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
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10$–$25°$
$\mu = 0.608$ 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 $I > 2\sigma(I)$

$\theta_{max} = 67°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 32$
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: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*92 (Vickovic, 1994). Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1983).

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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.

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Three Diphenyl Sulfones

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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

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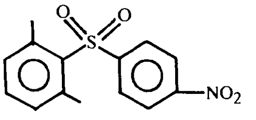
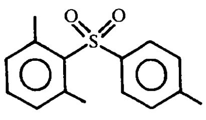
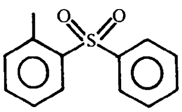
and other important biological activities (De Benedetti *et al.*, 1985). In each of compounds (I), (II) and (III), the phenyl rings are almost equidistant from the S atom [average C_{sp^2}—S 1.779 (3) Å, range 1.768 (3)–1.791 (2) Å], which agrees well with the literature (Tiwari *et al.*, 1984). The mean S═O distance is 1.439 Å. In all cases, the S atom is at the centre of a distorted tetrahedron. In all three structures, the phenyl-ring geometry is normal and the phenyl rings are nearly perpendicular to each other. Dihedral angles are given in Table 4. In many diphenyl sulfones (Sime & Woodhouse, 1974), the dihedral angles between the aromatic rings and the C—S—C plane are near 90°. In this study, it was found that the presence of the nitro group in compound (I) and the presence of the *p*-methyl substituent in (II) result in variations of these angles from 90° (Table 4). This phenomenon may be related to the electron-withdrawing nature of the nitro group in (I) and the slight electron-donating nature of the *p*-methyl substituent in (II). The deviation of the S atom from the plane of phenyl ring C1′–C6′ is evidence for the lack of conjugation with the ring (Sime & Woodhouse, 1974).



(I)



(II)



(III)

In compound (I), the nitro group has the O═N═O structure, which can be confirmed by comparison with data in the literature (Gzella *et al.*, 1989). This nitro group is nearly coplanar with the phenyl ring to which it is attached [dihedral angle 169 (1)°].

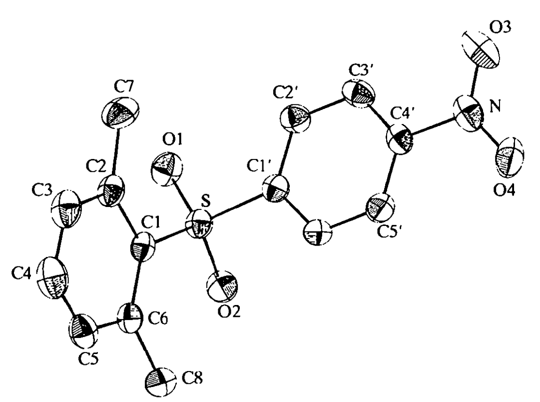


Fig. 1. The molecular structure of (I), with 50% probability displacement ellipsoids for non-H atoms (H atoms omitted).

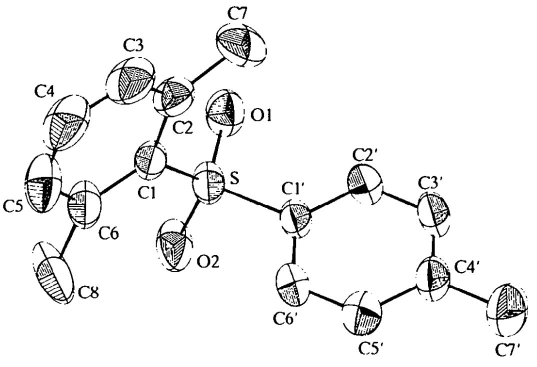


Fig. 2. The molecular structure of (II), with 50% probability displacement ellipsoids for non-H atoms (H atoms omitted).

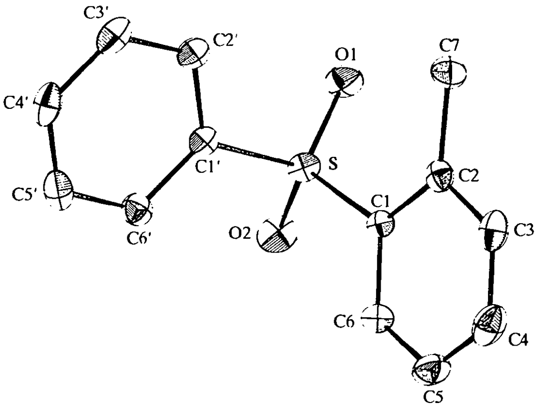


Fig. 3. The molecular structure of (III), with 50% probability displacement ellipsoids for non-H atoms (H atoms omitted).

Experimental

Compounds (I), (II) and (III) were prepared by the oxidation of the corresponding sulfides or sulfoxides with an excess of hydrogen peroxide in acetic acid (Chandrasekaran, 1991). Single crystals were obtained from ethanol by slow evaporation.

