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Di-μ-azido-κ^4N:N-bis({2-[(3-amino-2,2-dimethylpropyl)iminomethyl]-6-methoxyphenolato-1κ^3N,N',O^1}copper(II))

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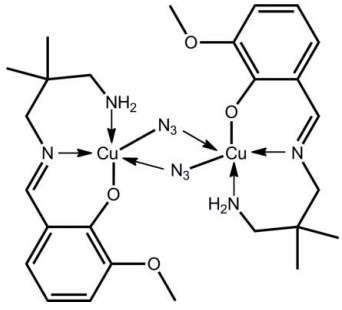
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 16.7.

The complete molecule of the title complex, $[Cu_2(C_{13}H_{19}N_2O_2)_2(N_3)_2]$, is generated by the application of a centre of inversion. The central Cu_2N_2 core is a rhombus as the μ_2-azide ligands bridge in an asymmetric fashion. Each Cu^{II} atom is also coordinated by a monoanionic tridentate Schiff base ligand *via* the anticipated oxide O, imine N and amine N atoms. The resulting N_4O coordination geometry is based on a square pyramid. No specific intermolecular interactions are noted in the crystal packing, but the amine H atoms form intramolecular N—H···O(oxide)/N(azide) hydrogen bonds.

Related literature

For background to azido derivatives of tridentate Schiff base copper(II) structures, see: Adhikary & Koner (2010). For a related structure, see: Ghaemi *et al.* (2012). For additional structural analysis, see: Addison *et al.* (1984).



Experimental

Crystal data

$[Cu_2(C_{13}H_{19}N_2O_2)_2(N_3)_2]$
M_r = 681.76
Monoclinic, $P2_1/c$
a = 9.1733 (5) Å
b = 12.2369 (5) Å
c = 13.0988 (6) Å
β = 98.203 (5)°
V = 1455.33 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.51 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.674, T_{max} = 1.000
5747 measured reflections
3314 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.06
3314 reflections
198 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu—O2	1.9047 (18)	Cu—N3	2.023 (2)
Cu—N1	1.960 (2)	Cu—N3^{i}	2.641 (2)
Cu—N2	2.001 (2)		

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
N2—H1N···O2^{i}	0.87 (1)	2.35 (2)	2.956 (3)	127 (2)
N2—H2N···N3	0.88 (1)	2.36 (3)	2.752 (3)	107 (3)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5249).

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supplementary materials

Acta Cryst. (2012). E**68**, m993–m994 [doi:10.1107/S1600536812028954]

Di-μ-azido-κ^4N:N-bis({2-[(3-amino-2,2-dimethylpropyl)iminomethyl]-6-methoxyphenolato-1κ^3N,N',O^1}copper(II))

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Comment

Azido-bridged copper(II) complexes continue to attract attention in relation to investigations of small molecule activation of copper-containing proteins and for new magnetic materials (Adhikary & Koner, 2010). Recently, the crystal structure of a related Ni^{II} complex was described in which the Schiff base ligand was shown to coordinate in two distinct modes, *i.e*. a tridentate mode towards one Ni^{II} atom and in a pentadentate mode, bridging two Ni^{II} atoms (Ghaemi *et al.*, 2012).

In the centrosymmetric binuclear complex (I), Fig. 1, the Cu^{II} atoms are bridged by one end of each of two μ_2-azido ligands to generate an Ni_2N_2 core with the shape of a rhombus as the bridge is asymmetric, Table 1. The coordination geometry for the Cu^{II} atom is completed by the oxido-O, imine-O and amino-N donor atoms derived from a tridentate uninegative Schiff base ligand. The N_4O donor set defines a coordination geometry close to square pyramidal. This is quantified by the value of τ = 0.12 which compares to the τ values of 0.0 and 1.0 for ideal square pyramidal and trigonal bipyramidal geometries, respectively (Addison *et al.*, 1984). The configuration is stabilized by an intramolecular N—H···O(oxido) and *N*—*H*···*N*(azido) hydrogen bonds, Table 2. Globally, molecules stack in columns aligned along the *a* axis, Fig. 2, without specific intermolecular interactions between them.

