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(*RS*)-Benzyl mandelate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.089
Data-to-parameter ratio = 11.0

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

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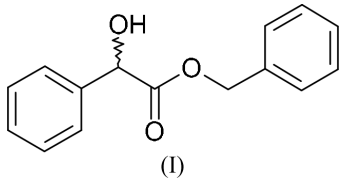
A benzyl ester of mandelic acid, $C_{15}H_{14}O_3$, was obtained by the crystallization of racemic mandelic acid from benzyl alcohol followed by vacuum drying at 363 K. The structure is composed of two hydrogen-bonded chains of *S* or *R* configuration, running along the shortest crystallographic *b* axis. There is one molecule in the asymmetric unit and each molecule forms four intermolecular hydrogen bonds with two other molecules of the same chirality.

Comment

During the crystallization of racemic mandelic acid from benzyl alcohol and drying off the solvent in a vacuum oven, colourless needle-shaped crystals of (*RS*)-benzyl mandelate (BM), (I), were obtained. The crystal structure of this compound was not found in the Cambridge Structural Database (CSD, Version 1.6; Allen, 2002) and hence its structure was determined by single-crystal X-ray diffraction at 150 K.



(I)

The compound BM has one molecule in the asymmetric unit. Fig. 1 shows the structure and the atom labelling. The bond lengths and angles are unexceptional. Each molecule forms four intermolecular hydrogen bonds to two neighbouring molecules, as shown in Fig. 2. The unit-cell contents of BM are shown in Fig. 3.

The crystal structure is composed of two types of chains that run along the shortest crystallographic axis, *b*, which is the needle axis. The $C_1^1(5)$ chain runs through the hydroxyl and carbonyl groups *via* —C═O···H—O— hydrogen bonding.

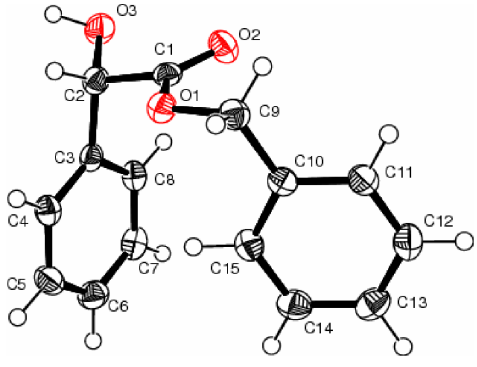


Figure 1
The crystallographically independent molecule in the asymmetric unit of BM; displacement ellipsoids are drawn at the 50% probability level.

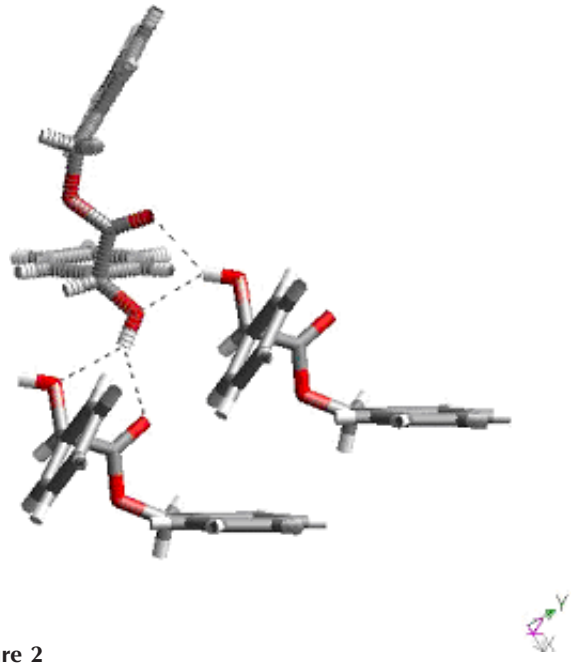


Figure 2
Hydrogen bonds (dashed lines) formed by each independent molecule with the neighbouring two molecules.

