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4-[(1,3-Thiazol-2-yl)sulfamoyl]phenyl 2,2,2-trifluoroacetate

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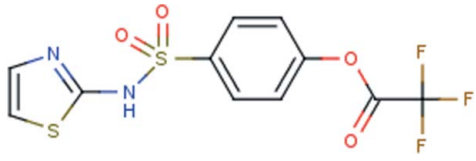
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.071; wR factor = 0.215; data-to-parameter ratio = 15.6.

In the title compound, $C_{11}H_7F_3N_2O_4S_2$, the 1,3-thiazol-2-amine residue is almost perpendicular to the central benzene ring [dihedral angle = 84.3 (2)°]. There is a small twist between the benzene ring and the ester group [C—O—C—C torsion angle = 9.8 (6)°]. Thus, the molecule has an L-shape. Inversion-related dimers are connected in the crystal packing by pairs of N—H···N hydrogen bonds formed between the amine H and thiazole N atom *via* eight-membered $\{\cdots HNCN\}_2$ synthons.

Related literature

For the biological efficacy of F and CF_3 in medicinal chemistry, see: Fokin & Kolomiyets (1988); Bonacorso *et al.* (2006). For background to the biological applications of sulfonamides, see: Croitoru *et al.* (2004); Dogruer *et al.* (2010). For related structures, see: Asiri *et al.* (2011, 2012).



Experimental

Crystal data

$C_{11}H_7F_3N_2O_4S_2$
M_r = 352.31
Monoclinic, $P2_1/n$
a = 8.7498 (5) Å
b = 14.4343 (9) Å
c = 10.7225 (5) Å
β = 96.749 (5)°
V = 1344.84 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 100 K
0.30 × 0.30 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.876, T_{max} = 0.956
12068 measured reflections
3105 independent reflections
2252 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.215
S = 1.06
3105 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1^{i}	0.88	1.99	2.858 (5)	171

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5192).

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4-[(1,3-Thiazol-2-yl)sulfamoyl]phenyl 2,2,2-trifluoroacetate

Abdullah M. Asiri, Hassan M. Faidallah, Khalid A. Alamry, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

The presence of fluoride and trifluoromethyl groups, in particular, has long been recognized in medicinal chemistry as a substituent of distinctive qualities (Fokin & Kolomiyets, 1988; Bonacorso *et al.*, 2006) owing to their ability to alter the physico-chemical and biological characteristics of molecules. In connection with on-going studies of sulphonamides, biological (Croitoru *et al.*, 2004; Dogruer *et al.*, 2010) and crystallographic (Asiri *et al.*, 2011; Asiri *et al.*, 2012), the title CF_3-derivatized sulphonamide (I), was investigated.

In (I), Fig. 1, with reference to the central benzene ring, the 1,3-thiazol-2-amine residue occupies an almost perpendicular position with the N2—S2—C4—C5 torsion angle being 122.7 (3)°. The dihedral angle between the benzene and thiazol rings [r.m.s. deviation = 0.011 Å] is 84.3 (2)°. There is a small twist between the benzene ring and the ester group with the C10—O3—C7—C6 torsion angle being 9.8 (6)°. To a first approximation, the molecule of (I) has the shape of the letter *L*.

In the crystal packing, N—H···N hydrogen bonds are formed between the amine-H and thiazol-N atoms of centrosymmetrically related molecules to form eight-membered $\{\cdots HNCN\}_2$ synthons, Fig. 2 and Table 1. Molecules pack with no specific intermolecular interactions between them.

S2. Experimental

A mixture of sulfamerazine (2.6 g, 10 mmol) in THF (30 ml) and trifluroacetic anhydride (2.2 g, 11 mmol) was refluxed for 2 h. The solid which separated on cooling was recrystallized from ethanol. Yield: 68%. *M*.pt: 513–514 K.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [N—H = 0.88 Å and C—H = 0.95 Å; U_{iso}(H) = 1.2U_{eq}(N,*C*)] and were included in the refinement in the riding model approximation. Owing to poor agreement, the (0 2 1) reflection was omitted from the final cycles of refinement.

