Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of 4-chloro-2-[(5-ethoxy-1,3,4-thiadiazol-2-yl)methyl]-5-(piperidin-1-yl)pyridazin-3(2*H*)-one

Hongsen Li,* Xinfeng Ren, Ya Li and Linjing Zhao

College of Chemistry and Chemical Engineering, Shanghai University of Engineering Science, 333 Longteng Road, Shanghai, People's Republic of China. *Correspondence e-mail: lihongsen19@163.com

Received 1 September 2014; accepted 15 September 2014

Edited by E. R. T. Tiekink, University of Malaya, Malaysia

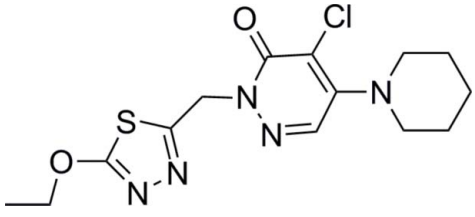
In the title molecule, $C_{14}H_{18}ClN_5O_2S$, the six atoms of the 1,6-dihydropyridazine ring are essentially coplanar (r.m.s. deviation = 0.008 Å), and the dihedral angle between this and the 1,3,4-thiadiazole ring is 62.06 (10)°. In the crystal, centrosymmetrically related molecules are linked by intermolecular C—H—O hydrogen bonding to form a supramolecular dimer. The terminal ethyl group is statistically disordered over two positions.

Keywords: pyridazinone derivatives; crystal structure; C—H—O hydrogen bonding.

CCDC reference: 1024313

1. Related literature

For the biological activity of pyridazinone derivatives, see: Abouzid *et al.* (2008); Siddiqui *et al.* (2010), and for their synthesis, see: Wang *et al.* (2010); Zhang *et al.* (2002).



2. Experimental

2.1. Crystal data

$C_{14}H_{18}ClN_5O_2S$ M_r = 355.84

Triclinic, $P\overline{1}$
a = 5.2840 (8) Å
b = 11.0323 (16) Å
c = 14.902 (2) Å
α = 107.318 (2)°
β = 91.590 (2)°
γ = 99.528 (2)°
V = 815.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 K
0.30 × 0.24 × 0.16 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.895, T_{max} = 0.942
4244 measured reflections
2828 independent reflections
2490 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.121
S = 1.58
2828 reflections
229 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14A—H14A···O1^{i}	0.96	2.45	3.366 (11)	160

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

Financial support from the Development Program of the Shanghai University of Engineering Science is gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5340).

References

Abouzid, K., Hakeem, M. A., Khalil, O. & Maklad, Y. (2008). *Bioorg. Med. Chem.* **16**, 382–389.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Gottingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siddiqui, A. A., Mishra, R. & Shaharyar, M. (2010). *Eur. J. Med. Chem.* **45**, 2283–2290.
Wang, T. T., Bing, G. F., Zhang, X. & Qin, Z. F. (2010). *Chem. J. Chin. Univ.* **31**, 708–713.
Zhang, Y., Liu, D. & Dai, C. F. (2002). *Chem. J. Chin. Univ.* **23**, 1882–1886.

supporting information

Acta Cryst. (2014). E**70**, o1113 [doi:10.1107/S1600536814020662]

Crystal structure of 4-chloro-2-[(5-ethoxy-1,3,4-thiadiazol-2-yl)methyl]-5-(piperidin-1-yl)pyridazin-3(2*H*)-one

Hongsen Li, Xinfeng Ren, Ya Li and Linjing Zhao

S1. Experimental

A mixture of 4,5-dichloro-2-[(5-ethoxy-1,3,4-thiadiazol-2-yl)methyl]-pyridazin-3(2H)-one (3.98 g, 1.3 mmol), piperidine (1.37 g, 19.5 mmol), potassium carbonate (3 g) and dry DMF (30mL) was stirred at 40°C for 8 h. The mixture was then poured into ice-water and a yellow precipitate -formed. The precipitate was washed with water, followed by vaccum drying, to give the pure title compound (3.38 g, yield: 73.2 %). The obtained compound was recrystallized from its ethyl acetate/petroleum ether (5:1) to give yellow crystals.

S1.1. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2–1.5U_{eq}(C). The terminal ethyl group (C13 and C14) was statistically disordered over two positions.

