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(2*R*,4*S*,5*S*)-5-Hydroxy-4-methyl-3-oxohept-6-en-2-yl benzoate

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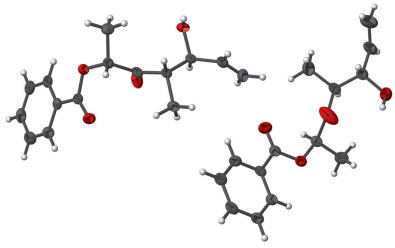
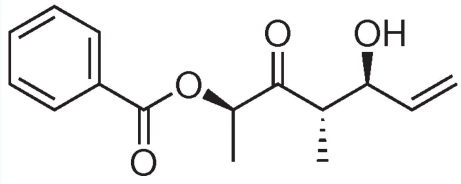
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{18}O_4$, which crystallizes with two molecules in the asymmetric unit, was obtained in the course of the total synthesis of curvicollides A–C and fusaequisin A. It features the relative configuration of the Western aldol part of the natural products. In the crystal, molecules are linked by C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

The asymmetric synthesis of the title compound (I) (Fig. 1) is based on Paterson's *anti*-aldol chemistry utilizing enantiomerically pure (*R*)-3-oxopentan-2-yl benzoate (II) synthesized in accordance to published procedures from commercially available (*R*)-ethyl lactate (Paterson *et al.*, 1994, Paterson, 1998). In the following hitherto unpublished example of a Paterson aldol reaction, chlorodicyclohexylborane in the presence of triethylamine was employed to generate the (*E*)-configured boron enolate of (*R*)-ethyl ketone (II), which was then treated with an excess of acroleine as the electrophile. The title compound (I) was obtained in good yields (80%) and excellent diastereoselectivities (dr > 95:5). From a synthetic perspective, compound (I) can be viewed as a versatile building block as it ensures high *enantio*- and diastereoselectivities as well as facile expandability in several directions. The latter is due to the fact that the benzoylated α-hydroxy ketone on one hand, and the vinyl group on the other can be orthogonally transformed into a whole variety of synthetic products. In particular, the title compound represents a synthetic precursor for the Western side chains of Curvicollides A–C (Che *et al.*, 2004) and Fusaequisin A (Shiono *et al.*, 2013).

Compound (I) crystallizes with two molecules in the asymmetric unit (Fig. 1) with similar conformations (r.m.s. overlay fit = 0.329 Å). The absolute structure (C8 *R*, C11 *S*, C13 *S*; C23 *R*, S26 *S*, C28 *S*) is well established based on refinement of the Flack

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O7	0.84	2.63	3.140 (3)	120
C8—H8A···O3^{i}	1.00	2.41	3.235 (3)	140
C23—H23···O7^{ii}	1.00	2.54	3.189 (3)	122
C26—H26···O7^{ii}	1.00	2.29	3.205 (3)	151

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

parameter (Table 2). In the crystal, weak C—H···O hydrogen bonds (Table 1) link the molecules.

Synthesis and crystallization

The reaction was carried out in two parallel batches under an argon atmosphere. To a solution of the ketone (II) ($C_{12}H_{14}O_3$, 206.24 g mol^{-1}, 778 mg, 3.772 mmol, 1 equiv.) in Et_2O (32 ml were successively added dried 3 Å molecular sieves (200 mg, 0.1 mbar, 473 K, 1 h), chlorodicyclohexylborane (*c*-Hex_2BCl, 1 *M* in hexane, 5.79 ml, 5.79 mmol,1.53 equiv.) and triethylamine ($C_6H_{15}N$, 101.19 g mol^{-1}, 0.726 g mol^{-1}, 0.97 ml, 704.2 mg, 6.959 mmol, 1.84 equiv.) at 223 K. The clear, colorless suspension was stirred for 20 min at 232 K and the color of the suspension turned to white. The white, turbid suspension was cooled to 193 K and to the solution freshly distilled acrolein (C_3H_4O, 56.06 g mol^{-1}, 0.839 g ml^{-1}, 1.05 ml, 881 mg, 15.715 mmol, 4.17 equiv.) was added dropwise over a period of 10 min at 193 K. The white suspension was stirred at 193 K for 1 h and was then diluted by the addition of aqueous phosphate pH 7 buffer (20 ml and CH_2Cl_2 (20 ml). The decolorized mixture was then warmed to room temperature and transferred into a separating funnel using CH_2Cl_2 (10 ml) for rinsing. The phase were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 30 ml). The combined organic phases were dried ($MgSO_4$) and concentrated under reduced pressure. The oily yellowish residue was purified by flash chromatography (cyclohexane–ethyl acetate, 50:1 to 20:1 to 10:1 to 5:1) to deliver the aldol (I) ($C_{15}H_{18}O_4$, 262.30 g mol^{-1}, 787 mg, 3.000 mmol, 80%) as a white solid. Crystallization of

