An electron microprobe analysis, secondary ion mass spectrometry, and single-crystal X-ray diffraction study of phlogopites from Mt. Vulture, Potenza, Italy: Consideration of cation partitioning

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ABSTRACT

Mt. Vulture trioctahedral micas-1M mainly consist of phlogopite-annite solid solutions with a minor component of brittle micas. However, both Li-free and Li- and F-rich compositions may coexist in the same volcano-stratigraphic level. We report the results of electron microprobe analysis (EMPA), secondary ion mass spectrometry (SIMS), and single-crystal X-ray diffraction (SCXRD) for three crystals (LC7-27Go, LC7-3Go, and LC7-1R) that are representative of both compositions.

EMPA and SIMS showed that sample LC7-1R is richer in TiO_2, Li_2O, and F and poorer in H_2O than the other samples.

Structure refinements using anisotropic displacement parameters for the three samples, performed in space group $C2/m$, converged at $3.46 \leq R \leq 4.34$, $3.89 \leq R_w \leq 4.39$. When considering bond distances, the three samples can be described as homo-octahedral whereas mean atomic numbers suggest that only LC7-1R is meso-octahedral. Significant differences occur among the samples for some distortion parameters commonly used for micas. In particular, LC7-1R displays higher values of BLD_{M2}, $shift_{M2}$ and lower values of Δ_{K-O4} and t_{K-O4} than those of samples LC7-3Go and LC7-27Go. These differences are ascribed to F- and Ti-substitutions.

Cation distributions were obtained by combining EMPA, SIMS, and SCXRD data after analysis of the effect of normalization schemes commonly used in mica formula recalculation. In LC7-3Go and LC7-27Go, Ti is incorporated according to the Ti-Tschermak mechanism, whereas more than one Ti-substitution mechanism occurs in LC7-1R. For the latter sample, octahedral vacancies are present leading to a complex substitution pattern and complex structural distortions related to the special octahedral compositions that were determined.

Keywords: Analysis, chemical (mineral), EPMA and SIMS investigation, crystal structure, trioctahedral micas –1M, major and minor elements, Ti-F-bearing phlogopites, trace elements and REE, Li-bearing micas, XRD data, single-crystal structure refinement