

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-[(2*S*,3*R*,4*S*,5*S*,6*R*)-3,4-Dihydroxy-6-hydroxymethyl-3-[(2*S*,3*R*,4*R*,5*R*,6*S*)-3,4,5-trihydroxy-6-methyltetrahydropyran-2-yloxy]tetrahydropyran-2-yloxy]-7-hydroxy-2-(4-hydroxyphenyl)chromen-4-one monohydrate

 Yuzhen Chen,^{a*} Airong Wang,^b Haiyan Gao^c and Shengyang Niu^c

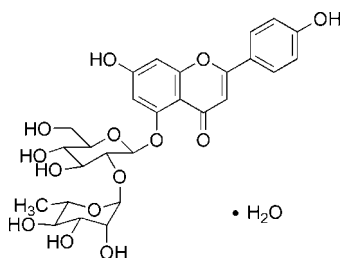
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Received 22 August 2008; accepted 30 August 2008

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.045; wR factor = 0.111; data-to-parameter ratio = 8.1.

In the title compound, $\text{C}_{27}\text{H}_{30}\text{O}_{14}\cdot\text{H}_2\text{O}$, the hydroxyphenyl ring makes a dihedral angle of $20.05(11)^\circ$ with the chromenone ring system. The crystal structure is stabilized by intra- and intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. The absolute configuration was assigned on the basis of an analogous structure.

Related literature

 For related literature, see: Li *et al.* (2007).


Experimental

Crystal data

$\text{C}_{27}\text{H}_{30}\text{O}_{14}\cdot\text{H}_2\text{O}$
 $M_r = 596.53$
 Orthorhombic, $P2_12_12_1$
 $a = 7.2392(14)$ Å
 $b = 9.832(2)$ Å
 $c = 36.782(7)$ Å

$V = 2618.1(9)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 113(2)$ K
 $0.08 \times 0.06 \times 0.04$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.990$, $T_{\max} = 0.995$

20457 measured reflections
 3332 independent reflections
 2875 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.090$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.110$
 $S = 1.05$
 3332 reflections
 410 parameters
 10 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 ⁱ ···O15	0.89 (3)	1.80 (3)	2.684 (3)	177 (5)
O6—H6 ⁱ ···O10 ⁱ	0.82 (3)	2.08 (3)	2.881 (3)	166 (3)
O7—H7 ⁱ ···O11 ⁱ	0.85 (3)	1.99 (3)	2.817 (3)	164 (4)
O8—H8 ⁱ ···O14 ⁱⁱ	0.82 (4)	2.17 (3)	2.821 (3)	136 (3)
O11—H11 ⁱ ···O2 ⁱⁱⁱ	0.87 (3)	2.19 (3)	3.006 (3)	158 (3)
O13—H13 ⁱ ···O2	0.87 (3)	1.79 (3)	2.653 (3)	172 (3)
O14—H14 ⁱ ···O13 ^{iv}	0.87 (3)	1.76 (3)	2.617 (3)	170 (3)
O15—H15A ⁱ ···O8	0.80 (3)	2.06 (4)	2.829 (3)	161 (4)
O15—H15B ⁱ ···O2 ^v	0.84 (3)	2.00 (3)	2.820 (4)	168 (4)

Symmetry codes: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{3}{2}, -z$; (iii) $x + 1, y, z$; (iv) $x - \frac{1}{2}, -y + \frac{5}{2}, -z$; (v) $x, y - 1, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2776).

References

- Li, W., Deng, Y. L., Dai, R. J., Yu, Y. H., Saeed, M. K., Li, L., Meng, W. W. & Zhang, X. S. (2007). *J. Pharm. Biomed. Anal.* **45**, 38–46.
 Rigaku (2005). *CrystalClear* and *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2008). E64, o1901 [doi:10.1107/S1600536808027839]

5-[(2*S*,3*R*,4*S*,5*S*,6*R*)-3,4-Dihydroxy-6-hydroxymethyl-3-[(2*S*,3*R*,4*R*,5*R*,6*S*)-3,4,5-trihydroxy-6-methyltetrahydropyran-2-yloxy]tetrahydropyran-2-yloxy]-7-hydroxy-2-(4-hydroxyphenyl)chromen-4-one monohydrate

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Comment

The title compound was prepared from compound (II) by hydrolysis (scheme 2).

The hydroxyphenyl ring makes a dihedral angle of 20.05 (11)° with the benzopyranone ring. The two saturated rings adopt chair configurations. The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds (Table 1), with O···O distances in the range 2.617 (3)–3.006 (3) Å.

