Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-(1,3-Benzoxazol-2-ylsulfanyl)-1-phenylethanone

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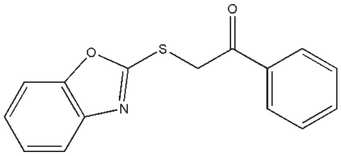
Received 10 August 2009; accepted 25 August 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.093; data-to-parameter ratio = 21.3.

In the title compound, $C_{15}H_{11}NO_2S$, a new thio-benzoxazole derivative, the dihedral angle between the benzoxazole ring and the phenyl ring is 9.91 (9)°. An interesting feature of the crystal structure is the short C···S [3.4858 (17) Å] contact, which is shorter than the sum of the van der Waals radii of these atoms. In the crystal structure, molecules are linked together by zigzag intermolecular C—H···N interactions into a column along the a axis. The crystal structure is further stabilized by intermolecular π–π interactions [centroid–centroid = 3.8048 (10) Å].

Related literature

For applications of 2-(benzo[*d*]oxazol-2-ylthio)-1-phenylethanone and β-keto-sulfones in organic synthesis, see: Marco *et al.* (1995); Fuju *et al.* (1988); Ni *et al.* (2006). For uses of haloalkyl sulfones, see: Grossert *et al.* (1984); Oishi *et al.* (1988); Antane *et al.* (2004). For their biological activity, see: Padmavathi *et al.* (2008). For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{15}H_{11}NO_2S$
M_r = 269.31
Orthorhombic, $P2_12_12_1$
a = 4.8580 (2) Å
b = 14.0780 (5) Å
c = 18.6840 (7) Å
V = 1277.82 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 296 K
0.50 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min}$ = 0.886, $T_{\max}$ = 0.976
13902 measured reflections
3659 independent reflections
3175 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 1.04
3659 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1514 Friedel pairs
Flack parameter: 0.07 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···N1$^{\rm i}$	0.93	2.56	3.395 (2)	149

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2004); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

We thank the University of Isfahan and the University of Malaya for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2859).

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supplementary materials

Acta Cryst. (2009). E**65**, o2287 [**doi:10.1107/S1600536809033960**]

2-(1,3-Benzoxazol-2-ylsulfanyl)-1-phenylethanone

H. Loghmani-Khouzani, D. Hajiheidari, W. T. Robinson, N. Abdul Rahman and **R. Kia**

Comment

2-(Benzo[d]oxazol-2-ylthio)-1-phenylethanone is of great importance in organic synthesis and β-Keto-sulfones are a very important group of intermediates as they are precursors for Michael and Knoevenagel reactions and are used in the preparation of acetylenes, allenes, chalcones, vinyl sulfones, polyfunctionalized 4H-pyrans and ketones (Marco *et al.*, 1995; Fuju *et al.*, 1988; Ni *et al.*, 2006). In addition, β-keto-sulfones can be converted into optically active β-hydroxy-sulfones, halomethyl sulfones and dihalomethyl sulfones. Halomethyl sulfones and dihalomethyl sulfones are very good α-carbanion stabilizing substituents and precursors for the preparation of alkenes, aziridines, epoxides, and β-hydroxy-sulfones. Haloalkyl sulfones are useful in preventing aquatic organisms from attaching to fishing nets and ship hulls (Grossert *et al.*, 1984; Oishi *et al.*, 1988; Antane *et al.*, 2004). They also possess other biological properties such as herbicidal, bactericidal antifungal and insecticidal. Recently sulfone-linked heterocycles were prepared and have been showed antimicrobial activity (Padmavathi *et al.*, 2008). We prepared this compound as a precursor for synthesis of gem-difluoromethylene- containing heterocycle.

