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9-Methoxy-6a,11a-dimethyl-6a,11a-dihydro-6*H*-1-benzofuro[3,2-*c*]chromen-3-ol from *Dalbergia oliveri*

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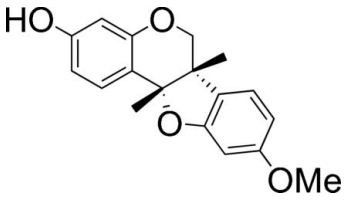
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 10.7.

The title compound, commonly known as (+)-(6a*S*,11a*S*)-medicarpin, $C_{16}H_{14}O_4$, was isolated from *Dalbergia oliveri* and displays a rigid molecule consisting of four fused rings. The benzofuran system is inclined at an angle of 76.49 (2)° with respect to the chroman unit. The compound exists as a polymeric chain arising from intermolecular O—H···O bonding.

Related literature

For general background to (+)-(6a*S*,11a*S*)-medicarpin, see: Deesamer *et al.* (2007); Hargreaves *et al.* (1976). For a related structure, see: Aree *et al.* (2003).



Experimental

Crystal data

$C_{16}H_{14}O_4$
M_r = 270.27
Monoclinic, $P2_1$
a = 6.6289 (3) Å
b = 8.7963 (4) Å
c = 11.3150 (5) Å
β = 99.4820 (10)°
V = 650.76 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.40 × 0.25 × 0.20 mm

Data collection

Bruker SMART diffractometer
Absorption correction: none
4783 measured reflections
1949 independent reflections
2867 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.09
1949 reflections
182 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O3^{i}	0.82	2.07	2.882 (2)	169

Symmetry code: (i) $x, y, z+1$.

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (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: NG2631).

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supporting information

Acta Cryst. (2009). E**65**, o2387 [doi:10.1107/S1600536809034485]

9-Methoxy-6a,11a-dimethyl-6a,11a-dihydro-6*H*-1-benzofuro[3,2-*c*]chromen-3-ol from *Dalbergia oliveri*

Sujittra Deesamer, Warinthorn Chavasiri, Narongsak Chaichit, Nongnuj Muangsin and Udom Kokpol

S1. Comment

Dalbergia Oliveri Gamble is widely found in Thailand and used in traditional Thai medicine for treament of chronic ulcer. One of major compositions of CH_2Cl_2 crude products extracted from the heartwoods of *Dalbergia Oliveri* (Deesamer *et al.*, 2007) was (+)(6*aS*,11*aS*)-Medicarpin. It was identified as phytoalexin (Hargreaves *et al.*, 1976).

The rigid molecule of the title compound consists of four fused rings adopts a bent-shaped conformation. The benzofuran ring system is inclined at the angle of 76.49 (2)° with respect to the chroman moiety. The tetrahydropyranyl group adopts an envelope conformation with atom C6 deviates from the plane by 0.4144 Å.

The compound exists as a polymeric chain arising from intermolecular O—H···O bonding.

S2. Experimental

Four kilograms of dried and powder heartwoods of *D. oliveri* were extracted with hexane. The marc was then extracted with CH_2Cl_2, EtOAc and MeOH, respectively. The CH_2Cl_2 crudeextract was subjected to silica gel colume chromatography eluting with 60%EtOAc:Hexane to afford the title compound (3.92 g). The suitable single crystals of the title compound were recrystallized from acetone-water as colourless needle crystals.

m.p. 132.0–133.5°C; m/*z*: 270[M^+]

The specific rotation of D3 as $[\alpha]_D^+$ 223.1° (c 0.16 in acetone, at 20°C) indicated the absolute configuration to be (+)(6*aS*,11*aS*)-medicarpin.

^{1}H-NMR ($CDCl_3$): δ (p.p.m.) 3.55(1*H*,m,H-6a), 3.65 (1*H*, dd, J =10.9 and 10.9 Hz, H-6*ax*), 4.26 (1*H*, dd, J = 4.8, 10.9 Hz, H-6*eq*) and 5.23 (1*H*, d, J = 6.7 Hz, H-116*a*),

S3. Refinement

All non-hydrogen atoms were anisotropically refined. The hydrogen atoms were positioned geometrically and refined using a riding model, with C—H = 0.93Å (aromatic), 0.97Å (CH_2) and 0.98Å (CH_3), and O—H = 0.82 Å, and U_{iso}(H) = 1.2*U*eq ($C_{aromatic}$), 1.5*U*eq (C_{CH2}), 1.5*U*eq (C_{CH3}) and 1.2*U*eq (C_O), respectively. In the structure, Friedel pairs [1949] were merged and the stereochemistry assumed from the specific rotation and the previously reported structure (Deesamer *et al.* 2007).

