

## 3-[(2-Chloro-1,3-thiazol-5-yl)methyl]-5-methyl-1,3,5-oxadiazinan-4-one

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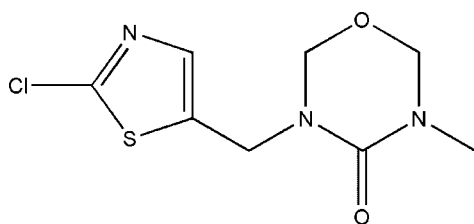
Received 12 September 2012; accepted 8 October 2012

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.081; data-to-parameter ratio = 15.7.

In the title compound,  $\text{C}_8\text{H}_{10}\text{ClN}_3\text{O}_2\text{S}$ , the oxadiazinane ring is in a sofa conformation with the ring O atom deviating from the best plane of the remaining five atoms by 0.636 (2) Å. A short intramolecular C-S $\cdots$ O=C contact [S $\cdots$ O 3.122 (2) Å, C-S $\cdots$ O 80.0 (2)°] is observed between the two molecular fragments bridged by the methylene group. In the crystal, C-H $\cdots$ O hydrogen bonds link molecules, forming chains along the  $b$  axis.

### Related literature

For the biological activity of thiamethoxam, see: Maienfisch *et al.* (2001, 2006); Suchail *et al.* (2001); Ford & Casida (2006). For the structure of thiamethoxam, see: Chopra *et al.* (2004). For ring conformations, see: Duax & Norton (1975).



### Experimental

#### Crystal data

$\text{C}_8\text{H}_{10}\text{ClN}_3\text{O}_2\text{S}$

$M_r = 247.70$

Orthorhombic,  $P2_12_12_1$

$a = 4.6141$  (2) Å

$b = 11.7335$  (4) Å

$c = 20.1460$  (8) Å

$V = 1090.70$  (7) Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 0.53$  mm<sup>-1</sup>

$T = 293$  K

$0.3 \times 0.2 \times 0.2$  mm

#### Data collection

Oxford Diffraction Xcalibur

Sapphire3 diffractometer

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)

$T_{\min} = 0.925$ ,  $T_{\max} = 1.000$

22323 measured reflections

2147 independent reflections

1974 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.081$

$S = 1.07$

2147 reflections

137 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.22$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

856 Friedel pairs

Flack parameter: 0.04 (9)

**Table 1**

Hydrogen-bond geometry (Å, °).

D—H $\cdots$ A	D—H	H $\cdots$ A	D $\cdots$ A	D—H $\cdots$ A
C12—H12 $\cdots$ O7 <sup>i</sup>	0.93	2.60	3.443 (3)	151

Symmetry code: (i)  $-x, y + \frac{1}{2}, -z + \frac{3}{2}$ .

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); 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: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

RK acknowledges the Department of Science & Technology for access to the single-crystal X-ray diffractometer sanctioned as a National Facility under project No. SR/S2/CMP-47/2003.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2519).

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## supplementary materials

*Acta Cryst.* (2012). E68, o3109 [doi:10.1107/S1600536812042031]

**3-[(2-Chloro-1,3-thiazol-5-yl)methyl]-5-methyl-1,3,5-oxadiazinan-4-one**

**Rajni Kant, Vivek K. Gupta, Kamini Kapoor, Chetan S. Shripanavar and Kaushik Banerjee**

**Comment**

An important milestone in the history of modern insect control is marked by the discovery of neonicotinoid insecticides (Maienfisch, 2006). In 1998 Novartis launched thiamethoxam as a novel second generation neonicotinoid with a unique structure and outstanding insecticidal activity (Maienfisch *et al.*, 2001). The major natural metabolite of thiamethoxam is the title compound, which is thiamethoxam urea derivative (Suchail *et al.*, 2001, Ford & Casida, 2006)

In the title compound (Fig.1) all bond lengths and angles are normal and correspond to those observed in the related structure (Chopra *et al.*, 2004). The oxadiazinane ring is in a sofa conformation [asymmetry parameter:  $\Delta C_s(O1-C4) = 7.47$  (Duax & Norton, 1975)]. In the crystal, the displacement of the atom O1 from the plane defined by atoms C2/N3/C4/N5/C6 is  $-0.636(2)$  Å. In thiametoxam and the title compound the two molecular fragments bridged by the methylene group are similarly oriented. C—H $\cdots$ O hydrogen bonds link molecules to form chains along *b* axis(Fig.2).

