ESRF	**Experiment title:** Single Crystal Structure Determination of a New Scandium Phosphate	**Experiment number**:
Beamline: ID-11	**Date of experiment**: from: 30/01/03 to: 03/02/03	**Date of report**: 27/02/04
Shifts:	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Hyunsoo Park*, Ivor Bull and John B. Parise

Departments of Geosciences and Chemistry

State University of New York at Stony Brook

Stony Brook, NY 11794

USA

Report:

Since the successful syntheses of a family of organically templated alumino-phosphates ($AlPO_4$-n) by Flanigen and co-workers[1], a great number of metal-phosphates have been induced to form open framework and layered materials. Interest in this class of materials is driven by the need for new functionality, selective catalysis and ion exchange for example, and the new insights provided by exploratory synthetic and structural chemistry. The incorporation of a wider variety of species, with coordination geometries different from the corner connected tetrahedral geometries traditionally associated with molecular sieves, resulted in the discovery of new structure types containing mixed tetrahedral-octahedral geometry. For instance, a number of transition metal phosphates such as $TiPO^2$ and $FePO^3$ have been discovered and structurally characterized.

Although a great variety of metals has been incorporated into new and known framework topologies, there are but few reports of the successful synthesis of scandium-based open framework materials. Our research focuses on the synthesis and structural characterization of new scandium phosphate frameworks. Since many of these materials are obtained only as microcrystals of 5 to 10 μm in size, they are unsuitable for conventional in-house X-ray sources.

Using the facilities at the ID-11 beamline we determined the crystal structure of organically templated scanium phosphate frameworks and one of those is reported here. After screening a large number of crystals, one suitable for data collection was found and the structure of a new scandium phosphate, namely $(C_6H_{14}N_2)Sc_2(HPO_4)_4.3H_2O$, was discovered. This material was synthesized under mild hydrothermal conditions with 1,2-diaminocyclohexane as a structure directing agent. Data were collected (λ = 0.50915 Å) at room temperature with a scan width of 0.1° in ϕ and an exposure time of 1 second per frame. A total of 1786 frames were collected. The raw intensity data were processed with programs SAINT[4] and SADABS[5]. The structure was solved using the SHELXTL package[6]. It crystallizes in the orthorhombic Pbcn space group with the unit cell paramters of a = 23.952(1) Å, b = 17.8281(8) Å, c = 10.6823(5) Å. At present, the anisotropic refinement yielded R and wR factors of 8.37% and 26.4%, respectively, for 3559 reflections $I > 2\sigma(I)$. Somewhat large values of reliability factors may be attributed to high residual electron densities caused by the disorder of water and organic molecules.

The crystal structure of $(C_6H_{14}N_2)Sc_2(HPO_4)_4.3H_2O$, illustrated in Figure 1, consists of alternating PO_4 and ScO_6 structural building units (SBU). Two crystallographically independent Sc atoms are octahedrally coordinated to oxygen with the Sc – O bond distances ranging from 2.00 to 2.13 Å. The Sc(2) atom is actually bonded to a H_2O molecule, as seen from the slightly longer Sc – O bond length of 2.20 Å. All four P atoms are tetrahedrally coordinated to three framework oxygens (1.50 to 1.54 Å) and one dangling oxygen which is protonated (1.58 to 1.60 Å). These ScO_6, $ScO_5(OH_2)$ and HPO_4 polyhedra are linked by corners to form a zig-zag chain along the (001) direction. The organic template and water molecules occupy the space between two adjacent chains. There is hydrogen bonding present between the inorganic chain and the organic molecules, as the terminal O atoms from the phosphate groups interact with the hydrogen atoms from the amine with the N - H …O distance of 3.09 Å. The free water molecules also interact with the amine via hydrogen bonding with N – H … O length of 2.88 Å.

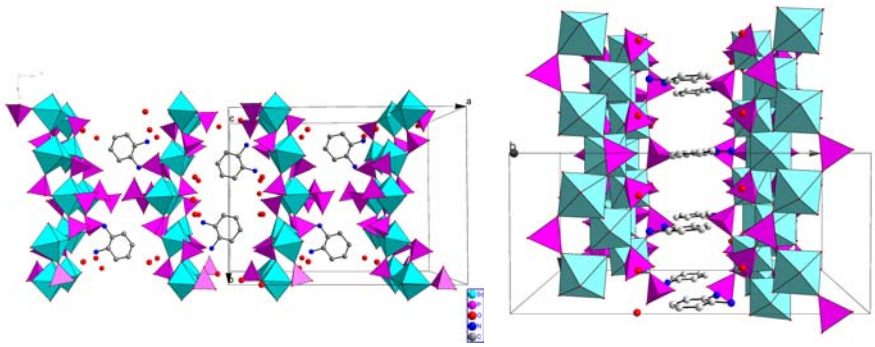

Figure 1: Crystal Structure of $(C_6H_{14}N_2)Sc_2(HPO_4)_4.3H_2O$ viewed along [001] and [010] directions

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