Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.162$
$S = 1.186$
1832 reflections
182 parameters
H atoms fixed (H atoms on C7 riding)
$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2 + 0.5159P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.033$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.006 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (III)*

S—O1	1.436 (2)	S—C1	1.777 (3)
S—O2	1.444 (2)	C2—C7	1.502 (4)
S—C1′	1.768 (3)		
O1—S—O2	118.41 (14)	C6—C1—S	115.9 (2)
O1—S—C1′	108.29 (13)	C2—C1—S	123.1 (2)
O2—S—C1′	107.43 (13)	C3—C2—C7	118.7 (3)
O1—S—C1	110.34 (13)	C1—C2—C7	125.4 (3)
O2—S—C1	106.90 (13)	C6′—C1′—S	119.2 (2)
C1′—S—C1	104.58 (11)	C2′—C1′—S	119.9 (2)

Table 4. *Selected dihedral angles (°) for (I), (II) and (III)*

	Ar/Ar	C—S—C/C1–C6	C—S—C/C1′–C6′
(I)	80.0 (1)	108.7 (1)	63.3 (1)
(II)	89.6 (1)	110.4 (1)	120.9 (1)
(III)	78.9 (1)	95.8 (1)	82.6 (1)

For all compounds, data collection: *SDP* (Frenz, 1978); cell refinement: *SDP*; data reduction: *SDP*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*92 (Vickovic, 1994); software used to prepare material for publication: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1058). Services for accessing these data are described at the back of the journal.

References

Chandrasekaran, R. (1991). *Studies on Organic Sulfur Compounds*. PhD thesis, Madurai Kamaraj University, India.

De Benedetti, P. G., Folli, U., Iarossi, D. & Frassineti, C. (1985). *J. Chem. Soc. Perkin Trans.* 2, pp. 1527–1532.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.

Gzella, A., Wrzeciono, U., Dudzinska-Usarewicz, J. & Borowiak, T. (1989). *Acta Cryst.* C**45**, 642–644.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Sime, J. G. & Woodhouse, D. J. (1974). *J. Cryst. Mol. Struct.* **4**, 269–285.

Tiwari, R. K., Haridas, M. & Singh, T. P. (1984). *Acta Cryst.* C**40**, 655–657.

Vickovic, I. (1994). *J. Appl. Cryst.* **27**, 437.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1998). C**54**, 633–635

9-(4-Methoxyphenyl)-10-phenyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione

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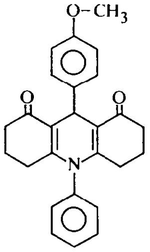
Abstract

In the title compound, $C_{26}H_{25}NO_3$, the central ring adopts a distorted boat conformation and the outer rings adopt half-chair conformations. The molecule is stabilized by C—H···O-type hydrogen bonds.

Comment

The interactions of acridine with DNA are generally known to be partly responsible for their biological activities (Neidle, 1979). Acridines of the same type as the title compound, (I), have been observed to fluoresce well as laser dyes in alcohol solvents (Selladurai *et al.*, 1990).



(I)

The bond lengths in the pyridine ring range from 1.348 (3) to 1.511 (3) Å and show the respective greater or lesser degree of single- or double-bond character as predicted from comparisons with related acridine structures (Selladurai *et al.*, 1989, 1990; Sivaraman *et al.*, 1994, 1996). The C—N distances in the acridinyl ring have an average value of 1.395 (5) Å and the mean C—N—C angle is 119.8 (3)°. These values agree well with those for related acridinyl derivatives (Chu & Van der Helm, 1977).

The ketone bond lengths C6—O6′ and C10—O10′ of 1.227 (3) and 1.229 (3) Å, respectively, are in agreement with values observed for related structures (Dideberg *et* *al.*, 1973). The central ring (pyridine) adopts a distorted boat conformation, while the outer two rings adopt half-chair conformations. The torsion angles C14—C9—C8—C21 of 98.6 (2) and C9—C14—N1—C20 of −173.7 (2)° show that the methoxyphenyl group is axial to the acridine moiety, while the phenyl ring is equatorial. The mean plane passing through phenyl ring C21–C26 is inclined by 86.6 (1)° to that of the acridine moiety, while the plane of phenyl ring C15–C20 is inclined by 89.0 (1)° to that of the acridine moiety. The central ring is not planar and so a large buckling [19.0 (1)°] is observed. Analysis of the acridine compounds reported earlier reveals that buckling of the acridine moiety is independent of the substituents at the N1 and C8 positions.

