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[8,17-Bis(5-nitro-2-oxidobenzyl-κO)-5,6:19,20-dibenzo-1,4,11,14-tetraoxa-8,17-diazacycloeicosane-$\kappa^4 N,N'$]-copper(II) chloroform solvate

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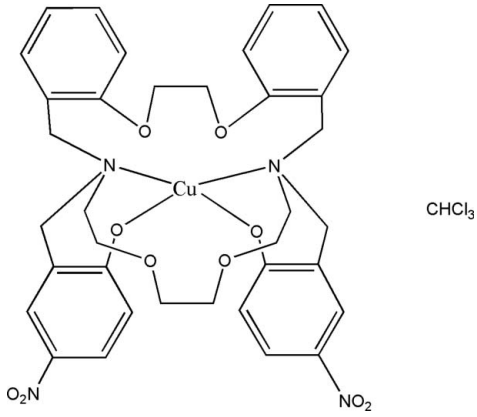
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; H-atom completeness 98%; disorder in solvent or counterion; R factor = 0.056; wR factor = 0.148; data-to-parameter ratio = 16.5.

In the title compound, $C_{36}H_{38}CuN_4O_{10}\cdot CHCl_3$, the Cu^{II} atom is coordinated by two phenolate O atoms and two amine N atoms and displays a distorted square-planar coordination geometry. The complex molecule has approximate C_2 symmetry. The chloroform solvent molecule is disordered equally over two positions.

Related literature

For related literature, see: Ma *et al.* (2005); Ma & Zhu (2002); Meyerstein (1990). For synthesis, see: López-Deber *et al.* (2005).



Experimental

Crystal data

$C_{36}H_{38}CuN_4O_{10}\cdot CHCl_3$
M_r = 869.61
Triclinic, $P\overline{1}$
a = 12.055 (3) Å
b = 12.819 (4) Å
c = 13.409 (4) Å
α = 82.532 (12)°
β = 82.378 (11)°
γ = 71.364 (9)°
V = 1937.7 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 293 (2) K
0.23 × 0.22 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.826, T_{max} = 0.883
18759 measured reflections
8707 independent reflections
6756 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.148
S = 1.07
8707 reflections
527 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.33 e Å^{-3}
$\Delta\rho_{min}$ = −1.03 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O5	1.874 (2)	Cu1—N2	2.144 (2)
Cu1—O6	1.878 (2)	Cu1—N1	2.147 (2)
O5—Cu1—O6	165.47 (10)	O5—Cu1—N1	92.40 (9)
O5—Cu1—N2	89.60 (9)	O6—Cu1—N1	92.16 (9)
O6—Cu1—N2	94.16 (9)	N2—Cu1—N1	146.51 (9)

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2091).

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supplementary materials

Acta Cryst. (2007). E**63**, m2301 [**doi:10.1107/S1600536807038202**]

[8,17-Bis(5-nitro-2-oxidobenzyl-κO)-5,6:19,20-dibenzo-1,4,11,14-tetraoxa-8,17-diazacycloeicosane-$\kappa^4 N,N'$]copper(II) chloroform solvate

T.-T. Han, J.-F. Ma, G.-J. Ping and Z.-F. Jia

Comment

In the last few decades, the modification of the macrocycles to control and tune properties of coordinated metal centers has been the subject of much interest (Meyerstein, 1990). We are involved in studies of oxaaza-macrocycles and their metal coordination compounds (Ma *et al.*, 2005). In this paper, we report the preparation and crystal structure of the title macrocyclic complex of Cu^{II}.

In the reported crystal structure, the Cu^{II} atom is four-coordinated by two phenolate O atoms and two N atoms from the macrocyclic ligand forming a distorted square planar coordination geometry (Fig. 1). The title molecule has an approximate C_2 symmetry. The geometry around the Cu atom is normal (Ma & Zhu, 2002). The chloroform molecule is disordered.

Experimental

5,6:19,20-Dibenzo-1,4,11,14-tetraoxa-8,17-diazacycloeicosane (L2) was synthesized according to the reported method (López-Deber *et al.*, 2005). An anhydrous toluene solution (200 ml) containing L2 (0.76 g, 2.00 mmol), paraformaldehyde (0.15 g, 5.00 mmol), and 4-nitrophenol (0.56 g, 4.00 mmol) was refluxed for 24 h. The solvent was evaporated by rotatory evaporation, and the yellow solid consisting of the title ligand H_2L was obtained.

A solution of $Cu(NO_3)_2{\cdot}3H_2O$ (0.024 g, 0.10 mmol) in 5 ml e thanol was added dropwise to a solution of H_2L (0.068 g, 0.10 mmol) in 6 ml chloroform. After stirring for 30 min, the mixture was filtered. Green crystals of of the title compound were obtained by evaporating the filtrate at room temperature (yield 50%).

Refinement

The H atoms bonded C atoms were placed at their idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene and aromatic H atoms. The chloroform molecule is disordered over two positions. It was refined with no restraints imposed on the molecular geometry and with the assumed occupancy factor of 0.5. The high residual peak on the final differnce map is located 0.70 Å from Cl2' atom. The H atom of the chloroform molecule was not localized and is not included in the structural model.

Figures

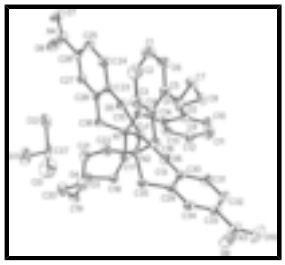


Fig. 1. A view of the molecule the title molecule. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity, Only one orintation of $CHCl_3$ is shown.

