Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-μ-chlorido-bis[(2-anilinobenzoato-κ²O,O′)(1,10-phenanthroline-κ²N,N′)-copper(II)]

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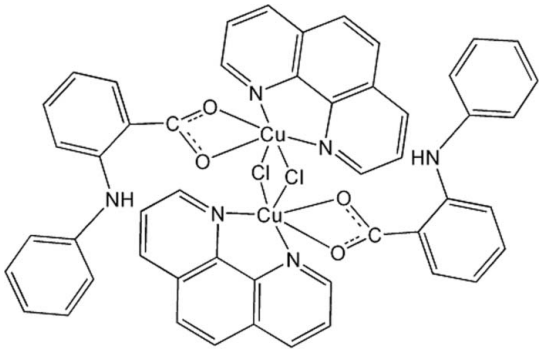
Received 16 May 2007; accepted 21 May 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.092; data-to-parameter ratio = 17.5.

In the title compound, $[Cu_2(C_{13}H_{10}NO_2)_2Cl_2(C_{12}H_8N_2)_2]$, the coordination geometry around the Cu^{II} atom is square-pyramidal, comprising one O atom from a 2-anilinobenzoate ligand, two N atoms from a 1,10-phenanthroline ligand and two Cl^- anions. In addition, there is a weak interaction of 2.700 (2) Å between the Cu^{II} atom and the other O atom of the 2-anilinobenzoate ligand. The two Cl^- anions bridge two Cu^{II} atoms to form a dimeric complex molecule, which lies on an inversion centre. There is an intramolecular N—H···O hydrogen bond in the 2-anilinobenzoate ligand.

Related literature

For a related structure, see: Atria *et al.* (1994).



Experimental

Crystal data

$[Cu_2(C_{13}H_{10}NO_2)_2Cl_2(C_{12}H_8N_2)_2]$
M_r = 982.83
Monoclinic, $P2_1/c$
a = 14.1685 (18) Å
b = 10.3545 (13) Å
c = 14.9955 (19) Å
β = 100.843 (2)°
V = 2160.7 (5) Å³
Z = 2
Mo $K\alpha$ radiation
μ = 1.16 mm⁻¹
T = 293 (2) K
0.40 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.643, T_{max} = 0.718
12962 measured reflections
5099 independent reflections
3284 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.092
S = 1.03
5099 reflections
292 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å⁻³
$\Delta\rho_{min}$ = −0.46 e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.9423 (16)	Cu1—Cl1	2.3010 (7)
Cu1—N1	2.0241 (19)	Cu1—O2	2.700 (2)
Cu1—N2	2.045 (2)	Cu1—Cl1^{i}	2.7011 (8)
O1—Cu1—N1	171.49 (8)	N2—Cu1—Cl1	170.08 (6)
O1—Cu1—N2	91.26 (8)	O1—Cu1—Cl1^{i}	94.02 (6)
O2—Cu1—N1	121.94 (6)	O2—Cu1—Cl1^{i}	147.97 (6)
O2—Cu1—N2	85.45 (8)	N1—Cu1—Cl1^{i}	89.91 (6)
N1—Cu1—N2	80.73 (8)	N2—Cu1—Cl1^{i}	97.64 (6)
O1—Cu1—Cl1	93.86 (6)	Cl1—Cu1—Cl1^{i}	90.47 (2)
O2—Cu1—Cl1	90.70 (6)	O1—Cu1—O2	53.97 (7)
N1—Cu1—Cl1	93.65 (6)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···O2	0.91 (3)	1.88 (3)	2.652 (3)	142 (3)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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*.

The authors thank the National Natural Science Foundation of China (grant No. 20471014), the Programme for New Century Excellent Talents in Chinese Universities (grant No. NCET-05-0320), the Fok Ying Tung Education Foundation and the Analysis and Testing Foundation of Northeast Normal University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2064).

References

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supplementary materials

Acta Cryst. (2007). E**63**, m1681 [**doi:10.1107/S1600536807024725**]

Di-μ-chlorido-bis[(2-anilinobenzoato-κ^2O,O')(1,10-phenanthroline-κ^2N,N')copper(II)]

H. Jiang, J.-F. Ma and W.-L. Zhang

Comment

As part of our investigation of the transition metal complexes, there is a need to prepare further examples of these compounds. In this paper, the structure of the title compound, (I), is described.