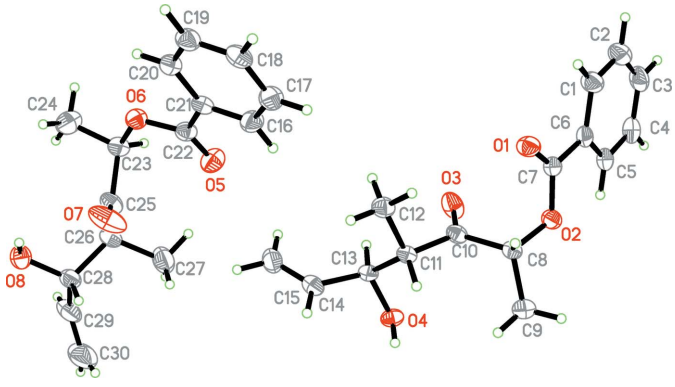


Figure 1
The molecular structure of the title compound, showing the labeling of all non-H atoms. Displacement ellipsoids are shown at the 50% probability level. The asymmetric unit contains two molecules.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{18}O_4$
M_r	262.29
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	100
a, b, c (Å)	15.6945 (8), 4.9102 (2), 20.1737 (10)
β (°)	112.712 (2)
V (Å^3)	1434.10 (12)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.72
Crystal size (mm)	0.98 × 0.10 × 0.08
Data collection	
Diffractometer	Bruker D8 VENTURE area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.350, 0.470
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22281, 5396, 5143
R_{int}	0.051
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.609
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.099, 1.04
No. of reflections	5396
No. of parameters	349
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.28
Absolute structure	Flack x determined using 2185 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.02 (6)

Computer programs: *SMART* and *SAINT* (Bruker, 2012), *SHELXD*, *SHELXL97* and *SHELXTL-Plus* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) was accomplished from a solution in hot cyclohexane (50 ml, 333 K) by slow cooling to room temperature. The product crystallized in colorless needles: m.p. 365–369 K; R_f 0.45 (cyclohexane–ethyl acetate, 2:1); $[\alpha]_D^{20} = -39.7$ ($c = 0.6$ in $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.23 (*d*, ^3J = 7.3 Hz, 3H), 1.57 (*d*, ^3J = 7.0 Hz, 3H), 2.36 (*d*, ^3J = 5.4 Hz, 1H), 2.93 (*quin*, ^{3}J = 7.3 Hz, 1H), 4.24–4.30 (*m*, 1H), 5.21 (*app d*, ^3J = 10.3 Hz, 1H), 5.31 (*app d*, ^3J = 17.1 Hz, 1H), 5.44 (*q*, ^3J = 7.0 Hz, 1H), 5.84 (*ddd*, ^3J = 17.1, 10.3, 6.8 Hz, 1H), 7.43–7.49 (*m*, 2H), 7.56–7.62 (*m*, 1H), 8.06–8.11 (*m*, 2H); ^{13}C NMR (151 MHz $CDCl_3$) δ 14.55 (CH_3), 15.84 (CH_3), 47.93 (CH), 74.91 (CH), 75.29 (CH), 117.32 (CH_2), 128.61 (CH), 129.61 (C), 129.96 (CH), 133.51 (CH), 138.40 (CH), 165.99 (C), 211.25 (C); IR ν 3330 (*w*), 2980 (*w*), 2935 (*w*), 1720 (*s*), 1605 (*w*), 1450 (*m*), 1380 (*m*), 1350 (*m*), 1315 (*m*), 1300 (*m*), 1265 (*s*), 1175 (*w*), 1115 (*s*), 1065 (*m*), 1015 (*m*), 1000 (*s*), 950 (*m*), 920 (*m*), 745 (*w*), 710 (*s*), 685 (*w*) cm^{-1}. Analysis calculated for $C_{15}H_{18}O_4$: C, 68.68; H, 6.92; found: C, 68.7; H, 7.0.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). **2**, x171539 [https://doi.org/10.1107/S2414314617015395]

