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Synthesis and crystal structures of a bis(3-hydroxy-cyclohex-2-en-1-one) and two hexahydroquinoline derivatives

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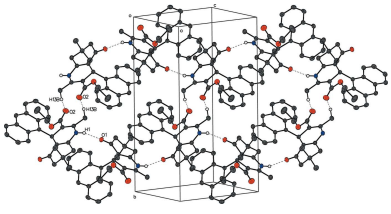
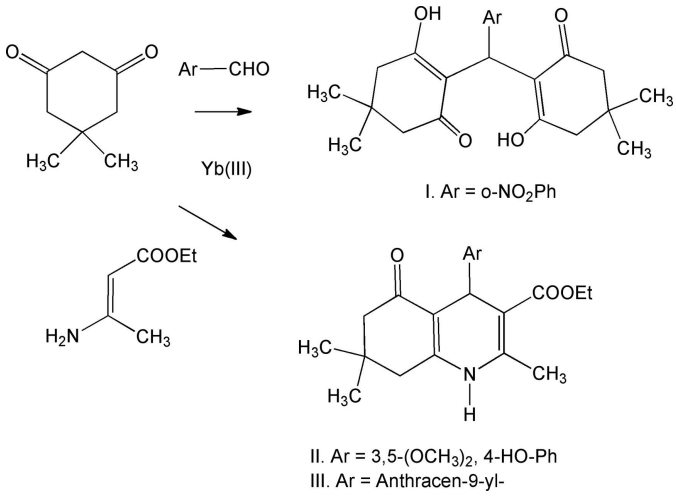
The title compound **I**, 2,2′-[(2-nitrophenyl)methylene]bis(3-hydroxy-5,5-dimethylcyclohex-2-enone), $C_{23}H_{27}NO_6$, features a 1,3-ketone–enol conformation which is stabilized by two intramolecular hydrogen bonds. The most prominent intermolecular interactions in compound **I** are C—H···O hydrogen bonds, which link molecules into a two-dimensional network parallel to the (001) plane and a chain perpendicular to $(1\bar{1}1)$. Both title compounds **II**, ethyl 4-(4-hydroxy-3,5-dimethoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{23}H_{29}NO_6$, and **III**, ethyl 4-(anthracen-9-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{29}H_{29}NO_3$, share the same structural features, such as a shallow boat conformation of the dihydropyridine group and an orthogonal aryl group attached to the dihydropyridine. Intermolecular N—H···O bonding is present in the crystal packing of both compound **II** and **III**.

1. Chemical context

4-Aryl-1,4-dihydropyridines (DHPs) that bind the L-type voltage-gated calcium channels (VGCC) have been applied in general medical practice for over three decades. (Zamponi, 2016). Many modifications on 1,4-DHP have been performed to obtain active compounds such as calcium-channel agonists or antagonists. (Martín *et al.*, 1995; Rose, 1990; Rose & Dräger, 1992; Trippier *et al.* 2013) One such modification is fusing a cyclohexanone ring to form hexahydroquinolone (HHQ), in which the orientation of the carbonyl group of the ester substituent at the 5-position in the 1,4-DHP ring has been fixed. This class of compounds has been shown to have calcium-channel antagonistic activity (Aygün Cevher *et al.*, 2019), inhibit the multidrug-resistance transporter (MDR) (Shahraki *et al.*, 2017), as well as possess anti-inflammatory and stem-cell differentiation properties, and has been implicated in slowing neurodegenerative disorders. (Trippier *et al.*, 2013). In the HHQ literature, specific substitution of the cyclohexenone ring can confer sub-type selectivity at the voltage-gated calcium channel (Schaller *et al.*, 2018). Our group has been interested in bioisosteric 4-isoxazolyl-dihydropyridines at the VGCC (Schauer *et al.*, 1986; Zamponi *et al.*, 2003; Natale *et al.*, 2014) and MDR (Steiger *et al.*, 2017), and continue our studies towards understanding stereoelectronic effects, which define selectivity, as well as to explore the scope and limitations of our synthetic methodologies (Steiger *et al.*, 2016). These interests led us to continue our pursuit of crystallographic studies in this area (Steiger *et al.*, 2014*a*,*b*; 2018).





2. Structural commentary

Compound **I** crystallizes in the triclinic $P\overline{1}$ space group with one independent molecule in the asymmetric unit (Fig. 1). As in other bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) compounds, in compound **I** the 1,3-ketone–enol conformation is stabilized by two internal hydrogen bonds between two pairs of enols and ketones that bridge the two hydroxycyclohexenones, in addition to the bridging carbon C7. The two hydroxycyclohexenones are arranged along a pseudo-mirror plane formed by atoms C15, C11, C8, C7, C16, C19, and C22, which has a root-mean-square deviation (RMSD) of 0.025 Å. The phenyl ring attached to C7 flaps to one side of the above plane, with a plane normal angle of 44.34 (4)°.

Both 3-hydroxy-5,5-dimethyl-cyclohex-2-en-1-one rings adopt an envelope conformation, with both methyl groups

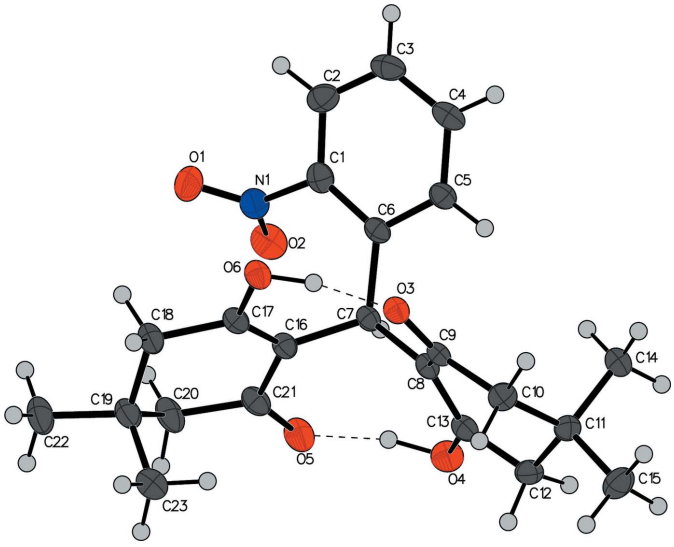


Figure 1
The asymmetric unit of compound **I** showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed lines indicate intramolecular O—H···O hydrogen bonds.

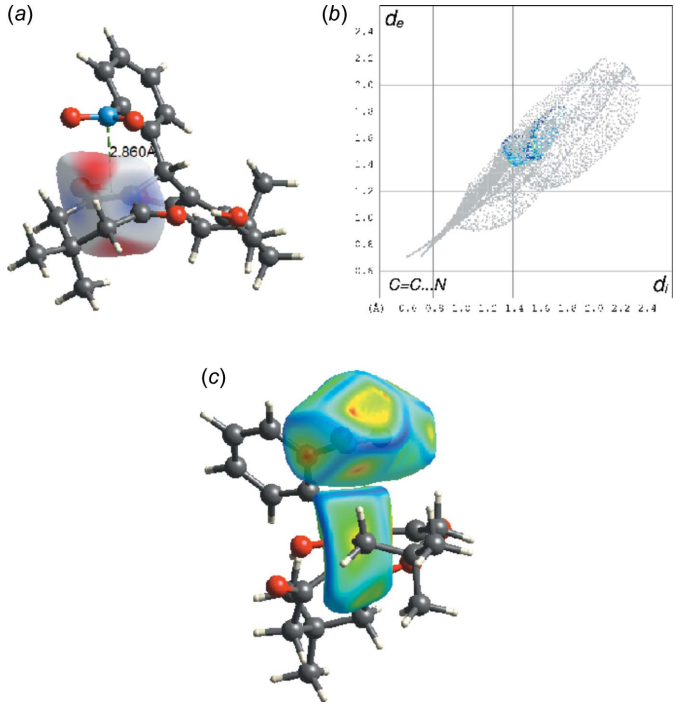


Figure 2
(*a*) View of the three-dimensional Hirshfeld surface of C16—C17 mapped over electrostatic potentials, over the range of −0.0221 to 0.9216 arbitrary units. (*b*) The two-dimensional fingerprint plot for the C=C···N interaction. (*c*) The Hirshfeld surfaces of NO_2 and C16—C17 mapped over curvedness.

