



Experiment title:
HIGH RESOLUTION FIBER DIFFRACTION DATA ON NATIVE
CELLULOSE

**Experiment
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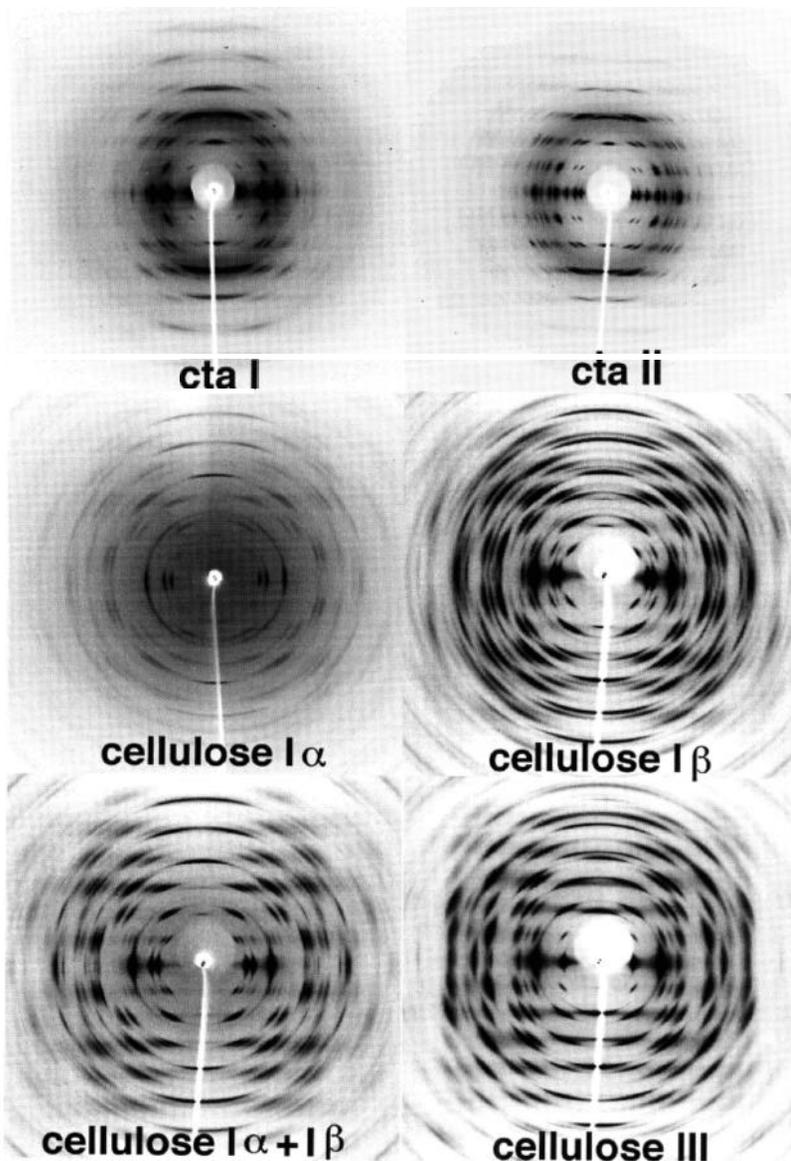
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Report: Our experiments have dealt with the recording of high resolution fiber diffraction data on native cellulose, some modified cellulose and cellulose derivatives. The samples consisted of stack of films made of oriented cellulose crystals. 6 types of samples were investigated, namely (i) a cellulose sample from the mantle of *Halocynthia* (a sea animal); this sample was of high crystallinity and contained essentially one crystalline phase : cellulose I β . (ii) a cellulose sample from the cell wall of *Cladophora* (a green alga); this sample was of high crystallinity and contained two crystalline phases: cellulose I α and cellulose I β (iii) a cellulose sample from the cell wall of *Glaucocystis* (a green alga) that contained only the I α phase of cellulose. (iv) a cellulose sample *Cladophora* treated with ammonia at 200°C under pressure. This sample was converted to the cellulose III allomorph. (v) a sample of cellulose triacetate CTA I, obtained by heterogeneous acetylation of *Cladophora* cellulose: this sample was annealed at 240°C under nitrogen atmosphere. (vi) a sample of cellulose triacetate CTAII: this sample was prepared from fibers of cellulose triacetate and was annealed under tension at 240°C in an autoclave under water vapors.

In the experiments, a wavelength of 0.0720006 nm was used and the diffraction data were recorded on the MAR 345 imaging plate system positioned at 170 mm from the sample. The 3 samples of cellulose I diffracted to the resolution limit of the system, i.e. at a resolution of around 0.1 nm. The sample of cellulose III had the best orientation and the same resolution as those of cellulose I. Both CTA I and CTA II had a lower resolution that could be estimated to be of the order of 0.2



nm. The 6 types or patterns are shown in this report. For clarity, we have cut the diagrams at around 0.17 nm.

Examples of the 6 types of patterns are shown in the adjacent Figure. The intensities of the patterns of cellulose I β , cellulose I α +I β and cellulose III are presently being deconvoluted with the CCP13 program series. These intensities will be used to refine the corresponding molecular and crystal structures of cellulose I β that of cellulose III and if possible that of cellulose I α . The present X-ray fiber data on cellulose I and III will be combined with ILL diffraction experiments achieved on the same samples and where the OH holding the structures together have been replaced by ODs.