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Crystal data

$C_{15}H_{18}O_4$
M_r = 262.29
Monoclinic, $P2_1$
a = 15.6945 (8) Å
b = 4.9102 (2) Å
c = 20.1737 (10) Å
β = 112.712 (2)°
V = 1434.10 (12) Å^3
Z = 4
$F(000)$ = 560
D_x = 1.215 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9866 reflections
θ = 3.1–72.5°
μ = 0.72 mm^{-1}
T = 100 K
Needle, colourless
0.98 × 0.10 × 0.08 mm

Data collection

Bruker D8 VENTURE area detector diffractometer
Radiation source: microfocus sealed X-ray tube
Detector resolution: 7.9 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.350, T_{max} = 0.470
22281 measured reflections
5396 independent reflections
5143 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 70.0°, θ_{min} = 3.1°
h = −19→19
k = −5→5
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.04
5396 reflections
349 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack x determined using 2185 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.02 (6)

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.

Refinement. H-atoms attached to C, except those in CH_3, were placed in calculated positions (C-H = 0.95-1.00Å and $U_{iso}(H) = 1.2\ U_{eq}(C)$). CH_3 hydrogen atoms, which were taken from a Fourier map (AFIX 137), were allowed to rotate but not to tip (C-H = 0.98 Å and $U_{iso}(H) = 1.5\ U_{eq}(C)$). H-atoms attached to O were placed in calculated positions (O-H = 0.84Å and $U_{iso}(H) = 1.5\ U_{eq}(C)$).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O6	0.44530 (10)	0.7087 (3)	0.23069 (8)	0.0297 (3)
O2	0.97206 (10)	0.2429 (3)	0.75670 (8)	0.0267 (3)
O4	0.97357 (9)	0.3000 (3)	0.51804 (8)	0.0270 (3)
H4	0.9934	0.1438	0.5149	0.040*
O1	0.82462 (10)	0.1587 (3)	0.74079 (9)	0.0313 (4)
O8	0.53681 (11)	0.7325 (4)	0.02037 (9)	0.0355 (4)
H8	0.5035	0.6201	0.0307	0.053*
O5	0.58656 (11)	0.7084 (4)	0.31915 (9)	0.0384 (4)
O3	0.88168 (14)	0.4843 (3)	0.63246 (9)	0.0388 (4)
O7	0.54639 (18)	0.4739 (4)	0.16545 (13)	0.0567 (6)
C10	0.89931 (14)	0.2484 (4)	0.62747 (11)	0.0247 (4)
C6	0.90938 (15)	0.4881 (4)	0.82787 (11)	0.0252 (4)
C11	0.86074 (14)	0.1032 (4)	0.55512 (11)	0.0223 (4)
H11	0.8946	−0.0721	0.5585	0.027*
C21	0.46950 (15)	0.4252 (4)	0.33115 (12)	0.0256 (4)
C13	0.87508 (13)	0.2880 (4)	0.49904 (11)	0.0226 (4)
H13	0.8529	0.4749	0.5040	0.027*
C5	0.98921 (15)	0.6447 (5)	0.85657 (11)	0.0282 (4)
H5	1.0384	0.6176	0.8409	0.034*
C22	0.50808 (15)	0.6266 (5)	0.29529 (12)	0.0278 (4)
C8	0.95719 (14)	0.0841 (4)	0.69332 (11)	0.0250 (4)
H8A	0.9242	−0.0884	0.6950	0.030*
C23	0.48191 (16)	0.8800 (5)	0.19033 (12)	0.0291 (5)
H23	0.5154	1.0378	0.2205	0.035*
C7	0.89546 (14)	0.2800 (4)	0.77118 (11)	0.0243 (4)
C14	0.82627 (15)	0.1980 (5)	0.42279 (11)	0.0287 (5)
H14	0.8401	0.0236	0.4091	0.034*
C25	0.54742 (16)	0.7171 (5)	0.16606 (12)	0.0314 (5)
C20	0.37594 (16)	0.3604 (5)	0.30452 (12)	0.0308 (5)
H20	0.3340	0.4491	0.2628	0.037*
C16	0.53049 (16)	0.2961 (5)	0.39209 (13)	0.0336 (5)
H16	0.5942	0.3418	0.4104	0.040*
C15	0.76517 (17)	0.3506 (6)	0.37411 (12)	0.0371 (5)
H15A	0.7504	0.5257	0.3867	0.044*
H15B	0.7356	0.2862	0.3262	0.044*

