Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methylsulfanyl-9*H*-1,3,4-thiadiazolo-[2,3-*b*]quinazolin-9-one

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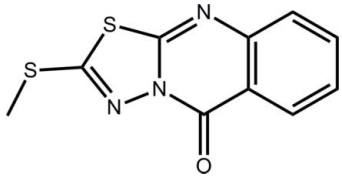
Received 9 June 2012; accepted 9 June 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.082; data-to-parameter ratio = 14.6.

In the title compound, $C_{10}H_7N_3OS_2$, the 16 non-H atoms are almost planar (r.m.s. deviation = 0.037 Å) and the S-bound methyl group is *syn* to the ketone O atom. In the crystal, centrosymmetrically related molecules are connected by pairs of C—H···O interactions between the ketone O and methyl H atoms. The dimeric aggregates are connected into a linear supramolecular chain along the *b* axis *via* π–π interactions occurring between the five-membered and benzene rings [centroid–centroid distance = 3.6123 (9) Å]. The chains assemble into layers in the *bc* plane *via* S···S interactions involving the endocyclic S atoms [S···S = 3.4607 (6) and 3.4792 (6) Å].

Related literature

For recent studies on the synthesis and biological properties of quinazoline-4(3*H*)-one derivatives, see: El-Azab & ElTahir (2012); El-Azab *et al.* (2011). For the synthesis and antimicrobial activity of the title compound, see: El-Azab (2007).



Experimental

Crystal data

$C_{10}H_7N_3OS_2$
M_r = 249.31
Monoclinic, $P2_1/c$
a = 11.8193 (4) Å
b = 4.9841 (2) Å
c = 17.4985 (6) Å
β = 91.453 (3)°
V = 1030.48 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 4.53 mm^{-1}
T = 100 K
0.30 × 0.10 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.344, T_{max} = 0.876
3781 measured reflections
2110 independent reflections
1937 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.082
S = 1.09
2110 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O1^{i}	0.98	2.32	3.170 (2)	145

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

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: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the Deanship of Scientific Research and the Research Center of the College of Pharmacy, King Saud University. We also thank the Ministry of Higher Education (Malaysia) for funding structural studies through the High-Impact Research scheme (UM.C/HIR/MOHE/SC/12).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6843).

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supporting information

Acta Cryst. (2012). E**68**, o2134 [https://doi.org/10.1107/S1600536812026189]

2-Methylsulfanyl-9*H*-1,3,4-thiadiazolo[2,3-*b*]quinazolin-9-one

Adel S. El-Azab, Alaa A.-M. Abdel-Aziz, Ibrahim A. Al-Swaidan, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

Quinazoline-4(3*H*)-one derivatives attract interest owing to their putative biological activity (El-Azab & ElTahir, 2012; El-Azab *et al.*, 2011). The title compound, 2-(methylthio)-5*H*-[1,3,4]thiadiazolo[2,3-*b*]quinazolin-5-one (I), has been synthesized previously and evaluated for its anti-microbial activity (El-Azab, 2007). Herein, we describe its crystal structure determination.

The 16 non-hydrogen atoms in (I), Fig. 1, are planar with the r.m.s. deviation being 0.037 Å. The maximum deviations from the least-squares plane are 0.068 (1) Å for the ketone-O1 atom and -0.065 (2) Å for the methyl-C10 atom. The *S*-bound methyl group is *syn* to the ketone-O1 atom.

In the crystal packing, centrosymmetrically related molecules are connected by C—H···O interactions between the ketone-O and methyl-H atoms, Table 1, *via* a 16-membered $\{\cdots HCSCN_2CO\}_2$ synthon, Fig. 2. The dimeric aggregates are connected into a linear supramolecular chain along the *b* axis *via* *π*—*π* interactions occurring between the five-membered and benzene rings [inter-centroid distance = 3.6123 (9) Å, angle of inclination = 2.09 (7)° for symmetry operation: *x*, 1 + *y*, *z*]. The chains assemble into layers in the *bc* plane *via* S···S interactions involving the endocyclic-S1 atoms whereby each S1 atom forms two such interactions [S1···S1^{i} = 3.4607 (6) Å for symmetry operation *i*: 2 - *x*,2 - *y*,1 - *z*; and S1···S1^{ii} = 3.4792 (6) Å for *ii*: 2 - *x*, 1 - *y*, 1 - *z*]. Layers stack along the *a* axis without specific interactions between them, Fig. 3.

