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Crystal Structure
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A comparison of 3,4,6a,7,10,10a-hexahydro-7,10-epoxyprymido[2,1-*a*]-isoindol-6(2*H*)-one and 2-(2-aminoethyl)-3a,4,7,7a-tetrahydro-1*H*-4,7-epoxyisoindole-1,3(2*H*)-dione: structural and reactivity differences of two homologous tricyclic imides

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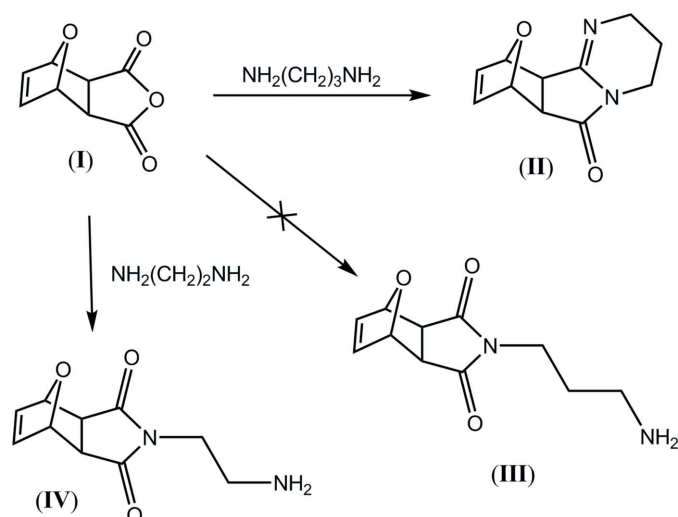
The crystal structures of 3,4,6a,7,10,10a-hexahydro-7,10-epoxyprymido[2,1-*a*]isoindol-6(2*H*)-one, C₁₁H₁₂N₂O₂, and 2-(2-aminoethyl)-3a,4,7,7a-tetrahydro-1*H*-4,7-epoxyisoindole-1,3(2*H*)-dione, C₁₀H₁₂N₂O₃, two tricyclic imides, show one and two molecules in the asymmetric unit, respectively. Intermolecular hydrogen-bonding interactions are observed in both compounds.

Comment

3,4,6a,7,10,10a-Hexahydro-7,10-epoxyprymido[2,1-*a*]isoindol-6(2*H*)-one, (II), is an important intermediate in the synthesis of phloeodictine A1, which has been shown to have antimicrobial properties (Neubert & Snider, 2003), as well as exhibiting significant cytotoxicity towards KB human nasopharyngeal carcinoma cells (Kourany-Lefoll *et al.*, 1992). Additionally, cyclic amidines are commonly used in medicinal chemistry (Hellal *et al.*, 2006). However, despite these important applications, the crystal structures of these compounds have not been well studied. In this work, 2-(2-aminoethyl)-3a,4,7,7a-tetrahydro-1*H*-4,7-epoxyisoindole-1,3(2*H*)-dione, (IV), serves as a model for understanding the preferential formation of the cyclic compound, (II), *via* intramolecular condensation over the homologous acyclic precursor 2-(3-aminopropyl)-3a,4,7,7a-tetrahydro-1*H*-4,7-epoxyisoindole-1,3(2*H*)-dione, (III) (see Scheme), and for probing how the formation of the amidine ring influences the structural and electronic properties of this biologically relevant molecule.

In initial work focused on preparing (III) from (I) (see Scheme), ¹H NMR data indicated a break in symmetry

between atoms H3 and H6 and between atoms H2 and H7. This suggested that the proton coupling could be affected by hydrogen bonding between the amine H atoms and one of the carbonyl O atoms, or that the actual structure differed from theoretical expectations altogether. Mass spectrometry was used to probe the first hypothesis and revealed that H₂O elimination had occurred, indicating that the desired product was not obtained. However, through single-crystal X-ray crystallography, we were able to identify the material as compound (II) (Fig. 1), an unexpected yet interesting product of the reaction. Notably, model compound (IV) (Fig. 2) was easily obtained by substituting ethylenediamine for propane-1,3-diamine during the synthesis, demonstrating that the longer carbon chain is required for facile formation of the amidine ring.



Compound (IV) contains two molecules in the asymmetric unit, which are hydrogen-bonded dimers with a noncrystallographic inversion center at $x = \frac{1}{4}$, $y = 0.53$ (near $\frac{1}{2}$) and $z = 0.12$ (near $\frac{1}{8}$). Molecules (IVA) and (IVB) are nearly identical, with the exception of the C3–C4 bond lengths [C3A–C4A = 1.518 (2) Å and C3B–C4B = 1.509 (2) Å]. When comparing this bond with the analogous C5–C6 bonds, molecules (IVA) and (IVB) both have a bond length of 1.517 (2) Å. Looking at the molecule of (II), the C3–C4 bond length is 1.5185 (17) Å.

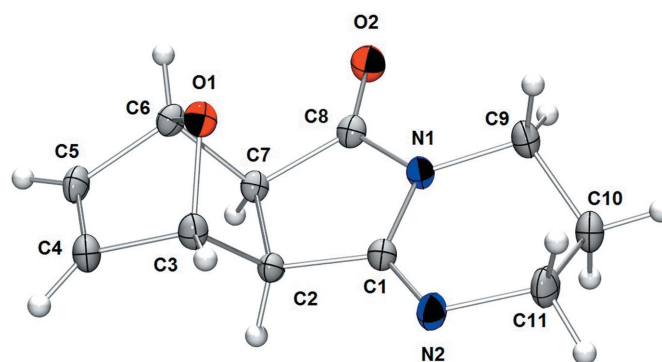


Figure 1
The molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

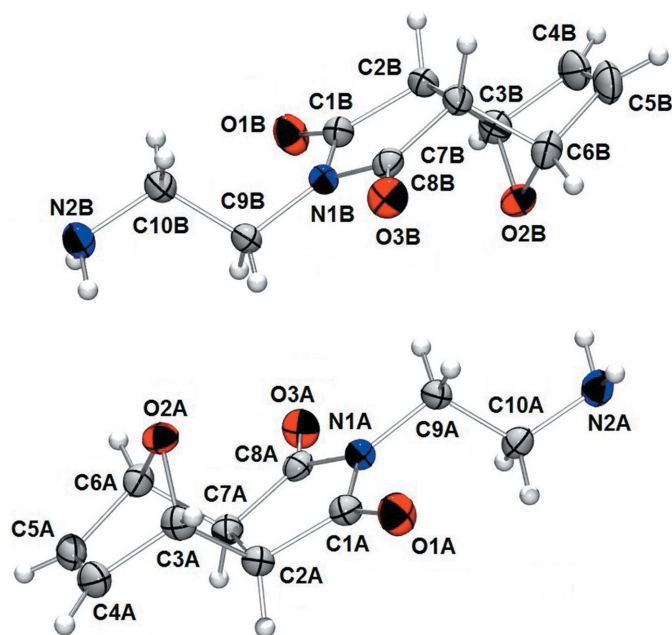


Figure 2

The two independent molecules of (IV), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

This leads us to believe that C3B–C4B has undergone some distortion.

With the exception of C3B–C4B, the bond lengths and angles in the bicyclic ring for both (II) and (IV) agree with previously reported compounds containing the same functionality (Trujillo-Ferrara *et al.*, 2004; Tan *et al.*, 2012). Therefore, further discussion will focus on the amidine and central five-membered rings. However, it is also important to note that the bicyclic ring, with inherent internal ring strain, gives the molecules ROMP (ring-opening metathesis polymerization) reactivity, making these compounds available for uses in polymer chemistry (Trnka & Grubbs, 2001; Runge & Bowden, 2007).