**Experimental**

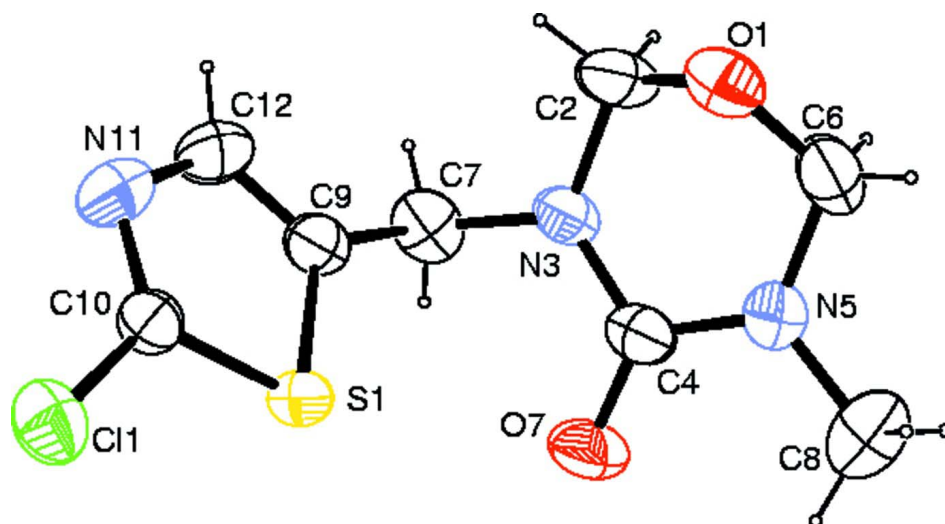
Thiamethoxam (0.291 g, 0.001 mol) was dissolved in 5 ml methanol and to it 5 ml of 1 N K<sub>2</sub>CO<sub>3</sub> solution was added. The reaction mixture was refluxed for about 10 h on a water bath at 343 K and then cooled. The reaction mixture was neutralized with 1 N HCl solution, until the solid compound was separated out. The synthesized compound was dissolved in minimum amount of methanol and was kept standing for slow evaporation until colourless transparent crystals were formed (m.p. = 372 K).

