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4-Hydroxy-3,6,9-trimethyl-2,3-dihydrobenzo[*de*]chromene-7,8-dione

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Key indicators

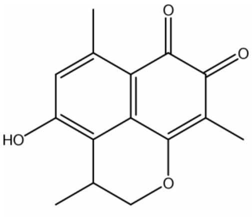
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.148
Data-to-parameter ratio = 9.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{15}H_{14}O_4$, also known as mansonone H, crystallizes in the space group $P2_12_12$ with two molecules in the asymmetric unit. In both molecules, the tetrahydropyran ring adopts an envelope conformation and the attached methyl group occupies an axial position. In the crystal structure, symmetry-related molecules are linked by O—H···O and C—H···O hydrogen bonds, to form chains along the *a* and *b* axes. They are interlinked by C—H···O hydrogen bonds to form a network.

Comment

Mansonone H, (I), was isolated from the roots of *Helicteres angustifolia* (*Sterculiaceae*), which is a known anti-inflammatory and antitumour medicine (Jiangsu New Medical Colleges, 1986). This cadinane sesquiterpenoid quinone has been previously isolated from the wood sawdust of *Mansonia altissima Chev* (Tanaka *et al.*, 1966), the root bark of *Ulmus davidiana* (Kim *et al.*, 1996) and the heartwood of *Mansonia gagei Drumm* (Tiew *et al.*, 2002). The structure of (I) was previously elucidated on the basis of spectroscopic analysis. Here we report the crystal structure of (I).



(I)

The X-ray study of (I) confirms the previously proposed molecular structure based on spectroscopic data (Fig. 1). The asymmetric unit of (I) consists of two independent molecules, *A* and *B*, linked by a C13*B*—H13*D*···O3*A* hydrogen bond (Table 1). Bond lengths and angles observed in these two molecules agree with each other. The C8*A*—C9*A* [1.523 (4) Å] and C8*B*—C9*B* [1.536 (4) Å] bond lengths are longer than the mean value of 1.478 (12) Å reported for unconjugated $Csp^2—Csp^2$ bonds by Allen *et al.* (1987). In both molecules, the tetrahydropyran ring adopts an envelope conformation and the attached methyl group occupies an axial position.

In the crystal structure, symmetry-related *A* molecules are linked by O1*A*—H1*A*···O3*A*$^{\rm i}$ and C2*A*—H2*A*···O3*A*$^{\rm ii}$ hydrogen bonds, forming chains along the *b* axis. Similarly, symmetry-related *B* molecules are linked by O1*B*—H1*B*···O3*B*$^{\rm i}$ and C2*B*—H2*B*···O3*B*$^{\rm ii}$ hydrogen bonds, forming chains along the *a* axis (symmetry codes are as in Table 1). The chains formed by molecules *A* and *B* are linked

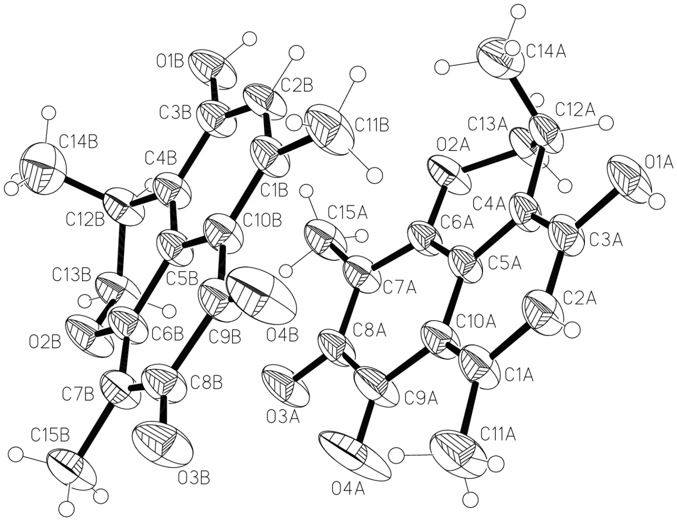


Figure 1
View of the molecules in the asymmetric unit of (I), with ellipsoids drawn at the 50% probability level.

