DETERMINATION OF PLATINUM GROUP ELEMENTS IN REFERENCE MATERIALS WITH NICKEL SULFIDE FIRE-ASSAY COLLECION AND INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

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The concentration of platinum group elements (PGE: ruthenium, rhodium, palladium, osmium, iridium, platinum) in common rocks and soils is generally less than 1 ng g^{-1}, so sensitive and reliable analytical methods are required to their determination. Few reference materials with certified values of PGE at the ng g^{-1} level are available to test analytical methodologies. The Chinese reference materials GPt 1-7, prepared by the Institute of Geophysical and Geochemical Exploration (IGGE), are adequate to this purpose.

In this work, the PGE have been determined in the reference materials, GPT-3 and GPT-4, after a pre-concentration by nickel sulfide fire-assay and Instrumental Neutron Activation Analysis (INAA).

The nickel sulfide fire assay is a classical pre-concentration method of PGE, and the use of large samples (10 to 50g) avoid problems of heterogeneous distribution [1]. INAA was one of the first analytical techniques used in the determination of PGE in geological samples. The combination of nickel sulfide fire assay and INAA have been employed successfully for the determination of PGE[2]. The NiS button is dissolved in hot hydrochloric acid and the insoluble PGE sulfides are filtered. The filter paper is irradiated with neutrons and the induced activity is determined by gamma-ray spectrometry.

The analytical procedure consisted of drying the sample for 200^0 C for 2 h followed by ashing at 650^0 C for 4 h. About 15g of the sample and a mixture of 20g of sodium tetraborate (anhydrous GR, Merck), 10g of sodium carbonate (anhydrous extra pure Merck), 1g of nickel powder (INCO Metals), and 0.75g of sulfur (Merck) were homogenized, transferred to a fire clay crucible and submitted to fusion (950^0 C for 30 min and 1050^0 C for 30 min). After the fusion, the crucible was allowed to cool and it was broken to separate the nickel sulfide button. The button was weighed, crushed and weighed again. A blank was prepared by using 15 g of silica instead of the sample. The dissolution of the NiS button was made with 20 mL of hot concentrate hydrochloric acid. The precipitate was filtered in a 0.45µm Millipore filter paper, rinsed with distilled water and dried in a desiccator. Standards containing 10µg of each of the PGE were prepared by pipetting a convenient aliquot of a PGE standard solution (Specpure ALFA AESAR) of PGE onto a 0.45µm Millipore filter paper. Standards and samples were packed in polyethylene vials and were irradiated at the research nuclear reactor IEA-R1m of IPEN for 5 min at a thermal neutron flux of 5.10^{11} n cm^{-2} s^{-1} for the determination of Rh and Pd, and for 8 h at a thermal neutron flux of 10^{12} n cm^{-2} s^{-1} for the determination of Pt, Ru, Os and Ir. The gamma-ray spectrometry was performed in a Canberra GMX20190 HPGe detector.

New results for the PGE in the analysed reference materials are presented. Some discrepancies with the recommended values are discussed on basis of the difficulties related to the accurate determination of the PGE by using NiS fire assay and INAA.

References

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