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<sup>1</sup>This paper is dedicated to Her Royal Highness Princess Chulabhorn Krom Phra Srisavangavadhana of Thailand for her contributions to science on the occasion of her 62th birthday, which fell on July 4th, 2019.

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# Crystal structure of (*E*)-*N'*-(3,4-dihydroxybenzylidene)-4-hydroxybenzohydrazide<sup>1</sup>

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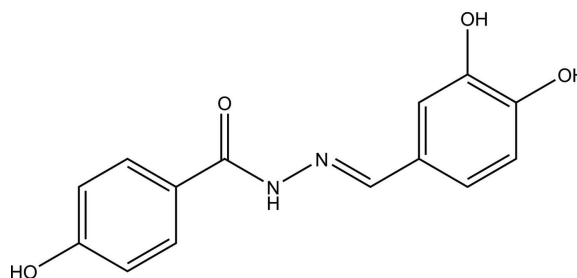
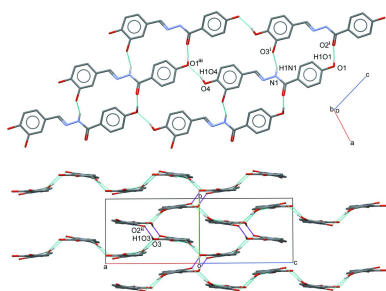
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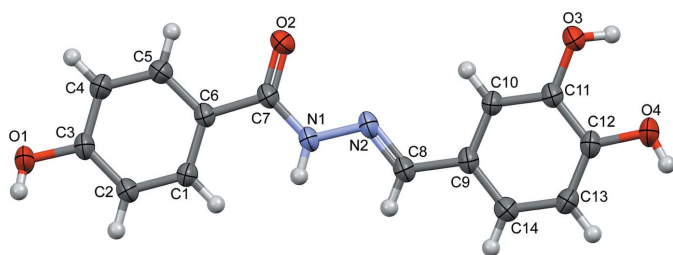
In the title benzohydrazide derivative, C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>, the azomethine C=N double bond has an *E* configuration. The hydrazide connecting bridge, (C=O)–(NH)–N=(CH), is nearly planar with C–C–N–N and C–N–N=C torsion angles of –177.33 (10) and –174.98 (12)°, respectively. The 4-hydroxyphenyl and 3,4-dihydroxyphenyl rings are slightly twisted, making a dihedral angle of 9.18 (6)°. In the crystal, molecules are connected by N–H···O and O–H···O hydrogen bonds into a three-dimensional network, while further consolidated *via*  $\pi$ – $\pi$  interactions [centroid–centroid distances = 3.6480 (8) and 3.7607 (8) Å]. The conformation is compared to those of related benzylidene-4-hydroxybenzohydrazide derivatives.

## 1. Chemical context

Hydrazides and hydrazones are important synthons for several transformations and have gained importance because of their various biological and clinical applications (Narasimhan *et al.*, 2010). Benzohydrazide derivatives containing an azomethine (–NHN=CH–) group have been reported to possess diverse biological activities such as antitumor (Xia *et al.*, 2007; Kumari & Bansal, 2018), antioxidant (Aziz *et al.*, 2014), antitubercular and antimicrobial (Maheswari & Manjula, 2015) and  $\alpha$ -glucosidase inhibition (Taha *et al.*, 2015) activities. The interesting biological activities of benzohydrazides led us to synthesize several benzohydrazides to study their bioactivities (Fun *et al.*, 2011; Horkaew *et al.*, 2011; Chantrapromma *et al.*, 2016), including the title compound (I), which was found to exhibit antioxidant activity with an IC<sub>50</sub> value of 0.035 ± 0.004 mM (ascorbic acid used as the reference standard; Thaipong *et al.*, 2006) and  $\alpha$ -glucosidase inhibitory activity with an IC<sub>50</sub> value of 0.014 ± 0.001 mM (acarbose as the reference standard; Bachhawat *et al.*, 2011).



