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7-Hydroxy-3,6,9-trimethyl-2,3,5,6-tetrahydronaphtho[1,8-*b*,*c*]pyran-4,8-dione

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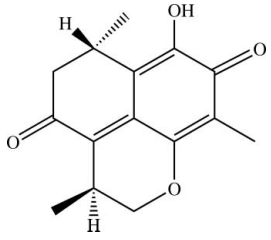
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.113; data-to-parameter ratio = 10.3.

The title sesquiterpenoid quinone compound, $C_{15}H_{16}O_4$, was isolated from *Thespesia populnea*. There are two independent molecules (*A* and *B*) with identical conformations in the asymmetric unit. In both molecules, the dihydropyran rings adopt envelope conformations, with the methylene C as the flap atom, whereas the cyclohenene rings are in screw-boat conformations. Intramolecular O—H···O hydrogen bonds generate *S*(5) ring motifs in both molecules. The molecules are linked into chains along the *a* axis through weak C—H···O intermolecular interactions. The crystal structure is stabilized by intramolecular O—H···O hydrogen bonds, and weak C—H···O intra- and intermolecular interactions. C—H···π interactions involving the cyclohexadiene ring are observed in the crystal structure.

Related literature

For details of the sources and biological activities of related sesquiterpenes, see Tiew *et al.* (2002); Duh *et al.* (2004); Wang *et al.* (2004); Silva *et al.* (2006). For related literature on hydrogen-bond motifs, see Bernstein *et al.* (1995), and on values of bond lengths and angles, see Allen *et al.* (1987). For a related structure, see Fun *et al.* (2007). For related literature, see: Cremer & Pople (1975); Milbrodt *et al.* (1997).



Experimental

Crystal data

$C_{15}H_{16}O_4$
M_r = 260.28
Orthorhombic, $P2_12_12_1$
a = 8.5390 (4) Å
b = 10.0913 (5) Å
c = 30.3769 (14) Å
V = 2617.6 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100.0 (1) K
0.51 × 0.19 × 0.11 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.953, T_{max} = 0.990
28942 measured reflections
3593 independent reflections
3004 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.02
3593 reflections
349 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3*A*—H3*AA*···O2*A*	0.82	2.28	2.703 (2)	112
O3*A*—H3*AA*···O2*B*	0.82	1.98	2.760 (2)	159
O3*B*—H3*BA*···O2*A*	0.82	2.05	2.829 (2)	158
O3*B*—H3*BA*···O2*B*	0.82	2.24	2.693 (3)	115
C7*A*—H7*AA*···O3*A*	0.98	2.51	2.865 (3)	101
C7*B*—H7*BA*···O3*B*	0.98	2.50	2.854 (3)	101
C12*A*—H12*A*···O4*B*^{i}	0.97	2.50	3.452 (3)	168
C12*B*—H12*D*···O4*B*^{ii}	0.97	2.44	3.281 (3)	145
C14*A*—H14*A*···O1*A*	0.96	2.43	2.853 (3)	106
C12*A*—H12*B*···*Cg*1^{iii}	0.97	2.78	3.657 (3)	151
C13*A*—H13*B*···*Cg*1^{iv}	0.97	2.67	3.387 (3)	132

Symmetry codes: (i) $-x+\frac{3}{2}, -y+2, z-\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{5}{2}, -z+1$; (iii) $x+\frac{3}{2}, -y-\frac{1}{2}, -z$; (iv) $x+\frac{5}{2}, -y-\frac{1}{2}, -z$. *Cg*1 is the centroid of C1*B*–C6*B*.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2300).

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supplementary materials

Acta Cryst. (2007). E**63**, o2749-o2750 [**doi:10.1107/S1600536807020193**]

7-Hydroxy-3,6,9-trimethyl-2,3,5,6-tetrahydronaphtho[1,8-*b*,*c*]pyran-4,8-dione

S. Chantrapromma, S. Boonsri, H.-K. Fun and C. Karalai

Comment

The heartwood of *Thespesia populnea* is a rich source of highly oxidized sesquiterpenes containing a cadinane skeleton (Milbrodt *et al.*, 1997). Some possess significant pharmacological effects such as cytotoxicity (Tiew *et al.*, 2002; Duh *et al.*, 2004; Wang *et al.*, 2004) and antifungal activity (Silva *et al.*, 2006). Previously we reported the stucture of mansonone E, a sesquiterpene isolated from *T. populnea* (Fun *et al.*, 2007). In continuation of our study of bioactive compounds from *T. populnea*, (Po-ta-lea in Thai) a plant in the *malvaceae*, we report the structure of the title compound, (I) isolated from the heartwood of *T. populnea* collected from the Suratthani province in Thailand. Biological activity tests show that (I) is inactive against bacteria and shows an IC_{50} > 5 µg/ml) against MCF-7 (breast), Hela (cervical), HT-29 (colon) and KB (oral cavity) cancer cell lines.

