



United States Department of the Interior

U. S. GEOLOGICAL SURVEY
Reston, VA 20192

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November 23, 2015

OFFICE OF WATER QUALITY TECHNICAL MEMORANDUM 2016.02

SUBJECT: Supplemental guidance for coding and publishing data from NWQL Custom Method LS2437

SUPPLEMENTAL: This technical memorandum contains supplemental information for NWQL Tech Memo [2015.04](#) – Retrospective Review and Guidance for Interpretation of Results from National Water-Quality Laboratory Custom Method (LS2437) “Determination of Pesticides and Pesticide Degradates by Direct-Aqueous-Injection Liquid Chromatography- Mass Spectrometry (DAI LC-MS/MS)”

CORRECTION: Supplemental Memo 2016.01 is revised here with a correction to a parameter code in Table 1. The parameter code for parathion methyl is 65089.

PURPOSE OF SUPPLEMENTAL MEMORANDUM

National Water-Quality Laboratory (NWQL) Method O-2437-15, for the determination of current-use pesticides and pesticide degradates, was implemented as Custom Method Laboratory Schedule 2437 (LS2437), from May 1, 2012 to May 31, 2015. The method was approved by the Office of Water Quality (OWQ) on June 1, 2015 and will be published in U.S. Geological Survey Techniques and Methods book 5, chap. B11 (Sandstrom and others, *in press*). Prior to approval, Custom Method LS2437 was applied in a number of national field studies for surface- and groundwater quality in the National Water-Quality Assessment (NAWQA) Program and the USGS Toxics Hydrology Program. Data from LS2437 have provided extensive testing to establish comparability and stability of the method over time. Subsequent to initial release as a Custom Method, there have been two changes to the approved list of parameters. In May 2013, ten parameters were deleted from the method due to poor performance, and in May 2015, two additional parameters were deleted (Table 1). This supplemental memorandum, TM-2016.01, provides guidance for appropriate NWIS coding, documentation, and possible publication for data from Method O-2437-15 including the 12 rejected parameters, that were released prior to final approval of the method.

Table 1. These compounds, specifically analyzed by USGS Custom Method O-2437-15 (LS2437), have been deleted from the method due to overall poor performance. Data already released to NWIS are coded as “U” and are unavailable to the public. [USGS, U.S. Geological Survey; NWIS, National Water Information System; U, Research or unapproved method or laboratory]

Pesticide Compound	USGS Parameter Code	Method Code	NWIS data quality indicator (DQI) code
Data released to NWIS from May 2012 to May 2013			
3-Ketocarbofuran	68509	LCM60	U
7-Hydroxycarbofuran	68518	LCM60	U
Aminopyralid	68534	LCM60	U
Ammelide	68535	LCM60	U
Bifenazate	68540	LCM60	U
Bifenazate diazene	68541	LCM60	U
Deisopropyliprodione	68565	LCM60	U
Flumiclorac-pentyl	68607	LCM60	U
Formetanate	68609	LCM60	U
Phosmet oxon	68674	LCM60	U
Data released to NWIS from May 2012 to May 2015			
Phosmet	65101	LCM60	U
Parathion methyl	65089	LCM60	U

POLICY

Office of Water Quality Policy Memorandum [04.01](#) provides a procedure for approval and release of water-quality sample results through the National Water Information System-Web Interface (NWIS-Web) or in other USGS information products from unapproved methods prior to method approval. The performance of the unapproved method must be examined and found to be comparable over time to performance of the approved method. If the performance of the unapproved method is comparable to the approved method, then samples may be further examined with respect to field quality control and eventually approved for wider dissemination to the public without a full description or reference to the method of analysis.

GUIDANCE

The NWQL has evaluated and the OWQ has independently reviewed the performance of Custom Method LS2437 from May 2012 through May 2015. They conclude that the data quality has been consistent, comparable, and performing stably over time. Overall, the relatively small differences in median recovery and variability of the pesticide compounds in laboratory reference samples during this time show that there were no major changes related to the method operation and performance from May 2012 through May 2015. Based on this careful analysis, data produced on or after May 1, 2012, with the exception of results for the pesticide parameters listed above (Table 1), are considered suitable to be included in USGS data and interpretive reports without a full description of or reference to the approved method. Results should be reviewed at the local level in light of field quality-control results for approval and updates in Data Quality Indicator (DQI) codes. The NWQL concludes and the OWQ concurs that, except for twelve compounds (Table 1), the data quality support a change of the NWIS data quality

indicator code from “U” (unapproved method) to “S” (presumed satisfactory) for results in NWIS prior to approval of the method from May 1, 2012 to May 31, 2015.

The twelve compounds listed in Table 1 had recoveries that were consistently outside of acceptable control limits established by NWQL and did not perform adequately to be included in the approved method. As per OWQ PM-04.01, the data for these twelve compounds that were already released to NWIS will continue to be coded “U”, and publication of these parameters must include a summary of the method and description of the method performance issues to inform future users who interpret the data. The twelve parameters (Table 1) are no longer included in the schedule for NWQL Method O-2437-15. Further questions or requests for unapproved data should be directed to NAWQA Component Leaders for Surface Water, Groundwater, or Regional Stream-Quality Assessment. Unapproved data will not otherwise be available to the public.

Supporting information is available in a summary document and in six data tables that are available at this internal NWQL website: <http://www.nwql.cr.usgs.gov/USGS/sh2437.shtml> Specific guidance to USGS employees such as any changes in detection levels, analytes dropped, improved recoveries of individual compounds over time, or other considerations are provided. The guidance is for individuals who will interpret the results for purposes of data review and approval in NWIS and publication in other USGS information products.

REFERENCES

- Office of Water Quality Technical Memorandum 2015.04, Retrospective Review and Guidance for Interpretation of Results from National Water Quality Laboratory Custom Method (LC2437) “Determination of Pesticides and Pesticide Degradates by Direct Aqueous Injection Liquid Chromatography- Mass Spectrometry (DAI LC-MS/MS)”, accessed October 26, 2015 at <http://water.usgs.gov/admin/memo/QW/qw2015.04.pdf>
- Office of Water Quality Technical Memorandum “Approval of New USGS Method (O-2437-15) Determination of Pesticides and Pesticide Degradates by Direct Aqueous Injection (DAI) LC-MS/MS” dated June 1, 2015 and accessed October 26, 2015 at http://water.usgs.gov/usgs/owq/Analytical_Methods/2015-06-01_DAI-pest-method.pdf
- Office of Water Quality Technical Memorandum 04.01 Revised Policy for the Approval of U.S. Geological Survey (USGS) Water-Quality Analytical Methods, accessed October 26, 2015 at <http://water.usgs.gov/admin/memo/QW/qw04.01.html>
- Sandstrom, M.W., Kanagy, L.K., Anderson, C.E., and Kanagy, C.J., *in press*, Determination of pesticides and pesticide degradates in filtered water by direct aqueous injection liquid chromatography tandem mass spectrometry: U.S. Geological Survey Techniques and Methods book 5, chap. B11.

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