

Crystal structures of 2-methoxyisindoline-1,3-dione, 1,3-dioxoisindolin-2-yl methyl carbonate and 1,3-dioxo-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-2-yl methyl carbonate: three anticonvulsant compounds

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Keywords: crystal structure; anticonvulsant; isoindoline; isoquinoline; indoline

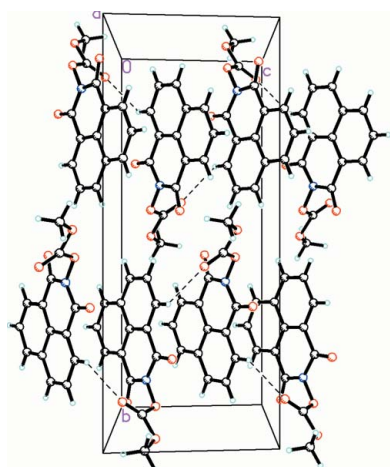
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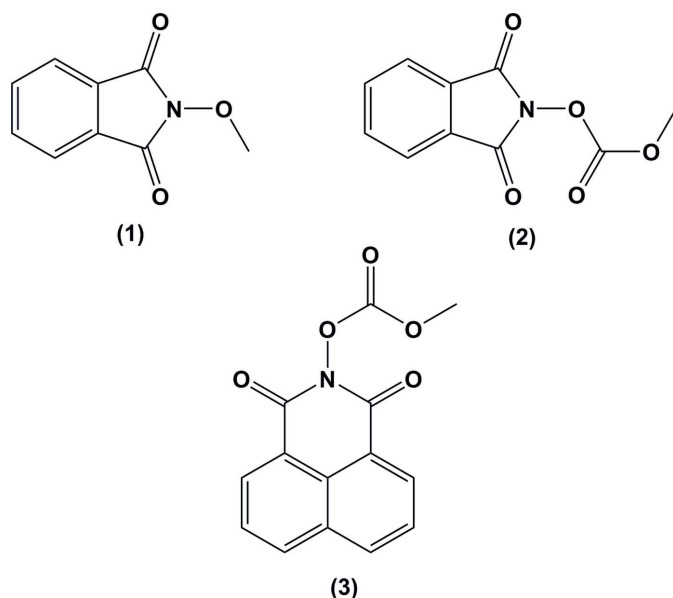
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The title compounds, C₉H₇NO₃ (1), C₁₀H₇NO₅ (2), and C₁₄H₉NO₅ (3), are three potentially anticonvulsant compounds. Compounds (1) and (2) are isoindoline derivatives and (3) is an isoquinoline derivative. Compounds (2) and (3) crystallize with two independent molecules (*A* and *B*) in their asymmetric units. In all three cases, the isoindoline and benzoisoquinoline moieties are planar [r.m.s. deviations are 0.021 Å for (1), 0.04 and 0.018 Å for (2), and 0.033 and 0.041 Å for (3)]. The substituents attached to the N atom are almost perpendicular to the mean planes of the heterocycles, with dihedral angles of 89.7 (3)° for the N—O—C_{methyl} group in (1), 71.01 (4) and 80.00 (4)° for the N—O—C(=O)O—C_{methyl} groups in (2), and 75.62 (14) and 74.13 (4)° for the same groups in (3). In the crystal of (1), there are unusual intermolecular C=O...C contacts of 2.794 (1) and 2.873 (1) Å present in molecules *A* and *B*, respectively. There are also C—H...O hydrogen bonds and π – π interactions [inter-centroid distance = 3.407 (3) Å] present, forming slabs lying parallel to (001). In the crystal of (2), the *A* and *B* molecules are linked by C—H...O hydrogen bonds, forming slabs parallel to (10 $\bar{1}$), which are in turn linked *via* a number of π – π interactions [the most significant centroid–centroid distances are 3.4202 (7) and 3.5445 (7) Å], forming a three-dimensional structure. In the crystal of (3), the *A* and *B* molecules are linked *via* C—H...O hydrogen bonds, forming a three-dimensional structure, which is consolidated by π – π interactions [the most significant inter-centroid distances are 3.575 (3) and 3.578 (3) Å].

1. Chemical context

Traumatic brain injury (TBI) is a neurological disorder that is defined as damage to the brain resulting from external mechanical force, including accelerating, decelerating and rotating forces (Langlois *et al.*, 2003, 2005; Ashman *et al.*, 2006; Coronado *et al.*, 2011). TBI also exacerbates seizure severity in individuals with pre-existing epilepsy (Ferraro *et al.*, 1999), being one example of the process of epileptogenesis (Christensen *et al.*, 2009). In this context, it has been demonstrated that early lesions in the central nervous system (CNS) alter the transport dynamic of the blood–brain barrier (BBB) and deteriorate the balance of the inhibitory and excitatory neurotransmitter system (Scantlebury *et al.*, 2005). This neuronal dysfunction predisposes to subsequent development of spontaneous recurrent seizures in the presence of prior subtle brain malformation (Love, 2005).





TBI is the major cause of death in young individuals (14–24 years) from industrialized countries, with head injuries accounting for 25–33% of all trauma-related deaths (Abdul-Muneer *et al.*, 2014). Disorders like memory loss, depression and seizures are some of the side effects to TBI. TBI affects people over 75 years of age because of falls and of 17–25 years of age because of accidents (Langlois *et al.*, 2003, 2005; Ashman *et al.*, 2006; Coronado *et al.*, 2011). At present, there are no effective treatments available for TBI and there is thus a critical need to develop novel and effective strategies to alter the disease course. As indicated above, this health condition is quite similar to epilepsy in some instances and thus our earlier work (Alexander *et al.*, 2013; Jackson *et al.*, 2012; Edafiogho *et al.*, 2007) on developing anticonvulsant compounds for the treatment of epilepsy is relevant.

Our research on pharmacologically active compounds is a multi-pronged approach, which involves synthesis, chemical characterization, computer modeling, pharmacological evaluation, and structure determination (North *et al.*, 2012; Gibson *et al.*, 2009). From this comprehensive approach, structure–activity correlations can be made to improve the existing pharmacologically active compounds. From our studies, we identified three imidoxy derivatives as potential drug candidates for TBI that underwent anticonvulsant evaluation to test their ability to inhibit the onset of seizures in the *in vivo* MES, scPTZ test models. The MES (maximal electroshock seizure evaluation) test presented activity in animals in phase 1 testing.

2-Methoxyisoindoline-1,3-dione, (1), studied by X-ray techniques, was inactive in MES and scPTZ in mice, but showed MES protection in rat studies at 50 mg kg⁻¹ at 4 h and also protected 1/4 mice at three different time intervals (0.50, 1 and 2 h) in the 6 Hz test (Jackson, 2009). For scPTZ studies, the compound was Class III (no activity at 300 mg kg⁻¹). The compound is a dual MES/6Hz active compound. Compounds (2) and (3) showed similar activity.

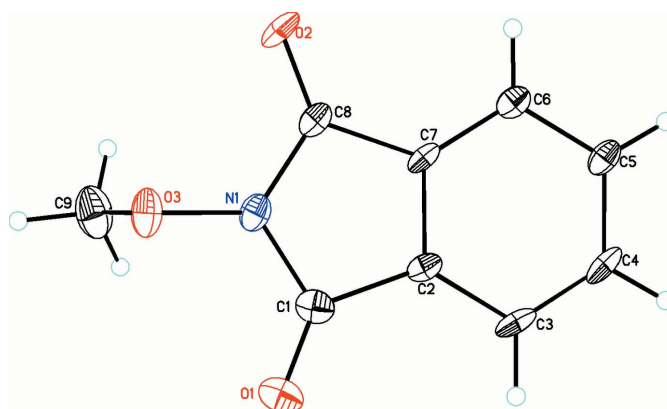


Figure 1
The molecular structure of compound (1), with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

The title compounds, containing either an isoindoline-1,3-dione moiety, (1) (Fig. 1) and (2) (Fig. 2), or an isoquinoline-1,3-dione moiety, (3) (Fig. 3), have been studied extensively for their anticonvulsant effects with promising results. Herein, we report on the crystal structures of these new structurally related compounds.

2. Structural commentary

In compound (1), the isoindoline ring is planar [r.m.s. deviation = 0.017 (4) Å]. The methoxy O atom, O3, deviates from this plane by 0.176 (6) Å while the methyl C atom, C9, is out of the plane by 1.105 (9) Å. The methoxy substituent is oriented almost perpendicular to the indoline ring with the dihedral angle between the mean planes of the indoline ring and the methoxy substituent being 89.7 (3)°.

In compound (2), there are two molecules (*A* and *B*) in the asymmetric unit. The isoindoline ring is planar [r.m.s. deviation = 0.0327 (9) for *A* and 0.0147 (9) Å for *B*] with the dione O atoms significantly out of the plane for molecule *A* but not for molecule *B* [0.172 (1) and 0.123 (1) Å for atoms O1 and

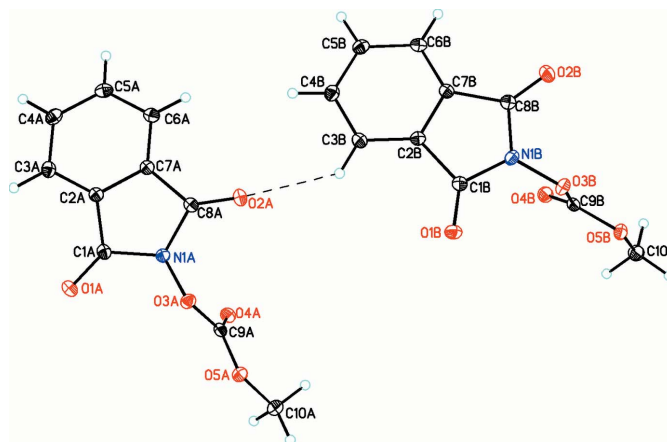


Figure 2
The molecular structures of the two independent molecules (*A* and *B*) of compound (2), with atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The C–H...O hydrogen bond is shown as a dashed line (see Table 2 for details).

