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7-Hydroxy-4′,5′-methylenedioxypterocarpan hemihydrate

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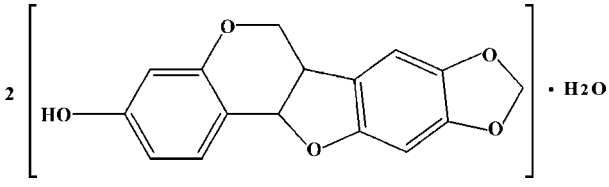
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.078; data-to-parameter ratio = 6.7.

The asymmetric unit of the title compound, $C_{16}H_{12}O_5\cdot0.5H_2O$, contains two essentially identical independent molecules and a water molecule of crystallization. The five fused rings form a non-planar structure in each molecule. The pyran ring is in a half-chair conformation, while the furan ring and the dioxolane ring adopt envelope conformations. The three molecules are linked through O—H···O hydrogen bonds. The O—H···O hydrogen bonds, C—H···π interactions and π–π stacking [centroid–centroid distance 3.7522 (2) Å] help to stabilize the crystal structure.

Related literature

For related literature, see: Kim *et al.* (2006); Li (2006); Liu *et al.* (1980); Salem & Werbovetz (2006).



Experimental

Crystal data

$C_{16}H_{12}O_5\cdot0.5H_2O$
M_r = 293.27
Monoclinic, $P2_1$
a = 6.5998 (13) Å
b = 7.6711 (16) Å
c = 26.547 (5) Å
β = 91.318 (7)°
V = 1343.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 273 (2) K
0.21 × 0.13 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.977, T_{max} = 0.990
14367 measured reflections
2588 independent reflections
2428 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.078
S = 1.06
2588 reflections
389 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

*Cg*2 and *Cg*3 are the centroids of the benzene rings C17–C22 and C26–C31, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O1	0.82	1.94	2.714 (3)	157
O1*W*—H1*WA*···O6	0.95	1.84	2.784 (3)	173
O1—H1···O1*W*^{i}	0.82	1.82	2.608 (3)	162
O1*W*—H1*WB*···O3^{ii}	1.00	1.91	2.870 (3)	159
C16—H16*B*···*Cg*2^{iii}	0.97	2.99	3.663 (4)	128
C28—H28···*Cg*3^{iv}	0.93	2.93	3.722 (3)	144
C29—H29···*Cg*3^{v}	0.93	2.93	3.776 (3)	151

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

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2211).

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supplementary materials

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7-Hydroxy-4',5'-methylenedioxypterocarpan hemihydrate

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Comment

Isoflavonoids are valuable secondary metabolites of plants. Many of them have biological activity, such as glycosidase inhibitory activity (Liu *et al.*, 1980), anti-microbial activity, anti-oxidant activity (Li, 2006), anti-protozoal activity (Salem & Werbovetz, 2006), *etc*. Here, the title isoflavone compound (I) was isolated from the seeds of Sophora davidii, found in China. This compound was discovered as effective inhibitors of α-glucosidase and β-amylase (Kim *et al.*, 2006).

The crystal structure of (I), $C_{16}H_{12}O_5$, contains two independent molecules and a lattice water molecule (Fig. 1). The five fused rings form a non-coplanar structure as seen in the dihedral angle of 43.79 (7)° formed between the C1—C6 and C10—C15 phenyl rings, and 36.26 (9)° formed between the C17—C22 and C26—C31 phenyl rings. The three molecules comprising the asymmetric unit are linked *via* O—H···O hydrogen bonds (Table 1). The crystal structure is further consolidated by C—H···π interactions as well as π···π stacking, see data in Table 1.

Experimental

Seeds of Sophora davidii were collected from Anshun in Guizhou Province (China) and the air-dried. Seeds (6 kg) were powdered and refluxed with 75% EtOH three times. The combined extract was evaporated under reduced pressure to give a residue which was suspended in water and fractionated with ethyl acetate (5*L* × 3 times) and n-butanol (5*L* × 3 times). The ethyl acetate fraction was subjected to column chromatography on silica gel with $CHCl_3$—CH_3OH (10:1) as the eluting solvent to afford (I) (Yield 1.5 g). Single crystals suitable for X-ray diffraction were obtained by the slow evaporation of an ethanol solution of (I) held at room temperature.

