

## Gabapentin-lactum–chloranilic acid (1/1)

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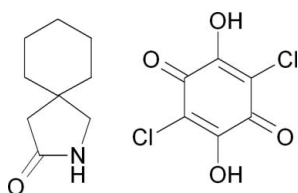
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Key indicators: single-crystal X-ray study;  $T = 110$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.119; data-to-parameter ratio = 14.9.

In the title compound,  $\text{C}_9\text{H}_{15}\text{NO}\cdot\text{C}_6\text{H}_2\text{Cl}_2\text{O}_4$  [systematic name: 2-azaspiro[4.5]decan-3-one–chloranilic acid (1/1)], the cyclohexane ring of the lactam molecule adopts a slightly distorted normal chair conformation and the five-membered 3-azaspiro ring is in a slightly distorted chair conformation. The dihedral angle between the least-squares planes of the cyclohexane and 3-azaspiro rings is  $84.0$  (3)°. In the crystal, the chloranilic acid molecule and the gabapentin-lactum molecules are held together by strong intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds with two bifurcated O acceptor atoms on the chloranilic acid molecule and one on the gabapentin-lactum molecule, each bonding with an inter- and intramolecular hydrogen bond. The molecules are linked into chains parallel to (011) and propagating along the  $b$  axis.

### Related literature

For the neuroprotective properties of gabapentin-lactam and related compounds, see: Lagreze *et al.* (2001); Henle *et al.* (2006); Bowery (1993). For the synthesis and spectroscopic studies of chloranilic acid charge-transfer complexes, see: Al-Attas *et al.* (2009). For related structures, see: Gotoh *et al.* (2008); Ibers (2001); Ishida (2004); Ishida & Kashino (2000); Jasinski *et al.* (2009). For density functional theory (DFT), see: Frisch *et al.* (2004); Hehre *et al.* (1986); Schmidt & Polik (2007).



### Experimental

#### Crystal data

$\text{C}_9\text{H}_{15}\text{NO}\cdot\text{C}_6\text{H}_2\text{Cl}_2\text{O}_4$   
 $M_r = 362.20$   
 Triclinic,  $P\bar{1}$   
 $a = 6.6127$  (9) Å  
 $b = 9.5800$  (11) Å  
 $c = 13.0724$  (13) Å  
 $\alpha = 102.679$  (9)°  
 $\beta = 91.934$  (9)°  
 $\gamma = 98.481$  (10)°  
 $V = 797.23$  (16) Å<sup>3</sup>  
 $Z = 2$   
 Cu  $K\alpha$  radiation  
 $\mu = 3.90$  mm<sup>-1</sup>  
 $T = 110$  K  
 $0.47 \times 0.42 \times 0.15$  mm

#### Data collection

Goniometer Xcalibur  
 diffractometer with a Ruby  
 (Gemini Cu) detector  
 Absorption correction: multi-scan  
 (*CrysAlis RED*; Oxford  
 Diffraction, 2007)  
 $T_{\min} = 0.200$ ,  $T_{\max} = 0.557$   
 5138 measured reflections  
 3123 independent reflections  
 2731 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.119$   
 $S = 1.05$   
 3123 reflections  
 210 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.40$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1A}-\text{H1A}\cdots\text{O2A}^i$	0.84	1.97	2.7493 (19)	153
$\text{O1A}-\text{H1A}\cdots\text{O2A}$	0.84	2.20	2.671 (2)	115
$\text{O3A}-\text{H3A}\cdots\text{O1B}^{ii}$	0.84	1.70	2.4807 (19)	153
$\text{O3A}-\text{H3A}\cdots\text{O4A}$	0.84	2.26	2.7148 (19)	114
$\text{N2B}-\text{H2BA}\cdots\text{O1B}^{ii}$	0.88	2.07	2.913 (2)	161
$\text{N2B}-\text{H2BA}\cdots\text{O4A}$	0.88	2.53	3.091 (2)	122

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); 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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2704).

