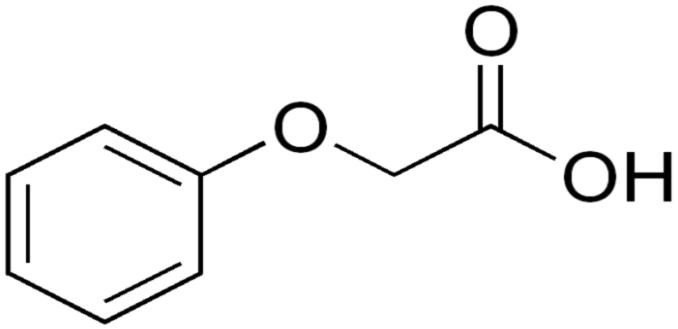
# Extraction of Phenoxyacetic Acid Herbicides From Soil By LC-MS/MS




## UCT Part Numbers

**EEC181M6**
ENVIRO-CLEAN®
6 mL cartridge

**ECUNIC18**
ENVIRO-CLEAN®
1100mg C18/
Universal Cartridge (83mL)

## Procedure:

### 1. Sample Pretreatment

a) Prepare an acid washed beaker*
b) Add 10-100 grams of soil sample
c) Add enough DI $H_2O$ to form a loose slurry
d) Insert a magnetic stir bar and extract for 15 minutes
e) Adjust pH to 2 using 50% aqueous sulfuric acid ($H_2SO_4$)
f) Continue extraction for 15 minutes adjusting pH as needed
g) Filter sample through previously acidified filter media

**Note:** Acid washed glassware must be used in this procedure. Soda lime glassware must be avoided as it may interfere with the analysis.

### 2. Condition C18 SPE Cartridge

a) Add 5 mL $CH_3OH$ and wait 1 minute
b) Add 5 mL DI $H_2O$

**Note:** Aspirate at low vacuum setting. Do not let cartridge dry out otherwise repeat steps a) and b)

### 3. Add Sample

a) Adjust vacuum and load cartridge at 10 mL/minute flow rate

### 4. Dry Cartridge

b) Dry cartridge for 10 minutes at full vacuum

### 5. Elute Phenoxyacetic acid Herbicides

a) Place a clean collection vial in manifold
b) Add 5 mL of $CH_3OH$ and wait 1 minute
c) Add a second 5 mL volume of $CH_3OH$
d) Adjust vacuum and collect at 1-2 mL/ minute

### 6. Dry Eluate

a) Evaporate to dryness at < 40˚C using $N_2$
b) Reconstitute in 100 µL of mobile phase for LC-MS/MS
c) Inject 10-100 µL

## HPLC Analysis and Instrumentation Requirements:

**Guard Column:** C18 10mm x 2.6mm with 0.5 µm frit

**Analytical Column:**
- C18 100 mm x 2 mm 5 µm particle ODS-Hypersil
- C18 100 mm x 2 mm 3 µm particle MOS2-Hypersil or equivalent

**HPLC/MS Interface:** Micromixer 10-µL interface HPLC column system with HPLC post-column addition solvent

**Interface:** Thermospray ionization interface and source capable of generating both positive and negative ions and have a discharge electrode or filament

**Mass Spectrometer System:**
- A single quadrupole mass spectrometer capable of scanning from 1 to 1000 amu
- Scanning from 150 to 450 amu in 1.5 sec. or less using 70 volts (nominal) in positive or negative electron modes
- Capable of producing a calibrated mass spectrum for polyethylene glycol (PEG 400, 600, or 800, average mol. wts.) or other compounds used as a calibrant
- Use PEG 400 for analysis of chlorinated phenoxyacid compounds. PEG is introduced via the Thermospray interface circumventing the HPLC

## Thermospray Temperatures:

**Vaporizer Control:** 110°C to 130°C
**Vaporizer Tip:** 200°C to 215°C
**Jet:** 210°C to 220°C
**Source Block:** 230°C to 265°C

## Recommended HPLC Chromatographic Conditions

### Chlorinated Phenoxyacid Compounds

| Initial Mobile Phase % | Initial Time (minutes) | Final (minutes) | Final Mobile Phase % | Time (minutes) |
|---|---|---|---|---|
| 75A/25 | 2 | 15 | 40/60 | |
| 40A/60 | 3 | 5 | 75/25 | 10 |

A=0.1 M ammonium acetate/methanol

## Limits of Detection in the Positive and Negative Ion Modes for HPLC Analysis of Chlorinated Phenoxyacid Herbicides and Esters

| Compound | Positive Ion Mode Quantitation LOD | | Negative Ion Mode Quantitation LOD | |
|---|---|---|---|---|
| | Ion | ng | Ion | ng |
| **Dalapon** | Not detected | 13 | 141 $(M^-H)^-$ | 11 |
| **Dicamba** | 238 $(M^+NH_4)^+$ | 2.9 | 184 $(M^-HCl)^-$ | 3.0 |
| **2,4-D** | 238 $(M^+NH_4)^+$ | 120 | 184 $(M^-HCl)^-$ | 50 |
| **MCPA** | 218 $(M^+NH_4)^+$ | 2.7 | 199 $(M^-1)^-$ | 28 |
| **Dichloroprop** | 252 $(M^+NH_4)^+$ | 5.0 | 235 $(M^-1)^-$ | 25 |
| **MCPP** | 232 $(M^+NH_4)^+$ | 170 | 213 $(M^-1)^-$ | 12 |
| **2,4,5- T** | 272 $(M^+NH_4)^+$ | 160 | 218 $(M^-HCl)^-$ | 6.5 |
| **2,4,5-TP Silvex** | 286 $(M^+NH_4)^+$ | 24 | 269 $(M^-1)^-$ | 43 |
| **Dinoseb** | 228 $(M^+NH_4^-NO)^+$ | 3.4 | 240 (M-) | 19 |
| **2,4-DB** | 266 $(M^+NH_4)^+$ | 1.4 | 247 (M-1)- | 110 |
| **2,4,5-D, butoxy ethanol ester** | 321 $(M^+H)^+$ | 1.4 | 185 $(M^-C_6H_{13}O1)^-$ | |
| **2,4,5-T,butoxy ethanol ester** | 372 $(M^+NH_4)^+$ | 0.6 | 195 $(M^-C_8H_{15}O_3)^-$ | |
| **2,4,5-T, butyl ester** | 328 $(M^+NH_4)^+$ | 8.6 | 195 $(M^-C_6H_{11}O_2)^-$ | |
| **2,4-D, ethyl hexyl ester** | 350 $(M^+NH_4)^+$ | 1.2 | 161$(M^-C_{10}H_{19}O_3)^-$ | |

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**DCN-900240-146**

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