THE CRYSTALLINE STRUCTURE OF NH_{4}LaP_{4}O_{12}.


During our studies on lanthanum phosphates' phase relations the apparently new compound was obtained in the pure, crystalline form. The results of some preparative procedures and X-ray powder data based on literature reports suggested it to be lanthanum oxyphosphate. Single-crystal X-ray analysis, however, proved that the compound is NH_{4}LaP_{4}O_{12} which is surprisingly stable at elevated temperatures. Preliminary X-ray data are: system monoclinic, space group C2/c with a = 7.941(4), b = 12.645(15), c = 10.702(9) Å β = 110.00(5)°. Diffractionometer collected data were reduced in a standard way the absorption correction being allowed for using program DIFABS by N. Walker and D. Stuart (1983). 1536 reflections with I > 1.966(I) were used for the refinement. H-atoms were partly resolved in a difference Fourier synthesis. The refinement led to R1 = 0.037 and R2 = 0.046. The structure consists of P_{4}O_{12} tetrahedras linked together to form tetracycles, LaO_{6} dodecahedras and ammonium tetrahedras. The La-O bond lengths vary between 2.475(5) and 2.516(5) Å. The figure resembling that found in LaP_{3}O_{9} (J. Matuszewski, T. Znamierowska, and T. Smallerowska, 1986). The crystals of NH_{4}LaP_{4}O_{12} are isomorphous with those of NH_{4}PrP_{4}O_{12} (Mausse R. et al., 1977).

A REFERENCE OF K_{4}V_{2}O_{14}.

By T. Ozeki, N. Ichida and Y. Sasaki, Department of Chemistry, Faculty of Science, The University of Tokyo, Japan.

K_{4}V_{2}O_{14}. Mr=96.0. triclinic, P31m, a=8.6509(4), c=8.973(3) Å, V=326.88(4) Å^{3}, Z=1, R=0.031 for 793 unique reflections (2θ<60°, F>3σ(F)). The structure, which was first determined by Byström and Evans (Acta Chem. Scand. 13(1959)377-378), was refined. It contains two independent V atoms, one in a VO_{6} octahedron and another in a VO_{4} tetrahedron. The other coordinated tetrahedrally by four O atoms. These polyhedras share edges to make up two dimensional networks. K atom is surrounded by five O atoms in the lower layer and five in the layer above. V-O distances in the network are 1.737(2), 1.739(3) Å. V-O distances for terminal O atoms are 1.621(5) Å and the tetragonal pyramid, respectively. They are slightly longer than the value of 1.577(3) Å for the VO_{4} tetragonal pyramid in the orthorhombic V_{2}O_{5} (Byström and Evans, Acta Chem. Scand. S2(1956)1467-1469). K-O distances vary from 2.829(5) to 3.280(6) Å.

THE CRYSTAL AND MOLECULAR STRUCTURE OF (Cu(Al),MoO_{4})_{2}NH_{4}LaP_{4}O_{12}.

By J.A. Costamagna and J.L. Culf, Universidad de Santiago de Chile, Casilla 10733, Santiago, Chile.

Light blue crystals of the title compound are formed when copper-bearing molybdenite concentrate is leached with nitric acid and ammonium aquous solution, in order to obtain molybdenum trioxide.

The molecular structure of the crystal was determined from data given by chemical analysis, IR spectrum, thermogravimetric behavior and magnetic susceptibility measurements.

The compound crystallizes with space group P2_{1} and the following cell dimensions:

a = 6.155(3), b = 7.168(3), c = 7.516(4) Å

R1 = 0.058, R2 = 0.060, T = 110.00(5)°

The structure was solved by Patterson and Fourier methods using 1071 unique reflections (Fourier-synthesis, MoKα-radiation, 0-2θ scan), and refined to R=0.036. The [Cu(NH_{4})_{2}(MoO_{4})_{2}]+ anion is a planar complex of Cu(II) and shows two MoO_{4} tetrahedras, acting as monodentate oxygen-donor ligands, and two NH_{4} in a trans configuration. Bond distances in the copper plane are 1.965 and 2.071 Å (Cu-N) and 1.662 and 2.000 Å (Cu-O), while in the tetrahedra they range from 1.677 to 1.840 Å.

STRUCTURAL STUDIES ON ALUMINATE SODALITES.

By W. Depmeier, Institut für Kristallographie, Universität, D-7500 Karlsruhe, Federal Republic of Germany.

Our research program on aluminate sodalites of general composition M[Al_{12}O_{24}](XO_{4})(M=Ca,Sr..., X=S,Cr,Mo,...) has been continued. These compounds are interesting for several reasons; the most important ones being: i) The occurrence of ferroic phase transitions; ferroelectric and ferroelastic species have been found. ii) Strong repulsive host-guest interactions lead to important structural strains (bond length and angular). iii) Complicated superstructures occur which in some cases are best described as commensurate modulations; iv) The possible existence of tricritical points. The actual investigations focused on:

a) The phase sequence of Ca[Al_{12}O_{24}](WO_{4})_{2} with its two phase transitions at 614 and 656 K.

b) The phase sequence of Sr[Al_{12}O_{24}](CRO_{4})_{2} with temperatures of transition near room temperature (-293 K and -297 K).

c) The room temperature phase of Sr[Al_{12}O_{24}](MoO_{4})_{2}. Its distribution of superstructure reflections suggests that it might be regarded as commensurately modulated.

d) The room temperature phase of Sr[Al_{12}O_{24}](WO_{4})_{2}.

Latest results will be presented.