08-Inorganic and Mineralogical Crystallography

We will demonstrate ways in which structural information on zeolites can be obtained from high resolution images and electron diffraction patterns. Quantitative agreement is obtained between experimental electron diffraction intensities and theoretical calculations. Under favorable circumstances, the weak-phase object approximation (WP0A) can be used to simplify image interpretation and quantification, leading to the determination of the secondary building units (SBUs) of the framework.

**MS-08.02.05**

C₆H₅(CH₃)₂Sb₅S₄ AND Cs₂Sb₅S₄(O,S): TWO ZEOLITE-LIKE PHASES WITH NANOSPORUS SULFOANTIMONATE(III) FRAMEWORKS.

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In the course of a systematic search for nanoporous materials with non-tetrahedral host frameworks, we synthesized single crystals of the two title compounds and determined their structures from X-ray diffraction data.

C₆H₅(CH₃)₂Sb₅S₄: triclinic red plates; a = 15.866(3) Å, b = 11.581(2) Å, c = 8.295(2) Å, α = 71.46(2)*, β = 75.71(2)*, γ = 82.25(2)*, Z = 2, space group P1. R = 0.061, Rw = 0.052 for 3172 independent reflections with I > 3σ(I) and 215 variables.

Cs₂Sb₅S₄(O,S): triclinic red needles; a = 11.872(2) Å, b = 13.277(6) Å, c = 14.859(9) Å, α = 84.58(5)*, β = 85.32(5)*, γ = 86.19(4)*, Z = 2. Reflections with h = 2n + 1 are weak but sharp. An average structure with m = n/2 was refined to R = 0.061, Rw = 0.052 for 1316 independent reflections with I > 2σ(I) and 92 variables.

Both structures contain [Sb₅S₄] pyramids with d(Sb-S) = 2.65 Å, most of which are complemented by one or two S atoms with d(Sb-S) between 2.85 Å and 3.3 Å to form [Sb₅S₄] octahedra with n = 4, 5, i.e. distorted octahedra with (6-n) ligands missing. In each of the two structures these γ-octahedra share edges and/or corners via common S atoms to form a 3-dimensional framework. The large cavities are located in channel-like pores of the respective frameworks (Fig. 1).

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**MS-08.02.06**

THE STRUCTURE OF A NEW COBALT CONTAINING ALUMINOPHOSPHATE.

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**SUMMARY.** The novel structure has been determined by using a single crystal of CoAlPO₄·H₂O to collect data with MoKα radiation on an Enraf-Nonius CAD4 diffractometer. From the method of preparation (Wilson & Flanigen) the AIPO₄-21 type was expected. The monoclinic unit cell has the following parameters: a = 8.539(1) Å, b = 15.540(1) Å, c = 7.736(1) Å, β = 105.65(1)°. The space group is P2₁/c. Determination of the structure shows distorted octahedral coordination of cobalt atom, and one AlO₄ and two PO₄ tetrahedra building the framework. The presence of template molecules ethylenediamine has also been determined. The cobalt atom is coordinated to five framework oxygen atoms and one nitrogen atom of the ethylenediamine.

**INTRODUCTION.** The structure determination of the discussed AIPO₄-based material was undertaken as part of our studies of aluminophosphates, where aluminium and phosphorus are replaced by small amounts of other elements, mainly transition metals. The substitution gives a new group of microporous materials, which indicate to be useful for catalytic and adsorption applications and many studies of CoAPO-molecular sieves have been reported recently, with respect to stability, redox behavior and associated acid properties (Kruseheer-Chernetzki et al.). Octahedral coordination of the cobalt atom in such compounds is rare, in spite of the fact, that for Co₆¹⁺ ion, as well as for several other ions of the first transition series, ligand-field stabilization energies distinguish the tetrahedral configuration relative to the octahedral one. From some points of view it is consistent, since the available data (Kruseheer-Chernetzki et al.) show, that the occurrence of Brønsted acidic properties is related to the presence of tetrahedral MeO₄Al⁻⁻ units.

**EXPERIMENTAL.** The synthesis of a new compound has been performed using the reaction gel of molar composition 0.4 Co(OH)₂ : 0.8 Al₂O₃ : 1.0 PO₄ : 1.0 en : 50 H₂O (en = ethylenediamine, ac = acetic), following the procedure of crystallization described by Wilson & Flanigen. In a beaker-based autoclave under static conditions at 468 K over 4 days, prismatic-shaped prismatic crystals were obtained. A crystal of 1.14 x 0.34 x 0.25 mm in size was used for data collection. The crystal structure was solved by direct methods. An absorption correction was made using Gaussian method (a = 2.217 mm⁻¹). Nonhydrogen atoms were refined anisotropically and hydrogen atoms, identified in an electron density map, isotropically. The final R (on F) was 0.037, Rw = 0.029 for 2326 contributing reflections and 182 parameters.