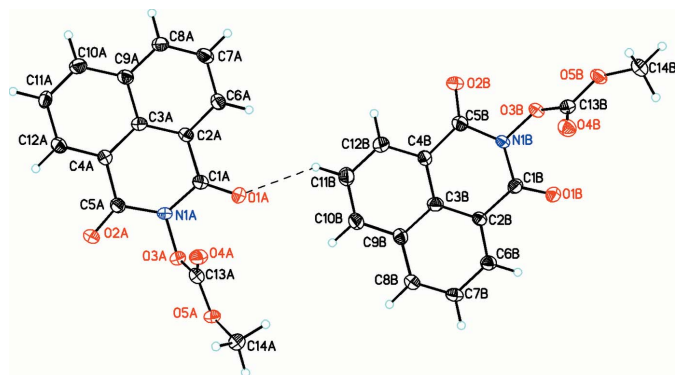


Figure 3
The molecular structures of the two independent molecules (*A* and *B*) of compound (3), with atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The C—H...O hydrogen bond is shown as a dashed line (see Table 3 for details).

O2, respectively, in *A* but by only 0.013 (1) and 0.002 (1) Å, respectively, in *B*]. The carbonato moiety is planar in both molecules [r.m.s. deviations of 0.0066 (2) and 0.0027 (5) Å for *A* and *B*, respectively] and makes dihedral angles of 71.50 (3) and 80.03 (4)° with the benzoisoquinoline ring in *A* and *B*, respectively, indicating that these substituents are oriented almost perpendicular to the benzoisoquinoline ring system.

In compound (3), there are also two molecules (*A* and *B*) in the asymmetric unit. In both molecules, the benzoisoquinoline ring systems are planar (r.m.s. deviations for *A* and *B* = 0.033 and 0.015 Å, respectively). The methoxy O atom deviates from this plane by 0.126 (1) for atom O5A in *A* and 0.156 (1) Å for atom O5B in *B*. The methyl carbonate moieties are planar [r.m.s. deviations of 0.007 (1) and 0.003 (1) Å for *A* and *B*, respectively] and these substituents are oriented almost perpendicular to the isoquinoline rings, making dihedral angles of 71.50 (3) and 80.04 (4)° for *A* and *B*, respectively. As in (2), these dihedral angles are significantly smaller than that found for (1).

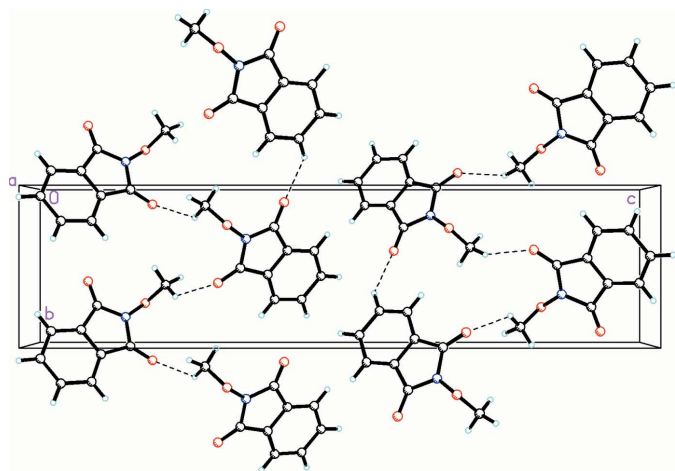


Figure 4
A view along the *a* axis of the crystal packing of compound (1), showing the formation of the three-dimensional array by an extensive network of C—H...O hydrogen bonds (shown as dashed lines; see Table 1 for details).

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

<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>
C4—H4A...O2 ⁱ	0.95	2.38	3.190 (4)	143
C9—H9A...O1 ⁱⁱ	0.98	2.54	3.428 (7)	151
C9—H9B...O1 ⁱⁱⁱ	0.98	2.53	3.260 (8)	131

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

Table 2
Hydrogen-bond geometry (Å, °) for (2).

<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>
C5A—H5AA...O3B ⁱ	0.95	2.54	3.3341 (15)	141
C6A—H6AA...O4A ⁱⁱ	0.95	2.51	3.4091 (15)	158
C3B—H3BA...O2A ⁱⁱⁱ	0.95	2.59	3.2281 (14)	125
C6B—H6BA...O3A ^{iv}	0.95	2.55	3.3086 (14)	137
C10B—H10F...O2B ^v	0.98	2.57	3.4956 (16)	157

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

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

<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>
C6A—H6AA...O4A ⁱ	0.95	2.51	3.159 (5)	125
C7B—H7BA...O2B ⁱⁱ	0.95	2.51	3.229 (5)	133
C10B—H10B...O5B ⁱⁱⁱ	0.95	2.60	3.428 (5)	146
C11B—H11B...O1A ⁱⁱⁱ	0.95	2.48	3.270 (6)	141
C14A—H14A...O1B ^{iv}	0.98	2.51	3.481 (5)	169
C14B—H14E...O4A ^{iv}	0.98	2.51	3.306 (6)	138

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

3. Supramolecular features

In the crystal of (1), there are C—H...O hydrogen bonds (Fig. 4 and Table 1) and π — π interactions present, forming slabs lying parallel to (001) [$Cg1...Cg2^{i,ii} = 3.407$ (3) Å; $Cg1$ and $Cg2$ are the centroids of rings N1/C1/C2/C7/C8 and C2—C7, respectively; symmetry codes: (i) $x - 1, y, z$; (ii) $x + 1, y, z$].

In the crystal of (2), the *A* and *B* molecules are linked by C—H...O hydrogen bonds (Fig. 5 and Table 2), forming slabs parallel to (10 $\bar{1}$). The slabs are in turn linked *via* π — π interactions, forming a three-dimensional structure with centroid—centroid distances of 3.4202 (7) for $Cg1...Cg5^{ii}$ and 3.5445 (7) Å for $Cg2...Cg4^{ii}$ [$Cg1, Cg2, Cg4$ and $Cg5$ are the

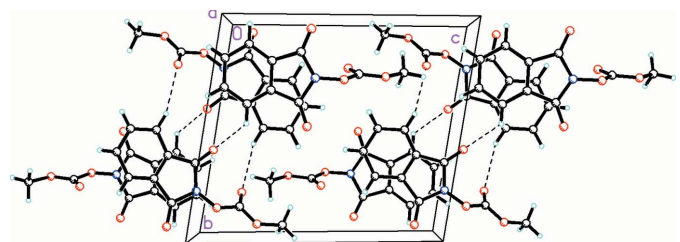
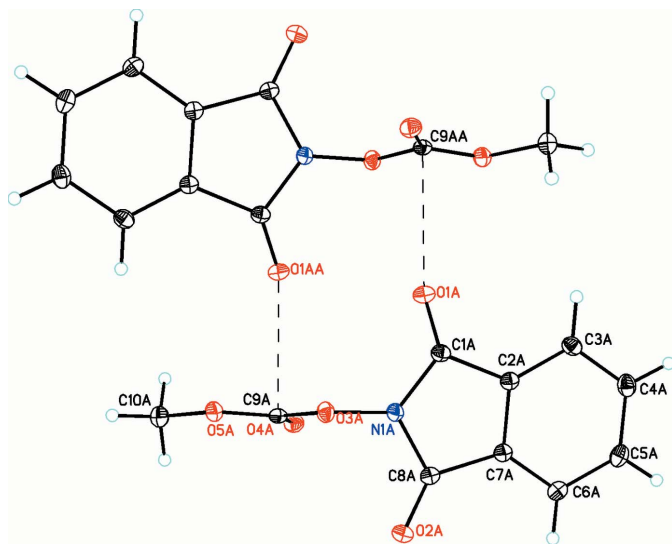


Figure 5
A view along the *a* axis of the crystal packing of compound (2), showing the three-dimensional array formed by an extensive network of C—H...O hydrogen bonds (dashed lines; see Table 2 for details).

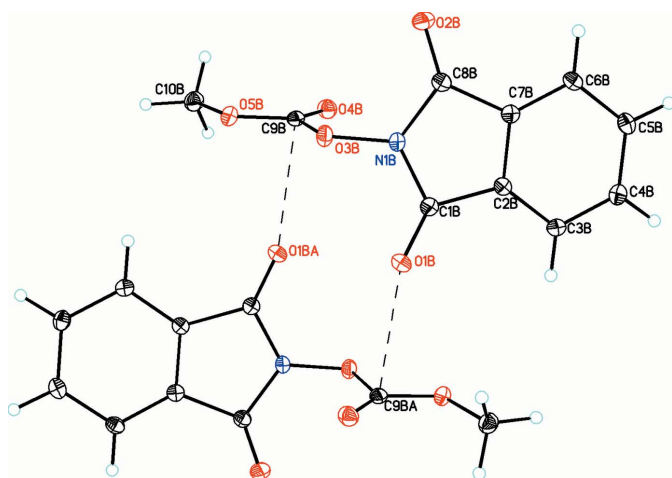

Figure 6

For molecule *A* in compound (2), perpendicular interactions between atoms O1A and C9A (shown as dashed lines) link the molecules into inversion dimers [symmetry code: (A) $-x + 1, -y + 2, -z$].

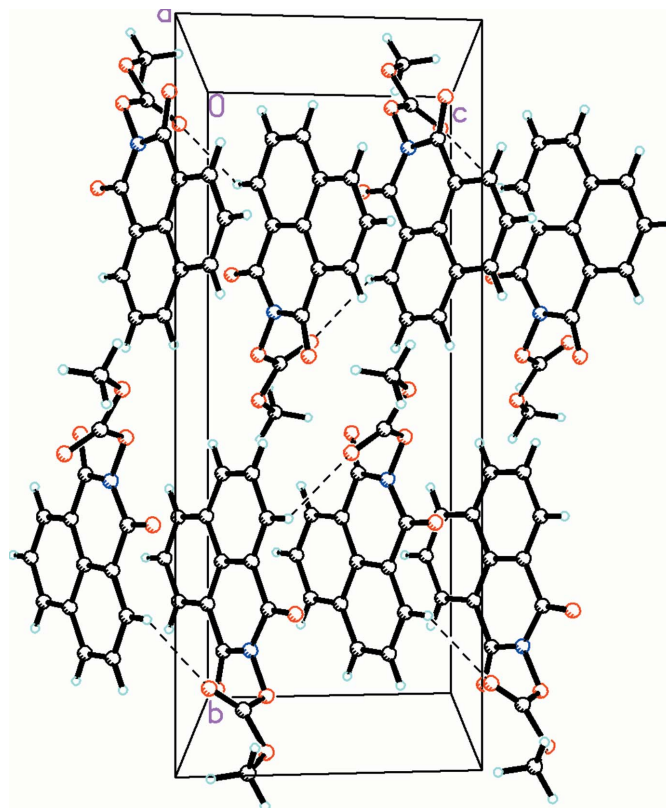
centroids of rings N1A/C1A/C2A/C7A/C8A, C2A–C7A, N1B/C1B/C2B/C7B/C8B and C2B–C7B, respectively; symmetry code: (ii) $x + 1, y, z - 1$].

