Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(3-Sulfanylidene-3,4-dihydroquinoxalin-2-yl)benzohydrazide dimethylformamide monosolvate

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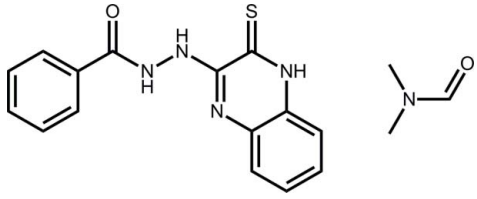
Received 28 June 2013; accepted 10 July 2013

Key indicators: single-crystal X-ray study; T = 180 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.119; data-to-parameter ratio = 17.7.

The 2-sulfanylidene-3,4-dihydroquinoxalin-2-yl ring system of the title solvate, $C_{15}H_{12}N_4OS\cdot C_3H_7NO$, is essentially planar, the maximum deviation from the mean plane being 0.024 (2) Å for the thione C atom. The mean plane through the fused-ring system is almost perpendicular to the terminal phenyl ring, as indicated by the dihedral angle of 70.05 (8)°. In the crystal, the main and solvent molecules are linked by N—H···O hydrogen bonds, forming a layer parallel to (010).

Related literature

For potential applications of quinoxaline derivatives, see: Cheon *et al.* (2004); Jackson *et al.* (1991); Benzeid *et al.* (2012).



Experimental

Crystal data

$C_{15}H_{12}N_4OS\cdot C_3H_7NO$
M_r = 369.44
Monoclinic, $P2_1/c$
a = 10.4053 (2) Å
b = 16.8563 (5) Å
c = 10.3624 (2) Å
β = 100.882 (2)°
V = 1784.83 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 180 K
0.20 × 0.12 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini ultra) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2012)
T_{min} = 0.960, T_{max} = 0.992
15848 measured reflections
4153 independent reflections
3090 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.119
S = 1.03
4153 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*N*···O2^{i}	0.88	2.14	2.906 (2)	146
N4—H4*N*···O2^{ii}	0.88	2.04	2.906 (2)	166
N1—H1···O1^{iii}	0.88	2.01	2.8331 (19)	154

Symmetry codes: (i) $x, y, z+1$; (ii) $-x+1, -y, -z$; (iii) $-x+2, -y, -z+1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2012); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2012); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5236).

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supplementary materials

Acta Cryst. (2013). E**69**, o1268 [doi:10.1107/S1600536813019181]

N′-(3-Sulfanylidene-3,4-dihydroquinoxalin-2-yl)benzohydrazide dimethylformamide monosolvate

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Comment

Quinoxaline derivatives have been discovered as leads for a novel series of dipeptidyl peptidase-IV molecules (Cheon *et al.*, 2004). They are also used as ligands for the strychnine-insensitive glycine site (Jackson *et al.*, 1991) and as new fluorescent probes for amyloid-*β* fibrils (Benzeid *et al.*, 2012).

The crystal structure of title compound is build up from two fused six-membered rings (N1, N2 C1–C8) linked to a benzohydrazide system (N3, N4, O1, C9–C15) and a dimethylformamide solvent molecule as shown in Fig. 1. The fused rings system is almost planar with the maximum deviation from the mean plane being -0.024 (2) Å for the C1 atom. The dihedral angle between the terminal phenyl ring and the fused ring system is 70.05 (8)°. In the crystal structure, the molecules and the solvent are linked by N—H···O hydrogen bond to form a layer parallel to (0 1 0), Table 1.

Experimental

A mixture of quinoxaline-2,3-dithione (1 g, 5.15 10 $^{-3}$ mol), benzhydrazide (1.4 g, 0.01 mol) and DMF (40 ml) was boiled under reflux for 48 h. The volume of DMF was reduced under reduced pressure (2 ml) and the residue was taken up into 100 ml of diethyl ether. An oily product precipitated quickly after addition of diethyl ether. After centrifugation the diethyl ether phase was recovered and the precipitate (oily product) was washed with diethyl ether (2 x 3 ml). The product crystallized in the diethyl ether phase overnight at room temperature. Crystals were collected, washed with diethyl ether (5 ml) and dried under vacuum. Yield: 700 mg, 46%.

