March 20, 2007

To: Water Resources Discipline

From: Janice R. Ward, Senior Hydrologist
Office of Water Quality

Subject: Approval of a Water-Quality Analytical Method O-2140-07, USGS method code LCM47, and OGRL code LCIX for the Determination of Dissolved Isoxaflutole and Its Sequential Degradation Products, Diketonitrile and Benzoic Acid, in Water Using Solid-Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry

The Office of Water Quality (OWQ) has approved a new water-quality analytical method O-2140-07, USGS method code LCM47, and OGRL code LCIX by the Organic Geochemistry Research Laboratory (OGRL) in Lawrence, Kansas, for the determination of low-level concentrations (in micrograms per liter) of isoxaflutole and its two sequential degradation products, diketonitrile and benzoic acid, in water samples. The method uses solid-phase extraction and liquid chromatography/tandem mass spectrometry, using electrospray ionization in negative-ion mode using multiple-reaction monitoring. Compounds are identified by comparing retention times against those of standards and by measuring pre-optimized multiple-reaction monitoring molecular ion and product ion transitions.

The calibration range for this method is equivalent to concentrations from 0.005 to 0.200 µg/L. Samples with higher concentrations are diluted and reanalyzed. Recoveries in buffered reagent water range from 105 to 109 percent and relative standard deviations range from 4 to 18 percent. The combined mean relative standard deviations of the compounds in four water matrices at two spiked concentrations ranged from 0.74 to 26 percent. The initial method detection limit for all compounds is 0.003 µg/L and the initial method reporting level is 0.010 µg/L.

This water-quality analytical method approval follows the technical procedure specified in OWQ Technical Memorandum 98.05, except that the report will be published as Techniques and Methods Report, Book 5, Chapter A9, instead of an Open-File Report. The reference for this method is:

> Michael T. Meyer, Edward A. Lee, and Elisabeth A. Scribner, in production, Methods of Analysis by the U.S. Geological Survey Organic Geochemistry Research Group--Determination of Dissolved Isoxaflutole and Its Sequential Degradation Products, Diketonitrile and Benzoic Acid, in Water Using Solid-Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry: U.S. Geological Survey Techniques and Methods, book 5, chapter A9 (pages to be determined).

After Bureau approval, the report will be made available through the USGS Publications Warehouse at http://infotrek.er.usgs.gov/pubs/. If you have questions about the new analytical method, please contact the senior author Mike Meyer (mmeyer@usgs.gov, 785-832-3564).

If you have questions about the method approval process, please contact Janice Ward (jward@usgs.gov, 303-236-1871).