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1,5,8-Trimethyl-1,2-dihydronaphtho-[2,1-*b*]furan-6,7-dione

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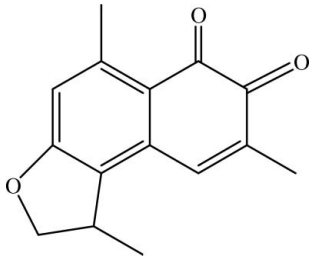
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.010 Å; R factor = 0.088; wR factor = 0.259; data-to-parameter ratio = 7.5.

The title sesquiterpene *ortho*-naphthoquinone compound, $C_{15}H_{14}O_3$, known as Mansonone D, was isolated from *Thespesia populnea*. There are two independent molecules in the asymmetric unit. In both molecules the dihydrofuran ring adopts an envelope conformation. The molecules are connected into sheets parallel to the *bc* plane by weak C—H···O interactions. The sheets are stacked along the *a* axis, with molecules of adjacent sheets linked by C—H···O hydrogen bonds, C—H···π and π–π [centroid–centroid distance = 3.579 (4) Å] interactions.

Related literature

For bond-length data, see: Allen *et al.* (1987). For ring puckering parameters, see: Cremer & Pople (1975). For related quinone structures, see: Chantrapromma *et al.* (2007); Fun *et al.* (2007); Milbrodt *et al.* (1997); Puckhaber & Stipanovic (2004).



Experimental

Crystal data

$C_{15}H_{14}O_3$
M_r = 242.26
Orthorhombic, $P2_12_12_1$
a = 7.1218 (2) Å
b = 10.2061 (2) Å
c = 32.9005 (7) Å
V = 2391.4 (1) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
0.37 × 0.16 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.966, T_{max} = 0.989
23139 measured reflections
2445 independent reflections
1873 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.259
S = 1.07
2445 reflections
325 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*1 is the centroid of the C3*A*/C4*A*/C9*A*–C12*A* ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1*B*—H1*BA*···O3*B*^{i}	0.97	2.47	3.413 (10)	164
C11*B*—H11*B*···O1*A*^{ii}	0.93	2.43	3.349 (8)	169
C13*B*—H13*E*···O2*A*^{iii}	0.96	2.42	3.204 (10)	138
C13*B*—H13*F*···O2*B*^{i}	0.96	2.54	3.500 (9)	174
C15*B*—H15*D*···*Cg*1^{iii}	0.96	2.87	3.539 (9)	127
C15*B*—H15*E*···*Cg*1^{iv}	0.96	2.86	3.632 (9)	138

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

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: CI2508).

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supplementary materials

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1,5,8-Trimethyl-1,2-dihydronaphtho[2,1-*b*]furan-6,7-dione

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

Comment

We have been investigating the chemical constituents of *Thespesia populnea*, or Po-ta-lae in Thai, a genus of *malvaceae* with basically tropical and subtropical worldwide distribution. The heartwood of this plant is a rich source of sesquiterpenoid quinines (Milbrodt *et al.*, 1997). We have previously reported the crystal structures of sesquiterpenoid quinone compounds from this plant namely 3,6,9-trimethyl-2,3-dihydrobenzo[*de*]-chromene-7,8-dione (Fun *et al.*, 2007) and 7-hydroxy-3,6,9-trimethyl-2,3,5,6-tetrahydronaphtho [1,8 − *b*,*c*]pyran-4,8-dione (Chantrapromma *et al.*, 2007). We report the crystal structure of the title compound known as Mansonone D (Puckhaber & Stipanovic, 2004) which was isolated from the heartwood of *T. populnea*, collected from the Suratthani province in the southern part of Thailand.

The title compound crystallizes with two molecules (*A* and *B*) per asymmetric unit (Fig. 1). The bond lengths and angles in the title compound are within normal ranges (Allen *et al.*, 1987) and comparable with closely related structures (Chantrapromma *et al.*, 2007; Fun *et al.*, 2007). The naphthoquinone ring system (C3–C12) is essentially planar, with atom C7A deviating by a maximum of 0.059 (7) Å in molecule *A* and atom C10B deviating by a maximum of 0.056 (7) Å in molecule *B*. The furan ring adopts an envelope conformation in both molecules, with atom C1 displaced from the C2/C3/C12/O1 plane by −0.096 (11) and −0.138 (9) Å, respectively, for molecules *A* and *B*. The puckering parameters (Cremer & Pople, 1975) are Q = 0.154 (7) Å and θ = 42 (3)° for molecule *A* and Q = 0.218 (7) Å and θ = 42.5 (19)° for molecule *B*. The methyl group at atom C2 is axially attached, as indicated by the torsion angles C13–C2–C3–C12 of 109.8 (7)° and 103.4 (7)°, respectively, in molecules *A* and *B*.