### References

- Al-Attas, A. S., Habeeb, M. M. & Al-Raimi, D. S. (2009). *J. Mol. Struct.* **928**, 158–170.  
 Bowery, N. G. (1993). *Annu. Rev. Pharmacol. Toxicol.* **33**, 109–147.  
 Frisch, M. J., *et al.* (2004). *GAUSSIAN03*, Revision C01. Gaussian Inc., Wallingford, CT, USA.  
 Gotoh, K., Asaji, T. & Ishida, H. (2008). *Acta Cryst.* **C64**, o550–o553.  
 Hehre, W. J., Random, L., Schleyer, P. V. R. & Pople, J. A. (1986). In *Ab Initio Molecular Orbital Theory*. New York: Wiley.  
 Henle, F., Leemhuis, J., Fischer, C., Bock, H. H., Lindemeyer, K., Feuerstein, T. J. & Meyer, D. K. (2006). *J. Pharmacol. Exp. Ther.* **319**, 181–191.  
 Ibers, J. A. (2001). *Acta Cryst.* **C57**, 641–643.

- Ishida, H. (2004). *Acta Cryst.* **E60**, o1900–o1901.
- Ishida, H. & Kashino, S. (2000). *Acta Cryst.* **C56**, e202–e204.
- Jasinski, J. P., Butcher, R. J., Yathirajan, H. S., Mallesha, L., Mohana, K. N. & Narayana, B. (2009). *J. Chem. Crystallogr.* **39**, 777–780.
- Lagrze, W. A., Muller-Velten, R. & Feuerstein, T. J. (2001). *Graefe's Arch. Clin. Exp. Ophthalmol.* **239**, 845–849.
- Oxford Diffraction (2007). *CrysAlis PRO* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
- Schmidt, J. R. & Polik, W. F. (2007). *WebMO Pro*. WebMO, LLC: Holland, MI, USA, available from <http://www.webmo.net>.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2010). E66, o163–o164 [doi:10.1107/S1600536809053410]

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**S1. Comment**

Gabapentin-lactum (systematic name: 3-azaspiro-[4,5]-decan-2-one), is an intermediate for the preparation of gabapentin. Gabapentin-lactam (GBP-*L*) is also a derivative of the anti-convulsant drug gabapentin. The neuroprotective properties of gabapentin-lactam are described (Lagreze *et al.* 2001). Gabapentin is currently used as a therapeutic agent against epilepsy as well as neuropathic pain. In contrast to gabapentin, its derivative gabapentin-lactam has a pronounced neuroprotective activity (Henle *et al.* 2006). Gabapentin is structurally related to the neurotransmitteraminobutyric acid (GABA), which has been widely studied for its significant inhibitory action in the central nervous system (Bowery, 1993). We have recently reported a crystal structure of a second polymorph of gabapentin hydrochloride hemihydrate with a three-center bifurcated hydrogen bond (Jasinski *et al.* 2009).

Chloranilic acid is a strong dibasic organic acid which exhibits electron-acceptor properties on one hand and acidic properties leading to formation of hydrogen bonds on the other hand. In the case of stronger bases the proton-transfer, hydrogen bonded ion pairs will be formed which is interesting from the point of view of electron transfer reactions in biological systems. Also, protonation of the donor from acidic acceptors are generally a route for the formation of ion pair adducts. The synthesis and spectroscopic studies of charge transfer complexes between chloranilic acid and some heterocyclic amines in ethanol (Al-Attas, Habeeb & Al-Raimi, 2009) have been studied. In view of the importance of gabapentin-lactam, this paper reports the interaction of Gabapentin-lactam as an electron donor with chloranilic acid as an electron acceptor resulting in the formation of a charge transfer complex (I) while the two molecules are held together by intermolecular hydrogen bonding interactions.