When comparing the angles and bond lengths of the central five-membered ring of (II) (see Table 1) with those of (IV) (see Table 2), it appears that no distortion has occurred due to the formation of the amidine ring. The C7–C8–N1 angle of (II) and the C7A–C8A–N1A angle of (IV) are 108.68 (10) and 108.63 (12)°, respectively. Additionally, the C1–N1–C8 angle in (II) [113.35 (9)°] and the C1A–N1A–C8A angle in (IV) [112.94 (12)°] are approximately equal, supporting the conjecture that little or no distortion has occurred. The N2–C1–N1 angle in (II) is 126.67 (11)°, which is slightly greater than the average N–C=N angle (121.53°) reported by Kosturkiewicz *et al.* (1992) for 28 amidine complexes. This is due to the additional flexibility during the formation of the compound, which is not uncommon for ring-fused cyclic amidine complexes. In fact, 2-[(1,3-benzodioxol-5-yl)methylene]-6,7-dihydro-5H-thiazolo[3,2-*a*]pyrimidin-3-one was reported to have an even larger N–C=N bond angle of 128.49 (18)° (Liang, 2004).

In both (II) and (IV), hydrogen bonding dictates the packing structure. In (II), there are no classical hydrogen

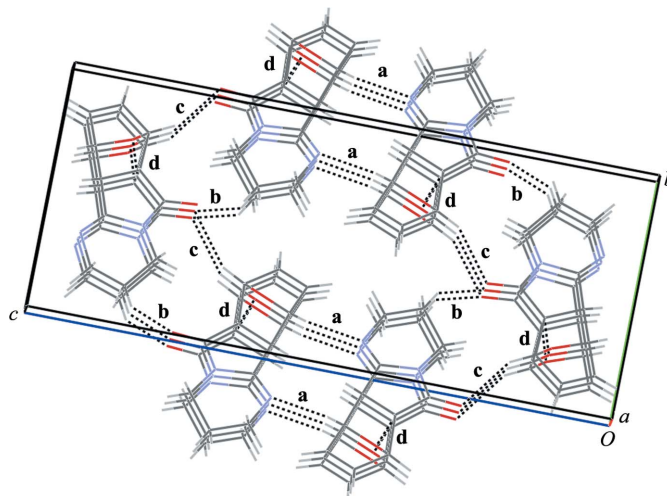


Figure 3

A three-dimensional packing diagram for (II), viewed along the *a* axis. Intermolecular hydrogen bonding (dashed lines) is shown between atoms N2 and H3 (marked **a**), O2 and H10B (**b**), O2 and H6 (**c**), and O1 and H7 (**d**).

bonds, but weaker interactions [compared with N–H···O hydrogen bonds in (IV)], such as C–H···O and C–H···N, exist between the molecules. Compound (II) packs in a head-to-tail fashion (see bond **d** in Fig. 3) with respect to the ether groups, with intermolecular hydrogen bonding occurring between atoms O1 and H7, while compound (IV) packs in a head-to-head fashion (see bonds **a** and **b** in Fig. 4), with hydrogen bonding present between atoms O2A and H11D and between O2B and H11B. This bonding structure causes a stair step in the packing of (II). In (II) (see bonds **a–c** in Fig. 3), the molecule is inverted in each row, so that bonding occurs in a head-to-head/tail-to-tail fashion, resulting in hydrogen

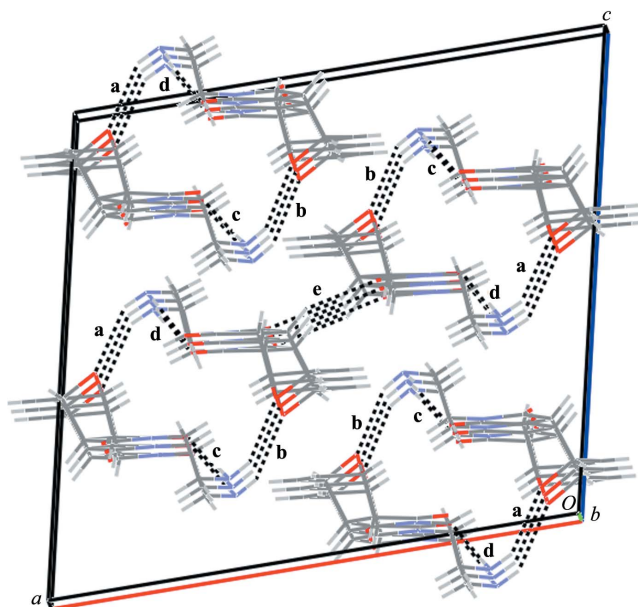


Figure 4

A three-dimensional packing diagram for (IV), viewed along the *b* axis. Intermolecular hydrogen bonding (dashed lines) is shown between atoms O2A and H11D (marked **a**), O2B and H11B (**b**), O3A and H11A (**c**), O3B and H11C (**d**), and O1B and H2B (**e**).

bonding occurring between atoms N2 and H3, between O2 and H6, and between O2 and H10B of adjacent molecules. The bonds between atoms N2 and H3 are significantly closer to linearity, as indicated by the bonding angle (see Table 3), which indicates that these bonds are stronger and therefore dictate the packing structure. In (IV), the bonds closest to linearity are between the carbonyl groups and the amine H atoms (see Table 4). Intermolecular hydrogen bonding also occurs between atoms O3B and H11C, and between O3A and H11A (see bonds **c** and **d** in Fig. 4).

In addition to affecting the structural properties of (II), the formation of the amidine ring seems to have had notable perturbations on the distribution of charge compared with that of the other ring-fused amidine compounds. The N—C and C=N bond lengths are highly indicative of electron-density distribution across bonds. Previous studies reported the difference between the bond lengths of the analogous N—C and C=N bonds in the amidine rings (Δ bond distance) as, on average, 0.0670 Å for the 28 compounds studied (Kosturkiewicz *et al.*, 1992), while the Δ bond distance in (II) was measured as 0.133 Å. From these data it was concluded that, compared with other ring-fused amidine complexes, (II) exhibits very little delocalization across the N—C and C=N bonds. Kosturkiewicz *et al.* (1992) speculated that the low level of delocalization is caused by the presence of an *R* group at the central C atom, which is consistent with the five-membered ring attached to atom C1 in (II). Other cyclic amidines also display large Δ bond distances, including 2-[(1,3-benzodioxol-5-yl)methylene]-6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-one, where the Δ bond distance is 0.131 Å (Liang, 2004).

This large discrepancy in the N—C bond lengths also contributes to the previously discussed higher-than-average N1—C1—N2 angle in the six-membered ring of (II). If there were a higher degree of delocalization across the N1—C1—N2 unit, it would be expected that the C10—C9—N1 [107.25 (10)°] and N2—C11—C10 [114.08 (10)°] bond angles would be equivalent, but this is not the case. The same reasoning can be applied when considering the C9—N1—C1 [121.17 (9)°] and C1—N2—C11 [116.62 (11)°] angles.

In conclusion, we have determined that the formation of cyclic compound (II) does not cause noticeable perturbations in the structure of the bicyclic ring or the central five-membered ring, compared with the linear compound, (IV). However, the formation of the cyclic compound does lead to perturbations in the N—C=N units, as indicated by the large bond angle and the large discrepancy in bond lengths between the N—C and C=N bonds.

