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(E)-N'-(4-Bromobenzylidene)-p-toluene-sulfonohydrazideReza Kia,^a Bijan Etemadi,^b Hoong-Kun Fun^{a*} and Hadi Kargar^c

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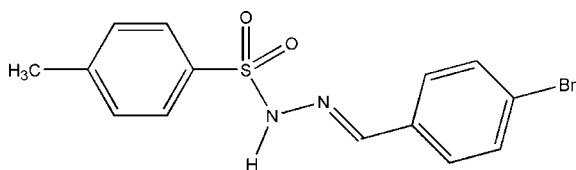
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.040; wR factor = 0.106; data-to-parameter ratio = 27.0.

In the title compound, $\text{C}_{14}\text{H}_{13}\text{BrN}_2\text{O}_2\text{S}$, a novel sulfonamide derivative, intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link neighbouring molecules into different dimers along the b axis, generating $R_2^2(8)$ and $R_2^2(16)$ ring motifs. The dihedral angle between the benzene rings is $82.39(13)^\circ$. The crystal structure is further stabilized by intermolecular $\pi-\pi$ stacking interactions [centroid-centroid distances = $3.867(2)-3.9548(8)$ Å].

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures and applications, see, for example: Kia *et al.* (2008a,b); Tabatabaee *et al.* (2007); Ali *et al.* (2007); Tierney *et al.* 2006; Krygowski *et al.* (1998); Mehrabi *et al.* (2008); Kayser *et al.* (2004). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{13}\text{BrN}_2\text{O}_2\text{S}$
 $M_r = 353.23$
Triclinic, $P\bar{1}$
 $a = 5.9565(3)$ Å
 $b = 9.4005(3)$ Å
 $c = 12.8020(6)$ Å
 $\alpha = 97.153(2)^\circ$
 $\beta = 96.350(2)^\circ$

$\gamma = 92.125(1)^\circ$
 $V = 705.95(5)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 3.06$ mm⁻¹
 $T = 100$ K
 $0.57 \times 0.15 \times 0.07$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2005)
 $T_{\min} = 0.276$, $T_{\max} = 0.819$

18030 measured reflections
5040 independent reflections
4098 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.106$
 $S = 1.11$
5040 reflections
187 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.63$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.56$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O1}^{\text{i}}$	0.85 (4)	2.06 (4)	2.902 (3)	171 (4)
$\text{C14}-\text{H14A}\cdots\text{O1}^{\text{ii}}$	0.98	2.57	3.420 (3)	145

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x, -y + 2, -z + 1$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2299).

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supplementary materials

Acta Cryst. (2009). E65, o821-o822 [doi:10.1107/S1600536809009751]

(*E*)-*N'*-(4-Bromobenzylidene)-*p*-toluenesulfonohydrazide

R. Kia, B. Etemadi, H.-K. Fun and H. Kargar

Comment

Sulfonamides were the first class of antimicrobial agents to be discovered. They inhibit dihydropteroate synthetase in the bacterial folic acid pathway. Although their clinical role has diminished, they are still useful in certain situations, because of its efficacy and low cost (Krygowski *et al.*, 1998). Sulfonamides (sulfanilamide, sulfamethoxazole, sulfafurazole) are structural analogues of *p*-aminobenzoic acid (PABA) and compete with PABA to block its conversion to dihydrofolic acid. These agents are generally used in combination with other drugs (usually sulfonamides) to prevent or treat a number of bacterial and parasitic infections (Tierney *et al.*, 2006). Because of the above important features, we report the crystal structure of the title compound.

