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Tris(1,10-phenanthroline-5,6-dione-κ^2N,N')copper(II) bis(perchlorate) 4.5-hydrate

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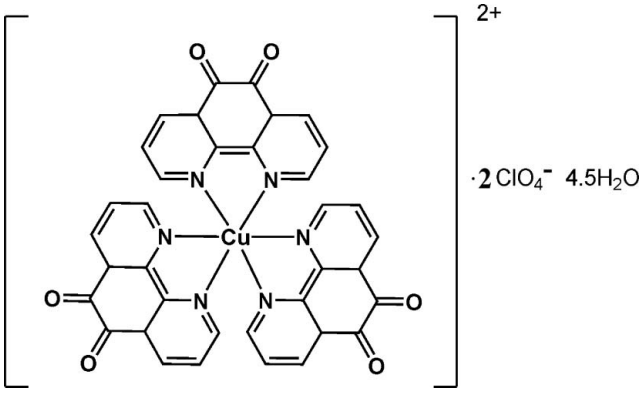
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.007 Å; disorder in solvent or counterion; R factor = 0.047; wR factor = 0.122; data-to-parameter ratio = 13.0.

In the title compound, $[Cu(C_{12}H_6N_2O_2)_3](ClO_4)_2\cdot4.5H_2O$, the Cu^{II} atom is in a distorted octahedral environment with unequal Cu—N distances, due to the Jahn–Teller elongation of the two Cu—N bonds in *trans* positions. The crystal studied was an inversion twin..

Related literature

For related literature, see: Majumdar *et al.* (1998); Hadadzadeh *et al.* (2006); Wang *et al.* (2007); Mansouri *et al.* (2007).



Experimental

Crystal data

$[Cu(C_{12}H_6N_2O_2)_3](ClO_4)_2\cdot4.5H_2O$
M_r = 974.08
Orthorhombic, $P2_12_12_1$
a = 13.136 (2) Å
b = 14.071 (2) Å
c = 20.636 (3) Å
V = 3814.2 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 93 (2) K
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2004)
T_{min} = 0.920, T_{max} = 0.923
24980 measured reflections
6940 independent reflections
6206 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.122
S = 0.95
6940 reflections
534 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Absolute structure: Flack (1983), with 3043 Friedel pairs
Flack parameter: 0.517 (14)

Table 1
Selected bond lengths (Å).

Cu1—N1	2.045 (3)	Cu1—N10	2.069 (3)
Cu1—N20	2.047 (3)	Cu1—N11	2.253 (3)
Cu1—N21	2.059 (4)	Cu1—N30	2.288 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O51—H51A···O10	0.98	1.99	2.937 (5)	162.8
O51—H51B···O55	0.98	2.39	3.099	129.1
O52—H52A···O15^{i}	0.98	1.87	2.810 (3)	158.6
O52—H52B···O55	0.98	1.96	2.623	123.1
O53—H53A···O2	0.98	1.99	2.956 (3)	168.3
O53—H53B···O26	0.98	2.06	2.878 (3)	139.3
O54—H54A···O53	0.98	1.90	2.824	157.1
O54—H54B···O51^{ii}	0.98	2.16	2.917	132.6
O55—H55A···O51	0.98	2.12	3.099	179.9
O55—H55B···O16^{i}	0.98	2.08	2.998 (3)	154.4

Symmetry codes: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2003); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2056).

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supplementary materials

Acta Cryst. (2007). E**63**, m2848 [**doi:10.1107/S1600536807052348**]

Tris(1,10-phenanthroline-5,6-dione-κ^2N,N')copper(II) bis(perchlorate) 4.5-hydrate

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Comment

Metal complexes with 1,10-phenantroline-5,6-dione (phen-dione) as a ligand have been increasingly studied over recent years (Hadadzadeh *et al.*, 2006; Mansouri *et al.*, 2007). The structure of monomeric hexacoordinated tris-chelated complexes of copper(II) have been reported previously (Majumdar *et al.*, 1998 Wang *et al.*, 2007). The six-coordinate copper center in the the title complex has distorted octahedral geometry (Fig. 1) being slightly tetragonally elongated due to the Jahn–Teller elongation of two Cu—N bonds in *trans* positions (average Cu—Neq = 2.05 and Cu—Nax = 2.27 Å) and the contraction of three of the N—Cu—N angles (average chelate angle N—Cu—N = 78°) from the ideal octahedral values, imposed by the three chelate ligands (Majumdar *et al.*, 1998).

Experimental

1 was prepared by the reaction of $Cu(ClO_4)2$ (37 mg, 0.1 mmol) and (phen-dione) (62 mg, 0.3 mmol) in a mixture CH_3CN–H_2O (2:1 *v*/*v*, 6 ml) at 298 K. After allowing the resulting green solution to stand at 298 K for 3 days, green crystals of the product were formed.

