April 30, 2001

To: Water Resources Division

From: Peter F. Rogerson, Senior Chemist
Office of Water Quality

For: LeRoy Schroder, Chief
Branch of Quality Systems

Subject: Approval of a Water Quality Analytical Method for the Determination of Polar Pesticides and Metabolites by the National Water Quality Laboratory

The Office of Water Quality (OWQ) has approved a new Water Quality Analytical Method for the determination of polar pesticides and metabolites in filtered water developed by the National Water Quality Laboratory (NWQL). This new method is designed to compliment and extend the existing NWQL method O-1131-95, known as Schedule 2050 (laboratory extraction) and Schedule 2051 (field extraction) as described in Werner, et.al., Open File Report 96-216, available at: http://wwwnwql.cr.usgs.gov/Public/pubs/OFR96-216/OFR_96-216.html. The new method uses solid-phase extraction for sample preparation and high-performance liquid chromatography/mass spectrometry (HPLC/MS) for determination of 65 polar pesticides and metabolites in filtered water. Of the 65 analytes, 38 are reported without data qualification, while the remaining 27 are routine reported with the "E" data qualifier, signifying that although the compounds are qualitatively identified as present, their reported concentrations have greater uncertainty. The new method number is O-2060-01 and can be requested through the NWQL as Schedule 2060. At this time there is not a field-extraction option for this method.

Data users are cautioned that compound concentrations reported for some surface water samples may be affected by an apparent matrix-enhancement effect for several classes of pesticides in this method. This matrix enhancement is inherent to the HPLC/MS analysis of these compounds in some environmental samples. It affects primarily sulfonylurea, sulfonamide, and imidazolinone herbicides and tribenuron, all of which are routinely reported with an "E" data qualifier. This effect has been shown to enhance apparent recovery in South Platte River water, with recoveries averaging 214 percent and ranging up to around 400 percent. There are indications that this effect may vary for different surface water sources, with the South Platte River water having some of the highest enhancement effects seen to date. This effect is being evaluated through surface water matrix spikes from National Water Quality Assessment study unit sites. In order to evaluate the impact of surface water matrix enhancement in specific waters, users are strongly encouraged to submit duplicate surface water samples for laboratory spiking. At this time, spiked duplicate surface water samples provide the

only way to assess this matrix enhancement effect for specific samples. For information on submitting field duplicates for laboratory spiking, please contact Mark Burkhardt, Ron Brenton, or Ed Furlong at the NWQL (see below).

This method approval process follows the technical procedure specified in OWQ Tech Memo 98.05, except that this method is described in a Water Resources Investigations Report instead of an Open File Report. The method performance is described in:

> Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry, by Edward T. Furlong, Bruce D. Anderson, Stephen L. Werner, Paul P. Soliven, Laura J. Coffey, and Mark R. Burkhardt. U.S. Geological Survey Water Resources Investigations Report 01-XXXX (number to be assigned upon Director's approval).

When approved by the Director, the report will be made available through the NWQL web site at: http://wwwnwql.cr.usgs.gov/USGS/Pubs/pubs.html .

The complete list of parent pesticides and degradates is available through the NWQL web site http://wwwnwql.cr.usgs.gov/USGS . Please click on SPiN and request Schedule 2060. Note: This announcement approves this method, so there may be a delay of a few days before Schedule 2060 is available in SPiN.

If you have questions about the new analytical method, how to submit a duplicate for spiking, or would like a copy of the report, when it is available, please contact Mark Burkhardt (mrburk@usgs.gov, (303) 236-3250), Ron Brenton (rbrenton@usgs.gov, (303) 236-3210), or Ed Furlong (efurlong@usgs.gov, (303) 236-3941) at the NWQL.

If you have questions about the method approval process, please contact Pete Rogerson (rogerson@usgs.gov, (303) 236-1836).