

In I-III and secondary glyoxylamides in the CSD the amide resonance makes the average α C=O longer than the β C=O, but by <0.01 Å, suggesting fairly similar abilities as hydrogen bond acceptors. From a secondary indol-2-ylglyoxylamide structure Black et al. [1] inferred that a set of 3 hydrogen-bonded rings, S(5) from amide H to β -O, R₂²(10) involving the same atoms intermolecularly, and S(6) from indole NH to α -O could be a robust supramolecular synthon. In a series of indol-3-yl analogues we find that the S(5) persists, and the S(6) replaces the indole NH with the adjacent CH as donor but remains fairly strong. However, C(4) chains linking amide H to α -O occur as often as the R₂²(10) motif.

[1] Black, D.St.C., Craig, D.C., McConnell, D.B., *J. Am. Chem. Soc.*, 1996, 118, 8148.

MS19 P06

Reaction of 1,3,5-triazacyclohexane with copper : formation of other complex Saida Latreche^{a*}, Ahcène Bouchemma^b, Abdelhamid Mousser^a, Randolph D.Köhn^c, Gabriel K Köhn^c. ^a*Département de Chimie, Centre Universitaire Larbi Tebessi Tebessa, Algérie.* ^b*Laboratoire de Chimie Appliquée et Technologie des Matériaux, Centre Universitaire Larbi Ben M'hidi, Oum El Bouaghi.* ^c*Department of Chemistry, University of Bath, Bath, BA2 7AY, UK.* E-mail:latrechesouad@yahoo.fr

Keywords: Triazacyclohexane , copper complexes , X-ray.

Copper complexes with three facially coordinating nitrogen atoms play an important role in the biological binding and activation of dioxygen^[1]. They are used, for example, as bioinorganic model systems, reagents with high metal ion selectivity or as olefin polymerisation catalysts.

Many complexes containing triazacyclohexane and larger macrocyclic amines are known^[2,3].

The title complex 1,3,5-(4-methylphenyl)-1,3,5-triazacyclohexane **1** is prepared by reaction of formaldehyde with primary amine (4-methyl aniline)^[4]. The complexation of 1,3,5-(4-methylphenyl)-1,3,5-triazacyclohexane **1** give a bimetallic complex in which the ligand is fragmented (Fig 1).

As the coordination number of these copper complexes is two short bonds between (1.879 and 1.881Å).

The Para magnetism of this complex made it difficult to get useful H-or C-NMR. The ligand and complex were characterized by X-ray crystallography.

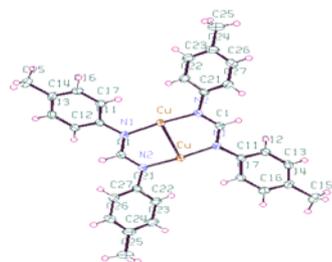


Figure 1: X-ray structure of (p-tol)NCHNH(p-tol)

[1] R.D.Kohn, G.Seifert, G.K.Kohn. *Chem. Ber.*1996, 129, 1327-1333.

[2] P.Chaudhuri, K. Wieghard, *Prog. Inorg. Chem.* 1987, 35, 329.

[3] R.D.Kohn, P.Kampe, G.K.Kohn. *Eur. J. Inorg.Chem.* 2005, 3217-3223.

[4] A. Bouchemma, P.H.McCabe, G.A. Sim. *Acta Cryst.* 1990. C46, 674-676.

MS19 P07

The Structural Class Concept, an Extension Ake Oskarsson, *Department of Chemistry, Lund University, P.O. Box 124, SE-221 00 Lund, Sweden.* E-mail: ake.oskarsson@inorg.lu.se

Keywords: structural-class, symmetry , packing

Novacki [1] was the first to study space group frequencies at a time when the space groups were only known for about 3000 crystal structures. Since then a number of studies has appeared. An important improvement in this field was introduced by Belsky et al. [2,3] with the structural class concept, which beside the space group also includes the Z value and the distribution of molecules over equivalent positions. Implicit in this notation is also unoccupied special positions, which is of importance in discussing crystal packing, since every unoccupied position is a waste of space [4]. Therefore it is suggested that these positions should be added to the structural class notation in the following way: a semicolon shows the end of the structural class and then the unoccupied special positions are given within brackets. No unoccupied special positions are denoted ;[0]. An obvious notation would be P2₁/c,Z=2(-1);[(-1³)] and P2₁/c,Z=4(1);[(-1⁴)] etc. This notation will thus explicit give an overview of all Wyckoff positions, occupied or not, and will be helpfull in addressing questions like *i*) which molecular symmetries are retained and avoided in the solid state and *ii*) which Wyckoff positions are unoccupied. A few examples regarding complexes *cis/trans*-PtX₂L₂ and *cis/trans*-PtX₄L₂, where X=halogen and L=neutral ligand, will be discussed.