Refinement

All H atoms could be located in a difference Fourier map. However, they were placed in calculated positions with C—H = 0.95 Å (aromatic), N—H = 0.88 Å and C—H = 0.98 Å (methyl) and refined as riding on their parent atoms with U_{iso}(H) = 1.2 U_{eq}(aromatic and N) and U_{iso}(H) = 1.5 U_{eq}(methyl).

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2012); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2012); data reduction: *CrysAlis RED* (Oxford Diffraction, 2012); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

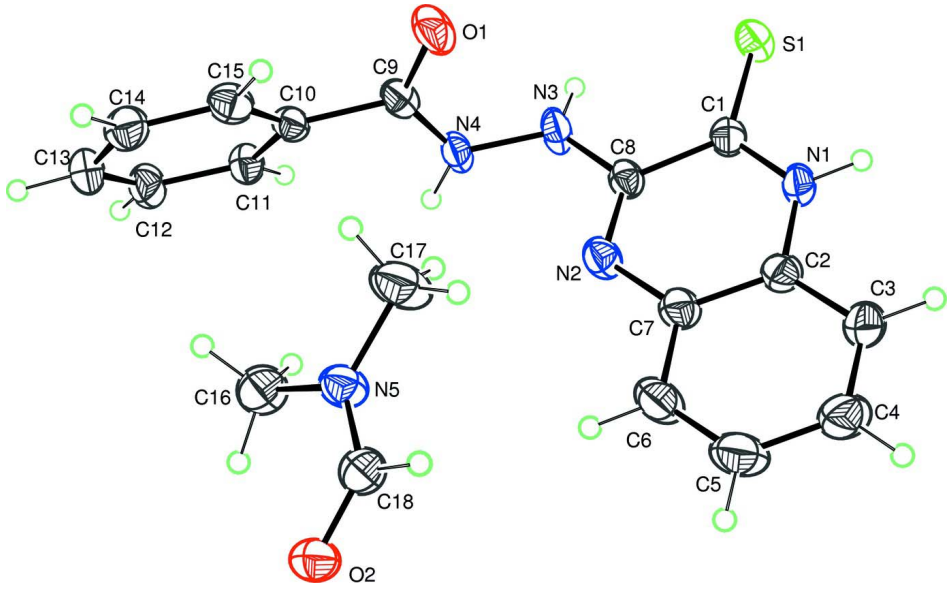

Figure 1

Molecular structures of the components of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

