

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

 Rashad Al-Salahi,^a Mohamed Al-Omar,^a Mohammed Abbas^a and Seik Weng Ng^{b,c*}

^aDepartment of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, Riyadh 11451, Saudi Arabia, ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and ^cChemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia
Correspondence e-mail: seikweng@um.edu.my

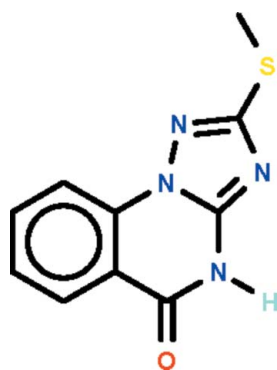
Received 10 May 2012; accepted 14 May 2012

 Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.089; data-to-parameter ratio = 13.6.

The non-H atoms of the title compound, $\text{C}_{10}\text{H}_8\text{N}_4\text{OS}$, lie approximately in a common plane (r.m.s. deviation = 0.058 Å). In the crystal, two molecules are linked across a center of inversion by a pair of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, forming a dimer.

Related literature

For the synthesis, see: Al-Salahi & Geffken (2011). For a related compound, see: Al-Salahi *et al.* (2011).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_8\text{N}_4\text{OS}$
 $M_r = 232.26$

Monoclinic, $P2_1/c$
 $a = 10.4150$ (1) Å
 $b = 5.0631$ (1) Å
 $c = 18.6564$ (3) Å
 $\beta = 96.857$ (1)°
 $V = 976.76$ (3) Å³

$Z = 4$
 Cu $K\alpha$ radiation
 $\mu = 2.81$ mm⁻¹
 $T = 294$ K
 $0.35 \times 0.15 \times 0.10$ mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
 Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
 $T_{\min} = 0.439$, $T_{\max} = 0.766$

15980 measured reflections
 2052 independent reflections
 1996 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.089$
 $S = 1.05$
 2052 reflections
 151 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N4}-\text{H4}\cdots\text{N3}^i$	0.85 (2)	2.05 (2)	2.896 (2)	174 (2)

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

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: *pubCIF* (Westrip, 2010).

We thank the Research Center of the College of Pharmacy College and Deanship of Scientific Research of King Saud University, and the Ministry of Higher Education of Malaysia (grant No. UM.C/HIR/MOHE/SC/12) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5914).

References

- Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
 Al-Salahi, R. & Geffken, D. (2011). *Synth. Commun.* **41**, 3512–3523.
 Al-Salahi, R., Detlef, G. & Ahmed, B. (2011). *Acta Cryst.* **E67**, o1861.
 Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2012). E68, o1805 [doi:10.1107/S1600536812021757]

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

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

Comment

The title compound (Scheme 1) was synthesized from 2-hydrazinobenzoic acid and dimethyl *N*-cyanoimidodithiocarbonate; further reactions on the inherent lactam unit yielded other derivatives (Al-Salahi & Geffken, 2011). The non-H atoms of C₁₀H₈N₄OS lie in a common plane (Fig. 1). Two molecules are linked across a center-of-inversion by N–H···N hydrogen bonds to form a dimer (Table 1). A related compound that has a benzyloxy group instead of the methylsulfanyl group also exists as a hydrogen-bonded dimer (Al-Salahi *et al.*, 2011).

Experimental

Under ice-cold conditions, 2-hydrazinobenzoic acid (10 mmol, 1.52 g) was added to a solution of dimethyl *N*-cyanoimidodithiocarbonate (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 was 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.