In the crystal structure (Fig. 2), the molecules are connected into sheets parallel to the *bc* plane by weak C—H···O interactions (Table 1), and the sheets are stacked along the *a* axis. In addition to C—H···O hydrogen bonds, C—H···π interactions (Table 1) involving the C3A/C4A/C9A—C12A ring (centroid *Cg*1), and π-π interactions involving the C4A—C9A and C3B/C4B/C9B—C12B rings [centroid-centroid distance is 3.579 (4) Å] are observed between the sheets.

Experimental

Air-dried heartwood of *T. populnea* 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 furnish a orange-brown residue (37.5 g) which was subjected to quick column chromatography on silica gel, eluting with CH_2Cl_2 and separated into 8 fractions (F1—F8). Fraction F7 was purified by quick column chromatography with a gradient of acetone-CH_2Cl_2 to give the title compound. Single crystals of the title compound were obtained by recrystalization from MeOH-CH_2Cl_2 (3:7 *v*/*v*) after several days (m.p. 432–434 K).

Refinement

H atoms were placed in calculated positions, with C—H = 0.93–0.98 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 total of 1761 Friedel pairs were merged before final

refinement as there is no large anomalous dispersion for the determination of the absolute configuration. Large anisotropic displacement parameters and difference density features indicate possible disorder in both independent molecules, but no suitable disorder model was found.

Figures

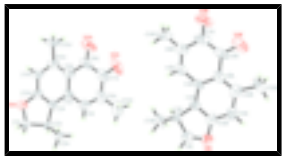


Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme.

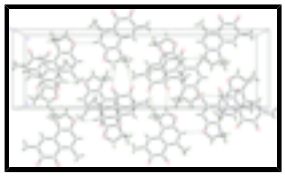


Fig. 2. The crystal packing of the title compound, viewed approximately along the *a* axis. Hydrogen bonds are shown as dashed lines.

1,5,8-Trimethyl-1,2-dihydronaphtho[2,1-*b*]furan-6,7-dione

Crystal data

$C_{15}H_{14}O_3$	$D_x = 1.346$ Mg m^{-3}
$M_r = 242.26$	Melting point: 432-434 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 2445 reflections
$a = 7.1218$ (2) Å	$\theta = 1.2$–25.0^o
$b = 10.2061$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 32.9005$ (7) Å	$T = 100.0$ (1) K
$V = 2391.4$ (1) Å^3	Plate, colourless
$Z = 8$	0.37 × 0.16 × 0.12 mm
$F_{000} = 1024$	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	2445 independent reflections
Radiation source: fine-focus sealed tube	1873 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.059$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 25.0^o$
$T = 100.0(1)$ K	$\theta_{min} = 1.2^o$
ω scans	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -11 \rightarrow 12$
$T_{min} = 0.966$, $T_{max} = 0.989$	$l = -39 \rightarrow 36$
23139 measured reflections	

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.088$

$wR(F^2) = 0.259$

$S = 1.07$

2445 reflections

325 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

$w = 1/[\sigma^2(F_o^2) + (0.1374P)^2 + 3.3697P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.68$ 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 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 $F^2 > 2$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	0.7860 (8)	0.1631 (5)	0.23458 (13)	0.0521 (13)
O2A	0.7413 (9)	0.7367 (6)	0.0870 (2)	0.082 (2)
O3A	0.6978 (10)	0.7477 (5)	0.1677 (2)	0.082 (2)
C1A	0.8304 (15)	0.0739 (8)	0.2016 (2)	0.067 (3)
H1AA	0.7599	−0.0068	0.2047	0.080*
H1AB	0.9632	0.0528	0.2020	0.080*
C2A	0.7783 (11)	0.1413 (6)	0.16119 (19)	0.0441 (17)
H2AA	0.8829	0.1344	0.1420	0.053*
C3A	0.7582 (10)	0.2811 (6)	0.17544 (18)	0.0384 (15)
C4A	0.7458 (10)	0.3985 (6)	0.15415 (19)	0.0374 (15)
C5A	0.7488 (10)	0.3972 (7)	0.1104 (2)	0.0480 (18)
H5AA	0.7557	0.3161	0.0976	0.058*
C6A	0.7425 (10)	0.5055 (8)	0.0863 (2)	0.0519 (19)
C7A	0.7335 (10)	0.6332 (8)	0.1061 (3)	0.058 (2)
C8A	0.7167 (11)	0.6400 (7)	0.1528 (3)	0.0567 (15)

