

mean plane. It makes dihedral angles of 17.4 (2), 85.22 (17) and 65.39 (16)°, respectively, with the phenyl, xanthen and anthracene ring systems. In the crystal structure, there are intramolecular C—H...O and C—H...N contacts and molecules are also linked by C—H... $\pi$  interactions.

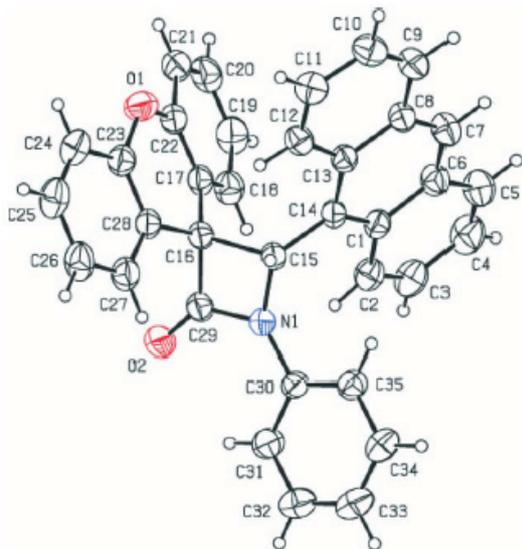


Fig 1: The molecular structure of title compound

The title compound,  $C_{35}H_{23}NO_2$ , crystallizes in the monoclinic  $P2_1/c$  space group with  $a = 13.6906$  (8) Å,  $b = 13.3085$  (7) Å,  $c = 17.3527$  (10) Å,  $\beta = 127.548$  (4)°,  $V = 2506.7$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $\mu = 0.08\text{mm}^{-1}$ ,  $R = 0.051$ ,  $wR = 0.110$ ,  $S = 0.90$ .

**Keyword:** crystal structure; 4-(9-Anthryl)-1-phenylspiro[azetidine-3,90-xanthen]-2-one; the  $\beta$ -lactam ring

#### FA4-MS01-P04

**Concentration Modulations and Short-Range vs. Long-Range Order in Layered Tellurides.** Oliver Oeckler<sup>a</sup>, Matthias N. Schneider<sup>a</sup>. <sup>a</sup>Department of Chemistry and Biochemistry, LMU Munich.

E-mail: [oliver.oeckler@gmx.de](mailto:oliver.oeckler@gmx.de)

Multinary tellurides have received much attention both as phase-change alloys for data storage and as promising thermoelectrics.[1,2] In the systems  $M\text{-Y-Te}$  ( $M = \text{Ge, Sn, Pb, Ag, In, ...}$ ;  $Y = \text{Sb, Bi}$ ) thermodynamically stable phases are found on pseudobinary lines  $(M\text{Te})_n(\text{Y}_2\text{Te}_3)_m$  and  $(\text{Y}_2\text{Te}_3)_m(\text{Y}_2)_k$ , they exhibit rocksalt-type as well as  $A7$ -type building blocks and can exhibit extremely long translation periods.

Quenching and subsequent annealing yields series of trigonal metastable phases  $(M\text{Te})_n(\text{Y}_2\text{Te}_3)_m(\text{Y}_2)_k$  with building blocks from both types of stable compounds. [3] Although long-periodic order is evidenced by sharp superstructure reflections, pronounced positional disorder is present. Single-crystal X-ray analysis as well as TEM show that the element concentrations vary continuously along the stacking direction of hexagonal atom layers.

Typical examples include  $(\text{Ge}_x\text{Sb}_{1-x/2})_2\text{Te}_3\text{:}[(\text{GeTe})_y\text{Sb}_{1-2y}]_8$  ( $x = 0.31$ ,  $y = 0.0325$ ;  $R\text{-}3m$ ,  $a = 4.266$ ,  $c = 75.02$  Å,  $R1 = 0.051$ ) and  $\text{Ge}_{2-x}\text{Sb}_{2+x}\text{Te}_2\text{Sb}_8$  ( $x = 0.43$ ;  $R\text{-}3m$ ,  $a = 4.258$ ,  $c = 97.23$  Å,  $R1 = 0.044$ ). This variation can be described as a long-range ordered occupational modulation which blurs the boundaries between “building blocks”. Quenching obviously leads to partial spinodal decomposition involving wavy concentration fluctuations. These “waves” are perfected by short-range diffusion during annealing, whereas decomposition is impossible as it requires long-range diffusion. Incommensurate concentration modulations have also been observed.