Compound (I)

Crystal data

$C_{14}H_{13}NO_4S$
$M_r = 291.31$
Monoclinic
$P2_1/n$
$a = 11.598\ (1)$ Å
$b = 10.959\ (1)$ Å
$c = 12.148\ (1)$ Å
$\beta = 117.41\ (1)°$
$V = 1370.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.412$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 21 reflections
$\theta = 15$–$30°$
$\mu = 2.2$ mm^{-1}
$T = 293\ (2)$ K
Tabular
$0.45 \times 0.40 \times 0.30$ mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.325$, $T_{\max} = 0.513$
2349 measured reflections
1788 independent reflections
1660 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.064$
$\theta_{\max} = 56.66°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 13$
3 standard reflections every 200 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.113$
$S = 0.613$
1788 reflections
234 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.121P)^2 + 1.59P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0080 (13)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

S—O2	1.437 (2)	N—O3	1.228 (3)
S—O1	1.441 (2)	N—C4′	1.480 (4)
S—C1	1.791 (2)	C2—C7	1.517 (4)
S—C1′	1.788 (3)	C6—C8	1.501 (5)
N—O4	1.231 (3)		
O2—S—O1	117.81 (13)	C6—C1—S	117.2 (2)
O2—S—C1	109.33 (12)	C3—C2—C7	116.6 (3)
O1—S—C1	110.06 (12)	C1—C2—C7	126.2 (2)
O2—S—C1′	108.36 (12)	C5—C6—C8	117.1 (3)
O1—S—C1′	105.53 (12)	C1—C6—C8	125.6 (3)
C1—S—C1′	104.92 (11)	C6′—C1′—S	119.7 (2)
O4—N—O3	124.2 (3)	C2′—C1′—S	118.7 (2)
O4—N—C4′	117.6 (3)	C3′—C4′—N	118.0 (3)
O3—N—C4′	118.2 (3)	C5′—C4′—N	118.7 (2)
C2—C1—S	121.0 (2)		

Compound (II)

Crystal data

$C_{15}H_{16}O_2S$
$M_r = 260.34$
Monoclinic
$C2/c$
$a = 20.339 (7)$ Å
$b = 11.024 (4)$ Å
$c = 15.220 (7)$ Å
$\beta = 126.80 (2)°$
$V = 2732.6 (19)$ Å^3
$Z = 8$
$D_x = 1.266$ Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 21 reflections
$\theta = 15–30°$
$\mu = 2.0$ mm^{-1}
$T = 293 (2)$ K
Tabular
0.4 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.518$, $T_{\max} = 0.666$
2257 measured reflections
1822 independent reflections
1616 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.037$
$\theta_{\max} = 56.63°$
$h = -22 \rightarrow 22$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 16$
3 standard reflections every 200 reflections intensity decay: <4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.133$
$S = 0.921$
1822 reflections
215 parameters
H atoms fixed (H atoms on C7′ riding)
$w = 1/[\sigma^2(F_o^2) + (0.0904P)^2 + 3.072P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. *Selected geometric parameters (Å, °) for (II)*

S—O2	1.434 (2)	C2—C7	1.495 (5)
S—O1	1.439 (2)	C6—C8	1.512 (5)
S—C1′	1.771 (3)	C4′—C7′	1.511 (5)
S—C1	1.781 (3)		
O2—S—O1	117.01 (15)	C3—C2—C7	117.0 (3)
O2—S—C1′	107.22 (14)	C1—C2—C7	125.7 (3)
O1—S—C1′	108.32 (14)	C1—C6—C8	126.1 (3)
O2—S—C1	109.99 (15)	C5—C6—C8	117.0 (3)
O1—S—C1	109.80 (14)	C2′—C1′—S	120.8 (2)
C1′—S—C1	103.61 (13)	C6′—C1′—S	118.6 (2)
C6—C1—S	121.2 (2)	C5′—C4′—C7′	120.8 (4)
C2—C1—S	117.0 (2)	C3′—C4′—C7′	121.1 (4)

Compound (III)

Crystal data

$C_{13}H_{12}O_2S$
$M_r = 232.29$
Monoclinic
$P2_1/c$
$a = 12.360 (5)$ Å
$b = 8.246 (4)$ Å
$c = 11.615 (7)$ Å
$\beta = 103.74 (3)°$
$V = 1149.9 (10)$ Å^3
$Z = 4$
$D_x = 1.342$ Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 21 reflections
$\theta = 13–28°$
$\mu = 2.3$ mm^{-1}
$T = 293 (2)$ K
Tabular
0.4 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.391$, $T_{\max} = 0.625$
1936 measured reflections
1832 independent reflections
1740 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.039$
$\theta_{\max} = 62.50°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 13$
3 standard reflections every 100 reflections intensity decay: <3%

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.

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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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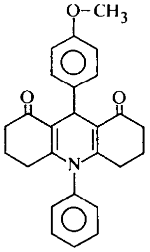
Abstract

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*