

Assay of urine Hg concentrations measurement Version 6

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Abstract

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Protocol

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 1.

All containers need to be soaked in nitric acid (10%) for 24h and then rinse with double-deionized water (DDW) .

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 2.

Collect 50ml midstream urine with clean plastic container.

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 3.

Transfer 2 ml of the urine sample to a into 25 ml capped glass tubes. Add 5 mL of 8mol/L nitric acid, anti-foaming agent (a few drops) and 2 ml of 0.0599mol/L potassium bromate - 0.0234mol/L potassium bromide reagent, respectively

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 4.

Tightly capped glass tubes and allow standing on bench for 20 min to complete breakdown of organic Hg compounds.

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 5.

Add 10ml DDW, then add a few drops of 1.439mol/L hydroxylamine hydrochloride to remove free bromine after digestion.

For total Hg measurement, the urine samples will be digested using the brominating procedure

Step 6.

Add DDW to fill the volume up to 25 ml.

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 7.

Switch on the HG - AFS 9700, and preheat 20 minutes. Urinary mercury is determined in the experimental conditions indicated in Table 1.

cmd **COMMAND (Table1 The operating parameters of the AFS-9700 HG-AFS for the Determination of Hg)**

Parameters High negative voltage of PMT 270V Atomizer temperature Rome Temperature Atomizer height 7 mm Lamp current 30 mA Flow rate of carrier gas 600 mL/min Flow rate of shield gas 800

mL/min Measurement method Std. curve Read method Peak area Delay time 3s Read time 24s
Injection volume 2.0 mL Resonance wavelength 253.7 nm

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 8.

Calibration regression curves at seven points (0, 0.2, 0.4, 0.8, 1.2, 1.6 and 2.0 ug/L) treated as the samples bromination procedure.

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 9.

Calibration regression curves according to experimental conditions.

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 10.

Spiked recovery test is performed to ensure the precision and accuracy of the analyses.

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 11.

To avoid possible contamination during the digestion procedure and sample manipulation, a blank solution is used through the preparation and device assay process along every twenty samples.

Hg fluorescent intensity is assayed by using a fully-automatic double-channel hydride generation atomic fluorescence spectrometer (HG - AFS 9700)

Step 12.

A sample of standard materials with known reference concentrations of Hg is prepared and carried through the preparation and assay processes along every twenty urine samples to verify the stability of the process.