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(*S*)-3-[4-(Benzyloxy)phenyl]-2-hydroxypropanoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.070
wR factor = 0.204
Data-to-parameter ratio = 7.4

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

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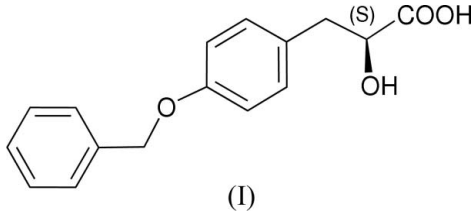
The title compound, $C_{16}H_{16}O_4$, has been obtained by the reaction of *O*-benzylated L-tyrosine with sodium nitrite as colorless blocks. The packing of the title compound exhibits two independent hydrogen bonds involving the hydroxy and carboxylic groups, giving rise to an infinite ladder parallel to the *b* axis.

Comment

The title compound, (I), is a key intermediate and widely used in the synthesis of PPARa/g dual agonists (Haigh *et al.*, 1999) and heteropeptides (Valls *et al.*, 2002). Much research has been carried out, but there are still some drawbacks in the existing synthetic processes. During our continuing study on asymmetric synthesis (Zeng, Liu, Cui *et al.* 2002; Zeng, Liu, Mi *et al.* 2002), we found a practical route for synthesis of the title compound, (I).



(I)

The two benzene ring of (I) are essentially coplanar. The packing exhibits wo independent hydrogen bonds involving the hydroxy and carboxylic acid groups (Fig. 2), forming an infinite ladder parallel to the *b* axis.

Experimental

To a solution of 1*M* sulfuric acid (39 ml) and DMF (19 ml), *O*-benzylated L-tyrosine (3.207 g) was added. The suspension was stirred until it dissolved and was then cooled with iced water. A solution of sodium nitrite (4.067 g) in water (10 ml) was added dropwise to the resulting solution. After one hour, 3.2 *M* sulfuric acid (9.8 ml) was added slowly, and the resulting solution was stirred overnight. The reaction mixture was extracted with ethyl acetate, and the organic layer was washed with water and saturated salt solution. It was then dried over anhydrous magnesium sulfate and filtered. The solvent was removed under reduced pressure, and a yellow liquid (3.090 g) was obtained in 96.4% crude yield. The crude product was recrystallized to give crystals (1.417 g) in 43.3% yield. ^{1}H NMR (600 MHz, $CDCl_3$): δ 2.60 (*br*, 2H), 2.96 (*dd*, 1H, J_1 = 14.4 Hz, J_2 = 7.8 Hz), 3.17 (*dd*, 1H, J_1 = 14.4 Hz, J_2 = 4.2 Hz), 4.49 (*dd*, 1H, J_1 = 7.2 Hz, J_2 = 4.2 Hz), 5.05 (*s*, 2H), 6.94 (*d*, 2H, *J* = 8.4 Hz), 7.18 (*d*, 2H, *J* = 8.4 Hz), 7.33 (*t*, 1H, *J* = 7.2 Hz), 7.39 (*t*, 2H, *J* = 7.2 Hz), 7.43 (*d*, 2H, *J* = 7.2 Hz) ^{13}C NMR (150 MHz, CD_3COCD_3): δ 39.6, 69.7, 71.4, 114.6, 127.6, 127.8, 128.5, 130.1, 130.7, 137.9, 157.8, 174.5.

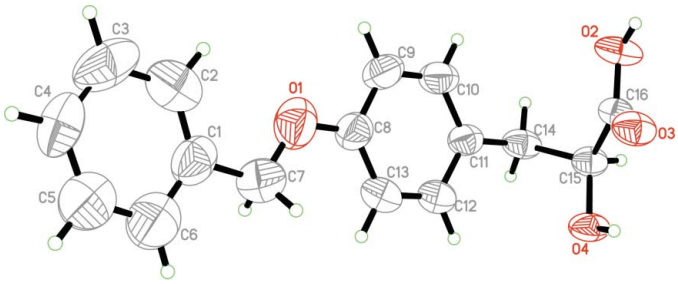


Figure 1
ORTEP3 (Farrugia, 1997) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Crystal data