C28	0.62499 (15)	0.7506 (5)	0.07868 (12)	0.0322 (5)
H28	0.6496	0.5616	0.0915	0.039*
C18	0.40574 (17)	0.0346 (5)	0.39970 (13)	0.0338 (5)
H18	0.3840	−0.1010	0.4229	0.041*
C12	0.75833 (15)	0.0434 (5)	0.53746 (12)	0.0304 (5)
H12A	0.7522	−0.0610	0.5768	0.046*
H12B	0.7329	−0.0624	0.4929	0.046*
H12C	0.7245	0.2153	0.5314	0.046*
C4	0.99678 (17)	0.8405 (5)	0.90809 (12)	0.0342 (5)
H4A	1.0511	0.9482	0.9274	0.041*
C19	0.34438 (16)	0.1657 (5)	0.33928 (13)	0.0343 (5)
H19	0.2805	0.1219	0.3217	0.041*
C1	0.83745 (16)	0.5302 (5)	0.85134 (13)	0.0338 (5)
H1	0.7826	0.4246	0.8318	0.041*
C17	0.49868 (17)	0.1007 (6)	0.42635 (13)	0.0359 (5)
H17	0.5405	0.0121	0.4681	0.043*
C26	0.61548 (16)	0.8774 (5)	0.14491 (12)	0.0307 (5)
H26	0.5934	1.0695	0.1339	0.037*
C3	0.92564 (18)	0.8797 (5)	0.93149 (13)	0.0371 (5)
H3	0.9315	1.0126	0.9672	0.045*
C2	0.84610 (18)	0.7253 (6)	0.90288 (13)	0.0400 (6)
H2	0.7971	0.7535	0.9187	0.048*
C9	1.05202 (17)	0.0179 (6)	0.69334 (14)	0.0389 (6)
H9A	1.0839	0.1873	0.6912	0.058*
H9B	1.0448	−0.0951	0.6515	0.058*
H9C	1.0884	−0.0813	0.7374	0.058*
C24	0.39994 (18)	0.9808 (7)	0.12633 (16)	0.0460 (7)
H24A	0.3655	0.8249	0.0984	0.069*
H24B	0.4216	1.0943	0.0960	0.069*
H24C	0.3596	1.0887	0.1430	0.069*
C29	0.6908 (2)	0.9059 (7)	0.05534 (15)	0.0485 (7)
H29	0.6718	1.0810	0.0349	0.058*
C27	0.7077 (2)	0.8744 (8)	0.21025 (15)	0.0509 (7)
H27A	0.6974	0.9289	0.2533	0.076*
H27B	0.7508	1.0020	0.2022	0.076*
H27C	0.7339	0.6904	0.2170	0.076*
C30	0.7708 (2)	0.8223 (12)	0.0606 (2)	0.0802 (14)
H30A	0.7925	0.6485	0.0807	0.096*
H30B	0.8082	0.9344	0.0444	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O6	0.0295 (7)	0.0313 (8)	0.0306 (8)	0.0010 (6)	0.0143 (6)	0.0062 (7)
O2	0.0254 (7)	0.0311 (8)	0.0252 (7)	−0.0017 (6)	0.0113 (6)	−0.0026 (6)
O4	0.0231 (7)	0.0244 (7)	0.0371 (8)	0.0000 (6)	0.0156 (6)	0.0022 (6)
O1	0.0256 (7)	0.0354 (9)	0.0338 (8)	−0.0041 (6)	0.0126 (6)	−0.0091 (7)
O8	0.0390 (8)	0.0325 (9)	0.0332 (8)	−0.0028 (7)	0.0120 (7)	−0.0013 (7)

