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1-(2-Naphthalenyl)-3-(3-nitrophenyl)-2-propen-1-one

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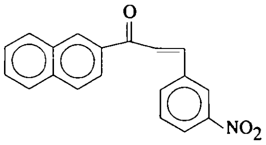
Abstract

Molecules of $C_{19}H_{13}NO_3$ have an *s-cis* conformation for the ketone system, with an O═C—C—C torsion angle of −14.9 (3)°, and are nearly planar.

Comment

Chalcones derived from acetonaphthones are potential germicides, fungicides and anticarcinogens (Misra *et al.*, 1971). The carbonyl system plays an important role in the antibacterial activity in chalcones (Pinaka & Edwards, 1963). The present structural study was undertaken to establish the conformation of the propenone group in the title compound, (I).



(I)

A drawing of the molecule with atomic numbering is shown in Fig. 1. The molecule assumes an *s-cis* conformation for the ketone system, defined by the torsion angle O20—C11—C12—C13 of −14.9 (3)°. A similar result was observed for α,β-unsaturated ketone systems by Tokuno *et al.* (1986) and by Eswaramoorthy *et al.* (1994). The C11═O20 bond length [1.233 (2) Å] is comparable with values reported by Zabel *et al.* (1980) and Shanmuga Sundara Raj *et al.* (1996, 1997). It is interesting to note that the whole molecule is nearly planar, as can be seen from the torsion angles C10—C1—C2—C11 −178.3 (1), C2—C11—C12—C13 165.7 (2), C11—C12—C13—C14 −176.7 (1) and C12—C13—C14—C19 12.8 (2)°.

The nitro group shows a tendency to be coplanar with the phenyl-ring system. The relevant torsion angles are C15—C16—N21—O22 179.3 (2), C15—C16—N21—O23 0.1 (2), C17—C16—N21—O22 1.8 (2) and C17—C16—N21—O23 −177.4 (2)°.

The naphthalene ring is planar and makes an angle of 2.2 (1)° with the nitrophenyl ring. The short contact H3···H12 [1.78 (4) Å] could be relieved by opening the C2—C11—C12 angle or by rotation around the C2—C11 bond, but is not so here. In contrast, the contact between H12···H19 [2.09 (3) Å] involves an opening of the C12—C13—C14 angle [to 124.83 (15)°] and slight rotation of the nitrophenyl group around the C13—C14 bond by *ca* 13°. The molecules run in a linear fashion along the **c** direction. The structure is stabilized by van der Waals interactions.

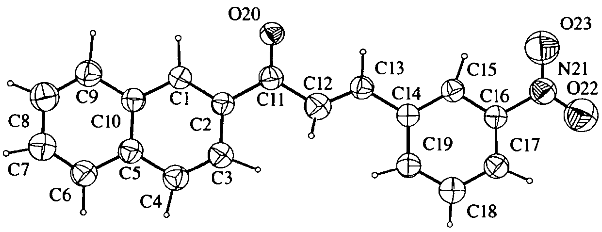


Fig. 1. *ORTEP*II (Johnson, 1976) plot of $C_{19}H_{13}NO_3$ showing 50% probability displacement ellipsoids.

Experimental

The title compound was synthesized according to the procedure of Bonsingnore *et al.* (1976). A solution of sodium hydroxide (2 g) in water (20 ml) and a solution of *m*-nitrobenzaldehyde (5.4 g) in ethanol (20 ml) were added simultaneously to a well stirred solution of methyl β-naphthyl ketone (6 g) in ethanol (25 ml). The stirring was continued for 3 h. The solid product which formed was collected on a Buchner funnel, washed thoroughly with water and then with ethanol. The crude sample was air dried and recrystallized twice (m.p. 453–454 K; yield 60%).

Crystal data

$C_{19}H_{13}NO_3$
$M_r = 303.30$
Monoclinic
$P2_1/c$
$a = 10.317\ (2)$ Å
$b = 5.864\ (4)$ Å
$c = 24.582\ (1)$ Å
$\beta = 100.15\ (2)°$
$V = 1463.9\ (10)$ Å^3
$Z = 4$
$D_x = 1.376$ Mg m^{-3}
D_m not measured

Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 23 reflections
$\theta = 13–24°$
$\mu = 0.766$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.24 × 0.22 × 0.16 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/2θ scans
Absorption correction: none

$\theta_{\max} = 70°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 7$
$l = -29 \rightarrow 29$

2924 measured reflections
2763 independent reflections
2537 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.072$

3 standard reflections every 200 reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.118$
$S = 1.079$
2763 reflections
260 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.012$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C11—O20	1.233 (2)	C13—C14	1.472 (2)
C11—C12	1.481 (2)	N21—O23	1.218 (2)
C12—C13	1.325 (2)	N21—O22	1.216 (2)
C3—C2—C11	121.87 (13)	C12—C11—C2	118.40 (14)
O20—C11—C12	120.72 (14)	C13—C12—C11	123.2 (2)
O20—C11—C2	120.88 (13)	C12—C13—C14	124.83 (15)
C1—C2—C11—O20	−1.0 (2)	O20—C11—C12—C13	−14.9 (3)
C3—C2—C11—O20	179.1 (1)	C2—C11—C12—C13	165.7 (2)
C1—C2—C11—C12	178.4 (1)	C11—C12—C13—C14	−176.7 (1)
C3—C2—C11—C12	−1.5 (2)	C12—C13—C14—C19	12.8 (2)

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1335). Services for accessing these data are described at the back of the journal.

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4-Methyl-7,10-dihydrobenzo[*h*]coumarin and 4-Methyl-7,8,9,10-tetrahydro-8,9-epoxybenzo[*h*]coumarin

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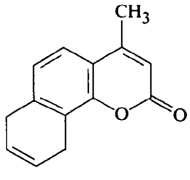
Abstract

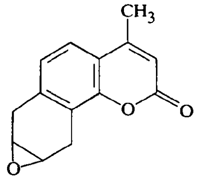
The coumarin rings in the title molecules, $C_{14}H_{12}O_2$ and $C_{14}H_{12}O_3$, are planar. In the epoxide derivative, the tetrahydrobenzene ring adopts a boat conformation and the oxirane plane is nearly perpendicular to the coumarin moiety.

Comment

Coumarin derivatives are used as laser dyes (Maeda, 1984). Some of them are found in natural products and exhibit antifungal and anticoagulant properties (Parrish *et al.*, 1974; Troste & Toste, 1996). The role of arene oxides in biological systems continues to attract attention due to their cytotoxicity, mutagenicity and carcinogenicity (Boyd & Sharma, 1996; Boyd & Jerina, 1985). The crystal structure determination of the title compounds, 4-methyl-7,10-dihydrobenzo[*h*]coumarin [$C_{14}H_{12}O_2$, (I)] and 4-methyl-7,8,9,10-tetrahydro-8,9-epoxybenzo[*h*]coumarin [$C_{14}H_{12}O_3$, (II)], was undertaken as part of structural studies on coumarin derivatives.



(I)



(II)

Bond lengths and angles in the coumarin rings are normal and are in agreement with those observed in other coumarin derivatives (Chinnakali *et al.*, 1992; Yip *et al.*, 1996). In (I), C13—C14 [1.324 (3) Å] shows double-bond character and the bond lengths C12—C13 [1.459 (3) Å] and C14—C15 [1.474 (2) Å] are shorter

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