Experimental Report

Proposal: **5-12-308** **Council:** 4/2014

Title: Guest-Induced Distortions in Urea Inclusion Compounds

This proposal is a new proposal

Researh Area: Chemistry

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Samples: Hexadecane/Urea
1,6-dibromohexane/urea

Instrument	Req. Days	All. Days	From	To
D19	10	10	15/09/2014	24/09/2014

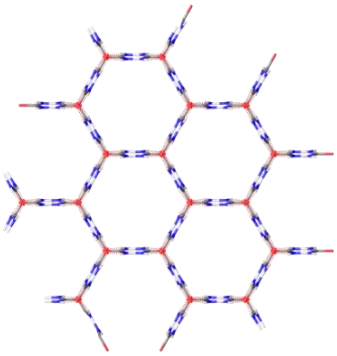
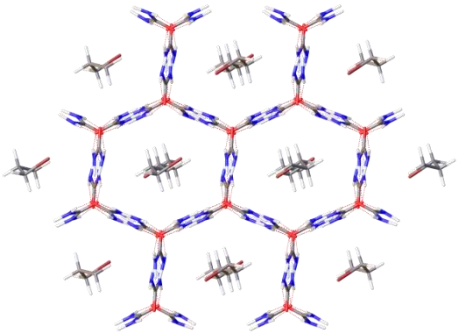
Abstract:

This project aims to study distortions from hexagonal symmetry in urea channel inclusion compounds induced by interactions with the guest species. These interactions between guest and channel wall are responsible for fascinating properties such as ferroelasticity. We have obtained single crystal neutron data on a hydrogen-bonded ferroelastic inclusion compound of 2,7-octanedione that is significantly distorted. We now wish to structurally characterise the urea hydrogen bonded network in two further compounds, namely 1,6-dibromohexane and hexadecane. The 1,6-dibromohexane inclusion compounds is also distorted from hexagonal symmetry but there are no apparent hydrogen bonds from the urea to the guest. The hexadecane sample is an undistorted control compound with a regular urea hydrogen bonded network. Combined with our existing (unpublished) data on the dione compound, these two samples will allow us to build up a picture of interactions between guests and host channel in these inclusion compounds and hence understand the fascinating properties that arise.

Experimental

Successful neutron diffraction experiments on two examples of a urea inclusion compound were carried out on instrument D19. Urea hexadecane diffraction data were collected at 150 and 260 K, as peak splitting owing to a phase transition occurs below 150 K. Data for urea 1,6-dibromohexane were collected at temperatures 30, 120 and 260 K. The high quality data, face based absorption correction, and vanadium can corrections allowed solution and refinement of both structures, allowing detailed comparison of the finer details of hydrogen bonding. Data integration was achieved using Shelx and final refinement was carried out in Olex2.

Table 1 Cell parameters and results of refinement

	Urea hexadecane (host structure only shown) 	Urea 1,6-dibromohexane 
Crystal dimensions	1.0 x 1.0 x 0.7 mm	2.3 x 1.9 x 1.6 mm
Unit cell	$P6_522$, $a = b$ = 8.1529(5)Å, c = 10.9819(6)Å, α = β = 90°, γ = 120°	a = 8.5518(2)Å, b = 10.8605(2)Å, c = 13.3296(3), α = γ = 120°, β = 92.92°
Temperature	150 K, (260 K)	30 K, (120 K, 260 K)
Refs(total) / Refs(Fo > 4sig(Fo))	2857/672	12505/12134
Data/parameters	717/53	3729/308
R(int)	0.0400	0.0353
R1(Fo > 4sig(Fo))	0.0682	0.0324
wR2	0.1882	0.0657