



# Crystal structure of methyl (3*RS*,4*SR*,4*aRS*,11*aRS*,11*bSR*)-5-oxo- 3,4,4*a*,5,7,8,9,10,11,11*a*-decahydro- 3,11*b*-epoxyazepino[2,1-*a*]isoindole-4- carboxylate

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Received 2 September 2015; accepted 7 September 2015

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

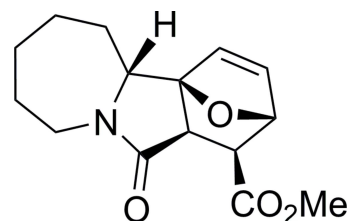
The title compound, C<sub>15</sub>H<sub>19</sub>NO<sub>4</sub>, is the a product of the esterification of the corresponding carbonic acid with methanol. The molecule comprises a fused tetracyclic system containing three five-membered rings (2-pyrrolidinone, tetrahydrofuran and dihydrofuran) and one seven-membered ring (azepane). The five-membered rings have the usual envelope conformations, with the quaternary C atom being the flap atom for the 2-pyrrolidinone ring, and the ether O atom being the common flap atom for the remaining rings. The seven-membered azepane ring adopts a chair conformation with the methine and middle methylene C atoms lying above and below the mean plane defined by the remaining five atoms. The carboxylate substituent is rotated by 77.56 (5)° with respect to the base plane of the tetrahydrofuran ring. In the crystal, the molecules are bound by weak C—H···O hydrogen-bonding interactions into puckered layers parallel to (001).

**Keywords:** crystal structure; 3,6a-epoxyisoindoles; azepane; intramolecular cycloaddition; C—H···O hydrogen bonds.

**CCDC reference:** 1422681

## 1. Related literature

For the synthesis of 2-(furan-2-yl)azepane, see: Asher *et al.* (1981); Shono *et al.* (1981); Nikolic & Beak (1997). For intramolecular cycloaddition reactions of  $\alpha,\beta$ -unsaturated acid anhydrides to  $\alpha$ -furylamines (IMDAF reactions), see: Vogel *et al.* (1999); Zubkov *et al.* (2005). For related compounds, see: Zylber *et al.* (1995); Evans *et al.* (1999); Kachkovskiy & Kolodiaznyi (2007); Kharitonov *et al.* (2009); Aabid *et al.* (2010); Zubkov *et al.* (2010, 2011, 2014); Toze *et al.* (2011); Wang & Li (2012); Zaytsev, Mikhailova *et al.* (2012); Zaytsev, Zubkov *et al.* (2012); Zaytsev *et al.* (2013); Chen *et al.* (2013); Hizartzidis *et al.* (2014).



## 2. Experimental

### 2.1. Crystal data

C <sub>15</sub> H <sub>19</sub> NO <sub>4</sub>	$\gamma = 106.620 (2)^\circ$
$M_r = 277.31$	$V = 691.24 (12) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.5460 (8) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.6984 (10) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 10.2894 (10) \text{ \AA}$	$T = 290 \text{ K}$
$\alpha = 103.857 (2)^\circ$	$0.30 \times 0.25 \times 0.25 \text{ mm}$
$\beta = 94.745 (2)^\circ$	

### 2.2. Data collection

Bruker APEXII CCD diffractometer	9699 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2003)	3268 independent reflections
$T_{\min} = 0.959$ , $T_{\max} = 0.969$	2613 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.018$

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	182 parameters
$wR(F^2) = 0.141$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
3268 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

**Table 1**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1···O5 <sup>i</sup>	0.93	2.59	3.4576 (19)	156
C3—H3···O13 <sup>ii</sup>	0.98	2.55	3.5259 (19)	174
C4A—H4A···O14 <sup>iii</sup>	0.98	2.51	3.4190 (17)	154
C14—H14A···O5 <sup>iv</sup>	0.96	2.56	3.279 (2)	132

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $-x + 2, -y, -z + 2$ ; (iii)  $-x + 2, -y + 1, -z + 2$ ; (iv)  $-x + 3, -y + 1, -z + 2$ .