Refinement

H atoms were included in calculated positions (O—H distances are 0.98 Å, C—H distances are 0.98 Å for methyl H atoms, 0.99 Å for methylene H atoms and 0.95 Å for aryl H atoms) and were refined as riding atoms with $U_{iso}(H) = 1.2U_{eq}$(parent atom, O, methylene and aryl H atoms) or $U_{iso}(H) = 1.5U_{eq}$(parent atom, methyl H atoms). O55 was refined as 0.5 occupancy to be compatable with microanlaytical data.

Figures

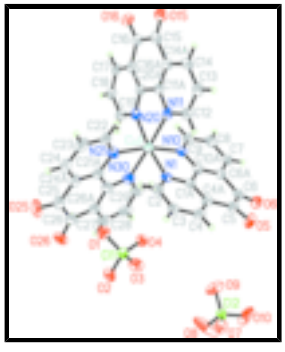


Fig. 1. The structure of (**1**) with displacement ellipsoids drawn at the 50% probability level.

Tris(1,10-phenanthroline-5,6-dione-κ^2*N*,*N*')copper(II) bis(perchlorate) 4.5-hydrate

Crystal data

$[Cu(C_{12}H_6N_2O_2)_3](ClO_4)_2·4.5H_2O$	F_{000} = 1984
M_r = 974.08	D_x = 1.696 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 12591 reflections
a = 13.136 (2) Å	θ = 1.6–28.8º
b = 14.071 (2) Å	μ = 0.81 mm^{-1}
c = 20.636 (3) Å	T = 93 (2) K
V = 3814.2 (10) Å^3	Prism, green
Z = 4	0.10 × 0.10 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer	6940 independent reflections
Radiation source: rotating anode	6206 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.061
T = 93(2) K	θ_{max} = 25.4º
ω and φ scans	θ_{min} = 2.1º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2004)	h = −11→15
T_{min} = 0.920, T_{max} = 0.923	k = −16→16
24980 measured reflections	l = −23→24

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 2.3835P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.122	$(\Delta/\sigma)_{max}$ = 0.016
S = 0.95	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
6940 reflections	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
534 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with how many Friedel pairs?
Secondary atom site location: difference Fourier map	Flack parameter: 0.517 (14)

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	0.80991 (4)	0.51514 (3)	−0.06737 (2)	0.02531 (13)	
C1A	0.6817 (3)	0.3910 (3)	0.00640 (17)	0.0245 (8)	
N1	0.7794 (3)	0.4193 (2)	0.00456 (15)	0.0246 (8)	
C2	0.8461 (4)	0.3783 (3)	0.04414 (19)	0.0293 (10)	
H2A	0.9149	0.3989	0.0429	0.035*	
C3	0.8192 (4)	0.3058 (3)	0.08770 (18)	0.0296 (9)	
H3A	0.8680	0.2788	0.1162	0.036*	
C4	0.7188 (4)	0.2751 (3)	0.0875 (2)	0.0326 (11)	
H4A	0.6978	0.2250	0.1154	0.039*	
C4A	0.6494 (4)	0.3183 (3)	0.04634 (19)	0.0292 (9)	
C5	0.5416 (4)	0.2886 (3)	0.0463 (2)	0.0311 (10)	
O5	0.5114 (3)	0.2206 (2)	0.07747 (16)	0.0402 (8)	
C6	0.4668 (4)	0.3469 (3)	0.0055 (2)	0.0318 (10)	
O6	0.3748 (3)	0.3364 (2)	0.01319 (15)	0.0411 (8)	
C6A	0.5092 (3)	0.4159 (3)	−0.04171 (19)	0.0271 (9)	
C7	0.4487 (4)	0.4575 (3)	−0.0889 (2)	0.0339 (10)	
H7A	0.3783	0.4428	−0.0919	0.041*	
C8	0.4929 (3)	0.5202 (3)	−0.13128 (19)	0.0340 (10)	
H8A	0.4534	0.5492	−0.1644	0.041*	
C9	0.5950 (4)	0.5410 (3)	−0.1255 (2)	0.0323 (10)	
H9A	0.6244	0.5856	−0.1546	0.039*	
N10	0.6545 (2)	0.5005 (2)	−0.08031 (14)	0.0252 (7)	
C10A	0.6118 (3)	0.4370 (3)	−0.03969 (19)	0.0259 (9)	
C11A	0.8654 (3)	0.4356 (3)	−0.19842 (18)	0.0231 (8)	
N11	0.8339 (3)	0.4002 (2)	−0.14182 (15)	0.0264 (8)	
C12	0.8182 (4)	0.3058 (3)	−0.1370 (2)	0.0296 (9)	
H12A	0.7941	0.2808	−0.0971	0.035*	
C13	0.8357 (4)	0.2443 (3)	−0.1878 (2)	0.0354 (11)	
H13A	0.8227	0.1782	−0.1831	0.042*	
C14	0.8727 (4)	0.2803 (3)	−0.2457 (2)	0.0318 (10)	
H14A	0.8868	0.2392	−0.2812	0.038*	
C14A	0.8887 (3)	0.3774 (3)	−0.25086 (19)	0.0284 (9)	
C15	0.9276 (3)	0.4188 (3)	−0.3117 (2)	0.0303 (10)	