Refinement

Water-bound H atoms was located in a difference Fourier map and fixed in these as-found positions with $U_{iso}(H) = 1.2U_{eq}(O)$, see Table 1 for distances. The other H atoms were placed in calculated positions and refined in the riding model approximation with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$. In the absence of significant anomalous scattering effects, 1747 Friedel pairs were averaged in the final refinement.

Figures

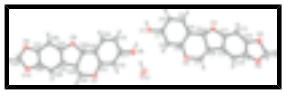


Fig. 1. The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

7-Hydroxy-4',5'-methylenedioxypterocarpan hemihydrate

Crystal data

$C_{16}H_{12}O_5 \cdot 0.5H_2O$	$F_{000} = 612$
$M_r = 293.27$	$D_x = 1.450$ Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 175-176° C K
Hall symbol: P 2yb	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.5998$ (13) Å	Cell parameters from 14367 reflections
$b = 7.6711$ (16) Å	$\theta = 0.8$–25.2º
$c = 26.547$ (5) Å	$\mu = 0.11$ mm^{-1}
$\beta = 91.318$ (7)º	$T = 273$ (2) K
$V = 1343.7$ (5) Å^3	Prism, colorless
$Z = 4$	0.21 × 0.13 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	2588 independent reflections
Radiation source: fine-focus sealed tube	2428 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.032$
$T = 273$(2) K	$\theta_{max} = 25.2$º
φ and ω scans	$\theta_{min} = 0.8$º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -7 \rightarrow 7$
$T_{min} = 0.977$, $T_{max} = 0.990$	$k = -9 \rightarrow 9$
14367 measured reflections	$l = -31 \rightarrow 28$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.2147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$(\Delta/\sigma)_{max} = <0.001$
$wR(F^2) = 0.078$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.12$ e Å^{-3}
2588 reflections	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
389 parameters	Extinction coefficient: 0.0094 (15)
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C17	0.3735 (4)	0.2865 (4)	0.28681 (9)	0.0410 (6)
C18	0.1732 (4)	0.2929 (4)	0.27036 (9)	0.0464 (7)
H18	0.0696	0.2652	0.2921	0.056*
C19	0.5265 (4)	0.3225 (4)	0.25426 (9)	0.0456 (6)
H19	0.6610	0.3173	0.2654	0.055*
C20	0.1291 (4)	0.3409 (4)	0.22136 (9)	0.0434 (6)
H20	−0.0057	0.3487	0.2106	0.052*
C21	0.4796 (4)	0.3666 (4)	0.20467 (9)	0.0401 (6)
C22	0.2810 (4)	0.3781 (3)	0.18742 (9)	0.0361 (5)
C23	0.2321 (4)	0.4413 (3)	0.13512 (9)	0.0370 (5)
H23	0.1476	0.5459	0.1374	0.044*
C24	0.5951 (4)	0.3719 (5)	0.12170 (9)	0.0475 (7)
H24A	0.5624	0.2500	0.1162	0.057*
H24B	0.7131	0.3998	0.1021	0.057*
C25	0.4173 (4)	0.4844 (4)	0.10406 (9)	0.0383 (6)
H25	0.4508	0.6087	0.1062	0.046*
C26	0.3451 (3)	0.4357 (3)	0.05191 (9)	0.0352 (5)
C27	0.1733 (4)	0.3339 (3)	0.05624 (9)	0.0370 (5)
C28	0.0649 (4)	0.2655 (4)	0.01535 (9)	0.0434 (6)
H28	−0.0514	0.1985	0.0187	0.052*
C29	0.4257 (4)	0.4705 (4)	0.00480 (9)	0.0388 (6)
H29	0.5434	0.5355	0.0011	0.047*
C30	0.3202 (4)	0.4027 (3)	−0.03548 (9)	0.0377 (6)
C31	0.1455 (4)	0.3060 (3)	−0.03058 (9)	0.0399 (6)
C32	0.1876 (4)	0.3583 (5)	−0.11197 (10)	0.0562 (8)
H32A	0.2212	0.2887	−0.1411	0.067*
H32B	0.1096	0.4586	−0.1234	0.067*
O6	0.4273 (3)	0.2438 (3)	0.33548 (7)	0.0539 (5)
H6	0.3249	0.2244	0.3515	0.081*
O7	0.6410 (2)	0.3999 (3)	0.17416 (6)	0.0542 (6)
O8	0.1194 (2)	0.3076 (3)	0.10559 (6)	0.0429 (4)
O9	0.0730 (3)	0.2572 (3)	−0.07755 (7)	0.0556 (5)
O10	0.3679 (3)	0.4137 (3)	−0.08601 (6)	0.0501 (5)

