H atoms were placed in idealized positions, but allowed to ride on the parent C atom. Methyl groups were allowed to rotate. Isotropic displacement parameters were refined for all H atoms.

The structure was solved using the *SHELXTL-Plus* program package (Sheldrick, 1988) on a MicroVAX II computer and refined using *SHELXL*93 (Sheldrick, 1993).

The authors would like to thank Dr Charles Campana at Siemens Industrial Automation Inc. for the final refinement of the structure using *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Nitro-*N*-phenyl-4-(phenylamino)-benzenesulfonamide

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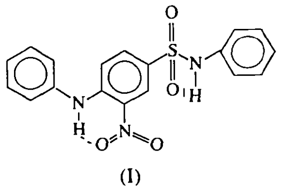
Abstract

The structure of the title compound, $C_{18}H_{15}N_3O_4S$, displays the characteristic features of benzenesulfonamide derivatives. The *N*-phenyl rings are twisted by 48.3 (1) and 77.8 (1)° with respect to the benzene ring of the phenylsulfonamide group. In the crystal, the molecules are linked to form dimers *via* N—H···O hydrogen bonds between sulfonamide groups. The amine H atom participates in an intramolecular hydrogen bond with the neighbouring nitro substituent.

Comment

The title compound, (I), is a well known commercial dye, Dispersol Yellow CT (CI Disperse Yellow 42-10338, ICI, Manchester, England), and may be considered as a derivative of either phenyl benzenesulfonamide or diphenylamine.



(I)

Structures of both systems are well known (Cambridge Structural Database, 1994) and their geometries are similar to that observed for the title compound. However, the structure of the title compound has three characteristic features. Firstly, the endocyclic bond angle C(3)—C(4)—C(5) of 115.9 (2)° is rather small; this is caused by the presence of an electron-withdrawing nitro group at the *ortho* position. This observation is in agreement with the values of 111.4, 112.2 and 113.0° found in all *N*-picrylanilides and their analogues (Gridunova, Shklover, Struchkov, Il'ina, Mikhalev & Potapov, 1989; Gridunova, Petrov, Struchkov, Il'ina & Mikhalev, 1990). Secondly, the S^{VI}—C(aryl) distance of 1.745 (2) Å found in this study is shorter than the average value of 1.76 Å for 120 arylsulfonamides (Kálmán, Czugler & Argay, 1981), although about 25 other benzenesulfonamide structures, mostly substituted by electron-donating groups at *ortho* and *para* positions, with similar S^{VI}—C(aryl) distances are known (Cambridge Structural Database, 1994). Finally, one N—C(aryl) bond, N(4)—C(4) of 1.347 (3) Å, is shortened relative to the other [N(4)—C(41) 1.438 (3) Å], which can be attributed to the strong electron-withdrawing substituents at the *ortho* and *para* positions of the benzene ring of the nitrobenzenesulfonamide system. Similar differences have been observed in all *N*-picrylanilides (Gridunova *et al.*, 1989, 1990).

There are two types of hydrogen bond in the title compound: an intermolecular N(11)—H···O sulfonamide–sulfonamide hydrogen bond which links the molecules into dimers and an intramolecular N(4)—H···O amine–nitro hydrogen bond. The latter maintains the coplanarity of the phenyl ring and the nitro group. There are several 2-nitrophenylamine structures in the literature, all showing similar intramolecular hydrogen-bonding schemes (Cambridge Structural Database, 1994). The other phenyl ring in the diphenylamine moiety is always twisted by about 60° [77.8 (1)° in this study] relative to the nitroaniline plane.

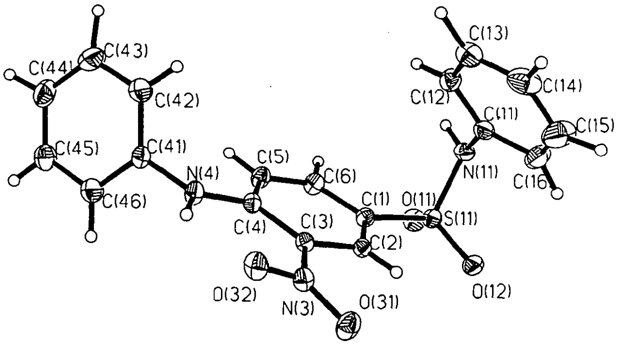


Fig. 1. A view of the title molecule. Displacement ellipsoids are plotted at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

Experimental

Crystals of the title compound suitable for the X-ray diffraction study were obtained by recrystallization of the commercial dye from toluene.