Experimental

Propane-1,3-diamine (14.9 g, 201.0 mmol) and 3a,4,7,7a-tetrahydro-4,7-epoxyisobenzofuran-1,3-dione (4.8 g, 28.9 mmol), (I), were refluxed at 353 K for 2 h. The resulting solution was poured into H₂O and extracted with dichloromethane (2 × 30 ml). The combined organic layers were dried with Na₂SO₄ and concentrated. A translucent solid was obtained by recrystallization from a solution in toluene to yield (II) [yield 23.5%; m.p. 393 K (decomposition)]. Crystals of (II) suitable for X-ray diffraction were obtained by slow

Table 1

Bond lengths and angles for the central five-membered ring (Å, °) of (II).

C1—C2	1.5100 (17)	N1—C8	1.3722 (16)
C2—C7	1.5485 (15)	O2—C8	1.2212 (15)
C7—C8	1.5147 (16)		
C1—C2—C7	104.45 (9)	N1—C8—C7	108.68 (10)
C2—C7—C8	104.99 (9)	C8—N1—C1	113.35 (9)
O2—C8—C7	126.84 (11)	N1—C1—C2	108.46 (9)
O2—C8—N1	124.47 (11)		

Table 2

Bond lengths and angles for the central five-membered ring (Å, °) of (IV).

C1A—C2A	1.509 (2)	C8A—N1A	1.3858 (19)
C2A—C7A	1.547 (2)	C8A—O3A	1.2197 (18)
C7A—C8A	1.518 (2)	C1A—N1A	1.3877 (19)
C1A—C2A—C7A	104.74 (12)	N1A—C8A—C7A	108.63 (12)
C8A—C7A—C2A	104.56 (11)	C8A—N1A—C1A	112.94 (12)
O3A—C8A—C7A	127.27 (14)	N1A—C1A—C2A	108.91 (12)
O3A—C8A—N1A	124.08 (14)		

evaporation from a saturated solution of (II) in dichloromethane that was layered with *n*-heptane. ¹H NMR and ¹³C{¹H} NMR data are available in the *Supplementary materials*.

Compound (IV) was prepared in an analogous manner to (II), using ethylenediamine (5 ml, 74.9 mmol) and (I) (5.0 g, 30.1 mmol). Compound (IV) was obtained as a white solid by recrystallization from a mixture of dichloromethane and ether (1:3 *v/v*) [yield 9.9%; m.p. 399 K (decomposition)]. Crystals of (IV) suitable for X-ray diffraction were obtained by precipitation from a dichloromethane–ether (1:3 *v/v*) solution at 253 K. ¹H NMR (300 MHz, CDCl₃): δ 6.45 (*s*, 2H, H4A and H5A), 5.20 (*s*, 2H, H3A and H6A), 3.48 (*t*, 2H, *J* = 6.3, H9A and H9B), 2.80 (*t*, 4H, *J* = 6.3 Hz, H2A, H7A, H10A and H10B), 1.01 (*br*, 2H, H11A and H11B); ¹³C{¹H} NMR (75 MHz, CDCl₃): δ 176.4 (C1A and C8A), 136.4 (C4A and C5A), 80.8 (C3A and C6A), 47.3 (C2A and C7A), 41.9 (C9A), 39.7 (C10A).

Compound (II)

Crystal data

C ₁₁ H ₁₂ N ₂ O ₂	<i>V</i> = 952.8 (6) Å ³
<i>M_r</i> = 204.23	<i>Z</i> = 4
Monoclinic, <i>P</i> 2 ₁ /c	Mo <i>K</i> α radiation
<i>a</i> = 4.916 (2) Å	μ = 0.10 mm ⁻¹
<i>b</i> = 8.998 (3) Å	<i>T</i> = 100 K
<i>c</i> = 21.604 (6) Å	0.38 × 0.14 × 0.09 mm
β = 94.445 (7)°	

Data collection

Rigaku AFC12 with Saturn 724+ CCD area-detector diffractometer	21174 measured reflections 2193 independent reflections 2031 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (<i>ABSCOR</i> ; Higashi, 1999)	<i>R</i> _{int} = 0.031
<i>T</i> _{min} = 0.822, <i>T</i> _{max} = 1.000	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	136 parameters
$wR(F^2) = 0.100$	H-atom parameters constrained
<i>S</i> = 1.05	$\Delta\rho_{\max} = 0.36 \text{ e \AA}^{-3}$
2193 reflections	$\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$

Table 3
Hydrogen-bond geometry (Å, °) for (II).

<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>
C3—H3...N2 ⁱ	0.98	2.61	3.570 (4)	165
C6—H6...O2 ⁱⁱ	0.98	2.48	3.3421 (19)	147
C7—H7...O1 ⁱⁱⁱ	0.98	2.32	3.2040 (18)	150
C10—H10B...O2 ^{iv}	0.97	2.53	3.393 (4)	148

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

Table 4
Hydrogen-bond geometry (Å, °) for (IV).

<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>
N2A—H11A...O3A ⁱ	0.95 (2)	2.42 (2)	3.3599 (18)	169.1 (18)
N2A—H11B...O2B	0.89 (2)	2.33 (2)	3.1480 (18)	151.8 (17)
N2B—H11C...O3B ⁱⁱ	0.92 (2)	2.30 (2)	3.2175 (19)	174.4 (18)
N2B—H11D...O2A	0.93 (2)	2.45 (2)	3.2838 (19)	148.7 (19)
C2B—H2B...O1B ⁱⁱⁱ	0.98	2.52	3.408 (2)	151

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

Compound (IV)*Crystal data*

C₁₀H₁₂N₂O₃ $V = 1941.9 (3) \text{ \AA}^3$
 $M_r = 208.22$ $Z = 8$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 17.840 (2) \text{ \AA}$ $\mu = 0.11 \text{ mm}^{-1}$
 $b = 6.8747 (6) \text{ \AA}$ $T = 153 \text{ K}$
 $c = 16.2125 (17) \text{ \AA}$ $0.48 \times 0.25 \times 0.07 \text{ mm}$
 $\beta = 102.419 (5)^\circ$

Data collection

Rigaku Mercury2 diffractometer 19826 measured reflections
 Absorption correction: multi-scan 4439 independent reflections
 (*ABSCOR*; Higashi, 1999) 3478 reflections with $I > 2\sigma(I)$
 $T_{\min} = 0.923$, $T_{\max} = 1.000$ $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$ H atoms treated by a mixture of
 $wR(F^2) = 0.125$ independent and constrained
 $S = 1.07$ refinement
 4439 reflections $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 287 parameters $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

The amine H atoms were located in a difference Fourier map and both positional and isotropic displacement parameters were refined. All other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

For both compounds, data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SIR97* within *WinGX* (Farrugia, 2012); molecular graphics: *ORTEP-3* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF3191). Services for accessing these data are described at the back of the journal.

