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4-Methyl-*N*-[2-(*p*-tolylsulfanyl)phenyl]benzenesulfonamide

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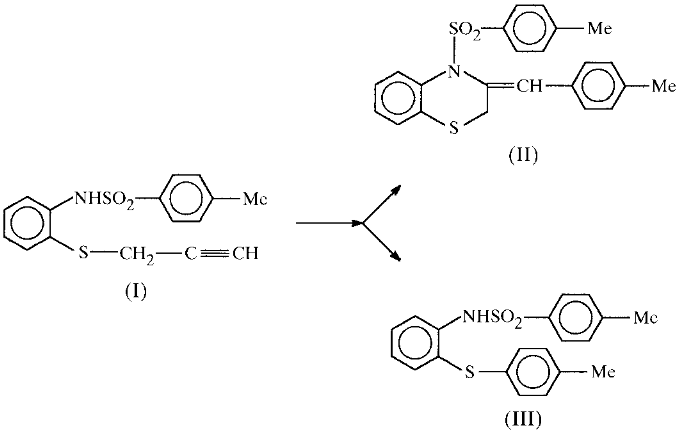
The title compound, $C_{20}H_{19}NO_2S_2$, is formed by a palladium–copper-catalyzed reaction between 4-methyl-*N*-[2-(prop-2-ynylsulfanyl)phenyl]benzenesulfonamide and *p*-iodotoluene. The molecules contain three essentially planar parts, namely an aminothiophenol moiety (*A*), a toluenesulfone moiety excluding the oxo ligands (*B*) and a tolyl group (*C*), approximately orthogonal to each other; the dihedral angles *A*/*B*, *A*/*C* and *B*/*C* are 111.6 (1), 89.3 (1) and 101.4 (1)°, respectively. Intermolecular N—H···O hydrogen bonds link the molecules into infinite one-dimensional chains.

Comment

Metal-catalyzed heteroannulation is an important synthetic tool for the formation of a variety of heterocyclic systems of biological importance (Mukherjee *et al.*, 2000; Kundu *et al.*, 1999). During the synthesis of a sulfur-containing heterocyclic compound, benzothiazine, (II), *via* a palladium–copper-catalyzed reaction between 4-methyl-*N*-[2-(prop-2-ynylsulfanyl)phenyl]benzenesulfonamide, (I), and *p*-iodotoluene, the title compound, 4-methyl-*N*-[2-(*p*-tolylsulfanyl)phenyl]benzenesulfonamide, (III), was obtained through a novel depropargylation and *S*-arylation. The X-ray structural study of (III) was undertaken in order to establish the regio- and stereospecificities of the reaction.

The structure of (III) (Fig. 1) consists of three essentially planar parts (*A*, *B* and *C*) approximately orthogonal to each other. The central aminothiophenol moiety *A* (atoms C8–C13, N1, S2) is planar to within 0.031 (4) Å. The other two parts, *p*-toluenesulfone excluding the two oxo ligands (*B*: atoms C1–C7, S1) and *p*-tolyl (*C*: atoms C14–C20), with a maximum deviation of 0.025 (4) Å for an in-plane atom (C1) from the corresponding least-squares plane, are inclined by 101.4 (1)° with respect to each other. The dihedral angles *A*/*B* and *A*/*C* are 111.6 (1) and 89.3 (1)°, respectively. The torsion angle C5—S1—N1—C8 of 62.4 (3)° establishes a *gauche* conformation of the molecule when viewed along the S—N axis.



The geometric parameters of (III) agree well with those found in other substituted *p*-toluenesulfonamide structures (Parvez *et al.*, 1999; Goswami *et al.*, 1998; Bachechi *et al.*, 1996; Gainsford & Lensink, 1996). The N atom with a bond angle sum of 349.5 (2)° indicates a pyramidal configuration. The angular disposition of the bonds about the sulfonyl S atom (S1) deviate significantly from that of regular tetrahedron. The widening of the O1—S1—O2 angle of 119.1 (1)° from the ideal tetrahedral value is presumably the result of the repulsive interaction between the short S═O bonds (Table 1). The lack of π bonding in the branches among the phenyl rings precludes any possible π conjugation across the whole molecule. The aromatic nature of the rings is therefore localized within the rings and on their direct substituents. One of the sulfonyl O atoms, O1, is nearly coplanar with the tolyl substituent [O1—S1—C5—C6 14.2 (3)°]; the other sulfonyl O atom, O2, forms a torsion angle O2—S1—C5—C6 of 144.8 (3)°.