As shown in Fig. 1, the coordination polyhedron around the Cu^{II} atom is a square pyramid, comprising one O atom from a 2-anilinobenzoate ligand, two N atoms from a 1,10-phenanthroline ligand and two Cl^- anions. In addition, there is a weak interaction of 2.700 (2)Å between Cu1 and O2 (Table 1). The two Cl^- anions bridge two Cu^{II} atoms to form a dimeric complex molecule, which lies on an inversion center. The bond distances and angles are normal (Atria *et al.*, 1994). There exists intramolecular N—H···O hydrogen bond in (I) (Table 2).

Experimental

A mixture of $CuCl_2{\cdot}2H_2O$ (0.171 g, 1 mmol), NaOH (0.08 g, 2 mmol) and 2-anilinobenzoic acid (0.426 g, 2 mmol) in ethanol (15 ml) was stirred for 10 min at room temperature. Then 1,10-phenanthroline (0.360 g, 1 mmol) was added to the solution with stirring for 30 min and a blue precipitate was obtained. The precipitate was dissolved by dropwise addition of ammonia (5 *M*). Green single crystals were obtained by slow evaporation of the solution at room temperature.

Refinement

H atoms on C atoms were poisitioned geometrically and refined as riding atoms, with C—H = 0.93 Å, and U_{iso}(H) = 1.2U_{eq}(C). H atom bonded to N atom was located in a difference Fourier map and refined with U_{iso}(H) = 1.5U_{eq}(N).

Figures

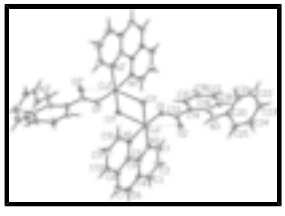


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The dashed lines denote the weak interactions between Cu1 and O2. [Symmetry code: (i) $-x+1, -y+1, -z+1$.]

Di-μ-chlorido-bis[(2-anilinobenzoato-κ$^2O,O'$)(1,10-phenanthroline- κ$^2N,N'$)copper(II)]

Crystal data

$[Cu_2(C_{13}H_{10}NO_2)_2Cl_2(C_{12}H_8N_2)_2]$ F_{000} = 1004

M_r = 982.83	D_x = 1.511 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: not measured K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.1685 (18) Å	Cell parameters from 5099 reflections
b = 10.3545 (13) Å	θ = 2.4–28.3º
c = 14.9955 (19) Å	μ = 1.16 mm^{-1}
β = 100.843 (2)º	T = 293 (2) K
V = 2160.7 (5) Å^3	Block, green
Z = 2	0.40 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	5099 independent reflections
Radiation source: fine-focus sealed tube	3284 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.041
T = 293(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −18→17
T_{min} = 0.643, T_{max} = 0.718	k = −6→13
12962 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.092	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
5099 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
292 parameters	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	U_{iso}*/U_{eq}
Cu1	0.38851 (2)	0.57806 (3)	0.46359 (2)	0.03554 (11)
C1	0.2915 (2)	0.7342 (3)	0.2981 (2)	0.0569 (8)
H1	0.2811	0.6567	0.2665	0.068*
C2	0.2601 (2)	0.8483 (4)	0.2536 (2)	0.0775 (11)
H2	0.2298	0.8463	0.1929	0.093*
C3	0.2735 (2)	0.9628 (4)	0.2984 (3)	0.0759 (10)

