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U.S. GEOLOGICAL SURVEY

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NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1995.03

October 14, 1994

To: Assistant Chief Hydrologist for PC&TS
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Assistant Chief, Office of Water Quality
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Area Hydrologists
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Chief, Yucca Mountain HIP
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Chief, Branch of Quality Assurance
Employees, National Water Quality Laboratory

From: Peter F. Rogerson, Chief
National Water Quality Laboratory
Branch of Analytical Services

Subject: Graphite Furnace Atomic Absorption Spectrophotometry to replace Atomic Absorption Spectrometric, Chelation-extraction for the determination of molybdenum in water

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Revision: None

SCOPE

Effective November 1, 1994, the National Water Quality Laboratory (NWQL) will be replacing the Atomic Absorption Spectrometric, Chelation-extraction method for the determination of molybdenum in water with a Graphite Furnace Atomic Absorption Spectrophotometry (GF-AAS) method. All molybdenum data released by the NWQL after November 1, 1994, will be determined by the provisionally approved GF-AAS method. Full U.S. Geological Survey (USGS) approval of this method will be pursued with an Open-file Report within 6 to 12 months.

The GF-AAS method is based on the Environmental Protection Agency's (EPA) standard method 246.2, Molybdenum (Atomic Absorption, furnace technique). The GF-AAS method for the determination of molybdenum has an applicable analytical range from 1 to 50 micrograms per liter ($\mu\text{g/L}$) with a method detection limit (MDL) 0.8 $\mu\text{g/L}$. These are the same or better than the flame-AA chelation-extraction method.

The lab code for dissolved molybdenum determinations will be changed from 110 Mo diss to 1998 Mo-GF diss. The lab code for whole-water recoverable molybdenum determination will be changed from 265 Mo Total to 1999 Mo-GF Total. The NWQL will update all schedules with the new lab codes.

The current flame-AA chelation-extraction method involves the use of hazardous and reactive reagents. These hazardous reagents are methyl isobutyl ketone (MIBK) and 8-hydroxyquinoline--both of which present health, safety, and hazardous waste disposal problems for the NWQL laboratory and staff. MIBK produces heavy vapors that are dangerous fire hazards; exposure to MIBK is irritating, narcotic, and toxic. On two recent occasions the entire lab had to be evacuated due to release of small amounts of MIBK. In order to protect our employees, the NWQL will change to the safer GF-AAS method.

Precision of the GF-AAS method at the detection limit is approximately 50 percent relative standard deviation (RSD), and is approximately 14 percent RSD throughout the analytical range of 1 to 50 $\mu\text{g/L}$. Accuracy has been demonstrated by analyzing multiple replicates of more than 12 standard reference materials.

These results indicate that the method is within one standard deviation of the established mean values for the determination of molybdenum. In addition, 15 different samples were spiked with known concentrations of molybdenum and were analyzed in replicate. Spike recoveries for the determination of molybdenum had an average recovery of 105 percent.

Other methods also available at the NWQL for the determination of molybdenum include Inductively Coupled Plasma (ICP, method reporting limit 10 $\mu\text{g/L}$) and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS, method reporting limit 1.0 $\mu\text{g/L}$). These are excellent choices for multielement determinations, while the GF-AAS determination is ideal for that single element.

Supersedes: None

Key Words: Molybdenum, Atomic Absorption Spectrophotometry Graphite Furnace

Distribution: See above plus the continua USGS.labnews & .waterquality and WRD Secys

Impact on National Data Base: None.