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3-Benzyl-8-methoxy-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

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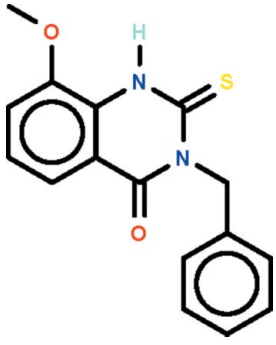
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.114; data-to-parameter ratio = 14.8.

The tetrahydroquinazole fused-ring system of the title compound, $C_{16}H_{14}N_2O_2S$, is roughly planar (r.m.s. deviation = 0.039 Å); the phenyl ring of the benzyl substituent is aligned at 78.1 (1)° with respect to the mean plane of the fused-ring system. In the crystal, two molecules are linked by a pair of N—H···S hydrogen bonds about a center of inversion, generating a dimer.

Related literature

For the synthesis, see: Al-Omar *et al.* (2004).



Experimental

Crystal data

$C_{16}H_{14}N_2O_2S$ M_r = 298.35
Triclinic, $P\overline{1}$
a = 6.3025 (5) Å
b = 10.8353 (5) Å
c = 11.0144 (7) Å
α = 101.728 (5)°
β = 102.419 (6)°
γ = 101.693 (5)°
V = 694.95 (8) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 2.12 mm^{-1}
T = 294 K
0.40 × 0.30 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.484, T_{max} = 0.676
11149 measured reflections
2888 independent reflections
2714 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.114
S = 1.06
2888 reflections
195 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.87 (1)	2.63 (1)	3.493 (1)	174 (2)

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

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: *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: BT5918).

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Al-Omar, M. A., Abdel-Hamide, S. G., Al-Khamees, H. A. & El-Subbagh, H. I. (2004). *Saudi Pharm. J.* **12**, 63–71.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2012). E**68**, o1807 [doi:10.1107/S1600536812021794]

3-Benzyl-8-methoxy-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

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

Comment

The compound (Scheme I) was previously synthesized for a study of its antimicrobial activity. The background to this class of compounds is discussed (Al-Omar *et al.*, 2004). The tetrahydroquinazole fused-ring of $C_{16}H_{14}N_2O_2S$ is flat; the phenyl ring of the benzyl substituent is aligned at 78.1 (1) ° with respect to the fused-ring (Fig. 1). Two molecules are linked by an N–H···S hydrogen bond about a center of inversion to generate a dimer (Table 1).

Experimental

Benzyl isothiocyanate (10 mmol, 1.35 g), 2-amino-3-methoxybenzoic acid (10 mmol,1.67 g) and triethylamine (5 mmol, 0.51 g) in ethanol (30 ml) was heated under reflux for two hours. After cooling, the mixture was poured into ice-cold water. The resulting solid was filtered, washed with water and dried. Recrystallization from ethanol gave 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 0.97 Å, U_{iso}(H) 1.2 to 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.

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).