In the crystal of (3), the *A* and *B* molecules are linked by C–H···O hydrogen bonds (Fig. 6 and Table 3), forming a three-dimensional structure, which is consolidated by π – π interactions [$Cg1 \cdots Cg3^{iii} = 3.578$ (3), $Cg2 \cdots Cg3^{iii} = 3.575$ (3) Å and $Cg9 \cdots Cg10^{iv}$; $Cg1, Cg2, Cg3, Cg9$ and $Cg10$ are the centroids of rings N1A/C1A–C5A, C2A/C3A/C6A–C9A, C3A/C4A/C9A–C12A, C2B/C3B/C6B–C9B and C3B/C4B/C9B–C12B, respectively; symmetry codes: (iii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $x, -y + \frac{3}{2}, z + \frac{1}{2}$].

Interestingly, in the crystal of (2) one of the two dione moieties for each molecule (O1A and O1B) has a short intermolecular interactions with the central C atom of the


Figure 7

For molecule *B* in compound (2), perpendicular interactions between atoms O1B and C9B (shown as dashed lines) link the molecules into inversion dimers [symmetry code: (A) $-x, -y + 1, -z - 1$].


Figure 8

A view along the *a* axis of the crystal packing of compound (3), showing the formation of the three-dimensional array by an extensive network of C–H···O hydrogen bonds (dashed lines; see Table 3 for details).

carbonato group [$O1A \cdots C9A = 2.794$ (1), $O1B \cdots C9B = 2.873$ (1) Å], which is perpendicular to the carbonato plane indicating that both atoms, C9A and C9B, must have significant positive character. These interactions link the molecules into dimers as shown in Figs. 6 and 7, respectively. This is also noticed to a lesser extent in (3) (Fig. 8) for molecule *A* (but not for molecule *B*), where a longer intermolecular interaction of 3.060 (3) Å is observed between atoms O2A and C13A, resulting in weakly associated dimers similar to that seen in the case of (2).

4. Database survey

A search of the Cambridge Structural Database (Version 5.35; Groom & Allen, 2014) for the indoline skeleton gave 26 hits. In all cases, the geometrical parameters of the indoline skeleton are similar to those observed in compounds (1) and (2). In the case of the isoquinoline structure, there are only two structures containing the planar isoquinoline moiety with similar geometrical parameters to the present structure, (3).

5. Synthesis and crystallization

Compound (1):

To a freshly prepared solution of sodium (2.3 g, 0.10 mol) in absolute ethanol (60 ml) was added a solution of *N*-hydroxy-

Table 4
Experimental details.

	(1)	(2)	(3)
Crystal data			
Chemical formula	C ₉ H ₇ NO ₃	C ₁₀ H ₇ NO ₅	C ₁₄ H ₉ NO ₅
<i>M_r</i>	177.16	221.17	271.22
Crystal system, space group	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	123	123	123
<i>a</i> , <i>b</i> , <i>c</i> (Å)	4.2987 (4), 7.0243 (10), 27.587 (4)	7.0363 (4), 11.0082 (5), 12.4239 (6)	16.512 (3), 18.579 (3), 7.6156 (13)
α , β , γ (°)	90, 90, 90	98.884 (4), 96.159 (4), 93.009 (4)	90, 99.434 (17), 90
<i>V</i> (Å ³)	832.98 (19)	942.95 (8)	2304.6 (7)
<i>Z</i>	4	4	8
Radiation type	Mo <i>K</i> α	Cu <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.11	1.10	0.12
Crystal size (mm)	0.66 × 0.23 × 0.04	0.35 × 0.25 × 0.08	0.44 × 0.12 × 0.07
Data collection			
Diffractometer	Agilent Xcalibur (Ruby, Gemini)	SuperNova (Dual, Cu at zero, Atlas)	Agilent Xcalibur (Ruby, Gemini)
Absorption correction	Analytical (<i>CrysAlis PRO</i> ; Agilent, 2012)	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2012)	Analytical (<i>CrysAlis PRO</i> ; Agilent, 2012)
<i>T</i> _{min} – <i>T</i> _{max}	0.946, 0.996	0.807, 1.000	0.995, 0.999
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5145, 2259, 1989	6437, 3803, 3516	9949, 4156, 1898
<i>R</i> _{int}	0.087	0.018	0.091
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.727	0.631	0.600
Refinement			
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.099, 0.229, 1.13	0.033, 0.089, 1.06	0.080, 0.224, 1.00
No. of reflections	2259	3803	4156
No. of parameters	119	291	363
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.50, -0.34	0.29, -0.21	0.33, -0.39

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXS2013*, *SHELXL2013* and *SHELXTL* (Sheldrick, 2008) and *SUPERFLIP* (Palatinus *et al.* 2007).

phthalimide (16.3 g, 0.10 mol) in absolute ethanol (350 ml), and the red reaction mixture was stirred at room temperature for 30 min. The brick-red precipitate was collected, washed with water, and dried in the oven at 373 K for 30 min to give 17.45 g (95%) of sodium phthalimide oxide as brick-red crystals; m.p. > 573 K. To the solution of sodium phthalimide oxide (0.92 g, 5 mmol) in water (15 ml) was added acetone (10 ml), followed by a solution of bromomethane (0.66 g, 7 mmol). The reaction mixture was stirred at room temperature for 16 h, during which the red color disappeared. On standing at room temperature for 48 h, the product solidified in the aqueous mixture and was collected. Recrystallization from 2-propanol gave 0.72 g (78%) of compound (1) as plate-like colorless crystals: m.p. 395–397 K; ¹H NMR (CDCl₃) δ 3.36 (s, 3H, *J* = 6 Hz, OCH₃), 5.52, s, 1 H, CH, 7.87 (*m*, 4 H, phthalimido ring).

Compound (2):

To a solution of sodium phthalimide oxide (0.92 g, 5 mmol) in water (15 ml) was added acetone (10 ml), followed by a solution of bromo(methoxy)methanone (0.97 g, 7 mmol). The reaction mixture was stirred at room temperature for 16 h, during which the red color disappeared. On standing at room temperature for 48 h, the product solidified in the aqueous mixture and was collected. Recrystallization from ethanol gave 0.82 g (74%) of compound (2) as colorless crystals: m.p. 410–411 K; ¹H NMR (CDCl₃) δ 3.8 (s, 3H, OCH₃), 7.86 (*m*, 4H, phthalimido ring).

Compound (3):

To a solution of sodium naphthalimide oxide, (1.18 g, 5 mmol), in water (50 ml), was added bromo(methoxy)methanone (1.25g, 7 mmol) in acetone (10 ml). The red reaction mixture was stirred at room temperature. The red color disappeared within 5 min and the reaction mixture was filled with a white precipitate. After standing for 4 h, the white precipitate was collected, washed with water, and recrystallized from ethanol to give 1.46 g (89%) of compound (3) as colorless crystals: m.p. 483–485 K; ¹H NMR (CDCl₃) δ 3.79 (s, 3H, OCH₃), 5.66 (s, 1H, CH), 7.65–8.50 (*m*, 6 H, naphthalimido ring).

6. Refinement

Crystal data, data collection and structure refinement details for (1), (2) and (3) are summarized in Table 4. For all three compounds, the H atoms were positioned geometrically and refined as riding: C–H = 0.93–0.99 Å with *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H atoms and = 1.2*U*_{eq}(C) for other H atoms.

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supporting information

Acta Cryst. (2014). E70, 456–461 [doi:10.1107/S1600536814023769]

Crystal structures of 2-methoxyisoindoline-1,3-dione, 1,3-dioxoisoindolin-2-yl methyl carbonate and 1,3-dioxo-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-2-yl methyl carbonate: three anticonvulsant compounds

Fortune Ezemobi, Henry North, Kenneth R. Scott, Anthohy K. Wutoh and Ray J. Butcher

Computing details

For all compounds, data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012). Program(s) used to solve structure: SUPERFLIP (Palatinus & Chapuis, 2007) for (1); *SHELXS2013* (Sheldrick, 2008) for (2); SUPERFLIP (Palatinus *et al.* 2007) for (3). For all compounds, program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(1) 2-Methoxyisoindoline-1,3-dione

Crystal data

$C_9H_7NO_3$

$M_r = 177.16$

Orthorhombic, $P2_12_12_1$

$a = 4.2987$ (4) Å

$b = 7.0243$ (10) Å

$c = 27.587$ (4) Å

$V = 832.98$ (19) Å³

$Z = 4$

$F(000) = 368$

$D_x = 1.413$ Mg m⁻³

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

Cell parameters from 1634 reflections

$\theta = 3.3$ – 31.1°

$\mu = 0.11$ mm⁻¹

$T = 123$ K

Plate, colorless

$0.66 \times 0.23 \times 0.04$ mm

Data collection

Agilent Xcalibur (Ruby, Gemini)
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Detector resolution: 10.5081 pixels mm⁻¹

ω scans

Absorption correction: analytical
(*CrysAlis PRO*; Agilent, 2012)

$T_{\min} = 0.946$, $T_{\max} = 0.996$

5145 measured reflections

2259 independent reflections

1989 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

$\theta_{\max} = 31.1^\circ$, $\theta_{\min} = 3.3^\circ$

$h = -5 \rightarrow 5$

$k = -10 \rightarrow 9$

$l = -25 \rightarrow 38$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.099$