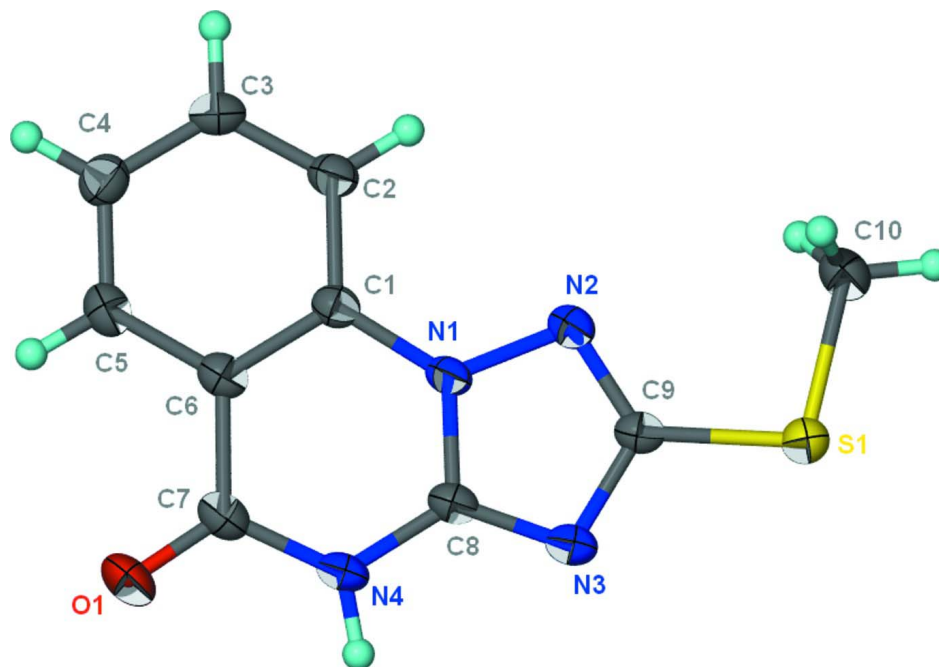
Refinement

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

The amino H-atom was freely refined.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); 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).

**Figure 1**

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{10}H_8N_4OS$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{10}H_8N_4OS$

$M_r = 232.26$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 10.4150(1)\ \text{\AA}$

$b = 5.0631(1)\ \text{\AA}$

$c = 18.6564(3)\ \text{\AA}$

$\beta = 96.857(1)^\circ$

$V = 976.76(3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 480$

$D_x = 1.579\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184\ \text{\AA}$

Cell parameters from 10679 reflections

$\theta = 4.3\text{--}76.7^\circ$

$\mu = 2.81\ \text{mm}^{-1}$

$T = 294\ \text{K}$

Prism, colorless

$0.35 \times 0.15 \times 0.10\ \text{mm}$

Data collection

Agilent SuperNova Dual

diffractometer with an Atlas detector

Radiation source: SuperNova (Cu) X-ray

Source

Mirror monochromator

Detector resolution: $10.4041\ \text{pixels mm}^{-1}$

ω scan

Absorption correction: multi-scan

(*CrysAlis PRO*; Agilent, 2012)

$T_{\min} = 0.439$, $T_{\max} = 0.766$

15980 measured reflections

2052 independent reflections

1996 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 76.9^\circ$, $\theta_{\min} = 4.3^\circ$

