



Crystal structure and absolute configuration of (4*S*,5*R*,6*S*)-4,5,6-trihydroxy-3-methylcyclohex-2-enone (gabosine H)

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Received 10 March 2017

Accepted 21 March 2017

Edited by C. Rizzoli, Università degli Studi di Parma, Italy

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Keywords: crystal structure; absolute configuration; Mitsunobu inversion reaction; natural product.

CCDC reference: 1539327

Supporting information: this article has supporting information at journals.iucr.org/e

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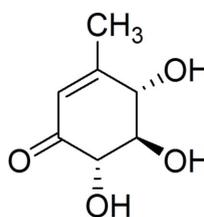
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The molecule of the title keto carbasugar, C₇H₁₀O₄, is formed by a cyclohexene skeleton with an envelope conformation, substituted by carbonyl, methyl and hydroxyl groups. The crystal structure is controlled mainly by a combination of strong O—H···O and weak C—H···O hydrogen bonds, forming nearly perpendicular chains running parallel to the [110] and $[\bar{1}10]$ directions. This perpendicularity is caused by a tetragonal pseudosymmetry influenced by the similarity between the *a* and *b* axes, the value of 90.9770 (10)^o of the β angle and the action of a 2₁ screw axis, which transform each chain into its corresponding nearly orthogonal one.

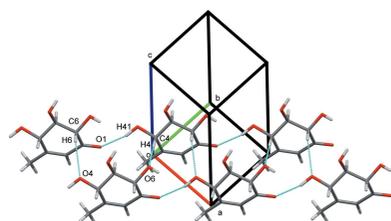
1. Chemical context

Gabosines are regarded as secondary metabolites and were first isolated in 1974 from *Streptomyces strains* (Tsushiya *et al.*, 1974). These compounds are closely related to carbasugars and exhibit DNA binding properties (Tang *et al.*, 2000). To date, 15 gabosines have been isolated, of which 14 have been synthesized. Gabosine H is one of such kind, whose total synthesis has recently been achieved by our research group (Tibhe *et al.*, 2017), starting from a biotransformation of toluene that introduces chirality. A further sequence of reactions, including Mitsunobu and final removal of the acetyl protective group, led to the title compound.



2. Structural commentary

Fig. 1 shows the molecule of the title compound. The absolute configuration of gabosine H with the carbonyl, methyl and hydroxyl groups in equatorial positions, determined as 4*S*,5*R*,6*S* on the basis of synthetic pathway, was confirmed by X-ray diffraction on the basis of anomalous dispersion of light atoms only. The six-membered ring (C1–C6) in the molecule



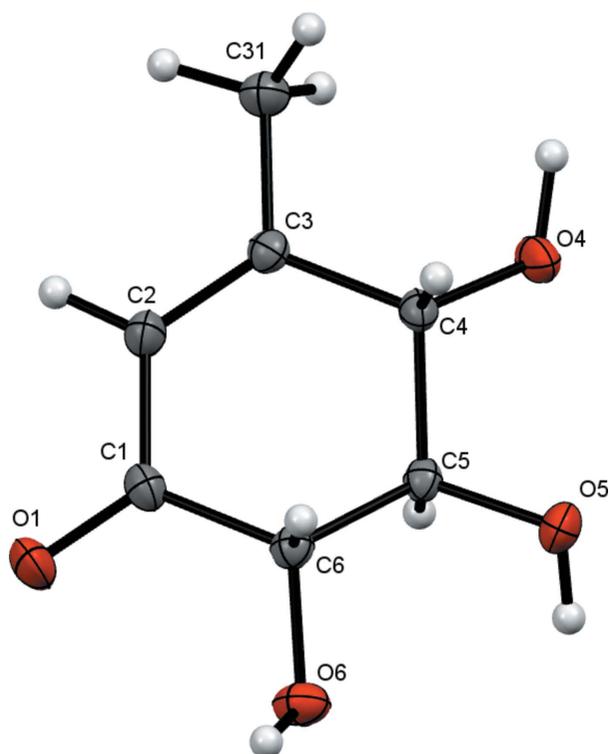


Figure 1
The molecular structure of the title compound, showing the anisotropic displacement ellipsoids drawn at the 50% probability level.

