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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1361). Services for accessing these data are described at the back of the journal.

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1,3,4,8-Tetraphenyl-7-oxa-1,2-diazaspiro-[4.4]nona-2,8-dien-6-one†

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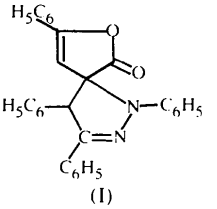
Abstract

In the title compound, $C_{30}H_{22}N_2O_2$, the pyrazoline ring conformation deviates slightly from an ideal envelope conformation. It is substituted by three planar phenyl rings, inclined to it at angles of 89.8 (1), 14.1 (1) and 7.3 (1)°. The substituted phenyl rings are in equatorial and axial positions with respect to the pyrazoline ring. The lactone ring is essentially planar, but the keto group O atom deviates from the least-squares plane through the ring atoms by −0.130 (1) Å. The lactone ring has one phenyl substituent, which adopts an axial position and is inclined at an angle of 11.3 (1)°. The dihedral angle between the pyrazoline and lactone rings is 87.6 (1)°. The crystal structure is stabilized by weak intermolecular hydrogen bonds.

Comment

Pyrazoline compounds have many important pharmacological properties, finding use as, for example, anti-inflammatory agents, herbicides, analgetic agents, antibacterial agents, moderate non-toxic local anaesthetics and antifungal agents (Gusar *et al.*, 1995; Sharma *et al.*, 1993; Ankhiwala & Hathi, 1996). They are also effective scintillation solutes and lubricating oil antioxidants (Beher *et al.*, 1967). Lactones serve as starting materials for the synthesis of natural products (Rao, 1976). The lactone derivatives α- and β-angelica lactones are cardiovascular agents, whereas the γ-lactone is used in the perfume industry (Rao, 1964; Jenkins & Hartung, 1950). Furthermore, lactones find use in the preparation of pyrrolidone (Lakhrissi & Chapleur, 1994). In view of the above importance of such compounds and to confirm the structure assignments and relative stereochemistries, a structure determination of the title spiro pyrazoline–lactone compound, (I), was carried out.



(I)

In the pyrazoline–lactone ring system (Fig. 1), the pyrazoline ring deviates slightly from an ideal envelope conformation [Q_2 = 0.238 (2) Å and Φ_2 = 3.2 (4)°; Cremer & Pople, 1975]. This is also confirmed by the sum of the bond angles within the pyrazoline ring [534.1 (11)°]. The pyrazoline and lactone rings are nearly orthogonal to each other [87.6 (1)°]. The bond lengths and angles of the pyrazoline ring differ slightly from the values found for acetone 4,4-dimethyl-5-oxo-2-pyrazolin-3-ylhydrazone (Meyers *et al.*, 1996). The three phenyl rings, *A*, *C* and *D*, attached to the pyrazoline ring at C7, N4 and C6, subtend angles of 89.8 (1), 14.1 (1) and 7.3 (1)°, respectively. Phenyl rings *A* and *C* are disposed equatorially, while ring *D* is in an axial position with respect to the pyrazoline ring. The planar lactone ring is inclined at an angle of 11.3 (1)° to the substituted phenyl ring *B*, which adopts an axial

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position. The O2 atom deviates by −0.130 (1) Å from the mean plane of the other five atoms in the lactone ring. The dihedral angle between phenyl rings are: *A/B* 68.4 (1), *B/C* 89.2 (1), *C/D* 9.7 (1) and *A/D* 84.7 (1)°. Phenyl rings *B* and *C* are mutually perpendicular. The crystal structure is stabilized by weak C—H···O and C—H···N intermolecular hydrogen bonds.

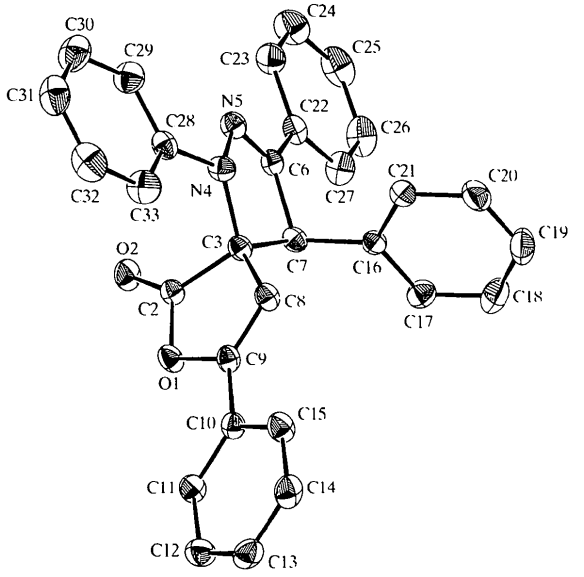

Fig. 1. Perspective view of the title molecule with the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

Experimental

Triethylamine (3.3 mmol) was added to a solution of α-benzylidene-γ-phenyl $\Delta^{\beta\gamma}$-butenolide (3 mmol) and *N*-phenylbenzhydrazidoyl chloride (3 mmol) in dry chloroform (10 ml). The reaction mixture was stirred at room temperature for 36 h, after which the solution was filtered under reduced pressure to remove triethylamine hydrochloride and solvent. The resulting crude product was purified by column chromatography (hexane–EtOAc, 9:1) and crystallized from methanol (Shanmuga Sundaram & Raghunathan, 1997).