$wR(F^2) = 0.229$

$S = 1.13$

2259 reflections

119 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2 + 1.043P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6646 (10)	0.6034 (6)	0.30567 (13)	0.0417 (10)
O2	0.6859 (9)	0.0978 (4)	0.41116 (14)	0.0358 (9)
O3	0.9419 (9)	0.2430 (6)	0.32341 (14)	0.0413 (10)
N1	0.7177 (10)	0.3273 (6)	0.35101 (15)	0.0293 (9)
C1	0.6031 (12)	0.5105 (7)	0.34055 (17)	0.0284 (10)
C2	0.3946 (11)	0.5531 (6)	0.38248 (16)	0.0229 (9)
C3	0.2197 (12)	0.7156 (6)	0.39295 (18)	0.0306 (11)
H3A	0.2110	0.8199	0.3711	0.037*
C4	0.0601 (13)	0.7184 (6)	0.4364 (2)	0.0318 (11)
H4A	-0.0591	0.8278	0.4445	0.038*
C5	0.0684 (12)	0.5655 (6)	0.46879 (18)	0.0275 (10)
H5A	-0.0436	0.5722	0.4984	0.033*
C6	0.2410 (10)	0.4030 (6)	0.45760 (16)	0.0250 (9)
H6A	0.2465	0.2977	0.4792	0.030*
C7	0.4026 (11)	0.3989 (6)	0.41468 (16)	0.0229 (9)
C8	0.6130 (11)	0.2513 (6)	0.39447 (17)	0.0247 (9)
C9	0.8051 (17)	0.1214 (10)	0.2870 (2)	0.0532 (18)
H9A	0.9693	0.0701	0.2661	0.080*
H9B	0.6583	0.1952	0.2674	0.080*
H9C	0.6948	0.0162	0.3028	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.039 (2)	0.049 (2)	0.0379 (19)	-0.010 (2)	0.0000 (17)	0.0107 (16)
O2	0.0325 (19)	0.0155 (14)	0.059 (2)	0.0085 (16)	-0.0026 (17)	0.0010 (15)
O3	0.028 (2)	0.051 (2)	0.045 (2)	0.005 (2)	0.0082 (16)	-0.0136 (17)
N1	0.022 (2)	0.030 (2)	0.036 (2)	0.0038 (17)	0.0013 (16)	-0.0028 (16)
C1	0.0186 (19)	0.034 (2)	0.033 (2)	-0.007 (2)	-0.0037 (18)	0.0048 (18)
C2	0.0163 (18)	0.0171 (19)	0.035 (2)	-0.0029 (16)	-0.0069 (17)	0.0032 (16)
C3	0.031 (3)	0.0115 (18)	0.049 (3)	-0.0008 (18)	-0.014 (2)	0.0041 (17)
C4	0.028 (3)	0.013 (2)	0.054 (3)	0.0040 (19)	-0.011 (2)	-0.0085 (19)
C5	0.021 (2)	0.019 (2)	0.043 (3)	-0.0001 (17)	-0.0024 (19)	-0.0055 (18)
C6	0.019 (2)	0.0183 (19)	0.038 (2)	-0.0048 (18)	-0.0031 (18)	-0.0007 (16)
C7	0.021 (2)	0.0122 (17)	0.035 (2)	0.0024 (19)	-0.0075 (17)	-0.0009 (16)
C8	0.0183 (19)	0.019 (2)	0.037 (2)	-0.0023 (17)	-0.0044 (18)	-0.0035 (17)

C9	0.050 (4)	0.062 (4)	0.047 (3)	-0.011 (4)	0.010 (3)	-0.025 (3)
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Geometric parameters (Å, °)

O1—C1	1.193 (6)	C4—C5	1.398 (7)
O2—C8	1.214 (5)	C4—H4A	0.9500
O3—N1	1.364 (5)	C5—C6	1.396 (6)
O3—C9	1.444 (7)	C5—H5A	0.9500
N1—C8	1.387 (6)	C6—C7	1.373 (6)
N1—C1	1.407 (6)	C6—H6A	0.9500
C1—C2	1.494 (7)	C7—C8	1.484 (6)
C2—C3	1.397 (6)	C9—H9A	0.9800
C2—C7	1.401 (6)	C9—H9B	0.9800
C3—C4	1.381 (8)	C9—H9C	0.9800
C3—H3A	0.9500		
N1—O3—C9	110.9 (4)	C6—C5—H5A	120.0
O3—N1—C8	123.0 (4)	C4—C5—H5A	120.0
O3—N1—C1	122.0 (4)	C7—C6—C5	118.5 (4)
C8—N1—C1	114.5 (4)	C7—C6—H6A	120.8
O1—C1—N1	126.0 (5)	C5—C6—H6A	120.8
O1—C1—C2	130.4 (5)	C6—C7—C2	121.2 (4)
N1—C1—C2	103.6 (4)	C6—C7—C8	130.3 (4)
C3—C2—C7	120.9 (4)	C2—C7—C8	108.5 (4)
C3—C2—C1	130.3 (4)	O2—C8—N1	125.9 (4)
C7—C2—C1	108.7 (4)	O2—C8—C7	129.4 (5)
C4—C3—C2	117.3 (4)	N1—C8—C7	104.7 (4)
C4—C3—H3A	121.3	O3—C9—H9A	109.5
C2—C3—H3A	121.3	O3—C9—H9B	109.5
C3—C4—C5	122.1 (4)	H9A—C9—H9B	109.5
C3—C4—H4A	119.0	O3—C9—H9C	109.5
C5—C4—H4A	119.0	H9A—C9—H9C	109.5
C6—C5—C4	120.0 (5)	H9B—C9—H9C	109.5
C9—O3—N1—C8	93.4 (6)	C5—C6—C7—C2	-0.4 (7)
C9—O3—N1—C1	-94.8 (6)	C5—C6—C7—C8	177.1 (4)
O3—N1—C1—O1	6.7 (8)	C3—C2—C7—C6	-0.4 (7)
C8—N1—C1—O1	179.1 (5)	C1—C2—C7—C6	177.4 (4)
O3—N1—C1—C2	-173.7 (4)	C3—C2—C7—C8	-178.4 (4)
C8—N1—C1—C2	-1.2 (5)	C1—C2—C7—C8	-0.6 (5)
O1—C1—C2—C3	-1.7 (9)	O3—N1—C8—O2	-6.2 (7)
N1—C1—C2—C3	178.6 (5)	C1—N1—C8—O2	-178.5 (4)
O1—C1—C2—C7	-179.2 (5)	O3—N1—C8—C7	173.2 (4)
N1—C1—C2—C7	1.1 (5)	C1—N1—C8—C7	0.9 (5)
C7—C2—C3—C4	0.9 (7)	C6—C7—C8—O2	1.5 (8)
C1—C2—C3—C4	-176.4 (5)	C2—C7—C8—O2	179.2 (5)
C2—C3—C4—C5	-0.5 (7)	C6—C7—C8—N1	-177.9 (5)
C3—C4—C5—C6	-0.2 (7)	C2—C7—C8—N1	-0.1 (5)

C4—C5—C6—C7

0.7 (7)

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>
C4—H4 <i>A</i> ···O2 ⁱ	0.95	2.38	3.190 (4)	143
C9—H9 <i>A</i> ···O1 ⁱⁱ	0.98	2.54	3.428 (7)	151
C9—H9 <i>B</i> ···O1 ⁱⁱⁱ	0.98	2.53	3.260 (8)	131

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

(2) 1,3-Dioxoisindolin-2-yl methyl carbonate

Crystal data

C₁₀H₇NO₅ $M_r = 221.17$ Triclinic, $P\bar{1}$ $a = 7.0363$ (4) Å $b = 11.0082$ (5) Å $c = 12.4239$ (6) Å $\alpha = 98.884$ (4)° $\beta = 96.159$ (4)° $\gamma = 93.009$ (4)° $V = 942.95$ (8) Å³ $Z = 4$ $F(000) = 456$ $D_x = 1.558$ Mg m⁻³Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 4882 reflections

 $\theta = 3.6$ – 76.2 ° $\mu = 1.10$ mm⁻¹ $T = 123$ K

Prism, colorless

 $0.35 \times 0.25 \times 0.08$ mm

Data collection

SuperNova (Dual, Cu at zero, Atlas)
diffractometer

Radiation source: SuperNova (Cu) X-ray

Source

Detector resolution: 5.3250 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrysAlis PRO; Agilent, 2012)

 $T_{\min} = 0.807$, $T_{\max} = 1.000$

6437 measured reflections

3803 independent reflections

3516 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.018$ $\theta_{\max} = 76.7$ °, $\theta_{\min} = 3.6$ ° $h = -6 \rightarrow 8$ $k = -12 \rightarrow 13$ $l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.033$ $wR(F^2) = 0.089$ $S = 1.06$

3803 reflections

291 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.2145P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.29$ e Å⁻³ $\Delta\rho_{\min} = -0.21$ e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1A	0.58069 (12)	0.94400 (7)	-0.10814 (7)	0.02474 (19)
O2A	0.70600 (12)	0.59365 (8)	0.03549 (7)	0.02446 (19)
O3A	0.71408 (11)	0.85375 (7)	0.08575 (6)	0.02145 (18)
O4A	0.45369 (12)	0.79308 (7)	0.16556 (7)	0.02328 (18)
O5A	0.69506 (12)	0.92945 (8)	0.25353 (7)	0.02361 (18)
N1A	0.62655 (14)	0.78226 (9)	-0.01005 (8)	0.0207 (2)
C1A	0.57541 (15)	0.83540 (10)	-0.10425 (9)	0.0194 (2)
C2A	0.52332 (15)	0.72621 (10)	-0.19136 (9)	0.0191 (2)
C3A	0.44765 (16)	0.72094 (11)	-0.29960 (9)	0.0224 (2)
H3AA	0.4198	0.7936	-0.3289	0.027*
C4A	0.41381 (17)	0.60441 (12)	-0.36426 (10)	0.0256 (2)
H4AA	0.3617	0.5976	-0.4389	0.031*
C5A	0.45539 (17)	0.49821 (11)	-0.32085 (10)	0.0255 (2)
H5AA	0.4321	0.4202	-0.3667	0.031*
C6A	0.53058 (16)	0.50392 (11)	-0.21126 (10)	0.0226 (2)
H6AA	0.5582	0.4315	-0.1815	0.027*
C7A	0.56299 (15)	0.61941 (10)	-0.14801 (9)	0.0190 (2)
C8A	0.64135 (15)	0.65422 (10)	-0.03083 (9)	0.0191 (2)
C9A	0.60062 (16)	0.85373 (10)	0.17055 (9)	0.0194 (2)
C10A	0.6040 (2)	0.94039 (12)	0.35373 (10)	0.0295 (3)
H10A	0.6854	0.9950	0.4124	0.044*
H10B	0.5859	0.8588	0.3749	0.044*
H10C	0.4793	0.9747	0.3415	0.044*
O1B	-0.08991 (12)	0.51108 (8)	0.60519 (7)	0.02492 (19)
O2B	-0.11026 (13)	0.93213 (8)	0.64789 (7)	0.0288 (2)
O3B	-0.16633 (12)	0.71284 (8)	0.49226 (6)	0.02337 (18)
O4B	0.14891 (12)	0.77011 (8)	0.48293 (7)	0.02594 (19)
O5B	-0.07693 (12)	0.72731 (8)	0.33471 (6)	0.02271 (18)
N1B	-0.11179 (15)	0.72022 (9)	0.60321 (8)	0.0242 (2)
C1B	-0.06927 (15)	0.61621 (10)	0.65099 (9)	0.0192 (2)
C2B	-0.00046 (15)	0.66970 (10)	0.76699 (9)	0.0184 (2)
C3B	0.06678 (16)	0.61061 (10)	0.85235 (9)	0.0204 (2)
H3BA	0.0703	0.5235	0.8430	0.025*
C4B	0.12944 (16)	0.68463 (11)	0.95314 (9)	0.0229 (2)
H4BA	0.1772	0.6470	1.0135	0.027*
C5B	0.12339 (16)	0.81246 (11)	0.96696 (9)	0.0232 (2)
H5BA	0.1673	0.8603	1.0364	0.028*
C6B	0.05364 (16)	0.87132 (10)	0.88024 (9)	0.0215 (2)
H6BA	0.0482	0.9583	0.8894	0.026*
C7B	-0.00701 (15)	0.79761 (10)	0.78058 (9)	0.0194 (2)
C8B	-0.08039 (16)	0.83365 (10)	0.67376 (9)	0.0213 (2)
C9B	-0.00857 (16)	0.74035 (10)	0.43860 (9)	0.0201 (2)
C10B	0.06721 (18)	0.74950 (12)	0.26295 (10)	0.0269 (3)
H10D	0.0097	0.7306	0.1863	0.040*
H10E	0.1729	0.6967	0.2745	0.040*

