Crystal structure of 2-[4-(methylsulfanyl)quinazolin-2-yl]-1-phenylethanol

Gamal A. El-Hiti,[a]* Keith Smith,[b] Amany S. Hegazy,[b] Mansour D. Ajarim[c] and Benson M. Kariuki[b]*

[a]Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, PO Box 10219, Riyadh 11433, Saudi Arabia, [b]School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, Wales, and [c]Criminal Evidence, Ministry of Interior, Riyadh 11632, PO Box 86985, Saudi Arabia. *Correspondence e-mail: gelhiti@ksu.edu.sa, kariukib@cardiff.ac.uk

In the molecule of the title compound, $C_{17}H_{16}N_2OS$, the almost planar methylsulfanylquinazoline group [the methyl C atom deviates by 0.032 (2) Å from the plane through the ring system] forms an interplanar angle of 76.26 (4)° with the plane of the phenyl group. An intramolecular O—H···N hydrogen bond is present between the quinazoline and hydroxy groups. In the crystal, molecules are stacked along the b-axis direction.

Keywords: crystal structure; 4-(methylsulfanyl)quinazoline derivative; hydrogen bonding.

CCDC reference: 1022918

1. Related literature

For the synthesis of 4-(methylsulfanyl)quinazoline derivatives, see: Smith *et al.* (2005a,b); Leonard & Curtin (1946); Meerwein *et al.* (1956). For the crystal structures of related compounds, see: Alshammari *et al.* (2014a,b).
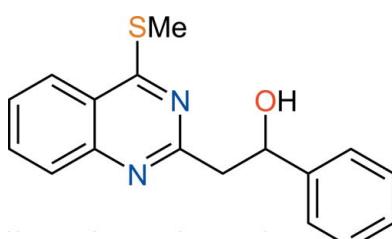
2. Experimental

2.1. Crystal data

$C_{17}H_{16}N_2OS$
$M_r = 296.38$

Monoclinic, $P2_1/n$
$a = 15.6142$ (3) Å
$b = 5.6142$ (1) Å
$c = 17.2355$ (3) Å
$\beta = 101.138$ (2)°
$V = 1482.43$ (5) Å^3
$Z = 4$

Cu $K\alpha$ radiation
$\mu = 1.93$ mm^{-1}
$T = 293$ K
$0.32 \times 0.19 \times 0.14$ mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014) $T_{min} = 0.867$, $T_{max} = 1.000$

9606 measured reflections
2938 independent reflections
2688 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.094$
$S = 1.06$
2938 reflections

192 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	2.12	2.7531 (15)	134

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006), *ORTEP-3 for Windows* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
Alshammari, M. B., Smith, K., Hegazy, A. S., Kariuki, B. M. & El-Hiti, G. A. (2014a). *Acta Cryst.* E**70**, o919–o920.
Alshammari, M. B., Smith, K., Hegazy, A. S., Kariuki, B. M. & El-Hiti, G. A. (2014b). *Acta Cryst.* E**70**, o953.
Cambridge Soft (2001). *CHEMDRAW Ultra*. Cambridge Soft Corporation, Cambridge, Massachusetts, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Leonard, N. J. & Curtin, D. Y. (1946). *J. Org. Chem.* **11**, 349–352.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Meerwein, H., Laasch, P., Mersch, R. & Nentwig, J. (1956). *Chem. Ber.* **89**, 224–238.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, K., El-Hiti, G. A. & Hegazy, A. S. (2005a). *J. Sulfur Chem.* **26**, 121–131.
Smith, K., El-Hiti, G. A. & Hegazy, A. S. (2005b). *Synthesis*, pp. 2951–2961.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Crystal structure of 2-[4-(methylsulfanyl)quinazolin-2-yl]-1-phenylethanol

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Mansour D. Ajarim and Benson M. Kariuki

S1. Comment

In $C_{17}H_{16}N_2OS$ (Fig. 1), the methylsulfanylquinazoline group is planar and is oriented at an angle of 76.26 (4)° with the phenyl group. The hydroxyl group forms an intramolecular hydrogen bond to one of the quinazoline ring nitrogen atoms (Table 1) whereas the second N atom is not involved in hydrogen bonding. In the crystal structure, molecules are stacked along the *b*-axis direction (Fig. 2).