In the crystal packing, the phenyl ring of one molecule partially stacks with the methoxyphenyl ring of another molecule. Intramolecular and intermolecular C—H···O hydrogen bonds stabilize the crystal structure (Table 2).

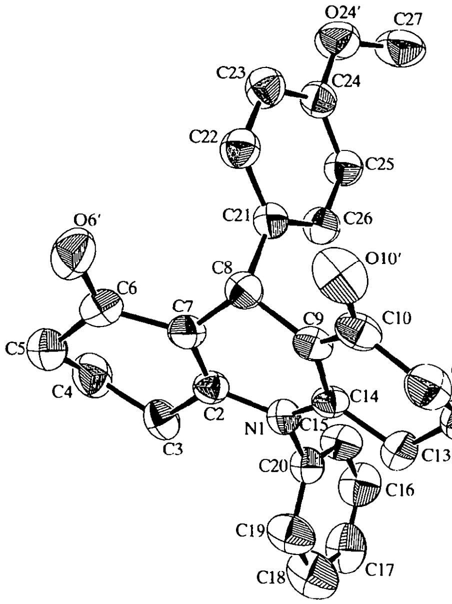


Fig. 1. An *ORTEP*II (Johnson, 1976) diagram of the title molecule with the atom-numbering scheme.

Experimental

For the preparation of (I), 2,2′-(*p*-methoxybenzylidene)bis-(cyclohexane-1,3-dione) in acetic acid was added to aniline and refluxed for 3 h. The reaction mixture was concentrated and poured into ice water. The solid was filtered off, dried and recrystallized from MeOH–$CHCl_3$ (1:1) (Shanmugasundaram *et al.*, 1996).

Crystal data

$C_{26}H_{25}NO_3$
$M_r = 399.47$
Monoclinic
$P2_1/n$
$a = 8.6696 (7)$ Å
$b = 11.1396 (7)$ Å
$c = 21.1975 (7)$ Å
$\beta = 92.49 (2)°$
$V = 2045.2 (2)$ Å^3
$Z = 4$
$D_x = 1.291$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 23 reflections
$\theta = 8–18°$
$\mu = 0.631$ mm^{-1}
$T = 293 (2)$ K
Needle
0.32 × 0.14 × 0.10 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4153 measured reflections
3884 independent reflections
2961 reflections with $I > 2\sigma(I)$

$R_{int} = 0.015$
$\theta_{max} = 69.93°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = -25 \rightarrow 25$
3 standard reflections every 200 reflections intensity decay: <0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.176$
$S = 0.610$
3884 reflections
372 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.1626P)^2 + 2.7584P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.029$

$\Delta\rho_{max} = 0.275$ e Å^{-3}
$\Delta\rho_{min} = -0.171$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0014 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C2	1.394 (3)	C7—C8	1.511 (3)
N1—C14	1.396 (3)	C8—C9	1.508 (3)
C2—C7	1.357 (3)	C9—C14	1.348 (3)
C20—N1—C2—C7	171.9 (2)	C21—C8—C9—C14	98.6 (2)
C7—C2—C3—C4	−17.0 (3)	C14—C9—C10—C11	−1.0 (3)
C2—C3—C4—C5	46.8 (3)	C9—C10—C11—C12	−31.4 (3)
C3—C4—C5—C6	−52.9 (3)	C10—C11—C12—C13	55.5 (3)
C4—C5—C6—C7	28.7 (3)	C11—C12—C13—C14	−47.3 (3)
C3—C2—C7—C6	−7.7 (3)	C8—C9—C14—N1	9.8 (3)
N1—C2—C7—C8	−5.9 (3)	C10—C9—C14—C13	9.5 (3)
C5—C6—C7—C2	1.8 (3)	C2—N1—C14—C9	12.7 (3)
C2—C7—C8—C9	24.9 (3)	C20—N1—C14—C9	−173.7 (2)
C7—C8—C9—C14	−26.9 (3)	C12—C13—C14—C9	15.3 (3)

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

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C8—H81···O6′	2.48 (2)	2.814 (3)	101 (2)
C8—H81···O10′	2.50 (2)	2.809 (3)	99 (2)
C26—H261···O10′	2.94 (3)	3.346 (3)	108 (2)
C3—H31···O24′^{i}	2.69 (3)	3.471 (3)	136 (2)
C18—H181···O10′^{ii}	2.52 (4)	3.348 (3)	144 (3)
C11—H112···O6′^{iii}	2.60 (3)	3.462 (3)	140 (2)

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $x, y - 1, z$; (iii) $1 + x, y, z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: PT1052). Services for accessing these data are described at the back of the journal.

