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4-Methyl-*N*-(3-oxo-2,3-dihydro-1,2-benzisothiazol-2-yl)benzenesulfonamide

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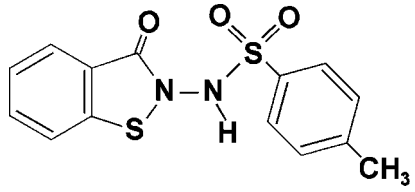
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.089; data-to-parameter ratio = 18.1.

In the title molecule, $C_{14}H_{12}N_2O_3S_2$, the benzisothiazolone ring system is essentially planar and forms a dihedral angle of 67.37 (6)° with the plane of the benzene ring. In the crystal structure, molecules are linked *via* intermolecular N—H···O and C—H···O hydrogen bonds to form chains parallel to the *b* axis.

Related literature

For the chemical and biological properties of 1,2-benzisothiazol-3(2*H*)-one derivatives, see: Clerici *et al.* (2007); Siegemund *et al.* (2002). For 2-amino-1,2-benzisothiazol-3(2*H*)-one derivatives with antiplatelet/spasmolitic effects, see: Vicini *et al.* (1997,2000). For derivatives with antimicrobial properties, see: Vicini *et al.* (2002); Zani *et al.* (2004). For the synthesis of the title compound, see: Vicini *et al.* (2009). For the crystal structures of related compounds, see: Cavalca *et al.* (1970); Ranganathan *et al.* (2002); Steinfeld & Kersting (2006); Kim *et al.* (1996); Xu *et al.* (2006); Sarma & Mugesh (2007); Kolberg *et al.* (1999).



Experimental

Crystal data

$C_{14}H_{12}N_2O_3S_2$
M_r = 320.38
Monoclinic, $P2_1/n$
a = 8.051 (3) Å
b = 7.655 (3) Å
c = 23.910 (10) Å
β = 98.490 (8)°
V = 1457.4 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 (2) K
0.28 × 0.26 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.892, T_{max} = 0.957
17685 measured reflections
3521 independent reflections
1888 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.089
S = 1.01
3521 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.848 (17)	1.948 (17)	2.784 (3)	168.3 (15)
C6—H6···O2^{ii}	0.93	2.56	3.492 (3)	175

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *SCHAKAL* (Keller, 1997); software used to prepare material for publication: *SHELXL97* and *PARST95* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2764).

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supporting information

Acta Cryst. (2009). E**65**, o425–o426 [doi:10.1107/S1600536809003201]

4-Methyl-*N*-(3-oxo-2,3-dihydro-1,2-benzisothiazol-2-yl)benzenesulfonamide

Corrado Rizzoli, Paola Vicini and Matteo Incerti

S1. Comment

Over the past decades a substantial number of 1,2-benzisothiazol-3(2*H*)-one derivatives have been reported to possess a wide range of biological activities including antimicrobial, antiviral, anticancer, anti-inflammatory, cartilage antidegenerative and other pharmacological activities (Clerici *et al.*, 2007; Siegemund *et al.*, 2002). As part of our program aimed at developing novel biologically active 1,2-benzisothiazol-3(2*H*)-ones, we have synthesized in the last years 2-amino-1,2-benzisothiazol-3(2*H*)-one derivatives resulted in the discovery of new compounds active as antiplatelet/spasmolitic agents (Vicini *et al.*, 1997; Vicini *et al.*, 2000) and of compounds endowed with very interesting antimicrobial properties (Vicini *et al.*, 2002; Zani *et al.*, 2004). Recently, in our continuing efforts to find novel effective 2-amino-1,2-benzisothiazol-3(2*H*)-one derivatives, we have synthesized a series of 2-(phenylsulfonyl)amino-1,2-benziso-thiazol-3(2*H*)-ones which exhibit anti-HIV-1 activity against wild type virus and against viral strains carrying clinically relevant mutations (Vicini *et al.*, 2009). Experimental evidences suggest non classical targets for this novel class of anti-HIV-1 agents. In order to study their binding sites at a molecular level we thought it appropriate to obtain X-ray crystallographic data for a prototype.

