ESRF	**Experiment title:** The structure of a novel wedge-shaped molecule: 3,5-di(diacetylene)hydroxyl-benzoic methylester	**Experiment number**: SC-2779
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Report:

Since polymerization of diacetylenes was achieved in supramolecular assemblies of amphiphilic molecules in monolayers, bilayers and multilayers, a large number of monomers containing polymerizable diacetylene group as well as different reactive groups such as amide, carboxyl, etc [1-3], have been developed. In the literature, there are also reports on wedge-shaped molecules with two or three polymerizable diacetylene groups.[4-6]

As far as it is known, the wedge-shaped amphiphilic molecules with a polar head at the tip of the wedge and a large nonpolar body can self-assemble into well-defined cylindrical structures. However, to the best of our knowledge, the papers reporting the self-assembly of wedge-shaped molecules with several diacetylene groups were rare. The aim of the present work is to synthesize novel wedge-shaped molecules with two polymerizable diacetylene groups, which can be successfully converted to the polydiacetylene as well as self-assemble to form a columnar phase. Here, we synthesized a wedge shaped molecule: 3,5-di(diacetylene) hydroxyl-benzoic acid methyl ester.

To identify the lattice parameters formed by this compound and understand the phase behaviour of this material, wide-angle X-ray diffraction

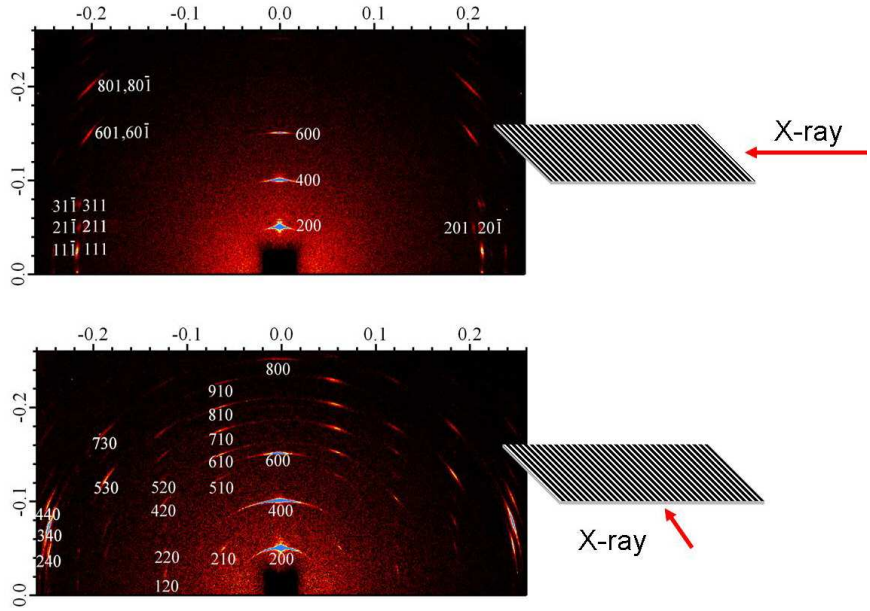

Fig. 1: 2D Grazing incidence X-ray diffraction patterns recalculated into the **s** planar space corresponding to oriented film of 3,5-di(diacetylene)hydroxyl-benzoic acid methyl ester deposited on a silicon wafer rubbed with PTFE annealed at RT for 24 hours. The incident X-ray beam was oriented perpendicular (top) and parallel (bottom) to the rubbing direction.

experiments in grazing incidence and transmission geometry at variable temperatures were carried out. The 2D GIWAXD pattern were measured on ca. 100 nm thick films of 3,5-di(diacetylene)hydroxyl-benzoic acid methyl ester deposited on a silicon wafer rubbed with PTFE. The films were subsequently annealed at RT for 24 hours. The recorded diffractograms are shown in Figure 1 for the cases where the X-ray beam was perpendicular and parallel to the rubbing direction. The strong *h*00 reflections are found to be strictly meridional indicating a smectic-like stacking of layers parallel to the Si substrate. Furthermore, the material reveals a single-crystal like orientation on the PTFE rubbed surface seen from the absence of any reflection with l different from 0 when viewed along the rubbing direction.

The unit cell of the annealed 3,5-di(diacetylene) hydroxyl-benzoic acid methyl ester is identified as monoclinic with angle β different from 90°. The lattice parameters are the following: a=39.89 Å, b=16.32 Å and c=4.78 Å, β=88.5°. The slight monoclinicity is visible from the split of the *hkl*, *hk*-l peaks, but even more clearly from the fact that the *h*0*l* and *hkl* peaks are not exactly on the same layer lines as it would be expected for orthorhombic unit cells (cf. Fig. 1 top). The first order peaks in the a-a nd b-direction are missing, indicating a centered unit-cell in the ab-projection.

From the measurements on large spherulites grown in thicker films the fast growth axis can be identified as the **c**-direction. The latetr coincides with the radial direction of the spherulitc crystals when investigated using a rather small beam size (200*200µm²) compared to the lateral dimensions of the spherulites. A characteristic 2D X-ray pattern is given in Fig. 2. Here, the pattern exhibits nice orientation, similar to what we observed previously in micro focus experiments on polymer spherulites.[7] The absence of the smectic peaks (except the very first peak) shows that the smectic layers preserve their orientation parallel to the substrate even in thick films.

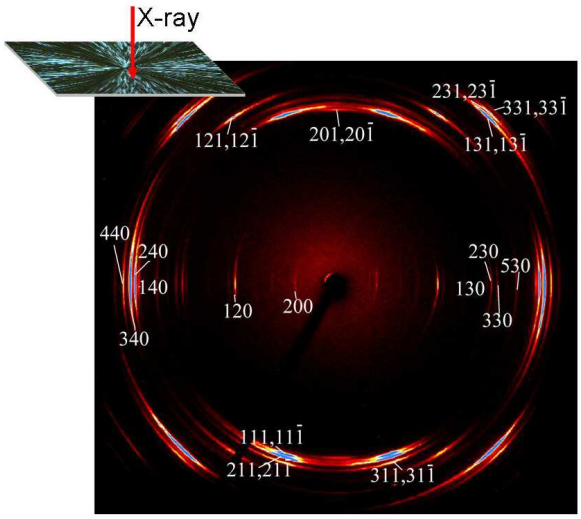

Fig. 2: 2D WAXD pattern recorded in transmission geometry on a large spherulites of the studied compound showing a good orientation. The *hk*0 reflexes are oriented strictly in the equatorial direction and are therefore perpendicular to the radial direction of the spherulite.

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