

Electron probe microanalysis of trace sulfur in experimental basaltic glasses and silicate minerals

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ABSTRACT

Sulfur (S) in the mantle is conventionally assumed to be exclusively stored in accessory sulfide phases, but recent work shows that the major silicate minerals that comprise >99% of the mantle could be capable of hosting trace amounts of S. Assessing the incorporation of trace S in nominally S-free mantle minerals and determining equilibrium S partitioning between these minerals and basaltic melt requires analyzing small experimental phases with low S contents. Here, we develop a protocol for EPMA analysis of the trace levels of S in silicate phases. We use a suite of natural and experimental basaltic glass primary and secondary standards with S contents ranging from 44 ppm to 1.5 wt%. The effects of beam current and counting time are assessed by applying currents ranging from 50 to 200 nA and total counting times between 200 and 300 s at 15 kV accelerating voltage. We find that the combination of 200 nA beam current with a 200 s counting time (80 s peak, 60 s each for upper and lower background, respectively) achieves precise yet cost-effective measurements of S down to a calculated detection limit of ~5 ppm and a blank-derived, effective detection limit of ~17 ppm. Close monitoring of the S peak intensity and position throughout the duration of each spot also shows that high currents and extended dwell times do not compromise the accuracy of measurements, and even low S contents of 44 ppm can be reproduced to within one standard deviation. Using our developed recipe, we analyzed a small suite of experimental clinopyroxenes (Cpx) and garnets (Gt) from assemblages of silicate partial melt + Cpx ± Gt ± sulfide, generated at 1.5 to 3.0 GPa and 1200 to 1300 °C. We find S contents of up to 71 ± 35 ppm in Cpx and 63 ± 28 ppm in Gt and calculate mineral-melt partition coefficients ($D_s^{\text{min/melt}}$) of up to 0.095 ± 0.064 and 0.110 ± 0.064 for $D_s^{\text{Cpx/melt}}$ and $D_s^{\text{Gt/melt}}$, respectively. The sulfur capacity and mineral-partitioning for Cpx are in good agreement with SXRF measurements in a prior study by Callegaro et al. (2020), serving as an independent validation of our EPMA analytical protocol.

Keywords: Electron probe microanalyzer, sulfur solubility, mantle, silicate glass, clinopyroxene, garnet, partitioning