The molecule of the title compound (Fig. 1) shows no unusual geometric features, with the S1—N1 (1.7116 (19) Å) and S1—C1 (1.721 (2) Å) bond distances corresponding to those observed in similar structures (Cavalca *et al.*, 1970; Ranganathan *et al.*, 2002; Steinfeld & Kersting, 2006; Kim *et al.*, 1996; Xu *et al.*, 2006; Sarma & Mugesh, 2007). The N1—N2 bond distance (1.364 (2) Å) is just significantly shorter than that observed in 4,5-dimethyl-2-(3-nitrobenzene-sulfonylamino)isothiazol-3(2*H*)-one 1,1-dioxide (1.387 (4) Å; Kolberg *et al.*, 1999). The benzoisothiazole rings system is essentially planar (maximum deviation 0.019 (4) Å for atom C4) and forms a dihedral angle of 67.37 (6)° with the plane of the C8–C13 benzene ring. In the crystal structure (Fig. 2), molecules are linked into chains running parallel to the *b* axis by intermolecular N—H···O and C—H···O hydrogen bonding interactions (Table 1).

S2. Experimental

The title compound was synthesized as described elsewhere (Vicini *et al.*, 2009). Freshly prepared chlorocarbonyl-sulfenylchloride (18 mmol) in dried CCl_4 (40 ml) was added dropwise to a stirred, ice-cooled solution of 2-tosylhydrazine (20 mmol) in pyridine (18 ml). After 2 h the reaction mixture was allowed to cool to room temperature and the crude product was filtered, purified by base-acid (Na_2CO_3—HCl) exchange and silica-gel column chromatography (eluent CH_2Cl_2—EtOH 95:5 *v*/*v*). Pale yellow single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature.

S3. Refinement

The H atoms bound to the N2 atom was located in a difference Fourier map and refined isotropically with the N—H distance constrained to 0.87 (1) Å. All other H atoms were placed at calculated positions and refined using a riding

model, with C—H = 0.93–0.96 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms.