Experimental

A mixture of 2,2-dimethylpropylenediamine (0.234 g, 2.3 mmol) was added to a clear solution of $Cu(NO_3)_2.3H_2O$ (0.50 g, 2.07 mmol) dissolved in methanol (25 ml), which immediately produced an intense-blue solution. The solution was then heated to boiling and a methanolic solution of 2-hydroxy-3-methoxybenzaldehyde (0.273 g, 1.8 mmol) was added drop-wise over 2 h under refluxing conditions. Reflux was continued for another 45 min. Then an excess sodium azide (0.5 g, 7.7 mmol) dissolved in water (2 ml) was added. The precipitate was filtered and dissolved in methanol. Brown crystals were formed within a few days from the methanolic solution. Anal. Calc. for $C_{26}H_{38}Cu_2N_{10}O_4$: C, 45.81; H, 5.62; N, 20.55. Found: C, 45.77; H, 5.57; N, 20.66%. IR (KBr) [ν, cm^{-1}]: $\nu_{as}(N_3)$ 2035 *versus*, ν(C═N) 1621 s, ν(C═C) 1540 s, ν(C—O) 1224 m. *M*.pt: 476–478. Yield: 60%.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95–0.99 Å, U_{iso}(H) = 1.2–1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The amino H-atoms were located from a difference map and refined with N—H = 0.88±0.01 and with U_{iso}(H) = 1.2U_{eq}(N).

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO* (Agilent, 2010); data reduction: *CrysAlis PRO* (Agilent, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