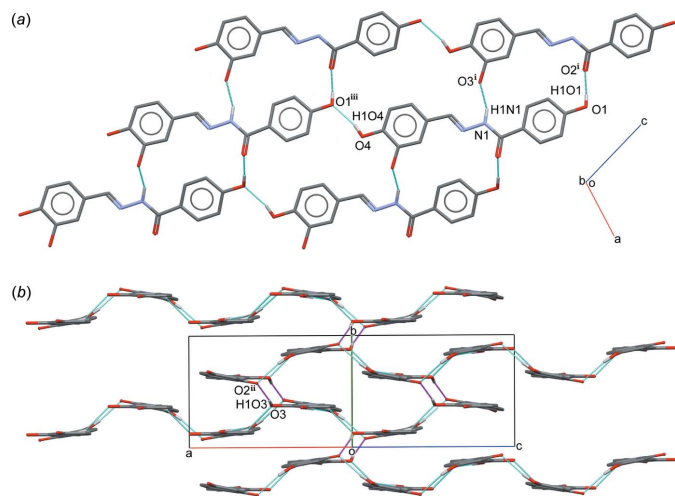
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**Figure 1**

The molecular structure of the title compound with the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

## 2. Structural commentary

The title hydrazide derivative, (I), consists of a 4-hydroxyphenyl ring, a 3,4-dihydroxyphenyl ring and a hydrazide ( $\text{C}=\text{O}$ )–(NH)–N=(CH) connecting bridge (Fig. 1). The C6–C7, C7–N1 and C8–C9 bond lengths of 1.4861 (15), 1.3385 (17) and 1.4584 (16) Å, respectively, confirm their single-bond character, whereas the C7=O2 and N2=C8 bond lengths of 1.2403 (15) and 1.2738 (17) Å, respectively, confirm the presence of the double bonds. The  $sp^2$ -hybridized character of atoms C7 and C8 is further supported by the bond angles C6–C7–N1 [116.49 (11)°] and N2–C8–C9 [120.86 (12)°]. The bond lengths and angles of the central hydrazide connecting bridge are consistent with those in related structures (Fun *et al.*, 2011; Chantrapromma *et al.*, 2016). The molecule exhibits an *E* configuration with respect to the azomethine C=N double bond. As the torsion angle C6–C7–N1–N2 [−177.33 (10)°] and C7–N1–N2–C8 [−174.98 (12)°] are both in an *anti-periplanar* conformation, the overall conformation for the hydrazide connecting bridge is almost planar. Furthermore, the 4-hydroxyphenyl and


**Figure 2**

(a) A partial packing diagram of the title compound, showing a two-dimensional plate formed by O–H...O and N–H...O hydrogen bonds (cyan dotted lines). [Symmetry codes: (i)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ ; (iii)  $x - 1, y, z - 1$ .] (b) A partial packing diagram of the title compound with additional O–H...O hydrogen bonds (magenta dotted lines). [Symmetry code: (ii)  $-x + 1, -y + 1, -z$ .] Hydrogen atoms not involved in with these interactions are omitted for clarity.

**Table 1**

Hydrogen-bond geometry (Å, °).

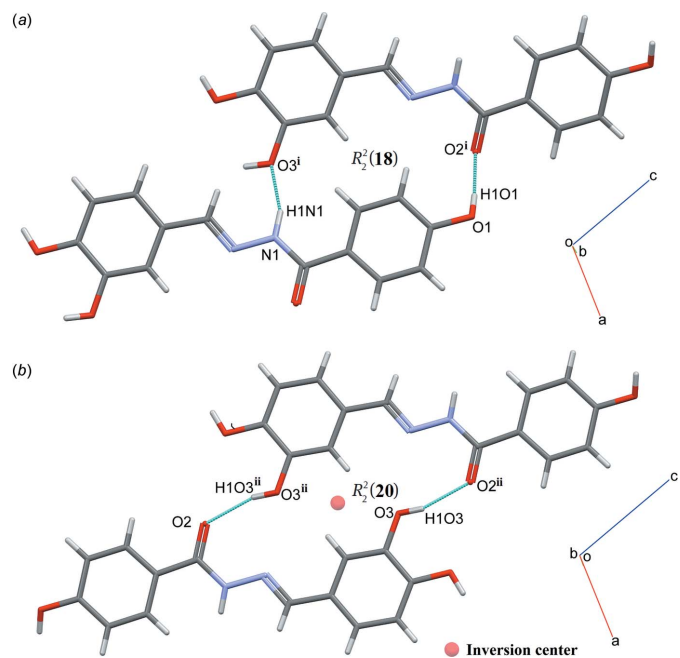
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1O1...O2 <sup>i</sup>	0.80 (2)	1.92 (2)	2.7203 (15)	171 (2)
O3–H1O3...O2 <sup>ii</sup>	0.88 (2)	2.17 (2)	3.0276 (13)	163 (2)
O4–H1O4...O1 <sup>iii</sup>	0.82 (2)	1.93 (2)	2.7379 (16)	166 (2)
N1–H1N1...O3 <sup>i</sup>	0.87 (2)	2.24 (2)	3.0017 (16)	146.1 (19)

