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(*S*)-*N*-(1-Benzyl-2-hydroxyethyl)phthalamic acid

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.012 Å
Disorder in main residue
R factor = 0.075
wR factor = 0.130
Data-to-parameter ratio = 8.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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A feature of the structure of the title compound, $C_{17}H_{17}NO_4$, is the three-dimensional connectivity generated by intermolecular hydrogen bonds.

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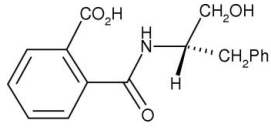
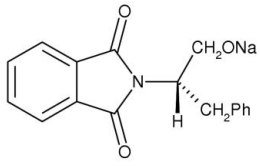
Comment

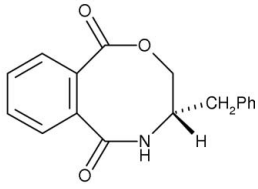
There have been only a few authenticated reports of the interesting [1,4]-oxazocine-5,8-dione ring system exemplified by (III). Aly (2003) used a strategy involving reaction of a diethyl phthalate with a 2-aminophenol derivative to generate the lactone and lactam functions. Assoumatine *et al.* (2004) applied the analogous reaction of a succinate diester with a β-amino alcohol derivative. Conceptually the system might also be constructed from a phthaloyl β-amino alcohol, for example (II), by intramolecular nucleophilic attack of the derived side chain alkoxide on a carbonyl group, involving carbon–oxygen bond formation followed by carbon–nitrogen bond cleavage to yield the eight-membered ring system (III). However, when the Na salt of (*S*)-*N*-(1-benzyl-2-hydroxyethyl)phthalimide, (II), was generated in dry tetrahydrofuran (THF), subsequent quenching with water resulted in hydrolysis to yield the title compound, (I), rather than the oxazocine dione, (III).



(I)



(II)



(III)

The molecule of (I) is shown in Fig. 1. Although the refinement of the structure has been compromised somewhat by limitations imposed by comparatively poor intensity data obtained from a weakly diffracting sample crystal and by disorder affecting the C12–C17 phenyl group (see later for details), the molecular geometry is well enough determined to

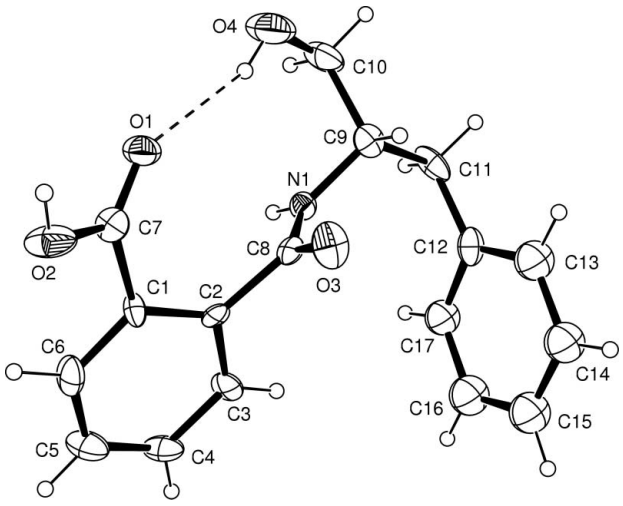


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii. The intramolecular hydrogen bond is indicated by the dashed line. Only the major component (see text) of the disordered C12–C17 phenyl group is shown.