H3	0.2520	1.0391	0.2686	0.091*
C4	0.3194 (2)	0.9658 (3)	0.3892 (2)	0.0517 (7)
C5	0.34946 (17)	0.8467 (2)	0.42921 (18)	0.0380 (6)
C6	0.3382 (2)	1.0797 (3)	0.4440 (3)	0.0643 (9)
H6	0.3188	1.1596	0.4186	0.077*
C7	0.3832 (2)	1.0736 (3)	0.5312 (3)	0.0610 (9)
H7	0.3949	1.1495	0.5646	0.073*
C8	0.41356 (19)	0.9538 (2)	0.5737 (2)	0.0463 (7)
C9	0.39598 (17)	0.8408 (2)	0.52190 (17)	0.0361 (6)
C10	0.4697 (2)	0.7138 (3)	0.64042 (18)	0.0504 (7)
H10	0.4905	0.6333	0.6635	0.060*
C11	0.4894 (2)	0.8213 (3)	0.6963 (2)	0.0618 (9)
H11	0.5216	0.8119	0.7560	0.074*
C12	0.4614 (2)	0.9394 (3)	0.6635 (2)	0.0592 (9)
H12	0.4741	1.0116	0.7008	0.071*
C13	0.2532 (2)	0.4353 (2)	0.36398 (18)	0.0423 (6)
C14	0.20479 (18)	0.3495 (2)	0.28849 (16)	0.0378 (6)
C15	0.2531 (2)	0.3170 (2)	0.21885 (17)	0.0437 (7)
H15	0.3153	0.3474	0.2215	0.052*
C16	0.2121 (2)	0.2417 (3)	0.14645 (18)	0.0502 (7)
H16	0.2463	0.2210	0.1012	0.060*
C17	0.1199 (2)	0.1976 (3)	0.14189 (19)	0.0556 (8)
H17	0.0913	0.1468	0.0931	0.067*
C18	0.0694 (2)	0.2279 (3)	0.2089 (2)	0.0560 (8)
H18	0.0068	0.1982	0.2042	0.067*
C19	0.1104 (2)	0.3023 (2)	0.28385 (19)	0.0465 (7)
C20	−0.00863 (19)	0.2604 (3)	0.38439 (19)	0.0488 (7)
C21	−0.0202 (2)	0.1287 (3)	0.3698 (2)	0.0632 (9)
H21	0.0167	0.0857	0.3340	0.076*
C22	−0.0867 (2)	0.0609 (3)	0.4082 (2)	0.0725 (10)
H22	−0.0947	−0.0273	0.3975	0.087*
C23	−0.1407 (2)	0.1224 (4)	0.4619 (2)	0.0719 (10)
H23	−0.1853	0.0761	0.4872	0.086*
C24	−0.1289 (2)	0.2532 (3)	0.4784 (2)	0.0618 (9)
H24	−0.1643	0.2949	0.5161	0.074*
C25	−0.0646 (2)	0.3212 (3)	0.43881 (19)	0.0516 (7)
H25	−0.0583	0.4098	0.4485	0.062*
N1	0.42259 (14)	0.72155 (19)	0.55568 (13)	0.0364 (5)
N2	0.33596 (14)	0.7321 (2)	0.38417 (14)	0.0389 (5)
O1	0.34218 (12)	0.45837 (16)	0.36536 (12)	0.0441 (4)
O2	0.20849 (14)	0.48232 (18)	0.41969 (14)	0.0598 (6)
Cl1	0.43055 (5)	0.41708 (6)	0.56938 (4)	0.04377 (17)
N3	0.05935 (19)	0.3350 (2)	0.35103 (18)	0.0620 (8)
H3N	0.090 (2)	0.397 (3)	0.389 (2)	0.093*

