Experimental report

13/09/2022

Proposal: EASY-745 **Council:** 4/2020

Title: Complete structure solution of a new strontium aluminosilicate Sr1.05Si1.9 Al0.1 O5

Research area: Chemistry

This proposal is a new proposal

Main proposer: MICHAEL PITCHER

Experimental team:

Local contacts: Stanislav SAVVIN

Samples: Sr1.13 Si1.87 Al0.13 O5

Instrument	Requested days	Allocated days	From	To
D2B	4	4	08/02/2021	09/02/2021

Abstract:

We have recently isolated a new metastable strontium aluminosilicate Sr1.05 Si1.9 Al0.1 O5 by a computationally-guided exploration approach. The PXRD pattern is indexed to a primitive monoclinic unit cell (volume ~740 A3). We have conducted a partial structure solution in-house using laboratory PXRD: charge flipping and simulated annealing have located the strongly-scattering Sr atom positions with a high degree of certainty, but we have very low precision on the Si and O positions. Neutron diffraction will allow us to locate these sites correctly and complete the structure solution.

Experiment EASY-745 aimed to complete the ab-initio structure solution of a newly-discovered strontium aluminosilicate synthesised by glass-crystallisation, thought at the time of the proposal to be "$Sr_{1.05}Si_{1.9}Al_{0.1}O_5$". Neutron diffraction data were collected at room temperature from D2B. A Rietveld refinement was used to (i) test an initial structural model originally solved ab-initio from PXRD, (ii) obtain precise atomic coordinates for the oxide sublattice, (iii) verify the presence of additional oxide sites. The final refined structure corresponds to the composition $Sr_2Si_3O_8$. It is a 1-dimensional structure comprised of infinite $[Si_3O_8]$ ribbons. The shift from the nominal composition "$Sr_{1.05}Si_{1.9}Al_{0.1}O_5$" results from the presence of residual glass, which contributes broad features to the background of the neutron diffraction patterns, confirmed later by solid state NMR and a series of laboratory powder diffraction experiments using Al_2O_3-spiked samples to quantify the amorphous content, and found to represent ~30 mol% of the sample.

The experiment will be included in the thesis of Euan Duncan (CEMHTI, CNRS UPR3079). It is being prepared for publication as part of a wider exploratory study of computationally-guided synthesis in the SrO-Al_2O_3-SiO_2 phase diagram.

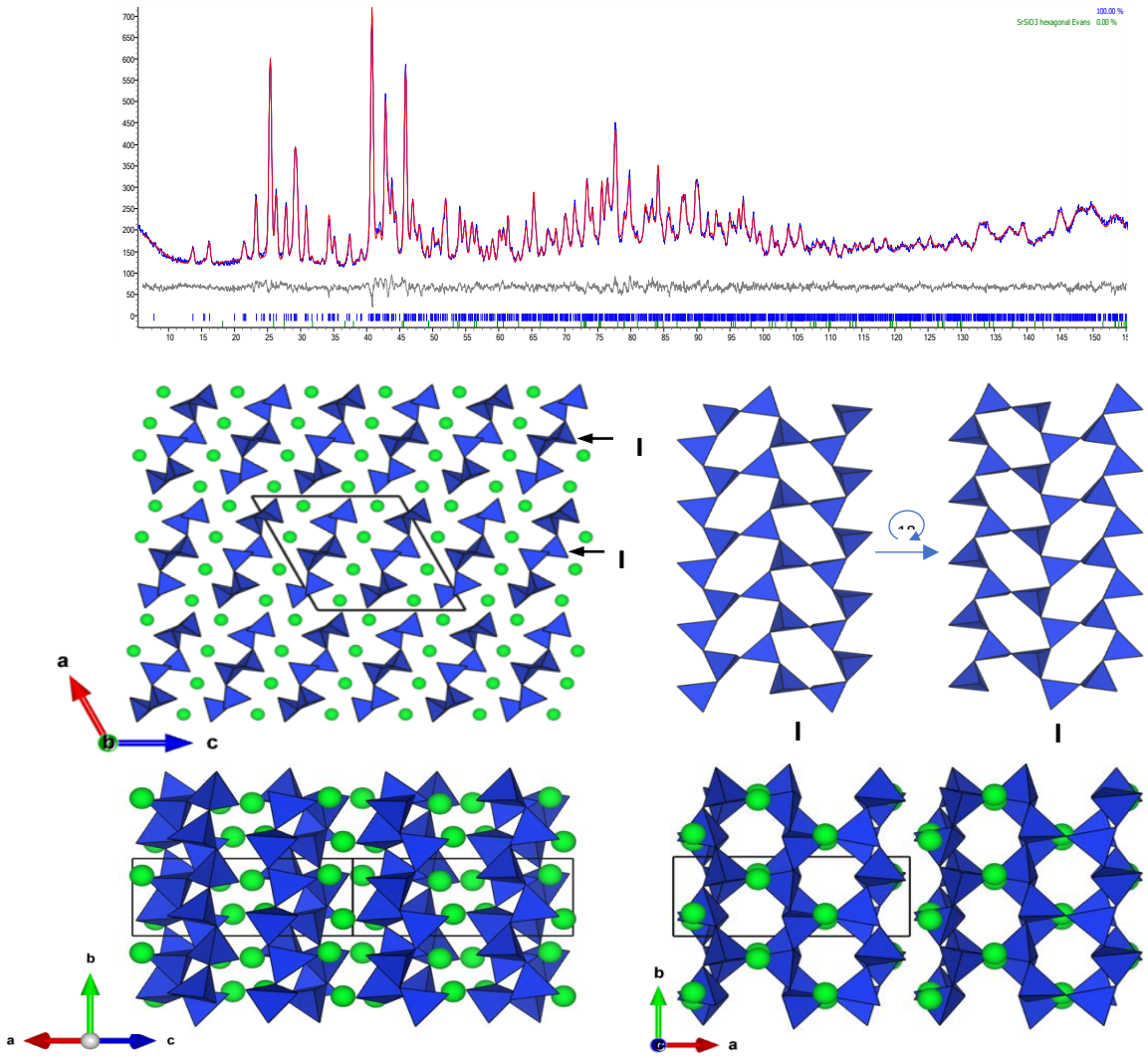

Figure: *Rietveld refinement against the obtained D2B data (above), and structural projections of $Sr_2Si_3O_8$ along differet cell axes, highlighting the $[Sr_3O_8]$ infinite ribbons directed along the crystallographic b axis.*