The title compound,  $C_9H_{15}NO.C_6H_2Cl_2O_4$ , (I), is composed of two independent molecules, gabapentin-lactum ( $C_9H_{15}NO$ ) and chloranilic acid ( $C_6H_2Cl_2O_4$ ), in the asymmetric unit (1:1) (Fig. 1). In the gabapentin-lactum molecule the cyclohexane ring (C4B—C9B) adopts a slightly distorted normal chair conformation and the 5-membered 3-Azaspiro ring is in a slightly distorted half-chair conformation. The N2B and C1B atoms are  $sp^2$  hybridized while the C3B, C4B and C10B atoms are  $sp^3$ . The C10B—C4B—C5B—C6B, N2B—C3B—C4B—C5B and N2B—C3B—C4B—C9B torsion angles are  $177.77(17)^\circ$ ,  $-136.89(16)^\circ$  and  $101.19(17)^\circ$ , respectively, indicating a significant twist between the 3-azaspiro and cyclohexane rings while sharing a corner C4B atom. The dihedral angle between the least squares planes of these two rings measures  $84.0(3)^\circ$ . The planar chloranilic acid molecule and gabapentin-lactum molecules are held together by N—H $\cdots$ O and O—H $\cdots$ O intermolecular hydrogen bonds with two bifurcated oxygen acceptor atoms on the chloranilic acid molecule (O2A & O4A) and one on the gabapentin-lactum molecule (O1B), each bifurcating with an inter and intra molecular hydrogen bond, respectively (Fig. 3, Table 1). This produces a set of O—H $\cdots$ O—H $\cdots$ O—H infinite chains along the *b* axis in (011). The O=C—N—H groups from the 3-azaspiro groups in adjacent gabapentin-lactum molecules form a R2,2(8) graph set motif, while the O=C—C—O—H groups from symmetry related chloranilic acid molecules form a R2,2(10) graph set motif in the unit cell (Fig. 3). The dihedral angles between mean planes of the chloranilic acid molecule and the 3-azaspiro and cyclohexane rings of the gabapentin-lactum molecule are  $7.0(1)^\circ$  and  $77.0(1)^\circ$ ,

respectively. In addition, weak  $Cg3 \cdots Cg3$  [ $= 3.680(1) \text{ \AA}$ ; slippage  $= 1.825 \text{ \AA}$ ;  $-x, 1 - y, 1 - z$ ] intermolecular interactions are observed where  $Cg3 = C1A-C6A$ , which contribute to crystal packing.

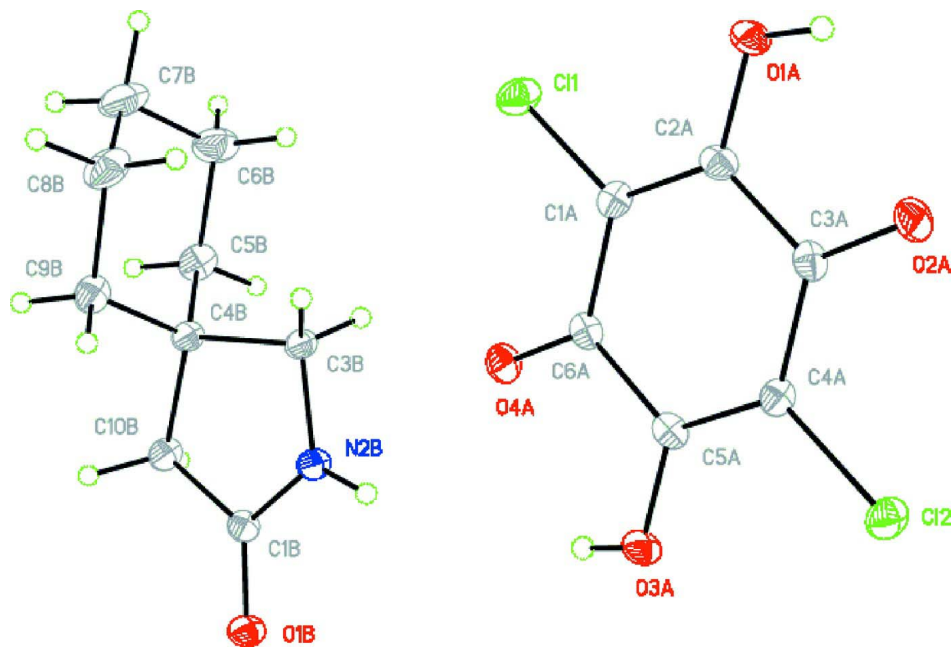
Following a geometry optimization, density functional theory (DFT) calculation at the B3LYP 6-31-G(*d*) level (Hehre *et al.*, 1986; Schmidt & Polik, 2007) with the Gaussian03 program package (Frisch *et al.*, 2004) the dihedral angle between the least squares planes of the 3-azaspiro and cyclohexane rings in the gabapentin-lactum molecule become  $79.2(9)^\circ$  compared to  $84.0(3)^\circ$  in the crystal. The dihedral angles between mean planes of the chloranilic acid molecule and the 3-azaspiro and cyclohexane rings of the gabapentin-lactum molecule become  $2.0(0)^\circ$  and  $77.2(9)^\circ$ , respectively, versus  $7.0(1)^\circ$  and  $77.0(1)^\circ$  observed in the crystal. Starting geometries were taken from X-ray refinement data. This suggests that strong  $N-H \cdots O$  and  $O-H \cdots O$  intermolecular hydrogen bonds and weak intermolecular  $Cg \cdots Cg$  intermolecular interactions, collectively, influence crystal packing.

