ESRF	**Experiment title:** Ferroelectric-ferroelastic phase transition in pentakis- (methylammonium) undeca-chlorodibismuthate monitored by DAFS at the K-edge of chlorine and the M_V -edge of bismuth.	**Experiment number:** He 520
Beamline: ID1	**Date of Experiment:** from: 19. Nov. 1998 to: 25 Nov. 1998	**Date of Report:** 24. Febr. 1999
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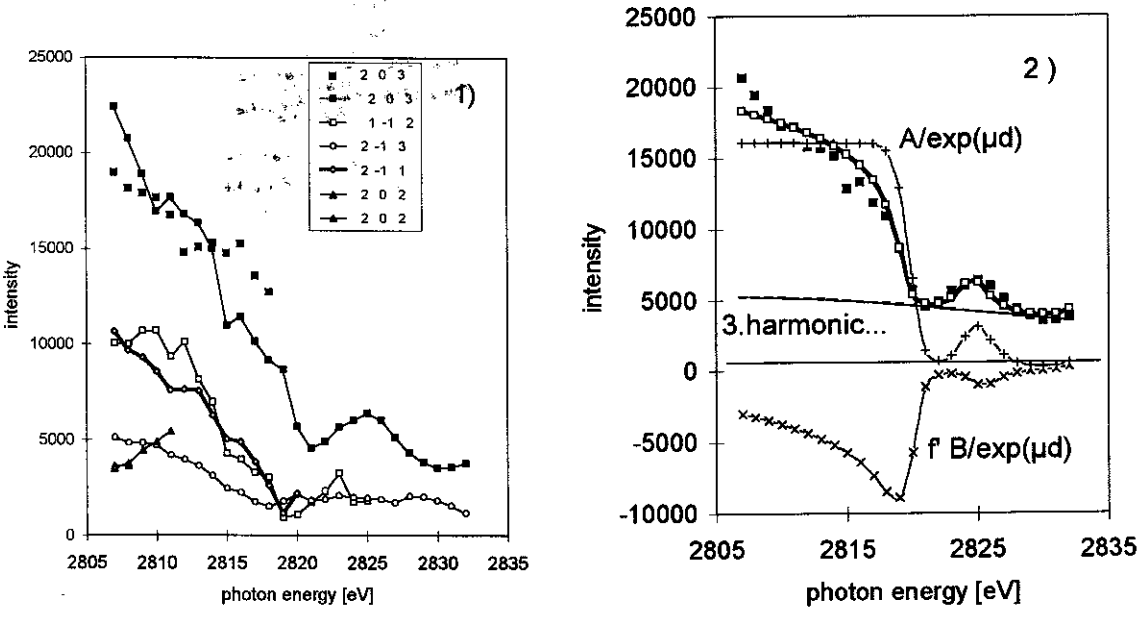
Report:

The X-ray intensity diffracted by a crystal of pentamethylammonium undekachlorobismuthate (PMACB) was measured at 30 wavelengths near the K edge of chlorine (4.40 Å). X-ray photons of that wavelength are strongly absorbed by any matter. Air in that sense is no exception.

The instrument ID1 with its large vacuum vessel meets the requirements X-ray diffraction with soft X-rays to a large extent. However, due to the presence of absorbers in the beam and due to the low sensitivity of the CCD camera to soft X-rays the probability to record 4.4 Å photon leaving the double crystal monochromator was about $1/10^5$.

The amount of higher harmonics emitted by the undulator had to be reduced to a level which allowed the observation of the diffraction of 4.4 Å photons. This was done in two ways: (1) the second crystal of the double monochromator was slightly detuned, and (2), a mirror had been installed at 1 m distance from the sample in order to eliminate efficiently shorter wavelengths. Nevertheless, the measured X-ray diffraction pattern came mainly from the 3^{rd} order harmonics (the 2^{nd} order is eliminated by the Si 111 monochromator). The first step of the data analysis was to index the whole diffraction pattern. This allowed to identify the fundamental reflections. Only four fundamental spots were measured over a significant energy range (see Fig. 1a). The accuracy of the data is not sufficient to give a quantitative analysis.

However the reflections reveal clearly a dispersion resulting essentially from f' of chlorine (Fig.2). The 202 reflection is particularly interesting for our purpose because the chlorine bridging the bismuth atoms in the $[Cl_5Bi - \mathbf{Cl} - BiCl_5]^{5-}$ anion and, in a strategic position during phase transition, contributes mainly to the partial structure factor of the 11 chlorine atoms. Unfortunately the energy scan for this reflection is incomplete.



The experiment shows that anomalous dispersion of X-ray diffraction can be measured at wavelengths beyond 4 Å, although the sensitivity of the CCD camera of hardly more than 1% to 4.4 Å photons is not ideal for this purpose. The recent replacement 240 μm kapton foil behind the double monochromator by a 12 μm foil will increase the intensity of 4.4 Å photons at the sample by two orders of magnitude in future experiments. The planned installation of focusing mirrors would reject the higher harmonics more efficiently and increase the intensity at the sample by another order of magnitude.

It is fair to say that the instrument ID1 is perfectly suited for DAFS experiments with soft X-rays on crystals which tolerate the evacuated environment. For many materials the vacuum and the absence of humidity is a real advantage. This is the case for the chlorobismuthates which are slowly hydrolysed by atmospheric moisture.

Some time has been spent on development of a program which controls the crystal orientation and the transfer of the data from the CCD camera to disks.