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Hellal, M., Bihel, F., Mongoet, A. & Bourguignon, J. (2006). *Org. Biomol. Chem.* **4**, 3142–3146.
- Higashi, T. (1999). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Kosturkiewicz, Z., Ciszak, E. & Tykarska, E. (1992). *Acta Cryst.* **B48**, 471–476.
- Kourany-Lefoll, E., Pais, E., Sevenet, T., Guittet, E., Montagnac, A., Fontain, C., Guenard, D. & Adeline, M. T. (1992). *J. Org. Chem.* **57**, 3832–3835.
- Liang, Z.-P. (2004). *Acta Cryst.* **E60**, o339–o340.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Neubert, B. J. & Snider, B. B. (2003). *Org. Lett.* **5**, 765–768.
- Rigaku (2008). *CrystalClear*. Rigaku Americas Corporation, The Woodlands, Texas, USA.
- Runge, M. B. & Bowden, N. B. (2007). *J. Am. Chem. Soc.* **129**, 10551–10560.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tan, X.-J., Li, C.-G., Xing, D.-X. & Liu, Y. (2012). *Acta Cryst.* **E68**, o866.
- Trnka, T. M. & Gurbbs, R. H. (2001). *Acc. Chem. Res.* **34**, 18–29.
- Trujillo-Ferrara, J. G., García-Báez, E. V., Padilla-Martínez, I. I., Martínez-Martínez, F. J. & Farfan-García, N. (2004). *Acta Cryst.* **C60**, o427–o430.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2013). C69, 638-641 [doi:10.1107/S0108270113010706]

A comparison of 3,4,6a,7,10,10a-hexahydro-7,10-epoxypyrimido[2,1-a]isoindol-6(2H)-one and 2-(2-aminoethyl)-3a,4,7,7a-tetrahydro-1H-4,7-epoxyisoindole-1,3(2H)-dione: structural and reactivity differences of two homologous tricyclic imides

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(II) 3,4,6a,7,10,10a-Hexahydro-7,10-epoxypyrimido[2,1-a]isoindol-6(2H)-one

Crystal data

$C_{11}H_{12}N_2O_2$

$M_r = 204.23$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 4.916$ (2) Å

$b = 8.998$ (3) Å

$c = 21.604$ (6) Å

$\beta = 94.445$ (7)°

$V = 952.8$ (6) Å³

$Z = 4$

$F(000) = 432$

$D_x = 1.424$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å

Cell parameters from 21982 reflections

$\theta = 7.3$ – 55.0 °

$\mu = 0.10$ mm⁻¹

$T = 100$ K

Block, colourless

$0.38 \times 0.14 \times 0.09$ mm

Data collection

Rigaku AFC12 with Saturn 724+ CCD area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1999)

$T_{\min} = 0.822$, $T_{\max} = 1.000$

21174 measured reflections

2193 independent reflections

2031 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.6$ °

$h = -6 \rightarrow 6$

$k = -11 \rightarrow 11$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.100$

$S = 1.05$

2193 reflections

136 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

$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.4632P]$

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

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

$\Delta\rho_{\max} = 0.36$ e Å⁻³

$\Delta\rho_{\min} = -0.18$ e Å⁻³

Special details

Experimental. ^1H NMR (300 MHz, CDCl_3 , δ , p.p.m.): 6.44 (s, 2H), 5.20 (s, 1H), 5.16 (s, 1H), 3.52 (t, 2H, $J = 6.0$), 3.47 (t, 2H, $J = 5.6$), 2.85 (d, 1H, $J = 7.2$), 2.69 (d, 1H, $J = 6.9$), 1.81–1.73 (m, 2H); $^{13}\text{C}\{^1\text{H}\}$ NMR (300 MHz, CDCl_3 , δ , p.p.m.): 173.4, 155.3, 136.0 (CH), 82.4 (CH), 80.1 (CH), 46.4 (CH), 45.3 (CH), 44.3 (CH_2), 37.5 (CH_2), 18.7 (CH_2). HRMS (CI+), calculated for $\text{C}_{11}\text{H}_{12}\text{O}_2\text{N}_2$: $[M + \text{H}]^+$ 205.0977; found: 205.0977.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	−0.0103 (3)	0.26831 (14)	0.40558 (6)	0.0228 (3)
H11B	0.0009	0.3512	0.4344	0.027*
H11A	−0.2009	0.2404	0.3988	0.027*
C10	0.0904 (3)	0.31934 (13)	0.34407 (6)	0.0230 (3)
H10B	−0.0324	0.3941	0.3255	0.028*
H10A	0.2699	0.3637	0.3514	0.028*
C9	0.1047 (3)	0.18828 (13)	0.29981 (6)	0.0212 (3)
H9A	−0.0775	0.1533	0.2868	0.025*
H9B	0.1938	0.2177	0.2632	0.025*
C8	0.4126 (2)	−0.03622 (13)	0.30657 (5)	0.0173 (2)
C7	0.5411 (2)	−0.13572 (12)	0.35729 (5)	0.0159 (2)
H7	0.7407	−0.1378	0.3583	0.019*
C6	0.4117 (2)	−0.29402 (12)	0.35416 (5)	0.0167 (2)
H6	0.4043	−0.3421	0.3134	0.020*
C5	0.5539 (2)	−0.38203 (13)	0.40695 (6)	0.0196 (2)
H5	0.6827	−0.4569	0.4039	0.024*
C4	0.4574 (2)	−0.33056 (13)	0.45853 (6)	0.0208 (3)
H4	0.5027	−0.3625	0.4990	0.025*
C3	0.2574 (2)	−0.20859 (13)	0.43766 (5)	0.0182 (2)
H3	0.1207	−0.1844	0.4668	0.022*
C2	0.4360 (2)	−0.07520 (12)	0.41816 (5)	0.0161 (2)
H2	0.5839	−0.0507	0.4495	0.019*
C1	0.2649 (2)	0.05812 (12)	0.39800 (5)	0.0163 (2)
N1	0.2610 (2)	0.07085 (11)	0.33328 (4)	0.0176 (2)
N2	0.1435 (2)	0.14183 (11)	0.43429 (5)	0.0208 (2)
O1	0.15021 (16)	−0.26290 (9)	0.37768 (4)	0.0184 (2)
O2	0.43549 (19)	−0.04727 (10)	0.25087 (4)	0.0246 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0250 (6)	0.0193 (6)	0.0246 (6)	0.0092 (5)	0.0052 (5)	0.0013 (5)
C10	0.0246 (6)	0.0171 (6)	0.0276 (6)	0.0049 (5)	0.0027 (5)	0.0040 (5)

C9	0.0224 (6)	0.0198 (6)	0.0213 (6)	0.0046 (5)	0.0016 (4)	0.0053 (4)
C8	0.0154 (5)	0.0168 (5)	0.0202 (5)	-0.0013 (4)	0.0042 (4)	0.0013 (4)
C7	0.0128 (5)	0.0163 (5)	0.0187 (5)	0.0015 (4)	0.0031 (4)	0.0002 (4)
C6	0.0156 (5)	0.0157 (5)	0.0193 (5)	0.0020 (4)	0.0044 (4)	-0.0014 (4)
C5	0.0175 (5)	0.0141 (5)	0.0274 (6)	0.0035 (4)	0.0022 (4)	0.0017 (4)
C4	0.0234 (6)	0.0161 (5)	0.0227 (6)	0.0027 (4)	0.0009 (5)	0.0041 (4)
C3	0.0204 (5)	0.0174 (5)	0.0175 (5)	0.0038 (4)	0.0058 (4)	0.0021 (4)
C2	0.0171 (5)	0.0150 (5)	0.0160 (5)	0.0028 (4)	0.0003 (4)	-0.0006 (4)
C1	0.0162 (5)	0.0150 (5)	0.0179 (5)	0.0005 (4)	0.0017 (4)	0.0007 (4)
N1	0.0191 (5)	0.0166 (5)	0.0171 (5)	0.0030 (4)	0.0021 (4)	0.0020 (4)
N2	0.0243 (5)	0.0174 (5)	0.0212 (5)	0.0058 (4)	0.0045 (4)	0.0004 (4)
O1	0.0131 (4)	0.0196 (4)	0.0227 (4)	0.0009 (3)	0.0027 (3)	0.0006 (3)
O2	0.0316 (5)	0.0250 (5)	0.0181 (4)	0.0024 (4)	0.0080 (4)	0.0016 (3)

