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N-(4-Bromophenylsulfonyl)-2,2,2-trimethylacetamide

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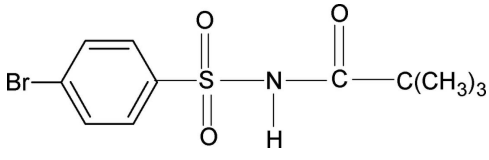
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 17.5.

The conformations of the N—H and C=O bonds in the SO_2—NH—CO—C group of the title compound (N4BPSTMAA), $C_{11}H_{14}BrNO_3S$, are *trans* to each other, similar to what is observed in *N*-(4-chlorophenylsulfonyl)-2,2,2-trimethylacetamide (N4CPSTMAA) and 2,2,2-trimethyl-*N*-(4-methylphenylsulfonyl)acetamide (N4MPSTMAA). The bond parameters in N4BPSTMAA are similar to those in N4CPSTMAA, N4MPSTMAA, *N*-aryl-2,2,2-trimethylacetamides and 4-bromobenzenesulfonamide. The benzene ring and the SO_2—NH—CO—C group in N4BPSTMAA form a dihedral angle of 82.8 (1)°, comparable with the values of 82.2 (1)° in N4CPSTMAA and 71.2 (1)° in N4MPSTMAA. N—H···O hydrogen bonds form a centrosymmetric ring characterized by an $R_2^2(8)$ motif.

Related literature

For related literature, see: Gowda *et al.* (2003, 2007, 2008); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{11}H_{14}BrNO_3S$
M_r = 320.20
Triclinic, $P\overline{1}$
a = 6.066 (1) Å
b = 10.858 (1) Å
c = 11.092 (2) Å
α = 68.19 (1)°
β = 78.66 (2)°
γ = 88.10 (2)°
V = 664.40 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.25 mm^{-1}
T = 299 (2) K
0.20 × 0.08 × 0.04 mm

Data collection

Oxford Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.563, T_{max} = 0.881
6843 measured reflections
2692 independent reflections
1551 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.101
S = 0.97
2692 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.86	2.23	2.982 (3)	146

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

BTG thanks the Alexander von Humboldt Foundation, Bonn, Germany, for extensions of his research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2154).

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supporting information

Acta Cryst. (2008). E**64**, o1389 [doi:10.1107/S1600536808019375]

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S1. Comment

In the present work, as part of a study of the substituent effects on the solid state geometries of *N*-(aryl)-sulfonamides and substituted amides, the structure of *N*-(4-bromophenylsulfonyl)-2,2,2-trimethylacetamide (N4BPSTMAA) has been determined (Gowda *et al.*, 2003, 2007, 2008). The conformations of the N—H and C═O bonds of the SO2—NH—CO—C group in N4CPSTMAA are *anti* to each other (Fig. 1), similar to that observed in *N*-(4-chlorophenylsulfonyl)-2,2,2-trimethylacetamide (N4CPSTMAA) and (4-methylphenylsulfonyl)-2,2,2-trimethylacetamide (N4MPSTMAA) (Gowda *et al.*, 2008). The bond parameters in N4BPSTMAA are similar to those in N4CPSTMAA, N4MPSTMAA (Gowda *et al.*, 2008), *N*-(aryl)-2,2,2-trimethylacetamides (Gowda *et al.*, 2007) and 4-bromobenzenesulfonamide (Gowda *et al.*, 2003). The N—H···O hydrogen bonds form a centrosymmetric macro-ring characterized by $R_2^2(8)$ motif (Bernstein *et al.*, 1995) (Table 1, Fig. 2).

S2. Experimental

The title compound was prepared by refluxing 4-bromobenzenesulfonamide (0.10 mole) with an excess pivalyl chloride (0.20 mole) for about an hour on a water bath. The reaction mixture was cooled and poured into ice cold water. The resulting solid was separated, washed thoroughly with water and dissolved in warm dilute sodium hydrogen carbonate solution. The title compound was precipitated by acidifying the filtered solution with glacial acetic acid. It was filtered, dried and recrystallized from ethanol. The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra. Single crystals of the title compound used for X-ray diffraction studies were obtained from a slow evaporation of an ethanolic solution.