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

## Acknowledgements

Funding from the US National Science Foundation (PREM DMR-0934212 and EPSCoR IIA-1301346) is gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5384).

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## supporting information

*Acta Cryst.* (2015). E71, o729–o730 [doi:10.1107/S2056989015016679]

## Crystal structure of methyl (3*RS*,4*SR*,4*aRS*,11*aRS*,11*bSR*)-5-oxo-3,4,4*a*,5,7,8,9,10,11,11*a*-decahydro-3,11*b*-epoxyazepino[2,1-*a*]isoindole-4-carboxylate

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### S1. Structural commentary

In the last decade our synthetic group has published some effective strategies for the synthesis of 3,6*a*-epoxyisoindoles annulated with various heterocycles (Zubkov *et al.* 2010, 2011, 2014; Zaytsev, Mikhailova *et al.* 2012; Zaytsev, Zubkov *et al.* 2012; Zaytsev *et al.* 2013). These strategies were based on the intramolecular cycloaddition reaction of  $\alpha,\beta$ -unsaturated acid anhydrides to the heterocycles containing an  $\alpha$ -furfurylamine fragment (IMDAF reaction) (Vogel *et al.*, 1999; Zubkov *et al.*, 2005). Therefore, within the scope of this investigation, the initial carboxylic acid was easily synthesized by the treatment of 2-(2-furyl)perhydroazepine (Asher *et al.*, 1981; Shono *et al.*, 1981; Nikolic *et al.*, 1997) with maleic anhydride.

This work reports the structural characterization of 3,6*a*-epoxyisoindole annulated with perhydroazepine ring. The esterification of the initial carboxylic acid obtained as fine-crystalline powder is due to its poor solubility in most common organic solvents (Fig. 1).

The molecule of the title compound, C<sub>15</sub>H<sub>19</sub>NO<sub>4</sub>, (I) comprises a fused tetracyclic system containing three five-membered rings (2-pyrrolidinone, tetrahydrofuran and dihydrofuran) and one seven-membered ring (1,4-diazepine) (Fig. 2). The 2-pyrrolidinone, tetrahydrofuran and dihydrofuran five-membered rings have the usual envelope conformations (Zylber *et al.*, 1995; Evans *et al.*, 1999; Kachkovskiy *et al.*, 2007; Kharitonov *et al.*, 2009; Aabid *et al.*, 2010; Toze *et al.*, 2011; Wang *et al.*, 2012; Chen *et al.*, 2013; Hizartzidis *et al.*, 2014). The seven-membered diazepine ring adopts a chair conformation. The nitrogen N6 atom has the slightly pyramidalized geometry (sum of the bond angles is 359.7 (4)°). The dihedral angle between the basal plane of the diazepine ring (C8—C9/C11—C11A) and the base plane of the pyrrolidinone ring (C4—C5—N6—C11A) is 66.55 (9)°. The carboxylate substituent is rotated by 77.56 (5)° to the base plane of the tetrahydrofuran ring.

The molecule of (I) possesses five asymmetric centers at the C3, C4, C4A, C11A and C11B carbon atoms and can have potentially numerous diastereomers. The crystal of (I) is racemic and consists of enantiomeric pairs with the following relative configuration of the centers: *rac*-3*R*\*,4*S*\*,4*AR*\*,11*AR*\*,11*BS*\*.

In the crystal, the molecules of (I) are bound by the weak intermolecular C—H⋯O hydrogen bonding interactions into puckered layers parallel to (001) (Fig. 3, Table 1).

