	Experiment title: Combined single crystal / powder diffraction study of beta-Cyclodextrin hydrates	**Experiment number**: **HS- 3426**
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Report:

High resolution powder X-Ray diffraction was carried out on beta-cyclodextrine (β-CD) crystalline powder at various relative humidity condition. The main goal of the experiment was to investigate β-CD structural water content as a function of environmental condition. Even though a full characterization of the β-Cd/H_20 system was not achieved, due to experimental difficulties, a high-quality diffraction pattern of anhydrous β-CD was collected.

Experimental

Hydration and de-hydration kinetic of cyclodextrins is quite fast and don't allow an off-situ sample preparation. In particular, anhydrous β-CD shows a quite strong tendency to

rehydration. To maintain a constant environmental relative humidity during the diffraction pattern collection, an in-capillary r.h. conditioning system was adopted. The fully hydrated sample was placed in a glass capillar in close contact with mother liquor, while the anhydrous sample, as obtained from a thermal dehydration at 200 C, was sealed in the capillary along with a small amount of silica gel, separated from it by a porous sect. High resolution patterns were collected, among others, of 100% hydrated β-CD, anhydrous β–CD and partially hydrated commercial grade β–CD (figure 1). A 2-θ range between 1 and 40 degrees was scanned; wavelength used was 0.8 Å. As no environmental humidity sample conditioning was available at ID31 at the time the experiment was carried out, we were not able to perform a complete in-situ study of the hydration/dehydration kinetic of the system.

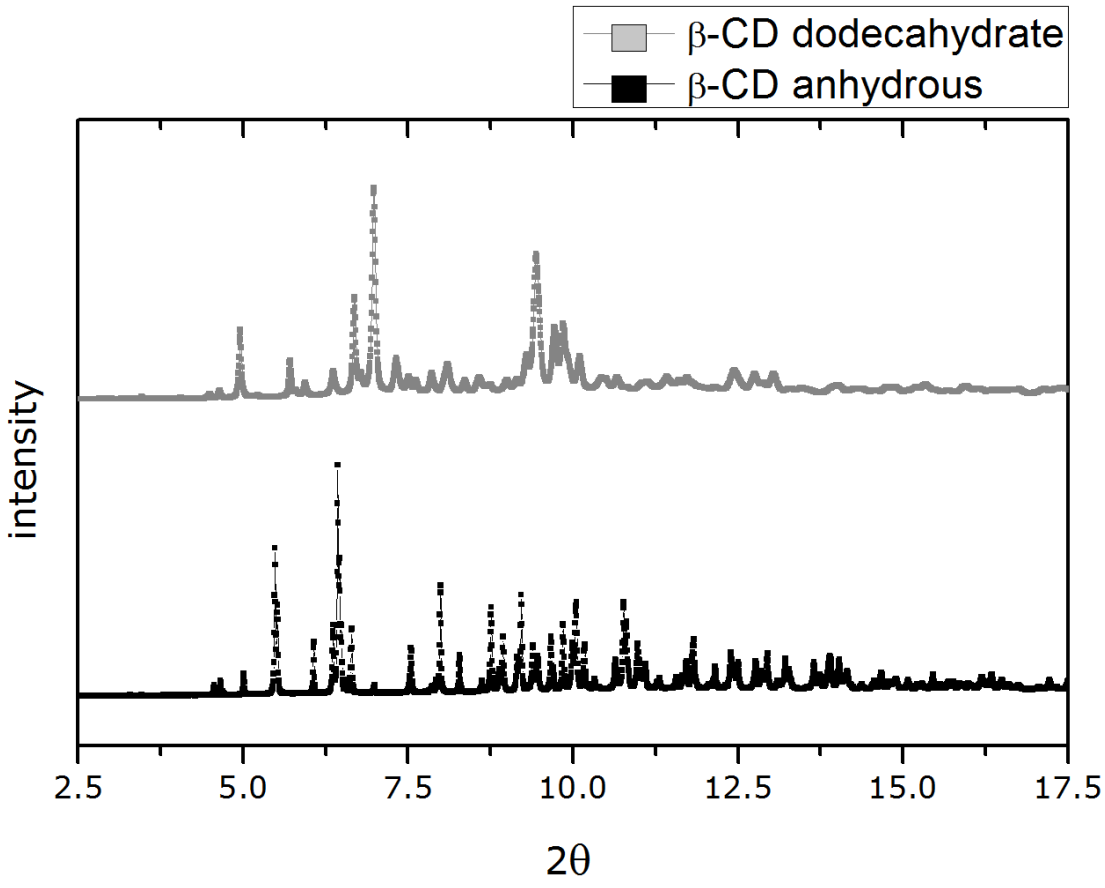

Figure 1 - Powder diffraction patterns of b-CD dodecahydrate and b-CD anhydrous collected on the high resolution powder diffractometer at ID31

Results

As the most important experimental result, a high-resolution pattern of pure anhydrous β–CD was collected, allowing us to succesfully perform cell indexing and space group

determination. The successive molecular packing determination using direct space methods should have been straightforward, given that no structural alteration are expected to happen in the cyclodextrin ring due to complete dehydration. Global optimization using simulated annealing was attempted with several different softwares, using the β–CD molecular structure, as found in the literature, as reference molecular fragment. However, no completely satisfactory results have been achieved so far. We believe that major alteration in the molecular conformation may appear as a consequence of the dehydration and loss of structural water, requiring the adoption of a more sophisticated model in the optimization. It is worth remembering that the last step in structure solution from powder diffraction is usually the most troublesome. Thus, despite the final difficulties, we are quite confident to be on the right path to a full structure determination of anhydrous β–CD, which would be of remarkable importance from both a scientific and a practical point of view.