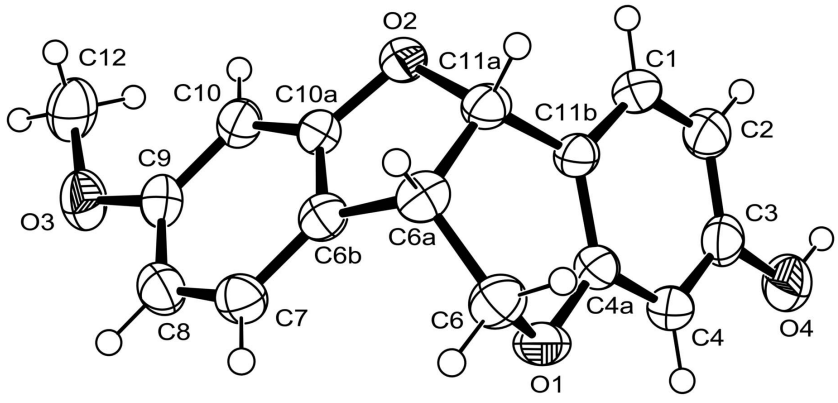

Figure 1

View of the title compound (50% probability displacement ellipsoids)

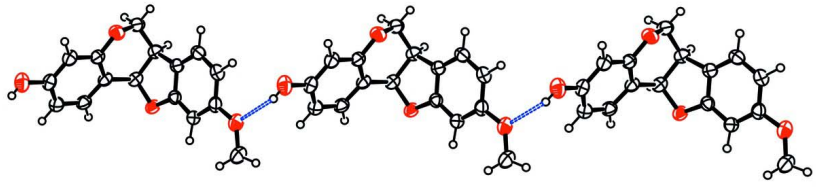

Figure 2

Packing diagram of a polymeric hydrogen bonding chain along the *c* axis.

9-Methoxy-6a,11a-dimethyl-6a,11a-dihydro-6*H*-1-benzofuro[3,2-*c*]chromen-3-ol from *Dalbergia* Oliveri

Crystal data

$C_{16}H_{14}O_4$
$M_r = 270.27$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.6289$ (3) Å
$b = 8.7963$ (4) Å
$c = 11.3150$ (5) Å
$\beta = 99.482$ (1)°
$V = 650.76$ (5) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.379$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Needle, colourless
0.40 × 0.25 × 0.20 mm

Data collection

Bruker SMART diffractometer
Radiation source: Mo
ω scans
4783 measured reflections
3198 independent reflections
1949 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 30.4°$, $\theta_{min} = 1.8°$
$h = -7 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.093$
$S = 1.09$
1949 reflections
182 parameters
1 restraint
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.0162P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0057 (2)	0.4390 (2)	0.69102 (16)	0.0414 (4)
H1	−0.1252	0.398	0.6736	0.05*
C2	0.0496 (3)	0.5393 (3)	0.78509 (16)	0.0458 (4)
H2	−0.0512	0.5664	0.8294	0.055*
C3	0.2455 (3)	0.6001 (2)	0.81356 (14)	0.0413 (4)
C4	0.3960 (3)	0.5576 (2)	0.74813 (15)	0.0399 (4)
H4	0.5278	0.5962	0.7678	0.048*
C4A	0.3482 (2)	0.45666 (19)	0.65285 (14)	0.0351 (3)
C6	0.4820 (3)	0.2767 (2)	0.53202 (17)	0.0426 (4)
H6A	0.4918	0.1964	0.5915	0.051*
H6B	0.5928	0.2632	0.4867	0.051*
C6A	0.2800 (2)	0.26311 (19)	0.44829 (15)	0.0365 (3)
H6A1	0.2669	0.1601	0.4149	0.044*
C6B	0.2447 (2)	0.37674 (19)	0.34730 (14)	0.0338 (3)