In the molecule of the title compound, (Fig. 1), a new thio-benzoxazole derivative, the dihedral angle between the benzoxazole ring and the phenyl ring is 9.91 (9)°. The interesting feature of the crystal structure is the short C6···S1^{i} [3.4858 (17) Å; (i) -1 + x, y, z] contact which is shorter than the sum of the van der Waals radii of these atoms. In the crystal structure, the molecules are linked together by a *zig-zag* intermolecular C—H···N interactions (Table 1) which packed into a column along the *a* axis (Fig. 2). The crystal structure is further stabilized by the intramolecular π–π interactions [$Cg1\cdots Cg2^{i}$ = 3.8048 (10) Å].

Experimental

Sodium carbonate (4.5 mmol) was added to a stirred solution of 2-mercaptobenzoxazole (3 mmol) in ethanol (15 mL) and water (15 mL) and stirred in room temperature for 30 min. α-Bromoacetophenone (3 mmol) was added to the reaction mixture and stirring was continued for 1h. The reaction was monitored by TLC and after 60 min. showed the complete disappearance of starting material. The reaction mixture was poured into 100 mL of 1M HCl containing 50 g of crushed ice. The product was filtered under vacuum and filtrate washed with 10 mL ice-cold ethanol and 10 mL water. Recrystalization from petrol ether and filtration gave the title compound. m.p.: 397-398 K; ^{1}H NMR (400 MHz; CDCl$_3$): δ 7.86-7.21 (m, 9H), 4.58 (s, 2H). ^{13}C NMR (126 MHz; CDCl$_3$): δ 194.1 (C═O), 164.3, 148.9, 140.8, 136.1, 132.6, 128.0, 127.8, 124.1, 122.9, 118.3, 109.6, 37.3. IR (KBr, cm^{-1}): 3027, 2581, 1671 (C═O), 1593, 1492, 1447, 1382, 1326, 1291, 1230, 1182, 1025, 993, 738. Analysis calculated for $C_{15}H_{11}NO_2S$: C 66.89, H 4.12, N 5.20%. Found: C 66.96, H 4.06, N 5.17%.

Refinement

All of the hydrogen atoms were positioned geometrically [C—H = 0.93–0.97 Å] and refined using a riding model approximation with U_{iso} (H) = 1.2 U_{eq} (C). In the presence of sufficient anomalous scattering the absolute structure was determined (1514 Friedel pairs).

Figures

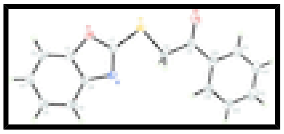

Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering.

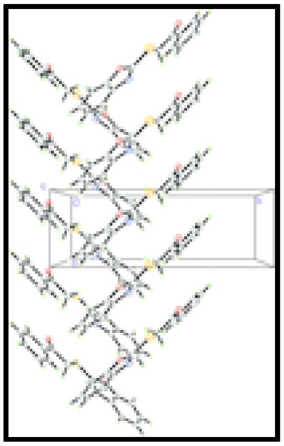

Fig. 2. The crystal packing of the title compound, viewed down the *c*-axis, showing linking of the molecules along the *a*-axis through intermolecular C—H···N interactions. Intermolecular interactions are drawn as dashed lines.

2-(1,3-Benzoxazol-2-ylsulfanyl)-1-phenylethanone

Crystal data

$C_{15}H_{11}NO_2S$

M_r = 269.31

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

a = 4.8580 (2) Å

b = 14.0780 (5) Å

c = 18.6840 (7) Å

V = 1277.82 (8) Å^3

Z = 4

F_{000} = 560

D_x = 1.400 Mg m^{-3}

Melting point: 398 K

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 4173 reflections

θ = 2.6–28.2°

μ = 0.25 mm^{-1}

T = 296 K

Needle, colourless

0.50 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

3659 independent reflections

3175 reflections with $I > 2\sigma(I)$

Monochromator: graphite

T = 296 K

φ and ω scans

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

$T_{\min}$ = 0.886, $T_{\max}$ = 0.976

13902 measured reflections

R_{int} = 0.037

$\theta_{\max}$ = 30.0°

$\theta_{\min}$ = 2.6°

$h = -6 \rightarrow 6$

$k = -19 \rightarrow 19$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.093$

S = 1.04

3659 reflections

172 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.1389P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max}$ = 0.001

