ESRF	**Experiment title:** Study of Charge Ordering in the Organic Superconductors $(TMTTF)_2X$	**Experiment number**: HS-1397
Beamline:	**Date of experiment**: from: 07-avr-01 to: 15-avr-01	**Date of report**: 29-aug-01
Shifts:	**Local contact(s)**: Dr. David LE BOLLOCH' / Dr Peter BOESECKE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Dr Sylvain RAVY, *Dr Pascale FOURY-LEYLEKIAN

Laboratoire de physique des solides, CNRS UMR8502, Bât. 510, Université Paris-sud,

91405 Orsay, France

***Dr Pierre MONCEAU, *Dr Felix NAD

Centre de Recherche sur les Très Basses Températures, C.N.R.S., BP 166X, F-38042, Grenoble, France.

Report:

The purpose of this experiment was to evidence a charge ordering in the organic superconductor series $(TMTTF)_2X$, where TMTTF is tetramethyltetrathiafulvalenium, and X an anion as PF_6, AsF_6 or SbF_6. We used the experimental setup of the previous experiment, performed by R. Kahn and coworkers : a plexiglas box in which an He flow could be directed on the sample. This box was built by H. Stuhrmann from IBS Grenoble and J.-M Bois from EMBL. We used image plates to measure the diffracted intensity.

We have experienced many difficulties, mainly due to leeks in the box. These leeks where responsible for

- a strong background due the diffusion by the residual oxygen in the box (see Fig. 1). This diffusion was attenuated by an adequate beamstop and tube around the incident beam.
- the presence of ice on the sample, whose weight broke the crystal many times. When present the ice gave a strong powder diffraction signal.

These effects made our measurements very difficult to reproduce. Moreover, this setup was not designed for regulating the temperature, which made extremely difficult a precise temperature regulation during the exposure time (around 30 mn).

We have studied 11 samples of $(TMTTF)_2AsF_6$ (T_{co}=100 K). We have performed energy scans from 2460 eV to 2490 eV with 2eV steps at low temperature (~40 K) and higher temperature (between 150 K and 300 K). At each energy, the crystal was rotated (rocking curve) in order to integrate the intensity.

Our best results are indicated on figure 2. The Bragg intensity was obtained by integrating the intensity from the image plate. They were obtained on the 10th crystal at 220 K and the 11th one at 40 K on a (0kl) Bragg reflection. The sulfur edge was found to be at 2471 $\pm$ 1 eV, which is consistent with known values: pure S Edge is 2469.1 eV and in benzothiophene ~2470.4 eV [1].

The curves show a very similar variation of the intensity above and below the transition temperature. The difference above and below the S edge could be due to the absorption, which depends on the exact shape of the crystal.

This experiment demonstrates that a chemical shift between the different charged states of the molecule, if any, is **less that 2 eV**.

In conclusion, we would like to stress the necessity of a good low temperature experimental setup (airproof box, good temperature regulation) to perform these difficult but promising low energy experiments.

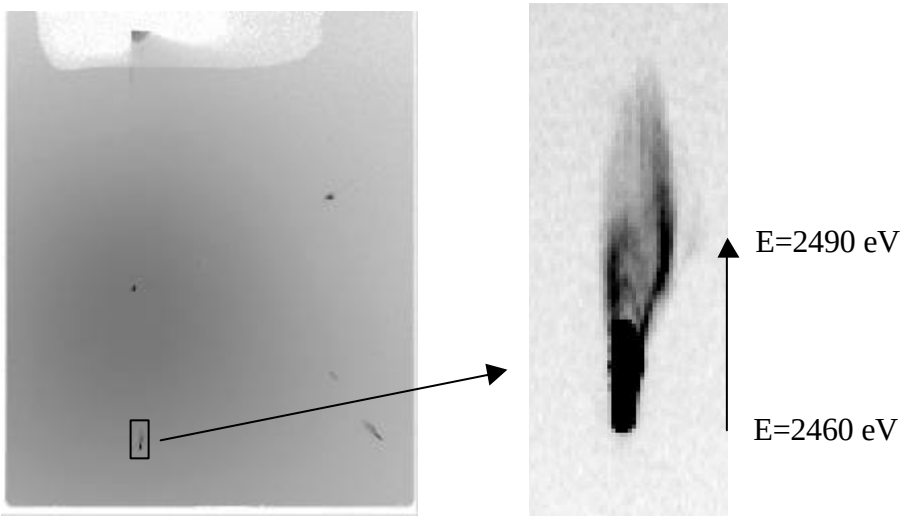

Fig. 1 Typical image plate from $(TMTTF)_2AsF_6$ at 300 K (left). Zoom of the previous image around a Bragg reflection (right). The flare effect is a size effect due to the absorption and was eliminated by closing the entrance slits.

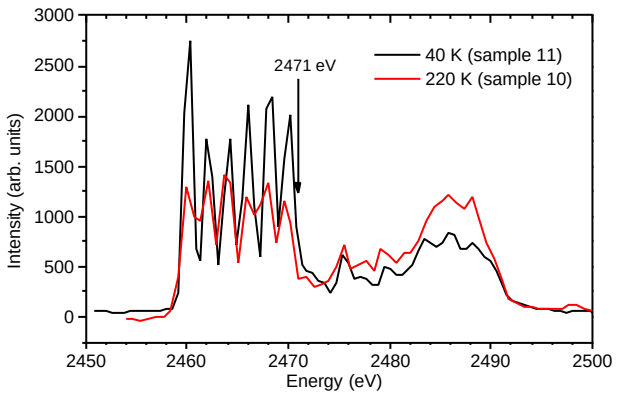

Fig. 2 : Energy scans of a (0kl) Bragg reflection at 40 K (black) and 220 K (red/thin).

[1] G. Georges and M. Gorbaty, J. Am. Chem. Soc. **111**, 3182 (1999).