S2. Experimental

Synthesis and crystallization: 2-(2-hydroxy-2-phenylethyl)-4-(methylsulfanyl)quinazoline was obtained in 83% yield from lithiation of 2-methyl-4-(methylsulfanyl)quinazoline with *n*-butyllithium at 78°C in anhydrous THF under nitrogen followed by reaction with benzaldehyde (Smith *et al.*, 2005*b*). Crystallization from a mixture of ethyl acetate and diethyl ether (1:3 by volume) gave the title compound as colorless crystals. The NMR and low and high resolution mass spectra for the title compound were consistent with those reported (Smith *et al.*, 2005*b*).

S3. Refinement

H atoms were positioned geometrically and refined using a riding model with $U_{iso}(H)$ constrained to be 1.2 times $U_{eq}(C)$ except for the methyl group where it was 1.5 times with free rotation about the C—C bond. For the OH group, $U_{iso}(H)$ 1.5 times $U_{eq}(O)$ was used with free rotation about the C—O bond.
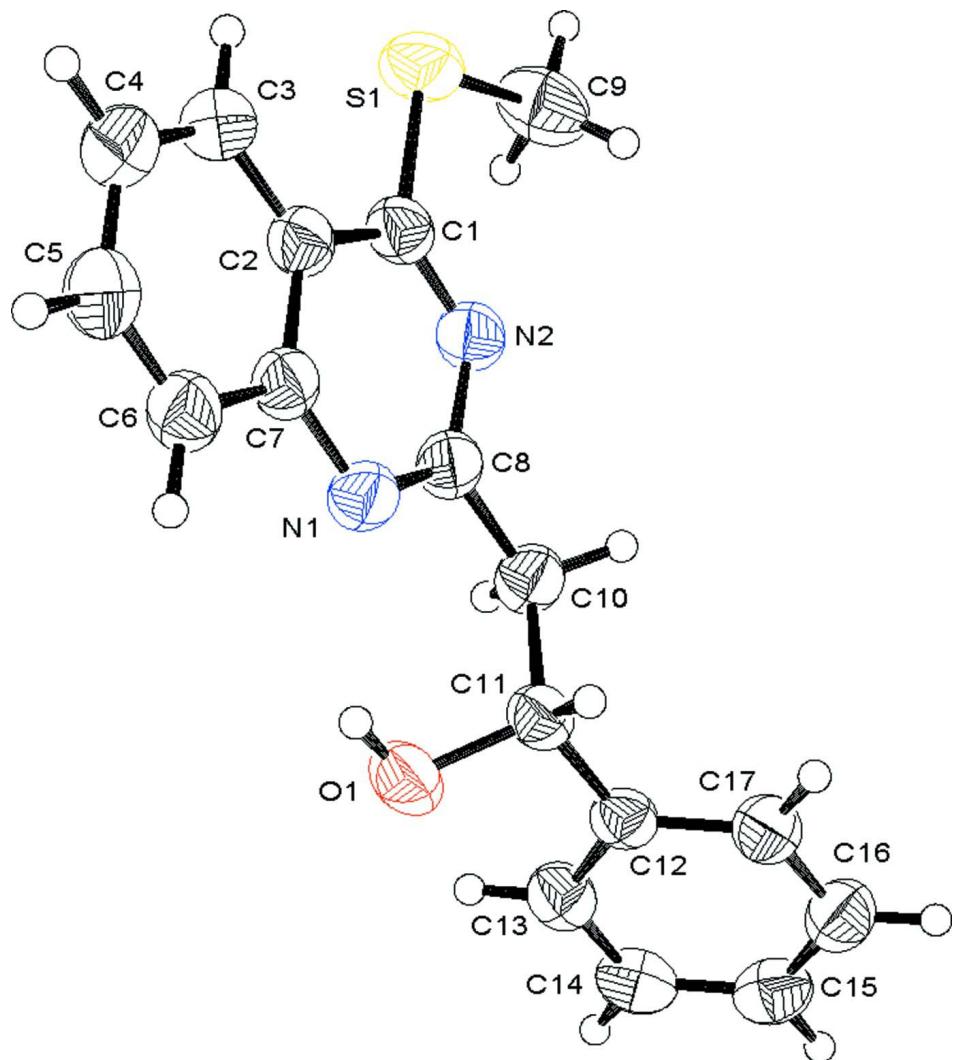
Figure 1