$\Delta\rho_{\max}$ = 0.35 e Å^{-3}

$\Delta\rho_{\min}$ = −0.19 e Å^{-3}

Extinction correction: none

Absolute structure: Flack (1983), 1514 Friedel pairs

Flack parameter: 0.07 (7)

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2sigma(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	0.01065 (10)	0.44145 (3)	0.66514 (2)	0.02920 (11)
O1	0.3102 (3)	0.29909 (9)	0.71157 (6)	0.0295 (3)
N1	0.4022 (3)	0.33643 (10)	0.59627 (7)	0.0250 (3)
C1	0.5079 (4)	0.23556 (11)	0.68726 (8)	0.0262 (3)
C2	0.6325 (4)	0.16254 (14)	0.72375 (10)	0.0346 (4)
H2A	0.5895	0.1480	0.7710	0.042*
C3	0.8271 (4)	0.11206 (14)	0.68500 (10)	0.0366 (4)
H3A	0.9193	0.0621	0.7071	0.044*
C4	0.8889 (4)	0.13370 (13)	0.61400 (10)	0.0341 (4)

H4A	1.0209	0.0980	0.5900	0.041*
C5	0.7576 (4)	0.20749 (13)	0.57839 (9)	0.0292 (4)
H5A	0.7982	0.2218	0.5310	0.035*
C6	0.5639 (3)	0.25876 (12)	0.61651 (8)	0.0244 (3)
C7	0.2612 (3)	0.35517 (12)	0.65300 (8)	0.0251 (3)
C8	−0.0098 (4)	0.47588 (12)	0.57236 (8)	0.0289 (3)
H8A	0.1664	0.5008	0.5567	0.035*
H8B	−0.0536	0.4209	0.5432	0.035*
C9	−0.2291 (3)	0.55059 (12)	0.56314 (9)	0.0266 (3)
C10	−0.2762 (4)	0.58747 (12)	0.48943 (9)	0.0264 (3)
C11	−0.4792 (4)	0.65637 (12)	0.47888 (10)	0.0337 (4)
H11A	−0.5823	0.6780	0.5175	0.040*
C12	−0.5275 (5)	0.69250 (13)	0.41129 (11)	0.0396 (5)
H12A	−0.6630	0.7383	0.4045	0.048*
C13	−0.3744 (4)	0.66049 (14)	0.35363 (11)	0.0397 (5)
H13A	−0.4070	0.6850	0.3082	0.048*
C14	−0.1733 (4)	0.59220 (15)	0.36330 (10)	0.0375 (4)
H14A	−0.0713	0.5707	0.3244	0.045*
C15	−0.1237 (4)	0.55572 (14)	0.43100 (10)	0.0312 (4)
H15A	0.0120	0.5099	0.4374	0.037*
O2	−0.3588 (3)	0.57915 (10)	0.61422 (7)	0.0390 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0334 (2)	0.0325 (2)	0.02181 (18)	0.0053 (2)	0.00128 (18)	−0.00418 (15)
O1	0.0362 (7)	0.0345 (6)	0.0179 (5)	0.0024 (5)	0.0020 (5)	0.0012 (5)
N1	0.0279 (7)	0.0290 (7)	0.0180 (6)	0.0018 (6)	−0.0013 (5)	−0.0011 (5)
C1	0.0289 (8)	0.0297 (7)	0.0200 (6)	−0.0035 (8)	0.0007 (7)	0.0003 (5)
C2	0.0439 (11)	0.0348 (9)	0.0253 (8)	−0.0017 (8)	−0.0021 (8)	0.0072 (7)
C3	0.0413 (11)	0.0305 (9)	0.0381 (10)	0.0046 (8)	−0.0074 (8)	0.0052 (8)
C4	0.0343 (9)	0.0313 (9)	0.0367 (10)	0.0049 (8)	0.0024 (8)	−0.0049 (8)
C5	0.0322 (9)	0.0335 (9)	0.0219 (8)	−0.0007 (8)	0.0020 (7)	−0.0034 (7)
C6	0.0283 (9)	0.0263 (8)	0.0185 (7)	−0.0028 (6)	−0.0038 (6)	−0.0009 (6)
C7	0.0276 (8)	0.0279 (8)	0.0198 (7)	−0.0024 (7)	−0.0030 (6)	−0.0012 (6)
C8	0.0290 (8)	0.0332 (8)	0.0243 (7)	0.0037 (8)	0.0008 (8)	0.0017 (6)
C9	0.0253 (8)	0.0227 (7)	0.0316 (8)	−0.0029 (7)	0.0008 (6)	−0.0021 (7)
C10	0.0251 (8)	0.0221 (8)	0.0322 (9)	−0.0040 (6)	−0.0028 (7)	0.0011 (6)
C11	0.0319 (9)	0.0282 (8)	0.0409 (9)	0.0010 (8)	−0.0054 (8)	−0.0018 (7)
C12	0.0377 (11)	0.0300 (9)	0.0512 (11)	0.0011 (9)	−0.0159 (10)	0.0046 (8)
C13	0.0438 (11)	0.0370 (10)	0.0383 (10)	−0.0096 (9)	−0.0134 (8)	0.0083 (8)
C14	0.0359 (10)	0.0449 (11)	0.0317 (9)	−0.0048 (9)	−0.0024 (8)	0.0045 (8)
C15	0.0287 (8)	0.0335 (9)	0.0313 (8)	0.0005 (8)	−0.0023 (7)	0.0035 (8)
O2	0.0440 (8)	0.0376 (7)	0.0355 (7)	0.0095 (6)	0.0076 (6)	−0.0020 (6)