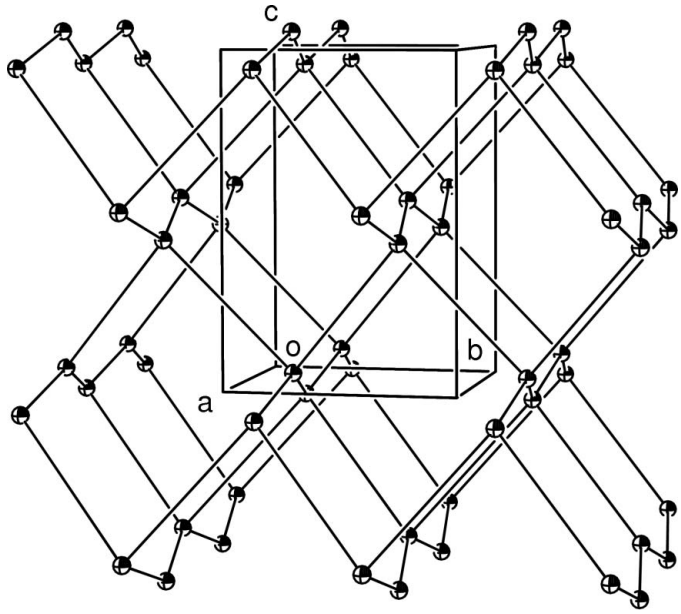


Figure 2
A schematic representation of the intermolecular hydrogen bonding in (I). The hydrogen bonds are represented by lines joining pseudo-atoms of arbitrary size which are coincident with the centroids of the molecules.

show that the bond lengths and bond angles lie within the usual ranges and do not merit further discussion here. The main interest in this structure lies in the hydrogen bonds given in Table 1. The first of these is intramolecular, creating a ten-membered ring (see Fig. 1). The other two are intermolecular and provide three-dimensional interconnection of the molecules as indicated schematically in Fig. 2. Notable in this figure is the predominance of six-membered, *i.e.* hexa-molecular, hydrogen-bonded rings. One such ring is shown in detail in Fig. 3. Here it can be seen that hydrogen bonds of the form O2—H2···O4^{i} [symmetry code: (i) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$] create infinite zigzag chains of molecules propagated in the *b* direction. Two such chains pass through the unit cell, one comprising the molecule of the asymmetric unit together with the molecules with symmetry codes (i) and (iii), and the other comprising the molecules with symmetry codes (iv), (v) and (vi) [symmetry codes as in Table 1]. Hydrogen bonds of the form N1—H1···O3^{ii}, on the other hand, create chains of molecules propagated in the *a* direction. These interconnect the chains previously described, by connecting molecules such as those in Fig. 3 with symmetry codes (iii) and (iv), and, in the process, complete the three-dimensional connectivity. The propagation of the chains in the direction of *a* just described also brings about a C—H···π interaction of the form C13*A*—H13*A*···*Cg*1^{vi} (*Cg*1 is the centroid of the C1–C6 ring) in which the critical parameters are the H···*Cg* distance and the C—H···*Cg* angle of 2.985 Å and 165°, respectively.

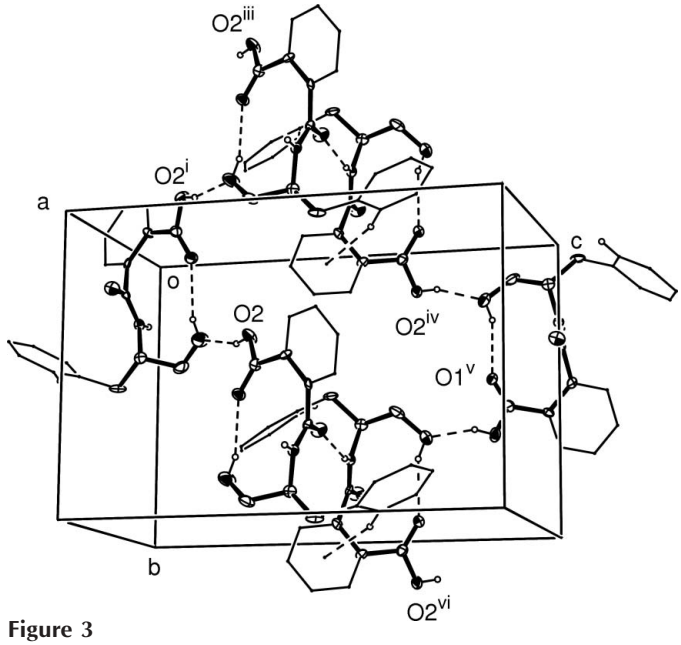


Figure 3
A view of the unit cell contents of (I). For clarity, bonds in the C1–C6 and C12–C17 rings are drawn as thin lines. Displacement ellipsoids are drawn at the 20% probability level. H atoms involved in intermolecular contacts (dashed lines) are shown as small circles of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (i) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $x, y-1, z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (v) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (vi) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$.]