C14 and C23 having an axial orientation being trans to each other. As a result of the steric effect of the neighboring atoms and groups, instead of being on the same plane as the phenyl ring, the mean plane formed by the NO_2 group is rotated out of the plane of the aromatic system with an angle of 52.85 (6)°. This may indicate a possible π–π interaction between the NO_2 group and the ketone–enol C═C bond, evidenced by a short-contact N1···C16 distance of 2.816 (2) Å and a short distance of 2.860 Å between N1 and the midpoint of the C16═C17 double bond. The interaction of the NO_2 group and the enol C16═C17 double bond were analyzed using Hirshfeld surface analysis and quantified using the associated two-dimensional fingerprint plot (Fig. 2), both performed with *CrystalExplorer17.5* (Turner *et al.*, 2017). The electrostatic potentials were calculated using *TONTO* integrated within *CrystalExplorer*. Hirshfeld surfaces of the NO_2 group and C16═C17 mapped over curvedness are shown in Fig. 2(c). The flat yellowish surfaces confirm that an intramolecular π–π interaction takes place between the NO_2 group and the enol double bond. This is also evidence that the π-hole interaction can stabilize conformers when the interacting atom is four or five bonds away from the N atom of a nitro aromatic compound (Franconetti *et al.*, 2019).

Compounds **II** and **III** both crystallized racemically in the monoclinic space group $P2_1/n$. The asymmetric unit of compound **II** contains two independent molecules (*A* and *B*), both in the same enantiomeric configuration. The overall unit cell is racemic with four pairs of racemates. Compound **III** has

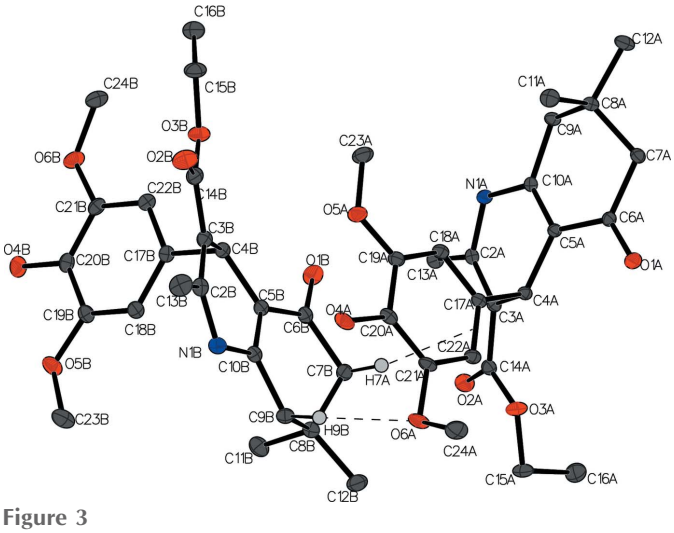


Figure 3
The asymmetric unit of compound **II** showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed lines indicate the C9*B*—H9*B*···O6*A* hydrogen bond and the C—H···π interaction between H7*A* and the C17*A*—C22*A* bond. Other hydrogen atoms have been omitted for clarity.

only one independent molecule in the asymmetric unit. The displacement ellipsoid plots showing the atomic numbering of compounds **II** and **III** are presented in Figs. 3 and 4, respectively.

As in the other 4-aryl-hexahydroquinoline derivatives (Steiger *et al.*, 2014*a*,*b*; 2018) that we have reported, compound **II** has a flattened boat conformation on the 1,4-DHP ring. The mean plane defined by atoms C2, C3, C5, and C10 is planar with an RMSD of 0.000 and 0.006 Å for *A* and *B*, respectively. Atoms N1 and C4 are displaced slightly from the mean plane at distances of 0.1696 (11) Å for N1*A* and 0.1867 (11) Å for N1*B*, and 0.3722 (13) Å for C4*A* and 0.3506 (13) Å for C4*B*, respectively. The 4-dihydroxylmethoxyphenyl ring is almost orthogonal to the 1,4-DHP basal plane comprising atoms C2, C3, C5, and C10, making dihedral angles of 88.03 (3) and 81.05 (3)° in **II*A*** and **II*B***, respectively. The ring puckering parameters for the cyclohexanone ring (C5–C10) indicate that it adopts an envelope conformation: Q = 0.4631 Å, θ = 58.01°, and φ = 168.1681° for **II*A*** and Q = 0.4592 Å, θ = 124.10°, and φ = 344.3794° for **II*B***.

In the molecule of compound **II**, the orientations of the ethyl groups on the ester and of the methoxy groups on the phenyl rings are different in molecules *A* and *B*. The hydroxyl and methoxy groups are mostly co-planar with the phenyl ring to which they are attached in both molecules *A* and *B*. The exception is one of the methyl groups in molecule *A*, C24*A*, which protrudes out of the phenyl plane with a displacement of 1.2802 (12) Å. The angle between the O6*A*—C24*A* bond and the normal to the phenyl plane is 154.38 (5)°. Similarly, the ethyl group on the ester group in molecule *B* is co-planar with the ester atoms O2*B*, O3*B*, and C14*B* whereas in molecule *A*, the ethyl group is folded with an angle of 14.94 (10)° between the C15*A*—C16*A* bond and the normal to the O2*A*/O3*A*/C14*A* plane with atom C16*A* displaced by 1.656 (3) Å from the plane. These orientations imply that these two functional groups are flexible in the structure.

Although compounds **II** and **III** share the same structural features, such as the envelope conformation of the cyclohexanone ring and the pseudo-axial position of the 4-aryl group, they exhibit differences, especially in the conformation of the 1,4-DHP ring. In compound **III**, atoms N1 and C4 are only slightly displaced from C2/C3/C5/C10 mean plane at distances of 0.107 (2) and 0.092 (2) Å, respectively. There is a short contact of 1.88 Å between hydrogen atoms H4 and H27. A C—H···π contact of 2.47 Å also exists between C19—H19 and the C5—C10 bond.

In compound **III**, the anthracenyl group bisects the basal plane of the 1,4-DHP ring, with N1···C4—C17—C18 torsion angle of 2.09 (15)°. As a result of the elongated aromatic system, the ethyl group on the ester is stabilized in a folded position by a weak C—H···π interaction between C16—H16*B* and C25–C30 ring, with an H16-to-plane distance of 2.82 Å. The O=C—O ester group is no longer co-planar with the 1,4-DHP basal plane and the O2—C14—C3—C2 torsion angle is −25.10 (19)°.

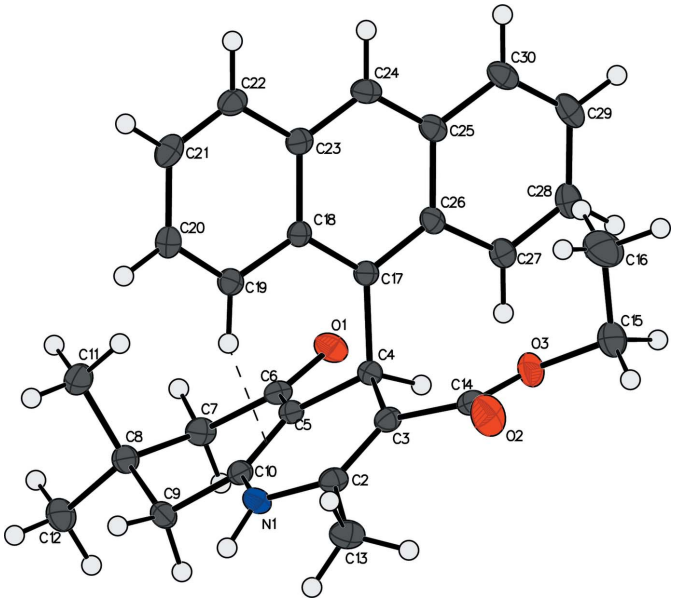


Figure 4
The asymmetric unit of compound **III** showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The C—H···π interaction is indicated by a dashed line.