adopts an envelope conformation with atom C5 as the flap [deviating from the plane through the other ring atoms by 0.639 (2) Å] and puckering parameters $Q = 0.4653$ (19) Å, $\theta = 129.5$ (2)° and $\varphi = 66.7$ (3)°.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H41 \cdots O1 ⁱ	0.94 (4)	1.96 (4)	2.873 (2)	163 (3)
C6—H6 \cdots O4 ⁱⁱ	0.98	2.46	3.195 (2)	131
C4—H4 \cdots O6 ⁱⁱⁱ	0.98	2.36	3.345 (3)	179
C6—H6 \cdots O6 ^{iv}	0.98	2.62	3.306 (2)	127
O6—H61 \cdots O5 ^v	0.87 (4)	1.99 (4)	2.811 (2)	155 (3)
O5—H51 \cdots O4 ^{vi}	0.85 (4)	2.45 (3)	3.050 (2)	128 (3)
O5—H51 \cdots O5 ^{vi}	0.85 (4)	2.26 (4)	3.0402 (12)	152 (3)

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

3. Supramolecular features

In the crystal structure, hydrogen bonds O4—H41 \cdots O1ⁱ [symmetry code: (i) $x - 1, y - 1, z$] link the molecules into chains that run along the [110] direction (Table 1). These chains are further connected by weaker C6—H6 \cdots O4ⁱⁱ and C4—H4 \cdots O6ⁱⁱⁱ [symmetry codes: (ii) $x + 1, y, z$; (iii) $x, y - 1, z$] hydrogen bonds along the $[\bar{1}10]$ direction, forming (001) sheets (Fig. 2). Considering that the chains run along the diagonal of the ab plane and the fact that $a \approx b$, it is possible to observe that the 2_1 screw axis parallel to b transforms each chain into a nearly orthogonal one along $[\bar{1}10]$ (Fig. 3). The orthogonal chains are connected by single C6—H6 \cdots O6^{iv}, O6—H61 \cdots O5^v and bifurcated O5—H51 \cdots O4^{vi} and O5—H51 \cdots O5^{vi} hydrogen bonds [symmetry codes: (iv) $-x + 1, y - \frac{1}{2}, -z + 1$; (v) $-x + 1, y + \frac{1}{2}$; (vi) $-x, y + \frac{1}{2}, -z + 1$] to define a three-dimensional array along the [001] direction. These hydrogen bonds connect the orthogonal chains by pairs along