O5	0.0304 (8)	0.0417 (10)	0.0430 (9)	−0.0039 (7)	0.0140 (7)	0.0119 (8)
O3	0.0645 (12)	0.0228 (8)	0.0302 (9)	0.0060 (8)	0.0196 (8)	−0.0015 (7)
O7	0.1004 (17)	0.0197 (9)	0.0799 (15)	−0.0014 (9)	0.0678 (14)	−0.0039 (9)
C10	0.0304 (10)	0.0210 (10)	0.0271 (10)	−0.0012 (8)	0.0160 (8)	−0.0004 (8)
C6	0.0301 (10)	0.0246 (10)	0.0192 (9)	0.0013 (8)	0.0077 (8)	0.0028 (8)
C11	0.0245 (10)	0.0200 (9)	0.0253 (10)	0.0001 (7)	0.0130 (8)	−0.0008 (8)
C21	0.0302 (11)	0.0225 (10)	0.0294 (11)	0.0006 (8)	0.0175 (9)	−0.0016 (8)
C13	0.0224 (9)	0.0229 (10)	0.0260 (10)	−0.0006 (8)	0.0130 (8)	−0.0013 (8)
C5	0.0323 (11)	0.0281 (11)	0.0213 (10)	−0.0003 (9)	0.0071 (8)	0.0041 (8)
C22	0.0302 (11)	0.0246 (10)	0.0315 (11)	0.0039 (8)	0.0152 (9)	0.0028 (9)
C8	0.0267 (10)	0.0260 (11)	0.0244 (10)	0.0020 (8)	0.0123 (8)	−0.0019 (8)
C23	0.0336 (11)	0.0253 (11)	0.0326 (11)	0.0028 (9)	0.0176 (9)	0.0041 (9)
C7	0.0246 (10)	0.0259 (10)	0.0223 (10)	0.0015 (8)	0.0088 (8)	0.0021 (8)
C14	0.0319 (11)	0.0296 (11)	0.0291 (11)	−0.0054 (9)	0.0168 (9)	−0.0055 (9)
C25	0.0446 (13)	0.0223 (12)	0.0324 (12)	0.0001 (9)	0.0205 (10)	−0.0028 (9)
C20	0.0301 (11)	0.0341 (12)	0.0306 (11)	0.0002 (9)	0.0145 (9)	−0.0011 (9)
C16	0.0279 (11)	0.0401 (13)	0.0348 (12)	−0.0007 (9)	0.0145 (9)	0.0059 (10)
C15	0.0368 (12)	0.0470 (14)	0.0261 (11)	−0.0045 (10)	0.0107 (10)	−0.0018 (10)
C28	0.0351 (11)	0.0318 (12)	0.0331 (12)	0.0018 (10)	0.0167 (10)	−0.0053 (10)
C18	0.0430 (13)	0.0312 (12)	0.0363 (12)	−0.0046 (10)	0.0254 (11)	−0.0001 (10)
C12	0.0276 (11)	0.0349 (12)	0.0327 (11)	−0.0033 (9)	0.0161 (9)	0.0009 (9)
C4	0.0399 (12)	0.0316 (12)	0.0244 (10)	−0.0055 (9)	0.0049 (9)	0.0017 (9)
C19	0.0325 (11)	0.0396 (13)	0.0355 (12)	−0.0077 (10)	0.0184 (10)	−0.0045 (10)
C1	0.0319 (12)	0.0396 (13)	0.0316 (12)	−0.0028 (10)	0.0140 (9)	−0.0076 (10)
C17	0.0371 (12)	0.0396 (13)	0.0344 (12)	0.0029 (10)	0.0176 (10)	0.0100 (10)
C26	0.0389 (12)	0.0249 (10)	0.0350 (11)	0.0001 (9)	0.0216 (10)	−0.0041 (9)
C3	0.0495 (14)	0.0340 (12)	0.0242 (10)	0.0017 (11)	0.0103 (10)	−0.0060 (10)
C2	0.0420 (13)	0.0467 (14)	0.0342 (12)	0.0023 (11)	0.0180 (10)	−0.0086 (11)
C9	0.0295 (12)	0.0530 (15)	0.0364 (13)	0.0085 (11)	0.0150 (10)	−0.0009 (11)
C24	0.0382 (13)	0.0566 (17)	0.0464 (15)	0.0067 (12)	0.0197 (12)	0.0228 (13)
C29	0.0509 (16)	0.0613 (18)	0.0454 (15)	−0.0170 (14)	0.0318 (13)	−0.0216 (14)
C27	0.0509 (16)	0.0655 (19)	0.0360 (14)	−0.0105 (14)	0.0164 (12)	−0.0122 (14)
C30	0.0534 (19)	0.130 (4)	0.071 (2)	−0.019 (2)	0.0389 (17)	−0.038 (3)

