Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of poly[[*trans*-diaqua-bis[μ_2-*trans*-4,4′-(diazenediyl)dipyridine]-nickel(II)] diiodide ethanol disolvate]

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Received 2 July 2014; accepted 11 July 2014

Edited by I. Brito, University of Antofagasta, Chile

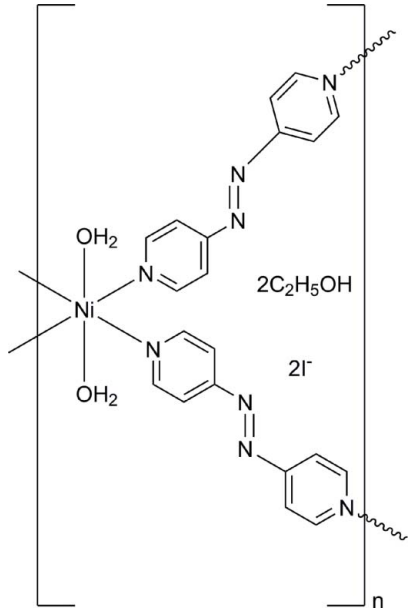
In the title compound, $\{[Ni(C_{10}H_8N_4)_2(H_2O)_2]I_2\cdot 2C_2H_5OH\}_n$, the complex shows an octahedral environment of the Ni^{2+} cation in which it is located on a centre of symmetry, linked to two water molecules and the pyridine-N atoms of four 4,4′-(diazenediyl)dipyridine ligands bridging Ni^{2+} cations along the *b*- and *c*-axis directions, giving rise to a two-dimensional arrangement. The Ni—N bond lengths are in the range 2.109 (4)–2.186 (3) Å and the Ni—O bond length is 2.080 (3) Å. The 4,4′-(diazenediyl)dipyridine ligand lies on an inversion centre. An O—H···O hydrogen-bond interaction is observed between water and ethanol molecules. The I^- ions can be regarded as free anions in the crystal lattice.

Keywords: crystal structure; nickel coordination compound; bidimensional MOF; cationic network.

CCDC reference: 1013422

1. Related literature

For related two-dimensional structures, see: Carlucci *et al.* (2003); Noro *et al.* (2005, 2006); Li *et al.* (2007); Pan *et al.* (2010); Aijaz *et al.* (2011).



2. Experimental

2.1. Crystal data

$[Ni(C_{10}H_8N_4)_2(H_2O)_2]I_2\cdot 2C_2H_6O$
M_r = 809.09
Monoclinic, $P2_1/n$
a = 8.6367 (11) Å
b = 13.2598 (16) Å
c = 13.4188 (14) Å
β = 101.737 (3)°
V = 1504.6 (3) Å^3
Z = 2
Mo *Kα* radiation
μ = 2.74 mm^{-1}
T = 100 K
0.12 × 0.08 × 0.06 mm

2.2. Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.77, T_{max} = 0.85
19224 measured reflections
2741 independent reflections
1948 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.097
S = 1.00
2741 reflections
185 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.83 (4)	1.91 (4)	2.703 (6)	161 (5)

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

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:

SHELXTL (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

Diffraction data were collected at the SCXRD laboratory from the Servicio Interdepartamental de Investigación (SIdI, UAM). Financial support received from the Spanish Ministerio de Economía y Competitividad (CTQ2011-23066) and the Comunidad de Madrid (S2009/MAT-1467) is gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BX2463).

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supporting information

Acta Cryst. (2014). E**70**, m314–m315 [doi:10.1107/S1600536814016158]

Crystal structure of poly[[*trans*-diaquabis[μ_2-*trans*-4,4′-(diazenediyl)dipyridine]-nickel(II)] diiodide ethanol disolvate]

Josefina Perles, Miguel Cortijo and Santiago Herrero

S1. Related Literature

A similar laminar structure was found for the compound $[Ni(NCS)_2(t\text{-}apy)_2]\cdot 3$toluene (Noro *et al.*, 2006) although in this latter case there is no one-dimensional H-bond chain. For related 2D structures see: Carlucci *et al.* (2003); Noro *et al.* (2005 and 2006); Li *et al.* (2007); Pan *et al.* (2010); and Aijaz *et al.* (2011).