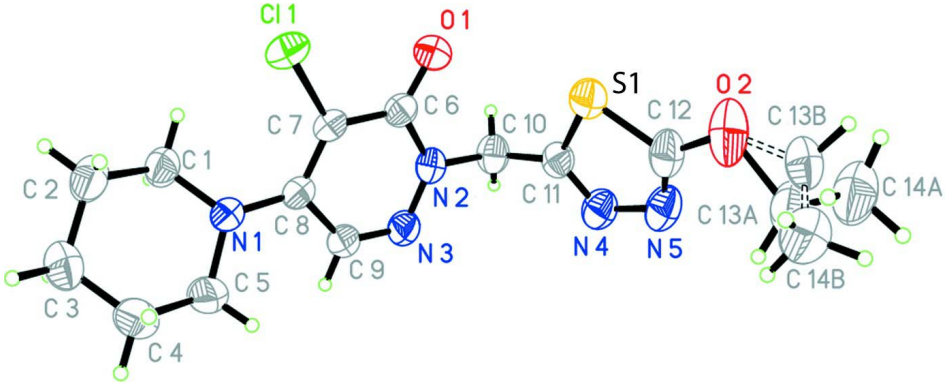


Figure 1

Molecular structure of the title compound showing atom labelling and displacement ellipsoids at 50%.

4-Chloro-2-[(5-ethoxy-1,3,4-thiadiazol-2-yl)methyl]-5-(piperidin-1-yl)pyridazin-3(2*H*)-one

Crystal data

$C_{14}H_{18}ClN_5O_2S$
M_r = 355.84
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.2840 (8) Å
b = 11.0323 (16) Å
c = 14.902 (2) Å
α = 107.318 (2)°
β = 91.590 (2)°
γ = 99.528 (2)°
V = 815.1 (2) Å^3
Z = 2
F(000) = 372
D_x = 1.450 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2456 reflections

θ = 2.8–27.3°
μ = 0.38 mm^{-1}
T = 296 K

Block, yellow
0.30 × 0.24 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.895, T_{max} = 0.942

4244 measured reflections
2828 independent reflections
2490 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.1°, θ_{min} = 2.0°
h = −6→6
k = −13→13
l = −13→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.121
S = 1.58
2828 reflections
229 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
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.050
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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}	Occ. (<1)
C1	1.1293 (4)	0.40442 (18)	0.64144 (14)	0.0529 (5)	
H1A	0.9963	0.4362	0.6807	0.064*	
H1B	1.1620	0.4550	0.5984	0.064*	
C2	1.3686 (5)	0.4198 (2)	0.70167 (19)	0.0710 (7)	
H2A	1.4236	0.5100	0.7376	0.085*	
H2B	1.5043	0.3928	0.6621	0.085*	
C3	1.3239 (5)	0.3396 (2)	0.76876 (18)	0.0726 (7)	
H3A	1.4850	0.3448	0.8038	0.087*	
H3B	1.2037	0.3733	0.8135	0.087*	
C4	1.2172 (5)	0.2004 (2)	0.71378 (17)	0.0623 (6)	
H4A	1.3494	0.1633	0.6768	0.075*	
H4B	1.1719	0.1521	0.7576	0.075*	
C5	0.9873 (5)	0.1870 (2)	0.65010 (17)	0.0654 (6)	

