



Determination of Phenoxyacid Herbicides in Water by Solid Phase Extraction and LC-MS/MS Detection

UCT Part Numbers:

ECHLD156-P (Enviro Clean[®] HL DVB 500mg/6mL, PE Frits)

VMFSTFR12 (Sample Transfer Tubes)

EPA Method 8321B*

Procedure:

1. Sample Pretreatment

- a) Adjust sample pH to <1 with 1:1 sulfuric acid in water, low pH is critical to obtain high recoveries.

2. Cartridge Conditioning

- a) Attach sample transfer tubes (**VMFSTFR12**) to the top of the SPE cartridges (**ECHLD156-P**), and attach the SPE cartridges to an SPE manifold.
- b) Wash the SPE cartridges (with transfer tubes connected) using 10 mL methylene chloride, let solvent soak sorbent for 2 min before drawing to waste, leave full vacuum on for 1 min.
- c) Condition the SPE cartridges with 10 mL methanol, leave a thin layer above the frit.
- d) Equilibrate the SPE cartridges with 15 mL DI water, leave a thin layer above the frit.

3. Sample Loading

- a) Insert the stainless steel ends of the sample transfer tubes into sample bottles, adjust vacuum for a fast dropwise sample flow (about 20-25 mL/min).
- b) After all sample is passed through, dry the SPE cartridges under full vacuum for 10 min.

4. Analyte Elution

- a) Insert the collection vials to the manifold.
- b) Rinse the sample bottles with 5 mL acetonitrile, apply the rinse to the SPE cartridges. Let the elution solvent soak the sorbent for 1-2 min before drawing through slowly.
- c) Repeat the elution (step 4b) with 2 additional aliquots of 5 mL acetonitrile.

Instrumental Analysis

Analyze the eluate directly by LC-MS/MS, or concentrate to 1 mL and analyze by HPLC.

Note: Use acid washed sodium sulfate and glassware if SPE eluates need be dried and concentrated.

Results

Compound	LCS1 Recovery%	LCS2 Recovery%	RPD%
2,4-D	96.6	96.2	0.4
MCPA	95.0	94.4	0.6
Dichlorprop	93.6	92.7	1.0
Mecoprop	94.4	93.4	1.1
2,4,5-T	94.2	93.0	1.3
Dichlorobenzoic acid	89.5	87.6	2.1
2,4-DB	85.1	83.7	1.7
Acifluorfen	108.6	88.4	20.5
Silvex	103.1	86.0	18.1
Bentazone	89.8	89.7	0.1

*EPA Method 8321B SOLVENT-EXTRACTABLE NONVOLATILE COMPOUNDS BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY/THERMOSPRAY/MASS SPECTROMETRY (HPLC/TS/MS) OR ULTRAVIOLET (UV) DETECTION