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Bis[μ-2,4-dibromo-6-(2-pyridylmethylaminomethyl)phenolato]bis[nitratocopper(II)]

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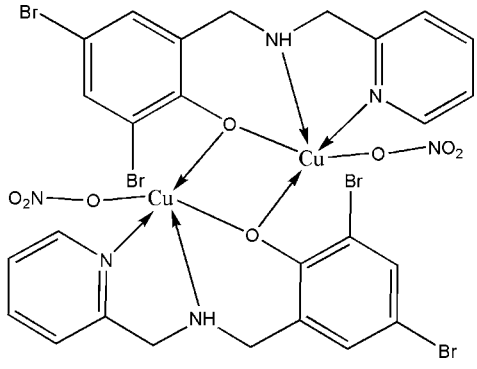
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.005 Å; R factor = 0.034; wR factor = 0.071; data-to-parameter ratio = 17.1.

In the centrosymmetric binuclear title compound, $[Cu_2(C_{13}H_{11}Br_2N_2O)_2(NO_3)_2]$, each Cu^{II} atom is five-coordinated by two phenolate O atoms, one nitrate O atom, one pyridine N atom and one amine N atom in a distorted trigonal–bipyramidal CuO_3N_2 environment. The Cu···Cu separation is 3.207 (4) Å.

Related literature

For related literature, see: Gavrilova & Bosnich (2004); Solomon *et al.* (1996); Collman, Zhong, Zhang & Costanzo (2001); Collman, Zhong, Zeng & Costanzo (2001); Xu *et al.* (2005); Yang *et al.* (2006).



Experimental

Crystal data

$[Cu_2(C_{13}H_{11}Br_2N_2O)_2(NO_3)_2]$
M_r = 993.22
Monoclinic, $P2_1/n$
a = 10.385 (5) Å
b = 11.252 (5) Å
c = 14.074 (5) Å
β = 104.810 (5)°
V = 1589.9 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.42 mm^{-1}
T = 290 (2) K
0.24 × 0.16 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.30, T_{max} = 0.43
14979 measured reflections
3629 independent reflections
2707 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.071
S = 1.05
3629 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—O1	1.923 (2)	Cu1—O2	2.038 (2)
Cu1—N1	1.985 (3)	Cu1—O1^{i}	2.243 (2)
Cu1—N2	2.021 (3)		

Symmetry code: (i) $-x+2, -y, -z+1$.

Data collection: *PROCESS-AUTO* (Rigaku, 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: HB2500).

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supplementary materials

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Bis[μ-2,4-dibromo-6-(2-pyridylmethylaminomethyl)phenolato]bis[nitratocopper(II)]

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Comment

The synthesis and characterization of binuclear copper(II) compounds have received increasing interest in bio-inorganic chemistry (Solomon *et al.*, 1996) and catalysis (Collman, Zhong, Zhang & Costanzo, 2001; Collman, Zhong, Zeng & Costanzo, 2001). It is well known that the phenolate anion is a good bridging ligand for the construction of binuclear transition metal compounds (Gavrilova & Bosnich 2004; Yang *et al.*, 2006). Modification of the phenolate anion in its *ortho* position can lead to the formation of stable binuclear compounds through chelation (Xu, *et al.*, 2005). The structure of a new binuclear complex $[Cu_2(dmp)_2(NO_3)_2]$, (I), where dmp is 2,4-dibromo-6-((pyridine-2-ylmethylamino)methyl)phenol, is presented here.

As shown in Fig. 1, the title compound, $[Cu_2(C_{13}H_{11}Br_2N_2O)(NO_3)_2]$, contains a binuclear copper(II) unit bridged by two phenolate O atoms with a Cu···Cu distance of 3.207 (4) Å. Each Cu^{II} atom is chelated by one dmp ligand and is also coordinated by a nitrate O atom. Finally, the trigonal-bipyramidal coordination environment is completed by bond to the phenolate O atom from another dmp ligand. The axial positions of the trigonal-bipyramid are occupied by one phenolate O atom and one pyridine N atom with the O—Cu—N angle being 171.25 (10)°.

Experimental

2,4-Dibromo-6-((pyridine-2-ylmethylamino)methyl)phenol (0.372 g, 1 mmol) was added to a methanol solution (20 ml) of $Cu(NO_3)_2{\cdot}3H_2O$ (0.241 g, 1 mmol) with stirring. The resulting solution was left to stand at room temperature and green crystals of (I) were obtained after several days.

