United States Department of the Interior

U. S. GEOLOGICAL SURVEY
Reston, VA 20192

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October 24, 2016

OFFICE OF WATER QUALITY TECHNICAL MEMORANDUM 2017.01

SUBJECT: Retrospective Review and Guidance for Interpretation and Approval of Results from National Water-Quality Laboratory Methods O-4437-16 for the determination of 37 heat-purgeable volatile organic compounds (VOCs) and O-4436-16 for the determination of 49 ambient-purgeable VOCs

PURPOSE OF MEMORANDUM

The Office of Water Quality (OWQ) approved two whole-water analytical methods at the USGS National Water Quality Laboratory (NWQL): Method O-4437-16 for the determination of 37 heat-purgeable volatile organic compounds (VOCs), and Method O-4436-16 for the determination of 49 ambient-purgeable VOCs (NWQL Rapi-Note 16-11). These two methods are used in the following NWQL Laboratory Schedules (LS):

- LS 4437, heat-purgeable VOCs (Method O-4437-16);
- LS 4443, a sub-schedule of LS 4437 (Method O-4437-16);
- LS 4436, ambient-purgeable VOCs (Method O-4436-16);
- LS 4439, a sub-schedule of LS 4436 (Method O-4436-16); and
- LS 4440, which is a combination of all compounds in the previously approved LS 2020 (Method O-4127-96) (Connor and others, 1998) and those in LS 4436 (Method O-4436-16).

The methods were approved on May 10, 2016 in OWQ Method-Approval Memorandum 30. Analytes approved in this method are listed in Appendixes A1 and A2 of the current memo. The methods were implemented for the time period from December 1, 2012 through May 10, 2016 as custom schedules 4437 and 4436, for testing by the National Water-Quality Assessment (NAWQA), the Toxics Hydrology Program, and the Groundwater Ambient Monitoring and Assessment (GAMA) Program in the USGS California Water Science Center. The methods were subsequently published in U.S. Geological Survey Techniques and Methods book 5, Chap. B12 (Rose and others, 2016). Data collected during the implementation phase have provided extensive testing to establish performance and stability of the methods over time. This memorandum, TM-2017.01, officially approves the coding change from “U”

(research or unapproved method or laboratory) to “S” (provisional data) for data from NWQL Methods O-4437-16 and O-4436-16 collected during the implementation phase. This memo and NWQL Rapi-Note 16-28 provide guidance for appropriate National Water Information System (NWIS) coding, documentation, and publication of the data. Final review and approval of those data will occur at the Program or Water Science Center level after review of field quality-control information.

POLICY

OWQ Policy Memorandum 04.01 provides policy and procedures for approval and release of water-quality results that were produced from unapproved methods. Data from unapproved methods that are entered into NWIS are coded as “U” and are restricted from the public until further review. If the performance of the unapproved method is stable over time, in this case from 2012 to 2016 for custom schedules 4437 and 4436, and those data are comparable to data produced by the approved method, then they may be recoded to “S.” The provisional data may be further examined with respect to field quality control by the Program or Water Science Center, and if they are approved, they may be coded 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 USGS Methods O-4436-16 and O-4437-16 from February 12, 2012 through May 10, 2016. They concluded that data quality has been consistent, comparable, and method performance has been stable over this time period. Continuing calibration verification standards (CCV) prepared by NWQL were compared between the method validation and method implementation periods, and there were relatively small differences in median recovery and variability of the VOC compounds. Based on this careful analysis, data produced by these methods on or before May 10, 2016 are considered suitable to be considered for inclusion in USGS data and interpretive reports without a full description of or reference to the approved method.

With the exception of five analytes that will remain with a Data-Quality Indicator (DQI) code of “U” due to poor method performance (Table 1), the NWQL concludes, and the OWQ concurs, that the data quality support a change of the DQI code from “U” (unapproved method) to a code of “S” (provisional method) for results in NWIS from NWQL Methods O-4437-16 and O-4436-16 that were released prior to approval of the method, during the time period February 12, 2012 to May 10, 2016.

Table 1. Parameters from USGS Methods O-4437-16 and O-4436-16 that are not approved for release to the public and will remain coded as unapproved method, DQI code "U".

