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Tricoccin R2

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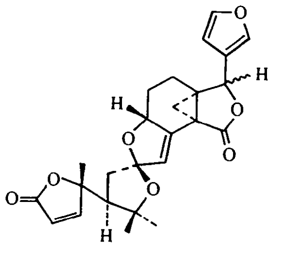
Abstract

In the title compound, (3′*S*-{3′α,3′$\alpha\beta$,5′$\alpha\alpha$,7′α[*R**-(*R**)],8′bβ}-4-(2,5-dihydro-2-methyl-5-oxo-2-furanyl)-3′-(3-furanyl)-4,5,5′,5′α-tetrahydro-5,5-dimethylspiro-[furan-2(3*H*),7′(4′*H*)-1*H*,3*H*-3α,8*b*]methanobenzo[1,2-*b*:3,4-*c*′]difuran)-1′-one, $C_{25}H_{26}O_7$, some C—C bonds and C—C—C angles deviate significantly from their expected values. Both the terminal five-membered rings are nearly planar. The fused five-membered rings, *B*, *C* and *E*, have half-chair, distorted envelope and half-chair conformations, respectively. The six-membered ring is in a slightly distorted sofa conformation. The structure is stabilized by van der Waals interactions.

Comment

Tricoccin R2, (I) was isolated by Epe & Mondon (Herz, Grisebach, Kirby, 1983) from *Cneorum tricoccin L.*, a shrub native to coastal areas of the western Mediterranean with hairless leaves, yellow blossoms and red fruits.



(I)

Some C—C bonds and C—C—C angles involved in the fused ring systems with axial substitutions of heavy bulky groups deviate by more than the 3σ level from their expected values (Engh & Huber, 1991). Two factors seem to be responsible for these deviations: (1) the degree of substitution at the C atom and (2) the presence of steric strain in the structure (Hall & Maslen, 1965; Gzella, Zaprutko, Wrezciono & Jaskólski, 1987; Sekar, Parthasarathy, Kundu & Barik, 1992, 1993). Rings *A* and *F* are planar within 2σ. Rings *B*, *C* and *E* are in half-chair, distorted envelope and half-chair conformations, respectively. Ring *D* is in a slightly distorted sofa conformation with a mean torsion angle of 33.5 (7)°. Puckering is pronounced near C11 and less so near C14. Packing of the molecules in the unit cell is stabilized by van der Waals interactions.

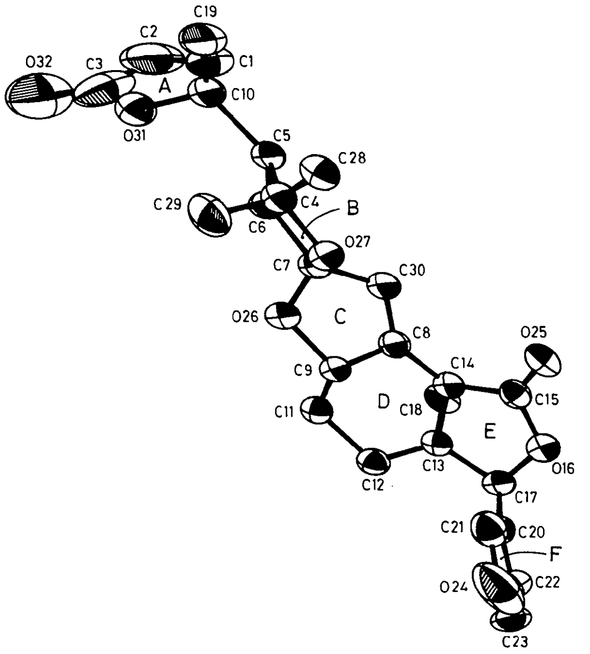


Fig. 1. Molecular structure with ellipsoids at the 50% probability level.

Experimental

Crystals were grown at room temperature from an ethanol/acetone mixture.

Crystal data

$C_{25}H_{26}O_7$
$M_r = 438.48$
Monoclinic
$C2$
$a = 22.939\ (1)$ Å
$b = 6.574\ (2)$ Å
$c = 16.481\ (2)$ Å
$\beta = 114.67\ (1)°$
$V = 2259\ (2)$ Å^3
$Z = 4$
$D_x = 1.29$ Mg m^{-3}

Cu *K*α radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20–30°$
$\mu = 0.739$ mm^{-1}
$T = 293$ K
Needle
0.30 × 0.25 × 0.20 mm
Colourless

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‡ Contribution No. 851.

