United States Department of the Interior

U.S. GEOLOGICAL SURVEY
National Water Quality Laboratory
P.O. Box 25585
Denver, CO 80225-0585

NATIONAL WATER QUALITY LABORATORY
TECHNICAL MEMORANDUM 2016.02

August 19, 2016

Subject: Changes to detection levels, reporting levels, and data-reporting conventions for steroid hormones and other analytes in water, solids, and suspended sediment for National Water Quality Laboratory schedules 2434, 4434, 6434, and 7434

Effective: June 10, 2016

Authors: William T. Foreman, Method Research and Development Program, NWQL, wforeman@usgs.gov, 303-236-3942
Patricia L. Toccalino, Regional Program Officer, Western Region, National Water Quality Program, Sacramento, Calif., ptocca@usgs.gov, 916-278-3090
Rhiannon C. ReVello, Analytical Services, NWQL, rcrevell@usgs.gov, 303-236-3258

Revision: Not applicable

PURPOSE

This memorandum describes changes to the detection levels (DLs) and reporting levels (RLs) for selected analytes and to the data-reporting conventions for many analytes for U.S. Geological Survey (USGS) National Water Quality Laboratory (NWQL) methods for steroid hormones, including methods requested via laboratory schedule (LS) 2434 (filtered water), LS 4434 (unfiltered water), LS 6434 (solids), and LS 7434 (suspended sediment on a glass-fiber filter). It also announces the need to reload selected sample data from LS 2434 and LS 4434 into the National Water Information System (NWIS) for samples collected in calendar years 2013–2015.

BACKGROUND

The NWQL has developed methods for steroid hormones and other analytes[1] in various matrices (collectively referred to as the hormone methods). The NWQL implemented LS 2434 (filtered water) and LS 4434 (unfiltered water) as official USGS methods on May 31, 2012. These two schedules are identical in all sample preparation and instrumental analysis procedures, differing only in whether the water sample

[1] Analytes are the chemicals specifically targeted for analysis by these methods and do not include surrogate or isotope dilution standard compounds also determined by the methods.

is filtered (preferably in the field) or unfiltered (Foreman and others, 2012). Additional research methods have been available via custom analysis request for hormones in solids (LS 6434) and suspended sediment collected onto a glass fiber filter (LS 7434). These two research methods likewise have identical sample preparation steps and differ only in the applied matrix.

In 2015–early 2016, the National Water-Quality Assessment (NAWQA) Project and the NWQL conducted an examination of quality assurance and quality control (QA/QC) data from LS 2434 as part of a review of results for nearly 900 groundwater samples collected by NAWQA in 2013–2014. This review focused primarily on laboratory and field-related blank results, and was expanded to include LS 4434. In addition, it included a review of false positive occurrence in spike samples submitted by the USGS Branch of Quality System's Organic Blind Sample Project (OBSP; see link to "False Positive and False Negative Data Table and Charts" for LS 4434 at https://bqs.usgs.gov/OBSP/ [internal USGS access only]).

The quality assurance and quality control assessment revealed two primary issues:

1. Laboratory contamination occurred during portions of 2013 and especially in 2014 (samples analyzed in late April through August 2014) for some analytes that may have resulted in low-level contamination (false positives) in some field samples. The potential for laboratory-derived contamination was implicated from this broader assessment of blank results, as contamination often was not indicated by results for the laboratory reagent-water blank (lab blank) sample that is prepared along with ten environmental samples for each sample-preparation set and used by the analyst during data review to censor or qualify environmental sample data. A summary of laboratory reagent-water blank sample results for LS 2434 / 4434 is shown in table 1.

2. Detection and reporting levels (reported using the interim reporting level [IRL] or minimum reporting level [MRL] conventions) were too low for some analytes relative to lab blank levels or based on instrumental response during routine analyses.

This memorandum discusses actions being taken by the NWQL to mitigate the contamination and its effects on how future data are reported, as well as the need for a data reload to mitigate the risk of false positives that may be attributable to laboratory contamination bias in previously reported data for field samples.

