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Reinvestigation of seselin

A. K. Bauri,[a] Sabine Foro,[b] Hans-Jörg Lindner[b] and Sandip K. Nayak[c]*

[a]Bioorganic Division, Bhabha Atomic Research Centre, Trombay, Mumbai 400 085, India, [b]Clemens Schöpf-Institut für Organische Chemie und Biochemie, Technische Universität Darmstadt, Petersenstrasse 22, D-64287 Darmstadt, Germany, and [c]Bio-organic Division, Bhabha Atomic Research Centre, Trombay, Mumbai 400 085, India

Correspondence e-mail: sknayak@magnum.barc.ernet.in

Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.119
Data-to-parameter ratio = 10.5

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

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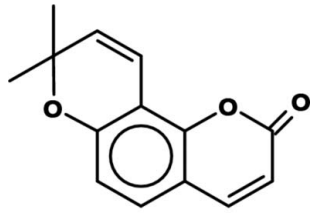
The crystal structure of seselin (2′,2′-dimethyl-3-pyreno[6,5:7,8]coumarin), $C_{14}H_{12}O_3$, a bioactive pyrenocoumarin, has been reinvestigated. The coumarin ring system is nearly planar and the α-pyran ring adopts a distorted half-chair conformation. An intermolecular C—H···O hydrogen bond is observed.

Comment

Seselin, a bioactive pyrenocoumarin (Shanghag *et al.*, 1964; Smith *et al.*, 1957; Stanley & Vannier, 1957; Huang *et al.*, 1994), has been isolated from a methanol extract of the seeds of *Trachyspermum stictocarpum* (Ajmoda in Hindi). There is no previous phytochemical report on this plant in the literature. Several earlier publications dealing with its isolation from different natural sources (Austin *et al.*, 1968; Tomer *et al.*, 1969), structure elucidation (Shanghag *et al.*, 1967), synthesis (Schroeder *et al.*, 1959) and bioactivity studies (Huang *et al.*, 1994; Smith *et al.*, 1957) have appeared. From this perspective, our discovery of *T. stictocarpum* as a rich source of medicinally important pyranocoumarin is significant. The compound is credited with various medicinal attributes such as vasodilatory (Shanghag *et al.*, 1967), antitumor and anti-HIV activities (Huang *et al.*, 1994). This paper deals with the reinvestigation and improved refinement of the crystal structure of this compound (Kato, 1970), isolated from *T. stictocarpum*.



(I)

As shown in Fig. 1, the coumarin ring system is nearly planar, the largest deviation from the least-squares mean plane being 0.036 (1)Å at C1, and the α-pyran ring adopts a distorted half-chair conformation.

In the crystal structure, the molecules are linked through intermolecular C—H···O hydrogen bonds, as shown in the packing diagram (Fig. 2). Details of the hydrogen bonding are given in Table 1.

Experimental

The title compound was isolated as a major product from a methanol extract of *Trachyspermum stictocarpum* by column chromatography

over silica gel with gradient elution by changing the polarity of the ethyl acetate–petroleum ether solvent system. Crystals suitable for X-ray diffraction were obtained by recrystallization from benzene–hexane (1:4) at room temperature by evaporation.

Crystal data

$C_{14}H_{12}O_3$
M_r = 228.24
Monoclinic, $P2_1/c$
a = 8.428 (1) Å
b = 11.112 (2) Å
c = 12.324 (2) Å
β = 103.08 (1)°
V = 1124.2 (3) Å^3
Z = 4
D_x = 1.348 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5.4–27.7°
μ = 0.78 mm^{-1}
T = 299 (2) K
Prism, light yellow
0.80 × 0.38 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3435 measured reflections
2004 independent reflections
1834 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 66.9°
$h = -9 \rightarrow 10$
$k = -13 \rightarrow 7$
$l = -14 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 0.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
S = 1.12
2004 reflections
191 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 0.1549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.082 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O3^{i}	0.947 (18)	2.400 (18)	3.2710 (17)	152.9 (14)

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

All H atoms were located in a difference map and refined with isotropic displacement parameters [$1.2U_{eq}$(parent atom)].

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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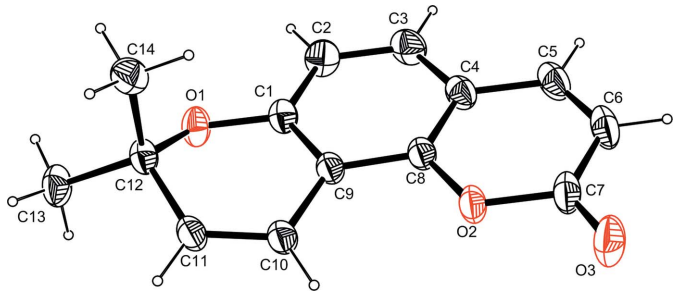

Figure 1
The molecular structure of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

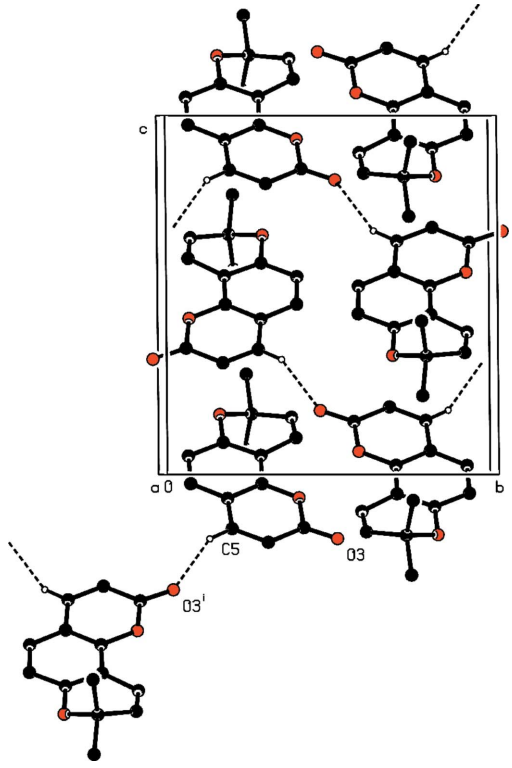

Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $-x, y-\frac{1}{2}, -z-\frac{1}{2}$.]

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