Experimental Report

Proposal: **5-11-406** **Council:** 4/2014

Title: Framework ordering and H-bonding network in the zeolite stilbite, NaCa2Al5Si13O36&#8226 - 14H2O

This proposal is a new proposal

Researh Area: Other...

Main proposer: **GATTA G. Diego**

Experimental Team:

Local Contact: FERNANDEZ DIAZ Maria Teresa

Samples: NaCa2Al5Si13O36 x 14H2O

Instrument	Req. Days	All. Days	From	To
D9	12	18	10/11/2014	28/11/2014
D19	12	1	05/12/2014	06/12/2014
CYCLOPS	2	2	06/12/2014	08/12/2014

Abstract:

The aim of the present study is a reinvestigation of the crystal structure of a the zeolite stilbite (NaCa2Al5Si13O36 x 14H2O) at 298 and 20 K by single-crystal neutron diffraction, in order to provide: a) a reliable description of the Si/Al-ordering of the tetrahedral framework (which governs the presence of Bronsted-Lewis acid sites and so the catalytic properties of this material); b) the location of the proton sites and the description of the complex H-bonding network; c) the anisotropic displacement parameters of all the atomic sites, including the H-sites. The vibrational regime of the extra-framework sites is significant at room-T in this class of materials, and so low-T data are needed. As X-ray diffraction data have proved to be inadequate to model the Si/Al-ordering and to locate directly the H atoms, single-crystal neutron diffraction represents the best experimental technique to answer the open questions about the crystal structure/crystal chemistry of stilbite.

Experimental report

Experiment n. 5-11-406

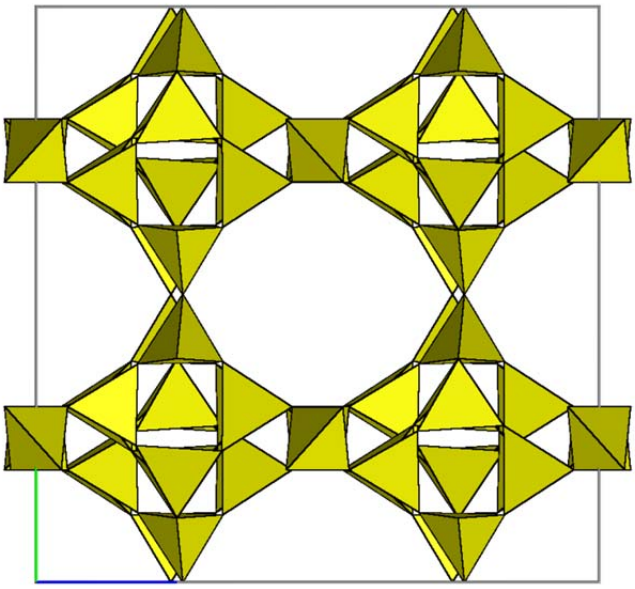
Title: Framework ordering and H-bonding network in the zeolite stilbite, $NaCa_2Al_5Si_{13}O_{36}\bullet 14H_2O$

by G. Diego Gatta

The aim of this experiment was a reinvestigation of the crystal structure and crystal chemistry of a natural stilbite (from Poona/India, ideally $NaCa_2Al_5Si_{13}O_{36}\cdot 14H_2O$) at ambient and low temperature (20 K) by single-crystal neutron diffraction, in order to provide:
a) a reliable description of the Si/Al-ordering of the tetrahedral framework;
b) the location of the proton sites and the description of the complex H-bonding network in the zeolitic voids, along its low-*T* induced rearrangement;
c) the anisotropic displacement parameters of all the atomic sites, including the H-sites.

The single-crystal diffraction data were successfully indexed in the in the non-standard *F*2/*m* space group with a = 13.62 Å, b = 18.25 Å , c = 17.84 Å, and β = 90.3°, according to the structure model of Cruciani et al. (1997) (Fig. 1). A preliminary anisotropic structure refinement showed a fully disordered Si/Al distribution between the tetrahedral sites. More difficult is the location of the proton sites, due to an apparent disorder of the extra-framework population. We are confident to obtain a final structure model soon.

Figure 1. Crystal framework of the zeolite stilbite, viewed down [100], based on the neutron structure refinement of this study.



References

Cruciani G., Artioli G., Gualtieri A., Stahl K., Hanson J.C. (1997) Dehydration dynamics of stilbite using synchrotron X-ray powder diffraction. American Mineralogist, 82, 729-739