POTENTIAL CONTAMINATION SOURCES AND MITIGATION STEPS

Contamination occurrences were rare for most analytes, with none apparent for six analytes based on lab-blank results alone (table 1). Foreman and others (2012) previously identified bisphenol A, cholesterol, and 3*beta*-coprostanol as "blank-limited analytes" [defined here as analytes having a detection frequency of 20 percent (%) or more in lab blanks]. Although these three analytes have a high detection frequency in lab blanks (table 1), concentrations in the blanks typically were less than the applied MRL concentration, a censoring level below which measured concentrations are not reported in environmental samples.

Contamination sources have been or could be attributed to:

1. *Carryover on the gas chromatograph inlet from preceding calibration-related standards, laboratory spike samples, or environmental samples having high concentrations.* Inlet carryover was observed for several compounds that are most responsive during analysis by gas chromatography/tandem mass spectrometry, particularly for *trans*-diethylstilbestrol. Carryover is a recognized consideration in

chromatographic analyses, and various strategies are used by the NWQL and other laboratories to minimize inlet carryover risk. For example, matrices such as wastewater treatment plant effluent or influent samples that are suspected to have high concentrations of hormone method analytes are intentionally positioned near the end of the gas chromatographic (GC) analysis sequence to minimize inlet carryover risk to samples anticipated to have low concentrations (Foreman and others, 2012). The NWQL has been pursuing enhanced solutions with the manufacturer of the instrumentation used for many NWQL GC-based methods to further reduce the risk of inlet carryover issues, which are not exclusive to the hormone methods.

2. *The unintended fortification of a subset of samples with very high concentrations of bisphenol A-d_{16} isotope dilution standard, a deuterium-labeled analog of bisphenol A (BPA), resulting in detection of the unlabeled BPA analyte.* Unlabeled BPA is present at a very low (well below the detection level) concentration in BPA-d_{16} at the concentration of this isotope that is normally fortified into all samples. Nearly all BPA results for those samples affected by this issue previously were censored by the NWQL prior to initial sample data release (NWIS null-result value qualifier code "r" or "x" reported; these codes defined in Appendix A table 10 of the NWIS user's manual).

3. *Use of the same syringes to prepare the isotope dilution standard or internal injection standard solutions that contain no analytes that are used for preparation of the calibration-related and analyte fortification (laboratory spike) standard solutions that do contain the analytes.* Although these syringes are solvent cleaned between use, separate syringes are now used for preparation of analyte- and non-analyte-containing solutions to eliminate this potential contamination source.

4. *Possible carryover from inadequate cleaning of the extraction tubes, glassware, and other apparatus that are non-disposable in the methods (Foreman and others, 2012).* Lab blank results have not specifically implicated inadequate cleaning of these method components as a contamination source, as uniform carryover of most/all analytes was not observed in the lab blanks.

5. *Possible carryover of analytes for some LS 2434 samples that were filtered by the NWQL (via request of laboratory code 4200) from proceeding filtered samples (either intended for LS 2434 or other filtered-water schedules) that contained very high concentrations of method analytes, especially wastewater treatment plant influent or effluent samples that often contain relatively high concentrations of cholesterol and 3beta-coprostanol.* Laboratory filtration is a separate step from the routine LS 2434 / 4434 sample preparation procedures. Thus, the LS 2434 / 4434 lab blank does not include the filtration step, as laboratory filtration is not applicable for many LS 2434 and all LS 4434 samples. The NWQL is currently assessing the scope of carryover during laboratory filtration for LS 2434 and LS 1433 (waste indicators in filtered water).

DATA REPORTING CHANGES

To address these quality control issues and help reduce the risk of reporting false positive results in the future, the NWQL implemented the following data-reporting changes for LS 2434, LS 4434, LS 6434, and LS 7434 effective June 10, 2016 (based on a sample's instrumental analysis date; see NWQL Rapi-Note 15.23 [internal USGS access only]); these changes will not be implemented retroactively:

1. Detection and reporting levels are increased for selected analytes as shown in tables 2–4.

2. Determined concentrations that are less than the detection level are no longer being reported for those analytes having the IRL report-level type code in NWIS (tables 2–4). Those concentrations will be

censored with the reported result being the less than "<" NWIS remark code and the reporting level concentration (< IRL).

Note: This change supersedes the data-reporting convention that allows reporting results below the detection level in Section 12.2 of Foreman and others (2012).