Data collection

Enraf–Nonius CAD-4 diffractometer	1996 observed reflections $[I \geq 3.0\sigma(I)]$
θ–2θ scans	$R_{\rm int} = 0.027$
Absorption correction: empirical, ψ scan	$\theta_{\rm max} = 70°$
$T_{\rm min} = 0.961$, $T_{\rm max} = 0.991$	$h = -26 \rightarrow 26$
	$k = 0 \rightarrow 8$
	$l = 0 \rightarrow 27$
2312 measured reflections	2 standard reflections
2230 independent reflections	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{\rm max} = 0.06$
$R = 0.049$	$\Delta\rho_{\rm max} = 0.298$ e Å^{-3}
$wR = 0.058$	$\Delta\rho_{\rm min} = -0.424$ e Å^{-3}
$S = 1.08$	Extinction correction: none
1996 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), Vol. IV, Table 2.2B)
392 parameters	
$w = 1/[\sigma^2(F) + 0.004351F^2]$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.4996 (3)	0.3367 (13)	0.1535 (3)	0.086 (2)
C2	0.5369 (4)	0.3187 (24)	0.1109 (4)	0.132 (5)
C3	0.5549 (3)	0.1046 (29)	0.1167 (3)	0.152 (7)
C4	0.5110 (2)	−0.0331 (9)	0.3509 (2)	0.049 (1)
C5	0.5155 (2)	0.1504 (8)	0.2947 (2)	0.045 (1)
C6	0.5859 (2)	0.2098 (9)	0.3415 (2)	0.054 (2)
C7	0.6052 (2)	0.1500 (8)	0.4406 (2)	0.049 (1)
C8	0.6672 (2)	0.2698 (8)	0.5839 (2)	0.043 (1)
C9	0.6970 (2)	0.0756 (8)	0.5694 (2)	0.044 (1)
C10	0.4897 (2)	0.1402 (9)	0.1906 (2)	0.053 (2)
C11	0.7689 (2)	0.0942 (9)	0.6017 (2)	0.051 (1)
C12	0.7992 (2)	0.1337 (9)	0.7048 (2)	0.050 (1)
C13	0.7687 (2)	0.3112 (8)	0.7325 (2)	0.046 (1)
C14	0.7005 (2)	0.3743 (8)	0.6719 (2)	0.042 (1)
C15	0.6691 (2)	0.4274 (8)	0.7337 (2)	0.049 (2)
O16	0.7131 (1)	0.4224 (7)	0.8206 (2)	0.059 (1)
C17	0.7742 (2)	0.3347 (8)	0.8297 (2)	0.049 (1)
C18	0.7574 (2)	0.5110 (9)	0.6846 (3)	0.060 (1)
C19	0.4207 (2)	0.0713 (11)	0.1437 (3)	0.067 (2)
C20	0.7852 (2)	0.1452 (9)	0.8843 (2)	0.053 (2)
C21	0.7453 (2)	−0.0300 (10)	0.8696 (3)	0.068 (2)
C22	0.8365 (3)	0.1102 (11)	0.9621 (3)	0.076 (2)
C23	0.8346 (3)	−0.0704 (11)	0.9983 (3)	0.072 (2)
O24	0.7819 (5)	−0.1497 (13)	0.9468 (6)	0.133 (6)
O25	0.6141 (1)	0.4674 (8)	0.7145 (2)	0.066 (1)
O26	0.6653 (1)	0.0426 (7)	0.4734 (2)	0.055 (1)
O27	0.5551 (1)	0.0250	0.4412 (2)	0.060 (1)
C28	0.4469 (2)	−0.0559 (11)	0.3558 (3)	0.066 (2)
C29	0.5331 (4)	−0.2345 (11)	0.3272 (4)	0.084 (2)
C30	0.6163 (2)	0.3144 (9)	0.5104 (2)	0.051 (1)
O31	0.5280 (2)	−0.0026 (10)	0.1646 (2)	0.087 (2)
O32	0.5862 (2)	0.0030 (23)	0.0846 (3)	0.0226 (7)

Table 2. *Selected geometric parameters (Å, °)*

C4—C5	1.550 (7)	C11—C12	1.566 (5)
C5—C10	1.566 (5)	C13—C17	1.561 (5)
C6—C7	1.554 (5)		
C2—C3—O32	133.3 (9)	C14—C15—O25	128.3 (4)
C4—C5—C10	122.2 (4)	O16—C15—O25	121.3 (5)
C14—C8—C30	133.5 (5)	C17—C20—C22	125.1 (6)
C12—C13—C17	122.2 (4)	C17—C20—C21	129.4 (5)
C8—C14—C15	124.4 (5)		