Experimental

Compound (II) was mixed with methanol and K₂CO₃. After stirring for 60 min at 40°C, hydrochloric acid was added to make pH 7.0. The mixture was kept for 24 h at 4°C. The resultant white precipitate was separated by filtration, washed with water and dried for X-ray analysis.

Refinement

In the absence of anomalous scatterers Friedel pairs were merged. The absolute configuration was set according to the literature (Li *et al.*, 2007). The O-bound H atoms were located in a difference map and refined with the restraint O—H = 0.85 (3) Å. All other H atoms were positioned geometrically and refined as riding atoms, with U_{iso}(H) = 1.2 U_{eq}(CH and CH₂) and C—H ranging from 0.95–1.0 Å or U_{iso}(H) = 1.5 U_{eq}(CH₃) and C_{methyl}—H = 0.99 Å.

Figures

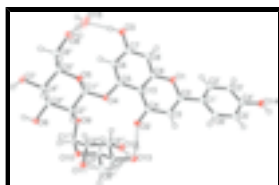


Fig. 1. The molecular structure of the title compound with the atom-numbering scheme and 50% probability displacement ellipsoids. The dashed lines indicates H-bonds.

5-[(2*S*,3*R*,4*S*,5*S*,6*R*)-3,4-Dihydroxy-6- hydroxymethyl-3-[(2*S*,3*R*,4*R*,5*R*,6*S*)-3,4,5- trihydroxy-6-methyltetrahydropyran-2-yloxy]tetrahydropyran-2-yloxy]-7-hydroxy- 2-(4-hydroxyphenyl)chromen-4-one monohydrate

Crystal data

C₂₇H₃₀O₁₄·H₂O

M_r = 596.53

*F*₀₀₀ = 1256

D_x = 1.513 Mg m⁻³

supplementary materials

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.2392$ (14) Å

$b = 9.832$ (2) Å

$c = 36.782$ (7) Å

$V = 2618.1$ (9) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5367 reflections

$\theta = 2.1$ – 27.1°

$\mu = 0.13$ mm⁻¹

$T = 113$ (2) K

Block, white

$0.08 \times 0.06 \times 0.04$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer

Radiation source: rotating anode

Monochromator: confocal

$T = 113$ (2) K

ω scans

Absorption correction: multi-scan (CrystalClear; Rigaku/MSO, 2005)

$T_{\min} = 0.990$, $T_{\max} = 0.995$

20457 measured reflections

3332 independent reflections

2875 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.090$

$\theta_{\max} = 27.1^\circ$

$\theta_{\min} = 2.1^\circ$

$h = -9 \rightarrow 9$

$k = -12 \rightarrow 12$

$l = -30 \rightarrow 47$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.110$

$S = 1.06$

3332 reflections

410 parameters

10 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

$$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.0488P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.31$ e Å⁻³

