



Method Summary

Determination of Trace Level Mercury in Waters and Leachates by PSA Cold Vapour Atomic Fluorescence Spectrometry

Scope and Range

This method is suitable for the determination of trace level dissolved mercury in drinking water, environmental (ground and surface) waters, treated and untreated aqueous effluents, leachates, and also unfiltered samples.

The LoD for this method is 0.01 µg/l for dissolved matrices, and 0.02 µg/l for unfiltered matrices.

Principle

Preparation and Extraction

The samples shall be collected in either a borosilicate glass bottle (500ml or 1L) or a 100ml HDPE bottle containing high purity hydrochloric acid (1.0 ml Hydrochloric Acid 21% (w/w)).

If dissolved analysis is required samples should be filtered on site.

10ml of sample is transferred to a glass tube and filtered for dissolved analyses, and for unfiltered samples 30ml is transferred into a plastic digestion tube.

Filtered samples are treated with hydrochloric acid and potassium bromide/bromate solution to generate bromine, which oxidises all mercury compounds present to Hg(II). Excess bromine is removed by ascorbic acid immediately prior to analysis. Hg(II) is reduced to Hg(0) by tin (II) chloride.

Analysis

Elemental mercury vapour is stripped from solution by an argon gas carrier stream. Moisture is removed from the sample gas stream, and the dry mercury vapour is detected by atomic fluorescence spectrometry.

Interferences

Mercury vapour can diffuse through the walls of non-fluorinated plastics; hence sampling and storage vessels must be of borosilicate glass or fluorinated plastic, e.g. PTFE. Instrument sample uptake tubing must be of fluorinated plastic, e.g. FEP. It should be noted that the unfiltered mercury extraction vessel is HDPE. However, the validation has passed the in house limits and is deemed acceptable.

Exchange reactions - adsorption and desorption of mercury may occur at the walls of sampling and reaction vessels.

Suppression of the fluorescence signal may be caused by:

- Water vapour or condensate in the fluorescence cell may cause quenching. The sample uptake apparatus and instrument drying membrane should be suitably maintained to avoid this.
- Quenching by dissolved gaseous compounds - these are removed by bromination
- Anions which complex strongly with mercury, e.g. sulphide, iodide and bromide (the potassium bromide-bromate digestion reagent causes no suppression if applied as directed)
- Noble metals e.g. gold, silver and platinum, which amalgamate with mercury vapour.