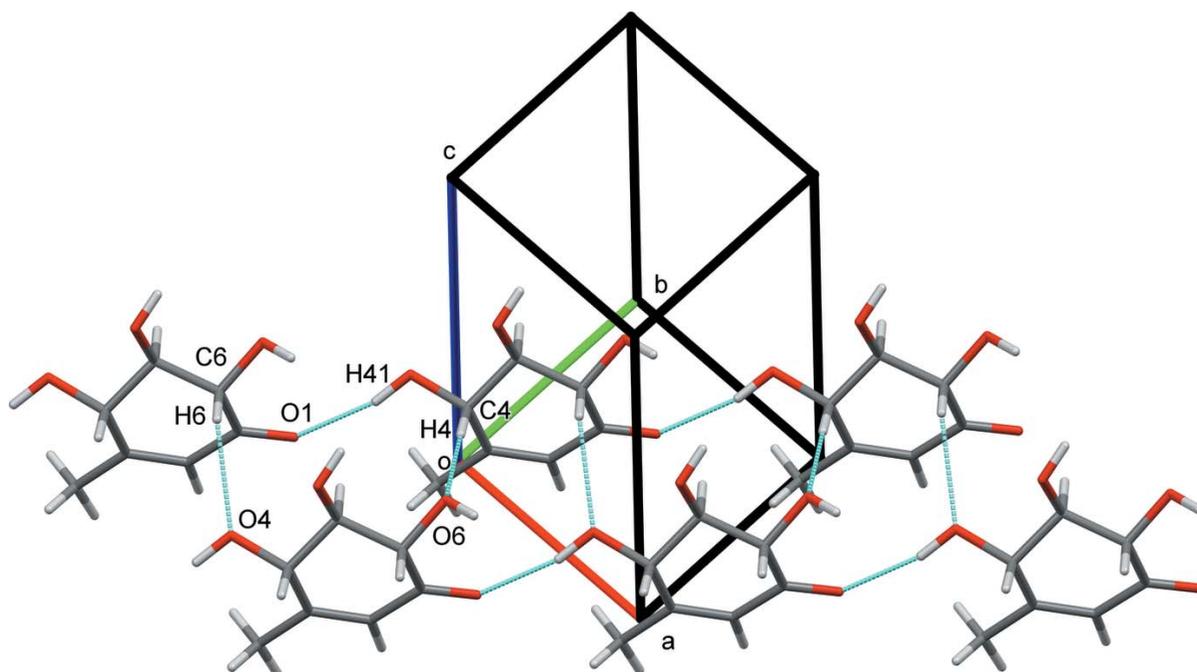


Figure 2
Partial crystal packing of the title compound showing the C—H \cdots O and O—H \cdots O hydrogen bonds (dotted lines) along [110] and $[\bar{1}10]$, forming sheets parallel to the (001) plane.

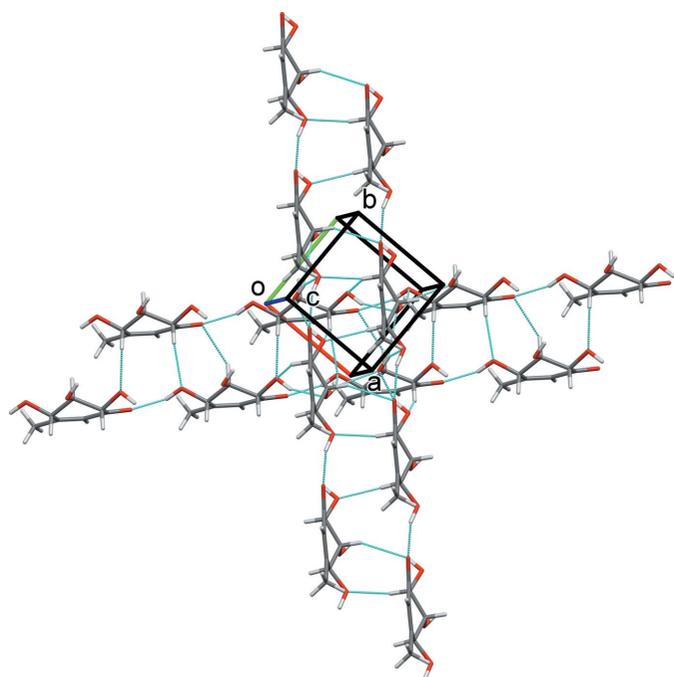


Figure 3
Partial crystal packing of the title compound connected into a nearly orthogonal assembly along [001] through C–H···O and O–H···O hydrogen bonds (dotted lines).

[001]. Between these neighboring [001] sheets, weak dipolar or van der Waals forces stabilize the assembly along the *c*-axis direction.

4. Database survey

A search of the Cambridge Structural Database (CSD Version 5.36 with one update; Groom *et al.*, 2016) was carried out considering molecular structures similar to gabosine and its derivatives. Among the natural compounds, only the structure of gabosine N, (4*R*,5*R*,6*R*)-4,5,6-trihydroxy-2-methylcyclohex-2-enone (Tang *et al.* 2000), has been reported. The remaining hits were mainly derivatives of other gabosines different from H or derivatives such as 5-hydroxy-4-methyl-7-oxabicyclo[4.1.0]hept-3-en-2-one (White *et al.*, 2010), which is an epoxide with different configuration of the asymmetric carbons compared with gabosine H.

5. Synthesis and crystallization

The synthesis of gabosine H was achieved by inversion of the allylic –OH group using Mitsunobu conditions followed by deprotection. (4*R*,5*R*,6*S*)-5-Acetoxy-4,5-dihydroxy-3-methylcyclohex-2-enone (**2**, Fig. 4; 0.149 mmol, 0.030 g) was dissolved in 1 ml of benzene and TPP (0.283 mmol, 0.078 g) was added along with *p*-nitrobenzoic acid (0.299 mmol, 0.050 g) and diisopropyl azodicarboxylate (DIAD; 0.297 mmol, 0.060 g). The reaction mixture was stirred at room temperature for 6 h. The solvent was evaporated and the crude mass was used for the next reaction without further purification. The crude