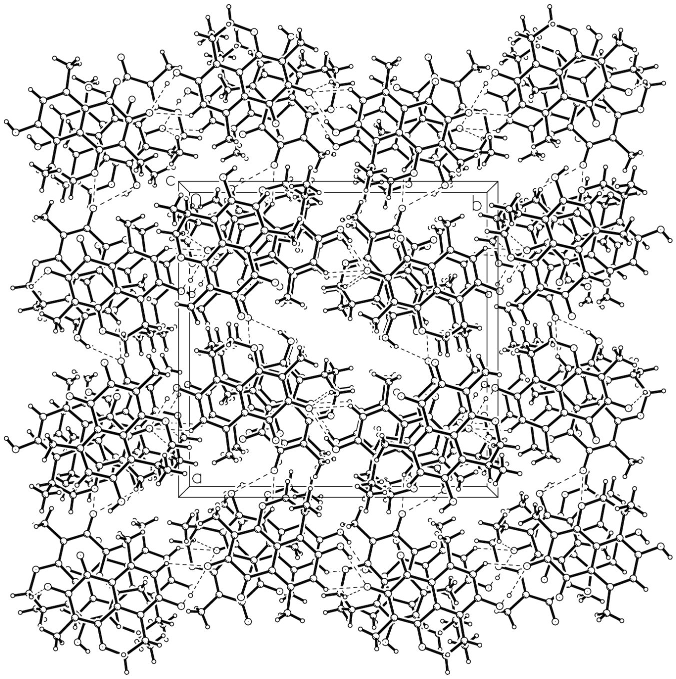


Figure 2
The molecular packing of (I), viewed down the *c* axis.

into a network by C13*B*—H13*D*···O3*A* hydrogen bonds (Fig. 2).

Experimental

Dried powder, obtained from the roots of *Helicteres angustifolia*, was extracted with EtOH. The extract was concentrated *in vacuo*, and the residue was extracted with EtOAc. The soluble portion of the EtOAc extract was subjected to silica-gel column chromatography, eluting with chloroform/methanol, to yield the title compound, mansonone H, (I). The compound identity was confirmed by the NMR spectra. Crystals of (I) were obtained from chloroform/methanol by solvent diffusion. ^{1}H NMR (500 MHz, DMSO-d_6): δ 6.78 (*s*, H5), 3.19 (*m*, H9), 4.43 (*dd*, *J* = 1.5, 5.0 Hz, H2*A*), 4.29 (*dd*, *J* = 1.5, 5.0 Hz, H2*B*), 1.83 (*s*, H9), 2.51 (*s*, H6), 1.20 (*d, J* = 7.0 Hz, H3).

Crystal data

$C_{15}H_{14}O_4$
$M_r = 258.26$
Orthorhombic, $P2_12_12$
$a = 17.917$ (1) Å
$b = 18.126$ (1) Å
$c = 7.668$ (1) Å
$V = 2490.3$ (4) Å^3
$Z = 8$
$D_x = 1.378$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 25$–31°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Plate, red
0.28 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: none
14655 measured reflections
3275 independent reflections
2108 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\rm max} = 28.3°$
$h = -23 \rightarrow 22$
$k = -15 \rightarrow 23$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.148$
$S = 1.00$
3275 reflections
344 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O3*A*$^{\rm i}$	0.82	1.97	2.785 (3)	169
O1*B*—H1*B*···O3*B*$^{\rm ii}$	0.82	2.13	2.892 (3)	154
C2*A*—H2*A*···O3*A*$^{\rm i}$	0.93	2.37	3.099 (4)	136
C2*B*—H2*B*···O3*B*$^{\rm ii}$	0.93	2.29	3.074 (4)	141
C13*B*—H13*D*···O3*A*	0.97	2.43	3.311 (5)	150

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

The H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with C—H distances in the range 0.93–0.98 Å and an O—H distance of 0.82 Å. The reflections (001) and (110) were omitted during the refinement as they fit very badly. One of the anisotropic displacement parameters (U_{33}) for the carbonyl atom O4*A* is large, indicating a possible disorder. Owing to a large fraction of weak data at higher angles, the completeness of the data is rather low. The Friedel opposites were merged during the refinement.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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