S2. Experimental

A mixture of 2-mercapto-5*H*-[1,3,4]thiadiazolo[2,3-*b*]quinazolin-5-one (470 mg, 2 mmol) and methyliodide (2.1 mmol) in acetone (10 ml) containing anhydrous potassium carbonate (300 mg) was stirred at room temperature for 12 h. The reaction mixture was filtered, the solvent removed under reduced pressure and the solid obtained was dried and recrystallized from ethanol. Yield 88%. ^{1}H NMR ($CDCl_3$): *δ* 8.42 (d, 1H, J = 7.5 Hz), 7.79 (t, 1H, J = 7.0 Hz), 7.63 (d, 1H, J = 8.0 Hz), 7.49 (t, 1H, J = 7.0 Hz), 2.84 (s, 3H) p.p.m.. ^{13}C NMR ($CDCl_3$): *δ* = 15.3, 118.9, 126.2, 127.6, 134.8, 147.2, 156.2, 157.1, 158.5 p.p.m..

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 to 0.98 Å, U_{iso}(H) = 1.2–1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

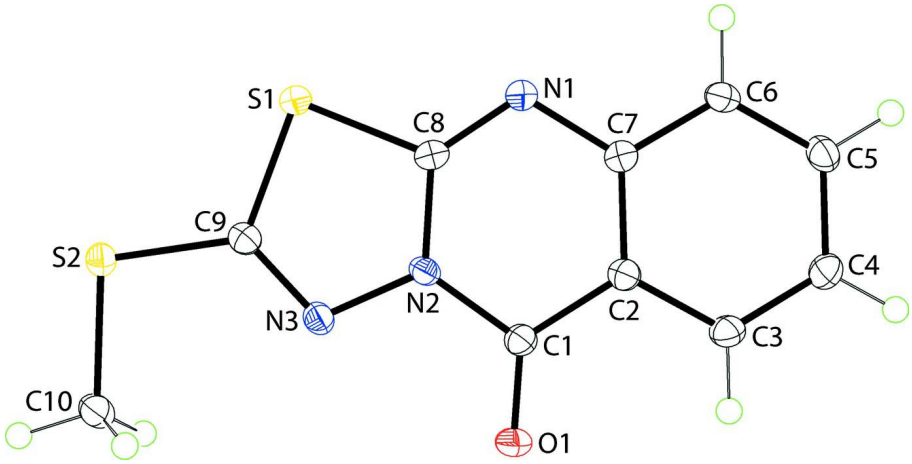

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

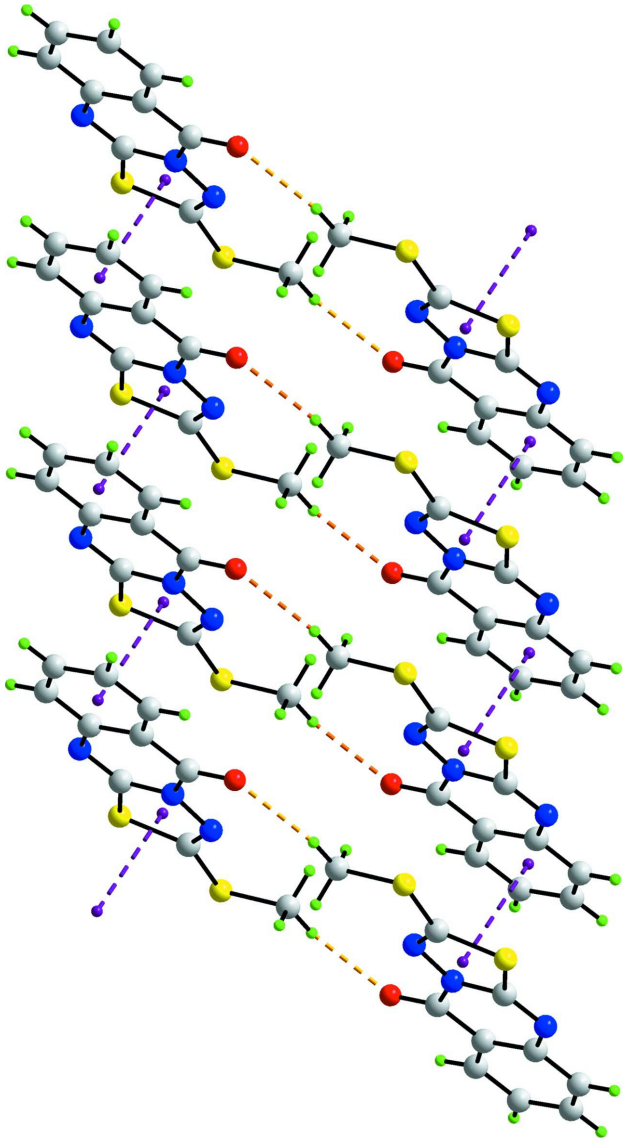

Figure 2

A view of the linear supramolecular chain along the *b* axis in (I). The C—H···O and π—π interactions are shown as orange and purple dashed lines respectively.

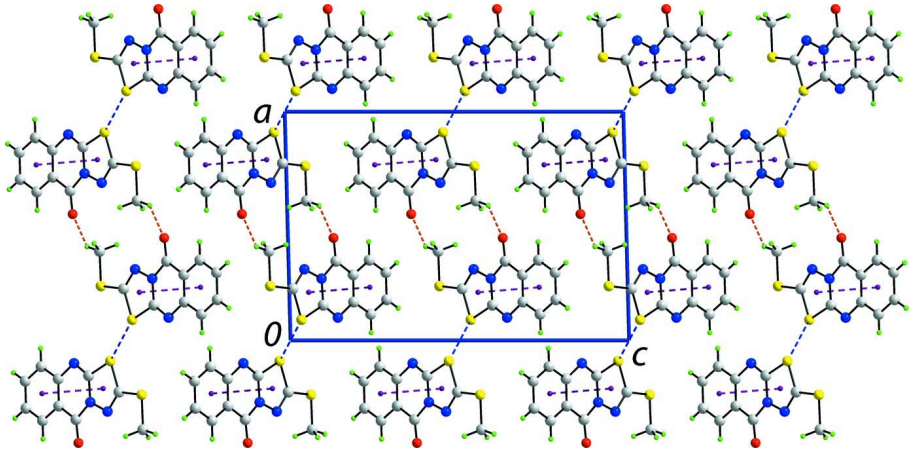

Figure 3

A view in projection down the *b* axis of the unit-cell contents for (I). The C—H···O, π—π and S···S interactions are shown as orange, purple and blue dashed lines respectively.