## S2. Experimental

The title compound was synthesized by adding a solution of chloranilic acid (0.42 g, 2 mmol) in 10 ml methanol to a solution of gabapentin-lactam (0.21 g, 2 mmol) in 10 ml methanol. A red color developed and the solution was allowed to evaporate slowly at room temperature. The red colored complex formed was filtered off, washed with diethyl ether and dried under vacuum. X-ray quality crystals were grown from methanol:water (80:20 v/v) solvent mixture (m.p.: 439–442 K). Analysis for  $C_{15}H_{17}Cl_2NO_5$ : Found (Calculated): C:49.68 (49.74); H: 4.70 (4.73); N:3.85 (3.87).

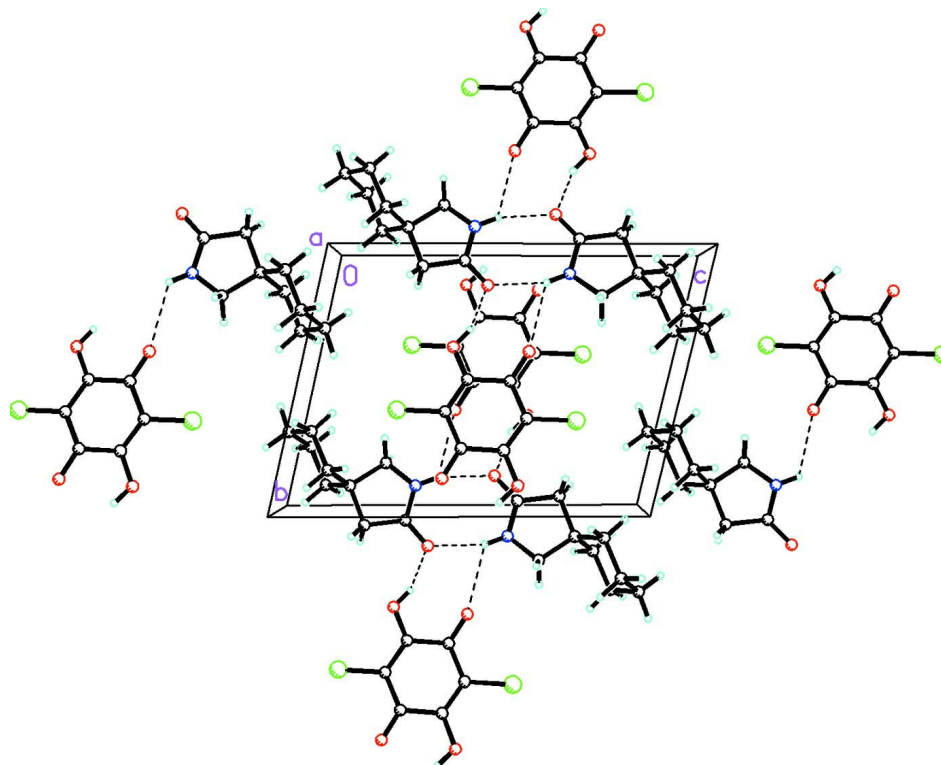
## S3. Refinement

The hydroxyl and aza hydrogen atoms (H1A, H3A & H2B) were obtained from a difference fourier map. The rest of the H atoms were placed in their calculated positions and then refined using the riding model with  $O-H = 0.84 \text{ \AA}$ ,  $N-H = 0.88 \text{ \AA}$ ,  $C-H = 0.99 \text{ \AA}$ , and with  $U_{iso}(H) = 1.18-1.22U_{eq}(C,O,N)$ .



**Figure 1**

Molecular structure of  $C_9H_{15}NO.C_6H_2Cl_2O_4$  showing the atom labeling scheme and 50% probability displacement ellipsoids. H atoms are presented as small circles of arbitrary radius.