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₇ H ₁₀ O ₄
<i>M_r</i>	158.15
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.4143 (2), 5.4176 (2), 11.9200 (5)
β (°)	90.977 (1)
<i>V</i> (Å ³)	349.59 (2)
<i>Z</i>	2
Radiation type	Cu Kα
μ (mm ⁻¹)	1.06
Crystal size (mm)	0.37 × 0.34 × 0.10
Data collection	
Diffractometer	Bruker D8 Venture/Photon 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2013)
<i>T_{min}</i> , <i>T_{max}</i>	0.588, 0.754
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	12187, 1492, 1472
<i>R_{int}</i>	0.046
(sin θ/λ) _{max} (Å ⁻¹)	0.637
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.033, 0.088, 1.08
No. of reflections	1492
No. of parameters	111
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.25, −0.18
Absolute structure	Flack <i>x</i> determined using 639 quotients [(<i>I</i> ⁺) − (<i>I</i> [−])] / [(<i>I</i> ⁺) + (<i>I</i> [−])] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.09 (11)

Computer programs: *SAINT* (Bruker, 2013), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008).

product was dissolved in MeOH (3.4 mL), a catalytic quantity of K₂CO₃ was added and the reaction mixture was stirred at room temperature for 5 min and filtered. Evaporation of the solvent from the filtrate afforded crude gabosine H, which was purified by column chromatography (CH₂Cl₂/MeOH, 9:1 *v/v*) to afford pure gabosine H as a white crystalline powder (yield: 7.2 mg, 30%; m.p. 390.6 K. Suitable crystals for X-ray analysis were obtained by dissolving the solid in a minimum amount of methanol and allowing it to evaporate at room temperature. IR (KBr): 3400, 2875, 1660 cm⁻¹. ¹H NMR (400 MHz, CD₃OD): δ = 2.07 (*s*, 3 H), 3.56 (*dd*, *J* = 10.8, 2.4 Hz, 1 H), 4.01 (*d*, *J* = 10.8 Hz, 1 H), 4.23 (*d*, *J* = 8.4 Hz, 1 H), 5.92 (*s*, 1 H).

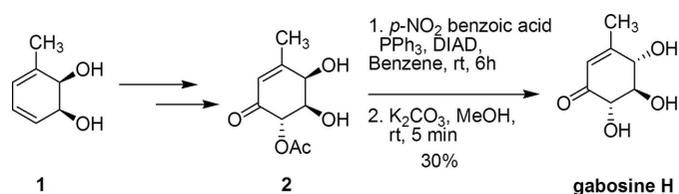


Figure 4
Reaction scheme.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms bonded to C were placed in calculated positions ($C-H = 0.93-0.98 \text{ \AA}$) and included as riding contributions with isotropic displacement parameters set to 1.2–1.5 times the U_{eq} of the parent atom. Hydroxy H atoms were located in difference density maps and were refined with $U_{iso}(H) = 1.5 U_{eq}(O)$. The absolute structure parameter γ was calculated using *PLATON* (Spek, 2009). The resulting value of 0.07 (7) indicates that the absolute structure was determined correctly (Hooft *et al.*, 2008).

Acknowledgements

The authors would like to thank ANII (EQC_2012_07), CSIC and the Facultad de Química for funds to purchase the diffractometer and the financial support of OPCW and PEDECIBA. GT and MM also thank ANII for their respective postdoctoral fellowships (PD_NAC_2014_1_102498 and PD_NAC_2014_1_102409).

Funding information

Funding for this research was provided by: Organisation for the Prohibition of Chemical Weapons; Agencia Nacional de

Investigación e Innovación (award Nos. PD_NAC_2014_1_102498, PD_NAC_2014_1_102409, EQC_2012_07).