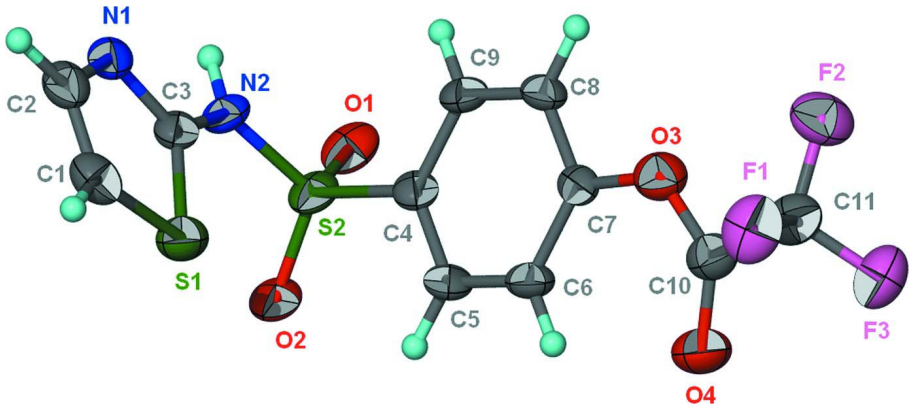

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

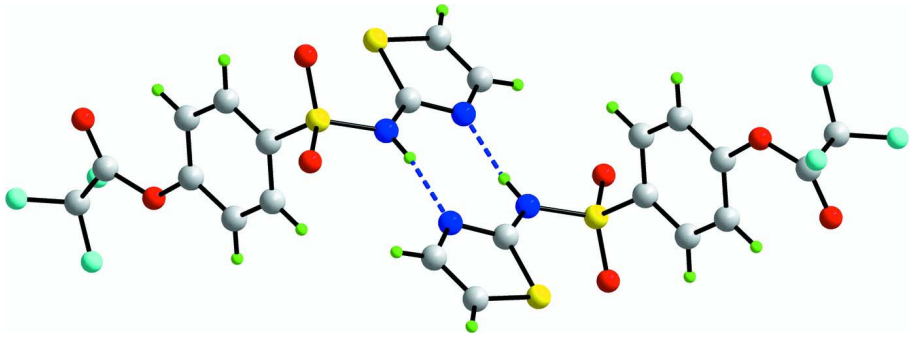

Figure 2

Centrosymmetric dimers in (I) sustained by N—H···N hydrogen bonds shown as blue dashed lines leading to eight-membered $\{\cdots HNCN\}_2$ synthons.

4-[(1,3-Thiazol-2-yl)sulfamoyl]phenyl 2,2,2-trifluoroacetate

Crystal data

$C_{11}H_7F_3N_2O_4S_2$
$M_r = 352.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.7498\ (5)$ Å
$b = 14.4343\ (9)$ Å
$c = 10.7225\ (5)$ Å
$\beta = 96.749\ (5)°$
$V = 1344.84\ (13)$ Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.740$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3265 reflections
$\theta = 2.3–27.5°$
$\mu = 0.45$ mm^{-1}
$T = 100$ K
Irregular, light-yellow
0.30 × 0.30 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.876$, $T_{max} = 0.956$
12068 measured reflections
3105 independent reflections