Compound (I) crystallizes with two conformationally similar independent molecules (A and B) per asymmetric unit (Fig. 1). The bond lengths and angles in (I) are normal (Allen *et al.*, 1987) and comparable to those in a related structure (Fun *et al.*, 2007). In both molecules, the cyclohexadiene rings (C1—C6) are essentially planar with maximum deviations of -0.037 (3) for C1A and 0.036 (3) Å for atom C4B. The dihydropyran rings adopt envelope conformations, with atom C12 displaced from the C1/C2/C10/C11/O1 plane by -0.340 (3)Å and -0.315 (3) Å for A and B, respectively. The puckering parameters (Cremer & Pople, 1975) are Q = 0.473 (3) Å, θ = 122.7 (3)° and φ = 121.6 (3)° for A and Q = 0.439 (2) Å, θ = 124.0 (3)° and φ = 119.3 (4)° for B. Both the cycloxene rings adopt screw boat conformations with puckering parameters Q = 0.434 (3) Å, θ = 57.4 (4)° and φ = 150.4 (4)° for A and Q = 0.416 (2) Å, θ = 54.1 (4)° and φ = 154.9 (4)° for B. In both molecules, the methyl group at C3 lies in the cyclohexadiene ring plane whereas the C7 and C11 methyl groups are axial to the cyclohexene and dihydropyran rings (Fig. 1).

In the crystal intramolecular O3A—H3AA···O2A and O3B—H3BA···O2B hydrogen bonds generate S(5) ring motifs with S(10) motifs formed by O3A—H3AA···O2B and O3B—H3BA···O2A interactions (Bernstein *et al.*, 1995). These link the two molecules into dimers which form chains along a through weak intermolecular C—H···O interactions (Fig. 2, Table 1). The crystal is further stabilized by C—H···π interactions; Cg_1 is the centroid of C1B–C6B (Table 1).

Experimental

Air-dried heartwood of *T. populnea* (2.1 kg) was extracted with CH_2Cl_2 over a period of 5 d at room temperature. The CH_2Cl_2 extract was evaporated under reduced pressure to yield a orange-brown gum (37.5 g), which was subjected to silica gel column chromatography using CH_2Cl_2 as eluent to afford 8 fractions (F1—F8). Fraction F7 was subjected to repeated column chromatography with acetone-CH_2Cl_2 as eluents for gradient elution to afford the title compound (I). Purple needle-shaped single crystals of (I) were obtained by recrystallization from MeOH-CH_2Cl_2 (3:7 v/v) after several days (m.p. 532–534 K).

Refinement

In the absence of significant anomalous scattering effects, 2730 Friedel pairs were averaged. All H atoms were positioned geometrically and allowed to ride on their parent atoms, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.96 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

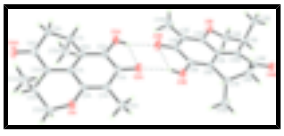


Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen bonds are shown as dashed lines.

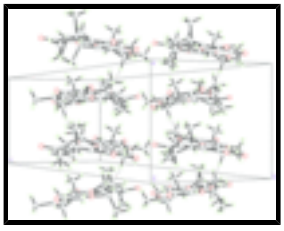


Fig. 2. The crystal packing of (I). Hydrogen bonds are shown as dashed lines.

7-Hydroxy-3,6,9-trimethyl-2,3,5,6-tetrahydronaphtho[1,8-b,c]pyran-4,8-dione

Crystal data

$C_{15}H_{16}O_4$	$D_x = 1.321$ Mg m^{-3}
$M_r = 260.28$	Melting point: 532-534 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 3593 reflections
$a = 8.5390$ (4) Å	$\theta = 1.3$–28.0º
$b = 10.0913$ (5) Å	$\mu = 0.10$ mm^{-1}
$c = 30.3769$ (14) Å	$T = 100.0$ (1) K
$V = 2617.6$ (2) Å^3	Needle, purple
$Z = 8$	0.51 × 0.19 × 0.11 mm
$F_{000} = 1104$	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	3593 independent reflections
Radiation source: fine-focus sealed tube	3004 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.067$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 28.0$º
$T = 297$(3) K	$\theta_{min} = 1.3$º
ω scans	$h = -11 \rightarrow 11$

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.953, T_{max} = 0.990

28942 measured reflections

$k = -12 \rightarrow 13$

$l = -39 \rightarrow 40$

Refinement

Refinement on F^2

Least-squares matrix: full

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

$wR(F^2)$ = 0.113

S = 1.03

3593 reflections

349 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.359P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.30 e Å^{-3}

