

## 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 <sub>18</sub> 2.1 x 100 mm, 3.5 µm |
| Part number:      | 186000404                                      |
| Mobile phase A:   | 15 mM NH <sub>4</sub> 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<br>(min) | Profile |    |
|---------------|---------|----|
|               | %A      | %B |
| 0.0           | 75      | 25 |
| 9.0           | 40      | 60 |
| 14.0          | 40      | 60 |
| 30.0          | 10      | 90 |

| Compound                   | RF (slope) | r <sup>2</sup> | 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.

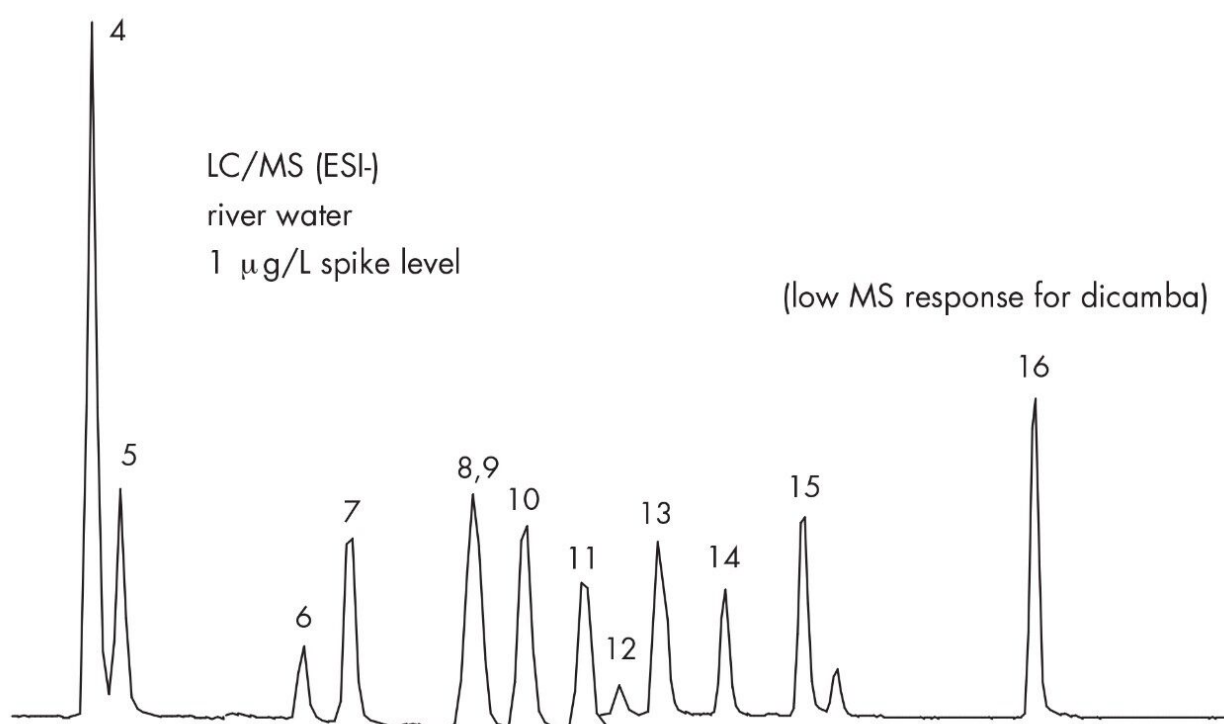
### ELUTE 2\*:

4 mL methanol(2 % TFA)

### EVAPORATE AND RECONSTITUTE

---

## Results and Discussion



---

## Featured Products

WA31764.27, June 2003