[8,17-Bis(5-nitro-2-oxidobenzyl-κ*O*)-5,6:19,20-dibenzo-1,4,11,14-tetraoxa-8,17- diazacycloeicosane-κ^4*N*,*N*']copper(II) chloroform solvate

Crystal data

$C_{36}H_{38}CuN_4O_{10}\cdot CHCl_3$	$Z = 2$
$M_r = 869.61$	$F_{000} = 898$
Triclinic, $P\bar{1}$	$D_x = 1.490$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 12.055$ (3) Å	Cell parameters from 13446 reflections
$b = 12.819$ (4) Å	$\theta = 3.1$–27.4º
$c = 13.409$ (4) Å	$\mu = 0.83$ mm^{-1}
$\alpha = 82.532$ (12)º	$T = 293$ (2) K
$\beta = 82.378$ (11)º	Block, green
$\gamma = 71.364$ (9)º	0.23 × 0.22 × 0.15 mm
$V = 1937.7$ (10) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	8707 independent reflections
Radiation source: rotor target	6756 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 27.4$º
$T = 293(2)$ K	$\theta_{min} = 3.1$º
ω scans	$h = -15\rightarrow15$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -16\rightarrow16$
$T_{min} = 0.826$, $T_{max} = 0.883$	$l = -17\rightarrow17$
18759 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.148$	$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 1.7805P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
8707 reflections	$\Delta\rho_{max} = 1.33$ e Å^{-3}
527 parameters	$\Delta\rho_{min} = -1.03$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. 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 $F^2 > \sigma(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.71823 (3)	0.20288 (3)	0.27584 (3)	0.03486 (12)	
C1	0.3790 (3)	0.3280 (4)	0.6013 (3)	0.0620 (10)	
H1	0.2997	0.3450	0.6247	0.074*	
C2	0.4616 (4)	0.2550 (4)	0.6590 (3)	0.0695 (11)	
H2	0.4386	0.2238	0.7217	0.083*	
C3	0.5793 (3)	0.2283 (3)	0.6231 (3)	0.0554 (9)	
H3	0.6352	0.1792	0.6625	0.067*	
C4	0.6155 (3)	0.2732 (3)	0.5297 (2)	0.0411 (7)	
C5	0.5300 (3)	0.3481 (3)	0.4729 (2)	0.0405 (7)	
C6	0.4121 (3)	0.3763 (3)	0.5092 (3)	0.0514 (8)	
H6	0.3557	0.4274	0.4716	0.062*	
C7	0.4897 (3)	0.4700 (3)	0.3214 (3)	0.0495 (8)	
H7A	0.4518	0.5357	0.3566	0.059*	
H7B	0.4296	0.4399	0.3076	0.059*	
C8	0.5560 (3)	0.4981 (3)	0.2251 (3)	0.0503 (8)	
H8A	0.5076	0.5633	0.1883	0.060*	
H8B	0.6264	0.5126	0.2389	0.060*	
C9	0.6373 (3)	0.4149 (3)	0.0713 (2)	0.0443 (7)	
C10	0.6663 (4)	0.5075 (3)	0.0251 (3)	0.0596 (9)	
H10	0.6535	0.5686	0.0606	0.072*	
C11	0.7148 (4)	0.5071 (4)	−0.0752 (3)	0.0704 (12)	
H11	0.7342	0.5690	−0.1063	0.084*	
C12	0.7345 (3)	0.4184 (4)	−0.1290 (3)	0.0633 (10)	
H12	0.7668	0.4196	−0.1959	0.076*	

C13	0.7055 (3)	0.3267 (3)	−0.0819 (3)	0.0519 (8)
H13	0.7185	0.2660	−0.1181	0.062*
C14	0.6576 (3)	0.3229 (3)	0.0180 (2)	0.0399 (7)
C15	0.6212 (2)	0.2252 (3)	0.0668 (2)	0.0375 (6)
H15A	0.6106	0.1853	0.0140	0.045*
H15B	0.5455	0.2522	0.1055	0.045*
C16	0.7440 (3)	0.2485 (3)	0.4927 (2)	0.0395 (6)
H16A	0.7571	0.3168	0.4617	0.047*
H16B	0.7891	0.2244	0.5508	0.047*
C17	0.8248 (2)	0.1088 (3)	0.0779 (2)	0.0395 (7)
H17A	0.8463	0.1743	0.0506	0.047*
H17B	0.8190	0.0716	0.0211	0.047*
C18	0.9229 (3)	0.0329 (3)	0.1370 (2)	0.0429 (7)
H18A	0.9980	0.0336	0.1008	0.052*
H18B	0.9176	0.0604	0.2021	0.052*
C19	1.0058 (3)	−0.1512 (3)	0.2126 (3)	0.0511 (8)
H19A	1.0679	−0.1186	0.2143	0.061*
H19B	1.0402	−0.2198	0.1811	0.061*
C20	0.9581 (4)	−0.1763 (3)	0.3186 (3)	0.0608 (10)
H20A	0.8890	−0.1990	0.3170	0.073*
H20B	1.0166	−0.2376	0.3510	0.073*
C21	0.8062 (3)	−0.0388 (3)	0.4051 (3)	0.0487 (8)
H21A	0.7770	−0.0964	0.4420	0.058*
H21B	0.7656	−0.0134	0.3445	0.058*
C22	0.7783 (3)	0.0561 (3)	0.4694 (2)	0.0430 (7)
H22A	0.6977	0.0702	0.4994	0.052*
H22B	0.8285	0.0327	0.5242	0.052*
C23	0.5085 (2)	0.1334 (3)	0.3020 (2)	0.0370 (6)
C24	0.3935 (3)	0.1536 (3)	0.3514 (2)	0.0470 (8)
H24	0.3720	0.1949	0.4069	0.056*
C25	0.3129 (3)	0.1135 (3)	0.3194 (2)	0.0455 (7)
H25	0.2374	0.1277	0.3523	0.055*
C26	0.3459 (3)	0.0513 (3)	0.2372 (2)	0.0417 (7)
C27	0.4568 (3)	0.0326 (2)	0.1851 (2)	0.0389 (6)
H27	0.4761	−0.0075	0.1288	0.047*
C28	0.5387 (2)	0.0730 (2)	0.2159 (2)	0.0347 (6)
C29	0.9426 (2)	0.2571 (2)	0.3520 (2)	0.0371 (6)
C30	0.8940 (2)	0.3123 (3)	0.2616 (2)	0.0375 (6)
C31	0.9198 (3)	0.4091 (3)	0.2205 (3)	0.0506 (8)
H31	0.8890	0.4459	0.1611	0.061*
C32	0.9903 (3)	0.4512 (3)	0.2668 (3)	0.0547 (9)
H32	1.0078	0.5149	0.2382	0.066*
C33	1.0340 (3)	0.3977 (3)	0.3554 (3)	0.0476 (8)
C34	1.0106 (2)	0.3018 (3)	0.3984 (2)	0.0427 (7)
H34	1.0407	0.2673	0.4588	0.051*
C35	0.9206 (2)	0.1509 (2)	0.3955 (2)	0.0376 (6)
H35A	0.9589	0.1250	0.4572	0.045*
H35B	0.9556	0.0955	0.3480	0.045*
C36	0.6608 (2)	0.0469 (2)	0.1620 (2)	0.0358 (6)