3. Supramolecular features

In compound **I**, C15—H15*B*···O3^{i} and C20—H20*B*···O5^{ii} and hydrogen bonds (Table 1) between the same enantiomers form a two-dimensional network parallel to (001), with one chain running along the *a*-axis direction and the other along the *b*-axis direction (Fig. 5). Other intermolecular O—H interactions such as C10—H10*B*···O5^{ii} and C2—H2···O1^{i} between a pair of enantiomers form a chain of alternating enantiomers (Fig. 6)

In compound **II**, there is a C9*B*—H9*B*···O6*A* hydrogen bond between molecules *A* and *B*, with distance of 2.59 Å and

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O5	0.97 (3)	1.62 (3)	2.5570 (16)	162 (3)
O6—H6···O4	1.08 (4)	1.58 (4)	2.6437 (19)	166 (3)
C2—H2···O1^{i}	0.95	2.63	3.538 (2)	160
C10—H10B···O5^{ii}	0.99	2.65	3.6138 (19)	165
C15—H15B···O3^{iii}	0.98	2.58	3.505 (2)	157
C18—H18B···O1	1.04 (2)	2.67 (2)	3.381 (2)	125.5 (16)
C20—H20B···O5^{iv}	0.99	2.43	3.332 (2)	152

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

a C—H···π interaction between C7B—H7A and the C17A—C22A bond with a distance of 2.6715 (6) Å. Links alternating

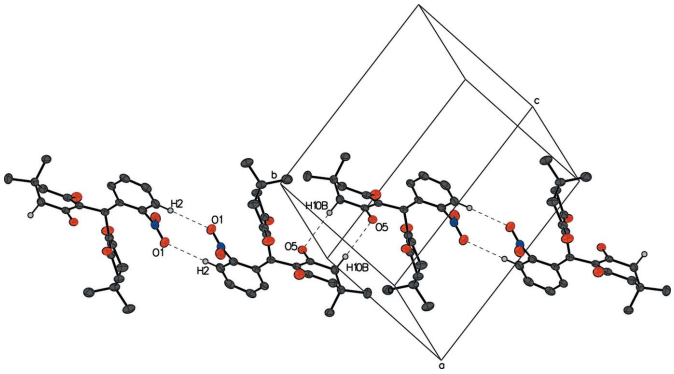


Figure 6
The packing of compound **I** showing a chain of alternating enantiomers. For clarity, H atoms not participating in hydrogen bonds are omitted, and participating atoms are labeled once.

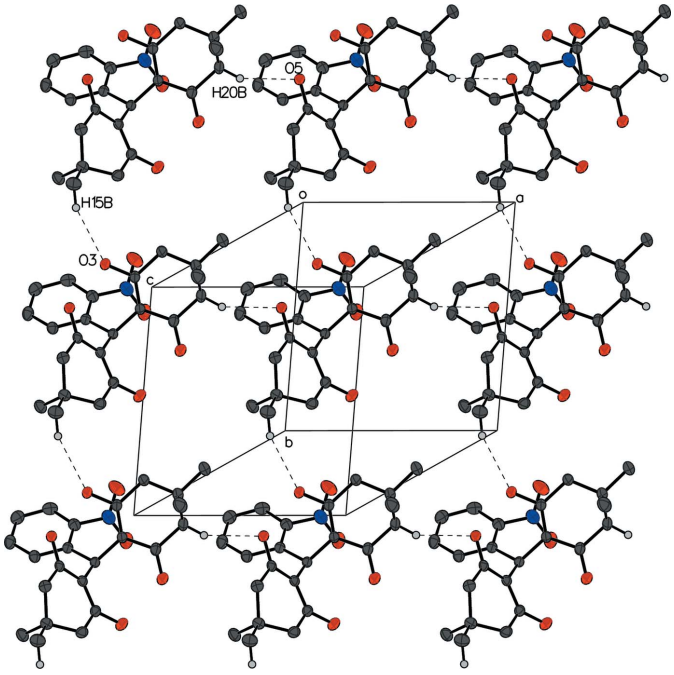


Figure 5
The packing of compound **I** showing the two-dimensional network parallel to the (001) plane. For clarity, H atoms not participating in hydrogen bonds are omitted, and participating atoms are labeled once.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4A—H4C···O1A^{i}	0.848 (17)	1.937 (17)	2.6948 (9)	148.0 (16)
N1A—H1A···O1B^{ii}	0.880 (15)	1.890 (15)	2.7666 (10)	174.1 (13)
C7A—H7C···O6B^{ii}	0.99	2.67	3.4510 (12)	136
C12A—H12D···O2A^{iii}	0.98	2.60	3.5237 (12)	157
C13A—H13D···O1B^{ii}	0.98	2.59	3.3590 (12)	136
C16A—H16D···O4A^{iv}	0.98	2.65	3.3136 (13)	126
C24A—H24E···O4B^{v}	0.98	2.43	3.3105 (13)	149
N1B—H1···O1A^{i}	0.888 (15)	2.166 (15)	2.9479 (10)	146.6 (12)
C7B—H7B···O2A	0.99	2.69	3.4992 (11)	139
C9B—H9B···O6A	0.99	2.59	3.5751 (11)	172
C15B—H15A···O5B^{iii}	0.99	2.60	3.4993 (12)	151
C23B—H23B···O2B^{vi}	0.98	2.55	3.4277 (13)	149

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

between the two independent molecules form a column through hydrogen bonds N1A—H1A···O1B^{ii} and N1B—H1···O1A^{i}, which run along the b-axis direction. This column branches out through the O4A—H4C···O1A^{i} and C24A—H24E···O4B^{v} hydrogen bonds to another parallel column, forming a sheet perpendicular to (101) (Fig. 7). Weak C23B—H23B···O2B^{vi} and C15B—H15A···O5B^{iii} interactions link the B molecules into a chain along the a-axis direction. A

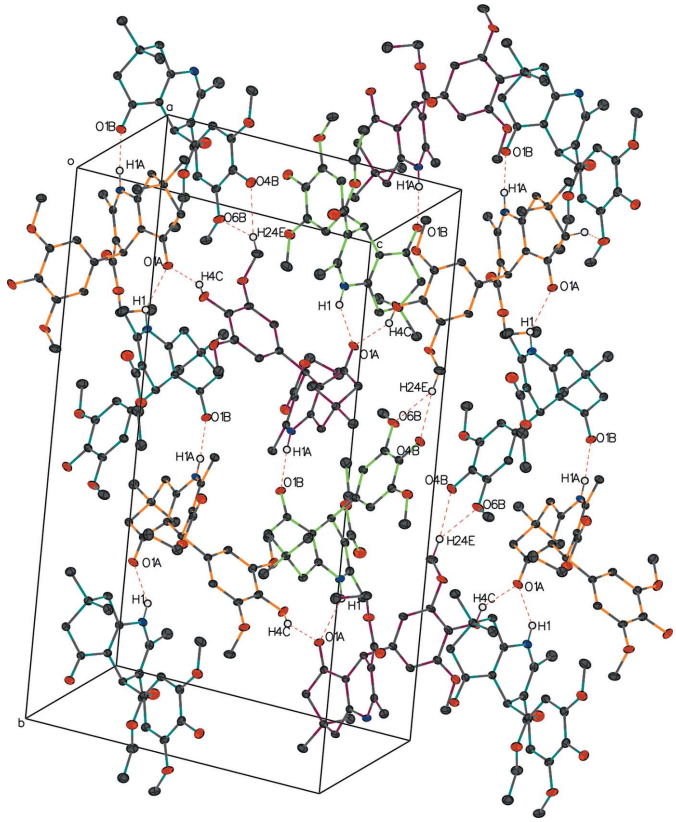


Figure 7
The packing of compound **II** showing an array of columns along the b axis formed by hydrogen bonds. Atoms involved in hydrogen bonds are labeled. H atoms not involved in hydrogen bonds are omitted for clarity. Molecules A and B are colored in orange and lime, respectively. Molecules colored in magenta are the enantiomers of molecule A, and those colored in teal are the enantiomers of molecule B.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.90 (2)	1.94 (2)	2.7776 (16)	154.2 (18)
C13—H13B···O2^{ii}	0.98	2.65	3.409 (2)	134
C19—H19···N1	0.95	2.48	3.4148 (19)	168

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

similar chain of *A* molecules is formed through weak C12*A*—H12*D*···O2*A*′ interactions (Fig. 8). Other hydrogen bonds are listed in the Table 2.

