

Diastereoselective formation of a dicopper(I) helicate with a chiral tetradentate pyridyl-thiazole ligand

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Electronic Supplementary Information

Experimental for X-ray Crystal Structure

Data was collected on a brown rod crystal with size 0.05 x 0.06 x 0.49 mm³ mounted in capillary using Bruker SMART 1000 CCD diffractometer. The Radiation source is fine-focus sealed tube with graphite monochromator. The Detector resolution is 512 x 512 pixels. A matrix of 3 runs of 20 frames each with exposure of 5 seconds was collected for a preliminary unit cell determination. Of which, 24 reflections out of total of 38 reflections were successfully indexed. Data, in 4 runs, totaling of 1421 frames with exposure time of 120 seconds each were collected, and a total of 28504 reflections was thus obtained after integration. The structure was solved by using Direct methods (SHELXS97), the remaining non-H atoms were located from difference Fourier map. The structure was refined using SHELXL97.

The total potential solvent area volume per unit cell volume is 1341.7Å³ [32.4%]. the largest void volume is about 660Å³ at (0, 0.5, -0.064). Attempts to locate the solvates were not successful, only one dichloromethane and one water solvates could be located, as such, a void volume of about 380Å³ still remains. As a result, the PLATON-SQUEEZE program was run and found that there may be as much as 191 electron counts in the void. Due to the fact that the data was collected at room temperature, 27 (1)°C, the probability of finding all the solvates which possibly were disordered is deemed to be low; the solvent-free reflection data were then obtained from the SQUEEZE program and used in subsequent SHELXL refinement until convergence. PLATON was then run again with the new .hkl and .res files with the instruction 'CALC FCF' to produce the final FoFc-CIF on .hkp, which was then renamed to the .fcf file. Thus this CIF only contain the major complex without any solvates located.

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0025P)^2]$
$wR(F^2) = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\max} = 0.004$
9405 reflections	$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$
455 parameters	Extinction correction: SHELXL, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
21 restraints	Extinction coefficient: 0.000266 (8)
0 constraints	Absolute structure: Flack (1983). 4118 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.008 (9)
Secondary atom site location: difference Fourier map	

Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

All non-H atoms were refined anisotropically. H-atoms could be located from difference Fourier map but were placed at geometrical sites with C-H = 0.93, 0.96, 0.97Å and 0.98Å for phenyl, methylene, methyl and methine hydrogen respectively. They were all refined using riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{Carrier})$.

21 restraints were used in the least-squares refinements which were related to the refinement of the disordered PF_6 ion. Atoms P1A and P1B were restrained to the same coordinates and the same thermal parameters, while all the P-F distances were restrained to be similar.

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* v7.34A(Bruker, 2006); data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku Corporation, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to

refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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