ESRF	**Experiment title:** Polymeric Fe(II)triazole: PDF from high Q_{max} powder diffraction for structure analysis	**Experiment number**: CH-2030
Beamline: ID31	**Date of experiment**: from: 23-sept-05 to: 30-sept-05	**Date of report**: 02-feb-06
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Report:

From a number of samples of polymeric iron(II)tri(4-hydroxyethyl-1,2,4-triazole)-di(*p*-chloro-benzene-sulfonate) (**1**), formula $FeCl_2S_2O_9N_9C_{24}H_{29}$, two samples have been selected during the experiment CH-2030 for temperature dependent powder diffractometry: One of best crystallinity (**1a**; label CH1-024-100-N2) and a second of lower crystallinity (**1b**; label JK1.03). Both show differences in their magnetic behaviour as $f(T)$. Data sets for both **1a** and **1b** were recorded at T = 350K, 295K, 250K and 80K for approximately 8h in the 16-bunch mode each using constant counting times $t(2\theta)$♣. Figure 1 compares a portion of their diffractograms recorded with λ = 0.35093(5)Å.

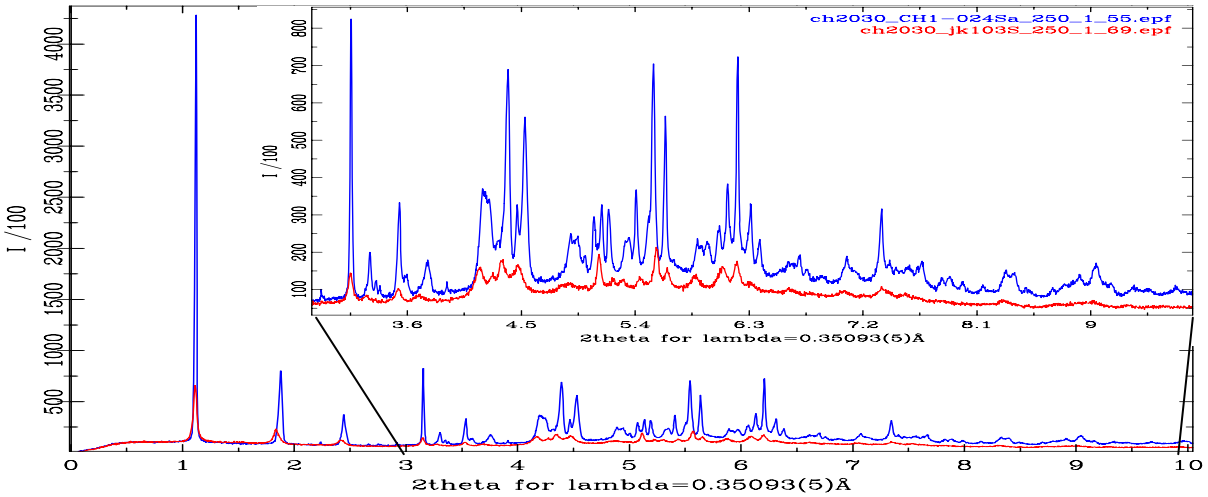
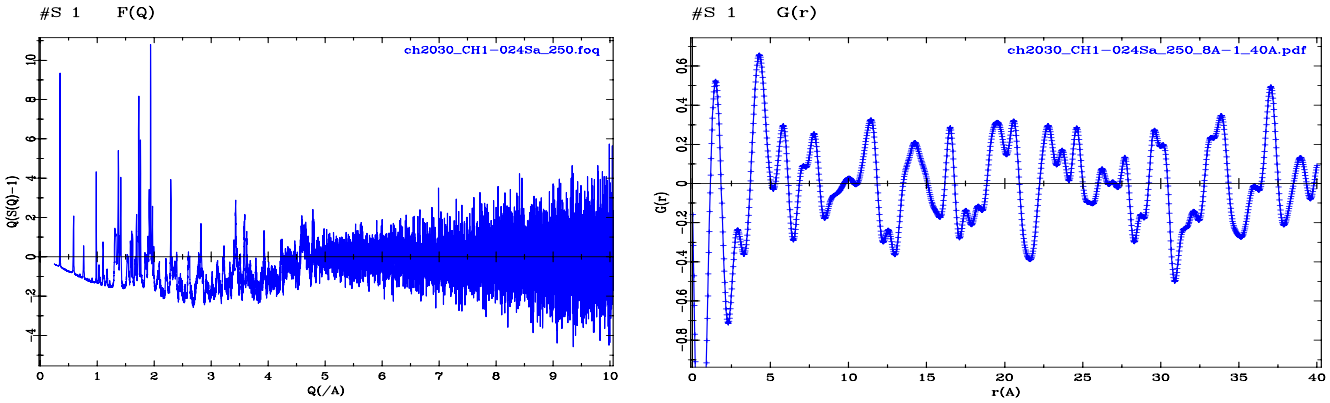