In compound **III**, an N1—H1···O1^{i} hydrogen bond (Table 3) alternating between two enantiomers results in a zigzag chain of racemic molecules running perpendicular to the (101) plane. The C13—H13*B*···O2^{ii} hydrogen bond cross-links a pair of enantiomers from different chains and forms a sheet of molecules parallel to (10$\bar{1}$) (Fig. 9). As a consequence of close packing, several short contacts are observed, *i.e.* an edge-to-edge π–π contact of 2.7636 (15) Å between C21 and C21^{ii}, H4···C29^{i} = 2.76 Å and H7*A*···H24^{i} = 2.60 Å (symmetry codes as in Table 3).

4. Database survey

A search for arylbis(3-hydroxy-5,5-dimethylcyclohex-2-enone) compounds in the Cambridge Structural Database (CSD Version 5.40, update of August 2019; Groom *et al.*, 2016) gave 29 hits, among which are two NO_2-phenylbis(3-hydroxy-5,5-dimethylcyclohex-2-enone) compounds. One is NO_2 substituted at the *para* position (CSD refcode IRODID; Yao *et al.*, 2005) while the other is NO_2 substituted at the *meta* position (VUZYIZ; Palakshi Reddy *et al.*, 2010) and both exhibit a similar structural configuration to that of compound **I**. However, with less steric effects surrounding the nitro group, both the *p*- and *m*-NO_2 groups are tilted only slightly

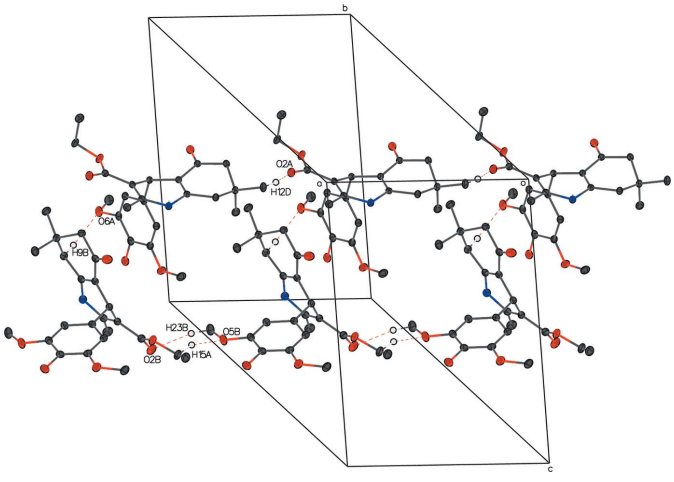


Figure 8
The packing of compound **II** showing the chains formed by *A* and *B* molecules along the *a* axis. For clarity, H atoms not participating in hydrogen bonds are omitted, and participating atoms are labeled once.

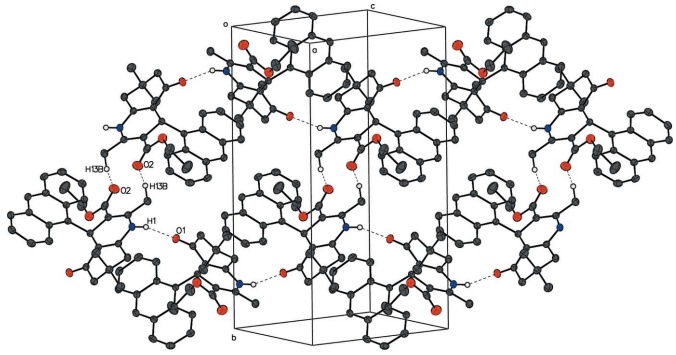


Figure 9
The packing of compound **III**. Cross-linked zigzag chains of alternating enantiomers form a sheet. For clarity, H atoms not participating in hydrogen bonds are omitted, and participating atoms are labeled once.

from the aromatic ring with torsion angles between the N═O and C═C bonds of *ca* 8.25 and 4.58°, respectively. In contrast, in compound **I** (an *o*-NO_2 group), the torsion angle is 49.68 (6)°. The database search also found 20 4-aryl-hexahydroquinoline-3-carboxylate derivatives. All of them display the same common structural features as compounds **II** and **III** in this report, such as the flat-boat conformation of the 1,4-DHP ring, the envelope conformation of the fused cyclohexanone ring, and the substituted phenyl ring at the pseudo-axial position and orthogonal to the 1,4-DHP ring.

5. Synthesis and crystallization

The synthesis was performed as outlined in the scheme. An oven-dried 100 ml round-bottom flask equipped with a magnetic stir bar was charged with 10 mmol of dimedone, 10 mmol of ethyl acetoacetate and 5 mol% of ytterbium(III) trifluoromethanesulfonate (Wang *et al.*, 2005). The mixture was then taken up in 30 ml of absolute ethanol, capped and placed under an inert atmosphere of argon, after which the solution was allowed to stir at room temperature for 20 min. The appropriate corresponding benzaldehyde (10 mmol) and 10 mmol of ammonium acetate were added to the stirring solution, the solution was allowed to stir at room temperature for 48 h. Reaction progress was monitored *via* TLC. Once the reaction was complete, excess solvent was removed *via* rotary evaporation. The solution was then purified *via* silica column chromatography. The title compound was recrystallized by slow evaporation from hexane and ethyl acetate (*v*:*v* = 3:1).

2,2′-[(2-Nitrophenyl)methylene]bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**I**). ^{1}H NMR ($CDCl_3$) δ ppm 7.99 (*d*, *J* = 7.8 Hz, 1H); 7.39 (*ddd*, *J* = 1.37, 6.88 & 8 Hz, 1H); 7.35 (*dd*, *J* = 1.83 & 8.24 Hz, 1H); 7.30 (*ddd*, *J* = 1.37, 1.83 & 7.58 Hz, 1H), 5.01 (*s*, 1H); 3.35 (*s*, 1H); 2. 82 (*s*, 1H); 2.45 (*dd*, *J* = 4.35 & 14.76 Hz, 2H); 2.25 (*m*, 4H); 2.10 (*dd*, *J* = 1.83 & 14.20 Hz, 1 Hz); 2.04 (*d*, *J* =14.20 Hz, 1H); 1.14 (*s*, 3H); 1.11 (*s*, 3H); 1.04 (*s*, 3H); 0.95 (*s*, 3H). ^{13}C NMR δ ppm 190.99, 189.51, 149.79, 132.16, 131.46, 129.67, 127.27, 124.44, 114.73, 46.93, 46.35, 32.00, 30.14, 28.62, 28.25. LC–MS calculated for $C_{23}H_{27}NO_6$, observed *m*/*z* 414 ([*M*+1]$^{+}$, 100% rel. intensity).

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{23}H_{27}NO_6$	$C_{23}H_{29}NO_6$	$C_{29}H_{29}NO_3$
M_r	413.45	415.47	439.53
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
Temperature (K)	100	100	100
a, b, c (Å)	8.7024 (3), 9.8709 (4), 13.1621 (5)	10.8854 (4), 25.2446 (10), 15.3665 (6)	11.6527 (3), 18.1986 (4), 12.3435 (3)
α, β, γ (°)	90.3822 (19), 108.9608 (18), 97.3571 (18)	90, 100.7606 (19), 90	90, 114.8758 (12), 90
V (Å^3)	1059.08 (7)	4148.4 (3)	2374.74 (10)
Z	2	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.10	0.08
Crystal size (mm)	0.39 × 0.25 × 0.13	0.48 × 0.43 × 0.31	0.45 × 0.12 × 0.11
Data collection			
Diffractometer	Bruker SMART BREEZE CCD	Bruker SMART BREEZE CCD	Bruker SMART BREEZE CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29458, 5315, 4254	168826, 12707, 11044	72579, 5902, 4515
R_{int}	0.031	0.045	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.683	0.716	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.054, 0.154, 1.04	0.039, 0.108, 1.04	0.048, 0.135, 1.04
No. of reflections	5315	12707	5902
No. of parameters	291	569	306
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.65, −0.25	0.52, −0.21	0.54, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Ethyl 4-(4-hydroxy-3,5-dimethoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (II). Spectra are similar to those for the product of the synthesis previously reported by Yang *et al.* (2011). ^{1}H NMR ($CDCl_3$) δ ppm 6.56 (*s*, 2H, Ar-H); 5.69 (*br.s*, 1H); 5.33 (*s*, 1H); 5.01 (*s*, 1H); 4.10 (*q*, 2H, J = 6Hz); 3.83 (*s*, 6H); 2.39 (*s*, 3H); 2.36, *s*, 1H); 2.225 (*q*, 2H, J = 16 Hz); 2.18 (*s*, 1H); 1.24 (*t*, 3H, J = 6Hz); 1.10(*s*, 3H); 0.99 (*s*, 3H). ^{13}C NMR δ ppm 195.47, 167.42, 147.49, 146.49, 133.15, 112.33, 106.26, 104.98, 59.82, 56.23, 50.69, 36.34, 32.69, 29.58, 26.84, 19.53, 14.33. LC–MS calculated for $C_{23}H_{29}NO_6$, observed m/z 831 ([M_2+1]$^+$, 100% rel. intensity), 416 ([M+1]$^+$, 74), 262 ([M-4-Ar-H]$^+$, 81).