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

3,4-dihydroxyphenyl rings are also coplanar to the corresponding azomethine and carbonyl double bonds, with torsion angles N2–C8–C9–C10 [−0.76 (19)°] and C5–C6–C7–O2 [−1.18 (19)°] both in a *syn-periplanar* conformation. Those torsion angles result in an overall flat shape of the title compound with the dihedral angle between the terminal benzene rings being 9.18 (6)°.

## 3. Supramolecular features

In the crystal, molecules are linked by N–H...O and O–H...O hydrogen bonds (Table 1) into a three-dimensional network. Molecules are connected into infinite chains along [011] through an O4–H1O4...O1<sup>iii</sup> hydrogen bond and those chains are further connected into two-dimensional plates parallel to the *ac* plane via N1–H1N1...O3<sup>i</sup> and O1–H1O1...O2<sup>i</sup> hydrogen bonds with an  $R_2^2(18)$  ring motif (Fig. 2a and 3a; symmetry codes as in Table 1). Those plate are interconnected via an O3–H1O3...O2<sup>ii</sup> hydrogen bond with an  $R_2^2(20)$  ring motif, forming a three-dimensional network (Fig. 2b and 3b; symmetry code as in Table 1). In addition, the molecules are further stabilized by  $\pi$ – $\pi$  interactions involving


**Figure 3**

A view of dimers with (a)  $R_2^2(18)$  and (b)  $R_2^2(20)$  ring motifs. [Symmetry codes: (i)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ ; (ii)  $-x + 1, -y + 1, -z$ .]

Table 2

Selected torsion angles ( $^{\circ}$ ) and the dihedral angle ( $^{\circ}$ ) between the terminal benzene rings.

