

**PS08.01.33 THE NEW TERNARY INTERMETALLIDE WITH A GIGANT UNIT CELL IN THE Nd-Ru-Sn SYSTEM.** J. Stepien-Damm\*, P. Salamakha, O. Bodak. \*Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Okolna 2, Wroclaw, Poland; Inorganic Chemistry Department, L'viv State University, Kyryla and Mefodiya str. 6, 290005 L'viv, Ukraine

The structure of  $\text{Nd}_{125}\text{Ru}_{67-x}\text{Sn}_{90}$  was studied by single crystal X-ray diffraction. The title compound crystallizes in the cubic system, space group  $Fm-3m$ ,  $a=30.785\text{\AA}$ ,  $Z=4$ .  $\text{Nd}_{125}\text{Ru}_{67-x}\text{Sn}_{90}$  is the first representative of a new structure type in the ternary intermetallic systems, however is related to  $\text{Tb}_{117}\text{Fe}_{52}\text{Ge}_{112}$  type of structure [1]. The differences are due to composition as well as the additional Ru atoms in 4(b) position in neodymium rhenium tin alloy, which in  $\text{Tb}_{117}\text{Fe}_{52}\text{Ge}_{112}$  is not filled. Hence, the number of atoms in the unit cell increases from 1124 to 1128. Coordination polyhedra for additional Ru atoms are cubes.

[1] V.K. Pecharsky, O. Bodak, V.K. Belsky, P.K. Starodub, I.R. Mokra, E.I. Gladyshevsky, *Kristallografiya*, vol.2, (1987), 344-348

**PS08.01.34 STRUCTURE REFINEMENT OF  $\text{CuInSe}_2$  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  $\text{CuInSe}_2$  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  $\omega$  scan width of a frame was taken to be  $0.15^\circ$ . The crystal of  $\text{CuInSe}_2$  is tetragonal with the space group  $I4_2d$ . Cell dimensions of  $a=5.7852(1)\text{\AA}$  and  $c=11.6254(4)\text{\AA}$  were determined from 1961 reflections. In all, 2542 frames were taken to cover a hemisphere of the reciprocal space in the range  $(\sin\theta)/\lambda \leq 0.81\text{\AA}^{-1}$ . Integrated intensities of 2334 reflections were retrieved from the frame data. It took about 12 hours to finish all measurement. The same crystal as used for the CCD experiment was mounted on a Ag rotating-anode four-circle diffractometer. The integrated intensities of 1024 reflections within a quadrant of reciprocal space in the range  $(\sin\theta)/\lambda \leq 0.81\text{\AA}^{-1}$  were measured using  $2\theta-\omega$  scan technique with  $\text{AgK}\alpha$  radiation monochromated by graphite (004). It took about 4 days to finish all of the measurement. Structural parameters obtained after the least-squares refinement generally agreed well within errors. Final R values were 0.0319 for 340 independent reflections obtained by the CCD diffractometer and 0.0171 for 402 independent reflections obtained by the four-circle diffractometer in the same range. Although the R factor of the CCD data set was slightly worse due to the relatively large disagreement between the observed and calculated structure factors of weak reflections, the CCD camera system was proved to be a useful equipment for the structural study of inorganic compounds as well as for organic and macromolecule compounds.

**PS08.01.35 TWINNING IN LANTHANUM OXYSULFATE.** Christer Svensson and Bengt Aurivillius†, Dept. of Inorganic Chemistry 2, Lund University, P.O. Box 124, S221 00 Lund, Sweden

There has long been some confusion about the structure of the lanthanide oxysulfates,  $\text{Ln}_2\text{O}_2\text{SO}_4$ . 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  $I222$ . Similarly, a single crystal study [2] in space group  $Immn$  indicated disordered sulfate oxygen atoms. In 1991 Kampf [3] succeeded in solving the related structure of grandreefite,  $\text{Pb}_2\text{O}_2\text{SO}_4$ , in space group  $A2/a$  but concluded that "... it is highly unlikely that any of the lanthanide oxide sulfates have the grandreefite structure".

We have prepared single crystals of  $\text{La}_2\text{O}_2\text{SO}_4$  and refined the structure in space group  $I2/c$  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 grandreefite.

There were two problems with the  $\text{La}_2\text{O}_2\text{SO}_4$  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  $I2/c$  with dimensions  $a = 14.352(2)$ ,  $b = 4.288(1)$ ,  $c = 8.391(1)\text{\AA}$  and  $\beta = 106.92(2)^\circ$ . A very similar  $C2/c$  cell is reached by the transformation  $(1\ 0\ 1/0\ -1\ 0/0\ 0\ -1) : a = 14.362(2)$ ,  $b = 4.288(1)$ ,  $c = 8.391(1)\text{\AA}$  and  $\beta = 107.06(2)^\circ$ .
- (2) The crystals are twinned on  $(1,0,-2)$  on either cell such that the twin reflections of, say, the  $I2/c$  cell are found at positions expected for the  $C2/c$  cell oriented as the  $I2/c$  cell. Structurally, the twinning only means different orientations, and slight displacements, of one half of the sulfate ions.

#### References:

- [1] J.A. Fahey (1976) in C.E. Lundin, Ed., Proceedings of the 12th Rare Earth Research Conference, Vail, Colorado, p. 762-771.
- [2] J.M. Haschke (1988) *J. Solid State Chem.* **73**, 71-79.
- [3] A.R. Kampf (1991) *Amer. Mineral.* **76**, 278-282.

**PS08.01.36 LITHIUM ALUMINIUM BORATE,  $\text{LiAl}_7\text{B}_2\text{O}_{17}$ .** 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  $\text{AlO}_6$  octahedra running parallel to the crystallographic  $c$ -axis. These chains are connected by five-fold coordinated  $\text{AlO}_5$  trigonal bipyramids and planar  $\text{BO}_3$ -groups. The lithium ions are located in the channels formed in the structure.

The title compound was obtained from a  $\text{PbO/B}_2\text{O}_3$  flux, as a by product when  $\text{LiAl}_5\text{O}_8$  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  $\text{Al}_7\text{B}_4\text{O}_{17}$ -framework, boron and lithium atoms are omitted for clarity.

