



Experiment title:  
**Cation location and its environment in  
Rb-exchanged zeolites**

Experiment  
number:  
**CH 107**

<b>Beamline:</b> <b>D16</b>	<b>Date of experiment:</b> from: May 24 1996                      to: May 26 1996	<b>Date of report:</b> <b>20 August 1996</b>
<b>Shifts:</b> <b>6</b>	<b>Local contact(s):</b> Andy Fitch and Gavin Vaughan	<b>Received at ESRF:</b>

**Names and affiliations of applicants** (\* indicates experimentalists):

C. Lamberti<sup>(a\*)</sup>, S. Bordiga<sup>(a)</sup>, G. Ricchiardi<sup>(a\*)</sup>, M. Salvalaggio<sup>(a\*)</sup>,  
A. Zecchina<sup>(a)</sup>, and G. L. Marra<sup>(b\*)</sup>,

(a) Dipartimento di Chimica I.F.M., Via P. Giuria **7, I- 10125** Torino, Italy

(b) ENICHEM Istituto Donegani Via G. Fauser 4, Novara, I-28100 Italy

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**Report:** In the last few years our group has undertaken an ongoing research program in the characterization of alkali-metal exchanged zeolites, mainly using infrared spectroscopy of adsorbed probe molecules [1-7]. In November 1995 we have performed at GILDA D8 beamline of ESRF, an EXAFS study on Rb-exchanged zeolites to characterize the local Rb environment (see corresponding exp. report). The aim of the present work is to localize cation sites in Rb-exchanged Y zeolites and to study the cation migration upon interaction with strong ligands like H<sub>2</sub>O and NH<sub>3</sub>. To fulfill this request a total control of the sample atmosphere is fundamental. We have thus prepared an *ad hoc* cell able to activate the sample under dynamical vacuum up to 700 K and to locate the so dehydrated sample in a boron silicate capillary used for XRD measurements; if desired a known amount of pure adsorbate can be dosed before the capillary occlusion. To minimize the sample absorptions XRD measurements have been performed just before the Rb  $k_{\alpha}$  edge, using  $\lambda = 0.8497 \text{ \AA}$ . The XRD pattern has been collected up to  $2\theta = 67^{\circ}$  ( $d \approx 0.75 \text{ \AA}$ ) using a continuous scan acquisition mode with a scan speed progressively decreased at high  $2\theta$  to increase the counts statistic. We were able to collect at RT three spectra: in **vacuum**, **+H<sub>2</sub>O** and **+NH<sub>3</sub>**. The data analysis has been performed by Rietveld method included in GSAS software package. As the observed spectra show a bump due to the amorphous scattering from the capillary, we have chosen an angular range for the refinement from  $7^{\circ}$  to  $67^{\circ}$ . Starting from structure data of Na-Y zeolite (space group *Fd-3m*) [8] we have identified two extraframework sites for Rb<sup>+</sup> cations (sites II and I') and one for unexchanged Na<sup>+</sup> cations (site I) with refined occupancies.

Even if for all the samples a good agreement between measured and refined data has been obtained, the here presented results must be considered as preliminary. Figure 1 shows the refinement of the spectrum collected in vacuo, while Figure 2 shows the Fourier map of the difference between the observed and the calculated structure factors centered in the II Rb site. Preliminary results from this experiment will be presented next days at [9]. We are indebted with all the D16 staff which has enabled us to work under optimal conditions and particularly with Gavin Vaughan for sophisticated data binning.

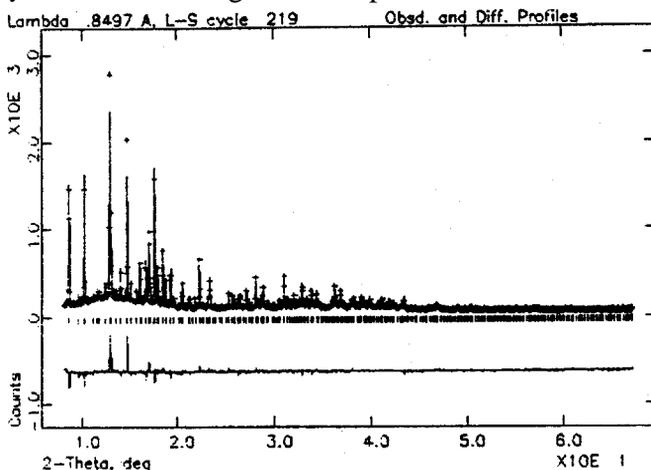


Figure 1: Rietveld refinement of Rb-Y in vacuo condition. Refinement of spectra collected in presence of H<sub>2</sub>O and NH<sub>3</sub> are of similar quality.

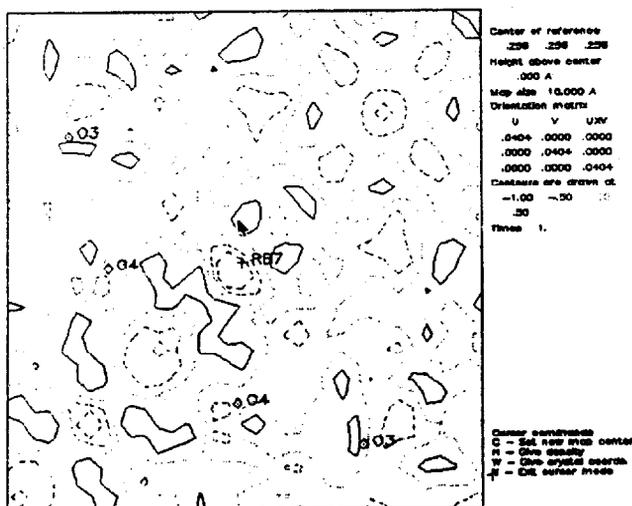


Figure 2: Fourier map of the difference between measured and simulated structure factors centred in site II.

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