ORGANIC COMPOUNDS

Acta Cryst. (1998). C**54**, 629–630

1′,3′-Diphenylspiro[anthracene-9(10*H*),5′(4′*H*)-pyrazol]-10-one

SANKARAN BANUMATHI,[a] DEVADASAN VELMURUGAN,[a] RAGHAVACHARY RAGHUNATHAN[b] AND MUTHIAN SHANMUGA SUNDARAM[b]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and* [b]*Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India. E-mail: crystal@giasmd01.vsnl.net.in*

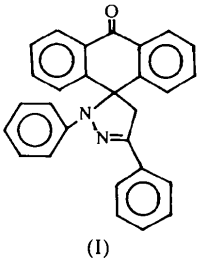
(*Received 20 January 1997; accepted 21 November 1997*)

Abstract

The title compound, $C_{28}H_{20}N_2O$, contains one anthrone moiety and one pyrazoline ring, which is connected to two phenyl rings. The central six-membered ring of the anthrone moiety adopts a sofa conformation, while the pyrazoline ring is planar. The anthrone moiety is nearly perpendicular to the pyrazoline ring, forming a dihedral angle of 92.5 (3)°.

Comment

Pyrazoline derivatives have been found to have moderate non-toxic local anaesthetic effects. As a consequence of their fluorescence, these derivatives are effective scintillation solutes. These compounds have also been utilized as lubricating oils and antioxidants (Jarboe, 1967). Anthrone derivatives are used to study the reaction rate of acid catalysts (Ghosh *et al.*, 1993). The crystal structure analysis of the title compound, (I), was undertaken to determine the conformational changes of the pyrazoline ring on its spiro union with the anthrone moiety.



(I)

Rings *A* and *C* of the three-fused-ring fragment *ABC* of the anthrone moiety are planar, with average deviations of 0.007 (2) and 0.004 (2) Å, respectively. The least-squares-plane calculations show that the central six-membered ring (*B*) of the anthrone moiety assumes a sofa conformation. Atoms C2, C7, C8, C9 and C14 form the base, and C1 deviates from it by −0.246 (2) Å. The widening of the angle C2—C1—C14 to 113.7 (2)° compared with that of free anthrone may be due to the overall molecular overcrowding [some of the shortest distances giving rise to the overcrowding are H6···O1 2.41, C1···C3 2.522 (2), C1···C13 2.529 (3), N2···C13 2.909 (2) and C3···C15 3.148 (2) Å]. The anthrone moiety is nearly perpendicular to the pyrazoline ring, with a dihedral angle of 92.5 (3)°. The geometric parameters of the anthrone moiety agree well with the literature values (Ghosh *et al.*, 1993).

The bond lengths and angles of the pyrazoline ring are in close agreement with the literature values (Ergin *et al.*, 1993, 1996; Lorand *et al.*, 1985) and the ring adopts a planar conformation. Phenyl rings 1 and 2 are attached to the pyrazoline ring at N2 and C16, respectively (Fig. 1), and they deviate from the plane of the pyrazoline ring with dihedral angles of 15.35 (8) and 10.49 (6)°, respectively. The dihedral angle between the two phenyl rings is 25.75 (8)°. The N2 atom of the pyrazoline ring is sp^2 hybridized.

In addition to the van der Waals interactions, weak inter- and intramolecular C—H···O and C—H···N hydrogen bonds stabilize the structure.

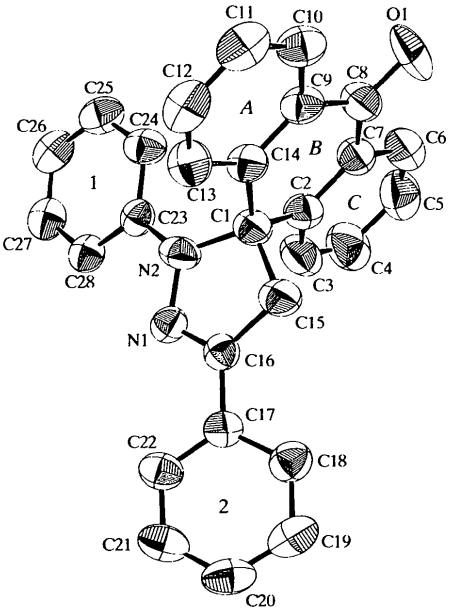


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids with the atomic numbering scheme.

Experimental

Triethylamine was added to a solution of 9-methyleneanthrone (1 mmol) and *N*-phenylbenzhydrazidoyl chloride (1 mmol) in

dry benzene. The solution was stirred until the starting material disappeared, as determined by thin-layer chromatography. The crude product was purified by column chromatography using petroleum ether–benzene (9:1) and crystallized from petroleum ether–benzene (1:1) solution; m.p. 503 K.

