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Tetra-μ-acetato-κ^8O:O'-bis[(*N*-ethylpyrimidin-2-amine)copper(II)](*Cu*—*Cu*)

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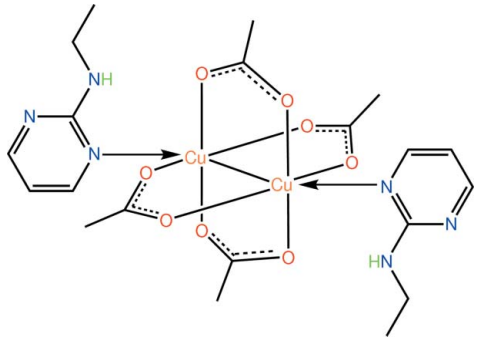
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.080; data-to-parameter ratio = 17.5.

In the centrosymmetric title molecule, $[Cu_2(CH_3COO)_4(C_6H_9N_3)_2]$, each of the four acetate groups bridges a pair of Cu^{II} atoms [Cu—Cu = 2.6540 (4) Å]. The distorted octahedral geometry of the metal atom is completed by an *N*-donor atom of the *N*-ethylpyrimidin-2-amine ligand: an intramolecular N—H···O hydrogen links its N—H group to an acetate carboxylate O atom. In the crystal, C—H···O interactions link the molecules into a supramolecular chain along the *b* axis.

Related literature

For related examples of tetrakisacetatobis[(substituted 2-aminopyridyl)copper(II)] complexes, see: Fairuz *et al.* (2010*a*,*b*).



Experimental

Crystal data

$[Cu_2(C_2H_3O_2)_4(C_6H_9N_3)_2]$
M_r = 609.58
Triclinic, $P\overline{1}$
a = 7.8488 (6) Å
b = 8.5114 (7) Å
c = 10.2999 (8) Å
α = 98.404 (1)°
β = 92.698 (1)°
γ = 105.599 (1)°
V = 652.92 (9) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.68 mm^{-1}
T = 293 K
0.40 × 0.35 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.613, T_{max} = 0.746
6208 measured reflections
2969 independent reflections
2669 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.080
S = 1.02
2969 reflections
170 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu—O1	1.978 (2)	Cu—O4^{i}	1.953 (1)
Cu—O2^{i}	1.963 (2)	Cu—N1	2.246 (2)
Cu—O3	1.955 (1)	Cu—Cu^{i}	2.6540 (4)

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—H3···O1	0.85 (1)	2.04 (1)	2.871 (2)	164 (2)
C4—H4a···O3^{ii}	0.96	2.51	3.458 (3)	171

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (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: HB5613).

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Tetra-μ-acetato-$\kappa^8O:O'$-bis[(*N*-ethylpyrimidin-2-amine)copper(II)](*Cu*—*Cu*)

Zaharah Aiyub, Edura Badaruddin, Zanariah Abdullah, Zainal A. Fairuz, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

In continuation of on-going structural studies of tetrakisacetatobis[(substituted 2-aminopyridyl)copper] complexes (Fairuz *et al.*, 2010*a*, 2010*b*), the title complex, (I), was investigated.

The binuclear copper(II) complex, Fig. 1, is situated about a centre of inversion and features two Cu atoms bridged by four acetate groups. The Cu–O bond distances lie in a narrow range of 1.953 (1) to 1.978 (2) Å, Table 1. The coordination environment for each Cu atom is completed by an N atom derived from the *N*-ethylpyrimidin-2-amine ligand and the second Cu atom [Cu···Cu^{i} = 2.6540 (4) Å for *i*: 1 - *x*, 1 - *y*, 1 - *z*]. The resulting hexa-coordinated geometry is based on an octahedron. An intramolecular N3–H···O1 interaction contributes to the stability of the dinuclear molecule, Table 2. The *N*-heterocycle is effectively planar as seen in the C8–N3–C9–C10 torsion angle of -166.6 (2) °. In the crystal packing, the presence of C–H···O interactions connect dinuclear molecules into supramolecular chains along the *b* axis, Fig. 2 and Table 2.