Experimental

Compound (I) was prepared by slow addition of (*S*)-*N*-(1-benzyl-2-hydroxyethyl)phthalimide (3.0 g, 10 mmol) to NaH (0.51 g, 17 mmol) in dry THF (20 ml) contained in a flask fitted with a $CaCl_2$ guard tube. After approximately 45 min, when hydrogen evolution had ceased, water (5 ml) was added slowly to remove excess sodium hydride. The mixture was then stirred for a further 10 min, and water (30 ml) and dilute HCl (10 ml) were added. The milky suspension was extracted three times with diethyl ether, the ether extracts were combined and dried over anhydrous magnesium sulfate, and the solvent was removed under reduced pressure to yield a white powdery solid which, on recrystallization from acetone, afforded (I) as colourless crystals [yield 1.6 g, 50%; m.p. 401–403 K (dec.)]. IR (cm^{-1}): ν_{max} 3488 (OH), 3302 (NH), 1701 and 1663 (C=O); ^{1}H NMR (DMSO,

p.p.m.): δ_H 2.73 (*dd*, 1H, J = 11.2 and 6.8 Hz, $PhCH_AH_{\underline{B}}$), 2.97 (*dd*, 1H, J = 11.2 and 3.2 Hz, $PhCH_{\underline{A}}H_B$), 3.38 (*dd*, 1H, J = 11.4 and 3.6 Hz, $CH_AH_{\underline{B}}OH$), 3.59 (*dd*, 1H, J = 11.4 and 7.2 Hz, $CH_{\underline{A}}H_BOH$), 4.05 (*m*, 1H, CH), 4.61 (*br s*, 1H, OH), 7.14–7.78 (*m*, 9H, aromatic), 8.23 (*d*, 1H, J = 8.4 Hz, NH) and 13.48 [*br s*, 1H, OH (acid)]; ^{13}C NMR (DMSO, p.p.m.): δ_C 35.7 ($PhCH_2$), 52.4 (CH_2OH), 62.0 (CH), 125.4, 126.9, 127.6, 128.5, 128.6, 128.7, 130.0, 138.3, 138.9 (aromatic), 167.5 and 167.8 (C═O). Analysis found: C 68.10, H 5.77, N 4.63%; $C_{17}H_{17}NO_4$ requires: C 68.22, H 5.72, N 4.68%.

Crystal data

$C_{17}H_{17}NO_4$
M_r = 299.32
Orthorhombic, $P2_12_12_1$
a = 9.569 (7) Å
b = 10.723 (6) Å
c = 15.416 (6) Å
V = 1581.8 (16) Å^3
Z = 4
D_x = 1.257 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 reflections
θ = 7.9–9.6°
μ = 0.09 mm^{-1}
T = 298 (2) K
Plate, colourless
0.55 × 0.30 × 0.10 mm

Data collection

Nicolet *P*3 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1626 measured reflections
1625 independent reflections
618 reflections with $I > 2\sigma(I)$
R_{int} = 0.008
θ_{max} = 25.1°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 18$
2 standard reflections every 50 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.130
S = 0.85
1625 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4O···O1	0.82	2.25	3.032 (8)	159
O2—H2···O4^{i}	0.82	1.82	2.591 (8)	156
N1—H1···O3^{ii}	0.87 (2)	2.09 (4)	2.883 (8)	149 (6)