C9A	0.7284 (9)	0.5177 (6)	0.1763 (2)	0.0428 (16)
C10A	0.7230 (10)	0.5159 (7)	0.2191 (2)	0.0491 (19)
C11A	0.7399 (10)	0.3971 (7)	0.2398 (2)	0.0479 (18)
H11A	0.7368	0.3942	0.2681	0.057*
C12A	0.7613 (11)	0.2844 (6)	0.2174 (2)	0.0439 (17)
C13A	0.6035 (12)	0.0846 (7)	0.1427 (2)	0.052 (2)
H13A	0.5734	0.1313	0.1182	0.078*
H13B	0.5015	0.0923	0.1616	0.078*
H13C	0.6240	−0.0062	0.1364	0.078*
C14A	0.7541 (12)	0.5032 (9)	0.0414 (2)	0.073 (3)
H14A	0.7541	0.4141	0.0321	0.109*
H14B	0.8677	0.5456	0.0329	0.109*
H14C	0.6480	0.5485	0.0302	0.109*
C15A	0.7062 (13)	0.6392 (8)	0.2456 (3)	0.068 (2)
H15A	0.7065	0.6145	0.2738	0.102*
H15B	0.5911	0.6838	0.2394	0.102*
H15C	0.8104	0.6963	0.2403	0.102*
O1B	0.6595 (8)	0.6338 (5)	−0.14493 (14)	0.0526 (14)
O2B	0.8305 (13)	1.2728 (5)	−0.0344 (2)	0.097 (3)
O3B	0.7238 (10)	1.2479 (5)	−0.1135 (2)	0.0796 (19)
C1B	0.6116 (12)	0.5724 (8)	−0.1074 (2)	0.061 (2)
H1BA	0.6657	0.4853	−0.1063	0.074*
H1BB	0.4762	0.5642	−0.1052	0.074*
C2B	0.6862 (10)	0.6546 (6)	−0.0722 (2)	0.0433 (17)
H2BA	0.5956	0.6582	−0.0498	0.052*
C3B	0.7019 (10)	0.7842 (6)	−0.0932 (2)	0.0398 (16)
C4B	0.7307 (10)	0.9114 (6)	−0.0810 (2)	0.0410 (16)
C5B	0.7446 (9)	0.9358 (7)	−0.0353 (2)	0.0435 (16)
H5BA	0.7309	0.8647	−0.0179	0.052*
C6B	0.7744 (10)	1.0504 (7)	−0.0194 (2)	0.0516 (19)
C7B	0.7858 (13)	1.1676 (8)	−0.0467 (3)	0.067 (2)
C8B	0.7482 (10)	1.1473 (7)	−0.0909 (3)	0.0567 (15)
C9B	0.7388 (10)	1.0164 (7)	−0.1085 (2)	0.0464 (17)
C10B	0.7339 (10)	0.9925 (8)	−0.1500 (2)	0.0517 (19)
C11B	0.7083 (11)	0.8608 (8)	−0.1639 (2)	0.0528 (19)
H11B	0.7048	0.8419	−0.1915	0.063*
C12B	0.6885 (10)	0.7611 (7)	−0.1350 (2)	0.0478 (18)
C13B	0.8766 (11)	0.6060 (7)	−0.0575 (2)	0.054 (2)
H13D	0.9099	0.6504	−0.0328	0.081*
H13E	0.9697	0.6235	−0.0779	0.081*
H13F	0.8705	0.5134	−0.0526	0.081*
C14B	0.7923 (13)	1.0738 (8)	0.0239 (2)	0.064 (2)
H14D	0.7974	0.9915	0.0380	0.097*
H14E	0.6861	1.1232	0.0333	0.097*
H14F	0.9054	1.1222	0.0291	0.097*
C15B	0.7494 (12)	1.0943 (7)	−0.1844 (3)	0.065 (2)
H15D	0.8544	1.1509	−0.1793	0.098*
H15E	0.6363	1.1454	−0.1854	0.098*
H15F	0.7670	1.0501	−0.2098	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.064 (3)	0.049 (3)	0.044 (3)	0.005 (3)	−0.012 (3)	−0.004 (2)
O2A	0.046 (3)	0.062 (4)	0.137 (6)	0.000 (3)	−0.012 (4)	0.048 (4)