S2. Experimental

Copper acetate (0.1 g, 0.5 mmol) was dissolved in acetonitrile (15 ml) and mixed with a solution of *N*-(pyrimidin-2-yl)ethylamine (0.2 g, 1.1 mmol) dissolved in acetonitrile (15 ml). The blue precipitate that formed was recrystallized from acetonitrile to yield blue prisms of (I).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 to 1.5U_{equiv}(C). The N-bound H-atom was located in a difference Fourier map and was refined with a distance restraint of N–H 0.86±0.01 Å; the U_{iso} value was freely refined

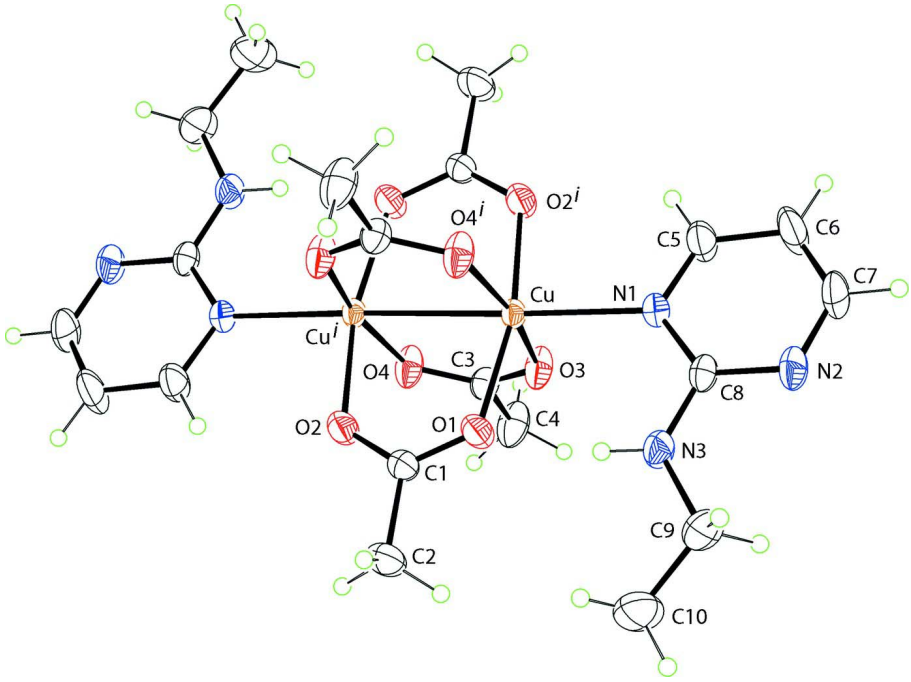

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 35% probability level. Primed atoms are related by the symmetry operation i: 1 - *x*, 1 - *y*, 1 - *z*.

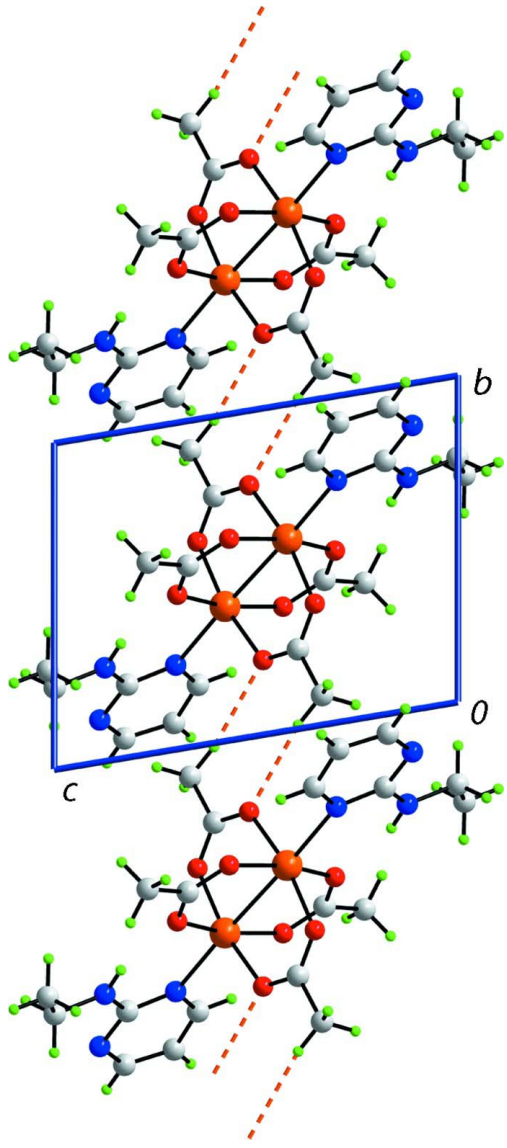

Figure 2

Supramolecular chain along the *b* axis in (I) mediated by C–H···O contacts shown as orange dashed lines.