H36A	0.7145	−0.0084	0.2042	0.043*	
H36B	0.6615	0.0148	0.1001	0.043*	
C37	0.8037 (11)	−0.2229 (8)	0.0476 (7)	0.056 (3)	0.50
C37'	0.8568 (17)	−0.239 (2)	0.0380 (19)	0.161 (12)	0.50
N1	0.70559 (19)	0.1447 (2)	0.13594 (17)	0.0332 (5)	
N2	0.79173 (19)	0.16288 (19)	0.41818 (17)	0.0339 (5)	
N3	1.1055 (3)	0.4422 (3)	0.4063 (3)	0.0629 (9)	
N4	0.2629 (3)	0.0064 (3)	0.2054 (2)	0.0550 (7)	
O1	0.57237 (18)	0.38994 (18)	0.38185 (16)	0.0439 (5)	
O2	0.5863 (2)	0.40529 (18)	0.16771 (16)	0.0485 (5)	
O3	0.9184 (2)	−0.07739 (19)	0.1522 (2)	0.0542 (6)	
O4	0.9275 (2)	−0.0835 (2)	0.3771 (2)	0.0641 (7)	
O5	0.58314 (18)	0.1697 (2)	0.33764 (16)	0.0462 (5)	
O6	0.82833 (19)	0.27311 (19)	0.21482 (15)	0.0439 (5)	
O7	0.1678 (2)	0.0182 (3)	0.2558 (2)	0.0804 (9)	
O8	0.2898 (3)	−0.0447 (3)	0.1304 (2)	0.0853 (10)	
O9	1.1372 (3)	0.3971 (3)	0.4884 (3)	0.0845 (10)	
O10	1.1309 (3)	0.5246 (3)	0.3661 (3)	0.0926 (11)	
Cl1	0.8225 (5)	−0.3148 (4)	0.1526 (4)	0.1063 (18)	0.50
Cl1'	0.9885 (4)	−0.3439 (4)	0.0298 (3)	0.1373 (14)	0.50
Cl2	0.6560 (2)	−0.19588 (18)	0.0184 (2)	0.0889 (8)	0.50
Cl2'	0.7794 (7)	−0.2300 (6)	−0.0623 (6)	0.209 (2)*	0.50
Cl3	0.8917 (5)	−0.2601 (4)	−0.0603 (4)	0.177 (2)	0.50
Cl3'	0.7727 (5)	−0.2745 (5)	0.1465 (5)	0.147 (2)	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0335 (2)	0.0416 (2)	0.03340 (19)	−0.01475 (15)	−0.00599 (13)	−0.00648 (14)
C1	0.046 (2)	0.074 (3)	0.064 (2)	−0.0170 (18)	0.0112 (17)	−0.023 (2)
C2	0.074 (3)	0.071 (3)	0.057 (2)	−0.024 (2)	0.021 (2)	−0.007 (2)
C3	0.058 (2)	0.052 (2)	0.0466 (19)	−0.0075 (16)	0.0037 (15)	−0.0049 (16)
C4	0.0439 (17)	0.0411 (16)	0.0385 (16)	−0.0113 (13)	−0.0002 (12)	−0.0127 (13)
C5	0.0388 (16)	0.0427 (17)	0.0411 (16)	−0.0096 (13)	−0.0046 (12)	−0.0147 (13)
C6	0.0414 (18)	0.060 (2)	0.053 (2)	−0.0092 (15)	−0.0051 (14)	−0.0194 (16)
C7	0.0468 (18)	0.0446 (18)	0.0496 (19)	0.0039 (14)	−0.0150 (14)	−0.0130 (15)
C8	0.064 (2)	0.0349 (16)	0.0484 (19)	−0.0048 (14)	−0.0174 (16)	−0.0056 (14)
C9	0.0452 (17)	0.0453 (18)	0.0415 (17)	−0.0124 (14)	−0.0113 (13)	0.0021 (14)
C10	0.072 (2)	0.048 (2)	0.063 (2)	−0.0260 (18)	−0.0138 (19)	0.0038 (18)
C11	0.078 (3)	0.064 (3)	0.068 (3)	−0.032 (2)	−0.007 (2)	0.025 (2)
C12	0.059 (2)	0.073 (3)	0.050 (2)	−0.0189 (19)	−0.0008 (17)	0.0120 (19)
C13	0.0490 (19)	0.059 (2)	0.0424 (18)	−0.0105 (16)	−0.0049 (14)	0.0011 (15)
C14	0.0354 (15)	0.0438 (17)	0.0380 (16)	−0.0089 (12)	−0.0086 (12)	0.0018 (13)
C15	0.0340 (15)	0.0454 (17)	0.0348 (15)	−0.0117 (12)	−0.0080 (11)	−0.0060 (12)
C16	0.0381 (16)	0.0432 (16)	0.0372 (15)	−0.0089 (12)	−0.0069 (12)	−0.0099 (13)
C17	0.0337 (15)	0.0486 (18)	0.0362 (15)	−0.0124 (13)	0.0015 (11)	−0.0088 (13)
C18	0.0362 (16)	0.0467 (18)	0.0481 (18)	−0.0134 (13)	−0.0041 (13)	−0.0105 (14)
C19	0.0394 (18)	0.0458 (19)	0.064 (2)	−0.0026 (14)	−0.0097 (15)	−0.0117 (16)