The title compound (Fig. 1), is a novel sulfonamide derivative. Bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges and are comparable with the related structures (Kia *et al.* 2008*a,b*; Mehrabi *et al.*, 2008; Ali *et al.* 2007). Intermolecular N—H...O and C—H...O hydrogen bonds link neighbouring molecules by $R_2^2(8)$ and $R_2^2(16)$ ring motifs (Bernstein *et al.*, 1995), respectively, into different dimers along the *b* axis. The dihedral angle between the two benzene rings is 82.39 (13)°. The crystal structure is further stabilized by intermolecular π - π stacking interactions [$Cg1 \cdots Cg1^{iii} = 3.9319(15)$ and $Cg2 \cdots Cg2^{iv} = 3.8677(16)$ Å; the perpendicular distances are 3.6548 (10) and 3.6189 (11) Å, respectively. Symmetry codes: (iii) $-x, 2 - y, 1 - z$; (iv) $2 - x, 1 - y, 2 - z$].

Experimental

p-Tosylhydrazine (2 mmol) was added to a 50 ml refluxing ethanolic solution of 4-bromobenzaldehyde (2 mmol). The mixture was stirred for 2 h. After cooling, the colorless crystalline solid was isolated by filtration, washed with cold ethanol, and re-crystallized from ethanol.

Refinement

H atom bound to N1 was located from the difference Fourier map and refined freely; see Table 1. The rest of the hydrogen atoms were positioned geometrically and refined as riding model. A rotating group model was used for the methyl group. The highest peak (0.63 e.Å⁻³) is located 1.77 Å from H2A and the deepest hole (-0.56 e.Å⁻³) is located 1.47 Å from H2A. The crystal structure was twinned by a pseudo-twofold rotation about (0 1 0) with a refined BASF ratio of 0.115 (1)/0.885 (1).

Figures

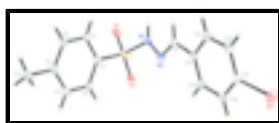


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering.

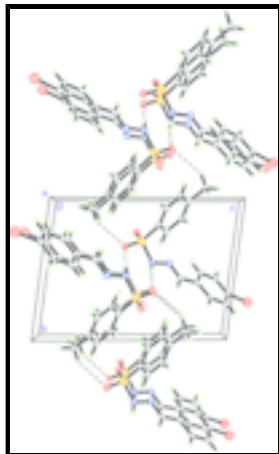


Fig. 2. The crystal packing of the title compound, viewed down the *a* axis, showing linking of different dimers along the *b* axis through N—H...O and C—H...O interactions. Intermolecular hydrogen bonds are shown as dashed lines.

(*E*)-*N'*-(4-Bromobenzylidene)-*p*-toluenesulfonohydrazide

Crystal data

C₁₄H₁₃BrN₂O₂S

M_r = 353.23

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

a = 5.9565 (3) Å

b = 9.4005 (3) Å

c = 12.8020 (6) Å

α = 97.153 (2)°

β = 96.350 (2)°

γ = 92.125 (1)°

V = 705.95 (5) Å³

Z = 2

*F*₀₀₀ = 356

D_x = 1.662 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 7222 reflections

θ = 2.5–34.1°

μ = 3.06 mm⁻¹

T = 100 K

Block, colourless

0.57 × 0.15 × 0.07 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 100 K

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

T_{min} = 0.276, *T_{max}* = 0.819

18030 measured reflections

5040 independent reflections

4098 reflections with *I* > 2 σ (*I*)

