

# Analysis of Chloroacetanilide and other Acetamide Herbicide Degradates in Drinking Water Using EPA Method 535 with Automated Solid Phase Extraction (FMS TurboTrace®)

## Introduction

Chloroacetanilide and its congeners are degradation products of acetamide-containing herbicides. Originally, conceived as a pharmaceutical agent, its liver and kidney toxicity, led to its being phased out. However, they can leach into the environment, due to weed killer usage, posing a serious threat to human health.

EPA Method 535 outlines the procedure for extraction and analysis of these compounds in water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 535 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of automating these processes apparent. Hence, FMS developed a simple automated system which is fast, inexpensive and yields high quality data.

## Instrumentation

- FMS TurboTrace® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC
- Waters Xevo TQD

## Consumables

- FMS, Inc. 0.5 g charcoal cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Ammonium acetate
- Fisher Pesticide Grade Methanol
- Restek 535 spiking standards

## Procedure

- 6 samples (250 mL water each) are prepared
- Spike with various 535 standards
- Put sample bottles in place and fill rinse bottles with 15 mL 10 mM ammonium acetate in methanol
- Cartridges are installed in each of the six positions.

### Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 2 x 10 mL ammonium acetate/methanol (keep wet) and 3 x 10 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 10 mL/min
- Cartridges are rinsed with 5 mL water and dried with nitrogen for 3 minutes.
- Sample bottles are automatically rinsed from the rinse bottles with 15 mL ammonium acetate/methanol.

### Stage 2:

- Ammonium acetate/methanol from sample bottles is loaded across the cartridges (5 mL, then 10 mL) and the eluent is collected for analysis into Direct to LC Vial Collection Vessels

## FMS SuperVap®

- Pre-heat temp: 65 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 65 °C under nitrogen (7-10 psi)
- Direct to LC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis



Table 1 with recoveries for a number of 535 compounds

| Compound         | %Recovery | Stdev |
|------------------|-----------|-------|
| Acetochlor ESA   | 96.2      | 4.2   |
| Acetochlor OA    | 83.5      | 8.2   |
| Alachlor ESA     | 89.1      | 6.0   |
| Alachlor OA      | 97.3      | 7.4   |
| Dimethenamid ESA | 102.5     | 3.8   |
| Dimethenamid OA  | 91.2      | 5.3   |
| Flufenacet ESA   | 102.4     | 5.9   |
| Flufenacet OA    | 79.5      | 4.7   |
| Metolachlor ESA  | 91.6      | 6.3   |
| Metolachlor OA   | 84.5      | 3.5   |
| Propachlor ESA   | 92.7      | 6.7   |
| Propachlor OA    | 100.3     | 4.5   |

### Conclusions

Reviewing the sample data shows high recoveries for a dozen acetanilide congeners, demonstrating excellent efficiency for these compounds. Samples can be taken from collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



For more information contact FMS:  
 FMS, Inc.  
 580 Pleasant Street  
 Watertown, MA 02472  
 Phone: (617) 393-2396  
 Fax: (617) 393-0194  
 Email: [onlineinfo@fms-inc.com](mailto:onlineinfo@fms-inc.com)  
 Web site: [www.fms-inc.com](http://www.fms-inc.com)

FMS TurboTrace® System