O15	0.9565 (3)	0.3708 (2)	−0.35700 (14)	0.0399 (8)
C16	0.9344 (3)	0.5279 (3)	−0.31652 (18)	0.0285 (9)
O16	0.9674 (2)	0.5632 (2)	−0.36579 (14)	0.0354 (7)
C16A	0.9003 (3)	0.5846 (3)	−0.26048 (19)	0.0264 (9)
C17	0.8983 (3)	0.6839 (3)	−0.2627 (2)	0.0299 (10)
H17A	0.9181	0.7163	−0.3011	0.036*
C18	0.8676 (4)	0.7345 (3)	−0.2094 (2)	0.0308 (10)
H18A	0.8641	0.8019	−0.2106	0.037*
C19	0.8418 (3)	0.6850 (3)	−0.1536 (2)	0.0286 (10)
H19A	0.8233	0.7202	−0.1161	0.034*
N20	0.8418 (3)	0.5896 (2)	−0.15016 (15)	0.0252 (8)
C20A	0.8708 (3)	0.5402 (3)	−0.20334 (19)	0.0262 (9)
C21A	0.9652 (4)	0.6034 (3)	0.0173 (2)	0.0406 (12)
N21	0.9543 (3)	0.5403 (3)	−0.03255 (16)	0.0378 (10)
C22	1.0360 (4)	0.4910 (3)	−0.0517 (2)	0.0401 (11)
H22A	1.0288	0.4475	−0.0867	0.048*
C23	1.1302 (4)	0.5004 (4)	−0.0230 (2)	0.0560 (14)
H23A	1.1860	0.4629	−0.0373	0.067*
C24	1.1423 (5)	0.5650 (5)	0.0266 (3)	0.0622 (17)
H24A	1.2070	0.5743	0.0462	0.075*
C24A	1.0580 (5)	0.6160 (4)	0.0474 (2)	0.0503 (14)
C25	1.0674 (5)	0.6847 (4)	0.1013 (3)	0.0605 (17)
O25	1.1476 (4)	0.7083 (4)	0.1246 (2)	0.0977 (19)
C26	0.9694 (5)	0.7303 (3)	0.1277 (2)	0.0470 (14)
O26	0.9727 (3)	0.7770 (2)	0.17717 (15)	0.0567 (11)
C26A	0.8755 (4)	0.7166 (3)	0.0904 (2)	0.0411 (13)
C27	0.7863 (5)	0.7642 (3)	0.1079 (2)	0.0474 (15)
H27A	0.7860	0.8066	0.1437	0.057*
C28	0.6988 (5)	0.7487 (3)	0.0725 (2)	0.0492 (14)
H28A	0.6370	0.7802	0.0829	0.059*
C29	0.7045 (5)	0.6855 (4)	0.0211 (2)	0.0452 (13)
H29A	0.6441	0.6740	−0.0030	0.054*
N30	0.7889 (4)	0.6394 (3)	0.00283 (17)	0.0392 (10)
C30A	0.8739 (4)	0.6550 (3)	0.0371 (2)	0.0387 (12)
Cl1	0.86111 (8)	0.51099 (7)	0.19790 (4)	0.0303 (2)
O1	0.9516 (2)	0.5291 (2)	0.15943 (14)	0.0410 (8)
O2	0.8557 (3)	0.5791 (2)	0.25014 (14)	0.0388 (8)
O3	0.8655 (3)	0.4168 (2)	0.22383 (14)	0.0427 (9)
O4	0.7724 (2)	0.5215 (2)	0.15703 (12)	0.0348 (7)
Cl2	0.32975 (8)	0.49483 (7)	0.29448 (4)	0.0328 (2)
O7	0.3919 (3)	0.4244 (2)	0.32687 (16)	0.0408 (8)
O8	0.3279 (6)	0.5780 (3)	0.3307 (2)	0.115 (3)
O9	0.3686 (3)	0.5139 (3)	0.23209 (16)	0.0647 (11)
O10	0.2305 (3)	0.4562 (4)	0.2882 (2)	0.0905 (17)
O51	0.1547	0.4507	0.1543	0.067
H51A	0.1842	0.4394	0.1973	0.080*
H51B	0.2066	0.4371	0.1215	0.080*
O52	0.4579	0.6529	0.0193	0.107
H52A	0.5011	0.6369	0.0564	0.128*

