

A stepwise mechanism and the role of water in the liquid-assisted grinding synthesis of metal-organic materials

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Supplementary Material

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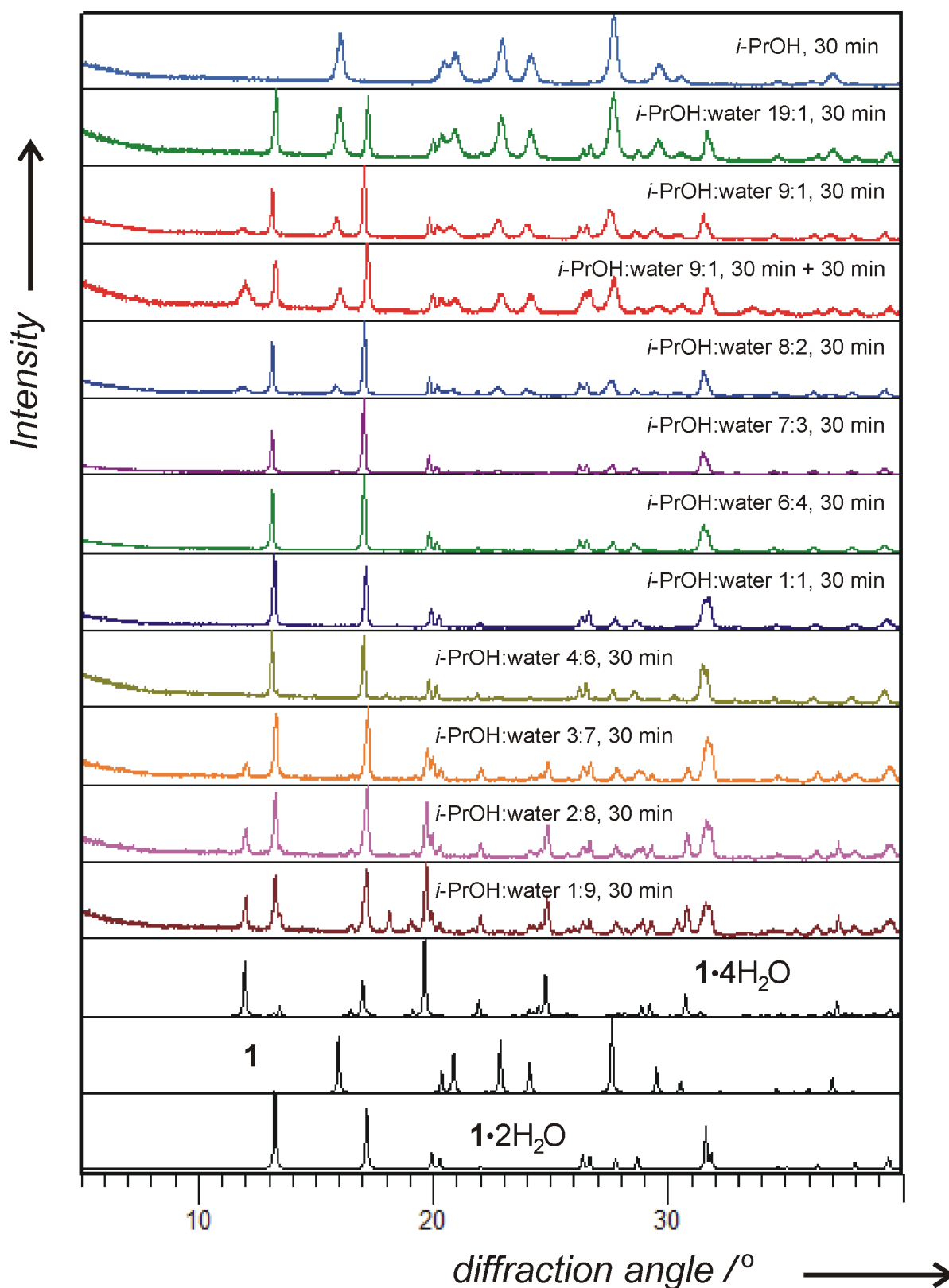


Figure 1. PXRD patterns for LAG experiments using *i*-PrOH and its aqueous mixtures as the grinding liquid. Patterns of experiments that differ only in the time of grinding have the same colour, and simulated patterns are shown in black.

