Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-{[4-(4-Pyridyl)pyrimidin-2-yl]-sulfanylmethyl}benzoic acid

Hai-Bin Zhu,* Hai Wang and Jun-Feng Ji

School of Chemistry and Chemical Engineering, Southeast University, Nanjing, People's Republic of China
Correspondence e-mail: zhuhaibin@seu.edu.cn

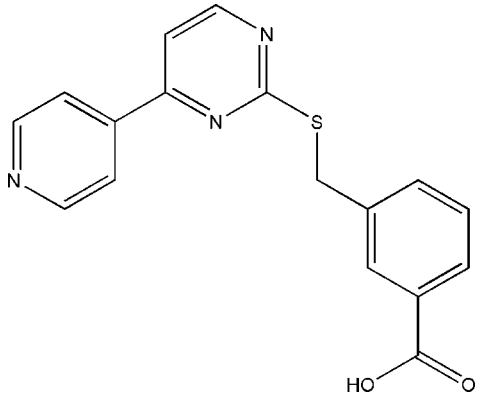
Received 28 October 2008; accepted 8 November 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.071; wR factor = 0.170; data-to-parameter ratio = 13.1.

The title compound, $C_{17}H_{13}N_3O_2S$, was prepared by reaction of 4-(4-pyridyl)pyrimidine-2-thiol with 3-(bromomethyl)-benzoic acid under basic conditions. Each pair of molecules is mutually linked *via* O—H···N hydrogen bonds, forming a dimer. The packing of the dimers is stablized by C—H···π interactions involving the methylene unit of the $-CH_2S-$ linkage and benzene rings.

Related literature

For monodentate and chelating ligands, see: Raper (1996). For the structures of binuclear and polynuclear complexes with bridging heterocyclic thionate ligands, see: Raper (1997). For O—H···N interactions, see: Han *et al.* (2008). For C—H···π interactions, see: Choi *et al.* (2008).



Experimental

Crystal data

$C_{17}H_{13}N_3O_2S$ M_r = 323.36
Triclinic, $P\overline{1}$
a = 4.4130 (9) Å
b = 10.458 (2) Å
c = 16.432 (3) Å
α = 87.79 (3)°
β = 89.90 (3)°
γ = 80.48 (3)°
V = 747.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.934, T_{max} = 0.977
3108 measured reflections
2724 independent reflections
1685 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.170
S = 1.04
2724 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 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
O2—H2A···N1^{i}	0.82	1.84	2.659 (5)	174
C10—H10B···$Cg3^{ii}$	0.97	2.56	3.398 (5)	145

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$. $Cg3$ is the centroid of the phenyl ring.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors are grateful for support from the China Postdoctoral Research Fund (20070411010) and the National Natural Science of Foundation of China (20801011).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2127).

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supporting information

Acta Cryst. (2008). E**64**, o2347 [doi:10.1107/S1600536808036799]

3-{[4-(4-Pyridyl)pyrimidin-2-yl]sulfanylmethyl}benzoic acid

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S1. Comment

Heterocyclic thionates (Raper, 1996, 1997) have been extensively studied due to not only their versatile coordination modes but also their close biological relativity. Herein, we report the crystal structure of a new heterocyclic thionate derivative, 3-((4-(4-pyridyl)pyrimidin-2-ylthio)methyl)benzoic acid.

In the molecular structure of the title compound (Fig.1), the pyrimidinyl and pyridinyl rings are not coplanar with the dihedral angle of 11.17 (3)*o*. Each pair of compound molecules is linked each other *via* O—H···N hydrogen bonds (Han *et.al*., 2008) to form a dimer (Fig. 2 and Table 1), and the packing of the dimers is stablized by C—H···π interactions (Choi *et al.*, 2008) between a methylene H atom of the $–CH_2S–$ linkage and the phenyl ring, with a C10—H10B···*Cg*3^{ii} separation of 2.56 Å (Fig. 3 and Table 1; *Cg*3 is the centroid of the C11/C12/C13/C14/C15/C16 phenyl ring, symmetry code as in Fig. 3).

