## 08. INORGANIC AND MINERALOGICAL CRYSTALLOGRAPHY

structure (P21/n, a=12.969(7), b=9.782(4), c=8.517(4) A,  $\beta$ =106.31(4). The central unit is a onedimensional infinite chain  $\pm [Na_2/2NaCsGe_4]$  which consists of linked cubanes {-[NaCsGe\_4] -Na - [Ge\_4CsNa] - Na} in trans-arrangement. A further Cs atom stays without direct contact to the Ge\_units and preserves again the compo-sition MX. The structure of this compound is sition MX. The structure of this compound is represented by the formula Cs1[CsNaNa2/2Ge4].

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SYNTHESES AND CRYSTAL STRUCTURES OF 08.2 - 41AMINOPYRIDINIUM POLYMOLYBDATES. By J. M. Gutiérrez-Zorrilla<sup>3</sup>, P. Román<sup>3</sup>, C. Esteban-Calderón<sup>5</sup>, M. Martínez-Ripoll<sup>5</sup> and S. García-Blanco<sup>5</sup>, a) Dept. Guímica (Inorsánica) Univ. del País Vasco, Artdo. 644, Bilbao, Spain, b) Dept. Rayos X, Instituto Rocasolano CSIC, Serrano 119, Madrid-6, Spain.

Aminopyridinium hepta- and octamolybdates have been prepared in aqueous or  $H_2O/DMF$  solutions. Preparation of these compounds can be formulated by equations (1) and (2):

a9.sol.  $MoO_3 + B$ (BH)<sub>6</sub> Mo<sub>7</sub> O<sub>24</sub> . nH<sub>2</sub> O (1) PH > 6.5

H<sub>2</sub>0/DMF -----> (B'H)<sub>4</sub> Mo<sub>8</sub>0<sub>26</sub> . 2DMF MoO<sub>3</sub> + B' (2)PH < 3.5

B = 2-aminopy (i), 3-aminopy (ii), 4-aminopy (iii), Z-amino-3-methylpy (iv), Z-amino-4-me-thylpy (v), Z-amino-5-methylpy (vi) and Z-amino-6-methylpy (vii) B' = 2-aminopy (viii). (py = pyridine)

Sinsle crystals of (i), (iii), (iv), (vi) and

(viii) have been obtained. Crystal data for (i) are: $(C_5H_7N_2)_c$  Mo<sub>7</sub>O<sub>24</sub>.3H<sub>2</sub>O a=14.8161(4), b=17.5073(4), c=20.8492(6)A, /3 = 107.503(2)°, V=5153.7(3)A<sup>3</sup>, Z=4, P2<sub>4</sub>/n, Do=2.15 Dx=2.17 g cm-3, R=0.030, wR=0.034 for 4959 observed reflexions. The structure contains isolated ( $Mo_7 O_{24}$ )6- anions linked to the 2-aminoeyridinium cations and water molecules through hydrogen bonds of N-H...O and O-H...O type. Crystal structures of (iii), (iv), (vi) and (viii) are in progress.

SYNTHESIS AND CRYSTAL SYMMETRY OF A 08.2 - 42MONOCLINIC MODIFICATION OF MoO<sub>3</sub>.H<sub>2</sub>O. by J.L. Garin and M.A. Solar. Departamento d de Metalurgia, Universidad de Santiago de Chile, Santiago, Chile.

Molybdenum trioxide mono-hydrate is formed as an intermediate substance during thermal decomposition of some ammonium molybdates, the final product being anhydrous molibdenum trioxide. The compound has been reported in earlier literature, as having at least three different pha-ses. The structure of the white modification or "a-molybdic acid", for instance, crystalli-zes with triclinic symmetry in the space group PI (Böschen and Krebs (1975), Acta Cryst. <u>B30</u>, as having at least three different pha-1.795).

The authors synthesized another modification of the compound, by leaching of molybdenite concen-trate with nitric acid. Single crystals gro-wing in well-sphaped white needles were obtai-ned. The chemical composition of the crystals, as determined by usual analysis techniques, was found to be very close to stoichiometry.

The crystal data for  $MoO_2$ ,  $H_2O$  where determined by recording three dimensional data on Weissenberg and Buerger precession photographs, using Ni-filtered Cu-radiation. The unit cell is monoclinic, with a=9.720Å, b=3.725Å, c=7.179Å,  $\beta$ =102,4° and space group P2<sub>1</sub>/m.

The calculated density, assuming four formula mit in the unit cell, is  $4.24~{\rm g~cm^{-3}}$ . The measured value resulted to be  $4.20~{\rm g~cm^{-3}}$ .

From our results we can conclude that the compound under study discloses another white isomer of Mo03.H20.

08.2-43 STRUCTURAL CHARACTERISTICS OF La<sub>2</sub>Ge<sup>IV</sup>Ge<sup>V</sup>Ge<sup>VIO</sup>9 AND RELATIONSHIPS TO THE RARE EARTH ALUMINIUM GERMANATES OF THE TYPES  $RE_2ALEGeO_4$ ] 20H AND REALGeVO5. By <u>K.-H. Klaska</u>, M. Werk and O. Jarchow, Mineralogisch-Petrogra-phisches Institut der Universität, Grindelallee 48, 2000 Hamburg 13, Germany.

Single Crystals of  $La_2Ge^{IV}Ge^{VI}O_q$  were synthesized hydrothermally at 800°C and 1500 bar.  $\begin{array}{l} {\tt La_2Ge^{IV}Ge^{V}Ge^{VI}0_9 \ crystallizes \ in \ space \ group} \\ {\tt AI \ with \ a=7.68_6\ } \mbox{$\mathbbmath$$$$}, \ b=5.71_2\ \ \mbox{$\mathbbmath$$$$$$$$$}, \ c=16.57_0\ \ \mbox{$\mathbbmath$$$$$$$$$}, \end{array}$  $\approx$ =90.59°,  $\beta$ =109.82°,  $\gamma$ =88.72° and Z=4. The direct method program MULTAN 80 was used for the structure determination. The structure is characterized by slightly distorted [GeO<sub>6</sub>] -

octahedra, [GeO<sub>5</sub>]-tetragonal pyramids and

 $\texttt{IGeO}_{\ensuremath{\Delta}}$  -tetrahedra. To the best of our knowledge this is the first structure with three different coordination numbers of the Ge-atoms. The [GeO<sub>6</sub>]-octahedra join into chains parallel [OlO] by sharing edges. Adjacent chains are cross-linked by pairs of edge-sharing tetra-gonal pyramids. In addition an inter-chain

linkage is provided by insular [GeO<sub>4</sub>]-tetra-

hedra. The Ge-polyhedra form layers running in the a,b-plane. These layers are connected by  $La^{3+}-0$  bonds to a three-dimensional network.

The new compound La  $e^{IV}Ge^{V}Ge^{VI}O_9$  has a structure closely related to those of

 $\text{RE}_2\text{AllGeO}_4$  20H and  $\text{RE}_2\text{AlGeVO}_5$  (Jarchow, Klaska, Werk, Z. Krist. (1982) 159, 65-67). All three structures are based on straight chains of edge-sharing octahedra.