**Figure 2**

Packing diagram of the title compound, (I), viewed down the  $a$  axis. Dashed lines indicate strong N—H $\cdots$ O and O—H $\cdots$ O intermolecular hydrogen bonds linking the C<sub>9</sub>H<sub>15</sub>NO and C<sub>6</sub>H<sub>2</sub>Cl<sub>2</sub>O<sub>4</sub> molecules into an infinite O—H $\cdots$ O—H $\cdots$ O—H chain network along the  $b$  axis in (011).

### 2-azaspiro[4.5]decan-3-one-chloranilic acid (1/1)

#### Crystal data

C<sub>9</sub>H<sub>15</sub>NO·C<sub>6</sub>H<sub>2</sub>Cl<sub>2</sub>O<sub>4</sub>

$M_r = 362.20$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 6.6127$  (9) Å

$b = 9.5800$  (11) Å

$c = 13.0724$  (13) Å

$\alpha = 102.679$  (9)°

$\beta = 91.934$  (9)°

$\gamma = 98.481$  (10)°

$V = 797.23$  (16) Å<sup>3</sup>

$Z = 2$

$F(000) = 376$

$D_x = 1.509$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 3438 reflections

$\theta = 4.8$ – $74.0$ °

$\mu = 3.90$  mm<sup>-1</sup>

$T = 110$  K

Irregular plate, red-brown

$0.47 \times 0.42 \times 0.15$  mm

#### Data collection

Goniometer Xcalibur  
diffractometer with a Ruby (Gemini Cu)  
detector

Radiation source: Enhance (Cu) X-ray Source

Graphite monochromator

Detector resolution: 10.5081 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2007)

$T_{\min} = 0.200$ ,  $T_{\max} = 0.557$

5138 measured reflections

3123 independent reflections

2731 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 74.2^\circ$ ,  $\theta_{\min} = 4.8^\circ$   
 $h = -8 \rightarrow 5$

$k = -10 \rightarrow 11$   
 $l = -15 \rightarrow 16$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.119$   
 $S = 1.05$   
 3123 reflections  
 210 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.4046P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$

*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.

**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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.07819 (8)	0.35536 (5)	0.25360 (3)	0.02058 (15)
C12	0.30441 (7)	0.39346 (5)	0.73387 (3)	0.01894 (15)
O1A	0.0035 (2)	0.11592 (14)	0.36708 (11)	0.0185 (3)
H1A	-0.0101	0.0550	0.4048	0.022*
O2A	0.1030 (2)	0.13961 (14)	0.57058 (11)	0.0198 (3)
O3A	0.3781 (2)	0.63101 (14)	0.62265 (11)	0.0168 (3)
H3A	0.3800	0.6958	0.5885	0.020*
O4A	0.2633 (2)	0.61192 (14)	0.41806 (11)	0.0174 (3)
C1A	0.1342 (3)	0.3619 (2)	0.38385 (15)	0.0142 (4)
C2A	0.0914 (3)	0.2440 (2)	0.42497 (15)	0.0149 (4)
C3A	0.1454 (3)	0.25105 (19)	0.53885 (15)	0.0142 (4)
C4A	0.2436 (3)	0.3875 (2)	0.60388 (14)	0.0139 (4)
C5A	0.2870 (3)	0.50807 (19)	0.56472 (15)	0.0134 (4)
C6A	0.2301 (3)	0.50147 (19)	0.45017 (15)	0.0132 (4)
O1B	0.5433 (2)	1.13507 (14)	0.43096 (11)	0.0189 (3)
C1B	0.4590 (3)	1.03933 (19)	0.35294 (15)	0.0147 (4)
N2B	0.3806 (3)	0.90640 (16)	0.35775 (12)	0.0158 (3)
H2BA	0.3923	0.8731	0.4149	0.019*
C3B	0.2729 (3)	0.8193 (2)	0.26005 (14)	0.0163 (4)
H3BA	0.1226	0.8081	0.2654	0.020*
H3BB	0.3141	0.7220	0.2424	0.020*
C4B	0.3395 (3)	0.90694 (19)	0.17620 (14)	0.0155 (4)
C5B	0.1561 (3)	0.9111 (2)	0.10248 (16)	0.0214 (4)