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β = 100.882 (2)°
V = 1784.83 (7) Å^3
Z = 4
$F(000)$ = 776
D_x = 1.375 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4153 reflections
θ = 3.1–27.9°
μ = 0.21 mm^{-1}
T = 180 K
Plate, yellow
0.20 × 0.12 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini ultra) diffractometer
Graphite monochromator
Detector resolution: 16.1978 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2012)
T_{min} = 0.960, T_{max} = 0.992
15848 measured reflections
4153 independent reflections
3090 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.9°, θ_{min} = 3.1°
h = −13→13
k = −21→21
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.119
S = 1.03
4153 reflections
235 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.657P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.90077 (17)	0.07517 (11)	0.52315 (18)	0.0238 (4)
C2	0.97172 (18)	0.13196 (12)	0.33400 (19)	0.0277 (4)
C3	1.0611 (2)	0.18006 (13)	0.2856 (2)	0.0353 (5)
H3	1.1320	0.2040	0.3438	0.042*
C4	1.0448 (2)	0.19226 (15)	0.1524 (2)	0.0451 (6)
H4	1.1046	0.2249	0.1179	0.054*
C5	0.9401 (2)	0.15666 (16)	0.0673 (2)	0.0475 (6)
H5	0.9300	0.1649	−0.0248	0.057*
C6	0.8519 (2)	0.11001 (15)	0.1155 (2)	0.0403 (5)
H6	0.7808	0.0868	0.0565	0.048*
C7	0.86557 (18)	0.09631 (12)	0.25101 (19)	0.0289 (4)
C8	0.79327 (17)	0.03925 (11)	0.42633 (17)	0.0231 (4)
C9	0.64594 (16)	−0.12910 (12)	0.37671 (17)	0.0245 (4)
C10	0.53941 (17)	−0.17643 (11)	0.29440 (17)	0.0230 (4)
C11	0.40700 (17)	−0.15997 (12)	0.28901 (18)	0.0264 (4)
H11	0.3817	−0.1175	0.3391	0.032*
C12	0.31276 (19)	−0.20563 (13)	0.2106 (2)	0.0339 (5)
H12	0.2226	−0.1951	0.2083	0.041*
C13	0.3494 (2)	−0.26669 (13)	0.1356 (2)	0.0357 (5)
H13	0.2843	−0.2962	0.0787	0.043*
C14	0.4802 (2)	−0.28490 (12)	0.14304 (19)	0.0332 (5)
H14	0.5049	−0.3278	0.0934	0.040*
C15	0.57487 (19)	−0.24034 (12)	0.22322 (18)	0.0290 (4)
H15	0.6647	−0.2534	0.2298	0.035*
C16	0.5145 (2)	−0.09973 (14)	−0.0642 (2)	0.0382 (5)
H16A	0.4905	−0.0777	0.0155	0.057*
H16C	0.5032	−0.1575	−0.0649	0.057*
H16B	0.4582	−0.0768	−0.1418	0.057*
C17	0.7469 (2)	−0.09952 (17)	0.0508 (2)	0.0474 (6)
H17A	0.8339	−0.0827	0.0381	0.071*
H17B	0.7472	−0.1568	0.0666	0.071*
H17C	0.7246	−0.0716	0.1265	0.071*
C18	0.6865 (2)	−0.05175 (12)	−0.17353 (19)	0.0314 (4)