C20	0.076 (3)	0.045 (2)	0.062 (2)	−0.0166 (18)	−0.0013 (19)	−0.0196 (17)
C21	0.0496 (19)	0.0390 (17)	0.057 (2)	−0.0115 (14)	−0.0107 (15)	−0.0037 (15)
C22	0.0452 (17)	0.0409 (17)	0.0412 (17)	−0.0126 (13)	−0.0030 (13)	−0.0006 (13)
C23	0.0345 (15)	0.0444 (16)	0.0371 (15)	−0.0179 (12)	−0.0021 (11)	−0.0082 (12)
C24	0.0417 (17)	0.061 (2)	0.0436 (17)	−0.0217 (15)	0.0056 (13)	−0.0199 (15)
C25	0.0344 (16)	0.057 (2)	0.0483 (18)	−0.0198 (14)	0.0020 (13)	−0.0085 (15)
C26	0.0410 (16)	0.0474 (18)	0.0447 (17)	−0.0232 (14)	−0.0071 (13)	−0.0049 (14)
C27	0.0435 (16)	0.0413 (16)	0.0368 (15)	−0.0183 (13)	−0.0042 (12)	−0.0077 (13)
C28	0.0353 (15)	0.0355 (15)	0.0350 (15)	−0.0132 (12)	−0.0027 (11)	−0.0045 (12)
C29	0.0305 (14)	0.0424 (16)	0.0386 (15)	−0.0096 (12)	−0.0058 (11)	−0.0063 (12)
C30	0.0333 (15)	0.0445 (16)	0.0374 (15)	−0.0146 (12)	−0.0034 (11)	−0.0065 (12)
C31	0.060 (2)	0.055 (2)	0.0447 (18)	−0.0293 (17)	−0.0127 (15)	0.0052 (15)
C32	0.060 (2)	0.061 (2)	0.056 (2)	−0.0377 (18)	−0.0046 (16)	−0.0030 (17)
C33	0.0377 (17)	0.064 (2)	0.0500 (19)	−0.0253 (15)	−0.0017 (13)	−0.0161 (16)
C34	0.0305 (15)	0.0555 (19)	0.0436 (17)	−0.0121 (13)	−0.0069 (12)	−0.0096 (14)
C35	0.0289 (14)	0.0417 (16)	0.0412 (16)	−0.0070 (12)	−0.0078 (11)	−0.0057 (13)
C36	0.0366 (15)	0.0363 (15)	0.0368 (15)	−0.0129 (12)	−0.0005 (11)	−0.0100 (12)
C37	0.101 (9)	0.042 (4)	0.041 (4)	−0.044 (5)	−0.014 (5)	−0.001 (3)
C37'	0.134 (17)	0.22 (2)	0.168 (19)	−0.111 (17)	−0.097 (15)	0.076 (16)
N1	0.0304 (12)	0.0388 (13)	0.0323 (12)	−0.0122 (10)	−0.0023 (9)	−0.0064 (10)
N2	0.0313 (12)	0.0359 (13)	0.0335 (12)	−0.0082 (9)	−0.0053 (9)	−0.0035 (10)
N3	0.0547 (18)	0.091 (3)	0.060 (2)	−0.0433 (18)	0.0025 (15)	−0.0228 (18)
N4	0.0504 (17)	0.070 (2)	0.0574 (18)	−0.0332 (15)	−0.0080 (13)	−0.0104 (15)
O1	0.0414 (12)	0.0473 (12)	0.0393 (12)	−0.0066 (9)	−0.0080 (9)	−0.0048 (9)
O2	0.0642 (15)	0.0424 (12)	0.0399 (12)	−0.0164 (11)	−0.0056 (10)	−0.0066 (10)
O3	0.0479 (13)	0.0438 (13)	0.0744 (17)	−0.0114 (10)	−0.0230 (11)	−0.0069 (11)
O4	0.0591 (16)	0.0587 (16)	0.0785 (18)	−0.0153 (12)	−0.0020 (13)	−0.0331 (14)
O5	0.0412 (12)	0.0688 (15)	0.0393 (11)	−0.0275 (11)	0.0017 (9)	−0.0220 (11)
O6	0.0500 (12)	0.0559 (13)	0.0354 (11)	−0.0286 (10)	−0.0116 (9)	0.0009 (10)
O7	0.0516 (16)	0.124 (3)	0.088 (2)	−0.0548 (17)	0.0049 (14)	−0.0307 (19)
O8	0.080 (2)	0.122 (3)	0.085 (2)	−0.063 (2)	0.0053 (16)	−0.052 (2)
O9	0.081 (2)	0.122 (3)	0.076 (2)	−0.058 (2)	−0.0289 (16)	−0.0100 (19)
O10	0.112 (3)	0.123 (3)	0.081 (2)	−0.091 (2)	−0.0047 (18)	−0.015 (2)
Cl1	0.145 (4)	0.089 (3)	0.103 (3)	−0.058 (3)	−0.064 (3)	0.034 (2)
Cl1'	0.136 (3)	0.143 (3)	0.107 (3)	−0.010 (2)	0.020 (2)	−0.035 (2)
Cl2	0.1068 (19)	0.0546 (12)	0.114 (2)	−0.0205 (12)	−0.0597 (16)	0.0009 (12)
Cl3	0.186 (5)	0.160 (4)	0.168 (4)	−0.063 (3)	0.127 (4)	−0.076 (3)
Cl3'	0.138 (5)	0.117 (4)	0.192 (6)	−0.070 (3)	0.070 (4)	−0.048 (4)