Geometric parameters (Å, °)

S1—C7	1.7343 (17)	C8—C9	1.507 (2)
S1—C8	1.8028 (16)	C8—H8A	0.9700

O1—C7	1.3704 (19)	C8—H8B	0.9700
O1—C1	1.389 (2)	C9—O2	1.212 (2)
N1—C7	1.290 (2)	C9—C10	1.489 (2)
N1—C6	1.398 (2)	C10—C15	1.393 (3)
C1—C2	1.374 (2)	C10—C11	1.397 (3)
C1—C6	1.389 (2)	C11—C12	1.381 (2)
C2—C3	1.387 (3)	C11—H11A	0.9300
C2—H2A	0.9300	C12—C13	1.385 (3)
C3—C4	1.394 (3)	C12—H12A	0.9300
C3—H3A	0.9300	C13—C14	1.383 (3)
C4—C5	1.389 (3)	C13—H13A	0.9300
C4—H4A	0.9300	C14—C15	1.386 (3)
C5—C6	1.383 (2)	C14—H14A	0.9300
C5—H5A	0.9300	C15—H15A	0.9300

C7—S1—C8	95.81 (8)	S1—C8—H8A	109.7
C7—O1—C1	103.29 (12)	C9—C8—H8B	109.7
C7—N1—C6	103.67 (14)	S1—C8—H8B	109.7
C2—C1—O1	128.62 (15)	H8A—C8—H8B	108.2
C2—C1—C6	124.18 (18)	O2—C9—C10	122.17 (16)
O1—C1—C6	107.20 (14)	O2—C9—C8	120.60 (16)
C1—C2—C3	115.13 (17)	C10—C9—C8	117.23 (14)
C1—C2—H2A	122.4	C15—C10—C11	119.18 (16)
C3—C2—H2A	122.4	C15—C10—C9	122.08 (16)
C2—C3—C4	122.13 (18)	C11—C10—C9	118.74 (16)
C2—C3—H3A	118.9	C12—C11—C10	120.30 (18)
C4—C3—H3A	118.9	C12—C11—H11A	119.8
C5—C4—C3	121.36 (18)	C10—C11—H11A	119.9
C5—C4—H4A	119.3	C11—C12—C13	120.01 (18)
C3—C4—H4A	119.3	C11—C12—H12A	120.0
C6—C5—C4	117.13 (16)	C13—C12—H12A	120.0
C6—C5—H5A	121.4	C14—C13—C12	120.29 (18)
C4—C5—H5A	121.4	C14—C13—H13A	119.9
C5—C6—C1	120.06 (16)	C12—C13—H13A	119.9
C5—C6—N1	130.61 (15)	C13—C14—C15	119.98 (19)
C1—C6—N1	109.33 (15)	C13—C14—H14A	120.0
N1—C7—O1	116.50 (15)	C15—C14—H14A	120.0
N1—C7—S1	128.59 (13)	C14—C15—C10	120.25 (17)
O1—C7—S1	114.90 (11)	C14—C15—H15A	119.9
C9—C8—S1	109.67 (12)	C10—C15—H15A	119.9
C9—C8—H8A	109.7		