$h = -13 \rightarrow 13$

$k = -6 \rightarrow 6$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.089$

$S = 1.05$

2052 reflections

151 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 atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.2828P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0089 (9)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.07695 (3)	0.09449 (7)	0.203762 (17)	0.03356 (15)
O1	0.19589 (12)	1.1102 (2)	-0.05498 (6)	0.0527 (3)
N1	0.25468 (10)	0.6522 (2)	0.12100 (5)	0.0284 (2)
N2	0.24857 (10)	0.4808 (2)	0.17833 (5)	0.0299 (2)
N3	0.07817 (11)	0.4211 (2)	0.09036 (6)	0.0327 (3)
N4	0.13609 (11)	0.7645 (3)	0.01027 (6)	0.0365 (3)
C1	0.34851 (12)	0.8433 (3)	0.11483 (6)	0.0282 (3)
C2	0.45835 (13)	0.8685 (3)	0.16496 (7)	0.0331 (3)
H2	0.4716	0.7559	0.2045	0.040*
C3	0.54687 (13)	1.0631 (3)	0.15483 (8)	0.0357 (3)
H3	0.6209	1.0803	0.1877	0.043*
C4	0.52720 (14)	1.2342 (3)	0.09627 (8)	0.0381 (3)
H4A	0.5867	1.3674	0.0908	0.046*
C5	0.41916 (13)	1.2058 (3)	0.04634 (7)	0.0365 (3)
H5	0.4066	1.3195	0.0070	0.044*
C6	0.32892 (12)	1.0088 (3)	0.05434 (7)	0.0311 (3)
C7	0.21660 (14)	0.9725 (3)	-0.00156 (7)	0.0359 (3)
C8	0.15228 (12)	0.6134 (3)	0.07104 (7)	0.0303 (3)
C9	0.14125 (12)	0.3490 (3)	0.15648 (7)	0.0291 (3)
C10	0.18994 (15)	0.0881 (3)	0.28453 (8)	0.0443 (4)
H10A	0.1713	-0.0598	0.3138	0.066*
H10B	0.1826	0.2487	0.3111	0.066*
H10C	0.2763	0.0721	0.2718	0.066*
H4	0.072 (2)	0.722 (4)	-0.0199 (11)	0.062 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0327 (2)	0.0351 (2)	0.0319 (2)	-0.00273 (12)	-0.00017 (14)	0.00156 (12)
O1	0.0552 (7)	0.0637 (8)	0.0356 (6)	-0.0047 (5)	-0.0093 (5)	0.0204 (5)
N1	0.0297 (5)	0.0316 (5)	0.0225 (5)	0.0011 (4)	-0.0028 (4)	0.0021 (4)
N2	0.0315 (5)	0.0323 (6)	0.0249 (5)	0.0010 (4)	-0.0010 (4)	0.0025 (4)
N3	0.0308 (5)	0.0381 (6)	0.0273 (5)	-0.0004 (4)	-0.0038 (4)	0.0003 (4)
N4	0.0364 (6)	0.0439 (7)	0.0263 (5)	-0.0022 (5)	-0.0087 (4)	0.0045 (5)

C1	0.0298 (6)	0.0300 (6)	0.0246 (6)	0.0029 (5)	0.0018 (5)	-0.0023 (5)
C2	0.0345 (6)	0.0356 (7)	0.0275 (6)	0.0012 (5)	-0.0031 (5)	0.0007 (5)
C3	0.0328 (6)	0.0399 (7)	0.0328 (7)	-0.0007 (5)	-0.0020 (5)	-0.0048 (5)
C4	0.0394 (7)	0.0364 (7)	0.0387 (7)	-0.0050 (6)	0.0051 (6)	-0.0020 (6)
C5	0.0428 (7)	0.0356 (7)	0.0312 (7)	0.0012 (6)	0.0049 (6)	0.0046 (5)
C6	0.0335 (6)	0.0335 (7)	0.0258 (6)	0.0039 (5)	0.0015 (5)	0.0000 (5)
C7	0.0385 (7)	0.0413 (7)	0.0269 (6)	0.0034 (6)	-0.0008 (5)	0.0035 (6)
C8	0.0310 (6)	0.0340 (7)	0.0245 (6)	0.0030 (5)	-0.0029 (5)	-0.0009 (5)
C9	0.0291 (6)	0.0314 (6)	0.0260 (6)	0.0027 (5)	0.0000 (5)	-0.0023 (5)
C10	0.0442 (8)	0.0510 (9)	0.0354 (7)	-0.0051 (6)	-0.0041 (6)	0.0098 (6)

Geometric parameters (Å, °)