2-Methylsulfanyl-9*H*-1,3,4-thiadiazolo[2,3-*b*]quinazolin-9-one

Crystal data

$C_{10}H_7N_3OS_2$
$M_r = 249.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.8193$ (4) Å
$b = 4.9841$ (2) Å
$c = 17.4985$ (6) Å
$\beta = 91.453$ (3)°
$V = 1030.48$ (6) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.607$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2166 reflections
$\theta = 3.7$–$76.5°$
$\mu = 4.53$ mm^{-1}
$T = 100$ K
Prism, colourless
0.30 × 0.10 × 0.03 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)
$T_{min} = 0.344$, $T_{max} = 0.876$
3781 measured reflections
2110 independent reflections
1937 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 76.7°$, $\theta_{min} = 3.7°$
$h = -12 \rightarrow 14$
$k = -5 \rightarrow 6$
$l = -15 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.082$
$S = 1.09$
2110 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.3686P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.90968 (3)	0.75129 (8)	0.46619 (2)	0.01702 (12)
S2	0.76245 (3)	1.12015 (8)	0.55613 (2)	0.01660 (12)
N1	0.90012 (11)	0.3645 (3)	0.35873 (8)	0.0160 (3)
N2	0.72981 (11)	0.5607 (3)	0.40394 (7)	0.0145 (3)
N3	0.68915 (11)	0.7516 (3)	0.45394 (7)	0.0160 (3)
O1	0.55197 (9)	0.4218 (3)	0.36566 (7)	0.0217 (3)
C1	0.65417 (13)	0.4007 (3)	0.36022 (9)	0.0159 (3)
C2	0.71312 (13)	0.2152 (3)	0.31034 (9)	0.0153 (3)
C3	0.64945 (13)	0.0467 (4)	0.26154 (9)	0.0183 (3)
H3	0.5691	0.0533	0.2616	0.022*
C4	0.70332 (14)	−0.1282 (4)	0.21351 (9)	0.0196 (3)
H4	0.6602	−0.2429	0.1806	0.024*
C5	0.82196 (14)	−0.1368 (3)	0.21325 (9)	0.0192 (3)
H5	0.8589	−0.2560	0.1796	0.023*
C6	0.88539 (13)	0.0261 (3)	0.26135 (9)	0.0179 (3)
H6	0.9657	0.0173	0.2609	0.021*
C7	0.83228 (13)	0.2050 (3)	0.31091 (9)	0.0149 (3)
C8	0.84595 (12)	0.5289 (3)	0.40102 (8)	0.0149 (3)
C9	0.77423 (13)	0.8644 (3)	0.48930 (9)	0.0152 (3)
C10	0.61018 (14)	1.1556 (4)	0.55743 (10)	0.0211 (3)
H10A	0.5903	1.2965	0.5938	0.032*
H10B	0.5762	0.9854	0.5730	0.032*
H10C	0.5815	1.2041	0.5062	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01263 (19)	0.0180 (2)	0.0204 (2)	−0.00013 (13)	−0.00028 (14)	−0.00389 (14)
S2	0.0170 (2)	0.0176 (2)	0.0151 (2)	0.00105 (14)	−0.00022 (14)	−0.00248 (14)
N1	0.0136 (6)	0.0176 (7)	0.0167 (6)	0.0001 (5)	0.0003 (5)	−0.0019 (5)
N2	0.0124 (6)	0.0179 (7)	0.0133 (6)	0.0031 (5)	0.0007 (5)	−0.0011 (5)
N3	0.0161 (6)	0.0180 (7)	0.0139 (6)	0.0031 (5)	0.0001 (5)	−0.0018 (5)
O1	0.0118 (5)	0.0294 (7)	0.0239 (6)	0.0024 (5)	−0.0007 (4)	−0.0067 (5)
C1	0.0148 (7)	0.0191 (7)	0.0138 (7)	0.0014 (6)	−0.0011 (6)	−0.0006 (6)
C2	0.0151 (7)	0.0176 (7)	0.0131 (7)	0.0011 (6)	−0.0001 (5)	0.0006 (6)
C3	0.0146 (7)	0.0227 (8)	0.0176 (7)	0.0013 (7)	−0.0018 (6)	−0.0009 (6)
C4	0.0190 (8)	0.0219 (8)	0.0177 (8)	0.0007 (6)	−0.0041 (6)	−0.0031 (6)
C5	0.0204 (8)	0.0207 (8)	0.0166 (7)	0.0037 (6)	0.0012 (6)	−0.0032 (6)
C6	0.0135 (7)	0.0206 (8)	0.0195 (7)	0.0022 (6)	0.0013 (6)	−0.0013 (6)
C7	0.0149 (7)	0.0160 (7)	0.0139 (7)	−0.0004 (6)	0.0001 (5)	0.0017 (6)
C8	0.0127 (7)	0.0159 (7)	0.0160 (7)	−0.0008 (6)	−0.0005 (5)	0.0023 (6)
C9	0.0155 (7)	0.0160 (7)	0.0141 (7)	0.0019 (6)	0.0005 (5)	0.0014 (6)
C10	0.0178 (7)	0.0261 (9)	0.0194 (8)	0.0048 (7)	0.0015 (6)	−0.0032 (7)

Geometric parameters (Å, º)