3. *trans*-Diethylstilbestrol data are being reported using the MRL report-level type code instead of the IRL report-level type code. Following the data reporting convention for all analytes with the MRL report-level type code (tables 2–4), determined concentrations that are less than the MRL concentration will be censored and the reported result will be < MRL.

ADDITIONAL NWQL ACTIONS

Laboratory schedules 2434 and 4434

The NWQL has undertaken a comprehensive review of results from LS 2434 and LS 4434 to identify whether previously reported results for environmental samples might be influenced by or attributed to laboratory contamination. Based on this review, the NWQL will reload data for some analytes in some environmental water samples collected from calendar years 2013–2015. Affected USGS Water Science Centers will be informed by NWQL Rapi-Note of this reload upon completion of the data review. The NWQL anticipates completing this review by October 1, 2016.

The following are the most common censoring or data qualification actions related to those results that will be reloaded:

1. All previously reported detections for cholesterol below 400 nanograms per liter (ng/L) will be censored and reported as "less than" the reported value.

The 400-ng/L censoring threshold is the new MRL value for cholesterol (table 2) and is being applied as a conservative censoring threshold for previously reported environmental water sample results for this blank-limited analyte because:

a. Cholesterol was detected in all lab blanks analyzed since May 2012 (fig. 1, table 1).

b. Cholesterol concentrations were greater than the 200 ng/L MRL threshold originally applied in 3.5% of the lab blank samples. This frequency exceeds the 1% false positive risk frequency at a concentration at or above the detection level (see NWQL technical memorandum 2014.01), and indicates that a MRL of 200 ng/L was insufficient to minimize false positive risk to 1% or less during 2013–2015 for this blank-limited analyte (fig. 1). Cholesterol also was detected in two of 50 unspiked OBSP samples at concentrations (227, 285 ng/L) above the applied MRL of 200 ng/L (see https://bqs.usgs.gov/OBSP/WY16fc/Cumulative/False_SCHED4434.html).

c. Cholesterol concentrations in 30.9% of the lab blanks exceeded 66.7 ng/L, a concentration that is 33% of the previous 200-ng/L MRL censoring threshold (table 1). Instances of relatively higher cholesterol concentrations in lab blanks require more censoring or qualification of results ("v" result-level value qualifier code application; see below) for field samples having concentration just above the MRL of 200 ng/L compared to instances of lower lab blank concentrations. Application of the 400-ng/L censoring threshold helps ensure that laboratory contamination is not a substantial portion of a reported cholesterol concentration for those environmental samples that are at or just above this higher minimum reporting-level threshold.

2. Concentrations previously reported for some analytes will be unchanged, but the original reported concentration will now include the NWIS estimated value (E) remark code and the "v" result-level value-qualifier code to denote that the reported result in the environmental sample was between three and ten times the amount in the lab blank that was specifically prepared along with the environmental sample (which is commonly referred to as the "set" blank) or the lab blank from a separately prepared set of samples that also is included as a part of the instrumental analysis batch [the batch blank] (see Office of Water Quality technical memorandum 2012.01).

3. Results for some analytes will be censored because of suspected laboratory contamination or because the result is less than three times the amount in the environmental sample's associated set blank or in the batch blank.

   a. If the original reported result was less than the reporting level, then "less than" (<) reporting level will be the new result.

   b. If the original result exceeded the reporting level, but was within the concentration range of the suspected laboratory contamination, the original reported concentration will now include a "less than" (<) remark code (a raised reporting level condition) and include the "v" result-level value qualifier code to indicate that the analyte was detected in the lab blank (see Office of Water Quality technical memorandum 2012.01).

4. Based on the NAWQA / NWQL review of previously reported results, all data that were less than the reporting level applicable at the time of analysis are being conservatively censored to less than the reporting level for all NAWQA groundwater samples. Similar censoring might be applied to groundwater samples from other projects depending on the NWQL's review of results.

Laboratory schedule 6434 (solids)

A review of LS 6434 sample data is pending. Any need for a data reload for LS 6434 will be communicated separately as that review progresses. Changes to detection and reporting levels for analytes in LS 6434 are summarized in table 3.

