ESRF	**Experiment title:** Structural determination of the liquid-liquid phase transition in a 4-methylpiridyne/alpha-cyclodextrin/water solution	**Experiment number**: HD104
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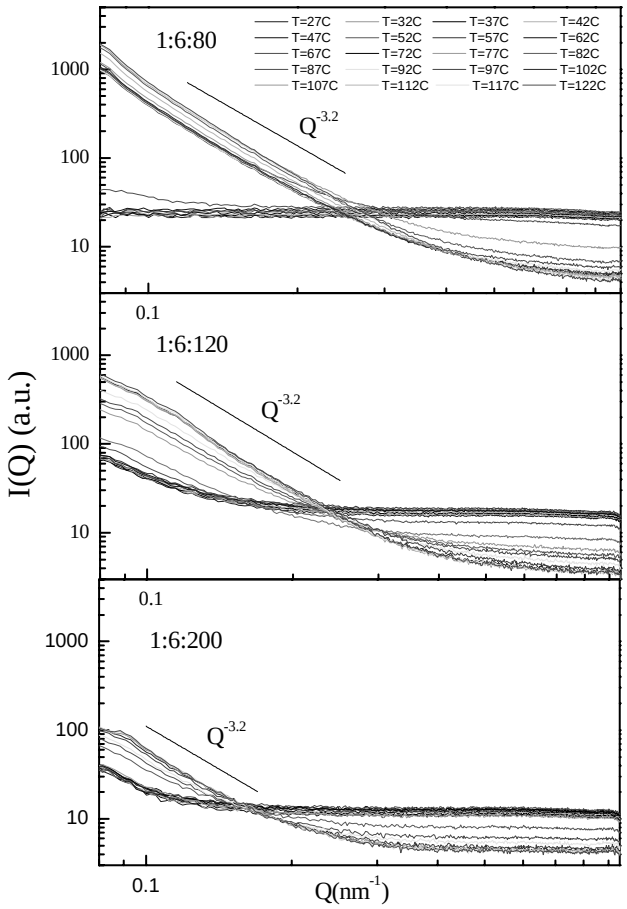
Report:

Small angle x ray scattering (SAXS) measurements have been performed on a solution of 4-methyl-piridyne (4MP) (C_6H_7N), α-cyclodextrin (αCD) ($C_{36}H_{60}O_{30}$) and water as a function of the temperature in the range 300÷475K. The incident photons had an energy of 10.2 keV (λ= 1.21 Å). The sample was confined in a 2 mm diameter borosilicate capillary. The detector was placed at a distance of almost 7.6m from the centre of the sample and the intensity was collected in the Q range 0.07÷1 nm^{-1}. The geometrical parameters of the measurements were calibrated via a standard of collagen and the two dimensional diffraction patterns were collapsed to a 1D patterns using the FIT2D software package. The sample has been prepared at three different concentrations of αCD, water and 4MP with molar ratios respectively of 1:6:*x*, and *x*=80,120,200. Previous x-ray diffraction experiment performed in the Q range 1.5÷32 nm^{-1}, as a function of the temperature in this concentration region confirmed the existence of a disordered structure both below and above the transition temperature detected by differential scanning calorimetry (DSC) [1]. These data showed an increasing of the scattered intensity in the low Q region suggesting the hypothesis of being in presence of a critical phenomenon.

The aim of this experiment was to try to understand the nature of the transition detected by DSC and x-ray diffraction and in particular to focus the attention on the low Q behaviour of the scattered intensity. In Fig.1 a sample of the scattered intensities at the three indicated

concentrations as a function of the temperature across the thermodynamic endothermic transition observed by DSC [1] is shown in double logarithmic scale.



Characteristic SAXS measurements of a solution of αCD, water and 4MP at three different concentrations with molar ratios respectively of 1:6:*x*, and *x*=80,120,200 and as a function of the temperature in the range 300÷495K. The data are subtracted of the empty cell contribution. A power law behaviour, with a slope of approximately -3.2, in double logarithmic scale is also shown.

At increasing temperature an abrupt increase of the low Q scattered intensity is observed for all the three investigated concentrations, showing therefore a different behaviour between the low and high temperature samples. This difference although less evident is still present as the concentration is lowered (1:6:200). A power law behaviour, with a slope of approximately -3.2, is shown in double logarithmic representation for $Q<0.26nm^{-1}$ in the solution 1:6:80, for $Q<0.23nm^{-1}$ in the solution 1:6:120 and for $Q<0.16nm^{-1}$ in the solution 1:6:200.

[1] R. Angelini et al. www.esrf.eu/news/spotlight/spotlight43/inverse-melting/