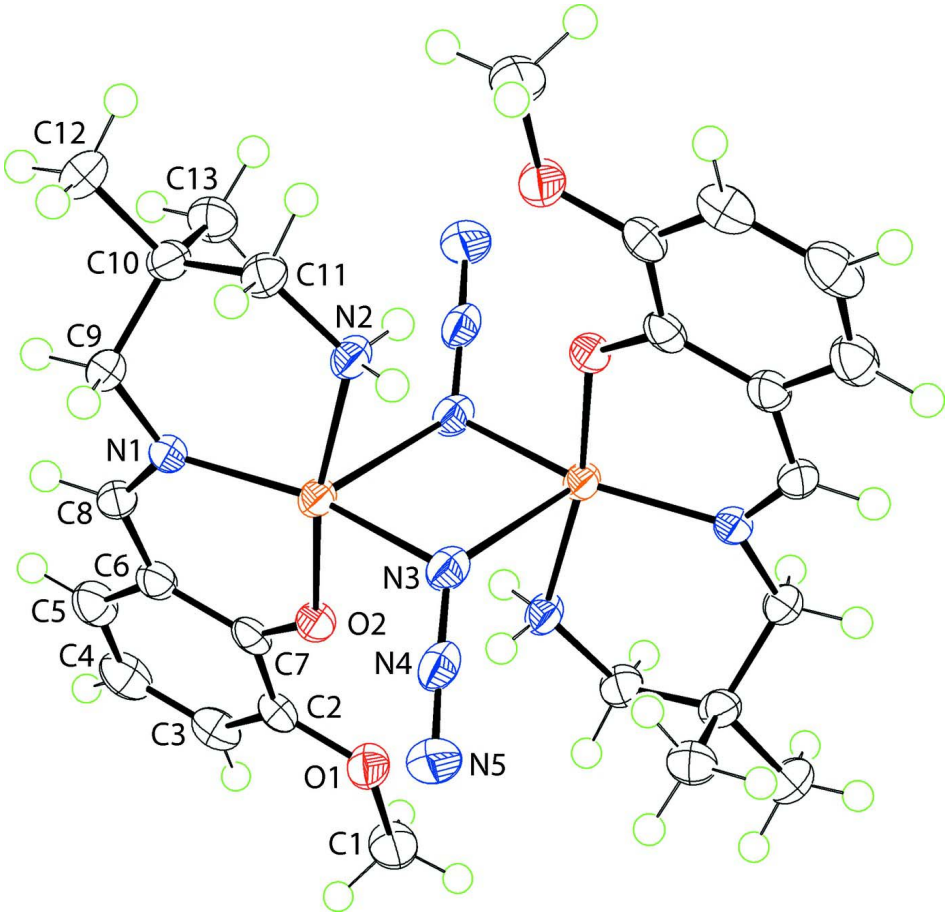

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

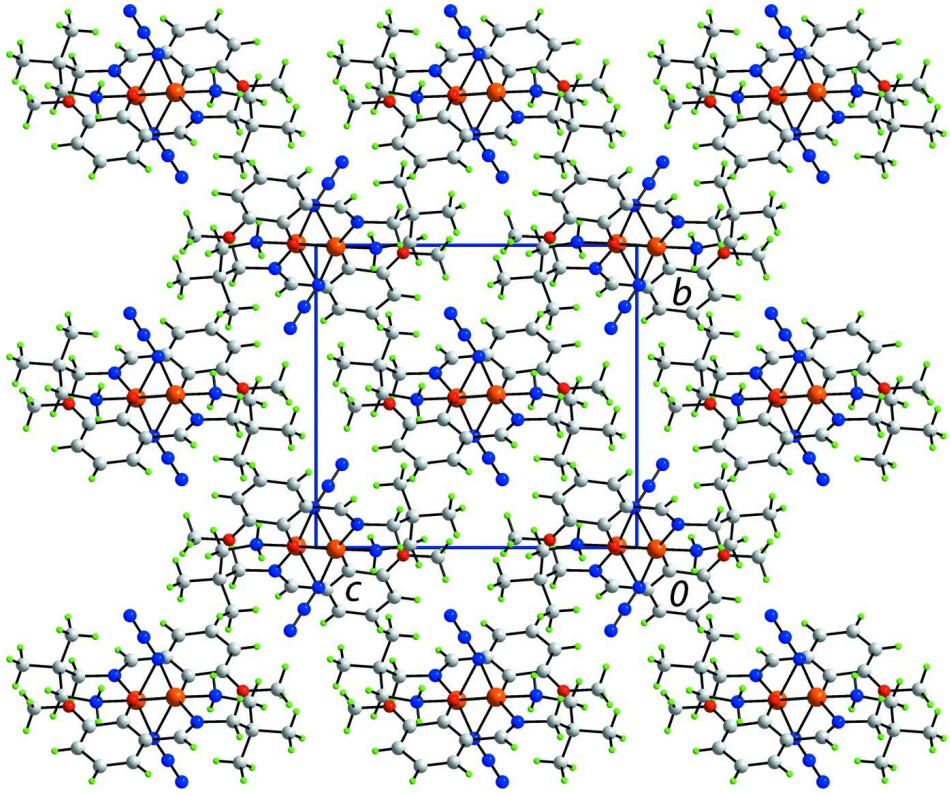

Figure 2

A view in projection down the *a* axis of the unit-cell contents of (I).

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Crystal data

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M_r = 681.76
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.1733 (5) Å
b = 12.2369 (5) Å
c = 13.0988 (6) Å
β = 98.203 (5)°
V = 1455.33 (12) Å^3
Z = 2
$F(000)$ = 708
D_x = 1.556 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2122 reflections
θ = 2.5–27.5°
μ = 1.51 mm^{-1}
T = 100 K
Prism, brown
0.20 × 0.15 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.674, T_{max} = 1.000
5747 measured reflections
3314 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.6°, θ_{min} = 2.8°
h = −8→11
k = −15→10
l = −14→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.105$
$S = 1.06$
3314 reflections
198 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.1866P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu	0.35165 (3)	0.49481 (2)	0.56108 (2)	0.01599 (12)
O1	0.1585 (2)	0.52540 (15)	0.22861 (14)	0.0225 (4)
O2	0.2389 (2)	0.50500 (12)	0.42756 (14)	0.0172 (4)
N1	0.2087 (2)	0.57697 (16)	0.62846 (16)	0.0171 (4)
N2	0.5049 (3)	0.48980 (18)	0.68604 (18)	0.0185 (5)
H1N	0.572 (2)	0.5342 (18)	0.670 (2)	0.016 (7)*
H2N	0.539 (4)	0.4236 (13)	0.679 (3)	0.056 (11)*
N3	0.4612 (2)	0.36868 (17)	0.50727 (15)	0.0191 (5)
N4	0.3900 (2)	0.29635 (18)	0.46239 (16)	0.0202 (5)
N5	0.3257 (3)	0.2250 (2)	0.4194 (2)	0.0331 (6)
C1	0.1093 (3)	0.5311 (3)	0.1204 (2)	0.0287 (6)
H1A	0.1670	0.4805	0.0842	0.043*
H1B	0.1222	0.6058	0.0960	0.043*
H1C	0.0049	0.5111	0.1066	0.043*
C2	0.0871 (3)	0.5912 (2)	0.29121 (19)	0.0184 (5)
C3	−0.0234 (3)	0.6633 (2)	0.2566 (2)	0.0217 (6)
H3	−0.0517	0.6728	0.1845	0.026*
C4	−0.0950 (3)	0.7229 (2)	0.3256 (2)	0.0264 (6)
H4	−0.1717	0.7723	0.3006	0.032*
C5	−0.0539 (3)	0.7096 (2)	0.4296 (2)	0.0237 (6)
H5	−0.1035	0.7492	0.4767	0.028*
C6	0.0620 (3)	0.6374 (2)	0.4675 (2)	0.0184 (5)
C7	0.1344 (3)	0.5761 (2)	0.39860 (19)	0.0168 (5)
C8	0.0970 (3)	0.6270 (2)	0.5777 (2)	0.0186 (5)
H8	0.0307	0.6606	0.6176	0.022*
C9	0.2165 (3)	0.5744 (2)	0.74125 (19)	0.0190 (5)
H9A	0.1890	0.5003	0.7621	0.023*
H9B	0.1427	0.6261	0.7615	0.023*
C10	0.3675 (3)	0.6033 (2)	0.80083 (19)	0.0183 (5)

