Acta Cryst. (2002). A58 (Supplement), C310

SUPRAMOLECULAR SELF-ASSEMBLY THROUGH DIFFERENT π-ARYL INTERACTIONS IN TWO ORGANOTELLURIUM COMPOUNDS

J. Zukerman-Schpector1 I. Caracelli1 I. Haiduc2
1Universidade Federal De Sao Carlos Quimica Dept. KM 235 Rodovia Washington Luis Caixa Postal 676, Sao Carlos SAO PAULO 13565-905 BRAZIL 2Facultatea de Chimie, Universitatea Babes-Bolyai, RO-3400 Cluj-Napoca, Romania

Te... centroid = 3.757 Å, resulting in the self-assembly of a chain like with one of the peripheric phenyl rings of a neighbouring molecule occupying the fifth equatorial position. Acknowledgements FAPESP, CNPq, CAPES.

Keywords: SELF ASSEMBLY, PI-ARYL INTERACTION, ORGANOTELLURIUM

Acta Cryst. (2002). A58 (Supplement), C310

PHASE TRANSFORMATIONS IN T-BUTYLCALIX[4]ARENE INCLUSION COMPOUNDS AT ELEVATED TEMPERATURES

National Research Council Steacie Institute for Molecular Sciences 100 Sussex OTTAWA ONTARIO K1A 0R6 CANADA

Thermal gravimetric analysis (TGA) studies have shown that upon heating a number of 1:1 t-butylicalx[4]arene (C4) inclusion compounds lose guest molecules and gradually convert to a stable 2:1 form. Remarkably this phase transformation occurs without loss of crystallinity. Single crystals (0.35 mm x 0.35 mm x 0.1 mm) of 1:1 C4-toluene (1) were mounted on goniometer heads and heated at 2° C/min (as in TGA studies). Those crystals that were removed at 150° C were completely transformed to single crystals of a 2:1 C4-toluene phase (2) (despite changes in crystal symmetry and unit cell volume). Those removed between 120° and 150° C were hybrid crystals. Diffraction studies revealed the simultaneous presence of domains of both 1 and 2, but only a single orientation of each phase. The 2:1 and hybrid crystals converted back to single crystals of 1 when heated overnight at 70° C in the presence of toluene vapor. Crystals heated beyond 150° C lost all guest molecules and were converted to a polycrystalline phase of neat, self-included C4 in (1), a Te ... Br [3.253(1) Å] secondary bond links the supramolecular arrangement. The chains are packed by van der Waals forces and show an interdigitated layering. The adjacent chains are extended into a three dimensional supramolecular array by a Br π-C6H5 inter-chain interaction (3.680 Å). Due to the secondary interactions, which includes intramolecular π-C6H5 interaction, the polyhedron around the tellurium can be best described as a distorted pseudo-pentagonal bipyramidal, with two halides in axial positions and the centroid of a phenyl ring in (1) and a bromine in (2) occupying the fifth equatorial position. Acknowledgements FAPESP, CNPq, CAPES.

Keywords: INCLUSION COMPOUNDS, PHASE TRANSITIONS, HYBRID CRYSTALS

Acta Cryst. (2002). A58 (Supplement), C310

INCLUSION OF PARABEN MOLECULES IN HEPTAKIS(2,3,6-TRIO-METHYL)-β-CYCLODEXTRIN

E. J. C. de Vries M. R. Caïra L. R. Nassimbeni
Department of Chemistry University of Cape Town Rondebosch CAPE TOWN 7701 SOUTH AFRICA

The aim of this study was to investigate the inclusion of methyl-, ethyl-, propyl- and butyl paraben in TRIMEB [heptakis(2,3,6-tri-O-methyl)β-cycloextrin] in the solid state. Powdered inclusion complexes were prepared by kneading. Thermal analysis and X-ray powder diffraction (XRPD) were used to confirm that complexation had occurred. Infrared spectroscopy was used to determine how the carbonyl stretching frequency was affected by complexation. X-ray diffraction from single crystals was used to determine structural and geometrical features. The thermal analytical results and XRPD patterns of the kneaded materials were different from those of physical mixtures, indicating complexation. The C=O stretching frequency of the complexed drug showed an increase frequency relative to the uncomplexed drug, which also indicated that the guest is included in the host. Preliminary X-ray photography revealed Laue mmm symmetry, corresponding to the orthorhombic system [space group P212121]. Accurate cell data are listed below (in Å), with Z=4 complex units with 1:1 host-guest ratio in each case. Methyl paraben complex: a=10.718(1), b=26.353(1), c=30.018(2) Å; Ethyl paraben complex: a=14.866(2), b=22.024(3), c=27.602(2) Å; Propyl paraben complex: a=14.863(1), b=21.862(2), c=27.627(2) Å; Butyl paraben complex: a=14.866(2), b=21.967(2), c=27.635(4) Å. Physicochemical methods of analysis show that methyl-, ethyl-, propyl- and butyl paraben form well defined inclusion complexes with TRIMEB. All guests, except ethyl paraben, are oriented with their hydroxyl groups at the host secondary rim.

Keywords: CYCLODEXTRIN, INCLUSION COMPLEX, PARABEN

Acta Cryst. (2002). A58 (Supplement), C310

THE INFLUENCE OF INTERMOLECULAR INTERACTIONS ON THE SYN/ANTI FORM OF 2,4,6-TRIHYDROXYBENZOIC ACID - CRYSTALLOGRAPHIC STUDIES.

W. Jankowski1 A. Kedzierski M. Gdaniec T. Polonka2
1Adam Mickiewicz University Faculty of Chemistry UL, Grunwaldzka 6, POZNAN 60-780 POLAND 2Department of Chemistry, Technical University, 80-952 Gdansk, Poland

2,6-Dihydroxybenzoic acid (DHB) is known to form two polymorphs in which two different intramolecular hydrogen-bond schemes are observed [1]. 2,4,6-Trihydroxybenzoic acid (THBA), similarly to DHB, has also two possibilities for intramolecular hydrogen-bond formation, one with two phenolic OH groups and second with one phenolic and one carboxylic OH group involved as donors in intramolecular hydrogen-bond formation. To examine the influence of intermolecular interactions on the preferred form of the THBA molecule the crystalline complexes of THBA with weak heteroaromatic bases like pyrazine, quinoxaline and phenazine have been prepared and their crystal structure determined by X-ray analyses. Pyrazine and quinoxaline form with THBA 1:1 complexes where THBA carboxylic group adopts syn conformation. THBA molecule and the hydrogen-bonded to the carboxylic group aromatic base are nearly coplanar. While no stacking interactions are observed in the pyrazine complex, quinoxaline and THBA are arranged into infinite stacks where base and acid molecules alternate. When THBA is co-crystallized with the largest base, phenazine, the 1:2 complex is formed and the THBA molecule adopts anti form. The phenolic OH groups at position 2 and 4 act as donors in the hydrogen bonds to phenazine N atoms and THBA molecules fasten together the phenazine molecules arranged into infinite stacks. Contrary to THBA, the 1:1 complex is formed by DHBa with phenazine and the acid molecule is in the syn form.


Keywords: MOLECULAR COMPLEXES IMTERMOLECULAR INTERACTIONS CRYSTAL STRUCTURE