Reaction and Recovery of Nucleation Particle Strontium Chloride Hexahydrate in Calcium Chloride Hexahydrate: Supplementary Information

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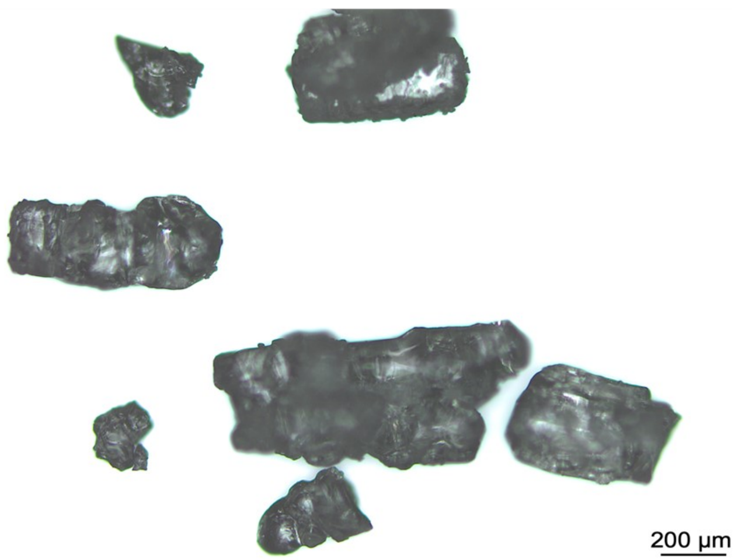

Figure SI-1: Optical image of neat strontium chloride hexahydrate particles. Particle size was observed to range from approximately 150 μm – 1000 μm, and was composed of irregularly shaped, faceted particles.

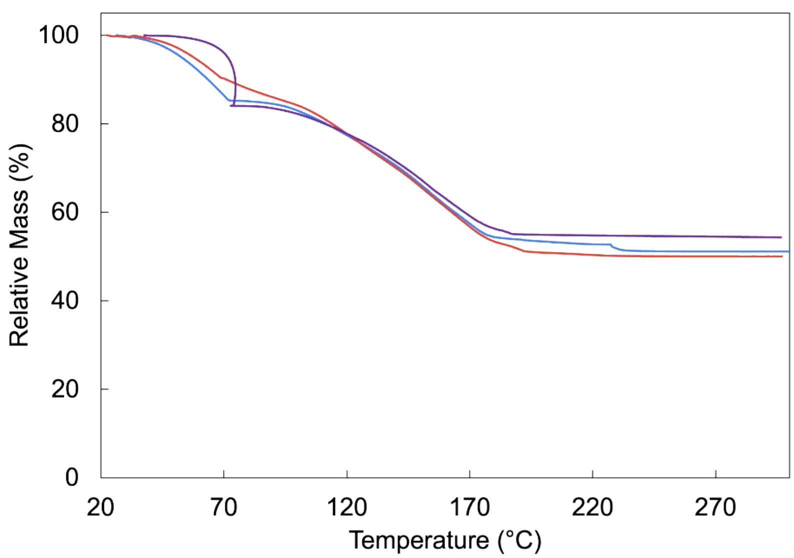

Figure SI-2: Thermal gravimetry analysis (TGA) of as-received calcium chloride hexahydrate (CCH), ramped from ambient temperature (approx. 24 °C) to 300 °C, at a rate of 5 °C·min^{-1}. The average remaining relative mass for all three samples at the maximum temperature was m_{rel} = (51.8 ± 2.1)%, where the number following the symbol ± is the estimated standard deviation, using a coverage factor k = 2, corresponding to a level of confidence of approximately 95 percent. Thus, the mass loss associated with dehydration from the initial as received sample to the dehydrated form is (48.2 ± 2.1)%, which is within uncertainty of the theoretical mass loss of complete dehydration of $CaCl_2 \cdot 6H_2O$ of 49.3%.