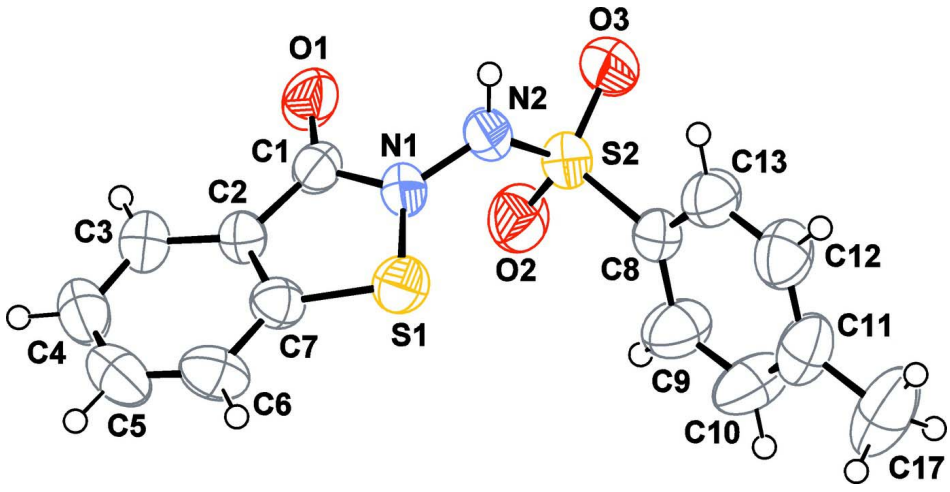

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

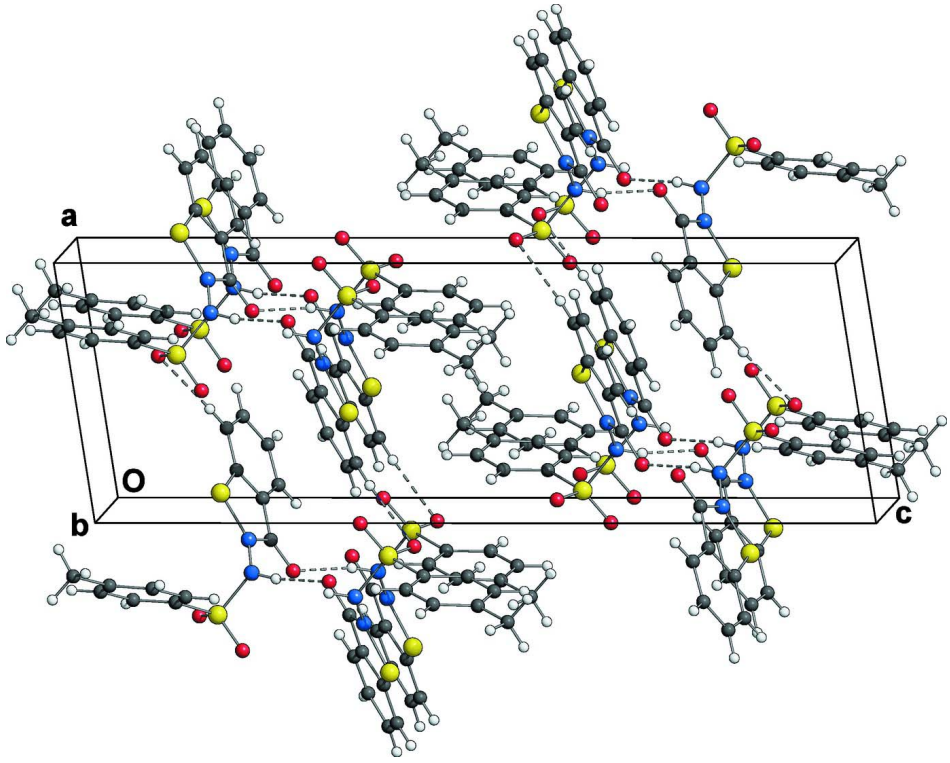

Figure 2

Crystal packing of the title compound viewed approximately along the *b* axis. Intemolecular N—H···O and C–H···O hydrogen bonds are shown as dashed lines.

4-Methyl-*N*-(3-oxo-2,3-dihydro-1,2-benzisothiazol-2-yl)benzenesulfonamide

Crystal data

$C_{14}H_{12}N_2O_3S_2$
$M_r = 320.38$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.051$ (3) Å
$b = 7.655$ (3) Å
$c = 23.91$ (1) Å
$\beta = 98.490$ (8)°
$V = 1457.4$ (10) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.460$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1208 reflections
$\theta = 3.1$–$54.7°$
$\mu = 0.38$ mm^{-1}
$T = 296$ K
Prism, pale yellow
0.28 × 0.26 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.892$, $T_{max} = 0.957$
17685 measured reflections
3521 independent reflections
1888 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 28.0°$, $\theta_{min} = 1.7°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.089$
$S = 1.01$
3521 reflections
194 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.07092 (8)	0.54275 (7)	0.15129 (2)	0.0674 (2)
S2	−0.38794 (7)	0.64261 (7)	0.12617 (2)	0.06196 (18)
O1	−0.25352 (19)	0.2912 (2)	0.21942 (6)	0.0740 (4)
O2	−0.4086 (2)	0.48129 (18)	0.09686 (6)	0.0795 (5)
O3	−0.5189 (2)	0.7130 (2)	0.15313 (7)	0.0826 (5)
N1	−0.1134 (2)	0.4976 (2)	0.17603 (7)	0.0585 (5)
N2	−0.2283 (3)	0.6277 (2)	0.17766 (8)	0.0692 (5)

