

trans-Bis(nitrato- κ O)tetrakis(1-vinyl-1*H*-imidazole- κ N³)copper(II)

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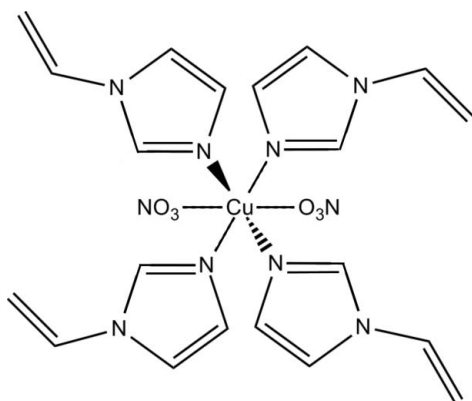
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.041; wR factor = 0.109; data-to-parameter ratio = 19.3.

In the title compound, $[\text{Cu}(\text{NO}_3)_2(\text{C}_5\text{H}_6\text{N}_2)_4]$, the Cu^{II} ion is located on an inversion centre. It features a Jahn–Teller-distorted octahedral coordination geometry, defined by four N atoms of four 1-vinylimidazole ligands in the equatorial plane and two nitrate O atoms in the axial positions. The nitrate anion is disordered over two sets of sites in a 0.801 (6):0.199 (6) ratio. In the crystal, the complex molecules are linked by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For applications and characterisation of related compounds, see: Sundberg & Martin (1974); Kurimura *et al.* (1994); Baran (1999); Zhao (2008).



Experimental

Crystal data

$[\text{Cu}(\text{NO}_3)_2(\text{C}_5\text{H}_6\text{N}_2)_4]$	$V = 1249.10$ (8) Å ³
$M_r = 564.03$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.9415$ (4) Å	$\mu = 0.93$ mm ⁻¹
$b = 8.7618$ (3) Å	$T = 296$ K
$c = 16.3172$ (6) Å	$0.1 \times 0.1 \times 0.1$ mm
$\beta = 102.281$ (4)°	

Data collection

Oxford Diffraction SuperNova diffractometer	6658 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2007)	3820 independent reflections
$T_{\text{min}} = 0.911$, $T_{\text{max}} = 0.911$	2908 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	1 restraint
$wR(F^2) = 0.109$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.26$ e Å ⁻³
3820 reflections	$\Delta\rho_{\text{min}} = -0.32$ e Å ⁻³
198 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the imidazole (N1/C1/N2/C3/C2) ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C7}-\text{H7}\cdots\text{O3A}$	0.93	2.41	3.273 (3)	155
$\text{C1}-\text{H1}\cdots\text{O1A}^{\text{i}}$	0.93	2.52	3.273 (3)	139
$\text{C4}-\text{H4}\cdots\text{O1A}^{\text{i}}$	0.93	2.38	3.217 (4)	149
$\text{C5}-\text{H5B}\cdots\text{Cg1}^{\text{ii}}$	0.93	2.78	3.6515	156
$\text{C10}-\text{H10A}\cdots\text{Cg1}^{\text{iii}}$	0.93	2.95	3.6691	136

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2655).

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supporting information

Acta Cryst. (2012). E68, m1045 [https://doi.org/10.1107/S1600536812030607]

trans*-Bis(nitrato- κ O)tetrakis(1-vinyl-1*H*-imidazole- κ N³)copper(II)*Fatih Şen, Ramazan Şahin, Ömer Andaç and Murat Taş****S1. Comment**

Numerous complexes derived from *d*-block metals and imidazole ligands are well-known (Sundberg & Martin, 1974). A large number of investigations on the complexation of copper(II) with imidazole ligands and vinylimidazol ligands have been reported, with some of them reporting structures determined crystallographically (Kurimura *et al.*, 1994). We report here the crystal structure of the title compound, [Cu(C₅H₆N₂)₄(NO₃)₂], a mixed-ligand Cu^{II} complex, (I).

The molecular structure of compound (I) (Fig. 1) is similar to those of analogous derivatives (Baran, 1999). The Cu^{II} atom displays a Jahn-Teller distorted octahedral coordination geometry, with four N atoms from four 1-vinylimidazole ligands in the equatorial plane (with Cu—N1 and Cu—N3 bond lengths of 2.0091 (15) and 2.0147 (16) Å) and two nitrate molecules in axial positions (with Cu—O2B and Cu—O2A bond lengths of 2.531 (9) and 2.651 (3) Å). These bond lengths are comparable with those reported by Baran (1999).

