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## Structure Reports

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## 2,2'-Dihydroxy-3,3'-[(1*E*,1'*E*)-hydrazine-1,2-diylidenedimethylidyne]dibenzoic acid *N,N*-dimethylformamide disolvate

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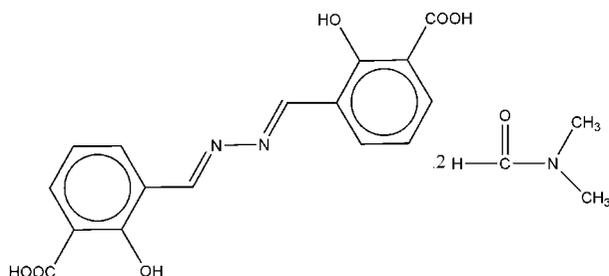
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Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.067;  $wR$  factor = 0.149; data-to-parameter ratio = 12.4.

The title compound,  $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_6 \cdot 2\text{C}_3\text{H}_7\text{NO}$ , lies across a crystallographic inversion centre which is situated at the midpoint of the central N—N bond. The substitution at the C=N bond adopts a *trans* configuration and it is essentially coplanar with the benzene ring [N—C—C torsion angles =  $-173.9$  (4) and  $6.4$  (6)°]. All torsion angles involving non-H atoms are close to 180°. Intramolecular O—H...O and weak C—H...O hydrogen bonds form  $S(6)$  and  $S(5)$  ring motifs, respectively, while intermolecular O—H...O and weak C—H...O hydrogen bonds connect the Schiff base molecule to solvent dimethylformamide molecules.

### Related literature

For information on Schiff base ligands, their complexes and their applications, see, for example: Pal *et al.* (2005); Hou *et al.* (2001); Ren *et al.* (2002). For bond-length data, see: Allen *et al.* (1987). For the structures and properties of related azine organic and metallorganic compounds, see, for example: Dreuw *et al.* (2005); Chattopadhyay *et al.* (2008); Cucos *et al.* (2006); Fu (2007); Mijanuddina *et al.* (2004); Sreerama *et al.* (2007); Butcher *et al.* (2007). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_6 \cdot 2\text{C}_3\text{H}_7\text{NO}$   
 $M_r = 474.47$   
 Monoclinic,  $P2_1/c$   
 $a = 5.9136$  (12) Å  
 $b = 10.837$  (2) Å  
 $c = 18.991$  (4) Å  
 $\beta = 98.96$  (3)°

$V = 1202.2$  (4) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 295$  K  
 $0.36 \times 0.20 \times 0.16$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.965$ ,  $T_{\max} = 0.984$

7616 measured reflections  
 1978 independent reflections  
 679 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.082$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$   
 $wR(F^2) = 0.149$   
 $S = 1.01$   
 1978 reflections

159 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.14$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.14$  e Å<sup>-3</sup>

**Table 1**

Selected torsion angles (°).

|             |            |                           |            |
|-------------|------------|---------------------------|------------|
| C7—C1—C2—C3 | 179.5 (4)  | C6—C1—C7—O2               | 174.1 (4)  |
| C3—C4—C5—C8 | 179.5 (4)  | N1 <sup>i</sup> —N1—C8—C5 | -179.4 (4) |
| C4—C5—C6—O3 | -179.5 (4) | C6—C5—C8—N1               | -173.9 (4) |
| C8—C5—C6—C1 | -180.0 (4) | C11—N2—C9—O4              | 178.3 (5)  |
| C7—C1—C6—C5 | -179.2 (4) |                           |            |

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

**Table 2**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$             | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------|-------|-------------|-------------|---------------|
| C9—H9...O1 <sup>ii</sup>  | 0.93  | 2.59        | 3.261 (6)   | 130           |
| C2—H2A...O2               | 0.93  | 2.43        | 2.742 (6)   | 99            |
| O3—H3...O1                | 0.82  | 1.85        | 2.568 (4)   | 146           |
| O2—H2...O4 <sup>iii</sup> | 0.82  | 1.76        | 2.557 (4)   | 162           |

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

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); 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: SHELXTL.

This work was supported by the Key Laboratory for Research and Development of Natural Drugs of Jiangxi Province. The data were collected and the structure solved and refined at the Department of Chemistry and Biology, Qinzhou University, Qinzhou, Guangxi 535000, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2745).