C11	0.4764 (3)	0.5101 (2)	0.7928 (2)	0.0205 (6)
H11A	0.5707	0.5277	0.8364	0.025*
H11B	0.4372	0.4425	0.8202	0.025*
C12	0.3474 (3)	0.6122 (2)	0.9145 (2)	0.0287 (6)
H12A	0.3124	0.5421	0.9380	0.043*
H12B	0.2750	0.6693	0.9226	0.043*
H12C	0.4418	0.6308	0.9558	0.043*
C13	0.4229 (3)	0.7114 (2)	0.7623 (2)	0.0248 (6)
H13A	0.5206	0.7277	0.8000	0.037*
H13B	0.3544	0.7701	0.7737	0.037*
H13C	0.4294	0.7056	0.6884	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.01581 (19)	0.01867 (19)	0.01328 (18)	0.00302 (11)	0.00137 (14)	−0.00186 (11)
O1	0.0205 (9)	0.0314 (10)	0.0153 (9)	0.0031 (8)	0.0016 (8)	0.0004 (8)
O2	0.0148 (9)	0.0211 (9)	0.0148 (9)	0.0036 (7)	−0.0008 (7)	−0.0003 (7)
N1	0.0155 (10)	0.0178 (10)	0.0185 (10)	−0.0036 (8)	0.0042 (9)	−0.0038 (9)
N2	0.0188 (12)	0.0232 (12)	0.0135 (11)	0.0058 (9)	0.0018 (9)	−0.0015 (9)
N3	0.0215 (11)	0.0181 (10)	0.0179 (11)	0.0026 (9)	0.0035 (9)	−0.0026 (9)
N4	0.0222 (11)	0.0223 (11)	0.0165 (10)	0.0073 (9)	0.0041 (9)	−0.0012 (10)
N5	0.0269 (13)	0.0330 (13)	0.0390 (15)	−0.0034 (11)	0.0035 (12)	−0.0176 (12)
C1	0.0240 (14)	0.0469 (17)	0.0138 (13)	−0.0002 (13)	−0.0018 (11)	0.0008 (13)
C2	0.0137 (12)	0.0193 (12)	0.0220 (13)	−0.0041 (10)	0.0023 (10)	0.0013 (11)
C3	0.0202 (13)	0.0198 (12)	0.0237 (13)	−0.0041 (10)	−0.0019 (11)	0.0063 (11)
C4	0.0195 (13)	0.0213 (13)	0.0360 (16)	0.0019 (11)	−0.0048 (12)	0.0053 (12)
C5	0.0188 (13)	0.0197 (12)	0.0319 (15)	0.0014 (10)	0.0017 (12)	−0.0015 (12)
C6	0.0157 (12)	0.0164 (11)	0.0228 (13)	−0.0020 (10)	0.0019 (11)	−0.0025 (11)
C7	0.0127 (11)	0.0148 (11)	0.0227 (13)	−0.0040 (9)	0.0015 (10)	0.0000 (10)
C8	0.0176 (12)	0.0160 (12)	0.0226 (13)	−0.0023 (10)	0.0046 (11)	−0.0057 (11)
C9	0.0186 (12)	0.0241 (13)	0.0149 (12)	−0.0015 (11)	0.0046 (10)	−0.0028 (11)
C10	0.0204 (13)	0.0192 (12)	0.0155 (12)	−0.0023 (10)	0.0032 (10)	−0.0022 (10)
C11	0.0214 (14)	0.0264 (14)	0.0136 (13)	0.0009 (10)	0.0021 (11)	0.0006 (10)
C12	0.0289 (15)	0.0392 (16)	0.0190 (13)	−0.0011 (13)	0.0071 (12)	−0.0093 (13)
C13	0.0234 (14)	0.0208 (13)	0.0303 (15)	−0.0049 (11)	0.0039 (12)	−0.0052 (12)