Geometric parameters (Å, °)

Cu1—O5	1.874 (2)	C20—H20B	0.9700
Cu1—O6	1.878 (2)	C21—O4	1.407 (4)
Cu1—N2	2.144 (2)	C21—C22	1.507 (5)
Cu1—N1	2.147 (2)	C21—H21A	0.9700
C1—C2	1.374 (6)	C21—H21B	0.9700
C1—C6	1.377 (5)	C22—N2	1.497 (4)
C1—H1	0.9300	C22—H22A	0.9700
C2—C3	1.383 (5)	C22—H22B	0.9700

C2—H2	0.9300	C23—O5	1.301 (3)
C3—C4	1.386 (5)	C23—C24	1.414 (4)
C3—H3	0.9300	C23—C28	1.416 (4)
C4—C5	1.397 (4)	C24—C25	1.371 (4)
C4—C16	1.506 (4)	C24—H24	0.9300
C5—O1	1.371 (4)	C25—C26	1.384 (5)
C5—C6	1.386 (4)	C25—H25	0.9300
C6—H6	0.9300	C26—C27	1.387 (4)
C7—O1	1.431 (4)	C26—N4	1.436 (4)
C7—C8	1.491 (5)	C27—C28	1.381 (4)
C7—H7A	0.9700	C27—H27	0.9300
C7—H7B	0.9700	C28—C36	1.506 (4)
C8—O2	1.424 (4)	C29—C34	1.384 (4)
C8—H8A	0.9700	C29—C30	1.419 (4)
C8—H8B	0.9700	C29—C35	1.499 (4)
C9—O2	1.365 (4)	C30—O6	1.318 (3)
C9—C10	1.389 (5)	C30—C31	1.402 (4)
C9—C14	1.396 (5)	C31—C32	1.385 (5)
C10—C11	1.394 (6)	C31—H31	0.9300
C10—H10	0.9300	C32—C33	1.373 (5)
C11—C12	1.366 (6)	C32—H32	0.9300
C11—H11	0.9300	C33—C34	1.384 (5)
C12—C13	1.384 (5)	C33—N3	1.452 (4)
C12—H12	0.9300	C34—H34	0.9300
C13—C14	1.389 (5)	C35—N2	1.505 (3)
C13—H13	0.9300	C35—H35A	0.9700
C14—C15	1.500 (4)	C35—H35B	0.9700
C15—N1	1.512 (4)	C36—N1	1.502 (4)
C15—H15A	0.9700	C36—H36A	0.9700
C15—H15B	0.9700	C36—H36B	0.9700
C16—N2	1.504 (4)	C37—C37'	0.61 (2)
C16—H16A	0.9700	C37—Cl3'	1.460 (12)
C16—H16B	0.9700	C37—Cl2'	1.560 (12)
C17—N1	1.503 (3)	C37—Cl3	1.702 (11)
C17—C18	1.512 (4)	C37—Cl1	1.705 (11)
C17—H17A	0.9700	C37—Cl2	1.788 (12)
C17—H17B	0.9700	C37'—Cl3	1.36 (3)
C18—O3	1.420 (4)	C37'—Cl2'	1.71 (2)
C18—H18A	0.9700	C37'—Cl1'	1.72 (2)
C18—H18B	0.9700	C37'—Cl3'	1.75 (2)
C19—O3	1.432 (4)	C37'—Cl1	1.78 (2)
C19—C20	1.495 (5)	N3—O9	1.223 (4)
C19—H19A	0.9700	N3—O10	1.231 (4)
C19—H19B	0.9700	N4—O8	1.223 (4)
C20—O4	1.433 (4)	N4—O7	1.226 (4)
C20—H20A	0.9700		
O5—Cu1—O6	165.47 (10)	C25—C24—C23	121.5 (3)
O5—Cu1—N2	89.60 (9)	C25—C24—H24	119.3
O6—Cu1—N2	94.16 (9)	C23—C24—H24	119.3