Refinement

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

Figures

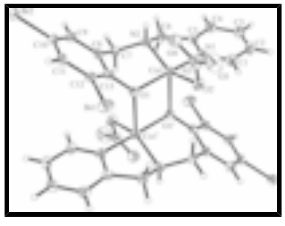


Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level (arbitrary spheres for the H atoms). Symmetry code: (i) $2-x, -y, 1-z$.

Bis[μ-2,4-dibromo-6-(2-pyridylmethylaminomethyl)phenolato]\ bis[nitratocopper(II)]

Crystal data

$[Cu_2(C_{13}H_{11}Br_2N_2O)_2(NO_3)_2]$	F_{000} = 964
M_r = 993.22	D_x = 2.075 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2yn	Cell parameters from 11121 reflections
a = 10.385 (5) Å	θ = 3.0–27.5º
b = 11.252 (5) Å	μ = 6.42 mm^{-1}
c = 14.074 (5) Å	T = 290 (2) K
β = 104.810 (5)º	Block, green
V = 1589.9 (12) Å^3	0.24 × 0.16 × 0.13 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	3629 independent reflections
Radiation source: rotating anode	2707 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.054
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.5º
T = 290(2) K	θ_{min} = 3º
ω scans	h = −13→13
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	k = −14→14
T_{min} = 0.30, T_{max} = 0.43	l = −15→18
14979 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.071	$w = 1/[\sigma^2(F_o^2) + (0.0224P)^2 + 0.8145P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.007
3629 reflections	$\Delta\rho_{max}$ = 0.48 e Å^{-3}
212 parameters	$\Delta\rho_{min}$ = −0.49 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}
Cu1	0.93487 (4)	0.01290 (3)	0.38420 (3)	0.03277 (11)
C1	0.9741 (4)	0.2204 (3)	0.2616 (3)	0.0414 (8)
H1	0.9214	0.2636	0.2936	0.050*
C2	1.0259 (4)	0.2770 (3)	0.1930 (3)	0.0461 (9)
H2	1.0085	0.3570	0.1789	0.055*
C3	1.1040 (4)	0.2130 (3)	0.1454 (3)	0.0444 (9)
H3	1.1398	0.2492	0.0986	0.053*
C4	1.1286 (4)	0.0938 (3)	0.1685 (3)	0.0432 (9)
H4	1.1806	0.0489	0.1370	0.052*
C5	1.0746 (3)	0.0432 (3)	0.2387 (2)	0.0340 (7)
C6	1.1035 (4)	−0.0829 (3)	0.2745 (3)	0.0416 (8)
H6A	1.1081	−0.1336	0.2198	0.050*
H6B	1.1891	−0.0858	0.3225	0.050*
C7	1.0454 (3)	−0.2280 (3)	0.3883 (2)	0.0352 (8)
H7A	1.1168	−0.2020	0.4432	0.042*
H7B	1.0793	−0.2915	0.3547	0.042*
C8	0.9315 (3)	−0.2731 (3)	0.4252 (2)	0.0314 (7)
C9	0.8915 (4)	−0.3920 (3)	0.4112 (2)	0.0374 (8)
H9	0.9362	−0.4439	0.3794	0.045*
C10	0.7862 (4)	−0.4316 (3)	0.4448 (2)	0.0377 (8)
C11	0.7165 (4)	−0.3581 (3)	0.4914 (3)	0.0394 (8)
H11	0.6448	−0.3865	0.5131	0.047*
C12	0.7563 (3)	−0.2396 (3)	0.5055 (2)	0.0344 (7)
C13	0.8643 (3)	−0.1944 (3)	0.4740 (2)	0.0314 (7)
N1	0.9972 (3)	0.1047 (2)	0.2840 (2)	0.0343 (6)
N2	1.0003 (3)	−0.1265 (2)	0.3190 (2)	0.0326 (6)
N3	0.6808 (3)	0.0902 (3)	0.3379 (2)	0.0450 (7)
O1	0.9028 (2)	−0.08086 (17)	0.49007 (15)	0.0322 (5)
O2	0.7906 (3)	0.13349 (19)	0.38969 (19)	0.0425 (6)
O3	0.5783 (3)	0.1497 (3)	0.3244 (2)	0.0759 (9)
O4	0.6836 (3)	−0.0106 (2)	0.3020 (2)	0.0554 (7)
Br1	0.65460 (4)	−0.13772 (4)	0.56476 (3)	0.05240 (12)
Br2	0.73308 (4)	−0.59426 (3)	0.42370 (3)	0.04830 (12)

