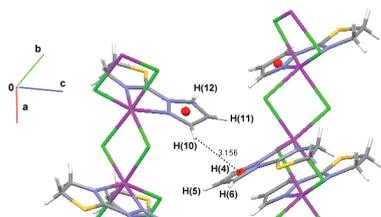


containing two crystallographically independent monomeric units that are enantiomers. A refinement of the Flack parameter led to a direct estimation of the enantiomeric composition. Moreover, these structural units show a disorder affecting to heterocycles, probably due to the presence of two enantiomers in the crystal lattice.

The coordination polymer framework consists of chains made up of  $[\text{Cd}(\mu\text{-Cl})_2(\text{PtTn})]$  structural units, each one bound to other two units through chloro bridging ligands. Chains are held together by van der Waals forces and aromatic interactions, forming parallel sheets in such a way that sheets belonging to each enantiomer alternate.



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**Keywords:** coordination polymer, racemic twinning

## MS87.P14

*Acta Cryst.* (2011) **A67**, C753

### Synthesis and structural study of a circular trimetallic Cu(I) helicate

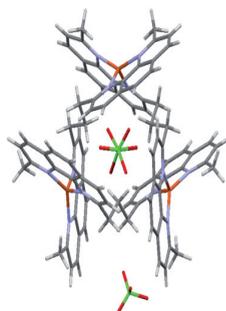
Luis Lemus,<sup>a</sup> Juan Guerrero,<sup>a</sup> Juan Costamagna,<sup>a</sup> Danilo Jara,<sup>a</sup> José Zagal,<sup>a</sup> Allen Oliver,<sup>b</sup> Guillermo Ferraudi,<sup>b</sup> Graham Lappin,<sup>b</sup> <sup>a</sup>*Facultad de Química y Biología, Universidad de Santiago de Chile, Santiago, (Chile)*. <sup>b</sup>*Department of Chemistry and Biochemistry, University of Notre Dame, IN, (USA)*. E-mail: luis.lemus@usach.cl

Bridged polypyridines ligands have been widely used for the building of multimetallic supramolecular structures upon coordination to copper (I). Adequate choice of ligand design allows the formation of a variety of linear and circular helicates. [1], [2]

This work presents the synthesis and X-ray structure of the circular trimetallic helicate  $[\text{Cu}(\text{mphenpr})]_3(\text{ClO}_4)_3$ , (see figure) where *mphenpr* = 1,3-bis(9-methyl-1,10-phenanthrolin-2-yl)propane. The complex crystallizes as a racemic mixture in the trigonal  $P3_1c$  space group, with  $a = b = 16.9700(5)$  Å,  $c = 17.6573(6)$  Å,  $V = 4403.7(2)$  Å<sup>3</sup>,  $Z = 2$ ,  $R(F^2 > 2\sigma(F^2)) = 0.065$  and  $wR(F^2) = 0.230$  for 3028 independent reflections (2063 with  $I > 2\sigma(I)$ ), and 190 parameters. The perchlorate counterions were found to be disordered.

The trimetallic helicate is produced from suspensions of its related bimetallic helicate [3] in coordinating solvents, such as acetonitrile. The coordination geometry around each copper atom is pseudo-tetrahedral with angles of 72.18° between the phenanthroline planes.

1D and 2D NMR studies show that the complex remains in its structural nature in concentrated solutions of non-coordinating solvents, while the decrease of the concentration and the use of coordinating solvents drive to the unfolding of the trimer, followed by the formation of an equilibrium between the bimetallic and monometallic related species.



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**Keywords:** circular helicates, copper (I) complexes, supramolecular

## MS87.P15

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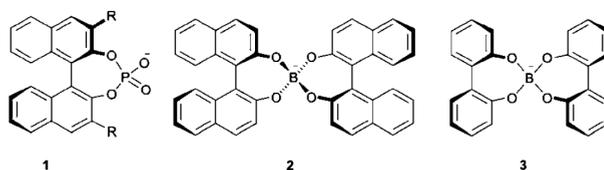
### Chiral selection in the formation of borates from racemic binaphthols and related diols

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Chiral counterions have received increasing attention from the asymmetric catalysis community over the past few years [1]. BINOL is the foremost example of axial chirality. The chiral backbone of BINOL is stereochemically stable (*atropos*) and the enantiomers can be readily resolved [2]; it has been one of the most extensively used motifs in the creation of chiral anions. Some spectacular applications of BINOL-based chiral phosphates in asymmetric catalysis have recently been demonstrated [3], [4]. The bis-BINOL borate **2**, initially introduced by Periasamy for the purpose of resolving amino alcohol derivatives [4], has been studied less extensively in the asymmetric catalysis context [5].

The synthesis of bis-BINOL borates from racemic BINOL can yield the homochiral (R,R)<sub>ax</sub> and (S,S)<sub>ax</sub> or the heterochiral (R,S)<sub>ax</sub> diastereomers. Recent studies of bis-BINOL **2** or biphenol **3** borates by Wuest and co-workers have demonstrated that both form homochiral anions upon crystallisation from a racemic solution in the presence of various amines as well as other counterions [6]. The bis-biphenol borate anion (**3**) framework is stereochemically labile (*tropos*) and the stereoselection is therefore limited to the solid state.