A molecule of C$_{17}$H$_{16}$N$_{2}$OS with atom labels and 50% probability displacement ellipsoids for nonhydrogen atoms.
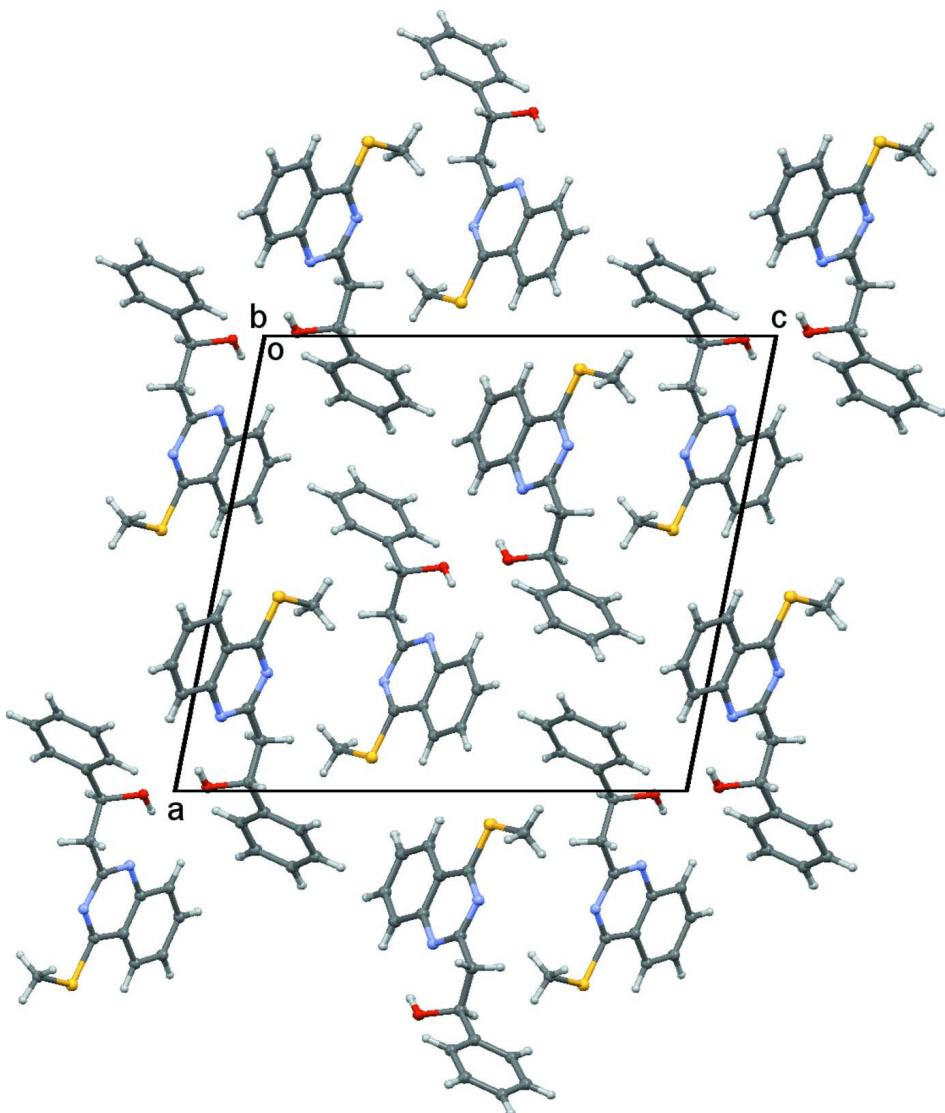
Figure 2

Crystal structure packing viewed down the *b* axis.