Atomic displacement parameters (Å^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Cu1	0.04285 (19)	0.02743 (17)	0.03428 (18)	−0.00175 (14)	0.00194 (13)	−0.00253 (13)
C1	0.0589 (19)	0.058 (2)	0.0487 (18)	0.0005 (15)	−0.0022 (15)	0.0070 (15)
C2	0.081 (3)	0.091 (3)	0.051 (2)	0.012 (2)	−0.0102 (18)	0.025 (2)
C3	0.076 (2)	0.067 (2)	0.081 (3)	0.015 (2)	0.006 (2)	0.035 (2)
C4	0.0479 (17)	0.0423 (17)	0.068 (2)	0.0088 (14)	0.0174 (15)	0.0180 (15)
C5	0.0344 (14)	0.0360 (16)	0.0454 (16)	0.0014 (12)	0.0123 (12)	0.0065 (12)
C6	0.070 (2)	0.0270 (16)	0.102 (3)	0.0102 (15)	0.034 (2)	0.0123 (17)
C7	0.067 (2)	0.0284 (16)	0.096 (3)	−0.0064 (15)	0.037 (2)	−0.0112 (17)
C8	0.0443 (16)	0.0332 (15)	0.066 (2)	−0.0051 (12)	0.0230 (15)	−0.0121 (14)
C9	0.0358 (14)	0.0302 (14)	0.0448 (15)	−0.0009 (11)	0.0135 (12)	−0.0032 (11)
C10	0.0628 (19)	0.0461 (17)	0.0387 (16)	0.0081 (14)	0.0005 (14)	−0.0069 (13)
C11	0.072 (2)	0.068 (2)	0.0414 (17)	0.0049 (18)	0.0004 (15)	−0.0214 (16)
C12	0.061 (2)	0.056 (2)	0.061 (2)	−0.0064 (16)	0.0123 (16)	−0.0331 (16)
C13	0.0507 (17)	0.0294 (14)	0.0457 (16)	−0.0048 (13)	0.0060 (13)	−0.0034 (12)
C14	0.0416 (15)	0.0302 (14)	0.0390 (15)	−0.0044 (11)	0.0009 (12)	−0.0004 (11)
C15	0.0470 (16)	0.0391 (16)	0.0428 (16)	−0.0031 (13)	0.0031 (13)	0.0001 (13)
C16	0.063 (2)	0.0471 (17)	0.0391 (16)	−0.0008 (15)	0.0070 (14)	−0.0035 (13)
C17	0.067 (2)	0.0500 (18)	0.0421 (17)	−0.0128 (15)	−0.0092 (15)	−0.0066 (14)
C18	0.0500 (18)	0.060 (2)	0.0540 (19)	−0.0205 (15)	0.0002 (15)	−0.0107 (15)
C19	0.0496 (17)	0.0396 (16)	0.0496 (17)	−0.0097 (13)	0.0078 (14)	−0.0030 (13)
C20	0.0397 (16)	0.0508 (18)	0.0544 (18)	−0.0098 (14)	0.0056 (13)	−0.0066 (14)
C21	0.064 (2)	0.0524 (19)	0.081 (2)	−0.0103 (16)	0.0342 (18)	−0.0120 (17)
C22	0.079 (2)	0.051 (2)	0.091 (3)	−0.0184 (18)	0.026 (2)	−0.0093 (18)
C23	0.058 (2)	0.081 (3)	0.084 (3)	−0.0147 (19)	0.0313 (19)	0.001 (2)
C24	0.051 (2)	0.072 (2)	0.064 (2)	0.0018 (17)	0.0158 (16)	−0.0056 (17)
C25	0.0470 (17)	0.0515 (18)	0.0541 (18)	−0.0023 (14)	0.0039 (14)	−0.0076 (14)
N1	0.0443 (13)	0.0316 (12)	0.0330 (12)	0.0019 (10)	0.0060 (10)	−0.0032 (9)
N2	0.0399 (12)	0.0388 (13)	0.0358 (12)	−0.0010 (10)	0.0016 (10)	0.0026 (10)
O1	0.0398 (11)	0.0433 (11)	0.0462 (11)	−0.0080 (8)	0.0009 (8)	−0.0105 (8)
O2	0.0577 (13)	0.0571 (13)	0.0682 (14)	−0.0152 (10)	0.0209 (11)	−0.0282 (11)
Cl1	0.0519 (4)	0.0330 (3)	0.0466 (4)	−0.0034 (3)	0.0096 (3)	0.0086 (3)
N3	0.0609 (17)	0.0593 (18)	0.0713 (19)	−0.0270 (14)	0.0264 (14)	−0.0223 (14)

Geometric parameters (Å, °)

