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2-Methylsulfonyl-1,2,4-triazolo[1,5-*a*]-quinazolin-5(4*H*)-one

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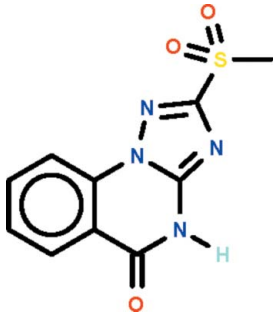
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.082; data-to-parameter ratio = 13.7.

The triazoloquinazoline fused-ring system of the title compound, $C_{10}H_8N_4O_3S$, is essentially planar (r.m.s. deviation = 0.027 Å). In the crystal, adjacent molecules are linked by N—H···$O_{sulfonyl}$ hydrogen bonds, generating a helical chain running along the *b* axis.

Related literature

For the synthesis of the precursor, see: Al-Salahi & Geffken (2011).



Experimental

Crystal data

$C_{10}H_8N_4O_3S$
M_r = 264.26
Monoclinic, $P2_1$
a = 9.6216 (2) Å
b = 4.9206 (1) Å
c = 12.1623 (3) Å
β = 106.628 (2)°
V = 551.73 (2) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 2.71 mm^{-1}
T = 294 K
0.20 × 0.10 × 0.02 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.613, T_{max} = 0.948
9640 measured reflections
2304 independent reflections
2237 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.082
S = 1.04
2304 reflections
168 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983), 1007 Friedel pairs
Flack parameter: 0.00 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.88 (1)	2.23 (1)	3.072 (2)	160 (3)

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

Data collection: *CrysAlis PRO* (Agilent, 2012); 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); 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: BT5915).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, o1806 [doi:10.1107/S1600536812021769]

2-Methylsulfonyl-1,2,4-triazolo[1,5-*a*]quinazolin-5(4*H*)-one

Rashad Al-Salahi, Mohamed Marzouk, Mohammed Abbas and Seik Weng Ng

S1. Comment

2-(Methylsulfanyl)-[1,2,4]triazolo[1,5-*a*]quinazolin-5-one was synthesized from 2-hydrazinobenzoic acid and dimethyl *N*-cyanoimidodithiocarbonate; further reactions on the inherent lactam unit yielded other derivatives (Al-Salahi & Geffken, 2011). In the present study, this compound is oxided by hydrogen peroxide. The triazoloquinazoline fused-ring system of $C_{10}H_8N_4O_3S$ (Scheme I, Fig. 1) is planar. Adjacent molecules are linked by an N–$H\cdots O_{sulfonyl}$ hydrogen bond to generate a helical chain running along the *b*-axis of the monoclinic unit cell (Fig. 2, Table 1).

S2. Experimental

Under ice-cold conditions, 2-hydrazinobenzoic acid (10 mmol, 1.52 g) was added to a solution of dimethyl *N*-cyano-dithioimidocarbonate (10 mmol, 1.46 g) in ethanol (20 ml). Triethylamine (30 mmol, 3.03 g) was added. The reaction mixture was stirred overnight at room temperature. Concentrated hydrochloric acid was added; the acidified mixture for heated for an hour. The mixture was poured into ice water; the solid that formed was collected and recrystallized from ethanol to give colorless crystals of 2-(methylsulfanyl)-[1,2,4]triazolo[1,5-*a*]quinazolin-5-one. The procedure was that reported earlier (Al-Salahi & Geffken, 2011).

To the boiling mixture of 2-methylsulfanyl-[1,2,4]triazolo[1,5-*a*]quinazolin-5-one (1 mmol, 0.23 g) in glacial acetic acid (5 ml) was added hydrogen peroxide. Colorless crystals of the oxidized product were obtained when the solution was allowed to cool.

S3. Refinement

All H-atom were located in a difference Fourier map. Carbon-bound H-atoms were placed in calculated positions [C–H 0.93 to 0.96 Å, U_{iso}(H) 1.2–1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The amino H-atom was refined isotropically with a distance restraint of N–H 0.88±0.01 Å.