$C_{16}H_{16}O_4$	D_x = 1.338 Mg m^{-3}
M_r = 272.29	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2099 reflections
a = 8.532 (4) Å	θ = 2.5–27.7°
b = 5.782 (2) Å	μ = 0.10 mm^{-1}
c = 14.050 (6) Å	T = 293 (2) K
β = 102.784 (7)°	Chunk, colorless
V = 676.0 (5) Å^3	0.50 × 0.40 × 0.20 mm
Z = 2	

Data collection

Bruker SMART APEX area-detector diffractometer	1335 independent reflections
φ and ω scans	1231 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.024
T_{min} = 0.954, T_{max} = 0.981	θ_{max} = 25.5°
3287 measured reflections	$h = -9 \rightarrow 10$
	$k = -6 \rightarrow 6$
	$l = -16 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1596P)^2 + 0.4615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.070	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.204	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
S = 0.86	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
1335 reflections	
181 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.320 (7)	O3—C16	1.177 (5)
O1—C8	1.346 (7)	O4—C15	1.388 (6)
O2—C16	1.297 (6)		
C7—O1—C8	121.4 (6)	C16—C15—C14	113.2 (4)
O1—C7—C1	113.1 (6)	O3—C16—O2	125.3 (4)
C11—C14—C15	114.5 (3)	O3—C16—C15	122.8 (4)
O4—C15—C16	110.5 (3)	O2—C16—C15	111.8 (3)
O4—C15—C14	109.6 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H17···O4^{i}	0.82	1.78	2.581 (5)	165
O4—H4A···O3^{ii}	0.82	2.09	2.769 (5)	141

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

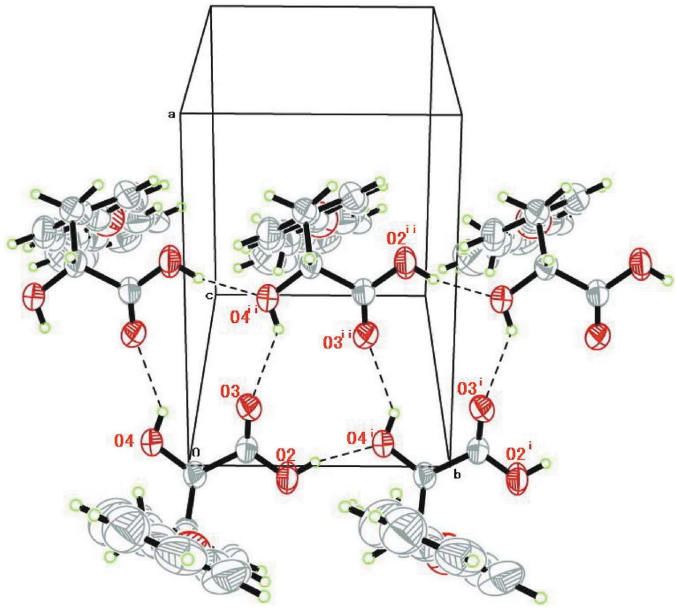


Figure 2
View of the intermolecular hydrogen bonds (dashed lines) in (I).

The H atoms were positioned geometrically (C—H = 0.93, 0.98 and 0.97 Å for phenyl, tertiary and methylene H atoms, respectively; O—H = 0.82 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(parent atom). Owing to the absence of any significant anomalous scatterers, Friedel pairs were merged before the final refinement. The absolute configuration has been determined from the chiral starting material.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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supporting information

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The two phenyl ring of (I) are effectively coplanar. The packing exhibits three kinds of hydrogen bond involving the hydroxy and carboxylic acid groups (Fig. 2), forming an infinite ladder parallel to the *b* axis.