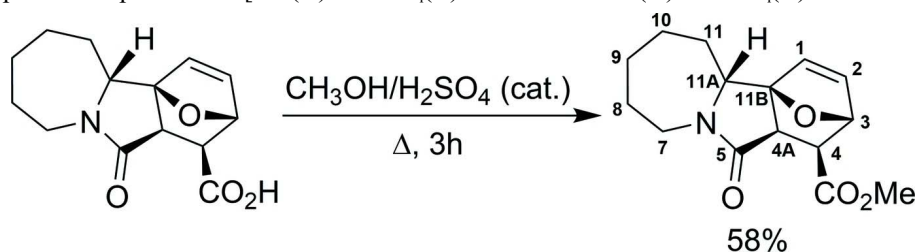
### S2. Synthesis and crystallization

A solution of the initial acid – (3*R*\*,4*S*\*,4*AR*\*,11*AR*\*,11*BS*\*)-5-oxo-3,4,4*a*,5,7,8,9,10,11,11*a*-decahydro-3,11*b*-epoxyazepino[2,1-*a*]isoindole-4-carboxylic acid (0.5 g, 1.8 mmol) in methanol (30 mL) was refluxed for 3 h in the presence of

catalytic amount of concentrated  $\text{H}_2\text{SO}_4$  (monitoring by TLC until disappearance of the starting compound (eluent – EtOAc:hexane (1:3), Sorbfil). Then the solvent was evaporated. The residual oil was passed through a thin layer of aluminum oxide (eluent – chloroform). Chloroform was removed under reduced pressure. The crude ester was recrystallized from a mixture of EtOAc-EtOH to give the target compound I as white crystals. Yield is 0.31 g (58%). Single-crystals were isolated as fine needles by slow re-crystallization from EtOAc-EtOH. M.pt = 388–389 K. IR (KBr),  $\nu/\text{cm}^{-1}$ : 1741 (C=O), 1693 (C=O). Mass spectrum, EI-MS (70 eV),  $m/z$  (I<sub>r</sub>(%)): 230 (5), 189 (52), 146 (96), 118 (19), 96 (100), 91 (17), 77 (18), 70 (17), 44 (33), 42 (25), 41 (18).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 300 K):  $\delta$  = 1.64–1.25 (m, 3H, H9B, H10A, H10B), 2.00–1.92 (m, 4H, H11B, H8A, H8B, H9A), 2.25–2.22 (m, 1H, H11A), 2.71 (d, 1H, H4,  $J_{4,4a}$  = 9.0), 2.86 (d, 1H, H4a,  $J_{4a,4}$  = 9.0), 3.18–3.14 (m, 1H, H7B), 3.78–3.73 (m, 1H, H11A), 3.79 (s, 3H,  $\text{CO}_2\text{Me}$ ), 3.96 (m, 1H, H7A), 5.15 (d, 1H, H3,  $J_{3,2}$  = 1.7), 6.47 (dd, 1H, H2,  $J_{2,1}$  = 6.5,  $J_{2,3}$  = 1.7), 6.53 (d, 1H, H1,  $J_{1,2}$  = 6.5).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 300 K):  $\delta$  = 26.6, 27.6, 29.2, 33.6 (C8, C9, C10, C11), 43.5, 45.4, 49.6, 52.1 (C7, C4A, C13, C4), 59.6 (C11A), 81.2 (C3), 92.1 (C11B), 135.2, 137.4 (C1, C2), 170.5, 172.5 ( $\text{CO}_2\text{Me}$ , NCO).