Crystal data

$C_{28}H_{20}N_2O$
M_r = 400.46
Orthorhombic
Pbca
a = 11.188 (4) Å
b = 13.672 (9) Å
c = 27.352 (2) Å
V = 4183.8 (4) Å^3
Z = 8
D_x = 1.272 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 10–25°
μ = 0.608 mm^{-1}
T = 293 (2) K
Rectangular
0.40 × 0.18 × 0.15 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3728 measured reflections
3727 independent reflections
2940 reflections with $I > 2\sigma(I)$

θ_{max} = 67°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 32$
3 standard reflections every 100 reflections
intensity decay: <1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.174
S = 1.103
3727 reflections
360 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1001P)^2 + 0.7638P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = −0.005
$\Delta\rho_{max}$ = 0.278 e Å^{-3}
$\Delta\rho_{min}$ = −0.155 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C16	1.278 (2)	C1—C14	1.534 (3)
N1—N2	1.378 (2)	C1—C15	1.562 (3)
N2—C23	1.398 (3)	C2—C7	1.394 (3)
N2—C1	1.490 (3)	C7—C8	1.476 (3)
O1—C8	1.216 (3)	C8—C9	1.479 (3)
C1—C2	1.515 (3)	C9—C14	1.390 (3)
C16—N1—N2	109.6 (2)	C2—C1—C15	109.6 (2)
N1—N2—C23	118.6 (2)	C14—C1—C15	109.0 (2)
N1—N2—C1	112.82 (15)	C16—C15—C1	102.7 (2)
C23—N2—C1	125.7 (2)	N1—C16—C17	121.9 (2)
N2—C1—C2	112.0 (2)	N1—C16—C15	113.6 (2)
N2—C1—C14	111.4 (2)	C17—C16—C15	124.4 (2)
C2—C1—C14	113.7 (2)	C24—C23—N2	121.0 (2)
N2—C1—C15	100.26 (15)	N2—C23—C28	120.3 (2)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP92* (Vickovic, 1994). Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1983).

SB thanks the CSIR, India, for financial support in the form of a Senior Research Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1463). Services for accessing these data are described at the back of the journal.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Ergin, O., Sillanpää, R. & Ezer, N. (1993). *Acta Cryst.* C**49**, 42–44.
Ergin, O., Sillanpää, R. & Yesilada, A. (1996). *Acta Cryst.* C**52**, 1770–1772.
Ghosh, R., Lynch, V. M., Simonsen, S. H., Prasad, R. S. & Roberts, R. M. (1993). *Acta Cryst.* C**49**, 1013–1015.
Jarboe, C. H. (1967). In *Pyrazoles, Pyrazolines, Pyrazolidines, Indazoles and Condensed Rings*, edited by R. H. Wiley. Gresham: John Wiley.
Lorand, T., Szabo, D., Foldesi, A., Parkanyi, L., Kalman, A. & Neszmelyi, A. (1985). *J. Chem. Soc. Perkin Trans.* 1, pp. 481–486.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Vickovic, I. (1994). *J. Appl. Cryst.* **27**, 437.

Acta Cryst. (1998). C**54**, 630–633

Three Diphenyl Sulfones

JEYARAMAN JEYAKANTHAN,[a] D. VELMURUGAN,[a] K. PANNEERSELVAM,[b] MANUEL SORIANO-GARCIA,[b] S. PERUMAL[c] AND R. CHANDRASEKARAN[c]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras 600 025, India,* [b]*Instituto de Quimica, UNAM, Circuito Exterior Ciudad Universitaria, Mexico DF 04510, Mexico, and* [c]*Department of Organic Chemistry, Madurai Kamaraj University, Palkalai Nagar, Madurai 21, India. E-mail: crystal@giasmd01.vsnl.net.in*

(*Received 3 April 1997; accepted 14 November 1997*)

Abstract

In 2,6-dimethylphenyl 4-nitrophenyl sulfone, $C_{14}H_{13}NO_4S$, (I), 2,6-dimethylphenyl 4-methylphenyl sulfone, $C_{15}H_{16}O_2S$, (II), and 2-methylphenyl phenyl sulfone, $C_{13}H_{12}O_2S$, (III), the S atoms have distorted tetrahedral bonding geometry. In each compound, the phenyl rings are nearly perpendicular to each other [dihedral angles: 80.0 (1) in (I), 89.6 (1) in (II) and 78.9 (1)° in (III)].

Comment

A number of diaryl sulfides, sulfones and sulfoxides display insecticidal, antibacterial, germicidal, microbial