Analysis of Glyphosate, Glufosinate and Metabolites Using UCT's Enviro-Clean® Glyphosate Push-Thru Purification Cartridge

UCT Part Numbers

ECGLYSC
Enviro-Clean® Glyphosate Push-Thru Purification Cartridge with Polyethylene Frits - Small

or

ECGLYLC
Enviro-Clean® Glyphosate Push-Thru Purification Cartridge with Polyethylene Frits - Large

FOOD

Summary:

Glyphosate (N-(phosphonomethyl)glycine) is a broad-spectrum systemic herbicide and crop desiccant that is used to kill weeds and grasses that compete with crops. Its herbicidal activity was first discovered in 1970 by researchers at Monsanto, who subsequently brought it to market in 1974 under the trade name Roundup®. Farmers quickly adopted glyphosate, especially after the introduction of genetically engineered crops that were resistant to glyphosate and enabled farmers to kill weeds without killing their crops. In 2012, glyphosate was the most used herbicide in the United States' agricultural and industry, commercial and government sectors, and the second most used in home and garden sector [1]. While glyphosate formulations have been approved by regulatory bodies worldwide, concerns about its effects on humans and the environment have grown as the global usage of glyphosate has increased. As a result, glyphosate now finds itself at the forefront of agricultural regulatory testing.

Commonly monitored in water, soil, vegetation and fruit, glyphosate is notoriously difficult to ionize. UCT's Enviro-Clean® Glyphosate Push-Thru Purification Cartridge simplifies the analysis of glyphosate and its metabolites by removing unwanted matrix components that can otherwise lead to significant ion-suppression. Samples that have been extracted with water or a buffer solution are simply pushed through a small or large purification cartridge prior to instrumental analysis. This application note outlines a suggested extraction procedure and an optional fluorenylmethyloxycarbonyl chloride (FMOC) derivatization step that allows for the analysis of glyphosate on a C18 HPLC column. However, the procedure can readily be modified to meet a laboratory's specific sample volume and analysis requirements.

Extraction Procedure:

1. Weigh 1-3 g of representative sample into a 50 mL centrifuge tube.
2. Add 20 mL of water (18 MΩ).
3. Shake the sample for 2 minutes on a Spex Geno/Grinder® (1500 rpm).
4. Centrifuge the sample at 3000 rpm for 5 minutes.
5. Remove the plunger from a disposable syringe and attach the syringe to an Enviro-Clean® Glyphosate Push-Thru purification cartridge (ECGLYSC or ECGLYLC).
6. Add 1-3 mL of the sample supernatant (dependent on the required volumes for sufficient analysis) to the syringe barrel, attach the plunger, and gently push the sample through the cartridge into a collection tube.
7. The sample can be filtered through a 0.45 µm nylon filter into an autosampler vial and analyzed directly by LC-MS/MS. Alternatively, the sample can be derivatized following the procedure outlined below.
    - The use of a 0.2 µm filter can result in the glyphosate-FMOC complex getting retained.

Derivatization Procedure (Optional):

1. Transfer 1 mL of purified extract into a polypropylene tube.
2. Add 0.5 mL of borate buffer and 0.5 mL of FMOC solution.
3. Cap the tube and immediately vortex for at least 30 seconds.
4. Sonicate the sample for at least 15 minutes.
5. Let the sample sit at room temperature for approximately 1 hour.
6. Vortex the sample again for at least 30 seconds.
7. Sonicate the samples again for approximately 15 minutes.
8. Let the samples sit for approximately 2.5 hours.
    - Total time has to be at least 4 hours from the time the FMOC was added to the sample. Alternatively, the samples can be derivatized overnight at room temperature.
9. Filter the samples through a 0.45 µm nylon filter into an autosampler vial.
10. All derivatized samples should be analyzed within 60 hours of FMOC addition.