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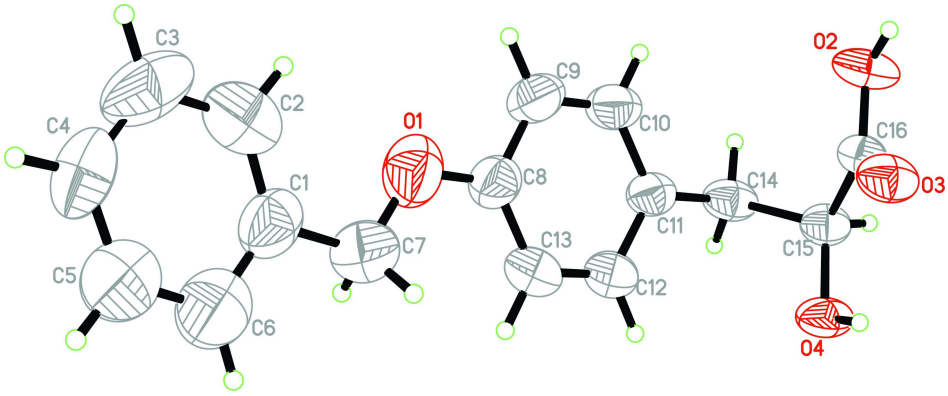
The H atoms were positioned geometrically (C—H = 0.93, 0.98 and 0.97 Å for phenyl, tertiary and methylene H atoms, respectively; O—H = 0.82 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(parent atom). Owing to the absence of any significant anomalous scatterers in the crystal, Friedel pairs were merged before the final refinement. The absolute structure has been determined from a reference chiral material.



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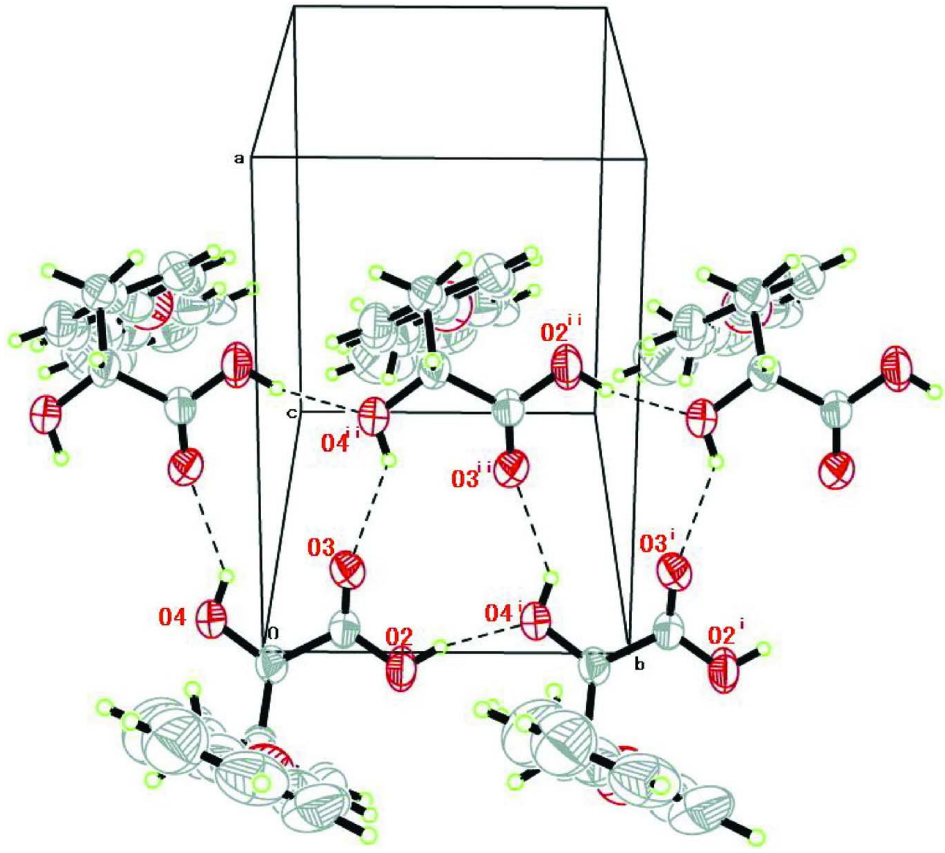

Figure 2

View of the intermolecular hydrogen bonds (dashed lines) in (I), viewed along the *c* axis [O3···O4^{ii} = 2.773 (7) Å and O2···O4^{i} = 2.582 (7) Å; symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, y+1/2, -z$].

