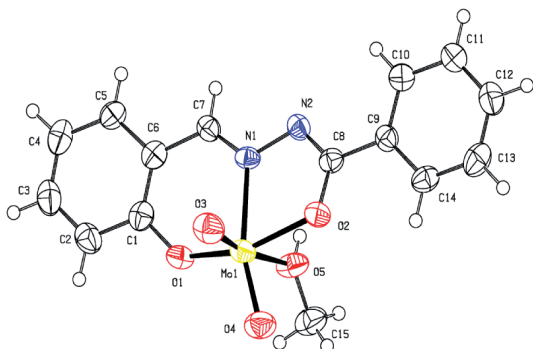


MS61.P18

Acta Cryst. (2011) A67, C611**X-ray structure of methanol {E-N/(2-hydroxybenzylidene)benzohydrazido}dioxidomolybdenum(VI)**

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In the structure of the title compound, [Mo(C₁₄H₁₀N₂O₄)(CH₃OH)], the Mo^{VI} ion is octahedrally coordinated by two oxido atoms, the N atom and two deprotonated OH groups of the tridentate Schiff base ligand(E)-N/(2-hydroxybenzylidene)benzohydrazid and by a methanol O atom. Its crystal data: C₁₅H₁₄MoN₂O₅ Triclinic, P, a = 7.8478 (13) Å, b = 9.7989 (16) Å, c = 10.3766 (17) Å, c = 10.3766 (17) Å, α = 94.315 (13)°, β = 107.834 (13)°, γ = 90.565 (13)°, Z = 2, μ = 0.89 mm⁻¹ T = 233 (2) K, 0.40 × 0.15 × 0.15 mm.



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Keywords: Dioxidomolybdenum(VI) complex

MS61.P19

Acta Cryst. (2011) A67, C611**Polymeric manganese(II) complex with isophthalate ion and 2,2'-dipyridylamine**

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In the past decade the design and synthesis of metal-organic coordination polymers with anions of isophthalic (1,3-benzenedicarboxylic) acid, ipht, have become a growing field in crystal engineering due to their structural diversity and potential application as functional materials [1]. We have been continually interested in synthesis and characterisation of ternary transition metal complexes containing polycarboxylate anions and some aromatic N-containing ligands [2,3]. As a continuation of our research, the polymeric complex, [Mn(dipya)(ipht)]_n, where dipya is 2,2'-dipyridylamine, represents a novel example.

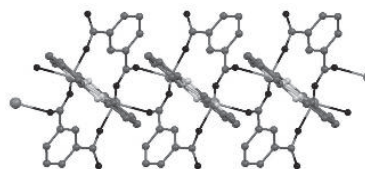
In [Mn(dipya)(ipht)]_n, ipht anion bridges three Mn atoms with bidentate-bridging and monodentate COO groups. In this way

centrosymmetric double chains extending along *c*-axis are formed (Figure). Mn(II) ions are in a deformed square pyramidal environment consisting of two N atoms from chelating dipya ligand and three O atoms from three different ipht ligands. The shortest intrachain Mn–Mn distance of only 3.76 Å could be the cause of possible strong magnetic interactions. Two crystallographically different Mn atoms are linked by two bridging ipht ligands to construct eight-membered [Mn₂O₄C₂] rings. Similar rings are already found in the ipht structures where at least one bidentate-bridging COO exist [3].

Double chains are stacked by face to face π-π interactions at centroid-centroid distances of 3.67 and 3.79 Å. Uncoordinated O atoms from monodentate COO groups and the amine H atoms of dipya build hydrogen bonds, which connect adjacent chains. Therefore through π-π interactions and hydrogen bonds the chains are packed into a three-dimensional framework.

The compound was hydrothermally synthesized in a Teflon-lined steel autoclave (T = 433 K, 5 days) starting from an aqueous solution containing Mn(NO₃)₂, dipya and sodium isophthalate. The structure was refined using single-crystal X-ray diffraction data (Oxford diffractometer, CCD detector, θ_{max} = 25.7°, 6711 measured reflections, R_{int} = 0.018, T = 293 K).