Tetra-μ-acetato-$\kappa^8O:O'$-bis[(*N*-ethylpyrimidin-2- amine)copper(II)](*Cu—Cu*)

Crystal data

$[Cu_2(C_2H_3O_2)_4(C_6H_9N_3)_2]$
M_r = 609.58
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.8488 (6) Å
b = 8.5114 (7) Å
c = 10.2999 (8) Å
α = 98.404 (1)°
β = 92.698 (1)°
γ = 105.599 (1)°
V = 652.92 (9) Å^3

Z = 1
F(000) = 314
D_x = 1.550 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3508 reflections
θ = 2.5–28.2°
μ = 1.68 mm^{-1}
T = 293 K
Prism, blue
0.40 × 0.35 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.613, T_{max} = 0.746
6208 measured reflections
2969 independent reflections
2669 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −10→10
k = −11→11
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.080
S = 1.02
2969 reflections
170 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.1167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu	0.60458 (3)	0.58733 (2)	0.42299 (2)	0.02818 (9)
O1	0.3830 (2)	0.5272 (2)	0.30471 (15)	0.0479 (4)
O2	0.2102 (2)	0.3821 (2)	0.43330 (16)	0.0491 (4)
O3	0.5310 (2)	0.77124 (17)	0.51727 (16)	0.0474 (4)
O4	0.3551 (2)	0.62484 (18)	0.64537 (16)	0.0473 (4)
N1	0.7898 (2)	0.7417 (2)	0.30101 (17)	0.0328 (3)
N2	0.8689 (3)	0.8709 (2)	0.11294 (19)	0.0473 (4)
N3	0.5862 (3)	0.7135 (2)	0.12539 (18)	0.0435 (4)
H3	0.513 (3)	0.648 (2)	0.165 (2)	0.048 (7)*
C1	0.2361 (3)	0.4432 (2)	0.3307 (2)	0.0364 (4)
C2	0.0792 (3)	0.4133 (4)	0.2317 (3)	0.0585 (7)
H2A	0.0917	0.5081	0.1889	0.088*
H2B	−0.0277	0.3946	0.2758	0.088*
H2C	0.0736	0.3180	0.1672	0.088*
C3	0.4244 (3)	0.7560 (2)	0.6037 (2)	0.0361 (4)
C4	0.3760 (4)	0.9086 (3)	0.6633 (3)	0.0595 (7)
H4A	0.3991	0.9880	0.6044	0.089*
H4B	0.4457	0.9552	0.7459	0.089*
H4C	0.2522	0.8799	0.6775	0.089*
C5	0.9529 (3)	0.8135 (3)	0.3561 (2)	0.0497 (6)
H5	0.9836	0.7926	0.4389	0.060*
C6	1.0798 (3)	0.9180 (4)	0.2965 (3)	0.0650 (8)
H6	1.1928	0.9706	0.3381	0.078*
C7	1.0308 (3)	0.9397 (3)	0.1740 (3)	0.0566 (6)