H2	−0.247 (3)	0.670 (3)	0.2089 (6)	0.085 (8)*
C1	−0.1300 (3)	0.3352 (2)	0.19864 (8)	0.0524 (5)
C2	0.0211 (2)	0.2380 (2)	0.19334 (8)	0.0469 (5)
C3	0.0556 (3)	0.0671 (3)	0.21027 (8)	0.0584 (5)
H3	−0.0221	0.0028	0.2269	0.070*
C4	0.2040 (3)	−0.0051 (3)	0.20233 (10)	0.0702 (6)
H4	0.2268	−0.1211	0.2122	0.084*
C5	0.3219 (3)	0.0936 (3)	0.17950 (10)	0.0796 (7)
H5	0.4249	0.0432	0.1758	0.096*
C6	0.2925 (3)	0.2611 (3)	0.16232 (10)	0.0722 (6)
H6	0.3724	0.3250	0.1467	0.087*
C7	0.1378 (3)	0.3337 (2)	0.16900 (8)	0.0527 (5)
C8	−0.3251 (2)	0.7991 (3)	0.08016 (8)	0.0544 (5)
C9	−0.3198 (3)	0.7587 (3)	0.02474 (10)	0.0815 (7)
H9	−0.3454	0.6466	0.0111	0.098*
C10	−0.2757 (4)	0.8877 (4)	−0.01057 (10)	0.0977 (9)
H10	−0.2729	0.8606	−0.0483	0.117*
C11	−0.2362 (3)	1.0529 (4)	0.00758 (12)	0.0789 (7)
C12	−0.2448 (3)	1.0894 (3)	0.06352 (11)	0.0793 (7)
H12	−0.2203	1.2017	0.0772	0.095*
C13	−0.2886 (3)	0.9645 (3)	0.09934 (10)	0.0708 (6)
H13	−0.2935	0.9922	0.1369	0.085*
C14	−0.1871 (4)	1.1911 (4)	−0.03204 (12)	0.1226 (12)
H14A	−0.1661	1.2993	−0.0120	0.184*
H14B	−0.2766	1.2068	−0.0629	0.184*
H14C	−0.0874	1.1549	−0.0464	0.184*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0837 (4)	0.0494 (3)	0.0742 (4)	−0.0037 (3)	0.0287 (3)	0.0081 (3)
S2	0.0711 (4)	0.0537 (3)	0.0623 (4)	0.0114 (3)	0.0140 (3)	0.0072 (3)
O1	0.0561 (9)	0.0904 (11)	0.0790 (10)	0.0106 (8)	0.0222 (8)	0.0324 (9)
O2	0.1055 (13)	0.0531 (9)	0.0784 (11)	−0.0047 (8)	0.0092 (9)	−0.0016 (8)
O3	0.0789 (11)	0.0863 (11)	0.0896 (12)	0.0260 (9)	0.0360 (9)	0.0181 (9)
N1	0.0668 (12)	0.0489 (10)	0.0626 (11)	0.0159 (9)	0.0193 (9)	0.0127 (8)
N2	0.0929 (14)	0.0660 (12)	0.0483 (11)	0.0358 (11)	0.0092 (10)	0.0007 (10)
C1	0.0538 (13)	0.0556 (13)	0.0484 (12)	0.0027 (10)	0.0099 (10)	0.0099 (10)
C2	0.0457 (12)	0.0437 (11)	0.0508 (11)	0.0001 (9)	0.0055 (9)	0.0021 (9)
C3	0.0598 (14)	0.0514 (13)	0.0610 (13)	0.0000 (10)	−0.0004 (11)	0.0053 (10)
C4	0.0759 (17)	0.0548 (13)	0.0760 (16)	0.0139 (13)	−0.0013 (13)	−0.0038 (12)
C5	0.0670 (16)	0.0808 (18)	0.0911 (18)	0.0205 (14)	0.0121 (14)	−0.0180 (14)
C6	0.0622 (15)	0.0765 (16)	0.0834 (16)	−0.0070 (13)	0.0287 (13)	−0.0146 (14)
C7	0.0552 (13)	0.0498 (12)	0.0543 (12)	−0.0030 (10)	0.0122 (10)	−0.0045 (9)
C8	0.0579 (13)	0.0556 (12)	0.0491 (12)	0.0104 (10)	0.0056 (10)	0.0034 (10)
C9	0.111 (2)	0.0751 (16)	0.0603 (16)	−0.0120 (15)	0.0195 (14)	−0.0075 (13)
C10	0.125 (2)	0.119 (2)	0.0504 (15)	−0.0115 (19)	0.0165 (15)	0.0051 (16)
C11	0.0669 (16)	0.0886 (19)	0.0774 (19)	−0.0017 (14)	−0.0021 (13)	0.0278 (16)