H5BA	0.0461	0.9484	0.1448	0.026*
H5BB	0.1986	0.9785	0.0568	0.026*
C6B	0.0727 (4)	0.7610 (2)	0.03391 (17)	0.0288 (5)
H6BA	0.0190	0.6956	0.0791	0.035*
H6BB	-0.0418	0.7686	-0.0143	0.035*
C7B	0.2397 (4)	0.6977 (3)	-0.02991 (17)	0.0320 (5)
H7BA	0.2827	0.7577	-0.0805	0.038*
H7BB	0.1841	0.5986	-0.0705	0.038*
C8B	0.4253 (4)	0.6919 (2)	0.04056 (17)	0.0278 (5)
H8BA	0.5350	0.6579	-0.0036	0.033*
H8BB	0.3866	0.6216	0.0847	0.033*
C9B	0.5062 (3)	0.8409 (2)	0.11141 (16)	0.0198 (4)
H9BA	0.5625	0.9073	0.0674	0.024*
H9BB	0.6193	0.8318	0.1598	0.024*
C10B	0.4297 (3)	1.0593 (2)	0.24256 (15)	0.0175 (4)
H10A	0.5621	1.0955	0.2170	0.021*
H10B	0.3340	1.1288	0.2394	0.021*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0306 (3)	0.0172 (2)	0.0145 (2)	0.00588 (19)	-0.00172 (18)	0.00410 (18)
C12	0.0277 (3)	0.0151 (2)	0.0153 (2)	0.00428 (18)	-0.00131 (18)	0.00606 (17)
O1A	0.0265 (7)	0.0099 (6)	0.0176 (7)	-0.0005 (5)	0.0004 (6)	0.0026 (5)
O2A	0.0281 (8)	0.0111 (6)	0.0211 (7)	0.0016 (6)	0.0049 (6)	0.0057 (5)
O3A	0.0228 (7)	0.0093 (6)	0.0179 (7)	-0.0007 (5)	-0.0029 (5)	0.0048 (5)
O4A	0.0214 (7)	0.0131 (7)	0.0194 (7)	0.0022 (5)	0.0020 (5)	0.0076 (5)
C1A	0.0154 (9)	0.0149 (9)	0.0141 (9)	0.0062 (7)	0.0023 (7)	0.0042 (7)
C2A	0.0144 (9)	0.0112 (8)	0.0183 (9)	0.0032 (7)	0.0023 (7)	0.0008 (7)
C3A	0.0154 (9)	0.0107 (9)	0.0184 (9)	0.0048 (7)	0.0048 (7)	0.0047 (7)
C4A	0.0168 (9)	0.0125 (9)	0.0138 (9)	0.0051 (7)	0.0017 (7)	0.0042 (7)
C5A	0.0112 (9)	0.0113 (9)	0.0184 (9)	0.0036 (7)	0.0014 (7)	0.0037 (7)
C6A	0.0122 (8)	0.0108 (9)	0.0179 (9)	0.0043 (7)	0.0027 (7)	0.0044 (7)
O1B	0.0276 (8)	0.0112 (6)	0.0166 (7)	-0.0010 (5)	-0.0015 (6)	0.0036 (5)
C1B	0.0170 (9)	0.0110 (8)	0.0166 (9)	0.0029 (7)	0.0014 (7)	0.0037 (7)
N2B	0.0236 (8)	0.0109 (7)	0.0130 (8)	0.0012 (6)	-0.0001 (6)	0.0044 (6)
C3B	0.0245 (10)	0.0116 (8)	0.0123 (9)	0.0004 (7)	0.0009 (7)	0.0032 (7)
C4B	0.0224 (10)	0.0106 (9)	0.0138 (9)	0.0024 (7)	0.0013 (7)	0.0038 (7)
C5B	0.0251 (10)	0.0216 (10)	0.0192 (10)	0.0078 (8)	-0.0004 (8)	0.0058 (8)
C6B	0.0350 (12)	0.0279 (12)	0.0208 (10)	0.0012 (9)	-0.0075 (9)	0.0034 (9)
C7B	0.0492 (15)	0.0252 (11)	0.0174 (10)	0.0041 (10)	-0.0010 (10)	-0.0022 (8)
C8B	0.0413 (13)	0.0195 (10)	0.0229 (10)	0.0115 (9)	0.0078 (9)	0.0002 (8)
C9B	0.0245 (10)	0.0173 (9)	0.0193 (9)	0.0054 (8)	0.0042 (8)	0.0060 (8)
C10B	0.0268 (10)	0.0112 (9)	0.0155 (9)	0.0030 (7)	0.0024 (8)	0.0049 (7)

