

In Reply Refer To:
Mail Stop 412

March 9, 2010

To: GS-W-ALL

From: Terry L. Schertz, Assistant Chief
Office of Water Quality

Subject: Approval of Method for the Analysis of Glyphosate,
Aminomethylphosphonic acid, and Glufosinate, in Water by Isotope
Dilution and Online Solid-Phase Extraction and Liquid
Chromatography/Tandem Mass Spectrometry (USGS Method Number O-
2141-09)

The Office of Water Quality has approved a new water-quality analytical method developed by the Kansas Water Science Center, Organic Geochemistry Research Laboratory (OGRL) for the determination of glyphosate, its degradation product, aminomethylphosphonic acid (AMPA), and glufosinate in filtered water using 9-fluorenylmethylchloroformate (FMOc) derivitization, online solid-phase Extraction (OLSPE) and liquid chromatography/tandem mass spectrometry (LC/MS/MS) with quantitation by isotope dilution (USGS Method Number O-2141-09). This method replaces USGS method O-2136-01 (Lee et al., 2002), for analysis of glyphosate, AMPA, and glufosinate, using FMOc derivitization and OLSPE LC/MS with quantitation by standard addition, which was adapted for LC/MS/MS analysis in 2003.

Glyphosate has become the most widely used herbicide in the world since the introduction of glyphosate tolerant (GT) soybeans and corn in the 1990's. However, comparatively little is known about the fate and transport of glyphosate in groundwater, surface water, and the atmosphere relative to other widely used herbicide. This is primarily due to difficulties in developing analytical methods capable of measuring trace levels glyphosate and AMPA in water. Advances in liquid chromatography/mass spectrometry ionization interfaces in the 1990's provided the means to develop robust methods for the routine analysis of glyphosate.

Acceptable performance for Method O-2141-09 was determined by comparing quantitation of glyphosate, AMPA, and glufosinate by isotope dilution and standard addition on 467 environmental samples over 2 years and also comparing isotope dilution to regressed multi-level extracted standard curves on 7 sets of samples spiked in duplicate at 3 concentration levels in distilled/deionized water, groundwater, and surface water over a period of 2 months. Holding time studies were also conducted on underivitized and derivitized samples. The utilization of isotope dilution eliminates the need to analyze a spiked sample with every unspiked environmental sample required for standard addition, thus decreasing analysis and data reduction costs. The use of the tandem mass spectrometer decreased the reporting levels of glyphosate, AMPA and glufosinate from

0.1 to 0.02 µg/L which increased the applicability of this method for environmental studies of glyphosate.

The method has been in use by the OGRL since October 24, 2000 and approximately 2000 results have been delivered to users. The results to date have been identified by the method code O-2136-01. A reload of those values with the new method code O-2141-09 will be provided. A memo will be sent out by the Office of Water Quality describing the details of this reload.

If you have any questions about the new analytical method, or would like a copy of the report, please contact Mike Meyer (mmeyer@usgs.gov, 785-832-3544).

The reference for this method is:

Meyer, M.T., Loftin, K.A., Lee, E.A., Hinshaw, G., Dietze, J.E, and Scribner, E.A., in press, Determination of Glyphosate, its Degradation Product Aminomethylphosphonic Acid, and Glufosinate in Water by Isotope Dilution and Online Solid-Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry: U.S. Geological Survey Techniques and Methods.