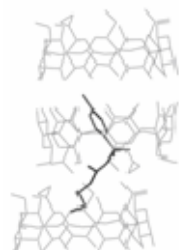


The asymmetric unit contains two  $\beta$ CD molecules and two guest molecules statistically disordered over 2 positions with s.o.f. of 0.5. The stacking of the two host molecules along the *c*-axis generates dimers held together by seven H-bonds via the secondary hydroxyl groups. The geometries of the two guest molecules and their interactions are very similar. The 2,4-dichlorophenyl moiety of the guest is inside the  $\beta$ CD cavity, whereas the 5-chlorophenol expands in the space between the primary sides of two host molecules. The dihedral angle between the two phenyl rings is  $81^\circ$ . Tolbutamide, 1-butyl-3-(*p*-tolylsulfonyl)urea (TBM), is an oral hypoglycemic agent insoluble in water. Complexation of TBM with  $\beta$ CD improves its dissolution rate and solubility, which is believed to be the rate-determining step for its absorption from the gastrointestinal tract.



The inclusion complex of  $\beta$ CD/TBM was crystallized and data were collected at room temperature (monoclinic, *C*2 with  $a = 19.225 \text{ \AA}$ ,  $b = 24.547 \text{ \AA}$ ,  $c = 15.708 \text{ \AA}$ ,  $\beta = 109.652^\circ$ ). The structure has been refined up to  $R_f = 0.1054$ . Two  $\beta$ CD molecules, related by the 2-fold axis and held together by seven H-bonds via the secondary hydroxyl groups, form also dimers, in the cavity of which a TBM molecule is encapsulated. The aromatic ring of the guest is located in the secondary side of the CD and the chain sticks out between the primary sides of two hosts. The N-atoms of the urea group H-bond to hydroxylic groups of the latter pointing inside. The 2-fold axis of the lattice generates a  $\beta$ CD dimer. However, one guest molecule occupies the dimer cavity in a statistical way (the host: guest ratio is 2:1). The dimers align head-to-head along the *c*-axis forming infinite channels. Channel formation is strengthened by H-bonds between primary hydroxyl groups existing in a variety of orientations.

#### MS18 P07

**Synthesis, characterisation and X-ray study of cobalt (II) complex: [Dimalonato tetraaqua cobalt (II)]**  $[\text{Co}(\text{C}_4\text{H}_3\text{O}_4)_2(\text{H}_2\text{O})_4]$  Jawher Abdelhak, Sawssen Namouchi Cherni, Mohamed Faouzi Zid & Ahmed Driss *Laboratoire de Matériaux et Cristallographie, Département de chimie, Faculté des Sciences, 2092 El Manar, Tunis, Tunisia.* E-Mail: [jawherabd@hotmail.com](mailto:jawherabd@hotmail.com)

**Keywords:** cobalt compounds, characterization method, magnetic crystal structures.

The investigations of novel complexes represent an intense interest of chemical research; this is driven to a large extent by their interesting properties and their potential practical applications [1-3]. In addition, mixed ligand-metal complexes of some organic oxygen-donors ligands continue to attract attention because they play an important role in biological systems [4].

The title compound was synthesized by slow evaporation of solution at room temperature it has been characterized by X-ray diffraction, IR spectral analysis and UV-vis spectroscopy. The natural elements are detected by the EDX on a scanning electron microscope. The thermal analysis shows that the dehydration of the compound occurs in agreement with the structural features. The temperature dependence of the molar magnetic susceptibility,  $\chi_M$ , was measured on a polycrystalline sample in the temperature range 5-300 K using a SQUID magnetometer.

The structure was determined by X-ray diffraction on single crystal. This salt crystallizes in the triclinic space group *P*-1 with  $a = 5.241(1) \text{ \AA}$ ,  $b = 7.362(1) \text{ \AA}$ ,  $c = 9.268(1) \text{ \AA}$ ,  $\alpha = 109.15(2)^\circ$ ,  $\beta = 104.39(3)^\circ$ ,  $\gamma = 93.15(1)^\circ$ ,  $V = 323.6(3) \text{ \AA}^3$  and  $Z = 2$ . Crystal structure has been determined and refined to  $R = 0.021$  and  $wR = 0.057$ .

The framework structure of  $[\text{Co}(\text{C}_4\text{H}_3\text{O}_4)_2(\text{H}_2\text{O})_4]$  is consisted by  $\text{Co}^{\text{II}}$  ions laying in inversion centers and are six fold coordinated by oxygen atoms from two malonate anions and four water molecules to form a slightly distorted octahedron. The H atoms form hydrogen bonds to neighboring O ensure the cohesion and stability of the molecular packing.

[1] Boudalis A.K., Tangoulis V., Raptopoulou C.P., Terzis A., Tuchagues J.-P. and Perlepes S.P., *J. Inorg.Chim. Acta*, 357 (2004) 1345-1354.

[2] Ganesan R., Viswantho B., *J. of Molecular Catalysis A: Chemical* 181 (2002) 99-107.

[3] Rojo J.M., Mesa J.L., Lezanna L., Fernandez J.R. and Pizarro J.L., *Int. J. Organ. Mater.* 3 (2001) 67.

[4] Divjak B., Franko M. and Novič M., *J. of Chromatography A*, 829 (1998) 167-174.