Compound	R	$\tau_1$	$\tau_2$	$\tau_3$	$\tau_4$	Dihedral angle
Planar						
(I)	3,4-dihydroxyphenyl	-1.2	-177.3	-175.0	-0.8	9.2
ABALIA (Fun <i>et al.</i> , 2011)	3-hydroxy-4-methoxyphenyl	3.2	178.4	170.1	-14.2	24.2
CECZOB (Subashini <i>et al.</i> , 2012)	4-chlorophenyl	26.1	-174.4	166.6	-8.9	5.8
CECZUH (Subashini <i>et al.</i> , 2012)	4-bromophenyl	25.6	-174.9	169.0	-7.2	9.8
ESOTUD (Chantrapromma <i>et al.</i> , 2016)	3-methoxyphenyl	-19.4, 20.7	-173.5, -177.8	-175.7, -173.0	1.2, 0.6	24.0, 29.3
HOZBII (Li & Ban, 2009)	4-nitrophenyl	2.0	177.7	178.3	-0.6	2.5
IJUKEE (Zhang, 2011)	4-hydroxy-3-nitrophenyl	-7.2	-177.0	-179.3	6.0	5.5
IRAXEF (Sánchez-Lozano <i>et al.</i> , 2011)	2,4-dihydroxyphenyl	-7.7	-177.8	-177.2	-4.1	6.9
MOZPEX (Ren, 2009)	3,5-dichloro-2-hydroxyphenyl	12.3	178.7	-179.4	-7.3	5.1
ROFMOP (Xue <i>et al.</i> , 2008)	3-bromo-5-chloro-2-hydroxyphenyl	-2.3	175.9	-176.5	-1.3	3.0
TEWLAL (Ayyannan <i>et al.</i> , 2016)	5-bromo-2-hydroxyphenyl	-15.7	-173.6	168.9	3.1	27.0
WACYON (Shalash <i>et al.</i> , 2010)	4-hydroxy-3-methoxyphenyl	-34.2	-175.5	174.7	15.4	28.6
WACXOP (Huang, 2010)	2,4-dichlorophenyl	-14.3	-179.8	-175.0	3.0	7.0
YAGYAI (Horkaew <i>et al.</i> , 2011)	3,4,5-trimethoxyphenyl	-10.6	172.2	175.8	2.8	19.4
YIFPAF (Salhin <i>et al.</i> , 2007)	2-hydroxyphenyl	18.8	179.5	178.7	3.3	21.7
ZAPKOS (Hou, 2012)	3-nitrophenyl	-14.6	169.4	177.4	13.8	9.2
ZIPLAO (Prachumrat <i>et al.</i> , 2018)	2,3-dimethoxyphenyl	9.6	-175.3	172.9	-1.3	9.3
Non-planar						
CABWUA (Meng <i>et al.</i> , 2014)	2-hydroxy-5-methylphenyl	18.4	-178.5	-169.8	8.0	40.8
CEDBAQ (Subashini <i>et al.</i> , 2012)	4-(diethylamino)phenyl	34.9	-178.5	-151.7	8.75	77.3
HUCVIL (Hao, 2009)	2-chlorophenyl	-22.5	-179.2	177.4	-4.2	30.5
HUCWOS (Shi, 2009)	4-methoxyphenyl	-50.9	-177.5	174.8	9.2	46.6
MOSPEQ (Qiu, 2009)	5-chloro-2-hydroxyphenyl	19.0	-178.5	-170.9	7.59	40.2
PAQJID (Gopal Reddy <i>et al.</i> , 2017)	4-ethylphenyl	-39.9	171.1	173.9	7.4	49.9
PAWVUG (Rassem <i>et al.</i> , 2012a)	2-methoxyphenyl	29.1	-166.8	-175.1	19.2	66.6
PEDGOW (Saad <i>et al.</i> , 2012)	3-chlorophenyl	-21.1	179.5	175.3	-9.3	39.0
XEBYUA (Rassem <i>et al.</i> , 2012b)	2-hydroxy-4-methoxyphenyl	28.7	178.1	-169.8	1.3	40.6

both aromatic rings with  $Cg1 \cdots Cg2^{iv} = 3.6480(8) \text{ \AA}$  and  $Cg1 \cdots Cg2^v = 3.7607(8) \text{ \AA}$  [symmetry codes: (iv)  $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ; (v)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ ;  $Cg1$  and  $Cg2$  are the centroids of the C1–C6 and C9–C14 aromatic rings, respectively.]

#### 4. Database survey

A search of the Cambridge Structural Database (CSD version 5.40, last update May 2019; Groom *et al.*, 2016) using (*E*)-*N'*-benzylidene-4-hydroxybenzohydrazide as the reference moiety resulted in 31 structures with different substituents at the benzylidene ring. The different substituent (**R**) together with selected torsion angles,  $\tau_1$  (C5–C6–C7–O2),  $\tau_2$  (C6–C7–N1–N2),  $\tau_3$  (C7–N1–N2–C8) and  $\tau_4$  (N2–C8–C9–C10) as shown in Fig. 4, and the dihedral angle between the terminal aromatic rings are summarized in Table 2. The torsion angles  $\tau_2$  and  $\tau_3$  are *anti-periplanar* ( $151.7$ – $179.8^{\circ}$ ), showing that the hydrazide connecting bridges are nearly planar. As for the torsion angle  $\tau_4$ , all structures adopt a *syn-periplanar* conformation ( $0.6$ – $19.6^{\circ}$ ). Similar to the title compound, the  $\tau_1$  torsion angles for most of the structures are *syn-periplanar* ( $2.0$ – $29.1^{\circ}$ ). However, there are three outliers (CEDBAQ, HUCWOS and PAQJID) whose  $\tau_1$  torsion angles are *syn-clinal* ( $34.9$ – $50.9^{\circ}$ ). By comparing the dihedral angles, the structures can be divided into planar compounds (dihedral angle =  $2.5$ – $29.3^{\circ}$ ) and non-planar compounds (dihedral angle =  $30.5$ – $77.3^{\circ}$ ). In general, as the hydrazide-connecting bridges are nearly planar, relatively flat  $\tau_1$  and  $\tau_4$  torsion angles are observed in the former compounds, while relatively twisted  $\tau_1$  and  $\tau_4$  torsion angles are observed in the latter.