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

The data set contains no Friedel pairs, and there is no significant anomalous scattering. The enantiomer chosen for the structural model is that for which the absolute configuration at the chiral centre (C9) is the same as in the phenylalanol precursor. Disorder of the C12–C17 phenyl group over two orientations related by a twist about the C11—C12 bond of approximately 20° was modelled by splitting the disordered atoms (C13–C17) into pairs as C13*A*/C13*B* with occupancies, as determined by refinement with the displacement parameters for the atoms of each pair constrained to be equal and isotropic displacement parameters for all atom pairs, of 0.61 (2) and 0.39 (2) for the major (suffix *A*) and minor (suffix *B*) components of the disorder, respectively. Restraints were applied to the disordered phenyl rings in terms of both their planarity (target r.m.s. displacement 0.02 Å) and their bond lengths and bond angles by means of a variable *d* for bond lengths and 1.732*d* for 1,3 distances for internal angles, respectively. The value of *d* after refinement was 1.382 (6) Å. In the final stages of refinement H atoms attached to C atoms were placed in calculated positions with C—H = 0.93, 0.97 and 0.98 Å for aryl, methylene and tertiary C atoms, respectively. The H atom of the NH group was placed initially as for an aryl H atom but its coordinates were then refined. Difference map peaks provided initial positions for the H atoms of the OH groups. The groups were then idealized and their torsion angles refined. In all cases the H atoms were refined with a riding model with $U_{iso}(H) = 1.2U_{eq}(C,N,O)$.

Data collection: *Nicolet P3 Software* (Nicolet, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

PM thanks Dublin City University for a studentship.

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supporting information

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Melting point: 401-403 (dec.) K
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Cell parameters from 14 reflections
θ = 7.9–9.6°
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T = 298 K
Plate, colourless
0.55 × 0.30 × 0.10 mm

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Nicolet P3 four-circle diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
1626 measured reflections
1625 independent reflections
618 reflections with $I > 2\sigma(I)$
R_{int} = 0.008
θ_{max} = 25.1°, θ_{min} = 2.3°
h = 0→11
k = 0→12
l = 0→18
2 standard reflections every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.130
S = 0.85
1625 reflections
196 parameters
31 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
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Experimental. Scan rates, dependent on prescan intensity (Ip), were in the range 58.6 (Ip>2500) to 5.33 (Ip<150) ° 2θ min^{-1}. Scan widths, dependent on 2θ, were in the range 2.4 to 2.7 ° 2θ. Stationary crystal, stationary counter background counts were taken on either side of the peak each for 25% of the total (peak plus background) count time.

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.
Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)
1.8355 (1073) *x* + 9.0224 (437) *y* + 7.7883 (762) *z* = 14.4770 (478)
* -0.0057 (0.0041) C12 * -0.0002 (0.0020) C13A_a * 0.0063 (0.0042) C14A_a * -0.0065 (0.0066) C15A_a * 0.0005 (0.0074) C16A_a * 0.0057 (0.0065) C17A_a -1.7277 (0.0158) C9 - 2.0892 (0.0234) C10 - 0.2565 (0.0146) C11
Rms deviation of fitted atoms = 0.0050
4.9368 (1220) *x* + 8.3 (987) *y* + 5.5912 (1591) *z* = 14.4245 (554)
Angle to previous plane (with approximate e.s.d.) = 20.74 (1.31)
* -0.0040 (0.0044) C12 * 0.0003 (0.0023) C13B_b * 0.0036 (0.0047) C14B_b * -0.0036 (0.0073) C15B_b * -0.0002 (0.0081) C16B_b * 0.0040 (0.0071) C17B_b -1.3705 (0.0274) C9 - 1.4237 (0.0439) C10 0.0353 (0.0221) C11
Rms deviation of fitted atoms = 0.0031
7.9027 (174) *x* + 2.9195 (316) *y* - 7.6120 (408) *z* = 3.0308 (341)
Angle to previous plane (with approximate e.s.d.) = 62.72 (0.78)
* 0.0025 (0.0057) C1 * -0.0030 (0.0056) C2 * -0.0028 (0.0058) C3 * 0.0092 (0.0068) C4 * -0.0098 (0.0065) C5 * 0.0038 (0.0056) C6
Rms deviation of fitted atoms = 0.0060
1.8355 (1073) *x* + 9.0224 (437) *y* + 7.7883 (762) *z* = 14.4770 (478)
Angle to previous plane (with approximate e.s.d.) = 82.07 (0.59)
* -0.0057 (0.0041) C12 * -0.0002 (0.0020) C13A_a * 0.0063 (0.0042) C14A_a * -0.0065 (0.0066) C15A_a * 0.0005 (0.0074) C16A_a * 0.0057 (0.0065) C17A_a
Rms deviation of fitted atoms = 0.0050