Laboratory schedule 7434 (suspended solids on filter)

No data updates are required for the few samples submitted for this schedule. Changes to detection and reporting levels for analytes in LS 7434 are summarized in table 4.

ADDITIONAL DATA REPORTING INFORMATION AND QUALITY CONTROL DATA ACCESS

The NWQL's laboratory reporting level (NWIS report-level type code LRL), and identical interim reporting level (IRL), data reporting conventions are given in Childress and others (1999) and in Office of Water Quality technical memorandum 2010.07. Analytes coded with an IRL report-level type code have detection levels that were estimated using a procedure other than the long-term method detection level procedure described in Childress and others (1999).

NWIS data reporting codes are defined in Appendix A of the NWIS user's manual. The data-reporting conventions for analytes that use the minimum reporting level convention (MRL report level type code), along with the reporting conventions for analytes having the newer NWIS report-level type codes DLDQC, DLBLK, RLDQC, and RLBLK are defined and described in NWQL technical memorandum 2015.02.

Historical detection and reporting level information for NWQL methods are available at http://nwql.cr.usgs.gov/usgs/limits/limits.cfm (USGS access only).

USGS personnel can access laboratory blank, spike, and other QA/QC data for NWQL methods via the Online NWQL QC Data portal (internal USGS access only); this information is available to non-USGS personnel by request at labhelp@usgs.gov.

Note: Queries of laboratory reagent-water blank and spike sample data for LS 2434 and LS 4434 using the Online NWQL QC Data portal require entry of "2434" as the schedule number because these QC sample types are grouped within the NWQL's database under LS 2434 only. However, these QC sample results are applicable to environmental samples prepared by LS 2434 or LS 4434 as all preparation and analysis steps are identical.

REFERENCES

Childress, C.J.O., Foreman, W.T., Connor, B.F., and Maloney, T.J., 1999, New reporting procedures based on long-term method detection levels and some considerations for interpretations of water-quality data provided by the U.S. Geological Survey National Water Quality Laboratory: U.S. Geological Survey Open-File Report 99-193, 19 p. Available at *http://water.usgs.gov/owq/OFR_99-193/index.html*.

Foreman, W.T., Gray, J.L., ReVello, R.C., Lindley, C.E., Losche, S.A., and Barber, L.B., 2012, Determination of steroid hormones and related compounds in filtered and unfiltered water by solid-phase extraction, derivatization, and gas chromatography with tandem mass spectrometry: U.S. Geological Survey Techniques and Methods, book 5, chap. B9, 118 p. Available at *http://pubs.usgs.gov/tm/5b9/*.

National Water Quality Laboratory online NWQL QC data. Available at *http://nwqlqc.cr.usgs.gov* (internal USGS access only).

National Water Quality Laboratory technical memorandum 2014.01, Reduction in the use of sample-weight-based scaling of reporting levels for National Water Quality Laboratory methods for organics in solids and sediments. Available at *http://nwql.usgs.gov/Public/tech_memos/nwql.2014-01.pdf*.

National Water Quality Laboratory technical memorandum 2015.02, Changes to National Water Quality Laboratory (NWQL) procedures used to establish and verify laboratory detection and reporting limits. Available at *http://nwql.usgs.gov/Public/tech_memos/nwql.2015-02.pdf*.

National Water Quality Laboratory Rapi-Note 15-23, Effective 20 October analytical results will be reported based upon analysis date. Available at *http://wwwnwql.cr.usgs.gov/USGS/rn.shtml?15-23*.

Office of Water Quality technical memorandum 2010.07, Changes to the reporting convention and to data qualification approaches for selected analyte results reported by the National Water Quality Laboratory (NWQL). Available at *http://water.usgs.gov/admin/memo/QW/qw10.07.html*.

Office of Water Quality technical memorandum 2012.01, Application of the result-level ‘v’ value qualifier code and ‘E’ remark code to selected organic results reported by the National Water Quality Laboratory (NWQL). Available at *http://water.usgs.gov/admin/memo/QW/qw12.01.pdf*.

U.S. Geological Survey, User’s manual for the National Water Information System of the U.S. Geological Survey, Water Quality System, version 5.2. Available at *http://nwis.usgs.gov/currentdocs/qw/QW.user.book.html*.