#### 5. Synthesis and crystallization

The title compound (I) was prepared by dissolving 4-hydroxybenzohydrazide (2 mmol, 0.30 g) in ethanol (10 ml). A solution of 3,4-dihydroxybenzaldehyde (2 mmol, 0.28 g) in ethanol (10 ml) was then added to the reaction. The mixture was refluxed for 6 h and the white solid of the product that appeared was collected by filtration, washed with ethanol and dried in air. Colourless single crystals of (I) were obtained after recrystallization from methanol at room temperature for several days.

M.p.  $572$ – $573$  K. UV–Vis (MeOH)  $\lambda_{\text{max}}$  213, 327 nm; FT–IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3121 (O–H stretching), 2800 (C–H aromatic stretching), 1615 (amide C=O stretching), 1570 (C=N stretching), 1506 (C=C stretching of aromatic compound)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $d_6$ -DMSO)  $\delta$  11.39 (*s*, 1H, NH), 10.10 (*s*, 1H, Ar–OH), 8.23 (*s*, 1H, N=CH), 7.77 (*d*,  $J =$

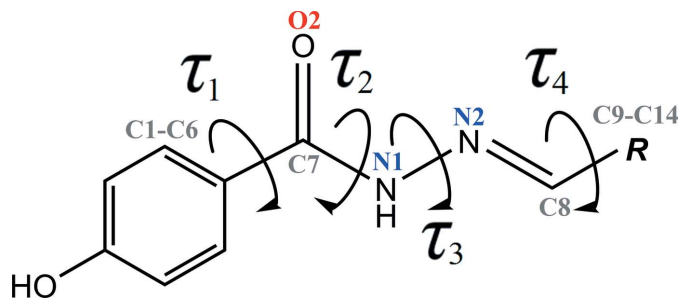


Figure 4  
General chemical diagram, showing torsion angles  $\tau_1$ ,  $\tau_2$ ,  $\tau_3$  and  $\tau_4$  in the benzylidene-4-hydroxybenzohydrazide derivative.

Table 3

Experimental details.

Crystal data	
Chemical formula	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub>
<i>M<sub>r</sub></i>	272.26
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.5352 (8), 7.1711 (5), 15.0606 (10)
$\beta$ (°)	108.548 (2)
<i>V</i> (Å <sup>3</sup> )	1181.10 (14)
<i>Z</i>	4
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.11
Crystal size (mm)	0.80 × 0.21 × 0.07
Data collection	
Diffraction	Bruker APEXII DUO CCD area- detector
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2012)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.924, 0.954
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	22559, 3200, 2453
<i>R<sub>int</sub></i>	0.024
(sin $\theta/\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.686
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.050, 0.161, 1.05
No. of reflections	3200
No. of parameters	197
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.35, -0.19

Computer programs: *APEX2* and *SAINTE* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2009).

8.4 Hz, 2H, Ar—H), 6.84 (*d*, *J* = 8.4, 2H, Ar—H), 9.33 (*s*, 2H, Ar—OH), 7.22 (*s*, 1H, Ar—H), 6.90 (*d*, *J* = 7.8 Hz, 1H, Ar—H), 6.77 (*d*, *J* = 8.1 Hz, 1H, Ar—H).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined using a riding model with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C). All O- and N-bound H atoms were located in a difference-Fourier map and refined freely [O—H = 0.80 (2)–0.88 (2) Å and N—H = 0.87 (2) Å].

