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Investigational compound GENE, is characterized as anhydrate. Anhydrate converts to trihydrate in aqueous slurry.

Hydrate dehydrates to form a “dehydrated hydrate/DH” which is X-ray amorphous. DH crystallizes to anhydrate at ≥192°C.

DSC traces (left) of BM samples. Crystallization exotherms overlap with T_g except for the sample ball milled for 60 min, (higher speed/HS) VTXRPD (right) of the BM-60 min (HS) sample. Crystallization to anhydrate starts at 125°C

Lyophilized

Crystallization observed at 100°C (hot stage microscopy)

Dehydrated (DH)

DSC (left) shows 2 crystallization exotherms for DH.VTXRPD (center) and SSNMR (right) reveal an “intermediate” phase at 155-160°C(1st DSC exotherm) which rearranges to form the anhydrate at 195°C

METHODS

- Lattice disorder was induced by ball milling, lyophilization and dehydration
- Characterization: DSC, TGA, XRPD, SSNMR, Water sorption, computational analysis

RESULTS

Disordered phases

Overlaid patterns (XRPD, left) and spectra (SSNMR, right)

BACKGROUND

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OBJECTIVE

- Investigate the nature of disorder/local order in a “disordered phase” generated through different routes
- Understand effects of disorder/ “existing order” on recrystallization tendency and nature of resulting phase

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