American Mineralogist, Volume 90, pages 1131–1138, 2005

Effect of current density on the electron microprobe analysis of alkali aluminosilicate glasses

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ABSTRACT

In a previous work (Morgan and London 1996), we proposed an optimized procedure for electron microprobe analysis (EMPA) of rhyolitic glasses using a broad (20 μm diameter), low-current (2 nA) fixed beam. Some important applications for EMPA of glass, such as vitreous inclusions in minerals and experimental run products, require smaller beam diameters that produce greater areal current densities (expressed as $nA/\mu m^2$). For these situations, we have assessed the effect of areal current density on the migration of Na and its concomitant effects on other elements and their ratios during EMPA of granitic glasses. Anhydrous and hydrous glasses of a haplogranite composition ($Ab_{38.23}Or_{29.31}Qtz_{33.37}C_{0.10}$) were analyzed at 20 kV accelerating potential, using 2–50 nA beam currents, fixed beam diameters of 2–20 μm, and counting times scaled to yield similar analytical uncertainty at each condition (~2.6% relative for Na_2O). There is almost no loss of Na (≤1.7–2.7% relative) using a current density of 0.006 $nA/\mu m^2$, minor (7–9%) Na loss for current densities up to 0.1 $nA/\mu m^2$, and increasing Na loss with higher current densities that becomes severe at >0.5 $nA/\mu m^2$ (e.g., 48–63% relative loss from hydrous glass at 50 nA and 2 μm during 3–6 s of irradiation). Sodium migration is more pronounced in hydrous glasses than in anhydrous ones, with significant loss from hydrous glass occurring during the first second of irradiation. The migration of Na results in increased concentrations of Al and Si, but little or no change in the concentration of K; if not fully corrected for, these effects produce systematic errors in important elemental ratios. With current densities <0.01 $nA/\mu m^2$, anhydrous glasses or crystalline materials are suitable standards and data correction may not be needed. Significant Na loss using current densities up to ~0.1–0.2 $nA/\mu m^2$, especially in hydrous glasses, requires data correction or primary standardization utilizing a glass having composition and water content similar to the unknown. Current densities ≥0.5–1.0 $nA/\mu m^2$ are not suitable for EMPA of glass because of large and uncertain corrections (~25% to >100% of the Na_2O value obtained).

The correlation of analytical condition (beam current and diameter) with current density and EMPA results provided here allows analysts to select beam conditions that optimize the quality of analyses. When current densities >~ 0.01 $nA/\mu m^2$ must be used (e.g., with beam spot sizes <20 μm), the results can lead to improved estimates of the systematic errors due to alkali migration. Natural and some experimental glasses contain a variety of other minor components among which Ca and Fe are important, and so the discussion of analytical methods is extended to more complex compositions. For example, Na migration is accelerated as glass structures become less polymerized than those of simple tectosilicate stoichiometry (e.g., due to increasing alkalinity and/or the presence of fluxing components such as F, Cl, B). Analysis using 20 kV accelerating voltage, as opposed to 15 kV, both slightly decreases Na migration and improves limits of detection and statistical accuracies for minor components such as Fe while providing reasonable beam penetration depths.