2-[4-(Methylsulfanyl)quinazolin-2-yl]-1-phenylethanol

Crystal data

$C_{17}H_{16}N_2OS$	$F(000) = 624$
$M_r = 296.38$	$D_x = 1.328$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$a = 15.6142$ (3) Å	Cell parameters from 5816 reflections
$b = 5.6142$ (1) Å	$\theta = 3.5–73.7°$
$c = 17.2355$ (3) Å	$\mu = 1.93$ mm^{-1}
$\beta = 101.138$ (2)°	$T = 293$ K
$V = 1482.43$ (5) Å^3	Block, colourless
$Z = 4$	$0.32 \times 0.19 \times 0.14$ mm

Data collection

Agilent SuperNova (Dual, Cu at 0, Atlas) diffractometer	2938 independent reflections
ω scans	2688 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.014$
(*CrysAlis PRO*; Agilent, 2014)	$\theta_{max} = 74.0°$, $\theta_{min} = 3.5°$
$T_{min} = 0.867$, $T_{max} = 1.000$	$h = -19\rightarrow19$
9606 measured reflections	$k = -6\rightarrow6$
	$l = -11\rightarrow21$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.094$	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.2343P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2938 reflections	$(\Delta/\sigma)_{max} = 0.001$
192 parameters	$\Delta\rho_{max} = 0.16$ e Å⁻³
0 restraints	$\Delta\rho_{min} = -0.28$ e Å⁻³

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.36.28 (release 01-02-2013 CrysAlis171 .NET) (compiled Feb 1 2013,16:14:44) Empirical absorption correction in SCALE3 ABSPACK.

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

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.67633 (8)	0.0261 (2)	0.10297 (7)	0.0458 (3)
C2	0.68632 (8)	−0.1600 (2)	0.04845 (7)	0.0450 (3)
C3	0.62090 (9)	−0.3225 (3)	0.01440 (8)	0.0553 (3)
H3	0.5652	−0.3110	0.0258	0.066*
C4	0.63923 (10)	−0.4967 (3)	−0.03523 (9)	0.0601 (4)
H4	0.5961	−0.6048	−0.0569	0.072*
C5	0.72249 (10)	−0.5136 (3)	−0.05366 (8)	0.0569 (3)
H5	0.7342	−0.6336	−0.0873	0.068*
C6	0.78660 (9)	−0.3561 (3)	−0.02273 (8)	0.0523 (3)
H6	0.8413	−0.3669	−0.0363	0.063*
C7	0.76994 (8)	−0.1775 (2)	0.02961 (7)	0.0444 (3)
C8	0.81859 (8)	0.1354 (2)	0.11131 (7)	0.0445 (3)
C9	0.59215 (12)	0.3019 (3)	0.19721 (11)	0.0734 (5)
H9A	0.6392	0.2626	0.2400	0.110*
H9B	0.5401	0.3312	0.2176	0.110*
H9C	0.6070	0.4421	0.1708	0.110*
C10	0.88942 (8)	0.3057 (2)	0.14745 (8)	0.0479 (3)
H10A	0.8870	0.3261	0.2029	0.058*
H10B	0.8775	0.4595	0.1220	0.058*
C11	0.98167 (8)	0.2298 (2)	0.14133 (7)	0.0429 (3)