#### MS18 P08

**Solvent induced transformations in a supramolecular organic zeolite** Michela Brunelli<sup>a</sup>, Consiglia Tedesco<sup>b</sup>, Loredana Erra<sup>b</sup>, Ivano Immediata<sup>b</sup>, Valeria Cipolletti<sup>b</sup>, Carmine Gaeta<sup>b</sup>, Placido Neri<sup>b</sup>, Andy Fitch<sup>a</sup>, *ESRF, BP 200 - 38043 Grenoble, France*, <sup>b</sup>*Department of Chemistry, University of Salerno, Italy.*

E-mail: [brunelli@esrf.fr](mailto:brunelli@esrf.fr), [ctedesco@unisa.it](mailto:ctedesco@unisa.it)

**Keywords:** calixarenes; supramolecular assemblies; synchrotron powder diffraction

At the Dept. of Chemistry of the University of Salerno we prepared and characterized a new crystalline solid based on *p*-Bu<sup>t</sup>-calix[4]dihydroquinone **1** revealing the simultaneous existence of water channels and very large hydrophobic cavities ( $988 \text{ \AA}^3$ ). The compound crystallizes from  $\text{CHCl}_3$  and ethylacetate with a cubic structure ( $a = 36.412(4) \text{ \AA}$ ) containing 48 calixarene molecules and 155 water molecules in the unit cell. The host supramolecular framework is surprisingly robust and that the material behaves as an organic supramolecular zeolite featuring the simultaneous presence of networked

channels, filled with easily removable water, and empty isolated hydrophobic cavities [1].

By crystallization of **1** from  $\text{CHCl}_3$  and water X-ray quality single crystals have been obtained at the interphase. The structure analysis displays a new triclinic structure ( $a = 11.259(3) \text{ \AA}$ ,  $b = 17.330(4) \text{ \AA}$ ,  $c = 9.993(3) \text{ \AA}$ ,  $\alpha = 92.73(2)^\circ$ ,  $\beta = 104.74(2)^\circ$ ,  $\gamma = 85.03(2)^\circ$ ) with 2 calixarene molecules, 2 water molecules and 2 chloroform molecules in the unit cell.

A powder mixture of cubic and triclinic phase has been characterized by High Resolution X-Ray Powder Diffraction (HRXRPD) at beam line ID31 (ESRF) before and after 3 days exposure to  $\text{CCl}_4$  showing that the triclinic phase disappears and the sample can be indexed as a single cubic phase ( $a = 36.66847(15) \text{ \AA}$ ). The increase in the lattice parameter with respect to the previously characterized cubic phase can be accounted for considering that possibly the  $\text{CCl}_4$  molecules could enter the channels and substitute the water molecules.

These results seem to highlight the assembly possibilities of **1** and these can be triggered by appropriate solvent choice.

[1] Tedesco C., Immediata I., Gregoli L., Vitagliano L., Immirzi A., Neri P., *CrystEngComm* 2005, 7, 449.

#### MS18 P09

**Chemical Induction of carbonyl Substitution Catalysed by Electron Transfer in tri Iron Complex  $(\mu\text{-S})_2\text{Fe}_3(\text{CO})_9$** , Mousser H.<sup>1</sup>, Mousser A.<sup>2</sup>, Darchen A.<sup>3</sup> and Mousset G.<sup>4</sup> <sup>1</sup>Département de Chimie Industrielle, Université Mentouri Constantine, Algérie, [hmbouzidi@yahoo.com](mailto:hmbouzidi@yahoo.com); <sup>2</sup>Département de Chimie, Université Mentouri Constantine, Algérie; <sup>3</sup>Laboratoire d'Electrochimie, Ecole Nationale Supérieure de Chimie, Rennes, France; <sup>4</sup>Laboratoire d'Electrochimie Organique, Université Blaise Pascal, Aubière, France.

**Key words: Induction, Substitution, Complex, X-ray analysis**

The reactions of carbonyl substitutions, by other ligands L, were the subject of many studies devoted to various organometallic substrates [1]. According to the nature of the substrate or ligand, these reactions can be led under various activation modes (thermal, photochemical, chemical, catalytic...) [2]. These catalytic reactions illustrate the great reactivity of the paramagnetic species "cluster anion" in the exchange ligand reactions [3, 4]. We show on the trinuclear iron complex  $(\mu\text{-S})_2\text{Fe}_3(\text{CO})_9$ , that not reducer chemical reagents can induce a catalysis by electron transfer.

The studied complex  $(\mu\text{-S})_2\text{Fe}_3(\text{CO})_9$  is easily reducible and very active in carbonyl replacement reactions which are catalysed by electron transfer. Spectrum EPR of the cluster, recorded in the presence of nucleophilic, shows a single signal, of increasing intensity, with  $g = 2.0178$  ascribable to the cluster anion  $(\mu\text{-S})_2\text{Fe}_3(\text{CO})_9^-$ . In the presence of an excess of ligand L ( $L = \text{P}(\text{OMe})_3$ ,  $\text{PPh}_3$ ,  $2,6\text{-Me}_2\text{PhCN}$ ) and nucleophilic (triethylamine), this cluster reacts quickly and leads quantitatively to the mono substituted complex. The spectroscopic information is insufficient to specify the regioselectivity and the stereochemistry of the exchange reaction. The X-ray structure of the mono substituted  $(\mu\text{-S})_2\text{Fe}_3(\text{CO})_8[\text{P}(\text{OMe})_3]$  complex was solved on single crystal by direct methods in P21/m space group and refined by least squares methods to  $R = 0.010$ . The ligand  $\text{P}(\text{OMe})_3$  is on the sulphured iron atom in axial position in trans with the iron-iron bond. The angle iron-iron-phosphorus is of  $144.2(4)$ .

[1] M. Chanon, *Acc. Chem. Res.*, 20 (1987) 214.

[2] J. M. Savean, *Acc. Chem. Res.*, 13 (1980).

[3] M. Chanon et M. L. Tobe, *Angew. Chem. Int. Edit. Engl.*, 21 (1982) 1.

[4] D. Astruc, *Comptes Rendus de l'Académie des Sciences, Série IIc : Chimie* 3 (2000), 189.