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Isoderrone

Jean Jules Bankeu Kezetas,[a] Bruno Ndjakou Lenta,[b]* Silvere Ngouela,[a] Etienne Tsamo,[a] Norbert Sewald,[b] Beate Neumann[b] and Hans-Georg Stammler[b]

[a]Department of Organic Chemistry, Faculty of Science, University of Yaoundé 1, PO Box 812 Yaoundé, Cameroon, and [b]Chemistry Department, Bielefeld University, PO Box 100131, 33615 Bielelеld, Germany

Correspondence e-mail: lentabruno@yahoo.fr

Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.117
Data-to-parameter ratio = 16.1

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

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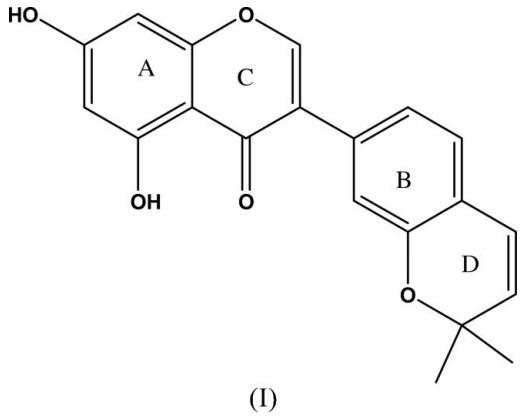
The title compound, $C_{20}H_{16}O_5$, also known as 5,7-dihyroxy-3-(2′,2′-dimethyl-2H'-chromen-7′-yl)chroman-4-one, was isolated from the medicinal plant *Ficus mucuso*. The benzopyranone (chromen-4-one) ring system is essentially planar and the dihedral angle between the benzopyranone ring system and the other benzene ring is 37.7 (10)°. In the crystal structure, one-dimensional chains are formed *via* intermolecular O—H···O hydrogen bonds.

Comment

Ficus species are widely used in traditional medicine in Cameroon. They are known as a source of biologically active compounds (Peraza-Sanchez *et al.*, 2002; Noumi & Fozi, 2003; Sandabe *et al.*, 2006). A mixture of powdered leaves of *Ficus mucuso* (Leguminosae) and palm oil is used in the treatment of epilepsy in Fongo Tongo, a village of the western Province of Cameroon (Noumi & Fozi, 2003). A decoction of stem bark is used in by Baka pygmies to treat jaundice (Betti, 2004). To the best of our knowledge, no phytochemical study has been carried out on this species. As part of our continuing phytochemical investigation of Cameroonian medicinal plants, we have examined the fruits of *Ficus micuso* and isolated isoderrone and alpinumisoflavone as major constituents. Isoderrone was previously isolated from other species of Leguminosae, such as *Lupinus albus* (Tahara *et al.*, 1989), *Ulex parviflorus* (Maximo & Lourenço, 1998), *Genista cortisa* (Pistelli *et al.*, 2000) and *Erythrina vogelii* (Emerson *et al.*, 2002). Although the structure of isoderrone has already been determined by spectroscopic methods (Pistelli *et al.*, 2000), we report here the single-crystal X-ray structure of isoderrone.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The isoflavone part of the molecule consists of three six-membered

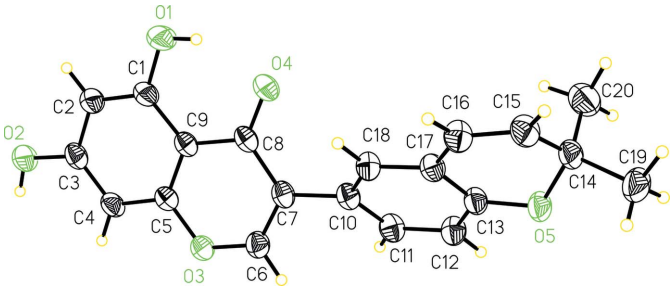


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

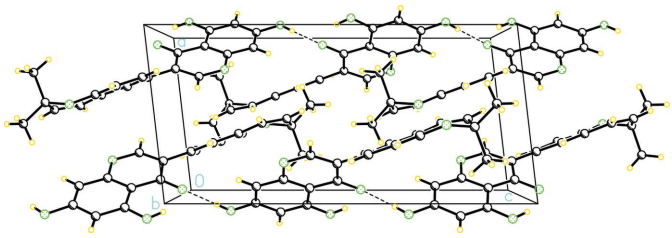


Figure 2
Part of the crystal structure of (I) showing intermolecular hydrogen bonds as dashed lines.

rings *A*, *C* and *B*. Ring *B* is fused to ring *D* to form a dimethylchromene unit. The benzopyranone fragment *A*/*C* and ring *B* are each essentially planar (the average deviations of contributing atoms from the least-squares planes of each system are 0.0246 and 0.0294 Å, respectively). The six-membered ring *D* is in a distorted half-chair conformation, with puckering parameters (Cremer & Pople, 1975) Q = 0.3319 (2) Å, θ = 114.34 (11)° and φ =145.55 (11)°. The dihedral angle between the benzopyranone system *A*/*C* and benzene ring *B* is 37.7 (10)°.