Geometric parameters (Å, °)

C1—C2	1.5100 (17)	C7—C6	1.5591 (17)
C2—C7	1.5485 (15)	C7—H7	0.9800
C7—C8	1.5147 (16)	C6—O1	1.4460 (18)
N1—C8	1.3722 (16)	C6—C5	1.5143 (16)
O2—C8	1.2212 (15)	C6—H6	0.9800
C11—N2	1.4762 (16)	C5—C4	1.3279 (17)
C11—C10	1.5245 (17)	C5—H5	0.9300
C11—H11B	0.9700	C4—C3	1.5185 (17)
C11—H11A	0.9700	C4—H4	0.9300
C10—C9	1.5233 (18)	C3—O1	1.4455 (14)
C10—H10B	0.9700	C3—C2	1.5642 (17)
C10—H10A	0.9700	C3—H3	0.9800
C9—N1	1.4642 (15)	C2—H2	0.9800
C9—H9A	0.9700	C1—N2	1.2690 (16)
C9—H9B	0.9700	C1—N1	1.4017 (15)
C1—C2—C7	104.45 (9)	O1—C6—C5	101.99 (9)
C2—C7—C8	104.99 (9)	O1—C6—C7	100.29 (9)
O2—C8—C7	126.84 (11)	C5—C6—C7	106.39 (10)
O2—C8—N1	124.47 (11)	O1—C6—H6	115.4
N1—C8—C7	108.68 (10)	C5—C6—H6	115.4
C8—N1—C1	113.35 (9)	C7—C6—H6	115.4
N1—C1—C2	108.46 (9)	C4—C5—C6	105.99 (10)
N2—C11—C10	114.08 (10)	C4—C5—H5	127.0
N2—C11—H11B	108.7	C6—C5—H5	127.0
C10—C11—H11B	108.7	C5—C4—C3	105.56 (10)
N2—C11—H11A	108.7	C5—C4—H4	127.2
C10—C11—H11A	108.7	C3—C4—H4	127.2
H11B—C11—H11A	107.6	O1—C3—C4	101.80 (9)
C9—C10—C11	110.36 (10)	O1—C3—C2	101.02 (9)
C9—C10—H10B	109.6	C4—C3—C2	105.69 (11)
C11—C10—H10B	109.6	O1—C3—H3	115.5
C9—C10—H10A	109.6	C4—C3—H3	115.5
C11—C10—H10A	109.6	C2—C3—H3	115.5

H10B—C10—H10A	108.1	C1—C2—C7	104.45 (9)
N1—C9—C10	107.25 (10)	C1—C2—C3	112.04 (11)
N1—C9—H9A	110.3	C7—C2—C3	100.96 (9)
C10—C9—H9A	110.3	C1—C2—H2	112.8
N1—C9—H9B	110.3	C7—C2—H2	112.8
C10—C9—H9B	110.3	C3—C2—H2	112.8
H9A—C9—H9B	108.5	N2—C1—N1	126.67 (11)
C8—C7—C6	111.27 (10)	N2—C1—C2	124.86 (11)
C2—C7—C6	101.33 (9)	C8—N1—C9	125.47 (10)
C8—C7—H7	112.8	C1—N1—C9	121.17 (9)
C2—C7—H7	112.8	C1—N2—C11	116.62 (11)
C6—C7—H7	112.8	C3—O1—C6	96.21 (9)
N2—C11—C10—C9	52.43 (15)	C4—C3—C2—C7	71.22 (11)
C11—C10—C9—N1	-51.50 (14)	C7—C2—C1—N2	-178.87 (11)
O2—C8—C7—C2	-177.88 (11)	C3—C2—C1—N2	72.70 (15)
N1—C8—C7—C2	1.78 (12)	C7—C2—C1—N1	2.03 (12)
O2—C8—C7—C6	-69.07 (16)	C3—C2—C1—N1	-106.39 (10)
N1—C8—C7—C6	110.59 (11)	O2—C8—N1—C1	179.14 (11)
C8—C7—C6—O1	-73.71 (11)	C7—C8—N1—C1	-0.53 (13)
C2—C7—C6—O1	37.46 (10)	O2—C8—N1—C9	-0.25 (19)
C8—C7—C6—C5	-179.57 (9)	C7—C8—N1—C9	-179.92 (10)
C2—C7—C6—C5	-68.40 (11)	N2—C1—N1—C8	179.92 (11)
O1—C6—C5—C4	-31.12 (12)	C2—C1—N1—C8	-1.00 (13)
C7—C6—C5—C4	73.52 (13)	N2—C1—N1—C9	-0.66 (18)
C6—C5—C4—C3	-0.78 (12)	C2—C1—N1—C9	178.41 (10)
C5—C4—C3—O1	32.45 (12)	C10—C9—N1—C8	-152.25 (11)
C5—C4—C3—C2	-72.72 (12)	C10—C9—N1—C1	28.41 (14)
C8—C7—C2—C1	-2.26 (11)	N1—C1—N2—C11	-2.07 (18)
C6—C7—C2—C1	-118.15 (10)	C2—C1—N2—C11	179.00 (11)
C8—C7—C2—C3	114.14 (11)	C10—C11—N2—C1	-24.52 (16)
C6—C7—C2—C3	-1.75 (10)	C4—C3—O1—C6	-49.60 (10)
O1—C3—C2—C1	76.14 (10)	C2—C3—O1—C6	59.19 (10)
C4—C3—C2—C1	-178.13 (9)	C5—C6—O1—C3	49.17 (10)
O1—C3—C2—C7	-34.51 (10)	C7—C6—O1—C3	-60.20 (9)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots N2 ⁱ	0.98	2.61	3.570 (4)	165
C6—H6 \cdots O2 ⁱⁱ	0.98	2.48	3.3421 (19)	147
C7—H7 \cdots O1 ⁱⁱⁱ	0.98	2.32	3.2040 (18)	150
C10—H10B \cdots O2 ^{iv}	0.97	2.53	3.393 (4)	148

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

(IV) 2-(2-Aminoethyl)-3a,4,7,7a-tetrahydro-1H-4,7-epoxyisoindole-1,3(2H)-dione

Crystal data

$C_{10}H_{12}N_2O_3$	$F(000) = 880$
$M_r = 208.22$	$D_x = 1.424 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71075 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 4078 reflections
$a = 17.840 (2) \text{ \AA}$	$\theta = 2.6\text{--}27.5^\circ$
$b = 6.8747 (6) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 16.2125 (17) \text{ \AA}$	$T = 153 \text{ K}$
$\beta = 102.419 (5)^\circ$	Prism, colourless
$V = 1941.9 (3) \text{ \AA}^3$	$0.48 \times 0.25 \times 0.07 \text{ mm}$
$Z = 8$	