Crystal data

$C_{18}H_{15}N_3O_4S$
$M_r = 369.4$ (1)
Monoclinic
$P2_1/c$
$a = 8.869$ (1) Å
$b = 21.561$ (3) Å
$c = 9.014$ (1) Å
$\beta = 98.33$ (2)°
$V = 1705.5$ (4) Å^3
$Z = 4$
$D_x = 1.439$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 32 reflections
$\theta = 20$–25°
$\mu = 1.905$ mm^{-1}
$T = 293$ K
Prism
0.41 × 0.15 × 0.09 mm
Yellow

Data collection

KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3708 measured reflections
2930 independent reflections
2491 observed reflections [$I > 6\sigma(I)$]
$R_{\rm int} = 0.010$

$\theta_{\rm max} = 81°$
$h = 0 \rightarrow 10$
$k = -27 \rightarrow 0$
$l = -11 \rightarrow 11$
2 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F
$R = 0.039$
$wR = 0.062$
$S = 1.072$
2491 reflections
296 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.00273F^2]$
$(\Delta/\sigma)_{\rm max} = 0.012$

$\Delta\rho_{\rm max} = 0.216$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.245$ e Å^{-3}
Extinction correction: secondary (Larson, 1967)
Extinction coefficient: 0.008 (1)
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	0.2922 (3)	0.47960 (10)	0.8877 (2)	0.039 (1)
C(2)	0.4189 (3)	0.46147 (9)	0.8285 (2)	0.037 (1)
C(3)	0.4937 (3)	0.50356 (9)	0.7467 (2)	0.036 (1)
C(4)	0.4403 (3)	0.56504 (9)	0.7198 (2)	0.038 (1)
C(5)	0.3144 (3)	0.58221 (10)	0.7897 (3)	0.046 (1)
C(6)	0.2411 (3)	0.54120 (11)	0.8690 (3)	0.047 (1)
S(11)	0.1870 (1)	0.42630 (3)	0.9762 (1)	0.042 (1)
O(11)	0.1271 (2)	0.45708 (9)	1.0962 (2)	0.056 (1)
O(12)	0.2767 (2)	0.37153 (8)	1.0067 (2)	0.054 (1)
N(11)	0.0371 (2)	0.40813 (9)	0.8545 (2)	0.044 (1)
C(11)	0.0619 (3)	0.38312 (11)	0.7123 (3)	0.044 (1)
C(12)	0.0306 (4)	0.41854 (12)	0.5845 (3)	0.053 (1)
C(13)	0.0549 (4)	0.39480 (15)	0.4473 (3)	0.061 (1)
C(14)	0.1106 (4)	0.33665 (19)	0.4374 (4)	0.081 (1)
C(15)	0.1419 (6)	0.3014 (2)	0.5650 (4)	0.102 (2)
C(16)	0.1160 (5)	0.32379 (14)	0.7027 (4)	0.079 (1)
N(3)	0.6266 (2)	0.48093 (8)	0.6893 (2)	0.043 (1)
O(31)	0.6841 (2)	0.43195 (8)	0.7395 (2)	0.062 (1)
O(32)	0.6793 (2)	0.51120 (8)	0.5926 (2)	0.055 (1)
N(4)	0.5045 (2)	0.60684 (8)	0.6371 (2)	0.047 (1)
C(41)	0.4526 (3)	0.66991 (10)	0.6182 (3)	0.046 (1)
C(42)	0.3268 (4)	0.68435 (14)	0.5171 (4)	0.070 (1)
C(43)	0.2770 (5)	0.74507 (16)	0.5026 (5)	0.084 (1)
C(44)	0.3538 (4)	0.79089 (13)	0.5872 (4)	0.074 (1)
C(45)	0.4774 (5)	0.77641 (13)	0.6890 (5)	0.077 (1)
C(46)	0.5281 (4)	0.71570 (13)	0.7055 (4)	0.064 (1)

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

C(11)—N(11)	1.437 (3)	C(3)—N(3)	1.439 (3)
N(11)—S(11)	1.643 (2)	N(3)—O(31)	1.230 (3)
S(11)—O(11)	1.435 (2)	N(3)—O(32)	1.233 (3)
S(11)—O(12)	1.428 (2)	C(4)—N(4)	1.347 (3)
S(11)—C(1)	1.745 (2)	N(4)—C(41)	1.438 (3)
C(11)—N(11)—S(11)	118.1 (2)	C(3)—N(3)—O(32)	119.2 (2)
N(11)—S(11)—O(11)	105.1 (1)	C(3)—C(4)—C(5)	115.9 (2)
N(11)—S(11)—O(12)	107.8 (1)	C(4)—N(4)—C(41)	123.0 (2)
C(3)—N(3)—O(31)	118.4 (2)		

C(11)—N(11)—S(11)—C(1)	57.3 (2)
O(11)—S(11)—N(11)—C(11)	172.6 (2)
O(12)—S(11)—N(11)—C(11)	−58.8 (2)
C(41)—N(4)—C(4)—C(3)	−176.7 (2)
C(4)—C(3)—N(3)—O(31)	166.2 (2)
C(4)—C(3)—N(3)—O(32)	−13.4 (3)
C(2)—C(3)—N(3)—O(31)	−14.7 (3)
C(2)—C(3)—N(3)—O(32)	165.7 (2)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(4)—H(4)···O(32)	0.90 (3)	1.99 (3)	2.645 (3)	128.1 (3)
N(11)—H(11)···O(11$^{\rm i}$)	0.90 (2)	2.48 (2)	3.309 (3)	154.2 (2)

Symmetry code: (i) $-x, 1-y, 2-z$.