H52B	0.3868	0.6411	0.0314	0.128*	
O53	1.0080	0.7094	0.3069	0.066	
H53A	0.9624	0.6593	0.2913	0.079*	
H53B	1.0080	0.7595	0.2739	0.079*	
O54	0.9179	0.7872	0.4193	0.051	
H54A	0.9425	0.7758	0.3751	0.061*	
H54B	0.8740	0.8435	0.4185	0.061*	
O55	0.3445	0.5341	0.0856	0.095	0.50
H55A	0.2844	0.5078	0.1073	0.114*	0.50
H55B	0.3979	0.5094	0.1141	0.114*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0312 (3)	0.0204 (2)	0.0243 (2)	−0.0016 (2)	0.00168 (19)	0.00085 (19)
C1A	0.028 (2)	0.0215 (19)	0.0244 (18)	0.0010 (19)	0.0033 (17)	−0.0046 (15)
N1	0.029 (2)	0.0223 (17)	0.0220 (15)	0.0037 (15)	−0.0006 (14)	−0.0029 (13)
C2	0.031 (2)	0.029 (2)	0.0281 (19)	0.000 (2)	0.0021 (18)	−0.0007 (17)
C3	0.039 (3)	0.028 (2)	0.0227 (18)	0.001 (2)	0.0014 (19)	−0.0005 (16)
C4	0.048 (3)	0.019 (2)	0.031 (2)	−0.001 (2)	0.010 (2)	0.0009 (17)
C4A	0.034 (3)	0.0209 (19)	0.032 (2)	0.0024 (19)	0.0056 (19)	0.0002 (17)
C5	0.038 (3)	0.021 (2)	0.034 (2)	0.000 (2)	0.0068 (19)	−0.0006 (18)
O5	0.0390 (19)	0.0274 (16)	0.0543 (19)	−0.0054 (15)	0.0096 (16)	0.0076 (15)
C6	0.033 (3)	0.031 (2)	0.032 (2)	0.000 (2)	0.0015 (19)	−0.0103 (18)
O6	0.033 (2)	0.048 (2)	0.0424 (17)	−0.0055 (16)	0.0050 (15)	−0.0027 (15)
C6A	0.025 (2)	0.024 (2)	0.0323 (19)	0.0042 (19)	0.0040 (18)	−0.0040 (17)
C7	0.032 (2)	0.033 (2)	0.037 (2)	0.002 (2)	−0.0027 (19)	−0.0071 (19)
C8	0.036 (2)	0.032 (2)	0.034 (2)	0.004 (2)	−0.0058 (18)	−0.004 (2)
C9	0.041 (3)	0.028 (2)	0.0287 (19)	0.002 (2)	−0.0001 (19)	−0.0019 (17)
N10	0.0296 (18)	0.0210 (17)	0.0249 (14)	0.0011 (16)	0.0003 (13)	−0.0015 (13)
C10A	0.031 (2)	0.0173 (18)	0.0291 (19)	0.0027 (18)	0.0038 (18)	−0.0038 (16)
C11A	0.021 (2)	0.0203 (19)	0.0281 (19)	−0.0011 (17)	−0.0015 (18)	0.0020 (16)
N11	0.029 (2)	0.0197 (16)	0.0307 (17)	−0.0023 (15)	−0.0020 (15)	0.0051 (14)
C12	0.034 (2)	0.022 (2)	0.033 (2)	0.000 (2)	−0.002 (2)	0.0076 (16)
C13	0.039 (3)	0.026 (2)	0.041 (2)	0.000 (2)	−0.006 (2)	0.0050 (19)
C14	0.035 (3)	0.029 (2)	0.031 (2)	0.003 (2)	−0.0039 (19)	−0.0046 (18)
C14A	0.028 (2)	0.026 (2)	0.032 (2)	0.0008 (19)	−0.0014 (18)	0.0006 (18)
C15	0.030 (2)	0.030 (2)	0.031 (2)	0.004 (2)	−0.0082 (19)	−0.0008 (19)
O15	0.051 (2)	0.0372 (18)	0.0319 (15)	0.0018 (17)	0.0037 (15)	−0.0037 (14)
C16	0.024 (2)	0.034 (2)	0.028 (2)	0.006 (2)	−0.0018 (16)	0.0024 (19)
O16	0.0331 (18)	0.0413 (18)	0.0317 (15)	0.0030 (15)	0.0064 (14)	0.0067 (14)
C16A	0.021 (2)	0.026 (2)	0.033 (2)	−0.0038 (18)	−0.0024 (18)	0.0060 (17)
C17	0.031 (2)	0.028 (2)	0.031 (2)	−0.007 (2)	0.0001 (19)	0.0129 (18)
C18	0.033 (3)	0.019 (2)	0.040 (2)	−0.0010 (19)	0.001 (2)	0.0044 (18)
C19	0.033 (3)	0.0178 (19)	0.035 (2)	0.0027 (18)	0.0045 (19)	0.0016 (17)
N20	0.0241 (19)	0.0213 (16)	0.0304 (17)	−0.0018 (15)	0.0005 (14)	0.0012 (14)
C20A	0.024 (2)	0.024 (2)	0.0300 (19)	0.0020 (17)	0.0019 (18)	−0.0004 (16)
C21A	0.061 (3)	0.030 (2)	0.031 (2)	−0.023 (3)	0.013 (2)	−0.0012 (19)

