Successful Low Level Mercury Analysis

using the Agilent 7700 Series ICP-MS

Analysis of Mercury by ICP-MS

Mercury (Hg) is a rare heavy metal, many forms of which are highly toxic by inhalation, ingestion or absorption through the skin. Hg has many industrial uses, including as a component of compact fluorescent light bulbs, and is typically monitored and regulated in commodities such as drinking water, seafood, consumer goods, pharmaceutical products and children's toys.

Traditional methods for Hg analysis require a separate sample preparation combined with a single-element Hg analyzer, which reduces sample throughput and productivity. Mercury analysis can be combined with the typical multi-element analysis performed by ICP-MS, but this presents several challenges:

- Hg has a very high first ionization potential (10.44eV) which means that it is only about 4% ionized in the plasma. This leads to low sensitivity for Hg, as only ions (not atoms) are measured by ICP-MS.
- Hg has a high number of naturally occurring isotopes (7), all less than 30% abundant. Since the total element concentration is divided among many separate isotopes, the number of ions (and therefore the sensitivity) is lower for each individual isotope.
- Hg is very easily lost from aqueous samples by adsorption to the walls of the container and sample introduction components resulting in low recovery and extended washout times. Hg is also volatile and can be lost if samples are digested in open vessels.

Samples requiring Hg analysis are typically prepared using closed vessel digestion, but successful analysis of low-level Hg by ICP-MS depends on several additional factors:

- The plasma must be operated at the highest possible temperature (lowest possible CeO^+/Ce^+ ratio) to maximize ionization of the Hg atoms.
- The system must provide high sensitivity to compensate for the low degree of ionization and the low abundance of each Hg isotope.
- Appropriate sample preparation and preservation chemistry must be used to ensure stability of Hg.

The Agilent 7700 Series ICP-MS addresses the first two requirements and the simplest approach to the third is to prepare all samples and standards using a low % level of HCl, which ensures that the Hg is converted to the stable Hg^{2+} form.

Chemical Stability of Hg

Sample preparation and preservation using HNO_3 alone (specifically excluding HCl) is recommended in many ICP-MS methods in order to avoid problems caused by the Cl-based polyatomic interferences on V, Cr As and Se. However, excluding HCl can cause problems with the stability of many elements including Hg, As, Se, Mo, Tl and Ag.

Even with the advent of collision/reaction cell (CRC) ICP-MS, the presence of chloride (whether naturally present in the sample or added in the form of HCl) is still a major problem for instruments that use reactive cell gases to remove interferences. Ammonia (NH_3) reaction gas is commonly recommended for removing the ClO and ClOH interferences on V and Cr, but NH_3 is not effective for the ArCl and CaCl interferences on As, so a second reaction gas (such as O_2, H_2, or CH_4) is required when V, Cr and As all have to be measured. None of these reactive cell gases is reliable in all sample types.

Advances in CRC technology for the 7700 Series ICP-MS now allow a simple approach to removing all the Cl-based interferences using a single, universal He mode (Figure 1).

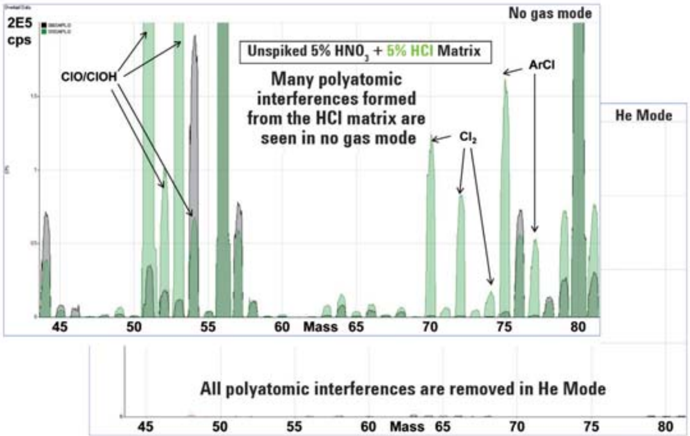

Figure 1. Comparison of polyatomic interferences from 5% HCl matrix in no gas mode (top) and He mode (bottom), shown on the same scale

As a result, HCl at 0.5 to 1.0% can and should be included in acidic sample preparation and stabilization of samples for ICP-MS analysis. In addition, samples with high or variable natural levels of Cl can now be measured reliably, without the extensive method development and multiple cell gases that are required when reaction mode is used. The presence of HCl allows Hg to be measured routinely at single ppt detection limits (DLs) on the 7700 (Figure 2). The ^{201}Hg isotope (13.18% abundance) was measured, as it is free from polyatomic overlap by WO.

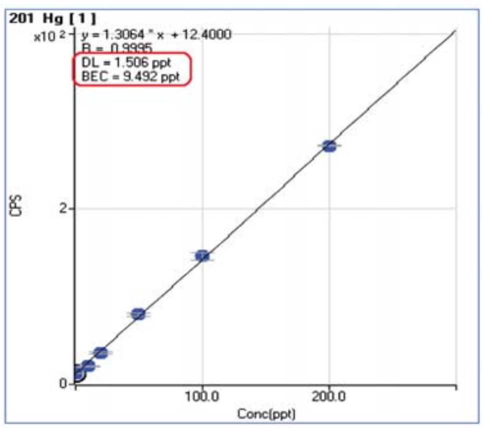

Figure 2. Calibration for mercury at ng/L (ppt) levels, demonstrating single ppt DL and BEC (Background Equivalent Concentration)

7700 Series ICP-MS Makes Low-Level Hg Analysis Routine

The 7700 Series ICP-MS provides a simple, universal He cell mode for effective removal of all Cl-based polyatomic interferences, allowing reliable analysis of V, Cr and As in the presence of % levels of chloride. As a result, HCl can now be routinely added to samples for ICP-MS analysis, improving the chemical stability of many elements including Hg. This enables ppt-level analysis of Hg by ICP-MS, without compromising the analysis of other elements in the typical ICP-MS multi-element suite.

For more information on the 7700 Series ICP-MS visit the Agilent Technologies web site at: **www.agilent.com/chem/icpms**