S1—C9	1.7402 (14)	C1—C6	1.4008 (18)
S1—C10	1.7980 (15)	C2—C3	1.378 (2)
O1—C7	1.2145 (18)	C2—H2	0.9300
N1—C8	1.3443 (15)	C3—C4	1.390 (2)
N1—N2	1.3848 (15)	C3—H3	0.9300
N1—C1	1.3897 (17)	C4—C5	1.3800 (19)
N2—C9	1.3237 (17)	C4—H4A	0.9300
N3—C8	1.3192 (18)	C5—C6	1.391 (2)
N3—C9	1.3759 (16)	C5—H5	0.9300
N4—C8	1.3614 (17)	C6—C7	1.4828 (18)
N4—C7	1.3800 (19)	C10—H10A	0.9600
N4—H4	0.85 (2)	C10—H10B	0.9600
C1—C2	1.3942 (17)	C10—H10C	0.9600
C9—S1—C10	100.67 (7)	C4—C5—C6	120.60 (13)
C8—N1—N2	109.76 (10)	C4—C5—H5	119.7
C8—N1—C1	123.49 (11)	C6—C5—H5	119.7
N2—N1—C1	126.72 (10)	C5—C6—C1	118.70 (12)
C9—N2—N1	101.13 (10)	C5—C6—C7	120.00 (12)
C8—N3—C9	102.03 (11)	C1—C6—C7	121.28 (13)
C8—N4—C7	123.00 (11)	O1—C7—N4	121.17 (13)
C8—N4—H4	114.7 (15)	O1—C7—C6	123.63 (14)
C7—N4—H4	122.3 (15)	N4—C7—C6	115.18 (12)
N1—C1—C2	122.47 (12)	N3—C8—N1	111.11 (11)
N1—C1—C6	116.55 (11)	N3—C8—N4	128.60 (11)
C2—C1—C6	120.97 (12)	N1—C8—N4	120.29 (12)
C3—C2—C1	118.83 (13)	N2—C9—N3	115.95 (12)
C3—C2—H2	120.6	N2—C9—S1	125.39 (9)
C1—C2—H2	120.6	N3—C9—S1	118.65 (10)
C2—C3—C4	121.01 (13)	S1—C10—H10A	109.5
C2—C3—H3	119.5	S1—C10—H10B	109.5
C4—C3—H3	119.5	H10A—C10—H10B	109.5
C5—C4—C3	119.85 (13)	S1—C10—H10C	109.5
C5—C4—H4A	120.1	H10A—C10—H10C	109.5
C3—C4—H4A	120.1	H10B—C10—H10C	109.5
C8—N1—N2—C9	0.85 (13)	C5—C6—C7—O1	-1.4 (2)

C1—N1—N2—C9	179.10 (12)	C1—C6—C7—O1	-179.72 (14)
C8—N1—C1—C2	-175.64 (12)	C5—C6—C7—N4	177.42 (13)
N2—N1—C1—C2	6.33 (19)	C1—C6—C7—N4	-0.88 (19)
C8—N1—C1—C6	3.12 (18)	C9—N3—C8—N1	1.23 (14)
N2—N1—C1—C6	-174.91 (11)	C9—N3—C8—N4	-178.64 (14)
N1—C1—C2—C3	179.92 (12)	N2—N1—C8—N3	-1.39 (15)
C6—C1—C2—C3	1.2 (2)	C1—N1—C8—N3	-179.71 (11)
C1—C2—C3—C4	0.7 (2)	N2—N1—C8—N4	178.50 (11)
C2—C3—C4—C5	-1.6 (2)	C1—N1—C8—N4	0.17 (19)
C3—C4—C5—C6	0.6 (2)	C7—N4—C8—N3	175.64 (14)
C4—C5—C6—C1	1.3 (2)	C7—N4—C8—N1	-4.2 (2)
C4—C5—C6—C7	-177.05 (13)	N1—N2—C9—N3	-0.07 (14)
N1—C1—C6—C5	179.03 (12)	N1—N2—C9—S1	179.08 (9)
C2—C1—C6—C5	-2.2 (2)	C8—N3—C9—N2	-0.71 (15)
N1—C1—C6—C7	-2.66 (18)	C8—N3—C9—S1	-179.93 (9)
C2—C1—C6—C7	176.13 (12)	C10—S1—C9—N2	2.50 (13)
C8—N4—C7—O1	-176.73 (14)	C10—S1—C9—N3	-178.36 (11)
C8—N4—C7—C6	4.4 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N4—H4...N3 ⁱ	0.85 (2)	2.05 (2)	2.896 (2)	174 (2)

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