

Acidic Herbicides in Drinking Water by LC-MS

Waters Corporation



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

Abstract

This application brief demonstrates analysis of acidic herbicides in drinking water by LC-MS.

Introduction

Compounds analysed in this study are:

1. picloram
2. chloramben
3. 4-nitrophenol bentazon
4. 2,4-D
5. MCPA
6. 2,4,5-T
7. dichlorprop
8. MCPP
9. dichlorobenzoic
10. acifluorfen
11. 2,4,5-TP
12. 2,4-DB
13. dinoseb
14. pentachloropheno

Experimental

HPLC Method

Column:	XTerra MS C ₁₈ 2.1 x 100 mm, 3.5 µm
Part number:	186000404

Mobile phase A:	15 mM NH ₄ COOH, pH 3.4
Mobile phase B:	ACN
Injection volume:	20 µL
Flow rate:	0.2 mL/min to MS
Temperature:	35 °C
Detection:	MS ESI
Instrument:	Alliance 2695, Micromass ZQ

Gradient

Time (min)	Profile	
	%A	%B
0.0	75	25
9.0	40	60
14.0	40	60
30.0	10	90

Compound	RF (slope)	r ²	LOQ	RSD*
1. picloram	3.7	0.999	300	16%
2. chloramben	1.8	0.989	200	9.3%
3. 4-nitrophenol				
4. (non-linear above 500)	474	0.990	<100	5.6%
5. bentazon				
6. (non-linear above 300)	181	—	<100	6.1%
7. 2,4-D	51	0.999	100	7.2%
8. MCPA	43	0.980	200	9.2%
9. 2,4,5-T	105	0.999	100	6.3%
10. dichlorprop	105	0.999	100	5.0%
11. MCPP	136	0.992	100	7.0%
12. dichlorobenzoic	64	0.988	100	5.6%
13. acifluorfen				
14. (non-linear above 300)	50	—	100	11%
15. 2,4,5-TP	91	0.997	100	6.6%
16. 2,4-DB	77	0.999	100	6.3%
17. dinoseb				
18. (non-linear above 200)	>500	—	<100	10.7%
19. pentachlorophenol	67	0.998	100	11.7%

*average of RSD from 4 levels

OASIS® MAX SPE METHOD FOR ACIDIC HERBICIDES

Conditions for Oasis® MAX Cartridge, 6 cc, 150 mg
Part Number 186000369

Sample is first hydrolyzed at pH 12 for 60 min. Then, pH is adjusted to approx. neutral with HCl before SPE.

PREPARE SAMPLE:

CONDITION:

3 mL methanol/ 3 mL water

LOAD:

300 mL sample

WASH #1:

3 mL 50 mM NaOAc (pH 7.5)

WASH #2 (methanol) will contain bases and neutrals retained by reversed-phase interaction. This fraction may be analyzed for those compounds if desired.

ELUTE 1 (WASH #2):

4 mL methanol

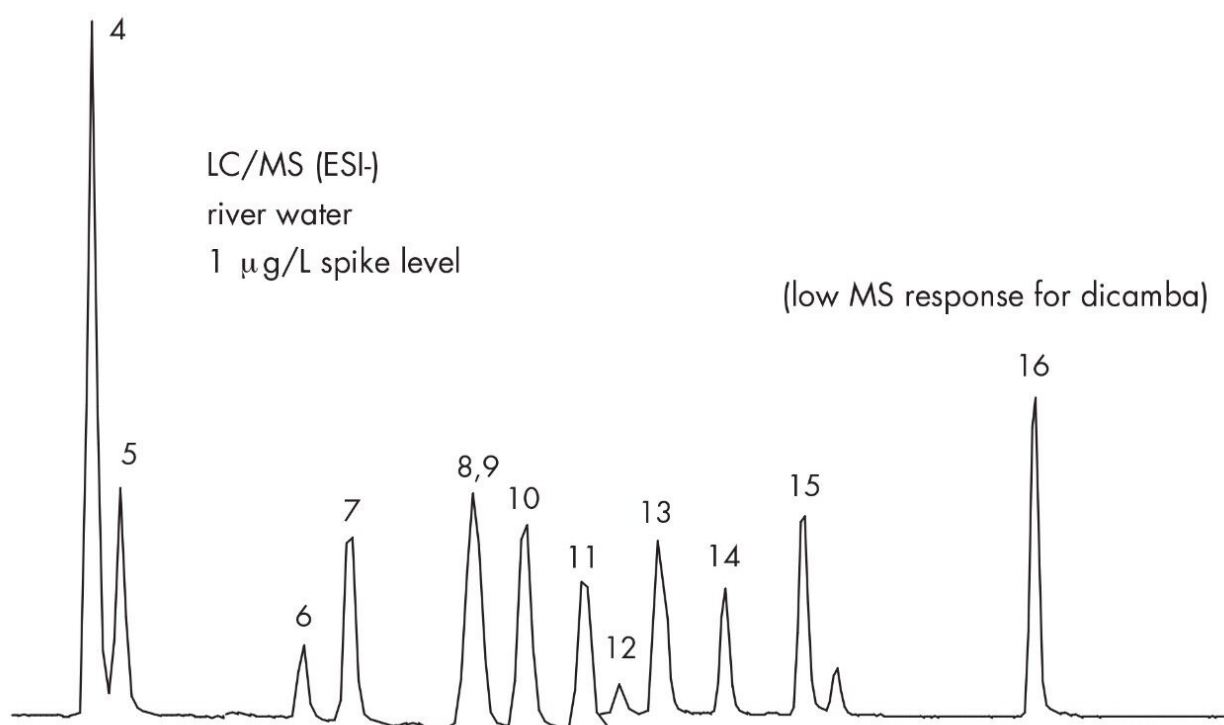
Analytes with $pK_a < 3$ require strong acid (i.e. trifluoroacetic) at this step.

ELUTE 2*:

4 mL methanol(2 % TFA)

EVAPORATE AND RECONSTITUTE

Results and Discussion



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