C1	0.1839 (4)	0.2268 (3)	0.45496 (9)	0.0363 (5)
C2	0.3757 (4)	0.2741 (4)	0.47033 (9)	0.0381 (6)
H2	0.4652	0.3213	0.4475	0.046*
C3	0.0490 (4)	0.1544 (4)	0.48846 (9)	0.0400 (6)
H3	−0.0814	0.1235	0.4779	0.048*
C4	0.1122 (4)	0.1293 (4)	0.53751 (9)	0.0392 (6)
H4	0.0234	0.0781	0.5598	0.047*
C5	0.3043 (3)	0.1777 (3)	0.55505 (8)	0.0334 (5)
C6	0.4352 (3)	0.2507 (3)	0.52047 (9)	0.0350 (5)
C7	0.6668 (4)	0.3281 (4)	0.58512 (9)	0.0397 (6)
H7A	0.5928	0.4313	0.5951	0.048*
H7B	0.8100	0.3502	0.5911	0.048*
C8	0.3761 (4)	0.1382 (3)	0.60767 (9)	0.0364 (5)
H8	0.3492	0.0152	0.6149	0.044*
C9	0.6023 (3)	0.1750 (3)	0.61713 (9)	0.0360 (5)
H9	0.6852	0.0715	0.6110	0.043*
C10	0.6054 (4)	0.2242 (3)	0.67194 (9)	0.0382 (6)
C11	0.4108 (4)	0.2677 (4)	0.68482 (9)	0.0395 (6)
C12	0.3581 (4)	0.3335 (4)	0.73156 (9)	0.0478 (7)
H12	0.2263	0.3636	0.7397	0.057*
C13	0.7658 (4)	0.2426 (4)	0.70657 (9)	0.0438 (6)
H13	0.8983	0.2141	0.6987	0.053*
C14	0.7161 (4)	0.3051 (4)	0.75271 (9)	0.0432 (6)
C15	0.5217 (4)	0.3496 (4)	0.76451 (9)	0.0464 (6)
C16	0.7197 (4)	0.4165 (4)	0.83025 (10)	0.0508 (7)
H16A	0.7354	0.3608	0.8629	0.061*
H16B	0.7606	0.5375	0.8336	0.061*
O1	0.1322 (3)	0.2541 (3)	0.40500 (6)	0.0450 (4)
H1	0.0151	0.2224	0.3996	0.068*
O2	0.6306 (2)	0.2972 (3)	0.53267 (6)	0.0446 (5)
O3	0.2721 (2)	0.2476 (3)	0.64510 (6)	0.0418 (4)
O4	0.8427 (3)	0.3305 (3)	0.79441 (7)	0.0614 (6)
O5	0.5157 (3)	0.4066 (4)	0.81333 (7)	0.0673 (7)
O1W	0.7950 (3)	0.1124 (3)	0.37226 (9)	0.0735 (7)
H1WA	0.6647	0.1553	0.3623	0.088*
H1WB	0.8030	−0.0173	0.3708	0.088*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C17	0.0395 (13)	0.0501 (16)	0.0333 (13)	0.0042 (12)	−0.0039 (10)	−0.0020 (11)
C18	0.0379 (14)	0.0651 (19)	0.0365 (13)	−0.0010 (13)	0.0062 (10)	−0.0003 (13)
C19	0.0313 (12)	0.0654 (18)	0.0398 (14)	0.0044 (13)	−0.0043 (10)	−0.0019 (13)
C20	0.0310 (12)	0.0611 (17)	0.0380 (14)	0.0038 (13)	−0.0022 (10)	−0.0026 (13)
C21	0.0308 (12)	0.0531 (16)	0.0365 (13)	0.0007 (12)	0.0009 (9)	−0.0018 (12)
C22	0.0325 (12)	0.0424 (14)	0.0334 (13)	0.0041 (11)	−0.0014 (9)	−0.0038 (11)
C23	0.0341 (12)	0.0413 (14)	0.0354 (13)	0.0038 (11)	−0.0027 (9)	−0.0017 (11)
C24	0.0333 (13)	0.075 (2)	0.0345 (13)	0.0004 (14)	0.0019 (10)	0.0012 (14)