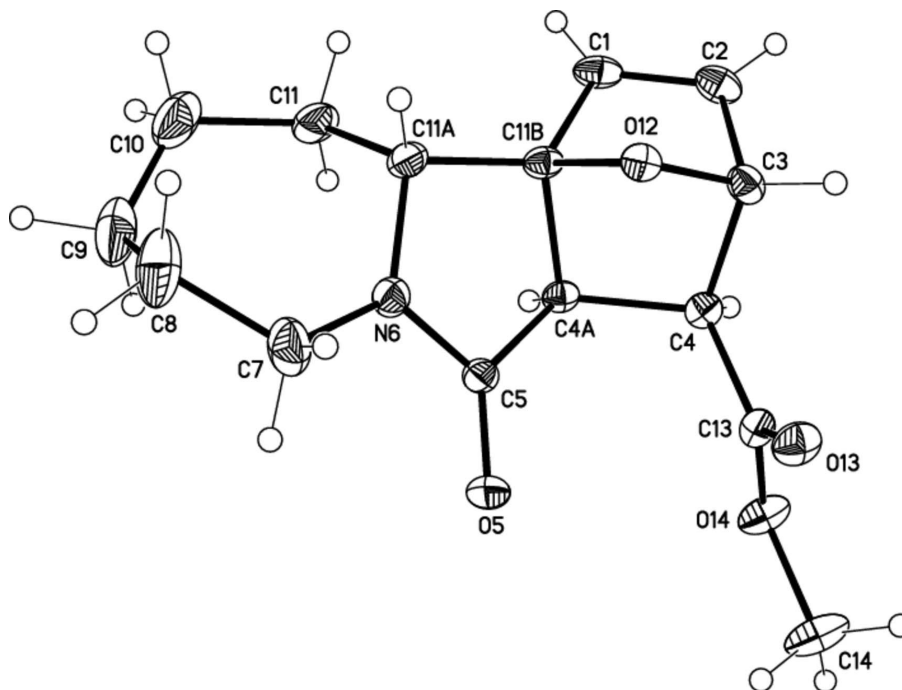
### S3. Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.93–0.98 Å and refined in the riding model with fixed isotropic displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for  $\text{CH}_3$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for remaining H].

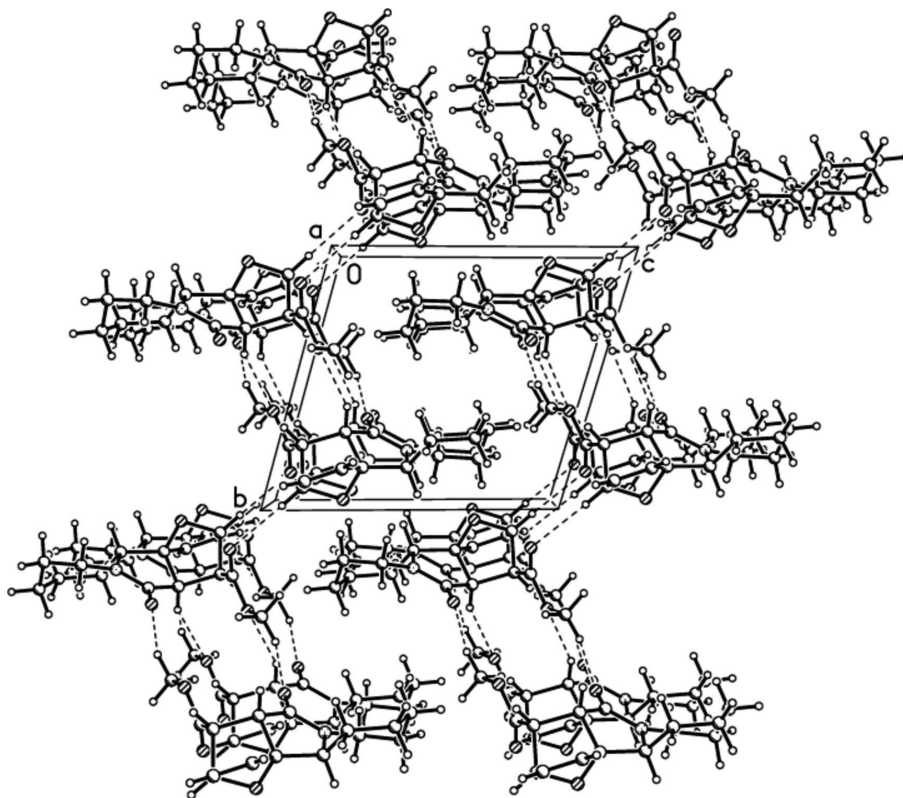


**Figure 1**

Esterification of 5-oxo-3,4,4a,5,7,8,9,10,11,11a-decahydro-3,11b-epoxyazepino[2,1-a]isoindole-4-carboxylic acid with methanol.

**Figure 2**

Molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

**Figure 3**

Crystal packing of (I) along the *a* axis demonstrating the H-bonded puckered layers parallel to (001). Dashed lines indicate the weak intermolecular C—H...O hydrogen-bonding interactions.

