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3-(Prop-2-en-1-yl)-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

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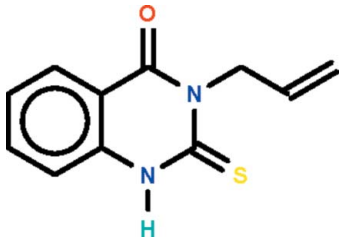
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.108; data-to-parameter ratio = 15.2.

The tetrahydroquinazoline fused-ring system of the title compound, $C_{11}H_{10}N_2OS$, is approximately planar (r.m.s. deviation = 0.019 Å). In the crystal, adjacent molecules are linked by N—H···O hydrogen bonds, forming a chain running along the b axis.

Related literature

For the synthesis, see: Shiau *et al.* (1990); Vassilev & Vassilev (2007).



Experimental

Crystal data

$C_{11}H_{10}N_2OS$
M_r = 218.27
Monoclinic, $P2_1/c$
a = 8.9823 (3) Å
b = 13.7271 (3) Å
c = 8.3137 (2) Å
β = 92.882 (3)°
V = 1023.79 (5) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.59 mm^{-1}
T = 294 K
0.30 × 0.30 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.511, T_{max} = 0.927
4913 measured reflections
2128 independent reflections
1855 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.06
2128 reflections
140 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.87 (1)	2.15 (1)	2.977 (2)	160 (2)

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

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: BT5919).

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supplementary materials

Acta Cryst. (2012). E**68**, o1810 [doi:10.1107/S1600536812021800]

3-(Prop-2-en-1-yl)-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

Rashad Al-Salahi, Mohamed Al-Omar, Mohamed Marzouk and Seik Weng Ng

Comment

The compound, 3-benzyl-8-methoxy-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one, was previously synthesized for a study of its antimicrobial activity. The related 2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one (Scheme I) exhibits cytokinin activity (Vassilev & Vassilev, 2007). The synthesis described in the present study is a more straightforward procedure than those previously reported (Shiau *et al.*, 1990; Vassilev & Vassilev, 2007). The tetrahydroquinazoline fused-ring of $C_{11}H_{10}N_2OS$ is planar (Fig. 1). Adjacent molecules are linked by an N–H···O hydrogen to form a chain running along the *b*-axis of the monoclinic unit cell (Table 1).

Experimental

Allyl isothiocyanate (10 mmol, 0.99 g), 2-amino-5-methylbenzoic acid (10 mmol, 1.51 g) and triethylamine (5 mmol, 0.51 g) in ethanol (30 ml) was heated for two hours. The mixture was poured into ice-cold water. The solid was collected and recrystallized from ethanol to give colorless crystals.

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 0.97 Å, U_{iso}(H) 1.2U_{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.

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).