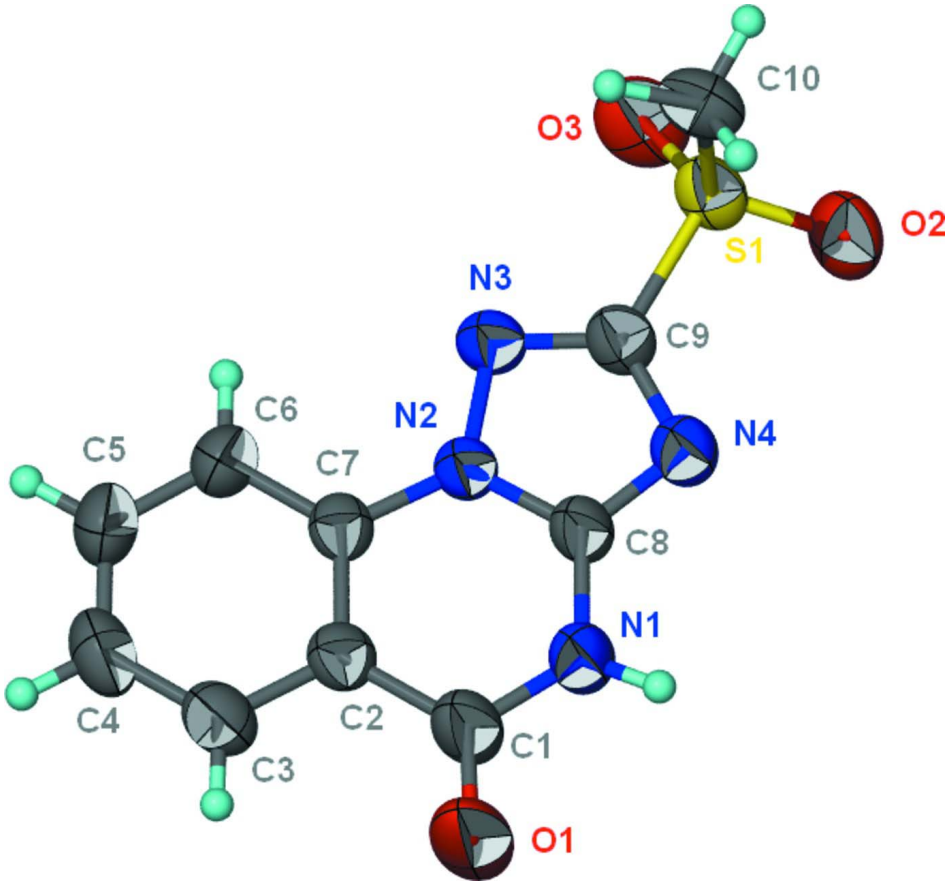

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{10}H_8N_4O_3S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