FIGURE AND TABLES

Figure 1. Concentrations of cholesterol in 376 laboratory reagent-water (set) blank samples analyzed from May 31, 2015 to March 18, 2016 for laboratory schedules 2434 and 4434 compared with the original (200 ng/L) and revised (400 ng/L) minimum reporting level (MRL) concentrations.

Table 1. Summary of detections of analytes in laboratory reagent-water (set) blank samples analyzed between May 31, 2012 and March 18, 2016 for laboratory schedules 2434 and 4434.

Table 2. Changes to detection levels, reporting levels, or report-level type codes for analytes in laboratory schedules 2434 and 4434.

Table 3. Changes to detection and reporting levels for analytes in laboratory schedule 6434.

Table 4. Changes to detection and reporting levels for analytes in laboratory schedule 7434.

David W. Reppert

/signed/
David W. Reppert, Assistant Chief
National Water Quality Laboratory
Branch of Analytical Services

Supersedes: The data reporting convention that allows reporting of results below the detection level in section 12.2 of Foreman and others (2012) is superseded by this technical memorandum.

Key words: blank, code, contamination, detection level, IRL, MRL NWIS, reporting level

Distribution: by Rapi-Note announcement
posted to http://wwwnwql.cr.usgs.gov/USGS (the NWQL USGS-visible intranet; internal USGS access only) and http://nwql.usgs.gov/Public (the NWQL public internet)

Any use of trade, product, or firm names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