S3. Refinement

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å, N—H = 0.86 Å, and were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

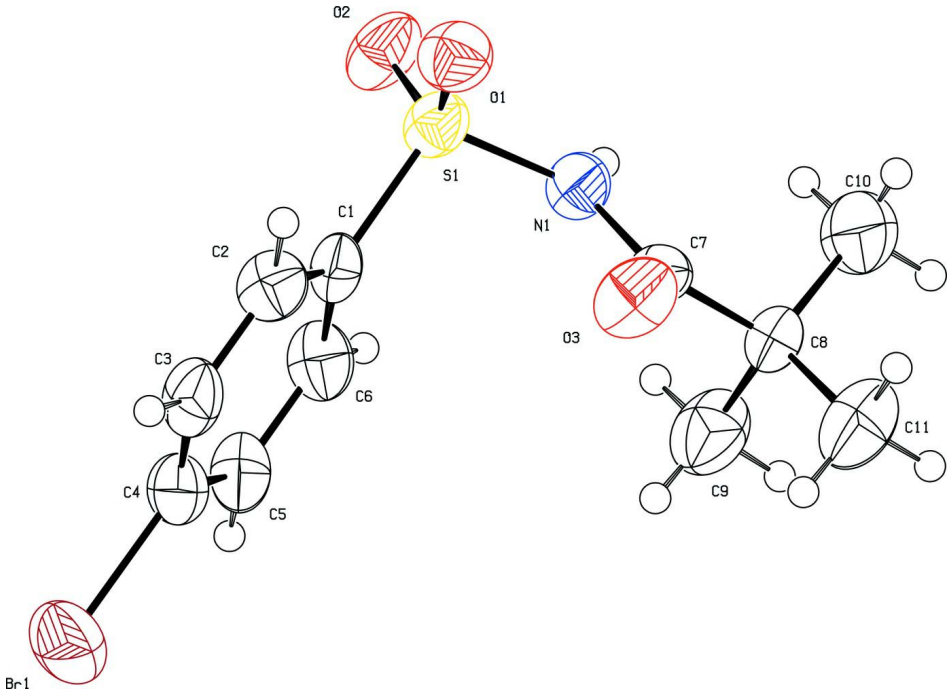

Figure 1

Molecular structure of the title compound, showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

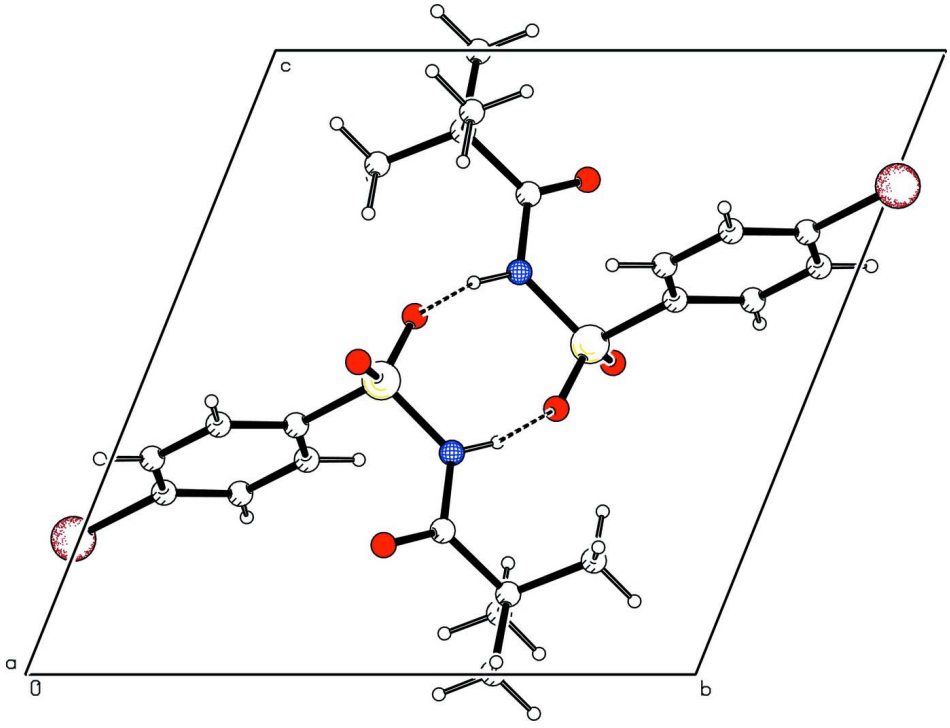

Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

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Crystal data

$C_{11}H_{14}BrNO_3S$
$M_r = 320.20$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.066$ (1) Å
$b = 10.858$ (1) Å
$c = 11.092$ (2) Å
$\alpha = 68.19$ (1)°
$\beta = 78.66$ (2)°
$\gamma = 88.10$ (2)°
$V = 664.40$ (17) Å^3
$Z = 2$
$F(000) = 324$
$D_x = 1.601$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2537 reflections
$\theta = 2.3$–28.0°
$\mu = 3.25$ mm^{-1}
$T = 299$ K
Needle, colourless
0.20 × 0.08 × 0.04 mm