Crystal data: C₁₈H₁₃MnN₃O₄, M_r = 390.25, monoclinic, space group C2/c, a = 14.8320(6), b = 21.9325(6), c = 11.9995(5) Å, β = 122.916(6)°, V = 3276.8(2) Å³, Z = 8, F(000) = 1592, ρ_x = 1.582 g cm⁻³, μ(Mo Kα) = 0.836 mm⁻¹. The refinement on F² (287 parameters) yielded R₁ = 0.037, wR₂ = 0.067, S = 0.97 for all data, and R₁ = 0.027 for 2478 observed reflections with I ≥ 2σ(I).



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Keywords: complex, carboxylate ligand, crystal structure

MS61.P20

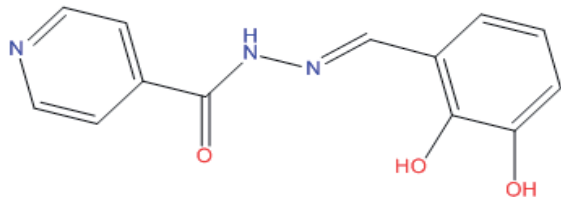
Acta Cryst. (2011) A67, C611–C612**Isonicotinic acid [1-(2,3 hydroxy phenyl) methylidene] hydrazide**

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In the title compound, C₁₃H₁₁N₃O₃, crystallizes with two molecules in the asymmetric unit which differ significantly in the conformation of hydrogen bonds, dihedral and torsion angle. The compound crystallizes in the monoclinic spacegroup P 2₁/c with a=7.7781(2)Å, b=30.0719(8)Å, c=10.5116(3)Å, α=90°, β=101.551(2)°, γ=90° and Z=8. The crystal structure is stabilized by intermolecular hydrogen bonds.

Schiff bases are typically formed by the condensation of a primary amine and an aldehyde. Also, schiff bases are a functional group that contains a carbon-nitrogen (C=N) double bond (an imine group). These

bases play an important role in inorganic chemistry as they easily form stable complexes with most transition metal ions. The development of the field of bioinorganic chemistry has increased the interest in Schiff base complexes, since it has been recognized that many of these complexes may serve as models for biologically important species [1], [2].



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Keywords: single-crystal X-ray study, isonicotinic acid

MS61.P21

Acta Cryst. (2011) **A67**, C612

Preparation and Evaluation of Anti-inflammatory Potential of Some Sesquiterpene lactones: X-ray Structure of Psilostachyin

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The natural sesquiterpene lactones (SLs) have displayed a diverse range of bioactivities from antiinflammatory to antibacterial, ameobicidal to anticancer, therefore, they constitute interesting targets for further investigations in the area of drug development. The spiro lactone psilostachyin (**1**) and acetylated pseudoguaianolides (**2**, **3**) are the SLs isolated from *Ambrosia psilostachya* and *Parthenium hysterophorus* respectively (Fig.1).

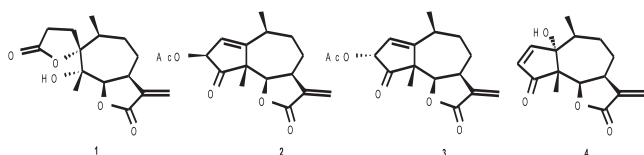


Fig.1 Naturally occurring sesquiterpene lactones

In continuity to our research programme of isolation and structural modifications of natural products to develop bioactive lead molecules particularly in the area of inflammation and cancer, we report the semi-synthesis of naturally occurring acetylated pseudoguaianolides (**2** and **3**) and spiro lactone psilostachyin (**1**) including their analogues from the major pseudoguaianolide parthenin from *Parthenium hysterophorus* for the evaluation of their anti-inflammatory potential estimated through *in vitro* expression of TNF- α , IL-1 β and IL-6 in murine neutrophils. The structure of semi-synthetic psilostachyin was also confirmed by X-ray crystallography.