**Methyl (3*RS*,4*SR*,4*aRS*,11*aRS*,11*bSR*)-5-oxo-3,4,4*a*,5,7,8,9,10,11,11*a*-decahydro-3,11*b*-epoxyazepino[2,1-*a*]isoindole-4-carboxylate**

*Crystal data*

$C_{15}H_{19}NO_4$

$M_r = 277.31$

Triclinic,  $P\bar{1}$

$a = 7.5460$  (8) Å

$b = 9.6984$  (10) Å

$c = 10.2894$  (10) Å

$\alpha = 103.857$  (2)°

$\beta = 94.745$  (2)°

$\gamma = 106.620$  (2)°

$V = 691.24$  (12) Å<sup>3</sup>

$Z = 2$

$F(000) = 296$

$D_x = 1.332$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4971 reflections

$\theta = 2.3$ – $29.5$ °

$\mu = 0.10$  mm<sup>-1</sup>

$T = 290$  K

Prism, colourless

$0.30 \times 0.25 \times 0.25$  mm

*Data collection*

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2003)

$T_{\min} = 0.959$ ,  $T_{\max} = 0.969$

9699 measured reflections

3268 independent reflections

2613 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 28.0$ °,  $\theta_{\min} = 2.1$ °

$h = -9 \rightarrow 9$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 13$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.141$

$S = 1.03$

3268 reflections

182 parameters

0 restraints

Primary atom site location: difference Fourier map

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 0.153P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.55178 (19)	0.17386 (17)	0.75675 (18)	0.0471 (4)
H1	0.4515	0.2009	0.7244	0.056*
C2	0.5710 (2)	0.12672 (19)	0.86511 (18)	0.0491 (4)
H2	0.4866	0.1136	0.9255	0.059*
C3	0.7587 (2)	0.09790 (16)	0.87191 (15)	0.0403 (3)
H3	0.7723	0.0281	0.9240	0.048*
C4	0.91211 (19)	0.25575 (15)	0.91807 (14)	0.0356 (3)
H4	0.8735	0.3221	0.9899	0.043*
C4A	0.89714 (17)	0.30474 (14)	0.78642 (13)	0.0320 (3)
H4A	0.8720	0.4006	0.8028	0.038*
C5	1.04722 (18)	0.29945 (15)	0.69682 (14)	0.0352 (3)
O5	1.21692 (14)	0.35060 (14)	0.73347 (12)	0.0513 (3)
N6	0.96070 (17)	0.23177 (15)	0.56738 (12)	0.0414 (3)
C7	1.0663 (3)	0.2066 (3)	0.45696 (19)	0.0663 (5)
H7A	1.0931	0.1134	0.4506	0.080*
H7B	1.1852	0.2864	0.4788	0.080*
C8	0.9697 (5)	0.1996 (3)	0.3221 (2)	0.0920 (8)
H8A	0.8776	0.1011	0.2861	0.110*
H8B	1.0616	0.2089	0.2615	0.110*
C9	0.8730 (4)	0.3136 (3)	0.3181 (2)	0.0914 (8)
H9A	0.9526	0.4088	0.3778	0.110*
H9B	0.8637	0.3246	0.2268	0.110*
C10	0.6832 (4)	0.2847 (3)	0.3564 (2)	0.0873 (8)
H10A	0.6030	0.1913	0.2941	0.105*
H10B	0.6353	0.3632	0.3411	0.105*
C11	0.6606 (3)	0.2749 (2)	0.4981 (2)	0.0620 (5)
H11A	0.5277	0.2389	0.5017	0.074*
H11B	0.7103	0.3751	0.5592	0.074*

