

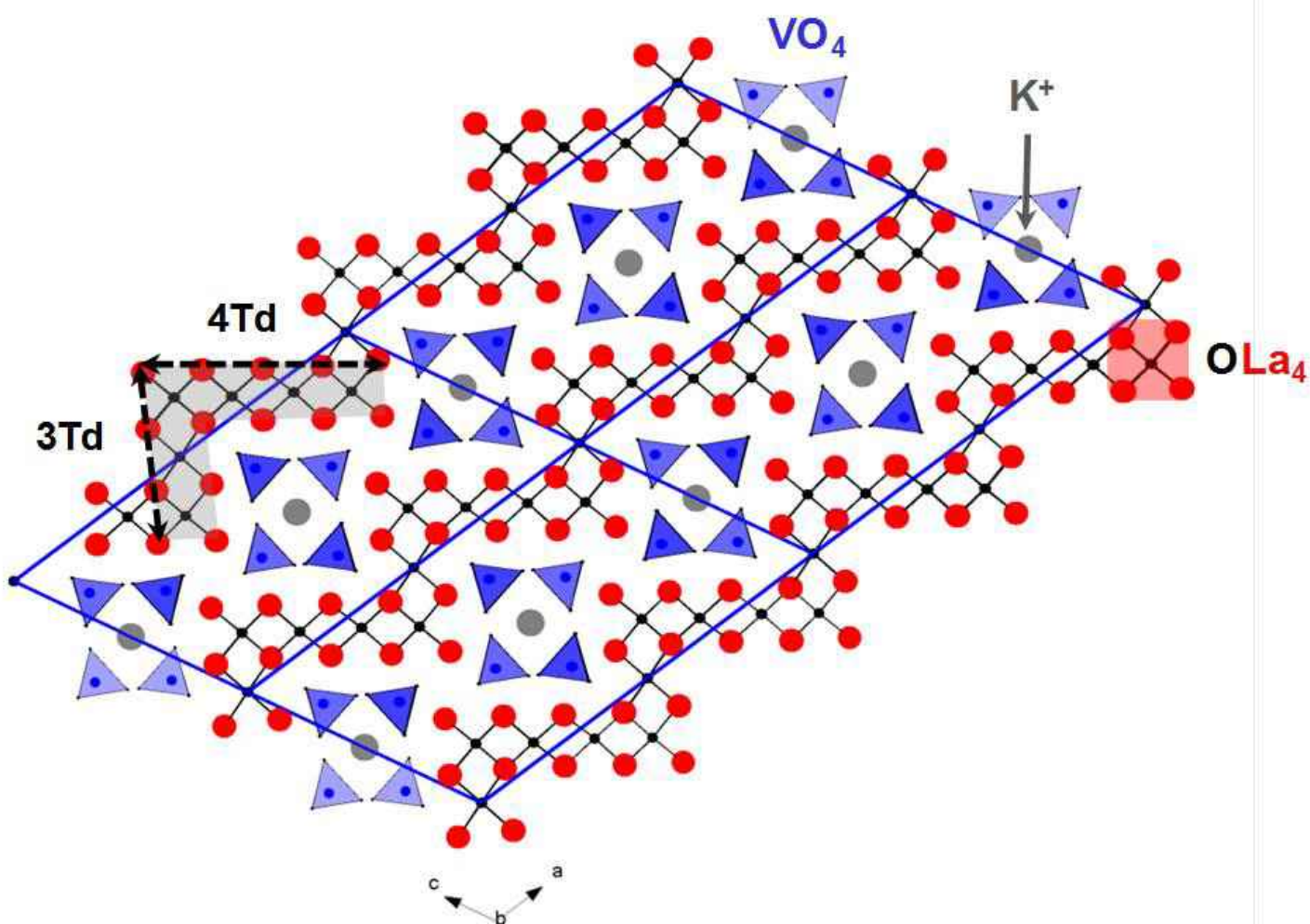
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Use of Precession Electron and X Powder for solution and refinement of materials

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This communication will present the case study of $\text{Ala}_5\text{O}_5(\text{VO}_4)_2$ (A= Li, Na, K, Rb), example of the use of a combination of Precession Electron and X-ray Powder Data for the solution and the refinement of new materials. Indeed, an original structural type has been evidenced in the system (A, La, V, O) with A=Li, Na, K, Rb. Attempts to solve the structure ab initio on X-ray powder data were unsuccessful (more particularly because the powder was a mixture of the title compound and of unreacted precursors). The structure was finally solved by charge flipping using Precession Electron Data (3D tomography) on a nanocrystal, enabling a posteriori the good formulation of a pure powder. This powder was then classically refined by Rietveld method showing the correctness of the electron-solved structure. It crystallizes in a monoclinic unit cell with space group $C2/m$ and $a=20.2282(14)$ $b=5.8639(4)$ $c=12.6060(9)$ Å and $\beta=117.64(1)^\circ$. The $\text{Ala}_5\text{O}_5(\text{VO}_4)_2$ structure is built of (OLa_4) tetrahedral units creating Crenel-like 2D ribbons. These ribbons, surrounded by four isolated VO_4 tetrahedra, are creating channels parallel to b axis in which A^+ ions are located.



Keywords: Precession Electron Data, Rietveld Refinement, New Materials