R_{int} = 0.034

θ_{max} = 32.5°

θ_{min} = 2.2°

h = -9→8

k = -14→14

l = -18→19

Refinement

Refinement on *F*²

Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 1.0631P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
5040 reflections	$(\Delta/\sigma)_{\max} < 0.001$
187 parameters	$\Delta\rho_{\max} = 0.63 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.81285 (5)	0.24366 (3)	-0.11229 (2)	0.02400 (8)
S1	0.25512 (10)	0.69735 (6)	0.50609 (4)	0.01269 (11)
O1	0.1164 (3)	0.66003 (19)	0.58555 (14)	0.0166 (3)
O2	0.4777 (3)	0.76093 (19)	0.53840 (15)	0.0171 (3)
N1	0.2703 (4)	0.5426 (2)	0.43423 (17)	0.0147 (4)
N2	0.3865 (4)	0.5374 (2)	0.34562 (16)	0.0149 (4)
C1	-0.1178 (4)	0.7666 (3)	0.3846 (2)	0.0162 (4)
H1A	-0.1895	0.6830	0.4034	0.019*
C2	-0.2334 (4)	0.8507 (3)	0.3168 (2)	0.0187 (5)
H2A	-0.3851	0.8231	0.2884	0.022*
C3	-0.1313 (5)	0.9747 (3)	0.2894 (2)	0.0179 (5)
C4	0.0912 (5)	1.0145 (3)	0.3328 (2)	0.0194 (5)
H4A	0.1614	1.1000	0.3162	0.023*
C5	0.2112 (4)	0.9315 (3)	0.3994 (2)	0.0162 (4)
H5A	0.3632	0.9587	0.4275	0.019*
C6	0.1059 (4)	0.8075 (2)	0.42462 (19)	0.0139 (4)
C7	0.3238 (4)	0.4296 (3)	0.2753 (2)	0.0159 (4)
H7A	0.1980	0.3695	0.2850	0.019*
C8	0.4385 (4)	0.3958 (3)	0.18089 (19)	0.0156 (4)

supplementary materials

C9	0.3420 (5)	0.2868 (3)	0.1030 (2)	0.0190 (5)
H9A	0.2005	0.2417	0.1106	0.023*
C10	0.4502 (5)	0.2435 (3)	0.0144 (2)	0.0208 (5)
H10A	0.3833	0.1699	-0.0387	0.025*
C11	0.6578 (4)	0.3098 (3)	0.0051 (2)	0.0176 (5)
C12	0.7559 (5)	0.4195 (3)	0.0805 (2)	0.0196 (5)
H12A	0.8971	0.4646	0.0722	0.023*
C13	0.6460 (5)	0.4627 (3)	0.1682 (2)	0.0180 (5)
H13A	0.7119	0.5381	0.2200	0.022*
C14	-0.2579 (5)	1.0639 (3)	0.2146 (2)	0.0255 (6)
H14A	-0.2504	1.1643	0.2471	0.038*
H14B	-0.4164	1.0282	0.2000	0.038*
H14C	-0.1893	1.0572	0.1481	0.038*
H1N1	0.160 (6)	0.483 (4)	0.436 (3)	0.020 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02205 (14)	0.03319 (15)	0.01596 (12)	0.00426 (11)	0.00449 (10)	-0.00286 (10)
S1	0.0128 (3)	0.0121 (2)	0.0130 (2)	-0.00060 (19)	0.0009 (2)	0.00153 (18)
O1	0.0189 (9)	0.0168 (8)	0.0144 (8)	-0.0017 (7)	0.0046 (7)	0.0014 (6)
O2	0.0138 (8)	0.0154 (8)	0.0213 (9)	-0.0018 (6)	-0.0020 (7)	0.0039 (6)
N1	0.0160 (10)	0.0117 (8)	0.0167 (9)	-0.0009 (7)	0.0047 (8)	0.0009 (7)
N2	0.0151 (10)	0.0159 (9)	0.0141 (9)	0.0022 (7)	0.0030 (7)	0.0020 (7)
C1	0.0137 (11)	0.0157 (10)	0.0186 (11)	-0.0015 (8)	0.0005 (9)	0.0017 (8)
C2	0.0155 (11)	0.0206 (11)	0.0190 (11)	0.0027 (9)	-0.0012 (9)	0.0010 (9)
C3	0.0232 (13)	0.0135 (10)	0.0158 (10)	0.0046 (9)	-0.0007 (9)	-0.0011 (8)
C4	0.0237 (13)	0.0123 (10)	0.0219 (12)	0.0007 (9)	0.0014 (10)	0.0027 (9)
C5	0.0166 (11)	0.0138 (10)	0.0174 (11)	-0.0009 (8)	0.0007 (9)	0.0006 (8)
C6	0.0150 (10)	0.0131 (9)	0.0136 (10)	0.0004 (8)	0.0020 (8)	0.0017 (8)
C7	0.0154 (11)	0.0151 (10)	0.0178 (11)	0.0011 (8)	0.0029 (9)	0.0031 (8)
C8	0.0159 (11)	0.0159 (10)	0.0154 (10)	0.0034 (8)	0.0011 (9)	0.0036 (8)
C9	0.0168 (12)	0.0195 (11)	0.0202 (12)	0.0015 (9)	0.0013 (9)	0.0007 (9)
C10	0.0207 (13)	0.0228 (12)	0.0169 (11)	0.0010 (10)	0.0005 (10)	-0.0033 (9)
C11	0.0175 (11)	0.0214 (11)	0.0142 (10)	0.0045 (9)	0.0022 (9)	0.0023 (9)
C12	0.0188 (12)	0.0212 (11)	0.0197 (12)	0.0010 (9)	0.0055 (10)	0.0037 (9)
C13	0.0195 (12)	0.0170 (11)	0.0172 (11)	-0.0014 (9)	0.0028 (9)	0.0008 (9)
C14	0.0335 (16)	0.0178 (11)	0.0226 (13)	0.0073 (11)	-0.0081 (11)	0.0010 (9)