N21	0.058 (3)	0.027 (2)	0.0290 (18)	−0.0123 (19)	0.0061 (18)	−0.0039 (15)
C22	0.047 (3)	0.033 (2)	0.040 (2)	−0.015 (2)	0.014 (2)	−0.006 (2)
C23	0.049 (3)	0.064 (4)	0.054 (3)	−0.008 (3)	0.024 (3)	−0.008 (3)
C24	0.044 (3)	0.088 (4)	0.055 (3)	−0.033 (3)	0.011 (3)	−0.009 (3)
C24A	0.052 (3)	0.054 (3)	0.045 (3)	−0.031 (3)	0.010 (2)	−0.010 (2)
C25	0.071 (4)	0.063 (4)	0.047 (3)	−0.039 (3)	0.013 (3)	−0.017 (3)
O25	0.067 (3)	0.141 (5)	0.084 (3)	−0.064 (3)	0.010 (3)	−0.054 (3)
C26	0.069 (4)	0.036 (3)	0.036 (2)	−0.024 (3)	0.007 (3)	0.000 (2)
O26	0.098 (3)	0.039 (2)	0.0333 (17)	−0.022 (2)	−0.0034 (19)	−0.0107 (15)
C26A	0.068 (4)	0.027 (2)	0.028 (2)	−0.018 (3)	0.011 (2)	−0.0004 (18)
C27	0.081 (4)	0.028 (2)	0.033 (2)	−0.008 (3)	0.015 (3)	0.003 (2)
C28	0.075 (4)	0.035 (2)	0.037 (2)	−0.003 (3)	0.015 (3)	0.006 (2)
C29	0.062 (4)	0.038 (3)	0.036 (2)	0.000 (3)	0.004 (2)	0.002 (2)
N30	0.057 (3)	0.031 (2)	0.0296 (18)	−0.008 (2)	0.0066 (19)	−0.0013 (15)
C30A	0.062 (4)	0.024 (2)	0.029 (2)	−0.015 (2)	0.006 (2)	0.0004 (18)
Cl1	0.0333 (5)	0.0283 (5)	0.0293 (4)	−0.0004 (5)	0.0005 (4)	−0.0001 (4)
O1	0.0320 (17)	0.050 (2)	0.0408 (16)	−0.0003 (16)	0.0051 (13)	0.0053 (16)
O2	0.0432 (19)	0.0342 (16)	0.0392 (16)	0.0003 (15)	−0.0021 (15)	−0.0133 (14)
O3	0.069 (3)	0.0270 (16)	0.0323 (16)	0.0022 (17)	−0.0044 (17)	0.0060 (13)
O4	0.0352 (16)	0.0389 (17)	0.0301 (14)	0.0020 (15)	−0.0035 (12)	0.0009 (14)
Cl2	0.0358 (6)	0.0303 (5)	0.0323 (5)	0.0006 (5)	0.0008 (4)	−0.0055 (4)
O7	0.046 (2)	0.0279 (16)	0.0488 (18)	0.0069 (16)	0.0003 (16)	0.0036 (14)
O8	0.224 (7)	0.045 (2)	0.077 (3)	0.066 (3)	−0.080 (4)	−0.037 (2)
O9	0.079 (3)	0.059 (2)	0.056 (2)	0.018 (2)	0.030 (2)	0.0241 (19)
O10	0.035 (2)	0.133 (4)	0.104 (3)	−0.023 (3)	−0.009 (2)	0.065 (3)
O51	0.060	0.080	0.060	0.018	−0.008	−0.015
O52	0.131	0.119	0.070	0.039	−0.033	0.004
O53	0.081	0.068	0.049	−0.021	−0.009	0.007
O54	0.042	0.052	0.058	−0.005	−0.007	0.013
O55	0.078	0.130	0.078	0.012	−0.018	0.026

Geometric parameters (Å, °)

Cu1—N1	2.045 (3)	C17—H17A	0.9500
Cu1—N20	2.047 (3)	C18—C19	1.387 (6)
Cu1—N21	2.059 (4)	C18—H18A	0.9500
Cu1—N10	2.069 (3)	C19—N20	1.345 (5)
Cu1—N11	2.253 (3)	C19—H19A	0.9500
Cu1—N30	2.288 (4)	N20—C20A	1.353 (5)
C1A—N1	1.344 (5)	C21A—C24A	1.380 (8)
C1A—C4A	1.380 (6)	C21A—N21	1.367 (5)
C1A—C10A	1.473 (6)	C21A—C30A	1.460 (8)
N1—C2	1.330 (5)	N21—C22	1.338 (6)
C2—C3	1.404 (6)	C22—C23	1.378 (7)
C2—H2A	0.9500	C22—H22A	0.9500
C3—C4	1.389 (7)	C23—C24	1.378 (8)
C3—H3A	0.9500	C23—H23A	0.9500
C4—C4A	1.386 (6)	C24—C24A	1.387 (9)
C4—H4A	0.9500	C24—H24A	0.9500

