ESRF	**Experiment title: Location by anomalous diffraction of cations in industrial zeolites**	**Experiment number**: 02-02-156
Beamline: BM2	**Date of experiment**: from: 04/04/01 to: 08/04/01	**Date of report**: 21/08/01
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Names and affiliations of applicants (* indicates experimentalists):

John LYNCH (IFP)
Bernadette REBOURS (IFP)
Christophe PICHON (IFP)
Jean-Louis HODEAU (Laboratoire de Cristallographie du CNRS de Grenoble)

Report:

Scientific context

In the field of petroleum industry, high performance industrial processes for catalysis, separation and purification of hydrocarbons mixtures are based on adsorption properties of materials such as X, Y zeolites (which are both support materials in catalysis and molecular sieves in separation and purification processes).
It is well know by industrial references that there is a high level of correlation between the catalytic or adsorbing properties of a zeolitic material and the location of their cations of compensation (or cation-like compounds) in the structure.

So it appears that to increase our comprehension of the selectivity of adsorbents or catalytic properties of supporting materials, and to be able to predict the properties of new solids, we can determine their cationic distribution. Location of cations (or cation-like compounds) through X-ray measurement is more relevant to those conditions than neutron diffraction location of molecules.
Four main crystallographic sites can be occupied by cations in X or Y zeolites. The occupancy factor of each site depends on the content of the zeolite. Due to the presence of several cations in the zeolite and to the very low content of some of them, anomalous diffraction is essential to perform such experiments.

Three diffractograms are needed (near the edge, some 10 eV from the edge and far from the edge) for each cation (or cation-like compounds) to be accurately localised. In addition, for the most sensitive diffraction peaks, data will be collected at a number of energies close to the edge in order to determine the best compromise between sensitivity to the anomalous effect and energy stability.

Diffraction experiments

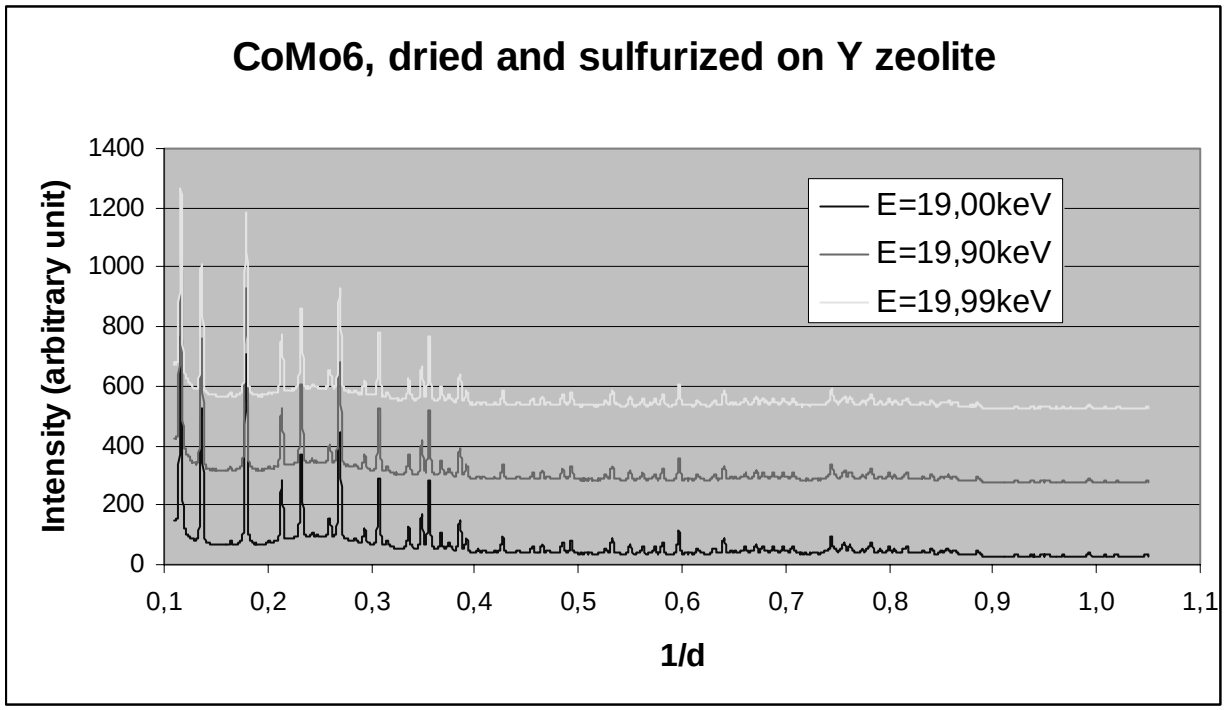
The system we chose to study during this run was a Na-Y zeolite partially exchanged with cation-like molybdenum species : molybdenum oxyde MoO_3 and an Anderson salt called $CoMo_6$ which general formula is $CoMo_6O_{24}H_6^{3-}/Co^{3+}$. For Y zeolite partially exchanged with Anderson salt, two different cases were studied : one composed by a dried zeolite and an other with a dried and then sulfurized zeolite.
In order to have some reference data with Anderson salt deposited on catalytic support material, we also studied a well know from laboratory system composed by $CoMo_6O_{24}H_6^{3-}/Co^{3+}$ on alumina.

The synchrotron diffraction patterns have been collected at the powder diffraction beam line BM02. The samples were prepared in sealed capillary glasses (1mm in diameter). For sulfurized samples the filling of the capillary glass was performed under pure nitrogen atmosphere to avoid destabilisation of the Anderson entity.

Each system was recorded at three different energies : 19.99keV (i.e. very close to the K-edge of molybdenum), 19.90keV (i.e. 10eV under the edge) and 19.00keV (i.e. far from the edge).

Results

The refinement of collected data is still in progress. The diffraction diagrams collected for a Y zeolite exchanged with $CoMo_6O_{24}H_6^{3-}/Co^{3+}$, dried and sulfurized, are reported on the following picture.



In spite of the fact that it is difficult to extract anomalous information, this study has given us some experience in anomalous experiments and have demonstrated the possibilities of the D2AM beamline for such studies.