C25	0.0359 (12)	0.0418 (14)	0.0371 (13)	−0.0049 (11)	−0.0028 (10)	−0.0033 (11)
C26	0.0327 (12)	0.0364 (13)	0.0363 (13)	−0.0030 (10)	−0.0023 (9)	0.0016 (10)
C27	0.0365 (12)	0.0401 (13)	0.0344 (12)	−0.0009 (11)	0.0003 (9)	0.0021 (11)
C28	0.0389 (13)	0.0497 (15)	0.0414 (14)	−0.0125 (13)	−0.0028 (10)	0.0008 (12)
C29	0.0351 (13)	0.0416 (14)	0.0398 (14)	−0.0069 (11)	0.0003 (10)	0.0014 (11)
C30	0.0370 (13)	0.0423 (14)	0.0337 (12)	0.0024 (11)	0.0007 (10)	0.0014 (11)
C31	0.0384 (13)	0.0441 (15)	0.0369 (13)	−0.0028 (12)	−0.0064 (10)	−0.0044 (12)
C32	0.0521 (16)	0.079 (2)	0.0375 (14)	−0.0138 (17)	−0.0072 (11)	−0.0003 (15)
O6	0.0437 (10)	0.0819 (15)	0.0359 (10)	0.0068 (10)	−0.0034 (7)	0.0060 (10)
O7	0.0291 (9)	0.0950 (17)	0.0385 (10)	−0.0041 (10)	−0.0018 (7)	0.0036 (11)
O8	0.0381 (9)	0.0558 (11)	0.0346 (9)	−0.0122 (8)	−0.0012 (7)	0.0028 (8)
O9	0.0567 (11)	0.0725 (14)	0.0373 (10)	−0.0221 (11)	−0.0049 (8)	−0.0043 (10)
O10	0.0496 (10)	0.0688 (14)	0.0318 (9)	−0.0112 (10)	0.0004 (7)	−0.0012 (9)
C1	0.0390 (13)	0.0385 (13)	0.0314 (12)	0.0027 (11)	0.0023 (9)	−0.0037 (10)
C2	0.0361 (12)	0.0443 (14)	0.0342 (13)	−0.0047 (11)	0.0061 (10)	0.0010 (11)
C3	0.0298 (13)	0.0512 (15)	0.0392 (14)	−0.0037 (11)	0.0029 (9)	−0.0020 (12)
C4	0.0347 (13)	0.0455 (14)	0.0375 (14)	−0.0063 (11)	0.0081 (10)	0.0005 (11)
C5	0.0308 (12)	0.0351 (12)	0.0345 (13)	−0.0027 (10)	0.0023 (9)	−0.0023 (10)
C6	0.0323 (12)	0.0365 (12)	0.0365 (12)	−0.0025 (10)	0.0041 (9)	−0.0034 (11)
C7	0.0336 (12)	0.0484 (14)	0.0372 (13)	−0.0063 (11)	−0.0010 (10)	−0.0049 (12)
C8	0.0387 (13)	0.0362 (13)	0.0345 (13)	−0.0031 (11)	0.0047 (10)	0.0004 (11)
C9	0.0331 (12)	0.0392 (13)	0.0357 (13)	0.0018 (10)	0.0004 (9)	−0.0023 (11)
C10	0.0350 (13)	0.0422 (14)	0.0373 (13)	0.0015 (11)	0.0008 (10)	0.0026 (11)
C11	0.0358 (13)	0.0493 (15)	0.0331 (12)	−0.0022 (12)	−0.0026 (9)	0.0014 (11)
C12	0.0336 (13)	0.0734 (19)	0.0363 (14)	0.0001 (14)	0.0032 (10)	−0.0014 (14)
C13	0.0360 (13)	0.0508 (15)	0.0442 (15)	0.0030 (12)	−0.0043 (10)	0.0006 (13)
C14	0.0389 (14)	0.0509 (16)	0.0394 (14)	−0.0033 (12)	−0.0079 (10)	0.0038 (12)
C15	0.0473 (15)	0.0604 (18)	0.0315 (13)	−0.0057 (14)	−0.0002 (10)	0.0025 (13)
C16	0.0572 (17)	0.0540 (17)	0.0406 (15)	−0.0056 (14)	−0.0083 (12)	0.0011 (13)
O1	0.0415 (9)	0.0591 (12)	0.0342 (9)	−0.0048 (9)	−0.0024 (7)	0.0015 (9)
O2	0.0337 (9)	0.0633 (12)	0.0370 (9)	−0.0131 (8)	0.0031 (7)	−0.0005 (8)
O3	0.0323 (9)	0.0607 (11)	0.0323 (9)	0.0013 (8)	0.0010 (6)	−0.0044 (9)
O4	0.0499 (11)	0.0897 (17)	0.0441 (11)	0.0017 (12)	−0.0129 (8)	−0.0078 (12)
O5	0.0530 (12)	0.116 (2)	0.0332 (10)	−0.0019 (13)	−0.0030 (8)	−0.0123 (12)
O1W	0.0548 (13)	0.0650 (14)	0.0989 (18)	−0.0079 (12)	−0.0338 (12)	0.0022 (13)