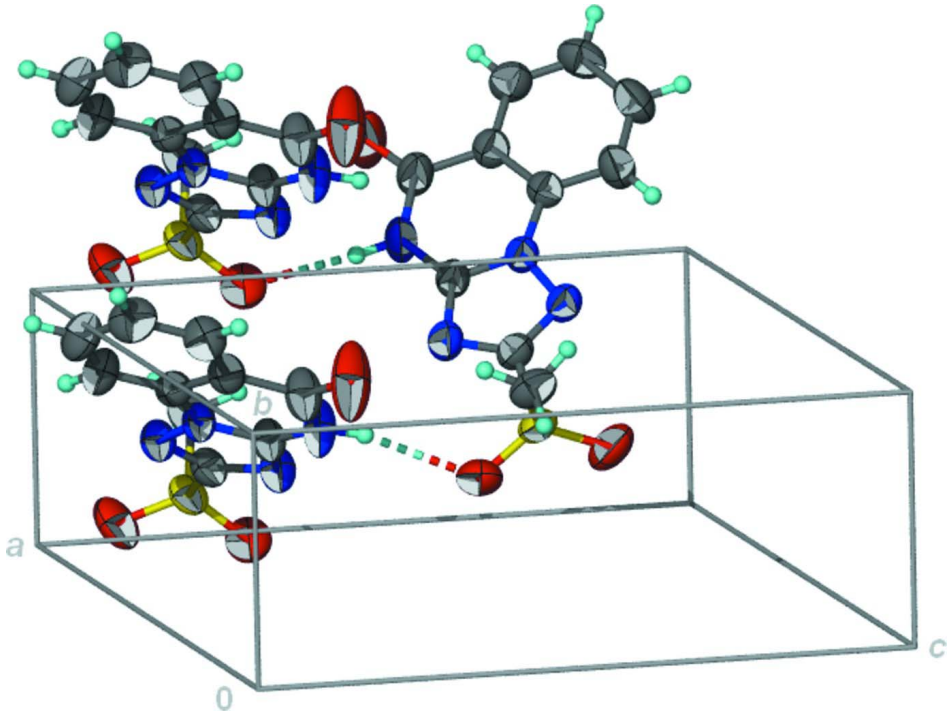

Figure 2

Hydrogen-bonded chain motif.

2-Methylsulfonyl-1,2,4-triazolo[1,5-*a*]quinazolin-5(4*H*)-one

Crystal data

$C_{10}H_8N_4O_3S$
$M_r = 264.26$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 9.6216$ (2) Å
$b = 4.9206$ (1) Å
$c = 12.1623$ (3) Å
$\beta = 106.628$ (2)°
$V = 551.73$ (2) Å^3
$Z = 2$
$F(000) = 272$
$D_x = 1.591$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 6133 reflections
$\theta = 3.8$–$76.5°$
$\mu = 2.71$ mm^{-1}
$T = 294$ K
Plate, colorless
0.20 × 0.10 × 0.02 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.613$, $T_{max} = 0.948$
9640 measured reflections
2304 independent reflections
2237 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 76.7°$, $\theta_{min} = 3.8°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.082$
$S = 1.04$
2304 reflections
168 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
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.056P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Flack (1983), 1007 Friedel pairs
Absolute structure parameter: 0.00 (2)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.24294 (4)	0.00109 (9)	0.18198 (3)	0.04202 (13)
O1	0.8628 (2)	0.7880 (6)	0.13678 (16)	0.0903 (8)
O2	0.24690 (17)	−0.1892 (3)	0.09276 (13)	0.0566 (4)
O3	0.23731 (18)	−0.0963 (4)	0.29138 (14)	0.0645 (4)
N1	0.67906 (18)	0.4984 (5)	0.12477 (13)	0.0542 (4)
H1	0.677 (3)	0.451 (7)	0.0548 (13)	0.086 (9)*
N2	0.56362 (13)	0.4813 (4)	0.27059 (11)	0.0354 (3)
N3	0.45055 (15)	0.3499 (3)	0.29552 (13)	0.0392 (3)
N4	0.47068 (16)	0.2190 (4)	0.12045 (12)	0.0436 (4)
C1	0.7743 (2)	0.6953 (5)	0.18009 (17)	0.0528 (5)
C2	0.75983 (17)	0.7852 (4)	0.29235 (15)	0.0396 (4)
C3	0.85191 (19)	0.9855 (5)	0.35516 (16)	0.0478 (4)
H3	0.9222	1.0634	0.3262	0.057*
C4	0.8387 (2)	1.0679 (4)	0.45993 (18)	0.0536 (5)
H4	0.8999	1.2021	0.5012	0.064*
C5	0.7349 (2)	0.9521 (5)	0.50437 (17)	0.0534 (5)
H5	0.7273	1.0095	0.5753	0.064*
C6	0.6429 (2)	0.7530 (5)	0.44484 (16)	0.0472 (4)
H6	0.5743	0.6733	0.4752	0.057*
C7	0.65534 (17)	0.6748 (3)	0.33865 (14)	0.0356 (3)
C8	0.57227 (18)	0.3991 (4)	0.16704 (14)	0.0389 (4)
C9	0.40156 (18)	0.2013 (4)	0.20289 (14)	0.0382 (4)
C10	0.1035 (2)	0.2349 (4)	0.12929 (18)	0.0500 (5)
H10A	0.0116	0.1442	0.1142	0.075*
H10B	0.1129	0.3136	0.0596	0.075*
H10C	0.1091	0.3755	0.1851	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0482 (2)	0.0365 (2)	0.0405 (2)	−0.01197 (18)	0.01137 (15)	0.00488 (17)
O1	0.0929 (13)	0.131 (2)	0.0609 (10)	−0.0698 (14)	0.0448 (10)	−0.0294 (12)

