응용 자료

EPA Method 535.0 Measurement of Chloracetanilide and Other Acetamide Herbicide Degradates in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS)

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief demonstrates the measurement of chloracetanilide and other acetamide herbicide degradates in drinking water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS).

Introduction

Chloroacetanillide and acetamide herbicides are widely used in the United States for the control of broadleaf and annual weeds on crops, such as corn and soybeans. These herbicides are thought to be degraded by microbial action to the oxanilic acid (OA) and ethanesulfonic acid (ESA) metabolites of the parent compounds. The OA and ESA metabolites are more water soluble and therefore more mobile than the parent herbicides. Several studies by the United States Geological Survey (USGS) and others have reported higher occurrences of the OA and ESA metabolites than the parent herbicides in both ground and surface water. The United States Environmental Protection Agency (US EPA) Method 535 was developed for the determination of OA and ESA metabolites of alachlor, acetochlor, metochlor, propachlor, flufenacet, and dimetheamide in drinking water. The European Union (EU) regulation regarding drinking water (EC Directive 98/83/EC), provides a general rule for pesticides and metabolites. This regulation limits the maximum admissible concentration (MAC) at 0.1 ppb for each individual component, with the total concentration not to exceed 0.5 ppb.

Experimental

LC Conditions

Instrument:	Waters ACQUITY UPLC System

Eluent: 5 mM aqeous ammomium acetate/methanol

Column: ACQUITY UPLC BEH C_{18}, 2.1 x 150 mm, 1.7 µm @ 60 °C

Injection: 50 µL

Flow rate: 0.45 mL/min

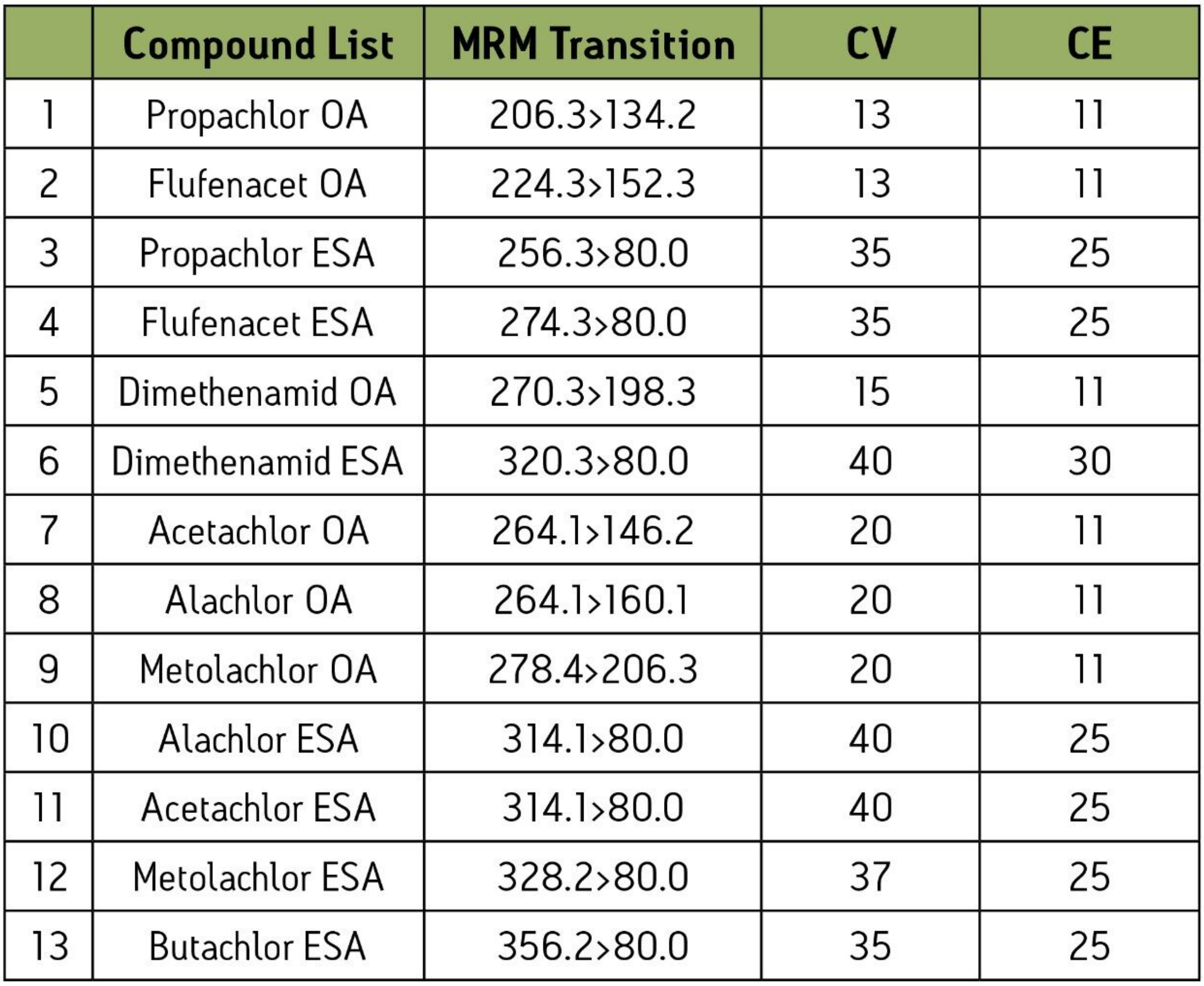
Data: Waters MassLynx Software

MS Conditions

Instrument: Waters TQ Detector or Quattro micro API Mass Spectrometer

Ion mode: Electrospray negative (ESI^-)

Mode: Multiple reaction monitoring (MRM). The MRM transitions, cone voltages (CV) and collision energies (CE) are listed in in the top right column.



	Compound List	MRM Transition	CV	CE
1	Propachlor OA	206.3>134.2	13	11
2	Flufenacet OA	224.3>152.3	13	11
3	Propachlor ESA	256.3>80.0	35	25
4	Flufenacet ESA	274.3>80.0	35	25
5	Dimethenamid OA	270.3>198.3	15	11
6	Dimethenamid ESA	320.3>80.0	40	30
7	Acetachlor OA	264.1>146.2	20	11
8	Alachlor OA	264.1>160.1	20	11
9	Metolachlor OA	278.4>206.3	20	11
10	Alachlor ESA	314.1>80.0	40	25
11	Acetachlor ESA	314.1>80.0	40	25
12	Metolachlor ESA	328.2>80.0	37	25
13	Butachlor ESA	356.2>80.0	35	25

Optimized MRM transition parameters for EPA method 535 in ES-.

Sample Preparation

A 250 mL water sample is extracted using a graphitized carbon SPE cartridge, dried and made up to 1 mL with 5 mM ammonium acetate.

