

PS19.00.05 STRUCTURE OF AMMONIUM HYDROGEN SUCCINATE AT LOW TEMPERATURES. S. Kashino, T. Yoshida, Y. Kubozono, T. Urakawa, Y. Yoshida, H. Ishida, H. Maeda, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan

Crystals of ammonium hydrogen succinate undergo the second order phase transition around 170 K, and the space group is $P\bar{1}$ with $Z=2$ at 293 K (Hausühl & Schreuer, 1993). We report the structural change accompanying the phase transition based on the structures determined at temperatures from 297 to 20 K. The space group $P\bar{1}$ is retained, and hydrogen atoms involved in two O—H...O hydrogen bonds between hydrogen succinate ions are disordered at all temperatures. Temperature dependency of the structure is remarkable in the O...O lengths of these hydrogen bonds. These lengths decrease with a decrease in temperature from room temperature to the phase-transition temperature, but increase after the phase transition. It is worthy of note that peak resolution of the difference Fourier maps in regions of these hydrogen bonds becomes lower at 80 and 20 K than at 150 K, suggesting some fluctuations in hydrogen-atom positions.

Intensity data were measured on a Hüber off-center four-circle diffractometer in 2θ range of 3-78° by using Mo K α X radiation. Final R values were 0.033 for 2720 reflections at 80 K, and 0.034 for 2819 reflections at 20 K.

Hausühl, S. G. & Schreuer, J. (1993). *Z. Kristallogr.* **206**, 255-265.

PS19.00.06 HIGH-TEMPERATURE RIETVELD REFINEMENTS: SYSTEMATIC ERRORS OF MEASUREMENT. A. Kern and W. Eysel, Mineralogisches Institut der Universität, Im Neuenheimer Feld 236, D-69120 Heidelberg, Germany

High temperature powder diagrams are influenced by systematic errors of measurement, which may strongly affect high-temperature Rietveld refinements.

The most important ones of these errors were investigated using a BraggBrentano diffractometer (D 500, Siemens) with a heating equipment (HTK 2.3, Bühler) up to 1500°C:

- Beam Overflow
- Temperature gradient in the flat sample
- Texture from sample preparation
- (Multi-) Texture from nucleation effects during polymorphic transitions
- Reflections from the material of the sample carrier.

These errors and how to take them into account and minimize them, were studied for selected materials: α -Al₂O₃, Quartz, MgO, Li₂SO₄, Ag₂SO₄.

Among others the refinement of a two phase sample (Li₂SO₄+Pt) will be presented, in which both phases exhibit multitexture. The refinement was carried out by using simultaneously the Rietveld method for Li₂SO₄ and the Whole Powder Pattern Fit method for Pt.

PS19.00.07 STRUCTURAL DETERMINATION OF THE LOW TEMPERATURE PHASES OF GLOBULAR ORGANIC MOLECULES BY POWDER X-RAY DIFFRACTION A.J.MORA^{1,2} and A.N.FITCH³ Department of Chemistry, Keele University, Keele, Staffordshire, ST5 2BG, UK ²Departamento de Química, Facultad de Ciencias, Universidad de los Andes, Mérida, 5101, Venezuela ³European Synchrotron Radiation Facility, BP220, 38043 Grenoble Cedex, France.

Globular molecules are often orientationally disordered at room temperature and exist in the so-called "plastic state". It is only at low temperatures (or high pressures) that they order. These ordered phases highlight the subtle interplay between steric and van-der-Waals interactions, neither of which are strong because of the globular nature of the molecules. Although carefully studied by calorimetric and spectroscopic techniques, the arrangement of the molecules at low temperature is often unknown, since crystals tend to fragment during the phase transitions. High resolution powder diffraction provides an alternative to the more traditional single-crystal approach. In particular, we have used this technique to study the ordered crystal structures of bicyclic organic molecules using the synchrotron source at Daresbury Laboratory, UK. The table shows the cell parameters, space group and transition temperature to ordered phases for some of the molecules we have investigated. We present in more detail the structural solution and Rietveld refinements of 3-azabicyclo[3.2.2.]nonane, bicyclo[3.3.1.]nonan-9-one and the racemic solid solution of R- and S-camphor.

Compound	<i>a</i> [Å]	<i>b</i> [Å]	<i>c</i> [Å]	β [°]	Space Group	Transition Temperature [K]
3-azabicyclo[3.2.2.]nonane	21.131	11.235	6.086	-	Aba2	297.8
bicyclo[3.3.1.]nonan-9-one	10.409	11.418	6.496	90.99	P2 ₁ /n	300.5
norbornylene	8.695	8.570	7.577	97.49	P2 ₁ /a	129
S-camphor	26.957	8.915	7.364	-	P2 ₁ 2 ₁ 2 ₁	240
R,S-camphor	6.832	11.656	11.496	89.98	Cc	205
thiocamphor	9.122	14.709	7.366	-	P2 ₁ 2 ₁ 2 ₁	-

PS19.00.08 SINGLE CRYSTAL X-RAY DIFFRACTION AT 9K IN AUSTRALIA. Alexander N. Sobolev and Brian N. Figgis, Department of Chemistry, University of Western Australia, Nedlands, Perth 6907, WA, Australia

A four-circle Huber 512 goniometer with a two-stage Displex DE202 cryocooler (APD, Cryogenics Inc.), following the Larsen design¹ has been constructed. This is the first such X-ray diffractometer in the southern hemisphere. It is controlled by locally-written Fortran software. The results of a study of a single crystal of Rb[Ru(NH₃)₆]. 2ClO₄ 2Cl are presented. I-MoK α , β -filter, 9.4(3)K, $w/2\theta$ data collection, $2\theta_{\max}$ 100°, scan speed 2.4°/min, whole sphere, 17500 reflections. The results obtained are compared with 293K and 92K structural data on same compound. A simple form of disorder is present at low temperature with atomic separations only 0.4Å, less than the resolution of the experiment, but which can be clearly modelled because of the large amount and high accuracy of the data. An analysis of the data in terms of the charge density distribution around the atoms is in progress.

1. Larsen, F.K. (1995). *Acta Cryst.*, B51, 468-482.