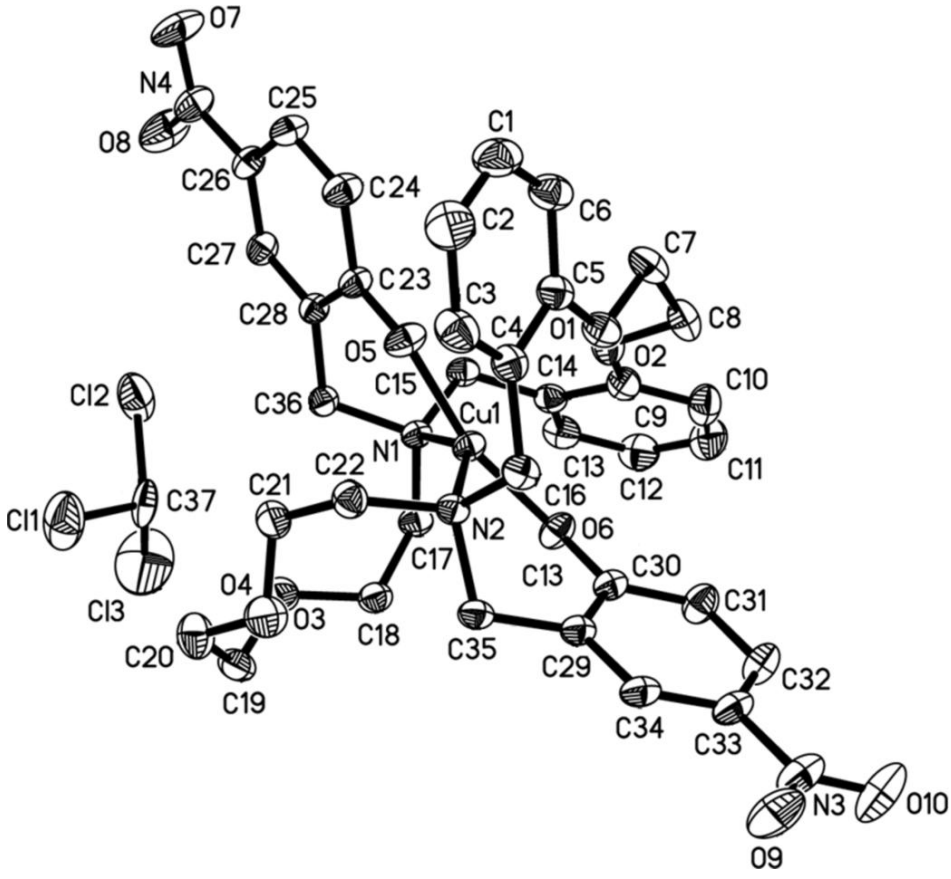
O5—Cu1—N1	92.40 (9)	C24—C25—C26	118.8 (3)
O6—Cu1—N1	92.16 (9)	C24—C25—H25	120.6
N2—Cu1—N1	146.51 (9)	C26—C25—H25	120.6
C2—C1—C6	120.8 (3)	C25—C26—C27	121.3 (3)
C2—C1—H1	119.6	C25—C26—N4	119.0 (3)
C6—C1—H1	119.6	C27—C26—N4	119.7 (3)
C1—C2—C3	119.5 (4)	C28—C27—C26	120.5 (3)
C1—C2—H2	120.3	C28—C27—H27	119.7
C3—C2—H2	120.3	C26—C27—H27	119.7
C2—C3—C4	121.2 (4)	C27—C28—C23	119.2 (3)
C2—C3—H3	119.4	C27—C28—C36	119.9 (3)
C4—C3—H3	119.4	C23—C28—C36	120.8 (2)
C3—C4—C5	118.2 (3)	C34—C29—C30	119.5 (3)
C3—C4—C16	121.3 (3)	C34—C29—C35	120.9 (3)
C5—C4—C16	120.3 (3)	C30—C29—C35	119.7 (3)
O1—C5—C6	124.3 (3)	O6—C30—C31	119.9 (3)
O1—C5—C4	114.9 (3)	O6—C30—C29	121.7 (3)
C6—C5—C4	120.7 (3)	C31—C30—C29	118.4 (3)
C1—C6—C5	119.5 (3)	C32—C31—C30	121.3 (3)
C1—C6—H6	120.3	C32—C31—H31	119.3
C5—C6—H6	120.3	C30—C31—H31	119.3
O1—C7—C8	107.5 (3)	C33—C32—C31	119.1 (3)
O1—C7—H7A	110.2	C33—C32—H32	120.5
C8—C7—H7A	110.2	C31—C32—H32	120.5
O1—C7—H7B	110.2	C32—C33—C34	121.4 (3)
C8—C7—H7B	110.2	C32—C33—N3	119.8 (3)
H7A—C7—H7B	108.5	C34—C33—N3	118.8 (3)
O2—C8—C7	106.8 (3)	C33—C34—C29	120.3 (3)
O2—C8—H8A	110.4	C33—C34—H34	119.9
C7—C8—H8A	110.4	C29—C34—H34	119.9
O2—C8—H8B	110.4	C29—C35—N2	112.7 (2)
C7—C8—H8B	110.4	C29—C35—H35A	109.1
H8A—C8—H8B	108.6	N2—C35—H35A	109.1
O2—C9—C10	125.1 (3)	C29—C35—H35B	109.1
O2—C9—C14	114.6 (3)	N2—C35—H35B	109.1
C10—C9—C14	120.4 (3)	H35A—C35—H35B	107.8
C9—C10—C11	118.9 (4)	N1—C36—C28	114.4 (2)
C9—C10—H10	120.5	N1—C36—H36A	108.7
C11—C10—H10	120.5	C28—C36—H36A	108.7
C12—C11—C10	121.8 (4)	N1—C36—H36B	108.7
C12—C11—H11	119.1	C28—C36—H36B	108.7
C10—C11—H11	119.1	H36A—C36—H36B	107.6
C11—C12—C13	118.7 (4)	C37'—C37—Cl3'	109 (3)
C11—C12—H12	120.7	C37'—C37—Cl2'	93 (3)
C13—C12—H12	120.7	Cl3'—C37—Cl2'	133.9 (8)
C12—C13—C14	121.6 (4)	C37'—C37—Cl3	47 (3)
C12—C13—H13	119.2	Cl3'—C37—Cl3	138.6 (8)
C14—C13—H13	119.2	Cl2'—C37—Cl3	46.2 (4)
C13—C14—C9	118.6 (3)	C37'—C37—Cl1	87 (3)