O3A	0.075 (4)	0.035 (3)	0.135 (5)	0.001 (3)	0.000 (4)	−0.006 (3)
C1A	0.091 (7)	0.050 (4)	0.060 (5)	0.022 (5)	−0.028 (5)	−0.022 (4)
C2A	0.051 (4)	0.041 (4)	0.041 (3)	0.015 (4)	−0.007 (3)	−0.014 (3)
C3A	0.033 (3)	0.045 (4)	0.037 (3)	0.000 (3)	0.002 (3)	−0.012 (3)
C4A	0.032 (4)	0.034 (3)	0.046 (4)	0.001 (3)	0.005 (3)	−0.006 (3)
C5A	0.029 (3)	0.058 (5)	0.056 (4)	0.003 (3)	0.006 (4)	−0.014 (4)
C6A	0.026 (3)	0.064 (5)	0.065 (5)	0.003 (4)	0.010 (4)	0.013 (4)
C7A	0.022 (3)	0.055 (5)	0.098 (6)	0.001 (4)	−0.003 (4)	0.012 (5)
C8A	0.034 (3)	0.031 (3)	0.106 (5)	0.003 (2)	0.007 (3)	0.000 (3)
C9A	0.028 (3)	0.037 (3)	0.063 (5)	0.000 (3)	0.014 (3)	−0.010 (3)
C10A	0.029 (3)	0.044 (4)	0.074 (5)	−0.005 (3)	0.012 (4)	−0.023 (4)
C11A	0.039 (4)	0.053 (4)	0.052 (4)	−0.004 (3)	0.005 (4)	−0.020 (3)
C12A	0.044 (4)	0.041 (4)	0.047 (4)	−0.001 (4)	−0.004 (4)	−0.013 (3)
C13A	0.069 (5)	0.040 (4)	0.047 (4)	0.003 (4)	−0.016 (4)	−0.010 (3)
C14A	0.041 (4)	0.103 (7)	0.073 (5)	−0.012 (5)	0.002 (4)	0.028 (5)
C15A	0.063 (5)	0.053 (5)	0.089 (6)	0.001 (4)	0.009 (5)	−0.035 (4)
O1B	0.064 (3)	0.046 (3)	0.048 (3)	−0.007 (3)	−0.005 (3)	−0.009 (2)
O2B	0.133 (7)	0.038 (3)	0.120 (5)	−0.021 (4)	0.019 (5)	−0.004 (3)
O3B	0.072 (4)	0.044 (3)	0.123 (5)	−0.001 (3)	0.012 (4)	0.012 (3)
C1B	0.060 (5)	0.052 (5)	0.072 (5)	−0.003 (4)	0.004 (5)	−0.006 (4)
C2B	0.044 (4)	0.037 (4)	0.048 (4)	−0.009 (3)	0.005 (3)	−0.002 (3)
C3B	0.041 (4)	0.030 (3)	0.049 (4)	−0.004 (3)	0.011 (3)	0.000 (3)
C4B	0.031 (3)	0.029 (3)	0.063 (4)	0.007 (3)	−0.016 (3)	0.003 (3)
C5B	0.022 (3)	0.041 (4)	0.068 (4)	−0.004 (3)	0.002 (3)	0.012 (3)
C6B	0.033 (4)	0.047 (4)	0.075 (5)	−0.001 (3)	0.005 (4)	−0.005 (4)
C7B	0.062 (5)	0.047 (5)	0.093 (6)	−0.024 (4)	0.023 (5)	−0.020 (4)
C8B	0.034 (3)	0.031 (3)	0.106 (5)	0.003 (2)	0.007 (3)	0.000 (3)
C9B	0.032 (3)	0.048 (4)	0.059 (4)	0.004 (3)	0.006 (4)	0.008 (3)
C10B	0.023 (3)	0.075 (5)	0.058 (4)	0.004 (4)	0.002 (3)	−0.004 (4)
C11B	0.048 (4)	0.062 (5)	0.048 (4)	−0.006 (4)	−0.008 (4)	0.006 (4)
C12B	0.038 (4)	0.054 (4)	0.051 (4)	−0.001 (4)	0.001 (3)	0.000 (4)
C13B	0.053 (4)	0.043 (4)	0.067 (5)	0.007 (4)	0.003 (4)	0.024 (4)
C14B	0.063 (5)	0.046 (4)	0.084 (6)	−0.007 (4)	0.018 (5)	−0.018 (4)
C15B	0.047 (4)	0.050 (4)	0.099 (6)	0.008 (4)	0.006 (5)	0.035 (4)