Data collection

Rigaku Mercury2 diffractometer	19826 measured reflections
Radiation source: fine-focus sealed tube	4439 independent reflections
Graphite monochromator	3478 reflections with $I > 2\sigma(I)$
Detector resolution: 13.6612 pixels mm^{-1}	$R_{\text{int}} = 0.033$
ω scans	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (<i>ABSCOR</i> ; Higashi, 1999)	$h = -23 \rightarrow 23$
$T_{\text{min}} = 0.923$, $T_{\text{max}} = 1.000$	$k = -8 \rightarrow 8$
	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.7213P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
4439 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
287 parameters	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. HRMS (CI⁺), calculated for $C_{10}H_{13}O_3N_2$: $[M + H]^+$ 209.0921; found: 209.0922. Analysis, calculated for $C_{10}H_{12}O_3N_2$: C 57.68, H 5.81, N 13.45; found: C 57.27, H 5.51, N 12.96.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1A	0.11191 (8)	0.5549 (2)	0.21220 (9)	0.0245 (3)

C8B	0.30204 (9)	0.7661 (2)	0.03009 (9)	0.0236 (3)
C2A	0.05896 (8)	0.3813 (2)	0.19748 (9)	0.0240 (3)
H2A	0.0264	0.3709	0.2389	0.029*
C7B	0.38202 (9)	0.8530 (2)	0.04465 (9)	0.0240 (3)
H7B	0.3878	0.9480	0.0014	0.029*
C3A	0.01235 (8)	0.3770 (2)	0.10399 (9)	0.0270 (3)
H3A	-0.0135	0.4988	0.0833	0.032*
C6B	0.40781 (9)	0.9334 (2)	0.13638 (9)	0.0292 (3)
H6B	0.3716	1.0224	0.1546	0.035*
C4A	-0.03908 (9)	0.1990 (3)	0.09612 (10)	0.0312 (4)
H4A	-0.0920	0.1969	0.0913	0.037*
C5B	0.48865 (10)	1.0111 (3)	0.14447 (10)	0.0375 (4)
H5B	0.5037	1.1408	0.1464	0.045*
C5A	0.00715 (9)	0.0472 (2)	0.09755 (9)	0.0303 (4)
H5A	-0.0067	-0.0834	0.0943	0.036*
C4B	0.53341 (10)	0.8565 (3)	0.14828 (11)	0.0368 (4)
H4B	0.5864	0.8554	0.1538	0.044*
C6A	0.08715 (9)	0.1305 (2)	0.10547 (9)	0.0257 (3)
H6A	0.1239	0.0454	0.0859	0.031*
C3B	0.48096 (9)	0.6824 (2)	0.14183 (10)	0.0285 (3)
H3B	0.5057	0.5604	0.1640	0.034*
C7A	0.11273 (8)	0.2044 (2)	0.19850 (9)	0.0235 (3)
H7A	0.1067	0.1060	0.2403	0.028*
C2B	0.43554 (8)	0.6755 (2)	0.04771 (9)	0.0250 (3)
H2B	0.4690	0.6848	0.0072	0.030*
C8A	0.19280 (8)	0.2911 (2)	0.21489 (8)	0.0232 (3)
C1B	0.38243 (9)	0.5025 (2)	0.03199 (9)	0.0252 (3)
C9A	0.25164 (8)	0.6263 (2)	0.22864 (9)	0.0259 (3)
H9A	0.2357	0.7442	0.1969	0.031*
H9B	0.2925	0.5673	0.2063	0.031*
C9B	0.24321 (9)	0.4292 (2)	0.01472 (9)	0.0262 (3)
H9C	0.2002	0.4915	0.0319	0.031*
H9D	0.2579	0.3165	0.0506	0.031*
C10A	0.28184 (9)	0.6778 (2)	0.32148 (9)	0.0265 (3)
H10A	0.2393	0.7229	0.3450	0.032*
H10B	0.3022	0.5611	0.3518	0.032*
C10B	0.21842 (9)	0.3630 (2)	-0.07725 (10)	0.0305 (4)
H10C	0.1980	0.4741	-0.1117	0.037*
H10D	0.2633	0.3175	-0.0963	0.037*
N1A	0.18672 (7)	0.49184 (18)	0.21772 (7)	0.0237 (3)
N1B	0.30777 (7)	0.56515 (18)	0.02627 (7)	0.0232 (3)
N2A	0.34167 (8)	0.8271 (2)	0.33524 (9)	0.0313 (3)
N2B	0.16070 (9)	0.2086 (2)	-0.09061 (9)	0.0316 (3)
O1A	0.09422 (7)	0.72480 (16)	0.21678 (8)	0.0350 (3)
O3B	0.24162 (6)	0.85307 (17)	0.02391 (7)	0.0328 (3)
O2A	0.07133 (6)	0.31402 (16)	0.06088 (6)	0.0266 (2)
O2B	0.42195 (6)	0.75223 (16)	0.18321 (6)	0.0280 (3)
O3A	0.25344 (6)	0.20386 (17)	0.22252 (7)	0.0330 (3)
O1B	0.40037 (7)	0.33208 (16)	0.02779 (8)	0.0360 (3)

H11A	0.3196 (12)	0.944 (3)	0.3100 (13)	0.055 (6)*
H11B	0.3779 (12)	0.794 (3)	0.3075 (12)	0.042 (5)*
H11C	0.1829 (12)	0.101 (3)	-0.0609 (13)	0.046 (6)*
H11D	0.1210 (14)	0.253 (3)	-0.0666 (14)	0.058 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0261 (8)	0.0258 (8)	0.0221 (7)	-0.0004 (6)	0.0066 (6)	-0.0001 (6)
C8B	0.0276 (8)	0.0259 (7)	0.0170 (6)	-0.0005 (6)	0.0040 (5)	0.0004 (6)
C2A	0.0232 (7)	0.0260 (8)	0.0239 (7)	-0.0017 (6)	0.0074 (6)	0.0001 (6)
C7B	0.0283 (8)	0.0222 (7)	0.0211 (7)	-0.0036 (6)	0.0043 (6)	0.0005 (6)
C3A	0.0231 (7)	0.0305 (8)	0.0270 (8)	0.0019 (6)	0.0042 (6)	0.0018 (6)
C6B	0.0365 (9)	0.0242 (8)	0.0245 (7)	0.0009 (6)	0.0016 (6)	-0.0029 (6)
C4A	0.0238 (8)	0.0413 (9)	0.0275 (8)	-0.0059 (7)	0.0030 (6)	-0.0023 (7)
C5B	0.0414 (10)	0.0347 (9)	0.0314 (9)	-0.0126 (8)	-0.0032 (7)	-0.0048 (7)
C5A	0.0319 (9)	0.0330 (9)	0.0241 (7)	-0.0087 (7)	0.0016 (6)	-0.0020 (6)
C4B	0.0277 (8)	0.0445 (10)	0.0350 (9)	-0.0103 (7)	-0.0003 (7)	-0.0051 (8)
C6A	0.0300 (8)	0.0243 (7)	0.0222 (7)	0.0000 (6)	0.0045 (6)	-0.0013 (6)
C3B	0.0218 (7)	0.0323 (8)	0.0305 (8)	0.0017 (6)	0.0039 (6)	-0.0012 (6)
C7A	0.0262 (8)	0.0230 (7)	0.0210 (7)	-0.0008 (6)	0.0046 (6)	0.0020 (6)
C2B	0.0226 (7)	0.0267 (8)	0.0271 (7)	-0.0040 (6)	0.0081 (6)	-0.0027 (6)
C8A	0.0274 (8)	0.0253 (7)	0.0166 (6)	0.0011 (6)	0.0040 (5)	0.0007 (5)
C1B	0.0256 (8)	0.0254 (8)	0.0255 (7)	-0.0022 (6)	0.0076 (6)	-0.0018 (6)
C9A	0.0263 (8)	0.0296 (8)	0.0224 (7)	-0.0061 (6)	0.0064 (6)	-0.0004 (6)
C9B	0.0251 (7)	0.0300 (8)	0.0241 (7)	-0.0080 (6)	0.0067 (6)	-0.0002 (6)
C10A	0.0277 (8)	0.0294 (8)	0.0222 (7)	0.0009 (6)	0.0053 (6)	-0.0026 (6)
C10B	0.0326 (9)	0.0330 (8)	0.0256 (8)	-0.0105 (7)	0.0055 (6)	0.0006 (6)
N1A	0.0234 (6)	0.0245 (6)	0.0232 (6)	-0.0026 (5)	0.0053 (5)	-0.0005 (5)
N1B	0.0215 (6)	0.0244 (6)	0.0243 (6)	-0.0037 (5)	0.0061 (5)	-0.0029 (5)
N2A	0.0265 (7)	0.0367 (8)	0.0299 (7)	-0.0024 (6)	0.0043 (6)	-0.0078 (6)
N2B	0.0306 (8)	0.0315 (8)	0.0318 (7)	-0.0080 (6)	0.0045 (6)	-0.0007 (6)
O1A	0.0358 (6)	0.0245 (6)	0.0455 (7)	0.0029 (5)	0.0104 (5)	-0.0029 (5)
O3B	0.0289 (6)	0.0349 (6)	0.0332 (6)	0.0055 (5)	0.0037 (5)	0.0013 (5)
O2A	0.0281 (6)	0.0301 (6)	0.0220 (5)	0.0009 (4)	0.0061 (4)	0.0037 (4)
O2B	0.0295 (6)	0.0317 (6)	0.0222 (5)	0.0040 (5)	0.0045 (4)	0.0028 (4)
O3A	0.0266 (6)	0.0342 (6)	0.0365 (6)	0.0056 (5)	0.0028 (5)	0.0007 (5)
O1B	0.0316 (6)	0.0257 (6)	0.0519 (7)	0.0003 (5)	0.0113 (5)	-0.0047 (5)

