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Standard Method Performance Requirements for Quantitative Determination of Estrone (E1) in Freshwater

Intended Use: Quantitative measurement by trained personnel

1 Applicability

Rapid screening-level determination of dissolved estrone at environmental levels in freshwater containing up to 1000 mg/L (ppm) of total suspended solids (TSS) and 1000 mg/L (ppm) of dissolved organic carbon (DOC). Estrone is also known as E1; oestrone; or 3-hydroxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydrocyclopenta[*a*]phenanthren-17-one. CAS No.: 53-16-7.

2 Analytical Technique

Any analytical technique that meets the following method performance requirements is acceptable.

3 Definitions

Freshwater.—Naturally occurring water on the Earth's surface in wetlands, ponds, lakes, rivers,streams, and aquifers exclusive of brines, seawater, and brackish water. (USGS definition: Less than 1000 mg/LTSS.)

DOC.—The fraction of total organic carbon (all carbon atoms covalently bonded in organic molecules) in water that passes through a 0.45 micron pore-diameter filter.

Limit of detection (LOD).—Equivalent to the term "Method Detection Limit" (MDL) used by the U.S. Environmental Protection Agency: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, and is determined from analysis of a sample in a given matrix containing the analyte.

Limit of quantification (LOQ).—The level above which quantitative results may be obtained with a specified degree of confidence.

Precision (repeatability).—Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator, and repeating during a short time period. Expressed as the % standard deviation (SD); or % relative standard deviation (%RSD).

Recovery factor.—The ratio of the observed mean test result to the true value. Recovery % = [$mean_{obs}$/true] × 100.

Reproducibility.—RSD calculated from among-laboratory data.

Table 1. Method performance requirements

Analytical range	2–200 ng/L (ppt)	
LOD	0.5 ng/L (ppt)	
LOQ	≤2 ng/L (ppt)	
Precision (repeatability; RSD_r)	2 ng/L (ppt)	≤110%
	20 ng/L (ppt)	≤50%
	200 ng/L (ppt)	≤30%
Recovery factor	50 to 120% of mean spiked recovery over the range of the assay	
Reproducibility (RSD_R)	2 ng/L (ppt)	≤165%
	20 ng/L (ppt)	≤75%
	200 ng/L (ppt)	≤45%

4 Method Performance Requirements

See Table 1.

5 System Suitability Tests and/or Analytical Quality Control

- Suitable methods will include blank check samples and check standards at the lowest point and midrange point of the applicability range.
- If a candidate method is a commercially provided method, then the method provider must provide documentation in the validation package to demonstrate (*1*) suitable lot-to-lot consistency, and (*2*) stability over the claimed shelf-life of the reagents.

6 Reference Method(s)

No reference method exists. (EPA 1698 could be used. GC-HRMS-MS could be used as an instrumental method with suitable sensitivity.)

7 Reference Material(s)

None available.

8 Validation Guidance

- Preservation of prepared samples must be considered and consistent among laboratories participating in a validation.
- Study materials should be analyzed by tandem mass spectrometry.
- Recommended level of validation: *Official Methods of Analysis*SM.

9 Maximum Time-to-Signal

No maximum time.

Approved by Stakeholder Panel on Endocrine Disruptors on September 20, 2010.