S1—C8	1.7473 (16)	C2—C7	1.409 (2)
S1—C9	1.7542 (16)	C3—C4	1.378 (2)
S2—C9	1.7378 (16)	C3—H3	0.9500
S2—C10	1.8091 (17)	C4—C5	1.403 (2)
N1—C8	1.286 (2)	C4—H4	0.9500
N1—C7	1.393 (2)	C5—C6	1.378 (2)
N2—C8	1.3840 (18)	C5—H5	0.9500
N2—N3	1.3866 (18)	C6—C7	1.403 (2)
N2—C1	1.409 (2)	C6—H6	0.9500
N3—C9	1.296 (2)	C10—H10A	0.9800
O1—C1	1.2186 (19)	C10—H10B	0.9800
C1—C2	1.461 (2)	C10—H10C	0.9800
C2—C3	1.403 (2)		
C8—S1—C9	88.48 (7)	C6—C5—H5	119.7
C9—S2—C10	100.19 (8)	C4—C5—H5	119.7
C8—N1—C7	114.96 (13)	C5—C6—C7	120.47 (14)
C8—N2—N3	117.51 (13)	C5—C6—H6	119.8
C8—N2—C1	122.09 (13)	C7—C6—H6	119.8
N3—N2—C1	120.37 (12)	N1—C7—C6	118.30 (14)
C9—N3—N2	108.78 (13)	N1—C7—C2	122.93 (14)
O1—C1—N2	121.69 (14)	C6—C7—C2	118.77 (14)
O1—C1—C2	126.16 (15)	N1—C8—N2	127.10 (14)
N2—C1—C2	112.15 (13)	N1—C8—S1	124.55 (12)
C3—C2—C7	120.23 (14)	N2—C8—S1	108.35 (11)
C3—C2—C1	119.08 (14)	N3—C9—S2	124.42 (12)
C7—C2—C1	120.69 (14)	N3—C9—S1	116.88 (12)
C4—C3—C2	120.06 (15)	S2—C9—S1	118.69 (9)
C4—C3—H3	120.0	S2—C10—H10A	109.5
C2—C3—H3	120.0	S2—C10—H10B	109.5
C3—C4—C5	119.90 (15)	H10A—C10—H10B	109.5
C3—C4—H4	120.0	S2—C10—H10C	109.5
C5—C4—H4	120.0	H10A—C10—H10C	109.5
C6—C5—C4	120.56 (15)	H10B—C10—H10C	109.5
C8—N2—N3—C9	−0.24 (19)	C3—C2—C7—N1	−179.16 (15)
C1—N2—N3—C9	177.60 (13)	C1—C2—C7—N1	0.9 (2)
C8—N2—C1—O1	176.66 (15)	C3—C2—C7—C6	0.7 (2)
N3—N2—C1—O1	−1.1 (2)	C1—C2—C7—C6	−179.20 (15)
C8—N2—C1—C2	−3.1 (2)	C7—N1—C8—N2	0.4 (2)
N3—N2—C1—C2	179.13 (13)	C7—N1—C8—S1	179.87 (11)
O1—C1—C2—C3	1.9 (3)	N3—N2—C8—N1	−179.87 (15)
N2—C1—C2—C3	−178.30 (14)	C1—N2—C8—N1	2.3 (2)
O1—C1—C2—C7	−178.17 (16)	N3—N2—C8—S1	0.61 (17)
N2—C1—C2—C7	1.6 (2)	C1—N2—C8—S1	−177.18 (12)
C7—C2—C3—C4	−0.5 (2)	C9—S1—C8—N1	179.88 (15)

C1—C2—C3—C4	179.44 (15)	C9—S1—C8—N2	−0.59 (11)
C2—C3—C4—C5	−0.3 (3)	N2—N3—C9—S2	178.77 (11)
C3—C4—C5—C6	0.8 (3)	N2—N3—C9—S1	−0.27 (17)
C4—C5—C6—C7	−0.5 (3)	C10—S2—C9—N3	0.76 (16)
C8—N1—C7—C6	178.13 (15)	C10—S2—C9—S1	179.79 (10)
C8—N1—C7—C2	−2.0 (2)	C8—S1—C9—N3	0.53 (13)
C5—C6—C7—N1	179.67 (15)	C8—S1—C9—S2	−178.57 (10)
C5—C6—C7—C2	−0.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O1^{i}	0.98	2.32	3.170 (2)	145

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