2252 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.4°$

$h = -11 \rightarrow 11$
$k = -13 \rightarrow 18$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.215$
$S = 1.06$
3105 reflections
199 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
$w = 1/[\sigma^2(F_o^2) + (0.1054P)^2 + 2.1576P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.83675 (13)	0.46698 (8)	0.30886 (10)	0.0500 (3)
S2	0.55748 (13)	0.61673 (8)	0.24923 (9)	0.0454 (3)
F1	1.4449 (3)	0.9101 (2)	0.4037 (3)	0.0707 (8)
F2	1.2781 (3)	1.0011 (2)	0.4699 (3)	0.0679 (8)
F3	1.3609 (4)	1.0282 (2)	0.2933 (3)	0.0684 (8)
O1	0.4182 (4)	0.6682 (2)	0.2516 (3)	0.0549 (8)
O2	0.5788 (4)	0.5696 (2)	0.1331 (2)	0.0547 (8)
O3	1.0875 (4)	0.8720 (2)	0.3708 (3)	0.0540 (8)
O4	1.2090 (4)	0.8779 (3)	0.1895 (3)	0.0621 (9)
N1	0.6932 (4)	0.4364 (2)	0.4965 (3)	0.0429 (8)
N2	0.5655 (4)	0.5461 (2)	0.3653 (3)	0.0407 (8)
H2	0.4899	0.5458	0.4127	0.049*
C1	0.9139 (5)	0.3890 (3)	0.4238 (4)	0.0525 (11)
H1	1.0078	0.3563	0.4215	0.063*
C2	0.8241 (5)	0.3813 (3)	0.5146 (4)	0.0487 (10)
H2A	0.8471	0.3419	0.5853	0.058*
C3	0.6816 (5)	0.4886 (3)	0.3920 (3)	0.0393 (9)
C4	0.7160 (5)	0.6916 (3)	0.2825 (3)	0.0390 (9)
C5	0.8250 (5)	0.7001 (3)	0.1993 (3)	0.0444 (10)
H5	0.8150	0.6645	0.1242	0.053*
C6	0.9482 (5)	0.7601 (3)	0.2248 (3)	0.0422 (9)
H6	1.0222	0.7663	0.1672	0.051*
C7	0.9628 (4)	0.8114 (3)	0.3362 (3)	0.0356 (8)
C8	0.8530 (5)	0.8018 (3)	0.4203 (3)	0.0408 (9)
H8	0.8636	0.8362	0.4963	0.049*
C9	0.7304 (5)	0.7431 (3)	0.3940 (3)	0.0414 (9)
H9	0.6556	0.7374	0.4510	0.050*
C10	1.1941 (5)	0.8985 (3)	0.2972 (4)	0.0484 (10)
C11	1.3199 (6)	0.9613 (4)	0.3672 (5)	0.0544 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0531 (7)	0.0573 (7)	0.0405 (6)	−0.0071 (5)	0.0083 (5)	−0.0031 (5)
S2	0.0539 (6)	0.0557 (6)	0.0244 (5)	−0.0088 (5)	−0.0045 (4)	0.0085 (4)
F1	0.0512 (16)	0.0725 (19)	0.086 (2)	0.0087 (14)	−0.0015 (14)	0.0160 (17)
F2	0.0624 (17)	0.081 (2)	0.0607 (17)	−0.0100 (15)	0.0071 (13)	−0.0193 (15)
F3	0.0685 (19)	0.0635 (18)	0.0730 (19)	−0.0053 (14)	0.0073 (15)	0.0189 (15)
O1	0.0508 (17)	0.073 (2)	0.0385 (15)	−0.0029 (15)	−0.0064 (13)	0.0167 (15)
O2	0.076 (2)	0.0631 (19)	0.0219 (13)	−0.0207 (17)	−0.0062 (13)	0.0018 (13)
O3	0.062 (2)	0.0564 (19)	0.0436 (16)	0.0032 (15)	0.0051 (14)	0.0015 (14)
O4	0.071 (2)	0.073 (2)	0.0442 (18)	−0.0039 (18)	0.0159 (15)	−0.0029 (16)
N1	0.059 (2)	0.0393 (18)	0.0288 (15)	0.0003 (16)	−0.0016 (14)	−0.0013 (13)
N2	0.0466 (18)	0.051 (2)	0.0247 (14)	−0.0051 (15)	0.0032 (13)	0.0077 (13)
C1	0.054 (3)	0.049 (2)	0.053 (3)	0.003 (2)	−0.002 (2)	−0.013 (2)
C2	0.063 (3)	0.042 (2)	0.038 (2)	0.003 (2)	−0.0043 (19)	−0.0034 (17)
C3	0.051 (2)	0.041 (2)	0.0248 (17)	−0.0051 (18)	−0.0011 (15)	−0.0027 (15)
C4	0.050 (2)	0.042 (2)	0.0229 (16)	−0.0013 (17)	−0.0038 (15)	0.0024 (15)
C5	0.063 (3)	0.048 (2)	0.0211 (16)	−0.004 (2)	0.0020 (16)	−0.0050 (15)
C6	0.054 (2)	0.047 (2)	0.0266 (17)	0.0037 (19)	0.0092 (16)	−0.0010 (16)
C7	0.046 (2)	0.0314 (18)	0.0282 (17)	0.0078 (16)	−0.0004 (15)	0.0033 (14)
C8	0.057 (2)	0.041 (2)	0.0239 (16)	0.0028 (18)	0.0035 (16)	−0.0042 (15)
C9	0.053 (2)	0.049 (2)	0.0231 (16)	−0.0024 (18)	0.0067 (15)	0.0023 (16)
C10	0.051 (2)	0.050 (2)	0.044 (2)	0.0062 (19)	0.0088 (19)	0.0039 (19)
C11	0.050 (3)	0.062 (3)	0.051 (3)	0.007 (2)	0.006 (2)	0.007 (2)

Geometric parameters (Å, º)