H7	1.1151	1.0064	0.1302	0.068*
C8	0.7529 (3)	0.7767 (2)	0.18107 (19)	0.0342 (4)
C9	0.5238 (4)	0.7487 (3)	0.0013 (2)	0.0564 (6)
H9A	0.5716	0.6922	−0.0707	0.068*
H9B	0.5645	0.8666	0.0000	0.068*
C10	0.3254 (4)	0.6923 (4)	−0.0163 (3)	0.0754 (9)
H10A	0.2848	0.7136	−0.0996	0.113*
H10B	0.2785	0.7513	0.0534	0.113*
H10C	0.2855	0.5759	−0.0140	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.02719 (13)	0.02865 (14)	0.02816 (14)	0.00393 (9)	0.00634 (9)	0.00911 (9)
O1	0.0309 (7)	0.0684 (10)	0.0392 (8)	−0.0011 (7)	−0.0011 (6)	0.0225 (7)
O2	0.0326 (7)	0.0646 (10)	0.0454 (9)	−0.0021 (7)	−0.0016 (6)	0.0253 (8)
O3	0.0602 (10)	0.0330 (7)	0.0548 (10)	0.0145 (7)	0.0295 (8)	0.0149 (7)
O4	0.0601 (10)	0.0336 (7)	0.0526 (9)	0.0139 (7)	0.0292 (8)	0.0122 (7)
N1	0.0319 (8)	0.0309 (8)	0.0339 (8)	0.0039 (6)	0.0071 (6)	0.0077 (6)
N2	0.0493 (11)	0.0492 (10)	0.0452 (10)	0.0069 (8)	0.0209 (9)	0.0206 (9)
N3	0.0444 (10)	0.0495 (10)	0.0344 (9)	0.0033 (8)	0.0042 (8)	0.0178 (8)
C1	0.0308 (9)	0.0396 (10)	0.0367 (10)	0.0061 (8)	−0.0001 (8)	0.0081 (8)
C2	0.0364 (11)	0.0801 (18)	0.0541 (15)	0.0041 (11)	−0.0089 (10)	0.0223 (13)
C3	0.0414 (10)	0.0314 (9)	0.0367 (10)	0.0108 (8)	0.0080 (8)	0.0068 (8)
C4	0.0823 (18)	0.0381 (12)	0.0678 (17)	0.0253 (12)	0.0358 (14)	0.0144 (11)
C5	0.0410 (12)	0.0578 (14)	0.0434 (12)	−0.0030 (10)	0.0060 (10)	0.0171 (11)
C6	0.0387 (12)	0.0742 (18)	0.0664 (18)	−0.0164 (12)	0.0095 (12)	0.0203 (14)
C7	0.0510 (14)	0.0589 (15)	0.0581 (15)	0.0006 (11)	0.0247 (12)	0.0254 (12)
C8	0.0419 (11)	0.0298 (9)	0.0325 (10)	0.0094 (8)	0.0137 (8)	0.0080 (7)
C9	0.0632 (16)	0.0740 (17)	0.0362 (12)	0.0191 (13)	0.0054 (11)	0.0222 (11)
C10	0.0622 (17)	0.116 (3)	0.0530 (16)	0.0251 (17)	−0.0021 (13)	0.0323 (17)

Geometric parameters (Å, º)

Cu—O1	1.978 (2)	C2—H2A	0.9600
Cu—O2^{i}	1.963 (2)	C2—H2B	0.9600
Cu—O3	1.955 (1)	C2—H2C	0.9600
Cu—O4^{i}	1.953 (1)	C3—C4	1.504 (3)
Cu—N1	2.246 (2)	C4—H4A	0.9600
Cu—Cu^{i}	2.6540 (4)	C4—H4B	0.9600
O1—C1	1.247 (2)	C4—H4C	0.9600
O2—C1	1.246 (3)	C5—C6	1.378 (3)
O3—C3	1.247 (2)	C5—H5	0.9300
O4—C3	1.250 (2)	C6—C7	1.355 (4)
N1—C5	1.321 (3)	C6—H6	0.9300
N1—C8	1.349 (3)	C7—H7	0.9300
N2—C7	1.331 (3)	C9—C10	1.494 (4)
N2—C8	1.340 (3)	C9—H9A	0.9700

N3—C8	1.338 (3)	C9—H9B	0.9700
N3—C9	1.448 (3)	C10—H10A	0.9600
N3—H3	0.85 (1)	C10—H10B	0.9600
C1—C2	1.502 (3)	C10—H10C	0.9600