O2	0.0684 (9)	0.0428 (8)	0.0593 (9)	−0.0175 (7)	0.0197 (7)	−0.0074 (7)
O3	0.0741 (10)	0.0654 (10)	0.0541 (8)	−0.0176 (8)	0.0183 (7)	0.0194 (7)
N1	0.0573 (9)	0.0762 (11)	0.0343 (7)	−0.0280 (10)	0.0216 (6)	−0.0137 (9)
N2	0.0342 (6)	0.0410 (7)	0.0312 (6)	−0.0058 (6)	0.0100 (5)	−0.0011 (6)
N3	0.0387 (7)	0.0435 (8)	0.0372 (7)	−0.0075 (6)	0.0138 (6)	−0.0003 (6)
N4	0.0456 (8)	0.0505 (9)	0.0342 (7)	−0.0125 (7)	0.0107 (6)	−0.0036 (6)
C1	0.0521 (10)	0.0692 (14)	0.0385 (9)	−0.0229 (10)	0.0150 (8)	−0.0051 (9)
C2	0.0359 (8)	0.0446 (9)	0.0360 (8)	−0.0031 (7)	0.0064 (6)	0.0006 (7)
C3	0.0431 (8)	0.0498 (10)	0.0471 (9)	−0.0101 (9)	0.0073 (7)	−0.0003 (10)
C4	0.0515 (11)	0.0499 (13)	0.0516 (11)	−0.0093 (8)	0.0022 (9)	−0.0136 (8)
C5	0.0555 (10)	0.0585 (15)	0.0452 (9)	−0.0048 (10)	0.0126 (8)	−0.0181 (9)
C6	0.0476 (9)	0.0554 (12)	0.0412 (9)	−0.0052 (9)	0.0169 (7)	−0.0098 (9)
C7	0.0342 (7)	0.0369 (8)	0.0332 (7)	0.0004 (6)	0.0055 (6)	−0.0014 (6)
C8	0.0389 (8)	0.0471 (9)	0.0296 (8)	−0.0095 (7)	0.0079 (6)	−0.0017 (7)
C9	0.0392 (8)	0.0380 (8)	0.0360 (8)	−0.0059 (7)	0.0084 (6)	0.0027 (7)
C10	0.0407 (9)	0.0498 (11)	0.0585 (11)	−0.0090 (8)	0.0127 (8)	0.0067 (9)

Geometric parameters (Å, º)