$\Delta\rho_{\min} = -0.29$ e Å⁻³

Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.4843 (3)	0.7580 (2)	0.00818 (5)	0.0211 (5)
O2	0.5796 (3)	0.9238 (2)	0.10827 (5)	0.0185 (5)
O3	0.4249 (3)	0.3145 (2)	0.05153 (5)	0.0241 (5)
H3	0.407 (5)	0.268 (4)	0.0720 (8)	0.036*
O4	0.5902 (2)	0.6692 (2)	0.13500 (5)	0.0165 (4)
O5	0.4557 (2)	0.5220 (2)	0.17460 (5)	0.0170 (4)
O6	0.8762 (3)	0.5651 (2)	0.24894 (5)	0.0205 (4)
H6	0.909 (5)	0.502 (3)	0.2620 (8)	0.031*
O7	0.6098 (3)	0.3398 (2)	0.25491 (5)	0.0231 (5)
H7	0.622 (5)	0.364 (4)	0.2769 (7)	0.035*
O8	0.2114 (3)	0.2890 (2)	0.17453 (5)	0.0252 (5)
H8	0.140 (5)	0.342 (4)	0.1646 (9)	0.038*
O9	0.9173 (2)	0.6586 (2)	0.17330 (5)	0.0167 (4)
O10	0.9519 (2)	0.8722 (2)	0.20277 (5)	0.0163 (4)
O11	1.3189 (2)	0.8698 (2)	0.17022 (5)	0.0189 (5)
H11	1.403 (4)	0.862 (4)	0.1536 (7)	0.028*
O12	1.1740 (3)	0.8979 (2)	0.09786 (5)	0.0224 (5)
H12	1.155 (5)	0.981 (3)	0.0934 (9)	0.034*
O13	0.8811 (3)	1.0723 (2)	0.11859 (5)	0.0219 (5)
H13	0.779 (4)	1.029 (4)	0.1135 (9)	0.033*
O14	0.4369 (3)	1.1650 (2)	-0.12150 (5)	0.0212 (5)
H14	0.406 (5)	1.250 (3)	-0.1214 (9)	0.032*
O15	0.3844 (4)	0.1713 (3)	0.11315 (6)	0.0375 (6)
H15A	0.358 (6)	0.199 (5)	0.1329 (8)	0.056*
H15B	0.438 (6)	0.096 (3)	0.1150 (12)	0.056*
C2	0.5093 (4)	0.8935 (3)	0.01211 (7)	0.0173 (6)
C3	0.5473 (4)	0.9489 (3)	0.04519 (7)	0.0190 (6)
H3A	0.5692	1.0439	0.0469	0.023*
C4	0.5552 (3)	0.8674 (3)	0.07760 (7)	0.0159 (6)
C5	0.5377 (3)	0.6235 (3)	0.10095 (7)	0.0149 (6)
C6	0.4977 (4)	0.4896 (3)	0.09418 (7)	0.0168 (6)
H6A	0.4939	0.4261	0.1136	0.020*
C7	0.4621 (4)	0.4463 (3)	0.05842 (7)	0.0186 (6)
C8	0.4641 (4)	0.5371 (3)	0.02980 (7)	0.0199 (6)
H8A	0.4449	0.5074	0.0055	0.024*
C9	0.4948 (4)	0.6725 (3)	0.03761 (7)	0.0178 (6)
C13	0.5313 (4)	0.7231 (3)	0.07286 (7)	0.0146 (6)
C1"	0.6285 (4)	0.5673 (3)	0.16087 (7)	0.0150 (5)
H1"	0.6935	0.4899	0.1488	0.018*
C2"	0.7487 (3)	0.6234 (3)	0.19134 (7)	0.0154 (6)
H2"	0.6905	0.7053	0.2027	0.019*
C3"	0.7742 (4)	0.5101 (3)	0.21932 (7)	0.0167 (6)