The imidazole rings have a maximum deviation of 0.004 Å and 0.002 Å from planarity for atoms N2 and N3, respectively. The bond lengths and angles of the 1-vinyl-imidazole molecules in (I) show no significant differences to those of the related 2-chlorobenzoato structure (Zhao, 2008).

In the crystal structure (Fig. 2) the molecules are linked by two intermolecular C—H \cdots O hydrogen bonds and two C—H \cdots π hydrogen-bonding associations (Table 1).

S2. Experimental

Complex (I) was prepared from a mixtures of solutions containing copper(II) nitrate trihydrate (2.42 g, 10 mmol) and 1-vinylimidazole (1.98 g, 20 mmol) in 20 ml ethanol. The reaction mixture was stirred for 1 h, then 20 ml dissolved succinic acid (1.18 g, 10 mmol) was added. Then the reaction mixture solution was again stirred for 30 min and finally the solution was filtered. Blue single crystals of (I) were isolated after one day. Suitable crystals of (I) for structure determination were obtained from ethanol by slow evaporation (yield %55).

S3. Refinement

H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.93 Å for the *sp*² C atoms. The displacement parameters of the H atoms were constrained with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The nitrate anion was shown to be disordered over two sets of sites in a 0.801 (6):0.199 (6) ratio.

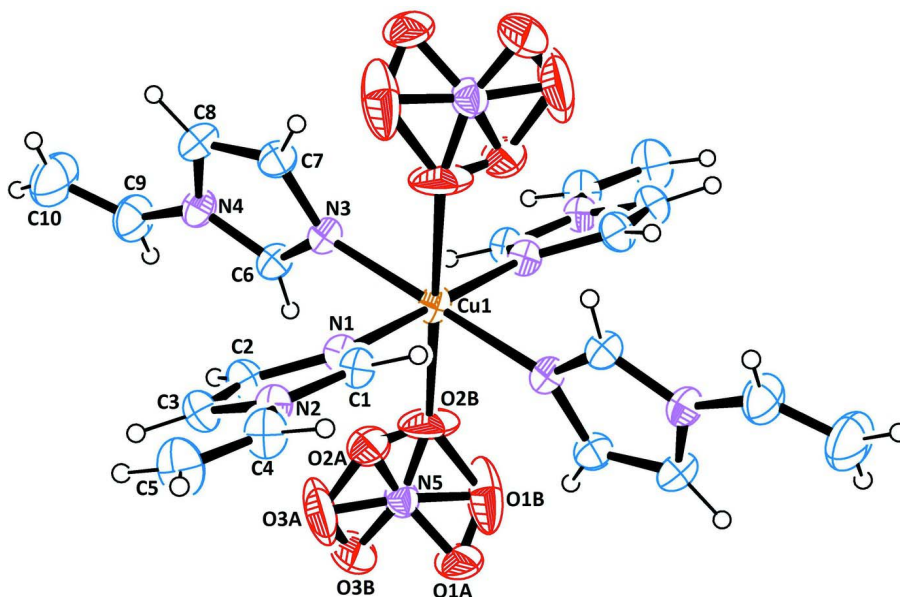


Figure 1

The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The disorder of the nitrate anion is shown, however only the shorter of the two Cu—O bonds is indicated.