S1—O3	1.4296 (16)	C1—C2	1.479 (3)
S1—O2	1.4421 (16)	C2—C7	1.395 (2)
S1—C10	1.744 (2)	C2—C3	1.397 (3)
S1—C9	1.7726 (17)	C3—C4	1.377 (3)
O1—C1	1.211 (2)	C3—H3	0.9300
N1—C8	1.364 (2)	C4—C5	1.387 (3)
N1—C1	1.370 (3)	C4—H4	0.9300
N1—H1	0.877 (10)	C5—C6	1.379 (3)
N2—C8	1.348 (2)	C5—H5	0.9300
N2—N3	1.3718 (19)	C6—C7	1.385 (2)
N2—C7	1.397 (2)	C6—H6	0.9300
N3—C9	1.312 (2)	C10—H10A	0.9600
N4—C8	1.321 (2)	C10—H10B	0.9600
N4—C9	1.355 (2)	C10—H10C	0.9600
O3—S1—O2	119.93 (11)	C3—C4—H4	119.8
O3—S1—C10	109.44 (11)	C5—C4—H4	119.8
O2—S1—C10	109.57 (10)	C6—C5—C4	120.88 (19)
O3—S1—C9	108.24 (9)	C6—C5—H5	119.6
O2—S1—C9	105.18 (9)	C4—C5—H5	119.6
C10—S1—C9	103.09 (9)	C5—C6—C7	118.24 (18)
C8—N1—C1	122.54 (16)	C5—C6—H6	120.9
C8—N1—H1	117 (2)	C7—C6—H6	120.9
C1—N1—H1	120 (2)	C6—C7—C2	122.18 (17)
C8—N2—N3	109.28 (14)	C6—C7—N2	122.22 (16)
C8—N2—C7	124.09 (14)	C2—C7—N2	115.60 (15)
N3—N2—C7	126.61 (13)	N4—C8—N2	111.47 (16)
C9—N3—N2	100.69 (13)	N4—C8—N1	128.56 (17)
C8—N4—C9	100.67 (15)	N2—C8—N1	119.95 (15)

O1—C1—N1	120.47 (19)	N3—C9—N4	117.88 (15)
O1—C1—C2	123.5 (2)	N3—C9—S1	121.05 (13)
N1—C1—C2	116.01 (16)	N4—C9—S1	120.94 (13)
C7—C2—C3	118.12 (18)	S1—C10—H10A	109.5
C7—C2—C1	121.66 (16)	S1—C10—H10B	109.5
C3—C2—C1	120.22 (17)	H10A—C10—H10B	109.5
C4—C3—C2	120.10 (18)	S1—C10—H10C	109.5
C4—C3—H3	119.9	H10A—C10—H10C	109.5
C2—C3—H3	119.9	H10B—C10—H10C	109.5
C3—C4—C5	120.47 (18)		

C8—N2—N3—C9	−0.34 (19)	C8—N2—C7—C2	0.5 (2)
C7—N2—N3—C9	178.37 (16)	N3—N2—C7—C2	−178.05 (17)
C8—N1—C1—O1	−176.2 (3)	C9—N4—C8—N2	0.2 (2)
C8—N1—C1—C2	3.1 (3)	C9—N4—C8—N1	178.8 (2)
O1—C1—C2—C7	179.4 (3)	N3—N2—C8—N4	0.1 (2)
N1—C1—C2—C7	0.1 (3)	C7—N2—C8—N4	−178.68 (16)
O1—C1—C2—C3	−0.6 (4)	N3—N2—C8—N1	−178.63 (19)
N1—C1—C2—C3	−179.9 (2)	C7—N2—C8—N1	2.6 (3)
C7—C2—C3—C4	0.4 (3)	C1—N1—C8—N4	177.0 (2)
C1—C2—C3—C4	−179.7 (2)	C1—N1—C8—N2	−4.5 (3)
C2—C3—C4—C5	0.4 (3)	N2—N3—C9—N4	0.5 (2)
C3—C4—C5—C6	−0.1 (3)	N2—N3—C9—S1	−175.38 (13)
C4—C5—C6—C7	−1.0 (3)	C8—N4—C9—N3	−0.5 (2)
C5—C6—C7—C2	1.8 (3)	C8—N4—C9—S1	175.42 (14)
C5—C6—C7—N2	−177.84 (18)	O3—S1—C9—N3	−34.09 (18)
C3—C2—C7—C6	−1.5 (3)	O2—S1—C9—N3	−163.43 (15)
C1—C2—C7—C6	178.53 (19)	C10—S1—C9—N3	81.78 (17)
C3—C2—C7—N2	178.19 (17)	O3—S1—C9—N4	150.12 (17)
C1—C2—C7—N2	−1.8 (3)	O2—S1—C9—N4	20.79 (18)
C8—N2—C7—C6	−179.84 (18)	C10—S1—C9—N4	−94.00 (18)
N3—N2—C7—C6	1.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.88 (1)	2.23 (1)	3.072 (2)	160 (3)

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