supplementary materials

H3''	0.8472	0.4347	0.2080	0.020*
C4''	0.5868 (4)	0.4542 (3)	0.23160 (7)	0.0167 (6)
H4''	0.5165	0.5270	0.2446	0.020*
C5''	0.4779 (4)	0.4074 (3)	0.19843 (7)	0.0158 (6)
H5''	0.5506	0.3357	0.1855	0.019*
C6''	0.2879 (4)	0.3529 (3)	0.20634 (7)	0.0203 (6)
H6''1	0.2948	0.2860	0.2264	0.024*
H6''2	0.2064	0.4283	0.2142	0.024*
C1'''	1.0404 (3)	0.7556 (3)	0.18874 (7)	0.0146 (5)
H1'''	1.1133	0.7111	0.2085	0.018*
C2'''	1.1695 (3)	0.7886 (3)	0.15686 (7)	0.0159 (6)
H2'''	1.2187	0.7025	0.1461	0.019*
C3'''	1.0581 (4)	0.8670 (3)	0.12806 (7)	0.0170 (6)
H3'''	0.9544	0.8080	0.1195	0.020*
C4'''	0.9774 (4)	0.9936 (3)	0.14530 (7)	0.0162 (6)
H4'''	1.0803	1.0497	0.1555	0.019*
C5'''	0.8485 (3)	0.9518 (3)	0.17625 (7)	0.0163 (6)
H5'''	0.7459	0.8949	0.1662	0.020*
C6'''	0.7673 (4)	1.0724 (4)	0.19609 (8)	0.0253 (7)
H6'''3	0.7005	1.0407	0.2177	0.038*
H6'''4	0.6819	1.1207	0.1800	0.038*
H6'''5	0.8668	1.1339	0.2035	0.038*
C1'	0.4910 (4)	0.9672 (3)	-0.02252 (7)	0.0182 (6)
C2'	0.5075 (4)	0.8970 (3)	-0.05549 (7)	0.0192 (6)
H2'	0.5297	0.8018	-0.0555	0.023*
C3'	0.4916 (4)	0.9662 (3)	-0.08794 (7)	0.0190 (6)
H3'	0.5052	0.9185	-0.1102	0.023*
C4'	0.4559 (4)	1.1048 (3)	-0.08840 (7)	0.0179 (6)
C5'	0.4378 (4)	1.1761 (3)	-0.05556 (7)	0.0202 (6)
H5'	0.4134	1.2710	-0.0556	0.024*
C6'	0.4557 (4)	1.1065 (3)	-0.02315 (7)	0.0223 (6)
H6'	0.4439	1.1545	-0.0009	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0317 (11)	0.0152 (12)	0.0164 (9)	-0.0001 (9)	-0.0024 (8)	0.0025 (9)
O2	0.0233 (10)	0.0176 (13)	0.0144 (9)	-0.0028 (8)	-0.0018 (7)	-0.0026 (8)
O3	0.0339 (12)	0.0184 (14)	0.0201 (10)	-0.0024 (9)	-0.0012 (8)	-0.0014 (9)
O4	0.0217 (9)	0.0151 (12)	0.0126 (8)	-0.0015 (8)	-0.0021 (7)	0.0012 (8)
O5	0.0143 (8)	0.0181 (12)	0.0187 (9)	-0.0011 (8)	-0.0007 (7)	0.0047 (9)
O6	0.0237 (10)	0.0176 (12)	0.0203 (10)	-0.0044 (9)	-0.0101 (8)	0.0030 (9)
O7	0.0326 (11)	0.0202 (12)	0.0164 (9)	-0.0053 (10)	-0.0043 (8)	0.0068 (9)
O8	0.0251 (11)	0.0260 (14)	0.0246 (10)	-0.0073 (9)	-0.0074 (8)	0.0007 (10)
O9	0.0157 (9)	0.0172 (12)	0.0173 (9)	-0.0046 (8)	0.0012 (7)	-0.0033 (8)
O10	0.0179 (9)	0.0153 (12)	0.0157 (8)	0.0015 (8)	-0.0008 (7)	-0.0011 (8)
O11	0.0153 (9)	0.0239 (13)	0.0175 (9)	-0.0033 (8)	0.0003 (7)	-0.0034 (9)
O12	0.0237 (10)	0.0250 (14)	0.0184 (10)	-0.0009 (9)	0.0081 (7)	0.0021 (10)