C11A	0.7550 (2)	0.17431 (17)	0.55118 (15)	0.0423 (3)
H11C	0.7081	0.0719	0.4914	0.051*
C11B	0.72833 (18)	0.17475 (15)	0.69564 (14)	0.0353 (3)
O12	0.76978 (13)	0.05080 (10)	0.73010 (10)	0.0362 (2)
C13	1.1022 (2)	0.24951 (16)	0.96810 (14)	0.0369 (3)
O13	1.15577 (16)	0.14282 (13)	0.93999 (12)	0.0503 (3)
O14	1.20359 (16)	0.38062 (13)	1.05547 (12)	0.0547 (3)
C14	1.3882 (3)	0.3891 (3)	1.1134 (2)	0.0785 (7)
H14A	1.4432	0.4823	1.1823	0.118*
H14B	1.4649	0.3831	1.0437	0.118*
H14C	1.3793	0.3076	1.1528	0.118*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0251 (7)	0.0436 (8)	0.0698 (11)	0.0131 (6)	0.0051 (7)	0.0090 (7)
C2	0.0352 (8)	0.0505 (9)	0.0634 (10)	0.0136 (6)	0.0208 (7)	0.0147 (7)
C3	0.0389 (7)	0.0414 (8)	0.0462 (8)	0.0148 (6)	0.0143 (6)	0.0171 (6)
C4	0.0346 (7)	0.0389 (7)	0.0350 (7)	0.0170 (6)	0.0074 (5)	0.0063 (5)
C4A	0.0276 (6)	0.0300 (6)	0.0393 (7)	0.0131 (5)	0.0028 (5)	0.0074 (5)
C5	0.0299 (6)	0.0378 (7)	0.0428 (7)	0.0134 (5)	0.0052 (5)	0.0169 (6)
O5	0.0271 (5)	0.0681 (8)	0.0603 (7)	0.0106 (5)	0.0047 (5)	0.0266 (6)
N6	0.0388 (7)	0.0508 (7)	0.0387 (6)	0.0172 (6)	0.0076 (5)	0.0158 (5)
C7	0.0728 (13)	0.0911 (15)	0.0516 (10)	0.0372 (11)	0.0287 (9)	0.0299 (10)
C8	0.153 (3)	0.0921 (17)	0.0439 (11)	0.0550 (17)	0.0284 (13)	0.0186 (11)
C9	0.135 (3)	0.0910 (17)	0.0566 (12)	0.0335 (16)	0.0119 (14)	0.0391 (12)
C10	0.105 (2)	0.0797 (15)	0.0733 (14)	0.0216 (14)	-0.0235 (14)	0.0358 (12)
C11	0.0454 (9)	0.0690 (12)	0.0734 (12)	0.0160 (8)	-0.0107 (8)	0.0323 (10)
C11A	0.0372 (7)	0.0424 (8)	0.0424 (8)	0.0088 (6)	-0.0039 (6)	0.0109 (6)
C11B	0.0273 (6)	0.0330 (7)	0.0450 (7)	0.0113 (5)	0.0011 (5)	0.0093 (5)
O12	0.0351 (5)	0.0309 (5)	0.0438 (5)	0.0132 (4)	0.0073 (4)	0.0085 (4)
C13	0.0395 (7)	0.0419 (7)	0.0327 (7)	0.0179 (6)	0.0056 (5)	0.0105 (5)
O13	0.0452 (6)	0.0456 (6)	0.0635 (7)	0.0234 (5)	0.0030 (5)	0.0123 (5)
O14	0.0458 (6)	0.0534 (7)	0.0541 (7)	0.0215 (5)	-0.0103 (5)	-0.0062 (5)
C14	0.0509 (11)	0.0831 (15)	0.0813 (14)	0.0250 (10)	-0.0251 (10)	-0.0073 (11)

*Geometric parameters (Å, °)*

C1—C2	1.315 (3)	C8—C9	1.495 (4)
C1—C11B	1.5182 (19)	C8—H8A	0.9700
C1—H1	0.9300	C8—H8B	0.9700
C2—C3	1.519 (2)	C9—C10	1.484 (4)
C2—H2	0.9300	C9—H9A	0.9700
C3—O12	1.4380 (18)	C9—H9B	0.9700
C3—C4	1.567 (2)	C10—C11	1.504 (3)
C3—H3	0.9800	C10—H10A	0.9700
C4—C13	1.5062 (19)	C10—H10B	0.9700
C4—C4A	1.5450 (19)	C11—C11A	1.531 (2)