C7	0.3662 (3)	0.4270 (2)	0.26630 (15)	0.0393 (3)
H7	0.5007	0.3937	0.2723	0.047*
C8	0.2854 (3)	0.5275 (2)	0.17639 (16)	0.0424 (4)
H8	0.367	0.5633	0.123	0.051*
C9	0.0829 (3)	0.5752 (2)	0.16556 (14)	0.0388 (4)
C10	−0.0419 (3)	0.5288 (2)	0.24674 (15)	0.0374 (3)
H10	−0.1764	0.5621	0.2409	0.045*
C10A	0.0455 (2)	0.43038 (19)	0.33679 (13)	0.0332 (3)
C11A	0.0957 (2)	0.29610 (19)	0.51234 (15)	0.0363 (3)
H11A	0.0382	0.2002	0.5358	0.044*
C11B	0.1513 (2)	0.39649 (18)	0.62068 (14)	0.0343 (3)
C12	−0.1997 (3)	0.7026 (3)	0.04448 (19)	0.0579 (5)
H12A	−0.2266	0.7714	−0.0221	0.087*
H12B	−0.2447	0.7474	0.113	0.087*
H12C	−0.2719	0.6091	0.0244	0.087*
O1	0.50439 (17)	0.42138 (16)	0.59175 (11)	0.0436 (3)
O2	−0.05777 (17)	0.37602 (17)	0.42345 (10)	0.0401 (3)
O3	0.0152 (2)	0.67276 (19)	0.07158 (11)	0.0518 (4)
O4	0.2992 (2)	0.7012 (2)	0.90549 (12)	0.0550 (4)
H4A	0.2102	0.7028	0.9479	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0296 (7)	0.0524 (10)	0.0410 (8)	−0.0031 (7)	0.0021 (6)	0.0049 (8)
C2	0.0390 (8)	0.0594 (11)	0.0391 (8)	0.0030 (8)	0.0070 (7)	0.0028 (8)
C3	0.0482 (9)	0.0424 (9)	0.0321 (7)	−0.0012 (7)	0.0026 (6)	0.0048 (7)
C4	0.0368 (7)	0.0448 (9)	0.0372 (7)	−0.0089 (6)	0.0034 (6)	0.0037 (7)
C4A	0.0323 (7)	0.0385 (8)	0.0343 (7)	−0.0031 (6)	0.0045 (6)	0.0059 (6)
C6	0.0361 (7)	0.0445 (10)	0.0460 (9)	0.0054 (7)	0.0030 (7)	0.0002 (7)
C6A	0.0372 (8)	0.0295 (7)	0.0414 (8)	0.0010 (6)	0.0022 (6)	−0.0007 (6)
C6B	0.0347 (7)	0.0306 (7)	0.0354 (7)	0.0000 (6)	0.0039 (6)	−0.0039 (6)
C7	0.0361 (7)	0.0411 (8)	0.0420 (8)	0.0012 (6)	0.0102 (6)	−0.0054 (7)
C8	0.0455 (9)	0.0459 (9)	0.0379 (8)	−0.0007 (7)	0.0130 (7)	−0.0007 (7)
C9	0.0474 (9)	0.0392 (9)	0.0296 (7)	0.0014 (7)	0.0055 (6)	−0.0027 (6)
C10	0.0359 (7)	0.0423 (8)	0.0331 (7)	0.0048 (6)	0.0035 (6)	−0.0023 (6)
C10A	0.0336 (7)	0.0359 (7)	0.0301 (6)	−0.0033 (6)	0.0049 (5)	−0.0032 (6)
C11A	0.0351 (7)	0.0338 (8)	0.0384 (8)	−0.0057 (6)	0.0010 (6)	0.0056 (6)
C11B	0.0310 (6)	0.0360 (8)	0.0342 (7)	−0.0033 (6)	0.0004 (5)	0.0071 (6)
C12	0.0621 (12)	0.0648 (13)	0.0431 (9)	0.0143 (11)	−0.0022 (9)	0.0089 (9)
O1	0.0305 (5)	0.0527 (8)	0.0482 (6)	−0.0086 (5)	0.0087 (4)	−0.0075 (6)
O2	0.0299 (5)	0.0536 (7)	0.0358 (5)	−0.0032 (5)	0.0020 (4)	0.0073 (5)
O3	0.0604 (8)	0.0588 (9)	0.0363 (6)	0.0084 (7)	0.0076 (5)	0.0106 (6)
O4	0.0639 (9)	0.0619 (9)	0.0394 (7)	−0.0071 (7)	0.0088 (6)	−0.0089 (6)