O13	0.0227 (10)	0.0177 (13)	0.0253 (10)	-0.0020 (9)	-0.0050 (8)	0.0049 (9)
O14	0.0330 (11)	0.0170 (13)	0.0137 (9)	0.0016 (10)	-0.0006 (7)	0.0029 (9)
O15	0.0558 (16)	0.0267 (17)	0.0299 (12)	0.0115 (13)	0.0134 (11)	0.0070 (12)
C2	0.0202 (13)	0.0136 (16)	0.0182 (12)	-0.0012 (11)	0.0004 (9)	0.0007 (12)
C3	0.0242 (13)	0.0166 (17)	0.0162 (12)	-0.0025 (12)	-0.0006 (10)	0.0015 (11)
C4	0.0138 (11)	0.0179 (17)	0.0160 (12)	-0.0024 (11)	-0.0004 (9)	-0.0009 (11)
C5	0.0151 (11)	0.0162 (16)	0.0134 (11)	-0.0001 (11)	-0.0011 (9)	-0.0008 (11)
C6	0.0195 (13)	0.0152 (17)	0.0158 (13)	0.0021 (11)	-0.0011 (9)	0.0017 (12)
C7	0.0182 (12)	0.0164 (17)	0.0213 (13)	-0.0012 (11)	0.0003 (10)	-0.0029 (12)
C8	0.0267 (14)	0.0188 (18)	0.0144 (12)	0.0005 (12)	-0.0027 (10)	-0.0027 (11)
C9	0.0185 (13)	0.0200 (18)	0.0149 (12)	-0.0014 (11)	-0.0014 (9)	0.0024 (12)
C13	0.0171 (12)	0.0136 (16)	0.0132 (12)	-0.0003 (11)	-0.0008 (9)	0.0007 (11)
C1"	0.0172 (12)	0.0134 (15)	0.0143 (11)	-0.0002 (11)	-0.0005 (9)	0.0013 (11)
C2"	0.0155 (12)	0.0155 (17)	0.0153 (12)	-0.0021 (11)	0.0009 (9)	-0.0001 (11)
C3"	0.0189 (13)	0.0154 (17)	0.0160 (12)	-0.0011 (11)	-0.0039 (9)	-0.0017 (11)
C4"	0.0195 (12)	0.0155 (16)	0.0152 (12)	-0.0018 (11)	-0.0010 (9)	0.0017 (11)
C5"	0.0210 (13)	0.0121 (16)	0.0143 (12)	-0.0006 (11)	-0.0006 (9)	0.0025 (11)
C6"	0.0195 (12)	0.0228 (18)	0.0186 (13)	-0.0053 (13)	-0.0021 (9)	0.0022 (13)
C1'''	0.0147 (11)	0.0114 (15)	0.0178 (12)	-0.0007 (11)	-0.0007 (9)	-0.0048 (11)
C2'''	0.0144 (12)	0.0152 (16)	0.0180 (12)	-0.0025 (10)	-0.0013 (9)	-0.0028 (11)
C3'''	0.0166 (12)	0.0195 (17)	0.0150 (12)	-0.0038 (11)	0.0007 (9)	0.0019 (11)
C4'''	0.0177 (12)	0.0156 (17)	0.0152 (12)	-0.0002 (11)	-0.0028 (9)	0.0046 (11)
C5'''	0.0149 (12)	0.0163 (16)	0.0178 (12)	-0.0005 (11)	-0.0001 (9)	0.0009 (12)
C6'''	0.0255 (14)	0.025 (2)	0.0250 (14)	0.0081 (13)	0.0017 (11)	-0.0009 (13)
C1'	0.0202 (13)	0.0202 (18)	0.0142 (12)	0.0015 (12)	-0.0005 (9)	0.0013 (12)
C2'	0.0243 (14)	0.0163 (17)	0.0172 (13)	0.0023 (12)	-0.0004 (10)	-0.0006 (12)
C3'	0.0264 (14)	0.0153 (17)	0.0153 (12)	0.0019 (12)	0.0007 (10)	-0.0023 (11)
C4'	0.0192 (12)	0.0196 (17)	0.0149 (12)	-0.0013 (12)	0.0005 (9)	0.0018 (12)
C5'	0.0306 (15)	0.0114 (17)	0.0186 (13)	0.0010 (12)	0.0000 (11)	-0.0017 (12)
C6'	0.0328 (15)	0.0180 (18)	0.0159 (13)	0.0025 (13)	0.0010 (11)	-0.0031 (12)

Geometric parameters (Å, °)

O1—C2	1.352 (4)	C8—C9	1.379 (5)
O1—C9	1.373 (3)	C8—H8A	0.9500
O2—C4	1.269 (3)	C9—C13	1.414 (4)
O3—C7	1.347 (4)	C1"—C2"	1.522 (4)
O3—H3	0.89 (2)	C1"—H1"	1.0000
O4—C5	1.384 (3)	C2"—C3"	1.528 (4)
O4—C1"	1.409 (3)	C2"—H2"	1.0000
O5—C1"	1.420 (3)	C3"—C4"	1.532 (4)
O5—C5"	1.436 (3)	C3"—H3"	1.0000
O6—C3"	1.423 (3)	C4"—C5"	1.524 (3)
O6—H6	0.82 (2)	C4"—H4"	1.0000
O7—C4"	1.424 (4)	C5"—C6"	1.505 (4)
O7—H7	0.85 (2)	C5"—H5"	1.0000
O8—C6"	1.439 (3)	C6"—H6"1	0.9900
O8—H8	0.82 (2)	C6"—H6"2	0.9900
O9—C1'''	1.423 (3)	C1'''—C2'''	1.534 (4)