C4—H4	0.9800	C11—H11A	0.9700
C4A—C5	1.5226 (18)	C11—H11B	0.9700
C4A—C11B	1.5508 (18)	C11A—C11B	1.515 (2)
C4A—H4A	0.9800	C11A—H11C	0.9800
C5—O5	1.2228 (17)	C11B—O12	1.4386 (16)
C5—N6	1.3502 (19)	C13—O13	1.1972 (17)
N6—C7	1.454 (2)	C13—O14	1.3389 (18)
N6—C11A	1.4718 (19)	O14—C14	1.440 (2)
C7—C8	1.490 (3)	C14—H14A	0.9600
C7—H7A	0.9700	C14—H14B	0.9600
C7—H7B	0.9700	C14—H14C	0.9600
C2—C1—C11B	105.23 (13)	C10—C9—C8	117.5 (2)
C2—C1—H1	127.4	C10—C9—H9A	107.9
C11B—C1—H1	127.4	C8—C9—H9A	107.9
C1—C2—C3	106.25 (13)	C10—C9—H9B	107.9
C1—C2—H2	126.9	C8—C9—H9B	107.9
C3—C2—H2	126.9	H9A—C9—H9B	107.2
O12—C3—C2	100.98 (12)	C9—C10—C11	118.87 (19)
O12—C3—C4	101.71 (10)	C9—C10—H10A	107.6
C2—C3—C4	106.12 (12)	C11—C10—H10A	107.6
O12—C3—H3	115.4	C9—C10—H10B	107.6
C2—C3—H3	115.4	C11—C10—H10B	107.6
C4—C3—H3	115.4	H10A—C10—H10B	107.0
C13—C4—C4A	115.38 (11)	C10—C11—C11A	116.12 (18)
C13—C4—C3	112.85 (12)	C10—C11—H11A	108.3
C4A—C4—C3	100.04 (11)	C11A—C11—H11A	108.3
C13—C4—H4	109.4	C10—C11—H11B	108.3
C4A—C4—H4	109.4	C11A—C11—H11B	108.3
C3—C4—H4	109.4	H11A—C11—H11B	107.4
C5—C4A—C4	119.20 (10)	N6—C11A—C11B	100.81 (11)
C5—C4A—C11B	100.41 (11)	N6—C11A—C11	112.40 (13)
C4—C4A—C11B	101.76 (10)	C11B—C11A—C11	112.17 (14)
C5—C4A—H4A	111.4	N6—C11A—H11C	110.4
C4—C4A—H4A	111.4	C11B—C11A—H11C	110.4
C11B—C4A—H4A	111.4	C11—C11A—H11C	110.4
O5—C5—N6	125.20 (13)	O12—C11B—C11A	111.09 (11)
O5—C5—C4A	126.66 (13)	O12—C11B—C1	101.52 (11)
N6—C5—C4A	108.11 (11)	C11A—C11B—C1	126.86 (12)
C5—N6—C7	121.55 (14)	O12—C11B—C4A	99.30 (10)
C5—N6—C11A	114.43 (12)	C11A—C11B—C4A	105.24 (11)
C7—N6—C11A	123.77 (14)	C1—C11B—C4A	109.56 (11)
N6—C7—C8	114.54 (19)	C3—O12—C11B	96.06 (10)
N6—C7—H7A	108.6	O13—C13—O14	123.64 (13)
C8—C7—H7A	108.6	O13—C13—C4	126.23 (13)
N6—C7—H7B	108.6	O14—C13—C4	110.07 (11)
C8—C7—H7B	108.6	C13—O14—C14	115.86 (13)
H7A—C7—H7B	107.6	O14—C14—H14A	109.5