C4A—C5	1.476 (7)	C24A—C25	1.480 (7)
C5—O5	1.218 (5)	C25—O25	1.205 (7)
C5—C6	1.532 (6)	C25—C26	1.538 (9)
C6—O6	1.228 (6)	C26—O26	1.215 (6)
C6—C6A	1.485 (6)	C26—C26A	1.466 (8)
C6A—C10A	1.381 (6)	C26A—C27	1.397 (8)
C6A—C7	1.387 (6)	C26A—C30A	1.400 (6)
C7—C8	1.371 (6)	C27—C28	1.379 (8)
C7—H7A	0.9500	C27—H27A	0.9500
C8—C9	1.377 (6)	C28—C29	1.386 (7)
C8—H8A	0.9500	C28—H28A	0.9500
C9—N10	1.344 (5)	C29—N30	1.338 (7)
C9—H9A	0.9500	C29—H29A	0.9500
N10—C10A	1.347 (5)	N30—C30A	1.340 (7)
C11A—N11	1.336 (5)	Cl1—O3	1.430 (3)
C11A—C14A	1.391 (6)	Cl1—O2	1.444 (3)
C11A—C20A	1.477 (5)	Cl1—O4	1.446 (3)
N11—C12	1.347 (5)	Cl1—O1	1.452 (3)
C12—C13	1.378 (6)	Cl2—O8	1.389 (4)
C12—H12A	0.9500	Cl2—O9	1.410 (3)
C13—C14	1.387 (6)	Cl2—O10	1.418 (4)
C13—H13A	0.9500	Cl2—O7	1.448 (3)
C14—C14A	1.387 (6)	O51—H51A	0.9799
C14—H14A	0.9500	O51—H51B	0.9801
C14A—C15	1.476 (6)	O52—H52A	0.9799
C15—O15	1.214 (5)	O52—H52B	0.9799
C15—C16	1.541 (6)	O53—H53A	0.9801
C16—O16	1.212 (5)	O53—H53B	0.9799
C16—C16A	1.475 (6)	O54—H54A	0.9800
C16A—C20A	1.389 (5)	O54—H54B	0.9801
C16A—C17	1.399 (6)	O55—H55A	0.9800
C17—C18	1.372 (6)	O55—H55B	0.9800
N1—Cu1—N20	169.47 (13)	O16—C16—C15	118.9 (4)
N1—Cu1—N21	92.32 (14)	C16A—C16—C15	118.1 (4)
N20—Cu1—N21	90.84 (14)	C20A—C16A—C17	118.2 (4)
N1—Cu1—N10	80.48 (13)	C20A—C16A—C16	120.5 (4)
N20—Cu1—N10	98.36 (13)	C17—C16A—C16	121.3 (4)
N21—Cu1—N10	166.19 (13)	C18—C17—C16A	119.8 (4)
N1—Cu1—N11	92.81 (12)	C18—C17—H17A	120.1
N20—Cu1—N11	76.69 (12)	C16A—C17—H17A	120.1
N21—Cu1—N11	103.45 (14)	C17—C18—C19	118.5 (4)
N10—Cu1—N11	88.75 (13)	C17—C18—H18A	120.8
N1—Cu1—N30	91.19 (12)	C19—C18—H18A	120.8
N20—Cu1—N30	99.32 (13)	N20—C19—C18	123.1 (4)
N21—Cu1—N30	76.04 (16)	N20—C19—H19A	118.5
N10—Cu1—N30	92.24 (15)	C18—C19—H19A	118.5
N11—Cu1—N30	175.99 (13)	C19—N20—C20A	118.0 (3)
N1—C1A—C4A	122.0 (4)	C19—N20—Cu1	123.7 (3)
N1—C1A—C10A	116.5 (4)	C20A—N20—Cu1	118.1 (3)