Geometric parameters (Å, º)

O6—C22	1.356 (3)	C16—C17	1.383 (3)
O6—C23	1.436 (3)	C16—H16	0.9500
O2—C7	1.355 (2)	C15—H15A	0.9500
O2—C8	1.438 (2)	C15—H15B	0.9500
O4—C13	1.443 (2)	C28—C29	1.499 (4)
O4—H4	0.8400	C28—C26	1.533 (3)
O1—C7	1.200 (3)	C28—H28	1.0000
O8—C28	1.432 (3)	C18—C17	1.384 (4)
O8—H8	0.8400	C18—C19	1.386 (4)
O5—C22	1.205 (3)	C18—H18	0.9500
O3—C10	1.204 (3)	C12—H12A	0.9800
O7—C25	1.194 (3)	C12—H12B	0.9800

C10—C8	1.519 (3)	C12—H12C	0.9800
C10—C11	1.524 (3)	C4—C3	1.383 (4)
C6—C5	1.392 (3)	C4—H4A	0.9500
C6—C1	1.398 (3)	C19—H19	0.9500
C6—C7	1.486 (3)	C1—C2	1.381 (4)
C11—C13	1.533 (3)	C1—H1	0.9500
C11—C12	1.534 (3)	C17—H17	0.9500
C11—H11	1.0000	C26—C27	1.536 (4)
C21—C16	1.386 (3)	C26—H26	1.0000
C21—C20	1.392 (3)	C3—C2	1.382 (4)
C21—C22	1.485 (3)	C3—H3	0.9500
C13—C14	1.496 (3)	C2—H2	0.9500
C13—H13	1.0000	C9—H9A	0.9800
C5—C4	1.387 (3)	C9—H9B	0.9800
C5—H5	0.9500	C9—H9C	0.9800
C8—C9	1.523 (3)	C24—H24A	0.9800
C8—H8A	1.0000	C24—H24B	0.9800
C23—C24	1.511 (3)	C24—H24C	0.9800
C23—C25	1.525 (3)	C29—C30	1.286 (5)
C23—H23	1.0000	C29—H29	0.9500
C14—C15	1.311 (4)	C27—H27A	0.9800
C14—H14	0.9500	C27—H27B	0.9800
C25—C26	1.515 (3)	C27—H27C	0.9800
C20—C19	1.386 (3)	C30—H30A	0.9500
C20—H20	0.9500	C30—H30B	0.9500

