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Extending the Boundaries of Beryllium Detection with the Agilent 7500ce ICP-MS

There is a clear requirement to measure beryllium at ultratrace levels. While it is relatively non-toxic in its natural ore state, all other commercially important Be compounds exhibit significant pulmonary toxicity. The US Environmental Protection Agency (EPA) has also classified Be as a "probable human carcinogen".

Traditional NIOSH and OSHA methods for Be in airborne samples utilize ICP-OES, with approximate detection limits of 0.005ug/filter (NIOSH 7300), which requires a sampling volume of up to 2000 liters. In contrast, the Agilent 7500ce ICP-MS offers far superior sensitivity compared with ICP-OES, enabling the use of much lower air sampling volumes and providing significantly lower absolute detection limits.

ICP-MS Conditions

An Agilent 7500ce in standard configuration was used for this work. While the 7500ce is a collision cell instrument, the device was unpressurized and no collision/reaction chemistry was employed. The 7500ce incorporates unique design features in the ion optics to minimize the effects of space charge on low mass analytes such as Be. The high efficiency solid state 27.12MHz RF generator ensures a very high temperature which improves ionization of the element. As a result, ultratrace determination of Be by ICP-MS is now possible, with detection limits at the ppq level. Calibrations were performed in dilute HNO_3 from 1 ppt to 50 ppt as shown in Figure 1. Estimated detection limits based on calibration linearity, response factor and background were calculated to be 5.2 x 10^{-5} ppb (52 ppq) or 0.000052 ng/mL, compared with published values of 0.2 ng/mL by ICP-OES (NIOSH 7300). This translates to about 4000x improvement in sensitivity.

Precision and Accuracy

Precision and accuracy at the low ppt level were evaluated by measuring replicate analyses of NIST 1640, alternating between samples diluted 1000x and 10000x. During the course of the 8.5 hour analytical sequence, NIST 1640 was analyzed 113 times, 56 times at 1000x dilution and 57 times at 10000x dilution. The certified value for Be in NIST 1640 is 34.94 ppb and the 7500ce measured results for Be in the diluted samples are summarized in Table 1.

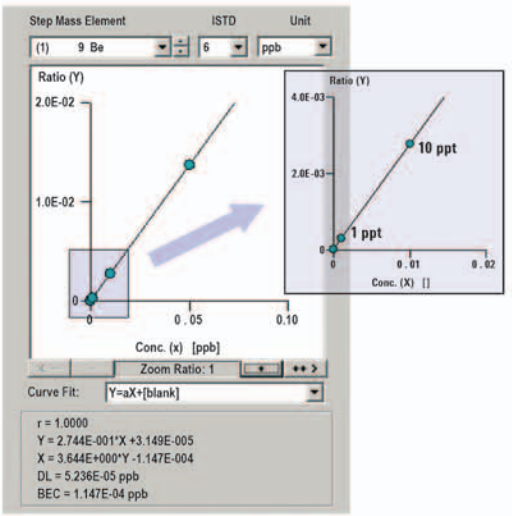

Figure 1. Calibration curve for Be from 1 ppt to 50 ppt

Analysis of Be in NIST 1640 - Standard Reference Water					
Dilution factor	Number of analyses	Mean result (ppb)	Measured conc. (ppt)	%RSD	Average % Recovery
1000	56	34.13	34	0.988	97.67
10000	57	34.06	3.4	2.51	97.49

Table 1. Accuracy and precision of alternating replicate measurements of NIST 1640 at dilution factors of 1000x and 10000x, during 8.5 hours of continuous analysis

Conclusions

The Agilent 7500ce ICP-MS is capable of measuring beryllium in acid digests comparable to those produced by digestion of air filters according to NIOSH 7300, but at levels up to 4000 times lower than the published DLs using ICP-OES.

In addition, the long term precision and accuracy, as determined by measuring highly diluted certified reference material (NIST 1640), shows recoveries of greater than 97% and precision in the order of 1-2% over more than 8 hours of continuous analysis.

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