S2. Experimental

The mixture of 4-(4-pyridyl)pyrimidine-2-thiol (0.1890 g,1.0 mmol), 3-(bromomethyl)benzoic acid (0.2150 g,1.0 mmol), NaOH (0.0400 g,1.0 mmol) in 30 ml of H_2O was refluxed for 10 h. After cooled to room temperature, the product was filtered and dried in vaccum. The single crystals suitable for X-ray diffraction were obtained by slow evaporation of the title compound in DMF solution.

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 Å for aromatic H atoms, 0.97 Å for methylene H atoms and O—H = 0.82 Å, respectively, and with with *U*iso(H) = 1.2*U*eq (C) for aromatic and methylene, *U*iso(H) =1.5*U*eq (O).

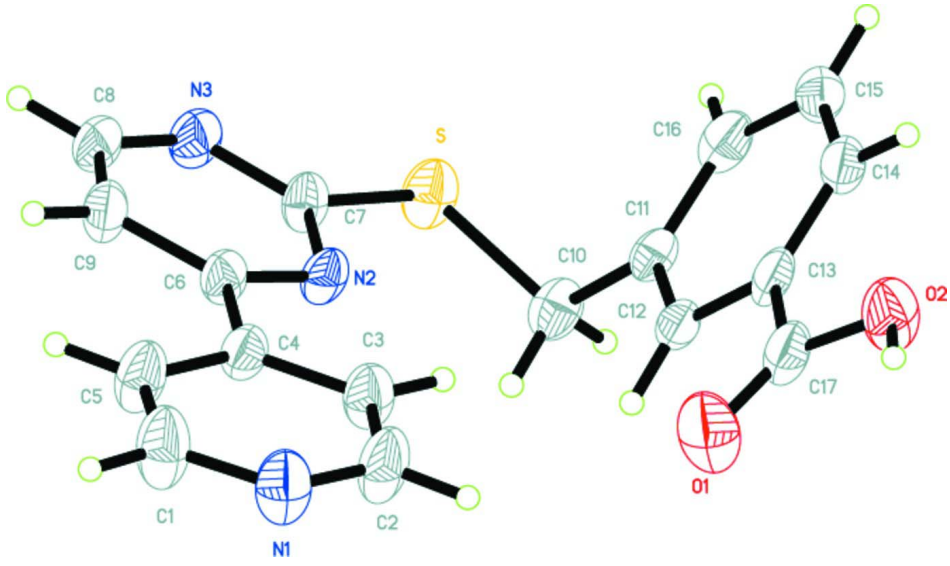

Figure 1

The molecular structure of the title compound with 30% displacement ellipsoids.