C4A—C1A—C10A	121.3 (4)	N20—C20A—C16A	122.4 (4)
C2—N1—C1A	118.9 (4)	N20—C20A—C11A	116.2 (4)
C2—N1—Cu1	127.1 (3)	C16A—C20A—C11A	121.3 (4)
C1A—N1—Cu1	113.8 (3)	C24A—C21A—N21	120.9 (5)
N1—C2—C3	122.9 (4)	C24A—C21A—C30A	122.4 (4)
N1—C2—H2A	118.6	N21—C21A—C30A	116.6 (5)
C3—C2—H2A	118.6	C22—N21—C21A	118.4 (4)
C4—C3—C2	117.6 (4)	C22—N21—Cu1	123.1 (3)
C4—C3—H3A	121.2	C21A—N21—Cu1	118.1 (3)
C2—C3—H3A	121.2	N21—C22—C23	122.9 (4)
C4A—C4—C3	119.3 (4)	N21—C22—H22A	118.5
C4A—C4—H4A	120.3	C23—C22—H22A	118.5
C3—C4—H4A	120.3	C22—C23—C24	119.1 (5)
C1A—C4A—C4	119.2 (4)	C22—C23—H23A	120.4
C1A—C4A—C5	120.3 (4)	C24—C23—H23A	120.4
C4—C4A—C5	120.4 (4)	C24A—C24—C23	118.6 (5)
O5—C5—C4A	122.3 (4)	C24A—C24—H24A	120.7
O5—C5—C6	120.0 (4)	C23—C24—H24A	120.7
C4A—C5—C6	117.6 (4)	C21A—C24A—C24	120.0 (5)
O6—C6—C6A	122.1 (4)	C21A—C24A—C25	119.7 (6)
O6—C6—C5	119.8 (4)	C24—C24A—C25	120.3 (5)
C6A—C6—C5	118.0 (4)	O25—C25—C24A	123.6 (6)
C10A—C6A—C7	119.3 (4)	O25—C25—C26	118.4 (5)
C10A—C6A—C6	119.2 (4)	C24A—C25—C26	118.0 (5)
C7—C6A—C6	121.5 (4)	O26—C26—C26A	122.9 (6)
C8—C7—C6A	118.4 (4)	O26—C26—C25	119.5 (5)
C8—C7—H7A	120.8	C26A—C26—C25	117.6 (4)
C6A—C7—H7A	120.8	C27—C26A—C30A	119.1 (5)
C7—C8—C9	119.6 (4)	C27—C26A—C26	120.5 (4)
C7—C8—H8A	120.2	C30A—C26A—C26	120.4 (5)
C9—C8—H8A	120.2	C28—C27—C26A	119.1 (4)
N10—C9—C8	122.4 (4)	C28—C27—H27A	120.4
N10—C9—H9A	118.8	C26A—C27—H27A	120.4
C8—C9—H9A	118.8	C27—C28—C29	117.4 (6)
C9—N10—C10A	118.1 (4)	C27—C28—H28A	121.3
C9—N10—Cu1	128.4 (3)	C29—C28—H28A	121.3
C10A—N10—Cu1	113.4 (3)	N30—C29—C28	124.8 (6)
N10—C10A—C6A	122.0 (4)	N30—C29—H29A	117.6
N10—C10A—C1A	115.7 (4)	C28—C29—H29A	117.6
C6A—C10A—C1A	122.2 (4)	C30A—N30—C29	117.6 (4)
N11—C11A—C14A	121.9 (4)	C30A—N30—Cu1	111.0 (3)
N11—C11A—C20A	116.6 (4)	C29—N30—Cu1	130.4 (4)
C14A—C11A—C20A	121.5 (4)	N30—C30A—C26A	121.9 (5)
C11A—N11—C12	118.7 (3)	N30—C30A—C21A	117.1 (4)
C11A—N11—Cu1	111.8 (2)	C26A—C30A—C21A	121.0 (5)
C12—N11—Cu1	129.5 (3)	O3—Cl1—O2	109.71 (18)
N11—C12—C13	122.5 (4)	O3—Cl1—O4	110.2 (2)
N11—C12—H12A	118.7	O2—Cl1—O4	109.14 (19)
C13—C12—H12A	118.7	O3—Cl1—O1	109.5 (2)