[1] Nowacki, W. *Helv. Chim. Acta* 1942, 25, 863.

[2] Zorkii, P.M., Razumaeva, A.E., Belsky, V.K. *Acta Cryst.* 1977, A33, 1001.

[3] Belsky, V.K., Zorkaya, O.N., Zorky, P.M. *Acta Cryst.* 1995, A51, 473.

[4] Brock, C.P., Dunitz, J.D. *Chem. Mater.* 1994, 6, 1118.

MS19 P08

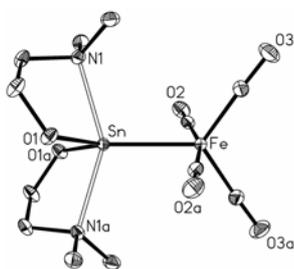
Unusual tin(II)-iron complexes (RO)₂Sn-Fe(CO)₄ and {[Li]((RO)₃Sn-Fe(CO)₄)}₂ (R = CH₂CH₂NMe₂)

Victor N. Khrustalev, *A.N.Nesmeyanov Institute of Organoelement Compounds RAS, Moscow, Russia* E-mail: vkx@xray.ineos.ac.ru

Keywords: tin(II), transition-metal complexes, X-ray diffraction analysis

Two new tin(II)-iron complexes (RO)₂Sn-Fe(CO)₄ (**3**) and {[Li]((RO)₃Sn-Fe(CO)₄)}₂ (**4**) (R = CH₂CH₂NMe₂) have been structurally characterized. Unusual types of heteronuclear bonding between heavier Group 14 elements and transition metals have been revealed. The iron atoms both in **3** and **4** have a trigonal-bipyramidal coordination typical for FeL₅-fragments. The "stannylene" ligand in **3** occupies the equatorial position, whereas the stannate ligand in **4** is axially disposed. The structures of **3** and **4** indicate that the monomeric tin(II) compound (RO)₂Sn (**1**) [1] is a better π -acceptor than a σ -donor toward iron [2] and thus displays the stannylene-like properties, while the

dimeric stannate $\{[Li]((RO)_3Sn)\}_2$ (**2**) [3] behaves as a σ -donor with the very weak π -acceptor ability. Data obtained show that compounds **1** and **2** are of value as nucleophiles with respect to transition metals.



3

This work was financially supported by the Russian Foundation for Basic Research (Project No. 07-03-01018).

[1] N.N.Zemlyansky, I.V.Borisova, M.G.Kuznetsova, V.N.Khrustalev, Yu.A.Ustynyuk, M.S.Nechaev, V.V.Lunin, J.Barrau, G.Rima, *Organometallics*, 2003, **22**, 1675.

[2] A.R.Rossi, R.Hoffmann, *Inorg. Chem.*, 1975, **14**, 365.

[3] V.N.Khrustalev, M.Yu.Antipin, N.N.Zemlyansky, I.V.Borisova, Yu.A.Ustynyuk, V.V.Lunin, J.Barrau, G.Rima, *J. Organomet. Chem.*, 2004, **689**, 478.

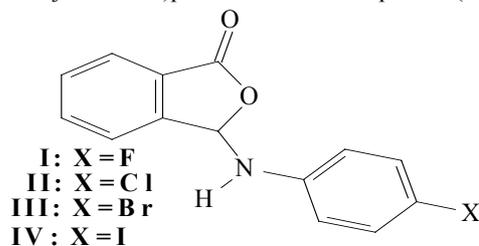
MS19 P09

3-(4-haloenoanilino)phthalides Orhan Büyükgüngör^a and Mustafa Odabaşoğlu^b, ^aDepartment of Physics, Ondokuz Mayıs University, TR-55139, Samsun, Turkey, ^bDepartment of Chemistry, Ondokuz Mayıs University, TR-55139, Samsun, Turkey. E-mail: orhanb@omu.edu.tr