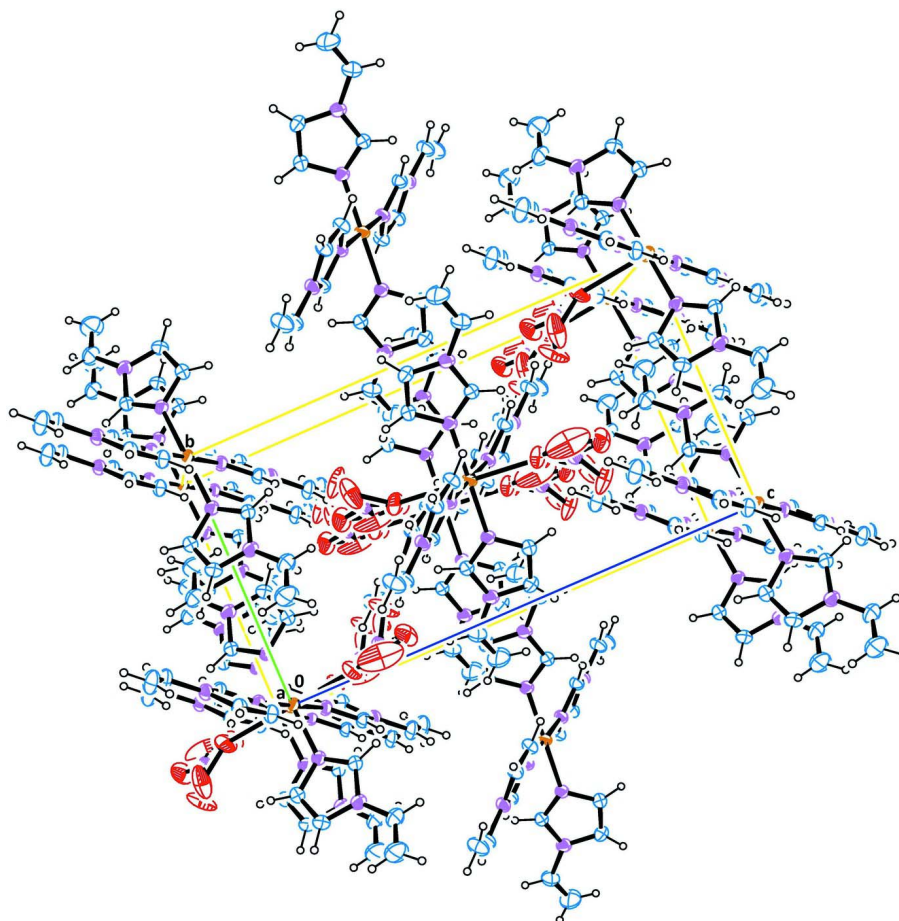


Figure 2
Packing of (I).

***trans*-Bis(nitrato- κ O)tetrakis(1-vinyl-1*H*-imidazole- κ N³)copper(II)**

Crystal data

[Cu(NO₃)₂(C₅H₆N₂)₄]

$M_r = 564.03$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.9415$ (4) Å

$b = 8.7618$ (3) Å

$c = 16.3172$ (6) Å

$\beta = 102.281$ (4)°

$V = 1249.10$ (8) Å³

$Z = 2$

$F(000) = 582$

$D_x = 1.500$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 452 reflections

$\theta = 3.3\text{--}30.4^\circ$

$\mu = 0.93$ mm⁻¹

$T = 296$ K

Prism, blue

$0.1 \times 0.1 \times 0.1$ mm

Data collection

Oxford Diffraction SuperNova
diffractometer

Radiation source: SuperNova (Mo) X-ray
Source

Mirror monochromator

Detector resolution: 16.0454 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2007)

$T_{\min} = 0.911$, $T_{\max} = 0.911$

6658 measured reflections

3820 independent reflections

2908 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\text{max}} = 30.5^\circ$, $\theta_{\text{min}} = 3.3^\circ$

