s9.m3.p17 Chiral Recognition And Crystal Structures Of Dca Inclusion Compound With Fenchone.

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Inclusion compounds have been extensively studied for their properties of storing guest compounds and separating isomers, tautomers or enantiomers. Deoxycholic acid (DCA) has been overlooked as a potential host in chiral recognition of guest compounds by most of these studies. DCA forms a channel-type inclusion complex with its guest. Bilayers of hydrogen-bonded DCA molecules separate hydrophobic channels which attract mostly organic substances as guests[1].

DCA co-crystallised with excess of R- or S-fenchone in alcoholic solution to form 2:1 inclusion compounds of DCA_2:R-fenchone (DCRFCH) or DCA_2:S-fenchone (DCSFCH). DCRFCH undergoes a phase change as the temperature is decreased from room temperature. This phase change involves the doubling of the *a*-axis and is of the order-disorder type. [T=200K, $P22_12_1$, a=7.086(1), b=13.810(1), c=27.251(3) Å; T=100K, $P2_12_12_1$, a=14.007(1), b=13.824(1), c=27.040(2) Å]. The phase change is reversible and occurs over a wide temperature range from 230K to 170K, as shown by single-crystal X-ray diffraction (SXRD) experiments. This is also observed by solid-state ^{13}C CP/MAS NMR. In the spectra the peaks due to C18, C19 and C21 atoms of the DCA molecule start to broaden from single peaks at 240K to doubles at lower temperature. These peaks suggest that in the asymmetric unit, there are one DCA molecule in the high-temperature form and two DCA molecules in the low-temperature form.

At ambient temperature the guest S-fenchone is ordered in the DCSFCH complex [T=295K, $P2_12_12_1$, a=14.103(1), b=14.004(1), c=27.159(1) Å]. As the temperature is increased, DCSFCH undergoes a phase change to a high-temperature form where the *a*-axis is halved. [T=355K, $P22_12_1$, a=7.218(1), b=14.016(1), c=27.231(1) Å]. However, the phase change occurs over a smaller temperature range compared to DCRFCH, from 350-353K in the SXRD experiments. The phase change is also reversible. In the ^{13}C CP/MAS NMR spectra, the phase transition is clearly observed from the peaks due to C18, C19 and C21 atoms of the DCA molecules. Reversible doubling of the peaks was seen from 346-348K. Due to the different phases adopted by the two inclusion compounds at ambient temperature, chiral recognition could be possible by recrystallisation of a racemate of the guests with DCA and will be investigated.

[1] Giglio, E., Inclusion Compounds, Vol. 2, Atwood, J.L., Davies, J.E.D., MacNicol, D.D., Eds., Academic Press: London, (1984), 207-229

s9.m3.p18 Supramolecular Assemblies Involving Benzenepolycarboxylic Acids.

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Keywords: benzenepolycarboxylates, crystal engineering, supramolecular.

Benzene polycarboxylic acids make good building blocks in supramolecular assemblies due to their rigid planar cores and relatively predictable hydrogen bond patterns. Micrporus materials produced from these compounds may have application in separation science, asymmetric synthesis and catalysis, with the exact function predominantly dependent on the detailed nature of the structure.

Our recent work has produced several new solvate co-crystals with common solvent molecules, a number of new alkylammonium, alkali and alkaline earth metal salts, and some microporus coordination polymers with late first-row transition metals. The most interesting new results from these three areas will be presented.

Structural analysis reveals a great variety of hydrogen-bonding motifs in the solvate co-crystals other than the common bis-carboxylic acid head to tail motif; a variety of stoichiometries in the group I and II metal salts; and novel 2D and 3D networks for the coordination polymers.

Diffraction data were collected using Bruker SMART 1K CCD diffactometers at low temperature both in Newcastle and using synchrotron radiation at Daresbury Laboratory Station 9.8 for small molecule crystallography.