[NWQL, National Water-Quality Laboratory; NWIS DQI, National Water-Information System data-quality-indicator code; LS, NWQL laboratory schedule; U, DQI code for research or unapproved method]

Compound	USGS Parameter Code	NWQL Method Code	Dates that data were released to NWIS	NWIS DQI code
Method O-4437-16, LS4437				
Acrolein	34210	GCM016	1/1/2012 – 4/1/2016	U
Method O-4436-16, LS4436 and LS4440				
1,2-Dimethylnaphthalene	68725	GCM66	1/1/2012 – 5/1/2014	U
1,6-Dimethylnapthalene	68727	GCM66	1/1/2012 – 5/1/2014	U
2,6-Di-*tert*-butyl phenol	61701	GCM66	1/1/2012 – 5/1/2014	U
2-Chloronaphthalene	34581	GCM66	1/1/2012 – 5/1/2014	U

The National Water Quality Laboratory (NWQL) is performing a reload of approximately 150,000 results to update the data-quality-indicator (DQI) codes for 50 compounds (48 analytes and two surrogates) analyzed during the development of U.S. Geological Survey (USGS) Methods O-4437-16 and O-4436-16 (Rose and others, 2016, NWQL Rapi-Note 16-28). The DQI for these compounds will be updated from "research or unapproved method or laboratory" to "provisional." This update enables the release of data to the public. After the NWQL reload of these data, Water Science Centers must review their data in conjunction with their quality-control results and determine the correct DQIs to set for final approval in NWIS.

Several compounds listed in Tables A1 and A2 had recoveries that were consistently outside of acceptable control limits established by NWQL, but were determined to be adequate for recoding to "S" with qualifiers on the reported values. Qualifiers included adding a Remark Code of "E" or revising the reporting level. Details on changes made to specific analytes during the implementation phase of the method, including analytes that were deleted early in the performance-testing phase, changes to parameter codes, and additions of surrogate compounds are provided in Appendixes B1 and B2. These changes have already been made in the NWQL Laboratory Information Management System (LIMS) system and have been reloaded into NWIS databases; no further action on these updates is needed by Water Science Centers, other than resetting the DQI codes once their data are reviewed and approved or rejected at the Water Science Center or Program level.

Supporting information is available in summary documents and data tables that are available at this internal NWQL website (http://wwwnwql.cr.usgs.gov/USGS/sh443x.shtml). Information for USGS employees, such as any changes in detection levels, analytes dropped, improved recoveries of individual compounds over time, or other considerations are provided at this website and in Appendixes B1 and B2. This guidance is for purposes of data review in NWIS and publication in other USGS information products.

REFERENCES

Connor, B.F., Rose, D.L., Noriega, M.C., Murtagh, L.K., and Abney, S.R., 1998, Methods of analysis by the U.S. Geological Survey National Water-Quality Laboratory – determination of 86 volatile organic compounds in water by gas chromatography/mass spectrometry, including detections less than reporting limits, U.S. Geological Survey Open-File Report 97-829, 78 p., accessible at http://wwwnwql.cr.usgs.gov/USGS/rpt.shtml?OFR-97-829.

National Water-Quality Laboratory Rapi-Note 16-02, Data reload to correct surrogate parameter codes for laboratory schedules 4436, 4437, 4439, 4440, and 4443, dated March 3, 2016, accessible at http://wwwnwql.cr.usgs.gov/rn.shtml?16-02.

National Water-Quality Laboratory Rapi-Note 16-05, Update to Rapi-Note 16-02—Reload files will be available by 16 March to correct surrogate parameter codes (Laboratory Schedules 4436, 4437, 4439, 4440, and 4443), dated March 11, 2016, accessible at http://wwwnwql.cr.usgs.gov/rn.shtml?16-05.

National Water-Quality Laboratory Rapi-Note 16-11, New methods available to determine volatile organic compounds (VOCs) using purge-and-trap gas chromatography / mass spectrometry (GC/MS) for the analysis of 37 heat purgeable and 49 ambient purgeable VOCs, dated June 1, 2016, accessible at http://wwwnwql.cr.usgs.gov/rn.shtml?16-11.

National Water-Quality Laboratory Rapi-Note 16-17, Release of data for surrogate compound tetrahydrofuran-d_8 for NWQL LS 4437, heat purgeable volatile organic compounds, dated July 11, 2016, accessible at http://wwwnwql.cr.usgs.gov/USGS/rn.shtml?16-17.