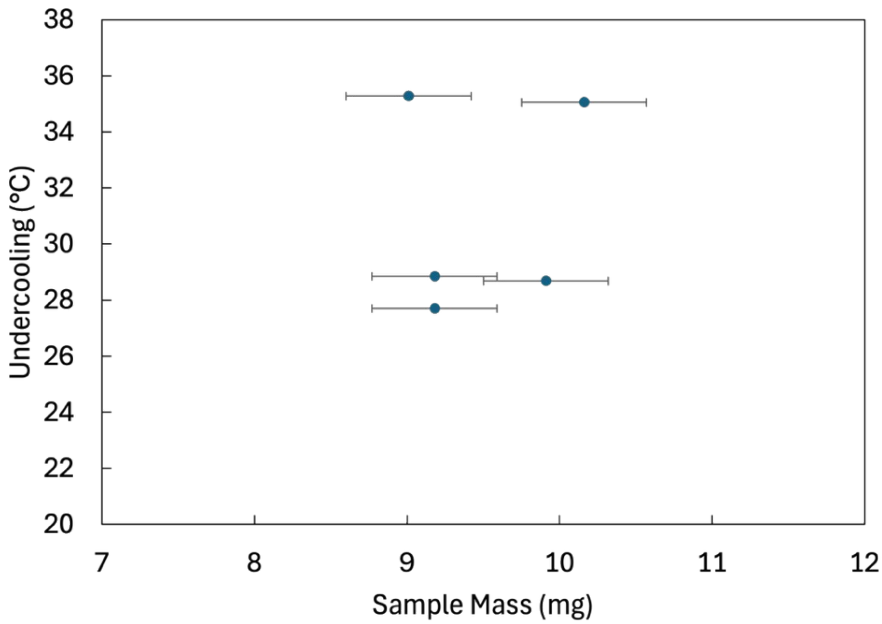

Figure SI-3: Comparison of DSC sample mass versus sample undercooling (T_m-T_{crys} = ΔT) for five neat CCH samples, all tested at a ramp rate of 1 °C·min^{-1}. Analytical uncertainty on the reported undercooling for an individual measurement is smaller than the symbol size. This mass variation is representative of the prepared samples. Across this variation of masses, there is no observed correlation between undercooling and sample mass, within the variation in mass of the prepared samples.