$h = -12 \rightarrow 12$
 $k = -7 \rightarrow 12$
 $l = -22 \rightarrow 23$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.109$
 $S = 1.03$
 3820 reflections
 198 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.3004P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0069 (15)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cu1	1.0000	0.0000	1.0000	0.03972 (13)	
O1A	0.8327 (4)	0.0665 (4)	1.25474 (18)	0.0831 (11)	0.801 (6)
O1B	0.7861 (14)	0.031 (2)	1.1897 (19)	0.185 (15)	0.199 (6)
O2A	0.8731 (3)	0.0287 (3)	1.13193 (14)	0.0624 (8)	0.801 (6)
O2B	0.9871 (18)	0.0315 (12)	1.1525 (6)	0.092 (5)	0.199 (6)
O3B	0.9566 (15)	0.1935 (15)	1.2426 (8)	0.096 (5)	0.199 (6)
O3A	1.0438 (4)	0.1280 (6)	1.22800 (15)	0.116 (2)	0.801 (6)
N1	1.20913 (18)	-0.05380 (19)	1.06665 (9)	0.0390 (3)	
N2	1.39690 (17)	-0.1596 (2)	1.15773 (9)	0.0411 (4)	
N3	1.07291 (18)	0.21679 (18)	0.99548 (9)	0.0396 (3)	
N4	1.1880 (2)	0.42052 (19)	0.96108 (10)	0.0460 (4)	
N5	0.91506 (19)	0.0750 (2)	1.20390 (10)	0.0474 (4)	
C1	1.2438 (2)	-0.1520 (2)	1.12881 (11)	0.0410 (4)	
H1	1.1725	-0.2085	1.1500	0.049*	
C2	1.3466 (2)	0.0048 (2)	1.05604 (13)	0.0464 (5)	
H2	1.3573	0.0776	1.0162	0.056*	
C3	1.4628 (2)	-0.0580 (3)	1.11142 (12)	0.0467 (5)	
H3	1.5666	-0.0373	1.1173	0.056*	
C4	1.4720 (3)	-0.2583 (3)	1.22233 (13)	0.0547 (5)	
H4	1.4121	-0.3254	1.2457	0.066*	

C5	1.6189 (3)	-0.2611 (3)	1.25094 (16)	0.0709 (7)
H5A	1.6820	-0.1954	1.2289	0.085*
H5B	1.6612	-0.3288	1.2935	0.085*
C6	1.1213 (2)	0.2877 (2)	0.93488 (11)	0.0430 (4)
H6	1.1103	0.2500	0.8806	0.052*
C7	1.1104 (2)	0.3105 (2)	1.06447 (11)	0.0459 (4)
H7	1.0898	0.2904	1.1169	0.055*
C8	1.1817 (3)	0.4360 (3)	1.04412 (13)	0.0526 (5)
H8	1.2192	0.5171	1.0793	0.063*
C9	1.2511 (3)	0.5242 (3)	0.90989 (19)	0.0670 (7)
H9	1.2270	0.5083	0.8522	0.080*
C10	1.3381 (4)	0.6372 (4)	0.9373 (2)	0.1013 (11)
H10A	1.3647	0.6568	0.9945	0.122*
H10B	1.3747	0.6997	0.8999	0.122*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.04072 (19)	0.03473 (19)	0.03982 (18)	-0.00283 (14)	-0.00020 (12)	0.00636 (13)
O1A	0.081 (2)	0.104 (2)	0.0780 (17)	-0.0103 (18)	0.0492 (15)	-0.0192 (16)
O1B	0.047 (7)	0.116 (13)	0.40 (5)	-0.024 (8)	0.072 (15)	-0.016 (19)
O2A	0.0630 (17)	0.0759 (16)	0.0460 (11)	-0.0037 (13)	0.0064 (10)	-0.0199 (10)
O2B	0.144 (15)	0.076 (7)	0.076 (8)	0.012 (8)	0.069 (9)	-0.023 (6)
O3B	0.060 (7)	0.116 (9)	0.105 (8)	0.002 (6)	0.002 (6)	-0.079 (7)
O3A	0.074 (2)	0.213 (5)	0.0536 (14)	-0.074 (3)	-0.0024 (13)	0.012 (2)
N1	0.0414 (8)	0.0401 (8)	0.0343 (7)	-0.0005 (7)	0.0051 (6)	0.0040 (6)
N2	0.0405 (8)	0.0454 (9)	0.0348 (7)	0.0000 (7)	0.0025 (6)	0.0032 (7)
N3	0.0436 (8)	0.0355 (8)	0.0369 (7)	-0.0018 (7)	0.0025 (6)	0.0019 (6)
N4	0.0513 (9)	0.0370 (8)	0.0487 (9)	-0.0059 (8)	0.0087 (7)	0.0027 (7)
N5	0.0426 (9)	0.0559 (11)	0.0433 (8)	-0.0038 (8)	0.0085 (7)	-0.0015 (8)
C1	0.0403 (9)	0.0416 (10)	0.0390 (8)	-0.0032 (8)	0.0039 (7)	0.0042 (8)
C2	0.0466 (10)	0.0526 (12)	0.0417 (9)	-0.0010 (9)	0.0132 (8)	0.0090 (8)
C3	0.0399 (9)	0.0564 (12)	0.0452 (10)	-0.0006 (10)	0.0121 (8)	0.0061 (9)
C4	0.0536 (12)	0.0599 (14)	0.0458 (10)	-0.0003 (11)	0.0000 (8)	0.0153 (10)
C5	0.0552 (13)	0.0848 (19)	0.0652 (14)	0.0027 (14)	-0.0041 (11)	0.0248 (14)
C6	0.0500 (10)	0.0401 (10)	0.0374 (8)	-0.0025 (9)	0.0061 (7)	0.0007 (8)
C7	0.0516 (11)	0.0469 (11)	0.0385 (9)	0.0001 (9)	0.0075 (8)	-0.0008 (8)
C8	0.0643 (13)	0.0415 (11)	0.0498 (11)	-0.0055 (11)	0.0071 (9)	-0.0087 (9)
C9	0.0806 (18)	0.0566 (14)	0.0671 (15)	-0.0165 (13)	0.0230 (13)	0.0094 (12)
C10	0.135 (3)	0.0703 (19)	0.110 (2)	-0.041 (2)	0.051 (2)	-0.0040 (18)