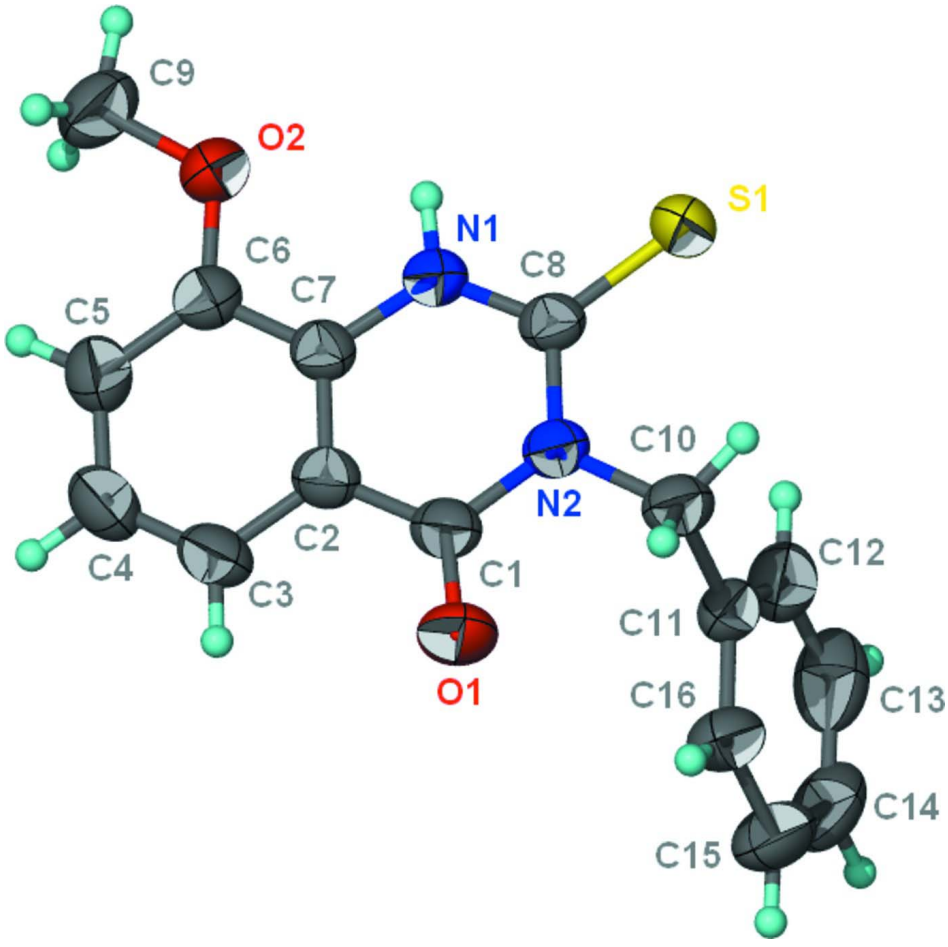

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{16}H_{14}N_2O_2S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3-Benzyl-8-methoxy-2-sulfanylidene-1,2,3,4-tetrahydroquinazolin-4-one

Crystal data

$C_{16}H_{14}N_2O_2S$
$M_r = 298.35$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.3025\ (5)$ Å
$b = 10.8353\ (5)$ Å
$c = 11.0144\ (7)$ Å
$\alpha = 101.728\ (5)°$
$\beta = 102.419\ (6)°$
$\gamma = 101.693\ (5)°$
$V = 694.95\ (8)$ Å^3

$Z = 2$
$F(000) = 312$
$D_x = 1.426$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 6739 reflections
$\theta = 4.3–76.5°$
$\mu = 2.12$ mm^{-1}
$T = 294$ K
Prism, colorless
0.40 × 0.30 × 0.20 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} = 0.484, T_{max} = 0.676
11149 measured reflections
2888 independent reflections
2714 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 76.7°, θ_{min} = 4.3°
h = −7→7
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.114
S = 1.06
2888 reflections
195 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
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.1365P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.84360 (6)	1.04442 (3)	0.64336 (3)	0.04455 (15)
O1	0.15151 (18)	0.69504 (12)	0.56998 (12)	0.0543 (3)
O2	0.8326 (2)	0.67738 (11)	0.27822 (11)	0.0509 (3)
N1	0.7112 (2)	0.81847 (12)	0.46983 (11)	0.0376 (3)
H1	0.829 (2)	0.8501 (18)	0.4456 (18)	0.051 (5)*
N2	0.47878 (18)	0.84651 (11)	0.60456 (11)	0.0355 (3)
C1	0.3257 (2)	0.72495 (14)	0.53999 (14)	0.0400 (3)
C2	0.3924 (2)	0.64174 (14)	0.44134 (13)	0.0388 (3)
C3	0.2672 (3)	0.51250 (15)	0.38286 (15)	0.0471 (3)
H3	0.1396	0.4778	0.4067	0.057*
C4	0.3356 (3)	0.43832 (15)	0.29020 (17)	0.0518 (4)
H4	0.2549	0.3520	0.2525	0.062*
C5	0.5233 (3)	0.48904 (16)	0.25080 (16)	0.0485 (4)
H5	0.5643	0.4374	0.1860	0.058*
C6	0.6480 (3)	0.61582 (14)	0.30800 (13)	0.0408 (3)
C7	0.5842 (2)	0.69243 (13)	0.40631 (13)	0.0363 (3)
C8	0.6691 (2)	0.89590 (13)	0.56926 (12)	0.0345 (3)
C9	0.8974 (3)	0.60835 (19)	0.17369 (17)	0.0571 (4)
H9A	1.0333	0.6606	0.1652	0.086*
H9B	0.7802	0.5900	0.0957	0.086*
H9C	0.9225	0.5279	0.1897	0.086*
C10	0.4113 (2)	0.92622 (14)	0.70865 (14)	0.0397 (3)
H10A	0.2568	0.9289	0.6768	0.048*
H10B	0.5053	1.0148	0.7331	0.048*
C11	0.4320 (2)	0.87257 (13)	0.82580 (13)	0.0401 (3)