H5A	0.9358	0.0973	0.6122	0.079*	
H5B	0.8461	0.2120	0.6873	0.079*	
C6	0.8041 (4)	0.24108 (16)	0.34196 (13)	0.0418 (4)	
C7	0.9639 (3)	0.28093 (16)	0.42889 (13)	0.0390 (4)	
C8	0.8999 (4)	0.23463 (16)	0.50280 (13)	0.0426 (4)	
C9	0.6649 (4)	0.14118 (19)	0.48425 (14)	0.0513 (5)	
H9	0.6167	0.1065	0.5323	0.062*	
C10	0.3987 (4)	0.1095 (2)	0.25530 (14)	0.0517 (5)	
H10A	0.3853	0.1833	0.2340	0.062*	
H10B	0.2307	0.0783	0.2729	0.062*	
C11	0.4730 (4)	0.00539 (18)	0.17587 (13)	0.0452 (5)	
C12	0.6399 (5)	−0.1324 (2)	0.05014 (15)	0.0605 (6)	
C13A	0.6909 (17)	−0.3381 (7)	−0.0461 (7)	0.074 (2)	0.503 (13)
H13A	0.6744	−0.3654	0.0099	0.089*	0.503 (13)
H13B	0.5282	−0.3668	−0.0845	0.089*	0.503 (13)
C14A	0.9045 (12)	−0.3878 (6)	−0.1004 (7)	0.083 (3)	0.503 (13)
H14A	0.9387	−0.3461	−0.1480	0.125*	0.503 (13)
H14B	0.8561	−0.4794	−0.1298	0.125*	0.503 (13)
H14C	1.0565	−0.3701	−0.0584	0.125*	0.503 (13)
C13B	0.7259 (16)	−0.3012 (8)	−0.0838 (6)	0.071 (2)	0.497 (13)
H13C	0.8075	−0.3014	−0.1413	0.085*	0.497 (13)
H13D	0.5414	−0.3283	−0.0987	0.085*	0.497 (13)
C14B	0.8311 (17)	−0.3870 (7)	−0.0377 (8)	0.086 (3)	0.497 (13)
H14D	1.0040	−0.3483	−0.0114	0.129*	0.497 (13)
H14E	0.8312	−0.4692	−0.0836	0.129*	0.497 (13)
H14F	0.7260	−0.3987	0.0116	0.129*	0.497 (13)
Cl1	1.24862 (9)	0.38323 (4)	0.42952 (4)	0.0529 (2)	
N1	1.0406 (3)	0.26824 (15)	0.58761 (11)	0.0507 (4)	
N2	0.5837 (3)	0.15084 (14)	0.33835 (11)	0.0437 (4)	
N3	0.5152 (3)	0.10080 (16)	0.40812 (12)	0.0519 (4)	
N4	0.3405 (4)	−0.10968 (17)	0.14941 (13)	0.0585 (5)	
N5	0.4391 (4)	−0.19254 (17)	0.07519 (14)	0.0630 (5)	
O1	0.8484 (3)	0.27791 (13)	0.27325 (10)	0.0585 (4)	
O2	0.7819 (4)	−0.1833 (2)	−0.01905 (13)	0.0901 (6)	
S1	0.73552 (11)	0.02873 (5)	0.11291 (4)	0.0556 (2)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0616 (13)	0.0406 (11)	0.0504 (12)	0.0058 (9)	−0.0067 (10)	0.0075 (9)
C2	0.0735 (16)	0.0530 (13)	0.0755 (16)	−0.0080 (11)	−0.0252 (13)	0.0164 (12)
C3	0.0772 (17)	0.0714 (15)	0.0639 (15)	0.0039 (12)	−0.0229 (13)	0.0205 (13)
C4	0.0673 (15)	0.0634 (14)	0.0622 (14)	0.0080 (11)	−0.0004 (11)	0.0307 (12)
C5	0.0680 (15)	0.0626 (14)	0.0651 (14)	−0.0112 (11)	−0.0107 (12)	0.0326 (12)
C6	0.0471 (11)	0.0336 (9)	0.0436 (11)	0.0097 (8)	0.0044 (8)	0.0091 (8)
C7	0.0404 (10)	0.0292 (8)	0.0459 (10)	0.0035 (7)	0.0023 (8)	0.0109 (7)
C8	0.0475 (11)	0.0324 (9)	0.0443 (11)	0.0013 (7)	−0.0012 (8)	0.0102 (8)
C9	0.0562 (13)	0.0474 (11)	0.0436 (11)	−0.0099 (9)	−0.0005 (9)	0.0145 (9)

C10	0.0438 (11)	0.0568 (12)	0.0496 (12)	0.0116 (9)	−0.0059 (9)	0.0085 (9)
C11	0.0427 (11)	0.0483 (11)	0.0413 (10)	0.0047 (8)	−0.0079 (8)	0.0117 (8)
C12	0.0638 (15)	0.0613 (13)	0.0476 (12)	0.0168 (11)	−0.0100 (11)	0.0022 (10)
C13A	0.088 (5)	0.058 (4)	0.058 (5)	0.008 (3)	0.015 (4)	−0.006 (3)
C14A	0.073 (4)	0.068 (4)	0.088 (6)	0.010 (3)	0.024 (4)	−0.007 (3)
C13B	0.080 (4)	0.073 (5)	0.045 (4)	0.009 (3)	−0.002 (3)	0.000 (3)
C14B	0.090 (6)	0.075 (4)	0.085 (6)	0.010 (4)	−0.001 (5)	0.017 (4)
Cl1	0.0456 (3)	0.0449 (3)	0.0658 (4)	−0.0037 (2)	0.0049 (2)	0.0198 (2)
N1	0.0600 (11)	0.0404 (9)	0.0476 (10)	−0.0061 (7)	−0.0102 (8)	0.0170 (7)
N2	0.0423 (9)	0.0424 (8)	0.0409 (9)	0.0042 (7)	−0.0018 (7)	0.0069 (7)
N3	0.0518 (10)	0.0487 (9)	0.0467 (10)	−0.0064 (7)	0.0007 (8)	0.0107 (8)
N4	0.0539 (11)	0.0553 (11)	0.0567 (11)	−0.0004 (8)	−0.0072 (9)	0.0092 (9)
N5	0.0615 (12)	0.0521 (11)	0.0610 (12)	0.0042 (9)	−0.0115 (10)	0.0007 (9)
O1	0.0772 (10)	0.0514 (8)	0.0479 (8)	0.0030 (7)	−0.0008 (7)	0.0220 (7)
O2	0.0843 (13)	0.0960 (14)	0.0663 (11)	0.0270 (11)	0.0062 (10)	−0.0163 (10)
S1	0.0575 (4)	0.0545 (3)	0.0502 (3)	0.0046 (2)	0.0026 (3)	0.0120 (2)

