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The optimization and calibration of spark-optical emission spectroscopy for the analysis of trace impurities in ultra-pure Pt, Pd and Rh

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ABSTRACT

Since the industrialization of platinum group metals (PGMs), particularly platinum (Pt), palladium (Pd) and rhodium (Rh), the control of trace impurities in these metals has become vital. Reliable analysis of impurities in these metals is, however a difficult task. Nobel metals are difficult to dissolve quickly and quantitatively. Thus, analytical techniques which determine samples in liquid form have become less favourable. They require time consuming digestions and are prone to contamination both from the chemicals and equipment used for the preparation. Direct-analysis techniques are increasingly being used in the platinum group metal (PGM) industry for the determination of impurities in the final products.

Spark-optical emission spectroscopy (Spark-OES) for the analysis of metals offers rapid turnaround times. Since the technique is almost non-destructive, little of the product is lost during analysis. The technique is also well established in the PGM industry. It is routinely used by two of the three largest platinum producing companies for the determination of impurities in their products. It is also used for the determination of PGMs after Fire Assay procedure by Anglo American Platinum. The greatest challenge for this technique remains the availability of certified reference materials (CRM) and calibration standards.

This study investigates the use of the Spark-OES for the determination of impurities in PGMs (notably gold (Au), silver (Ag), iron (Fe), nickel (Ni), copper (Cu), lead (Pb), magnesium (Mg), manganese (Mn), silicon (Si), aluminium (Al), antimony (Sb), chromium (Cr), tin (Sn), titanium (Ti), zirconium (Zr), calcium (Ca), zinc (Zn), boron (B), cobalt (Co), vanadium (V), molybdenum (Mo), bismuth (Bi), arsenic (As), selenium (Se), tellurium (Te), cadmium (Cd) in refined platinum, palladium and rhodium metals). It is to be used at Anglo American Platinum's final metal's laboratory. A method to be used routinely in the laboratory is also developed. The concentration of the impurities determined is used to quantify the overall purity of the PGMs. PGMs, other than the matrix (the metal whose purity is being quantified), are also determined. The use of

Spark-OES was evaluated as an alternative to inductively coupled plasma-optical emission spectroscopy (ICP-OES).

Due to the lack of CRMs and calibration standards, the study included the preparation of in-house reference material (IRM) for calibration and quality control purposes. The standards were prepared by spiking pure PGM metal sponges (produced by Anglo Platinum) with the metal oxides of the elements of interest. These were melted together using a vacuum induction furnace to produce metal disks. The disks were ground and analysed after dissolution using ICP-OES. The metal disks, and the shavings, were distributed to three other independent laboratories and analyzed by ICP-OES, inductively coupled plasma-mass spectroscopy (ICP-MS) and Spark-OES. The assigned consensus values were used for the calibration of the Spark-OES. The method was validated for linearity, accuracy, precision, robustness, bias and the measurement uncertainty of the method.

The metal disks were first tested for homogeneity. It was found that the bottom surface of the rhodium metal was not homogenous. Rapidly cooled moulds, will facilitate almost instantaneous cooling of the metal. This eliminates the migration of elements during cooling. This could assist with homogenizing the metal. Limits of detections (LODs) achieved for the methods ranged from 0.1 $mg.kg^{-1}$ to 4 $mg.kg^{-1}$. The highest LOD was for silicon, which was caused by contamination from the crucibles used. The precision for all impurity elements, except ruthenium (Ru), of the three methods (analysis of platinum, palladium and rhodium) was satisfactory. Ru showed poor precision in all the matrices due to the channel installed in the spectrometer. Due to the lack of CRMs, the traceability of the method could not be validated and the accuracy could only be validated by comparing it to in-house reference material.

Although the method met the validation criteria, it cannot be used to certify the purity of the product as the traceability could not be validated. It suggests that the method be used for twin stream analysis in conjunction with a primary method. Because of its rapid turnaround time, and its non-destructive nature, the method can be used for plant

control purposes, where the level of accuracy required is not as stringent as required on a certificate of analysis.