

diffraction studies of structural evolution, but also the study of hydrides rather than deuterides can often mitigate synthesis issues (a major issue for chemically complex materials). Breakthroughs in tackling these issues with neutron powder diffraction on modern instrumentation can have a profound effect on the study of such materials.

Keywords: neutron powder diffraction, hydrogen, molecular complexes

### MS.53.4

*Acta Cryst.* (2008). A64, C95

#### Powder diffraction and DFT optimization in structural characterization of macrocyclic compounds

Vladimir V. Chernyshev<sup>1</sup>, Nadezhda M. Logacheva<sup>2</sup>, Alexander O. Terent'ev<sup>3</sup>, Aslan Y. Tsivadze<sup>2</sup>, Leonid A. Aslanov<sup>1</sup>

<sup>1</sup>Moscow State University, Chemistry, Leninskie Gory, 1, Moscow, Moscow, 119991, Russia, <sup>2</sup>A.N. Frumkin Institute of Physical Chemistry and Electrochemistry, Leninsky prospect 31, 119991 Moscow GSP-1, Russia, <sup>3</sup>A.N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 ul. Vavilova, 119991 Moscow, Russia, E-mail: vladimir@struct.chem.msu.ru

A series of novel 1,2,4,5,7,8-hexaoxonanes [1], crown-containing compounds Cu(L1)<sub>2</sub> and LiNCS.HL1 (HL1 = 4'-[2-(tosylamino)benzylideneamino]-2,3-b15c5; b15c5 = benzo-15-crown-5) [2], and NaNCS.L2, [M(L2)<sub>2</sub>]<sup>2+</sup>.2PF<sub>6</sub><sup>-</sup>, [Co(L2)<sub>2</sub>]<sup>2+</sup>.2NCS<sup>-</sup>, [M(NaNCS.L2)<sub>2</sub>]<sup>2+</sup>.2PF<sub>6</sub><sup>-</sup>, L3 [3], [M(L3)<sub>2</sub>]<sup>2+</sup>.2PF<sub>6</sub><sup>-</sup> (L2 = terpyridine-O-CH<sub>2</sub>-b15c5, L3 = terpyridine-O-b15c5, M = Co, Ni, Cu, Zn) were structurally characterized by powder diffraction methods. For most of these compounds, it was rather difficult to obtain not only single-crystals but even crystalline powders suitable for structure determination, that required either repeat crystallizations or repeat full synthesis. Powder patterns for two compounds - Cu(L1)<sub>2</sub> and LiNCS.HL1, respectively - were obtained using synchrotron radiation, and the rest powder patterns were obtained using the laboratory diffractometers. The fourteen new crystal structures of the aforementioned compounds were determined from powder diffraction data, which provide a reliable validation of the molecular structure for each compound. Due to the large molecular dimensions, the structure determination process did not take too much time, in contrast to the subsequent Rietveld refinement because of a large number of variables and unknown (in advance) conformations of flexible macrocycles. To overcome these obstacles in each case, a number of molecular models with various conformations of macrocycles was subjected to DFT optimization. The optimized molecular geometries were used as templates in the bond-restrained Rietveld refinement allowing a choice of the correct conformations of macrocycles.

[1] Terent'ev, A.O. *et al.* (2007). *J. Org. Chem.* **72**, 7237-7243.

[2] Dorokhov, A.V. *et al.* (2007). *Acta Cryst.* **B63**, 402-410.

[3] Logacheva, N.M. *et al.* (2008). *Acta Cryst.* **E64**, o225.

Keywords: structure from powder diffraction, rietveld refinement, DFT

### MS.53.5

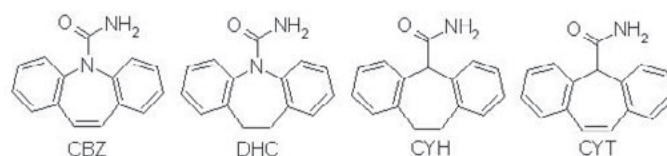
*Acta Cryst.* (2008). A64, C95

#### SDPD: A key component in populating the carbamazepine crystal structure landscape

Kenneth Shankland<sup>1</sup>, Alastair J Florence<sup>2</sup>, Norman Shankland<sup>2</sup>

<sup>1</sup>STFC Rutherford Appleton Laboratory, ISIS Facility, Chilton, Didcot, Oxon, OX11 0QX, UK, <sup>2</sup>Solid-State Research Group, Strathclyde Institute of Pharmacy and Biomedical Sciences, University of Strathclyde, Glasgow, U.K., E-mail: k.shankland@rl.ac.uk

The anticonvulsant agent carbamazepine (CBZ) has received a lot of attention from both those interested in polymorphism and those interested in crystal structure prediction (CSP). Our own interest in CBZ spans both these disciplines; in particular, we are intrigued by the fact that predicted structures for CBZ point to the existence of a favourable C(4) catemer that has yet to be observed experimentally. Given that the C(4) catemer is observed amongst analogues of CBZ, the focus of our work has broadened to include these closely-related compounds, specifically dihydrocarbamazepine (DHC), cyheptamide (CYH) and cytenamide (CYT). This presentation will illustrate the relationships between these four compounds and, in particular, will highlight the role that SDPD has played in obtaining the crystal structures of various polymorphs. Many of the crystal structures have presented considerable challenges to SDPD ( $Z'=4$ ,  $V \sim 2400 \text{ \AA}^3$ , 72 non-H atoms, 28 DOF) both in the solution and refinement aspects. Evidence for the existence of CBZ in a C(4) catemeric configuration in a CBZ:DHC 1:1 solid solution will also be presented.



Keywords: *ab-initio* structure determination, powder diffraction, pharmaceutical compounds

### MS.54.1

*Acta Cryst.* (2008). A64, C95

#### Martensitic transformations in the Ni-based ferromagnetic shape memory alloys

Ryosuke Kainuma<sup>1</sup>, Katsunari Oikawa<sup>2</sup>, Kiyohito Ishida<sup>2</sup>

<sup>1</sup>Tohoku University, IMRAM, 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi, 980-8577, Japan, <sup>2</sup>Tohoku University, Dept. Mater. Sci., Aoba-yama, Sendai, Miyagi, 980-8579, Japan, E-mail: kainuma@tagen.tohoku.ac.jp

Since Ullakko *et al.* reported a large magnetic field-induced strain (MFIS) in the Ni<sub>2</sub>MnGa single-crystalline alloy in 1996, ferromagnetic shape memory alloys (FSMAs) have received much attention as high performance actuator materials. The Ni<sub>2</sub>MnGa ferromagnetic Heusler-type alloy showing a martensitic transformation has first been found by Webster and Ziebeck in 1984, and several Ni-based FSMAs besides the Ni<sub>2</sub>MnGa have recently been reported by the present authors in Ni-Mn-Al, Ni-Co-Al, Ni-Co-Ga, Ni-Fe-Ga, Ni-Mn-In and Ni-Mn-Sn based alloy systems. In the present talk, basic features on the martensitic transformations and the magnetic properties in those new FSMAs except the Ni<sub>2</sub>MnGa alloy will be reviewed.

Keywords: ferromagnetic alloys, martensitic transformation, shape-memory alloys