$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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. 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}
O1A	0.7493 (2)	0.62788 (16)	0.10729 (5)	0.0247 (4)
O2A	0.8398 (2)	0.77230 (17)	0.25156 (6)	0.0279 (4)
O3A	0.7968 (2)	0.52747 (17)	0.28561 (5)	0.0277 (4)
H3AA	0.7890	0.5996	0.2979	0.042*
O4A	0.7326 (3)	0.13687 (19)	0.12509 (6)	0.0436 (6)
C1A	0.7573 (3)	0.4667 (2)	0.16700 (7)	0.0194 (5)
C2A	0.7636 (3)	0.6047 (2)	0.15122 (7)	0.0198 (5)
C3A	0.7918 (3)	0.7077 (2)	0.17842 (8)	0.0214 (5)
C4A	0.8115 (3)	0.6818 (2)	0.22519 (8)	0.0212 (5)
C5A	0.7907 (3)	0.5448 (2)	0.24131 (7)	0.0217 (5)
C6A	0.7665 (3)	0.4418 (2)	0.21395 (7)	0.0201 (5)

C7A	0.7381 (3)	0.3024 (2)	0.23054 (8)	0.0244 (5)
H7AA	0.7908	0.2922	0.2590	0.029*
C8A	0.8087 (3)	0.2023 (3)	0.19815 (8)	0.0296 (6)
H8AA	0.9220	0.2079	0.1995	0.035*
H8AB	0.7787	0.1135	0.2070	0.035*
C9A	0.7560 (3)	0.2260 (3)	0.15131 (8)	0.0286 (6)
C10A	0.7422 (3)	0.3664 (2)	0.13727 (8)	0.0207 (5)
C11A	0.7211 (3)	0.3933 (2)	0.08885 (8)	0.0228 (5)
H11A	0.6446	0.3301	0.0771	0.027*
C12A	0.6554 (3)	0.5315 (3)	0.08381 (8)	0.0262 (5)
H12A	0.6522	0.5547	0.0528	0.031*
H12B	0.5490	0.5335	0.0950	0.031*
C13A	0.8749 (3)	0.3761 (3)	0.06367 (8)	0.0304 (6)
H13A	0.9186	0.2906	0.0701	0.046*
H13B	0.9473	0.4439	0.0725	0.046*
H13C	0.8553	0.3830	0.0326	0.046*
C14A	0.7978 (3)	0.8499 (2)	0.16384 (8)	0.0265 (5)
H14A	0.7826	0.8545	0.1326	0.040*
H14B	0.8979	0.8870	0.1712	0.040*
H14C	0.7166	0.8991	0.1784	0.040*
C15A	0.5626 (3)	0.2783 (3)	0.23716 (9)	0.0338 (6)
H15A	0.5210	0.3443	0.2567	0.051*
H15B	0.5468	0.1919	0.2497	0.051*
H15C	0.5100	0.2835	0.2093	0.051*
O1B	0.7010 (2)	0.87759 (16)	0.49008 (5)	0.0273 (4)
O2B	0.7770 (3)	0.73126 (18)	0.34594 (6)	0.0386 (5)
O3B	0.8073 (2)	0.97874 (18)	0.31392 (5)	0.0291 (4)
H3BA	0.8132	0.9062	0.3019	0.044*
O4B	0.7987 (2)	1.36536 (17)	0.47624 (6)	0.0274 (4)
C1B	0.7519 (3)	1.0400 (2)	0.43210 (7)	0.0189 (5)
C2B	0.7216 (3)	0.9027 (2)	0.44669 (8)	0.0218 (5)
C3B	0.7241 (3)	0.7987 (2)	0.41856 (8)	0.0254 (5)
C4B	0.7622 (3)	0.8228 (2)	0.37285 (8)	0.0258 (6)
C5B	0.7823 (3)	0.9617 (2)	0.35765 (7)	0.0226 (5)
C6B	0.7778 (3)	1.0649 (2)	0.38527 (8)	0.0194 (5)
C7B	0.7933 (3)	1.2061 (2)	0.36968 (8)	0.0217 (5)
H7BA	0.8575	1.2062	0.3429	0.026*
C8B	0.8782 (3)	1.2897 (2)	0.40482 (7)	0.0237 (5)
H8BA	0.9876	1.2640	0.4057	0.028*
H8BB	0.8733	1.3824	0.3965	0.028*
C9B	0.8086 (3)	1.2736 (2)	0.44997 (8)	0.0219 (5)
C10B	0.7607 (3)	1.1374 (2)	0.46290 (8)	0.0200 (5)
C11B	0.7364 (3)	1.1094 (2)	0.51100 (8)	0.0219 (5)
H11B	0.6778	1.1837	0.5237	0.026*
C12B	0.6393 (3)	0.9857 (2)	0.51640 (8)	0.0260 (5)
H12C	0.6384	0.9598	0.5472	0.031*
H12D	0.5323	1.0038	0.5076	0.031*
C13B	0.8948 (3)	1.1007 (3)	0.53461 (8)	0.0304 (6)
H13D	0.9493	1.1834	0.5316	0.046*

