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NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1998.05

December 9, 1997

Subject: Changes in Minimum Reporting Levels for Inductively Coupled Plasma - Atomic Emission Spectrometry

Effective date
of changes: December 22, 1997

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Revision: None

Supplemental: None

BACKGROUND

Minimum reporting levels currently in place for Inductively Coupled Plasma - Atomic Emission Spectrometric (ICP-AES) analytical results were determined at the time the methods were developed in about 1979 (Fishman and Friedman, 1989). The procedure to assess reporting levels assessed the sensitivity of the methods and the standard deviation of blank sample results (Skogerbue and Grant, 1970). In addition, the reporting levels were determined using a different generation of ICP-AES instrumentation with different sample introduction apparatus than is used now. Such differences would have a significant impact on the reporting levels.

Even after newer ICP-AES instrumentation was put into use, the analysts at the NWQL continued to use the originally established reporting levels as goals to achieve. This was sometimes done by repeatedly analyzing sample sets until quality control results satisfied control limits. This practice led to logistical problems in managing Plasma Unit operations because of an increasing number of reanalyses, and it raised questions regarding the accuracy of the reporting levels. Reporting levels were reassessed in the winter of 1995 using EPA guidelines (U.S. Environmental Protection Agency, 1997a). Because of this assessment, changes in acceptance criteria were implemented in March 1995. In September 1995, procedural changes for ICP-AES methods were announced via electronic mail to the Water Resources Division with a statement that the NWQL was planning to reassess reporting levels (T.J. Maloney, U. S. Geological Survey, written commun., 1995).

The periodic summary reports of the Blind Sample Program (BSP) operated by the Branch of Quality Systems provide further evidence that reporting levels should be changed for some ICP-AES

constituents. The BSP summary reports prepared after March 1995 indicate that several ICP-AES constituents demonstrated increased variability and bias after the procedural changes were started.

The new minimum reporting levels announced in this Technical Memorandum were determined by using a multielement spiked nanopure water matrix. Low concentration spikes are used in this procedure. These reporting levels represent the best possible performance to be expected because of the ultrapure nature of the water matrix used. In addition, for aluminum and zinc, bias caused by ubiquitous nature of these elements further contributes to the overall variance used to determine the reporting level.

The reporting levels are applicable to water-sample matrices having specific conductance less than 2,500 uS/cm at 25o C because most samples with conductivities in this range do not demonstrate matrix effects. However, concerned customers can discuss ways to verify this for their individual matrices with Ed Zayhowski, Plasma Unit Chief at the NWQL.

SCOPE

This technical memorandum announces changes, effective December 22, 1997, in minimum reporting levels for constituents determined by ICP-AES methods at the NWQL. The changes are the result of a systematic evaluation of reporting levels for NWQL methods. Four ICP-AES constituents did not require changes of reporting levels: barium, calcium, copper, and lithium. Those constituents requiring reporting level changes are summarized in Table 1.

TABLE 1. --Reporting level changes for inductively coupled plasma - atomic emission spectrometric (ICP-AES) methods at the NWQL

Constituent	Parameter (WATSTORE) code *	NWQL Lab code	Minimum reporting level (microgram/liter)	
			Current	Effective Dec. 22, 1997
Aluminum	01106 F	2111	5	10
Beryllium	01010 B	655	0.5	1
Boron	01020 F	2110	4	16
Cadmium	01025 D	673	1	8
Chromium	01030 E	722	5	14
Cobalt	01035 C	644	3	12
Iron	01046 D	645	3	10
Lead	01049 C	646	10	100
Magnesium**	00925 C	663	0.01	0.004
Manganese	01056 C	648	1	4
Molybdenum	01060 A	649	10	60
Nickel	01065 E	721	10	40
Silica**	00955 D	667	0.01	0.1
Silver	01075 C	723	1	4
Sodium**	00930 C	675	0.2	0.1
Strontium	01080 B	652	0.5	1
Vanadium	01085 B	653	6	10
Zinc	01090 B	671	3	20

* The letter following the 5-digit parameter code represents the method code.

**Reporting level information for these constituents are in milligrams per liter (mg/L).

ALTERNATE METHODS AVAILABLE

The reporting level changes described here for individual lab codes will impact numerous laboratory schedules using ICP-AES. The higher minimum reporting levels for selected constituents may not be adequate for some water-quality studies. The NWQL recommends that Districts review the laboratory schedules they are using to decide if these changes will affect data-quality objectives, and if so, consider an alternative method of analysis. Two additional methods are typically available through the NWQL for these constituents that produce analytical results that are more sensitive and provide highly reproducible and accurate results. These methods are Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) and Graphite Furnace Atomic Absorption Spectrophotometry (GFAAS). The SPiN and Catalog applications on the NWQL homepage <http://wwwnwql.cr.usgs.gov/> can be searched to determine the lab codes and reporting levels of alternate methods. Steve Glodt (srglodt) at the NWQL should be contacted to make changes to existing laboratory schedules or create new laboratory schedules.

EFFECT ON DATA BASE

Minimum reporting level changes for the constituents in Table 1 will cause a shift in non-detection (less than) concentrations in the data base. Minimum reporting levels for historical data will not be changed in the data base. During FY1998, the Office of Water Quality, along with other Water Resources Division representatives, will be developing guidance on interpreting analytical results, both new and historical, for the effected determinations. Data users should be cautious when interpreting historical ICP-AES data in light of new higher reporting levels. Recent U.S. Environmental Protection Agency (1997b) guidelines on good laboratory practices recommend periodic assessment of method detection and reporting levels. The NWQL is developing plans to assess this information annually.

References:

Skogerbue, R. K. and Grant, C. L., 1970, Comments on the definitions of the terms sensitivity and detection limit. *Spectroscopy Letters*, 3, p.215-220.

U.S. Environmental Protection Agency, 1997a, Guidelines establishing test procedures for the analysis of pollutants (App.B to Part 136) Definition and procedure for the determination of the method detection limit): U.S. Code of Federal Regulations, Title 40, revised as of July 1, 1997, p. 265-267.

U.S. Environmental Protection Agency, 1997b, National Environmental Laboratory Accreditation Conference, Chapter 5, Quality Systems Standards.

/signed/

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Key words: Reporting levels, trace elements, Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

Supersedes: None

Distribution: E (all WRD employees) and <http://wwwnwql.cr.usgs.gov/>