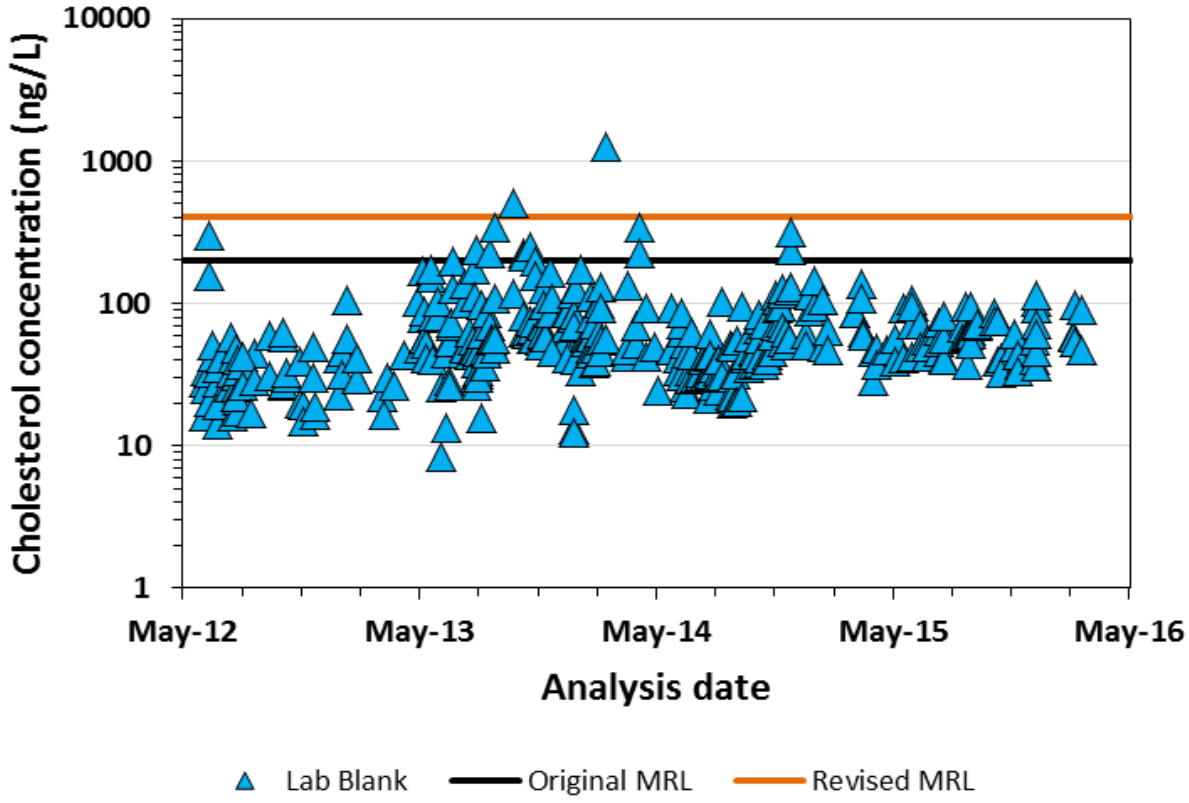

Figure 1. Concentrations of cholesterol in 376 laboratory reagent-water (set) blank samples analyzed from May 31, 2015 to March 18, 2016 for laboratory schedules 2434 and 4434 compared with the original (200 ng/L) and revised (400 ng/L) minimum reporting level (MRL) concentrations.

Table 1. Summary of detections of analytes in laboratory reagent-water (set) blank samples analyzed between May 31, 2012 and March 18, 2016 for laboratory schedules 2434 and 4434.[a]

[%, percent; ng/L, nanograms per liter; DL, detection level; RL, reporting level; #, number; ≥, greater than or equal to; Std dev, standard deviation; IRL, interim reporting level; --, not shown because no detections; NA, not applicable; MRL, minimum reporting level; ND, analyte not detected at or less than this percentile level; NC, not calculated because of insufficient number of detections; LS, laboratory schedule; USGS, U.S. Geological Survey; NWQL, National Water Quality Laboratory]

	Applied (ng/L)				Detection frequency (%)					Laboratory reagent-water blank concentration (ng/L)								
Analyte[b]	DL	RL	RL type	# of lab blanks[c]	All detections	≥10% of RL	≥33% of RL	≥50% of RL [or ≥DL][d]	≥RL	Maximum	2nd highest value	99th percentile	75th percentile	Median	25th percentile	Minimum	Mean	Std dev
4-Androstene-3,17-dione	0.4	0.8	IRL	376	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
cis-Androsterone	0.4	0.8	IRL	376	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
Bisphenol A	NA	100	MRL	369	65.0	4.6	1.9	1.6	1.1	161	159	84.5	3.6	1.8	ND	ND	4.2	14.8
Bisphenol F	NA	10	MRL	145	11.0	1.4	0.0	0.0	0.0	3.3	2.3	1.7	ND	ND	ND	ND	NC	NC
Cholesterol	NA	200	MRL	376	100	94.9	30.9	14.4	3.5	1260	509	320	75.0	50.5	36.9	8.4	68.7	81.7
3*beta*-Coprostanol	NA	200	MRL	376	94.4	42.6	0.5	0.3	0.0	138	99.2	39.8	23.0	19.0	15.0	0.0	19.1	10.6
trans-Diethylstilbestrol	0.4	0.8	IRL	377	3.4	3.4	2.9	2.4	1.9	2.6	2.5	1.3	ND	ND	ND	ND	NC	NC
Dihydrotestosterone	2	4	IRL	376	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
Epitestosterone	1	2	IRL	377	1.1	1.1	1.1	0.5	0.0	1.4	1.0	0.2	ND	ND	ND	ND	NC	NC
Equilenin	1	2	IRL	377	0.5	0.5	0.5	0.0	0.0	0.95	0.90	ND	ND	ND	ND	ND	NC	NC
Equilin	4	8	IRL	376	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
17*alpha*-Estradiol	0.4	0.8	IRL	377	2.7	2.7	2.4	2.4	1.1	6.4	2.2	0.9	ND	ND	ND	ND	NC	NC
17*beta*-Estradiol	0.4	0.8	IRL	377	2.4	2.4	2.1	2.1	1.9	4.4	4.1	1.2	ND	ND	ND	ND	NC	NC
Estriol	1	2	IRL	375	3.2	3.2	3.2	3.2	2.1	7.4	5.7	3.2	ND	ND	ND	ND	NC	NC
Estrone	0.4	0.8	IRL	372	3.0	3.0	3.0	2.7	2.4	5.1	4.8	1.7	ND	ND	ND	ND	NC	NC
17*alpha*-Ethynylestradiol	0.4	0.8	IRL	377	3.2	3.2	3.2	3.2	2.7	6.0	2.5	1.4	ND	ND	ND	ND	NC	NC
11-Ketotestosterone	NA	2	MRL	376	0.3	0.3	0.3	0.3	0.3	4.2	ND	ND	ND	ND	ND	ND	NC	NC
Mestranol	0.4	0.8	IRL	377	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
Norethindrone	0.4	0.8	IRL	376	0.0	--	--	--	--	--	--	--	--	--	--	--	--	--
Progesterone	2	8	IRL	376	0.3	0.0	0.0	0.0	0.0	0.6	ND	ND	ND	ND	ND	ND	NC	NC
Testosterone	0.8	1.6	IRL	376	1.1	1.1	1.1	0.8	0.5	1.6	1.6	0.2	ND	ND	ND	ND	NC	NC