Fig. 1. Comparison diffraction data $I(2\theta)$ of **1a** (blue) *vs.* **1b** (red) at 250K

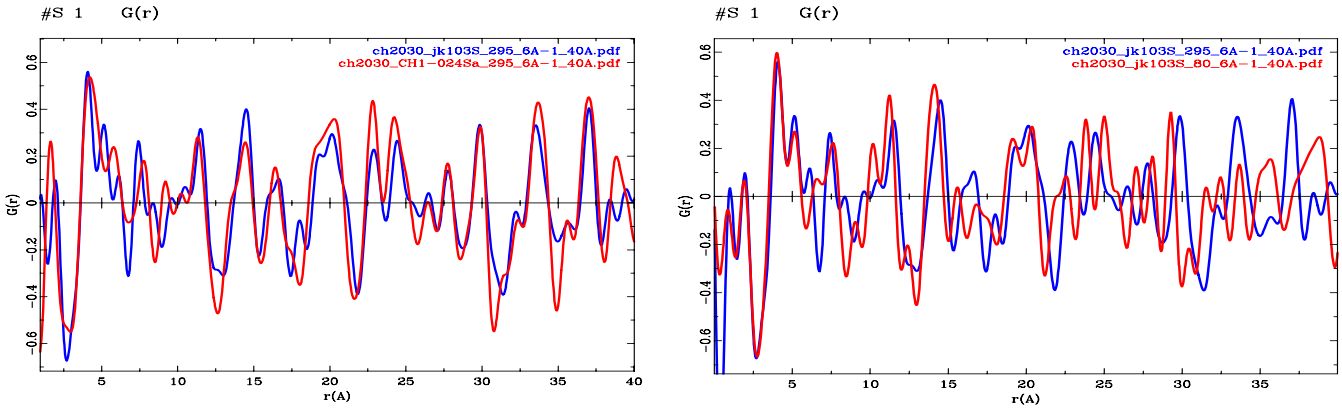
Figure 2 depicts the reduced structure function $F(Q)$ ($Q = 4\pi\sin(\theta_{max})/\lambda$) and its sine Fourier transform $G(r)$ (atomic pair distribution function, short: PDF) of **1a** at 250K as a typical example for the measurements at ID31. Bragg diffraction ceases for $Q > 6\text{Å}^{-1}$. To minimise noise effects, for the calculation of $G(r)$ the $F(Q)$ data sets were terminated at $Q_{max} = 8\text{Å}^{-1}$ (see also ♣). However, well pronounced PDFs were calculated up to $r = 40\text{Å}$ (Fig. 2b).



(a) (b)

Fig. 2 (a) The reduced structure function $F(Q) = Q[S(Q)-1]$ and (b) the PDF $G(r)$ of **1a** at 250K

A qualitative result concerning the Fe-Triazoles' PDFs is given in Figure 3: (a) **1a** and **1b** show differences mainly locally in the low-r region ($r < 10\text{Å}$) whereas in (b) decreasing T leads to a more dramatic change for $r > 20\text{Å}$ (**1b**).



(a) (b)

Fig. 3 (a) Comparison of PDFs of **1a** (blue) *vs.* **1b** (red) at 295K and (b) of **1b** at 295K (blue) *vs.* 80K (red)

The solving of the crystal structure of **1a** from the $I(2\theta)$ data set is currently in progress, using another ID31 data set of **1a** recorded at longer wavelength. It is corroborated by energy minimisation and especially accompanied by PDF local structure model least squares refinements. The latter is expected to resolve the differences in chain order or packing inbetween **1a** and **1b** which in turn influence the magnetic features of the material.

♣ For future measurements, we expect an improvement of the counting statistics in higher diffraction angles using higher counting times at higher θ. To avoid artifacts in the Fourier transform (see also experiment report on HS-2629), a variable counting time (VCT) scheme, which is a continiuous function of θ, should be applied.