C12	0.0940 (19)	0.0613 (15)	0.0808 (19)	−0.0026 (13)	0.0065 (14)	0.0065 (14)
C13	0.0920 (18)	0.0637 (15)	0.0571 (14)	0.0010 (13)	0.0125 (13)	−0.0009 (12)
C14	0.108 (2)	0.143 (3)	0.114 (2)	−0.014 (2)	0.0065 (18)	0.070 (2)

Geometric parameters (Å, º)

S1—N1	1.7116 (19)	C5—H5	0.9300
S1—C7	1.721 (2)	C6—C7	1.394 (3)
S2—O2	1.4175 (16)	C6—H6	0.9300
S2—O3	1.4205 (15)	C8—C13	1.364 (3)
S2—N2	1.647 (2)	C8—C9	1.368 (3)
S2—C8	1.751 (2)	C9—C10	1.380 (3)
O1—C1	1.223 (2)	C9—H9	0.9300
N1—N2	1.364 (2)	C10—C11	1.359 (3)
N1—C1	1.370 (2)	C10—H10	0.9300
N2—H2	0.844 (9)	C11—C12	1.378 (3)
C1—C2	1.448 (3)	C11—C14	1.511 (3)
C2—C3	1.385 (3)	C12—C13	1.364 (3)
C2—C7	1.386 (2)	C12—H12	0.9300
C3—C4	1.355 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—H14A	0.9600
C4—C5	1.387 (3)	C14—H14B	0.9600
C4—H4	0.9300	C14—H14C	0.9600
C5—C6	1.356 (3)		

N1—S1—C7	89.03 (9)	C5—C6—H6	121.3
O2—S2—O3	120.93 (11)	C7—C6—H6	121.3
O2—S2—N2	109.30 (10)	C2—C7—C6	120.65 (19)
O3—S2—N2	103.69 (10)	C2—C7—S1	112.83 (15)
O2—S2—C8	107.99 (10)	C6—C7—S1	126.50 (17)
O3—S2—C8	109.17 (10)	C13—C8—C9	120.0 (2)
N2—S2—C8	104.56 (10)	C13—C8—S2	119.47 (17)
N2—N1—C1	123.02 (17)	C9—C8—S2	120.50 (18)
N2—N1—S1	119.29 (14)	C8—C9—C10	118.6 (2)
C1—N1—S1	117.36 (13)	C8—C9—H9	120.7
N1—N2—S2	119.16 (15)	C10—C9—H9	120.7
N1—N2—H2	120.6 (16)	C11—C10—C9	122.7 (2)
S2—N2—H2	114.6 (16)	C11—C10—H10	118.7
O1—C1—N1	122.89 (18)	C9—C10—H10	118.7
O1—C1—C2	129.65 (18)	C10—C11—C12	117.1 (2)
N1—C1—C2	107.45 (17)	C10—C11—C14	121.4 (3)
C3—C2—C7	120.20 (18)	C12—C11—C14	121.5 (3)
C3—C2—C1	126.50 (18)	C13—C12—C11	121.5 (2)
C7—C2—C1	113.30 (17)	C13—C12—H12	119.3
C4—C3—C2	119.2 (2)	C11—C12—H12	119.3
C4—C3—H3	120.4	C12—C13—C8	120.2 (2)
C2—C3—H3	120.4	C12—C13—H13	119.9
C3—C4—C5	120.1 (2)	C8—C13—H13	119.9

C3—C4—H4	119.9	C11—C14—H14A	109.5
C5—C4—H4	119.9	C11—C14—H14B	109.5
C6—C5—C4	122.3 (2)	H14A—C14—H14B	109.5
C6—C5—H5	118.8	C11—C14—H14C	109.5
C4—C5—H5	118.8	H14A—C14—H14C	109.5
C5—C6—C7	117.5 (2)	H14B—C14—H14C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.85 (2)	1.95 (2)	2.784 (3)	168 (2)
C6—H6···O2^{ii}	0.93	2.56	3.492 (3)	175

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