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$c = 14.050$ (6) Å
$\beta = 102.784$ (7)°
$V = 676.0$ (5) Å^3
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Cell parameters from 2099 reflections
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$\mu = 0.10$ mm^{-1}
$T = 293$ K
Chunk, colorless
0.50 × 0.40 × 0.20 mm

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Radiation source: fine-focus sealed tube
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$T_{min} = 0.954$, $T_{max} = 0.981$
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1335 independent reflections
1231 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.5°$
$h = -9 \rightarrow 10$
$k = -6 \rightarrow 6$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.204$
$S = 0.86$
1335 reflections
181 parameters
20 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
$w = 1/[\sigma^2(F_o^2) + (0.1596P)^2 + 0.4615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.2228 (5)	1.0039 (10)	0.5075 (3)	0.0853 (14)
O2	1.1636 (4)	0.6401 (6)	0.9529 (3)	0.0593 (10)
H17	1.2155	0.5198	0.9562	0.089*
O3	1.3989 (3)	0.8040 (6)	0.9615 (3)	0.0543 (9)
O4	1.2780 (4)	1.2256 (6)	0.9607 (3)	0.0545 (9)

H4A	1.3701	1.1791	0.9810	0.082*
C1	1.3479 (7)	1.1014 (10)	0.3791 (4)	0.0776 (17)
C2	1.3012 (7)	0.8932 (11)	0.3355 (5)	0.0860 (19)
H2A	1.2429	0.7897	0.3647	0.103*
C3	1.3409 (7)	0.8360 (14)	0.2473 (5)	0.107 (3)
H3A	1.3074	0.6972	0.2159	0.128*
C4	1.4317 (7)	0.9922 (12)	0.2082 (4)	0.086 (2)
H4B	1.4636	0.9593	0.1505	0.104*
C5	1.4734 (9)	1.1960 (13)	0.2560 (5)	0.101 (2)
H5A	1.5338	1.2993	0.2282	0.122*
C6	1.4343 (10)	1.2614 (15)	0.3411 (5)	0.107 (3)
H6A	1.4641	1.4033	0.3708	0.129*
C7	1.3036 (9)	1.1671 (13)	0.4735 (5)	0.087 (2)
H7A	1.4011	1.2008	0.5218	0.104*
H7B	1.2393	1.3069	0.4633	0.104*
C8	1.1809 (6)	1.0272 (11)	0.5939 (4)	0.0582 (12)
C9	1.0973 (6)	0.8482 (11)	0.6234 (4)	0.0643 (13)
H9	1.0715	0.7178	0.5843	0.077*
C10	1.0524 (6)	0.8631 (9)	0.7108 (4)	0.0590 (13)
H10	0.9967	0.7410	0.7310	0.071*
C11	1.0881 (5)	1.0556 (9)	0.7695 (3)	0.0476 (10)
C12	1.1686 (6)	1.2319 (10)	0.7373 (4)	0.0597 (12)
H12	1.1922	1.3642	0.7754	0.072*
C13	1.2156 (7)	1.2199 (11)	0.6506 (4)	0.0658 (14)
H13	1.2709	1.3424	0.6304	0.079*
C14	1.0367 (5)	1.0708 (9)	0.8647 (3)	0.0504 (11)
H15	0.9563	0.9528	0.8657	0.060*
H14	0.9864	1.2200	0.8684	0.060*
C15	1.1717 (5)	1.0415 (8)	0.9542 (3)	0.0435 (10)
H16	1.1246	1.0462	1.0117	0.052*
C16	1.2593 (5)	0.8154 (8)	0.9557 (3)	0.0410 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.099 (3)	0.094 (4)	0.063 (2)	0.006 (3)	0.020 (2)	−0.004 (3)
O2	0.0546 (19)	0.0288 (16)	0.099 (3)	−0.0011 (14)	0.0264 (19)	0.0002 (17)
O3	0.0415 (15)	0.0387 (18)	0.082 (2)	0.0054 (14)	0.0110 (13)	0.0008 (16)
O4	0.0488 (17)	0.0288 (16)	0.080 (2)	0.0020 (13)	0.0012 (15)	−0.0013 (15)
C1	0.079 (4)	0.077 (4)	0.066 (3)	0.010 (3)	−0.007 (3)	−0.005 (3)
C2	0.058 (3)	0.084 (4)	0.112 (5)	0.001 (3)	0.012 (3)	0.020 (4)
C3	0.076 (4)	0.105 (6)	0.117 (5)	−0.007 (4)	−0.026 (3)	−0.037 (5)
C4	0.076 (3)	0.120 (6)	0.058 (3)	0.022 (4)	0.004 (2)	−0.013 (4)
C5	0.102 (5)	0.102 (6)	0.098 (5)	0.000 (5)	0.018 (4)	−0.005 (5)
C6	0.115 (6)	0.116 (7)	0.091 (4)	−0.016 (6)	0.022 (4)	−0.009 (5)
C7	0.105 (5)	0.069 (5)	0.083 (4)	−0.005 (4)	0.015 (3)	−0.009 (4)
C8	0.062 (3)	0.053 (3)	0.055 (2)	0.003 (3)	0.0012 (19)	−0.002 (2)
C9	0.071 (3)	0.054 (3)	0.063 (3)	−0.007 (3)	0.004 (2)	−0.013 (3)