Geometric parameters (Å, °)

O1A—C12A	1.372 (8)	O1B—C12B	1.355 (9)
O1A—C1A	1.451 (9)	O1B—C1B	1.427 (9)
O2A—C7A	1.231 (9)	O2B—C7B	1.191 (9)
O3A—C8A	1.211 (9)	O3B—C8B	1.280 (9)
C1A—C2A	1.543 (10)	C1B—C2B	1.525 (10)
C1A—H1AA	0.97	C1B—H1BA	0.97

C1A—H1AB	0.97	C1B—H1BB	0.97
C2A—C13A	1.501 (10)	C2B—C3B	1.497 (9)
C2A—C3A	1.508 (9)	C2B—C13B	1.522 (10)
C2A—H2AA	0.98	C2B—H2BA	0.98
C3A—C12A	1.380 (9)	C3B—C4B	1.374 (9)
C3A—C4A	1.390 (9)	C3B—C12B	1.400 (10)
C4A—C9A	1.424 (9)	C4B—C9B	1.406 (9)
C4A—C5A	1.441 (9)	C4B—C5B	1.525 (10)
C5A—C6A	1.360 (10)	C5B—C6B	1.299 (10)
C5A—H5AA	0.93	C5B—H5BA	0.93
C6A—C7A	1.458 (11)	C6B—C14B	1.449 (11)
C6A—C14A	1.479 (11)	C6B—C7B	1.498 (11)
C7A—C8A	1.543 (12)	C7B—C8B	1.492 (12)
C8A—C9A	1.471 (10)	C8B—C9B	1.459 (10)
C9A—C10A	1.410 (10)	C9B—C10B	1.386 (10)
C10A—C11A	1.396 (10)	C10B—C11B	1.431 (11)
C10A—C15A	1.535 (9)	C10B—C15B	1.540 (10)
C11A—C12A	1.376 (9)	C11B—C12B	1.400 (10)
C11A—H11A	0.93	C11B—H11B	0.93
C13A—H13A	0.96	C13B—H13D	0.96
C13A—H13B	0.96	C13B—H13E	0.96
C13A—H13C	0.96	C13B—H13F	0.96
C14A—H14A	0.96	C14B—H14D	0.96
C14A—H14B	0.96	C14B—H14E	0.96
C14A—H14C	0.96	C14B—H14F	0.96
C15A—H15A	0.96	C15B—H15D	0.96
C15A—H15B	0.96	C15B—H15E	0.96
C15A—H15C	0.96	C15B—H15F	0.96
C12A—O1A—C1A	106.6 (5)	C12B—O1B—C1B	104.4 (5)
O1A—C1A—C2A	108.2 (6)	O1B—C1B—C2B	109.4 (6)
O1A—C1A—H1AA	110.1	O1B—C1B—H1BA	109.8
C2A—C1A—H1AA	110.1	C2B—C1B—H1BA	109.8
O1A—C1A—H1AB	110.1	O1B—C1B—H1BB	109.8
C2A—C1A—H1AB	110.1	C2B—C1B—H1BB	109.8
H1AA—C1A—H1AB	108.4	H1BA—C1B—H1BB	108.2
C13A—C2A—C3A	114.3 (6)	C3B—C2B—C13B	111.6 (6)
C13A—C2A—C1A	112.1 (7)	C3B—C2B—C1B	99.3 (6)
C3A—C2A—C1A	100.2 (5)	C13B—C2B—C1B	111.8 (7)
C13A—C2A—H2AA	109.9	C3B—C2B—H2BA	111.2
C3A—C2A—H2AA	109.9	C13B—C2B—H2BA	111.2
C1A—C2A—H2AA	109.9	C1B—C2B—H2BA	111.2
C12A—C3A—C4A	118.9 (6)	C4B—C3B—C12B	117.2 (6)
C12A—C3A—C2A	109.4 (6)	C4B—C3B—C2B	135.3 (6)
C4A—C3A—C2A	131.6 (5)	C12B—C3B—C2B	107.5 (6)
C3A—C4A—C9A	119.0 (6)	C3B—C4B—C9B	122.5 (7)
C3A—C4A—C5A	119.7 (6)	C3B—C4B—C5B	116.9 (6)
C9A—C4A—C5A	121.4 (6)	C9B—C4B—C5B	120.6 (6)
C6A—C5A—C4A	125.0 (7)	C6B—C5B—C4B	123.7 (6)
C6A—C5A—H5AA	117.5	C6B—C5B—H5BA	118.2

