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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1042). Services for accessing these data are described at the back of the journal.

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4-(4-Methylbenzoyl)-6-(4-methylbenzylidene)-3-phenyl-2-oxa-3-azabicyclo[3.3.0]-oct-7-ene

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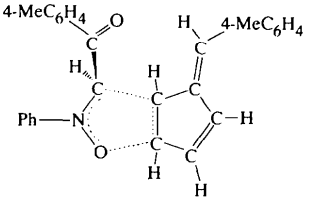
Abstract

The title compound, $C_{28}H_{25}NO_2$, is a minor product resulting from a 1,3-dipolar nitrone–fulvene [3+2] cycloaddition.

Comment

In the course of our work on periselectivity of [3+2] cycloadditions of *C*-aroyl-*N*-phenylnitrones on 6-arylfulvenes we have already shown that the major product (60% yield) results from an unexpected approach of the nitrone dipole on the side of dipolarophile fulvene close to the bulky aryl substituent (Kubicki *et al.*, 1998). By varying the substituents on both the nitrone and the fulvene reactants we were able to isolate and crystallize the minor adduct (40%), (1). Cycloaddition involves one of the two fulvenic double bonds, as expected. In contrast to the major product already described, cycloaddition leading to the minor product occurs on the less hindered side of the fulvene. Significant bond lengths are C2—C3 1.522 (4) and C4—C5 1.326 (4) Å.



(1)

The central saturated cycle is puckered and exhibits a 'boat-like' geometry over the planar C1—C2—C3—O1 unit. The dihedral angles involving this unit and the O1—N—C1 and C2–C6 planes are equal to 39.2 (2) and 58.4 (2)°, respectively. The fulvene derived fragment in (1) is roughly planar with the dihedral angle between the C2–C6 and C8–C13 planes equal to 19.9 (2)°. A perspective view of the title molecule is shown in Fig. 1.

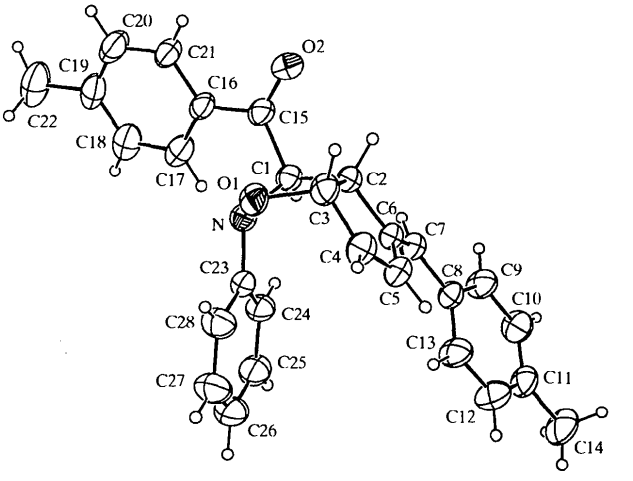


Fig. 1. The molecular structure of (1) showing 30% probability displacement ellipsoids.

Experimental

A mixture of 3 mmol of 6-*p*-tolylpentafulvene and 5 mmol of nitrone was refluxed for 15 h in toluene. After evaporation

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of the solvent, the crude oil was dissolved in ethanol. The major and the minor products were separated by thin-layer chromatography on silica gel (R_F major 0.76, R_F minor 0.62). Crystals of the minor product suitable for X-ray analysis were grown from ethanol.

Crystal data

$C_{28}H_{25}NO_2$
M_r = 407.49
Monoclinic
$P2_1/n$
a = 12.7246 (15) Å
b = 9.5273 (17) Å
c = 18.451 (2) Å
β = 93.098 (12)°
V = 2233.5 (6) Å^3
Z = 4
D_x = 1.212 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9.06–18.03°
μ = 0.076 mm^{-1}
T = 296 (1) K
Irregular
0.30 × 0.25 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3563 measured reflections
3409 independent reflections
1620 reflections with $I > 2\sigma(I)$

R_{int} = 0.024
θ_{max} = 24.63°
$h = -14 \rightarrow 0$
$k = -11 \rightarrow 0$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.156
S = 0.995
3409 reflections
280 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.196 e Å^{-3}
$\Delta\rho_{min}$ = −0.162 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—N	1.438 (3)	C2—C6	1.524 (4)
O1—C3	1.468 (3)	C3—C4	1.491 (4)
N—C23	1.430 (4)	C4—C5	1.326 (4)
N—C1	1.470 (4)	C5—C6	1.443 (4)
C1—C2	1.520 (4)	C6—C7	1.331 (4)
C2—C3	1.522 (4)	C7—C8	1.468 (4)
N—O1—C3	107.2 (2)	O1—C3—C4	114.0 (3)
C23—N—O1	111.8 (2)	O1—C3—C2	106.2 (2)
C23—N—C1	120.4 (2)	C4—C3—C2	104.5 (2)
O1—N—C1	102.4 (2)	C5—C4—C3	111.5 (3)
N—C1—C2	104.8 (2)	C4—C5—C6	112.3 (3)
N—C1—C15	106.8 (2)	C7—C6—C5	131.2 (3)
C2—C1—C15	112.9 (3)	C7—C6—C2	122.8 (3)
C1—C2—C3	102.5 (2)	C5—C6—C2	106.0 (3)
C1—C2—C6	115.0 (3)	C6—C7—C8	130.8 (3)
C3—C2—C6	105.2 (3)		

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*b*). Molecular graphics: *ORTEP3* (Farrugia, 1997). Software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1028). Services for accessing these data are described at the back of the journal.

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A flavone 1,4-dihydropyridine calcium antagonist

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Abstract

In the title compound, diethyl 2,6-dimethyl-4-(2-phenyl-4-oxo-4*H*-1-benzopyran-6-yl)-1,4-dihydropyridine-3,5-dicarboxylate, $C_{28}H_{27}NO_6$, the 1,4-dihydropyridine ring exhibits a boat conformation. The benzopyran moiety of the flavone is nearly planar, and is approximately perpendicular to the 1,4-dihydropyridine ring [dihedral angle 87.1 (1)°]. The phenyl ring is twisted 10.7 (1)° from the plane of the benzopyran ring system.

Comment

The 1,4-dihydropyridine-type (1,4-DHP) calcium antagonists (CAs), such as nifedipine and structurally related drugs, are known as a subset of a wider class of CAs, which are among the most commonly used drugs for patients with cardiovascular diseases (Hirakawa *et al.*, 1972; Nayler, 1988). In the search for new

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