Geometric parameters (\AA , $^\circ$)

C1A—C2A	1.509 (2)	C6A—O2A	1.4515 (17)
C2A—C7A	1.547 (2)	C6A—C7A	1.5639 (19)
C7A—C8A	1.518 (2)	C6A—H6A	0.9800
C8A—N1A	1.3858 (19)	C3B—O2B	1.4465 (18)
C8A—O3A	1.2197 (18)	C3B—C2B	1.567 (2)
C1A—N1A	1.3877 (19)	C3B—H3B	0.9800
C1A—O1A	1.2166 (19)	C7A—H7A	0.9800
C8B—O3B	1.2177 (18)	C2B—C1B	1.508 (2)
C8B—N1B	1.3878 (19)	C2B—H2B	0.9800

C8B—C7B	1.518 (2)	C1B—O1B	1.2203 (19)
C2A—C3A	1.565 (2)	C1B—N1B	1.3838 (19)
C2A—H2A	0.9800	C9A—N1A	1.4624 (18)
C7B—C2B	1.543 (2)	C9A—C10A	1.527 (2)
C7B—C6B	1.560 (2)	C9A—H9A	0.9700
C7B—H7B	0.9800	C9A—H9B	0.9700
C3A—O2A	1.4490 (18)	C9B—N1B	1.4637 (18)
C3A—C4A	1.518 (2)	C9B—C10B	1.530 (2)
C3A—H3A	0.9800	C9B—H9C	0.9700
C6B—O2B	1.4520 (19)	C9B—H9D	0.9700
C6B—C5B	1.517 (2)	C10A—N2A	1.463 (2)
C6B—H6B	0.9800	C10A—H10A	0.9700
C4A—C5A	1.327 (2)	C10A—H10B	0.9700
C4A—H4A	0.9300	C10B—N2B	1.462 (2)
C5B—C4B	1.323 (3)	C10B—H10C	0.9700
C5B—H5B	0.9300	C10B—H10D	0.9700
C5A—C6A	1.517 (2)	N2A—H11A	0.95 (2)
C5A—H5A	0.9300	N2A—H11B	0.89 (2)
C4B—C3B	1.509 (2)	N2B—H11C	0.93 (2)
C4B—H4B	0.9300	N2B—H11D	0.93 (2)
C1A—C2A—C7A	104.74 (12)	O2B—C3B—H3B	115.4
C8A—C7A—C2A	104.56 (11)	C4B—C3B—H3B	115.4
O3A—C8A—C7A	127.27 (14)	C2B—C3B—H3B	115.4
O3A—C8A—N1A	124.08 (14)	C8A—C7A—C6A	111.40 (12)
N1A—C8A—C7A	108.63 (12)	C2A—C7A—C6A	101.17 (11)
C8A—N1A—C1A	112.94 (12)	C8A—C7A—H7A	112.9
N1A—C1A—C2A	108.91 (12)	C2A—C7A—H7A	112.9
O1A—C1A—N1A	123.67 (14)	C6A—C7A—H7A	112.9
O1A—C1A—C2A	127.40 (14)	C1B—C2B—C7B	104.84 (12)
O3B—C8B—N1B	123.88 (14)	C1B—C2B—C3B	111.28 (12)
O3B—C8B—C7B	127.28 (14)	C7B—C2B—C3B	101.20 (11)
N1B—C8B—C7B	108.83 (12)	C1B—C2B—H2B	112.9
C1A—C2A—C3A	110.64 (12)	C7B—C2B—H2B	112.9
C7A—C2A—C3A	101.22 (11)	C3B—C2B—H2B	112.9
C1A—C2A—H2A	113.1	O1B—C1B—N1B	123.73 (14)
C7A—C2A—H2A	113.1	O1B—C1B—C2B	127.16 (14)
C3A—C2A—H2A	113.1	N1B—C1B—C2B	109.06 (12)
C8B—C7B—C2B	104.39 (12)	N1A—C9A—C10A	111.62 (12)
C8B—C7B—C6B	111.23 (12)	N1A—C9A—H9A	109.3
C2B—C7B—C6B	101.29 (11)	C10A—C9A—H9A	109.3
C8B—C7B—H7B	113.0	N1A—C9A—H9B	109.3
C2B—C7B—H7B	113.0	C10A—C9A—H9B	109.3
C6B—C7B—H7B	113.0	H9A—C9A—H9B	108.0
O2A—C3A—C4A	101.96 (12)	N1B—C9B—C10B	111.67 (12)
O2A—C3A—C2A	100.56 (11)	N1B—C9B—H9C	109.3
C4A—C3A—C2A	106.40 (12)	C10B—C9B—H9C	109.3
O2A—C3A—H3A	115.4	N1B—C9B—H9D	109.3
C4A—C3A—H3A	115.4	C10B—C9B—H9D	109.3

