

P.07.04.9*Acta Cryst.* (2005). A61, C307**Crystal Structure of [Sn(Bu)₃(O=PPh₃)₂][BPh₄]**

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The crystal structure of the title complex was determined by single crystal X-ray analysis. Data were collected on a Nonius KappaCCD diffractometer equipped with a fine focus molybdenum X-ray source, 0.3 mm *ifg* capillary collimator, and an Oxford Cryosystems crystal cooler. Structure solution and refinement utilized SIR97, MaXus, and the SHELXTL system.

The cation consists of trigonal bipyramidal tin(IV), with three butyl groups coordinated in the three equatorial positions and triphenylphosphine oxide ligands in the axial positions. The supramolecular structure is dominated by extensive concerted phenyl-phenyl interactions [1] among the six phenyl groups of the cation triphenylphosphine moieties and the four phenyl groups of the anion tetraphenylborate moieties.

Crystal data: C₇₂H₇₇BO₂P₂Sn; *M_r* = 1165.78 Daltons; transparent colorless flat slabs; 0.17 x 0.20 x 0.23 mm; orthorhombic; *Pna*2₁; *a* = 19.2421(3), *b* = 14.9077(4), *c* = 21.9461(5) Å, *V* = 6295.4 Å³; *Z* = 4; *D_{calc}* = 1.230 Mg/m³; λ_{MoKα} = 0.71073 Å; μ = 5.0 cm⁻¹; *T* = 200 K. Data collection: Bruker-Nonius KappaCCD, 0.3 mm *ifg* capillary collimator, 60,132 data collected, *R_{int}* = 0.0775, 8209 unique data, 6431 observed data (*I_o* > 4σ(*I_o*)).

[1] Dance I., Scudder M., *J. Chem. Soc., Chem. Commun.*, 1995, 1039.

Keywords: noncovalent bonding, crystal structure, tin compound

P.07.04.10*Acta Cryst.* (2005). A61, C307**Supramolecular Decoration of Metal-Organic Hosts From Products of Template-Directed Synthesis**

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Metal-mediated self-assembly is the process whereby organic subunits come together in solution by way of coordination bonding to metal centers to form well-defined infinite (*i.e.* coordination polymers, metal-organic frameworks) or discrete (*i.e.* metal-organic polygons, polyhedra) aggregates. Metal-organic polygons and polyhedra have found utility in the last year in the fields of host-guest chemistry and materials science. Congruous design of the organic subunit is imperative in metal-mediated self-assembly, as the subunit must contain appropriate functionalities to recognize the metal in such a way that the desired aggregate will form. Most self-assembling systems studied to date have been based on symmetrical ligands, those with multiple copies of identical binding functionalities. We have used template-directed synthesis in the solid state to produce unsymmetrical tetrapyrrolyl cyclobutane molecules in quantitative yield and in gram quantities. When these polyfunctional molecules self-assemble with metals, they give rise to metal-organic polyhedra [1]. In addition, the outer surfaces of these polyhedra can be decorated in a supramolecular fashion with various organic groups, opening doors to applications in anion transport and biomedical chemistry. That template-directed synthesis is used to produce molecules otherwise difficult or impossible to obtain, and that these molecules, in turn, assemble with metals to produce interesting and useful metal-organic assemblies augurs well for the future study of the crystalline solid state as a reaction medium.

[1] Hamilton T. D., Papaefstathiou G. S., MacGillivray L. R., *J. Am. Chem. Soc.*, 2002, 124, 11606.

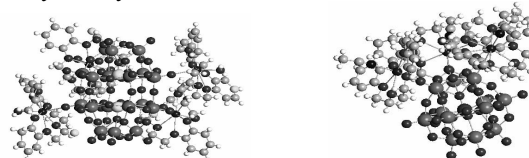
Keywords: metal-organic complexes, solid-state photochemistry, self-assembly supramolecular chemistry

P.07.04.11*Acta Cryst.* (2005). A61, C307**DB18C6 Sodium Polyoxometalate Supermolecular Complexes With α-Dawson and α-Keggin Structure**

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Since 1996, Lu et al have reported a series of supermolecular complexes containing polyoxometalates and crown ethers.¹ In this paper, continuing our work, we describe two novel crown ether sodium heteropolyoxometalate supermolecular complexes obtained by solvothermal reaction and characterized by X-ray, IR, UV-vis, multinuclear NMR and gUMBC NMR.

As shown in the Figures, [Na(DB18C8)(H₂O)]₄[X₂M₁₈O₆₂] (X=S, M=Mo,W) (**1**) consists of four dibenzo-18-crown-6 (DB18C6) sodium complex cations and one α-Dawson heteropolyoxometalate, while [Na(DB18C8)(CH₃CN)]₃[XM₁₂O₄₀] (X=As and P, M=Mo and W) (**2**) consists of three DB18C6 sodium units and an α-Keggin heteropolyanion. Each sodium ion is located in the cavity of DB18C6, and the four [Na(DB18C8)]⁺ ions in **1** are linked to four terminal O atoms from two "belt" layers of the Dawson-type polyanion, while the three complex cations in **2** are bound to terminal O atoms from one M₃O₁₃ unit of the α-Keggin structure. The effects dominating the self-assembly of the two types of supermolecules are charge compensation and higher symmetry.



[Na(DB18C8)(H₂O)]₄[S₂Mo₁₈O₆₂] [Na(DB18C8)(CH₃CN)]₃[PMo₁₂O₄₀]

[1] Lu X.M., Zhong R.F., Liu S.C., Liu Y., *Polyhedron*, 1997, 16, 3865.

Keywords: crown ether, heteropolyoxometalates, self-assembly

P.07.04.12*Acta Cryst.* (2005). A61, C307**1-D and 2-D Uranium(VI) Coordination Polymers**

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For this study we chose the reactions of uranyl nitrate with oxalic acid and 2,5-pyridine dicarboxylic acid. The resultant products were a 1-D polymer sesquihydrate {[UO₂(C₂O₄)(CH₃OH)]1.5H₂O}_n (**1**) and a 2-D polymer sheet {[UO₂(C₇H₃NO₄)(CH₃OH)]0.5CH₂Cl₂}_n (**2**) respectively. Both structures are based on the seven coordinate uranium(VI) ion centres.

The polymer chain in compound **1** is propagated by the oxalate groups acting as bridges between the adjacent metal centres. The packing of polymer strands forms a 3-dimensional interpenetrated network of channels running parallel to the three axes and which are filled with water molecules.

The polymeric compound **2** is based on the uranium(VI) ion centres coordinated to three 2,5-pyridine dicarboxylates, two oxo groups and one methanol molecule. The structure consists of stacked layers of polymer sheets separated by dichloromethane guest molecules.

Both **1** and **2** are very stable in air. We have dried samples of these complexes that have been standing in the air for over six months with no loss of integrity (checked and confirmed by X-ray powder diffraction). Complex **2** is also very stable in mother liquor while crystals of **1**, if left in mother liquor, undergo a transformation over a period of 3-4 weeks. There is a replacement of coordinated MeOH with water and an inclusion of an extra half water molecule.

Keywords: uranyl complexes, oxalate, 2,5-pyridine dicarboxylate