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 (WPOA) can be used to simplify image interpretation and quantification, leading to the determination of the secondary building units (SBU) of the framework.

MS-08.02.05 $[CH_3NH_3]_2Sb_8S_{13}$ AND $Cs_4Sb_{14}S_{20}(O,S)_3$: TWO ZEOLITE-LIKE PHASES WITH NANOPOROUS SULFOANTIMONATE(III) FRAMEWORKS. By F. Liebau* and X. Wang, Mineralogisches Institut der Universität Kiel, Germany.

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.

$[CH_3NH_3]_2Sb_8S_{13}$: triclinic red plates; a=15.866(3)Å, b=11.581(2)Å, c=8.295(2)Å, α=71.46(2)°, ß=75.71(2)°, γ=82.25(2)°, Z=2, space group $P\bar{1}$. R=0.061, R_w=0.052 for 3172 independent reflexions with $I>3\sigma(I)$ and 215 variables.

$Cs_4Sb_{14}S_{20}(O,S)_3$: triclinic red needles; a=11.872(12)Å, b=13.277(5)Å, c=14.859(9)Å, α=84.58(5)°, ß=85.52(5)°, γ= 86.19(4)°, Z=2. Reflexions with h=2n+1 are weak but sharp. An average structure with $a'=a/2$ was refined to R=0.061, R_w=0.052 in $P\bar{1}$ using 1136 independent reflexions with $I>2.5\sigma(I)$ and 92 variables.

Both structures contain $[SbS_3]$ pyramids with d(Sb-S)≤2.65Å, most of which are complemented by one or two S atoms with d(Sb-S) between 2.85Å and 3.3Å to form ψ-$[SbS_n]$ octahedra with n=4, 5, i.e. distorted octahedra with (6-n) ligands missing. In each of the two structures these ψ-octahedra share edges and/or corners via common S atoms to form a 3-dimensional framework. The large cations are located in channel-like pores of the respective frameworks (Fig.1).

Support from the DFG is gratefully acknowledged.

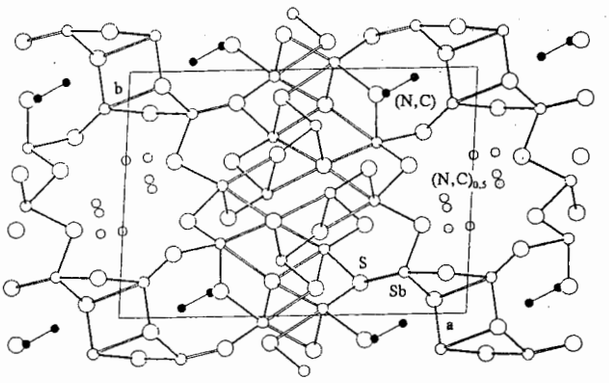

Fig.1 Projection of the structure of $[CH_3NH_3]_2Sb_8S_{13}$ along [001]. Open bonds:2.85-3.3Å, Solid bonds:2.39-2.65Å.

MS-08.02.06 THE STRUCTURE OF A NEW COBALT CONTAINING ALUMINOPHOSPHATE. By Primož Fajdiga, Ljubo Golič, Department of Chemistry and Chemical Technology, University of Ljubljana, Slovenia, Nataša Zabukovec, National Institute of Chemistry, Ljubljana, Slovenia, Venčeslav Kaučič*, National Institute of Chemistry, Ljubljana and Department of Chemistry and Chemical Technology, Ljubljana, Slovenia.

SUMMARY. The novel structure has been determined by using a single crystal of $CoAlP_2O_8C_2H_9N_2$ to collect data with MoKα radiation on an Enraf-Nonius CAD4 diffractometer. From the method of preparation (Wilson&Flanigen) the AlPO-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 = 110.65(1)°. The space group is $P2_1/c$. Determination of the structure shows distorted octahedral coordination of cobalt atom, and one AlO_4- and two PO_4-tetrahedra building the framework. The presence of template molecule 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 $AlPO_4$-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 absorption applications and many studies of CoAPO-molecular sieves have been reported recently, with respect to stability, redox behavior and associated acidis properties (Krausheer-Czarnetski et al.). Octahedral coordination of the cobalt atom in such compounds is rare, in spite of the fact, that for Co d^7 ion, as well as for several other ions of the first transition series, ligand-field stabilization energies disfavour the tetrahedral configuration relative to the octahedral one. From some points of view it is convenient, since the available data (Krausheer-Czarnetski et al.) show, that the occurrence of Brönsted acidic properties is related to the presence of tetrahedral $MeO_{4/2}^{2-}$ units.

EXPERIMENTAL. The synthesis of a new compound has been performed using the reaction gel of molar composition 0.4 $Co(ac)_2$: 0.8 Al_2O_3 : 1.0 P_2O_5 : 1.0 en : 50 H_2O (en = ethylenediamine, ac = acetate), following the procedure of crystallisation described by Wilson&Flanigen. In a teflon-lined autoclave under static conditions at 468 K over 4 days, purple needle-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 (μ = 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.

