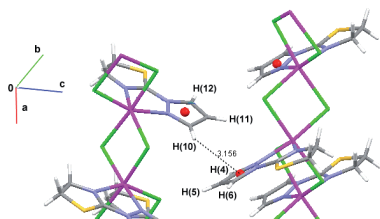


containing two crystallographically independent monomeric units that are enantiomers. A refinement of the Flack parameter led to a direct estimation of the enantiomeric composition. Moreover, these structural units show a disorder affecting to heterocycles, probably due to the presence of two enantiomers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



[1] A. Bernalte-García, A.M. Lozano-Vila, F. Luna-Giles, R. Pedrero-Marín, *Polyhedron* **2006**, *25*, 1399-1407.

Keywords: coordination polymer, racemic twinning

MS87.P14

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

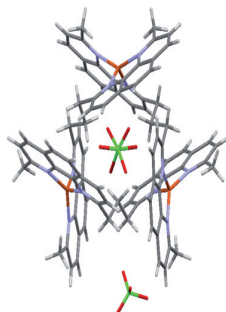
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Bridged polypyridines ligands have been widely used for the building of multimetallic supramolecular structures upon coordination to copper (I). Adequate choice of ligand design allows the formation of a variety of linear and circular helicates. [1], [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, $V = 4403.7(2)$ Å³, $Z = 2$, $R(F^2 > 2\sigma(F^2)) = 0.065$ 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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[1] J. Hamblin, F. Tuna, S. Bunce, L. Childs, A. Jackson, W. Errington, N. Alcock, H. Nierengarten, A. Van Dorsselaer, E. Leize-Wagner, M. Hannon, *Chem. Eur. J.* **2007**, *13*, 9286-9296. [2] M. Albrecht, *Chem. Rev.* **2001**, *101*, 3457-3497. [3] L. Lemus, J. Guerrero, J. Costamagna, G. Estiú, G. Ferraudi, G. Lappin, A. Oliver, B. Noll, *Inorg. Chem.* **2010**, *49*, 4023-4035.

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

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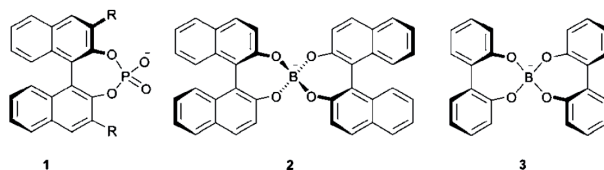
Chiral selection in the formation of borates from racemic binaphthols and related diols

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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)_{ax} and (S,S)_{ax} or the heterochiral (R,S)_{ax} 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.



[1] J. Lacour, R. Frantz, *Org. Biomol. Chem.* **2005**, *3*, 15; M. Terada, *Chem. Commun.* **2008**, 4097. [2] M. Oki, *Top. Stereochem.* **1983**, *14*, 4193. [3] T. Akiyama, J. Itoh, K. Yokota, K. Fuchibe, *Angew. Chem. Int. Ed.*, **2004**, *43*, 1566;