O4^{i}—Cu—O3	167.23 (6)	H2B—C2—H2C	109.5
O4^{i}—Cu—O2^{i}	88.86 (8)	O3—C3—O4	125.51 (18)
O3—Cu—O2^{i}	89.53 (8)	O3—C3—C4	117.20 (18)
O4^{i}—Cu—O1	89.76 (8)	O4—C3—C4	117.29 (18)
O3—Cu—O1	88.95 (7)	C3—C4—H4A	109.5
O2^{i}—Cu—O1	166.94 (6)	C3—C4—H4B	109.5
O4^{i}—Cu—N1	97.38 (6)	H4A—C4—H4B	109.5
O3—Cu—N1	95.36 (6)	C3—C4—H4C	109.5
O2^{i}—Cu—N1	93.54 (6)	H4A—C4—H4C	109.5
O1—Cu—N1	99.52 (6)	H4B—C4—H4C	109.5
O4^{i}—Cu—Cu^{i}	83.74 (4)	N1—C5—C6	123.0 (2)
O3—Cu—Cu^{i}	83.49 (4)	N1—C5—H5	118.5
O2^{i}—Cu—Cu^{i}	84.07 (5)	C6—C5—H5	118.5
O1—Cu—Cu^{i}	82.87 (5)	C7—C6—C5	116.3 (2)
N1—Cu—Cu^{i}	177.35 (4)	C7—C6—H6	121.9
C1—O1—Cu	124.63 (14)	C5—C6—H6	121.9
C1—O2—Cu^{i}	123.95 (14)	N2—C7—C6	123.7 (2)
C3—O3—Cu	123.73 (13)	N2—C7—H7	118.2
C3—O4—Cu^{i}	123.48 (13)	C6—C7—H7	118.2
C5—N1—C8	115.85 (17)	N3—C8—N2	116.97 (19)
C5—N1—Cu	115.99 (14)	N3—C8—N1	117.50 (17)
C8—N1—Cu	128.08 (13)	N2—C8—N1	125.53 (19)
C7—N2—C8	115.5 (2)	N3—C9—C10	109.8 (2)
C8—N3—C9	123.88 (19)	N3—C9—H9A	109.7
C8—N3—H3	118.0 (17)	C10—C9—H9A	109.7
C9—N3—H3	118.1 (17)	N3—C9—H9B	109.7
O2—C1—O1	124.4 (2)	C10—C9—H9B	109.7
O2—C1—C2	117.62 (19)	H9A—C9—H9B	108.2
O1—C1—C2	117.9 (2)	C9—C10—H10A	109.5
C1—C2—H2A	109.5	C9—C10—H10B	109.5
C1—C2—H2B	109.5	H10A—C10—H10B	109.5
H2A—C2—H2B	109.5	C9—C10—H10C	109.5
C1—C2—H2C	109.5	H10A—C10—H10C	109.5
H2A—C2—H2C	109.5	H10B—C10—H10C	109.5

O4^{i}—Cu—O1—C1	84.62 (19)	Cu^{i}—O2—C1—C2	−177.97 (16)
O3—Cu—O1—C1	−82.68 (19)	Cu—O1—C1—O2	−2.0 (3)
O2^{i}—Cu—O1—C1	0.7 (4)	Cu—O1—C1—C2	178.01 (16)
N1—Cu—O1—C1	−177.93 (18)	Cu—O3—C3—O4	2.6 (3)
Cu^{i}—Cu—O1—C1	0.90 (18)	Cu—O3—C3—C4	−177.68 (17)
O4^{i}—Cu—O3—C3	−2.8 (4)	Cu^{i}—O4—C3—O3	−2.2 (3)
O2^{i}—Cu—O3—C3	−85.53 (19)	Cu^{i}—O4—C3—C4	178.11 (17)
O1—Cu—O3—C3	81.49 (18)	C8—N1—C5—C6	−0.1 (3)

N1—Cu—O3—C3	−179.05 (18)	Cu—N1—C5—C6	−177.0 (2)
Cu^{i}—Cu—O3—C3	−1.45 (17)	N1—C5—C6—C7	−2.4 (4)
O4^{i}—Cu—N1—C5	−93.37 (16)	C8—N2—C7—C6	0.3 (4)
O3—Cu—N1—C5	85.80 (16)	C5—C6—C7—N2	2.3 (4)
O2^{i}—Cu—N1—C5	−4.07 (16)	C9—N3—C8—N2	−3.6 (3)
O1—Cu—N1—C5	175.63 (16)	C9—N3—C8—N1	175.8 (2)
Cu^{i}—Cu—N1—C5	21.6 (10)	C7—N2—C8—N3	176.3 (2)
O4^{i}—Cu—N1—C8	90.11 (16)	C7—N2—C8—N1	−3.1 (3)
O3—Cu—N1—C8	−90.72 (16)	C5—N1—C8—N3	−176.44 (19)
O2^{i}—Cu—N1—C8	179.42 (16)	Cu—N1—C8—N3	0.1 (3)
O1—Cu—N1—C8	−0.89 (17)	C5—N1—C8—N2	3.0 (3)
Cu^{i}—Cu—N1—C8	−154.9 (8)	Cu—N1—C8—N2	179.51 (15)
Cu^{i}—O2—C1—O1	2.0 (3)	C8—N3—C9—C10	−166.6 (2)

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*
N3—H3···O1	0.85 (1)	2.04 (1)	2.871 (2)	164 (2)
C4—H4a···O3^{ii}	0.96	2.51	3.458 (3)	171

Symmetry code: (ii) −*x*+1, −*y*+2, −*z*+1.