National Water-Quality Laboratory Rapi-Note 16-28, Data reload for approved methods of purgeable volatile organic compounds (VOCs) determined using U.S Geological Survey Methods O-4437-16 and O-4436-16, dated October 24, 2017, accessible at http://wwwnwql.cr.usgs.gov/rn.shtml?16-28.

Office of Water Quality Method Approval Memorandum 30, Approval of new USGS methods: determination of heat purgeable (Method O-4437-16) and ambient purgeable (Method O-4436-16) volatile organic compounds in water by gas chromatography/mass spectrometry, dated May 10, 2016, accessible at http://water.usgs.gov/usgs/owq/Analytical_Methods/2016-05-10_PurgeableVOCsInWaters.pdf.

Office of Water Quality Technical Memorandum 04.01, Revised Policy for the Approval of U.S. Geological Survey (USGS) Water-Quality analytical methods, dated February 11, 2004, accessible at http://water.usgs.gov/admin/memo/QW/qw04.01.html.

Olsen, L.D., Valder, J.F., Carter, J.M., and Zogorski, J.S., 2013, Prioritization of constituents for national- and regional-scale ambient monitoring of water and sediment in the United States: U.S. Geological Survey Scientific Investigations Report 2012–5218, 203 p., plus supplemental tables, accessible at http://pubs.usgs.gov/sir/2012/5218.

Rose, D.L., Sandstrom, M.W., and Murtagh, L.K., 2016, Determination of heat purgeable and ambient purgeable volatile organic compounds in water by gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods, book 5, chap. B12, 62 p., accessible at http://dx.doi.org/10.3133/tm5B12.

Donna N. Myers
Chief, Office of Water Quality

Distribution: All WMA Employees

Appendix A – Coding information for analytes in VOC Methods O-4437-16 and O-4436-16

Table A1. Heat-purgeable volatile organic compounds determined by Method O-4437-16, simultaneous full scan/selected-ion-monitoring mass spectrometry.

[Validation quality codes: A, acceptable, no remark code qualification; E, acceptable with estimated "E" remark code (**in bold**); CASRN, Chemical Abstracts Service Registry Number; NTAS, National Target Analyte Strategy; --, not applicable]

Validation Quality Code	Compound	CASRN[1]	Parameter Code	Method Code	NTAS tier[2,3]
	Heat-Purgeable Volatile Organic Compounds				
A	1,1-Dichloro-2-propanone	513-88-2	80336	GCM016	1
A	1,2,3-Trichloropropane	96-18-4	77443	GCM016	1–U
A	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	82625	GCM016	1–R
A	1,2-Dibromoethane (EDB)	106-93-4	77651	GCM016	1–R
A	1,2-Dichloropropane	78-87-5	34541	GCM016	1–R
A	1,3-Dioxolane	646-06-0	81583	GCM016	1
A	1,4-Dioxane	123-91-1	81582	GCM016	1–U
A	1-Butanol	71-36-3	77034	GCM016	1–U
A	1-Methoxy-4 (2-propenyl) benzene	140-67-0	68066	GCM016	1
A	1-Octanol	111-87-5	77310	GCM016	1
A	2,6-Dimethyl-4-heptanone	108-83-8	77419	GCM016	1
A	2-Ethoxyethyl acetate	111-15-9	68728	GCM016	1
A	2-Ethyl-1-hexanol	104-76-7	77311	GCM016	1
A	2-Methylpropyl acetate	110-19-0	77201	GCM016	1
A	2-Nitropropane	79-46-9	77076	GCM016	1
A	2-Propen-1-ol	107-18-6	68729	GCM016	1–U
A	4-Methyl-2-pentanol	108-11-2	77113	GCM016	1
A	5-Methyl-2-hexanone	110-12-3	77179	GCM016	1
A	Acetonitrile	75-05-8	76997	GCM016	1
A	*alpha*-Terpineol	98-55-5	68730	GCM016	1
A	Butanal	123-72-8	68732	GCM016	1
A	Chloropicrin	76-06-2	77548	GCM016	1
A	Cyclohexanone	108-94-1	77097	GCM016	1
A	Dimethoxymethane	109-87-5	81578	GCM016	1
A	Ethyl acetate	141-78-6	81585	GCM016	1
E	**Hexachlorocyclopentadiene**	77-47-4	34386	GCM016	1
A	Isophorone	78-59-1	34408	GCM016	1
A	Isopropyl acetate	108-21-4	45013	GCM016	1
A	Isopropyl alcohol	67-63-0	77015	GCM016	1
A	Methyl acetate	79-20-9	77032	GCM016	1
A	Nitrobenzene	98-95-3	34447	GCM016	1–U
A	N-Nitrosodiethylamine	55-18-5	78200	GCM016	1–U
A	*n*-Pentanal	110-62-3	77061	GCM016	1
A	Propyl acetate	109-60-4	45022	GCM016	1
A	*tert*-Butyl alcohol	75-65-0	77035	GCM016	1
A	*tert*-Butyl methyl ether (MTBE)	1634-04-4	78032	GCM016	1–U
A	*trans*-Crotonaldehyde	123-73-9	68733	GCM016	1