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## supporting information

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## Crystal structure of (*E*)-*N'*-(3,4-dihydroxybenzylidene)-4-hydroxybenzohydrazide

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### Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *S SAINT* (Bruker, 2012); data reduction: *S SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXL2013* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

### (*E*)-*N'*-(3,4-Dihydroxybenzylidene)-4-hydroxybenzohydrazide

#### Crystal data

$C_{14}H_{12}N_2O_4$

$M_r = 272.26$

Monoclinic,  $P2_1/c$

$a = 11.5352$  (8) Å

$b = 7.1711$  (5) Å

$c = 15.0606$  (10) Å

$\beta = 108.548$  (2)°

$V = 1181.10$  (14) Å<sup>3</sup>

$Z = 4$

$F(000) = 568$

$D_x = 1.531$  Mg m<sup>-3</sup>

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

Cell parameters from 6293 reflections

$\theta = 2.9$ – $29.2$ °

$\mu = 0.11$  mm<sup>-1</sup>

$T = 296$  K

Plate, colourless

$0.80 \times 0.21 \times 0.07$  mm

#### Data collection

Bruker APEXII DUO CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2012)

$T_{\min} = 0.924$ ,  $T_{\max} = 0.954$

22559 measured reflections

3200 independent reflections

2453 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 29.2$ °,  $\theta_{\min} = 1.9$ °

$h = -15$ → $15$

$k = -9$ → $9$

$l = -20$ → $20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.161$

$S = 1.05$

3200 reflections

197 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2 + 0.1317P]$

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

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

$\Delta\rho_{\max} = 0.35$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.18$  e Å<sup>-3</sup>

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.87093 (9)	0.31797 (18)	0.75119 (6)	0.0493 (3)
O2	0.72990 (8)	0.42338 (15)	0.31133 (6)	0.0439 (3)
O3	0.36583 (9)	0.37514 (15)	-0.12560 (6)	0.0432 (3)
O4	0.11997 (10)	0.35694 (18)	-0.18715 (7)	0.0547 (3)
N1	0.54178 (10)	0.37659 (17)	0.32223 (7)	0.0392 (3)
N2	0.48862 (10)	0.38068 (17)	0.22612 (7)	0.0387 (3)
C1	0.64041 (11)	0.3493 (2)	0.52044 (8)	0.0360 (3)
H1A	0.555998	0.341901	0.493279	0.043*
C2	0.69125 (11)	0.32969 (19)	0.61626 (8)	0.0370 (3)
H2A	0.641478	0.308993	0.653221	0.044*
C3	0.81673 (12)	0.34105 (19)	0.65681 (8)	0.0359 (3)
C4	0.89049 (12)	0.3773 (2)	0.60204 (9)	0.0429 (3)
H4A	0.974622	0.389056	0.629625	0.051*
C5	0.83860 (12)	0.3960 (2)	0.50639 (9)	0.0398 (3)
H5A	0.888474	0.419686	0.469812	0.048*
C6	0.71320 (11)	0.38002 (16)	0.46381 (8)	0.0312 (3)
C7	0.66306 (11)	0.39454 (18)	0.36014 (8)	0.0334 (3)
C8	0.37218 (12)	0.37651 (18)	0.19709 (8)	0.0357 (3)
H8A	0.329660	0.374477	0.240199	0.043*
C9	0.30468 (12)	0.37489 (17)	0.09713 (8)	0.0332 (3)
C10	0.36617 (11)	0.38007 (18)	0.03124 (8)	0.0339 (3)
H10A	0.451058	0.387612	0.051040	0.041*
C11	0.30251 (11)	0.37414 (17)	-0.06263 (8)	0.0332 (3)
C12	0.17505 (12)	0.36368 (19)	-0.09316 (9)	0.0377 (3)
C13	0.11375 (12)	0.3624 (2)	-0.02790 (9)	0.0434 (3)
H13A	0.028756	0.358471	-0.047790	0.052*
C14	0.17809 (12)	0.3669 (2)	0.06705 (9)	0.0400 (3)
H14A	0.136252	0.364586	0.110600	0.048*
H1O1	0.8288 (18)	0.256 (3)	0.7732 (15)	0.074 (6)*
H1O3	0.324 (2)	0.421 (3)	-0.1808 (16)	0.092 (7)*
H1O4	0.046 (2)	0.345 (3)	-0.1964 (16)	0.085 (7)*
H1N1	0.4954 (19)	0.342 (3)	0.3546 (14)	0.069 (6)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0364 (5)	0.0836 (8)	0.0215 (4)	-0.0105 (5)	0.0004 (4)	0.0057 (4)
O2	0.0376 (5)	0.0700 (7)	0.0267 (4)	-0.0036 (4)	0.0139 (4)	-0.0016 (4)
O3	0.0344 (5)	0.0719 (7)	0.0236 (4)	0.0037 (4)	0.0095 (4)	0.0026 (4)

