ESRF	**Experiment title:** Probing the local order in organic superconductors by means of pair distribution function (PDF) analysis from high energy synchrotron diffraction data	**Experiment number:** HS-3281
Beamline: ID15B	**Date of experiment:** from: 07-march-07 to: 11-march-07	**Date of report:** 21-january-08
Shifts: 12	**Local contact(s):** Gabriela Gonzalez Aviles	*Received at ESRF:*

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Report:

Powderized material of a single crystal of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ (sample size about 5 mg), formula $CuN_2H_{16}S_{18}C_{22}$, has been used to perform powder diffraction patterns up to high $Q_{max}=4\pi\sin(\theta)/\lambda$ = 30 Å^{-1} for 3 different temperatures (50, 90 and 300 K) above and below the glass-like transition, which has previously been reported and often been claimed to stem from configurational disorder of the terminal ethylenes on the (BEDT-TTF) molecule. As organic materials, these superconductors tend to be very soft and highly susceptible to irradiation-induced defects. Therefore, for our experiments a rapid acquisition technique of the PDFs was chosen to collect the data at the ID15B, *i.e.,* a high energy of 88.876 keV in combination with an image-plate detector in order to minimize the total duration of exposure for our sample and to reduce the irradiation damages. Here, because of the organic nature of the material the measuring time still lies in the range of 1 hour per temperature point. During the first 6 shifts the liquid-helium-cooled gas flow cryostat from the ID31 has been mounted. Due to contamination of the cryostat window, leading to a fast saturation of the image-plate, the experimental set-up has been modified for the last 6 shifts, using a displex cryostat from the ID15 without any spinning

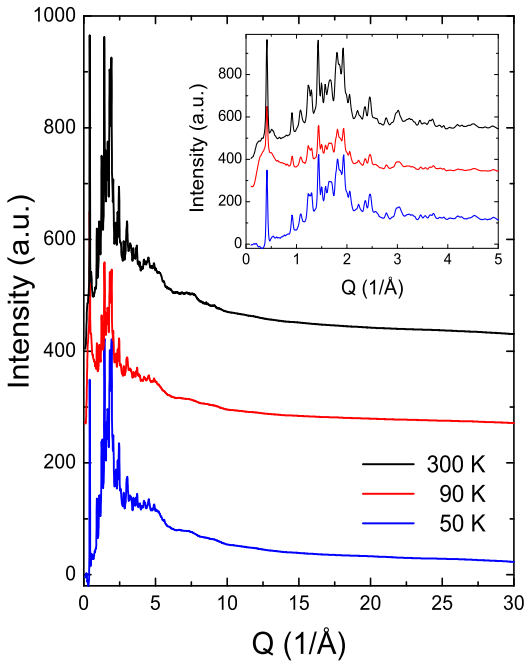

Fig.1: Intensity data I (Q) of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ measured using X-rays of λ = 0.1395 Å at the ID15B. Note the shift of the data for 90 K and 300 K along the intensity axis.

option, yielding reasonable data quality for our low diffracting organic compound.

Fig. 1 depicts the intensity data I(Q) of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ measured using X-rays of λ = 0.1395 Å at ID15B. In order to obtain high-resolution data for the whole Q-regime 0 ≤ Q ≤ 30 1/Å, three different sample-to-detector distances have been employed. Note, that the raw data have been preprocessed with respect to counting rate-dependencies of the detector and to an exact determination of the sample-to-detector distance. Then, the scans have been merged together and an accurate subtraction of the background (borosilicate container and cryostat background) has been performed, leading to the intensity data shown in Fig. 1 for temperatures 300 K, 90 K, and 50 K.