Geometric parameters (\AA , $^\circ$)

Br1—C11	1.901 (2)	C5—C6	1.393 (3)
S1—O2	1.4297 (19)	C5—H5A	0.9500
S1—O1	1.4469 (18)	C7—C8	1.460 (3)
S1—N1	1.633 (2)	C7—H7A	0.9500
S1—C6	1.756 (2)	C8—C9	1.396 (4)
N1—N2	1.389 (3)	C8—C13	1.400 (4)
N1—H1N1	0.85 (3)	C9—C10	1.390 (4)
N2—C7	1.283 (3)	C9—H9A	0.9500

C1—C2	1.390 (3)	C10—C11	1.388 (4)
C1—C6	1.395 (3)	C10—H10A	0.9500
C1—H1A	0.9500	C11—C12	1.385 (4)
C2—C3	1.396 (4)	C12—C13	1.387 (4)
C2—H2A	0.9500	C12—H12A	0.9500
C3—C4	1.398 (4)	C13—H13A	0.9500
C3—C14	1.509 (4)	C14—H14A	0.9800
C4—C5	1.386 (4)	C14—H14B	0.9800
C4—H4A	0.9500	C14—H14C	0.9800
O2—S1—O1	119.47 (11)	N2—C7—C8	122.5 (2)
O2—S1—N1	109.68 (11)	N2—C7—H7A	118.8
O1—S1—N1	102.07 (11)	C8—C7—H7A	118.8
O2—S1—C6	108.68 (11)	C9—C8—C13	119.0 (2)
O1—S1—C6	109.36 (11)	C9—C8—C7	118.2 (2)
N1—S1—C6	106.81 (11)	C13—C8—C7	122.7 (2)
N2—N1—S1	118.59 (16)	C10—C9—C8	120.9 (2)
N2—N1—H1N1	120 (2)	C10—C9—H9A	119.5
S1—N1—H1N1	114 (2)	C8—C9—H9A	119.5
C7—N2—N1	113.5 (2)	C11—C10—C9	118.7 (2)
C2—C1—C6	118.6 (2)	C11—C10—H10A	120.6
C2—C1—H1A	120.7	C9—C10—H10A	120.6
C6—C1—H1A	120.7	C12—C11—C10	121.6 (2)
C1—C2—C3	121.4 (2)	C12—C11—Br1	119.75 (19)
C1—C2—H2A	119.3	C10—C11—Br1	118.67 (19)
C3—C2—H2A	119.3	C11—C12—C13	119.3 (2)
C2—C3—C4	118.5 (2)	C11—C12—H12A	120.4
C2—C3—C14	120.8 (2)	C13—C12—H12A	120.4
C4—C3—C14	120.6 (2)	C12—C13—C8	120.5 (2)
C5—C4—C3	121.2 (2)	C12—C13—H13A	119.8
C5—C4—H4A	119.4	C8—C13—H13A	119.8
C3—C4—H4A	119.4	C3—C14—H14A	109.5
C4—C5—C6	119.1 (2)	C3—C14—H14B	109.5
C4—C5—H5A	120.5	H14A—C14—H14B	109.5
C6—C5—H5A	120.5	C3—C14—H14C	109.5
C5—C6—C1	121.2 (2)	H14A—C14—H14C	109.5
C5—C6—S1	120.30 (19)	H14B—C14—H14C	109.5
C1—C6—S1	118.52 (18)		
O2—S1—N1—N2	-54.7 (2)	O2—S1—C6—C1	-179.30 (19)
O1—S1—N1—N2	177.61 (18)	O1—S1—C6—C1	-47.3 (2)
C6—S1—N1—N2	62.9 (2)	N1—S1—C6—C1	62.4 (2)
S1—N1—N2—C7	-156.20 (19)	N1—N2—C7—C8	-173.9 (2)
C6—C1—C2—C3	-0.8 (4)	N2—C7—C8—C9	-172.5 (2)
C1—C2—C3—C4	-0.6 (4)	N2—C7—C8—C13	10.9 (4)
C1—C2—C3—C14	179.2 (2)	C13—C8—C9—C10	0.7 (4)
C2—C3—C4—C5	1.6 (4)	C7—C8—C9—C10	-176.0 (2)
C14—C3—C4—C5	-178.3 (2)	C8—C9—C10—C11	0.6 (4)
C3—C4—C5—C6	-1.0 (4)	C9—C10—C11—C12	-1.5 (4)
C4—C5—C6—C1	-0.5 (4)	C9—C10—C11—Br1	176.8 (2)