O4	0.0352 (6)	0.0991 (9)	0.0227 (5)	-0.0083 (5)	-0.0009 (4)	0.0033 (5)
N1	0.0326 (6)	0.0651 (7)	0.0185 (5)	-0.0038 (5)	0.0060 (4)	0.0023 (4)
N2	0.0380 (6)	0.0576 (7)	0.0182 (5)	-0.0033 (5)	0.0056 (4)	0.0010 (4)
C1	0.0254 (6)	0.0554 (7)	0.0248 (6)	-0.0019 (5)	0.0046 (4)	-0.0004 (5)
C2	0.0319 (6)	0.0558 (8)	0.0232 (6)	-0.0014 (5)	0.0086 (5)	0.0005 (5)
C3	0.0335 (6)	0.0485 (7)	0.0220 (5)	-0.0024 (5)	0.0037 (5)	-0.0001 (5)
C4	0.0272 (6)	0.0688 (9)	0.0289 (6)	-0.0039 (6)	0.0037 (5)	0.0022 (6)
C5	0.0307 (6)	0.0611 (8)	0.0284 (6)	-0.0029 (5)	0.0105 (5)	0.0014 (5)
C6	0.0300 (6)	0.0411 (6)	0.0213 (5)	0.0007 (4)	0.0063 (4)	-0.0010 (4)
C7	0.0337 (6)	0.0440 (7)	0.0222 (6)	-0.0001 (5)	0.0086 (5)	-0.0019 (4)
C8	0.0352 (7)	0.0481 (7)	0.0231 (6)	0.0003 (5)	0.0081 (5)	0.0012 (5)
C9	0.0332 (6)	0.0416 (6)	0.0219 (5)	0.0005 (5)	0.0046 (5)	0.0009 (4)
C10	0.0261 (5)	0.0492 (7)	0.0234 (6)	0.0004 (5)	0.0038 (4)	-0.0001 (5)
C11	0.0311 (6)	0.0443 (7)	0.0234 (5)	0.0008 (5)	0.0073 (4)	0.0010 (4)
C12	0.0313 (6)	0.0521 (7)	0.0243 (6)	-0.0013 (5)	0.0012 (5)	0.0026 (5)
C13	0.0255 (6)	0.0686 (9)	0.0321 (7)	-0.0003 (5)	0.0038 (5)	0.0046 (6)
C14	0.0329 (6)	0.0582 (8)	0.0298 (6)	0.0015 (5)	0.0110 (5)	0.0037 (5)

*Geometric parameters (Å, °)*

O1—C3	1.3688 (14)	C4—C5	1.3794 (18)
O1—H1O1	0.80 (2)	C4—H4A	0.9300
O2—C7	1.2403 (15)	C5—C6	1.3880 (18)
O3—C11	1.3688 (15)	C5—H5A	0.9300
O3—H1O3	0.88 (2)	C6—C7	1.4861 (15)
O4—C12	1.3556 (15)	C8—C9	1.4584 (16)
O4—H1O4	0.83 (3)	C8—H8A	0.9300
N1—C7	1.3385 (17)	C9—C14	1.3856 (18)
N1—N2	1.3811 (14)	C9—C10	1.3918 (17)
N1—H1N1	0.87 (2)	C10—C11	1.3709 (16)
N2—C8	1.2738 (17)	C10—H10A	0.9300
C1—C2	1.3812 (16)	C11—C12	1.3959 (18)
C1—C6	1.3916 (17)	C12—C13	1.3815 (19)
C1—H1A	0.9300	C13—C14	1.3861 (17)
C2—C3	1.3828 (18)	C13—H13A	0.9300
C2—H2A	0.9300	C14—H14A	0.9300
C3—C4	1.3852 (19)		
C3—O1—H1O1	111.0 (15)	O2—C7—N1	121.71 (11)
C11—O3—H1O3	113.5 (15)	O2—C7—C6	121.79 (11)
C12—O4—H1O4	107.2 (16)	N1—C7—C6	116.49 (11)
C7—N1—N2	120.02 (11)	N2—C8—C9	120.86 (12)
C7—N1—H1N1	122.4 (13)	N2—C8—H8A	119.6
N2—N1—H1N1	116.6 (13)	C9—C8—H8A	119.6
C8—N2—N1	115.30 (11)	C14—C9—C10	119.41 (11)
C2—C1—C6	121.20 (11)	C14—C9—C8	119.93 (12)
C2—C1—H1A	119.4	C10—C9—C8	120.66 (11)
C6—C1—H1A	119.4	C11—C10—C9	120.47 (11)