Ethyl 4-(9′-Anthryl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**III**). ^{1}H NMR ($CDCl_3$) δ ppm 9.09 (*d*, 1H); 8.29 (*s*, 2H); 7.93 (*m*, 2H); 7.57 (*m*, 1H); 7.43 (*m*, 1H); 7.33 (*m*, 2H); 6.68 (*s*, 1H); 5.92 (*br. s*, 1H); 3.7 (*m*, 2H, O*CH*$_2$CH$_3$), 2.06 (*d*, 1H, J = 16 Hz); 1.97 (*d*, 1H, J = 16 Hz); 0.5 (*t*, 3H, OCH$_2$*CH*$_3$, J = 8 Hz). ^{13}C NMR δppm 195.69, 167.41, 159.11, 147.37, 112.69, 111.72, 107.7, 59.39, 50.49, 32.27, 30.93, 29.11, 27.38, 19.11, 13.44. $C_{29}H_{29}NO_3$, observed m/z 440 [M+1]$^+$, 11.5% rel. intensity), 262 ([M-4-Ar-H]$^+$, 100).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. Hydrogen atoms attached to carbon were placed in calculated positions (C—H = 0.95–0.98 A) and refined with isotropic displacement parameters 1.2–1.5 times those of the parent atoms. Hydrogen atoms attached to nitrogen and oxygen were found in difference-Fourier maps and refined freely. In compound **III**, three reflections ($\overline{1}$01, 110, and 020) affected by the beam stop were omitted because of poor agreement between the observed and calculated intensities.

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Synthesis and crystal structures of a bis(3-hydroxy-cyclohex-2-en-1-one) and two hexahydroquinoline derivatives

Scott A. Steiger, Chun Li, Christina Gates and Nicholas R. Natale

Computing details

For all structures, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

2,2′-[(2-Nitrophenyl)methylene]bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (I)

Crystal data

$C_{23}H_{27}NO_6$
M_r = 413.45
Triclinic, $P\overline{1}$
a = 8.7024 (3) Å
b = 9.8709 (4) Å
c = 13.1621 (5) Å
α = 90.3822 (19)°
β = 108.9608 (18)°
γ = 97.3571 (18)°
V = 1059.08 (7) Å^3
Z = 2
$F(000)$ = 440
D_x = 1.297 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9400 reflections
θ = 2.5–28.8°
μ = 0.09 mm^{-1}
T = 100 K
Prism, yellow
0.39 × 0.25 × 0.13 mm

Data collection

Bruker SMART BREEZE CCD diffractometer
φ and ω scans
29458 measured reflections
5315 independent reflections
4254 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 29.1°, θ_{min} = 2.5°
h = −11→11
k = −13→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.154
S = 1.04
5315 reflections
291 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2 + 0.5356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O5	0.04549 (13)	0.40295 (11)	0.15867 (9)	0.0208 (2)
O3	0.21250 (13)	0.20362 (12)	0.17985 (9)	0.0236 (3)
O4	0.68127 (15)	0.53646 (13)	0.29954 (11)	0.0320 (3)
O6	0.50510 (15)	0.73583 (13)	0.29901 (10)	0.0296 (3)
C9	0.11023 (18)	0.52479 (16)	0.18099 (11)	0.0185 (3)
O2	0.61739 (15)	0.30380 (15)	0.47643 (10)	0.0346 (3)
O1	0.50744 (18)	0.09861 (14)	0.41032 (11)	0.0380 (3)
C16	0.44538 (19)	0.37141 (16)	0.25549 (12)	0.0203 (3)
C8	0.27365 (18)	0.56290 (16)	0.25447 (11)	0.0183 (3)
C7	0.37049 (18)	0.45661 (15)	0.31983 (11)	0.0182 (3)
H7	0.466554	0.510901	0.375098	0.022*
N1	0.49857 (19)	0.21541 (16)	0.43905 (11)	0.0279 (3)
C10	0.0114 (2)	0.63357 (17)	0.12509 (13)	0.0235 (3)
H10A	−0.104225	0.606543	0.120725	0.028*
H10B	0.014314	0.636767	0.050574	0.028*
C17	0.36806 (18)	0.25256 (16)	0.19662 (12)	0.0209 (3)
C13	0.34611 (19)	0.69472 (17)	0.25407 (12)	0.0218 (3)
C6	0.28101 (18)	0.36902 (16)	0.38462 (11)	0.0191 (3)
C21	0.61154 (19)	0.42118 (18)	0.26078 (13)	0.0237 (3)
C18	0.4540 (2)	0.15999 (18)	0.14946 (14)	0.0249 (3)
C12	0.2536 (2)	0.80723 (17)	0.20066 (13)	0.0249 (3)
H12A	0.273724	0.823572	0.131642	0.030*
H12B	0.296854	0.892238	0.246928	0.030*
C11	0.0687 (2)	0.77713 (16)	0.17870 (13)	0.0230 (3)
C1	0.3365 (2)	0.25007 (17)	0.43280 (12)	0.0238 (3)
C2	0.2498 (2)	0.16111 (18)	0.48286 (13)	0.0285 (4)
H2	0.288304	0.078128	0.509312	0.034*
C4	0.0550 (2)	0.31725 (19)	0.45528 (13)	0.0277 (4)
H4	−0.039275	0.344284	0.466757	0.033*
C19	0.6040 (2)	0.23166 (18)	0.12570 (14)	0.0267 (4)
C14	0.0311 (2)	0.78794 (18)	0.28423 (14)	0.0280 (4)
H14A	−0.086246	0.759417	0.270562	0.042*
H14B	0.060734	0.882821	0.313448	0.042*
H14C	0.094698	0.728498	0.336144	0.042*
C5	0.13841 (19)	0.40093 (17)	0.40030 (12)	0.0220 (3)
H5	0.097129	0.482417	0.372502	0.026*
C20	0.7057 (2)	0.3260 (2)	0.22307 (15)	0.0310 (4)
H20A	0.756393	0.268957	0.283196	0.037*
H20B	0.795669	0.381263	0.205032	0.037*