H12	0.925 (4)	−0.148 (3)	0.276 (3)	0.063 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0386 (2)	0.02697 (19)	0.0346 (2)	0.00306 (17)	0.01283 (18)	−0.00048 (15)
C1	0.045 (2)	0.0328 (17)	0.046 (2)	0.0052 (17)	0.0120 (17)	0.0002 (15)
C2	0.050 (2)	0.0345 (18)	0.053 (2)	−0.0009 (17)	0.0119 (19)	0.0097 (16)
C3	0.046 (2)	0.0450 (19)	0.045 (2)	−0.0064 (18)	0.0167 (18)	0.0034 (16)
C4	0.048 (2)	0.0435 (19)	0.042 (2)	−0.0010 (18)	0.0188 (18)	−0.0030 (15)
C5	0.0352 (19)	0.0360 (17)	0.0312 (19)	−0.0010 (15)	0.0090 (15)	−0.0030 (13)
C6	0.045 (2)	0.0329 (17)	0.051 (2)	0.0065 (16)	0.0208 (18)	0.0013 (15)
C7	0.041 (2)	0.0278 (15)	0.036 (2)	0.0064 (15)	0.0079 (15)	0.0016 (13)
C8	0.0329 (18)	0.0285 (15)	0.0303 (18)	0.0021 (14)	0.0034 (14)	0.0039 (12)
C9	0.043 (2)	0.0276 (16)	0.039 (2)	0.0022 (16)	0.0068 (16)	−0.0019 (14)
C10	0.047 (2)	0.0260 (15)	0.0346 (19)	−0.0049 (16)	0.0008 (16)	0.0031 (13)
C11	0.040 (2)	0.0364 (18)	0.038 (2)	−0.0081 (16)	0.0044 (16)	0.0049 (14)
C12	0.0349 (19)	0.0359 (17)	0.0319 (19)	0.0042 (15)	0.0074 (15)	−0.0014 (13)
C13	0.0328 (18)	0.0306 (16)	0.0273 (18)	0.0009 (14)	0.0015 (14)	0.0005 (12)
N1	0.0351 (16)	0.0331 (14)	0.0340 (16)	0.0030 (13)	0.0073 (12)	−0.0016 (11)
N2	0.0336 (16)	0.0290 (13)	0.0352 (17)	0.0028 (13)	0.0090 (13)	−0.0009 (11)
N3	0.048 (2)	0.0483 (18)	0.0425 (19)	0.0085 (17)	0.0188 (16)	0.0103 (14)
O1	0.0383 (13)	0.0278 (10)	0.0305 (12)	−0.0039 (10)	0.0091 (10)	−0.0040 (9)
O2	0.0408 (14)	0.0330 (12)	0.0528 (17)	0.0040 (11)	0.0107 (12)	−0.0054 (10)
O3	0.0446 (17)	0.101 (2)	0.086 (2)	0.0342 (18)	0.0253 (17)	0.0162 (18)
O4	0.0572 (18)	0.0503 (15)	0.0514 (17)	−0.0076 (14)	0.0007 (14)	0.0011 (12)
Br1	0.0483 (2)	0.0566 (2)	0.0579 (3)	−0.00440 (19)	0.02369 (19)	−0.01596 (18)
Br2	0.0671 (3)	0.02887 (17)	0.0439 (2)	−0.00976 (17)	0.00495 (19)	0.00350 (14)

Geometric parameters (Å, °)