C12	0.6395 (3)	0.88228 (16)	0.90532 (16)	0.0501 (4)
H12	0.7699	0.9204	0.8860	0.060*
C13	0.6537 (4)	0.8349 (2)	1.01459 (18)	0.0662 (5)
H13	0.7937	0.8417	1.0682	0.079*
C14	0.4613 (5)	0.77823 (19)	1.04350 (18)	0.0698 (6)
H14	0.4714	0.7466	1.1164	0.084*
C15	0.2558 (4)	0.76845 (19)	0.96517 (19)	0.0666 (5)
H15	0.1258	0.7299	0.9846	0.080*
C16	0.2402 (3)	0.81568 (17)	0.85676 (16)	0.0524 (4)
H16	0.0994	0.8091	0.8041	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0432 (2)	0.0386 (2)	0.0452 (2)	−0.00269 (15)	0.01814 (16)	0.00262 (15)
O1	0.0405 (6)	0.0597 (7)	0.0568 (7)	−0.0045 (5)	0.0229 (5)	0.0089 (5)
O2	0.0567 (7)	0.0465 (6)	0.0483 (6)	0.0031 (5)	0.0278 (5)	0.0052 (5)
N1	0.0370 (6)	0.0377 (6)	0.0364 (6)	0.0019 (5)	0.0151 (5)	0.0081 (5)
N2	0.0347 (6)	0.0383 (6)	0.0344 (6)	0.0057 (4)	0.0133 (4)	0.0109 (4)
C1	0.0357 (7)	0.0444 (8)	0.0375 (7)	0.0020 (6)	0.0101 (5)	0.0133 (6)
C2	0.0385 (7)	0.0392 (7)	0.0358 (7)	0.0029 (5)	0.0093 (5)	0.0112 (5)
C3	0.0429 (8)	0.0429 (8)	0.0493 (8)	−0.0027 (6)	0.0117 (6)	0.0124 (6)
C4	0.0542 (9)	0.0365 (7)	0.0540 (9)	−0.0016 (6)	0.0102 (7)	0.0059 (6)
C5	0.0572 (9)	0.0399 (7)	0.0443 (8)	0.0088 (6)	0.0140 (7)	0.0046 (6)
C6	0.0451 (7)	0.0410 (7)	0.0359 (7)	0.0069 (6)	0.0130 (6)	0.0109 (6)
C7	0.0381 (7)	0.0356 (6)	0.0329 (6)	0.0042 (5)	0.0086 (5)	0.0103 (5)
C8	0.0341 (6)	0.0378 (6)	0.0322 (6)	0.0060 (5)	0.0102 (5)	0.0125 (5)
C9	0.0654 (10)	0.0620 (10)	0.0522 (9)	0.0205 (8)	0.0308 (8)	0.0125 (8)
C10	0.0408 (7)	0.0401 (7)	0.0436 (7)	0.0107 (6)	0.0197 (6)	0.0131 (6)
C11	0.0488 (8)	0.0365 (7)	0.0373 (7)	0.0100 (6)	0.0194 (6)	0.0073 (5)
C12	0.0569 (9)	0.0475 (8)	0.0447 (8)	0.0141 (7)	0.0152 (7)	0.0070 (6)
C13	0.0895 (14)	0.0593 (10)	0.0468 (9)	0.0304 (10)	0.0055 (9)	0.0092 (8)
C14	0.1235 (19)	0.0547 (10)	0.0453 (9)	0.0316 (11)	0.0372 (11)	0.0198 (8)
C15	0.0951 (15)	0.0585 (10)	0.0608 (11)	0.0161 (10)	0.0477 (11)	0.0228 (9)
C16	0.0576 (9)	0.0531 (9)	0.0520 (9)	0.0094 (7)	0.0291 (7)	0.0149 (7)

Geometric parameters (Å, º)

S1—C8	1.6818 (14)	C6—C7	1.407 (2)
O1—C1	1.2141 (18)	C9—H9A	0.9600
O2—C6	1.3582 (18)	C9—H9B	0.9600
O2—C9	1.4241 (19)	C9—H9C	0.9600
N1—C8	1.3475 (18)	C10—C11	1.5110 (19)
N1—C7	1.3867 (18)	C10—H10A	0.9700
N1—H1	0.868 (9)	C10—H10B	0.9700
N2—C8	1.3787 (17)	C11—C12	1.379 (2)
N2—C1	1.4074 (18)	C11—C16	1.385 (2)
N2—C10	1.4822 (17)	C12—C13	1.394 (3)
C1—C2	1.457 (2)	C12—H12	0.9300
C2—C7	1.3888 (19)	C13—C14	1.376 (3)

C2—C3	1.402 (2)	C13—H13	0.9300
C3—C4	1.367 (2)	C14—C15	1.364 (3)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.394 (2)	C15—C16	1.384 (2)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.378 (2)	C16—H16	0.9300
C5—H5	0.9300		