Cu1—O1	1.9423 (16)	C12—H12	0.9300
Cu1—N1	2.0241 (19)	C13—O2	1.239 (3)
Cu1—N2	2.045 (2)	C13—O1	1.279 (3)
Cu1—Cl1	2.3010 (7)	C13—C14	1.500 (3)
Cu1—O2	2.700 (2)	C14—C15	1.393 (3)
Cu1—Cl1^{i}	2.7011 (8)	C14—C19	1.413 (3)
C1—N2	1.326 (3)	C15—C16	1.373 (3)
C1—C2	1.388 (4)	C15—H15	0.9300
C1—H1	0.9300	C16—C17	1.374 (4)
C2—C3	1.359 (5)	C16—H16	0.9300
C2—H2	0.9300	C17—C18	1.374 (4)
C3—C4	1.395 (4)	C17—H17	0.9300
C3—H3	0.9300	C18—C19	1.396 (4)
C4—C5	1.402 (3)	C18—H18	0.9300

C4—C6	1.433 (4)	C19—N3	1.388 (3)
C5—N2	1.361 (3)	C20—C21	1.386 (4)
C5—C9	1.423 (3)	C20—C25	1.391 (4)
C6—C7	1.345 (4)	C20—N3	1.399 (3)
C6—H6	0.9300	C21—C22	1.384 (4)
C7—C8	1.424 (4)	C21—H21	0.9300
C7—H7	0.9300	C22—C23	1.367 (4)
C8—C12	1.396 (4)	C22—H22	0.9300
C8—C9	1.400 (3)	C23—C24	1.382 (4)
C9—N1	1.361 (3)	C23—H23	0.9300
C10—N1	1.322 (3)	C24—C25	1.372 (4)
C10—C11	1.389 (4)	C24—H24	0.9300
C10—H10	0.9300	C25—H25	0.9300
C11—C12	1.350 (4)	Cl1—Cu1^{i}	2.7011 (8)
C11—H11	0.9300	N3—H3N	0.91 (3)
O1—Cu1—N1	171.49 (8)	O2—C13—O1	122.9 (2)
O1—Cu1—N2	91.26 (8)	O2—C13—C14	121.5 (2)
O2—Cu1—N1	121.94 (6)	O1—C13—C14	115.6 (2)
O2—Cu1—N2	85.45 (8)	C15—C14—C19	118.3 (2)
N1—Cu1—N2	80.73 (8)	C15—C14—C13	119.3 (2)
O1—Cu1—Cl1	93.86 (6)	C19—C14—C13	122.4 (2)
O2—Cu1—Cl1	90.70 (6)	C16—C15—C14	122.4 (3)
N1—Cu1—Cl1	93.65 (6)	C16—C15—H15	118.8
N2—Cu1—Cl1	170.08 (6)	C14—C15—H15	118.8
O1—Cu1—Cl1^{i}	94.02 (6)	C15—C16—C17	119.0 (3)
O2—Cu1—Cl1^{i}	147.97 (6)	C15—C16—H16	120.5
N1—Cu1—Cl1^{i}	89.91 (6)	C17—C16—H16	120.5
N2—Cu1—Cl1^{i}	97.64 (6)	C18—C17—C16	120.5 (3)
Cl1—Cu1—Cl1^{i}	90.47 (2)	C18—C17—H17	119.7
O1—Cu1—O2	53.97 (7)	C16—C17—H17	119.7
N2—C1—C2	122.2 (3)	C17—C18—C19	121.4 (3)
N2—C1—H1	118.9	C17—C18—H18	119.3
C2—C1—H1	118.9	C19—C18—H18	119.3
C3—C2—C1	120.1 (3)	N3—C19—C18	121.7 (3)
C3—C2—H2	119.9	N3—C19—C14	119.8 (2)
C1—C2—H2	119.9	C18—C19—C14	118.5 (3)
C2—C3—C4	119.9 (3)	C21—C20—C25	118.3 (3)
C2—C3—H3	120.0	C21—C20—N3	123.8 (3)
C4—C3—H3	120.0	C25—C20—N3	117.8 (3)
C3—C4—C5	116.5 (3)	C22—C21—C20	120.2 (3)
C3—C4—C6	125.4 (3)	C22—C21—H21	119.9
C5—C4—C6	118.0 (3)	C20—C21—H21	119.9
N2—C5—C4	123.5 (3)	C23—C22—C21	120.6 (3)
N2—C5—C9	116.3 (2)	C23—C22—H22	119.7
C4—C5—C9	120.2 (2)	C21—C22—H22	119.7
C7—C6—C4	121.4 (3)	C22—C23—C24	120.0 (3)
C7—C6—H6	119.3	C22—C23—H23	120.0