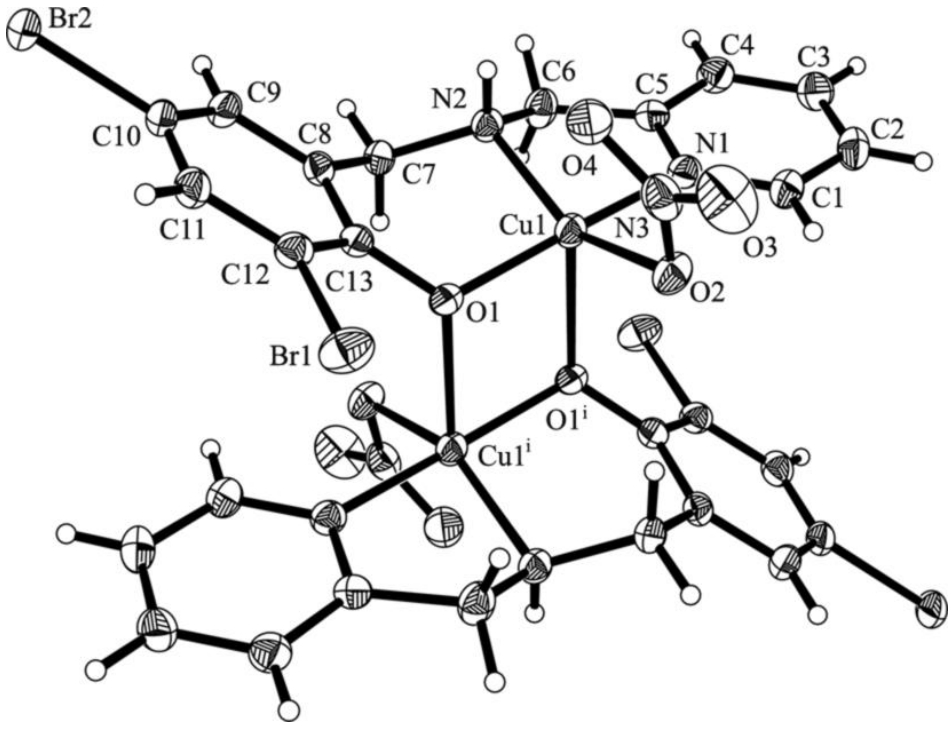
Cu1—O1	1.923 (2)	C7—C8	1.497 (4)
Cu1—N1	1.985 (3)	C7—H7A	0.9700
Cu1—N2	2.021 (3)	C7—H7B	0.9700
Cu1—O2	2.038 (2)	C8—C9	1.399 (4)
Cu1—O1^{i}	2.243 (2)	C8—C13	1.410 (4)
C1—N1	1.347 (4)	C9—C10	1.371 (5)
C1—C2	1.375 (5)	C9—H9	0.9300
C1—H1	0.9300	C10—C11	1.372 (5)
C2—C3	1.379 (5)	C10—Br2	1.913 (3)
C2—H2	0.9300	C11—C12	1.396 (4)
C3—C4	1.387 (5)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.402 (4)
C4—C5	1.378 (5)	C12—Br1	1.891 (3)
C4—H4	0.9300	C13—O1	1.341 (3)
C5—N1	1.339 (4)	N2—H12	0.90 (4)
C5—C6	1.509 (4)	N3—O3	1.230 (4)
C6—N2	1.457 (4)	N3—O4	1.245 (4)

C6—H6A	0.9700	N3—O2	1.282 (4)
C6—H6B	0.9700	O1—Cu1^{i}	2.243 (2)
C7—N2	1.497 (4)		
O1—Cu1—N1	171.25 (10)	H7A—C7—H7B	108.2
O1—Cu1—N2	94.09 (10)	C9—C8—C13	120.2 (3)
N1—Cu1—N2	82.67 (11)	C9—C8—C7	120.5 (3)
O1—Cu1—O2	93.51 (10)	C13—C8—C7	119.3 (3)
N1—Cu1—O2	93.27 (11)	C10—C9—C8	119.7 (3)
N2—Cu1—O2	150.47 (12)	C10—C9—H9	120.1
O1—Cu1—O1^{i}	79.63 (9)	C8—C9—H9	120.1
N1—Cu1—O1^{i}	93.86 (10)	C9—C10—C11	122.3 (3)
N2—Cu1—O1^{i}	110.53 (11)	C9—C10—Br2	118.8 (3)
O2—Cu1—O1^{i}	98.90 (9)	C11—C10—Br2	119.0 (3)
N1—C1—C2	122.3 (3)	C10—C11—C12	118.1 (3)
N1—C1—H1	118.9	C10—C11—H11	121.0
C2—C1—H1	118.9	C12—C11—H11	121.0
C1—C2—C3	118.9 (3)	C11—C12—C13	122.2 (3)
C1—C2—H2	120.6	C11—C12—Br1	117.6 (3)
C3—C2—H2	120.6	C13—C12—Br1	120.1 (2)
C2—C3—C4	119.1 (3)	O1—C13—C12	121.4 (3)
C2—C3—H3	120.4	O1—C13—C8	121.1 (3)
C4—C3—H3	120.4	C12—C13—C8	117.5 (3)
C5—C4—C3	118.9 (3)	C5—N1—C1	118.7 (3)
C5—C4—H4	120.6	C5—N1—Cu1	114.4 (2)
C3—C4—H4	120.6	C1—N1—Cu1	126.8 (2)
N1—C5—C4	122.1 (3)	C6—N2—C7	113.3 (3)
N1—C5—C6	114.8 (3)	C6—N2—Cu1	107.64 (19)
C4—C5—C6	123.1 (3)	C7—N2—Cu1	112.4 (2)
N2—C6—C5	110.8 (3)	C6—N2—H12	114 (3)
N2—C6—H6A	109.5	C7—N2—H12	108 (3)
C5—C6—H6A	109.5	Cu1—N2—H12	100 (2)
N2—C6—H6B	109.5	O3—N3—O4	122.8 (4)
C5—C6—H6B	109.5	O3—N3—O2	119.3 (3)
H6A—C6—H6B	108.1	O4—N3—O2	117.9 (3)
N2—C7—C8	110.0 (3)	C13—O1—Cu1	119.73 (19)
N2—C7—H7A	109.7	C13—O1—Cu1^{i}	126.11 (18)
C8—C7—H7A	109.7	Cu1—O1—Cu1^{i}	100.37 (9)
N2—C7—H7B	109.7	N3—O2—Cu1	106.3 (2)
C8—C7—H7B	109.7		
N1—C1—C2—C3	0.0 (6)	O1^{i}—Cu1—N1—C5	96.5 (2)
C1—C2—C3—C4	−0.3 (6)	N2—Cu1—N1—C1	169.3 (3)
C2—C3—C4—C5	−0.4 (5)	O2—Cu1—N1—C1	18.7 (3)
C3—C4—C5—N1	1.3 (5)	O1^{i}—Cu1—N1—C1	−80.5 (3)
C3—C4—C5—C6	−175.3 (3)	C5—C6—N2—C7	−158.1 (3)
N1—C5—C6—N2	24.0 (4)	C5—C6—N2—Cu1	−33.1 (3)
C4—C5—C6—N2	−159.2 (3)	C8—C7—N2—C6	−176.0 (3)
N2—C7—C8—C9	122.3 (3)	C8—C7—N2—Cu1	61.6 (3)