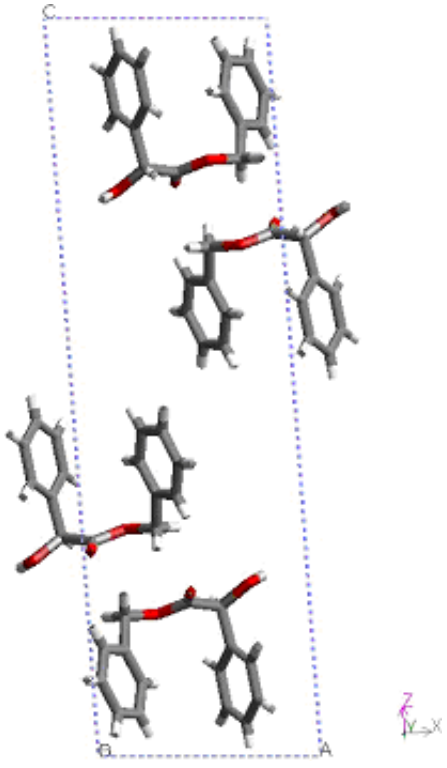


Figure 3
The unit-cell contents of BM, viewed along *b*.

The $C_1^1(2)$ chain arises from the linking of OH in one molecule to OH of another molecule. Fig. 4 shows the packing of the two chains and the resulting bilayer sandwich. Layers of hydrogen-bonded chains are sandwiched between bilayers of phenyl rings. There is face–edge interaction between the phenyl rings of each molecule, and also between the phenyl rings of adjacent molecules in the same chain. Each $C_1^1(5)$ and

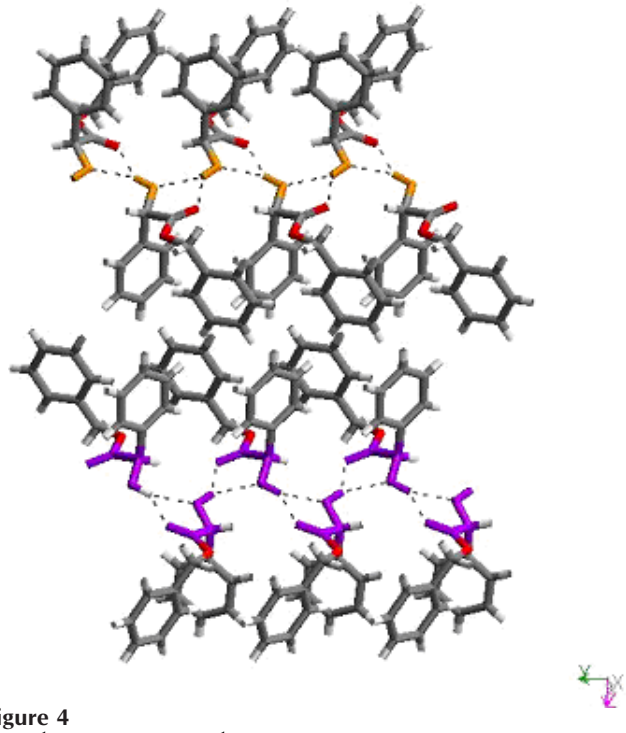


Figure 4
The $C_1^1(5)$ (purple) and $C_1^1(2)$ (orange) hydrogen-bonded chains (dashed lines) of BM, viewed along *c*.

$C_1^1(2)$ chain is composed of either all-*S* configuration molecules or all-*R* molecules, and the chains pack such that there are alternating *R* and *S* chains, as shown in Fig. 5. There are no hydrogen-bonding interactions between *R* and *S* molecules. The hydrogen bonds are listed in Table 1.