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}	Occ. (<1)
C1	0.6558 (8)	0.4460 (8)	0.4535 (5)	0.032 (2)	
C2	0.6717 (8)	0.5481 (7)	0.5098 (5)	0.032 (2)	
C3	0.7512 (8)	0.5306 (7)	0.5856 (6)	0.040 (2)	
H3	0.7632	0.5963	0.6243	0.048*	
C4	0.8122 (10)	0.4146 (8)	0.6029 (5)	0.050 (3)	
H4	0.8664	0.4043	0.6524	0.059*	
C5	0.7932 (10)	0.3173 (8)	0.5483 (6)	0.057 (3)	
H5	0.8315	0.2398	0.5613	0.068*	
C6	0.7165 (9)	0.3331 (7)	0.4729 (6)	0.048 (3)	
H6	0.7058	0.2664	0.4349	0.058*	
C7	0.5831 (9)	0.4630 (9)	0.3677 (5)	0.040 (2)	
O1	0.5804 (6)	0.5591 (5)	0.3276 (3)	0.0494 (18)	
O2	0.5259 (7)	0.3581 (5)	0.3411 (4)	0.063 (2)	
H2	0.4762	0.3720	0.2989	0.076*	
C8	0.5951 (9)	0.6701 (7)	0.4950 (5)	0.031 (2)	
O3	0.4697 (6)	0.6716 (5)	0.4985 (4)	0.0549 (18)	
N1	0.6798 (7)	0.7637 (6)	0.4781 (4)	0.0333 (18)	
H1	0.771 (2)	0.757 (6)	0.475 (4)	0.040*	
C9	0.6251 (8)	0.8885 (7)	0.4603 (6)	0.040 (2)	

H9	0.5229	0.8841	0.4630	0.048*	
C10	0.6657 (11)	0.9206 (7)	0.3672 (6)	0.065 (3)	
H10A	0.6385	1.0059	0.3547	0.078*	
H10B	0.7664	0.9148	0.3610	0.078*	
O4	0.6011 (9)	0.8397 (6)	0.3068 (4)	0.087 (2)	
H4O	0.6018	0.7683	0.3258	0.104*	
C11	0.6732 (9)	0.9838 (6)	0.5275 (6)	0.049 (3)	
H11A	0.7738	0.9937	0.5240	0.059*	
H11B	0.6304	1.0640	0.5153	0.059*	
C12	0.6336 (8)	0.9425 (7)	0.6170 (5)	0.048 (3)	
C13A	0.4920 (15)	0.9465 (16)	0.6464 (9)	0.067 (4)*	0.61 (2)
H13A	0.4271	0.9888	0.6125	0.080*	0.61 (2)
C14A	0.4450 (16)	0.8907 (19)	0.7229 (9)	0.068 (5)*	0.61 (2)
H14A	0.3515	0.8975	0.7388	0.082*	0.61 (2)
C15A	0.5364 (18)	0.8258 (17)	0.7749 (10)	0.073 (4)*	0.61 (2)
H15A	0.5072	0.7866	0.8256	0.087*	0.61 (2)
C16A	0.674 (2)	0.8220 (17)	0.7477 (12)	0.073 (3)*	0.61 (2)
H16A	0.7388	0.7795	0.7818	0.088*	0.61 (2)
C17A	0.7211 (18)	0.8782 (14)	0.6723 (11)	0.057 (3)*	0.61 (2)
H17A	0.8154	0.8724	0.6583	0.069*	0.61 (2)
C13B	0.5116 (19)	0.997 (2)	0.6448 (14)	0.067 (4)*	0.39 (2)
H13B	0.4613	1.0504	0.6093	0.080*	0.39 (2)
C14B	0.467 (2)	0.968 (3)	0.7274 (14)	0.068 (5)*	0.39 (2)
H14B	0.3850	1.0022	0.7501	0.082*	0.39 (2)
C15B	0.547 (3)	0.888 (3)	0.7742 (15)	0.073 (4)*	0.39 (2)
H15B	0.5164	0.8682	0.8297	0.087*	0.39 (2)
C16B	0.669 (3)	0.834 (3)	0.7472 (18)	0.073 (3)*	0.39 (2)
H16B	0.7194	0.7805	0.7829	0.088*	0.39 (2)
C17B	0.715 (2)	0.8630 (19)	0.6644 (16)	0.057 (3)*	0.39 (2)
H17B	0.7979	0.8300	0.6421	0.069*	0.39 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.026 (5)	0.028 (5)	0.042 (5)	0.008 (5)	−0.001 (5)	−0.010 (5)
C2	0.032 (5)	0.039 (5)	0.024 (5)	0.001 (5)	0.016 (5)	0.009 (5)
C3	0.053 (6)	0.020 (5)	0.047 (6)	−0.011 (5)	0.000 (5)	0.004 (5)
C4	0.057 (7)	0.053 (6)	0.039 (6)	−0.005 (6)	−0.018 (5)	0.016 (5)
C5	0.063 (7)	0.050 (6)	0.057 (7)	0.002 (6)	−0.020 (6)	0.018 (6)
C6	0.056 (6)	0.031 (5)	0.057 (7)	−0.008 (5)	0.008 (6)	−0.017 (5)
C7	0.037 (5)	0.042 (6)	0.041 (6)	−0.005 (6)	−0.003 (5)	−0.003 (6)
O1	0.068 (4)	0.040 (4)	0.040 (4)	−0.001 (4)	−0.014 (4)	0.005 (3)
O2	0.092 (5)	0.056 (4)	0.043 (4)	−0.035 (4)	−0.025 (4)	0.012 (3)
C8	0.041 (5)	0.033 (5)	0.019 (5)	−0.004 (5)	−0.007 (5)	−0.010 (5)
O3	0.022 (3)	0.060 (4)	0.083 (5)	−0.004 (4)	0.005 (3)	−0.009 (4)
N1	0.029 (4)	0.038 (4)	0.033 (5)	0.006 (4)	−0.001 (4)	−0.001 (4)
C9	0.025 (5)	0.042 (5)	0.052 (6)	−0.001 (5)	0.002 (5)	0.001 (5)
C10	0.083 (8)	0.042 (6)	0.071 (8)	0.011 (6)	0.000 (7)	0.025 (6)