Reagents:

1. Borate Buffer Solution, 5% w/v:

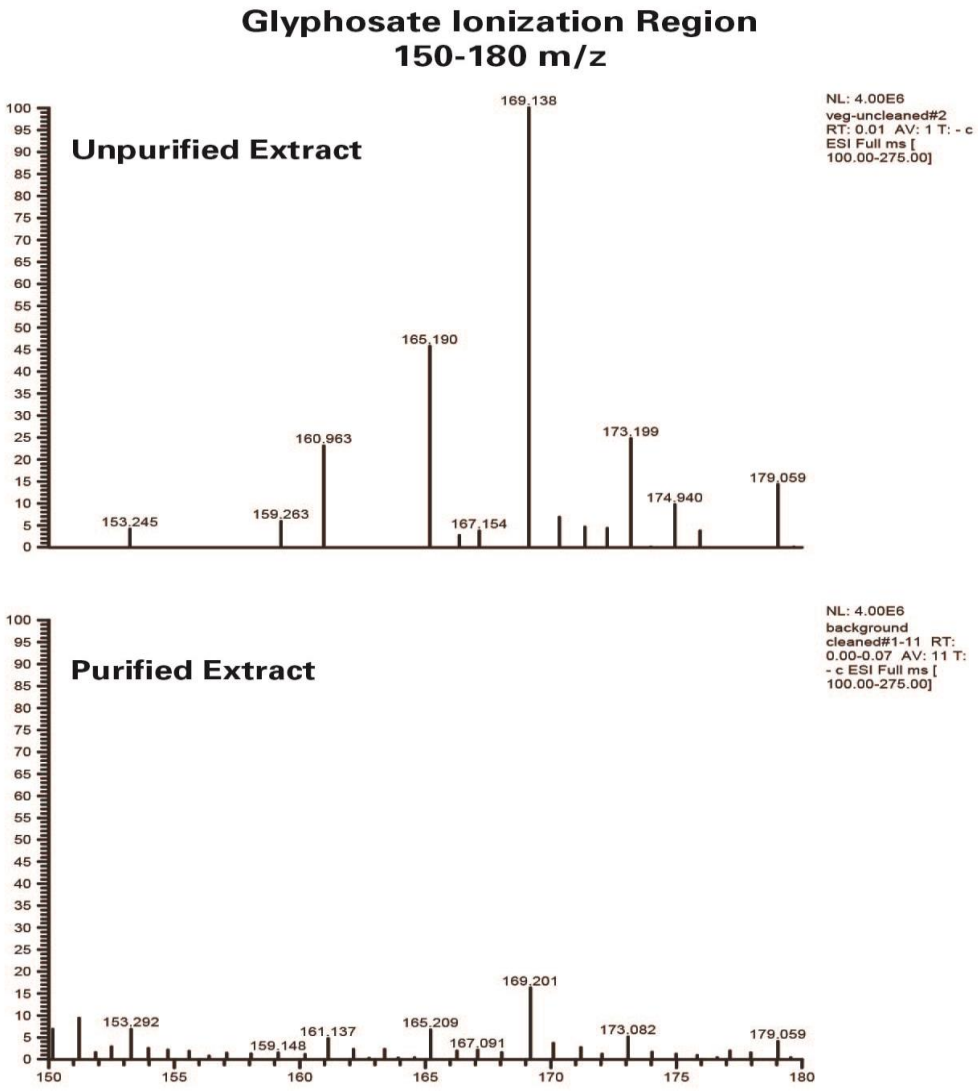
Weigh 5 g of sodium tetraborate (ACS grade or better) into a 100 mL volumetric flask. Add 70 mL of water (18 MΩ) and sonicate until the sodium tetraborate is completely dissolved. Bring to a final volume of 100 mL.

2. Fluorenylmethyloxycarbonyl chloride (FMOC), 1% w/v:

Dissolve 1 g of FMOC in 100 mL acetonitrile.

FOOD

Example of an MS Spectrum Before and After Cleanup:



Vegetation samples were infused onto an Ion Trap LC/MS in full-scan mode for an average of 30 seconds pre and post clean-up using UCT's Glyphosate Purification Cartridge. Background matrix that can lead to significant ion suppression and compete with glyphosate and glufosinate during analysis were significantly reduced following clean-up.

* Source: Steven C. Moser and OK Department of Agriculture, Food & Forestry.
**Note: glyphosate is traditionally found at *m/z* 168.

References:

[1] https://www.epa.gov/sites/production/files/2017-01/documents/pesticides-industry-sales-usage-2016_0.pdf

FOOD

UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 800.385.3153 • 215.781.9255
www.unitedchem.com Email: methods@unitedchem.com

UCT