

Extraction of Chlorophenoxy Acid Herbicides from an Aqueous Matrix



Procedure:

1. Base Hydrolysis

- Etch bottle for volume determination.
If necessary, transfer sample to a larger bottle.
- Add surrogates and matrix spike as necessary.
- Add 17 ml of 6N NaOH to sample. Shake and verify pH to > 12. Adjust if necessary.
- Let sample sit for at least 1 hour.
Check sample pH periodically.

2. Acidification

- Acidify the sample by adding 17 ml of cold 12N H₂SO₄.
- Shake and verify that the pH is < 1 using a pH meter.
- All glassware must be acid rinsed with dilute sulfuric acid.

3. Cartridge Conditioning

- Add 5 mLs of methanol to the cartridge.
- Draw a few drops through and hold for 1 minute.
- Rinse cartridge with 15 mLs of water adjusted to pH < 2 with 12N H₂SO₄.

Note: Do not let the cartridge dry out otherwise repeat conditioning steps.

4. Sample Extraction

- Draw the water sample through the cartridge adjusting the vacuum setting so that flow is about 25 mL/min.
- Draw air through the cartridge under full vacuum for 10 minutes to dry the cartridge. Tap the cartridge to removed excess water.

UCT Part Numbers

ECUNIDVB500
500 mg DVB, 83 mL cartridge

ECDVB156
500 mg DVB, 6 mL cartridge

Herbicides Determined Using this Procedure

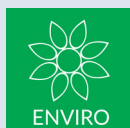
DCPA (surrogate)
MCPP (Mecoprop)
Dicamba
MCPA
Dichloroprop
2,4-D
2,4,5-TP (Silvex)
2,4,5-T
2,4-DB
Dinoseb

5. Elution

- Insert a collection tube containing 2 grams of acidified sodium sulfate.
- Add 10 mLs of acetone to the sample bottle and rinse. Add to cartridge.
- Allow to soak for 2 minutes and collect.
- Add 10 mLs of methylene chloride to the sample bottle and rinse. Add to cartridge.
- Allow to soak for 2 minutes and collect.
- Allow the extract to remain in contact with the acidified sodium sulfate for 2 hours. Incomplete drying will result in low recoveries for some analytes.

6. Concentration and Exchange

- Concentrate eluant using a Kuderna-Danish (KD) concentrator to 5 mLs.
- Alternatively, use an analytical evaporator and N₂ blow down to 5 mLs.
- Derivatize 1 ml with diazomethane and solvent exchange to 10 mLs hexane.
 - Methylation is detailed in Method 8151 or 515. Extract must be very dry otherwise methylation may not be complete.
 - Add internal standard.
 - Sample is ready for GC analysis.



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