S2. Preparation

Nickel(II) iodide (0.30 g, 1.0 mmol), *trans*-4,4′-(diazenediyl)dipyridine (0.18 g, 1.0 mmol), ethanol (9 mL), and water (3 mL) were placed into an 85 mL Teflon vessel with a magnetic stirrer. The vessel was sealed with a lid equipped with a temperature sensor and placed in a ETHOS ONE microwave oven. The reaction mixture was heated for 3 hours at 120°C and left to cool afterwards. Slow interdiffusion of diethyl ether in the obtained solution gave rise to red crystals suitable for single-crystal X-ray diffraction after a few days.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

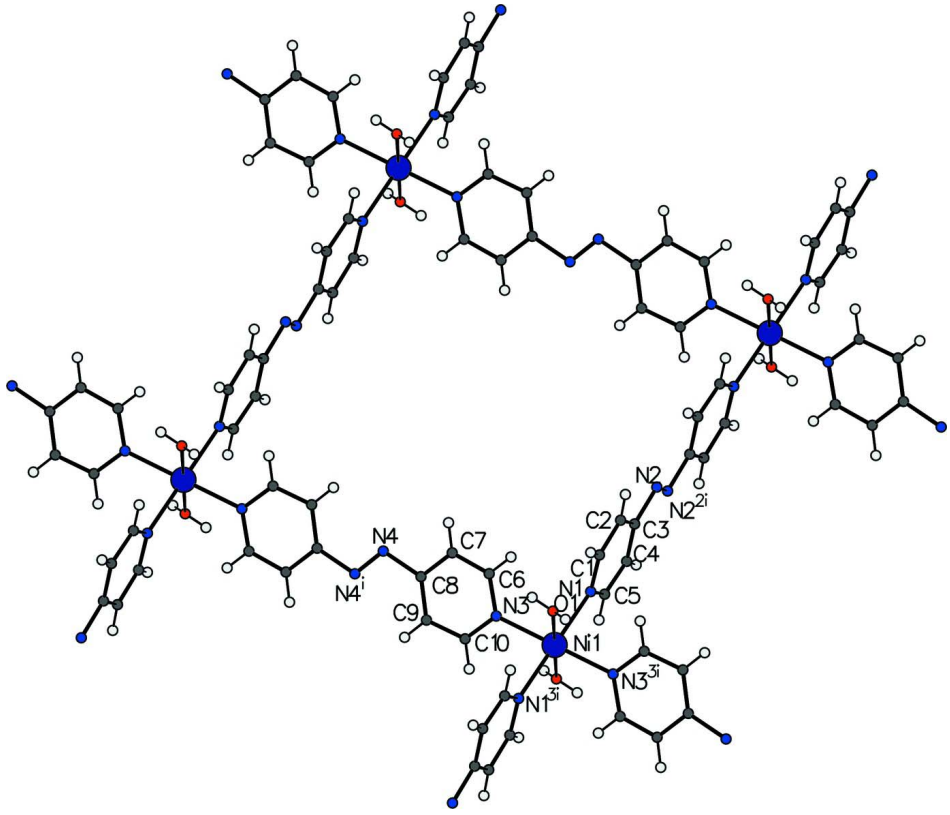

Figure 1

Part of the polymeric structure for the title compound. Symmetry code for compound (i):-x, -y+2, -z+2; (2i): -x-y+1,-z+1; (3i):-x,-y+1,-z+2.

