

**MS11.03.04 ENERGY DISPERSIVE DIFFUSE SCATTERING STUDIES OF ADAMANTANE (C<sub>10</sub>H<sub>16</sub>) AND DIAMOND.** John S. Reid, School of Physics, The University, Aberdeen, Scotland. Moreton Moore & Grzegorz Kowalski<sup>†</sup>, Department of Physics, Royal Holloway, University of London, Egham, Surrey UK. <sup>†</sup>on leave from the Institute of Experimental Physics, University of Warsaw, Poland

We have used the technique of energy dispersive diffuse scattering (Reid, 1993) to explore rotational disorder scattering in adamantane and structural disorder scattering from platelets in natural diamond.

Adamantane is a plastic molecular crystal which shows lobes of diffuse scattering along  $\langle 111 \rangle$  directions at room temperature, due to constrained rotational motion of the highly symmetric C<sub>10</sub>H<sub>16</sub> molecules. We set out to measure the absolute diffuse scattering in the lobes and the structure of that scattering, in order to provide a much needed comparison with the rotational predictions of molecular dynamics models. Single crystals of adamantane were grown by sublimation. They were left to anneal for over 25 years. Results of diffuse scattering measurements are presented over a sector of the (110) plane around [111] and in out-of-plane sections through the lobe. A comparison is made with results measured over the same sector of the (110) plane at an indicated temperature of 201 K.

The energy dispersive technique has also been applied to measuring the diffuse scattering seen as short spikes extending out in [100] directions from certain reflections in type Ia natural diamonds (Moore et al 1993). The sensitivity of the energy dispersive technique is such that these diffuse spikes can be traced out to the zone boundary.

Moore, Moreton et al. (1993), *Diamond Rel. Mat.*, 2, 115 - 120  
Reid, John S. (1993), *Acta Cryst. A* 49, 190 - 198

**MS.11.03.05 ORDER-DISORDER PHENOMENA IN CYCLODEXTRIN INCLUSION COMPOUNDS.** G.Le Bas, CEP, Univ. Paris-Sud, 92290 ChatenayMalabry, S.A.Mason, ILL, BP 156X, 38042 Grenoble Cedex 9, and J.Doucet, LURE, Univ. Paris-Sud, 91405 Orsay Cedex. FRANCE

X-ray diffuse scattering from the hexagonal crystals of  $\alpha$ -cyclodextrin cyclopentanone hydrate has been investigated. The study was carried out on several single crystals, using high intensity X-rays from the DCI storage ring at LURE.

Scattering features on the X-ray images revealed a variety of disorder phenomena, some of which could not have been predicted from the averaged structure determination. Two main results will be discussed:

- 1) The occurrence of a superstructure has allowed us to confirm some hypotheses concerning the correlated disorder phenomena involving host-guest and solvent molecules with alternative hydrogen bonded networks shown by the neutron diffraction structure at 20K; the correlations are limited to small domains.
- 2) Thin layers of intense diffuse scattering perpendicular to the c\* direction have been shown to be related to the host framework.

These features have revealed different twinning arrangements, which will be explained.

**MS11.03.06 STRUCTURE AND DYNAMICS OF t-BUTYLCALIX[4]ARINE INCLUSION COMPOUNDS.** G.D. Enright, E.B. Brouwer, E.J. Gabe, C.I. Ratcliffe and J.A. Ripmeester, Steacie Institute for Molecular Sciences, National Research Council of Canada, Ottawa K1A 0R6, Canada

We have carried out an extensive study of the structure and dynamics of several *t*-butylcalix[4]arine inclusion compounds with toluene, nitrobenzene, benzene, pyridine, cyclohexane, pentane, chlorobutane, xenon and methylcyclopentanone as guests. Overlap of the partially occupied symmetry-related guest sites is common in these compounds and often leads to difficulties in fully resolving the guest. By employing solid state NMR (a technique that is particularly sensitive to dynamics and microscopic structure) together with single crystal X-ray diffraction a greatly improved understanding of these systems has been obtained. We have clearly resolved a guest-induced distortion of the host lattice that is present in almost all of these compounds. The prevalence of a similar distortion, which can be described as a slight bending outward of the calix phenyl unit accompanied by a fixed rotation of the *t*-butyl group, with such a wide variety of guest molecules suggests that the interaction between the guest and host is dominated by steric effects. No contact distances significantly less than that determined from Van der Waals radii are observed. A comparison of the structure of the benzene and pyridine compounds reveals that the in-plane guest rotation observed in benzene is absent in pyridine despite the similar positions occupied by the guests. Although it has been suggested that weak N...H-C hydrogen bonding may stabilize the pyridine guest in such compounds, we believe that steric effects can explain these observations as well.

**MS11.03.07 COMPARISON OF 158 K THERMAL PARAMETERS FOR (NH<sub>4</sub>)H<sub>2</sub>PO<sub>4</sub> OBTAINED FROM NEUTRON AND FROM HIGH-ORDER CONVENTIONAL AND SYNCHROTRON X-RAY DATA.** M. Souhassou, N. Peres, C.Lecomte, H. Graafsma, and E. Espinosa, Université de Nancy, LCM3B, URA CNRS 809, BP 239, BP 239, 54506 Vandoeuvre-lès-Nancy, France

Ammonium dihydrogen phosphate undergoes a phase transition from a high-temperature paraelectric to a low-temperature antiferroelectric phase at 148 K. In order to do experimental electron density studies, we have measured three diffraction data sets at 10 K above the transition temperature:

- (1) neutron data at LLB Saclay,
- (2) conventional Mo K- $\alpha$  X-ray data in Nancy, and
- (3) short-wavelength synchrotron X-ray data at ESRF in Grenoble.

The neutron data, which were measured in order to accurately determine the positions and mean-square displacements of the hydrogen atoms, suffer from anisotropic extinction. The synchrotron X-ray measurements at a wavelength  $\lambda = 0.2 \text{ \AA}$  yielded data to the very high resolution  $\sin(\theta)/\lambda = 1.7 \text{ reciprocal \AA}$ . Results of refinements that fitted positional and thermal parameters to the neutron data and to the high-order synchrotron data, and multipolar electron density parameters to the conventional X-ray data, will be presented.