H18	0.7771	−0.0417	−0.1684	0.038*
N1	0.98526 (15)	0.11775 (9)	0.46798 (15)	0.0263 (4)
H1	1.0539	0.1380	0.5203	0.032*
N2	0.77741 (15)	0.04876 (10)	0.29977 (15)	0.0277 (4)
N3	0.70812 (15)	−0.00590 (10)	0.47938 (15)	0.0306 (4)
H3N	0.7111	−0.0052	0.5648	0.037*
N4	0.61647 (15)	−0.05320 (10)	0.39948 (15)	0.0264 (4)
H4N	0.5395	−0.0336	0.3639	0.032*
N5	0.65059 (16)	−0.08079 (10)	−0.06642 (15)	0.0312 (4)
O1	0.75344 (13)	−0.15906 (9)	0.42053 (14)	0.0373 (4)
O2	0.61339 (14)	−0.03660 (9)	−0.27891 (13)	0.0342 (3)
S1	0.91904 (5)	0.06336 (3)	0.68523 (5)	0.03039 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0196 (8)	0.0200 (9)	0.0310 (10)	0.0026 (7)	0.0027 (7)	−0.0020 (7)
C2	0.0236 (9)	0.0272 (10)	0.0317 (10)	0.0012 (8)	0.0033 (8)	0.0048 (8)
C3	0.0263 (10)	0.0356 (12)	0.0422 (12)	−0.0059 (9)	0.0017 (9)	0.0084 (9)
C4	0.0325 (11)	0.0529 (15)	0.0495 (13)	−0.0053 (10)	0.0066 (10)	0.0223 (11)
C5	0.0351 (12)	0.0684 (18)	0.0373 (12)	−0.0015 (12)	0.0028 (10)	0.0218 (12)
C6	0.0291 (11)	0.0580 (15)	0.0308 (11)	−0.0051 (10)	−0.0018 (9)	0.0078 (10)
C7	0.0219 (9)	0.0338 (11)	0.0306 (10)	−0.0001 (8)	0.0037 (8)	0.0043 (8)
C8	0.0192 (8)	0.0234 (10)	0.0256 (9)	0.0012 (7)	0.0016 (7)	−0.0026 (7)
C9	0.0180 (8)	0.0364 (11)	0.0193 (9)	−0.0036 (8)	0.0043 (7)	0.0022 (8)
C10	0.0222 (8)	0.0265 (10)	0.0197 (8)	−0.0018 (7)	0.0020 (7)	0.0027 (7)
C11	0.0229 (9)	0.0269 (10)	0.0290 (10)	−0.0018 (8)	0.0040 (8)	−0.0027 (8)
C12	0.0233 (9)	0.0340 (12)	0.0411 (11)	−0.0028 (8)	−0.0020 (8)	−0.0018 (9)
C13	0.0369 (11)	0.0318 (12)	0.0340 (11)	−0.0097 (9)	−0.0041 (9)	−0.0025 (9)
C14	0.0454 (12)	0.0270 (11)	0.0274 (10)	0.0004 (9)	0.0072 (9)	−0.0033 (8)
C15	0.0275 (9)	0.0335 (11)	0.0262 (9)	0.0027 (8)	0.0060 (8)	0.0025 (8)
C16	0.0374 (11)	0.0440 (13)	0.0349 (11)	−0.0037 (10)	0.0114 (9)	0.0034 (10)
C17	0.0407 (13)	0.0691 (17)	0.0308 (11)	0.0010 (12)	0.0030 (10)	0.0126 (11)
C18	0.0308 (10)	0.0328 (11)	0.0312 (10)	−0.0021 (9)	0.0075 (8)	0.0017 (8)
N1	0.0198 (7)	0.0260 (9)	0.0312 (9)	−0.0047 (6)	0.0006 (6)	−0.0010 (7)
N2	0.0214 (8)	0.0350 (10)	0.0260 (8)	−0.0024 (7)	0.0025 (6)	−0.0003 (7)
N3	0.0284 (8)	0.0413 (10)	0.0213 (8)	−0.0152 (7)	0.0031 (7)	−0.0067 (7)
N4	0.0199 (7)	0.0328 (9)	0.0246 (8)	−0.0067 (7)	−0.0008 (6)	−0.0023 (7)
N5	0.0300 (9)	0.0392 (10)	0.0241 (8)	0.0000 (7)	0.0048 (7)	0.0041 (7)
O1	0.0192 (7)	0.0457 (9)	0.0432 (8)	0.0007 (6)	−0.0041 (6)	−0.0027 (7)
O2	0.0356 (8)	0.0374 (8)	0.0286 (7)	−0.0017 (6)	0.0033 (6)	0.0069 (6)
S1	0.0267 (2)	0.0378 (3)	0.0253 (3)	−0.0027 (2)	0.00135 (19)	−0.0026 (2)

Geometric parameters (Å, º)

C1—N1	1.343 (2)	C11—H11	0.9500
C1—C8	1.483 (2)	C12—C13	1.386 (3)
C1—S1	1.6661 (19)	C12—H12	0.9500
C2—N1	1.390 (2)	C13—C14	1.383 (3)
C2—C3	1.396 (3)	C13—H13	0.9500