C2A—C3A—H3A	115.4	H9C—C9B—H9D	107.9
O2B—C6B—C5B	101.63 (12)	N2A—C10A—C9A	113.85 (12)
O2B—C6B—C7B	100.20 (11)	N2A—C10A—H10A	108.8
C5B—C6B—C7B	106.68 (13)	C9A—C10A—H10A	108.8
O2B—C6B—H6B	115.5	N2A—C10A—H10B	108.8
C5B—C6B—H6B	115.5	C9A—C10A—H10B	108.8
C7B—C6B—H6B	115.5	H10A—C10A—H10B	107.7
C5A—C4A—C3A	105.67 (14)	N2B—C10B—C9B	114.02 (12)
C5A—C4A—H4A	127.2	N2B—C10B—H10C	108.7
C3A—C4A—H4A	127.2	C9B—C10B—H10C	108.7
C4B—C5B—C6B	105.93 (15)	N2B—C10B—H10D	108.7
C4B—C5B—H5B	127.0	C9B—C10B—H10D	108.7
C6B—C5B—H5B	127.0	H10C—C10B—H10D	107.6
C4A—C5A—C6A	105.94 (14)	C8A—N1A—C1A	112.94 (12)
C4A—C5A—H5A	127.0	C8A—N1A—C9A	124.59 (13)
C6A—C5A—H5A	127.0	C1A—N1A—C9A	122.43 (13)
C5B—C4B—C3B	105.95 (14)	C1B—N1B—C8B	112.70 (12)
C5B—C4B—H4B	127.0	C1B—N1B—C9B	121.96 (13)
C3B—C4B—H4B	127.0	C8B—N1B—C9B	125.31 (12)
O2A—C6A—C5A	101.96 (12)	C10A—N2A—H11A	107.9 (13)
O2A—C6A—C7A	100.54 (11)	C10A—N2A—H11B	109.0 (13)
C5A—C6A—C7A	106.15 (12)	H11A—N2A—H11B	106.3 (18)
O2A—C6A—H6A	115.4	C10B—N2B—H11C	107.2 (13)
C5A—C6A—H6A	115.4	C10B—N2B—H11D	105.9 (14)
C7A—C6A—H6A	115.4	H11C—N2B—H11D	109.0 (18)
O2B—C3B—C4B	101.94 (13)	C3A—O2A—C6A	95.91 (10)
O2B—C3B—C2B	100.73 (11)	C3B—O2B—C6B	95.97 (11)
C4B—C3B—C2B	105.93 (13)		
O1A—C1A—C2A—C7A	-179.51 (15)	O2B—C3B—C2B—C7B	34.90 (14)
N1A—C1A—C2A—C7A	2.19 (15)	C4B—C3B—C2B—C7B	-70.95 (14)
O1A—C1A—C2A—C3A	72.18 (19)	C2A—C7A—C8A—O3A	178.38 (14)
N1A—C1A—C2A—C3A	-106.12 (13)	C6A—C7A—C8A—O3A	-73.16 (18)
O3B—C8B—C7B—C2B	179.53 (14)	C2A—C7A—C8A—N1A	-3.32 (15)
N1B—C8B—C7B—C2B	0.71 (15)	C6A—C7A—C8A—N1A	105.14 (13)
O3B—C8B—C7B—C6B	71.08 (19)	C7B—C2B—C1B—O1B	178.86 (15)
N1B—C8B—C7B—C6B	-107.74 (14)	C3B—C2B—C1B—O1B	-72.5 (2)
C1A—C2A—C3A—O2A	74.43 (14)	C7B—C2B—C1B—N1B	-3.73 (15)
C7A—C2A—C3A—O2A	-36.17 (13)	C3B—C2B—C1B—N1B	104.86 (14)
C1A—C2A—C3A—C4A	-179.63 (12)	N1A—C9A—C10A—N2A	-174.08 (13)
C7A—C2A—C3A—C4A	69.77 (14)	N1B—C9B—C10B—N2B	172.66 (14)
C8B—C7B—C6B—O2B	72.99 (14)	O3A—C8A—N1A—C1A	-176.58 (13)
C2B—C7B—C6B—O2B	-37.46 (14)	C7A—C8A—N1A—C1A	5.05 (16)
C8B—C7B—C6B—C5B	178.53 (13)	O3A—C8A—N1A—C9A	1.2 (2)
C2B—C7B—C6B—C5B	68.07 (14)	C7A—C8A—N1A—C9A	-177.21 (12)
O2A—C3A—C4A—C5A	32.38 (15)	O1A—C1A—N1A—C8A	177.01 (14)
C2A—C3A—C4A—C5A	-72.54 (15)	C2A—C1A—N1A—C8A	-4.61 (16)
O2B—C6B—C5B—C4B	31.39 (16)	O1A—C1A—N1A—C9A	-0.8 (2)
C7B—C6B—C5B—C4B	-73.11 (16)	C2A—C1A—N1A—C9A	177.59 (12)

C3A—C4A—C5A—C6A	-0.51 (16)	C10A—C9A—N1A—C8A	-93.08 (16)
C6B—C5B—C4B—C3B	0.50 (18)	C10A—C9A—N1A—C1A	84.46 (16)
C4A—C5A—C6A—O2A	-31.43 (15)	O1B—C1B—N1B—C8B	-178.00 (14)
C4A—C5A—C6A—C7A	73.40 (15)	C2B—C1B—N1B—C8B	4.48 (16)
C5B—C4B—C3B—O2B	-32.42 (16)	O1B—C1B—N1B—C9B	0.1 (2)
C5B—C4B—C3B—C2B	72.55 (16)	C2B—C1B—N1B—C9B	-177.36 (12)
C1A—C2A—C7A—C8A	0.67 (14)	O3B—C8B—N1B—C1B	177.87 (13)
C3A—C2A—C7A—C8A	115.74 (12)	C7B—C8B—N1B—C1B	-3.27 (16)
C1A—C2A—C7A—C6A	-115.15 (12)	O3B—C8B—N1B—C9B	-0.2 (2)
C3A—C2A—C7A—C6A	-0.08 (13)	C7B—C8B—N1B—C9B	178.66 (12)
O2A—C6A—C7A—C8A	-74.40 (14)	C10B—C9B—N1B—C1B	-79.01 (17)
C5A—C6A—C7A—C8A	179.74 (12)	C10B—C9B—N1B—C8B	98.90 (17)
O2A—C6A—C7A—C2A	36.24 (13)	C4A—C3A—O2A—C6A	-49.59 (12)
C5A—C6A—C7A—C2A	-69.62 (14)	C2A—C3A—O2A—C6A	59.87 (12)
C8B—C7B—C2B—C1B	1.77 (14)	C5A—C6A—O2A—C3A	49.24 (12)
C6B—C7B—C2B—C1B	117.39 (12)	C7A—C6A—O2A—C3A	-59.95 (12)
C8B—C7B—C2B—C3B	-114.03 (12)	C4B—C3B—O2B—C6B	49.64 (13)
C6B—C7B—C2B—C3B	1.59 (14)	C2B—C3B—O2B—C6B	-59.36 (12)
O2B—C3B—C2B—C1B	-76.04 (14)	C5B—C6B—O2B—C3B	-49.14 (13)
C4B—C3B—C2B—C1B	178.11 (13)	C7B—C6B—O2B—C3B	60.42 (12)

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>
N2A—H11A...O3A ⁱ	0.95 (2)	2.42 (2)	3.3599 (18)	169.1 (18)
N2A—H11B...O2B	0.89 (2)	2.33 (2)	3.1480 (18)	151.8 (17)
N2B—H11C...O3B ⁱⁱ	0.92 (2)	2.30 (2)	3.2175 (19)	174.4 (18)
N2B—H11D...O2A	0.93 (2)	2.45 (2)	3.2838 (19)	148.7 (19)
C2B—H2B...O1B ⁱⁱⁱ	0.98	2.52	3.408 (2)	151

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