Simultaneous Direct Analysis of Glyphosate and Aminomethylphosphonic Acid in Surface Water Using UPLC-MS in Selected Ion Recording Mode

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Abstract

Glyphosate, a nonselective, broad-range weed killer, is one of the most widely used herbicides in applications for weed control due to its low toxicity. Although glyphosate has a very low toxicity, it can be harmful to aquatic life in surface water at higher concentrations.Glyphosate and aminomethylphosphonic acid (AMPA), its main degradation product, are very polar and have a high solubility in water. Methods that are usually used to analyze glyphosate are HPLC-fluorescence detection, GC-MS or HPLC-MS/MS. Due to glyphosate’s high polarity, these current methods require a derivatization step which can extend the time of analysis. Additionally, if HPLC is selected as the analytical technique, it is very challenging to retain underivatized glyphosate utilizing reverse phase chromatography. A validated analytical methodology has been developed for the direct analysis (without derivatization) of glyphosate and AMPA in surface water, utilizing UPLC-MS, a UPLC amide column and single ion recording (SIR). The presented analytical approach is significantly faster compared to the existing methods and eliminates any complications associated with analyte derivatization.

Instrument Specifications

- Waters® Acquity UPLC-MS/MS
- Ultra High Performance Liquid Chromatography (UPLC) Conditions:
  - Column: Acquity Column BEH-Amide 2.1 x 100 mm 1.7 μm
  - Run Time: 5.00 minutes
  - Solvent A: Water
  - Solvent B: Aetonitrile
  - Column Temperature: 25.0°C
  - Injection Volume: 5.0 μL
  - Isocratic

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Flow Rate (mL/min)</th>
<th>%A</th>
<th>%B</th>
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<tbody>
<tr>
<td>Initial</td>
<td>0.300</td>
<td>30.0</td>
<td>70.0</td>
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Major Questions

- What is the potential harm of glyphosate and AMPA to both the environment and humans?
- What are the advantages of the proposed analytical method?
- How is the analyte sample recovery compared to existing methods?

Simple Sample Preparation

1. 20mL of DI water
2. Spike sample with Glyphosate
3. Evaporate samples to dryness (on Genevac® EZ-2 evaporator)
4. Add 200μL of DI water
5. Transfer solution into 0.3mL low recovery vials and inject

Results

Targeted Analytes

- Glyphosate
- Aminomethylphosphonic Acid (AMPA)

Conclusions

- A rugged, efficient analytical method was developed for glyphosate in water that provides very high sensitivity and eliminates complications associated with analyte derivatization.
- The preparation is simple and uses common laboratory equipment, minimizing cost and time.
- This preparation demonstrated a MDL of 1.49 ng/mL for glyphosate.
- The precision and accuracy study demonstrated a relative standard deviation of 2.9% and a recovery of 91%.

References


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