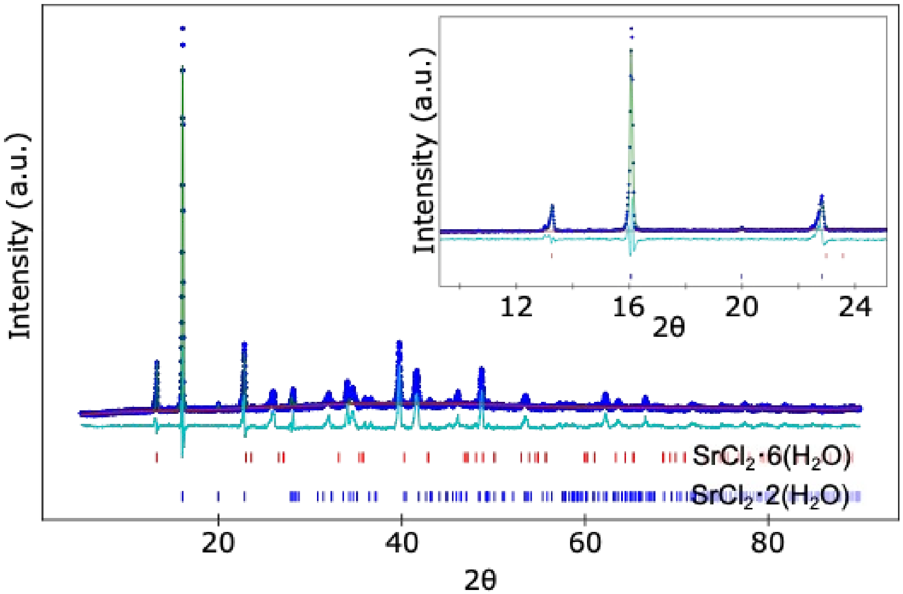

Figure SI-4: Refined powder diffraction data for reacted strontium chloride hexahydrate ($SrCl_2 \cdot 6(H_2O)$) recovered from immersion and equilibration in liquid CCH. This sample was equilibrated at 30 °C for a period of approximately 1 week, at a concentration of (5 wt. % SCH/ 95 wt. % CCH). Observed XRD spectra is consistent with the calculated powder diffraction patterns for strontium chloride hexahydrate ($SrCl_2 \cdot 6(H_2O)$) and strontium chloride dihydrate ($SrCl_2 \cdot 2(H_2O)$) (R_{wp} = 23.394, reduced χ^2 = 12.797). The quality of Rietveld refinements (specifically, the intensity of peaks) were limited by the poor sampling statistics due to relatively large grain sizes. This observation supports the claim that even at temperatures just above the melting temperature, the hexahydrate form will undergo a dehydration reaction, resulting in the dihydrate form.

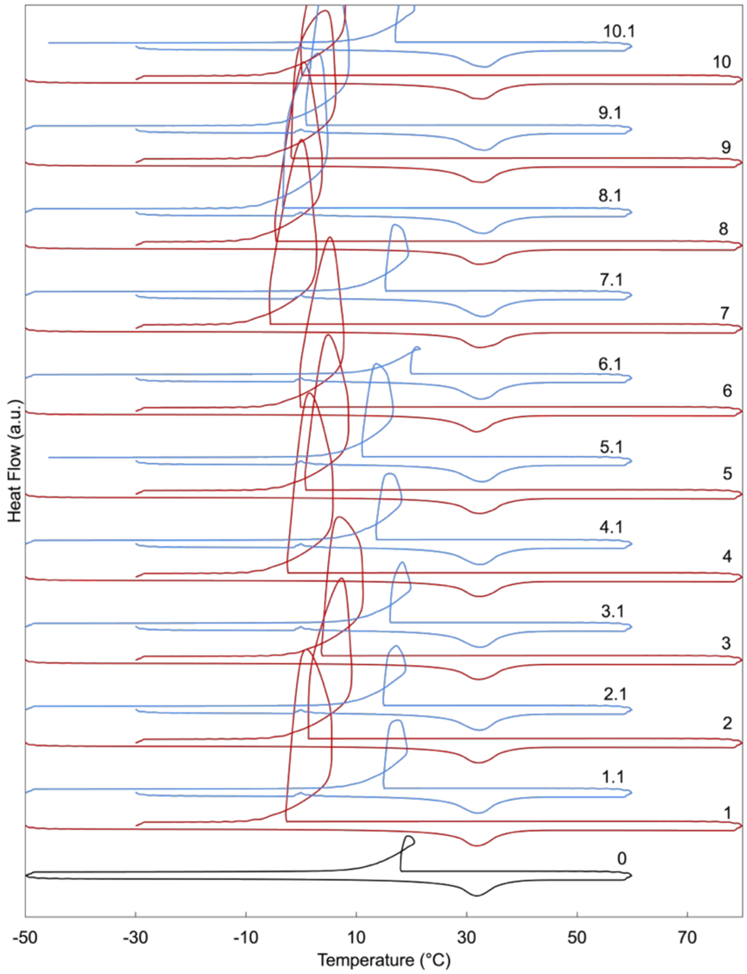

Figure SI-5: Full DSC data for short-term cycling experiment (10 cycles total) of a CCH sample containing approximately 5 wt. % SCH. Bottom cycle (black line) is the first cycle (time 0 test of sample), with cycle number increasing as you move up the figure. Here, we refer to a 'cycle' as a complete set of 1) a thermal loop with a maximum temperature of 80 °C, to fully melt the system and induce the SCH → SCD reaction, 2) followed by cooling and solidification to show that undercooling is large, and further cooling to -30 °C with a 15 minute hold at 0°C, to promote re-emergence of SCD → SCH. Parts 1 and 2 are illustrated with red lines above. 3) Followed by a final heating to a maximum temperature of 60 °C (sufficient to completely melt, but insufficient to allow for complete SCH → SCD conversion, and 4) cooling back down to a low temperature to demonstrate that (generally) undercooling has decreased. Parts 3 and 4 are illustrated with blue lines above. Thus, each "cycle" contains 2 melting and 2 solidification components, one of which generally exhibits a large undercooling, the other generally exhibits small undercooling (although there is some variability due to the reasons identified and discussed in the text).