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## supporting information

*Acta Cryst.* (2009). E65, o194–o195 [doi:10.1107/S1600536808043134]

## 2,2'-Dihydroxy-3,3'-[(1*E*,1'*E*)-hydrazine-1,2-diylidenedimethylidyne]dibenzoic acid *N,N*-dimethylformamide disolvate

Sheng-Sen Zhang, Geng-Jin-Sheng Cheng, Yu Lei and Yin-Bao Li

### S1. Comment

Schiff bases are one of most prevalent mixed-donor ligands in the field of coordination chemistry. Schiff bases have been used widely as ligands in the formation of transition metal complexes. There has been growing interest in Schiff base ligands, mainly because of their wide application in the field of biochemistry, synthesis, and catalysis (Pal *et al.*, 2005; Hou *et al.*, 2001; Ren *et al.*, 2002).

Herein, we report the synthesis and crystal structure of the Schiff-base compound, (I). The molecule lies across a crystallographic inversion centre which is situated at the midpoint of the N—N (1.393 (6) Å) bond. The molecular structure of (I) is shown in Fig.1. All bond lengths are within in normal ranges (Allen *et al.*, 1987). The N1—C8 [1.281 (5) Å] and N1—N1<sup>i</sup> [1.393 (6) Å] (symmetry code: (i) -x+2, -y+2, -z+1) distances indicate these correspond to double and single bonds, respectively. The torsion angles indicate that the molecule is essentially planar with the C=N bond adjacent to the benzene rings adopting a *trans* configuration with respect to its substitution. Intramolecular O—H⋯O and C—H⋯O hydrogen bonds form S(6) and S(5) ring motifs, respectively (Bernstein *et al.*, 1995). The Schiff-base molecule and solvent DMF molecules are connected by intermolecular hydrogen bonds (Fig.1 and Table. 2). Some crystal structures which are closely related to the title compound have already been studied (Chattopadhyay *et al.*, 2008; Cucos *et al.*, 2006; Fu, 2007; Mijanuddina *et al.*, 2004; Dreuw *et al.*, 2005; Sreerama *et al.*, 2007).

### S2. Experimental

Reagents and solvents used were of commercially available quality. To a stirred solution of 3-formylsalicylic acid (0.332 g, 2 mmol) in absolute methanol (10 ml) was added dropwise hydrazine hydrate (0.050 g, 1 mmol). After a few minutes, an orange precipitate appeared, which was isolated by filtration, washed with methanol, and dried in air. Crystals of (I) suitable for X-ray diffraction were obtained by recrystallized the crude product from DMF solution.

### S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.96 Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C})$  for methyl H atoms.

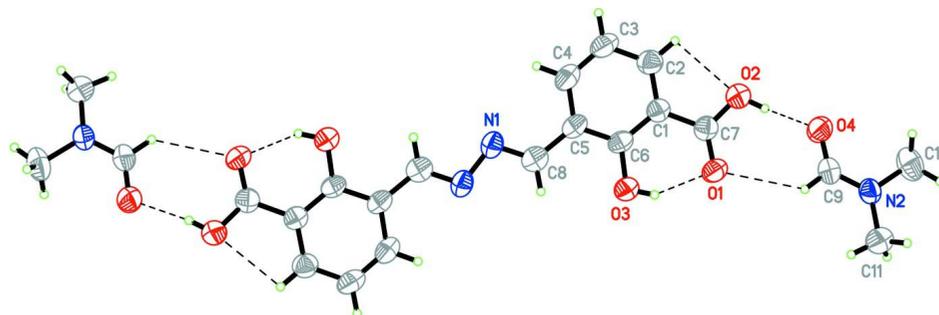


Figure 1

The molecular structure with displacement ellipsoids at the 30% probability level. Hydrogen bonds show as dashed lines.