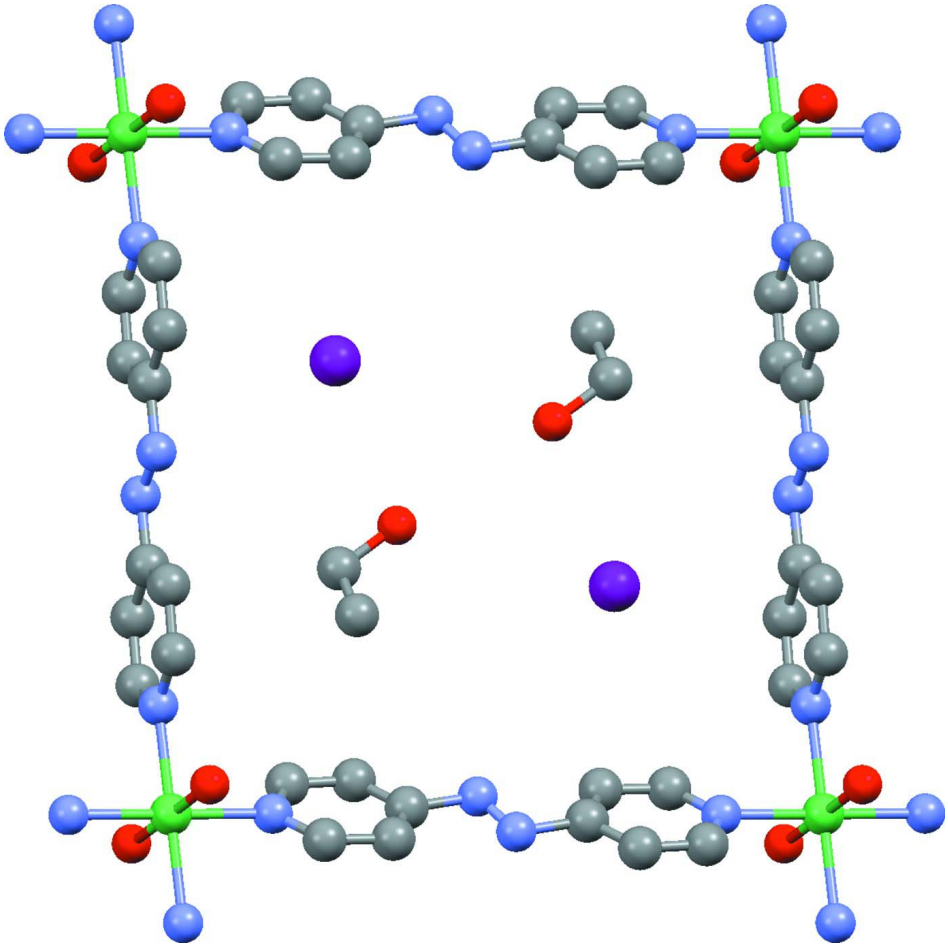

Figure 2

Simplified drawing of a layer parallel to (011). Hydrogen atoms have been omitted for clarity.

Poly[[*trans*-diaquabis[μ_2-*trans*-4,4′-(diazenediyl)dipyridine]nickel(II)] diiodide ethanol disolvate]

Crystal data

$[Ni(C_{10}H_8N_4)_2(H_2O)_2]I_2{\cdot}2C_2H_6O$
M_r = 809.09
Monoclinic, $P2_1/n$
a = 8.6367 (11) Å
b = 13.2598 (16) Å
c = 13.4188 (14) Å
β = 101.737 (3)°
V = 1504.6 (3) Å^3
Z = 2
$F(000)$ = 796
D_x = 1.786 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3456 reflections
θ = 2.9–21.6°
μ = 2.74 mm^{-1}
T = 100 K
Prismatic, clear orange–red
0.12 × 0.08 × 0.06 mm

Data collection

Bruker Kappa APEXII diffractometer
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
single crystal scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.77, T_{max} = 0.85
19224 measured reflections
2741 independent reflections
1948 reflections with $I > 2\sigma(I)$

R_{int} = 0.067
θ_{max} = 25.4°, θ_{min} = 2.2°
h = −10→10
k = −15→15
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.097
S = 1.00
2741 reflections
185 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 2.7231P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.01318 (5)	0.80017 (3)	0.33435 (3)	0.05632 (18)
Ni1	0	0.5	1.0	0.0183 (2)
C1	0.1600 (6)	0.4962 (4)	0.8111 (3)	0.0278 (11)
H1	0.2538	0.4945	0.8623	0.033*
C2	0.1749 (6)	0.4983 (4)	0.7105 (3)	0.0304 (12)
H2	0.2763	0.5	0.6934	0.036*
C3	0.0387 (6)	0.4978 (4)	0.6352 (3)	0.0290 (12)
C4	−0.1044 (6)	0.4962 (4)	0.6634 (4)	0.0365 (13)
H4	−0.1996	0.4952	0.6134	0.044*
C5	−0.1092 (6)	0.4962 (4)	0.7655 (3)	0.0363 (13)
H5	−0.2096	0.496	0.784	0.044*
C6	0.1181 (6)	0.7073 (4)	0.9562 (4)	0.0320 (12)
H6	0.1913	0.668	0.9286	0.038*
C7	0.1276 (7)	0.8108 (4)	0.9503 (4)	0.0416 (14)
H7	0.2052	0.842	0.9196	0.05*
C8	0.0228 (7)	0.8671 (4)	0.9897 (4)	0.0402 (15)
C9	−0.0855 (7)	0.8207 (4)	1.0356 (4)	0.0444 (15)
H9	−0.1575	0.8591	1.0651	0.053*
C10	−0.0877 (7)	0.7150 (4)	1.0381 (4)	0.0388 (14)
H10	−0.1634	0.6824	1.0694	0.047*
C11	0.8888 (11)	0.7821 (7)	0.7076 (6)	0.095 (3)