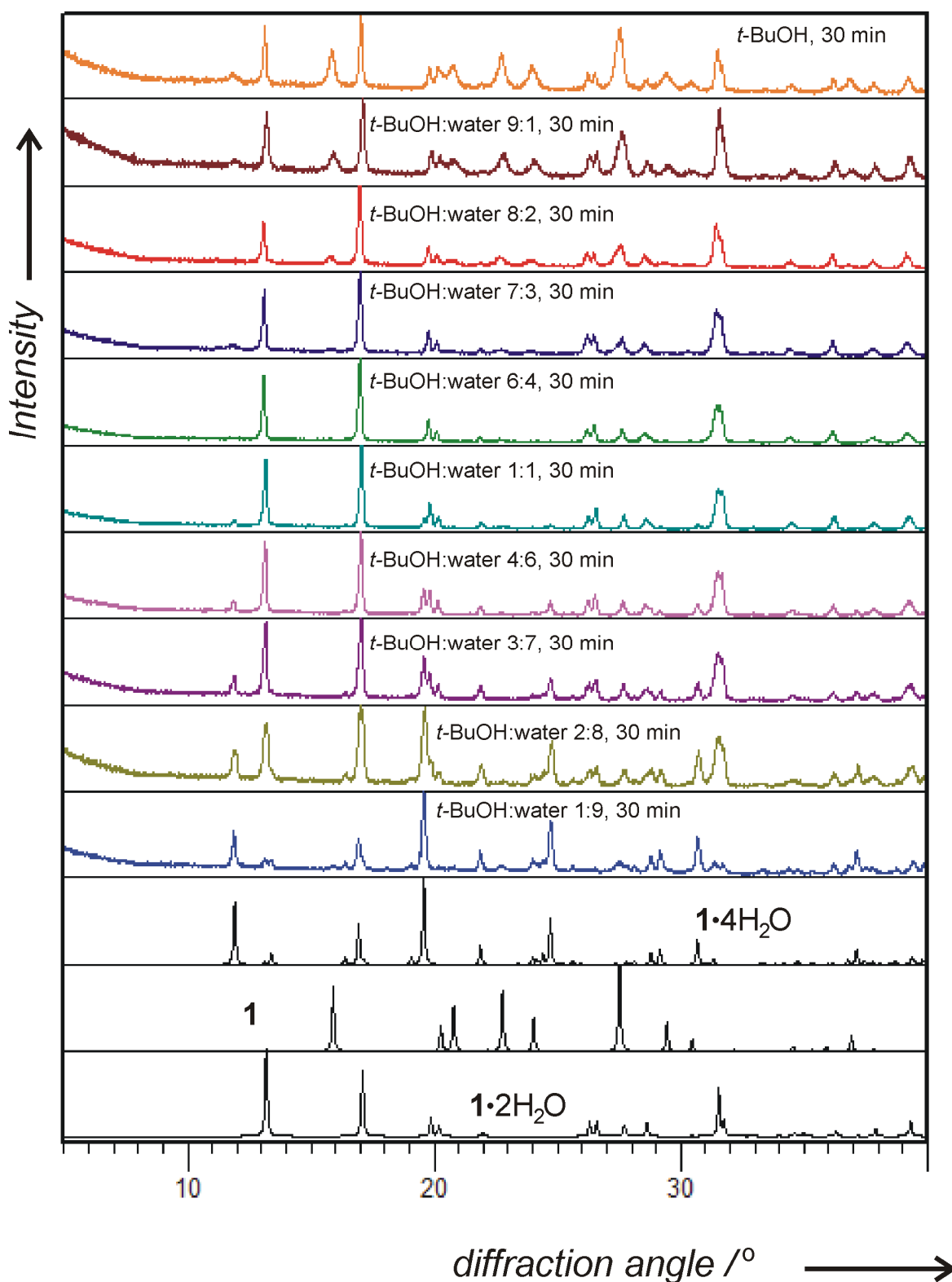


Figure 2. PXRD patterns for LAG experiments using *t*-BuOH and its aqueous mixtures as the grinding liquid. Patterns of experiments that differ only in the time of grinding have the same colour, and simulated patterns are shown in black.

