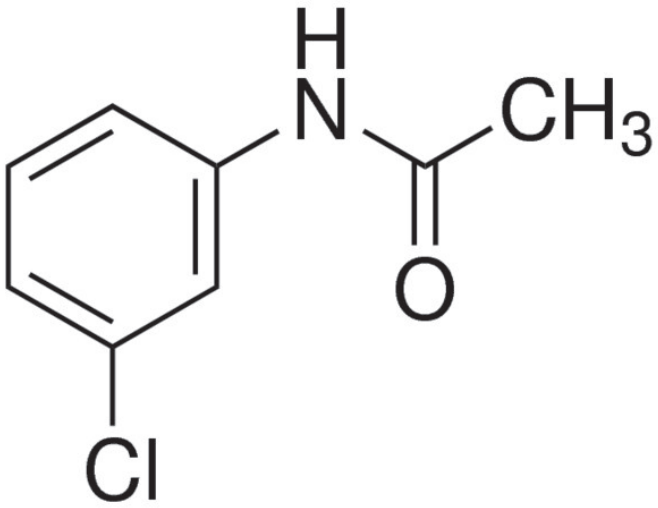
# Measurement of Chloroacetanilide and Other Acetamide Herbicide Degradates in Drinking Water by Solid-Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)




## UCT Part Numbers

**EC535156**
(500 mg graphitized carbon black (GCB) 90 m2/g, 6 mL)

## Method Summary:

A 250 mL water sample is drawn through and captured on a EC535156 cartridge containing 0.5 grams of non-porous graphitized carbon. Acetanilide and acetamide compounds are eluted from the cartridge using a small quantity of methanol containing 10 mM ammonium acetate. The methanol extract is concentrated to dryness by blow down with N2 in a water bath at 65OC then reconstituted with 1 mL of water containing 5 mM ammonium acetate. A 100 µL portion of the aqueous reconstitution is injected into an HPLC fitted with a C18 reverse phase analytical column. Detection occurs by tandem mass spectrometry and is compared to internal standards. A surrogate analyte of known concentration is measured with the same internal standard calibration procedure.

## Interferences:

Humic and/ or fulvic acid material, if present in the water source, is co-extracted with this method. High concentrations of these compounds can cause enhancement or suppression of the in the electrospray ionization source or low recoveries on the carbon SPE. Total organic carbon (TOC) is a good indicator of these interferences if present in the water sample.

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## Procedure:

### 1. Condition Cartridge

a) Rinse the cartridge with 20 mL of 10 mM ammonium acetate/methanol solution.

b) Rinse cartridge with 30 mL of reagent water. Do not let water drop below level of cartridge packing.

c) Add about 3 mL of reagent water to the top of the cartridge.

**Note:** Do not let the cartridge go dry during any step otherwise start over

### 2. Sample Addition

a) Add sample water to the cartridge and adjust vacuum so the flow is about 10-15 mL/minute.

b) Rinse cartridge with 5 mL of reagent water.

c) Draw air or $N_2$ through the cartridge at high vacuum (10-15 in/Hg) for 3 minutes.

### 3. Extract Elution

**Note:** All glassware must be meticulously washed to avoid contamination

a) Insert a clean collection tube into the extraction manifold.

b) Use 15 mL of 10 mM ammonium acetate/methanol and adjust vacuum to draw through at 5 mL/minute. Solvent will exit the cartridge in a dropwise fashion at this vacuum setting.

### 4. Eluate Drying

a) Concentrate the extract to dryness under a gentle stream of N2 in a heated water bath at 60°-70° C to remove all of the ammonium acetate/methanol.

b) Reconstitute the dried eluate by adding 1 mL of 5 mM ammonium acetate/methanol solution.

### 5. Extract Analysis

a) Establish operating conditions for the liquid chromatograph and mass spectrometer according to Tables 1-4 in Section 17. See Table A below for RT and precursor ions.

b) If the analyte peak area exceed the range of the initial calibration curve, the extract may be diluted with 5 mM ammonium acetate/reagent water and adjusting internal standards to compensate for this dilution.

**Table A - Triple Quadrupole MS/MS Method Conditions**

| Analyte | Retention Time | Precursor Ion | Product Energy | Collision Energy |
|---|---|---|---|---|
| Propachlor OA | 7.33 | 206 | 134 | 8 |
| Flufenacet OA | 8.67 | 224 | 152 | 10 |
| Propachlor ESA | 10.01 | 256 | 80 | 25 |
| Flufenacet ESA | 10.81 | 274 | 80 | 25 |
| Dimethenamid OA | 13.25 | 270 | 198 | 10 |
| Dimethenamid ESA | 14.87&15.11 | 320 | 80 | 25 |
| Alachlor OA | 15.86 | 264 | 160 | 10 |
| Acetochlor OA | 16.34 | 264 | 146 | 10 |
| Alachlor ESA | 18.46 | 314 | 80 | 25 |
| Metolachlor OA | 18.60 | 278 | 206 | 8 |
| Acetochlor ESA | 19.12 | 314 | 80 | 30 |
| Metolachlor ESA | 20.95 | 328 | 80 | 25 |
| Dimethachlor ESA (sur) | 12.18 | 300 | 80 | 25 |
| Butachlor ESA (IS) | 36.95 | 356 | 80 | 25 |

## References:

[1] For complete details on Method 535 Version 1.1 the analyst is referred to: J. A. Shoemaker and M. V. Bassett, April 2005, National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

**DCN-903020-114**

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