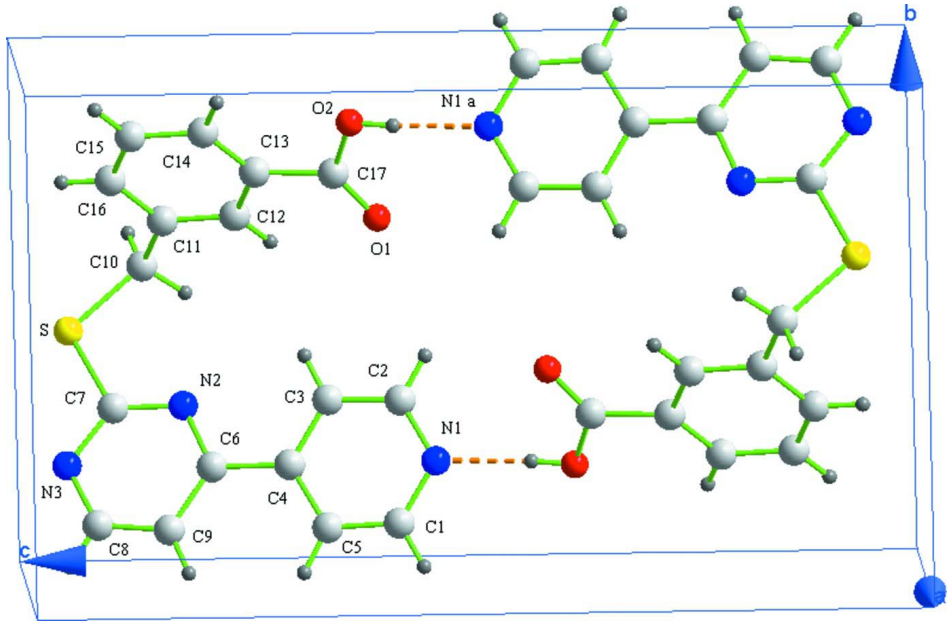

Figure 2

The hydrogen bonded dimer with O—H···N interactions (dotted lines) [symmetry code: -*x* + 2, -*y* + 1, -*z* + 1].

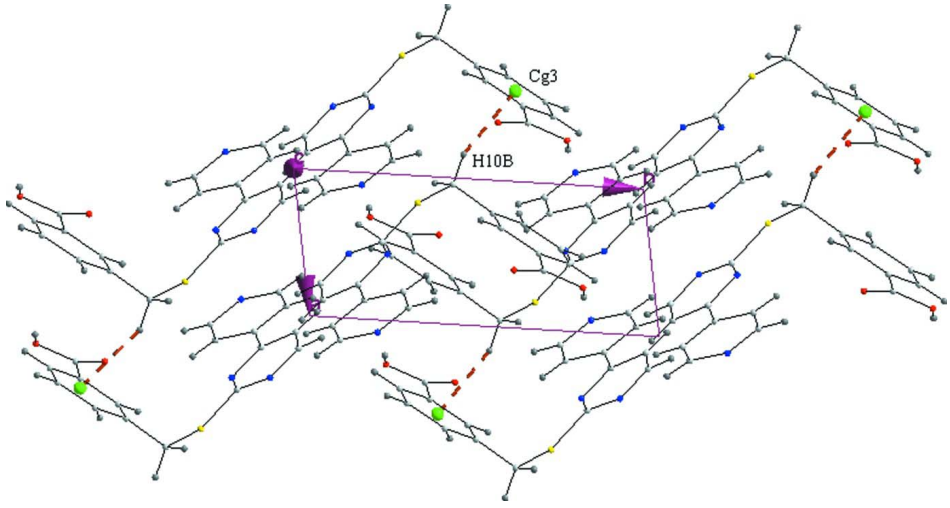

Figure 3

A section of the hydrogen bonded dimers viewed down the *c* axis. C—H···π interactions are shown as dotted lines. *Cg*3 denotes the phenyl ring centroid [symmetry code: *x* - 1, *y*, *z*].

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Crystal data

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.4130\ (9)$ Å
$b = 10.458\ (2)$ Å
$c = 16.432\ (3)$ Å
$\alpha = 87.79\ (3)°$
$\beta = 89.90\ (3)°$
$\gamma = 80.48\ (3)°$
$V = 747.4\ (3)$ Å^3
$Z = 2$
$F(000) = 336$
$D_x = 1.437$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 0.23$ mm^{-1}
$T = 298$ K
Prism, colorless
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.934$, $T_{max} = 0.977$
3108 measured reflections
2724 independent reflections
1685 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.2°$
$h = 0\rightarrow5$
$k = -12\rightarrow12$
$l = -19\rightarrow19$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.170$
$S = 1.04$
2724 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

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.