H11	0.9941	0.0764	0.1683	0.051*
C12	1.04767 (8)	0.4094 (2)	0.18105 (7)	0.0414 (3)
C13	1.06970 (9)	0.6065 (2)	0.14024 (8)	0.0483 (3)
H13	1.0441	0.6280	0.0873	0.058*
C14	1.12977 (9)	0.7713 (2)	0.17802 (9)	0.0538 (3)
H14	1.1449	0.9011	0.1499	0.065*
C15	1.16716 (9)	0.7448 (2)	0.25659 (9)	0.0544 (3)
H15	1.2075	0.8556	0.2815	0.065*
C16	1.14426 (9)	0.5519 (3)	0.29825 (8)	0.0541 (3)
H16	1.1681	0.5350	0.3517	0.065*
C17	1.08585 (8)	0.3841 (2)	0.26030 (8)	0.0477 (3)
H17	1.0720	0.2526	0.2883	0.057*
N1	0.83603 (7)	−0.0253 (2)	0.06177 (6)	0.0475 (3)
N2	0.74020 (7)	0.1686 (2)	0.13399 (6)	0.0475 (3)
O1	0.99198 (7)	0.2036 (2)	0.06187 (6)	0.0606 (3)
H1	0.9563	0.1070	0.0393	0.091*
S1	0.57374 (2)	0.05921 (7)	0.12865 (2)	0.06185 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0408 (6)	0.0551 (7)	0.0426 (6)	0.0077 (5)	0.0110 (5)	0.0052 (5)
C2	0.0415 (6)	0.0531 (7)	0.0399 (6)	0.0044 (5)	0.0071 (5)	0.0040 (5)
C3	0.0463 (7)	0.0683 (9)	0.0517 (7)	−0.0049 (6)	0.0101 (6)	−0.0006 (6)
C4	0.0609 (8)	0.0649 (8)	0.0529 (8)	−0.0122 (7)	0.0070 (6)	−0.0060 (6)
C5	0.0654 (9)	0.0565 (8)	0.0487 (7)	0.0011 (7)	0.0105 (6)	−0.0077 (6)
C6	0.0504 (7)	0.0597 (8)	0.0474 (7)	0.0065 (6)	0.0107 (5)	−0.0063 (6)
C7	0.0414 (6)	0.0508 (7)	0.0405 (6)	0.0059 (5)	0.0065 (5)	0.0007 (5)
C8	0.0398 (6)	0.0514 (7)	0.0418 (6)	0.0076 (5)	0.0072 (5)	−0.0004 (5)
C9	0.0777 (11)	0.0691 (10)	0.0845 (11)	0.0061 (8)	0.0435 (9)	−0.0105 (8)
C10	0.0431 (6)	0.0510 (7)	0.0492 (7)	0.0052 (5)	0.0078 (5)	−0.0062 (5)
C11	0.0433 (6)	0.0459 (6)	0.0410 (6)	0.0028 (5)	0.0123 (5)	0.0009 (5)
C12	0.0376 (6)	0.0434 (6)	0.0451 (6)	0.0061 (5)	0.0129 (5)	−0.0006 (5)
C13	0.0508 (7)	0.0475 (7)	0.0482 (6)	0.0057 (5)	0.0139 (5)	0.0037 (5)
C14	0.0538 (7)	0.0434 (6)	0.0691 (8)	0.0010 (6)	0.0244 (6)	0.0017 (6)
C15	0.0451 (7)	0.0510 (7)	0.0677 (8)	−0.0014 (6)	0.0123 (6)	−0.0110 (6)
C16	0.0479 (7)	0.0604 (8)	0.0517 (7)	0.0045 (6)	0.0038 (6)	−0.0037 (6)
C17	0.0468 (6)	0.0489 (7)	0.0477 (6)	0.0037 (5)	0.0098 (5)	0.0039 (5)
N1	0.0390 (5)	0.0554 (6)	0.0482 (5)	0.0046 (4)	0.0084 (4)	−0.0067 (5)
N2	0.0429 (5)	0.0546 (6)	0.0462 (5)	0.0072 (5)	0.0116 (4)	−0.0021 (5)
O1	0.0655 (6)	0.0740 (7)	0.0477 (5)	−0.0177 (5)	0.0247 (4)	−0.0148 (5)
S1	0.0459 (2)	0.0755 (3)	0.0698 (2)	0.00317 (16)	0.02499 (17)	−0.00692 (17)