Surrogate-Standard Compounds					
A	1-Bromo-4-fluorobenzene	460-00-4	90701	GCM016	--
A	1,2-Dichloroethane-d_4	17060-07-0	99832	GCM016	--
A	Isobutyl alcohol-d_6	72182-69-5	62835	GCM016	--
A	Tetrahydrofuran-d_8	1693-74-9	90607	GCM016	--
A	Toluene-d_8	2037-26-5	90703	GCM016	--
Internal-Standard Compounds					
--	1-Bromo-3-chloropropane-d_6	1173018-46-6	--	GCM016	--
--	1,4-Dioxane-d_8	17647-74-4	--	GCM016	--
--	Fluorobenzene	462-06-6	--	GCM016	--
--	*tert*-Butyl alcohol-d_{10}	53001-22-2	--	GCM016	--

[1]This table contains CAS Registry Numbers®, which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS Client Services SM.
[2] For more information on the National Target Analyte Strategy (NTAS) tier ranking for analytes, refer to Olsen and others (2013)
[3] "–R" indicates a regulated U.S. Environmental Protection Agency (EPA) drinking water compound; "–U" indicates an unregulated compound on the EPA Contaminant Candidate List 3 (CCL 3) and (or) EPA Unregulated Contaminant Monitoring Rule 3 (UCMR 3).

Table A2. Ambient-purgeable volatile organic compounds determined by Method O-4436-16, full-scan mass spectrometry.

[Validation quality code: A, acceptable, no remark code qualification; E, acceptable with estimated "E" remark code (**in bold**); CASRN, Chemical Abstracts Service Registry Number; NTAS, National Target Analyte Strategy; --, not applicable]

Validation Quality Code	Compound	CASRN[1]	Parameter Code	Method Code	NTAS tier[2,3]
High-Priority Ambient-Purgeable Volatile Organic Compounds[4]					
A	1,1-Difluoroethane	75-37-6	49538	GCM66	1
A	1,2,3,4-Tetrahydronaphthalene	119-64-2	77323	GCM66	1
A	1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	50985	GCM66	1
A	1,3-Butadiene	106-99-0	68726	GCM66	1–U
A	1-Chloro-1,1-difluoroethane	75-68-3	85668	GCM66	1
A	2,2-Dichloro-1,1,1-trifluoroethane	306-83-2	62174	GCM66	1
A	Butane	106-97-8	81563	GCM66	1
A	Chlorodifluoromethane	75-45-6	45028	GCM66	1–U
A	Dichlorofluoromethane	75-43-4	77119	GCM66	--
E	**Hexane**	110-54-3	81590	GCM66	1–U
A	Pentane	109-66-0	81604	GCM66	1
Ambient-Purgeable Volatile Organic Compounds					
A	1,1,1,2-Tetrachloroethane	630-20-6	77562	GCM66	2–U
A	1,1,1-Trichloroethane	71-55-6	34506	GCM66	1–R
A	1,1,2-Trichloroethane	79-00-5	34511	GCM66	2–R
A	1,1-Dichloroethane	75-34-3	34496	GCM66	1–U
A	1,1-Dichloroethene	75-35-4	34501	GCM66	1–R
A	1,2,4-Trichlorobenzene	120-82-1	34551	GCM66	2–R
A	1,2,4-Trimethylbenzene	95-63-6	77222	GCM66	1
A	1,2-Dichlorobenzene	95-50-1	34536	GCM66	2–R
A	1,2-Dichloroethane	107-06-2	32103	GCM66	2–R
A	1,4-Dichlorobenzene	106-46-7	34571	GCM66	2–R
A	Benzene	71-43-2	34030	GCM66	1–R
A	Bromochloromethane	74-97-5	77297	GCM66	2–U
A	Bromodichloromethane	75-27-4	32101	GCM66	1–R
A	Bromoform	75-25-2	32104	GCM66	1–R
E	**Bromomethane**	74-83-9	34413	GCM66	1–U
E	**Carbon disulfide**	75-15-0	77041	GCM66	1
A	Chlorobenzene	108-90-7	34301	GCM66	2–R
A	Chloroform	67-66-3	32106	GCM66	1–R
E	**Chloromethane**	74-87-3	34418	GCM66	1–U
A	*cis*-1,2-Dichloroethene	156-59-2	77093	GCM66	1–R
A	*cis*-1,3-Dichloropropene	10061-01-5	34704	GCM66	1