H10F 0.1161 0.8362 0.2796 0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0279 (4)	0.0182 (4)	0.0291 (4)	0.0039 (3)	0.0032 (3)	0.0063 (3)
O2A	0.0259 (4)	0.0239 (4)	0.0252 (4)	0.0039 (3)	0.0015 (3)	0.0092 (3)
O3A	0.0221 (4)	0.0214 (4)	0.0190 (4)	-0.0023 (3)	-0.0001 (3)	0.0006 (3)
O4A	0.0230 (4)	0.0202 (4)	0.0266 (4)	0.0001 (3)	0.0024 (3)	0.0045 (3)
O5A	0.0266 (4)	0.0226 (4)	0.0199 (4)	0.0007 (3)	-0.0017 (3)	0.0016 (3)
N1A	0.0254 (5)	0.0175 (5)	0.0178 (4)	0.0002 (4)	-0.0014 (4)	0.0016 (4)
C1A	0.0164 (5)	0.0207 (5)	0.0223 (5)	0.0033 (4)	0.0037 (4)	0.0057 (4)
C2A	0.0158 (5)	0.0204 (5)	0.0217 (5)	0.0021 (4)	0.0038 (4)	0.0039 (4)
C3A	0.0191 (5)	0.0265 (6)	0.0228 (5)	0.0030 (4)	0.0024 (4)	0.0071 (4)
C4A	0.0208 (5)	0.0336 (6)	0.0212 (5)	0.0009 (5)	0.0017 (4)	0.0012 (5)
C5A	0.0226 (5)	0.0251 (6)	0.0266 (6)	-0.0011 (4)	0.0047 (4)	-0.0029 (5)
C6A	0.0209 (5)	0.0199 (5)	0.0270 (6)	0.0008 (4)	0.0055 (4)	0.0024 (4)
C7A	0.0163 (5)	0.0202 (5)	0.0210 (5)	0.0011 (4)	0.0040 (4)	0.0039 (4)
C8A	0.0167 (5)	0.0181 (5)	0.0235 (5)	0.0010 (4)	0.0042 (4)	0.0047 (4)
C9A	0.0219 (5)	0.0161 (5)	0.0206 (5)	0.0046 (4)	-0.0001 (4)	0.0046 (4)
C10A	0.0387 (7)	0.0284 (6)	0.0210 (5)	0.0059 (5)	0.0034 (5)	0.0019 (5)
O1B	0.0289 (4)	0.0194 (4)	0.0248 (4)	0.0009 (3)	0.0027 (3)	-0.0011 (3)
O2B	0.0352 (5)	0.0208 (4)	0.0308 (4)	0.0050 (3)	0.0000 (4)	0.0070 (3)
O3B	0.0251 (4)	0.0280 (4)	0.0163 (4)	-0.0002 (3)	-0.0002 (3)	0.0039 (3)
O4B	0.0239 (4)	0.0249 (4)	0.0272 (4)	0.0005 (3)	-0.0025 (3)	0.0027 (3)
O5B	0.0236 (4)	0.0258 (4)	0.0192 (4)	0.0026 (3)	0.0009 (3)	0.0058 (3)
N1B	0.0348 (5)	0.0211 (5)	0.0156 (4)	0.0012 (4)	-0.0006 (4)	0.0023 (4)
C1B	0.0169 (5)	0.0207 (5)	0.0205 (5)	0.0012 (4)	0.0040 (4)	0.0032 (4)
C2B	0.0155 (5)	0.0193 (5)	0.0201 (5)	0.0006 (4)	0.0035 (4)	0.0015 (4)
C3B	0.0184 (5)	0.0195 (5)	0.0241 (5)	0.0018 (4)	0.0037 (4)	0.0046 (4)
C4B	0.0196 (5)	0.0283 (6)	0.0214 (5)	0.0031 (4)	0.0022 (4)	0.0054 (4)
C5B	0.0195 (5)	0.0275 (6)	0.0211 (5)	0.0010 (4)	0.0023 (4)	-0.0009 (4)
C6B	0.0196 (5)	0.0194 (5)	0.0244 (5)	0.0010 (4)	0.0044 (4)	-0.0006 (4)
C7B	0.0167 (5)	0.0205 (5)	0.0214 (5)	0.0017 (4)	0.0037 (4)	0.0035 (4)
C8B	0.0203 (5)	0.0205 (5)	0.0227 (5)	0.0013 (4)	0.0029 (4)	0.0021 (4)
C9B	0.0237 (5)	0.0154 (5)	0.0209 (5)	0.0034 (4)	0.0000 (4)	0.0033 (4)
C10B	0.0300 (6)	0.0279 (6)	0.0248 (6)	0.0036 (5)	0.0079 (5)	0.0062 (5)

Geometric parameters (Å, °)

O1A—C1A	1.2027 (14)	O1B—C1B	1.2023 (14)
O2A—C8A	1.2048 (14)	O2B—C8B	1.1998 (15)
O3A—N1A	1.3849 (12)	O3B—N1B	1.3794 (12)
O3A—C9A	1.3885 (14)	O3B—C9B	1.3980 (14)
O4A—C9A	1.1914 (15)	O4B—C9B	1.1890 (15)
O5A—C9A	1.3148 (14)	O5B—C9B	1.3116 (14)
O5A—C10A	1.4517 (15)	O5B—C10B	1.4555 (14)
N1A—C8A	1.4045 (14)	N1B—C1B	1.4002 (15)

N1A—C1A	1.4087 (14)	N1B—C8B	1.4027 (15)
C1A—C2A	1.4891 (15)	C1B—C2B	1.4888 (15)
C2A—C3A	1.3837 (15)	C2B—C3B	1.3817 (16)
C2A—C7A	1.3956 (15)	C2B—C7B	1.3958 (15)
C3A—C4A	1.3986 (17)	C3B—C4B	1.3982 (16)
C3A—H3AA	0.9500	C3B—H3BA	0.9500
C4A—C5A	1.3928 (18)	C4B—C5B	1.3945 (17)
C4A—H4AA	0.9500	C4B—H4BA	0.9500
C5A—C6A	1.3975 (17)	C5B—C6B	1.3989 (17)
C5A—H5AA	0.9500	C5B—H5BA	0.9500
C6A—C7A	1.3815 (16)	C6B—C7B	1.3833 (16)
C6A—H6AA	0.9500	C6B—H6BA	0.9500
C7A—C8A	1.4845 (15)	C7B—C8B	1.4908 (15)
C10A—H10A	0.9800	C10B—H10D	0.9800
C10A—H10B	0.9800	C10B—H10E	0.9800
C10A—H10C	0.9800	C10B—H10F	0.9800
N1A—O3A—C9A	111.33 (8)	N1B—O3B—C9B	110.40 (9)
C9A—O5A—C10A	114.27 (9)	C9B—O5B—C10B	113.62 (9)
O3A—N1A—C8A	121.52 (9)	O3B—N1B—C1B	122.20 (9)
O3A—N1A—C1A	120.85 (9)	O3B—N1B—C8B	121.91 (9)
C8A—N1A—C1A	114.42 (9)	C1B—N1B—C8B	115.69 (9)
O1A—C1A—N1A	125.64 (11)	O1B—C1B—N1B	125.87 (10)
O1A—C1A—C2A	131.20 (10)	O1B—C1B—C2B	131.10 (10)
N1A—C1A—C2A	103.13 (9)	N1B—C1B—C2B	103.02 (9)
C3A—C2A—C7A	121.36 (11)	C3B—C2B—C7B	121.65 (10)
C3A—C2A—C1A	129.54 (10)	C3B—C2B—C1B	129.07 (10)
C7A—C2A—C1A	109.10 (9)	C7B—C2B—C1B	109.26 (9)
C2A—C3A—C4A	117.42 (11)	C2B—C3B—C4B	117.10 (10)
C2A—C3A—H3AA	121.3	C2B—C3B—H3BA	121.5
C4A—C3A—H3AA	121.3	C4B—C3B—H3BA	121.5
C5A—C4A—C3A	120.97 (11)	C5B—C4B—C3B	121.40 (10)
C5A—C4A—H4AA	119.5	C5B—C4B—H4BA	119.3
C3A—C4A—H4AA	119.5	C3B—C4B—H4BA	119.3
C4A—C5A—C6A	121.41 (11)	C4B—C5B—C6B	121.06 (11)
C4A—C5A—H5AA	119.3	C4B—C5B—H5BA	119.5
C6A—C5A—H5AA	119.3	C6B—C5B—H5BA	119.5
C7A—C6A—C5A	117.16 (11)	C7B—C6B—C5B	117.24 (11)
C7A—C6A—H6AA	121.4	C7B—C6B—H6BA	121.4
C5A—C6A—H6AA	121.4	C5B—C6B—H6BA	121.4
C6A—C7A—C2A	121.67 (11)	C6B—C7B—C2B	121.55 (10)
C6A—C7A—C8A	129.38 (10)	C6B—C7B—C8B	129.39 (10)
C2A—C7A—C8A	108.95 (9)	C2B—C7B—C8B	109.03 (10)
O2A—C8A—N1A	124.89 (11)	O2B—C8B—N1B	125.16 (11)
O2A—C8A—C7A	131.55 (11)	O2B—C8B—C7B	131.84 (11)
N1A—C8A—C7A	103.55 (9)	N1B—C8B—C7B	103.00 (9)
O4A—C9A—O5A	129.96 (11)	O4B—C9B—O5B	130.60 (11)
O4A—C9A—O3A	125.15 (10)	O4B—C9B—O3B	124.73 (10)

