

**In Reply Refer To:
Mail Stop 412**

May 31, 2012

To: GS-W-ALL

**From: Terry L. Schertz
Assistant Chief, Office of Water Quality**

**Subject: Approval of New USGS Methods for Steroid Hormones and Related
Compounds in Filtered (Method O-2434-12) and Unfiltered (Method O-4434-
12) Water by Solid-Phase Extraction, Derivatization, and Gas
Chromatography with Tandem Mass Spectrometry**

The Office of Water Quality (OWQ) has approved new filtered- and unfiltered-water analytical methods, O-2434-12 and O-4434-12, respectively—National Water Quality Laboratory (NWQL) lab schedule (LS) 2434 and 4434, respectively—developed by chemists at the NWQL for the determination of 20 steroid hormones and related compounds.

The U.S. Geological Survey (USGS) developed these methods to support studies of selected steroid hormones in a variety of filtered or unfiltered water-matrix types ranging from groundwater and surface water to wastewater-treatment plant effluent and influent samples and animal feeding operation contaminated runoff. Laboratory processing of 0.5-liter samples for LS 2434 and LS 4434 is identical. Samples for LS 2434 are filtered (preferably in the field) before analysis.

All method analytes are determined by using an isotope-dilution quantification procedure that automatically applies a correction for procedural losses in the reported analyte concentration based on the absolute recovery of deuterium- or carbon-13-labeled isotope-dilution standard (IDS) compounds that are added to a sample just prior to extraction. The IDS compounds are either exact or structurally-similar isotopic analogs of the method analytes, which makes them suitable for recovery correction of the quantified analyte result.

For 16 analytes, the estimated detection levels range from 0.4 to 4 ng/L and applied interim reporting levels range from 0.8 to 8 ng/L. Minimum reporting levels of 2 ng/L for 11-ketotestosterone, 100 ng/L for bisphenol A, and 200 ng/L for cholesterol and 3-beta-coprostanol are used to prevent bias and false positives associated with the presence of these analytes in blanks.

Field samples are collected using USGS protocols for organic contaminants (section 5.6.1.F of Wilde and others, 2004), except that the samples are contained in 0.5-liter high-density polyethylene bottles which facilitates freezer storage of samples at the NWQL prior to extraction.

This method approval process follows the technical procedures specified in OWQ Technical Memorandum 2004.01 and the method is described in a USGS Techniques and Methods Report:

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, section B, chapter 9.

The report will be made available through the USGS Publications Warehouse.

If you have any questions about the new analytical method, or would like a copy of the report, when it is available, please contact Bill Foreman (wforeman@usgs.gov, 303-236-3942), James Gray (jlgray@usgs.gov, 303-236-3776) or Jeff McCoy (jefmccoy@usgs.gov, 303-236-3940).

If you have questions about the method approval process, please contact Terry Schertz (tschertz@usgs.gov, 703-648-6864).

Reference cited

Wilde, F.D., Radtke, D.B., Gibbs, Jacob, and Iwatsubo, R.T., eds., 2004 with updates through 2009, Processing of water samples (ver. 2.2): U.S. Geological Survey, Techniques of Water-Resources Investigations, book 9, chap. A5, April 2004. (Also available at <http://pubs.water.usgs.gov/twri9A5/>.)