Crystal data

$C_{30}H_{22}N_2O_2$
M_r = 442.50
Triclinic
$P\bar{1}$
a = 9.554 (1) Å
b = 10.401 (1) Å
c = 12.999 (1) Å
α = 71.92 (1)°
β = 70.72 (1)°
γ = 74.95 (1)°
V = 1141.15 (18) Å^3
Z = 2
D_x = 1.288 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 39 reflections
θ = 5.38–12.51°
μ = 0.081 mm^{-1}
T = 293 (2) K
Square prism
0.48 × 0.34 × 0.32 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
6113 measured reflections
5184 independent reflections
3039 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.50°
h = −1 → 12
k = −12 → 13
l = −16 → 16
3 standard reflections every 97 reflections
intensity variation: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 0.783
5184 reflections
308 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.209 e Å^{-3}
$\Delta\rho_{min}$ = −0.146 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.020 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C2	1.3630 (17)	N4—C28	1.413 (2)
O1—C9	1.4181 (16)	N5—C6	1.2826 (19)
O2—C2	1.1894 (17)	C6—C22	1.471 (2)
C2—C3	1.543 (2)	C6—C7	1.5184 (19)
C3—N4	1.4819 (19)	C7—C16	1.516 (2)
C3—C8	1.4927 (19)	C8—C9	1.321 (2)
C3—C7	1.571 (2)	C9—C10	1.460 (2)
N4—N5	1.3944 (16)		
C2—O1—C9	107.93 (11)	C2—C3—C7	109.39 (11)
O2—C2—O1	122.29 (14)	N5—N4—C28	116.39 (12)
O2—C2—C3	128.84 (13)	N5—N4—C3	109.88 (11)
O1—C2—C3	108.87 (11)	C28—N4—C3	124.10 (12)
N4—C3—C8	119.15 (12)	C6—N5—N4	109.78 (12)
N4—C3—C2	111.61 (12)	N5—C6—C22	121.66 (13)
C8—C3—C2	100.73 (11)	N5—C6—C7	113.42 (12)
N4—C3—C7	101.20 (11)	C22—C6—C7	124.84 (13)
C8—C3—C7	114.89 (12)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2^{i}	0.98	2.54	3.497 (2)	164
C13—H13···O2^{iii}	0.93	2.50	3.250 (2)	137
C20—H20···N5^{iii}	0.93	2.73	3.574 (2)	151
C26—H26···O2^{iv}	0.93	2.79	3.570 (2)	142

Symmetry codes: (i) 1 − x, 1 − y, − z; (ii) x, 1 + y, z; (iii) − x, 1 − y, 1 − z; (iv) x − 1, y, z.

Each H atom was placed geometrically and allowed to ride on its parent atom.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

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3-Phenylspiro[bicyclo[2.2.1]hept-5-ene-2,3′-chroman]-4′-one†

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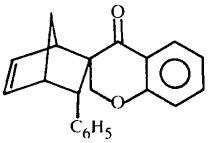
Abstract

The title compound, $C_{21}H_{18}O_2$, resulted from a Diels–Alder reaction. The chromanone moiety consists of one benzene ring fused with a six-membered heterocyclic ring, which adopts a half-chair conformation. In the bicyclo[2.2.1]heptene (norbornene) unit, the two five-membered rings are in envelope conformations, while the six-membered ring adopts a boat conformation. The dihedral angle between the chromanone system and the norbornene six-membered-ring moiety is 54.5 (4)°, and that between the norbornene six-membered ring and the phenyl substituent is 68.0 (1)°.

Comment

The chromanone part of the title compound, (I), comprising rings *A* and *B* in Fig. 1, has useful medicinal properties. Chromanone derivatives dilate the heart



(I)

and act as remedies for angina pectoris (Hasegaida, 1967). They also show vasodilating activity on the coronary vascular bed (Nagao *et al.*, 1972). The structural analysis of the title spiro chromanone derivative was performed in order to define the conformation of the 4′-chromanone system with respect to the bicyclo-[2.2.1]heptene (norbornene) moiety.

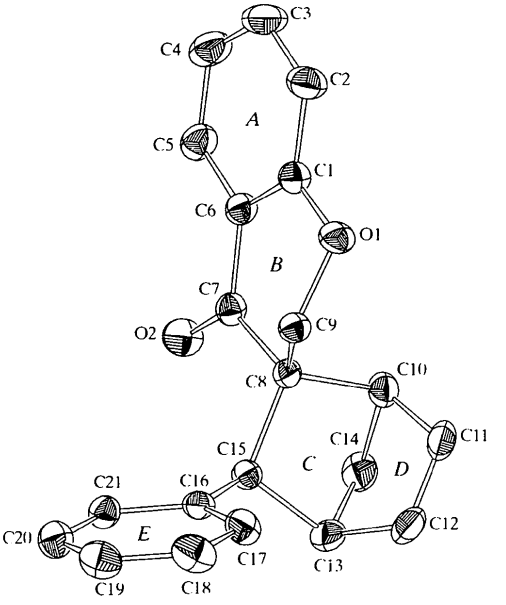


Fig. 1. A perspective view of the title molecule showing displacement ellipsoids drawn at 50% probability and the atomic numbering scheme.

In the chromanone moiety, ring *B* has a half-chair conformation with Cremer & Pople (1975) parameters q_2 = 0.392 (14) Å, q_3 = −0.285 (13) Å, φ_2 = 48.3 (2)°, θ_2 = 126.0 (2)° and Q_T = 0.484 (13) Å. The O2 atom deviates by 0.162 (12) Å from the mean plane of ring *B*. The boat conformation of the norbornene six-membered

† DCB contribution No. 881.

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