O5A—C9A—O3A	104.85 (9)	O5B—C9B—O3B	104.66 (9)
O5A—C10A—H10A	109.5	O5B—C10B—H10D	109.5
O5A—C10A—H10B	109.5	O5B—C10B—H10E	109.5
H10A—C10A—H10B	109.5	H10D—C10B—H10E	109.5
O5A—C10A—H10C	109.5	O5B—C10B—H10F	109.5
H10A—C10A—H10C	109.5	H10D—C10B—H10F	109.5
H10B—C10A—H10C	109.5	H10E—C10B—H10F	109.5
C9A—O3A—N1A—C8A	-84.35 (12)	C9B—O3B—N1B—C1B	-96.65 (12)
C9A—O3A—N1A—C1A	117.04 (10)	C9B—O3B—N1B—C8B	78.04 (12)
O3A—N1A—C1A—O1A	-8.90 (17)	O3B—N1B—C1B—O1B	-6.58 (18)
C8A—N1A—C1A—O1A	-168.93 (10)	C8B—N1B—C1B—O1B	178.42 (11)
O3A—N1A—C1A—C2A	169.42 (9)	O3B—N1B—C1B—C2B	174.59 (9)
C8A—N1A—C1A—C2A	9.39 (12)	C8B—N1B—C1B—C2B	-0.41 (13)
O1A—C1A—C2A—C3A	-7.3 (2)	O1B—C1B—C2B—C3B	3.3 (2)
N1A—C1A—C2A—C3A	174.49 (11)	N1B—C1B—C2B—C3B	-178.00 (11)
O1A—C1A—C2A—C7A	172.60 (11)	O1B—C1B—C2B—C7B	-178.47 (11)
N1A—C1A—C2A—C7A	-5.58 (11)	N1B—C1B—C2B—C7B	0.27 (12)
C7A—C2A—C3A—C4A	-0.50 (16)	C7B—C2B—C3B—C4B	-0.42 (16)
C1A—C2A—C3A—C4A	179.42 (10)	C1B—C2B—C3B—C4B	177.66 (10)
C2A—C3A—C4A—C5A	-0.12 (17)	C2B—C3B—C4B—C5B	0.31 (16)
C3A—C4A—C5A—C6A	0.57 (18)	C3B—C4B—C5B—C6B	0.19 (17)
C4A—C5A—C6A—C7A	-0.38 (17)	C4B—C5B—C6B—C7B	-0.57 (16)
C5A—C6A—C7A—C2A	-0.24 (16)	C5B—C6B—C7B—C2B	0.46 (16)
C5A—C6A—C7A—C8A	-179.74 (10)	C5B—C6B—C7B—C8B	-177.49 (11)
C3A—C2A—C7A—C6A	0.70 (17)	C3B—C2B—C7B—C6B	0.03 (16)
C1A—C2A—C7A—C6A	-179.24 (10)	C1B—C2B—C7B—C6B	-178.39 (10)
C3A—C2A—C7A—C8A	-179.71 (10)	C3B—C2B—C7B—C8B	178.36 (10)
C1A—C2A—C7A—C8A	0.35 (12)	C1B—C2B—C7B—C8B	-0.06 (12)
O3A—N1A—C8A—O2A	10.35 (17)	O3B—N1B—C8B—O2B	5.73 (18)
C1A—N1A—C8A—O2A	170.23 (10)	C1B—N1B—C8B—O2B	-179.25 (11)
O3A—N1A—C8A—C7A	-169.10 (9)	O3B—N1B—C8B—C7B	-174.64 (9)
C1A—N1A—C8A—C7A	-9.22 (12)	C1B—N1B—C8B—C7B	0.37 (13)
C6A—C7A—C8A—O2A	5.2 (2)	C6B—C7B—C8B—O2B	-2.4 (2)
C2A—C7A—C8A—O2A	-174.35 (11)	C2B—C7B—C8B—O2B	179.42 (12)
C6A—C7A—C8A—N1A	-175.40 (11)	C6B—C7B—C8B—N1B	177.99 (11)
C2A—C7A—C8A—N1A	5.05 (11)	C2B—C7B—C8B—N1B	-0.17 (12)
C10A—O5A—C9A—O4A	-0.38 (17)	C10B—O5B—C9B—O4B	2.22 (17)
C10A—O5A—C9A—O3A	-178.02 (8)	C10B—O5B—C9B—O3B	-178.74 (8)
N1A—O3A—C9A—O4A	5.52 (14)	N1B—O3B—C9B—O4B	-2.43 (15)
N1A—O3A—C9A—O5A	-176.69 (8)	N1B—O3B—C9B—O5B	178.46 (8)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5A—H5AA \cdots O3B ⁱ	0.95	2.54	3.3341 (15)	141
C6A—H6AA \cdots O4A ⁱⁱ	0.95	2.51	3.4091 (15)	158
C3B—H3BA \cdots O2A ⁱⁱⁱ	0.95	2.59	3.2281 (14)	125

C6B—H6BA···O3A ^{iv}	0.95	2.55	3.3086 (14)	137
C10B—H10F···O2B ^v	0.98	2.57	3.4956 (16)	157

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

(3) 1,3-Dioxo-2,3-dihydro-1H-benzo[de]isoquinolin-2-yl methyl carbonate

Crystal data

C ₁₄ H ₉ NO ₅	$F(000) = 1120$
$M_r = 271.22$	$D_x = 1.563 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 16.512 (3) \text{ \AA}$	Cell parameters from 1261 reflections
$b = 18.579 (3) \text{ \AA}$	$\theta = 3.4\text{--}26.9^\circ$
$c = 7.6156 (13) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$\beta = 99.434 (17)^\circ$	$T = 123 \text{ K}$
$V = 2304.6 (7) \text{ \AA}^3$	Needle, colorless
$Z = 8$	$0.44 \times 0.12 \times 0.07 \text{ mm}$

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer	9949 measured reflections
Radiation source: Enhance (Mo) X-ray Source	4156 independent reflections
Detector resolution: 10.5081 pixels mm ⁻¹	1898 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.091$
Absorption correction: analytical (<i>CrysAlis PRO</i> ; Agilent, 2012)	$\theta_{\text{max}} = 25.3^\circ, \theta_{\text{min}} = 3.3^\circ$
$T_{\text{min}} = 0.995, T_{\text{max}} = 0.999$	$h = -15 \rightarrow 19$
	$k = -22 \rightarrow 21$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2]$
$wR(F^2) = 0.224$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4156 reflections	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
363 parameters	$\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$
0 restraints	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1A	0.3491 (2)	0.31532 (15)	0.1257 (5)	0.0428 (9)
O2A	0.5630 (2)	0.44236 (15)	0.4206 (4)	0.0441 (9)
O3A	0.4152 (2)	0.43894 (14)	0.2299 (4)	0.0384 (9)
O4A	0.3270 (2)	0.41710 (15)	0.4258 (4)	0.0413 (9)
O5A	0.31114 (19)	0.50794 (14)	0.2257 (4)	0.0367 (8)
N1A	0.4539 (3)	0.37502 (17)	0.2925 (5)	0.0375 (10)

C1A	0.4144 (3)	0.3116 (2)	0.2245 (7)	0.0370 (12)
C2A	0.4578 (3)	0.2448 (2)	0.2868 (6)	0.0334 (11)
C3A	0.5339 (3)	0.2484 (2)	0.4016 (6)	0.0335 (11)
C4A	0.5716 (3)	0.3145 (2)	0.4574 (6)	0.0359 (12)
C5A	0.5329 (3)	0.3829 (2)	0.3934 (6)	0.0344 (12)
C6A	0.4237 (3)	0.1799 (2)	0.2309 (6)	0.0361 (12)
H6AA	0.3722	0.1785	0.1543	0.043*
C7A	0.4646 (3)	0.1149 (2)	0.2864 (7)	0.0400 (13)
H7AA	0.4415	0.0700	0.2451	0.048*
C8A	0.5375 (3)	0.1174 (2)	0.3996 (7)	0.0369 (12)
H8AA	0.5642	0.0735	0.4376	0.044*
C9A	0.5748 (3)	0.1828 (2)	0.4622 (7)	0.0365 (12)
C10A	0.6507 (3)	0.1873 (2)	0.5762 (7)	0.0411 (13)
H10A	0.6786	0.1443	0.6171	0.049*
C11A	0.6852 (3)	0.2519 (2)	0.6293 (6)	0.0385 (12)
H11A	0.7365	0.2534	0.7069	0.046*
C12A	0.6457 (3)	0.3158 (2)	0.5705 (6)	0.0378 (12)
H12A	0.6701	0.3606	0.6087	0.045*
C13A	0.3473 (3)	0.4514 (2)	0.3079 (7)	0.0354 (12)
C14A	0.2353 (3)	0.5276 (2)	0.2859 (6)	0.0427 (13)
H14A	0.2134	0.5717	0.2253	0.064*
H14B	0.2459	0.5358	0.4147	0.064*
H14C	0.1953	0.4886	0.2583	0.064*
O1B	0.1669 (2)	0.82517 (15)	0.5198 (4)	0.0392 (8)
O2B	-0.0410 (2)	0.95254 (15)	0.2005 (4)	0.0468 (10)
O3B	0.1021 (2)	0.94818 (14)	0.4052 (4)	0.0384 (9)
O4B	0.1866 (2)	0.92546 (16)	0.2003 (4)	0.0434 (9)
O5B	0.1998 (2)	1.02184 (14)	0.3847 (4)	0.0392 (9)
N1B	0.0622 (3)	0.88420 (17)	0.3479 (5)	0.0363 (10)
C1B	0.1017 (3)	0.8213 (2)	0.4202 (7)	0.0359 (12)
C2B	0.0594 (3)	0.7545 (2)	0.3564 (6)	0.0325 (11)
C3B	-0.0186 (3)	0.7576 (2)	0.2461 (6)	0.0358 (12)
C4B	-0.0567 (3)	0.8244 (2)	0.1926 (6)	0.0349 (12)
C5B	-0.0156 (3)	0.8933 (2)	0.2436 (7)	0.0376 (12)
C6B	0.0945 (3)	0.6896 (2)	0.4060 (6)	0.0363 (12)
H6BA	0.1462	0.6879	0.4820	0.044*
C7B	0.0547 (3)	0.6251 (2)	0.3450 (7)	0.0392 (13)
H7BA	0.0803	0.5802	0.3780	0.047*
C8B	-0.0194 (3)	0.6266 (2)	0.2403 (7)	0.0400 (13)
H8BA	-0.0454	0.5825	0.2016	0.048*
C9B	-0.0595 (3)	0.6923 (2)	0.1864 (6)	0.0356 (12)
C10B	-0.1376 (3)	0.6966 (2)	0.0811 (7)	0.0432 (13)
H10B	-0.1652	0.6535	0.0395	0.052*
C11B	-0.1745 (3)	0.7611 (2)	0.0371 (7)	0.0459 (13)
H11B	-0.2282	0.7626	-0.0307	0.055*
C12B	-0.1335 (3)	0.8257 (2)	0.0917 (7)	0.0415 (13)
H12B	-0.1592	0.8705	0.0585	0.050*
C13B	0.1661 (3)	0.9614 (2)	0.3143 (7)	0.0365 (12)