Experimental

A saturated solution of racemic mandelic acid (supplied by Sigma–Aldrich, 99%) in benzyl alcohol was prepared at 323 K and stirred at 343 K for 2 h. On cooling to 298–303 K, needle-shaped crystals of racemic mandelic acid formed; these were vacuum-filtered and then dried in a vacuum oven at 363 K to remove benzyl alcohol mother liquor. After a few weeks in the vacuum oven, crystals of (*RS*)-benzyl mandelate were found alongside an orange–yellow amorphous glass-like residue.

Crystal data

$C_{15}H_{14}O_3$
$M_r = 242.15$
Monoclinic, $P2_1/n$
$a = 8.0627\ (3)$ Å
$b = 5.6494\ (2)$ Å
$c = 26.7944\ (11)$ Å
$\beta = 94.130\ (1)^\circ$
$V = 1217.30\ (8)$ Å^3
$Z = 4$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7192 reflections
$\theta = 1$–26°
$\mu = 0.09$ mm^{-1}
$T = 150\ (2)$ K
Needle, colourless
0.25 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.978$, $T_{max} = 0.994$
7192 measured reflections
2425 independent reflections
1732 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 26.5^\circ$
$h = -7 \rightarrow 10$
$k = -5 \rightarrow 7$
$l = -32 \rightarrow 33$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.1653P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.089$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.18$ e Å^{-3}
2425 reflections	Extinction correction: *SHELXL*97
220 parameters	Extinction coefficient: 0.026 (4)
All H-atom parameters refined	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.94 (2)	2.08 (2)	2.8711 (15)	141.2 (18)
O3—H3···O3^{i}	0.94 (2)	2.17 (2)	2.9522 (6)	140.1 (19)

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

All H atoms were located in a difference Fourier map and refined isotropically.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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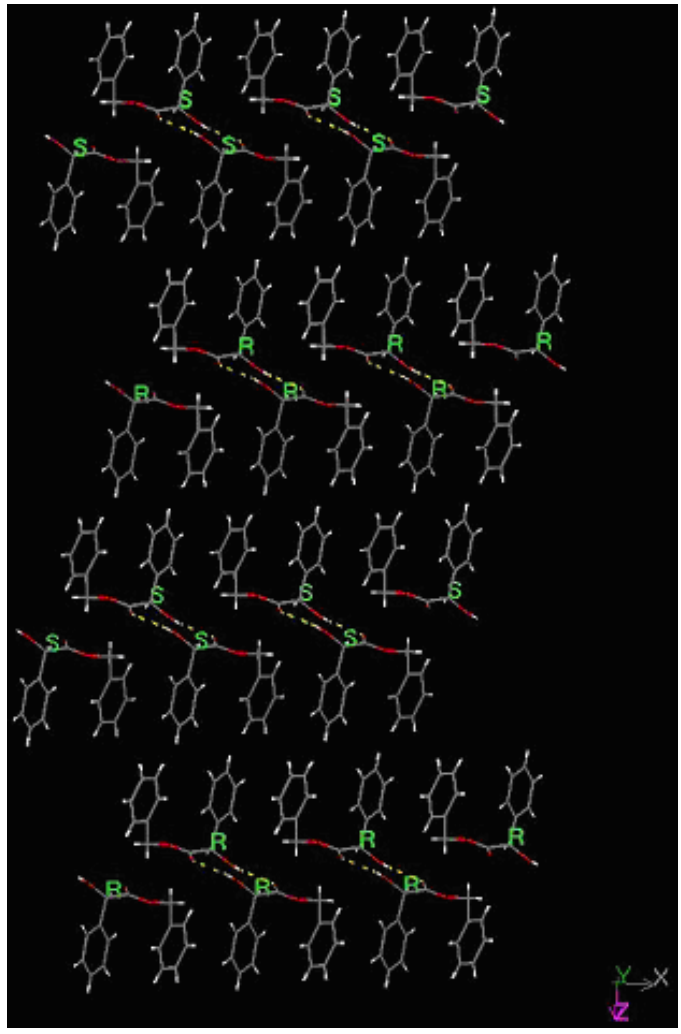