Amine and sulfonamide H atoms were refined with the N—H distances constrained to 0.90 Å. Other H atoms were refined freely with isotropic displacement parameters. The *SHELXTL/PC* program package (Sheldrick, 1990) was used throughout the analysis.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: KA1106). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(±)-1-Acetoxy-8-hydroxy-1,4,4a,9a-tetrahydroanthraquinone

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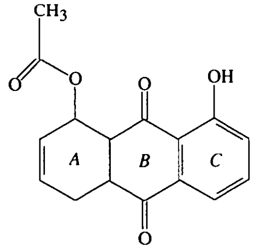
Abstract

The crystal structure of the title compound, methyl 8-hydroxy-9,10-dioxo-1,4,4a,9,9a,10-hexahydro-1-anthraceneacetate, $C_{16}H_{14}O_5$, is stabilized by intramolecular O—H···O and both intra- and intermolecular C—H···O hydrogen bonds. Ring *A*, carrying the acetoxy substituent, has a sofa conformation, the central ring *B* has an envelope conformation, while the aromatic ring *C* is planar.

Comment

The crystal and molecular structures of the title compound, (I), were investigated in order to determine the conformation and crystal packing, and also to confirm the stereochemistry.



(I)

A view of the molecule with the numbering scheme used is given in Fig. 1. The bond distances and angles are quite similar to those found in related compounds. From the torsion angles (see Table 2), it can be seen that rings *A* [puckering parameters φ_2 = 160.6 (3)°, θ_2 = 129.9 (2)°, Q = 0.434 (4) Å] and *B* [puckering parameters φ_2 = 172.4 (2)°, θ_2 = 120.2 (2)°, Q = 0.436 (4) Å] occur in sofa ($^6S_1$) and envelope (E_1) conformations, respectively (Boeyens, 1978). The aromatic ring *C* is planar to within 0.004 (2) Å.

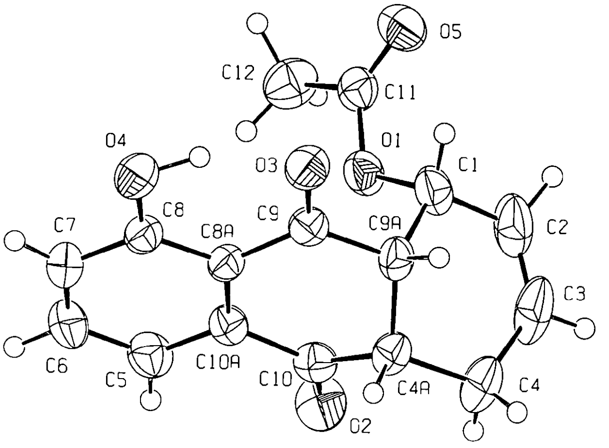


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme and 50% probability displacement ellipsoids.

The hydroxy O4 atom forms an intramolecular hydrogen bond with the carbonyl O3 atom [O···O 2.561 (3) Å; see Table 3] (Allen, Kennard & Taylor, 1983), forming a six-membered ring composed of atoms C9, C8a, C8, O4, HO4 and O3, which is planar to within 0.02 (1) Å. This ring makes an interplanar angle of 4.8 (7)° with the aromatic ring *C* (C10a, C5–C8, C8a). The crystal structure (Fig. 2) is stabilized by O—H···O hydrogen bonds (Desiraju, 1991) and there are two intra- and three intermolecular C—H···O hydrogen bonds (Table 3).

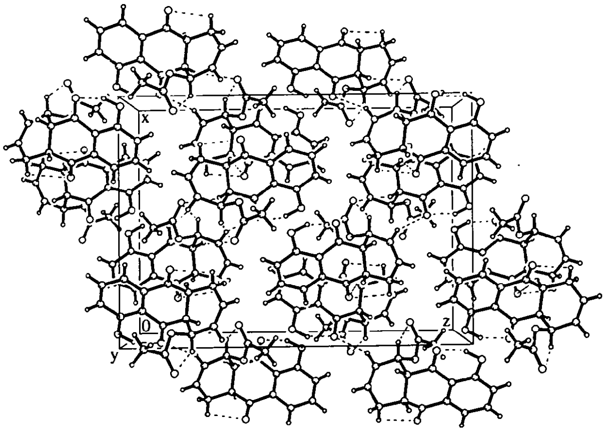


Fig. 2. A perspective drawing of the packing arrangement. The dashed lines indicate the hydrogen-bond contacts, details of which are given in Table 3.

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