Improvement of rapid quench techniques to obtain reliable quantitative analysis data from historical copper-slag compositions

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Quench techniques to produce glasses from rock powders have been reported for more than 25 years (Foland and Wagner 1981). As a first aim, these glasses were used to perform rapid quantitative analyses by electron microprobe. The method became increasingly important with developing Laser-ICP-MS analysis. Matrix-free preparation, no sample weight errors, rapidness and a great flexibility are the key benefits of the method. Although the method works fine for basic rock samples, several restrictions exist if unusual materials are used. They are rarely discussed in the literature or simply ignored:

1. Oxygen fugacity plays a crucial role, when iron rich (>30% FeO) samples are prepared.
2. Although a loss of elements appears to be negligible in basaltic rocks it can be observed in phonolitic rock samples and silica-poor slags.
3. Segregation and heterogeneities are observed in silica rich and refractory materials.
4. Slow quench gradients lead to crystallisation in low-silica systems.

Iridium or tungsten strip heaters are normally used for preparing the glass beads.

For our investigations we use a vertically mounted graphite resistance furnace (Perkin Elmer HGA 500) formerly used for flame-less atomic emission spectroscopy. Sample powder is fixed on a Pt-wire and, after fusion, dropped into a water-filled cylinder. Rapid quenching in water is essential to obtain glasses from materials containing less than 35 % SiO_2.

Although the method is established for fayalitic slags since 1994 (Kronz), it did not produce reliable results for slags from medieval non-ferrous metal production. The slags are characterised by high amounts of ZnO (10-27 mass-%), high FeO (30-50 mass-%), low SiO_2 (15-32 mass-%) and considerable concentrations of PbO, CuO, As_2O_3, Sb_2O_3 and S. Loss of Zn and other "volatiles" lead to erratic analytic results even at low fusion temperatures. Because the crystallisation of spinels started at 1200 ^oC there was no possibility to lower the fusion temperature to minimize above mentioned effects. The addition of 25-30 % lithium-tetraborate to the sample powders enabled us to produce homogenous glasses at 1000 ^oC without any visible crystallisation. Electron microprobe analysis gave reproducible results and a good concordance of reported concentrations in Zn-rich standard materials as proof of the validity of the method. For a proper matrix correction by EPMA the $Li_2B_4O_7$ amounts have to be taken into account.

References

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