H13E	0.9562	1.0311	0.5217	0.046*
H13F	0.8780	1.0821	0.5652	0.046*
C14B	0.6971 (5)	0.6582 (3)	0.43310 (9)	0.0420 (8)
H14D	0.6267	0.6574	0.4577	0.063*
H14E	0.7950	0.6190	0.4415	0.063*
H14F	0.6522	0.6085	0.4093	0.063*
C15B	0.6336 (3)	1.2662 (3)	0.35835 (8)	0.0275 (6)
H15E	0.5832	1.2127	0.3364	0.041*
H15F	0.6479	1.3544	0.3472	0.041*
H15G	0.5697	1.2691	0.3843	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0370 (9)	0.0204 (8)	0.0166 (8)	−0.0034 (7)	−0.0031 (7)	−0.0001 (7)
O2A	0.0407 (10)	0.0216 (9)	0.0213 (9)	−0.0030 (8)	0.0021 (8)	−0.0051 (8)
O3A	0.0459 (10)	0.0225 (9)	0.0149 (8)	−0.0034 (8)	0.0008 (8)	−0.0041 (7)
O4A	0.0871 (17)	0.0196 (9)	0.0242 (10)	−0.0047 (11)	−0.0080 (10)	−0.0051 (8)
C1A	0.0231 (11)	0.0168 (11)	0.0181 (11)	−0.0006 (9)	0.0002 (9)	0.0006 (10)
C2A	0.0243 (11)	0.0196 (12)	0.0155 (11)	−0.0007 (9)	0.0016 (9)	0.0005 (9)
C3A	0.0256 (11)	0.0185 (12)	0.0202 (11)	0.0008 (9)	0.0028 (10)	−0.0009 (10)
C4A	0.0243 (11)	0.0190 (12)	0.0202 (12)	−0.0009 (9)	0.0026 (10)	−0.0043 (10)
C5A	0.0261 (11)	0.0240 (13)	0.0149 (11)	−0.0023 (10)	−0.0001 (9)	−0.0004 (10)
C6A	0.0249 (11)	0.0193 (12)	0.0161 (11)	−0.0001 (9)	0.0004 (9)	0.0002 (9)
C7A	0.0370 (13)	0.0195 (12)	0.0168 (11)	−0.0015 (10)	−0.0030 (10)	0.0011 (10)
C8A	0.0482 (15)	0.0182 (12)	0.0224 (13)	0.0002 (11)	−0.0051 (12)	0.0003 (11)
C9A	0.0430 (14)	0.0206 (13)	0.0221 (12)	−0.0006 (11)	−0.0015 (11)	−0.0017 (11)
C10A	0.0256 (11)	0.0187 (12)	0.0177 (11)	−0.0019 (10)	−0.0007 (9)	0.0003 (10)
C11A	0.0298 (12)	0.0216 (12)	0.0169 (11)	−0.0048 (10)	−0.0011 (9)	−0.0057 (10)
C12A	0.0316 (12)	0.0283 (13)	0.0187 (12)	−0.0018 (11)	−0.0053 (10)	−0.0009 (11)
C13A	0.0345 (14)	0.0336 (15)	0.0230 (13)	−0.0043 (12)	0.0038 (10)	−0.0080 (12)
C14A	0.0380 (13)	0.0191 (12)	0.0225 (12)	−0.0003 (10)	0.0037 (11)	−0.0004 (11)
C15A	0.0403 (14)	0.0339 (16)	0.0272 (14)	−0.0122 (12)	0.0014 (11)	0.0032 (13)
O1B	0.0467 (10)	0.0195 (9)	0.0158 (8)	0.0016 (8)	0.0045 (8)	0.0000 (7)
O2B	0.0733 (14)	0.0223 (10)	0.0201 (9)	−0.0025 (10)	0.0051 (10)	−0.0060 (8)
O3B	0.0487 (11)	0.0227 (9)	0.0158 (8)	−0.0002 (9)	0.0033 (8)	−0.0037 (7)
O4B	0.0365 (9)	0.0221 (9)	0.0234 (9)	−0.0015 (8)	−0.0008 (8)	−0.0042 (8)
C1B	0.0210 (10)	0.0197 (11)	0.0160 (11)	−0.0007 (9)	0.0009 (8)	0.0000 (10)
C2B	0.0282 (12)	0.0201 (12)	0.0171 (11)	0.0023 (10)	0.0028 (9)	0.0020 (9)
C3B	0.0376 (13)	0.0188 (12)	0.0199 (12)	0.0016 (11)	0.0019 (11)	0.0002 (10)
C4B	0.0390 (14)	0.0212 (13)	0.0172 (12)	−0.0009 (10)	−0.0006 (10)	−0.0024 (10)
C5B	0.0305 (12)	0.0231 (12)	0.0143 (11)	−0.0003 (10)	−0.0003 (9)	0.0015 (10)
C6B	0.0246 (11)	0.0175 (11)	0.0160 (11)	0.0014 (9)	0.0002 (9)	0.0014 (9)
C7B	0.0300 (12)	0.0185 (12)	0.0167 (11)	−0.0016 (10)	0.0038 (10)	0.0011 (10)
C8B	0.0296 (12)	0.0212 (12)	0.0203 (12)	−0.0028 (10)	0.0028 (10)	−0.0001 (10)
C9B	0.0245 (11)	0.0209 (12)	0.0204 (11)	−0.0001 (10)	−0.0010 (9)	−0.0005 (10)
C10B	0.0224 (11)	0.0198 (11)	0.0179 (11)	0.0028 (9)	−0.0011 (9)	0.0008 (10)
C11B	0.0285 (12)	0.0221 (12)	0.0149 (11)	0.0048 (10)	−0.0012 (9)	−0.0032 (10)