The O1-hydroxy group has a *gauche* arrangement, with an H1—O1—C1—C9 torsion angle of −0.53°, giving rise to a short intramolecular contact (Table 2). In the crystal structure, intermolecular O—H···O hydrogen bonds link molecules into one-dimensional chains propagating in the *c*-axis direction.

Experimental

Leaves of *Ficus mucuso* (Moraceae) were collected at Ngoa-Ekelle Yaoundé in the central province of Cameroon and identified by M. Nana Victor, Botanist at the National Herbarium of Cameroon, where the specimen is deposited under n°41204.

The air-dried powdered leaves of *Ficus mucuso* were extracted with hexane at room temperature and the extract concentrated to dryness to obtain a viscous residue (3.90 g).

The hexane extract residue was subjected to flash column chromatography over silica gel (230–400 mesh) as stationary phase, eluting with hexane–EtOAc of increasing polarity to yield alpinumisoflavone (160 mg) and isoderrone (300 mg) as major products. X-ray quality crystals were grown from a hexane solution of the title compound.

Crystal data

$C_{20}H_{16}O_5$
M_r = 336.33
Monoclinic, $P2_1/c$
a = 7.9241 (2) Å
b = 12.4116 (3) Å
c = 16.4595 (3) Å
β = 96.5733 (13)°
V = 1608.15 (6) Å^3
Z = 4
D_x = 1.389 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 220 (2) K
Rhomb, yellow
0.30 × 0.30 × 0.30 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.971, T_{max} = 0.971
38284 measured reflections
3678 independent reflections
2962 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.117
S = 1.06
3678 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.2171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3496 (14)	C5—C9	1.4029 (15)
O2—C3	1.3530 (13)	C6—C7	1.3498 (16)
O3—C6	1.3468 (13)	C7—C8	1.4535 (17)
O3—C5	1.3698 (14)	C7—C10	1.4867 (15)
O4—C8	1.2661 (13)	C10—C11	1.3984 (17)
O5—C13	1.3655 (13)	C13—C17	1.4004 (17)
O5—C14	1.4675 (14)	C14—C15	1.5039 (18)
C1—C2	1.3733 (16)	C14—C19	1.5166 (18)
C6—O3—C5	119.15 (9)	C6—C7—C10	119.71 (11)
O1—C1—C2	118.73 (11)	O4—C8—C9	120.66 (11)
O1—C1—C9	120.40 (10)	O4—C8—C7	122.81 (10)
O2—C3—C4	122.20 (10)	O5—C13—C12	118.85 (11)
O2—C3—C2	116.38 (11)	O5—C13—C17	121.02 (10)
O3—C5—C4	116.61 (10)	O5—C14—C15	110.67 (9)
O3—C6—C7	125.57 (11)	C16—C15—C14	121.41 (11)
C6—C7—C8	118.02 (10)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4	0.83	1.86	2.5995 (15)	147
O2—H2···O4^{i}	0.83	1.86	2.6842 (16)	170

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

H atoms were positioned geometrically and treated as riding with C—H = 0.94 or 0.97 Å and O—H = 0.83 Å, and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl},O).

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990);

program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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References

Betti, J. L. (2004). *African Study Monographs*, **25**, 1–27.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Emerson, F. Q., Kagoyire, K. A., Terreaux, C., Antus, S. & Hostettmann, K. (2002). *J. Nat. Prod.* **65**, 403–406.
Maximo, P. & Lourenço, A. (1998). *Phytochemistry*, **48**, 359–362.
Nonius (2002). *COLLECT*. Nonius BV, Delft, The Netherlands.
Noumi, E. & Fozi, F. L. (2003). *Pharm. Biol.* **41**, 330–339.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Peraza-Sanchez, S. R., Chai, H. B., Shin, Y. G., Santisuk, T., Reutrakul, V., Farnsworth, N. R., Cordell, G. A., Pezzuto, J. M. & Kinghom, A. D. (2002). *Planta Med.* **68**, 186–188.
Pistelli, L., Giachi, I., Potenza, D. & Morelli, I. (2000). *J. Nat. Prod.* **63**, 504–506.
Sandabe, U. K., Onyeyili, P. A. & Chibuzo, G. A. (2006). *J. Ethnopharmacol.* **104**, 283–285.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Gottingen, Germany.
Siemens (1994). *XP* in *SHELXTL*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tahara, S., Orihara, S., Ingham, J. L. & Mizutan, J. I. (1989). *Phytochemistry*, **28**, 90–911.