Keywords: phthalide, isobenzofuranon, benzolacton

Phthalides (isobenzofuranones) are five-membered lactones found in plants and are known to show diverse biological activities as hormones, pheromones and antibiotics. These compounds possess several important properties, such as fungicidal, ctericidal and herbicidal, analgesic and hypotensive and vasorelaxant activities. In addition, phthalide derivatives are useful in the treatment of circulatory and heart-related diseases. They are also found to be associated with pesticidal activities. Considering the potential interest of such phthalide-3-phosphonates in synthetic organic chemistry, and as agrochemical and pharmaceutical agents, we decided to investigate the solid-state structures of 3-substituted phthalides by X-ray diffraction methods and present here the

3-(4-haloenoanilino)phthalides of the compounds (I-IV).



Crystals of $C_{14}H_{10}FNO_2$, (I) show paired N-H...O hydrogen bonds that link the molecules into $R_2^2(12)$ dimmers; the hydrogen-bonded dimmers are linked into chains by an aromatic π - π stacking interaction [1]. The structure of $C_{14}H_{10}ClNO_2$, (II), is stabilized by N-H...O, C-H... π and π - π interactions [2]. Crystals of $C_{14}H_{10}BrNO_2$,

(III), contain N-H...O hydrogen bond interactions that generate C(6) chains; these chains are linked by C-H...O hydrogen bonds, generating an $R_4^3(21)$ ring motif [3]. The structure of $C_{14}H_{10}INO_2$, (IV), whose asymmetric unit contains three independent molecules, are stabilized by three N-H...O, three C-H...O and one N-H...I intermolecular hydrogen bonds, the N-H...O and C-H...O hydrogen bonds generating an edge-fused $R_2^2(11)$ ring motif which appears as a molecular ladder in the structure [4]. The phthalide parts of all four molecules are planar and the dihedral angles between the phthalide group and the benzene ring are 74.10 (9)°, 75.58 (15)°, 62.2 (2)° and mean 80.5 (4)° for I, II, III and IV, respectively.

[1] Odabaşoğlu M. & Büyükgüngör O., *Acta Cryst.*, 2006, E62, o4138–o4139.

[2] Büyükgüngör O. & Odabaşoğlu M., *Acta Cryst.*, 2006, E62, o2003–o2004.

[3] Odabaşoğlu M. & Büyükgüngör O., *Acta Cryst.*, 2006, E62, o4366–o4367.

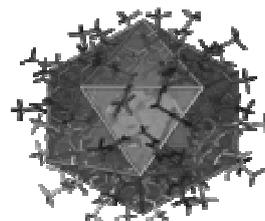
[4] Büyükgüngör O. & Odabaşoğlu M., *Acta Cryst.*, 2007, E63, o25–o27.

MS19 P10

Large supramolecular assemblies of p-sulfonatocalix[4]arene Clive L. Oliver, Leonard J. Barbour, *Department of Chemistry, University of Stellenbosch, South Africa* E-mail: oli@sun.ac.za

Keywords: supramolecular, self-assembly, architecture

The supramolecular enclosure of chemical space is ubiquitous in biological systems and serves as inspiration for mimicry of their architecture with synthetic systems. In this regard, the self-assembly of *p*-sulfonatocalix[4]arene, which usually occurs in an "up-down" (bi-layer) fashion, have been induced to occur in an "up-up" arrangement on the addition of lanthanide cations [1,2]. This results in two unique structures with either an icosahedral or a cuboctahedral arrangement of calixarene anions, the former mimicking the geometry of the cowpea chloritic mottle virus.



Here we report the discovery of a further two structures [1 and 2] with spherical arrangements of *p*-sulfonatocalix[4]arene anions that are not consistent with the geometry of Platonic or Archimedean solids. Crystal data for **1** are $a = 51.248(4)$, $b = 53.333(4)$, $c = 28.223(2)$ Å, space group $P2_12_12$ whilst that for **2** are $a = b = 59.262(1)$, $c = 49.342(1)$ Å, space group $I-4$. The arrangements of the calixarene anions are unique in both structures with **2** consisting of an unprecedented two different spherical arrangements.

[1] Orr, W.G., Barbour, L.J., Atwood, J.L., *Science*, 1999, 285, 1049.

[2] Atwood, J.L., Barbour, L.J., Dalgarno, S.J., Hardie, M.J., Raston, C.L., Webb, H.R., *J. Am. Chem. Soc.* 2004, 126, 13170.