High Throughput Analysis of Rock Digests

using the 7700x ICP-MS with HMI and ISIS-DS

ICP-MS in the Geological Exploration and Mining Industries

Geological exploration and mining generate tens of thousands of samples per week worldwide which must be analyzed quickly and accurately for a wide range of metals. Depending on the required analytes and detection limits, samples may be acid digested and measured by flame AA, ICP-OES, or ICP-MS. ICP-MS offers the lowest detection limits, but limited tolerance to dissolved solids and higher maintenance requirements have kept it from being used universally.

Combining HMI with Discrete Sampling using ISIS-DS

Agilent's HMI (High Matrix Introduction) system introduced the concept of aerosol dilution (Figure 1), which uniquely allows ICP-MS to directly measure samples containing percent levels of dissolved solids. HMI is standard on the Agilent 7700x ICP-MS, and provides several major benefits when analyzing high TDS samples, including:

- Aerosol dilution reduces the amount of matrix that reaches the plasma, thereby allowing higher matrix samples to be aspirated
- Improved plasma robustness decreases matrix effects and reduces matrix deposition, minimizing interface/lens maintenance
- Lower water vapor loading reduces oxides and other interferences

By combining HMI with Agilent's Integrated Sample Introduction System for Discrete Sampling (ISIS-DS), previously unachievable throughput and stability can become routine. Compared with traditional sample introduction, ISIS-DS shortens analysis times and reduces the sample loading reaching the instrument by introducing sample only during the data acquisition phase of the analysis. The combined effect of HMI and ISIS-DS can increase sample throughput 4-fold while reducing the matrix load on the plasma and interface by more than 90% compared to conventional ICP-MS.

Parameter	Initial Value (before sequence)	Final Value (after sequence)
Sensitivity (cps/ppb) m/z = 7	23610	22429
Sensitivity (cps/ppb) m/z = 89	97178	96206
Sensitivity (cps/ppb) m/z = 205	70994	71703
Oxides (%) m/z 156/140	0.418	0.431
Doubly Charged (%) 70/140	0.960	1.012

Table 1. Comparison of instrument tuning before and after the sample run

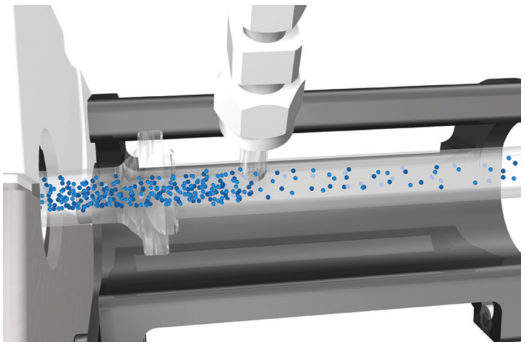

Figure 1. Schematic of HMI, showing aerosol dilution

Analysis of Rock Digests using Agilent 7700x with HMI and ISIS-DS

Four rock reference materials (0.5 g or 0.25 g) were digested using a mixed acid attack, and diluted to 125 mL final volume in 1% HNO_3, giving a nominal total dissolved solids level of 0.4% or 0.2%. The digests were analyzed directly using the 7700x ICP-MS with ISIS-DS. A 2-point initial calibration was performed and the samples were analyzed repeatedly in a random order for a total of ~700 analyses. 42 isotopes were measured in 61 seconds, run to run, and the internal standard recoveries for the whole sequence are shown in Figure 2.

Visual inspection of the sampler and skimmer cones before and after the 700 sample run showed virtually no matrix accumulation, and comparison of the instrument tune conditions before and after the sequence confirmed negligible impact on the interface or ion lens from the prolonged exposure to these high matrix samples (Table 1).

The analysis of the four rock reference materials showed accurate analysis of all elements in the concentration range from <1 ppb to >100 ppm in the solutions measured, with %RSD values typically around 5% RSD (external precision from randomized analysis). This exceptional stability and matrix tolerance demonstrates a robust new high-throughput ICP-MS method for analysis of mining samples.

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

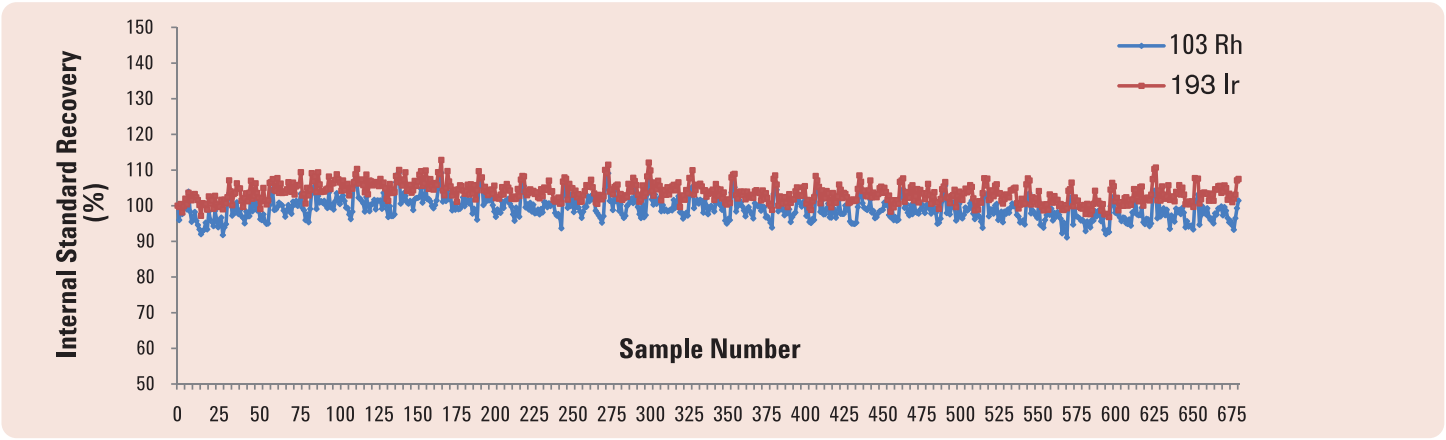

Figure 2. Internal standard recovery for the entire sequence of ~700 high-matrix rock digests, showing excellent stability throughout the sequence

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