### 2,2'-Dihydroxy-3,3'-[(1*E*,1'*E*)-hydrazine-1,2-diylidenedimethyldiylidene]dibenzoic acid *N,N*-dimethylformamide disolvate

#### Crystal data

$C_{16}H_{12}N_2O_6 \cdot 2C_3H_7NO$

$M_r = 474.47$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 5.9136\ (12)\ \text{\AA}$

$b = 10.837\ (2)\ \text{\AA}$

$c = 18.991\ (4)\ \text{\AA}$

$\beta = 98.96\ (3)^\circ$

$V = 1202.2\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 500$

$D_x = 1.311\ \text{Mg m}^{-3}$

Melting point: 443 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 812 reflections

$\theta = 2.1\text{--}15.4^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 295\ \text{K}$

Needle, red

$0.36 \times 0.20 \times 0.16\ \text{mm}$

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.965$ ,  $T_{\max} = 0.984$

7616 measured reflections

1978 independent reflections

679 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.082$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -6 \rightarrow 7$

$k = -12 \rightarrow 12$

$l = -22 \rightarrow 22$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.149$

$S = 1.01$

1978 reflections

159 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.022P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.14\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.14\ \text{e \AA}^{-3}$

Extinction correction: *SHELXL*,

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.016 (4)

*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 ( $\text{\AA}^2$ )*

|      | $x$         | $y$        | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|------------|--------------|----------------------------------|
| N1   | 0.9469 (6)  | 0.9471 (3) | 0.51035 (19) | 0.0850 (12)                      |
| N2   | 0.2954 (7)  | 0.6509 (3) | 0.23059 (19) | 0.0830 (11)                      |
| O1   | -0.0194 (5) | 0.7647 (3) | 0.34383 (15) | 0.0925 (10)                      |
| O2   | -0.0718 (5) | 0.5919 (3) | 0.40230 (16) | 0.0978 (10)                      |
| H2   | -0.1983     | 0.5948     | 0.3775       | 0.147*                           |
| O3   | 0.3501 (5)  | 0.8896 (3) | 0.37878 (14) | 0.0913 (10)                      |
| H3   | 0.2293      | 0.8697     | 0.3541       | 0.137*                           |
| O4   | 0.5660 (5)  | 0.5621 (3) | 0.31107 (17) | 0.1024 (11)                      |
| C1   | 0.2520 (8)  | 0.7119 (4) | 0.4437 (2)   | 0.0735 (12)                      |
| C2   | 0.3105 (8)  | 0.6335 (4) | 0.5012 (2)   | 0.0901 (14)                      |
| H2A  | 0.2151      | 0.5678     | 0.5079       | 0.108*                           |
| C3   | 0.5105 (10) | 0.6525 (5) | 0.5488 (2)   | 0.1046 (16)                      |
| H3A  | 0.5491      | 0.6001     | 0.5877       | 0.126*                           |
| C4   | 0.6512 (8)  | 0.7496 (5) | 0.5380 (2)   | 0.0977 (16)                      |
| H4   | 0.7858      | 0.7614     | 0.5698       | 0.117*                           |
| C5   | 0.5984 (8)  | 0.8308 (4) | 0.4808 (2)   | 0.0753 (13)                      |
| C6   | 0.3970 (8)  | 0.8102 (4) | 0.4342 (2)   | 0.0717 (12)                      |
| C7   | 0.0427 (8)  | 0.6924 (5) | 0.3928 (3)   | 0.0782 (13)                      |
| C8   | 0.7514 (9)  | 0.9333 (4) | 0.4714 (2)   | 0.0829 (13)                      |
| H8   | 0.7050      | 0.9909     | 0.4357       | 0.099*                           |
| C9   | 0.4849 (9)  | 0.6507 (5) | 0.2750 (3)   | 0.0863 (14)                      |
| H9   | 0.5674      | 0.7240     | 0.2801       | 0.104*                           |
| C10  | 0.1512 (9)  | 0.5403 (4) | 0.2220 (2)   | 0.1212 (18)                      |
| H10A | 0.2158      | 0.4811     | 0.1933       | 0.182*                           |
| H10B | 0.0001      | 0.5622     | 0.1993       | 0.182*                           |
| H10C | 0.1433      | 0.5052     | 0.2680       | 0.182*                           |
| C11  | 0.2066 (9)  | 0.7591 (5) | 0.1912 (3)   | 0.1217 (18)                      |
| H11A | 0.3080      | 0.8274     | 0.2039       | 0.183*                           |
| H11B | 0.0579      | 0.7784     | 0.2024       | 0.183*                           |
| H11C | 0.1949      | 0.7432     | 0.1410       | 0.183*                           |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|    | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$   | $U^{13}$  | $U^{23}$   |
|----|-----------|-----------|-----------|------------|-----------|------------|
| N1 | 0.066 (3) | 0.093 (3) | 0.091 (3) | 0.006 (2)  | 0.000 (2) | -0.007 (2) |
| N2 | 0.079 (3) | 0.082 (3) | 0.085 (3) | -0.017 (2) | 0.005 (2) | -0.005 (2) |