C12B	0.0373 (13)	0.0248 (13)	0.0159 (11)	0.0015 (11)	0.0045 (10)	−0.0011 (10)
C13B	0.0361 (14)	0.0360 (16)	0.0190 (12)	0.0061 (12)	−0.0038 (10)	−0.0023 (12)
C14B	0.081 (2)	0.0204 (14)	0.0248 (14)	−0.0058 (15)	0.0088 (15)	0.0020 (12)
C15B	0.0340 (13)	0.0257 (14)	0.0229 (13)	0.0004 (11)	−0.0028 (10)	0.0045 (11)

Geometric parameters (Å, °)

O1A—C2A	1.360 (3)	O1B—C2B	1.353 (3)
O1A—C12A	1.448 (3)	O1B—C12B	1.451 (3)
O2A—C4A	1.239 (3)	O2B—C4B	1.240 (3)
O3A—C5A	1.358 (3)	O3B—C5B	1.356 (3)
O3A—H3AA	0.8200	O3B—H3BA	0.8200
O4A—C9A	1.218 (3)	O4B—C9B	1.225 (3)
C1A—C10A	1.363 (3)	C1B—C10B	1.359 (3)
C1A—C6A	1.450 (3)	C1B—C6B	1.461 (3)
C1A—C2A	1.474 (3)	C1B—C2B	1.478 (3)
C2A—C3A	1.349 (3)	C2B—C3B	1.353 (3)
C3A—C4A	1.454 (3)	C3B—C4B	1.447 (3)
C3A—C14A	1.503 (3)	C3B—C14B	1.503 (4)
C4A—C5A	1.477 (3)	C4B—C5B	1.485 (3)
C5A—C6A	1.347 (3)	C5B—C6B	1.338 (3)
C6A—C7A	1.514 (3)	C6B—C7B	1.508 (3)
C7A—C15A	1.532 (4)	C7B—C15B	1.531 (3)
C7A—C8A	1.533 (4)	C7B—C8B	1.541 (3)
C7A—H7AA	0.9800	C7B—H7BA	0.9800
C8A—C9A	1.512 (3)	C8B—C9B	1.503 (3)
C8A—H8AA	0.9700	C8B—H8BA	0.9700
C8A—H8AB	0.9700	C8B—H8BB	0.9700
C9A—C10A	1.485 (3)	C9B—C10B	1.487 (3)
C10A—C11A	1.506 (3)	C10B—C11B	1.503 (3)
C11A—C12A	1.511 (3)	C11B—C12B	1.508 (3)
C11A—C13A	1.530 (3)	C11B—C13B	1.533 (3)
C11A—H11A	0.9800	C11B—H11B	0.9800
C12A—H12A	0.9700	C12B—H12C	0.9700
C12A—H12B	0.9700	C12B—H12D	0.9700
C13A—H13A	0.9600	C13B—H13D	0.9600
C13A—H13B	0.9600	C13B—H13E	0.9600
C13A—H13C	0.9600	C13B—H13F	0.9600
C14A—H14A	0.9600	C14B—H14D	0.9600
C14A—H14B	0.9600	C14B—H14E	0.9600
C14A—H14C	0.9600	C14B—H14F	0.9600
C15A—H15A	0.9600	C15B—H15E	0.9600
C15A—H15B	0.9600	C15B—H15F	0.9600
C15A—H15C	0.9600	C15B—H15G	0.9600
C2A—O1A—C12A	114.68 (18)	C2B—O1B—C12B	116.32 (18)
C5A—O3A—H3AA	109.5	C5B—O3B—H3BA	109.5
C10A—C1A—C6A	121.9 (2)	C10B—C1B—C6B	122.5 (2)
C10A—C1A—C2A	119.3 (2)	C10B—C1B—C2B	118.8 (2)
C6A—C1A—C2A	118.8 (2)	C6B—C1B—C2B	118.7 (2)