Figure 5
Packing of alternating *R* and *S* chains, together with the phenyl bilayer and the sandwiched hydrogen-bonded chains.

supporting information

Acta Cryst. (2004). E**60**, o1984–o1986 [https://doi.org/10.1107/S1600536804025024]

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$D_x = 1.321$ Mg m^{-3}
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Cell parameters from 19781 reflections
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Needle, colourless
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Radiation source: Enraf–Nonius FR590
Graphite monochromator
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$\theta_{max} = 26.5°$, $\theta_{min} = 2.6°$
$h = -7 \rightarrow 10$
$k = -5 \rightarrow 7$
$l = -32 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.089$
$S = 1.02$
2425 reflections
220 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.1653P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.026 (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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.45128 (18)	0.6686 (2)	0.20888 (5)	0.0254 (3)
C2	0.59343 (18)	0.4981 (3)	0.20329 (5)	0.0266 (4)
C3	0.62714 (17)	0.4970 (2)	0.14824 (5)	0.0245 (3)
C4	0.70938 (18)	0.6880 (3)	0.12828 (6)	0.0283 (4)
C5	0.7309 (2)	0.6970 (3)	0.07748 (6)	0.0332 (4)
C6	0.6696 (2)	0.5168 (3)	0.04634 (6)	0.0349 (4)
C7	0.5876 (2)	0.3270 (3)	0.06609 (6)	0.0336 (4)
C8	0.56580 (19)	0.3168 (3)	0.11684 (6)	0.0283 (4)
C9	0.16228 (19)	0.7244 (3)	0.19014 (6)	0.0287 (4)
C10	0.15042 (17)	0.8770 (2)	0.14424 (5)	0.0243 (3)
C11	0.06166 (19)	1.0879 (2)	0.14517 (6)	0.0293 (4)
C12	0.0375 (2)	1.2255 (3)	0.10280 (6)	0.0345 (4)
C13	0.1025 (2)	1.1563 (3)	0.05884 (7)	0.0352 (4)
C14	0.1933 (2)	0.9489 (3)	0.05773 (6)	0.0350 (4)
C15	0.21728 (19)	0.8091 (3)	0.10006 (6)	0.0294 (4)
O1	0.30683 (12)	0.57077 (16)	0.19239 (4)	0.0278 (3)
O2	0.46677 (12)	0.86900 (17)	0.22456 (4)	0.0339 (3)
O3	0.72933 (13)	0.58119 (18)	0.23477 (4)	0.0356 (3)
H2	0.5569 (16)	0.344 (2)	0.2129 (5)	0.020 (3)*
H3	0.798 (3)	0.452 (4)	0.2455 (8)	0.090 (7)*
H4	0.7517 (18)	0.817 (3)	0.1497 (5)	0.032 (4)*
H5	0.789 (2)	0.832 (3)	0.0638 (6)	0.041 (4)*
H6	0.683 (2)	0.524 (3)	0.0117 (6)	0.042 (5)*
H7	0.5475 (19)	0.197 (3)	0.0447 (6)	0.034 (4)*
H8	0.5101 (19)	0.187 (3)	0.1302 (5)	0.034 (4)*
H9A	0.0632 (19)	0.610 (2)	0.1888 (5)	0.030 (4)*
H9B	0.1662 (18)	0.818 (2)	0.2197 (5)	0.026 (4)*
H11	0.0162 (19)	1.137 (3)	0.1761 (6)	0.036 (4)*
H12	−0.025 (2)	1.368 (3)	0.1032 (6)	0.040 (4)*
H13	0.083 (2)	1.247 (3)	0.0281 (6)	0.042 (4)*
H14	0.241 (2)	0.899 (3)	0.0274 (6)	0.040 (5)*
H15	0.2794 (19)	0.659 (3)	0.0991 (5)	0.033 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0300 (9)	0.0265 (8)	0.0198 (8)	−0.0025 (6)	0.0028 (6)	0.0021 (6)
C2	0.0261 (8)	0.0223 (7)	0.0308 (8)	−0.0013 (7)	−0.0025 (6)	0.0013 (6)
C3	0.0202 (8)	0.0220 (7)	0.0310 (8)	0.0043 (6)	0.0002 (6)	0.0015 (6)
C4	0.0249 (8)	0.0239 (8)	0.0363 (9)	0.0024 (6)	0.0032 (7)	−0.0003 (7)
C5	0.0282 (9)	0.0285 (8)	0.0434 (10)	0.0029 (7)	0.0060 (7)	0.0066 (8)
C6	0.0335 (10)	0.0417 (9)	0.0299 (9)	0.0077 (7)	0.0038 (7)	0.0024 (8)
C7	0.0338 (9)	0.0327 (9)	0.0340 (9)	0.0009 (7)	0.0003 (7)	−0.0078 (7)
C8	0.0259 (8)	0.0241 (8)	0.0350 (9)	−0.0009 (6)	0.0025 (7)	−0.0009 (7)
C9	0.0252 (9)	0.0321 (8)	0.0291 (9)	0.0021 (7)	0.0048 (7)	−0.0011 (7)
C10	0.0196 (8)	0.0251 (7)	0.0282 (8)	−0.0041 (6)	0.0008 (6)	−0.0024 (6)
C11	0.0282 (9)	0.0297 (8)	0.0302 (9)	0.0004 (6)	0.0029 (7)	−0.0031 (7)
C12	0.0309 (9)	0.0260 (8)	0.0462 (11)	0.0027 (7)	−0.0002 (8)	−0.0006 (7)
C13	0.0343 (10)	0.0352 (9)	0.0355 (10)	−0.0040 (7)	−0.0015 (7)	0.0087 (8)
C14	0.0373 (10)	0.0392 (9)	0.0293 (9)	−0.0021 (7)	0.0076 (7)	−0.0001 (7)
C15	0.0281 (9)	0.0270 (8)	0.0336 (9)	0.0007 (7)	0.0061 (7)	−0.0014 (7)
O1	0.0249 (6)	0.0264 (5)	0.0321 (6)	0.0004 (4)	0.0014 (4)	0.0010 (4)
O2	0.0322 (6)	0.0297 (6)	0.0397 (7)	0.0005 (5)	0.0017 (5)	−0.0096 (5)
O3	0.0328 (7)	0.0329 (6)	0.0388 (7)	0.0021 (5)	−0.0120 (5)	−0.0024 (5)

