

Electronic Supplementary Information (ESI)

One-pot Solvothermal Synthesis of Well-ordered Layered Sodium Aluminoalcoholate Complex: A Useful Precursor for the Preparation of Porous Al₂O₃ Particles

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1. Single-crystal X-ray diffraction (SCXRD)

As shown in Fig. S1, the asymmetric unit of the compound is composed of one tetranuclear sodium hexakis(glycolato)tris(methanolato)aluminate complex $\text{Na}_3[\text{Al}_4(\text{OCH}_3)_3(\text{OCH}_2\text{CH}_2\text{O})_6]$. Three of the four Al^{3+} ions are 5-coordinate; each is bound by two bidentate glycolate chelators and one methanolate ligand. The carbon atoms of two of the three methanolate ligands (C13 and C15) are disordered over two positions, as are the two carbon atoms (C5 and C6) of one of the ethylene glycolate chelators. Similarity restraints were used for the 1-2 and 1-3 distances for all atoms involved in the disorders, and rigid bond restraints and similarity restraints were used on the displacement parameters for the entire structure. The displacement parameters of the carbon atom in the minor component of one of the disordered methanolate ligands (C13a) were refined using an isotropy restraint. One free methanol solvent molecule unbound to any Al^{3+} ions but tightly occluded in the interplanar voids is present in combination with the minor component of one of the disordered methanolate ligands; its partial occupancy (~10%) leads to the empirical formula of the title Al complex ($\text{C}_{15}\text{H}_{33}\text{Al}_4\text{Na}_3\text{O}_{15} \cdot 0.1\text{CH}_4\text{O}$).

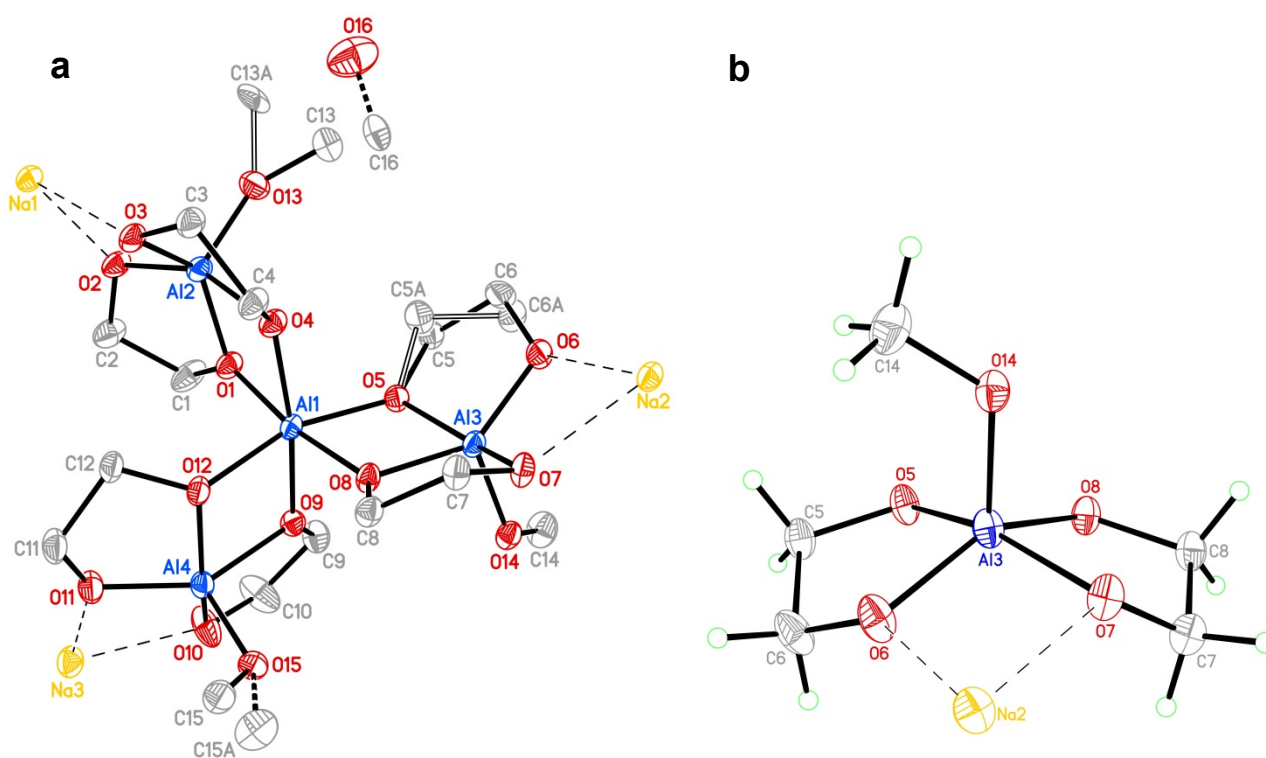


Fig. S1 (a) Asymmetric unit of the title Al complex showing the empirical formula $\text{C}_{15}\text{H}_{33}\text{Al}_4\text{Na}_3\text{O}_{15} \cdot 0.1\text{CH}_4\text{O}$ bearing ~10% occupancy of MeOH solvent. Hydrogen atoms are omitted for clarity, and thermal ellipsoids are set to 50% probability. The two carbon atoms (C5 and C6) of one of the glycolate ligands are disordered over two positions, as are the two carbon atoms (C13 and C15) of these three methanolate ligands. One free methanol solvent molecule (C16 and O16) is present in conjunction with the minor component of one of the two disordered methanolate ligands and thus has only ~10% occupancy. (b) Isolated $[\text{Al}]$ moiety with hydrogen atoms (green circle) from (a).

