



Experiment title:

**CATION POSITIONS FOR SELECTIVE GAS
SEPARATION IN FAUJASITE**

**Experiment
number:**
CH 183

Beamline:

D2 am

Date of experiment:

from: 31/07/96 to: 01/08/96

Date of report:

27/08/97

Shifts:

6

Local contact(s):

J.F. BERAR

Received at ESRF:

1 SEP. 1997

Names and affiliations of applicants (* indicates experimentalists):

*PLEVERT Jacques
DI RENZO Francesco
GOURSOT Annick
*DUTARTRE Roger

Laboratoire des Matériaux Catalytiques et Catalyse en Chimie Organique
8, rue de l'école normale
34296 Montpellier cedex 5
France

*CHIARI Giacomo

Universita di Torino
Dipartimento di scienze mineralogiche e petrologiche
Via Valperga Caluso, 37 - 10125 Torino - Italy

Report:

The experiment aimed at refining framework atoms and at finding the cation positions in two dehydrated faujasite-type zeolites LSX with 100% and 85% lithium-exchange (LSX: Low Silica X with Si/Al ratio 1).

Series of breakdowns delayed the measurements, nevertheless three data set were collected with $\lambda = 1.0735 \text{ \AA}$ on D2am beam line after the adaptation of the "7-cercle" goniometer to powder diffraction experiments. A powder pattern of the zeolite $\text{Li}_{96}\text{Si}_{96}\text{Al}_{96}\text{O}_{384}$ was recorded at room temperature after *in situ* dehydration. The two other data collections were performed on the zeolite $\text{Li}_{84}\text{Na}_{12}\text{Si}_{96}\text{Al}_{96}\text{O}_{384}$ at 420°C and next at room temperature.

Two types of problem appeared during the refinement of data:

1- The fully-exchanged Li-LSX sample was not properly dehydrated, probably due to an insufficient vacuum obtained with a primary vacuum pump, also responsible for the brown color taken by the sample. It may be supposed that the part of the

sample on the top of the container adsorbed the oil of the pump resulting in non-homogeneous activation of the zeolite.

2- The line positions in the third powder pattern show a shift compared with the estimated positions calculated with a cell parameter $a=24.70 \text{ \AA}$, as showed in Fig. 1. This effect may be due to a displacement of the sample holder combined with a misalignment of the instrument as suggested by the hump under each intense line of the powder pattern (Fig. 2).

The Rietveld refinement technique combined with Fourier methods applied to the second powder pattern collected at 420°C gave a solution leading to $R_{wp}=7.8\%$ and $R_B=6.0\%$ (Fig. 3), in which nearly all the cations were found (76 lithium cations + 5 sodium cations).

The objective was to refine simultaneously the SR data and the neutron data to obtain unambiguous information on the lithium cation positions in the supercage. Despite some uncertainty about cation site occupations, we used in a first stage the neutron diffraction data alone (J. Plevret, F. Di Renzo, F. Fajula and G. Chiari. *J. Phys. Chem.*, accepted).

Fig. 1
SHIFT (2-THETA)

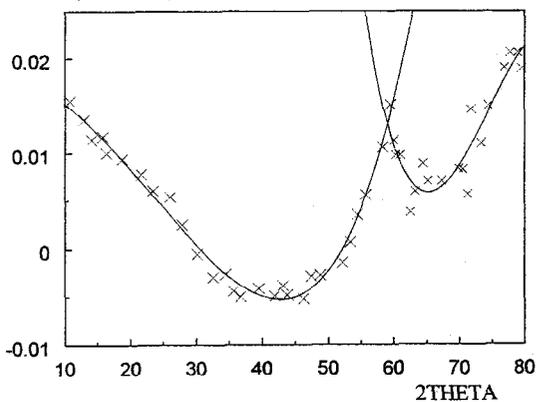


Fig. 2

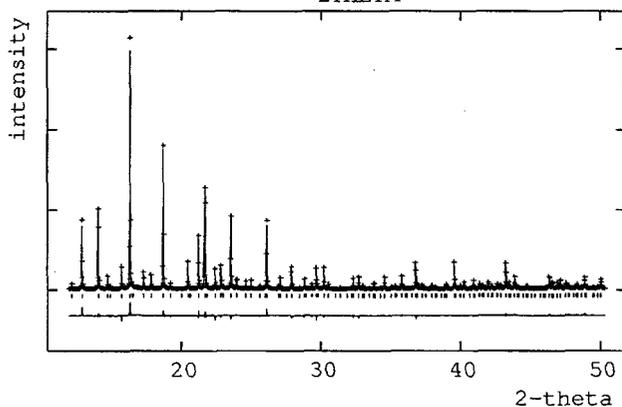
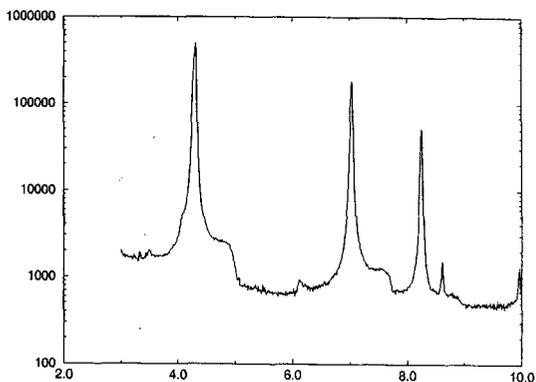


Fig. 3