References

Chu, S. S. C. & Van der Helm, D. (1977). *Acta Cryst.* B**32**, 1012–1016.

Dideberg, O., Campsteyn, H. & Dupont, L. (1973). *Acta Cryst.* B**29**, 103–112.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. University of Cambridge, England.

Neidle, S. (1979). *Prog. Med. Chem.* **16**, 151–221.

Selladurai, S., Subramanian, E. & Natarajan, S. (1989). *Acta Cryst* C**45**, 1346–1348.

Selladurai, S., Subramanian, K. & Ramakrishnan, V. T. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 227–232.

Shanmugasundaram, P., Murugan, P. & Ramakrishnan, V. T. (1996). *Heteroatom Chem.* **7**, 17–22.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Cambridge, England.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Ramakrishnan, V. T. (1994). *Acta Cryst.* C**50**, 2011–2013.

Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Shanmugasundaram, P. S. (1996). *Acta Cryst.* C**52**, 481–483.

Acta Cryst. (1998). C**54**, 635–637

(3-Bromo-1-phenylsulfonyl-2-indolyl)methyl Benzoate

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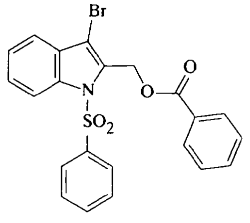
(Received 16 April 1996; accepted 17 November 1997)

Abstract

In the title compound, $C_{22}H_{16}BrNO_4S$, the phenylsulfonyl group is nearly perpendicular [94.7 (1)°] to the indole ring system, while the other phenyl ring (of the benzoyloxymethyl substituent) is inclined at an angle of 85.9 (2)° to it. The two phenyl rings are inclined at an angle of 112.9 (3)° with respect to one another. The geometry around the S atom is distorted from the ideal tetrahedral geometry. This is evident from the deviations of the values of the bond angles around sulfur from 108.8°.

Comment

The indole ring system is present in a number of natural products, many of which are found to possess psychotropic, antidepressant and hypertensive properties (Seetharaman & Rajan, 1995). They also exhibit antimicrobial (El-sayed *et al.*, 1986), anti-inflammatory (Rodriguez *et al.*, 1985) and anti-implantation activity in rats. The study of the title compound, (I), was undertaken because very little crystallographic information is available on non-steroidal indole derivatives (Chakraborthy & Talapatra, 1986).



(I)

A *ZORTEP* (Zsolnai, 1995) plot of the molecule of (I) is shown in Fig. 1. The S—O, S—C and S—N bond distances are 1.435 (5), 1.767 (7) and 1.685 (5) Å, respectively, and are comparable with those found for another phenylsulfonyl group attached to an indole ring system (Seetharaman & Rajan, 1995). The C3—Br bond distance, 1.871 (6) Å, is in agreement with the reported value of 1.898 (2) Å (Shanmuga Sundara Raj *et al.*, 1994). The relatively large values of the C—N distances in the indole moiety [C2—N1 1.422 (8) and C9—N1 1.450 (8) Å] are due to the electron-withdrawing character of the phenylsulfonyl group. The sum of the angles about the N1 atom [354.9 (4)°] is less than the sum of the angles about the corresponding atom of a related structure [359.2 (3)°; Hökelek *et al.*, 1994]. The bond angles around the S10 atom are distorted from the ideal values for tetrahedral geometry (108.8°; Singh *et al.*, 1985); this distortion is further supported by the short non-bonded distances in this moiety, *i.e.* C13···O11 2.604 (8), C13···O12 2.634 (8) and N1···O12 2.502 (5) Å.

The indole ring system is nearly planar, as observed in many natural product derivatives (Vijayalakshmi & Srinivasan, 1975; Yamane *et al.*, 1977). The phenylsulfonyl group is perpendicular to the indole moiety. The plane through the phenyl ring of the phenylsulfonyl substituent makes an angle of 92.5 (2)° with the indole

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