If the basic structure is close to cubic, e. g. in  $(\text{GeTe})_x(\text{Sb}_2\text{Te}_3)$  ( $x \geq 3$ ), high-temperature phases with disordered rocksalt-type structures are sometimes observed. Superstructure formation (i. e. either concentration waves or block formation) upon cooling consequently involves multiple twinning. Strain can impede this ordering, resulting in pseudocubic crystals with remarkable diffuse scattering; the maxima coincide with the positions of the strongest superstructure reflections of the corresponding long-range ordered (meta-)stable phases.

Concerning metastable crystalline phases of phase-change alloys, disordered cubic rocksalt-type structures are often assumed.[1] Investigations on comparable bulk samples (e. g. with compositions  $\text{Ge}_{0.73}\text{Sb}_{0.20}\text{Te}$  or  $\text{Ge}_{0.57}\text{Sb}_{0.34}\text{Te}$ ) evidence that pronounced short-range ordering matches with small (distorted) cutouts of the corresponding stable phases. This leads to the hypothesis that metastable crystalline samples may contain twinned and strained nanoscale domains of stable compounds and long-range order is impeded by the stress associated with volume change resulting from partial superstructure formation.

[1] Wuttig, M., Yamada N., *Nat. Mater.* **2007**, 6, 824. [2] Snyder G. J., Toberer E. S., *Nat. Mater.* **2008**, 7, 105. [3] Schneider M. N., Seibald M., Oeckler O., *Dalton Trans.* **2009**, 2004.

**Keywords:** phase-change technology; modulated structures; tellurium compounds

#### FA4-MS01-P05

**The Modulated Structure of  $\Lambda$ -Co(Sepulchrates)-Trinitrate at  $T = 100$  K.** Andreas Schönleber<sup>a</sup>, Sander van Smaalen<sup>a</sup>, Wolfgang Morgenroth<sup>b</sup>, Finn Krebs Larsen<sup>c</sup>. <sup>a</sup>Laboratory of Crystallography, University of Bayreuth, Bayreuth, Germany. <sup>b</sup>Institute of Geosciences, University of Frankfurt, Frankfurt am Main, Germany. <sup>c</sup>Department of Chemistry, University of Aarhus, Aarhus, Denmark.

E-mail: [andreas.schoenleber@uni-bayreuth.de](mailto:andreas.schoenleber@uni-bayreuth.de)

$\Lambda$ -Co(sepulchrates)-trinitrate,  $C_{12}H_{18}N_8Co^{3+} \cdot 3(NO_3)^-$  crystal-lizes at room temperature in symmetry  $P6_322$  [1]. Sepulchrates is (*S*)-(1,3,6,8,10,13,16,19-octa-azabicyclo-[6.6.6]eicosane). The cation and two of the nitrate anions are centred on three fold rotation axes, linked to each other by  $N\text{-H}\cdots\text{O}$  hydrogen bonds. The third nitrate anion is centred on the intersection of the two fold rotation axes and shows orientational disorder.

Applying light microscopy and spectroscopic measurements, two phase transitions have been observed at  $T_1 = 133$  K and at  $T_2 = 106$  K, respectively. The transition at  $T_1$  is interpreted as freezing of the disordered anions into two non-equivalent configurations [2]. By means of single crystal neutron diffraction the structural changes have been investigated as function of temperature [3]. Upon cooling the appearance of satellite reflections in the diffraction pattern at  $T_1 = 133$  K was discovered. The positions of these satellite peaks and therefore the length of the  $\mathbf{q}$ -vector varies continuously with temperature. Therefore the structure is incommensurately modulated. Two other phase transitions have been observed at  $T_2 = 107$  K and at  $T_3 = 98$  K.

We have performed single crystal X-ray diffraction at beam line D3 of Hasylab (DESY, Hamburg) at  $T = 100$  K. A first inspection of the data has shown, that the main reflections are regularly surrounded by satellite reflections up to second order. All observed peaks can be indexed in an hexagonal unit cell with  $a = b = 8.441$  Å and  $c = 15.732$  Å and the two  $\mathbf{q}$ -vectors  $(\sigma, \sigma, 0)$  and  $(-\sigma, 2\sigma, 0)$  with  $\sigma = 0.0882$ . We will present the results of the data analysis.