Geometric parameters (Å, °)

C17—O6	1.372 (3)	C1—O1	1.378 (3)
C17—C19	1.372 (4)	C1—C3	1.389 (3)
C17—C18	1.384 (3)	C2—C6	1.391 (3)
C18—C20	1.377 (4)	C2—H2	0.9300
C18—H18	0.9300	C3—C4	1.372 (3)
C19—C21	1.388 (3)	C3—H3	0.9300
C19—H19	0.9300	C4—C5	1.391 (3)
C20—C22	1.393 (3)	C4—H4	0.9300
C20—H20	0.9300	C5—C6	1.392 (3)
C21—O7	1.377 (3)	C5—C8	1.496 (3)
C21—C22	1.382 (3)	C6—O2	1.369 (3)

C22—C23	1.498 (3)	C7—O2	1.427 (3)
C23—O8	1.481 (3)	C7—C9	1.516 (4)
C23—C25	1.526 (3)	C7—H7A	0.9700
C23—H23	0.9800	C7—H7B	0.9700
C24—O7	1.435 (3)	C8—O3	1.481 (3)
C24—C25	1.522 (4)	C8—C9	1.534 (3)
C24—H24A	0.9700	C8—H8	0.9800
C24—H24B	0.9700	C9—C10	1.503 (3)
C25—C26	1.501 (3)	C9—H9	0.9800
C25—H25	0.9800	C10—C11	1.378 (4)
C26—C27	1.383 (3)	C10—C13	1.393 (3)
C26—C29	1.396 (3)	C11—O3	1.389 (3)
C27—O8	1.380 (3)	C11—C12	1.391 (4)
C27—C28	1.389 (3)	C12—C15	1.379 (3)
C28—C31	1.377 (4)	C12—H12	0.9300
C28—H28	0.9300	C13—C14	1.362 (4)
C29—C30	1.365 (3)	C13—H13	0.9300
C29—H29	0.9300	C14—C15	1.371 (4)
C30—C31	1.380 (4)	C14—O4	1.385 (3)
C30—O10	1.388 (3)	C15—O5	1.369 (3)
C31—O9	1.377 (3)	C16—O5	1.411 (3)
C32—O10	1.426 (3)	C16—O4	1.426 (4)
C32—O9	1.428 (3)	C16—H16A	0.9700
C32—H32A	0.9700	C16—H16B	0.9700
C32—H32B	0.9700	O1—H1	0.8200
O6—H6	0.8200	O1W—H1WA	0.9528
C1—C2	1.370 (3)	O1W—H1WB	0.9977
O6—C17—C19	117.6 (2)	C2—C1—C3	121.1 (2)
O6—C17—C18	122.0 (2)	O1—C1—C3	122.0 (2)
C19—C17—C18	120.4 (2)	C1—C2—C6	119.3 (2)
C20—C18—C17	119.2 (2)	C1—C2—H2	120.3
C20—C18—H18	120.4	C6—C2—H2	120.3
C17—C18—H18	120.4	C4—C3—C1	118.6 (2)
C17—C19—C21	119.7 (2)	C4—C3—H3	120.7
C17—C19—H19	120.1	C1—C3—H3	120.7
C21—C19—H19	120.1	C3—C4—C5	122.4 (2)
C18—C20—C22	121.8 (2)	C3—C4—H4	118.8
C18—C20—H20	119.1	C5—C4—H4	118.8
C22—C20—H20	119.1	C4—C5—C6	117.5 (2)
O7—C21—C22	122.3 (2)	C4—C5—C8	121.7 (2)
O7—C21—C19	116.4 (2)	C6—C5—C8	120.6 (2)
C22—C21—C19	121.3 (2)	O2—C6—C2	115.9 (2)
C21—C22—C20	117.7 (2)	O2—C6—C5	122.9 (2)
C21—C22—C23	120.7 (2)	C2—C6—C5	121.1 (2)
C20—C22—C23	121.5 (2)	O2—C7—C9	112.0 (2)
O8—C23—C22	111.2 (2)	O2—C7—H7A	109.2
O8—C23—C25	105.23 (18)	C9—C7—H7A	109.2
C22—C23—C25	114.38 (19)	O2—C7—H7B	109.2
O8—C23—H23	108.6	C9—C7—H7B	109.2