C3	0.1069 (2)	0.19581 (19)	0.49334 (14)	0.0304 (4)
H3A	0.044832	0.136339	0.526547	0.036*
C22	0.7073 (2)	0.1264 (2)	0.10677 (18)	0.0361 (4)
H22A	0.740886	0.071941	0.170262	0.054*
H22B	0.804872	0.173597	0.093904	0.054*
H22C	0.642313	0.066101	0.043989	0.054*
C15	−0.0190 (2)	0.87981 (19)	0.10242 (15)	0.0321 (4)
H15A	0.001219	0.871620	0.033763	0.048*
H15B	0.022549	0.972726	0.134428	0.048*
H15C	−0.137191	0.860984	0.090111	0.048*
C23	0.5504 (3)	0.3151 (2)	0.02618 (16)	0.0377 (4)
H23A	0.491440	0.253400	−0.036919	0.057*
H23B	0.647292	0.366841	0.015346	0.057*
H23C	0.478164	0.378662	0.036190	0.057*
H3	0.160 (4)	0.282 (3)	0.187 (2)	0.069 (9)*
H18A	0.377 (3)	0.114 (2)	0.093 (2)	0.042 (6)*
H18B	0.486 (3)	0.084 (2)	0.2037 (19)	0.042 (6)*
H6	0.569 (5)	0.647 (4)	0.307 (3)	0.103 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0178 (5)	0.0222 (6)	0.0217 (5)	0.0004 (4)	0.0066 (4)	−0.0023 (4)
O3	0.0172 (5)	0.0246 (6)	0.0287 (6)	−0.0011 (4)	0.0089 (4)	−0.0045 (5)
O4	0.0232 (6)	0.0310 (7)	0.0418 (7)	−0.0051 (5)	0.0142 (5)	−0.0105 (5)
O6	0.0217 (6)	0.0284 (6)	0.0351 (7)	−0.0051 (5)	0.0080 (5)	−0.0042 (5)
C9	0.0194 (7)	0.0225 (7)	0.0163 (7)	0.0031 (6)	0.0095 (6)	−0.0003 (5)
O2	0.0232 (6)	0.0471 (8)	0.0292 (6)	0.0049 (6)	0.0029 (5)	0.0016 (6)
O1	0.0496 (8)	0.0338 (7)	0.0369 (7)	0.0206 (6)	0.0170 (6)	0.0081 (6)
C16	0.0182 (7)	0.0224 (7)	0.0216 (7)	0.0027 (6)	0.0083 (6)	−0.0001 (6)
C8	0.0177 (7)	0.0213 (7)	0.0167 (6)	0.0027 (5)	0.0068 (5)	−0.0018 (5)
C7	0.0163 (7)	0.0209 (7)	0.0172 (7)	0.0005 (5)	0.0061 (5)	−0.0027 (5)
N1	0.0304 (8)	0.0335 (8)	0.0209 (7)	0.0114 (6)	0.0070 (6)	0.0047 (6)
C10	0.0233 (8)	0.0249 (8)	0.0202 (7)	0.0034 (6)	0.0043 (6)	0.0017 (6)
C17	0.0178 (7)	0.0246 (8)	0.0211 (7)	0.0024 (6)	0.0075 (6)	0.0004 (6)
C13	0.0210 (7)	0.0249 (8)	0.0203 (7)	−0.0007 (6)	0.0095 (6)	−0.0032 (6)
C6	0.0185 (7)	0.0236 (7)	0.0143 (6)	0.0000 (6)	0.0054 (5)	−0.0029 (5)
C21	0.0181 (7)	0.0310 (9)	0.0213 (7)	0.0006 (6)	0.0068 (6)	−0.0029 (6)
C18	0.0211 (8)	0.0265 (8)	0.0280 (8)	0.0008 (6)	0.0103 (7)	−0.0065 (7)
C12	0.0299 (8)	0.0208 (8)	0.0260 (8)	−0.0003 (6)	0.0135 (7)	0.0017 (6)
C11	0.0278 (8)	0.0218 (8)	0.0210 (7)	0.0052 (6)	0.0095 (6)	0.0034 (6)
C1	0.0234 (8)	0.0280 (8)	0.0186 (7)	0.0048 (6)	0.0044 (6)	−0.0028 (6)
C2	0.0382 (10)	0.0257 (8)	0.0194 (7)	0.0022 (7)	0.0072 (7)	0.0019 (6)
C4	0.0255 (8)	0.0383 (10)	0.0210 (7)	0.0000 (7)	0.0115 (6)	−0.0012 (7)
C19	0.0241 (8)	0.0288 (9)	0.0311 (8)	0.0032 (6)	0.0144 (7)	−0.0028 (7)
C14	0.0333 (9)	0.0289 (9)	0.0278 (8)	0.0079 (7)	0.0166 (7)	0.0018 (7)
C5	0.0224 (8)	0.0264 (8)	0.0179 (7)	0.0037 (6)	0.0076 (6)	0.0005 (6)
C20	0.0176 (7)	0.0369 (10)	0.0395 (10)	−0.0011 (7)	0.0128 (7)	−0.0102 (8)

C3	0.0365 (9)	0.0335 (9)	0.0222 (8)	−0.0030 (7)	0.0142 (7)	0.0013 (7)
C22	0.0283 (9)	0.0356 (10)	0.0493 (11)	0.0062 (7)	0.0190 (8)	−0.0077 (8)
C15	0.0384 (10)	0.0259 (9)	0.0312 (9)	0.0091 (7)	0.0084 (8)	0.0071 (7)
C23	0.0470 (11)	0.0377 (11)	0.0379 (10)	0.0098 (9)	0.0254 (9)	0.0049 (8)

Geometric parameters (Å, º)

O5—C9	1.2507 (19)	C17—C18	1.499 (2)
O3—C17	1.3234 (19)	C13—C12	1.499 (2)
O4—C21	1.240 (2)	C6—C1	1.404 (2)
O6—C13	1.3228 (19)	C6—C5	1.395 (2)
C9—C8	1.437 (2)	C21—C20	1.502 (2)
C9—C10	1.507 (2)	C18—C19	1.528 (2)
O2—N1	1.227 (2)	C12—C11	1.528 (2)
O1—N1	1.231 (2)	C11—C14	1.533 (2)
C16—C7	1.529 (2)	C11—C15	1.527 (2)
C16—C17	1.370 (2)	C1—C2	1.391 (2)
C16—C21	1.445 (2)	C2—C3	1.380 (3)
C8—C7	1.523 (2)	C4—C5	1.389 (2)
C8—C13	1.372 (2)	C4—C3	1.375 (3)
C7—C6	1.533 (2)	C19—C20	1.526 (2)
N1—C1	1.470 (2)	C19—C22	1.526 (2)
C10—C11	1.527 (2)	C19—C23	1.526 (3)
O5—C9—C8	122.69 (14)	O4—C21—C16	122.67 (15)
O5—C9—C10	117.37 (13)	O4—C21—C20	118.99 (14)
C8—C9—C10	119.93 (13)	C16—C21—C20	118.26 (15)
C17—C16—C7	124.87 (14)	C17—C18—C19	114.49 (14)
C17—C16—C21	119.05 (14)	C13—C12—C11	113.81 (13)
C21—C16—C7	116.07 (13)	C10—C11—C12	107.37 (13)
C9—C8—C7	121.06 (13)	C10—C11—C14	111.47 (14)
C13—C8—C9	117.49 (14)	C10—C11—C15	109.11 (14)
C13—C8—C7	121.00 (13)	C12—C11—C14	110.01 (14)
C16—C7—C6	112.82 (12)	C15—C11—C12	110.07 (14)
C8—C7—C16	113.54 (12)	C15—C11—C14	108.80 (14)
C8—C7—C6	114.51 (12)	C6—C1—N1	121.11 (15)
O2—N1—O1	124.15 (16)	C2—C1—N1	114.97 (15)
O2—N1—C1	117.60 (14)	C2—C1—C6	123.83 (15)
O1—N1—C1	118.19 (15)	C3—C2—C1	118.67 (16)
C9—C10—C11	115.19 (13)	C3—C4—C5	120.92 (16)
O3—C17—C16	123.87 (14)	C20—C19—C18	107.98 (14)
O3—C17—C18	112.79 (14)	C20—C19—C22	109.38 (15)
C16—C17—C18	123.25 (14)	C22—C19—C18	110.18 (15)
O6—C13—C8	123.82 (15)	C23—C19—C18	110.18 (15)
O6—C13—C12	112.72 (14)	C23—C19—C20	109.85 (16)
C8—C13—C12	123.46 (14)	C23—C19—C22	109.26 (15)
C1—C6—C7	122.42 (13)	C4—C5—C6	122.05 (15)
C5—C6—C7	122.82 (14)	C21—C20—C19	114.88 (14)

