

## **ABSTRACT**

An improved slurry method for determination of PGMs, gold and base metals (Ni, Cu, Fe and Co) using ICP-OES analytical instrument has been developed. This method has been successfully applied to quantitate metals in mineral concentrates and biological samples (grass, lichens, leaves and tree trunks) and validated using analytical figure of merit that include limits of detection, limits of quantitation, accuracy and precision.

Sample grinding using Fritsch GmbH Pulverisette Planetary Mono Mill was optimized to get > 60% and >80% of the particles to < 5.0  $\mu\text{m}$  and < 10  $\mu\text{m}$  respectively. The optimum grinding conditions for a sample mass of 0.4 g was determined as 130 rpm grinding speed and 30 minutes grinding time. An additional 15 minutes mixing with reagent was employed to ensure better slurry homogeneity for improved precisions.

Five slurry dispersants; tetrasodium pyrophosphate, Triton X-100, dodecylbenzene sulfonic acid sodium salt, potassium cyanide and potassium thiocyanate, were evaluated at varied concentrations and 0.1% v/v Triton X-100 established as the best dispersant for slurry preparation.

The ICP-OES sample delivery system was modified by introducing an extension elbow between the torch and the spray chamber that not only effectively reduced the sample residence time in the plasma for efficient atomization of the particles but also acted as a gravitational sieve eliminating larger particles and droplets.

Prepared slurries were continuously agitated using an ultrasonic bath prior to and during aspiration into ICP-OES to prevent sedimentation and ensure constant stability of the slurry. Optimum operating conditions for ICP-OES established as

1600 W and 1450 W plasma power for PGMs and base metals respectively, using a nebulizer flow rate of 0.8 mL min<sup>-1</sup>, coolant gas flow rate of 13.0 mL min<sup>-1</sup>, auxiliary gas flow rate of 1.0 L min<sup>-1</sup> with a sample uptake rate of 0.2 mL min<sup>-1</sup>.

Limits of detection for the developed technique were determined in µg g<sup>-1</sup> as 0.075, 0.039, 0.115, 0.071, 0.035 and 0.118 for Au, Ir, Pd, Pt, Rh and Ru respectively and 0.15, 0.391, 0.345 and 0.217 for Ni, Cu, Fe and Co respectively. Limits of quantitation in µg g<sup>-1</sup> were obtained as 0.250, 0.130, 0.383, 0.237, 0.117 and 0.393 for Ir, Pd, Pt, Rh and Ru respectively and 0.5, 1.31, 1.15 and 0.723 for Ni, Cu, Fe and Co respectively. Good accuracy was recorded for all the base metals as well as for the platinum group metals and gold except iridium that was susceptible to copper interference.

Two measurements of precision, reproducibility and repeatability were assessed. Reproducibility was obtained in percentages as 12, 7.93, 5.54, 10 and 12 for Au, Pd, Pt, Rh and Ru respectively and 9.9, 10, 9.8 and 10 for Ni, Cu, Fe and Co respectively. Repeatability was obtained in percentages as 11, 7.03, 4.94, 8.08 and 9.85 for Au, Pd, Pt, Rh and Ru respectively and 9.4, 9.6, 9.7 and 10 for Ni, Cu, Fe and Co respectively. Good percentage recoveries were obtained of 104.3%, 98.3%, 98%, 90.5% and 92.8% for Au, Pd, Pt, Rh and Ru respectively. Percentage recoveries of 109%, 111%, 89.9% and 100% for Ni, Cu, Fe and Co respectively were obtained.

The simplicity and low cost of sample preparation used in this method enables it to be easily adopted in any ICP-OES laboratory. A single analyst may achieve more sample through-puts than before per day (including re-grinding and analysis), resulting in a highly economical and rapid technique for determination of metals.