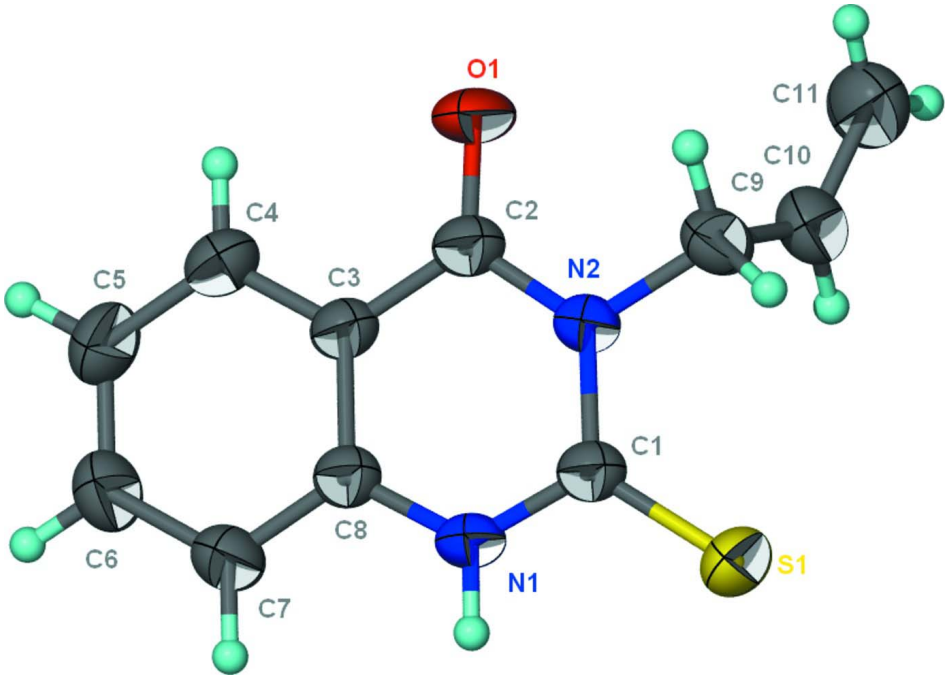

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{11}H_{10}N_2OS$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

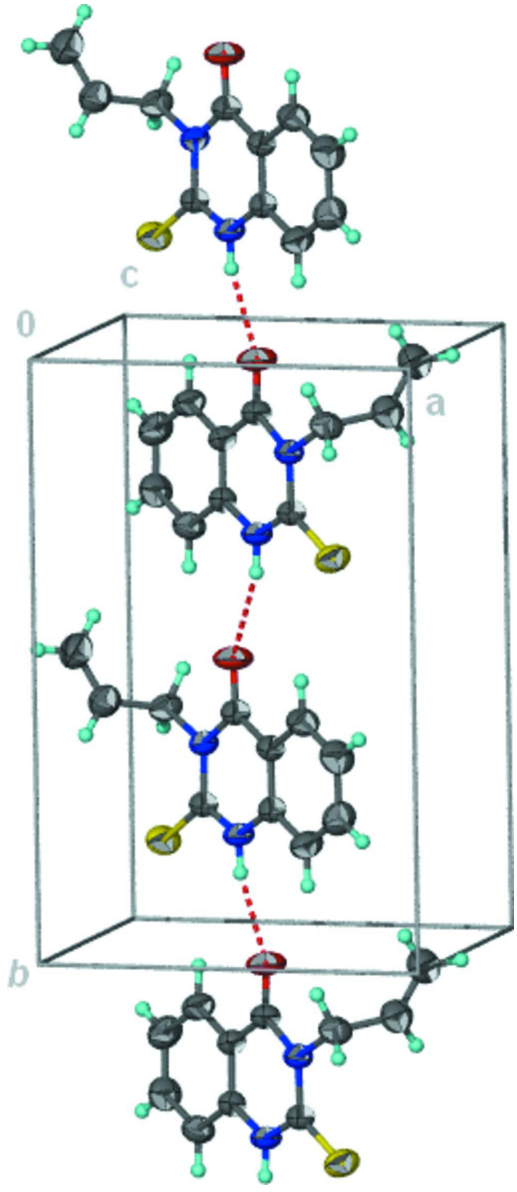

Figure 2

Hydrogen-bond chain structure.

3-(Prop-2-en-1-yl)-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

Crystal data

$C_{11}H_{10}N_2OS$
$M_r = 218.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.9823$ (3) Å
$b = 13.7271$ (3) Å
$c = 8.3137$ (2) Å
$\beta = 92.882$ (3)°
$V = 1023.79$ (5) Å^3
$Z = 4$

