

## **Synthesis and characterization of low-OH<sup>-</sup> fluor-chlorapatite: A single-crystal XRD and NMR spectroscopic study**

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### **ABSTRACT**

Low-OH apatite of the compositional range  $\text{Ca}_{4.99-5.06}(\text{PO}_4)_{2.98-3.00}\text{F}_{0.51-0.48}\text{Cl}_{0.38-0.36}\text{OH}_{0.14-0.12}$  was synthesized and characterized structurally by synchrotron-based single-crystal X-ray diffraction (XRD), and multiple nuclear magnetic resonance (NMR) spectroscopic techniques. The average structure is hexagonal with space group  $P6_3/m$ . The presence of scattering in the single-crystal diffraction data set, which is incommensurate within the average hexagonal structure, suggests the presence of localized short-range monoclinic domains. Complex lineshapes in the <sup>31</sup>P and <sup>19</sup>F MAS NMR spectra are also consistent with the presence of an incommensurate phase. No evidence was detected for splitting of the Ca2 site into two distinct sites (as had been previously reported for hexagonal ternary apatites). Structure refinement and <sup>19</sup>F{<sup>35</sup>Cl} TRAPDOR NMR experiments verified intercolumnal neighboring of F and Cl atoms (inter-column distance of 2.62 Å) within this low-OH<sup>-</sup> apatite suggesting that long-range neighboring of F and Cl within the apatite anion channels is feasible.

**Keywords:** Apatite, NMR spectroscopy, single-crystal XRD, Mars, Moon, PGE, synthesis