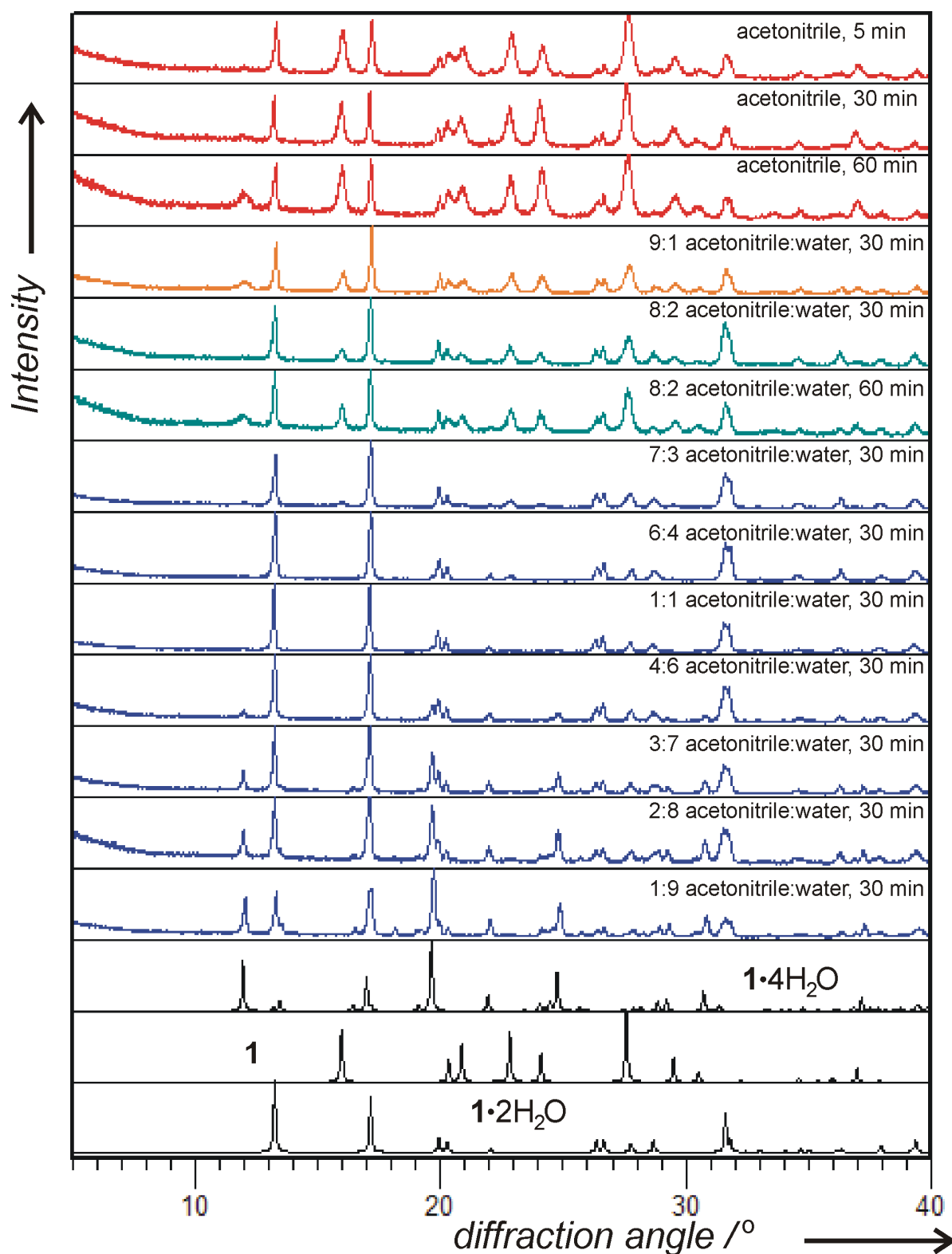


Figure 3. PXRD patterns for LAG experiments using acetonitrile and its aqueous mixtures as the grinding liquid. Patterns of experiments that differ only in the time of grinding have the same colour, and simulated patterns are shown in black.

