

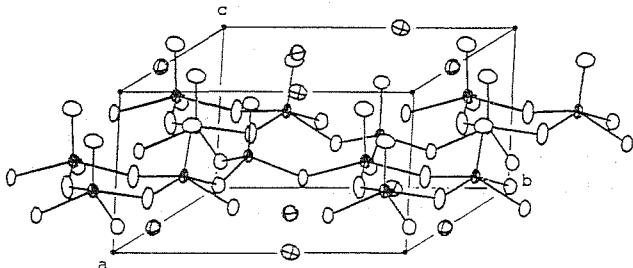
08.2-12 THE CRYSTAL STRUCTURE OF  $\text{NH}_4\text{LaP}_4\text{O}_{12}$ .  
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During our studies on lanthanum phosphates' phase relations the apparently new compound was obtained in the pure, crystalline form. Both preparative procedures and X-ray powder data based on literature reports suggested it to be lanthanum oxyphosphate. Single-crystal X-ray analysis, however, proved that the compound is  $\text{NH}_4\text{LaP}_4\text{O}_{12}$  which is surprisingly stable at elevated temperatures. Preliminary X-ray data are: system monoclinic, space group  $C2/c$  with  $a = 7.941(4)$ ,  $b = 12.645(13)$ ,  $c = 10.702(9)$  Å  $\beta = 110.00(5)^\circ$ . Diffractometer collected data were reduced in a standard way the absorption correction being allowed for using program DIFABS by N. Walker and D. Stuart (1983). 1366 reflexions with  $I > 1.966(I)$  were used for structure solution (Patterson) and refinement. H-atoms were partly resolved in a difference Fourier synthesis. The refinement led to  $R_1 = 0.037$  and  $R_2 = 0.046$ . The structure consists of  $\text{PO}_4$  tetrahedra linked together to form tetracycles,  $\text{LaO}_3$  dodecahedra and ammonium tetrahedra. The La - O bonds lengths vary between 2.475(5) and 2.516(5) Å the figure resembling that found in  $\text{LaP}_3\text{O}_9$  (J. Matuszewski, J. Kropiwnicka and T. Znamierowska, 1986). The crystals of  $\text{NH}_4\text{LaP}_4\text{O}_{12}$  are isomorphous with those of  $\text{NH}_4\text{PrP}_4\text{O}_{12}$  (Masse R. et al., 1977).

08.2-13 A REFINEMENT OF  $\text{K}_3\text{V}_5\text{O}_{14}$ .

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

$\text{K}_3\text{V}_5\text{O}_{14}$ ,  $M_r = 596.0$ , trigonal,  $P31m$ ,  $a = 8.6909(4)$ ,  $c = 4.9973(3)$  Å,  $U = 326.88(4)$  Å<sup>3</sup>,  $Z = 1$ ,  $R = 0.031$  for 793 unique reflections ( $2 < \theta < 40$ ,  $F > 3\sigma(F)$ ). The structure, which was first determined by Byström & Evans (Acta Chem. Scand. 13(1959)377-378), was refined. It contains two independent V atoms, one forming a  $\text{VO}_5$  tetragonal pyramid and the other coordinated tetrahedrally by four O atoms. These polyhedra share apices 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), 1.820(5) & 1.923(2) Å. V-O distances for terminal O atoms are 1.621(5) & 1.577(3) Å for the tetrahedron and the tetragonal pyramid, respectively. They are slightly longer than the value of 1.577(3) Å for the  $\text{VO}_5$  tetragonal pyramid in the orthorhombic  $\text{V}_2\text{O}_5$  (Enjalbert & Galy, Acta Cryst. C42(1986) 1467-1469). K-O distances vary from 2.829(5) to 3.280(6) Å.



08.2-14 THE CRYSTAL AND MOLECULAR STRUCTURE OF  $[\text{Cu}(\text{NH}_3)_2(\text{MoO}_4)_2](\text{NH}_4)_2$ . By J.A. Costamagna and J.L. Garin, Universidad de Santiago de Chile, Casilla 10233, Santiago, Chile.

Light blue crystals of the title compound are formed when copper-bearing molybdenite concentrate is leached with nitric acid and ammonia aqueous 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  $P1$ ,  $Z = 1$  and the following cell dimensions:

$$a = 6.155(3), \quad b = 7.158(3), \quad c = 7.516(4) \text{ \AA} \\ \alpha = 108.52(6), \quad \beta = 93.99(6), \quad \gamma = 113.72(4)^\circ$$

The structure was solved by Patterson and Fourier methods using 1071 unique reflexions (Four-circle diffractometer,  $\text{MoK}\alpha$ -radiation,  $\theta = 2\theta$  scan), and refined to  $R = 0.046$ .

The  $[\text{Cu}(\text{NH}_3)_2(\text{MoO}_4)_2]^-$  anion is a planar complex of Cu(II) and shows two  $\text{MoO}_4$  tetrahedra, acting as monodentate oxygen-donor ligands, and two  $\text{NH}_3$  in a trans configuration. Bond distances in the copper plane are 1.965 and 2.071 Å (Cu-N) and 1.962 and 2.000 Å (Cu-O), while in the tetrahedra they range from 1.677 to 1.840 Å.

08.2-15 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  $M_8[\text{Al}_{12}\text{O}_{24}](\text{XO}_4)_2$  ( $M = \text{Ca}, \text{Sr} \dots$ ,  $X = \text{S}, \text{Cr}, \text{Mo}, \text{W} \dots$ ) 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 focussed on:

- The phase sequence of  $\text{Ca}_8[\text{Al}_{12}\text{O}_{24}](\text{WO}_4)_2$  with its two phase transitions at 614 and 656 K.
- The phase sequence of  $\text{Sr}_8[\text{Al}_{12}\text{O}_{24}](\text{CrO}_4)_2$  with temperatures of transition near room temperature ( $\sim 291$  K and  $\sim 297$  K).
- The room temperature phase of  $\text{Sr}_8[\text{Al}_{12}\text{O}_{24}](\text{MoO}_4)_2$ . Its distribution of superstructure reflections suggests that it might be regarded as commensurately modulated.
- The room temperature phase of  $\text{Sr}_8[\text{Al}_{12}\text{O}_{24}](\text{WO}_4)_2$ .

Latest results will be presented.