supplementary materials

O9—C2''	1.432 (3)	C1'''—H1'''	1.0000
O10—C1'''	1.412 (3)	C2'''—C3'''	1.539 (4)
O10—C5'''	1.457 (3)	C2'''—H2'''	1.0000
O11—C2'''	1.431 (3)	C3'''—C4'''	1.514 (4)
O11—H11	0.86 (2)	C3'''—H3'''	1.0000
O12—C3'''	1.425 (3)	C4'''—C5'''	1.529 (4)
O12—H12	0.85 (3)	C4'''—H4'''	1.0000
O13—C4'''	1.432 (3)	C5'''—C6'''	1.511 (4)
O13—H13	0.87 (2)	C5'''—H5'''	1.0000
O14—C4'	1.360 (3)	C6'''—H6'''3	0.9800
O14—H14	0.87 (3)	C6'''—H6'''4	0.9800
O15—H15A	0.80 (3)	C6'''—H6'''5	0.9800
O15—H15B	0.84 (3)	C1'—C6'	1.393 (5)
C2—C3	1.361 (4)	C1'—C2'	1.400 (4)
C2—C1'	1.472 (4)	C2'—C3'	1.379 (4)
C3—C4	1.437 (4)	C2'—H2'	0.9500
C3—H3A	0.9500	C3'—C4'	1.386 (4)
C4—C13	1.440 (4)	C3'—H3'	0.9500
C5—C6	1.371 (4)	C4'—C5'	1.403 (4)
C5—C13	1.424 (4)	C5'—C6'	1.381 (4)
C6—C7	1.406 (4)	C5'—H5'	0.9500
C6—H6A	0.9500	C6'—H6'	0.9500
C7—C8	1.381 (4)		
C2—O1—C9	120.8 (2)	O5—C5''—C4''	108.0 (2)
C7—O3—H3	111 (2)	C6''—C5''—C4''	115.2 (2)
C5—O4—C1''	115.7 (2)	O5—C5''—H5''	108.8
C1''—O5—C5'''	111.37 (19)	C6''—C5''—H5''	108.8
C3''—O6—H6	108 (3)	C4''—C5''—H5''	108.8
C4''—O7—H7	112 (3)	O8—C6''—C5''	110.5 (2)
C6''—O8—H8	109 (3)	O8—C6''—H6''1	109.6
C1'''—O9—C2''	120.72 (19)	C5''—C6''—H6''1	109.6
C1'''—O10—C5'''	115.13 (19)	O8—C6''—H6''2	109.6
C2'''—O11—H11	104 (2)	C5''—C6''—H6''2	109.6
C3'''—O12—H12	105 (3)	H6''1—C6''—H6''2	108.1
C4'''—O13—H13	107 (2)	O10—C1'''—O9	113.9 (2)
C4'—O14—H14	116 (2)	O10—C1'''—C2'''	112.6 (2)
H15A—O15—H15B	110 (4)	O9—C1'''—C2'''	102.6 (2)
O1—C2—C3	121.1 (3)	O10—C1'''—H1'''	109.2
O1—C2—C1'	112.4 (2)	O9—C1'''—H1'''	109.2
C3—C2—C1'	126.5 (3)	C2'''—C1'''—H1'''	109.2
C2—C3—C4	121.8 (3)	O11—C2'''—C1'''	108.4 (2)
C2—C3—H3A	119.1	O11—C2'''—C3'''	110.7 (2)
C4—C3—H3A	119.1	C1'''—C2'''—C3'''	108.2 (2)
O2—C4—C3	120.0 (3)	O11—C2'''—H2'''	109.8
O2—C4—C13	123.7 (2)	C1'''—C2'''—H2'''	109.8
C3—C4—C13	116.4 (2)	C3'''—C2'''—H2'''	109.8
C6—C5—O4	122.3 (2)	O12—C3'''—C4'''	112.2 (2)
C6—C5—C13	121.4 (2)	O12—C3'''—C2'''	109.6 (2)
O4—C5—C13	116.3 (2)	C4'''—C3'''—C2'''	109.0 (2)