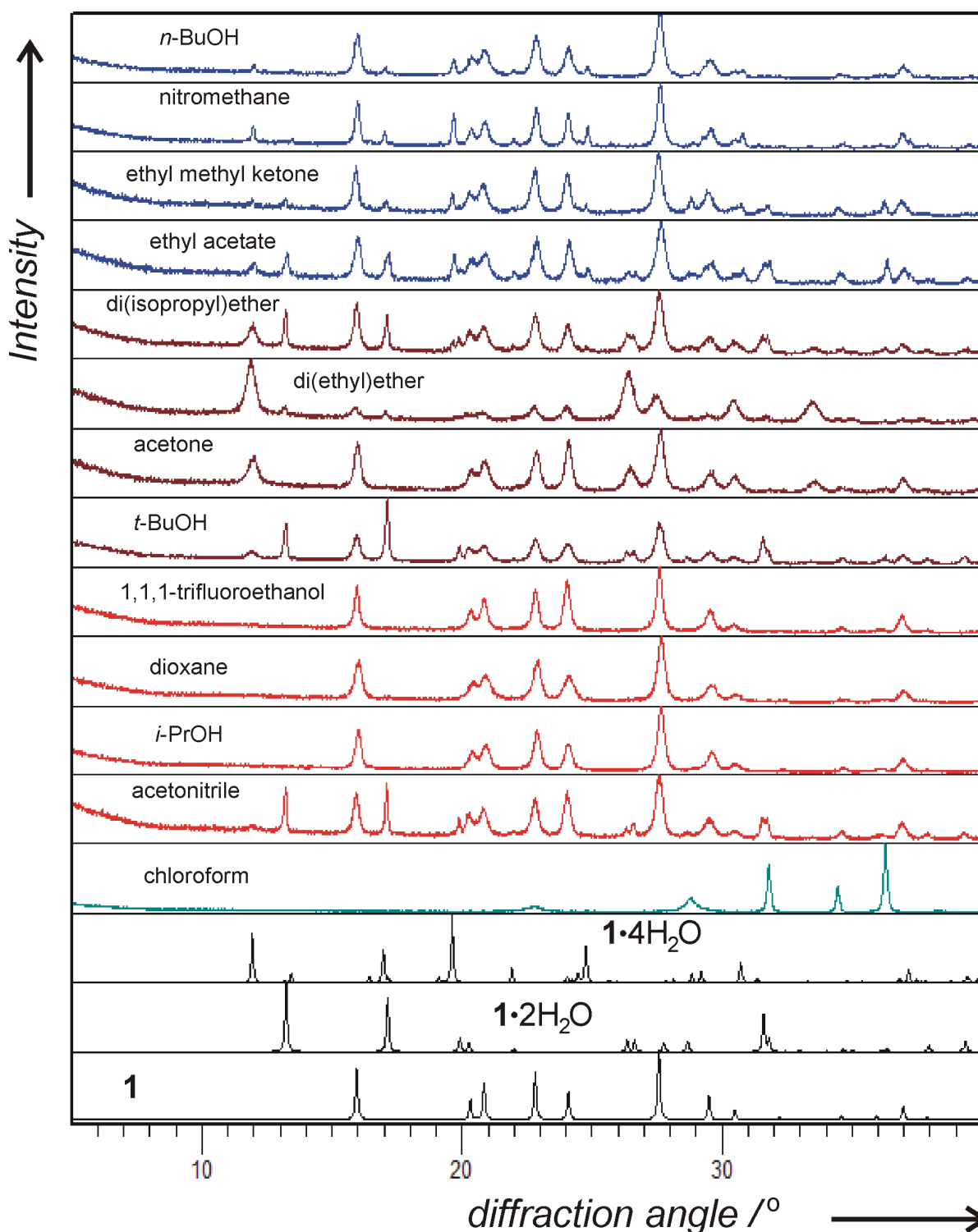


Figure 4. PXRD patterns for LAG experiments involving different organic liquids. The patterns have been colour-coded based on the appearance of 1·4H₂O (blue), 2 (brown) or 1 (red). The LAG experiment involving chloroform is unique and is shown in green, and simulated patterns are shown in black.