C22—O6—C23	114.68 (17)	O8—C28—C26	110.66 (18)
C7—O2—C8	114.31 (16)	C29—C28—C26	112.2 (2)
C13—O4—H4	109.5	O8—C28—H28	108.0
C28—O8—H8	109.5	C29—C28—H28	108.0
O3—C10—C8	121.1 (2)	C26—C28—H28	108.0
O3—C10—C11	120.7 (2)	C17—C18—C19	120.2 (2)
C8—C10—C11	118.12 (17)	C17—C18—H18	119.9
C5—C6—C1	119.5 (2)	C19—C18—H18	119.9
C5—C6—C7	122.80 (19)	C11—C12—H12A	109.5
C1—C6—C7	117.7 (2)	C11—C12—H12B	109.5
C10—C11—C13	108.21 (16)	H12A—C12—H12B	109.5
C10—C11—C12	107.76 (16)	C11—C12—H12C	109.5
C13—C11—C12	112.39 (17)	H12A—C12—H12C	109.5
C10—C11—H11	109.5	H12B—C12—H12C	109.5
C13—C11—H11	109.5	C3—C4—C5	120.4 (2)
C12—C11—H11	109.5	C3—C4—H4A	119.8
C16—C21—C20	120.2 (2)	C5—C4—H4A	119.8
C16—C21—C22	117.7 (2)	C20—C19—C18	120.2 (2)
C20—C21—C22	122.1 (2)	C20—C19—H19	119.9
O4—C13—C14	110.86 (16)	C18—C19—H19	119.9
O4—C13—C11	106.00 (16)	C2—C1—C6	120.0 (2)
C14—C13—C11	114.91 (17)	C2—C1—H1	120.0

O4—C13—H13	108.3	C6—C1—H1	120.0
C14—C13—H13	108.3	C16—C17—C18	119.9 (2)
C11—C13—H13	108.3	C16—C17—H17	120.1
C4—C5—C6	119.9 (2)	C18—C17—H17	120.1
C4—C5—H5	120.1	C25—C26—C28	110.55 (18)
C6—C5—H5	120.1	C25—C26—C27	106.7 (2)
O5—C22—O6	122.4 (2)	C28—C26—C27	111.5 (2)
O5—C22—C21	125.0 (2)	C25—C26—H26	109.3
O6—C22—C21	112.60 (19)	C28—C26—H26	109.3
O2—C8—C10	109.07 (16)	C27—C26—H26	109.3
O2—C8—C9	107.01 (18)	C2—C3—C4	119.9 (2)
C10—C8—C9	111.59 (18)	C2—C3—H3	120.0
O2—C8—H8A	109.7	C4—C3—H3	120.0
C10—C8—H8A	109.7	C1—C2—C3	120.4 (2)
C9—C8—H8A	109.7	C1—C2—H2	119.8
O6—C23—C24	106.33 (18)	C3—C2—H2	119.8
O6—C23—C25	109.98 (18)	C8—C9—H9A	109.5
C24—C23—C25	110.8 (2)	C8—C9—H9B	109.5
O6—C23—H23	109.9	H9A—C9—H9B	109.5
C24—C23—H23	109.9	C8—C9—H9C	109.5
C25—C23—H23	109.9	H9A—C9—H9C	109.5
O1—C7—O2	123.20 (19)	H9B—C9—H9C	109.5
O1—C7—C6	124.51 (19)	C23—C24—H24A	109.5
O2—C7—C6	112.29 (17)	C23—C24—H24B	109.5
C15—C14—C13	122.5 (2)	H24A—C24—H24B	109.5
C15—C14—H14	118.7	C23—C24—H24C	109.5
C13—C14—H14	118.7	H24A—C24—H24C	109.5
O7—C25—C26	121.7 (2)	H24B—C24—H24C	109.5
O7—C25—C23	121.3 (2)	C30—C29—C28	125.5 (4)
C26—C25—C23	117.00 (19)	C30—C29—H29	117.2
C19—C20—C21	119.5 (2)	C28—C29—H29	117.2
C19—C20—H20	120.3	C26—C27—H27A	109.5
C21—C20—H20	120.3	C26—C27—H27B	109.5
C17—C16—C21	120.1 (2)	H27A—C27—H27B	109.5
C17—C16—H16	120.0	C26—C27—H27C	109.5
C21—C16—H16	120.0	H27A—C27—H27C	109.5
C14—C15—H15A	120.0	H27B—C27—H27C	109.5
C14—C15—H15B	120.0	C29—C30—H30A	120.0
H15A—C15—H15B	120.0	C29—C30—H30B	120.0
O8—C28—C29	109.8 (2)	H30A—C30—H30B	120.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H8···O7	0.84	2.63	3.140 (3)	120
C8—H8*A*···O3^{i}	1.00	2.41	3.235 (3)	140

C23—H23···O7^{ii}	1.00	2.54	3.189 (3)	122
C26—H26···O7^{ii}	1.00	2.29	3.205 (3)	151

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