

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

547.0

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.

HPLC CONDITIONS

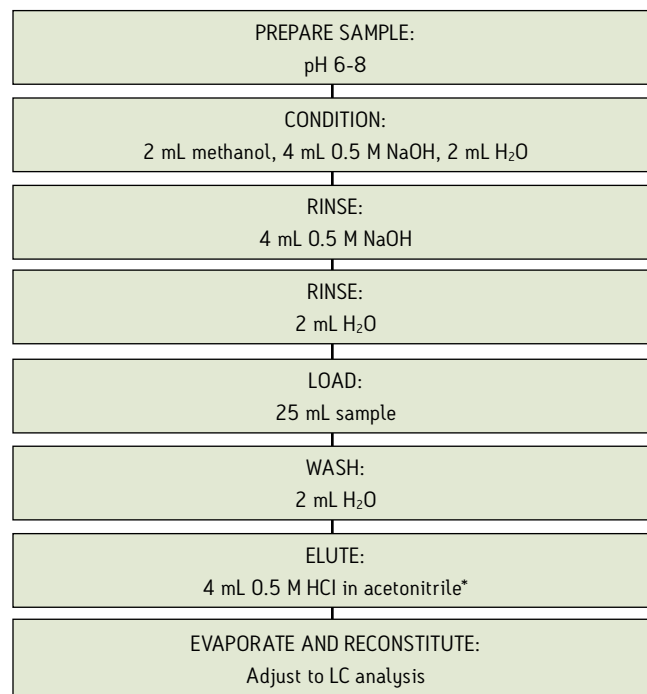
Instrument: Waters Alliance® system for carbamate analysis
 Eluent: 0.05% phosphoric acid
 Column: Ion Exclusion, 7.8 mm x 150 mm @ 55 °C
 Guard: Guard-Pak™ module and inserts
 Injection: 200 µL of standard mix
 Flow Rate: 1.5 mL / min
 Detection: Fluorescence, Ex- 340 nm, Em- 455 nm, Gain- 10

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

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.

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POST COLUMN REAGENT PREPARATION

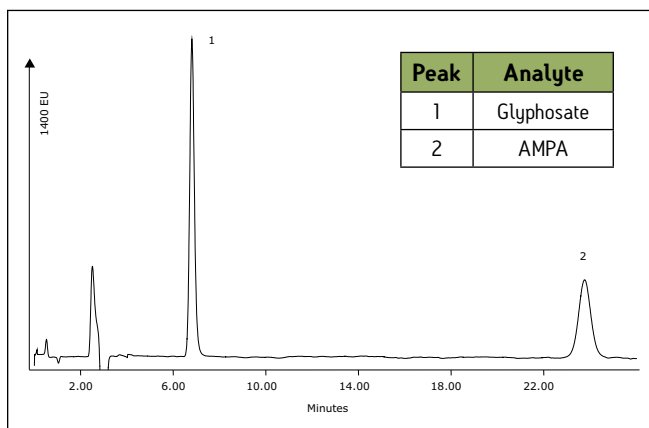
Reagent 1: Hypochlorite

Dissolve 1.35 g KH_2PO_4 , 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-phthaldialdehyde) 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.



Standard chromatogram, 100 ppb each analyte.

ORDERING INFORMATION

Related Parts	Part Number
IC-Pak™ Ion-Exclusion Column, 7.8 x 150 mm	WAT010295
Guard-Pak Holder	WAT88141
Semivolatiles #2 Herbicide Standard	186004271
Oasis MAX Cartridge, 6 cc, 150 mg	186000370
Oasis MAX Cartridge, 6cc, 500 mg	186000865

Related Documents	Literature Code
Environmental System Solutions	720001601EN
Glyphosate and AMPA in Drinking Water	WA31764.94
An LC/MS/MS Multi-Analyte Detection Method for Deleterious Organics in Drinking Water	720001090EN

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