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supporting information

Acta Cryst. (2017). E73, 606-609 [https://doi.org/10.1107/S2056989017004509]

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Computing details

Data collection: *SAINT* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

(4*S*,5*R*,6*S*)-4,5,6-Trihydroxy-3-methylcyclohex-2-enone

Crystal data

$C_7H_{10}O_4$
 $M_r = 158.15$
 Monoclinic, $P2_1$
 $a = 5.4143$ (2) Å
 $b = 5.4176$ (2) Å
 $c = 11.9200$ (5) Å
 $\beta = 90.977$ (1)°
 $V = 349.59$ (2) Å³
 $Z = 2$

$F(000) = 168$
 $D_x = 1.502$ Mg m⁻³
 Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
 Cell parameters from 9784 reflections
 $\theta = 3.7$ – 78.8 °
 $\mu = 1.06$ mm⁻¹
 $T = 298$ K
 Parallelepiped, colorless
 $0.37 \times 0.34 \times 0.10$ mm

Data collection

Bruker D8 Venture/Photon 100 CMOS
 diffractometer
 Radiation source: Cu Incoatec microsource
 Detector resolution: 10.4167 pixels mm⁻¹
 ω and ϕ scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2013)
 $T_{\min} = 0.588$, $T_{\max} = 0.754$

12187 measured reflections
 1492 independent reflections
 1472 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\max} = 79.1$ °, $\theta_{\min} = 3.7$ °
 $h = -6 \rightarrow 6$
 $k = -6 \rightarrow 6$
 $l = -15 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.088$
 $S = 1.08$
 1492 reflections
 111 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.0518P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³

Extinction correction: SHELXL2014
(Sheldrick, 2015),
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.161 (15)

Absolute structure: Flack x determined using
639 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons *et al.*,
2013)
Absolute structure parameter: 0.09 (11)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4992 (3)	0.2954 (4)	0.22586 (16)	0.0293 (4)
C2	0.3815 (4)	0.1293 (4)	0.14457 (16)	0.0313 (5)
H2	0.4304	0.1373	0.0702	0.038*
C3	0.2068 (3)	-0.0338 (3)	0.17173 (15)	0.0262 (4)
C31	0.0921 (4)	-0.2029 (5)	0.08715 (17)	0.0374 (5)
H31A	0.1711	-0.1813	0.0163	0.056*
H31B	-0.0807	-0.1656	0.0790	0.056*
H31C	0.1119	-0.3706	0.1116	0.056*
C4	0.1234 (3)	-0.0631 (3)	0.29155 (15)	0.0249 (4)
H4	0.2190	-0.1965	0.3269	0.030*
C5	0.1658 (3)	0.1719 (4)	0.35829 (15)	0.0237 (4)
H5	0.0534	0.3005	0.3302	0.028*
C6	0.4314 (3)	0.2607 (3)	0.34819 (14)	0.0258 (4)
H6	0.5418	0.1368	0.3818	0.031*
O1	0.6472 (3)	0.4524 (3)	0.19769 (14)	0.0453 (5)
O4	-0.1316 (3)	-0.1192 (3)	0.29994 (14)	0.0398 (4)
H41	-0.172 (6)	-0.273 (8)	0.269 (3)	0.060*
O5	0.1120 (3)	0.1206 (3)	0.47319 (11)	0.0336 (4)
H51	0.096 (5)	0.263 (7)	0.502 (3)	0.050*
O6	0.4549 (3)	0.4821 (3)	0.40965 (13)	0.0374 (4)
H61	0.606 (7)	0.485 (7)	0.437 (3)	0.056*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0252 (8)	0.0305 (10)	0.0322 (9)	-0.0046 (8)	0.0038 (7)	0.0021 (8)
C2	0.0322 (9)	0.0360 (11)	0.0257 (8)	-0.0051 (8)	0.0055 (7)	-0.0002 (8)
C3	0.0251 (8)	0.0263 (9)	0.0274 (8)	0.0013 (7)	0.0018 (6)	-0.0011 (7)
C31	0.0429 (11)	0.0358 (11)	0.0335 (10)	-0.0084 (10)	0.0007 (8)	-0.0050 (9)
C4	0.0241 (8)	0.0219 (9)	0.0289 (9)	0.0001 (7)	0.0061 (6)	0.0011 (7)
C5	0.0227 (8)	0.0242 (9)	0.0243 (8)	0.0024 (6)	0.0037 (6)	0.0007 (6)
C6	0.0240 (8)	0.0267 (10)	0.0267 (8)	0.0011 (7)	0.0004 (6)	-0.0019 (7)
O1	0.0464 (9)	0.0459 (10)	0.0440 (8)	-0.0236 (8)	0.0080 (7)	-0.0002 (7)
O4	0.0287 (8)	0.0363 (9)	0.0548 (9)	-0.0099 (6)	0.0152 (6)	-0.0124 (7)
O5	0.0363 (7)	0.0388 (8)	0.0260 (7)	-0.0005 (6)	0.0091 (5)	-0.0016 (6)