Geometric parameters (Å, º)

C1—O2	1.2110 (16)	C9—O1	1.4508 (17)
C1—O1	1.3352 (17)	C9—C10	1.499 (2)
C1—C2	1.513 (2)	C9—H9A	1.026 (15)
C2—O3	1.4139 (17)	C9—H9B	0.950 (14)
C2—C3	1.519 (2)	C10—C15	1.390 (2)
C2—H2	0.959 (13)	C10—C11	1.391 (2)
C3—C8	1.389 (2)	C11—C12	1.378 (2)
C3—C4	1.394 (2)	C11—H11	0.971 (15)
C4—C5	1.385 (2)	C12—C13	1.379 (2)
C4—H4	0.974 (15)	C12—H12	0.953 (16)
C5—C6	1.385 (2)	C13—C14	1.383 (2)
C5—H5	0.980 (17)	C13—H13	0.974 (16)
C6—C7	1.385 (2)	C14—C15	1.384 (2)
C6—H6	0.944 (16)	C14—H14	0.964 (17)
C7—C8	1.385 (2)	C15—H15	0.984 (15)
C7—H7	0.970 (15)	O3—H3	0.95 (2)
C8—H8	0.945 (16)		
O2—C1—O1	124.58 (13)	O1—C9—C10	112.44 (12)
O2—C1—C2	124.72 (13)	O1—C9—H9A	104.2 (8)
O1—C1—C2	110.66 (11)	C10—C9—H9A	109.2 (8)
O3—C2—C1	106.85 (11)	O1—C9—H9B	108.6 (9)
O3—C2—C3	113.31 (12)	C10—C9—H9B	111.1 (8)
C1—C2—C3	106.78 (11)	H9A—C9—H9B	111.1 (12)

