Acta Cryst. (1993). C**49**, 2148–2149

N,*N*′-(1,2-Ethynylenedi-2,2′-phenylene)bis(4-methylbenzenesulfonamide)

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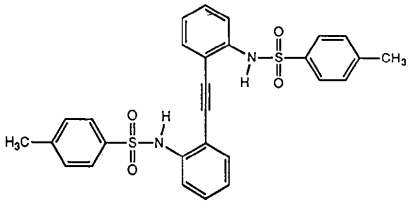
(*Received* 13 *November* 1992; *accepted* 10 *May* 1993)

Abstract

The title compound is centrosymmetric and contains an ethynyl bond of length 1.189 (2) Å. The bond angle about the ethynyl C atom is 178.2 (2)°. The two independent aromatic rings form a dihedral angle of 65.75 (7)°. Molecules are linked by weak intermolecular N—H···O hydrogen bonds each having an N···O distance of 3.032 (2) Å and an angle about H of 161 (2)°.

Comment

The title compound is an intermediate in the synthesis of diarylethynes that we designed to probe intramolecular recognition (Evans, Prince, Huang, Boss & Gandour, 1990). The determination of this structure contributes to knowledge of the geometrical effects of various substituents in the *ortho* positions of diarylethynes. This molecule was prepared by the procedure of Knops & Vögtle (1991). Tan plates of (1) suitable for X-ray analysis were obtained by recrystallization of the crude reaction mixture from methanol.



(1)

A search of the January 1992 version (4.6) of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed no structures composed of a diarylethyne backbone with N atoms in the *ortho*,*ortho*′ positions. Compound (1) has a triple-bond distance of 1.189 (2) Å and a bond angle of 178.2 (2)° at the ethynylic C atoms. Ethynyl bond lengths and angles in the title compound are comparable with those in diphenylethyne (Mavridis & Moustakali-Mavridis, 1977) and 2-(*N*,*N*-dimethylamino)diphenylethyne (Wallis & Dunitz, 1984). We conclude that these *ortho* substituents create no detectable structural changes in the ethyndiyl linkage.

The O1—S—N and O2—S—N bond angles are 107.95 (7) and 104.95 (7)°, respectively. The torsion angle O1—S—C8—C9 of −168.21 (14)° places O1 of the sulfonamide 0.271 (1) Å out of the plane of the tolyl ring. The maximum deviations from planarity of the aromatic rings are 0.014 (2) Å for the ring bearing the N atom and 0.005 (2) Å for the tolyl ring.

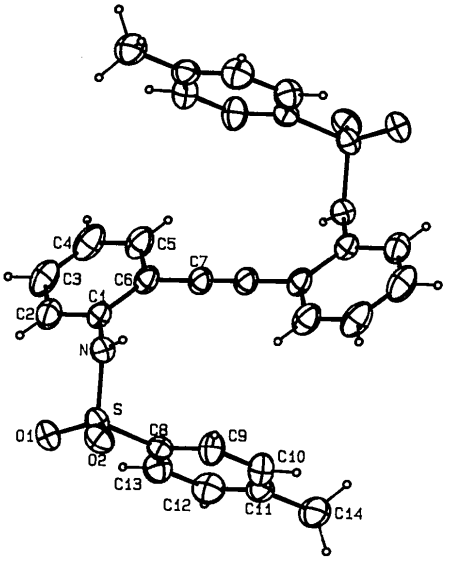


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound with thermal ellipsoids at the 40% probability level.

Experimental

Crystal data

$C_{28}H_{24}N_2O_4S_2$
M_r = 516.6
Triclinic
$P\bar{1}$
a = 8.1358 (6) Å
b = 8.6407 (4) Å
c = 9.6854 (5) Å
α = 81.098 (4)°
β = 87.352 (5)°
γ = 72.702 (5)°
V = 642.25 (6) Å^3
Z = 1
D_x = 1.334 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 25 reflections
θ = 26–30°
μ = 2.13 mm^{-1}
T = 297 K
Plate
0.42 × 0.37 × 0.10 mm
Tan

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
T_{min} = 0.6763, T_{max} = 0.9749
4777 measured reflections
2625 independent reflections
2350 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.020
θ_{max} = 75° (full sphere of data to 65°, hemisphere for 65 < θ < 75°)
$h = -9 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 167 min
intensity variation: 1%

Refinement

Refinement on F
R = 0.037
wR = 0.059
S = 2.642
2350 reflections
212 parameters
All H-atom parameters refined
$w = 4F^2[\sigma^2(I) + (0.02F^2)^2]^{-1}$
$(\Delta/\sigma)_{max}$ = 0.02