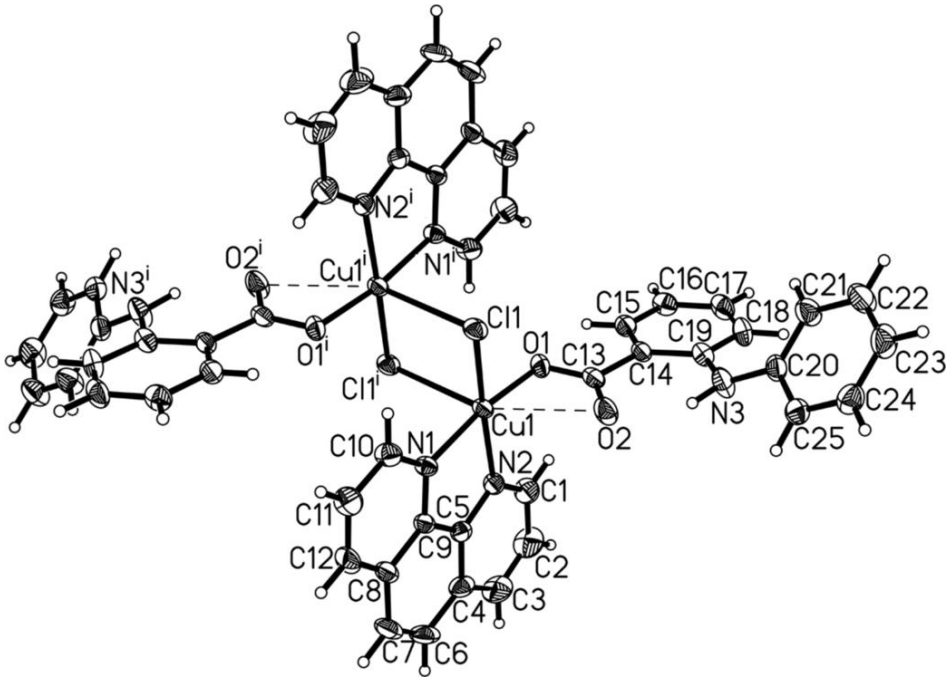
C4—C6—H6	119.3	C24—C23—H23	120.0
C6—C7—C8	121.6 (3)	C25—C24—C23	119.5 (3)
C6—C7—H7	119.2	C25—C24—H24	120.2
C8—C7—H7	119.2	C23—C24—H24	120.2
C12—C8—C9	116.7 (3)	C24—C25—C20	121.3 (3)
C12—C8—C7	125.1 (3)	C24—C25—H25	119.3
C9—C8—C7	118.2 (3)	C20—C25—H25	119.3
N1—C9—C8	122.9 (2)	C10—N1—C9	117.8 (2)
N1—C9—C5	116.6 (2)	C10—N1—Cu1	128.62 (18)
C8—C9—C5	120.5 (2)	C9—N1—Cu1	113.47 (16)
N1—C10—C11	122.6 (3)	C1—N2—C5	117.7 (2)
N1—C10—H10	118.7	C1—N2—Cu1	129.35 (19)
C11—C10—H10	118.7	C5—N2—Cu1	112.90 (16)
C12—C11—C10	119.6 (3)	C13—O1—Cu1	108.82 (16)
C12—C11—H11	120.2	Cu1—Cl1—Cu1^{i}	89.53 (2)
C10—C11—H11	120.2	C19—N3—C20	127.8 (2)
C11—C12—C8	120.3 (3)	C19—N3—H3N	112 (2)
C11—C12—H12	119.9	C20—N3—H3N	116 (2)
C8—C12—H12	119.9		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···O2	0.91 (3)	1.88 (3)	2.652 (3)	142 (3)

Fig. 1


N2i
O2i
Cu1i
N1i
N3i
O1i
Cl1
Cl1i
O1
C13
C14
C15
C16
C17
C18
C19
C20
C21
C22
C23
C24
C25
N3
O2
Cu1
C10
N1
N2
C1
C11
C5
C9
C12
C8
C2
C4
C3
C7
C6