**Refinement**

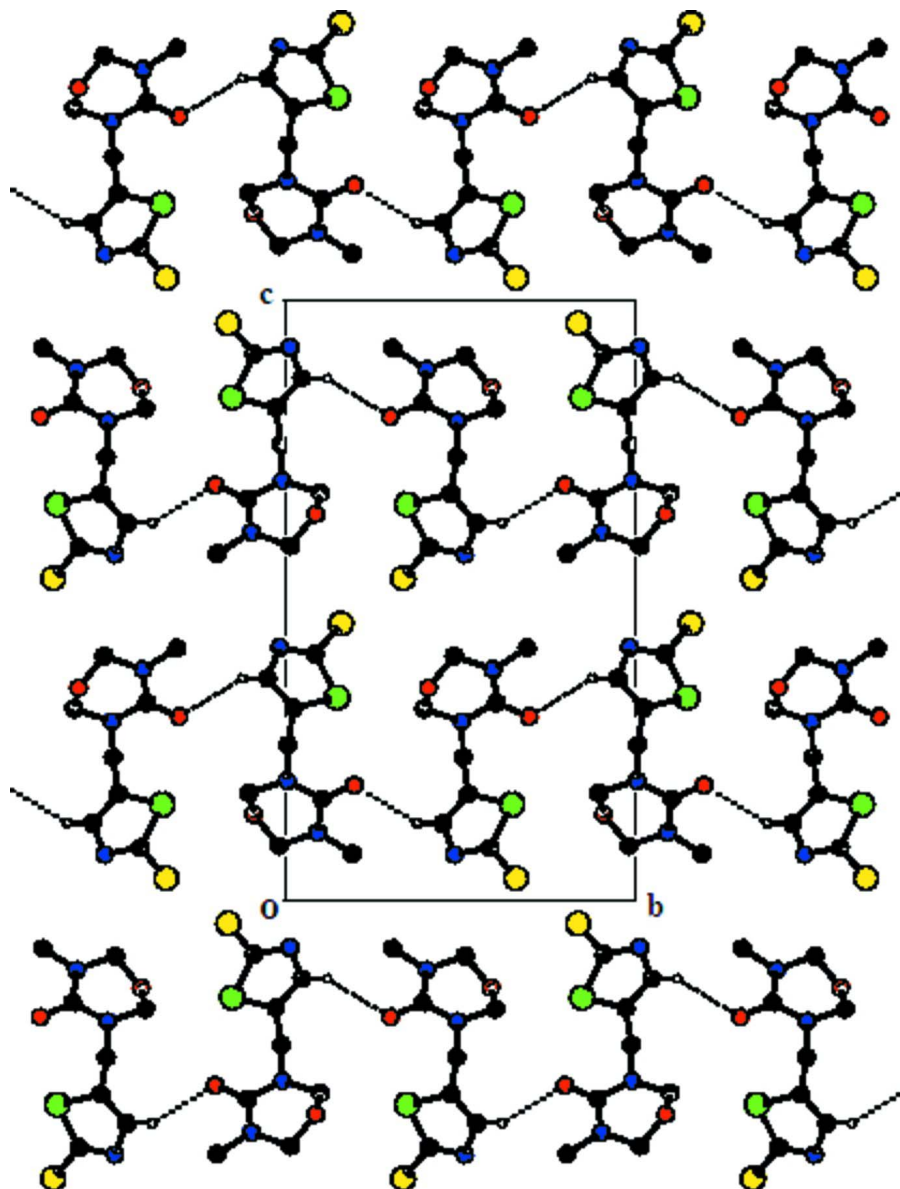
All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å and with  $U_{iso}(H) = 1.2U_{eq}(C)$  or  $1.5U_{eq}(\text{methyl C})$ .

**Computing details**

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

**Figure 1**

*ORTEP* view of the molecule with the atom-labeling scheme. The thermal ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.



**Figure 2**

The packing arrangement of molecules viewed down the *a* axis. The dotted lines show intermolecular C—H...O hydrogen bonds.

**3-[(2-Chloro-1,3-thiazol-5-yl)methyl]-5-methyl-1,3,5-oxadiazinan-4-one**

*Crystal data*

$C_8H_{10}ClN_3O_2S$

$M_r = 247.70$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.6141(2) \text{ \AA}$

$b = 11.7335(4) \text{ \AA}$

$c = 20.1460(8) \text{ \AA}$

$V = 1090.70(7) \text{ \AA}^3$

$Z = 4$

$F(000) = 512$

$D_x = 1.508 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 11280 reflections

$\theta = 3.5\text{--}29.0^\circ$

$\mu = 0.53 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Needle, white

$0.3 \times 0.2 \times 0.2 \text{ mm}$

*Data collection*

Oxford Diffraction Xcalibur Sapphire3 diffractometer	22323 measured reflections
Radiation source: fine-focus sealed tube	2147 independent reflections
Graphite monochromator	1974 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1049 pixels mm <sup>-1</sup>	$R_{\text{int}} = 0.034$
$\omega$ scan	$\theta_{\text{max}} = 26.0^\circ$ , $\theta_{\text{min}} = 3.5^\circ$
Absorption correction: multi-scan ( <i>CrysAlis PRO</i> ; Oxford Diffraction, 2010)	$h = -5 \rightarrow 5$
$T_{\text{min}} = 0.925$ , $T_{\text{max}} = 1.000$	$k = -14 \rightarrow 14$
	$l = -24 \rightarrow 24$

*Refinement*

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.2799P]$
$wR(F^2) = 0.081$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2147 reflections	$\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{\AA}^{-3}$
137 parameters	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$
0 restraints	Absolute structure: Flack (1983), 856 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (9)
Secondary atom site location: difference Fourier map	