C22—C23—H23	108.6	H7A—C7—H7B	107.9
C25—C23—H23	108.6	O3—C8—C5	111.7 (2)
O7—C24—C25	111.0 (2)	O3—C8—C9	104.47 (18)
O7—C24—H24A	109.4	C5—C8—C9	113.68 (19)
C25—C24—H24A	109.4	O3—C8—H8	108.9
O7—C24—H24B	109.4	C5—C8—H8	108.9
C25—C24—H24B	109.4	C9—C8—H8	108.9
H24A—C24—H24B	108.0	C10—C9—C7	110.5 (2)
C26—C25—C24	111.6 (2)	C10—C9—C8	101.33 (18)
C26—C25—C23	101.73 (19)	C7—C9—C8	109.6 (2)
C24—C25—C23	109.4 (2)	C10—C9—H9	111.7
C26—C25—H25	111.2	C7—C9—H9	111.7
C24—C25—H25	111.2	C8—C9—H9	111.7
C23—C25—H25	111.2	C11—C10—C13	120.7 (2)
C27—C26—C29	120.8 (2)	C11—C10—C9	108.0 (2)
C27—C26—C25	107.9 (2)	C13—C10—C9	131.1 (2)
C29—C26—C25	131.2 (2)	C10—C11—O3	112.7 (2)
O8—C27—C26	113.0 (2)	C10—C11—C12	124.3 (2)
O8—C27—C28	123.2 (2)	O3—C11—C12	122.9 (2)
C26—C27—C28	123.8 (2)	C15—C12—C11	112.9 (2)
C31—C28—C27	113.9 (2)	C15—C12—H12	123.5
C31—C28—H28	123.1	C11—C12—H12	123.5
C27—C28—H28	123.1	C14—C13—C10	115.7 (2)
C30—C29—C26	115.5 (2)	C14—C13—H13	122.1
C30—C29—H29	122.2	C10—C13—H13	122.1
C26—C29—H29	122.2	C13—C14—C15	122.6 (2)
C29—C30—C31	122.9 (2)	C13—C14—O4	127.9 (2)
C29—C30—O10	127.7 (2)	C15—C14—O4	109.4 (2)
C31—C30—O10	109.4 (2)	O5—C15—C14	110.1 (2)
C28—C31—O9	127.4 (2)	O5—C15—C12	126.1 (2)
C28—C31—C30	123.0 (2)	C14—C15—C12	123.8 (2)
O9—C31—C30	109.5 (2)	O5—C16—O4	108.4 (2)
O10—C32—O9	107.5 (2)	O5—C16—H16A	110.0
O10—C32—H32A	110.2	O4—C16—H16A	110.0
O9—C32—H32A	110.2	O5—C16—H16B	110.0
O10—C32—H32B	110.2	O4—C16—H16B	110.0
O9—C32—H32B	110.2	H16A—C16—H16B	108.4
H32A—C32—H32B	108.5	C1—O1—H1	109.5
C17—O6—H6	109.5	C6—O2—C7	114.17 (18)
C21—O7—C24	113.23 (18)	C11—O3—C8	105.37 (18)
C27—O8—C23	105.31 (18)	C14—O4—C16	104.9 (2)
C31—O9—C32	104.7 (2)	C15—O5—C16	105.7 (2)
C30—O10—C32	104.10 (19)	H1WA—O1W—H1WB	112.4
C2—C1—O1	116.9 (2)		
O6—C17—C18—C20	178.4 (3)	O1—C1—C2—C6	179.5 (2)
C19—C17—C18—C20	−1.9 (4)	C3—C1—C2—C6	−0.7 (4)
O6—C17—C19—C21	−179.9 (3)	C2—C1—C3—C4	−0.5 (4)
C18—C17—C19—C21	0.4 (4)	O1—C1—C3—C4	179.2 (2)
C17—C18—C20—C22	2.0 (4)	C1—C3—C4—C5	1.6 (4)

