



ESRF

Experiment title:

Influence of the chirality in a 2D crystallization of alcohol monolayers. Studies by grazing incidence diffraction.

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SC-306

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14

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Report:

We are interested in monolayers of chiral alcohols at the water surface in order to determine the role of the chirality in the 2D stacking. We already knew that all 2-alcohols monolayers at the water surface (racemic and enantiomer between 12 and 16 carbons) exhibit a hexagonal phase. Preliminary results show that for less than 14 carbons the hexagonal phase is stable with time for odd alcohols and none for even ones (Renault A., Alonso C., Artzner F., Berge B., Goldmann M. and Zakri C. submitted to J. Phys. II). The aim of this experiment (SC-306) was to analyse the shape of diffraction peaks in the case of 2-tridecanol, for monolayers of racemic mixture and pure enantiomer. The high performances of TROIKA beamline allows us to do this with an accurate precision.

We have used a high resolution set-up of X-Ray grazing incidence diffraction to measure the Bragg peak of alcohol monolayer. The incident beam was shaped at $\lambda=1.409 \text{ \AA}$ by a diamond (111) monochromator, followed by a flat mirror. The incident angle on the water surface was 2.0 mrad. The incident beam was sent on the sample inside our specially designed trough having a good thermal stability and homogeneity and a water level regulation better than $10 \mu\text{m}$. We analysed the diffracted intensity with a Ge (111) analyser (resolution 0.011") and a PSD.

The high resolution allows us to analyse precisely the line shape of peaks with a power law description (Zakri C., Renault A., Rieu J. P., Vallade M., Berge B., Legrand J. F., Vignault G. and Grübel G. Phys. Rev. B 55 1997 p14163) to estimate the coherence length and the shear elastic constant of the monolayer.

Monolayers were deposited in their liquid phase, the temperature was decreased until 10°C ($T_m=21\pm 1^\circ\text{C}$ for the both compounds) and we recorded spectra during the increasing of temperature until the melting of the monolayer.

In the case of the racemic mixture we observed a single very sharp peak corresponding to an hexagonal phase with vertical chains and a coherence length of $4500\pm 200\text{Å}$. This peak move to smaller diffraction angles -without broadening when temperature increases and disappear suddenly at the melting of the monolayer (Figure 1). These show that the melting of the monolayer is a first order transition. This result is confirmed by ellipsometry and surface tension measurements.

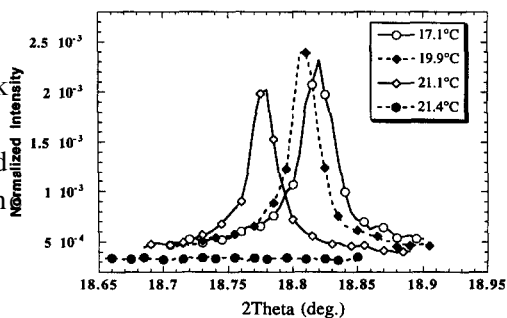
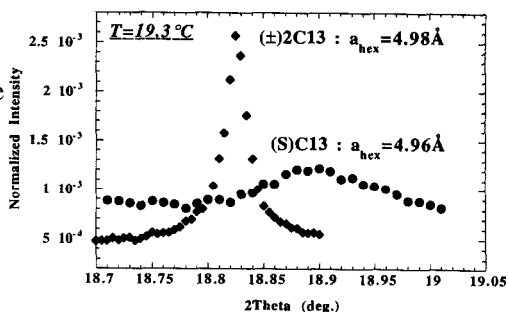


Figure 1

Concerning the monolayer of pure enantiomer, we also observed a single diffraction peak and rods can with maximum at $q_z=0\text{Å}^{-1}$: this is also an hexagonal structure.

A comparison of Bragg peaks obtained for monolayers of $(\pm)2\text{C}13$ and $(S)\text{C}13$ at the same temperature is presented on figure 2. The coherence length is smaller for the monolayer of pure enantiomer than for the racemic mixture ($1500\pm 200\text{Å}$). The sudden disappearance of the diffraction peak at the melting temperature indicates that the fusion-crystallisation transition is also first order.



These results show that the 2D stacking is little more compact for the pure enantiomer ($a_{\text{hex}}=4.960\pm 0.005\text{Å}$ for $(S)\text{C}13$ and $a_{\text{hex}}=4.980\pm 0.005\text{Å}$ for $(\pm)2\text{C}13$ at 19.3°C) but the long range order is more extended for the racemic mixture than for the optical active compound: it seems that there is no chiral segregation in the case of 2-tridecanol.

We want now to study mixtures of both enantiomers in different concentrations to establish phase diagrams and compare them to these obtained for 3D samples.