Geometric parameters (Å, º)

C1—N1	1.466 (2)	C10—N2	1.464 (2)
C1—C2	1.486 (3)	C10—C11	1.496 (3)
C1—H1A	0.9700	C10—H10A	0.9700
C1—H1B	0.9700	C10—H10B	0.9700
C2—C3	1.520 (3)	C11—N4	1.284 (3)
C2—H2A	0.9700	C11—S1	1.723 (2)
C2—H2B	0.9700	C12—N5	1.282 (3)
C3—C4	1.512 (3)	C12—O2	1.334 (3)
C3—H3A	0.9700	C12—S1	1.725 (2)
C3—H3B	0.9700	C13A—C14A	1.491 (13)
C4—C5	1.480 (3)	C13A—O2	1.619 (8)
C4—H4A	0.9700	C13A—H13A	0.9700
C4—H4B	0.9700	C13A—H13B	0.9700
C5—N1	1.476 (2)	C14A—H14A	0.9600
C5—H5A	0.9700	C14A—H14B	0.9600
C5—H5B	0.9700	C14A—H14C	0.9600
C6—O1	1.224 (2)	C13B—O2	1.349 (7)
C6—N2	1.390 (2)	C13B—C14B	1.489 (14)
C6—C7	1.436 (3)	C13B—H13C	0.9700
C7—C8	1.374 (3)	C13B—H13D	0.9700
C7—Cl1	1.7228 (18)	C14B—H14D	0.9600
C8—N1	1.366 (2)	C14B—H14E	0.9600
C8—C9	1.438 (3)	C14B—H14F	0.9600
C9—N3	1.282 (3)	N2—N3	1.347 (2)
C9—H9	0.9300	N4—N5	1.385 (3)
N1—C1—C2	110.36 (17)	N2—C10—H10A	109.0
N1—C1—H1A	109.6	C11—C10—H10A	109.0
C2—C1—H1A	109.6	N2—C10—H10B	109.0