Ring *B*	
C4—C5—C6—C7	−30.3 (6)
C5—C6—C7—O27	13.4 (6)
C6—C7—O27—C4	10.6 (6)
C7—O27—C4—C5	−29.6 (6)
O27—C4—C5—C6	36.3 (6)
Ring *C*	
C7—C30—C8—C9	2.4 (7)
C30—C8—C9—O26	12.3 (7)
C8—C9—O26—C7	−22.7 (6)
C9—O26—C7—C30	23.8 (6)
O26—C7—C30—C8	−16.4 (7)
Ring *D*	
C8—C9—C11—C12	62.5 (6)
C9—C11—C12—C13	−52.3 (7)
C11—C12—C13—C14	24.0 (8)
C12—C13—C14—C8	−4.3 (8)
C13—C14—C8—C9	13.9 (8)
C14—C8—C9—C11	−44.1 (7)
Ring *E*	
C13—C14—C15—O16	6.4 (7)
C14—C15—O16—C17	−9.7 (7)
C15—O16—C17—C13	8.8 (7)
O16—C17—C13—C14	−4.3 (6)
C17—C13—C14—C15	−0.9 (6)

Refinement was by the full-matrix least-squares method. H atoms were refined isotropically taking the starting values of the displacement parameters to be those of the heavy atoms to which they are covalently bonded.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Program used to determine the space group: *STATCW* (Sekar, 1991). Program used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Program used to calculate the molecular parameters: *PARST* (Nardelli, 1983). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1017). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Methyl-3-(1-phenylethylidene)-2,3-dihydrobenzo[*b*]furan-2-one

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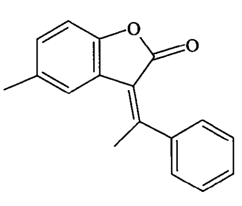
Abstract

The benzofuran ring in the title compound, $C_{17}H_{14}O_2$, is planar with a methyl group substituted in the 5-position. The 5-methyl and 3-ethylidene groups are in *trans* positions with respect to the benzofuran ring. The structure is stabilized by intermolecular van der Waals interactions.

Comment

The title compound, (I), was prepared as part of a study of diastereoselectivities of free-radical reactions. The crystal structure was determined in order to establish whether a phenylethylidene group would be formed *trans* or *gauche* to the benzofuran ring.



(I)

The benzofuran ring is planar (with a maximum deviation from the plane of 0.75 Å for C8) and shows typical aromaticity and delocalization of π electrons. The phenyl ring is twisted out of the plane of the benzofuran moiety by 130°. The 5-methyl and ethylidene groups are in *trans* positions with respect to the benzofuran ring. Bond lengths and bond angles are as expected.

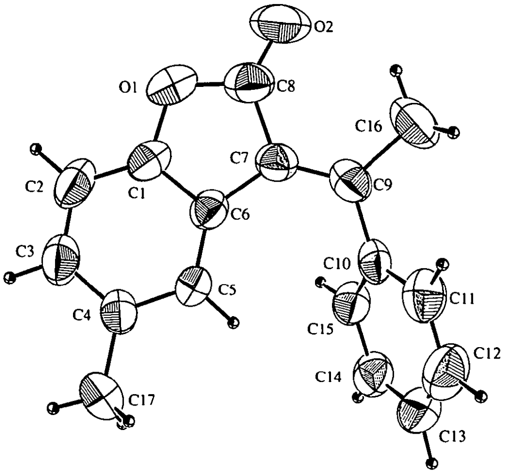


Fig. 1. View of the title molecule with displacement ellipsoids plotted at the 50% probability level.

Experimental

Crystal supplied by S. Selladurai.

Crystal data

$C_{17}H_{14}O_2$
M_r = 250.3
Triclinic
$P\bar{1}$
a = 9.890 (5) Å
b = 9.258 (4) Å
c = 8.878 (5) Å
α = 108.26 (4)°
β = 78.48 (4)°
γ = 67.38 (4)°
V = 663.3 (6) Å^3
Z = 2
D_x = 1.253 Mg m^{-3}
D_m = 1.249 Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11.5–15°
μ = 0.0757 mm^{-1}
T = 293 K
Block
0.35 × 0.29 × 0.12 mm
Red

Data collection

Syntex $P3$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1901 measured reflections
1705 independent reflections
913 observed reflections [$I > 3\sigma(I)$]

θ_{max} = 25°
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 10$
$l = -10 \rightarrow 10$
3 standard reflections monitored every 97 reflections intensity decay: <5%

Refinement

Refinement on F
R = 0.063
wR = 0.083
S = 1.271
913 reflections
214 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.58
$\Delta\rho_{max}$ = 2.2 e Å^{-3}
$\Delta\rho_{min}$ = 0.6 e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Mann (1968)