O3—C2—H2	112.4 (8)	C15—C10—C11	118.90 (14)
C1—C2—H2	107.5 (8)	C15—C10—C9	122.29 (13)
C3—C2—H2	109.6 (8)	C11—C10—C9	118.72 (13)
C8—C3—C4	119.58 (14)	C12—C11—C10	120.71 (15)
C8—C3—C2	120.71 (13)	C12—C11—H11	120.3 (9)
C4—C3—C2	119.52 (12)	C10—C11—H11	119.0 (9)
C5—C4—C3	120.14 (14)	C11—C12—C13	120.25 (15)
C5—C4—H4	119.3 (9)	C11—C12—H12	120.8 (9)
C3—C4—H4	120.6 (9)	C13—C12—H12	119.0 (9)
C6—C5—C4	120.04 (15)	C12—C13—C14	119.52 (15)
C6—C5—H5	120.5 (9)	C12—C13—H13	121.6 (10)
C4—C5—H5	119.5 (9)	C14—C13—H13	118.8 (10)
C7—C6—C5	119.91 (15)	C13—C14—C15	120.56 (16)
C7—C6—H6	120.3 (10)	C13—C14—H14	120.5 (9)
C5—C6—H6	119.8 (10)	C15—C14—H14	118.9 (9)
C6—C7—C8	120.36 (15)	C14—C15—C10	120.05 (14)
C6—C7—H7	120.5 (9)	C14—C15—H15	120.6 (9)
C8—C7—H7	119.2 (9)	C10—C15—H15	119.3 (8)
C7—C8—C3	119.97 (14)	C1—O1—C9	116.43 (11)
C7—C8—H8	120.1 (9)	C2—O3—H3	109.7 (14)
C3—C8—H8	119.9 (9)		

O2—C1—C2—O3	20.15 (19)	C2—C3—C8—C7	175.46 (13)
O1—C1—C2—O3	−162.16 (11)	O1—C9—C10—C15	27.5 (2)
O2—C1—C2—C3	−101.39 (15)	O1—C9—C10—C11	−156.07 (12)
O1—C1—C2—C3	76.30 (14)	C15—C10—C11—C12	1.2 (2)
O3—C2—C3—C8	142.87 (13)	C9—C10—C11—C12	−175.39 (14)
C1—C2—C3—C8	−99.78 (15)	C10—C11—C12—C13	−0.5 (2)
O3—C2—C3—C4	−42.13 (17)	C11—C12—C13—C14	−0.6 (2)
C1—C2—C3—C4	75.22 (16)	C12—C13—C14—C15	1.0 (2)
C8—C3—C4—C5	−0.5 (2)	C13—C14—C15—C10	−0.3 (2)
C2—C3—C4—C5	−175.59 (13)	C11—C10—C15—C14	−0.8 (2)
C3—C4—C5—C6	0.5 (2)	C9—C10—C15—C14	175.64 (14)
C4—C5—C6—C7	−0.4 (2)	O2—C1—O1—C9	4.6 (2)
C5—C6—C7—C8	0.4 (2)	C2—C1—O1—C9	−173.07 (11)
C6—C7—C8—C3	−0.4 (2)	C10—C9—O1—C1	80.69 (16)
C4—C3—C8—C7	0.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.94 (2)	2.08 (2)	2.8711 (15)	141.2 (18)
O3—H3···O3^{i}	0.94 (2)	2.17 (2)	2.9522 (6)	140.1 (19)

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