H11A	0.9057	0.8424	0.7508	0.142*
H11B	0.9055	0.7217	0.7504	0.142*
H11C	0.7804	0.7823	0.6675	0.142*
C12	1.0001 (11)	0.7823 (7)	0.6391 (7)	0.093 (3)
H12A	1.1095	0.7802	0.6796	0.112*
H12B	0.9832	0.7214	0.5957	0.112*
N1	0.0212 (5)	0.4964 (3)	0.8404 (3)	0.0226 (9)
N2	0.0617 (5)	0.4987 (3)	0.5323 (3)	0.0345 (10)
N3	0.0113 (5)	0.6588 (3)	0.9985 (3)	0.0240 (9)
N4	0.0347 (6)	0.9783 (4)	0.9742 (4)	0.0490 (13)
O1	0.2450 (4)	0.4913 (3)	1.0428 (2)	0.0263 (8)
H1A	0.305 (5)	0.539 (3)	1.062 (4)	0.039*
H1B	0.293 (6)	0.442 (3)	1.074 (3)	0.039*
O2	0.9803 (5)	0.8708 (3)	0.5759 (3)	0.0568 (12)
H2A	0.9192	0.8576	0.5203	0.085*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0540 (3)	0.0502 (3)	0.0633 (3)	−0.0059 (2)	0.0085 (2)	−0.0198 (2)
Ni1	0.0260 (5)	0.0134 (4)	0.0159 (4)	0.0006 (4)	0.0054 (3)	0.0000 (3)
C1	0.025 (3)	0.038 (3)	0.019 (2)	0.001 (2)	0.001 (2)	0.000 (2)
C2	0.028 (3)	0.041 (3)	0.024 (2)	−0.001 (2)	0.011 (2)	−0.001 (2)
C3	0.040 (3)	0.030 (3)	0.017 (2)	0.002 (2)	0.005 (2)	−0.002 (2)
C4	0.024 (3)	0.061 (4)	0.025 (3)	0.002 (3)	0.005 (2)	0.003 (3)
C5	0.030 (3)	0.057 (4)	0.024 (3)	0.000 (3)	0.010 (2)	−0.001 (2)
C6	0.033 (3)	0.024 (3)	0.038 (3)	0.001 (2)	0.007 (2)	0.003 (2)
C7	0.041 (3)	0.024 (3)	0.058 (4)	−0.001 (3)	0.006 (3)	0.009 (3)
C8	0.041 (4)	0.016 (3)	0.055 (3)	−0.009 (3)	−0.010 (3)	0.001 (2)
C9	0.054 (4)	0.027 (3)	0.052 (4)	0.013 (3)	0.008 (3)	−0.011 (3)
C10	0.055 (4)	0.025 (3)	0.038 (3)	0.000 (3)	0.016 (3)	−0.001 (2)
C11	0.103 (7)	0.109 (7)	0.068 (5)	−0.001 (6)	0.008 (5)	0.033 (5)
C12	0.085 (6)	0.091 (7)	0.106 (7)	0.004 (5)	0.025 (5)	0.033 (5)
N1	0.028 (2)	0.019 (2)	0.0208 (19)	−0.0026 (18)	0.0066 (17)	0.0008 (17)
N2	0.038 (3)	0.050 (3)	0.018 (2)	0.000 (2)	0.0098 (16)	0.000 (2)
N3	0.032 (2)	0.019 (2)	0.0202 (19)	−0.0020 (19)	0.0026 (17)	−0.0018 (16)
N4	0.044 (3)	0.050 (3)	0.056 (3)	0.002 (3)	0.017 (2)	−0.008 (2)
O1	0.027 (2)	0.024 (2)	0.0267 (17)	0.0012 (15)	0.0034 (15)	0.0030 (14)
O2	0.056 (3)	0.054 (3)	0.060 (3)	0.006 (2)	0.011 (2)	0.008 (2)

Geometric parameters (Å, º)

