

Novel method for measuring ultra-trace levels of U and Th in Au, Pt, Ir, and W matrices using QQQ-ICP-MS and an O₂ reaction gas

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The advent of commercial elemental MS/MS instrumentation has given rise to multiple opportunities for method development aimed at analytes in challenging matrices that either reduce or eliminate the need of extensive sample pre-processing. Here, ultra-trace concentrations (ppq level) of natural ²³²Th and ²³⁸U and non-natural tracer isotopes ²²⁹Th and ²³³U were measured in a solution of 10 ppm each of Au, Pt, Ir, and W in 2% HNO₃ using a QQQ-ICP-MS. Polyatomic interferences across a *m/z* range of 227-239 were characterized: the major interferants with ²²⁹Th⁺ is ¹⁹⁴Pt³⁵Cl⁺, with ²³²Th⁺ are ¹⁸⁴W¹⁶O₃⁺, ¹⁸³W¹⁶O₃H⁺, ¹⁹²Pt⁴⁰Ar⁺, ¹⁹⁶Pt³⁶Ar⁺, ¹⁹⁵Pt³⁷Cl⁺, and ¹⁹⁷Au³⁵Cl⁺, with ²³³U⁺ are ¹⁹³Ir⁴⁰Ar⁺, ¹⁹⁷Au³⁶Ar⁺, ¹⁸⁴W¹⁶O₃H⁺, and with ²³⁸U⁺ is ¹⁹⁸Pt⁴⁰Ar⁺. Upon reaction with O₂ gas, the highest sensitivity analyte species formed were ThO⁺ and UO₂⁺. Scanning the selected *m/z* range of 227-270 amu showed that higher oxide polyatomic species either did not form or did not create significant background on the target analyte masses. All measured concentrations in standard solutions matched the target values within the 98% confidence interval. The Th measurements were 80% accurate or better at the 10 ppq level and above, and the U measurements were 90% accurate or better at the 10 ppq level and above. While measurements at the 1 ppq level were consistent with target values within 1 standard deviation, the standard deviations of all three replicates were greater than 20% of the measured concentration value. Method detection limits in the matrix solutions were 2.74 fg Th and 12.9 fg U, which are comparable to detection limits of 1.2 fg Th and U previously measured in pristine 2% HNO₃ blank solutions. This method is but one example of how state of the art quadrupole mass spectrometry and collision reaction cell technology can be leveraged to develop novel analytical capability.