

8-Hydroxy-7-phenyl-2,6-dioxabicyclo[3.3.1]-nonan-3-one

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.049
 wR factor = 0.147
Data-to-parameter ratio = 8.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

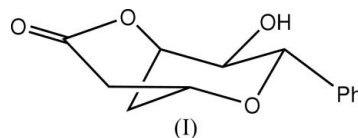
The title compound, $\text{C}_{13}\text{H}_{14}\text{O}_4$, is isolated from the plant *Goniothalamus wightii*. There are four independent molecules in the asymmetric unit, having similar conformations. Two molecules are linked *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains along the a axis in the crystal structure.

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Comment

Goniothalamus wightii Hook. f and Thomas (Annonaceae) is a medium-sized aromatic tree, growing in the Peppara Wild Life Sanctuary of the Nedumangadu forest region, Kerala, India. The plant material was collected during March 2004 and authenticated by Dr N. Mohan [Tropical Botanical Garden and Research Institute (TBGRI), Palode, Kerala, India, where a voucher specimen has been deposited]. Neither phytochemical nor biological studies on this plant are available in the literature. We are interested in the chemistry of this plant as it belongs to the genus *Goniothalamus* which is peculiar for its bioactive acetogenin and styryllactone constituents. As part of our ongoing phytochemical investigations of this plant we have isolated and performed single-crystal X-ray diffraction studies on the title compound, (I), a novel goniopyrone derivative. The structure of a diastereomer of (I) was reported by Fang *et al.* (1991). Even though the space group ($P2_1$) and unit-cell parameters of (I) were reported by Mu *et al.* (1999) [$a = 7.137$ (1) Å, $b = 35.495$ (3) Å, $c = 9.312$ (1) Å, $\beta = 91.14$ (1)°], three-dimensional structural data are not available in the literature.



There are four independent molecules in the asymmetric unit (Fig. 1), having similar conformations. They are denoted by labels a, b, c, d . The six-membered pyran ring has a chair conformation while that of the pyranone ring is half-chair. The molecules b and c are linked through fairly strong hydrogen bonds ($\text{O}3b-\text{H}31b\cdots\text{O}4c$ and $\text{O}3c-\text{H}31c\cdots\text{O}4b^i$; for symmetry code, see Table 1), forming chains along the a axis in the crystal structure (Fig. 2). The $\text{O}3d-\text{H}31d\cdots\text{O}3c^{ii}$ interaction might be a weak hydrogen bond. Molecule a is not involved in intermolecular hydrogen bonds.

Experimental

Air-dried and powdered bark (835 g) of *Goniothalamus wightii* was Soxhlet extracted with n -hexane (24 h) followed by ethyl acetate