O4	0.141 (7)	0.059 (4)	0.060 (5)	0.003 (6)	−0.038 (5)	0.014 (4)
C11	0.050 (6)	0.018 (5)	0.081 (8)	−0.004 (5)	−0.023 (6)	−0.002 (5)
C12	0.051 (6)	0.038 (6)	0.055 (6)	0.001 (5)	0.003 (5)	−0.019 (5)

Geometric parameters (Å, º)

C1—C6	1.376 (9)	C11—C12	1.497 (10)
C1—C2	1.406 (10)	C11—H11A	0.9700
C1—C7	1.506 (10)	C11—H11B	0.9700
C2—C3	1.406 (9)	C12—C17B	1.369 (17)
C2—C8	1.517 (10)	C12—C13B	1.373 (16)
C3—C4	1.400 (10)	C12—C17A	1.380 (14)
C3—H3	0.9300	C12—C13A	1.430 (14)
C4—C5	1.353 (10)	C13A—C14A	1.397 (14)
C4—H4	0.9300	C13A—H13A	0.9300
C5—C6	1.385 (10)	C14A—C15A	1.375 (14)
C5—H5	0.9300	C14A—H14A	0.9300
C6—H6	0.9300	C15A—C16A	1.386 (15)
C7—O1	1.202 (9)	C15A—H15A	0.9300
C7—O2	1.316 (8)	C16A—C17A	1.383 (14)
O2—H2	0.8200	C16A—H16A	0.9300
C8—O3	1.201 (8)	C17A—H17A	0.9300
C8—N1	1.316 (9)	C13B—C14B	1.378 (17)
N1—C9	1.463 (9)	C13B—H13B	0.9300
N1—H1	0.87 (2)	C14B—C15B	1.361 (16)
C9—C11	1.526 (9)	C14B—H14B	0.9300
C9—C10	1.527 (10)	C15B—C16B	1.369 (18)
C9—H9	0.9800	C15B—H15B	0.9300
C10—O4	1.415 (9)	C16B—C17B	1.385 (18)
C10—H10A	0.9700	C16B—H16B	0.9300
C10—H10B	0.9700	C17B—H17B	0.9300
O4—H4O	0.8200		
C6—C1—C2	120.4 (8)	C12—C11—H11A	109.5
C6—C1—C7	119.5 (8)	C9—C11—H11A	109.5
C2—C1—C7	119.9 (8)	C12—C11—H11B	109.5
C1—C2—C3	117.9 (8)	C9—C11—H11B	109.5
C1—C2—C8	121.8 (7)	H11A—C11—H11B	108.1
C3—C2—C8	120.1 (7)	C17B—C12—C13B	125.7 (16)
C4—C3—C2	120.2 (7)	C17A—C12—C13A	113.2 (12)
C4—C3—H3	119.9	C17B—C12—C11	122.1 (14)
C2—C3—H3	119.9	C13B—C12—C11	112.2 (12)
C5—C4—C3	120.7 (8)	C17A—C12—C11	124.3 (11)
C5—C4—H4	119.6	C13A—C12—C11	121.5 (10)
C3—C4—H4	119.6	C14A—C13A—C12	124.1 (13)
C4—C5—C6	119.9 (9)	C14A—C13A—H13A	118.0
C4—C5—H5	120.0	C12—C13A—H13A	118.0
C6—C5—H5	120.0	C15A—C14A—C13A	120.3 (13)