C5—C6—C7	120.0 (3)	O12—C3'''—H3'''	108.7
C5—C6—H6A	120.0	C4'''—C3'''—H3'''	108.7
C7—C6—H6A	120.0	C2'''—C3'''—H3'''	108.7
O3—C7—C8	118.7 (2)	O13—C4'''—C3'''	110.2 (2)
O3—C7—C6	120.3 (3)	O13—C4'''—C5'''	111.0 (2)
C8—C7—C6	121.0 (3)	C3'''—C4'''—C5'''	109.0 (2)
C9—C8—C7	117.8 (3)	O13—C4'''—H4'''	108.9
C9—C8—H8A	121.1	C3'''—C4'''—H4'''	108.9
C7—C8—H8A	121.1	C5'''—C4'''—H4'''	108.9
O1—C9—C8	114.7 (2)	O10—C5'''—C6'''	107.3 (2)
O1—C9—C13	121.2 (3)	O10—C5'''—C4'''	109.2 (2)
C8—C9—C13	124.1 (3)	C6'''—C5'''—C4'''	112.7 (3)
C9—C13—C5	115.4 (3)	O10—C5'''—H5'''	109.2
C9—C13—C4	118.7 (2)	C6'''—C5'''—H5'''	109.2
C5—C13—C4	125.8 (2)	C4'''—C5'''—H5'''	109.2
O4—C1''—O5	106.9 (2)	C5'''—C6'''—H6'''3	109.5
O4—C1''—C2''	110.6 (2)	C5'''—C6'''—H6'''4	109.5
O5—C1''—C2''	110.8 (2)	H6'''3—C6'''—H6'''4	109.5
O4—C1''—H1''	109.5	C5'''—C6'''—H6'''5	109.5
O5—C1''—H1''	109.5	H6'''3—C6'''—H6'''5	109.5
C2''—C1''—H1''	109.5	H6'''4—C6'''—H6'''5	109.5
O9—C2''—C1''	103.53 (19)	C6'—C1'—C2'	119.1 (3)
O9—C2''—C3''	112.7 (2)	C6'—C1'—C2	121.0 (2)
C1''—C2''—C3''	107.5 (2)	C2'—C1'—C2	119.9 (3)
O9—C2''—H2''	110.9	C3'—C2'—C1'	119.9 (3)
C1''—C2''—H2''	110.9	C3'—C2'—H2'	120.0
C3''—C2''—H2''	110.9	C1'—C2'—H2'	120.0
O6—C3''—C2''	107.5 (2)	C2'—C3'—C4'	120.7 (3)
O6—C3''—C4''	111.7 (2)	C2'—C3'—H3'	119.6
C2''—C3''—C4''	110.7 (2)	C4'—C3'—H3'	119.6
O6—C3''—H3''	109.0	O14—C4'—C3'	117.2 (2)
C2''—C3''—H3''	109.0	O14—C4'—C5'	122.9 (3)
C4''—C3''—H3''	109.0	C3'—C4'—C5'	119.9 (3)
O7—C4''—C5''	107.7 (2)	C6'—C5'—C4'	119.2 (3)
O7—C4''—C3''	110.9 (2)	C6'—C5'—H5'	120.4
C5''—C4''—C3''	109.3 (2)	C4'—C5'—H5'	120.4
O7—C4''—H4''	109.6	C5'—C6'—C1'	121.2 (3)
C5''—C4''—H4''	109.6	C5'—C6'—H6'	119.4
C3''—C4''—H4''	109.6	C1'—C6'—H6'	119.4
O5—C5''—C6''	107.1 (2)		
C9—O1—C2—C3	0.7 (4)	O6—C3''—C4''—C5''	175.5 (2)
C9—O1—C2—C1'	-179.5 (2)	C2''—C3''—C4''—C5''	55.7 (3)
O1—C2—C3—C4	-2.6 (4)	C1''—O5—C5''—C6''	-170.6 (2)
C1'—C2—C3—C4	177.6 (2)	C1''—O5—C5''—C4''	64.7 (3)
C2—C3—C4—O2	-175.6 (3)	O7—C4''—C5''—O5	-179.4 (2)
C2—C3—C4—C13	3.5 (4)	C3''—C4''—C5''—O5	-58.9 (3)
C1''—O4—C5—C6	9.7 (3)	O7—C4''—C5''—C6''	60.8 (3)
C1''—O4—C5—C13	-169.0 (2)	C3''—C4''—C5''—C6''	-178.6 (3)
O4—C5—C6—C7	-174.0 (2)	O5—C5''—C6''—O8	70.4 (3)