Ni1—O1^{i}	2.080 (3)	C7—C8	1.360 (8)
Ni1—O1	2.080 (3)	C7—H7	0.95
Ni1—N3	2.109 (4)	C8—C9	1.366 (8)
Ni1—N3^{i}	2.109 (4)	C8—N4	1.496 (7)
Ni1—N1	2.186 (3)	C9—C10	1.403 (8)
Ni1—N1^{i}	2.186 (3)	C9—H9	0.95

C1—N1	1.336 (6)	C10—N3	1.324 (7)
C1—C2	1.381 (6)	C10—H10	0.95
C1—H1	0.95	C11—C12	1.458 (12)
C2—C3	1.386 (7)	C11—H11A	0.98
C2—H2	0.95	C11—H11B	0.98
C3—C4	1.365 (7)	C11—H11C	0.98
C3—N2	1.435 (6)	C12—O2	1.437 (9)
C4—C5	1.378 (7)	C12—H12A	0.99
C4—H4	0.95	C12—H12B	0.99
C5—N1	1.348 (6)	N2—N2^{ii}	1.229 (8)
C5—H5	0.95	N4—N4^{iii}	1.156 (9)
C6—N3	1.342 (7)	O1—H1A	0.82 (2)
C6—C7	1.378 (7)	O1—H1B	0.833 (19)
C6—H6	0.95	O2—H2A	0.84

O1^{i}—Ni1—O1	180.00 (19)	C6—C7—H7	120.9
O1^{i}—Ni1—N3	89.33 (15)	C7—C8—C9	119.9 (5)
O1—Ni1—N3	90.68 (15)	C7—C8—N4	114.7 (5)
O1^{i}—Ni1—N3^{i}	90.67 (15)	C9—C8—N4	125.3 (6)
O1—Ni1—N3^{i}	89.33 (15)	C8—C9—C10	118.3 (6)
N3—Ni1—N3^{i}	180.0 (2)	C8—C9—H9	120.9
O1^{i}—Ni1—N1	90.79 (13)	C10—C9—H9	120.9
O1—Ni1—N1	89.21 (13)	N3—C10—C9	122.7 (5)
N3—Ni1—N1	89.97 (15)	N3—C10—H10	118.7
N3^{i}—Ni1—N1	90.03 (15)	C9—C10—H10	118.7
O1^{i}—Ni1—N1^{i}	89.21 (13)	C12—C11—H11A	109.5
O1—Ni1—N1^{i}	90.79 (13)	C12—C11—H11B	109.5
N3—Ni1—N1^{i}	90.03 (15)	H11A—C11—H11B	109.5
N3^{i}—Ni1—N1^{i}	89.97 (15)	C12—C11—H11C	109.5
N1—Ni1—N1^{i}	180.0 (2)	H11A—C11—H11C	109.5
N1—C1—C2	123.7 (4)	H11B—C11—H11C	109.5
N1—C1—H1	118.1	O2—C12—C11	111.0 (7)
C2—C1—H1	118.1	O2—C12—H12A	109.4
C1—C2—C3	118.6 (5)	C11—C12—H12A	109.4
C1—C2—H2	120.7	O2—C12—H12B	109.4
C3—C2—H2	120.7	C11—C12—H12B	109.4
C4—C3—C2	118.7 (4)	H12A—C12—H12B	108.0
C4—C3—N2	125.3 (4)	C1—N1—C5	116.3 (4)
C2—C3—N2	116.0 (5)	C1—N1—Ni1	123.2 (3)
C3—C4—C5	119.2 (5)	C5—N1—Ni1	120.5 (3)
C3—C4—H4	120.4	N2^{ii}—N2—C3	114.1 (5)
C5—C4—H4	120.4	C10—N3—C6	117.1 (4)
N1—C5—C4	123.5 (5)	C10—N3—Ni1	121.6 (4)
N1—C5—H5	118.2	C6—N3—Ni1	121.3 (3)
C4—C5—H5	118.2	N4^{iii}—N4—C8	110.5 (7)
N3—C6—C7	123.7 (5)	Ni1—O1—H1A	126 (4)
N3—C6—H6	118.1	Ni1—O1—H1B	124 (4)
C7—C6—H6	118.1	H1A—O1—H1B	103 (5)

C8—C7—C6	118.2 (5)	C12—O2—H2A	109.5
C8—C7—H7	120.9		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{iv}	0.83 (4)	1.91 (4)	2.703 (6)	161 (5)

Symmetry code: (iv) *x*−1/2, −*y*+3/2, *z*+1/2.