C10	0.056 (2)	0.045 (3)	0.071 (3)	−0.007 (2)	0.002 (2)	0.000 (2)
C11	0.0414 (19)	0.037 (2)	0.059 (2)	0.0041 (19)	−0.0010 (17)	0.000 (2)
C12	0.076 (3)	0.039 (3)	0.062 (3)	0.001 (3)	0.011 (2)	0.000 (2)
C13	0.082 (3)	0.044 (3)	0.070 (3)	−0.004 (3)	0.015 (3)	0.007 (3)
C14	0.0383 (19)	0.037 (2)	0.075 (3)	0.0011 (18)	0.0103 (18)	0.001 (2)
C15	0.044 (2)	0.033 (2)	0.055 (2)	0.0042 (19)	0.0145 (18)	−0.005 (2)
C16	0.046 (2)	0.031 (2)	0.048 (2)	−0.0011 (18)	0.0141 (16)	−0.0011 (18)

Geometric parameters (Å, º)

O1—C7	1.320 (7)	C7—H7A	0.9700
O1—C8	1.346 (7)	C7—H7B	0.9700
O2—C16	1.297 (6)	C8—C13	1.363 (8)
O2—H17	0.8200	C8—C9	1.372 (8)
O3—C16	1.177 (5)	C9—C10	1.367 (8)
O4—C15	1.388 (6)	C9—H9	0.9300
O4—H4A	0.8200	C10—C11	1.379 (7)
C1—C6	1.363 (5)	C10—H10	0.9300
C1—C2	1.370 (5)	C11—C12	1.361 (7)
C1—C7	1.506 (9)	C11—C14	1.500 (6)
C2—C3	1.394 (5)	C12—C13	1.366 (8)
C2—H2A	0.9300	C12—H12	0.9300
C3—C4	1.381 (5)	C13—H13	0.9300
C3—H3A	0.9300	C14—C15	1.515 (6)
C4—C5	1.364 (5)	C14—H15	0.9700
C4—H4B	0.9300	C14—H14	0.9700
C5—C6	1.364 (5)	C15—C16	1.504 (6)
C5—H5A	0.9300	C15—H16	0.9800
C6—H6A	0.9300		
C7—O1—C8	121.4 (6)	C10—C9—C8	119.4 (5)
C16—O2—H17	109.5	C10—C9—H9	120.3
C15—O4—H4A	109.5	C8—C9—H9	120.3
C6—C1—C2	123.4 (7)	C9—C10—C11	121.3 (5)
C6—C1—C7	115.9 (6)	C9—C10—H10	119.4
C2—C1—C7	120.7 (6)	C11—C10—H10	119.4
C1—C2—C3	120.1 (7)	C12—C11—C10	117.8 (5)
C1—C2—H2A	119.9	C12—C11—C14	121.4 (4)
C3—C2—H2A	119.9	C10—C11—C14	120.7 (5)
C4—C3—C2	117.8 (6)	C11—C12—C13	121.9 (5)
C4—C3—H3A	121.1	C11—C12—H12	119.1
C2—C3—H3A	121.1	C13—C12—H12	119.1