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 $F^2 > \sigma(F^2)$ 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}
S	0.1838 (3)	0.34695 (12)	0.92472 (7)	0.0633 (4)
O1	0.6060 (10)	0.6024 (3)	0.5857 (2)	0.0916 (13)
O2	0.6987 (9)	0.7953 (3)	0.61531 (19)	0.0795 (11)
H2A	0.7614	0.7948	0.5683	0.119*
N1	1.0847 (10)	0.1910 (4)	0.5353 (2)	0.0737 (12)
N2	0.5470 (7)	0.2426 (3)	0.80536 (18)	0.0457 (8)
N3	0.5608 (9)	0.1325 (4)	0.9366 (2)	0.0597 (10)
C1	1.1506 (13)	0.0803 (5)	0.5789 (3)	0.0832 (17)
H1B	1.2665	0.0098	0.5544	0.100*
C2	0.9037 (14)	0.2874 (5)	0.5702 (3)	0.0819 (17)
H2B	0.8459	0.3649	0.5403	0.098*
C3	0.7973 (11)	0.2779 (4)	0.6488 (3)	0.0635 (13)
H3B	0.6757	0.3490	0.6713	0.076*
C4	0.8710 (10)	0.1634 (4)	0.6942 (2)	0.0506 (10)
C5	1.0591 (12)	0.0636 (5)	0.6570 (3)	0.0693 (14)
H5A	1.1230	−0.0148	0.6854	0.083*
C6	0.7669 (10)	0.1494 (4)	0.7790 (2)	0.0470 (10)
C7	0.4682 (9)	0.2307 (4)	0.8824 (2)	0.0462 (10)
C8	0.7742 (11)	0.0419 (4)	0.9077 (2)	0.0577 (12)
H8A	0.8544	−0.0276	0.9425	0.069*
C9	0.8839 (10)	0.0436 (4)	0.8303 (3)	0.0558 (11)
H9A	1.0315	−0.0232	0.8123	0.067*
C10	0.0460 (9)	0.4535 (4)	0.8386 (3)	0.0566 (11)
H10A	0.0430	0.4012	0.7912	0.068*
H10B	−0.1644	0.4929	0.8493	0.068*
C11	0.2261 (8)	0.5595 (4)	0.8179 (2)	0.0449 (9)
C12	0.3212 (9)	0.5779 (4)	0.7389 (2)	0.0448 (9)
H12A	0.2784	0.5227	0.6991	0.054*
C13	0.4809 (10)	0.6787 (4)	0.7186 (2)	0.0516 (11)
C14	0.5392 (10)	0.7623 (4)	0.7761 (2)	0.0516 (10)
H14A	0.6428	0.8305	0.7619	0.062*
C15	0.4440 (10)	0.7455 (4)	0.8553 (3)	0.0544 (11)