[1] Dubicki L., Ferguson J., Williamson B., *J. Phys. Chem.*, **1984**, 88, 4254. [2] Dubicki L., Ferguson J., Geue R.J., Sargeson A.M., *Chem. Phys. Lett.*, **1980**, 74, 393. [3] Larsen F.K., Jørgensen P., Grønbaek Hazell R., Lebech B., Thomas R., Geue R.J., Sargeson A.M., in *Molecular structure: Chemical reactivity and biological activity*, Stezowski *et al.* (ed.) IUCr, Oxford University Press **1988**.

**Keywords: modulated structure; molecular compound; X-ray diffraction**

#### FA4-MS01-P06

**DRAWxtl 5.4 An Open-Source Program to Produce Crystal Structure Drawings.** Martin Kroeker<sup>a</sup>, Brian H. Toby<sup>b</sup>, Larry W. Finger<sup>c</sup>. <sup>a</sup>*Department of Inorganic Chemistry, University of Freiburg, Germany.* <sup>b</sup>*Advanced Light Source, ANL, Argonne, USA.* <sup>c</sup>*Retired from Carnegie Institution, Washington D.C, USA.*

E-mail: [martin@ruby.chemie.uni-freiburg.de](mailto:martin@ruby.chemie.uni-freiburg.de)

DRAWxtl is a versatile crystal structure drawing program that supports all the conventional drawing modes such as bonds, atomic spheres, thermal ellipsoids and coordination polyhedra of arbitrary complexity. Although it is mainly designed for the rendering of inorganic crystal structures, with import filters for CIF, CSD, Fullprof, GSAS and Shelx format, it is equally well suited for displaying results of LAPW calculations done with the popular VASP, WIEN2k and ELK/Exciting programs.

A significant new feature highlighted in our most recent publication[1] is its support for 3+1D to 3+3D modulated and composite crystal structures, including an interactive “walk through” the local structures that correspond to varying  $t$  values.

The upcoming 5.4 release, which should be available by the time of the conference, adds support for calculating and rendering cavity volumes and solvent-accessible surface areas and for displaying atomic basins as they result from

AIM calculations done with WIEN2k.

The program is freely available under the GPLv2 license from <http://www.lwfinger.net/drawxtl/> where we provide source code as well as pre-compiled versions for Linux, OS X and Windows.

[1] Finger L.W., Toby B.H., Kroeker, M., *J.Appl.Cryst.*, **2007**, 40, 188.

**Keywords: computer programs; computer graphics; linux crystallographic computing**

#### FA4-MS01-P07

**RASTGUI - A Free Software Package for Reciprocal Space Mapping.** Carsten Paulmann<sup>a</sup>, Thomas Malcherek<sup>a</sup>. <sup>a</sup>*Mineralogisch-Petrographisches Institut, Universität Hamburg, Hamburg, Germany.*  
E-mail: [carsten.paulmann@desy.de](mailto:carsten.paulmann@desy.de)

Besides powerful X-ray sources and modern 2D-detectors on the experimental side, processing and visualisation of diffraction phenomena from disordered crystals require advanced software features covering non-integer indexed positions in reciprocal space (modulated structures, twinning) as well as a complete pixel-wise data processing to remap the irregular spaced experimental data to a regular grid. A range of command-line driven software tools for reciprocal space remapping were developed which include full four-circle support (Eulerian, Kappa) as well as advanced pixel-wise corrections (polarisation, scaling, background, phosphor incidence). The programs also provide internal binning options, definition of arbitrary planes of interest and most recently, masking options of obscured detector areas. Special versions are tailored to laboratory setups (Bruker-Nonius/Smart, Bruker-Nonius/KappaCCD) with fixed geometry whereas other versions aim for typical synchrotron hard- and software setups like MarCCD/XDS and also recently Mar345/XDS. With respect to the number of different experimental setups at synchrotron sources the diffractometer geometry is fully customisable. Additional tools provide enhanced numeric data processing features with special emphasis on synchrotron specific details (eg. primary beam scaling). However, the increasing number of different detectors, diffraction geometries as well as specific setup files and data processing control input files demanded the development of a common graphical user interface to guide less experienced users through the complete range of different data processing features and special advanced options. The interface was developed using the Qt4-library. Among the commonly supported hardware options, the users are able to define their specific experimental setup as well as program and data directories. Depending on the setup, the user interface provides special input masks for each software tool, generates specific input files and starts the basic command-line driven programs as background processes. Experienced users may still use the command-line versions to ease up the use of script files and cross-networking. Most recently, an OpenGL-based reciprocal space viewer was added which also features advanced options for a semi-automatic definition of the sample