Geometric parameters (Å, º)

C1—C2	1.376 (3)	C6B—C10A	1.389 (2)
C1—C11B	1.399 (2)	C7—C8	1.387 (3)
C1—H1	0.93	C7—H7	0.93
C2—C3	1.393 (3)	C8—C9	1.393 (2)
C2—H2	0.93	C8—H8	0.93
C3—O4	1.370 (2)	C9—O3	1.382 (2)
C3—C4	1.388 (3)	C9—C10	1.395 (2)
C4—C4A	1.392 (2)	C10—C10A	1.389 (2)
C4—H4	0.93	C10—H10	0.93
C4A—O1	1.372 (2)	C10A—O2	1.3709 (19)
C4A—C11B	1.400 (2)	C11A—O2	1.484 (2)
C6—O1	1.437 (2)	C11A—C11B	1.507 (2)
C6—C6A	1.512 (2)	C11A—H11A	0.98
C6—H6A	0.97	C12—O3	1.431 (3)
C6—H6B	0.97	C12—H12A	0.96
C6A—C6B	1.507 (2)	C12—H12B	0.96
C6A—C11A	1.547 (2)	C12—H12C	0.96
C6A—H6A1	0.98	O4—H4A	0.82
C6B—C7	1.389 (2)		

C2—C1—C11B	122.26 (15)	C6B—C7—H7	120.3
C2—C1—H1	118.9	C7—C8—C9	120.45 (16)
C11B—C1—H1	118.9	C7—C8—H8	119.8
C1—C2—C3	119.68 (17)	C9—C8—H8	119.8
C1—C2—H2	120.2	O3—C9—C8	116.15 (16)
C3—C2—H2	120.2	O3—C9—C10	122.38 (15)
O4—C3—C4	117.40 (16)	C8—C9—C10	121.46 (16)
O4—C3—C2	122.73 (17)	C10A—C10—C9	116.39 (15)
C4—C3—C2	119.87 (17)	C10A—C10—H10	121.8
C3—C4—C4A	119.56 (15)	C9—C10—H10	121.8
C3—C4—H4	120.2	O2—C10A—C6B	113.57 (14)
C4A—C4—H4	120.2	O2—C10A—C10	123.03 (14)
O1—C4A—C4	116.13 (14)	C6B—C10A—C10	123.39 (15)
O1—C4A—C11B	122.15 (14)	O2—C11A—C11B	108.81 (14)
C4—C4A—C11B	121.72 (14)	O2—C11A—C6A	106.09 (13)
O1—C6—C6A	112.14 (14)	C11B—C11A—C6A	112.62 (13)
O1—C6—H6A	109.2	O2—C11A—H11A	109.7
C6A—C6—H6A	109.2	C11B—C11A—H11A	109.7
O1—C6—H6B	109.2	C6A—C11A—H11A	109.7
C6A—C6—H6B	109.2	C1—C11B—C4A	116.87 (15)
H6A—C6—H6B	107.9	C1—C11B—C11A	121.33 (14)
C6B—C6A—C6	115.68 (14)	C4A—C11B—C11A	121.74 (14)
C6B—C6A—C11A	101.24 (12)	O3—C12—H12A	109.5
C6—C6A—C11A	112.20 (14)	O3—C12—H12B	109.5
C6B—C6A—H6A1	109.1	H12A—C12—H12B	109.5
C6—C6A—H6A1	109.1	O3—C12—H12C	109.5

C11A—C6A—H6A1	109.1	H12A—C12—H12C	109.5
C7—C6B—C10A	118.82 (15)	H12B—C12—H12C	109.5
C7—C6B—C6A	132.64 (14)	C4A—O1—C6	114.15 (13)
C10A—C6B—C6A	108.46 (13)	C10A—O2—C11A	106.43 (12)
C8—C7—C6B	119.42 (15)	C9—O3—C12	117.65 (15)
C8—C7—H7	120.3	C3—O4—H4A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···O3^{i}	0.82	2.07	2.882 (2)	169

Symmetry code: (i) *x*, *y*, *z*+1.