A	Dibromochloromethane	124-48-1	32105	GCM66	1–R
A	Ethylbenzene	100-41-4	34371	GCM66	2–R
A	*meta*- and *para*-Xylene	179601-23-1	85795	GCM66	1
A	Methylene chloride	75-09-2	34423	GCM66	1–R
A	Naphthalene	91-20-3	34696	GCM66	1
A	*n*-Propylbenzene	103-65-1	77224	GCM66	2–U
A	*ortho*-Xylene	95-47-6	77135	GCM66	1
A	*sec*-Butylbenzene	135-98-8	77350	GCM66	2–U
A	Styrene	100-42-5	77128	GCM66	2–R
A	*tert*-Butyl methyl ether	1634-04-4	78032	GCM66	1–U
A	Tetrachloroethene	127-18-4	34475	GCM66	1–R
A	Tetrachloromethane	56-23-5	32102	GCM66	1–R
A	Toluene	108-88-3	34010	GCM66	1–R
A	*trans*-1,2-Dichloroethene	156-60-5	34546	GCM66	2–R
A	*trans*-1,3-Dichloropropene	10061-02-6	34699	GCM66	1
A	Trichloroethene	79-01-6	39180	GCM66	1–R
A	Vinyl chloride	75-01-4	39175	GCM66	1–R
Surrogate-Standard Compounds					
A	1-Bromo-3-chloropropane-d_6	1173018-46-6	90576	GCM66	--
A	1-Bromo-4-fluorobenzene	460-00-4	90695	GCM66	--
A	Toluene-d_8	2037-26-5	90696	GCM66	--
Internal-Standard Compounds					
--	1,2-Dichloroethane-d_4	17060-07-0	--	GCM66	--
--	Fluorobenzene	462-06-6	--	GCM66	--

[1]This table contains CAS Registry Numbers®, which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS Client Services SM.
[2] For more information on the USGS National Target Analyte Strategy (NTAS) Tier ranking, refer to Olsen and others, 2013.
[3] "–R" indicates a regulated U.S. Environmental Protection Agency (EPA) drinking-water compound; "–U" indicates an unregulated compound on the EPA Contaminant Candidate List 3 (CCL 3) and (or) EPA Unregulated Contaminant Monitoring Rule 3 (UCMR 3).
[4] High-priority VOCs are ranked as Tier 1 compounds by the NTAS team. High-priority VOCs have not been routinely monitored as targeted compounds on USGS VOC methods.

Appendix B – Timeline for qualifying analytes that did not meet quality criteria or were updated in the USGS NWIS databases.

Table B1 – Heat-Purgeable Compounds, Method O-4437-16, NWQL Custom Laboratory Schedule 4437 and sub-schedule 4443[1]. Thirty-seven compounds were approved for heat-purgeable Method O-4437-16 after being implemented as NWQL custom laboratory schedules 4437 and 4443 for testing. This table summarizes the history of important changes that were made for specific analytes during method development and implementation.

[NWQL, National Water-Quality Laboratory; LS, laboratory schedule; CASRN, Chemical Abstracts Service Registry Number; "E", estimated remark code; NWIS, National Water-Information System; DQI, data-quality-indicator code]