C3A—C2A—O1A	119.0 (2)	C3B—C2B—O1B	118.2 (2)
C3A—C2A—C1A	122.3 (2)	C3B—C2B—C1B	122.3 (2)
O1A—C2A—C1A	118.56 (19)	O1B—C2B—C1B	119.3 (2)
C2A—C3A—C4A	118.7 (2)	C2B—C3B—C4B	118.7 (2)
C2A—C3A—C14A	124.1 (2)	C2B—C3B—C14B	122.9 (2)
C4A—C3A—C14A	117.1 (2)	C4B—C3B—C14B	118.4 (2)
O2A—C4A—C3A	121.4 (2)	O2B—C4B—C3B	122.0 (2)
O2A—C4A—C5A	119.9 (2)	O2B—C4B—C5B	119.1 (2)
C3A—C4A—C5A	118.6 (2)	C3B—C4B—C5B	118.9 (2)
C6A—C5A—O3A	121.2 (2)	C6B—C5B—O3B	121.3 (2)
C6A—C5A—C4A	122.4 (2)	C6B—C5B—C4B	122.4 (2)
O3A—C5A—C4A	116.4 (2)	O3B—C5B—C4B	116.3 (2)
C5A—C6A—C1A	118.8 (2)	C5B—C6B—C1B	118.7 (2)
C5A—C6A—C7A	122.4 (2)	C5B—C6B—C7B	122.4 (2)
C1A—C6A—C7A	118.7 (2)	C1B—C6B—C7B	118.8 (2)
C6A—C7A—C15A	110.3 (2)	C6B—C7B—C15B	111.5 (2)
C6A—C7A—C8A	109.6 (2)	C6B—C7B—C8B	109.93 (19)
C15A—C7A—C8A	111.4 (2)	C15B—C7B—C8B	111.0 (2)
C6A—C7A—H7AA	108.5	C6B—C7B—H7BA	108.1
C15A—C7A—H7AA	108.5	C15B—C7B—H7BA	108.1
C8A—C7A—H7AA	108.5	C8B—C7B—H7BA	108.1
C9A—C8A—C7A	112.5 (2)	C9B—C8B—C7B	112.8 (2)
C9A—C8A—H8AA	109.1	C9B—C8B—H8BA	109.0
C7A—C8A—H8AA	109.1	C7B—C8B—H8BA	109.0
C9A—C8A—H8AB	109.1	C9B—C8B—H8BB	109.0
C7A—C8A—H8AB	109.1	C7B—C8B—H8BB	109.0
H8AA—C8A—H8AB	107.8	H8BA—C8B—H8BB	107.8
O4A—C9A—C10A	120.3 (2)	O4B—C9B—C10B	120.5 (2)
O4A—C9A—C8A	123.2 (2)	O4B—C9B—C8B	122.7 (2)
C10A—C9A—C8A	116.4 (2)	C10B—C9B—C8B	116.7 (2)
C1A—C10A—C9A	120.7 (2)	C1B—C10B—C9B	120.1 (2)
C1A—C10A—C11A	121.6 (2)	C1B—C10B—C11B	121.7 (2)
C9A—C10A—C11A	117.5 (2)	C9B—C10B—C11B	117.9 (2)
C10A—C11A—C12A	108.0 (2)	C10B—C11B—C12B	109.7 (2)
C10A—C11A—C13A	111.4 (2)	C10B—C11B—C13B	110.1 (2)
C12A—C11A—C13A	111.9 (2)	C12B—C11B—C13B	112.7 (2)
C10A—C11A—H11A	108.5	C10B—C11B—H11B	108.1
C12A—C11A—H11A	108.5	C12B—C11B—H11B	108.1
C13A—C11A—H11A	108.5	C13B—C11B—H11B	108.1
O1A—C12A—C11A	111.35 (19)	O1B—C12B—C11B	111.3 (2)
O1A—C12A—H12A	109.4	O1B—C12B—H12C	109.4
C11A—C12A—H12A	109.4	C11B—C12B—H12C	109.4
O1A—C12A—H12B	109.4	O1B—C12B—H12D	109.4
C11A—C12A—H12B	109.4	C11B—C12B—H12D	109.4
H12A—C12A—H12B	108.0	H12C—C12B—H12D	108.0
C11A—C13A—H13A	109.5	C11B—C13B—H13D	109.5
C11A—C13A—H13B	109.5	C11B—C13B—H13E	109.5
H13A—C13A—H13B	109.5	H13D—C13B—H13E	109.5
C11A—C13A—H13C	109.5	C11B—C13B—H13F	109.5