supplementary materials

C4—C5—C6—S1	178.40 (19)	C10—C11—C12—C13	1.0 (4)
C2—C1—C6—C5	1.4 (4)	Br1—C11—C12—C13	-177.21 (19)
C2—C1—C6—S1	-177.52 (19)	C11—C12—C13—C8	0.3 (4)
O2—S1—C6—C5	1.8 (2)	C9—C8—C13—C12	-1.2 (4)
O1—S1—C6—C5	133.8 (2)	C7—C8—C13—C12	175.4 (2)
N1—S1—C6—C5	-116.5 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N1···O1 ⁱ	0.85 (4)	2.06 (4)	2.902 (3)	171 (4)
C14—H14A···O1 ⁱⁱ	0.98	2.57	3.420 (3)	145

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x, -y+2, -z+1$.

Fig. 1

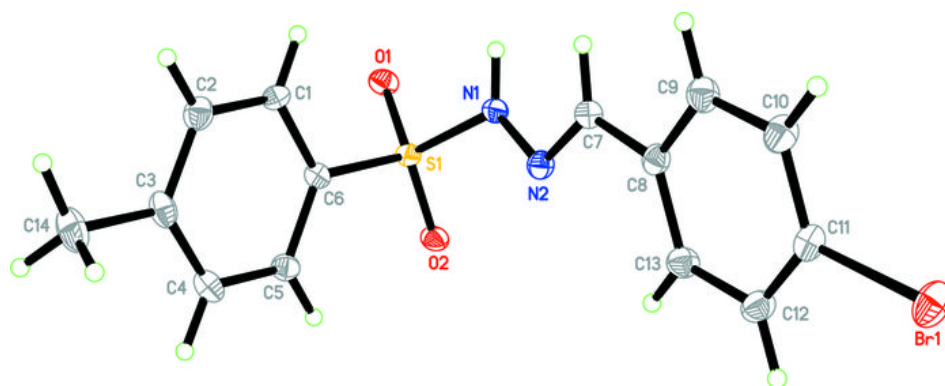


Fig. 2

