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10,12,12-Tris(3,3-dimethylallyl)-6,7-dihydroxy-2,2,9-trimethyl-1*H*-pyrano[2,3-*b*]anthracen-5(12*H*)-one

Simeon F. Kouam,[a] Bonaventure T. Ngadjui,[a] M. Iqbal Choudhary,[b] Atta-ur-Rahman,[b] Shazia Anjum[b]* and Shamsher Ali[b]

[a]Department of Chemistry, Higher Teacher Training College, University of Yaounde I, PO Box 47, Cameroon, and [b]HEJ Research Institute of Chemistry, International Centre for Chemical Sciences, University of Karachi, Karachi 75270, Pakistan

Correspondence e-mail: anjumshazia@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.048
wR factor = 0.137
Data-to-parameter ratio = 13.9

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

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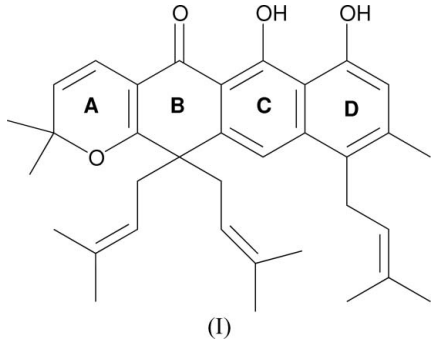
In the title compound, $C_{35}H_{42}O_4$, the chromene ring is in a distorted half-chair conformation and the cyclohexadienone ring adopts a screw-boat conformation. Intramolecular O—H···O hydrogen bonds involving the carbonyl and hydroxyl groups are observed.

Comment

Harungana madagascariensis (Hypericaceae family) is the only species of the genus *Harungana* that is found in Cameroon (Berhaut, 1975). It is an important medicinal plant in the Bamileke tribe where the plant is called Nketto. Parts of the plant are used in ethnomedicine for the treatment of jaundice, diarrhea, typhoid fever, constipation, dysentery and as a laxative and abortifacient (Berhaut, 1975; Okoli *et al.*, 2002; Prajapati *et al.*, 2003). A survey of the chemical literature of *H. madagascariensis* revealed that its stem barks and leaves have been investigated, showing a number of anthraquinones, triterpenoids (Buckley *et al.*, 1972), xanthones (Inuma *et al.*, 1995) and anthronoids (Ritchie & Taylor, 1964; Inuma *et al.*, 1995). Our previous study on this plant has yielded two new anthronoids, namely harunmadagascarin A and harunmadagascarin B (Kouam *et al.*, 2005). In this paper, we report the isolation and crystal structure of harunmadagascarin B, (I).



(I)

The bond lengths in compound (I) show normal values (Allen *et al.*, 1987). The chromene ring *A* is in a distorted half-chair conformation, with puckering parameters (Cremer & Pople, 1975) Q = 0.239 (2) Å, θ = 112.9 (4)° and φ = 136.3 (5)°. The two methyl groups are axially and bisectionally attached to the chromene ring at atom C19, with torsion angles C17—C18—C19—C20 of −94.1 (2)° and C17—C18—C19—C21 of 140.2 (2)°. The cyclohexadienone ring *B* adopts a screw-boat conformation, with Q = 0.101 (2) Å, θ = 114.0 (10)° and φ = 28.7 (10)°. The two 3-methylbut-2-enyl substituents are axially and bisectionally attached to ring C at atom C4, with torsion angles C22—C23—C4—C11 of −134.12 (14)° and C22—C23—C4—C26 of 107.81 (15)°. The naphthalene skeleton

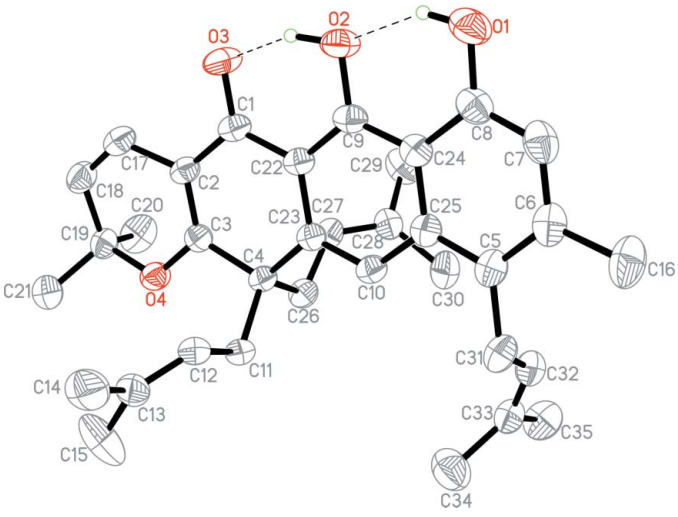


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering. For clarity, only H atoms involved in hydrogen bonding (dashed lines) are shown.