Data collection

Oxford Xcalibur diffractometer with Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and φ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007) (Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm)
$T_{min} = 0.563$, $T_{max} = 0.881$
6843 measured reflections
2692 independent reflections
1551 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.3°$
$h = -7 \rightarrow 7$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.101$
$S = 0.97$
2692 reflections
154 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.10728 (8)	−0.00925 (4)	0.21716 (5)	0.0810 (2)
S1	0.28409 (14)	0.35519 (8)	0.47093 (8)	0.0451 (2)
O1	0.4965 (4)	0.3098 (2)	0.5007 (2)	0.0548 (6)
O2	0.1120 (4)	0.3668 (2)	0.5741 (2)	0.0580 (6)
O3	0.5763 (4)	0.4572 (2)	0.2073 (2)	0.0615 (7)
N1	0.3085 (4)	0.5038 (2)	0.3551 (3)	0.0449 (7)
H1N	0.2309	0.5653	0.3720	0.054*
C1	0.1807 (5)	0.2548 (3)	0.3993 (3)	0.0399 (8)
C2	0.2864 (6)	0.1387 (3)	0.4005 (3)	0.0478 (8)
H2	0.4134	0.1145	0.4378	0.057*
C3	0.2013 (6)	0.0601 (3)	0.3458 (3)	0.0513 (9)
H3	0.2703	−0.0176	0.3456	0.062*
C4	0.0124 (6)	0.0983 (3)	0.2914 (3)	0.0487 (9)
C5	−0.0926 (6)	0.2124 (3)	0.2896 (4)	0.0521 (9)
H5	−0.2193	0.2364	0.2520	0.062*
C6	−0.0080 (5)	0.2915 (3)	0.3443 (4)	0.0506 (9)
H6	−0.0778	0.3691	0.3440	0.061*
C7	0.4476 (6)	0.5366 (3)	0.2305 (3)	0.0428 (8)
C8	0.4154 (5)	0.6724 (3)	0.1284 (3)	0.0451 (8)
C9	0.1793 (6)	0.6700 (4)	0.0986 (4)	0.0740 (12)
H9A	0.1665	0.6008	0.0657	0.089*
H9B	0.1552	0.7540	0.0331	0.089*
H9C	0.0686	0.6536	0.1782	0.089*
C10	0.4365 (8)	0.7799 (4)	0.1826 (4)	0.0856 (14)
H10A	0.3232	0.7639	0.2611	0.103*
H10B	0.4169	0.8649	0.1172	0.103*
H10C	0.5828	0.7788	0.2037	0.103*
C11	0.5872 (7)	0.6969 (4)	0.0018 (4)	0.0759 (12)
H11A	0.7360	0.6951	0.0197	0.091*
H11B	0.5660	0.7821	−0.0628	0.091*
H11C	0.5684	0.6290	−0.0318	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1133 (4)	0.0498 (3)	0.0888 (4)	−0.0078 (2)	−0.0360 (3)	−0.0269 (2)
S1	0.0570 (6)	0.0369 (5)	0.0389 (5)	0.0063 (4)	−0.0065 (4)	−0.0130 (4)
O1	0.0597 (15)	0.0513 (14)	0.0543 (15)	0.0087 (12)	−0.0183 (13)	−0.0177 (12)
O2	0.0759 (16)	0.0479 (14)	0.0402 (14)	0.0118 (12)	0.0055 (13)	−0.0145 (11)
O3	0.0650 (16)	0.0606 (16)	0.0540 (16)	0.0187 (14)	−0.0034 (13)	−0.0213 (13)
N1	0.0600 (17)	0.0323 (14)	0.0429 (17)	0.0063 (13)	−0.0063 (14)	−0.0169 (13)
C1	0.0397 (18)	0.0325 (17)	0.0390 (18)	−0.0038 (14)	0.0030 (15)	−0.0084 (14)
C2	0.051 (2)	0.0410 (19)	0.051 (2)	0.0136 (16)	−0.0139 (17)	−0.0153 (16)
C3	0.065 (2)	0.0285 (17)	0.054 (2)	0.0035 (16)	−0.0028 (19)	−0.0118 (16)
C4	0.060 (2)	0.0319 (17)	0.052 (2)	0.0014 (16)	−0.0134 (19)	−0.0121 (16)

C5	0.0446 (19)	0.042 (2)	0.059 (2)	−0.0033 (16)	−0.0081 (17)	−0.0081 (17)
C6	0.048 (2)	0.0362 (18)	0.065 (2)	0.0086 (16)	−0.0116 (19)	−0.0167 (17)
C7	0.045 (2)	0.049 (2)	0.0371 (19)	−0.0025 (17)	−0.0069 (16)	−0.0188 (16)
C8	0.048 (2)	0.0432 (19)	0.0356 (19)	−0.0061 (15)	−0.0020 (16)	−0.0076 (15)
C9	0.063 (3)	0.076 (3)	0.065 (3)	0.001 (2)	−0.021 (2)	−0.002 (2)
C10	0.144 (4)	0.041 (2)	0.070 (3)	−0.005 (2)	−0.029 (3)	−0.016 (2)
C11	0.071 (3)	0.077 (3)	0.057 (3)	−0.001 (2)	0.001 (2)	−0.006 (2)