Keywords: sesquiterpenes lactones, Parthenin, crystal structure

MS61.P22

Acta Cryst. (2011) **A67**, C612-C613

Crystal structures of novel CB[6] complexes with *p*-Xylylenediammine derivatives

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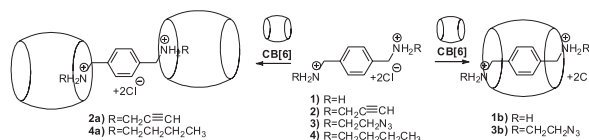
The ability of cucurbit[6]uril, **CB[6]**, to form host-guest complex with diammonium salts, in particular with linear aliphatic diammonium salts, has been widely used for the construction of pseudorotaxanes and rotaxanes that exhibited interesting dynamic, structural and functional properties. By contrast, the ability of **CB[6]** to host aromatic compounds has been less obvious. It has been shown that **CB[6]** and the dihydrochloride salt of bis-1,4-(allylaminomethyl)benzene, form only [3] pseudorotaxane. This mode of binding with two molecules of **CB[6]** hosting the allylic side chains of the guest was led to the interpretation that the aromatic moiety is too big to be encapsulated in the cavity of **CB[6]**. [1] However, it has already been reported that **CB[6]** and *p*-xylylenediammonium salt, **1**, form a stable inclusion complex, **1b**. [2] Furthermore, 4-aminobipyridine, and 4-(2-pyridyl)aniline, form strong, 1:1 inclusion complexes with **CB[6]**, whereas the bifunctional analog forms strong 1:2 complex with **CB[6]**, exhibiting remarkably large enhancements of fluorescence intensity and quantum yields. [3] These led us to speculate that the encapsulation of aromatic guest molecules, within the inner cavity of **CB[6]** can be thermodynamically favored but their formation may involve unexpectedly high kinetic barriers.

Novel pseudorotaxane compounds based on **CB[6]** and *p*-xylylenediammonium salts derivatives, **1**, **3-4**, were obtained by crystallization from aqueous solutions that contain a mixture of each diammonium guest and **CB[6]** at room temperature. The X-ray diffraction analysis of their complexes, **1b**, **3b** and **4a** was also supported by ¹H NMR in solution. The 1:2 complex of guest **2**, namely **2a**, was evident by NMR, but suitable for X-ray analysis single crystal were not be able to obtain. Nevertheless, upon heating and crystallization, the 1:1 complex, **2b**, was obtained.

Further support for these two different binding modes was obtained from ITC measurements at room temperature.

Each 1:1 complex exhibits two types of hydrogen bonding between the portal oxygen atoms and the guest. The more obvious bonds are formed with the ammonium hydrogen atoms (NH...O=C, 1.980(3)–2.420(3)Å), The less obvious, but quite visible from the solid state structure, are the hydrogen bonding between the carbonyl oxygens of **CB[6]** and the benzylic methylene hydrogens of the guest: CH...O=C, 2.317(3)–2.508(2) Å. Complex **4a** exhibited two distinguished hydrogen bond interactions: one corresponds to donor water molecule that is bridged by carbonyl oxygen of each cucurbituril unit (C=O(10)...O_w... (6A)O=C, 2.84-- 2.97Å); second, where two water molecules formed a string bridge between the two cucurbituril units. These strong interactions (C=O(8)...O_w... (04A)O=C, 2.73-2.88Å) could explain the asymmetry observed in the structure of **4a** where the **CB** units are obviously attracted to one another, creating an angle of 42.56° between them.

Our results show that 2:1 host-guest complexes, **2a** and **4a**, are kinetic products, which may be converted to the thermodynamic products upon prolonged heating.



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