[a]The LS became approved USGS methods May 2012. Data for laboratory blank (and spike) samples are grouped and accessed (USGS only) under LS 2434 in the NWQL's database, but are applicable to both schedules as all preparation and analysis steps are identical (see text).

[b]Parameter and method codes are provided in table 2.

[c]Numbers less than 377 indicate that the analyte was not reported in all blanks. Bisphenol F was added to laboratory schedules on July 23, 2014.

[d]The RL is twice the DL for many analytes; see table 2.

Table 2. Changes to detection levels, reporting levels, or report-level type codes for analytes in laboratory schedules 2434 and 4434.

[NWIS, National Water Information System; ng/L, nanograms per liter; IRL, interim reporting level; –, no change; NA, not applicable; MRL, minimum reporting level]

Analyte	NWIS parameter code for laboratory schedule		Effective May 31, 2012			Effective June 10, 2016		
	2434[a]	4434[b]	Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code	Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code
4-Androstene-3,17-dione	64513	64533	0.4	0.8	IRL	1	2	–
cis-Androsterone	64515	64535	0.4	0.8	IRL	1	2	–
Bisphenol A	67304	67305	NA	100	MRL	–	–	–
Bisphenol F[c]	51292	51294	NA	10	MRL	–	–	–
Cholesterol	64514	64534	NA	200	MRL	–	400	–
3*beta*-Coprostanol	64512	64532	NA	200	MRL	–	–	–
trans-Diethylstilbestrol	64516	64536	0.4	0.8	IRL	–	1.3	MRL
Dihydrotestosterone	64524	64544	2	4	IRL	–	–	–
Epitestosterone	64517	64537	1	2	IRL	–	–	–
Equilenin	64518	64538	1	2	IRL	–	–	–
Equilin	64519	64539	4	8	IRL	8	16	–
17*alpha*-Estradiol	64508	64528	0.4	0.8	IRL	–	–	–
17*beta*-Estradiol	64510	64530	0.4	0.8	IRL	0.8	1.6	–
Estriol	64520	64540	1	2	IRL	–	–	–
Estrone	64521	64541	0.4	0.8	IRL	1	2	–
17*alpha*-Ethynylestradiol	64509	64529	0.4	0.8	IRL	–	–	–
11-Ketotestosterone	64507	64527	NA	2	MRL	–	–	–
Mestranol	64522	64542	0.4	0.8	IRL	0.8	1.6	–
Norethindrone	64511	64531	0.4	0.8	IRL	0.8	1.6	–
Progesterone	64523	64543	2	8	IRL	6	12	–
Testosterone	64525	64545	0.8	1.6	IRL	–	–	–

[a] NWIS method code GM004.

[b] NWIS method code GM005.

[c] Bisphenol F is not classified as an approved method analyte in these schedules. It was included as a method add-on analyte beginning July 23, 2014. It has a NWIS data-quality indicator code of "U."