C14B	0.2722 (3)	1.0450 (2)	0.3140 (7)	0.0454 (14)
H14D	0.2953	1.0880	0.3779	0.068*
H14E	0.2569	1.0562	0.1871	0.068*
H14F	0.3132	1.0063	0.3292	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.037 (2)	0.0392 (18)	0.047 (2)	0.0021 (16)	-0.0087 (18)	-0.0011 (15)
O2A	0.044 (2)	0.0267 (16)	0.058 (2)	-0.0019 (16)	-0.0013 (18)	-0.0040 (15)
O3A	0.037 (2)	0.0299 (16)	0.046 (2)	0.0043 (15)	-0.0004 (17)	0.0045 (14)
O4A	0.048 (2)	0.0313 (16)	0.044 (2)	0.0042 (16)	0.0033 (18)	0.0056 (16)
O5A	0.039 (2)	0.0245 (15)	0.045 (2)	0.0049 (15)	0.0022 (17)	0.0062 (14)
N1A	0.038 (3)	0.0219 (19)	0.049 (3)	0.0043 (18)	-0.003 (2)	-0.0012 (17)
C1A	0.041 (4)	0.029 (2)	0.040 (3)	0.001 (2)	0.003 (3)	0.000 (2)
C2A	0.034 (3)	0.026 (2)	0.040 (3)	-0.003 (2)	0.005 (2)	0.001 (2)
C3A	0.036 (3)	0.027 (2)	0.037 (3)	0.002 (2)	0.005 (2)	0.003 (2)
C4A	0.036 (3)	0.032 (2)	0.038 (3)	-0.002 (2)	0.000 (2)	-0.002 (2)
C5A	0.038 (3)	0.031 (3)	0.033 (3)	-0.003 (2)	0.004 (2)	0.000 (2)
C6A	0.036 (3)	0.035 (3)	0.037 (3)	-0.006 (2)	0.005 (2)	-0.001 (2)
C7A	0.043 (4)	0.029 (2)	0.048 (3)	-0.002 (2)	0.010 (3)	-0.006 (2)
C8A	0.043 (4)	0.029 (2)	0.038 (3)	0.004 (2)	0.006 (3)	0.002 (2)
C9A	0.037 (3)	0.029 (2)	0.044 (3)	0.002 (2)	0.007 (3)	-0.005 (2)
C10A	0.039 (3)	0.035 (3)	0.048 (4)	0.007 (2)	0.006 (3)	0.002 (2)
C11A	0.029 (3)	0.045 (3)	0.039 (3)	0.002 (2)	-0.005 (2)	-0.001 (2)
C12A	0.042 (3)	0.034 (2)	0.037 (3)	0.000 (2)	0.004 (3)	-0.009 (2)
C13A	0.036 (3)	0.031 (2)	0.038 (3)	-0.004 (2)	0.003 (3)	-0.003 (2)
C14A	0.041 (3)	0.037 (3)	0.046 (3)	0.002 (2)	-0.002 (3)	0.002 (2)
O1B	0.035 (2)	0.0361 (17)	0.044 (2)	0.0022 (16)	-0.0005 (17)	-0.0033 (15)
O2B	0.050 (3)	0.0270 (17)	0.059 (3)	0.0022 (16)	-0.0037 (19)	0.0046 (15)
O3B	0.040 (2)	0.0257 (16)	0.048 (2)	-0.0046 (15)	0.0019 (18)	-0.0046 (14)
O4B	0.050 (2)	0.0384 (18)	0.041 (2)	-0.0032 (16)	0.0047 (18)	-0.0054 (16)
O5B	0.046 (2)	0.0265 (16)	0.043 (2)	-0.0059 (15)	0.0031 (17)	-0.0044 (14)
N1B	0.037 (3)	0.0213 (19)	0.046 (3)	-0.0027 (18)	-0.006 (2)	-0.0020 (17)
C1B	0.033 (3)	0.036 (3)	0.037 (3)	0.005 (2)	0.002 (3)	0.001 (2)
C2B	0.035 (3)	0.027 (2)	0.035 (3)	0.001 (2)	0.002 (2)	-0.007 (2)
C3B	0.040 (3)	0.030 (2)	0.038 (3)	0.001 (2)	0.005 (2)	0.003 (2)
C4B	0.033 (3)	0.032 (2)	0.038 (3)	-0.004 (2)	0.001 (2)	0.000 (2)
C5B	0.040 (4)	0.032 (3)	0.041 (3)	-0.001 (2)	0.006 (3)	-0.002 (2)
C6B	0.039 (3)	0.031 (2)	0.039 (3)	0.001 (2)	0.008 (2)	0.003 (2)
C7B	0.047 (4)	0.025 (2)	0.046 (3)	0.002 (2)	0.010 (3)	0.003 (2)
C8B	0.040 (4)	0.030 (2)	0.050 (4)	-0.004 (2)	0.006 (3)	-0.002 (2)
C9B	0.037 (3)	0.036 (3)	0.032 (3)	-0.005 (2)	0.003 (2)	-0.002 (2)
C10B	0.041 (4)	0.037 (3)	0.049 (3)	-0.009 (2)	0.000 (3)	0.005 (2)
C11B	0.042 (3)	0.048 (3)	0.045 (3)	-0.006 (3)	-0.001 (3)	0.001 (2)
C12B	0.040 (4)	0.037 (3)	0.046 (3)	0.001 (2)	0.002 (3)	0.006 (2)
C13B	0.036 (3)	0.030 (3)	0.042 (3)	0.002 (2)	0.000 (3)	0.007 (2)
C14B	0.044 (4)	0.037 (3)	0.055 (4)	-0.010 (2)	0.007 (3)	0.001 (2)

Geometric parameters (Å, °)

O1A—C1A	1.211 (6)	O1B—C1B	1.212 (5)
O2A—C5A	1.215 (5)	O2B—C5B	1.204 (5)
O3A—C13A	1.371 (5)	O3B—C13B	1.377 (5)
O3A—N1A	1.395 (4)	O3B—N1B	1.394 (4)
O4A—C13A	1.193 (5)	O4B—C13B	1.188 (5)
O5A—C13A	1.316 (5)	O5B—C13B	1.326 (5)
O5A—C14A	1.449 (5)	O5B—C14B	1.455 (5)
N1A—C1A	1.403 (6)	N1B—C5B	1.405 (6)
N1A—C5A	1.408 (6)	N1B—C1B	1.406 (6)
C1A—C2A	1.473 (6)	C1B—C2B	1.468 (6)
C2A—C6A	1.370 (6)	C2B—C6B	1.365 (6)
C2A—C3A	1.409 (6)	C2B—C3B	1.419 (6)
C3A—C4A	1.410 (6)	C3B—C4B	1.419 (6)
C3A—C9A	1.432 (6)	C3B—C9B	1.427 (6)
C4A—C12A	1.377 (7)	C4B—C12B	1.371 (7)
C4A—C5A	1.471 (6)	C4B—C5B	1.472 (6)
C6A—C7A	1.413 (6)	C6B—C7B	1.408 (6)
C6A—H6AA	0.9500	C6B—H6BA	0.9500
C7A—C8A	1.362 (7)	C7B—C8B	1.347 (7)
C7A—H7AA	0.9500	C7B—H7BA	0.9500
C8A—C9A	1.410 (6)	C8B—C9B	1.417 (6)
C8A—H8AA	0.9500	C8B—H8BA	0.9500
C9A—C10A	1.405 (7)	C9B—C10B	1.404 (7)
C10A—C11A	1.361 (6)	C10B—C11B	1.362 (6)
C10A—H10A	0.9500	C10B—H10B	0.9500
C11A—C12A	1.392 (6)	C11B—C12B	1.406 (6)
C11A—H11A	0.9500	C11B—H11B	0.9500
C12A—H12A	0.9500	C12B—H12B	0.9500
C14A—H14A	0.9800	C14B—H14D	0.9800
C14A—H14B	0.9800	C14B—H14E	0.9800
C14A—H14C	0.9800	C14B—H14F	0.9800
C13A—O3A—N1A	110.9 (3)	C13B—O3B—N1B	111.0 (3)
C13A—O5A—C14A	113.6 (3)	C13B—O5B—C14B	114.6 (4)
O3A—N1A—C1A	115.4 (4)	O3B—N1B—C5B	114.5 (3)
O3A—N1A—C5A	115.4 (3)	O3B—N1B—C1B	114.9 (4)
C1A—N1A—C5A	128.4 (4)	C5B—N1B—C1B	130.2 (4)
O1A—C1A—N1A	119.6 (4)	O1B—C1B—N1B	120.2 (4)
O1A—C1A—C2A	125.8 (4)	O1B—C1B—C2B	125.7 (4)
N1A—C1A—C2A	114.6 (5)	N1B—C1B—C2B	114.0 (5)
C6A—C2A—C3A	120.9 (4)	C6B—C2B—C3B	120.2 (4)
C6A—C2A—C1A	119.3 (5)	C6B—C2B—C1B	119.8 (5)
C3A—C2A—C1A	119.8 (4)	C3B—C2B—C1B	119.9 (4)
C2A—C3A—C4A	122.2 (4)	C2B—C3B—C4B	121.4 (4)
C2A—C3A—C9A	119.0 (4)	C2B—C3B—C9B	119.3 (4)
C4A—C3A—C9A	118.8 (5)	C4B—C3B—C9B	119.2 (5)