Geometric parameters (Å, °)

Cu1—N1 ⁱ	2.0091 (15)	N2—C3	1.378 (3)
Cu1—N1	2.0091 (15)	N2—C4	1.418 (2)
Cu1—N3 ⁱ	2.0147 (16)	N3—C6	1.316 (2)
Cu1—N3	2.0147 (16)	N3—C7	1.376 (2)
Cu1—O2B	2.531 (9)	N4—C6	1.336 (2)

Cu1—O2B ⁱ	2.531 (9)	N4—C8	1.375 (3)
Cu1—O2A ⁱ	2.651 (3)	N4—C9	1.428 (3)
Cu1—O2A	2.651 (3)	C1—H1	0.9300
O1A—O1B	1.10 (3)	C2—C3	1.342 (3)
O1A—N5	1.223 (3)	C2—H2	0.9300
O1A—O3B	1.612 (16)	C3—H3	0.9300
O1B—N5	1.191 (13)	C4—C5	1.297 (3)
O1B—O2A	1.34 (2)	C4—H4	0.9300
O2A—O2B	1.004 (15)	C5—H5A	0.9300
O2A—N5	1.223 (3)	C5—H5B	0.9300
O2B—N5	1.221 (10)	C6—H6	0.9300
O2B—O3A	1.490 (12)	C7—C8	1.347 (3)
O3B—O3A	1.036 (10)	C7—H7	0.9300
O3B—N5	1.231 (11)	C8—H8	0.9300
O3A—N5	1.226 (3)	C9—C10	1.280 (4)
N1—C1	1.316 (2)	C9—H9	0.9300
N1—C2	1.376 (3)	C10—H10A	0.9300
N2—C1	1.351 (2)	C10—H10B	0.9300
N1 ⁱ —Cu1—N1	180.000 (1)	C6—N4—C9	124.9 (2)
N1 ⁱ —Cu1—N3 ⁱ	88.27 (6)	C8—N4—C9	128.2 (2)
N1—Cu1—N3 ⁱ	91.73 (6)	C8—N4—Cu1	80.44 (12)
N1 ⁱ —Cu1—N3	91.73 (6)	C9—N4—Cu1	151.17 (15)
N1—Cu1—N3	88.27 (6)	O1B—N5—O2B	113.3 (16)
N3 ⁱ —Cu1—N3	180.000 (1)	O2A—N5—O1A	121.6 (3)
N1 ⁱ —Cu1—O2B	106.0 (3)	O2A—N5—O3A	120.6 (2)
N1—Cu1—O2B	74.0 (3)	O1A—N5—O3A	117.7 (3)
N3 ⁱ —Cu1—O2B	89.1 (3)	O1B—N5—O3B	123.1 (12)
N3—Cu1—O2B	90.9 (3)	O2B—N5—O3B	118.0 (9)
N1 ⁱ —Cu1—O2B ⁱ	74.0 (3)	N1—C1—N2	110.89 (17)
N1—Cu1—O2B ⁱ	106.0 (3)	N2—C1—Cu1	142.94 (13)
N3 ⁱ —Cu1—O2B ⁱ	90.9 (3)	N1—C1—H1	124.6
N3—Cu1—O2B ⁱ	89.1 (3)	N2—C1—H1	124.6
O2B—Cu1—O2B ⁱ	180.000 (3)	Cu1—C1—H1	92.5
N1 ⁱ —Cu1—O2A ⁱ	95.32 (7)	C3—C2—N1	110.28 (17)
N1—Cu1—O2A ⁱ	84.68 (7)	C3—C2—Cu1	142.29 (14)
N3 ⁱ —Cu1—O2A ⁱ	97.93 (7)	C3—C2—H2	124.9
N3—Cu1—O2A ⁱ	82.07 (7)	N1—C2—H2	124.9
O2B—Cu1—O2A ⁱ	157.8 (3)	Cu1—C2—H2	92.8
O2B ⁱ —Cu1—O2A ⁱ	22.2 (3)	C2—C3—N2	105.89 (17)
N1 ⁱ —Cu1—O2A	84.68 (7)	N2—C3—Cu1	79.50 (10)
N1—Cu1—O2A	95.32 (7)	C2—C3—H3	127.1
N3 ⁱ —Cu1—O2A	82.07 (7)	N2—C3—H3	127.1
N3—Cu1—O2A	97.93 (7)	Cu1—C3—H3	153.4
O2B—Cu1—O2A	22.2 (3)	C5—C4—N2	124.2 (2)
O2B ⁱ —Cu1—O2A	157.8 (3)	C5—C4—H4	117.9
O2A ⁱ —Cu1—O2A	180.000 (1)	N2—C4—H4	117.9
O1B—O1A—N5	61.4 (8)	C4—C5—H5A	120.0