*Special details*

**Experimental.** *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 *CrysAlis171.NET*) (compiled Aug 27 2010, 11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.29172 (13)	0.85031 (5)	0.83926 (3)	0.04611 (15)
C11	0.64157 (15)	0.83860 (6)	0.96232 (3)	0.0642 (2)
O1	0.4809 (4)	1.08899 (16)	0.64448 (10)	0.0653 (5)
C2	0.2262 (7)	1.1021 (2)	0.68003 (16)	0.0668 (8)
H2A	0.0844	1.1412	0.6527	0.080*
H2B	0.2630	1.1487	0.7189	0.080*
N3	0.1108 (5)	0.99332 (17)	0.70052 (10)	0.0512 (5)
C4	0.1836 (6)	0.8940 (2)	0.67011 (12)	0.0516 (6)
N5	0.3433 (6)	0.90503 (19)	0.61443 (11)	0.0640 (6)
C6	0.4364 (9)	1.0158 (3)	0.59173 (14)	0.0778 (9)
H6A	0.6149	1.0079	0.5667	0.093*
H6B	0.2905	1.0475	0.5624	0.093*

C7	−0.0657 (6)	0.9905 (3)	0.76014 (14)	0.0613 (7)
H7A	−0.2048	0.9289	0.7564	0.074*
H7B	−0.1734	1.0613	0.7634	0.074*
O7	0.1066 (5)	0.80128 (15)	0.69297 (10)	0.0744 (6)
C8	0.4470 (9)	0.8075 (3)	0.57872 (19)	0.0975 (12)
H8A	0.3872	0.7392	0.6012	0.146*
H8B	0.6547	0.8098	0.5765	0.146*
H8C	0.3684	0.8080	0.5346	0.146*
C9	0.1052 (5)	0.9747 (2)	0.82176 (12)	0.0494 (6)
C10	0.4123 (5)	0.9127 (2)	0.91085 (12)	0.0484 (6)
N11	0.3276 (6)	1.01443 (19)	0.92255 (12)	0.0692 (7)
C12	0.1516 (7)	1.0488 (2)	0.87096 (15)	0.0680 (8)
H12	0.0683	1.1210	0.8704	0.082*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0529 (3)	0.0353 (2)	0.0501 (3)	0.0000 (2)	0.0022 (2)	0.0000 (2)
C11	0.0651 (4)	0.0728 (4)	0.0546 (4)	−0.0027 (4)	−0.0063 (3)	0.0091 (3)
O1	0.0633 (11)	0.0579 (11)	0.0748 (12)	−0.0128 (9)	−0.0003 (10)	0.0120 (10)
C2	0.0732 (18)	0.0395 (12)	0.088 (2)	0.0011 (13)	0.0047 (16)	0.0129 (13)
N3	0.0552 (12)	0.0421 (10)	0.0563 (12)	−0.0002 (9)	0.0042 (10)	0.0105 (9)
C4	0.0570 (13)	0.0446 (11)	0.0533 (14)	−0.0083 (11)	−0.0123 (13)	0.0084 (11)
N5	0.0871 (17)	0.0551 (13)	0.0499 (12)	−0.0056 (13)	0.0064 (12)	−0.0020 (10)
C6	0.102 (3)	0.079 (2)	0.0529 (17)	−0.0165 (19)	0.0084 (16)	0.0135 (15)
C7	0.0480 (14)	0.0691 (16)	0.0669 (17)	0.0082 (13)	0.0019 (12)	0.0110 (14)
O7	0.1021 (17)	0.0435 (9)	0.0775 (13)	−0.0221 (11)	−0.0017 (13)	0.0082 (9)
C8	0.121 (3)	0.089 (2)	0.083 (2)	0.003 (2)	0.011 (2)	−0.025 (2)
C9	0.0472 (13)	0.0450 (12)	0.0561 (14)	0.0058 (10)	0.0112 (11)	0.0061 (11)
C10	0.0505 (13)	0.0459 (13)	0.0487 (13)	−0.0061 (11)	0.0058 (11)	0.0009 (10)
N11	0.0892 (19)	0.0501 (12)	0.0683 (15)	0.0053 (13)	0.0002 (14)	−0.0144 (11)
C12	0.087 (2)	0.0429 (14)	0.0741 (19)	0.0176 (14)	0.0026 (17)	−0.0061 (12)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S1—C10	1.710 (2)	N5—C6	1.443 (4)
S1—C9	1.731 (2)	C6—H6A	0.9700
C11—C10	1.718 (3)	C6—H6B	0.9700
O1—C6	1.382 (4)	C7—C9	1.482 (4)
O1—C2	1.385 (4)	C7—H7A	0.9700
C2—N3	1.443 (3)	C7—H7B	0.9700
C2—H2A	0.9700	C8—H8A	0.9600
C2—H2B	0.9700	C8—H8B	0.9600
N3—C4	1.359 (3)	C8—H8C	0.9600
N3—C7	1.452 (3)	C9—C12	1.336 (4)
C4—O7	1.233 (3)	C10—N11	1.278 (3)
C4—N5	1.348 (3)	N11—C12	1.379 (4)
N5—C8	1.434 (4)	C12—H12	0.9300
C10—S1—C9	88.42 (12)	N3—C7—C9	113.4 (2)