Table 3. Changes to detection and reporting levels for analytes in laboratory schedule 6434.[a]

[NWIS, National Water Information System; ng/L, nanograms per liter; IRL, interim reporting level; –, no change; NA, not applicable; MRL, minimum reporting level]

Analyte	NWIS parameter code[b]	Effective October 1, 2014			Effective June 10, 2016		
		Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code	Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code
4-Androstene-3,17-dione	64473	0.1	0.2	IRL	0.25	0.5	–
cis-Androsterone	63607	0.1	0.2	IRL	0.25	0.5	–
Bisphenol A	63188	NA	10	MRL	NA	20	–
Cholesterol	63196	NA	50	MRL	NA	120	–
3*beta*-Coprostanol	63170	NA	50	MRL	–	–	–
trans-Diethylstilbestrol	63620	0.1	0.2	IRL	NA	0.33	MRL
Dihydrotestosterone	64484	0.1	0.2	IRL	0.5	1	–
Epitestosterone	64477	0.5	1	IRL	–	–	–
Equilenin	63204	0.26	0.52	IRL	–	–	–
Equilin	64479	1	2	IRL	2	4	–
17*alpha*-Estradiol	64468	0.1	0.2	IRL	–	–	–
17*beta*-Estradiol	63164	0.1	0.2	IRL	0.2	0.4	–
Estriol	64480	0.26	0.52	IRL	–	–	–
Estrone	63205	0.1	0.2	IRL	0.25	0.5	–
17*alpha*-Ethynylestradiol	63207	0.1	0.2	IRL	–	–	–
11-Ketotestosterone	64467	NA	0.52	MRL	–	–	–
Mestranol	63638	0.1	0.2	IRL	0.2	0.4	–
Norethindrone	63644	0.1	0.2	IRL	0.2	0.4	–
Progesterone	63657	0.5	1	IRL	1.5	3	–
Testosterone	64485	0.1	0.2	IRL	0.2	0.4	–

[a] Detection and reporting levels listed are based on 5 grams dry-weight of extracted samples. These levels are scaled to higher values for sample weights less than 2.5 grams as described in NWQL technical memorandum 14.01.

[b] Laboratory schedule 6434 has NWIS method code GM006 and all method analytes have NWIS data-quality indicator code "U" (defined as results from a research or unapproved method or laboratory). Data from research or unapproved methods are not released to the public via NWISWeb, but can be published in interpretive reports as long as the method description and performance data are provided as described in Office of Water Quality technical memorandum 2004.01.

Table 4. Changes to detection and reporting levels for analytes in laboratory schedule 7434.[a]

[NWIS, National Water Information System; ng/L, nanograms per liter; IRL, interim reporting level; –, no change; NA, not applicable; MRL, minimum reporting level; L, liter]

Analyte	NWIS parameter code[c]	Effective October 1, 2010[b]			Effective June 10, 2016		
		Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code	Detection level (ng/L)	Reporting level (ng/L)	NWIS report-level type code
4-Androstene-3,17-dione	64493	0.4	0.8	IRL	1	2	–
cis-Androsterone	64495	0.4	0.8	IRL	1	2	–
Bisphenol A	67324	NA	200	MRL	NA	–	–
Cholesterol	64494	NA	200	MRL	NA	400	–
3*beta*-Coprostanol	64492	NA	200	MRL	–	–	–
trans-Diethylstilbestrol	64496	0.4	0.8	IRL	NA	1.3	MRL
Dihydrotestosterone	64504	2	4	IRL	–	–	–
Epitestosterone	64497	2	4	IRL	–	–	–
Equilenin	64498	1	2	IRL	–	–	–
Equilin	64499	4	8	IRL	8	16	–
17*alpha*-Estradiol	64488	0.4	0.8	IRL	–	–	–
17*beta*-Estradiol	64490	0.4	0.8	IRL	0.8	1.6	–
Estriol	64500	1	2	IRL	–	–	–
Estrone	64501	0.4	0.8	IRL	1	2	–
17*alpha*-Ethynylestradiol	64489	0.4	0.8	IRL	–	–	–
11-Ketotestosterone	64487	NA	2	MRL	NA	–	–
Mestranol	64502	0.4	0.8	IRL	0.8	1.6	–
Norethindrone	64491	0.4	0.8	IRL	0.8	1.6	–
Progesterone	64503	2	8	IRL	6	12	–
Testosterone	64505	0.4	0.8	IRL	0.8	1.6	–

[a] Detection and reporting levels listed are based on a sample consisting of 1 L of water passed through a glass-fiber filter. These levels are scaled to lower values for sample volumes greater than 2 L.

[b] Values shown for equilin were effective beginning October 1, 2011.

[c] Laboratory schedule 7434 has NWIS method code GM007. All method analytes have NWQL data-quality indicator code "U."