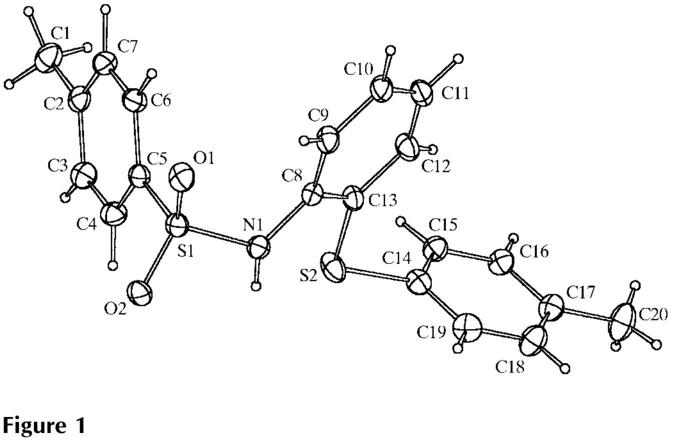


Figure 1
ZORTEP (Zsolnai, 1995) view (50% probability level) of the title molecule.

In the solid state, the molecules translated in the *b* direction are linked through N1—H8···O2 intermolecular hydrogen bonds (Table 2) to form infinite one-dimensional chains.

Experimental

Compound (III) [m.p. 357 (1) K] was synthesized by stirring a mixture of 4-methyl-*N*-[2-(prop-2-ynylsulfanyl)phenyl]benzenesulfonamide, (I) (1.32 mmol), and *p*-iodotoluene (1.33 mmol) in the presence of bis(triphenylphosphine)palladium(II) chloride (0.04 mmol), cuprous iodide (0.52 mmol) and triethylamine (5.23 mmol) followed by refluxing with tetrahydrofuran for 36 h under an argon atmosphere. After usual work-up, the crude product was purified by column chromatography through silica gel using an ethyl acetate–petroleum spirit (333–353 K) mixture (1:9) as eluant, yielded the title compound, (III). Single crystals suitable for X-ray analysis were obtained from a $CHCl_3$–light petroleum (333–353 K) mixture. Elemental analysis: calculated for $C_{20}H_{19}NO_2S_2$: C 65.01, H 5.18, N 3.79%; found C 65.32, H 5.25, N 3.74%.

Crystal data

$C_{20}H_{19}NO_2S_2$
$M_r = 369.48$
Triclinic, $P\overline{1}$
$a = 10.530$ (4) Å
$b = 11.873$ (3) Å
$c = 8.798$ (4) Å
$\alpha = 90.76$ (4)°
$\beta = 113.31$ (4)°
$\gamma = 112.05$ (2)°
$V = 919.2$ (6) Å^3

$Z = 2$
$D_x = 1.335$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 33.5$–39.8°
$\mu = 2.727$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.35 × 0.25 × 0.20 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.449$, $T_{\max} = 0.580$
3497 measured reflections
3301 independent reflections
2610 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.039$
$\theta_{\max} = 70.11°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 13$
$l = -9 \rightarrow 10$
3 standard reflections every 150 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.147$
$S = 1.031$
3301 reflections
227 parameters
H atoms were constrained

$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.3704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.427 (2)	S2—C13	1.783 (3)
S1—O2	1.437 (2)	S2—C14	1.790 (3)
S1—N1	1.647 (2)	N1—C8	1.437 (3)
S1—C5	1.765 (3)		
O1—S1—O2	119.09 (13)	O2—S1—C5	108.79 (13)
O1—S1—N1	108.45 (13)	N1—S1—C5	106.84 (12)
O2—S1—N1	104.96 (12)	C13—S2—C14	101.85 (14)
O1—S1—C5	108.09 (13)	C8—N1—S1	122.21 (18)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H8···S2	0.98	2.55	3.025 (3)	110
N1—H8···O2$^{\rm i}$	0.98	2.13	3.039 (4)	154

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

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1105). Services for accessing these data are described at the back of the journal.

References

Bachechi, F., Coiro, V. M. & Delfini, M. (1996). *Acta Cryst.* C**52**, 2915–2917.

Debaerdemaeker, T., Germain, G., Main, P., Refaat, L. S., Tate, C. & Woolfson, M. M. W. (1988). *MULTAN*88. Universities of York, England, and Louvain, Belgium.

Gainsford, G. J. & Lensink, C. (1996). *Acta Cryst.* C**52**, 472–474.

Goswami, S., Mahapatra, A. K., Nigam, G. D., Chinnakali, K. & Fun, H.-K. (1998). *Acta Cryst.* C**54**, 1301–1302.

Kundu, N. G., Khan, M. W., Guha, S. & Mukherjee, A. K. (1999). *Acta Cryst.* C**55**, 239–241.

Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1995). *TEXSAN*. Version 1.7-1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Mukherjee, A. K., Guha, S., Khan, M. W., Kundu, N. G. & Helliwell, M. (2000). *Acta Cryst.* C**56**, 85–87.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

North, A. C. T., Philips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Parvez, M., Arayne, M. S., Kamranzaman, M. & Sultana, N. (1999). *Acta Cryst.* C**55**, 74–75.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Zsolnai, L. (1995). *ZORTEP*. University of Heidelberg, Germany.