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METHOD 245.1

I. SCOPE AND APPLICATION:

This method measures total mercury (organic + inorganic) in drinking water, surface, ground, sea, brackish waters as well as for industrial and domestic wastewater. The range of the method is 0.2 to 10µ Hg/liter. The range can be extended above or below the normal range by increasing or decreasing the sample size. However, the actual detection limit and linear working range will be dependent on the sample matrix, type of instrumentation configuration, and selected operating parameters. This method is applicable to the following analyte:

Analyte

Mercury (Hg)

**Chemical Abstract Services
Registry Numbers (CASRN)**

7439-97-6

II. REAGENTS:

- Nitric acid (ultra high purity grade) 1:1 dilution. Add 500 ml of concentrated ultra pure nitric acid to 400 ml of ASTM type I water (reagent water-- free of analytes) and dilute to a volume of 1 liter.

III. MATERIALS:

- 1 liter (32 oz) high density polyethylene bottles with poly-foam lined screw on caps.
- Collect duplicate samples if 500 ml sample bottles are used.
- pH indicator paper (low end)
- Latex gloves
- Paper towels & Kim Wipes napkins
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Protective eyewear

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.
2. Open the tap and allow the system to flush for approximately 10 minutes. This should be sufficiently long enough to get a representative sample.
3. Remove the cap from the polyethylene container. Do not rinse the container as it has already been acid rinsed and may already contain acid as a preservative.
4. To fill, tip the bottle at about a 45° angle into the stream of water. Slow the stream sufficiently so as to be able to anticipate when the bottle is nearly full and thus avoid overflowing. Fill the bottle to the fill line or within ½ inch of the top. This will allow enough space for mixing and the addition of any additional acid if required.
5. Remove the bottle from the flow and recap. Invert the container five times.
6. Place a pH indicator test strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the pH indicator test strip with the aliquot from the glass pipette and immediately flick the pH indicator test strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference pH range. A determination must be made within 30 seconds.
7. If the pH is ≤ 2 , recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in field notebook and place the sample bottle in the ice chest.
8. If the pH is not ≤ 2 , add 3 ml of 1:1 nitric acid (HNO_3), recap the bottle firmly and invert the bottle 5 times.
9. Place a pH indicator test strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the pH indicator test strip with the aliquot from the glass pipette and immediately flick the pH indicator test strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference pH range. A determination must be made within 30 seconds.

IV. PROCEDURE (continued):

10. If the pH is ≤ 2 , recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in field notebook and place the sample bottle in the ice chest to cool to 4°C.
11. Continue the process of adding acid to the sample, recapping, mixing, and testing until the pH of the sample reaches a pH of ≤ 2 . Remember to note the amount of acid added to the water sample in the field notebook.

V. SAMPLE TRANSPORT:

After obtaining the water samples (in duplicate if necessary), attach the preprinted sample label onto the bottle as well as the completed chain of custody seal around the plastic cap of each sample bottle. Place the sample bottle(s) into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the samples for transportation. Never use “blue ice” as the samples may not chill adequately.

Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory. Samples that cannot be acid preserved at the time of collection because of sampling limitations or transport restrictions should be acidified with nitric acid (HNO₃) to a pH < 2 upon receipt in the laboratory and allowed to sit approximately sixteen hours. This waiting period is to allow any inorganic metals to return back into solution in the event they may have precipitated or “plated out” along the inside of the sample container as a result of inadequate acidification at the time of collection.

VI. SAMPLE STORAGE:

Store samples at 4°C until analysis.

VII. DEFINITIONS:

- A. *Aliquot*: A measured portion of a sample taken for analysis.
- B. *Matrix effects*: The influence of the sample matrix or sample components upon the ability of analytical methods to qualitatively identify and quantitatively measure target compounds in environmental samples

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 245.1--A known portion of a water sample is transferred to a BOD (biological oxygen demand) bottle or equivalent ground glass stoppered flask. It is digested in a diluted potassium permanganate-potassium persulfate solution and oxidized for 2 hours at 95°C so that any mercury in the digested water sample is reduced with stannous chloride to elemental mercury. Elemental mercury is readily purged from the sample using any pressure system (such as a compressed gas cylinder) capable of passing gas at 1-liter/minute. The mercury is then measured by the conventional cold vapor atomic absorption.