C4A—C5A—H5AA	117.5	C4B—C5B—H5BA	118.2
C5A—C6A—C7A	117.9 (7)	C5B—C6B—C14B	124.0 (7)
C5A—C6A—C14A	124.5 (8)	C5B—C6B—C7B	119.1 (7)
C7A—C6A—C14A	117.5 (7)	C14B—C6B—C7B	116.9 (7)
O2A—C7A—C6A	122.5 (8)	O2B—C7B—C8B	120.2 (8)
O2A—C7A—C8A	118.3 (8)	O2B—C7B—C6B	122.1 (9)
C6A—C7A—C8A	119.2 (7)	C8B—C7B—C6B	117.6 (6)
O3A—C8A—C9A	124.4 (8)	O3B—C8B—C9B	119.8 (7)
O3A—C8A—C7A	116.9 (7)	O3B—C8B—C7B	118.6 (7)
C9A—C8A—C7A	118.7 (6)	C9B—C8B—C7B	121.6 (7)
C10A—C9A—C4A	120.2 (6)	C10B—C9B—C4B	120.0 (7)
C10A—C9A—C8A	122.3 (6)	C10B—C9B—C8B	123.7 (7)
C4A—C9A—C8A	117.5 (6)	C4B—C9B—C8B	116.3 (7)
C11A—C10A—C9A	119.8 (6)	C9B—C10B—C11B	118.9 (7)
C11A—C10A—C15A	116.2 (7)	C9B—C10B—C15B	127.0 (7)
C9A—C10A—C15A	124.0 (7)	C11B—C10B—C15B	114.1 (7)
C12A—C11A—C10A	118.3 (6)	C12B—C11B—C10B	118.6 (7)
C12A—C11A—H11A	120.9	C12B—C11B—H11B	120.7
C10A—C11A—H11A	120.9	C10B—C11B—H11B	120.7
O1A—C12A—C11A	123.1 (6)	O1B—C12B—C3B	114.1 (6)
O1A—C12A—C3A	113.2 (6)	O1B—C12B—C11B	123.2 (6)
C11A—C12A—C3A	123.7 (7)	C3B—C12B—C11B	122.6 (7)
C2A—C13A—H13A	109.5	C2B—C13B—H13D	109.5
C2A—C13A—H13B	109.5	C2B—C13B—H13E	109.5
H13A—C13A—H13B	109.5	H13D—C13B—H13E	109.5
C2A—C13A—H13C	109.5	C2B—C13B—H13F	109.5
H13A—C13A—H13C	109.5	H13D—C13B—H13F	109.5
H13B—C13A—H13C	109.5	H13E—C13B—H13F	109.5
C6A—C14A—H14A	109.5	C6B—C14B—H14D	109.5
C6A—C14A—H14B	109.5	C6B—C14B—H14E	109.5
H14A—C14A—H14B	109.5	H14D—C14B—H14E	109.5
C6A—C14A—H14C	109.5	C6B—C14B—H14F	109.5
H14A—C14A—H14C	109.5	H14D—C14B—H14F	109.5
H14B—C14A—H14C	109.5	H14E—C14B—H14F	109.5
C10A—C15A—H15A	109.5	C10B—C15B—H15D	109.5
C10A—C15A—H15B	109.5	C10B—C15B—H15E	109.5
H15A—C15A—H15B	109.5	H15D—C15B—H15E	109.5
C10A—C15A—H15C	109.5	C10B—C15B—H15F	109.5
H15A—C15A—H15C	109.5	H15D—C15B—H15F	109.5
H15B—C15A—H15C	109.5	H15E—C15B—H15F	109.5
C12A—O1A—C1A—C2A	−15.4 (9)	C12B—O1B—C1B—C2B	−22.2 (8)
O1A—C1A—C2A—C13A	−106.3 (7)	O1B—C1B—C2B—C3B	22.6 (8)
O1A—C1A—C2A—C3A	15.4 (9)	O1B—C1B—C2B—C13B	−95.3 (7)
C13A—C2A—C3A—C12A	109.8 (7)	C13B—C2B—C3B—C4B	−74.1 (11)
C1A—C2A—C3A—C12A	−10.3 (9)	C1B—C2B—C3B—C4B	167.9 (9)
C13A—C2A—C3A—C4A	−73.1 (10)	C13B—C2B—C3B—C12B	103.4 (7)
C1A—C2A—C3A—C4A	166.8 (8)	C1B—C2B—C3B—C12B	−14.6 (8)
C12A—C3A—C4A—C9A	−2.7 (11)	C12B—C3B—C4B—C9B	2.7 (11)
C2A—C3A—C4A—C9A	−179.7 (7)	C2B—C3B—C4B—C9B	−179.9 (7)

