

**[s9.m3.p35]** Chiral recognition and crystal structures of DCA inclusion compound with  $\alpha$ -methylbenzylamine. M.I. Mohamed Tahir and K. Prout, *Chemical Crystallography Laboratory, 9 Parks Road, Oxford OX1 3PD, UK*, S.J. Heyes, *Inorganic Chemistry Laboratory, South Parks Road, Oxford OX1 3QR, UK*.  
Keywords: molecular interactions, supramolecular assemblies.

Deoxycholic acid (DCA) generally forms channel type inclusion compounds with organic molecules<sup>1</sup>. Hydrogen-bonded DCA molecules arrange themselves in a head to tail fashion to form bilayers. These bilayers then pack themselves antiparallel to each other to create channels which run normal to them. Shifts between these bilayers create cavities of varying size and shape of the cavities hence accommodating different types of guests. Previous studies have shown that no guests residing in these channels have intermolecular contacts with the host.

The recrystallisation of DCA with R- and S- $\alpha$ -methylbenzylamine in methanol produced a 1:1 inclusion compound with each of the enantiomers, DCRMBA and DCSMBA respectively. Both inclusion compounds crystallised out with monoclinic lattices and the structures at 100K were solved in the space group  $P2_1$  with two molecules in the asymmetric unit [DCRMBA,  $a=11.027(1)$ ,  $b=7.620(1)$ ,  $c=17.941(1)$  Å,  $\beta=101.92$  (1); DCSMBA,  $a=11.534(4)$ ,  $b=7.793(3)$ ,  $c=16.004(15)$ ,  $\beta=102.76(5)$ ]. However, both structures differ significantly in the arrangement of the host and guest in the lattice. These is our second example of DCA complexes where two enantiomers of the same chiral molecule produce different packing arrangement (previously with enantiomers of camphorquinone).

The DCA molecules in both complexes form helical chains through hydrogen bonding of the hydroxyl and the carboxyl group which run parallel to the  $b$ -axis. The DCA helical stacks are then connected to each other through hydrogen bonding of the hydroxyl and the carboxyl group of the DCA with the amine group of the  $\alpha$ -methylbenzylamine, forming sheets. The two complexes differ in the arrangement of the sheets relative to each other. In DCRMBA the sheets are 'eclipsed' to each other and runs parallel to the  $bc$  plane whereas for DCSMBA the sheets are 'staggered' to each other and run parallel to the plane  $\bar{1}01$ . No phase changes have been observed at high or low temperature from single-crystal X-ray diffraction and <sup>13</sup>C cross-polarisation magic angle spinning solid state nuclear magnetic resonance experiments.

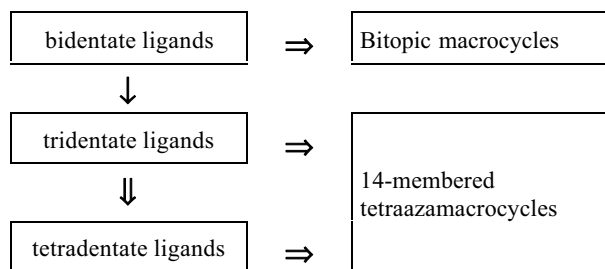
This is the first example of such 1:1 DCA inclusion compounds with this type of arrangement. No structures have been reported where the DCA molecule interact with the guest through hydrogen bonding. We expect due to the differences in structure of DCRMBA and DCSMBA chiral resolution is possible for the racemic amine and will be investigated.

**[s9.m3.p36]** From Coordination Compounds to Supramolecular systems. The Structure of Ni(II) Complexes Based on S-alkylisothiosemicarbazide and Related Macrocyces Yu.A. Simonov, V.Ch. Kravtsov, P.N. Bourosh, J. Lipkowski, N.V. Gerbeleu, J.I. Gradinaru, V.B. Arion, M.D. Revenco. *Institute of Applied Physics and Institute of Chemistry of the Academy of Sciences of Moldova, MD-2028 Chishinau, R. Moldova*, *Institute of Physical Chemistry, Polish Academy of Sciences, Kasprzaka 44/52, 01-224 Warsaw, Poland*.  
Keywords: X-ray structures, macrocycles complexes.

Template-controlled syntheses on the metal matrix provide the most elegant and effective route for building macrocyclic systems and their open chain precursors. 14, 22 and 25 membered rings including one ( $3d$ ) or two ( $\beta d$  and  $s$  or  $f$ ) metal centres have been obtained on the base of S-alkylisothiosemicarbazide<sup>1</sup>. Their composition strongly depends on the nature of metal centers and used building blocks, counter anions type and synthesis conditions.

The template condensation [1+2], [2+1] and [2+2] of S-alkylisothiosemicarbazide and  $\beta$ -dicarbonyl building blocks (salicylaldehyde, 2,4-pentanedione, 1-phenylbutane-1,3-dione, 1,1,3,3-tetraethoxypropane, sodium salt of nitromalonic dialdehyde etc.) lead to a variety of coordination compounds. All of them are diamagnetic products with square-planar surrounding of Ni(II). The structural aspects of *cis-trans*-azomacrocycles assembling with  $MN_4$  coordination core and their open-chain analogues with  $MN_2O_2$  and  $MN_3O$  type of cores as well as bitopic macrocycles containing two different binding centers have been studied by X-ray crystallography. In the last case the 22 and 25 atoms ring systems incorporate 'soft'  $N_2O_2$  cavity with  $3d$  metals and a 'hard' polyether cavity metal empty or bearing  $Ba^{2+}$  or  $K^+$  cations.

The general strategy of transition from coordination compounds to macrocyclic supramolecular systems:



**Ni(II) complexes based on S-alkylisothiosemicarbazide**

**Macrocyclic system**

⇒ - template-controlled route

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[1] Gerbeleu N.V., Arion V.B., Burgess J. "Template Synthesis of Macrocyclic Compounds" WILEY-VCH (1999), Weinheim, 565p.