

PGE-sulfide standard synthesis for LA-ICP-MS

Cora C. Wohlgemuth-Ueberwasser

Institute of Mineralogy, University of Muenster, Corrensstr. 24, 48149 Muenster, Germany

ueberwas@nwz.uni-muenster.de

Chris Ballhaus

Institute of Mineralogy, University of Muenster, Corrensstr. 24, 48149 Muenster, Germany

chrisb@uni-muenster.de

Thomas Meisel

General and Analytical Chemistry, University of Leoben, Franz-Josef-Str. 18, 8700 Leoben, Austria

thomas.meisel@notes.unileoben.ac.at

Jasper Berndt

Institute of Mineralogy, University of Muenster, Corrensstr. 24, 48149 Muenster, Germany

jberndt@uni-muenster.de

Abstract:

Laser-ablation-ICP-MS is a powerful tool for the situ trace element analysis of Re and platinum-group elements (PGE) in natural and synthetic samples. Re and the PGE occur to a large extent in solid solution in sulfide, and therefore the most suitable standards are sulfides.

Sulfides were synthesized from metal powders and elemental S in evacuated SiO₂ glass tubes. Re and PGE concentrations are around 30 ppm and were added as chloride solutions. Reaction to sulfide powders proceeded in evacuated silica glass tubes for about 40 h between 250 and 750°C, then sintered in a piston-cylinder press at 1 GPa to obtain a sulfide pellet with theoretical density. About one third of each sulfide pellet was analyzed with isotope dilution for bulk Re plus PGE. The remainder was polished and analyzed with an ArF excimer laser ($\lambda=193\text{nm}$) coupled to a single collector ICP-MS. Count rates were normalized to ³⁴S and the S content of each charge. To achieve homogeneity, FeS-based sulfides should always be slightly metal-deficient for high defect concentrations promote PGE substitution in sulfide. Partial melting needs to be avoided during all synthesis steps as PGE are strongly fractionated between crystalline monosulfide and sulfide melt. Synthesis should preferably be in capsule materials that do not interfere with sulfide via O₂-S₂ redox equilibria, and therefore graphite, boron nitride, or MgO should not be used.

Our most homogeneous standard is a Fe_{1-x}S composition with 2 wt. % Ni and an atomic metal/S ratio of 0.89. The 1-sigma variations in ³⁴S-normalized count rates of 16 replicate Re-PGE analyses are < 3.6%. Nearly as homogeneous is a pure Fe_{1-x}S standard with metal/S = 0.90 and 1 sigma variations < 5.8. Fe_{1-x}S monosulfide with 2 wt. % Cu proved difficult to homogenize since Cu in solid solution appears to promote PGE heterogeneity. The most problematic composition was pure NiS. Contrary to previous experiences for synthetic glass (Eggins 1998) partial melting of a sulfide target under the laser beam does not seem to cause increase apparent standard heterogeneity.

References

Eggins, S. M., Kinsley, L. P. J., Shelley, J. M. G. (1998). Deposition and element fractionation processes during atmosphere pressure laser sampling for analysis by ICP-MS. Applied Surface Science, 127-129, 278-286