C6—O1—C2	109.9 (2)	N3—C7—H7A	108.9
O1—C2—N3	111.3 (2)	C9—C7—H7A	108.9
O1—C2—H2A	109.4	N3—C7—H7B	108.9
N3—C2—H2A	109.4	C9—C7—H7B	108.9
O1—C2—H2B	109.4	H7A—C7—H7B	107.7
N3—C2—H2B	109.4	N5—C8—H8A	109.5
H2A—C2—H2B	108.0	N5—C8—H8B	109.5
C4—N3—C2	122.6 (2)	H8A—C8—H8B	109.5
C4—N3—C7	119.5 (2)	N5—C8—H8C	109.5
C2—N3—C7	117.6 (2)	H8A—C8—H8C	109.5
O7—C4—N5	123.6 (2)	H8B—C8—H8C	109.5
O7—C4—N3	121.1 (2)	C12—C9—C7	128.7 (2)
N5—C4—N3	115.3 (2)	C12—C9—S1	108.5 (2)
C4—N5—C8	121.5 (3)	C7—C9—S1	122.7 (2)
C4—N5—C6	120.9 (2)	N11—C10—S1	117.1 (2)
C8—N5—C6	117.4 (3)	N11—C10—C11	123.4 (2)
O1—C6—N5	111.1 (2)	S1—C10—C11	119.52 (14)
O1—C6—H6A	109.4	C10—N11—C12	108.3 (2)
N5—C6—H6A	109.4	C9—C12—N11	117.6 (2)
O1—C6—H6B	109.4	C9—C12—H12	121.2
N5—C6—H6B	109.4	N11—C12—H12	121.2
H6A—C6—H6B	108.0		
C6—O1—C2—N3	54.5 (3)	C4—N3—C7—C9	85.9 (3)
O1—C2—N3—C4	-20.9 (4)	C2—N3—C7—C9	-87.9 (3)
O1—C2—N3—C7	152.7 (2)	N3—C7—C9—C12	111.4 (3)
C2—N3—C4—O7	172.1 (3)	N3—C7—C9—S1	-66.6 (3)
C7—N3—C4—O7	-1.3 (4)	C10—S1—C9—C12	-0.2 (2)
C2—N3—C4—N5	-7.7 (4)	C10—S1—C9—C7	178.1 (2)
C7—N3—C4—N5	178.8 (2)	C9—S1—C10—N11	0.3 (2)
O7—C4—N5—C8	-2.7 (5)	C9—S1—C10—C11	-178.75 (16)
N3—C4—N5—C8	177.2 (3)	S1—C10—N11—C12	-0.2 (3)
O7—C4—N5—C6	-177.5 (3)	C11—C10—N11—C12	178.8 (2)
N3—C4—N5—C6	2.4 (4)	C7—C9—C12—N11	-178.0 (3)
C2—O1—C6—N5	-59.9 (4)	S1—C9—C12—N11	0.2 (4)
C4—N5—C6—O1	31.4 (4)	C10—N11—C12—C9	0.0 (4)
C8—N5—C6—O1	-143.6 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C12—H12...O7 <sup>i</sup>	0.93	2.60	3.443 (3)	151

Symmetry code: (i)  $-x, y+1/2, -z+3/2$ .