

DETERMINATION OF PLATINUM GROUP ELEMENTS AND GOLD IN REFERENCE MATERIALS BY INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS AND INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY WITH NICKEL SULPHIDE FIRE-ASSAY COLLECTION

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The natural abundance of PGE (Ru, Rh, Pd, Os, Ir, Pt) and Au is very low (ng g^{-1} or sub ng g^{-1}) and the usual methods for the determination of these elements in geological samples consist of a pre-concentration procedure followed by detection using a high sensitive analytical technique. The NiS fire assay, used as a collector to concentrate all noble metals, is a classical pre-concentration method due to the possibility of using large samples, eliminating problems of heterogeneous distribution in geological matrices.

In this work, Instrumental Neutron Activation Analysis (INAA) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) after NiS fire assay were used to determine PGE and Au in the geological reference materials peridotite GPt-3 and pyroxene peridotite GPt-4 (IGGE, China). INAA has been one of the most useful analytical techniques for PGEs and Au determination, due to its high sensitivity and accuracy. After the fire assay, the NiS button is dissolved in concentrated hydrochloric acid, leaving a residue of insoluble PGE sulphides; the solution is filtered and the filter is directly irradiated with neutrons. In more recent years, ICP-MS with nickel fire assay collection and tellurium coprecipitation has been used as a mean of analysing PGE, with the main advantage of avoiding problems of losses of PGE during the HCl digestion of the NiS button. Buttons were prepared by mixing the sample (10-15g) with fluxes, nickel and sulphur in a fire clay crucible and fused at temperatures around 1000°C . The ICP-MS analysis were performed with an HR-ICP-MS Instrument, Element, Finnigan MAT. For NAA, the filters containing the PGEs and Au were irradiated at the research nuclear reactor IEA-R1 of IPEN. The measurements of the induced gamma-ray activity were carried out in an hyperpure Ge detector (Canberra).

In general, the results obtained by both techniques were in good agreement with recommended values. INAA results exhibited higher values than the recommended values for Pd and Pt in GPt-3, while the opposed effect was observed for sample GPt-4. Ru was not detected by INAA. On the other hand, Rh and Ir were determined more accurately by INAA (relative errors better than 10%). The ICP-MS analytical technique showed better detection limits, and all the PGE were determined. Results obtained for Pt and Pd presented accuracy better than 5% while losses were observed for Os and Ir.