|     |           |           |           |              |              |             |
|-----|-----------|-----------|-----------|--------------|--------------|-------------|
| O1  | 0.093 (2) | 0.093 (2) | 0.084 (2) | 0.0066 (18)  | -0.0070 (18) | 0.0125 (17) |
| O2  | 0.089 (2) | 0.093 (2) | 0.107 (3) | -0.003 (2)   | 0.0012 (19)  | 0.0162 (19) |
| O3  | 0.096 (2) | 0.094 (2) | 0.079 (2) | -0.0056 (18) | -0.0022 (17) | 0.0103 (17) |
| O4  | 0.098 (3) | 0.085 (2) | 0.116 (3) | -0.001 (2)   | -0.010 (2)   | 0.0036 (19) |
| C1  | 0.071 (3) | 0.078 (3) | 0.072 (3) | 0.009 (3)    | 0.014 (3)    | 0.000 (3)   |
| C2  | 0.092 (4) | 0.098 (3) | 0.080 (3) | 0.001 (3)    | 0.015 (3)    | 0.008 (3)   |
| C3  | 0.104 (4) | 0.124 (4) | 0.080 (3) | 0.018 (4)    | -0.003 (3)   | 0.022 (3)   |
| C4  | 0.085 (4) | 0.119 (4) | 0.082 (4) | 0.009 (3)    | -0.009 (3)   | 0.005 (3)   |
| C5  | 0.083 (3) | 0.077 (3) | 0.063 (3) | 0.021 (3)    | 0.002 (3)    | -0.008 (3)  |
| C6  | 0.078 (3) | 0.075 (3) | 0.062 (3) | 0.021 (3)    | 0.011 (3)    | 0.001 (2)   |
| C7  | 0.075 (3) | 0.069 (3) | 0.090 (4) | 0.013 (3)    | 0.012 (3)    | -0.006 (3)  |
| C8  | 0.089 (4) | 0.079 (3) | 0.082 (3) | 0.020 (3)    | 0.018 (3)    | -0.009 (2)  |
| C9  | 0.076 (4) | 0.090 (4) | 0.094 (4) | -0.012 (3)   | 0.014 (3)    | -0.007 (3)  |
| C10 | 0.126 (5) | 0.116 (4) | 0.123 (4) | -0.042 (4)   | 0.023 (4)    | -0.037 (3)  |
| C11 | 0.118 (4) | 0.118 (4) | 0.120 (4) | -0.009 (3)   | -0.008 (3)   | 0.023 (3)   |

*Geometric parameters (Å, °)*

|                       |           |               |           |
|-----------------------|-----------|---------------|-----------|
| N1—C8                 | 1.281 (5) | C2—H2A        | 0.9300    |
| N1—N1 <sup>i</sup>    | 1.393 (6) | C3—C4         | 1.377 (6) |
| N2—C9                 | 1.293 (5) | C3—H3A        | 0.9300    |
| N2—C11                | 1.445 (5) | C4—C5         | 1.395 (5) |
| N2—C10                | 1.465 (5) | C4—H4         | 0.9300    |
| O1—C7                 | 1.228 (5) | C5—C6         | 1.387 (5) |
| O2—C7                 | 1.310 (5) | C5—C8         | 1.462 (6) |
| O2—H2                 | 0.8200    | C8—H8         | 0.9300    |
| O3—C6                 | 1.354 (4) | C9—H9         | 0.9300    |
| O3—H3                 | 0.8200    | C10—H10A      | 0.9600    |
| O4—C9                 | 1.232 (5) | C10—H10B      | 0.9600    |
| C1—C2                 | 1.384 (5) | C10—H10C      | 0.9600    |
| C1—C6                 | 1.397 (5) | C11—H11A      | 0.9600    |
| C1—C7                 | 1.462 (5) | C11—H11B      | 0.9600    |
| C2—C3                 | 1.388 (6) | C11—H11C      | 0.9600    |
| C8—N1—N1 <sup>i</sup> | 109.8 (5) | C5—C6—C1      | 121.6 (4) |
| C9—N2—C11             | 123.2 (4) | O1—C7—O2      | 122.3 (4) |
| C9—N2—C10             | 120.1 (4) | O1—C7—C1      | 122.1 (5) |
| C11—N2—C10            | 116.6 (4) | O2—C7—C1      | 115.6 (4) |
| C7—O2—H2              | 109.5     | N1—C8—C5      | 122.5 (4) |
| C6—O3—H3              | 109.5     | N1—C8—H8      | 118.7     |
| C2—C1—C6              | 119.1 (4) | C5—C8—H8      | 118.7     |
| C2—C1—C7              | 121.0 (5) | O4—C9—N2      | 125.9 (5) |
| C6—C1—C7              | 119.9 (4) | O4—C9—H9      | 117.0     |
| C1—C2—C3              | 120.4 (5) | N2—C9—H9      | 117.0     |
| C1—C2—H2A             | 119.8     | N2—C10—H10A   | 109.5     |
| C3—C2—H2A             | 119.8     | N2—C10—H10B   | 109.5     |
| C4—C3—C2              | 119.3 (4) | H10A—C10—H10B | 109.5     |
| C4—C3—H3A             | 120.4     | N2—C10—H10C   | 109.5     |