H13A—C13A—H13C	109.5	H13D—C13B—H13F	109.5
H13B—C13A—H13C	109.5	H13E—C13B—H13F	109.5
C3A—C14A—H14A	109.5	C3B—C14B—H14D	109.5
C3A—C14A—H14B	109.5	C3B—C14B—H14E	109.5
H14A—C14A—H14B	109.5	H14D—C14B—H14E	109.5
C3A—C14A—H14C	109.5	C3B—C14B—H14F	109.5
H14A—C14A—H14C	109.5	H14D—C14B—H14F	109.5
H14B—C14A—H14C	109.5	H14E—C14B—H14F	109.5
C7A—C15A—H15A	109.5	C7B—C15B—H15E	109.5
C7A—C15A—H15B	109.5	C7B—C15B—H15F	109.5
H15A—C15A—H15B	109.5	H15E—C15B—H15F	109.5
C7A—C15A—H15C	109.5	C7B—C15B—H15G	109.5
H15A—C15A—H15C	109.5	H15E—C15B—H15G	109.5
H15B—C15A—H15C	109.5	H15F—C15B—H15G	109.5
C12A—O1A—C2A—C3A	−154.1 (2)	C12B—O1B—C2B—C3B	−156.9 (2)
C12A—O1A—C2A—C1A	29.6 (3)	C12B—O1B—C2B—C1B	27.5 (3)
C10A—C1A—C2A—C3A	−174.2 (2)	C10B—C1B—C2B—C3B	−174.4 (2)
C6A—C1A—C2A—C3A	6.2 (3)	C6B—C1B—C2B—C3B	3.1 (4)
C10A—C1A—C2A—O1A	2.0 (3)	C10B—C1B—C2B—O1B	1.0 (3)
C6A—C1A—C2A—O1A	−177.67 (19)	C6B—C1B—C2B—O1B	178.5 (2)
O1A—C2A—C3A—C4A	−178.3 (2)	O1B—C2B—C3B—C4B	−173.7 (2)
C1A—C2A—C3A—C4A	−2.1 (4)	C1B—C2B—C3B—C4B	1.8 (4)
O1A—C2A—C3A—C14A	4.5 (4)	O1B—C2B—C3B—C14B	3.2 (4)
C1A—C2A—C3A—C14A	−179.4 (2)	C1B—C2B—C3B—C14B	178.6 (3)
C2A—C3A—C4A—O2A	179.0 (2)	C2B—C3B—C4B—O2B	175.0 (3)
C14A—C3A—C4A—O2A	−3.6 (4)	C14B—C3B—C4B—O2B	−1.9 (4)
C2A—C3A—C4A—C5A	−3.5 (3)	C2B—C3B—C4B—C5B	−5.7 (4)
C14A—C3A—C4A—C5A	174.0 (2)	C14B—C3B—C4B—C5B	177.3 (3)
O2A—C4A—C5A—C6A	−177.1 (2)	O2B—C4B—C5B—C6B	−175.7 (3)
C3A—C4A—C5A—C6A	5.4 (4)	C3B—C4B—C5B—C6B	5.0 (4)
O2A—C4A—C5A—O3A	3.1 (3)	O2B—C4B—C5B—O3B	3.6 (4)
C3A—C4A—C5A—O3A	−174.5 (2)	C3B—C4B—C5B—O3B	−175.6 (2)
O3A—C5A—C6A—C1A	178.5 (2)	O3B—C5B—C6B—C1B	−179.4 (2)
C4A—C5A—C6A—C1A	−1.3 (4)	C4B—C5B—C6B—C1B	−0.1 (4)
O3A—C5A—C6A—C7A	2.6 (4)	O3B—C5B—C6B—C7B	2.6 (4)
C4A—C5A—C6A—C7A	−177.3 (2)	C4B—C5B—C6B—C7B	−178.1 (2)
C10A—C1A—C6A—C5A	176.1 (2)	C10B—C1B—C6B—C5B	173.5 (2)
C2A—C1A—C6A—C5A	−4.3 (3)	C2B—C1B—C6B—C5B	−3.9 (3)
C10A—C1A—C6A—C7A	−7.8 (3)	C10B—C1B—C6B—C7B	−8.5 (3)
C2A—C1A—C6A—C7A	171.9 (2)	C2B—C1B—C6B—C7B	174.2 (2)
C5A—C6A—C7A—C15A	90.4 (3)	C5B—C6B—C7B—C15B	89.7 (3)
C1A—C6A—C7A—C15A	−85.6 (3)	C1B—C6B—C7B—C15B	−88.3 (3)
C5A—C6A—C7A—C8A	−146.6 (2)	C5B—C6B—C7B—C8B	−146.8 (2)
C1A—C6A—C7A—C8A	37.4 (3)	C1B—C6B—C7B—C8B	35.2 (3)
C6A—C7A—C8A—C9A	−51.6 (3)	C6B—C7B—C8B—C9B	−50.2 (3)
C15A—C7A—C8A—C9A	70.8 (3)	C15B—C7B—C8B—C9B	73.6 (3)
C7A—C8A—C9A—O4A	−144.8 (3)	C7B—C8B—C9B—O4B	−143.4 (2)
C7A—C8A—C9A—C10A	38.7 (3)	C7B—C8B—C9B—C10B	40.5 (3)
C6A—C1A—C10A—C9A	−8.3 (4)	C6B—C1B—C10B—C9B	−4.2 (3)