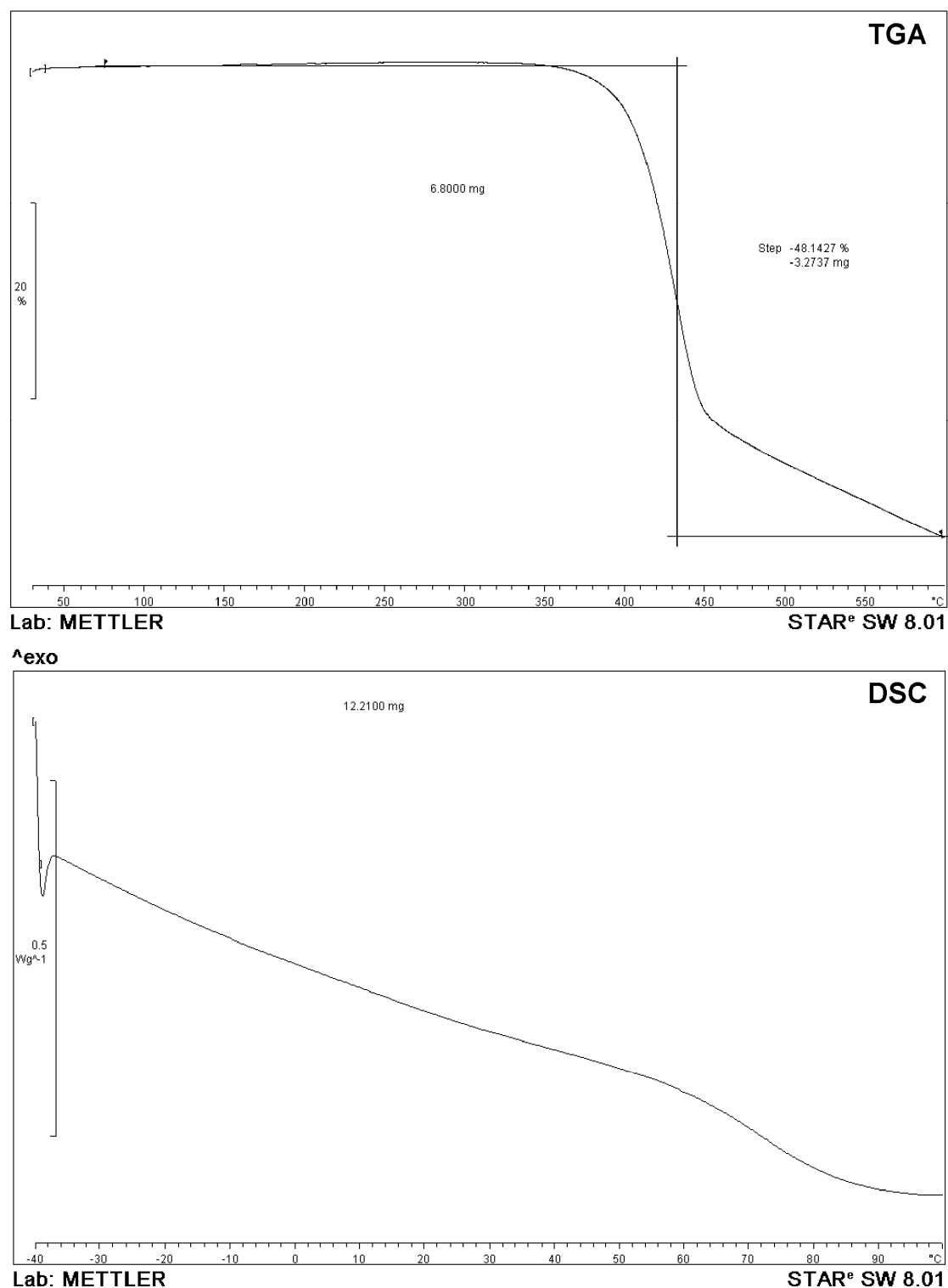


Figure 5. TGA (top) and DSC (bottom) thermograms for **2**. The changes in each thermogram are too small to be assigned to any desolvation process, suggesting **2** is a polymorph of anhydrous zinc fumarate (**1**).

