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U.S. GEOLOGICAL SURVEY

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NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1996.08

May 22, 1996

To: Assistant Chief Hydrologist for Technical Support
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From: Peter F. Rogerson, Chief
National Water Quality Laboratory
Branch of Analytical Services

Subject: Implementation of EPA Method 1664 for the Analysis of Oil and Grease or Total
Petroleum Hydrocarbons in water

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Revision: None

SCOPE

The National Water Quality Laboratory (NWQL) is changing the method used to analyze oil and grease in water samples. This method can also be used to determine Total Petroleum Hydrocarbons (TPH) in water samples. We will be implementing U.S. Environmental Protection Agency (EPA) proposed method 1664 "n-hexane Extractable Material (HEM) and Silica Gel Treated n-hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry." HEM is commonly called "oil and grease" and SGT-HEM is commonly called "Total Petroleum Hydrocarbons."

BACKGROUND

The current U.S. Geological Survey method for analyzing oil and grease in water samples uses Freon-113 as the extraction solvent. Freon-113 is a chlorofluorocarbon identified as a substance that depletes the ozone layer. Under the Clean Air Act Amendments of 1990, the United States is committed to controlling and eventually phasing out chlorofluorocarbons such as Freon-113. To avoid contributing to depletion of the ozone layer, the NWQL will eliminate its use of Freon-113 by implementing EPA method 1664, which uses n-hexane as the extraction solvent.

In the past, the NWQL has determined TPH as a custom analysis, using EPA method 418.1 "Petroleum Hydrocarbons, total recoverable," by infrared spectrophotometry. In its recent review of oil and grease methods, EPA discredited the infrared technique for EPA programs. The NWQL will discontinue custom analysis for TPH using the infrared technique.

DISCUSSION

The NWQL will start analyzing samples for oil and grease using EPA method 1664 on June 1, 1996, at which time the determination of Total Petroleum Hydrocarbons in water samples will also be available.

To request an analysis for oil and grease, use Lab Code 2125. To request an analysis for TPH, use lab code 2126. The current lab code for oil and grease is 127. Requests for Lab Code 127 received after June 1, 1996, will be converted to requests for Lab Code 2125. Do not request Lab Code 127 after June 1, 1996.

EPA projects require pH adjustment in the field and shipment in an iced (chilled) container. USGS protocol also prescribes pH adjustment and chilled shipment. Adjust the sample pH to less than 2 by adding 2 milliliters (m/L) of sulfuric acid to about 1 liter (L) of sample and mix. Put the sample into a baked 1-L glass bottle, chill to 4 degrees C, and ship to the NWQL in a chilled container. If pH is not adjusted in the field, it will be adjusted at the NWQL prior to analysis. Sulfuric acid ampules, used for this purpose, can be obtained from the Ocala facility.

The EPA-published method detection limit (MDL) for oil and grease is 1.4 milligram per liter (mg/L), and for TPH the MDL is 1.6 mg/L. The NWQL MDL for oil and grease is 1.3 mg/L, and the NWQL MDL for TPH is 1.2 mg/L.

The NWQL initially documented the accuracy of the EPA method by spiking four replicates of reagent water with 40 mg/L of total oil and grease and 20 mg/L of TPH. The mean recovery for total oil and grease was 92.5 percent and the relative standard deviation (RSD) was 4.5 percent. The mean recovery for TPH was 84.2 percent and the RSD was 1.8 percent.

Effects on data base: The available EPA information indicates that there is no consistent correlation between the data obtained using Freon-113 extraction with the data obtained using the n-hexane extraction. There may or may not be abrupt changes in the data from individual sampling sites when the new method is implemented.

Supersedes: None

Key words: Total Recoverable oil and grease Analysis, Total Recoverable Petroleum Hydrocarbons Analysis

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