As part of a project designed to study chiral ion pairs of relevance to asymmetric catalysis [7], homochiral racemates of various substituted bis-BINOL borates with sodium as counterion were required. A series of racemic or stereochemically labile chiral borate anions based on the 2,20-biphenol motif was investigated [8]. All borates were homochiral in the solid state, although in some cases the heterochiral diastereomers were computed to be thermodynamically preferred (DFT). The crystallographic preference for the homochiral diastereomer was attributed to its lower bulk, higher molecular symmetry, and the therewith associated better packing ability. We report the results of our systematic investigation.



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**Keywords:** chirality, chiral, borate

## MS87.P16

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### Recent developments in the SuperNova dual source micro-focus diffractometer.

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The introduction of the **SuperNova** in late 2008 being the first dual wavelength micro-focus diffractometer gave a tremendous boost to the capabilities and throughput for in-house crystallographic research. The combination of the high intensity Cu and Mo micro-focus sources (50W) co-mounted along with a 135mm Atlas CCD detector allows for true flexibility on a per-experiment basis.

Continued research and development in core technologies used in the **SuperNova** means that key aspects of the instrument have been updated and improved since its initial launch. Here we would like to highlight the overall benefits of these evolutionary changes and refinements in both hardware and software by presenting a number of absolute configuration determination studies. The examples presented include a broad spectrum of materials with greatly varying anomalous signal, including the first, purely hydrocarbon based molecule's experimental chirality determination using Bayesian methods.

**Keywords:** absolute configuration determination

## MS87.P17

*Acta Cryst.* (2011) **A67**, C754

### A three-dimensional framework structure of vanadium and 1,2,4,5-benzenetetracarboxylate

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Blue block crystals were obtained from the hydrothermal synthesis in aqueous solution of vanadium pentoxide, titanium tetraisopropoxide and 1,2,4,5-benzenetetracarboxylic acid in the molar ratio 1:2:2 at 150°C for 4 days, and structurally analyzed by single crystal X-ray diffraction. The interest in solving this structure is the difficulty in defining the correct space group. The unit cell parameters are consistent with a tetragonal unit cell, yet the systematic absence data cannot unambiguously identify an appropriate space group.

Assuming the structure to be orthorhombic with *a* and *b* approximately equivalent provides for the space group  $P2_12_12_1$  with  $a = 11.1302(6)$ ,  $b = 11.1307(6)$ ,  $c = 13.4114(7)$  Å,  $Z = 4$  and  $V = 1660.457(15)$  Å<sup>3</sup>. To solve the structure, both racemic and pseudo merohedral twin law were applied; the Flack parameter for the current refinement is 0.20(8), which indicates inversion twinning. However, the best model obtained so far refines to give  $R_1 = 0.1286$ , and further investigation is needed. The structure contains two crystallographically-independent vanadium atoms in octahedral geometry coordinated by four carboxylate-oxygen atoms from fully deprotonated 1,2,4,5-benzenecarboxylate ligands in the equatorial positions and two aqua ligands occupying the axial positions generate the three dimensional framework structure.

**Keywords:** pseudo merohedral twin, vanadium carboxylate, framework structure

## MS87.P18

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### Synthesis and structural characterization of two new isoindol derivatives

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Isoindol derivatives are versatile intermediates for the synthesis of a variety of bioactive compounds, and possess a broad spectrum of biological activity such as insecticidal, antibacterial, antibiotic, antitumor, antifungal, psychotherapeutic, antihypertensive, HIV and other activities. In this study, two new derivatives of Isoindol, N-(1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2H-4,7-methanoisoindol-2-yl)-2,2-dimethylpropanamide and 4,8-Methano-3-(2,5-dimethoxyphenyl)-6-(methylamino)4,4a,8,exo-8a-tetrahydro-exo-3aH isoxazolo [5,4-f]isoindole-5,7(6H,7aH)-endo-dione, have been synthesized and characterized by elemental analysis, FT-IR, UV-Vis and 1H NMR spectroscopy. The crystal structure of this compounds has been determined by single crystal diffraction method. The first compound (C<sub>14</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>), which is an oxalamide derivative, crystallized in the orthorhombic system with space group Pca21,  $a = 24.038$  (2) Å,  $b = 9.3387$  (7) Å,  $c = 12.3004$  (9) Å,  $Z = 8$ . The final R value is 0.0066 for 5227 measured reflections. The compound has two molecules in the asymmetric unit, is a chiral compound with 8 stereogenic center. This compound occurs non-centrosymmetric dimeric structure with N-H...O type H-bond. The crystal structure of the compound is stabilized by intermolecular N – H...O type and intramolecular N – H...O, C – H...O type H-bonds, also C – O...pi weak interactions[1,2]. The second compound (C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>O<sub>5</sub>) is an isoxazoline derivative, crystallized in the monoclinic system with space group P21/n,  $a = 9.7183$  (1)Å,  $b = 13.1735$  (2) Å,  $c = 13.2880$  (2)Å,  $\beta = 94.058$  (1)°,  $Z = 4$ . The final R value is 0.0036 for 3404 reflections. The molecule adopts Z-configuration. The structure is a chiral compound with 4 stereogenic center. Molecule occurs the fusion of the isoxazoline ring and dicarboximide onto norbornane unit. The crystal structure is stabilized by C – H...O type intra-molecular, C – H...N and C – H...O type inter-molecular interactions. In the crystal structure intermolecular N – H...N and C – H...O hydrogen bonds link the molecules to form an infinite one dimensional polymeric chain along b-axis. Crystallographic results are consistent with the literature [3], [4].