Compound	CASRN	Parameter Code	Method Code	Testing Start Date	Testing End Date	Action	Comments
Compounds Tested with No Results Submitted to NWIS Databases							
2-Chloromethyl oxirane	106-89-8	77084	--	--	--	Tested and dropped during initial method development	These three compounds were dropped from LS4437 during method development due to incompatibility with preservation with hydrochloric acid. No data were submitted to NWIS databases for these compounds.
Methyl oxirane	75-56-9	77011	--	--	--		
Oxirane	75-21-8	76999	--	--	--		
Compounds Tested with Results Submitted to NWIS Databases and Subsequently Deleted from the Method							
Acrolein	107-02-8	34210	GCM016	1/1/2012	4/1/2016	Deleted After 4/1/2016; Prior data remain coded as "U"	Acrolein was deleted from LS4437 due to instability within the prescribed 14-day holding time (tables 15, 16, Rose and others, 2016). Data were submitted to NWIS databases during the testing phase with a remark code of "E", but the DQI code will remain "U" for all data for this analyte and method.
Compounds Reported with an Estimated "E" Remark-Qualifier Code							
Hexachlorocylcopentadiene	77-47-4	34386	GCM016	1/1/2012	--	Estimated	Hexachlorocyclopentadiene is permanently flagged with a remark code of "E", due to high variability observed during method development. Calibration for this compound is biased by the presence of sulfide, resulting in lowered recoveries.
Compounds Reported with a Revised Minimum Reporting Level (MRL)							
2-Ethyl-1-hexanol	104-76-7	77311	GCM016	1/1/2012	--	MRL <2 µg/L	2-Ethyl-1-hexanol was consistently detected in laboratory reagent-water blanks (LRBs), potentially due to contamination from sample-bottle septa. A minimum reporting level of <2 was calculated from the statistical analysis of 84 LRBs (table 20, Rose and others, 2016).

Fumigant Compounds							
1,2-Dichloropropane	78-87-5	34541	GCM016	1/1/2012	--	Compounds tested from ambient-purge method	Four fumigant compounds from LS2020 were included for testing on LS4437 to achieve lower reporting levels. LS4437 (including sub-schedule 4443) is the preferred method for these compounds. The four compounds are still on LS2020 for projects that do not require lower reporting levels. LS4437 and LS4443 also are the preferred method to LS1306 for EDB and DBCP.
1,2-Dibromoethane (EDB)	106-93-4	77651	GCM016	1/1/2012	--		
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	82625	GCM016	1/1/2012	--		
1,2,3-Trichloropropane	96-18-4	77443	GCM016	1/1/2012	--		
Surrogate-Standard Compounds							
Tetrahydrofuran-d_8 (THF-d_8)	1693-74-9	90607	GCM016	1/25/2013	--	Surrogate added to the method	THF-d_8 was added to LS4437 as a surrogate compound in January 2013, assigned to internal standard 1,4-dioxane-d_8. Initially data for this surrogate were not released until enough data were available to determine if the surrogate performed within method specifications. The release flag was changed in December 2015. All data have been released (NWQL Rapi-Note 16-17) for samples received from January 2013 to December 2015.
1-Bromo-4-fluorobenzene (BFB)	460-00-4	90701	GCM016	1/1/12	--	Data reloaded with new parameter code	The original parameter code for surrogate standard compound BFB in LS4437 and LS4443was reported as 99834 from January 1, 2012 to July 10, 2014. All data for BFB in LS4437 and LS4443 have been retroactively recoded to parameter code 90701 and have been reloaded into NWIS (NWQL Rapi-Notes 16-02 and 16-05).
Toluene-d_8	2037-26-5	90703	GCM016	1/1/12	--	Data reloaded with new parameter code	The original parameter code for surrogate-standard compound toluene-d_8 in LS4437 and LS4443 was reported as 99833 from January 1, 2012 to July 10, 2014. All data for Toluene-d_8 in LS4437 and LS4443 have been retroactively recoded to parameter code 90703 and have been reloaded into NWIS databases (NWQL Rapi-Notes 16-02 and 16-05).

[1]NWQL LS4443 is a subset of LS4437. Data for LS4443 are reported only for the fumigant compounds: 1,2-dichloropropane, EDB, DBCP, 1,2,3-trichloropropane, and chloropicrin.

Table B2 – Ambient-purgeable volatile organic compounds, Method O-4436, NWQL Custom Laboratory Schedules 4436, 4440[1], and 4439[2]. Forty-nine compounds were approved for ambient-purgeable Method O-4436, after being implemented during the testing phase as custom LS4436 and LS4439. This table summarizes the history of important changes for specific analytes during method development and implementation.