O1B—O1A—O3B	101.9 (10)	C4—C5—H5B	120.0
N5—O1A—O3B	49.2 (4)	H5A—C5—H5B	120.0
O1A—O1B—N5	64.4 (11)	N3—C6—N4	111.49 (17)
O1A—O1B—O2A	121.6 (11)	N4—C6—Cu1	141.50 (13)
N5—O1B—O2A	57.3 (8)	N3—C6—H6	124.3
O1A—O1B—Cu1	128.6 (8)	N4—C6—H6	124.3
N5—O1B—Cu1	64.4 (12)	Cu1—C6—H6	93.8
N5—O2A—Cu1	135.6 (2)	C8—C7—N3	109.33 (17)
N5—O2B—Cu1	148.2 (9)	C8—C7—H7	125.3
N5—O3A—Cu1	71.46 (17)	N3—C7—H7	125.3
C1—N1—C2	105.69 (16)	C7—C8—N4	106.46 (18)
C1—N1—Cu1	127.61 (14)	N4—C8—Cu1	80.51 (12)
C2—N1—Cu1	126.69 (13)	C7—C8—H8	126.8
C1—N2—C3	107.24 (15)	N4—C8—H8	126.8
C1—N2—C4	125.05 (18)	Cu1—C8—H8	152.4
C3—N2—C4	127.69 (18)	C10—C9—N4	125.0 (3)
C3—N2—Cu1	81.48 (10)	C10—C9—H9	117.5
C4—N2—Cu1	150.75 (13)	N4—C9—H9	117.5
C6—N3—C7	105.73 (16)	C9—C10—H10A	120.0
C6—N3—Cu1	129.27 (13)	C9—C10—H10B	120.0
C7—N3—Cu1	123.86 (13)	H10A—C10—H10B	120.0
C6—N4—C8	106.98 (17)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the imidazole (N1/C1/N2/C3/C2) ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 \cdots O3A	0.93	2.41	3.273 (3)	155
C1—H1 \cdots O1A ⁱⁱ	0.93	2.52	3.273 (3)	139
C4—H4 \cdots O1A ⁱⁱ	0.93	2.38	3.217 (4)	149
C5—H5B \cdots Cg1 ⁱⁱⁱ	0.93	2.78	3.6515	156
C10—H10A \cdots Cg1 ^{iv}	0.93	2.95	3.6691	136

Symmetry codes: (ii) $-x+2, y-1/2, -z+5/2$; (iii) $-x+1, y-1/2, -z+1/2$; (iv) $x, y+1, z$.