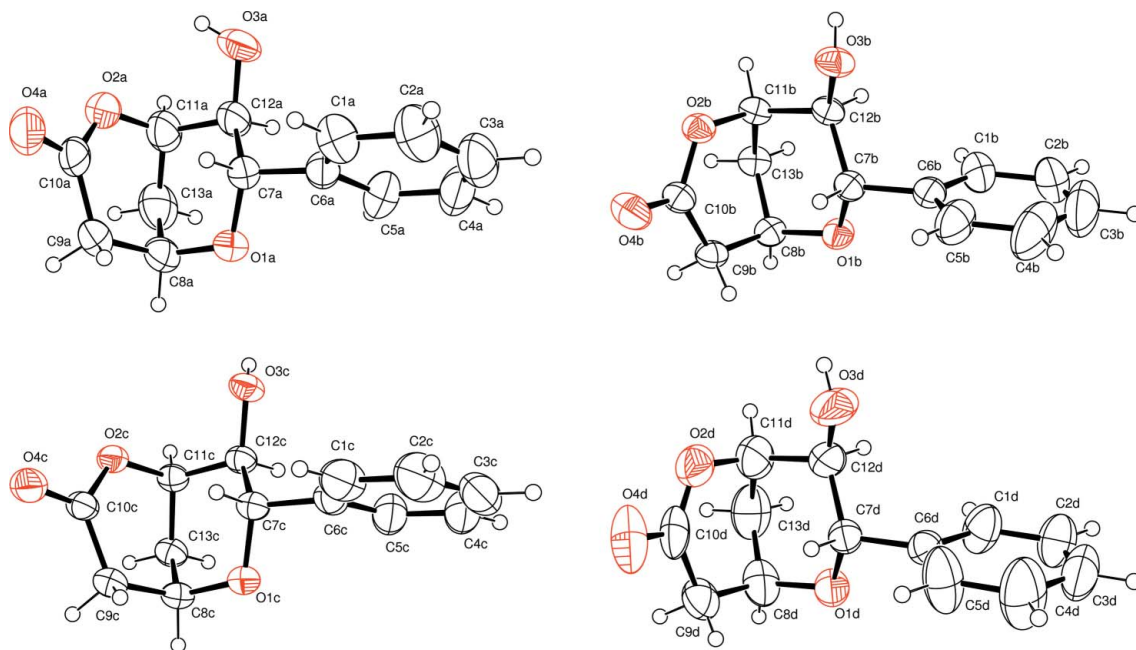


Figure 1
The molecular structures of the four independent molecules in (I), showing the atomic numbering scheme and 50% probability displacement ellipsoids.

(24 h). The ester extract was concentrated, resulting in 18 g of a gummy mass. It was then column chromatographed (20 mm × 1.5 m) through silica gel (100–200 mesh) and eluted with *n*-hexane, chloroform and their mixtures in increasing order of polarity. The compound (I) was obtained as colourless crystals from hexane–chloroform (1:1 *v/v*) fractions. Recrystallization from chloroform–methanol (1:1 *v/v*) yielded colourless prisms which were used for X-ray diffraction.

Crystal data

$C_{13}H_{14}O_4$ $Z = 8$
 $M_r = 234.24$ $D_x = 1.323 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1$ $Cu \text{ K}\alpha$ radiation
 $a = 7.1313 (5) \text{ \AA}$ $\mu = 0.81 \text{ mm}^{-1}$
 $b = 35.442 (3) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $c = 9.3061 (16) \text{ \AA}$ Prism, colourless
 $\beta = 90.454 (9)^\circ$ $0.3 \times 0.2 \times 0.2 \text{ mm}$
 $V = 2352.0 (5) \text{ \AA}^3$

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω – 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.943$, $T_{\max} = 0.999$
 (expected range = 0.802–0.850)
 4627 measured reflections
 4350 independent reflections
 3927 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 67.9^\circ$
 2 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.147$
 $S = 1.06$
 4350 reflections
 542 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.8943P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0083 (6)
 Absolute structure: Flack (1983),
 with no Friedel pairs
 Flack parameter = 0.0 (3)

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3b-H31b\cdots O4c$	0.82	1.97	2.783 (4)	173
$O3c-H31c\cdots O4b^i$	0.82	1.97	2.784 (4)	176
$O3d-H31d\cdots O3c^{ii}$	0.82	2.40	3.185 (7)	161

Symmetry codes: (i) $x - 1, y, z$; (ii) $x - 1, y, z - 1$.

Benzene rings were constrained to be ideal hexagons with Csp^2 – Csp^2 distances fixed at 1.39 \AA in order to avoid abnormal geometry. The hydroxy H atoms were located in difference Fourier maps and treated as riding with $O-H = 0.82 \text{ \AA}$, and with $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(O)$.

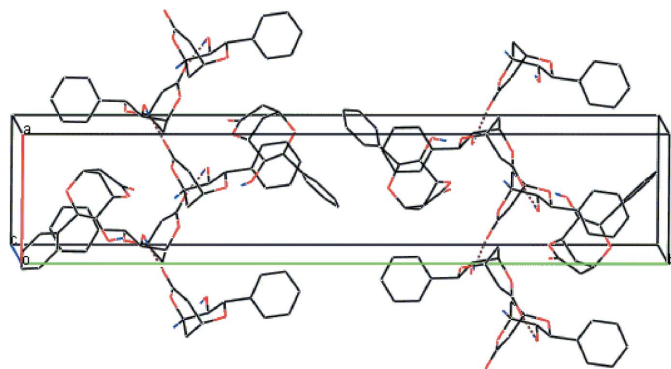


Figure 2
A packing diagram of (I). Dotted lines indicate hydrogen bonds. H atoms have been omitted.

Other H atoms were positioned geometrically and treated as riding with C–H = 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Since Friedel pairs were not measured, the refined Flack (1983) parameter is rather imprecise and does not provide unambiguous confirmation of the absolute configuration.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *WinGX* (Farrugia, 1999).

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References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Fang, X.-P., Anderson, J. E., Chang, C.-J., McLaughlin, J. L. & Fanwick, P. E. (1991). *J. Nat. Prod.* **54**, 1034–1043.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Mu, Q., Tang, W.-D., Li, C.-M., Lu, Y., Sun, H.-D., Zong, H.-L., Hao, X.-J., Zhong, Q.-T., Wu, N., Lou, L.-G. & Xu, B. (1999). *Heterocycles*, **51**, 2969–2976.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.