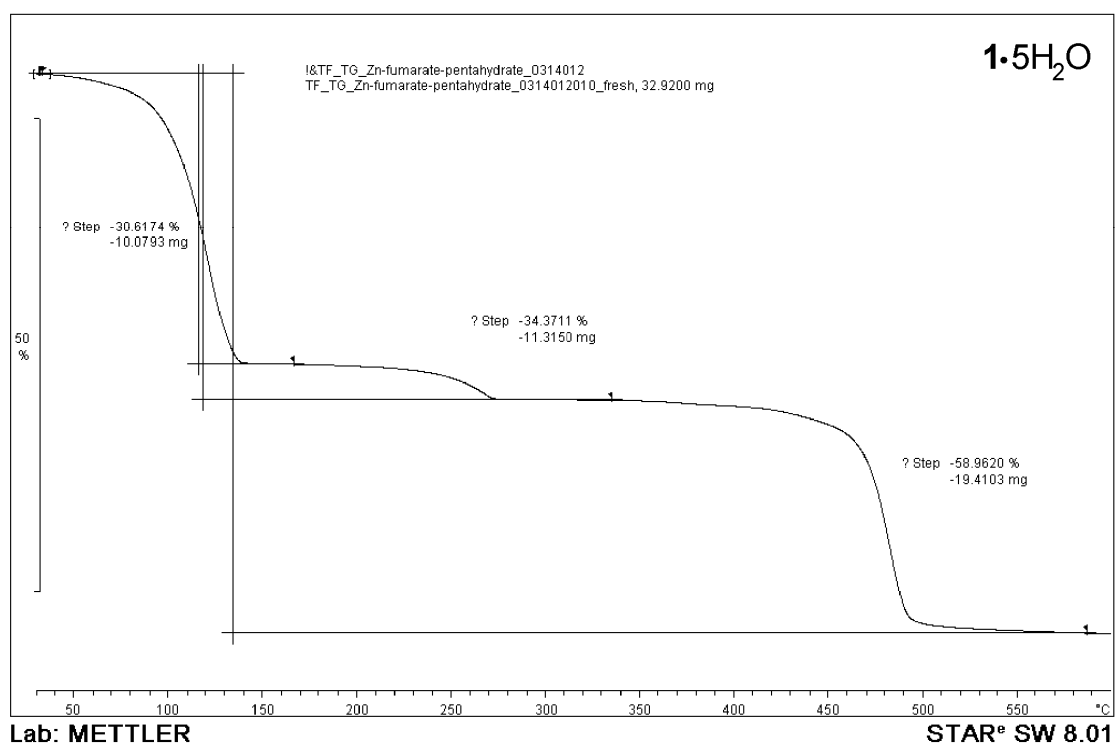
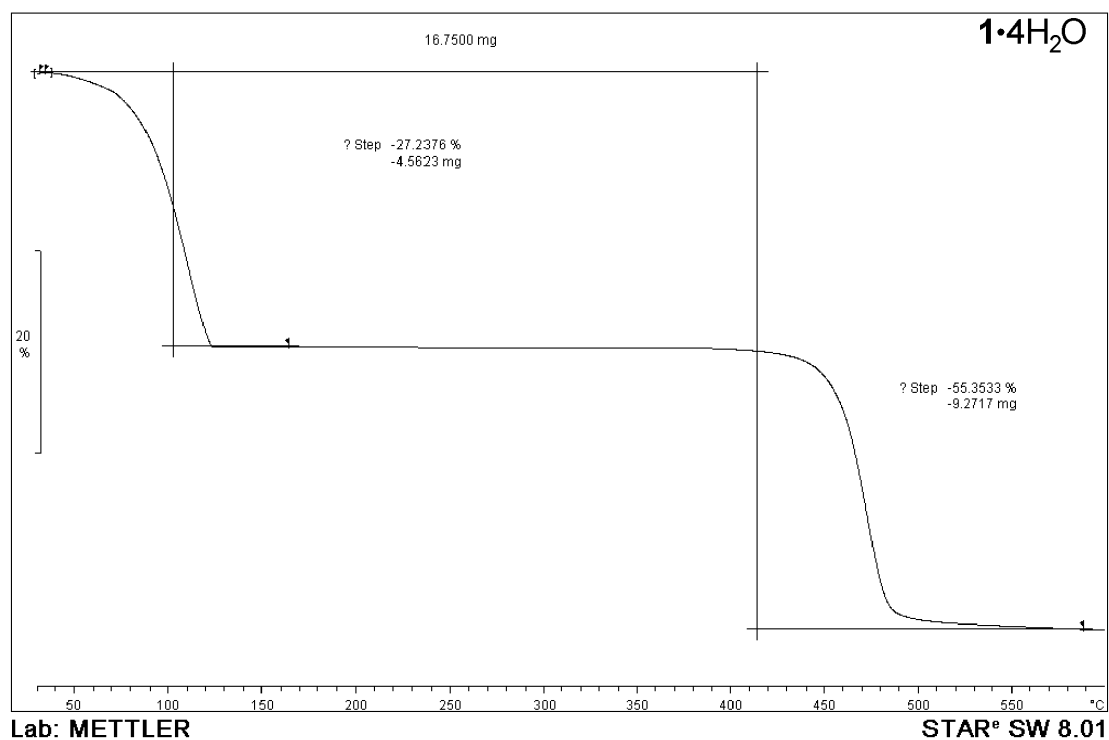


Figure 6. TGA thermograms for 1·4H₂O and (top) and 1·5H₂O (bottom). The first step in the thermogram for 1·4H₂O is 27.2% and roughly corresponds to the content of water (28.6%). The sum of the first two steps for 1·5H₂O is 34.4% and roughly corresponds to the content of water (33.4%).