$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: $(I + gI_c)^{-1}$ applied to F_c
Extinction coefficient: 2.7 (7) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S	0.08659 (4)	0.33167 (5)	0.71043 (4)	4.426 (8)
O1	0.0951 (2)	0.1814 (2)	0.7978 (1)	5.84 (3)
O2	−0.0705 (1)	0.4233 (2)	0.6391 (1)	5.45 (3)
N	0.2298 (2)	0.2928 (2)	0.5868 (1)	4.34 (3)
C1	0.4032 (2)	0.1948 (2)	0.6157 (2)	4.22 (3)
C2	0.4375 (3)	0.0322 (2)	0.6728 (2)	5.62 (4)
C3	0.6049 (3)	−0.0648 (3)	0.6956 (2)	6.71 (5)
C4	0.7389 (3)	−0.0026 (3)	0.6582 (2)	6.90 (5)
C5	0.7075 (2)	0.1574 (2)	0.5998 (2)	5.99 (4)
C6	0.5394 (2)	0.2613 (2)	0.5796 (2)	4.40 (3)
C7	0.5100 (2)	0.4299 (2)	0.5229 (2)	4.51 (3)
C8	0.1478 (2)	0.4602 (2)	0.8091 (2)	4.18 (3)
C9	0.1188 (2)	0.6232 (2)	0.7534 (2)	5.58 (4)
C10	0.1650 (2)	0.7267 (2)	0.8282 (2)	5.83 (4)
C11	0.2409 (2)	0.6710 (2)	0.9579 (2)	5.22 (4)
C12	0.2691 (3)	0.5085 (3)	1.0112 (2)	6.16 (5)
C13	0.2222 (2)	0.4012 (2)	0.9385 (2)	5.36 (4)
C14	0.2897 (3)	0.7873 (3)	1.0375 (2)	7.34 (5)

Table 2. *Geometric parameters* (Å, °)

S—O1	1.421 (1)	C5—C6	1.398 (2)
S—O2	1.431 (1)	C6—C7	1.428 (2)
S—N	1.633 (1)	C7—C7′	1.189 (2)
S—C8	1.759 (2)	C8—C9	1.382 (2)
N—C1	1.428 (2)	C8—C13	1.373 (2)
C1—C2	1.377 (2)	C9—C10	1.376 (3)
C1—C6	1.401 (2)	C10—C11	1.378 (3)
C2—C3	1.378 (3)	C11—C12	1.373 (3)
C3—C4	1.364 (3)	C11—C14	1.506 (3)
C4—C5	1.363 (3)	C12—C13	1.393 (3)
O1—S—O2	119.49 (8)	C1—C6—C5	118.1 (1)
O1—S—N	107.95 (7)	C1—C6—C7	121.7 (1)
O1—S—C8	108.50 (8)	C5—C6—C7	120.2 (2)
O2—S—N	104.95 (7)	C6—C7—C7′	178.2 (2)
O2—S—C8	108.42 (7)	S—C8—C9	118.4 (1)
N—S—C8	106.85 (7)	S—C8—C13	121.3 (1)
S—N—C1	122.1 (1)	C9—C8—C13	120.4 (2)
N—C1—C2	120.1 (2)	C8—C9—C10	119.7 (2)
N—C1—C6	120.1 (1)	C9—C10—C11	121.3 (2)
C2—C1—C6	119.8 (1)	C10—C11—C12	118.2 (2)
C1—C2—C3	120.4 (2)	C10—C11—C14	120.1 (2)
C2—C3—C4	120.5 (2)	C12—C11—C14	121.7 (2)
C3—C4—C5	119.9 (2)	C11—C12—C13	121.8 (2)
C4—C5—C6	121.3 (2)	C8—C13—C12	118.7 (2)

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles involving H atoms, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71321 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1052]

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Acta Cryst. (1993). C**49**, 2149–2151

Damnacanthal

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(*Received* 21 *January* 1993; *accepted* 4 *June* 1993)

Abstract

The structure of a natural product isolated from the plant *Morinda citrifolia* L. was determined by X-ray diffraction; the compound is shown to be 9,10-dihydro-3-hydroxy-1-methoxy-9,10-dioxo-2-anthracenecarboxaldehyde, $C_{16}H_{10}O_5$. An intramolecular hydrogen bond exists between the hydroxyl and formyl groups with the O···O distance being 2.591 (4) Å.

Comment

A natural product isolated from the plant *Morinda citrifolia* L. was found to inhibit ras oncogene functions (Hiramatsu, Imoto, Koyano & Umezawa, 1993). The X-ray crystal structure analysis revealed