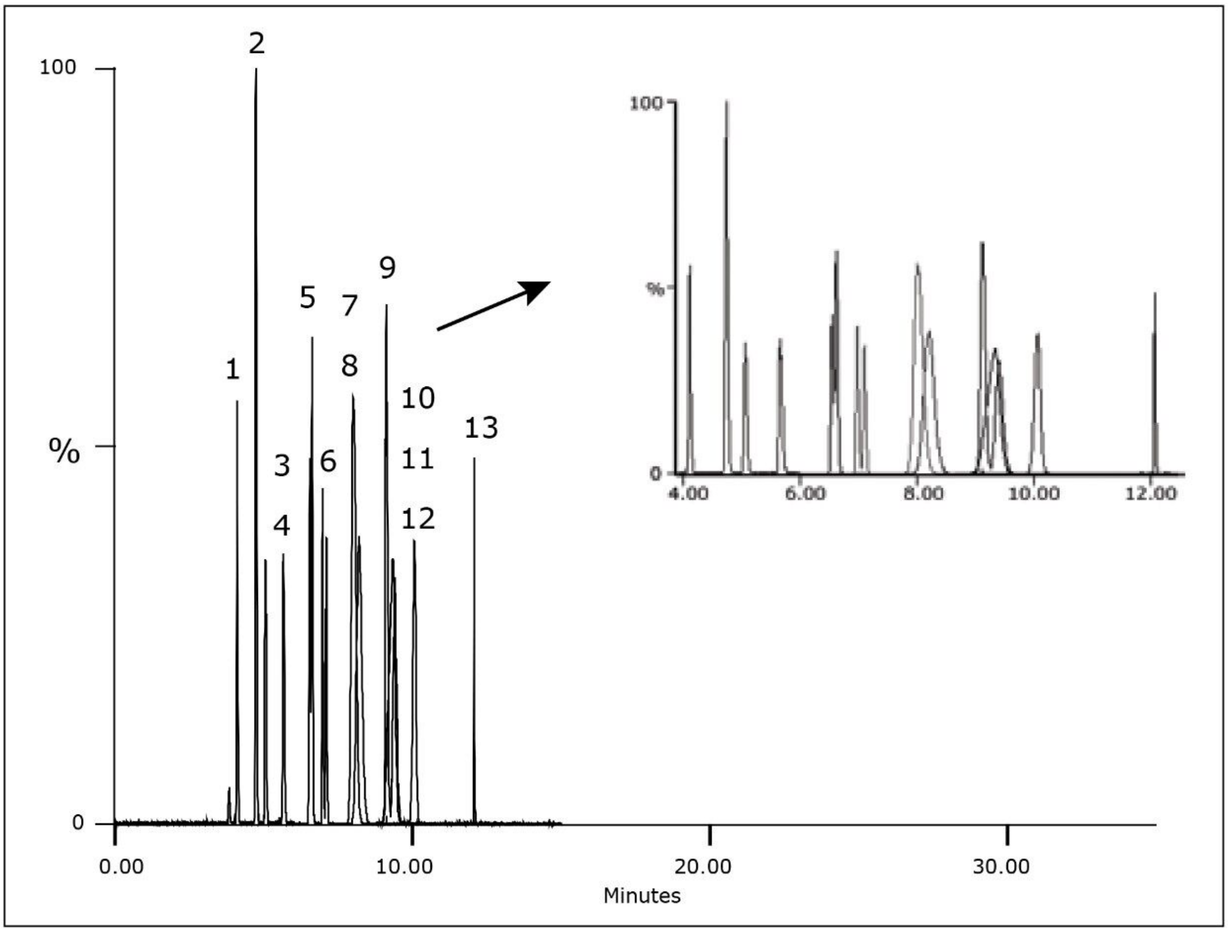
Eluent Preparation

A: 5 mM aqueous ammonium acetate

B: Methanol

Time	Flow	%A	%B
Initial	0.45	90	10
5.0	0.45	75	25
7.5	0.45	66	34
10.5	0.45	65	35
11.0	0.45	10	90
13.0	0.45	90	10

Eluent gradient.



UPLC®/MS/MS chromatogram for EPA method 535 analytes.

Related Documents

- Acetamide Herbicides and Metabolites in Drinking Water - WA31763.10 < https://www.waters.com/webassets/cms/library/docs/symmetry10.pdf>
- Analysis of Chloroacetanilide and Acetamide Herbicide Degradates in Drinking Water by UPLC-MS/MS - 720001999EN <https://www.waters.com/nextgen/us/en/library/application-notes/2006/analysis-of-chloroacetanilide-and-acetamide-herbicide-degradates-in-drinking-water-by-uplc-ms-ms.html>

Featured Products

ACQUITY UPLC System <https://www.waters.com/514207>

MassLynx MS Software <https://www.waters.com/513662>

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