C1—C2—C3	119.49 (11)	C11—C10—H10A	119.8
C1—C2—H2A	120.3	C9—C10—H10A	119.8
C3—C2—H2A	120.3	O3—C11—C10	119.04 (11)
O1—C3—C2	121.31 (12)	O3—C11—C12	120.68 (11)
O1—C3—C4	118.49 (11)	C10—C11—C12	120.26 (11)
C2—C3—C4	120.20 (11)	O4—C12—C13	124.53 (12)
C5—C4—C3	119.69 (12)	O4—C12—C11	116.14 (12)
C5—C4—H4A	120.2	C13—C12—C11	119.33 (11)
C3—C4—H4A	120.2	C12—C13—C14	120.43 (12)
C4—C5—C6	121.12 (12)	C12—C13—H13A	119.8
C4—C5—H5A	119.4	C14—C13—H13A	119.8
C6—C5—H5A	119.4	C9—C14—C13	120.07 (12)
C5—C6—C1	118.24 (11)	C9—C14—H14A	120.0
C5—C6—C7	118.67 (11)	C13—C14—H14A	120.0
C1—C6—C7	123.09 (11)		
C7—N1—N2—C8	-174.98 (12)	N1—N2—C8—C9	-178.29 (10)
C6—C1—C2—C3	0.1 (2)	N2—C8—C9—C14	178.83 (12)
C1—C2—C3—O1	-178.44 (12)	N2—C8—C9—C10	-0.76 (19)
C1—C2—C3—C4	1.9 (2)	C14—C9—C10—C11	-1.11 (18)
O1—C3—C4—C5	178.19 (13)	C8—C9—C10—C11	178.47 (11)
C2—C3—C4—C5	-2.1 (2)	C9—C10—C11—O3	-178.51 (12)
C3—C4—C5—C6	0.3 (2)	C9—C10—C11—C12	0.29 (19)
C4—C5—C6—C1	1.6 (2)	O3—C11—C12—O4	-0.71 (19)
C4—C5—C6—C7	-177.78 (12)	C10—C11—C12—O4	-179.50 (12)
C2—C1—C6—C5	-1.9 (2)	O3—C11—C12—C13	179.81 (13)
C2—C1—C6—C7	177.51 (12)	C10—C11—C12—C13	1.03 (19)
N2—N1—C7—O2	3.3 (2)	O4—C12—C13—C14	179.03 (13)
N2—N1—C7—C6	-177.33 (10)	C11—C12—C13—C14	-1.5 (2)
C5—C6—C7—O2	-1.18 (19)	C10—C9—C14—C13	0.60 (19)
C1—C6—C7—O2	179.44 (13)	C8—C9—C14—C13	-178.99 (12)
C5—C6—C7—N1	179.42 (12)	C12—C13—C14—C9	0.7 (2)
C1—C6—C7—N1	0.04 (18)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1O1 $\cdots$ O2 <sup>i</sup>	0.80 (2)	1.92 (2)	2.7203 (15)	171 (2)
O3—H1O3 $\cdots$ O2 <sup>ii</sup>	0.88 (2)	2.17 (2)	3.0276 (13)	163 (2)
O4—H1O4 $\cdots$ O1 <sup>iii</sup>	0.82 (2)	1.93 (2)	2.7379 (16)	166 (2)
N1—H1N1 $\cdots$ O3 <sup>i</sup>	0.87 (2)	2.24 (2)	3.0017 (16)	146.1 (19)

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