*Geometric parameters (Å, °)*

C11—C1A	1.7158 (19)	C3B—H3BB	0.9900
C12—C4A	1.7196 (18)	C4B—C5B	1.534 (3)
O1A—C2A	1.330 (2)	C4B—C9B	1.538 (3)
O1A—H1A	0.8400	C4B—C10B	1.546 (2)
O2A—C3A	1.227 (2)	C5B—C6B	1.530 (3)
O3A—C5A	1.301 (2)	C5B—H5BA	0.9900
O3A—H3A	0.8400	C5B—H5BB	0.9900
O4A—C6A	1.215 (2)	C6B—C7B	1.523 (3)
C1A—C2A	1.352 (3)	C6B—H6BA	0.9900
C1A—C6A	1.465 (3)	C6B—H6BB	0.9900
C2A—C3A	1.504 (3)	C7B—C8B	1.525 (3)
C3A—C4A	1.441 (3)	C7B—H7BA	0.9900
C4A—C5A	1.361 (3)	C7B—H7BB	0.9900
C5A—C6A	1.517 (3)	C8B—C9B	1.531 (3)
O1B—C1B	1.262 (2)	C8B—H8BA	0.9900
C1B—N2B	1.318 (2)	C8B—H8BB	0.9900
C1B—C10B	1.507 (2)	C9B—H9BA	0.9900
N2B—C3B	1.460 (2)	C9B—H9BB	0.9900
N2B—H2BA	0.8800	C10B—H10A	0.9900
C3B—C4B	1.558 (2)	C10B—H10B	0.9900
C3B—H3BA	0.9900		
C2A—O1A—H1A	109.5	C10B—C4B—C3B	103.66 (14)
C5A—O3A—H3A	109.5	C6B—C5B—C4B	111.76 (17)
C2A—C1A—C6A	120.62 (17)	C6B—C5B—H5BA	109.3
C2A—C1A—C11	121.97 (15)	C4B—C5B—H5BA	109.3
C6A—C1A—C11	117.41 (14)	C6B—C5B—H5BB	109.3
O1A—C2A—C1A	122.13 (17)	C4B—C5B—H5BB	109.3
O1A—C2A—C3A	116.63 (16)	H5BA—C5B—H5BB	107.9
C1A—C2A—C3A	121.23 (16)	C7B—C6B—C5B	110.90 (19)
O2A—C3A—C4A	124.02 (17)	C7B—C6B—H6BA	109.5
O2A—C3A—C2A	117.66 (17)	C5B—C6B—H6BA	109.5
C4A—C3A—C2A	118.32 (16)	C7B—C6B—H6BB	109.5
C5A—C4A—C3A	121.67 (17)	C5B—C6B—H6BB	109.5
C5A—C4A—C12	120.66 (14)	H6BA—C6B—H6BB	108.0
C3A—C4A—C12	117.67 (14)	C6B—C7B—C8B	111.52 (18)
O3A—C5A—C4A	121.97 (17)	C6B—C7B—H7BA	109.3
O3A—C5A—C6A	118.01 (16)	C8B—C7B—H7BA	109.3
C4A—C5A—C6A	120.03 (16)	C6B—C7B—H7BB	109.3
O4A—C6A—C1A	123.11 (17)	C8B—C7B—H7BB	109.3
O4A—C6A—C5A	118.78 (16)	H7BA—C7B—H7BB	108.0
C1A—C6A—C5A	118.10 (15)	C7B—C8B—C9B	111.18 (17)
O1B—C1B—N2B	123.96 (17)	C7B—C8B—H8BA	109.4
O1B—C1B—C10B	125.82 (16)	C9B—C8B—H8BA	109.4
N2B—C1B—C10B	110.21 (16)	C7B—C8B—H8BB	109.4
C1B—N2B—C3B	114.19 (15)	C9B—C8B—H8BB	109.4