C2—C7	1.401 (3)	C14—C15	1.384 (3)
C3—C4	1.374 (3)	C14—H14	0.9500
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.401 (3)	C16—N5	1.456 (3)
C4—H4	0.9500	C16—H16A	0.9800
C5—C6	1.372 (3)	C16—H16C	0.9800
C5—H5	0.9500	C16—H16B	0.9800
C6—C7	1.404 (3)	C17—N5	1.456 (3)
C6—H6	0.9500	C17—H17A	0.9800
C7—N2	1.384 (2)	C17—H17B	0.9800
C8—N2	1.300 (2)	C17—H17C	0.9800
C8—N3	1.360 (2)	C18—O2	1.234 (2)
C9—O1	1.233 (2)	C18—N5	1.330 (3)
C9—N4	1.347 (3)	C18—H18	0.9500
C9—C10	1.495 (2)	N1—H1	0.8800
C10—C15	1.394 (3)	N3—N4	1.390 (2)
C10—C11	1.396 (2)	N3—H3N	0.8800
C11—C12	1.383 (3)	N4—H4N	0.8800

N1—C1—C8	113.62 (16)	C14—C13—H13	119.8
N1—C1—S1	122.24 (13)	C12—C13—H13	119.8
C8—C1—S1	124.13 (14)	C13—C14—C15	119.64 (19)
N1—C2—C3	120.68 (17)	C13—C14—H14	120.2
N1—C2—C7	117.32 (17)	C15—C14—H14	120.2
C3—C2—C7	122.00 (18)	C14—C15—C10	120.48 (18)
C4—C3—C2	118.92 (19)	C14—C15—H15	119.8
C4—C3—H3	120.5	C10—C15—H15	119.8
C2—C3—H3	120.5	N5—C16—H16A	109.5
C3—C4—C5	120.2 (2)	N5—C16—H16C	109.5
C3—C4—H4	119.9	H16A—C16—H16C	109.5
C5—C4—H4	119.9	N5—C16—H16B	109.5
C6—C5—C4	120.7 (2)	H16A—C16—H16B	109.5
C6—C5—H5	119.7	H16C—C16—H16B	109.5
C4—C5—H5	119.7	N5—C17—H17A	109.5
C5—C6—C7	120.7 (2)	N5—C17—H17B	109.5
C5—C6—H6	119.7	H17A—C17—H17B	109.5
C7—C6—H6	119.7	N5—C17—H17C	109.5
N2—C7—C2	121.65 (17)	H17A—C17—H17C	109.5
N2—C7—C6	120.77 (17)	H17B—C17—H17C	109.5
C2—C7—C6	117.58 (18)	O2—C18—N5	126.27 (19)
N2—C8—N3	120.54 (16)	O2—C18—H18	116.9
N2—C8—C1	124.59 (17)	N5—C18—H18	116.9
N3—C8—C1	114.87 (16)	C1—N1—C2	124.49 (15)
O1—C9—N4	123.04 (17)	C1—N1—H1	117.8
O1—C9—C10	120.98 (18)	C2—N1—H1	117.8
N4—C9—C10	115.98 (15)	C8—N2—C7	118.23 (16)
C15—C10—C11	119.35 (17)	C8—N3—N4	120.40 (15)
C15—C10—C9	118.19 (16)	C8—N3—H3N	119.8
C11—C10—C9	122.44 (17)	N4—N3—H3N	119.8

C12—C11—C10	119.86 (18)	C9—N4—N3	119.73 (15)
C12—C11—H11	120.1	C9—N4—H4N	120.1
C10—C11—H11	120.1	N3—N4—H4N	120.1
C11—C12—C13	120.21 (19)	C18—N5—C17	121.23 (17)
C11—C12—H12	119.9	C18—N5—C16	121.41 (17)
C13—C12—H12	119.9	C17—N5—C16	117.25 (17)
C14—C13—C12	120.34 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O2^{i}	0.88	2.14	2.906 (2)	146
N4—H4*N*···O2^{ii}	0.88	2.04	2.906 (2)	166
N1—H1···O1^{iii}	0.88	2.01	2.8331 (19)	154

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, −*y*, −*z*; (iii) −*x*+2, −*y*, −*z*+1.