N1—C1—H1B	109.6	C11—C10—H10B	109.0
C2—C1—H1B	109.6	H10A—C10—H10B	107.8
H1A—C1—H1B	108.1	N4—C11—C10	121.30 (19)
C1—C2—C3	110.7 (2)	N4—C11—S1	114.96 (16)
C1—C2—H2A	109.5	C10—C11—S1	123.74 (14)
C3—C2—H2A	109.5	N5—C12—O2	126.0 (2)
C1—C2—H2B	109.5	N5—C12—S1	116.59 (17)
C3—C2—H2B	109.5	O2—C12—S1	117.4 (2)
H2A—C2—H2B	108.1	C14A—C13A—O2	102.4 (7)
C4—C3—C2	109.89 (19)	C14A—C13A—H13A	111.3
C4—C3—H3A	109.7	O2—C13A—H13A	111.3
C2—C3—H3A	109.7	C14A—C13A—H13B	111.3
C4—C3—H3B	109.7	O2—C13A—H13B	111.3
C2—C3—H3B	109.7	H13A—C13A—H13B	109.2
H3A—C3—H3B	108.2	O2—C13B—C14B	104.2 (7)
C5—C4—C3	112.46 (19)	O2—C13B—H13C	110.9
C5—C4—H4A	109.1	C14B—C13B—H13C	110.9
C3—C4—H4A	109.1	O2—C13B—H13D	110.9
C5—C4—H4B	109.1	C14B—C13B—H13D	110.9
C3—C4—H4B	109.1	H13C—C13B—H13D	108.9
H4A—C4—H4B	107.8	C13B—C14B—H14D	109.5
N1—C5—C4	111.07 (17)	C13B—C14B—H14E	109.5
N1—C5—H5A	109.4	H14D—C14B—H14E	109.5
C4—C5—H5A	109.4	C13B—C14B—H14F	109.5
N1—C5—H5B	109.4	H14D—C14B—H14F	109.5
C4—C5—H5B	109.4	H14E—C14B—H14F	109.5
H5A—C5—H5B	108.0	C8—N1—C1	120.60 (15)
O1—C6—N2	119.39 (17)	C8—N1—C5	119.35 (15)
O1—C6—C7	125.93 (18)	C1—N1—C5	111.72 (16)
N2—C6—C7	114.67 (16)	N3—N2—C6	125.32 (15)
C8—C7—C6	122.23 (17)	N3—N2—C10	115.33 (15)
C8—C7—Cl1	123.29 (14)	C6—N2—C10	119.26 (16)
C6—C7—Cl1	114.37 (14)	C9—N3—N2	116.97 (16)
N1—C8—C7	125.69 (17)	C11—N4—N5	113.00 (19)
N1—C8—C9	120.06 (17)	C12—N5—N4	110.34 (17)
C7—C8—C9	114.23 (17)	C12—O2—C13B	127.2 (5)
N3—C9—C8	126.57 (19)	C12—O2—C13A	105.7 (4)
N3—C9—H9	116.7	C13B—O2—C13A	29.3 (3)
C8—C9—H9	116.7	C11—S1—C12	85.12 (11)
N2—C10—C11	112.73 (15)		

N1—C1—C2—C3	−58.3 (3)	C7—C6—N2—N3	−0.2 (3)
C1—C2—C3—C4	54.5 (3)	O1—C6—N2—C10	−4.9 (3)
C2—C3—C4—C5	−52.6 (3)	C7—C6—N2—C10	176.21 (15)
C3—C4—C5—N1	53.7 (3)	C11—C10—N2—N3	−100.33 (19)
O1—C6—C7—C8	−179.95 (18)	C11—C10—N2—C6	82.9 (2)
N2—C6—C7—C8	−1.1 (3)	C8—C9—N3—N2	−0.1 (3)
O1—C6—C7—Cl1	−3.6 (2)	C6—N2—N3—C9	0.8 (3)

N2—C6—C7—Cl1	175.26 (12)	C10—N2—N3—C9	−175.75 (18)
C6—C7—C8—N1	−179.63 (17)	C10—C11—N4—N5	−179.86 (16)
Cl1—C7—C8—N1	4.3 (3)	S1—C11—N4—N5	0.5 (2)
C6—C7—C8—C9	1.6 (3)	O2—C12—N5—N4	180.0 (2)
Cl1—C7—C8—C9	−174.41 (14)	S1—C12—N5—N4	0.7 (2)
N1—C8—C9—N3	−179.9 (2)	C11—N4—N5—C12	−0.8 (3)
C7—C8—C9—N3	−1.0 (3)	N5—C12—O2—C13B	9.5 (7)
N2—C10—C11—N4	112.2 (2)	S1—C12—O2—C13B	−171.2 (5)
N2—C10—C11—S1	−68.2 (2)	N5—C12—O2—C13A	−13.1 (5)
C7—C8—N1—C1	50.6 (3)	S1—C12—O2—C13A	166.2 (4)
C9—C8—N1—C1	−130.8 (2)	C14B—C13B—O2—C12	−87.8 (7)
C7—C8—N1—C5	−163.8 (2)	C14B—C13B—O2—C13A	−38.9 (11)
C9—C8—N1—C5	14.9 (3)	C14A—C13A—O2—C12	−163.5 (7)
C2—C1—N1—C8	−152.6 (2)	C14A—C13A—O2—C13B	55.1 (11)
C2—C1—N1—C5	59.4 (2)	N4—C11—S1—C12	−0.12 (17)
C4—C5—N1—C8	154.6 (2)	C10—C11—S1—C12	−179.72 (17)
C4—C5—N1—C1	−56.8 (3)	N5—C12—S1—C11	−0.36 (18)
O1—C6—N2—N3	178.74 (16)	O2—C12—S1—C11	−179.71 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14*A*—H14*A*···O1^{i}	0.96	2.45	3.366 (11)	160

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