H15A	0.4859	0.8015	0.8948	0.065*
C16	0.2852 (10)	0.6446 (4)	0.8758 (3)	0.0575 (12)
H16A	0.2182	0.6343	0.9290	0.069*
C17	0.5941 (11)	0.6869 (4)	0.6328 (3)	0.0579 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0761 (8)	0.0604 (8)	0.0493 (7)	−0.0024 (6)	0.0165 (5)	0.0109 (5)
O1	0.161 (4)	0.067 (2)	0.0460 (19)	−0.016 (2)	0.026 (2)	−0.0079 (18)
O2	0.125 (3)	0.060 (2)	0.054 (2)	−0.019 (2)	0.0138 (19)	0.0092 (16)
N1	0.104 (3)	0.062 (3)	0.051 (2)	0.000 (2)	0.025 (2)	0.003 (2)
N2	0.056 (2)	0.0419 (19)	0.0373 (18)	−0.0057 (16)	0.0016 (15)	0.0116 (14)
N3	0.087 (3)	0.049 (2)	0.040 (2)	−0.006 (2)	−0.0012 (18)	0.0092 (17)
C1	0.119 (5)	0.054 (3)	0.067 (3)	0.015 (3)	0.027 (3)	0.000 (3)
C2	0.136 (5)	0.057 (3)	0.042 (3)	0.012 (3)	0.011 (3)	0.013 (2)
C3	0.091 (4)	0.048 (3)	0.047 (3)	0.002 (2)	0.010 (2)	0.005 (2)
C4	0.063 (3)	0.041 (2)	0.045 (2)	−0.002 (2)	−0.0035 (19)	0.0036 (18)
C5	0.094 (4)	0.054 (3)	0.051 (3)	0.013 (3)	0.017 (2)	0.011 (2)
C6	0.062 (3)	0.039 (2)	0.039 (2)	−0.007 (2)	−0.0010 (18)	0.0058 (17)
C7	0.056 (2)	0.041 (2)	0.041 (2)	−0.0115 (19)	0.0064 (18)	0.0108 (18)
C8	0.085 (3)	0.046 (3)	0.040 (2)	−0.007 (2)	−0.010 (2)	0.013 (2)
C9	0.074 (3)	0.037 (2)	0.052 (3)	0.004 (2)	−0.002 (2)	0.0017 (19)
C10	0.040 (2)	0.060 (3)	0.065 (3)	0.001 (2)	0.007 (2)	0.009 (2)
C11	0.033 (2)	0.046 (2)	0.051 (2)	0.0036 (17)	−0.0046 (17)	0.0045 (19)
C12	0.049 (2)	0.041 (2)	0.040 (2)	0.0058 (18)	−0.0048 (17)	0.0020 (17)
C13	0.064 (3)	0.040 (2)	0.044 (2)	0.011 (2)	−0.0021 (19)	0.0105 (19)
C14	0.065 (3)	0.039 (2)	0.048 (2)	0.000 (2)	0.001 (2)	0.0047 (19)
C15	0.069 (3)	0.043 (2)	0.049 (3)	−0.002 (2)	−0.006 (2)	−0.0028 (19)
C16	0.064 (3)	0.060 (3)	0.040 (2)	0.016 (2)	0.008 (2)	0.002 (2)
C17	0.074 (3)	0.044 (3)	0.050 (3)	0.005 (2)	0.001 (2)	0.010 (2)

Geometric parameters (Å, º)

S—C7	1.759 (4)	C5—H5A	0.9300
S—C10	1.809 (4)	C6—C9	1.393 (5)
O1—C17	1.191 (5)	C8—C9	1.360 (6)
O2—C17	1.314 (5)	C8—H8A	0.9300
O2—H2A	0.8200	C9—H9A	0.9300
N1—C2	1.327 (6)	C10—C11	1.496 (6)
N1—C1	1.327 (6)	C10—H10A	0.9700
N2—C7	1.318 (5)	C10—H10B	0.9700
N2—C6	1.341 (5)	C11—C12	1.379 (5)
N3—C8	1.323 (5)	C11—C16	1.382 (6)
N3—C7	1.344 (5)	C12—C13	1.392 (6)
C1—C5	1.358 (6)	C12—H12A	0.9300
C1—H1B	0.9300	C13—C14	1.365 (6)
C2—C3	1.380 (6)	C13—C17	1.499 (6)

C2—H2B	0.9300	C14—C15	1.381 (5)
C3—C4	1.378 (5)	C14—H14A	0.9300
C3—H3B	0.9300	C15—C16	1.390 (6)
C4—C5	1.383 (6)	C15—H15A	0.9300
C4—C6	1.476 (5)	C16—H16A	0.9300