C17—C19—C21—O7	−179.6 (3)	C3—C4—C5—C6	−1.3 (4)
C17—C19—C21—C22	1.1 (4)	C3—C4—C5—C8	−175.6 (3)
O7—C21—C22—C20	179.7 (3)	C1—C2—C6—O2	178.5 (2)
C19—C21—C22—C20	−1.0 (4)	C1—C2—C6—C5	1.0 (4)
O7—C21—C22—C23	−4.7 (4)	C4—C5—C6—O2	−177.3 (2)
C19—C21—C22—C23	174.5 (3)	C8—C5—C6—O2	−3.0 (4)
C18—C20—C22—C21	−0.5 (4)	C4—C5—C6—C2	0.0 (4)
C18—C20—C22—C23	−176.0 (3)	C8—C5—C6—C2	174.4 (2)
C21—C22—C23—O8	120.6 (2)	C4—C5—C8—O3	−71.5 (3)
C20—C22—C23—O8	−64.0 (3)	C6—C5—C8—O3	114.4 (2)
C21—C22—C23—C25	1.6 (3)	C4—C5—C8—C9	170.6 (2)
C20—C22—C23—C25	177.0 (2)	C6—C5—C8—C9	−3.6 (3)
O7—C24—C25—C26	−170.5 (2)	O2—C7—C9—C10	−169.33 (19)
O7—C24—C25—C23	−58.7 (3)	O2—C7—C9—C8	−58.5 (3)
O8—C23—C25—C26	24.6 (2)	O3—C8—C9—C10	27.2 (2)
C22—C23—C25—C26	146.9 (2)	C5—C8—C9—C10	149.2 (2)
O8—C23—C25—C24	−93.6 (2)	O3—C8—C9—C7	−89.6 (2)
C22—C23—C25—C24	28.7 (3)	C5—C8—C9—C7	32.4 (3)
C24—C25—C26—C27	101.0 (3)	C7—C9—C10—C11	97.6 (3)
C23—C25—C26—C27	−15.6 (3)	C8—C9—C10—C11	−18.5 (3)
C24—C25—C26—C29	−76.3 (3)	C7—C9—C10—C13	−77.1 (4)
C23—C25—C26—C29	167.1 (3)	C8—C9—C10—C13	166.8 (3)
C29—C26—C27—O8	177.7 (2)	C13—C10—C11—O3	177.6 (2)
C25—C26—C27—O8	0.1 (3)	C9—C10—C11—O3	2.2 (3)
C29—C26—C27—C28	−2.6 (4)	C13—C10—C11—C12	0.8 (4)
C25—C26—C27—C28	179.8 (2)	C9—C10—C11—C12	−174.6 (3)
O8—C27—C28—C31	−179.5 (2)	C10—C11—C12—C15	−0.7 (4)
C26—C27—C28—C31	0.9 (4)	O3—C11—C12—C15	−177.2 (3)
C27—C26—C29—C30	2.0 (4)	C11—C10—C13—C14	0.0 (4)
C25—C26—C29—C30	179.0 (3)	C9—C10—C13—C14	174.1 (3)
C26—C29—C30—C31	0.0 (4)	C10—C13—C14—C15	−0.8 (4)
C26—C29—C30—O10	−178.5 (2)	C10—C13—C14—O4	177.6 (3)
C27—C28—C31—O9	179.9 (3)	C13—C14—C15—O5	179.4 (3)
C27—C28—C31—C30	1.3 (4)	O4—C14—C15—O5	0.7 (4)
C29—C30—C31—C28	−1.8 (4)	C13—C14—C15—C12	1.0 (5)
O10—C30—C31—C28	177.0 (2)	O4—C14—C15—C12	−177.7 (3)
C29—C30—C31—O9	179.4 (2)	C11—C12—C15—O5	−178.4 (3)
O10—C30—C31—O9	−1.8 (3)	C11—C12—C15—C14	−0.2 (5)
C22—C21—O7—C24	−25.6 (4)	C2—C6—O2—C7	159.9 (2)
C19—C21—O7—C24	155.1 (3)	C5—C6—O2—C7	−22.6 (3)
C25—C24—O7—C21	57.8 (3)	C9—C7—O2—C6	54.0 (3)
C26—C27—O8—C23	16.2 (3)	C10—C11—O3—C8	16.0 (3)
C28—C27—O8—C23	−163.5 (2)	C12—C11—O3—C8	−167.2 (3)
C22—C23—O8—C27	−149.7 (2)	C5—C8—O3—C11	−150.2 (2)
C25—C23—O8—C27	−25.3 (2)	C9—C8—O3—C11	−26.9 (2)
C28—C31—O9—C32	169.6 (3)	C13—C14—O4—C16	173.4 (3)
C30—C31—O9—C32	−11.6 (3)	C15—C14—O4—C16	−8.0 (3)
O10—C32—O9—C31	20.6 (3)	O5—C16—O4—C14	12.2 (3)
C29—C30—O10—C32	−166.9 (3)	C14—C15—O5—C16	7.0 (4)