C5—C6—C1	114.76 (14)	C4—C3—C2	119.41 (16)
O5—C9—C8—C7	−7.3 (2)	C7—C6—C1—C2	−173.36 (14)
O5—C9—C8—C13	165.07 (14)	C7—C6—C5—C4	177.16 (14)
O5—C9—C10—C11	161.40 (13)	N1—C1—C2—C3	171.47 (15)
O3—C17—C18—C19	156.51 (14)	C10—C9—C8—C7	174.32 (13)
O4—C21—C20—C19	−146.42 (16)	C10—C9—C8—C13	−13.3 (2)
O6—C13—C12—C11	−162.31 (13)	C17—C16—C7—C8	−88.49 (18)
C9—C8—C7—C16	78.45 (17)	C17—C16—C7—C6	43.9 (2)
C9—C8—C7—C6	−53.11 (18)	C17—C16—C21—O4	169.75 (16)
C9—C8—C13—O6	−164.77 (14)	C17—C16—C21—C20	−13.6 (2)
C9—C8—C13—C12	14.0 (2)	C17—C18—C19—C20	45.7 (2)
C9—C10—C11—C12	49.40 (17)	C17—C18—C19—C22	165.08 (15)
C9—C10—C11—C14	−71.15 (18)	C17—C18—C19—C23	−74.29 (19)
C9—C10—C11—C15	168.67 (14)	C13—C8—C7—C16	−93.69 (17)
O2—N1—C1—C6	51.4 (2)	C13—C8—C7—C6	134.75 (14)
O2—N1—C1—C2	−125.14 (16)	C13—C12—C11—C10	−48.40 (17)
O1—N1—C1—C6	−131.16 (16)	C13—C12—C11—C14	73.07 (17)
O1—N1—C1—C2	52.2 (2)	C13—C12—C11—C15	−167.05 (14)
C16—C7—C6—C1	35.67 (19)	C6—C1—C2—C3	−5.0 (2)
C16—C7—C6—C5	−144.27 (14)	C21—C16—C7—C8	92.93 (16)
C16—C17—C18—C19	−26.7 (2)	C21—C16—C7—C6	−134.69 (14)
C16—C21—C20—C19	36.8 (2)	C21—C16—C17—O3	−174.70 (14)
C8—C9—C10—C11	−20.2 (2)	C21—C16—C17—C18	8.9 (2)
C8—C7—C6—C1	167.58 (13)	C18—C19—C20—C21	−51.4 (2)
C8—C7—C6—C5	−12.4 (2)	C1—C6—C5—C4	−2.8 (2)
C8—C13—C12—C11	18.8 (2)	C1—C2—C3—C4	−0.7 (2)
C7—C16—C17—O3	6.8 (3)	C5—C6—C1—N1	−169.69 (14)
C7—C16—C17—C18	−169.69 (15)	C5—C6—C1—C2	6.6 (2)
C7—C16—C21—O4	−11.6 (2)	C5—C4—C3—C2	4.3 (3)
C7—C16—C21—C20	165.04 (15)	C3—C4—C5—C6	−2.5 (2)
C7—C8—C13—O6	7.6 (2)	C22—C19—C20—C21	−171.31 (16)
C7—C8—C13—C12	−173.56 (14)	C23—C19—C20—C21	68.8 (2)
C7—C6—C1—N1	10.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O5	0.97 (3)	1.62 (3)	2.5570 (16)	162 (3)
O6—H6···O4	1.08 (4)	1.58 (4)	2.6437 (19)	166 (3)
C2—H2···O1^{i}	0.95	2.63	3.538 (2)	160
C10—H10*B*···O5^{ii}	0.99	2.65	3.6138 (19)	165
C15—H15*B*···O3^{iii}	0.98	2.58	3.505 (2)	157
C18—H18*B*···O1	1.04 (2)	2.67 (2)	3.381 (2)	125.5 (16)
C20—H20*B*···O5^{iv}	0.99	2.43	3.332 (2)	152

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*, −*y*+1, −*z*; (iii) *x*, *y*+1, *z*; (iv) *x*+1, *y*, *z*.

Ethyl 4-(4-hydroxy-3,5-dimethoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (II)

Crystal data

$C_{23}H_{29}NO_6$
$M_r = 415.47$
Monoclinic, $P2_1/n$
$a = 10.8854$ (4) Å
$b = 25.2446$ (10) Å
$c = 15.3665$ (6) Å
$\beta = 100.7606$ (19)°
$V = 4148.4$ (3) Å^3
$Z = 8$
$F(000) = 1776$
$D_x = 1.330$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9684 reflections
$\theta = 2.7$–30.5°
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Prism, colourless
0.48 × 0.43 × 0.31 mm

Data collection

Bruker SMART BREEZE CCD diffractometer
φ and ω scans
168826 measured reflections
12707 independent reflections
11044 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 30.6°$, $\theta_{min} = 1.6°$
$h = -15 \rightarrow 15$
$k = -36 \rightarrow 36$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.04$
12707 reflections
569 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 1.3541P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.09761 (6)	0.68726 (3)	0.28219 (5)	0.01536 (13)
O2A	0.66193 (6)	0.61973 (3)	0.32843 (5)	0.01652 (13)
O3A	0.54423 (6)	0.69363 (3)	0.31163 (5)	0.01527 (13)
O4A	0.40914 (7)	0.74895 (3)	0.70657 (5)	0.01676 (14)
H4C	0.4593 (16)	0.7750 (7)	0.7112 (11)	0.035 (4)*
O5A	0.31079 (7)	0.65461 (3)	0.68643 (4)	0.01866 (14)
O6A	0.47618 (6)	0.80349 (3)	0.56333 (5)	0.01668 (14)
N1A	0.34484 (7)	0.54131 (3)	0.39339 (5)	0.01093 (13)
H1A	0.3460 (13)	0.5068 (6)	0.4013 (9)	0.021 (3)*

C2A	0.45480 (8)	0.56515 (3)	0.38033 (5)	0.01037 (15)
C3A	0.45444 (8)	0.61775 (3)	0.36062 (6)	0.01007 (14)
C4A	0.33953 (8)	0.65156 (3)	0.36544 (5)	0.00946 (14)
H4B	0.332567	0.679421	0.318505	0.011*
C5A	0.22391 (8)	0.61725 (3)	0.34576 (5)	0.00925 (14)
C6A	0.10568 (8)	0.63990 (3)	0.30421 (5)	0.00999 (14)
C7A	−0.00768 (8)	0.60412 (3)	0.28469 (6)	0.01162 (15)
H7C	−0.013501	0.588937	0.224662	0.014*
H7D	−0.083739	0.625617	0.284713	0.014*
C8A	−0.00446 (8)	0.55865 (3)	0.35161 (6)	0.01072 (15)
C9A	0.11952 (8)	0.52866 (3)	0.35798 (6)	0.01071 (15)
H9C	0.130811	0.504944	0.410176	0.013*
H9D	0.115282	0.506253	0.304626	0.013*
C10A	0.23072 (8)	0.56462 (3)	0.36583 (5)	0.00927 (14)
C11A	−0.01825 (9)	0.58146 (4)	0.44184 (6)	0.01572 (17)
H11D	0.049914	0.606478	0.462131	0.024*
H11E	−0.014841	0.552601	0.484888	0.024*
H11F	−0.098635	0.599854	0.436132	0.024*
C12A	−0.11296 (9)	0.52050 (4)	0.31995 (7)	0.01625 (17)
H12D	−0.192403	0.539584	0.314712	0.024*
H12E	−0.110612	0.491581	0.362771	0.024*
H12F	−0.105306	0.505920	0.262095	0.024*
C13A	0.56241 (8)	0.52727 (4)	0.38723 (6)	0.01339 (16)
H13D	0.543503	0.494967	0.417571	0.020*
H13E	0.638014	0.543801	0.420913	0.020*
H13F	0.575997	0.518286	0.327703	0.020*
C14A	0.56421 (8)	0.64179 (4)	0.33347 (6)	0.01162 (15)
C15A	0.63909 (9)	0.71842 (4)	0.27011 (6)	0.01674 (17)
H15C	0.723206	0.707623	0.301464	0.020*
H15D	0.632745	0.757422	0.274256	0.020*
C16A	0.62173 (10)	0.70211 (4)	0.17397 (7)	0.02087 (19)
H16D	0.674331	0.724303	0.143533	0.031*
H16E	0.533831	0.706570	0.145851	0.031*
H16F	0.645760	0.664893	0.170167	0.031*
C17A	0.35248 (8)	0.67935 (3)	0.45549 (6)	0.01023 (15)
C18A	0.32110 (8)	0.65290 (3)	0.52864 (6)	0.01201 (15)
H18A	0.286818	0.618226	0.521374	0.014*
C19A	0.33951 (8)	0.67677 (4)	0.61162 (6)	0.01263 (16)
C20A	0.39194 (8)	0.72784 (4)	0.62385 (6)	0.01266 (16)
C21A	0.42098 (8)	0.75402 (3)	0.55069 (6)	0.01235 (16)
C22A	0.40129 (8)	0.73027 (3)	0.46704 (6)	0.01162 (15)
H22A	0.421364	0.749009	0.417934	0.014*
C23A	0.25765 (10)	0.60304 (4)	0.67836 (7)	0.02005 (19)
H23D	0.179341	0.603652	0.634894	0.030*
H23E	0.240715	0.591604	0.735908	0.030*
H23F	0.316250	0.578294	0.658694	0.030*
C24A	0.38733 (10)	0.84600 (4)	0.54853 (7)	0.02017 (19)
H24D	0.337479	0.843172	0.488624	0.030*