|             |            |                           |            |
|-------------|------------|---------------------------|------------|
| C2—C3—H3A   | 120.4      | H10A—C10—H10C             | 109.5      |
| C3—C4—C5    | 122.1 (5)  | H10B—C10—H10C             | 109.5      |
| C3—C4—H4    | 118.9      | N2—C11—H11A               | 109.5      |
| C5—C4—H4    | 118.9      | N2—C11—H11B               | 109.5      |
| C6—C5—C4    | 117.5 (5)  | H11A—C11—H11B             | 109.5      |
| C6—C5—C8    | 122.0 (4)  | N2—C11—H11C               | 109.5      |
| C4—C5—C8    | 120.5 (4)  | H11A—C11—H11C             | 109.5      |
| O3—C6—C5    | 116.5 (5)  | H11B—C11—H11C             | 109.5      |
| O3—C6—C1    | 121.9 (4)  |                           |            |
|             |            |                           |            |
| C6—C1—C2—C3 | 0.1 (6)    | C2—C1—C6—C5               | 0.3 (6)    |
| C7—C1—C2—C3 | 179.5 (4)  | C7—C1—C6—C5               | -179.2 (4) |
| C1—C2—C3—C4 | -0.5 (7)   | C2—C1—C7—O1               | 175.7 (4)  |
| C2—C3—C4—C5 | 0.6 (8)    | C6—C1—C7—O1               | -4.9 (6)   |
| C3—C4—C5—C6 | -0.3 (7)   | C2—C1—C7—O2               | -5.4 (6)   |
| C3—C4—C5—C8 | 179.5 (4)  | C6—C1—C7—O2               | 174.1 (4)  |
| C4—C5—C6—O3 | -179.5 (4) | N1 <sup>i</sup> —N1—C8—C5 | -179.4 (4) |
| C8—C5—C6—O3 | 0.7 (6)    | C6—C5—C8—N1               | -173.9 (4) |
| C4—C5—C6—C1 | -0.2 (6)   | C4—C5—C8—N1               | 6.4 (6)    |
| C8—C5—C6—C1 | -180.0 (4) | C11—N2—C9—O4              | 178.3 (5)  |
| C2—C1—C6—O3 | 179.5 (4)  | C10—N2—C9—O4              | 2.7 (7)    |
| C7—C1—C6—O3 | 0.1 (6)    |                           |            |

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

*Hydrogen-bond geometry (Å, °)*

| <i>D—H...A</i>            | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|---------------------------|------------|--------------|--------------|----------------|
| C9—H9...O1 <sup>ii</sup>  | 0.93       | 2.59         | 3.261 (6)    | 130            |
| C2—H2A...O2               | 0.93       | 2.43         | 2.742 (6)    | 99             |
| O3—H3...O1                | 0.82       | 1.85         | 2.568 (4)    | 146            |
| O2—H2...O4 <sup>iii</sup> | 0.82       | 1.76         | 2.557 (4)    | 162            |

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