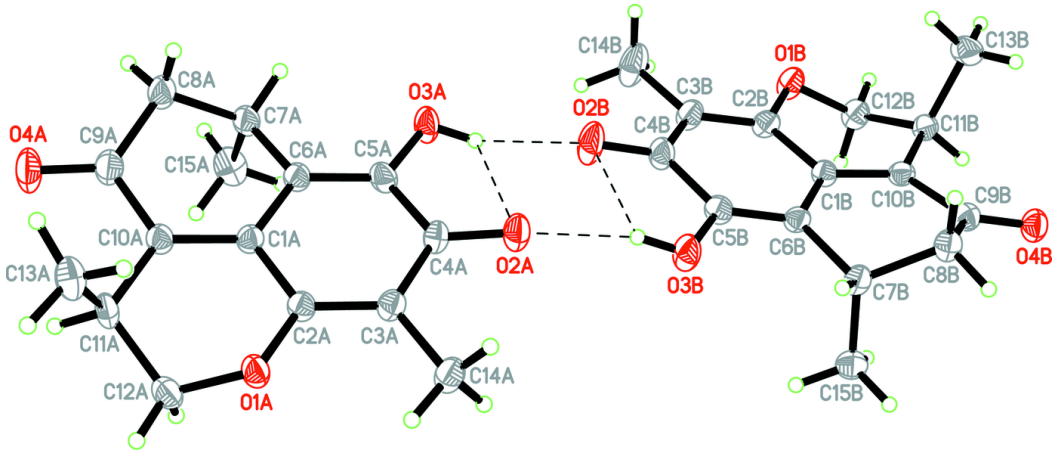
C2A—C1A—C10A—C9A	172.1 (2)	C2B—C1B—C10B—C9B	173.1 (2)
C6A—C1A—C10A—C11A	175.6 (2)	C6B—C1B—C10B—C11B	−178.9 (2)
C2A—C1A—C10A—C11A	−4.1 (4)	C2B—C1B—C10B—C11B	−1.6 (3)
O4A—C9A—C10A—C1A	175.4 (3)	O4B—C9B—C10B—C1B	171.1 (2)
C8A—C9A—C10A—C1A	−8.0 (4)	C8B—C9B—C10B—C1B	−12.7 (3)
O4A—C9A—C10A—C11A	−8.3 (4)	O4B—C9B—C10B—C11B	−14.0 (3)
C8A—C9A—C10A—C11A	168.3 (2)	C8B—C9B—C10B—C11B	162.2 (2)
C1A—C10A—C11A—C12A	−22.9 (3)	C1B—C10B—C11B—C12B	−23.9 (3)
C9A—C10A—C11A—C12A	160.8 (2)	C9B—C10B—C11B—C12B	161.3 (2)
C1A—C10A—C11A—C13A	100.4 (3)	C1B—C10B—C11B—C13B	100.7 (3)
C9A—C10A—C11A—C13A	−75.9 (3)	C9B—C10B—C11B—C13B	−74.1 (3)
C2A—O1A—C12A—C11A	−58.4 (3)	C2B—O1B—C12B—C11B	−53.8 (3)
C10A—C11A—C12A—O1A	52.5 (3)	C10B—C11B—C12B—O1B	49.7 (3)
C13A—C11A—C12A—O1A	−70.5 (3)	C13B—C11B—C12B—O1B	−73.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3A—H3AA···O2A	0.82	2.28	2.703 (2)	112
O3A—H3AA···O2B	0.82	1.98	2.760 (2)	159
O3B—H3BA···O2A	0.82	2.05	2.829 (2)	158
O3B—H3BA···O2B	0.82	2.24	2.693 (3)	115
C7A—H7AA···O3A	0.98	2.51	2.865 (3)	101
C7B—H7BA···O3B	0.98	2.50	2.854 (3)	101
C12A—H12A···O4B^{i}	0.97	2.50	3.452 (3)	168
C12B—H12D···O4B^{ii}	0.97	2.44	3.281 (3)	145
C14A—H14A···O1A	0.96	2.43	2.853 (3)	106
C12A—H12B···Cg1^{iii}	0.97	2.78	3.657 (3)	151
C13A—H13B···Cg1^{iv}	0.97	2.67	3.387 (3)	132

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

Fig. 1


C14B
C13B
01B
C3B
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02B
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C15A
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04B
C13A
C4A
02A
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C8B
C7B
C11A
C2A
C3A
C14A
C12A
01A
C15B

Fig. 2