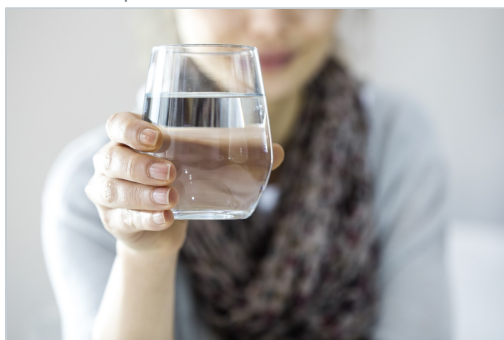


Application Note

Determination of Glyphosate in Drinking Water by Direct Aqueous Injection HPLC, Post Column Derivatization and Fluorescence Detection

Waters Corporation



Abstract

Glyphosate is a non-selective herbicide which is adsorbed through leaves and was first sold by Monsanto under the Roundup trade name. This is one of the most widely used herbicides, regularly used for agriculture, horticulture, and silviculture applications.

Introduction

Glyphosate is a non-selective herbicide which is adsorbed through leaves and was first sold by Monsanto under the Roundup trade name. This is one of the most widely used herbicides, regularly used for agriculture, horticulture, and silviculture applications. The United States Environmental Protection Agency (US EPA) requires that drinking water and raw surface water be monitored for the presence of glyphosate and related compounds using EPA Method 547.0. The European Union (EU) regulation (EC Directive 2005/70/EU) provides guidance with regards to the presence of glyphosate in drinking water supplies.

Experimental

HPLC conditions

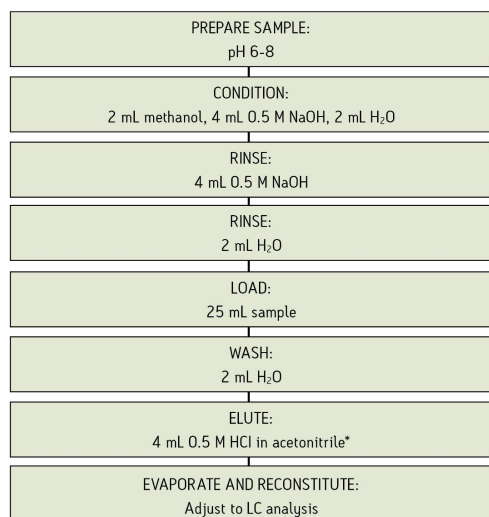
Instrument:	Waters Alliance system for carbamate analysis
Eluent:	0.05% phosphoric acid
Column:	Ion Exclusion, 7.8 mm x 150 mm @ 55 °C
Guard:	Guard-Pak module and inserts
Injection:	200 µL of standard mix
Flow Rate:	1.5 mL / min
Detection:	Fluorescence, Ex- 340 nm, Em- 455 nm, Gain- 10

Sample preparation

Refer to EPA Method 547 for preparation of field grab samples.

Filter through 0.45 µm Acrodisc. filters is described.

Alternative sample preparation



* Alternate eluent is 4 mL 0.6 M sodium citrate

Oasis. MAX SPE Method for Glyphosate and
Metabolite Method for Oasis MAX Cartridge, 6 cc, 150
mg

* Alternate eluent is 4 mL 0.6 M sodium citrate

Use 6 cc, 500 mg Oasis MAX for samples > 50 mL.

Standard mix preparation

Pipette 100 µL of AccuStandard. mix (M-547) into 100 mL of acidified water for a concentration of 100. Prepare acidified water by adjusting the pH of HPLC grade water to 3.0 by dropwise addition of hydrochloric acid (HCL). Use EPA method 547-02 as above for AMPA (aminomethyl phosphonic acid).

Eluent preparation

Dilute 0.5 mL of 85% phosphoric acid (H₃PO₄) to 1 L, mix well, filter and degas.

Post column preparation

Reagent 1: Hypochlorite

Dissolve 1.35 g KH₂PO₄, 11.6 g NaCl, 0.4 g NaOH, and 0.2 mL Clorox. Bleach (plain) in water and dilute to 1 L, filter and degas.

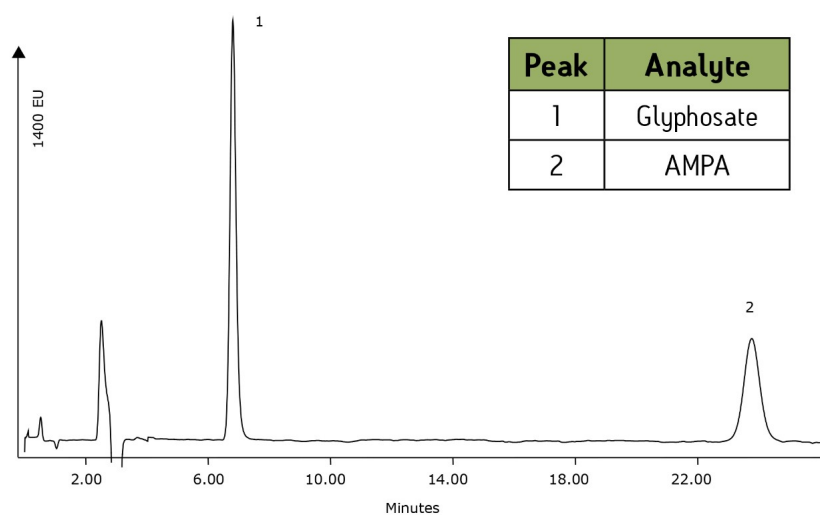
Reagent 2: OPA

Dissolve 0.8 g of OPA (o-phthdialdehyde) in 10 mL of methanol, add this to an aqueous solution of

19.1 g of Borax $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$. Make to a final volume of 1 L, filter and degas. To this, add 2 mL of 2-mercaptoethanol, swirl gently to mix. Protect from light.

Note: Post-column flow rate for both reagents is 0.5 mL/min, post column reaction temperature is 38 °C. Insert second reaction coil in line before the fluorescence detector.

Results and Discussion



Standard chromatogram, 100 ppb each analyte.

References

1. Environmental System Solutions 720001601EN
2. Glyphosate and AMPA in Drinking Water WA31764.94
3. An LC-MS/MS Multi-Analyte Detection Method for Deleterious Organics in Drinking Water
720001090EN

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