$F(000) = 456$
$D_x = 1.416$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2303 reflections
$\theta = 3.2$–$76.4°$
$\mu = 2.59$ mm^{-1}
$T = 294$ K
Prism, colorless
0.30 × 0.30 × 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} = 0.511, T_{max} = 0.927
4913 measured reflections
2128 independent reflections
1855 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 76.6°, θ_{min} = 5.9°
h = −11→10
k = −17→10
l = −10→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.06
2128 reflections
140 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
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.1451P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.77201 (5)	0.32274 (3)	0.07911 (5)	0.04374 (16)
O1	0.54446 (14)	0.00715 (8)	0.22169 (16)	0.0488 (3)
N1	0.53208 (14)	0.29760 (9)	0.24120 (16)	0.0337 (3)
H1	0.530 (2)	0.3608 (7)	0.239 (2)	0.052 (6)*
N2	0.64135 (14)	0.15416 (9)	0.15732 (15)	0.0317 (3)
C1	0.64308 (17)	0.25505 (10)	0.16333 (17)	0.0317 (3)
C2	0.53764 (17)	0.09571 (11)	0.23320 (18)	0.0341 (3)
C3	0.42514 (16)	0.14680 (11)	0.32027 (18)	0.0320 (3)
C4	0.31809 (19)	0.09589 (12)	0.4035 (2)	0.0404 (4)
H4	0.3200	0.0282	0.4068	0.048*
C5	0.2099 (2)	0.14630 (14)	0.4804 (2)	0.0458 (4)
H5	0.1379	0.1125	0.5345	0.055*
C6	0.20775 (19)	0.24781 (14)	0.4776 (2)	0.0445 (4)
H6	0.1339	0.2813	0.5293	0.053*
C7	0.3140 (2)	0.29896 (11)	0.3990 (2)	0.0393 (4)
H7	0.3127	0.3667	0.3982	0.047*
C8	0.42342 (16)	0.24836 (11)	0.32063 (17)	0.0311 (3)
C9	0.76129 (18)	0.10324 (12)	0.07515 (18)	0.0376 (4)
H9A	0.7907	0.1420	−0.0155	0.045*
H9B	0.7242	0.0412	0.0340	0.045*
C10	0.89456 (19)	0.08610 (13)	0.1881 (2)	0.0433 (4)
H10	0.9300	0.1382	0.2505	0.052*
C11	0.9639 (2)	0.00289 (15)	0.2043 (3)	0.0568 (5)
H11A	0.9313	−0.0506	0.1436	0.068*
H11B	1.0459	−0.0029	0.2765	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0415 (3)	0.0325 (2)	0.0586 (3)	−0.00599 (15)	0.01623 (19)	0.00349 (16)
O1	0.0559 (7)	0.0207 (5)	0.0712 (8)	−0.0015 (5)	0.0180 (6)	−0.0008 (5)
N1	0.0363 (7)	0.0203 (6)	0.0448 (7)	0.0004 (5)	0.0068 (6)	0.0020 (5)
N2	0.0324 (6)	0.0236 (6)	0.0393 (6)	0.0015 (5)	0.0055 (5)	−0.0004 (5)
C1	0.0328 (7)	0.0252 (6)	0.0369 (7)	−0.0002 (5)	0.0014 (6)	0.0009 (5)
C2	0.0358 (8)	0.0252 (7)	0.0414 (8)	−0.0004 (6)	0.0014 (6)	0.0025 (6)
C3	0.0319 (7)	0.0254 (7)	0.0387 (7)	−0.0019 (6)	0.0015 (6)	0.0016 (5)
C4	0.0406 (9)	0.0305 (7)	0.0505 (9)	−0.0053 (6)	0.0072 (7)	0.0044 (6)
C5	0.0414 (9)	0.0461 (9)	0.0511 (9)	−0.0086 (7)	0.0141 (7)	0.0039 (7)
C6	0.0383 (9)	0.0460 (9)	0.0502 (9)	0.0042 (7)	0.0124 (7)	−0.0006 (7)
C7	0.0407 (9)	0.0301 (7)	0.0477 (9)	0.0047 (6)	0.0086 (7)	0.0007 (6)
C8	0.0308 (7)	0.0265 (7)	0.0358 (7)	0.0001 (5)	0.0009 (6)	0.0020 (5)
C9	0.0408 (9)	0.0316 (8)	0.0413 (8)	0.0052 (6)	0.0103 (7)	−0.0015 (6)
C10	0.0382 (8)	0.0422 (9)	0.0502 (9)	0.0042 (7)	0.0092 (7)	−0.0025 (7)
C11	0.0431 (10)	0.0497 (10)	0.0774 (13)	0.0049 (8)	0.0004 (9)	0.0047 (9)

Geometric parameters (Å, º)