N2—C7—C8—C13	−57.5 (4)	O1—Cu1—N2—C6	−146.0 (2)
C13—C8—C9—C10	0.4 (5)	N1—Cu1—N2—C6	25.8 (2)
C7—C8—C9—C10	−179.4 (3)	O2—Cu1—N2—C6	109.5 (3)
C8—C9—C10—C11	0.6 (5)	$O1^{i}$—Cu1—N2—C6	−65.6 (2)
C8—C9—C10—Br2	179.5 (2)	O1—Cu1—N2—C7	−20.5 (2)
C9—C10—C11—C12	−0.6 (5)	N1—Cu1—N2—C7	151.3 (2)
Br2—C10—C11—C12	−179.6 (2)	O2—Cu1—N2—C7	−125.1 (2)
C10—C11—C12—C13	−0.3 (5)	$O1^{i}$—Cu1—N2—C7	59.9 (2)
C10—C11—C12—Br1	177.3 (3)	C12—C13—O1—Cu1	−132.0 (3)
C11—C12—C13—O1	−178.5 (3)	C8—C13—O1—Cu1	48.3 (4)
Br1—C12—C13—O1	3.9 (4)	C12—C13—O1—$Cu1^{i}$	95.7 (3)
C11—C12—C13—C8	1.2 (5)	C8—C13—O1—$Cu1^{i}$	−84.0 (3)
Br1—C12—C13—C8	−176.4 (2)	N2—Cu1—O1—C13	−32.5 (2)
C9—C8—C13—O1	178.5 (3)	O2—Cu1—O1—C13	119.0 (2)
C7—C8—C13—O1	−1.7 (4)	$O1^{i}$—Cu1—O1—C13	−142.6 (3)
C9—C8—C13—C12	−1.2 (5)	N2—Cu1—O1—$Cu1^{i}$	110.14 (11)
C7—C8—C13—C12	178.6 (3)	O2—Cu1—O1—$Cu1^{i}$	−98.42 (10)
C4—C5—N1—C1	−1.5 (5)	$O1^{i}$—Cu1—O1—$Cu1^{i}$	0.0
C6—C5—N1—C1	175.3 (3)	O3—N3—O2—Cu1	−175.6 (3)
C4—C5—N1—Cu1	−178.7 (3)	O4—N3—O2—Cu1	3.1 (3)
C6—C5—N1—Cu1	−1.9 (4)	O1—Cu1—O2—N3	−84.6 (2)
C2—C1—N1—C5	0.9 (5)	N1—Cu1—O2—N3	101.0 (2)
C2—C1—N1—Cu1	177.7 (3)	N2—Cu1—O2—N3	20.1 (3)
N2—Cu1—N1—C5	−13.7 (2)	$O1^{i}$—Cu1—O2—N3	−164.6 (2)
O2—Cu1—N1—C5	−164.4 (2)		

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

Fig. 1


Br2
C9
C10
C8
C7
N2
C6
C5
C4
C3
C2
C1
N1
O4
O3
N3
Cu1
O2
C11
C12
C13
O1
Br1
O1i
Cu1i