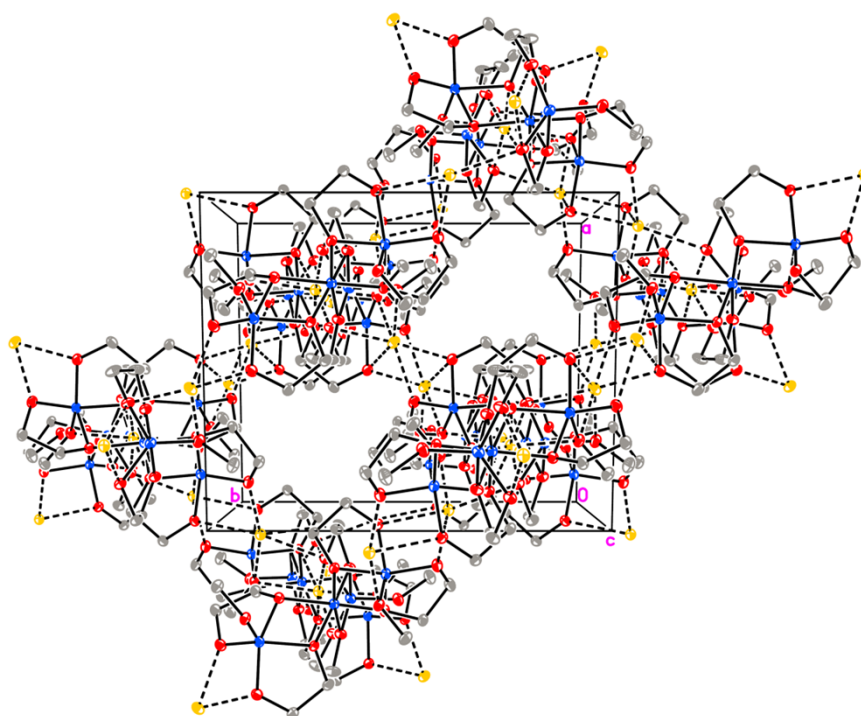


Fig. S2 Packing plot of the Al complex viewed along the crystallographic *c* axis. It is self-assembled into well-ordered layers parallel to the (110) plane. Hydrogen atoms are omitted for clarity. Blue: Al; red: O; gray: C; and yellow: Na.

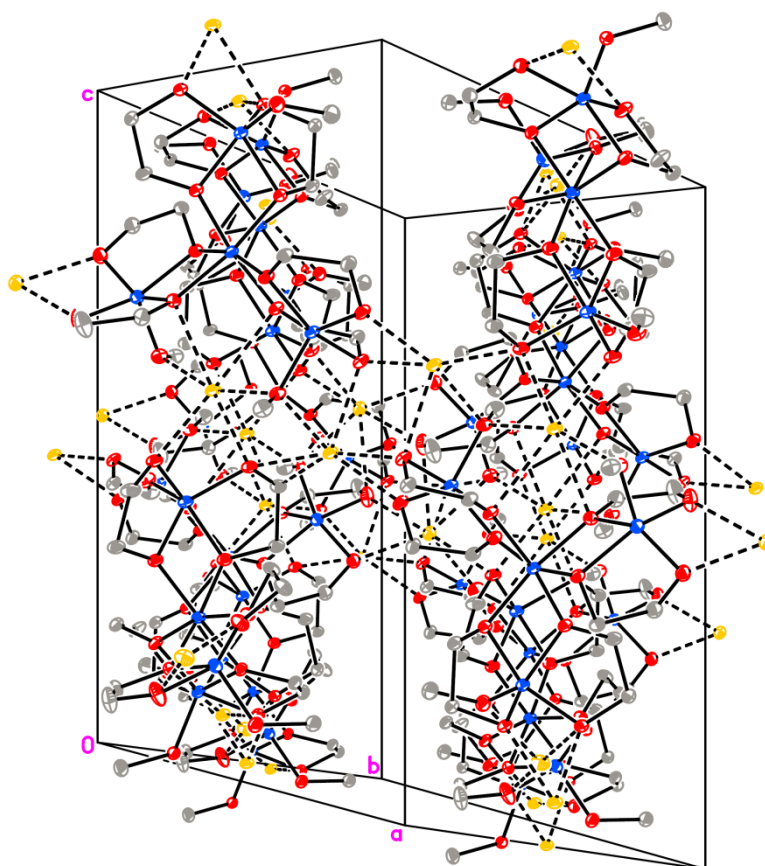


Fig. S3 Packing plot of the Al complex viewed along the crystallographic $[110]$ axis. Hydrogen atoms are omitted for clarity. Blue: Al; red: O; gray: C; and yellow: Na.