C1B—N2B—H2BA	122.9	H8BA—C8B—H8BB	108.0
C3B—N2B—H2BA	122.9	C8B—C9B—C4B	112.62 (17)
N2B—C3B—C4B	104.11 (15)	C8B—C9B—H9BA	109.1
N2B—C3B—H3BA	110.9	C4B—C9B—H9BA	109.1
C4B—C3B—H3BA	110.9	C8B—C9B—H9BB	109.1
N2B—C3B—H3BB	110.9	C4B—C9B—H9BB	109.1
C4B—C3B—H3BB	110.9	H9BA—C9B—H9BB	107.8
H3BA—C3B—H3BB	109.0	C1B—C10B—C4B	105.01 (15)
C5B—C4B—C9B	109.57 (15)	C1B—C10B—H10A	110.7
C5B—C4B—C10B	111.96 (15)	C4B—C10B—H10A	110.7
C9B—C4B—C10B	109.81 (16)	C1B—C10B—H10B	110.7
C5B—C4B—C3B	111.08 (16)	C4B—C10B—H10B	110.7
C9B—C4B—C3B	110.64 (15)	H10A—C10B—H10B	108.8
C6A—C1A—C2A—O1A	179.69 (16)	C4A—C5A—C6A—C1A	-1.8 (3)
C11—C1A—C2A—O1A	0.0 (3)	O1B—C1B—N2B—C3B	174.06 (18)
C6A—C1A—C2A—C3A	-1.6 (3)	C10B—C1B—N2B—C3B	-5.0 (2)
C11—C1A—C2A—C3A	178.76 (13)	C1B—N2B—C3B—C4B	14.0 (2)
O1A—C2A—C3A—O2A	-0.8 (3)	N2B—C3B—C4B—C5B	-136.89 (16)
C1A—C2A—C3A—O2A	-179.58 (18)	N2B—C3B—C4B—C9B	101.19 (17)
O1A—C2A—C3A—C4A	179.11 (16)	N2B—C3B—C4B—C10B	-16.48 (19)
C1A—C2A—C3A—C4A	0.3 (3)	C9B—C4B—C5B—C6B	55.7 (2)
O2A—C3A—C4A—C5A	-179.95 (18)	C10B—C4B—C5B—C6B	177.77 (17)
C2A—C3A—C4A—C5A	0.2 (3)	C3B—C4B—C5B—C6B	-66.9 (2)
O2A—C3A—C4A—C12	0.0 (3)	C4B—C5B—C6B—C7B	-57.1 (2)
C2A—C3A—C4A—C12	-179.94 (13)	C5B—C6B—C7B—C8B	55.9 (2)
C3A—C4A—C5A—O3A	-179.05 (16)	C6B—C7B—C8B—C9B	-54.4 (3)
C12—C4A—C5A—O3A	1.1 (3)	C7B—C8B—C9B—C4B	54.4 (2)
C3A—C4A—C5A—C6A	0.6 (3)	C5B—C4B—C9B—C8B	-54.5 (2)
C12—C4A—C5A—C6A	-179.28 (13)	C10B—C4B—C9B—C8B	-177.91 (16)
C2A—C1A—C6A—O4A	-176.63 (18)	C3B—C4B—C9B—C8B	68.3 (2)
C11—C1A—C6A—O4A	3.0 (3)	O1B—C1B—C10B—C4B	174.58 (18)
C2A—C1A—C6A—C5A	2.3 (3)	N2B—C1B—C10B—C4B	-6.4 (2)
C11—C1A—C6A—C5A	-178.02 (13)	C5B—C4B—C10B—C1B	133.80 (17)
O3A—C5A—C6A—O4A	-3.2 (3)	C9B—C4B—C10B—C1B	-104.25 (17)
C4A—C5A—C6A—O4A	177.16 (17)	C3B—C4B—C10B—C1B	13.99 (19)
O3A—C5A—C6A—C1A	177.86 (15)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1A—H1A $\cdots$ O2A <sup>i</sup>	0.84	1.97	2.7493 (19)	153
O1A—H1A $\cdots$ O2A	0.84	2.20	2.671 (2)	115
O3A—H3A $\cdots$ O1B <sup>ii</sup>	0.84	1.70	2.4807 (19)	153
O3A—H3A $\cdots$ O4A	0.84	2.26	2.7148 (19)	114

$N2B-H2BA\cdots O1B^{ii}$	0.88	2.07	2.913 (2)	161
$N2B-H2BA\cdots O4A$	0.88	2.53	3.091 (2)	122

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