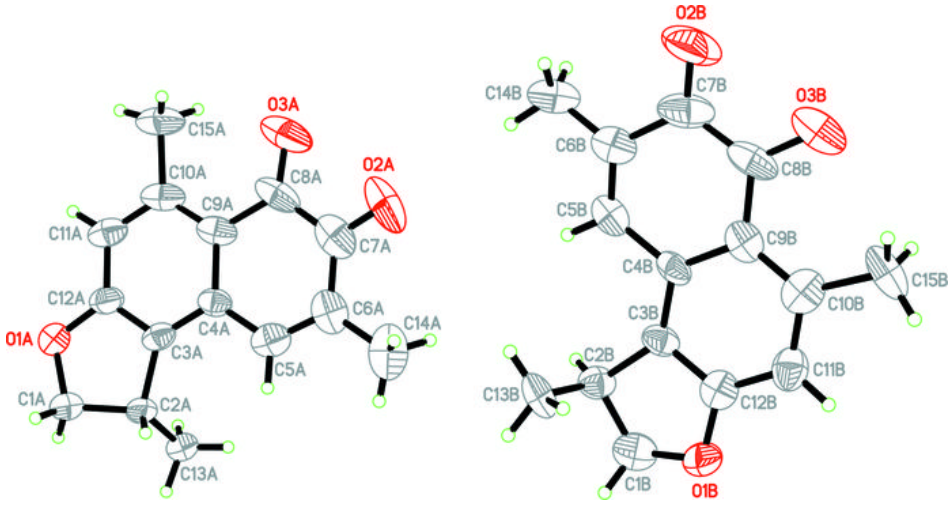
C12A—C3A—C4A—C5A	177.9 (6)	C12B—C3B—C4B—C5B	179.9 (6)
C2A—C3A—C4A—C5A	0.9 (13)	C2B—C3B—C4B—C5B	−2.8 (13)
C3A—C4A—C5A—C6A	−178.3 (7)	C3B—C4B—C5B—C6B	178.9 (7)
C9A—C4A—C5A—C6A	2.4 (12)	C9B—C4B—C5B—C6B	−3.9 (11)
C4A—C5A—C6A—C7A	0.6 (11)	C4B—C5B—C6B—C14B	−178.4 (7)
C4A—C5A—C6A—C14A	177.2 (7)	C4B—C5B—C6B—C7B	3.5 (11)
C5A—C6A—C7A—O2A	174.5 (7)	C5B—C6B—C7B—O2B	−172.6 (9)
C14A—C6A—C7A—O2A	−2.3 (11)	C14B—C6B—C7B—O2B	9.1 (13)
C5A—C6A—C7A—C8A	−4.6 (10)	C5B—C6B—C7B—C8B	4.3 (11)
C14A—C6A—C7A—C8A	178.6 (7)	C14B—C6B—C7B—C8B	−173.9 (7)
O2A—C7A—C8A—O3A	5.5 (12)	O2B—C7B—C8B—O3B	−15.4 (13)
C6A—C7A—C8A—O3A	−175.4 (7)	C6B—C7B—C8B—O3B	167.6 (7)
O2A—C7A—C8A—C9A	−173.3 (6)	O2B—C7B—C8B—C9B	164.7 (9)
C6A—C7A—C8A—C9A	5.8 (11)	C6B—C7B—C8B—C9B	−12.4 (11)
C3A—C4A—C9A—C10A	−0.3 (11)	C3B—C4B—C9B—C10B	−5.6 (11)
C5A—C4A—C9A—C10A	179.1 (7)	C5B—C4B—C9B—C10B	177.3 (6)
C3A—C4A—C9A—C8A	179.6 (7)	C3B—C4B—C9B—C8B	173.1 (7)
C5A—C4A—C9A—C8A	−1.0 (10)	C5B—C4B—C9B—C8B	−4.0 (10)
O3A—C8A—C9A—C10A	−1.7 (12)	O3B—C8B—C9B—C10B	10.5 (11)
C7A—C8A—C9A—C10A	177.0 (6)	C7B—C8B—C9B—C10B	−169.5 (7)
O3A—C8A—C9A—C4A	178.4 (8)	O3B—C8B—C9B—C4B	−168.1 (7)
C7A—C8A—C9A—C4A	−2.9 (10)	C7B—C8B—C9B—C4B	11.9 (10)
C4A—C9A—C10A—C11A	1.7 (11)	C4B—C9B—C10B—C11B	4.0 (11)
C8A—C9A—C10A—C11A	−178.2 (7)	C8B—C9B—C10B—C11B	−174.6 (7)
C4A—C9A—C10A—C15A	179.5 (7)	C4B—C9B—C10B—C15B	−177.3 (7)