supplementary materials

C13—C5—C6—C7	4.6 (4)	C4"—C5"—C6"—O8	-169.4 (2)
C5—C6—C7—O3	179.2 (3)	C5'''—O10—C1'''—O9	-61.0 (3)
C5—C6—C7—C8	-1.0 (4)	C5'''—O10—C1'''—C2'''	55.4 (3)
O3—C7—C8—C9	177.3 (3)	C2''—O9—C1''—O10	-43.8 (3)
C6—C7—C8—C9	-2.5 (4)	C2''—O9—C1''—C2'''	-165.8 (2)
C2—O1—C9—C8	179.2 (2)	O10—C1''—C2''—O11	66.0 (3)
C2—O1—C9—C13	0.2 (4)	O9—C1''—C2''—O11	-171.1 (2)
C7—C8—C9—O1	-176.4 (2)	O10—C1''—C2''—C3'''	-54.2 (3)
C7—C8—C9—C13	2.5 (4)	O9—C1''—C2''—C3'''	68.7 (3)
O1—C9—C13—C5	179.7 (2)	O11—C2''—C3''—O12	62.0 (3)
C8—C9—C13—C5	0.9 (4)	C1''—C2''—C3''—O12	-179.3 (2)
O1—C9—C13—C4	0.8 (4)	O11—C2''—C3''—C4'''	-61.2 (3)
C8—C9—C13—C4	-178.1 (3)	C1''—C2''—C3''—C4'''	57.5 (3)
C6—C5—C13—C9	-4.5 (4)	O12—C3''—C4''—O13	55.4 (3)
O4—C5—C13—C9	174.2 (2)	C2''—C3''—C4''—O13	176.9 (2)
C6—C5—C13—C4	174.4 (3)	O12—C3''—C4''—C5'''	177.5 (2)
O4—C5—C13—C4	-6.9 (4)	C2''—C3''—C4''—C5'''	-61.0 (3)
O2—C4—C13—C9	176.5 (2)	C1''—O10—C5'''—C6'''	-179.1 (2)
C3—C4—C13—C9	-2.5 (4)	C1''—O10—C5'''—C4'''	-56.6 (3)
O2—C4—C13—C5	-2.3 (4)	O13—C4''—C5''—O10	-179.9 (2)
C3—C4—C13—C5	178.7 (2)	C3''—C4''—C5''—O10	58.5 (3)
C5—O4—C1''—O5	-80.2 (3)	O13—C4''—C5''—C6'''	-60.8 (3)
C5—O4—C1''—C2''	159.1 (2)	C3''—C4''—C5''—C6'''	177.7 (2)
C5''—O5—C1''—O4	173.8 (2)	O1—C2—C1'—C6'	160.3 (3)
C5''—O5—C1''—C2''	-65.7 (3)	C3—C2—C1'—C6'	-19.9 (4)
C1'''—O9—C2''—C1''	158.0 (2)	O1—C2—C1'—C2'	-19.2 (3)
C1'''—O9—C2''—C3''	-86.2 (3)	C3—C2—C1'—C2'	160.6 (3)
O4—C1''—C2''—O9	-64.0 (3)	C6'—C1'—C2'—C3'	0.9 (4)
O5—C1''—C2''—O9	177.7 (2)	C2—C1'—C2'—C3'	-179.6 (3)
O4—C1''—C2''—C3''	176.6 (2)	C1'—C2'—C3'—C4'	-1.1 (4)
O5—C1''—C2''—C3''	58.3 (3)	C2'—C3'—C4'—O14	-178.2 (2)
O9—C2''—C3''—O6	70.4 (3)	C2'—C3'—C4'—C5'	0.7 (4)
C1''—C2''—C3''—O6	-176.2 (2)	O14—C4'—C5'—C6'	178.8 (3)
O9—C2''—C3''—C4''	-167.4 (2)	C3'—C4'—C5'—C6'	0.0 (4)
C1''—C2''—C3''—C4''	-53.9 (3)	C4'—C5'—C6'—C1'	-0.2 (4)
O6—C3''—C4''—O7	-66.0 (3)	C2'—C1'—C6'—C5'	-0.3 (4)
C2''—C3''—C4''—O7	174.2 (2)	C2—C1'—C6'—C5'	-179.7 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O15	0.89 (3)	1.80 (3)	2.684 (3)	177 (5)
O6—H6 \cdots O10 ⁱ	0.82 (3)	2.08 (3)	2.881 (3)	166 (3)
O7—H7 \cdots O11 ⁱ	0.85 (3)	1.99 (3)	2.817 (3)	164 (4)
O8—H8 \cdots O14 ⁱⁱ	0.82 (4)	2.17 (3)	2.821 (3)	136 (3)
O11—H11 \cdots O2 ⁱⁱⁱ	0.87 (3)	2.19 (3)	3.006 (3)	158 (3)
O13—H13 \cdots O2	0.87 (3)	1.79 (3)	2.653 (3)	172 (3)
O14—H14 \cdots O13 ^{iv}	0.87 (3)	1.76 (3)	2.617 (3)	170 (3)

O15—H15A···O8	0.80 (3)	2.06 (4)	2.829 (3)	161 (4)
O15—H15B···O2 ^v	0.84 (3)	2.00 (3)	2.820 (4)	168 (4)

Symmetry codes: (i) $-x+2, y-1/2, -z+1/2$; (ii) $x-1/2, -y+3/2, -z$; (iii) $x+1, y, z$; (iv) $x-1/2, -y+5/2, -z$; (v) $x, y-1, z$.

Fig. 1