C7—C8—C9	117.0 (2)	O14—C14—H14B	109.5
C7—C8—H8A	108.0	H14A—C14—H14B	109.5
C9—C8—H8A	108.0	O14—C14—H14C	109.5
C7—C8—H8B	108.0	H14A—C14—H14C	109.5
C9—C8—H8B	108.0	H14B—C14—H14C	109.5
H8A—C8—H8B	107.3		
C11B—C1—C2—C3	-0.09 (17)	C10—C11—C11A—N6	65.8 (2)
C1—C2—C3—O12	-32.37 (16)	C10—C11—C11A—C11B	178.61 (16)
C1—C2—C3—C4	73.37 (16)	N6—C11A—C11B—O12	-76.52 (13)
O12—C3—C4—C13	-91.15 (13)	C11—C11A—C11B—O12	163.70 (12)
C2—C3—C4—C13	163.64 (12)	N6—C11A—C11B—C1	159.66 (13)
O12—C3—C4—C4A	32.02 (12)	C11—C11A—C11B—C1	39.9 (2)
C2—C3—C4—C4A	-73.20 (13)	N6—C11A—C11B—C4A	30.01 (13)
C13—C4—C4A—C5	17.25 (17)	C11—C11A—C11B—C4A	-89.77 (14)
C3—C4—C4A—C5	-104.12 (13)	C2—C1—C11B—O12	32.57 (15)
C13—C4—C4A—C11B	126.38 (12)	C2—C1—C11B—C11A	160.29 (14)
C3—C4—C4A—C11B	5.01 (12)	C2—C1—C11B—C4A	-71.75 (15)
C4—C4A—C5—O5	-49.29 (19)	C5—C4A—C11B—O12	82.53 (11)
C11B—C4A—C5—O5	-159.17 (14)	C4—C4A—C11B—O12	-40.49 (11)
C4—C4A—C5—N6	132.54 (12)	C5—C4A—C11B—C11A	-32.47 (12)
C11B—C4A—C5—N6	22.66 (13)	C4—C4A—C11B—C11A	-155.48 (11)
O5—C5—N6—C7	3.1 (2)	C5—C4A—C11B—C1	-171.63 (11)
C4A—C5—N6—C7	-178.69 (14)	C4—C4A—C11B—C1	65.35 (13)
O5—C5—N6—C11A	177.51 (13)	C2—C3—O12—C11B	50.39 (12)
C4A—C5—N6—C11A	-4.29 (16)	C4—C3—O12—C11B	-58.82 (11)
C5—N6—C7—C8	-152.76 (19)	C11A—C11B—O12—C3	171.91 (11)
C11A—N6—C7—C8	33.4 (3)	C1—C11B—O12—C3	-50.81 (12)
N6—C7—C8—C9	43.2 (3)	C4A—C11B—O12—C3	61.51 (11)
C7—C8—C9—C10	-81.4 (3)	C4A—C4—C13—O13	-91.08 (18)
C8—C9—C10—C11	62.4 (3)	C3—C4—C13—O13	23.1 (2)
C9—C10—C11—C11A	-48.4 (3)	C4A—C4—C13—O14	91.77 (14)
C5—N6—C11A—C11B	-16.60 (16)	C3—C4—C13—O14	-154.06 (12)
C7—N6—C11A—C11B	157.66 (15)	O13—C13—O14—C14	1.6 (2)
C5—N6—C11A—C11	103.02 (16)	C4—C13—O14—C14	178.86 (16)
C7—N6—C11A—C11	-82.7 (2)		

*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>
C1—H1...O5 <sup>i</sup>	0.93	2.59	3.4576 (19)	156
C3—H3...O13 <sup>ii</sup>	0.98	2.55	3.5259 (19)	174
C4A—H4A...O14 <sup>iii</sup>	0.98	2.51	3.4190 (17)	154
C14—H14A...O5 <sup>iv</sup>	0.96	2.56	3.279 (2)	132

Symmetry codes: (i)  $x-1, y, z$ ; (ii)  $-x+2, -y, -z+2$ ; (iii)  $-x+2, -y+1, -z+2$ ; (iv)  $-x+3, -y+1, -z+2$ .