[NWQL, National Water Quality Laboratory; LS, laboratory schedule; CASRN, Chemical Abstracts Service Registry Number; EPA, Environmental Protection Agency; "E", estimated remark code; DQI, data quality indicator code; MRL, minimum reporting level]

Compound	CAS Number	Parameter Code	Method Code	Start Date	End Date	Action	Comments
Compounds Tested with No Results Submitted to NWIS Databases							
Benzyl chloride	100-44-7	68731	--	--	--	Dropped during initial method development	Benzyl chloride was deleted during initial method development due to instability over the holding period (table 17, Rose and others, 2016). No data were submitted to NWIS databases. (table 30, Rose and other, 2016).
Compounds Tested with Results Submitted to NWIS Databases and Subsequently Deleted from the Method							
1,2-Dimethylnaphthalene	573-98-8	68725	GCM66	1/1/2012	5/1/2014	Deleted After 5/1/2014; Prior data remain coded as "U"	After initial testing, these compounds did not perform within quality criteria. Initial method detection limits were determined to not be representative of long-term performance, and data were qualified and (or) the reporting level was raised in datasets where identification criteria failed. Data were submitted to NWIS prior to deletion of these compounds from the method on 5/1/2014. The DQI codes will remain "U" for these compounds in Method O-4436. (tables 12, 14, 26, 27, Rose and others, 2016).
1,6-Dimethylnapthalene	575-43-9	68727	GCM66	1/1/2012	5/1/2014		
2,6-Di-*tert*-butyl phenol	128-39-2	61701	GCM66	1/1/2012	5/1/2014		
2-Chloronaphthalene	91-58-7	34581	GCM66	1/1/2012	5/1/2014		
Compounds Reported with an Estimated "E" Remark Code							
Hexane	110-54-3	81590	GCM66	1/1/2012	--	Estimated	Hexane is reported with an estimated remark code due to lower recoveries observed in the holding time study (tables 17, 18, 30, Rose and others, 2016). Hexane is retained in the method until more data can be collected. Hexane may be deleted at a later date if further studies indicate it is not suitable for this method.
Bromomethane	74-83-9	34413	GCM66	1/1/2012	--	Estimated	Bromomethane, Carbon disulfide, and chloromethane are reported as estimated compounds in LS2020, and will continue to be reported as estimated compounds in LS4436 and LS4440. All 3 compounds also show higher variability in the presence of sulfide.
Carbon disulfide	75-15-0	77041	GCM66	1/1/2012	--	Estimated	
Chloromethane	74-87-3	34418	GCM66	1/1/2012	--	Estimated	

Compounds Reported with a Minimum Reporting Level (MRL)							
Chloromethane	74-87-3	34418	GCM66	1/1/2012	--	MRL <0.1 µg/L	Chloromethane, methylene chloride, and toluene are MRL compounds on LS2020, LS4436, and LS4440.
Methylene chloride	75-09-2	34423	GCM66	1/1/2012	--	MRL <0.04 µg/L	
Toluene	108-88-3	34010	GCM66	1/1/2012	--	MRL <0.02 or <0.025 µg/L	
Surrogate-Standard Compounds							
1-Bromo-4-fluorobenzene (BFB)	460-00-4	90695	GCM66	7/10/2014	--	Data reloaded with new parameter code	The original parameter code for BFB in LS4436, LS4439, and LS4440 was reported as 99834 from January 1, 2012 to July 10, 2014. All data for BFB in LS4436, LS4439, and LS4440 have been retroactively recoded to parameter code 90695 and have been reloaded into NWIS databases (NWQL Rapi-Note 16-02).
Toluene-d_8	2037-26-5	90696	GCM66	7/10/2014	--	Data reloaded with new parameter code	The original parameter code for surrogate toluene-d_8 in LS4436, LS4439, and LS4440 was reported as 99833 from January 1, 2012 to July 10, 2014. All data for Toluene-d_8 in LS4436, LS4439, and LS4440 have been retroactively recoded to parameter code 90696 and have been reloaded into NWIS (NWQL Rapi-Note 16-02).

[1] NWQL LS4440, which contains all of the compounds on LS2020 and LS4436, was created for internal quality-assurance/quality-control purposes. It has been used as a custom schedule by the USGS Toxics Hydrology Program. Documentation for LS2020 is in OFR 97-829 (Connor and others, 1998). LS4440 reports data for 100 compounds, including tentatively identified compounds, and surrogate compounds.

[2] NWQL LS4439 is a subset of schedule 4436. Data in LS4439 are reported only for benzene, toluene, ethylbenzene, meta-, para-, and ortho-xylene (BTEX), and tert-butyl methyl ether..