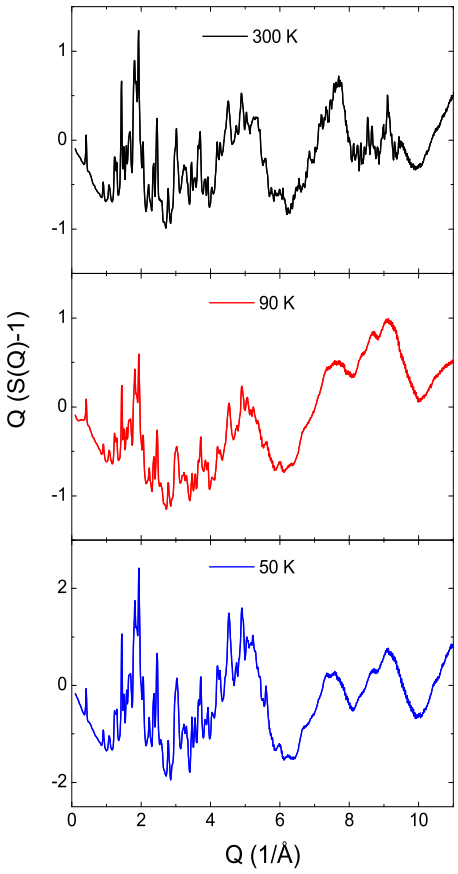

Fig. 2: The reduced structure function F(Q) = Q(S(Q)-1) of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ at temperatures 300 K, 90 K and 50 K.

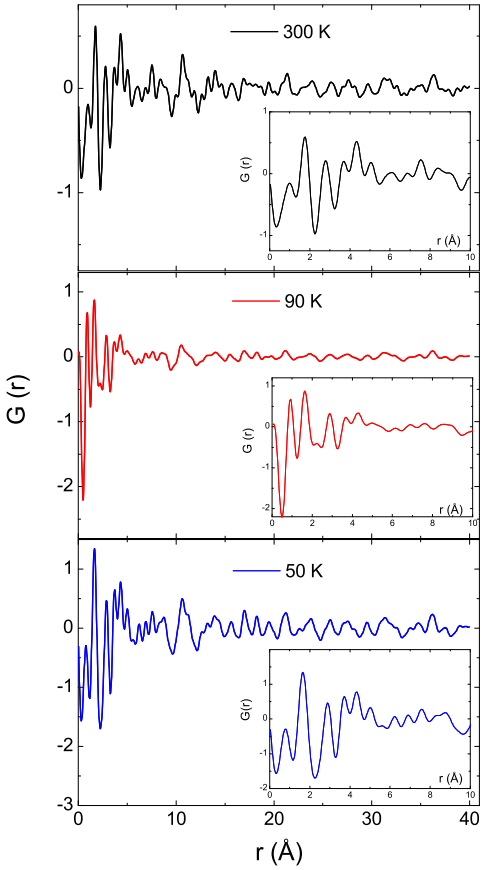

Fig. 3: The PDFs of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ at temperatures 300 K, 90 K and 50 K obtained by a Fourier trans-formation of the data for 0 ≤ Q ≤ 10 1/Å.

The reduced structure function F(Q) = Q(S(Q)-1), where S(Q) is the corrected and normalized X-ray powder scattering intensity, is given in Fig. 2 for temperatures T = 300 K, 90 K and 50 K. All corrections and data processing was done with the help of the programm PDFgetX2[1]. Since Bragg diffraction ceases for Q > 10 1/Å, in order to minimize noise effects for the calculation of G(r) the F(Q) data were terminated at Q_{max} = 10 1/Å. However, well pronounced PDFs were calculated up to r = 40 Å (see Fig. 3). Here, for future experiments both an enhanced sample mass as well as an improved experimental set-up regarding the cryostat environment should be used to improve the counting statistics in higher diffraction angles.

A qualitative result concerning the PDFs of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ is given in Fig. 3 for temperatures above (300 K), at (90 K) and well below (50 K) the glass-like transition obtained by a Fourier transformation of the data for 0 ≤ Q ≤ 10 1/Å. Apart from truncation effects at very low distances below r = 1 Å the PDFs have many peaks at similar characteristic distances separating pairs of atoms. In the region 5 Å ≤ r ≤ 7 Å, however, besides overall deviations in the absolute value of G(r) for the 3 different temperatures, the peaks appear at different positions in real space, indicating a change in the local structure of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ as function of temperature.

The solving of the local crystal structure by PDF local structure model least square refinements is presently carried out. From these refinements we expect to resolve the intrinsic structural change on an atomic lengthscale, which in turn might elucidate the nature of the glass-like transition.

[1] X.Qiu et al. J.Appl. Crystallogr. **37** 678 (2004)