C13—C14—C15	121.2 (3)	Cl2'—C37—Cl1	133.2 (6)
C9—C14—C15	120.1 (3)	Cl3—C37—Cl1	118.4 (7)
C14—C15—N1	115.7 (2)	C37'—C37—Cl2	153 (3)
C14—C15—H15A	108.3	Cl3'—C37—Cl2	88.9 (7)
N1—C15—H15A	108.3	Cl2'—C37—Cl2	60.2 (5)
C14—C15—H15B	108.3	Cl3—C37—Cl2	106.3 (6)
N1—C15—H15B	108.3	Cl1—C37—Cl2	107.0 (6)
H15A—C15—H15B	107.4	C37—C37'—Cl3	114 (3)
N2—C16—C4	116.3 (2)	C37—C37'—Cl2'	66 (2)
N2—C16—H16A	108.2	Cl3—C37'—Cl2'	48.0 (7)
C4—C16—H16A	108.2	C37—C37'—Cl1'	152 (3)
N2—C16—H16B	108.2	Cl3—C37'—Cl1'	70.0 (13)
C4—C16—H16B	108.2	Cl2'—C37'—Cl1'	111.5 (16)
H16A—C16—H16B	107.4	C37—C37'—Cl3'	52 (2)
N1—C17—C18	115.9 (2)	Cl3—C37'—Cl3'	143.3 (15)
N1—C17—H17A	108.3	Cl2'—C37'—Cl3'	106.9 (10)
C18—C17—H17A	108.3	Cl1'—C37'—Cl3'	107.1 (12)
N1—C17—H17B	108.3	C37—C37'—Cl1	73 (3)
C18—C17—H17B	108.3	Cl3—C37'—Cl1	136.9 (18)
H17A—C17—H17B	107.4	Cl2'—C37'—Cl1	118.6 (10)
O3—C18—C17	112.2 (2)	Cl1'—C37'—Cl1	85.7 (10)
O3—C18—H18A	109.2	C36—N1—C17	109.0 (2)
C17—C18—H18A	109.2	C36—N1—C15	105.9 (2)
O3—C18—H18B	109.2	C17—N1—C15	108.3 (2)
C17—C18—H18B	109.2	C36—N1—Cu1	107.17 (16)
H18A—C18—H18B	107.9	C17—N1—Cu1	110.15 (16)
O3—C19—C20	113.2 (3)	C15—N1—Cu1	116.07 (17)
O3—C19—H19A	108.9	C22—N2—C16	107.6 (2)
C20—C19—H19A	108.9	C22—N2—C35	109.0 (2)
O3—C19—H19B	108.9	C16—N2—C35	106.1 (2)
C20—C19—H19B	108.9	C22—N2—Cu1	111.25 (18)
H19A—C19—H19B	107.8	C16—N2—Cu1	116.04 (17)
O4—C20—C19	112.6 (3)	C35—N2—Cu1	106.49 (17)
O4—C20—H20A	109.1	O9—N3—O10	122.4 (3)
C19—C20—H20A	109.1	O9—N3—C33	118.7 (3)
O4—C20—H20B	109.1	O10—N3—C33	119.0 (3)
C19—C20—H20B	109.1	O8—N4—O7	121.9 (3)
H20A—C20—H20B	107.8	O8—N4—C26	119.2 (3)
O4—C21—C22	112.8 (3)	O7—N4—C26	118.8 (3)
O4—C21—H21A	109.0	C5—O1—C7	117.8 (2)
C22—C21—H21A	109.0	C9—O2—C8	118.1 (3)
O4—C21—H21B	109.0	C18—O3—C19	114.2 (2)
C22—C21—H21B	109.0	C21—O4—C20	114.6 (3)
H21A—C21—H21B	107.8	C23—O5—Cu1	131.38 (19)
N2—C22—C21	117.1 (3)	C30—O6—Cu1	126.55 (19)
N2—C22—H22A	108.0	Cl3'—Cl1—C37	57.5 (9)
C21—C22—H22A	108.0	Cl3'—Cl1—C37'	77.0 (11)
N2—C22—H22B	108.0	Cl3'—Cl1—Cl1'	122.8 (10)
C21—C22—H22B	108.0	C37—Cl1—Cl1'	65.3 (5)

