PS08.01.33 THE NEW TERNARY INTERMETALLIDE WITH A GIANT UNIT CELL IN THE Nd-Ru-Sn SYSTEM. J. Stupien-Dunn, P. Salamakha, O. Bodak. "Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Okolina 2, Wroclaw, Poland; Inorganic Chemistry Department, L'viv State University, Kyiv and Melfodya str. 6, 290005 L'viv, Ukraine.

The structure of Nd$_2$Ru$_{17}$Sn$_30$ was studied by single crystal X-ray diffraction. The title compound crystallizes in the cubic space, space group Fm-3m, a=30.785A, Z=4. Nd$_2$Ru$_{17}$Sn$_30$ is the first representative of a new structure type in the ternary intermetallic systems, however is related to Tb$_11$Fe$_2$Ge$_{12}$ type of structure [1]. The differences are due to composition as well as the additional Ru atoms in 4(b) position in neodymium ruthenium tin alloy, which in Th$_{11}$Fe$_2$Ge$_{12}$ is not filled. Hence, the number of atoms in the unit cell increases from 112 to 1128. Coordination polyhedra for additional Ru atoms are cubes.


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PS08.01.34 STRUCTURE REFINEMENT OF CuInSe$_2$ BY CCD CAMERA. K. Suda, H. Kitahara, N. Ishizawa and Y. Noda, Research Laboratory of Engineering Materials, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-Ku, Yokohama 226, Japan, Department of Materials Science, Faculty of Engineering, Tohoku University, Aramaki, Aoba-ku, Sendai 980-77, Japan.

Three-circle diffractometer equipped with a charge-coupled device (CCD) camera has been used for collecting three-dimensional diffraction data of CuInSe$_2$ inorganic crystal and the results were compared with those obtained by the conventional four-circle diffractometer. Crystals were grown by the traveling heater method using pure In as the solvent. The distance between the crystal and the phosphor plate was fixed at about 30 mm. Since the peaks have sharp profiles compared to the organic ones, the scan width of a frame was taken to be 0.15°. CuInSe$_2$ is tetragonal with the space group 142d. Cell dimensions of a=5.7852(1) Å and c=11.6254(4) Å were determined from 1961 reflections. In all, 2542 frames were taken to cover a hemisphere of reciprocal space. All 1198 observed reflections in a hemisphere of reciprocal space. All atoms are ordered, and the structure is indeed isomorphous with grandellite.

We have prepared single crystals of La$_2$O$_3$SO$_4$ and refined the structure in space group P2$_1$/c to an R-factor of 0.027 based on 1198 observed reflections in a hemisphere of reciprocal space. All atoms are ordered, and the structure is indeed isomorphous with grandellite.

There were two problems with the La$_2$O$_3$SO$_4$ structure solution: (1) There is a set of weak superstructure reflections that are not seen at all in powder diffraction. These lower the symmetry to monoclinic. The doubled cell has space group P2$_1$/c with dimensions $a=14.352(2)$, $b=4.288(1)$, $c=8.391(1)$ Å and $\beta=106.92(2)°$. A very similar C2/c cell is reached by the transformation (1 0 1) -1 (0 0 -1): $a=14.362(2)$, $b=4.288(1)$, $c=8.391(1)$ Å and $\beta=107.06(2)°$.

(2) The crystals are twinned on (1,0,-2) on either cell such that the twinning only means different mientations, and slight displacements, of one half of the sulfate ions.

References:

PS08.01.35 TWINNING IN LANTHANUM OXYSULFATE. Christer Svensson and Bengt Aurivillius. Dep. of Inorganic Chemistry, Lund University, P.O. Box 124, S221 00 Lund, Sweden.

There has long been some confusion about the structure of the lanthanide oxysulfates, Ln$_2$O$_3$SO$_4$. The intensity weighted reciprocal lattice can easily be mistaken for that of a tetragonal I-centered unit cell. Fahey [1] managed a partial structure solution, based on powder data, in the orthorhombic space group F222. Similarly, a single crystal study [2] in space group Cmcm indicated disordered sulfate oxygen atoms. In 1991 Kampf [3] succeeded in solving the related structure of grandellite, Pb$_2$O$_3$SO$_4$, in space group A2/c but concluded that "... it is highly unlikely that any of the lanthanide oxide sulfates have the grandellite structure".

We have prepared single crystals of La$_2$O$_3$SO$_4$ and refined the structure in space group P2$_1$/c to an R-factor of 0.027 based on 1198 observed reflections in a hemisphere of reciprocal space. All atoms are ordered, and the structure is indeed isomorphous with grandellite.

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PS08.01.36 LITHIUM ALUMINIUM BORATE, Li$_3$AlB$_2$O$_7$. By Göran Svensson and Johan Åhman, Inorganic Chemistry, Chalmers University of Technology, S-412 96 Göteborg, Sweden.

The most prominent features of this structure are isolated chains of edge-sharing AlO$_6$ octahedra running parallel to the crystallographic c-axis. These chains are connected by five-fold coordinated AlO$_6$ trigonal bipyramids and planar BO$_2$-groups. The lithium ions are located in the channels formed in the structure.

The title compound was obtained from a PbO/B$_2$O$_3$ flux, as a by product when Li$_2$O was grown. Needle shaped, clear crystals with lengths up to 4 mm was obtained. The crystal structure was determined from X-ray data collected on an Enraf-Nonius CAD-4 diffractometer. Two views of the Al$_2$B$_2$O$_7$-framework, boron and lithium atoms are omitted for clarity.