C8A—C9A—C10A—C15A	−0.4 (11)	C8B—C9B—C10B—C15B	4.1 (12)
C9A—C10A—C11A—C12A	−0.1 (11)	C9B—C10B—C11B—C12B	0.2 (11)
C15A—C10A—C11A—C12A	−178.0 (7)	C15B—C10B—C11B—C12B	−178.6 (7)
C1A—O1A—C12A—C11A	−171.8 (8)	C1B—O1B—C12B—C3B	12.5 (8)
C1A—O1A—C12A—C3A	8.8 (9)	C1B—O1B—C12B—C11B	−169.0 (7)
C10A—C11A—C12A—O1A	177.6 (7)	C4B—C3B—C12B—O1B	−179.8 (6)
C10A—C11A—C12A—C3A	−3.2 (12)	C2B—C3B—C12B—O1B	2.2 (9)
C4A—C3A—C12A—O1A	−176.0 (6)	C4B—C3B—C12B—C11B	1.7 (11)
C2A—C3A—C12A—O1A	1.5 (9)	C2B—C3B—C12B—C11B	−176.3 (7)
C4A—C3A—C12A—C11A	4.6 (12)	C10B—C11B—C12B—O1B	178.5 (7)
C2A—C3A—C12A—C11A	−177.8 (7)	C10B—C11B—C12B—C3B	−3.1 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1B—H1BA···O3B^{i}	0.97	2.47	3.413 (10)	164
C11B—H11B···O1A^{ii}	0.93	2.43	3.349 (8)	169
C13B—H13E···O2A^{iii}	0.96	2.42	3.204 (10)	138
C13B—H13F···O2B^{i}	0.96	2.54	3.500 (9)	174
C15B—H15D···Cg1^{iii}	0.96	2.87	3.539 (9)	127
C15B—H15E···Cg1^{iv}	0.96	2.86	3.632 (9)	138

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

Fig. 1


O2B
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C7B
O3B
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C8B
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C2B
C11B
C13B
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C15A
C10A
C8A
O2A
C9A
C11A
C7A
C12A
C6A
C14A
O1A
C4A
C3A
C5A
C1A
C2A
C13A

Fig. 2