Geometric parameters (Å, º)

Cu—O2	1.9047 (18)	C4—C5	1.370 (4)
Cu—N1	1.960 (2)	C4—H4	0.9500
Cu—N2	2.001 (2)	C5—C6	1.417 (4)
Cu—N3	2.023 (2)	C5—H5	0.9500
Cu—N3^{i}	2.641 (2)	C6—C7	1.410 (3)
O1—C2	1.380 (3)	C6—C8	1.440 (3)
O1—C1	1.427 (3)	C8—H8	0.9500
O2—C7	1.310 (3)	C9—C10	1.532 (3)
N1—C8	1.293 (3)	C9—H9A	0.9900
N1—C9	1.469 (3)	C9—H9B	0.9900
N2—C11	1.480 (3)	C10—C13	1.528 (3)

N2—H1N	0.869 (10)	C10—C11	1.529 (4)
N2—H2N	0.877 (10)	C10—C12	1.530 (3)
N3—N4	1.203 (3)	C11—H11A	0.9900
N4—N5	1.154 (3)	C11—H11B	0.9900
C1—H1A	0.9800	C12—H12A	0.9800
C1—H1B	0.9800	C12—H12B	0.9800
C1—H1C	0.9800	C12—H12C	0.9800
C2—C3	1.371 (3)	C13—H13A	0.9800
C2—C7	1.424 (3)	C13—H13B	0.9800
C3—C4	1.397 (4)	C13—H13C	0.9800
C3—H3	0.9500		

O2—Cu—N1	93.97 (8)	C6—C5—H5	119.7
O2—Cu—N2	168.34 (9)	C7—C6—C5	120.4 (2)
N1—Cu—N2	94.85 (9)	C7—C6—C8	122.5 (2)
O2—Cu—N3	87.81 (8)	C5—C6—C8	117.1 (2)
N1—Cu—N3	161.12 (9)	O2—C7—C6	124.0 (2)
N2—Cu—N3	86.30 (9)	O2—C7—C2	118.7 (2)
O2—Cu—N3^{i}	86.64 (7)	C6—C7—C2	117.3 (2)
N1—Cu—N3^{i}	109.71 (7)	N1—C8—C6	127.2 (2)
N2—Cu—N3^{i}	83.22 (8)	N1—C8—H8	116.4
N3—Cu—N3^{i}	89.15 (8)	C6—C8—H8	116.4
C2—O1—C1	116.9 (2)	N1—C9—C10	114.7 (2)
C7—O2—Cu	126.05 (16)	N1—C9—H9A	108.6
C8—N1—C9	116.6 (2)	C10—C9—H9A	108.6
C8—N1—Cu	122.94 (17)	N1—C9—H9B	108.6
C9—N1—Cu	120.14 (16)	C10—C9—H9B	108.6
C11—N2—Cu	124.71 (18)	H9A—C9—H9B	107.6
C11—N2—H1N	110.5 (18)	C13—C10—C11	111.8 (2)
Cu—N2—H1N	102.8 (18)	C13—C10—C12	110.6 (2)
C11—N2—H2N	111 (2)	C11—C10—C12	106.9 (2)
Cu—N2—H2N	99 (2)	C13—C10—C9	110.5 (2)
H1N—N2—H2N	106 (3)	C11—C10—C9	110.2 (2)
N4—N3—Cu	117.98 (17)	C12—C10—C9	106.6 (2)
N5—N4—N3	177.8 (3)	N2—C11—C10	113.3 (2)
O1—C1—H1A	109.5	N2—C11—H11A	108.9
O1—C1—H1B	109.5	C10—C11—H11A	108.9
H1A—C1—H1B	109.5	N2—C11—H11B	108.9
O1—C1—H1C	109.5	C10—C11—H11B	108.9
H1A—C1—H1C	109.5	H11A—C11—H11B	107.7
H1B—C1—H1C	109.5	C10—C12—H12A	109.5
C3—C2—O1	124.8 (2)	C10—C12—H12B	109.5
C3—C2—C7	121.1 (2)	H12A—C12—H12B	109.5
O1—C2—C7	114.1 (2)	C10—C12—H12C	109.5
C2—C3—C4	121.1 (2)	H12A—C12—H12C	109.5
C2—C3—H3	119.5	H12B—C12—H12C	109.5
C4—C3—H3	119.5	C10—C13—H13A	109.5
C5—C4—C3	119.5 (2)	C10—C13—H13B	109.5
C5—C4—H4	120.2	H13A—C13—H13B	109.5