C7—S—C10	103.54 (19)	C8—C9—H9A	121.3
C17—O2—H2A	109.5	C6—C9—H9A	121.3
C2—N1—C1	116.5 (4)	C11—C10—S	116.1 (3)
C7—N2—C6	115.9 (3)	C11—C10—H10A	108.3
C8—N3—C7	113.1 (3)	S—C10—H10A	108.3
N1—C1—C5	124.1 (4)	C11—C10—H10B	108.3
N1—C1—H1B	118.0	S—C10—H10B	108.3
C5—C1—H1B	118.0	H10A—C10—H10B	107.4
N1—C2—C3	123.0 (4)	C12—C11—C16	118.9 (4)
N1—C2—H2B	118.5	C12—C11—C10	120.0 (4)
C3—C2—H2B	118.5	C16—C11—C10	121.0 (4)
C4—C3—C2	120.1 (4)	C11—C12—C13	120.4 (4)
C4—C3—H3B	119.9	C11—C12—H12A	119.8
C2—C3—H3B	119.9	C13—C12—H12A	119.8
C3—C4—C5	116.3 (4)	C14—C13—C12	120.4 (4)
C3—C4—C6	122.1 (4)	C14—C13—C17	122.4 (4)
C5—C4—C6	121.6 (4)	C12—C13—C17	117.1 (4)
C1—C5—C4	119.9 (4)	C13—C14—C15	119.8 (4)
C1—C5—H5A	120.1	C13—C14—H14A	120.1
C4—C5—H5A	120.1	C15—C14—H14A	120.1
N2—C6—C9	120.2 (4)	C14—C15—C16	119.8 (4)
N2—C6—C4	117.1 (3)	C14—C15—H15A	120.1
C9—C6—C4	122.6 (4)	C16—C15—H15A	120.1
N2—C7—N3	128.7 (4)	C11—C16—C15	120.6 (4)
N2—C7—S	120.5 (3)	C11—C16—H16A	119.7
N3—C7—S	110.6 (3)	C15—C16—H16A	119.7
N3—C8—C9	124.3 (4)	O1—C17—O2	122.4 (4)
N3—C8—H8A	117.9	O1—C17—C13	124.5 (4)
C9—C8—H8A	117.9	O2—C17—C13	113.0 (4)
C8—C9—C6	117.5 (4)		

C2—N1—C1—C5	−3.9 (9)	N3—C8—C9—C6	1.3 (7)
C1—N1—C2—C3	2.9 (9)	N2—C6—C9—C8	−2.2 (6)
N1—C2—C3—C4	−1.9 (9)	C4—C6—C9—C8	179.0 (4)
C2—C3—C4—C5	1.6 (7)	C7—S—C10—C11	−84.7 (3)
C2—C3—C4—C6	178.9 (5)	S—C10—C11—C12	129.7 (3)
N1—C1—C5—C4	3.9 (9)	S—C10—C11—C16	−53.9 (5)
C3—C4—C5—C1	−2.5 (8)	C16—C11—C12—C13	1.6 (5)
C6—C4—C5—C1	−179.9 (5)	C10—C11—C12—C13	178.1 (4)
C7—N2—C6—C9	4.1 (6)	C11—C12—C13—C14	−1.5 (6)
C7—N2—C6—C4	−177.0 (4)	C11—C12—C13—C17	176.1 (4)
C3—C4—C6—N2	13.3 (6)	C12—C13—C14—C15	1.1 (6)

C5—C4—C6—N2	−169.5 (4)	C17—C13—C14—C15	−176.3 (4)
C3—C4—C6—C9	−167.9 (4)	C13—C14—C15—C16	−1.0 (6)
C5—C4—C6—C9	9.3 (7)	C12—C11—C16—C15	−1.4 (6)
C6—N2—C7—N3	−5.8 (6)	C10—C11—C16—C15	−177.8 (4)
C6—N2—C7—S	179.7 (3)	C14—C15—C16—C11	1.1 (6)
C8—N3—C7—N2	4.8 (7)	C14—C13—C17—O1	163.6 (5)
C8—N3—C7—S	179.7 (3)	C12—C13—C17—O1	−14.0 (7)
C10—S—C7—N2	4.9 (4)	C14—C13—C17—O2	−12.5 (6)
C10—S—C7—N3	−170.5 (3)	C12—C13—C17—O2	170.0 (4)
C7—N3—C8—C9	−2.2 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1^{i}	0.82	1.84	2.659 (5)	174
C10—H10*B*···*Cg*3^{ii}	0.97	2.56	3.398 (5)	145

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