S1—C1	1.6663 (15)	C5—C6	1.394 (3)
O1—C2	1.2212 (18)	C5—H5	0.9300
N1—C1	1.3487 (19)	C6—C7	1.376 (2)
N1—C8	1.3822 (19)	C6—H6	0.9300
N1—H1	0.869 (9)	C7—C8	1.392 (2)
N2—C1	1.3858 (17)	C7—H7	0.9300
N2—C2	1.4031 (19)	C9—C10	1.502 (2)
N2—C9	1.4799 (18)	C9—H9A	0.9700
C2—C3	1.453 (2)	C9—H9B	0.9700
C3—C8	1.394 (2)	C10—C11	1.305 (3)
C3—C4	1.399 (2)	C10—H10	0.9300
C4—C5	1.377 (2)	C11—H11A	0.9300
C4—H4	0.9300	C11—H11B	0.9300
C1—N1—C8	125.05 (13)	C7—C6—C5	120.60 (15)
C1—N1—H1	116.1 (14)	C7—C6—H6	119.7
C8—N1—H1	118.8 (14)	C5—C6—H6	119.7
C1—N2—C2	124.18 (12)	C6—C7—C8	119.37 (15)
C1—N2—C9	118.79 (12)	C6—C7—H7	120.3
C2—N2—C9	116.91 (12)	C8—C7—H7	120.3
N1—C1—N2	116.28 (13)	N1—C8—C7	120.78 (14)
N1—C1—S1	120.42 (11)	N1—C8—C3	118.67 (13)
N2—C1—S1	123.29 (11)	C7—C8—C3	120.55 (14)
O1—C2—N2	119.80 (14)	N2—C9—C10	111.18 (12)
O1—C2—C3	123.95 (14)	N2—C9—H9A	109.4
N2—C2—C3	116.24 (13)	C10—C9—H9A	109.4
C8—C3—C4	119.36 (14)	N2—C9—H9B	109.4
C8—C3—C2	119.46 (13)	C10—C9—H9B	109.4
C4—C3—C2	121.18 (14)	H9A—C9—H9B	108.0

C5—C4—C3	119.84 (15)	C11—C10—C9	124.25 (18)
C5—C4—H4	120.1	C11—C10—H10	117.9
C3—C4—H4	120.1	C9—C10—H10	117.9
C4—C5—C6	120.25 (15)	C10—C11—H11A	120.0
C4—C5—H5	119.9	C10—C11—H11B	120.0
C6—C5—H5	119.9	H11A—C11—H11B	120.0
C8—N1—C1—N2	3.4 (2)	C2—C3—C4—C5	−177.85 (15)
C8—N1—C1—S1	−177.21 (12)	C3—C4—C5—C6	−0.9 (3)
C2—N2—C1—N1	−3.4 (2)	C4—C5—C6—C7	−0.4 (3)
C9—N2—C1—N1	−179.29 (13)	C5—C6—C7—C8	0.6 (3)
C2—N2—C1—S1	177.16 (11)	C1—N1—C8—C7	179.38 (14)
C9—N2—C1—S1	1.30 (19)	C1—N1—C8—C3	−0.9 (2)
C1—N2—C2—O1	−179.93 (14)	C6—C7—C8—N1	−179.70 (15)
C9—N2—C2—O1	−4.0 (2)	C6—C7—C8—C3	0.5 (2)
C1—N2—C2—C3	1.0 (2)	C4—C3—C8—N1	178.43 (14)
C9—N2—C2—C3	176.97 (12)	C2—C3—C8—N1	−1.7 (2)
O1—C2—C3—C8	−177.37 (15)	C4—C3—C8—C7	−1.8 (2)
N2—C2—C3—C8	1.6 (2)	C2—C3—C8—C7	178.01 (14)
O1—C2—C3—C4	2.4 (2)	C1—N2—C9—C10	86.69 (17)
N2—C2—C3—C4	−178.57 (14)	C2—N2—C9—C10	−89.46 (16)
C8—C3—C4—C5	2.0 (2)	N2—C9—C10—C11	132.15 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.87 (1)	2.15 (1)	2.977 (2)	160 (2)

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