C3—C4—H4	120.2	C10—C13—H13C	109.5
C4—C5—C6	120.6 (3)	H13A—C13—H13C	109.5
C4—C5—H5	119.7	H13B—C13—H13C	109.5
N1—Cu—O2—C7	19.51 (19)	C4—C5—C6—C7	−1.5 (4)
N2—Cu—O2—C7	−119.6 (4)	C4—C5—C6—C8	−179.2 (2)
N3—Cu—O2—C7	−179.31 (19)	Cu—O2—C7—C6	−17.6 (3)
N3^{i}—Cu—O2—C7	−90.04 (19)	Cu—O2—C7—C2	164.15 (17)
O2—Cu—N1—C8	−8.9 (2)	C5—C6—C7—O2	−177.6 (2)
N2—Cu—N1—C8	163.5 (2)	C8—C6—C7—O2	−0.1 (4)
N3—Cu—N1—C8	−103.8 (3)	C5—C6—C7—C2	0.7 (3)
N3^{i}—Cu—N1—C8	78.9 (2)	C8—C6—C7—C2	178.2 (2)
O2—Cu—N1—C9	164.68 (17)	C3—C2—C7—O2	179.0 (2)
N2—Cu—N1—C9	−22.95 (18)	O1—C2—C7—O2	0.9 (3)
N3—Cu—N1—C9	69.8 (3)	C3—C2—C7—C6	0.6 (3)
N3^{i}—Cu—N1—C9	−107.47 (17)	O1—C2—C7—C6	−177.5 (2)
O2—Cu—N2—C11	156.8 (3)	C9—N1—C8—C6	−177.6 (2)
N1—Cu—N2—C11	17.8 (2)	Cu—N1—C8—C6	−3.8 (4)
N3—Cu—N2—C11	−143.3 (2)	C7—C6—C8—N1	11.6 (4)
N3^{i}—Cu—N2—C11	127.1 (2)	C5—C6—C8—N1	−170.8 (2)
O2—Cu—N3—N4	−49.87 (19)	C8—N1—C9—C10	−133.4 (2)
N1—Cu—N3—N4	46.0 (4)	Cu—N1—C9—C10	52.6 (3)
N2—Cu—N3—N4	140.2 (2)	N1—C9—C10—C13	51.7 (3)
N3^{i}—Cu—N3—N4	−136.5 (2)	N1—C9—C10—C11	−72.3 (3)
C1—O1—C2—C3	−2.2 (4)	N1—C9—C10—C12	172.0 (2)
C1—O1—C2—C7	175.8 (2)	Cu—N2—C11—C10	−39.6 (3)
O1—C2—C3—C4	176.8 (2)	C13—C10—C11—N2	−60.1 (3)
C7—C2—C3—C4	−1.1 (4)	C12—C10—C11—N2	178.8 (2)
C2—C3—C4—C5	0.3 (4)	C9—C10—C11—N2	63.3 (3)
C3—C4—C5—C6	1.0 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*···O2^{i}	0.87 (1)	2.35 (2)	2.956 (3)	127 (2)
N2—H2*N*···N3	0.88 (1)	2.36 (3)	2.752 (3)	107 (3)

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