



Determination of Glyphosate, Its Degradation Product Aminomethylphosphonic Acid, and Glufosinate, in Water by Isotope Dilution and Online Solid-Phase ... Chromatography/Tandem Mass Spectrometry

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The U.S. Geological Survey method (0-2141-09) presented is approved for the determination of glyphosate, its degradation product aminomethylphosphonic acid (AMPA), and glufosinate in water. It was validated to demonstrate the method detection levels (MDL), compare isotope dilution to standard addition, and evaluate method and compound stability. The original method USGS analytical method 0-2136-01 was developed using liquid chromatography/mass spectrometry and quantitation by standard addition. Lower method detection levels and increased specificity were achieved in the modified method, 0-2141-09, by using liquid chromatography/tandem mass spectrometry (LC/MS/MS). The use of isotope dilution for glyphosate and AMPA and pseudo isotope dilution of glufosinate in place of standard addition was evaluated. Stable-isotope labeled AMPA and glyphosate were used as the isotope dilution standards. In addition, the stability of glyphosate and AMPA was studied in raw filtered and derivatized water samples. The stable-isotope labeled glyphosate and AMPA standards were added to each water sample and the samples then derivatized with 9-fluorenylmethylchloroformate. After derivatization, samples were concentrated using automated online solid-phase extraction (SPE) followed by elution in-line with the LC mobile phase; the compounds separated and then were analyzed by LC/MS/MS using electrospray ionization in negative-ion mode with multiple-reaction monitoring. The deprotonated derivatized parent molecule and two daughter-ion transition pairs were identified and optimized for glyphosate, AMPA, glufosinate, and the glyphosate and AMPA stable-isotope labeled internal standards. Quantitative comparison between standard addition and isotope dilution was conducted using 473 samples analyzed between April 2004 and June 2006. The mean percent difference and relative standard deviation between the two quantitation methods was 7.6 plus or minus 6.30 (n = 179), AMPA 9.6 plus or minus 8.35

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