C12A—C4A—C3A	120.5 (4)	C12B—C4B—C3B	120.1 (4)
C12A—C4A—C5A	119.1 (4)	C12B—C4B—C5B	118.5 (4)
C3A—C4A—C5A	120.4 (5)	C3B—C4B—C5B	121.4 (5)
O2A—C5A—N1A	120.3 (4)	O2B—C5B—N1B	120.7 (4)
O2A—C5A—C4A	125.9 (5)	O2B—C5B—C4B	126.7 (5)
N1A—C5A—C4A	113.8 (4)	N1B—C5B—C4B	112.5 (4)
C2A—C6A—C7A	120.5 (5)	C2B—C6B—C7B	120.4 (5)
C2A—C6A—H6AA	119.7	C2B—C6B—H6BA	119.8
C7A—C6A—H6AA	119.7	C7B—C6B—H6BA	119.8
C8A—C7A—C6A	119.3 (4)	C8B—C7B—C6B	120.5 (4)
C8A—C7A—H7AA	120.3	C8B—C7B—H7BA	119.8
C6A—C7A—H7AA	120.3	C6B—C7B—H7BA	119.8
C7A—C8A—C9A	122.3 (4)	C7B—C8B—C9B	121.7 (4)
C7A—C8A—H8AA	118.8	C7B—C8B—H8BA	119.1
C9A—C8A—H8AA	118.8	C9B—C8B—H8BA	119.1
C10A—C9A—C8A	123.8 (4)	C10B—C9B—C8B	123.8 (4)
C10A—C9A—C3A	118.3 (4)	C10B—C9B—C3B	118.4 (4)
C8A—C9A—C3A	117.9 (5)	C8B—C9B—C3B	117.8 (5)
C11A—C10A—C9A	121.6 (4)	C11B—C10B—C9B	121.5 (5)
C11A—C10A—H10A	119.2	C11B—C10B—H10B	119.2
C9A—C10A—H10A	119.2	C9B—C10B—H10B	119.2
C10A—C11A—C12A	120.3 (5)	C10B—C11B—C12B	120.2 (5)
C10A—C11A—H11A	119.9	C10B—C11B—H11B	119.9
C12A—C11A—H11A	119.9	C12B—C11B—H11B	119.9
C4A—C12A—C11A	120.5 (4)	C4B—C12B—C11B	120.5 (5)
C4A—C12A—H12A	119.7	C4B—C12B—H12B	119.8
C11A—C12A—H12A	119.7	C11B—C12B—H12B	119.8
O4A—C13A—O5A	128.5 (4)	O4B—C13B—O5B	128.3 (5)
O4A—C13A—O3A	126.0 (4)	O4B—C13B—O3B	126.9 (4)
O5A—C13A—O3A	105.6 (4)	O5B—C13B—O3B	104.7 (4)
O5A—C14A—H14A	109.5	O5B—C14B—H14D	109.5
O5A—C14A—H14B	109.5	O5B—C14B—H14E	109.5
H14A—C14A—H14B	109.5	H14D—C14B—H14E	109.5
O5A—C14A—H14C	109.5	O5B—C14B—H14F	109.5
H14A—C14A—H14C	109.5	H14D—C14B—H14F	109.5
H14B—C14A—H14C	109.5	H14E—C14B—H14F	109.5
C13A—O3A—N1A—C1A	75.2 (5)	C13B—O3B—N1B—C5B	-107.2 (4)
C13A—O3A—N1A—C5A	-113.9 (4)	C13B—O3B—N1B—C1B	79.7 (5)
O3A—N1A—C1A—O1A	-3.6 (6)	O3B—N1B—C1B—O1B	-2.2 (6)
C5A—N1A—C1A—O1A	-173.0 (5)	C5B—N1B—C1B—O1B	-173.9 (5)
O3A—N1A—C1A—C2A	177.8 (4)	O3B—N1B—C1B—C2B	-179.2 (3)
C5A—N1A—C1A—C2A	8.3 (7)	C5B—N1B—C1B—C2B	9.1 (7)
O1A—C1A—C2A—C6A	0.3 (8)	O1B—C1B—C2B—C6B	-1.7 (8)
N1A—C1A—C2A—C6A	178.8 (4)	N1B—C1B—C2B—C6B	175.1 (4)
O1A—C1A—C2A—C3A	179.8 (5)	O1B—C1B—C2B—C3B	177.2 (4)
N1A—C1A—C2A—C3A	-1.6 (6)	N1B—C1B—C2B—C3B	-6.0 (7)
C6A—C2A—C3A—C4A	178.2 (4)	C6B—C2B—C3B—C4B	180.0 (4)

C1A—C2A—C3A—C4A	-1.3 (7)	C1B—C2B—C3B—C4B	1.1 (7)
C6A—C2A—C3A—C9A	-1.1 (7)	C6B—C2B—C3B—C9B	-0.5 (7)
C1A—C2A—C3A—C9A	179.4 (4)	C1B—C2B—C3B—C9B	-179.4 (4)
C2A—C3A—C4A—C12A	179.6 (4)	C2B—C3B—C4B—C12B	-177.4 (4)
C9A—C3A—C4A—C12A	-1.1 (7)	C9B—C3B—C4B—C12B	3.1 (7)
C2A—C3A—C4A—C5A	-1.3 (7)	C2B—C3B—C4B—C5B	2.3 (7)
C9A—C3A—C4A—C5A	178.0 (4)	C9B—C3B—C4B—C5B	-177.2 (4)
O3A—N1A—C5A—O2A	-0.2 (7)	O3B—N1B—C5B—O2B	4.6 (6)
C1A—N1A—C5A—O2A	169.3 (4)	C1B—N1B—C5B—O2B	176.3 (4)
O3A—N1A—C5A—C4A	179.8 (3)	O3B—N1B—C5B—C4B	-177.6 (3)
C1A—N1A—C5A—C4A	-10.7 (7)	C1B—N1B—C5B—C4B	-5.9 (7)
C12A—C4A—C5A—O2A	5.6 (8)	C12B—C4B—C5B—O2B	-3.0 (8)
C3A—C4A—C5A—O2A	-173.4 (5)	C3B—C4B—C5B—O2B	177.3 (5)
C12A—C4A—C5A—N1A	-174.3 (4)	C12B—C4B—C5B—N1B	179.4 (4)
C3A—C4A—C5A—N1A	6.6 (7)	C3B—C4B—C5B—N1B	-0.3 (7)
C3A—C2A—C6A—C7A	-0.5 (7)	C3B—C2B—C6B—C7B	1.4 (7)
C1A—C2A—C6A—C7A	179.0 (4)	C1B—C2B—C6B—C7B	-179.7 (4)
C2A—C6A—C7A—C8A	1.5 (7)	C2B—C6B—C7B—C8B	-1.5 (7)
C6A—C7A—C8A—C9A	-0.9 (7)	C6B—C7B—C8B—C9B	0.6 (8)
C7A—C8A—C9A—C10A	-178.7 (5)	C7B—C8B—C9B—C10B	-178.4 (5)
C7A—C8A—C9A—C3A	-0.6 (7)	C7B—C8B—C9B—C3B	0.3 (7)
C2A—C3A—C9A—C10A	179.8 (4)	C2B—C3B—C9B—C10B	178.4 (4)
C4A—C3A—C9A—C10A	0.5 (7)	C4B—C3B—C9B—C10B	-2.1 (7)
C2A—C3A—C9A—C8A	1.6 (7)	C2B—C3B—C9B—C8B	-0.4 (7)
C4A—C3A—C9A—C8A	-177.7 (4)	C4B—C3B—C9B—C8B	179.2 (4)
C8A—C9A—C10A—C11A	178.2 (4)	C8B—C9B—C10B—C11B	178.1 (5)
C3A—C9A—C10A—C11A	0.2 (8)	C3B—C9B—C10B—C11B	-0.6 (8)
C9A—C10A—C11A—C12A	-0.3 (7)	C9B—C10B—C11B—C12B	2.3 (8)
C3A—C4A—C12A—C11A	1.0 (8)	C3B—C4B—C12B—C11B	-1.4 (8)
C5A—C4A—C12A—C11A	-178.0 (4)	C5B—C4B—C12B—C11B	178.9 (4)
C10A—C11A—C12A—C4A	-0.3 (7)	C10B—C11B—C12B—C4B	-1.3 (8)
C14A—O5A—C13A—O4A	-2.6 (7)	C14B—O5B—C13B—O4B	-1.5 (7)
C14A—O5A—C13A—O3A	177.5 (3)	C14B—O5B—C13B—O3B	177.3 (3)
N1A—O3A—C13A—O4A	6.8 (6)	N1B—O3B—C13B—O4B	0.7 (7)
N1A—O3A—C13A—O5A	-173.3 (3)	N1B—O3B—C13B—O5B	-178.1 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6A—H6AA \cdots O4A ⁱ	0.95	2.51	3.159 (5)	125
C7B—H7BA \cdots O2B ⁱⁱ	0.95	2.51	3.229 (5)	133
C10B—H10B \cdots O5B ⁱⁱ	0.95	2.60	3.428 (5)	146
C11B—H11B \cdots O1A ⁱⁱⁱ	0.95	2.48	3.270 (6)	141
C14A—H14A \cdots O1B ^{iv}	0.98	2.51	3.481 (5)	169
C14B—H14E \cdots O4A ^{iv}	0.98	2.51	3.306 (6)	138

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