C7—O1—C1—C2	−179.71 (19)	C1—O1—C7—S1	178.11 (12)
C7—O1—C1—C6	0.52 (17)	C8—S1—C7—N1	8.41 (18)
O1—C1—C2—C3	−178.94 (17)	C8—S1—C7—O1	−170.44 (13)
C6—C1—C2—C3	0.8 (3)	C7—S1—C8—C9	177.42 (12)
C1—C2—C3—C4	−0.5 (3)	S1—C8—C9—O2	0.7 (2)
C2—C3—C4—C5	0.0 (3)	S1—C8—C9—C10	−179.75 (13)
C3—C4—C5—C6	0.3 (3)	O2—C9—C10—C15	178.88 (17)
C4—C5—C6—C1	0.0 (2)	C8—C9—C10—C15	−0.7 (2)

C4—C5—C6—N1	179.14 (16)	O2—C9—C10—C11	−0.8 (3)
C2—C1—C6—C5	−0.5 (3)	C8—C9—C10—C11	179.60 (16)
O1—C1—C6—C5	179.26 (15)	C15—C10—C11—C12	0.0 (3)
C2—C1—C6—N1	−179.86 (17)	C9—C10—C11—C12	179.69 (17)
O1—C1—C6—N1	−0.08 (18)	C10—C11—C12—C13	0.0 (3)
C7—N1—C6—C5	−179.68 (17)	C11—C12—C13—C14	0.2 (3)
C7—N1—C6—C1	−0.43 (18)	C12—C13—C14—C15	−0.2 (3)
C6—N1—C7—O1	0.83 (19)	C13—C14—C15—C10	0.1 (3)
C6—N1—C7—S1	−178.00 (13)	C11—C10—C15—C14	0.0 (3)
C1—O1—C7—N1	−0.89 (19)	C9—C10—C15—C14	−179.73 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···N1^{i}	0.93	2.56	3.395 (2)	149

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1.

Fig. 1

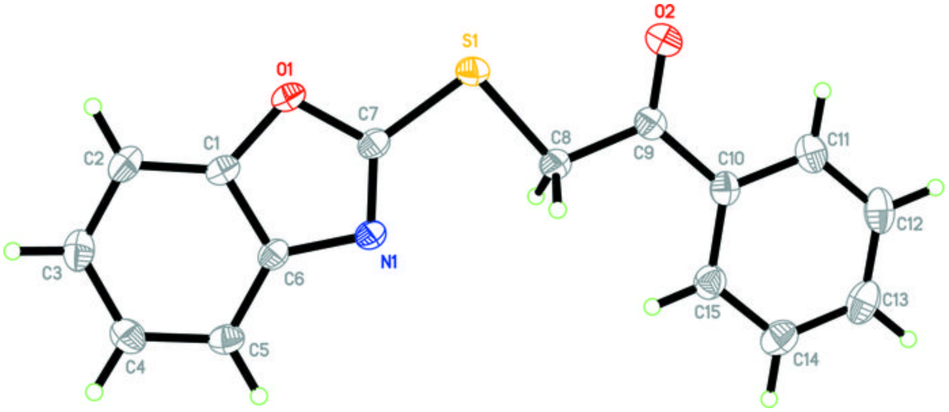

Fig. 2