



|  |  |                                      |
|--|--|--------------------------------------|
|  | <b>Experiment title:</b><br>Structural characterization of a new Rb <sup>+</sup> lithosilicate porous framework with the RUB-23 topology | <b>Experiment number:</b><br>CH 1346 |
| <b>Beamline:</b><br>ID11   | <b>Date of experiment:</b><br>from: 30/01/2003 to: 04/02/2003  | <b>Date of report:</b><br>25/02/2004 |
| <b>Shifts:</b>   | <b>Local contact(s):</b><br>Gavin Vaughan  | <i>Received at ESRF:</i>             |
| <b>Names and affiliations of applicants</b> (* indicates experimentalists):<br><b>*Aaron J. Celestian, Stony Brook University, Geosciences</b><br><b>John B. Parise, Stony Brook University, Geosciences and Chemistry</b> |  |                                      |

## Report:

### Introduction

Zeolites and synthetic nanoporous analogues have a widespread geologic distribution and are often used for industrial purposes. Zeolites are usually characterized by a corner shared aluminosilicate framework with a negative charge induced by the replacement of Si<sup>4+</sup> by Al<sup>3+</sup>. To maintain electroneutrality, cationic extra-framework species are incorporated into the nanoporous channels. The extra-framework species are exchanged for guest cations or molecules and give rise to the unique characteristics that molecular sieves embody. In order to maximize the cation exchange capacity, one method is to further reduce the charge of the framework by replacing some of the Si<sup>4+</sup> with Li<sup>+</sup>. This work shows the crystal structure determination of a Rb<sup>+</sup> lithosilicate with the RUB-23<sup>1</sup> topology.

### Experimental

Single crystals were grown by hydrothermal methods similar to those used by Park et al.<sup>1</sup>. Using a Bruker SMART 1500 CCD detector at the ID-11 beamline, diffracted intensities were collected at 293K to a maximum 2-theta 42.6° ( $\lambda=0.5095\text{\AA}$ ) using -0.2°  $\omega$  scans. The unit cell was determined to be P2<sub>1</sub> a=12.1817(6) $\text{\AA}$  b=10.2976(3) $\text{\AA}$  c=12.1867(5) $\text{\AA}$   $\beta=118.08(1)^\circ$  using 695 reflections in the program SMART. After integration using SAINT and absorption corrections using SADABS, the final R(int) of 0.04 was used for structure solution by Direct Methods in SHELXTL. The final R and wR were 0.33 and 0.60 respectively.

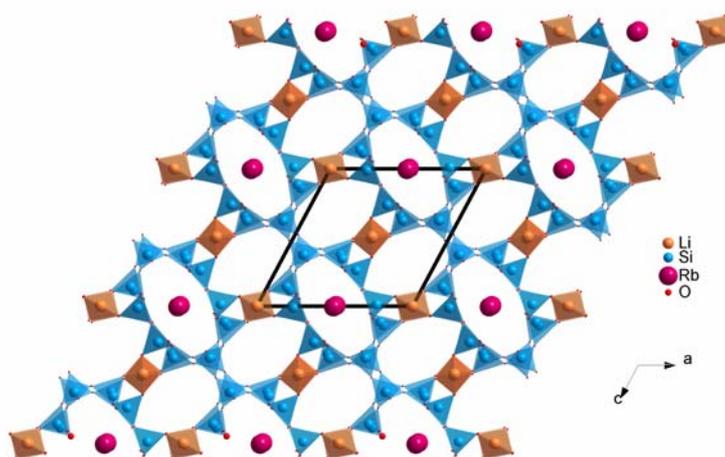
### Results

The partial structure of Rb-RUB-23 is shown in Figure 1. The structure consists of one dimensional 8 and 10 member rings (MR) parallel to [010]. The Rb<sup>+</sup> cations are located in

the 10MR bonded to 8 framework  $O^{2-}$  and interstitial water molecules. The Fourier difference map indicated residual electron density in the 8MR which may be attributed to  $Li^+$  and water molecules.

Reasons for the high final R values may be due to twinning. Two likely twin laws have been found. The first in the  $P2_1$  setting, resembles hexagonal  $P6_3$  with 2 twin components generated from the  $[(0\ 0\ 1)(0\ 1\ 0)(-1\ 0\ -1)]$ . The second in a C2 unit cell setting, has a  $\beta$  angle approximately  $90^\circ$  where a one component twin law would be  $[(1\ 0\ 0)(0\ -1\ 0)(0\ 0\ -1)]$ . Both tests have been conducted and have not yielded significant decreases in the final R values. Final structure refinements are ongoing.

<sup>1</sup>Park, S. H., Daniels, P. & Gies, H. (2000) *Micropor. Mesopor. Mat.* **37** 129-143



*Figure 1* The Rb form of the RUB-23 structure as determined from single crystal XRD.