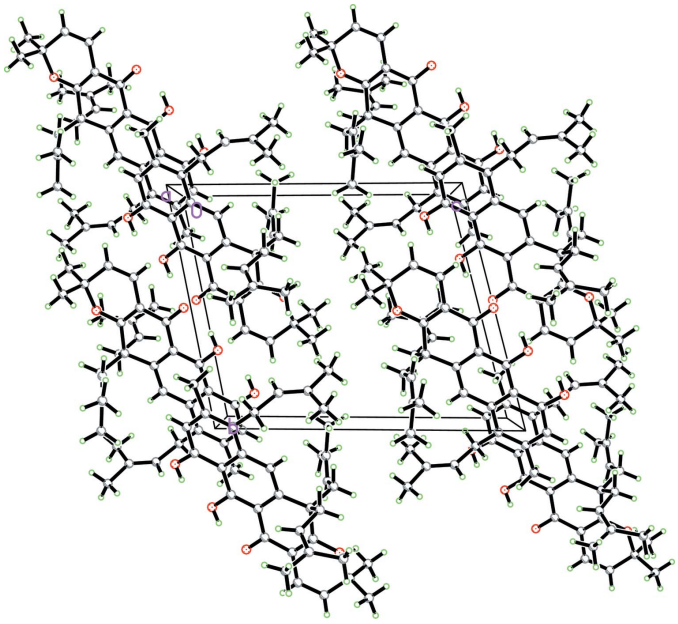


Figure 2
The packing of (I), viewed down the *a* axis.

(rings *C* and *D*) is essentially planar and the third 3-methylbut-2-enyl substituent is attached to ring *D* at C5, with C32—C31—C5—C6 = −96.10 (19)°, indicating a (−)anticlinal conformation (Fig. 1). Intramolecular O—H···O hydrogen bonds involving the carbonyl and hydroxyl groups are observed (Table 1). A view of the crystal packing is shown in Fig. 2.

Experimental

Air-dried and finely powdered stem bark (2.3 kg) of *Harungana madagascariensis* was macerated in methanol for 48 h. Filtration and evaporation yielded a crude methanol extract (250.7 g) which was re-extracted with hexane followed by ethyl acetate. Removal of the solvent yielded 50 g of hexane-soluble extract and 80.3 g of non-soluble extract. The hexane extract was subjected to column chromatography over silica gel (60 mesh) eluting with pure hexane and followed by a hexane–ethyl acetate mixture with increasing polarity. A total of 150 fractions of *ca* 200 ml each were collected and combined on the basis of thin-layer chromatography analysis. A fractions obtained with hexane–ethyl acetate (95:5 *v/v;* 15.5 g), was subjected to further column chromatography which, on elution with pure hexane, gave compound (I) (7.5 mg) which was then recrystallized from pure methanol (m.p. 395–396 K).

Crystal data

$C_{35}H_{42}O_4$
M_r = 526.69
Triclinic, $P\overline{1}$
a = 9.4790 (4) Å
b = 12.2440 (5) Å
c = 13.9979 (6) Å
α = 75.869 (1)°
β = 83.737 (1)°
γ = 70.930 (1)°
V = 1488.23 (11) Å^3
Z = 2
D_x = 1.175 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10469 reflections
θ = 1.8–25.0°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, brown
0.48 × 0.37 × 0.13 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.965, T_{max} = 0.991
14446 measured reflections
5239 independent reflections
4278 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.137
S = 1.04
5239 reflections
377 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.3259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.036
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O1*···O2	0.93 (2)	1.75 (2)	2.579 (2)	147 (2)
O2—H1*O2*···O3	0.98 (2)	1.55 (2)	2.463 (2)	154 (2)

H atoms attached to O atoms and atoms H12 and H27 (attached to C12 and C27, respectively) were located in a difference map and were refined isotropically; the range of O—H bond lengths is 0.93 (2)–0.98 (3) Å. All other H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 or 1.5 (methyl) times U_{eq} of the carrier atom. A rotating group model was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); 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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