C6—O2—C9	117.68 (13)	O2—C9—H9A	109.5
C8—N1—C7	124.75 (12)	O2—C9—H9B	109.5
C8—N1—H1	116.9 (14)	H9A—C9—H9B	109.5
C7—N1—H1	118.4 (14)	O2—C9—H9C	109.5
C8—N2—C1	123.99 (12)	H9A—C9—H9C	109.5
C8—N2—C10	120.75 (11)	H9B—C9—H9C	109.5
C1—N2—C10	115.03 (11)	N2—C10—C11	112.13 (11)
O1—C1—N2	119.33 (14)	N2—C10—H10A	109.2
O1—C1—C2	124.39 (13)	C11—C10—H10A	109.2
N2—C1—C2	116.28 (12)	N2—C10—H10B	109.2
C7—C2—C3	120.22 (14)	C11—C10—H10B	109.2
C7—C2—C1	118.79 (12)	H10A—C10—H10B	107.9
C3—C2—C1	120.99 (13)	C12—C11—C16	118.91 (14)
C4—C3—C2	118.92 (14)	C12—C11—C10	121.20 (13)
C4—C3—H3	120.5	C16—C11—C10	119.87 (14)
C2—C3—H3	120.5	C11—C12—C13	120.01 (18)
C3—C4—C5	121.60 (14)	C11—C12—H12	120.0
C3—C4—H4	119.2	C13—C12—H12	120.0
C5—C4—H4	119.2	C14—C13—C12	120.3 (2)
C6—C5—C4	119.91 (15)	C14—C13—H13	119.9
C6—C5—H5	120.0	C12—C13—H13	119.9
C4—C5—H5	120.0	C15—C14—C13	119.94 (17)
O2—C6—C5	125.90 (14)	C15—C14—H14	120.0
O2—C6—C7	114.79 (12)	C13—C14—H14	120.0
C5—C6—C7	119.31 (14)	C14—C15—C16	120.18 (19)
N1—C7—C2	119.13 (13)	C14—C15—H15	119.9
N1—C7—C6	120.88 (12)	C16—C15—H15	119.9
C2—C7—C6	119.99 (13)	C15—C16—C11	120.70 (18)
N1—C8—N2	116.47 (12)	C15—C16—H16	119.6
N1—C8—S1	120.25 (10)	C11—C16—H16	119.6
N2—C8—S1	123.28 (10)		

C8—N2—C1—O1	−172.81 (13)	O2—C6—C7—N1	−3.0 (2)
C10—N2—C1—O1	1.76 (19)	C5—C6—C7—N1	177.19 (13)
C8—N2—C1—C2	8.29 (19)	O2—C6—C7—C2	177.30 (12)
C10—N2—C1—C2	−177.14 (11)	C5—C6—C7—C2	−2.5 (2)
O1—C1—C2—C7	173.21 (14)	C7—N1—C8—N2	−3.3 (2)
N2—C1—C2—C7	−7.96 (19)	C7—N1—C8—S1	176.82 (10)
O1—C1—C2—C3	−7.2 (2)	C1—N2—C8—N1	−2.82 (19)
N2—C1—C2—C3	171.67 (12)	C10—N2—C8—N1	−177.09 (11)
C7—C2—C3—C4	−0.8 (2)	C1—N2—C8—S1	177.03 (10)

C1—C2—C3—C4	179.61 (14)	C10—N2—C8—S1	2.76 (18)
C2—C3—C4—C5	−1.4 (3)	C8—N2—C10—C11	−112.55 (14)
C3—C4—C5—C6	1.7 (3)	C1—N2—C10—C11	72.68 (15)
C9—O2—C6—C5	3.9 (2)	N2—C10—C11—C12	72.02 (17)
C9—O2—C6—C7	−175.88 (14)	N2—C10—C11—C16	−109.81 (15)
C4—C5—C6—O2	−179.43 (14)	C16—C11—C12—C13	0.2 (2)
C4—C5—C6—C7	0.3 (2)	C10—C11—C12—C13	178.36 (14)
C8—N1—C7—C2	3.3 (2)	C11—C12—C13—C14	0.1 (3)
C8—N1—C7—C6	−176.38 (12)	C12—C13—C14—C15	−0.1 (3)
C3—C2—C7—N1	−176.96 (12)	C13—C14—C15—C16	−0.1 (3)
C1—C2—C7—N1	2.7 (2)	C14—C15—C16—C11	0.4 (3)
C3—C2—C7—C6	2.7 (2)	C12—C11—C16—C15	−0.4 (2)
C1—C2—C7—C6	−177.66 (12)	C10—C11—C16—C15	−178.65 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.87 (1)	2.63 (1)	3.493 (1)	174 (2)

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