S1—C3	1.738 (4)	N2—H2	0.8800
S1—C1	1.745 (5)	C1—C2	1.326 (6)
S2—O1	1.430 (4)	C1—H1	0.9500
S2—O2	1.450 (3)	C2—H2A	0.9500
S2—N2	1.604 (3)	C4—C5	1.386 (6)
S2—C4	1.762 (4)	C4—C9	1.400 (5)
F1—C11	1.339 (5)	C5—C6	1.385 (6)
F2—C11	1.331 (5)	C5—H5	0.9500
F3—C11	1.325 (5)	C6—C7	1.398 (5)
O3—C10	1.346 (5)	C6—H6	0.9500
O3—C7	1.414 (5)	C7—C8	1.399 (5)
O4—C10	1.214 (5)	C8—C9	1.370 (6)
N1—C3	1.344 (5)	C8—H8	0.9500
N1—C2	1.389 (6)	C9—H9	0.9500
N2—C3	1.317 (5)	C10—C11	1.550 (7)
C3—S1—C1	90.8 (2)	C6—C5—C4	120.4 (3)
O1—S2—O2	117.08 (19)	C6—C5—H5	119.8
O1—S2—N2	106.00 (19)	C4—C5—H5	119.8
O2—S2—N2	111.73 (19)	C5—C6—C7	119.4 (4)

O1—S2—C4	109.3 (2)	C5—C6—H6	120.3
O2—S2—C4	106.50 (19)	C7—C6—H6	120.3
N2—S2—C4	105.72 (17)	C6—C7—C8	119.8 (4)
C10—O3—C7	126.0 (3)	C6—C7—O3	122.8 (3)
C3—N1—C2	114.7 (4)	C8—C7—O3	117.3 (3)
C3—N2—S2	122.1 (3)	C9—C8—C7	120.6 (3)
C3—N2—H2	119.0	C9—C8—H8	119.7
S2—N2—H2	119.0	C7—C8—H8	119.7
C2—C1—S1	111.0 (4)	C8—C9—C4	119.5 (4)
C2—C1—H1	124.5	C8—C9—H9	120.2
S1—C1—H1	124.5	C4—C9—H9	120.2
C1—C2—N1	113.6 (4)	O4—C10—O3	130.4 (5)
C1—C2—H2A	123.2	O4—C10—C11	117.0 (4)
N1—C2—H2A	123.2	O3—C10—C11	112.6 (4)
N2—C3—N1	121.0 (4)	F3—C11—F2	107.4 (4)
N2—C3—S1	129.1 (3)	F3—C11—F1	107.9 (4)
N1—C3—S1	109.9 (3)	F2—C11—F1	106.9 (4)
C5—C4—C9	120.2 (4)	F3—C11—C10	111.3 (4)
C5—C4—S2	120.6 (3)	F2—C11—C10	113.9 (4)
C9—C4—S2	119.2 (3)	F1—C11—C10	109.2 (4)

O1—S2—N2—C3	−178.9 (3)	S2—C4—C5—C6	179.0 (3)
O2—S2—N2—C3	52.5 (4)	C4—C5—C6—C7	0.7 (6)
C4—S2—N2—C3	−63.0 (4)	C5—C6—C7—C8	−0.1 (6)
C3—S1—C1—C2	0.9 (4)	C5—C6—C7—O3	178.1 (3)
S1—C1—C2—N1	−0.1 (5)	C10—O3—C7—C6	9.8 (6)
C3—N1—C2—C1	−1.1 (5)	C10—O3—C7—C8	−171.9 (4)
S2—N2—C3—N1	173.6 (3)	C6—C7—C8—C9	−0.6 (6)
S2—N2—C3—S1	−6.0 (5)	O3—C7—C8—C9	−178.9 (3)
C2—N1—C3—N2	−178.0 (4)	C7—C8—C9—C4	0.7 (6)
C2—N1—C3—S1	1.7 (4)	C5—C4—C9—C8	−0.1 (6)
C1—S1—C3—N2	178.2 (4)	S2—C4—C9—C8	−179.7 (3)
C1—S1—C3—N1	−1.5 (3)	C7—O3—C10—O4	−0.2 (8)
O1—S2—C4—C5	−123.6 (3)	C7—O3—C10—C11	−176.8 (3)
O2—S2—C4—C5	3.7 (4)	O4—C10—C11—F3	41.5 (6)
N2—S2—C4—C5	122.7 (3)	O3—C10—C11—F3	−141.4 (4)
O1—S2—C4—C9	56.0 (4)	O4—C10—C11—F2	163.1 (4)
O2—S2—C4—C9	−176.7 (3)	O3—C10—C11—F2	−19.8 (5)
N2—S2—C4—C9	−57.7 (4)	O4—C10—C11—F1	−77.6 (5)
C9—C4—C5—C6	−0.6 (6)	O3—C10—C11—F1	99.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.88	1.99	2.858 (5)	171

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