O6	0.0329 (7)	0.0364 (9)	0.0429 (8)	-0.0016 (6)	-0.0044 (6)	-0.0125 (7)
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Geometric parameters (Å, °)

C1—O1	1.220 (3)	C4—C5	1.517 (2)
C1—C2	1.461 (3)	C4—H4	0.9800
C1—C6	1.521 (2)	C5—O5	1.432 (2)
C2—C3	1.338 (3)	C5—C6	1.523 (2)
C2—H2	0.9300	C5—H5	0.9800
C3—C31	1.490 (3)	C6—O6	1.410 (2)
C3—C4	1.514 (2)	C6—H6	0.9800
C31—H31A	0.9600	O4—H41	0.94 (4)
C31—H31B	0.9600	O5—H51	0.85 (4)
C31—H31C	0.9600	O6—H61	0.87 (4)
C4—O4	1.419 (2)		
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O1—C1—C2	121.89 (18)	O4—C4—H4	108.6
O1—C1—C6	121.36 (18)	C3—C4—H4	108.6
C2—C1—C6	116.75 (16)	C5—C4—H4	108.6
C3—C2—C1	123.26 (17)	O5—C5—C4	107.89 (15)
C3—C2—H2	118.4	O5—C5—C6	110.15 (14)
C1—C2—H2	118.4	C4—C5—C6	110.99 (14)
C2—C3—C31	122.04 (17)	O5—C5—H5	109.3
C2—C3—C4	121.40 (16)	C4—C5—H5	109.3
C31—C3—C4	116.52 (16)	C6—C5—H5	109.3
C3—C31—H31A	109.5	O6—C6—C1	111.83 (16)
C3—C31—H31B	109.5	O6—C6—C5	107.72 (14)
H31A—C31—H31B	109.5	C1—C6—C5	110.99 (14)
C3—C31—H31C	109.5	O6—C6—H6	108.7
H31A—C31—H31C	109.5	C1—C6—H6	108.7
H31B—C31—H31C	109.5	C5—C6—H6	108.7
O4—C4—C3	113.27 (15)	C4—O4—H41	113 (2)
O4—C4—C5	106.33 (14)	C5—O5—H51	103 (2)
C3—C4—C5	111.22 (14)	C6—O6—H61	106 (2)
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O1—C1—C2—C3	-175.5 (2)	O4—C4—C5—C6	-175.86 (15)
C6—C1—C2—C3	5.4 (3)	C3—C4—C5—C6	-52.12 (19)
C1—C2—C3—C31	-179.29 (19)	O1—C1—C6—O6	28.4 (3)
C1—C2—C3—C4	-1.9 (3)	C2—C1—C6—O6	-152.56 (18)
C2—C3—C4—O4	145.37 (18)	O1—C1—C6—C5	148.7 (2)
C31—C3—C4—O4	-37.2 (2)	C2—C1—C6—C5	-32.2 (2)
C2—C3—C4—C5	25.7 (2)	O5—C5—C6—O6	-62.1 (2)
C31—C3—C4—C5	-156.85 (17)	C4—C5—C6—O6	178.49 (14)
O4—C4—C5—O5	63.37 (19)	O5—C5—C6—C1	175.17 (16)
C3—C4—C5—O5	-172.89 (14)	C4—C5—C6—C1	55.8 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O4—H41 \cdots O1 ⁱ	0.94 (4)	1.96 (4)	2.873 (2)	163 (3)
C6—H6 \cdots O4 ⁱⁱ	0.98	2.46	3.195 (2)	131
C4—H4 \cdots O6 ⁱⁱⁱ	0.98	2.36	3.345 (3)	179
C6—H6 \cdots O6 ^{iv}	0.98	2.62	3.306 (2)	127
O6—H61 \cdots O5 ^v	0.87 (4)	1.99 (4)	2.811 (2)	155 (3)
O5—H51 \cdots O4 ^{vi}	0.85 (4)	2.45 (3)	3.050 (2)	128 (3)
O5—H51 \cdots O5 ^{vi}	0.85 (4)	2.26 (4)	3.0402 (12)	152 (3)

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