Mechanochemical and Solution Reactions Between AgCH₃COO and [H₂NC₆H₁₀NH₂] Yield Three Isomers of the Coordination Network \{Ag[H₂NC₆H₁₀NH₂]\}ₙ

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Figure 1. Observed X-ray powder pattern for 1·nH₂O
Figure 2. Observed X-ray powder pattern for 1·MeOH·0.5H₂O after grinding. Note how the pattern coincides with that observed for 1·nH₂O.

Figure 3. Calculated X-ray powder pattern for 1·MeOH·0.5H₂O.
Figure 4. Observed X-ray powder pattern for $\text{1·}3\text{H}_2\text{O}$ after grinding. Note how the pattern coincides with that observed for $\text{1·}n\text{H}_2\text{O}$.

Figure 5. Calculated X-ray powder pattern for $\text{1·}n\text{H}_2\text{O}$.
Figure 6. Observed X-ray powder pattern for $\text{1}\cdot\text{4H}_2\text{O}$

Figure 7. Calculated X-ray powder pattern for $\text{1}\cdot\text{4H}_2\text{O}$
Figure 8. TGA analysis for $1 \cdot n\text{H}_2\text{O}$, showing that the weight loss corresponds to approximately 2.5 water molecules. At 170°C the sample starts to decompose.
Figure 9. Crystalline 1-MeOH·0.5H₂O. (a) The nitrogen atoms belonging to the organic base interact via hydrogen bonding with the acetate anion and the water molecule [N(1)···O(1)acetate 2.972(5), N(2)···O(2)acetate 3.019(4), N(2)···O(2)acetate 2.993(5), N(1)···O(4)water 3.036(3)Å], and, in turn, the acetate anion interacts both with the water molecule and the methanol ligand [O(3)MeOH···O(1)acetate 2.682(5); O(2)acetate···O(4)water 2.740(4) Å]. (b) The methanol molecule asymmetrically bridges two silver ions belonging to adjacent chains [Ag(1)···O(3)MeOH 2.693(5), Ag(1)···O(3)MeOH 3.139(4)Å] [I: x,1-y,1/2+z; II: 1-x,y,1/2-z; III: x,-1+y,z].

Observations:
1) The OMeOH···Ag distance is comparable to or slightly longer than the ones observed for similar interactions in the CSD [2.394 (QEHCAH); 2.446 (QEHCIP); 2.636 Å (TIDJOF)]

2) A search has been run in the CSD for intermolecular interactions (in the range 2.5-3.5 Å) between silver atoms bound to two N atoms each. A total of 63 compounds has been found, with a mean Ag···Ag distance of 3.266 (19) Å. Therefore the Ag···Ag distance of 3.323(1) Å observed in crystalline 1-MeOH·0.5H₂O is comparable to this mean value.
Figure 10. Crystalline 1·3H₂O. The hydrogen bonded network established between the N-H donor groups on the [H₂NC₆H₁₀NH₂] ligand, the acetate anions [O(1) and O(2)] and the water molecules [O(3), O(4) and O(5)]. Relevant hydrogen bonding interactions (e.s.d.s. 0.005 Å): N(1)···O(2) 3.136, N(2)···O(1) 3.099, N(1)···O(5) 3.014, O(5)···O(3) 2.799, N(2)···O(4) 3.040, O(5)···O(1) 2.904, O(4)···O(2) 2.886, O(3)···O(1) 2.817, O(3)···O(2) 2.753 Å.

(a)     (b)

Figure 11. Crystalline 1·4H₂O. (a) A view of the packing down the c-axis, as shown in Figure 3 (main article), together with the acetate anion and water molecules. (b) The same packing portion rotated by 90 deg around the y (vertical) axis, showing how the acetate anions and water molecules form a layer that is “sandwiched” in between layers constituted of parallel \{Ag[H₂NC₆H₁₀NH₂]⁺\}_∞.
chains. There are two such (crystallographically independent) acetate-water layers in the structure; one of the two layers is shown in details in figure 11c. Relevant hydrogen bonding parameters are 

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\begin{align*}
\text{[O(1), O(2), O(3) and O(4) are the acetate oxygens]:} & \\
N(1)\cdotsO(3)& 3.185(4), N(1)\cdotsO(8A) 3.144(4), \\
N(2)\cdotsO(1) & 3.209(4) 151, N(2)\cdotsO(3) 3.284(4), N(3)\cdotsO(4A) 3.113(4), N(3)\cdotsO(5A) 3.267(4), \\
N(4)\cdotsO(1) & 3.111(4), N(4)\cdotsO(3) 3.021(4), O(1A)\cdotsO(1) 2.884(4), O(1A)\cdotsO(1) 2.757(4), \\
O(2A)\cdotsO(1A) & 2.702(4), O(2A)\cdotsO(4A) 2.779(4), O(3A)\cdotsO(2) 2.849(4), O(3A)\cdotsO(2A) 2.761(4), \\
O(4A)\cdotsO(2) & 2.757(4), O(4A)\cdotsO(3A) 2.781(3), O(5A)\cdotsO(4) 2.761(4), O(5A)\cdotsO(6A) 2.742(4), \\
O(6A)\cdotsO(7A) & 2.748(4), O(6A)\cdotsO(8A) 2.761(4), O(7A)\cdotsO(3) 2.891(4), O(7A)\cdotsO(3) 2.772(4), \\
O(8A)\cdotsO(4) & 2.797(4), O(8A)\cdotsO(5A) 2.789(4) \text{ Å.}
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