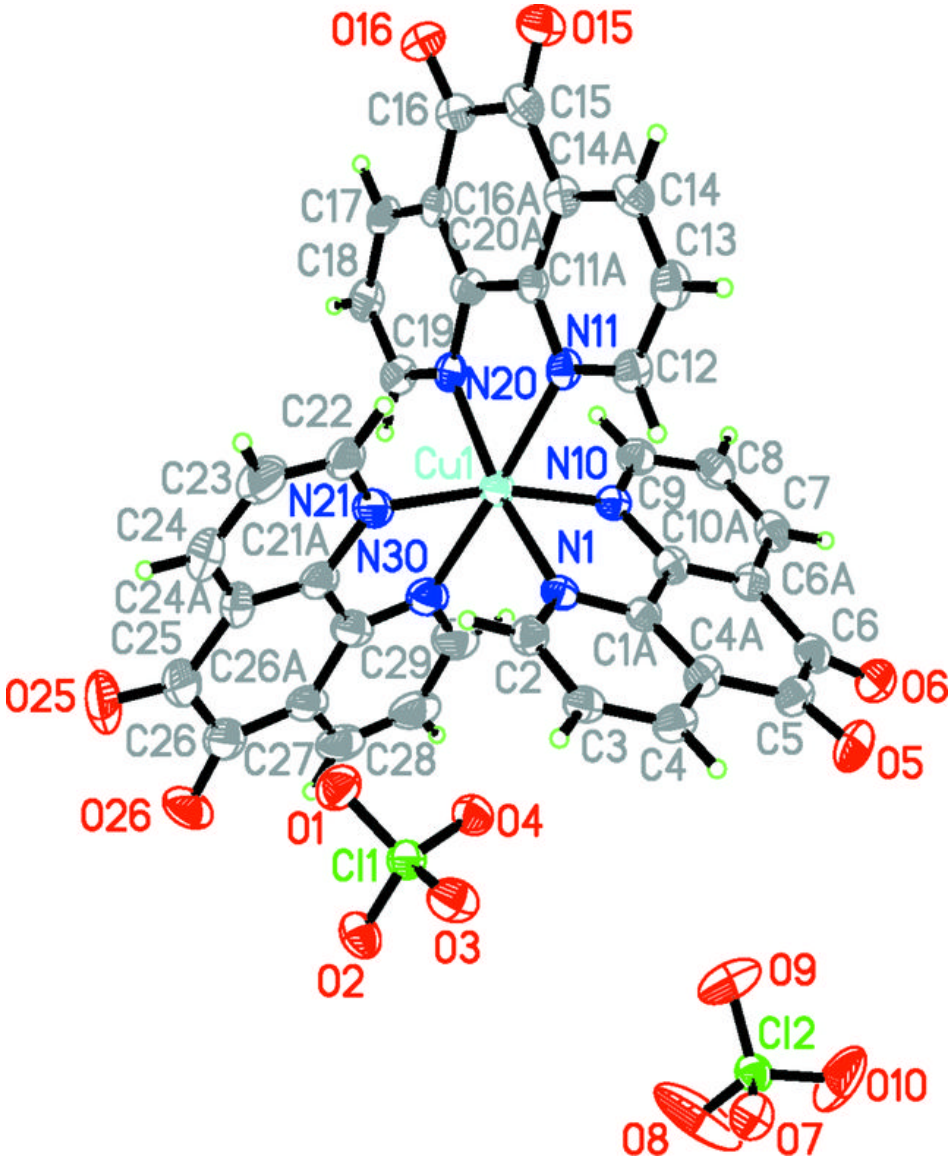
C14—C13—C12	119.0 (4)	O2—Cl1—O1	109.4 (2)
C14—C13—H13A	120.5	O4—Cl1—O1	108.86 (16)
C12—C13—H13A	120.5	O8—Cl2—O9	109.8 (3)
C13—C14—C14A	118.6 (4)	O8—Cl2—O10	110.8 (4)
C13—C14—H14A	120.7	O9—Cl2—O10	108.8 (3)
C14A—C14—H14A	120.7	O8—Cl2—O7	109.8 (2)
C11A—C14A—C14	119.2 (4)	O9—Cl2—O7	110.3 (2)
C11A—C14A—C15	120.4 (4)	O10—Cl2—O7	107.3 (2)
C14—C14A—C15	120.4 (4)	H51A—O51—H51B	108.5
O15—C15—C14A	122.9 (4)	H52A—O52—H52B	108.3
O15—C15—C16	119.1 (4)	H53A—O53—H53B	106.8
C14A—C15—C16	117.9 (4)	H54A—O54—H54B	108.2
O16—C16—C16A	123.0 (4)	H55A—O55—H55B	99.7

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O51—H51A···O10	0.98	1.99	2.937 (5)	162.8
O51—H51B···O55	0.98	2.39	3.099	129.1
O52—H52A···O15^{i}	0.98	1.87	2.810 (3)	158.6
O52—H52B···O55	0.98	1.96	2.623	123.1
O53—H53A···O2	0.98	1.99	2.956 (3)	168.3
O53—H53B···O26	0.98	2.06	2.878 (3)	139.3
O54—H54A···O53	0.98	1.90	2.824	157.1
O54—H54B···O51^{ii}	0.98	2.16	2.917	132.6
O55—H55A···O51	0.98	2.12	3.099	179.9
O55—H55B···O16^{i}	0.98	2.08	2.998 (3)	154.4

Symmetry codes: (i) −*x*+3/2, −*y*+1, *z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1


O16
O15
C16
C15
C14A
C17
C16A
C14
C20A
C13
C18
C11A
C19
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N20
C12
C22
Cu1
N10
C8
C23
N21
C9
C7
C24
C21A
C10A
N30
N1
C6A
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C25
C4A
C6
C26A
C29
C1A
C2
O25
O6
C26
C27
C28
C3
C4
C5
O5
O26
O1
O4
Cl1
O3
O2
O9
Cl2
O10
O8
O7