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Crystal Structure of $\text{Cd}_3(\text{O}_3\text{PC}_2\text{H}_4\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$: Influence of the Solid State NMR in the Structure Determination. Abraham Clearfield, Naima Bestaoui, Xiang Ouyang, Dept. of Chemistry, Texas A&M Univ., College Station, TX 77843, Florence Fredoueil, Bruno Bujoli, Laboratoire de Synthèse Organique, 2 Rue de la Houssinière, 44322 Nantes, France.

$\text{Cd}_3(\text{O}_3\text{PC}_2\text{H}_4\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$ crystal structure was determined from in-house powder data. This compound crystallizes in the monoclinic system, with the unit cell parameters: $a=8.7774(2)$, $b=9.4844(2)$, $c=9.1695(3)\text{Å}$ and $\beta=113.601(4)^\circ$. The systematic absences were consistent with $\text{P}2_1/c$ and $\text{P}2_1$. This structure was solved and refined at first in the more symmetrical space group. The refinement converged with $R_p=0.1046$, $R_{wp}=0.1378$ and $R_f=0.0763$. However, a few problems remained: (i) all the water molecules could not be detected; (ii) the solid state NMR spectrum could not be explained; the crystal structure showed only two types of cadmium and one type of phosphorus, whereas the ^{113}Cd and ^{31}P MS NMR data showed three cadmium and two phosphorus environments respectively. Clearly, the structure was wrong. Therefore, we lowered the symmetry to $\text{P}2_1$. All the atoms were found and the refinement converged ($R_p=0.0721$, $R_{wp}=0.1014$ and $R_f=0.0407$). In this case, the NMR data can be explained. We will present the needs to have another techniques to prove the accuracy of a powder structure.