in 20 minutes. A chromatogram of each compound and the internal standards is shown in Figure 1. All peaks are chromatographically resolved from one another. Calibration curves were generated for each compound over the range 0.25-5 ppb. All calibration curves exhibited excellent linearity, ranging from 0.9964 for Atrazine-desethyl to 0.9982 for Atrazine. The calibration curve for Simazine is shown in Figure 2. The other compounds exhibit similar linearity, and are not shown in this application note.

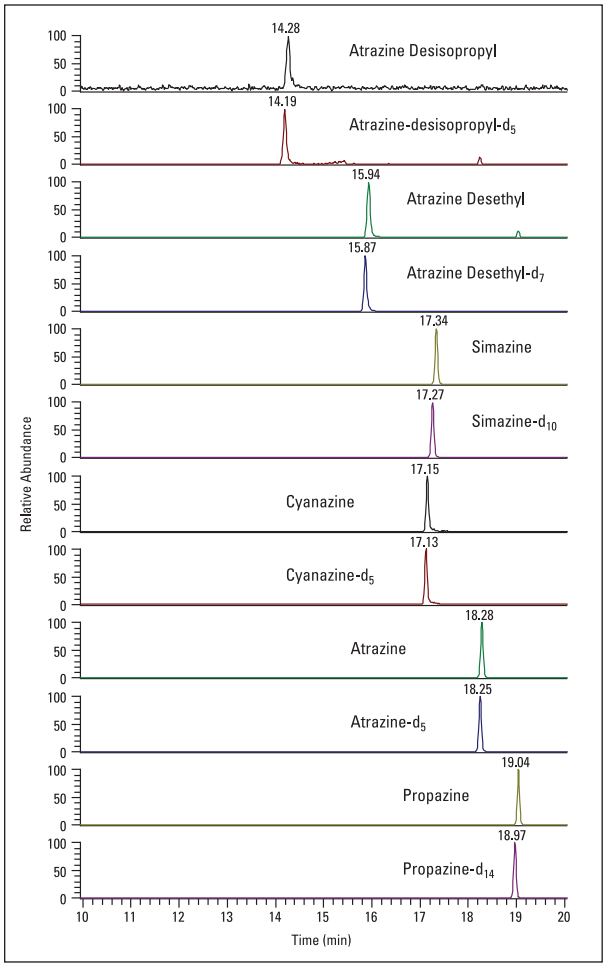

Figure 1: Chromatogram of the triazine compounds at 2 ppb, and their internal standards

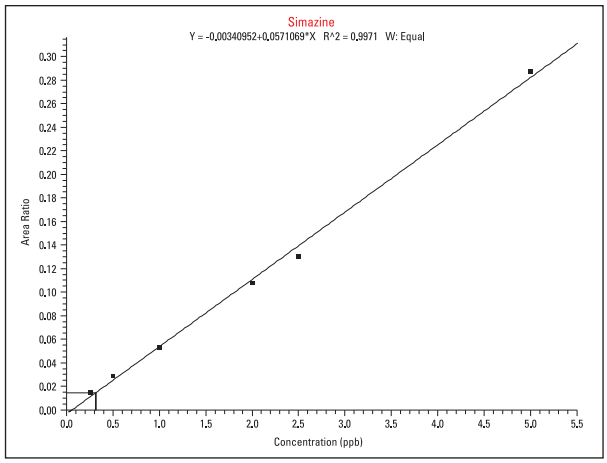

Figure 2: Calibration curve for Simazine, 0.25-5 ppb

Conclusion

The TSQ Quantum Access LC-MS/MS is an excellent choice for the analysis of triazine compounds and their degradates. Linearity over the entire calibration range of 0.25 to 5 ppb is observed. Separation of all the analytes is achieved with the Hypersil GOLD column allowing for unambiguous identification and quantitation of all of the compounds in this application note.

References

1. Smith, G.A., Pepich, B.V., Munch, D.J. "Determination of Triazine Pesticides and their Degradates in Drinking Water by Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI/MS)" Draft 5.0, April 2007.

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