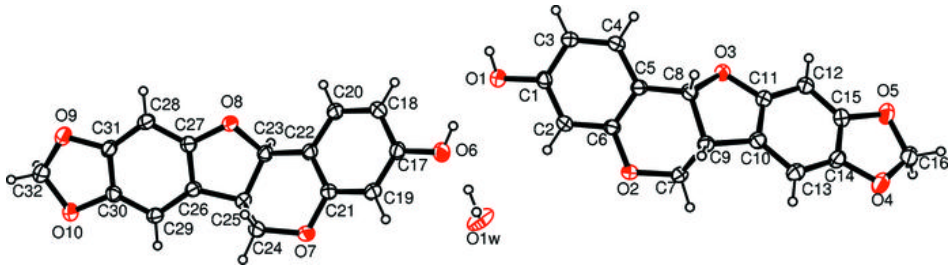
C31—C30—O10—C32	14.5 (3)	C12—C15—O5—C16	−174.6 (3)
O9—C32—O10—C30	−21.6 (3)	O4—C16—O5—C15	−11.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O1	0.82	1.94	2.714 (3)	157
O1W—H1WA···O6	0.95	1.84	2.784 (3)	173
O1—H1···O1W^{i}	0.82	1.82	2.608 (3)	162
O1W—H1WB···O3^{ii}	1.00	1.91	2.870 (3)	159
C16—H16B···Cg2^{iii}	0.97	2.99	3.663 (4)	128
C28—H28···Cg3^{iv}	0.93	2.93	3.722 (3)	144
C29—H29···Cg3^{v}	0.93	2.93	3.776 (3)	151

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

Fig. 1


O9
C31
C28
C27
O8
C23
C22
C20
C18
O6
C17
C19
C21
C32
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C30
C29
C26
C25
C24
O7
O1w
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C4
C5
C8
O3
C11
C12
C15
O5
C1
C2
C6
C9
C10
C14
C16
O2
C7
C13
O4