Geometric parameters (Å, º)

Br1—C4	1.891 (3)	C5—H5	0.9300
S1—O1	1.420 (2)	C6—H6	0.9300
S1—O2	1.430 (2)	C7—C8	1.524 (5)
S1—N1	1.636 (3)	C8—C11	1.514 (5)
S1—C1	1.760 (3)	C8—C10	1.517 (5)
O3—C7	1.208 (4)	C8—C9	1.535 (5)
N1—C7	1.394 (4)	C9—H9A	0.9600
N1—H1N	0.8600	C9—H9B	0.9600
C1—C6	1.380 (4)	C9—H9C	0.9600
C1—C2	1.392 (4)	C10—H10A	0.9600
C2—C3	1.378 (4)	C10—H10B	0.9600
C2—H2	0.9300	C10—H10C	0.9600
C3—C4	1.380 (5)	C11—H11A	0.9600
C3—H3	0.9300	C11—H11B	0.9600
C4—C5	1.369 (4)	C11—H11C	0.9600
C5—C6	1.381 (4)		
O1—S1—O2	118.80 (15)	O3—C7—N1	120.2 (3)
O1—S1—N1	111.33 (14)	O3—C7—C8	124.0 (3)
O2—S1—N1	103.71 (14)	N1—C7—C8	115.7 (3)
O1—S1—C1	108.67 (15)	C11—C8—C10	110.9 (3)
O2—S1—C1	109.40 (15)	C11—C8—C7	109.5 (3)
N1—S1—C1	103.87 (14)	C10—C8—C7	110.3 (3)
C7—N1—S1	123.8 (2)	C11—C8—C9	108.6 (3)
C7—N1—H1N	118.1	C10—C8—C9	110.0 (3)
S1—N1—H1N	118.1	C7—C8—C9	107.6 (3)
C6—C1—C2	120.7 (3)	C8—C9—H9A	109.5
C6—C1—S1	119.2 (2)	C8—C9—H9B	109.5
C2—C1—S1	120.1 (2)	H9A—C9—H9B	109.5
C3—C2—C1	119.4 (3)	C8—C9—H9C	109.5
C3—C2—H2	120.3	H9A—C9—H9C	109.5
C1—C2—H2	120.3	H9B—C9—H9C	109.5
C2—C3—C4	119.1 (3)	C8—C10—H10A	109.5
C2—C3—H3	120.5	C8—C10—H10B	109.5
C4—C3—H3	120.5	H10A—C10—H10B	109.5
C5—C4—C3	122.0 (3)	C8—C10—H10C	109.5
C5—C4—Br1	118.6 (3)	H10A—C10—H10C	109.5
C3—C4—Br1	119.5 (2)	H10B—C10—H10C	109.5

C4—C5—C6	119.2 (3)	C8—C11—H11A	109.5
C4—C5—H5	120.4	C8—C11—H11B	109.5
C6—C5—H5	120.4	H11A—C11—H11B	109.5
C1—C6—C5	119.7 (3)	C8—C11—H11C	109.5
C1—C6—H6	120.1	H11A—C11—H11C	109.5
C5—C6—H6	120.1	H11B—C11—H11C	109.5
O1—S1—N1—C7	55.9 (3)	C3—C4—C5—C6	−0.3 (5)
O2—S1—N1—C7	−175.2 (2)	Br1—C4—C5—C6	179.6 (3)
C1—S1—N1—C7	−60.9 (3)	C2—C1—C6—C5	−0.1 (5)
O1—S1—C1—C6	−170.6 (3)	S1—C1—C6—C5	−179.0 (3)
O2—S1—C1—C6	58.2 (3)	C4—C5—C6—C1	0.2 (5)
N1—S1—C1—C6	−52.0 (3)	S1—N1—C7—O3	−7.6 (4)
O1—S1—C1—C2	10.5 (3)	S1—N1—C7—C8	169.3 (2)
O2—S1—C1—C2	−120.7 (3)	O3—C7—C8—C11	−6.7 (4)
N1—S1—C1—C2	129.1 (3)	N1—C7—C8—C11	176.5 (3)
C6—C1—C2—C3	0.1 (5)	O3—C7—C8—C10	−129.0 (4)
S1—C1—C2—C3	179.0 (3)	N1—C7—C8—C10	54.2 (4)
C1—C2—C3—C4	−0.2 (5)	O3—C7—C8—C9	111.0 (4)
C2—C3—C4—C5	0.3 (5)	N1—C7—C8—C9	−65.7 (4)
C2—C3—C4—Br1	−179.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.86	2.23	2.982 (3)	146

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.