Geometric parameters (Å, º)

C1—N2	1.3092 (17)	C9—H9C	0.9600
C1—C2	1.4340 (18)	C10—C11	1.5252 (16)
C1—S1	1.7521 (13)	C10—H10A	0.9700

C2—C7	1.4083 (17)	C10—H10B	0.9700
C2—C3	1.4100 (19)	C11—O1	1.4173 (14)
C3—C4	1.365 (2)	C11—C12	1.5082 (17)
C3—H3	0.9300	C11—H11	0.9800
C4—C5	1.400 (2)	C12—C17	1.3880 (17)
C4—H4	0.9300	C12—C13	1.3894 (18)
C5—C6	1.365 (2)	C13—C14	1.3873 (19)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.4065 (18)	C14—C15	1.375 (2)
C6—H6	0.9300	C14—H14	0.9300
C7—N1	1.3713 (17)	C15—C16	1.384 (2)
C8—N1	1.3067 (16)	C15—H15	0.9300
C8—N2	1.3678 (16)	C16—C17	1.3843 (19)
C8—C10	1.5032 (18)	C16—H16	0.9300
C9—S1	1.7902 (17)	C17—H17	0.9300
C9—H9A	0.9600	O1—H1	0.8200
C9—H9B	0.9600		
N2—C1—C2	122.82 (11)	C11—C10—H10A	108.5
N2—C1—S1	119.58 (10)	C8—C10—H10B	108.5
C2—C1—S1	117.60 (10)	C11—C10—H10B	108.5
C7—C2—C3	119.24 (12)	H10A—C10—H10B	107.5
C7—C2—C1	115.14 (11)	O1—C11—C12	108.24 (10)
C3—C2—C1	125.62 (12)	O1—C11—C10	112.40 (10)
C4—C3—C2	120.08 (13)	C12—C11—C10	110.70 (10)
C4—C3—H3	120.0	O1—C11—H11	108.5
C2—C3—H3	120.0	C12—C11—H11	108.5
C3—C4—C5	120.48 (13)	C10—C11—H11	108.5
C3—C4—H4	119.8	C17—C12—C13	118.57 (12)
C5—C4—H4	119.8	C17—C12—C11	120.28 (11)
C6—C5—C4	120.70 (13)	C13—C12—C11	121.12 (11)
C6—C5—H5	119.7	C14—C13—C12	120.31 (12)
C4—C5—H5	119.7	C14—C13—H13	119.8
C5—C6—C7	120.01 (13)	C12—C13—H13	119.8
C5—C6—H6	120.0	C15—C14—C13	120.65 (13)
C7—C6—H6	120.0	C15—C14—H14	119.7
N1—C7—C6	119.03 (12)	C13—C14—H14	119.7
N1—C7—C2	121.49 (11)	C14—C15—C16	119.52 (13)
C6—C7—C2	119.47 (12)	C14—C15—H15	120.2
N1—C8—N2	126.28 (12)	C16—C15—H15	120.2
N1—C8—C10	118.73 (11)	C15—C16—C17	119.99 (13)
N2—C8—C10	114.98 (11)	C15—C16—H16	120.0
S1—C9—H9A	109.5	C17—C16—H16	120.0
S1—C9—H9B	109.5	C16—C17—C12	120.92 (12)
H9A—C9—H9B	109.5	C16—C17—H17	119.5
S1—C9—H9C	109.5	C12—C17—H17	119.5
H9A—C9—H9C	109.5	C8—N1—C7	117.30 (11)
H9B—C9—H9C	109.5	C1—N2—C8	116.95 (11)

| C8—C10—C11 | 115.00 (10) | C11—O1—H1 | 109.5 |
| C8—C10—H10A | 108.5 | C1—S1—C9 | 102.08 (7) |

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	2.12	2.7531 (15)	134