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Multi-analytical characterization of an unusual epidote-supergroup mineral from Malmkärra, Sweden: Toward the new (OH)-analog of dollaseite-(Ce)

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ABSTRACT

A study of a skarn sample from the Malmkärra iron mines, Norberg, Västmanland (Sweden), revealed the occurrence of a peculiar epidote-supergroup mineral. It was examined using electron microprobe, single-crystal X-ray diffraction, Mössbauer, and Fourier-transform infrared spectroscopy (FTIR) techniques. Structure refinements combined with electron microprobe data indicate the following cation populations: $A1 = \text{Ca}_{0.96}\text{REE}^{3+}_{0.03}\text{Mn}_{0.01}$; $A2 = \text{REE}^{3+}_{0.99}\text{Ca}_{0.01}$; $M1 = \text{Mg}_{0.40}\text{Al}_{0.32}\text{Fe}^{3+}_{0.26}\text{Fe}^{2+}_{0.02}$; $M2 = \text{Al}_{0.98}\text{Fe}^{3+}_{0.02}$; $M3 = \text{Mg}_{0.72}\text{Fe}^{2+}_{0.17}\text{Fe}^{3+}_{0.11}$; $T1,2,3 = \text{Si}_{2.93}\text{Al}_{0.07}$, accounting for a total positive charge of 24.64. The presence of Fe^{2+} is confirmed by Mössbauer data. The remarkable number of divalent cations at both *M*1 and *M*3 (>1 pfu, per formula unit) demands more than one monovalent anion pfu in the structure. As the mineral lacks fluorine, charge neutrality must be achieved through additional H^+ (about 0.4 apfu). Only one independent hydrogen atom is located within the structure, with O10 as donor and O4 as acceptor, as in other epidote-supergroup minerals. Nonetheless, another O-O distance is suitable for a hydrogen bond, namely O10-O2. Although the existence of the additional OH group was not directly proved by vibrational spectroscopy, FTIR data provided information related to this potential O10-O2 bridge. In the IR spectrum acquired, several bands are observed in the OH-stretching region, and a secondary peak at 2140 cm^{-1} could be assigned to the bending mode of the O10-H···O2 group. To shed light on this puzzling observation, one single crystal was subjected to annealing experiments at temperatures from 500 to 700 °C, in 50 °C steps, while a second one underwent a heat treatment at 700 °C. After the heat treatment, the IR spectrum showed a decreased intensity of all observed bands, in agreement with a dehydrogenation occurring at high temperatures. Although the structural position of the second hydrogen is still uncertain, it is reasonable to describe the composition of the epidote-supergroup mineral from Malmkärra as a solid solution between dissakisite-(Ce) (32%), ferriallanite-(Ce) (28%), and a yet undescribed end-member (40%), the (OH)-analog of dollaseite-(Ce), ideally $\text{CaREE}^{3+}\text{Mg}_2\text{AlSi}_3\text{O}_{11}(\text{OH})_2$.

Keywords: Epidote supergroup, allanite group, dollaseite, rare earth mineral, hydrogen, single-crystal X-ray diffraction, infrared spectroscopy, skarn