C1—C6—C5	120.9 (8)	C15A—C14A—H14A	119.9
C1—C6—H6	119.5	C13A—C14A—H14A	119.9
C5—C6—H6	119.5	C14A—C15A—C16A	116.4 (14)
O1—C7—O2	124.4 (8)	C14A—C15A—H15A	121.8
O1—C7—C1	124.4 (9)	C16A—C15A—H15A	121.8
O2—C7—C1	111.2 (8)	C17A—C16A—C15A	123.3 (16)
C7—O2—H2	109.5	C17A—C16A—H16A	118.3
O3—C8—N1	127.9 (9)	C15A—C16A—H16A	118.3
O3—C8—C2	119.1 (8)	C12—C17A—C16A	122.7 (15)
N1—C8—C2	113.0 (7)	C12—C17A—H17A	118.6
C8—N1—C9	121.0 (7)	C16A—C17A—H17A	118.6
C8—N1—H1	124 (5)	C12—C13B—C14B	117.5 (18)
C9—N1—H1	115 (5)	C12—C13B—H13B	121.3
N1—C9—C11	112.2 (7)	C14B—C13B—H13B	121.3
N1—C9—C10	106.9 (7)	C15B—C14B—C13B	116.8 (19)
C11—C9—C10	114.3 (7)	C15B—C14B—H14B	121.6
N1—C9—H9	107.7	C13B—C14B—H14B	121.6
C11—C9—H9	107.7	C14B—C15B—C16B	126 (2)
C10—C9—H9	107.7	C14B—C15B—H15B	116.9
O4—C10—C9	111.7 (7)	C16B—C15B—H15B	116.9
O4—C10—H10A	109.3	C15B—C16B—C17B	117 (2)
C9—C10—H10A	109.3	C15B—C16B—H16B	121.4
O4—C10—H10B	109.3	C17B—C16B—H16B	121.4
C9—C10—H10B	109.3	C12—C17B—C16B	117 (2)
H10A—C10—H10B	107.9	C12—C17B—H17B	121.7
C10—O4—H4O	109.5	C16B—C17B—H17B	121.7
C12—C11—C9	110.5 (6)		
C10—C11—C12—C13A	−74.9 (13)	C10—C11—C12—C13B	−99.0 (13)
C10—C11—C12—C17A	93.3 (14)	C10—C11—C12—C17B	83.1 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*O*···O1	0.82	2.25	3.032 (8)	159
O2—H2···O4^{i}	0.82	1.82	2.591 (8)	156
N1—H1···O3^{ii}	0.87 (2)	2.09 (4)	2.883 (8)	149 (6)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) *x*+1/2, −*y*+3/2, −*z*+1.