C5—C4—C3	118.5 (6)	C8—C13—C12	119.6 (6)
C5—C4—H4B	120.8	C8—C13—H13	120.2
C3—C4—H4B	120.8	C12—C13—H13	120.2
C4—C5—C6	125.7 (8)	C11—C14—C15	114.5 (3)
C4—C5—H5A	117.2	C11—C14—H15	108.6
C6—C5—H5A	117.2	C15—C14—H15	108.6

C1—C6—C5	114.4 (8)	C11—C14—H14	108.6
C1—C6—H6A	122.8	C15—C14—H14	108.6
C5—C6—H6A	122.8	H15—C14—H14	107.6
O1—C7—C1	113.1 (6)	O4—C15—C16	110.5 (3)
O1—C7—H7A	109.0	O4—C15—C14	109.6 (4)
C1—C7—H7A	109.0	C16—C15—C14	113.2 (4)
O1—C7—H7B	109.0	O4—C15—H16	107.8
C1—C7—H7B	109.0	C16—C15—H16	107.8
H7A—C7—H7B	107.8	C14—C15—H16	107.8
O1—C8—C13	122.8 (6)	O3—C16—O2	125.3 (4)
O1—C8—C9	117.1 (5)	O3—C16—C15	122.8 (4)
C13—C8—C9	120.0 (5)	O2—C16—C15	111.8 (3)
C6—C1—C2—C3	−0.4 (11)	C9—C10—C11—C12	0.6 (7)
C7—C1—C2—C3	179.0 (6)	C9—C10—C11—C14	179.4 (4)
C1—C2—C3—C4	1.9 (10)	C10—C11—C12—C13	−1.1 (8)
C2—C3—C4—C5	−1.9 (10)	C14—C11—C12—C13	−179.8 (5)
C3—C4—C5—C6	0.5 (12)	O1—C8—C13—C12	179.7 (5)
C2—C1—C6—C5	−1.0 (12)	C9—C8—C13—C12	0.9 (8)
C7—C1—C6—C5	179.6 (6)	C11—C12—C13—C8	0.3 (8)
C4—C5—C6—C1	0.9 (13)	C12—C11—C14—C15	−74.8 (6)
C8—O1—C7—C1	176.3 (5)	C10—C11—C14—C15	106.4 (5)
C6—C1—C7—O1	−178.8 (6)	C11—C14—C15—O4	65.9 (5)
C2—C1—C7—O1	1.7 (9)	C11—C14—C15—C16	−58.0 (5)
C7—O1—C8—C13	1.3 (9)	O4—C15—C16—O3	−0.2 (5)
C7—O1—C8—C9	−179.9 (6)	C14—C15—C16—O3	123.1 (5)
O1—C8—C9—C10	179.8 (5)	O4—C15—C16—O2	177.9 (4)
C13—C8—C9—C10	−1.3 (8)	C14—C15—C16—O2	−58.8 (5)
C8—C9—C10—C11	0.6 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H17···O4^{i}	0.82	1.78	2.581 (5)	165
O4—H4*A*···O3^{ii}	0.82	2.09	2.769 (5)	141

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