H22A—C22—H22B	107.3	C37'—Cl1—Cl1'	46.1 (8)
O5—C23—C24	119.1 (3)	C37'—Cl1'—Cl3	45.6 (8)
O5—C23—C28	122.3 (3)	C37'—Cl1'—Cl1	48.2 (8)
C24—C23—C28	118.6 (3)		
C6—C1—C2—C3	−1.2 (6)	Cl2'—C37—C37'—Cl3'	−138.8 (15)
C1—C2—C3—C4	−0.4 (6)	Cl3—C37—C37'—Cl3'	−140.0 (16)
C2—C3—C4—C5	1.1 (5)	Cl1—C37—C37'—Cl3'	−5.6 (12)
C2—C3—C4—C16	177.0 (3)	Cl2—C37—C37'—Cl3'	−128 (7)
C3—C4—C5—O1	179.4 (3)	Cl3'—C37—C37'—Cl1	5.6 (12)
C16—C4—C5—O1	3.4 (4)	Cl2'—C37—C37'—Cl1	−133.1 (6)
C3—C4—C5—C6	−0.2 (5)	Cl3—C37—C37'—Cl1	−134 (2)
C16—C4—C5—C6	−176.2 (3)	Cl2—C37—C37'—Cl1	−123 (6)
C2—C1—C6—C5	2.1 (6)	C28—C36—N1—C17	−177.0 (2)
O1—C5—C6—C1	179.1 (3)	C28—C36—N1—C15	−60.7 (3)
C4—C5—C6—C1	−1.4 (5)	C28—C36—N1—Cu1	63.8 (3)
O1—C7—C8—O2	72.7 (3)	C18—C17—N1—C36	−68.8 (3)
O2—C9—C10—C11	−178.0 (3)	C18—C17—N1—C15	176.5 (2)
C14—C9—C10—C11	0.5 (5)	C18—C17—N1—Cu1	48.6 (3)
C9—C10—C11—C12	0.0 (6)	C14—C15—N1—C36	−171.3 (2)
C10—C11—C12—C13	−0.1 (6)	C14—C15—N1—C17	−54.4 (3)
C11—C12—C13—C14	−0.2 (6)	C14—C15—N1—Cu1	70.0 (3)
C12—C13—C14—C9	0.7 (5)	O5—Cu1—N1—C36	−36.01 (18)
C12—C13—C14—C15	177.1 (3)	O6—Cu1—N1—C36	157.78 (17)
O2—C9—C14—C13	177.8 (3)	N2—Cu1—N1—C36	56.9 (2)
C10—C9—C14—C13	−0.9 (5)	O5—Cu1—N1—C17	−154.52 (19)
O2—C9—C14—C15	1.4 (4)	O6—Cu1—N1—C17	39.28 (19)
C10—C9—C14—C15	−177.3 (3)	N2—Cu1—N1—C17	−61.6 (3)
C13—C14—C15—N1	101.8 (3)	O5—Cu1—N1—C15	82.01 (19)
C9—C14—C15—N1	−81.9 (3)	O6—Cu1—N1—C15	−84.20 (19)
C3—C4—C16—N2	100.6 (3)	N2—Cu1—N1—C15	174.92 (17)
C5—C4—C16—N2	−83.6 (4)	C21—C22—N2—C16	172.5 (3)
N1—C17—C18—O3	76.9 (3)	C21—C22—N2—C35	−72.8 (3)
O3—C19—C20—O4	−70.6 (4)	C21—C22—N2—Cu1	44.3 (3)
O4—C21—C22—N2	72.0 (4)	C4—C16—N2—C22	−61.8 (3)
O5—C23—C24—C25	−177.4 (3)	C4—C16—N2—C35	−178.4 (3)
C28—C23—C24—C25	1.8 (5)	C4—C16—N2—Cu1	63.5 (3)
C23—C24—C25—C26	0.4 (5)	C29—C35—N2—C22	−175.1 (2)
C24—C25—C26—C27	−2.4 (5)	C29—C35—N2—C16	−59.4 (3)
C24—C25—C26—N4	178.1 (3)	C29—C35—N2—Cu1	64.8 (3)
C25—C26—C27—C28	2.0 (5)	O5—Cu1—N2—C22	46.72 (19)
N4—C26—C27—C28	−178.5 (3)	O6—Cu1—N2—C22	−147.32 (19)
C26—C27—C28—C23	0.3 (4)	N1—Cu1—N2—C22	−47.0 (3)
C26—C27—C28—C36	176.7 (3)	O5—Cu1—N2—C16	−76.8 (2)
O5—C23—C28—C27	177.0 (3)	O6—Cu1—N2—C16	89.2 (2)
C24—C23—C28—C27	−2.2 (4)	N1—Cu1—N2—C16	−170.52 (18)
O5—C23—C28—C36	0.6 (4)	O5—Cu1—N2—C35	165.42 (18)
C24—C23—C28—C36	−178.5 (3)	O6—Cu1—N2—C35	−28.63 (18)
C34—C29—C30—O6	−179.8 (3)	N1—Cu1—N2—C35	71.7 (2)
C35—C29—C30—O6	0.7 (4)	C32—C33—N3—O9	175.3 (4)

C34—C29—C30—C31	2.0 (4)	C34—C33—N3—O9	−4.1 (5)
C35—C29—C30—C31	−177.5 (3)	C32—C33—N3—O10	−3.8 (5)
O6—C30—C31—C32	−178.8 (3)	C34—C33—N3—O10	176.8 (4)
C29—C30—C31—C32	−0.5 (5)	C25—C26—N4—O8	177.0 (4)
C30—C31—C32—C33	−1.0 (6)	C27—C26—N4—O8	−2.5 (5)
C31—C32—C33—C34	1.0 (6)	C25—C26—N4—O7	−4.7 (5)
C31—C32—C33—N3	−178.4 (3)	C27—C26—N4—O7	175.8 (3)
C32—C33—C34—C29	0.5 (5)	C6—C5—O1—C7	1.6 (4)
N3—C33—C34—C29	179.9 (3)	C4—C5—O1—C7	−178.0 (3)
C30—C29—C34—C33	−2.0 (4)	C8—C7—O1—C5	−177.3 (3)
C35—C29—C34—C33	177.5 (3)	C10—C9—O2—C8	2.4 (5)
C34—C29—C35—N2	122.6 (3)	C14—C9—O2—C8	−176.2 (3)
C30—C29—C35—N2	−57.9 (4)	C7—C8—O2—C9	173.4 (3)
C27—C28—C36—N1	133.4 (3)	C17—C18—O3—C19	−176.8 (3)
C23—C28—C36—N1	−50.3 (4)	C20—C19—O3—C18	103.1 (3)
Cl3'—C37—C37'—Cl3	140.0 (16)	C22—C21—O4—C20	178.1 (3)
Cl2'—C37—C37'—Cl3	1(3)	C19—C20—O4—C21	112.7 (4)
Cl1—C37—C37'—Cl3	134 (2)	C24—C23—O5—Cu1	−156.6 (2)
Cl2—C37—C37'—Cl3	12 (8)	C28—C23—O5—Cu1	24.3 (5)
Cl3'—C37—C37'—Cl2'	138.8 (15)	O6—Cu1—O5—C23	103.5 (4)
Cl3—C37—C37'—Cl2'	−1(3)	N2—Cu1—O5—C23	−151.3 (3)
Cl1—C37—C37'—Cl2'	133.1 (6)	N1—Cu1—O5—C23	−4.7 (3)
Cl2—C37—C37'—Cl2'	11 (5)	C31—C30—O6—Cu1	−144.3 (3)
Cl3'—C37—C37'—Cl1'	48 (8)	C29—C30—O6—Cu1	37.4 (4)
Cl2'—C37—C37'—Cl1'	−91 (7)	O5—Cu1—O6—C30	84.9 (4)
Cl3—C37—C37'—Cl1'	−92 (7)	N2—Cu1—O6—C30	−19.7 (3)
Cl1—C37—C37'—Cl1'	42 (7)	N1—Cu1—O6—C30	−166.8 (3)
Cl2—C37—C37'—Cl1'	−80 (10)		

Fig. 1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