H24E	0.431736	0.879953	0.554867	0.030*
H24F	0.332068	0.843913	0.591977	0.030*
O1B	0.64618 (7)	0.56795 (3)	0.59302 (5)	0.01898 (14)
O2B	0.42404 (7)	0.61173 (3)	0.93863 (5)	0.02210 (15)
O3B	0.47350 (6)	0.54093 (3)	0.86392 (5)	0.01724 (14)
O4B	1.07150 (7)	0.47496 (3)	0.91945 (5)	0.01745 (14)
H4	1.1277 (17)	0.4973 (7)	0.9396 (12)	0.040 (5)*
O5B	1.09254 (6)	0.57723 (3)	0.88846 (5)	0.01832 (14)
O6B	0.85544 (7)	0.42479 (3)	0.88741 (5)	0.01802 (14)
N1B	0.67195 (7)	0.70086 (3)	0.81054 (5)	0.01272 (14)
H1	0.6769 (13)	0.7356 (6)	0.8200 (9)	0.022 (3)*
C2B	0.60016 (8)	0.67286 (4)	0.86093 (6)	0.01315 (16)
C3B	0.57467 (8)	0.62086 (4)	0.84315 (6)	0.01213 (15)
C4B	0.63347 (8)	0.59179 (3)	0.77391 (6)	0.01085 (15)
H4A	0.570391	0.566145	0.742480	0.013*
C5B	0.66228 (8)	0.63135 (3)	0.70649 (6)	0.01071 (15)
C6B	0.66639 (8)	0.61434 (3)	0.61710 (6)	0.01161 (15)
C7B	0.69106 (8)	0.65543 (3)	0.55138 (6)	0.01271 (15)
H7A	0.610642	0.671873	0.523744	0.015*
H7B	0.725884	0.637650	0.503929	0.015*
C8B	0.78141 (8)	0.69911 (3)	0.59211 (6)	0.01258 (15)
C9B	0.73133 (8)	0.72350 (3)	0.67009 (6)	0.01263 (15)
H9A	0.798126	0.745108	0.705925	0.015*
H9B	0.660884	0.747437	0.646616	0.015*
C10B	0.68745 (8)	0.68288 (3)	0.72894 (6)	0.01098 (15)
C11B	0.91219 (9)	0.67533 (4)	0.62358 (7)	0.01783 (18)
H11A	0.906685	0.645916	0.664297	0.027*
H11B	0.968228	0.702619	0.654158	0.027*
H11C	0.945007	0.662303	0.572359	0.027*
C12B	0.78685 (10)	0.74186 (4)	0.52223 (7)	0.01908 (18)
H12A	0.817987	0.726305	0.472081	0.029*
H12B	0.843131	0.770322	0.548282	0.029*
H12C	0.702898	0.756306	0.501600	0.029*
C13B	0.55601 (10)	0.70687 (4)	0.92881 (7)	0.02043 (19)
H13A	0.617141	0.735037	0.947664	0.031*
H13B	0.546865	0.685161	0.980088	0.031*
H13C	0.475127	0.722665	0.903090	0.031*
C14B	0.48511 (8)	0.59282 (4)	0.88766 (6)	0.01479 (16)
C15B	0.37849 (9)	0.51189 (4)	0.89925 (7)	0.01923 (18)
H15A	0.294455	0.525795	0.873705	0.023*
H15B	0.392024	0.516062	0.964409	0.023*
C16B	0.38731 (10)	0.45419 (4)	0.87558 (7)	0.02153 (19)
H16A	0.376740	0.450555	0.811117	0.032*
H16B	0.321567	0.434209	0.896828	0.032*
H16C	0.469351	0.440325	0.903427	0.032*
C17B	0.75057 (8)	0.56042 (3)	0.81518 (6)	0.01136 (15)
C18B	0.86636 (8)	0.58599 (4)	0.83344 (6)	0.01318 (16)
H18	0.871998	0.622799	0.821919	0.016*

C19B	0.97326 (8)	0.55741 (4)	0.86850 (6)	0.01334 (16)
C20B	0.96602 (8)	0.50345 (4)	0.88589 (6)	0.01323 (16)
C21B	0.85061 (9)	0.47788 (3)	0.86888 (6)	0.01301 (16)
C22B	0.74257 (8)	0.50638 (4)	0.83344 (6)	0.01278 (16)
H22	0.663772	0.489024	0.821767	0.015*
C23B	1.10806 (9)	0.63320 (4)	0.88435 (8)	0.02108 (19)
H23A	1.077197	0.645485	0.823712	0.032*
H23B	1.196897	0.642070	0.901795	0.032*
H23C	1.060743	0.650516	0.924718	0.032*
C24B	0.74049 (10)	0.39706 (4)	0.88304 (7)	0.01957 (19)
H24A	0.689946	0.400674	0.823490	0.029*
H24B	0.694746	0.411935	0.926559	0.029*
H24C	0.757687	0.359498	0.896071	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0138 (3)	0.0100 (3)	0.0212 (3)	0.0012 (2)	0.0004 (2)	0.0044 (2)
O2A	0.0114 (3)	0.0183 (3)	0.0206 (3)	0.0018 (2)	0.0049 (2)	0.0016 (3)
O3A	0.0153 (3)	0.0116 (3)	0.0209 (3)	−0.0018 (2)	0.0086 (3)	0.0018 (2)
O4A	0.0174 (3)	0.0172 (3)	0.0155 (3)	−0.0066 (3)	0.0026 (2)	−0.0073 (2)
O5A	0.0266 (4)	0.0172 (3)	0.0127 (3)	−0.0096 (3)	0.0049 (3)	−0.0020 (2)
O6A	0.0150 (3)	0.0086 (3)	0.0256 (3)	−0.0037 (2)	0.0018 (3)	−0.0039 (2)
N1A	0.0099 (3)	0.0076 (3)	0.0152 (3)	0.0012 (2)	0.0019 (3)	0.0017 (3)
C2A	0.0098 (3)	0.0115 (4)	0.0096 (3)	0.0008 (3)	0.0012 (3)	−0.0006 (3)
C3A	0.0088 (3)	0.0107 (3)	0.0106 (3)	0.0002 (3)	0.0017 (3)	−0.0005 (3)
C4A	0.0087 (3)	0.0078 (3)	0.0117 (3)	−0.0001 (3)	0.0014 (3)	0.0002 (3)
C5A	0.0088 (3)	0.0084 (3)	0.0104 (3)	0.0001 (3)	0.0016 (3)	0.0004 (3)
C6A	0.0101 (3)	0.0097 (3)	0.0102 (3)	0.0007 (3)	0.0020 (3)	0.0005 (3)
C7A	0.0095 (3)	0.0113 (4)	0.0134 (4)	−0.0005 (3)	0.0004 (3)	0.0006 (3)
C8A	0.0096 (3)	0.0098 (3)	0.0130 (4)	−0.0010 (3)	0.0027 (3)	−0.0007 (3)
C9A	0.0102 (3)	0.0081 (3)	0.0138 (4)	−0.0010 (3)	0.0024 (3)	−0.0005 (3)
C10A	0.0095 (3)	0.0092 (3)	0.0090 (3)	0.0005 (3)	0.0014 (3)	−0.0003 (3)
C11A	0.0166 (4)	0.0174 (4)	0.0146 (4)	−0.0002 (3)	0.0064 (3)	−0.0019 (3)
C12A	0.0119 (4)	0.0147 (4)	0.0220 (4)	−0.0042 (3)	0.0026 (3)	−0.0009 (3)
C13A	0.0118 (4)	0.0126 (4)	0.0159 (4)	0.0035 (3)	0.0031 (3)	0.0003 (3)
C14A	0.0113 (4)	0.0126 (4)	0.0107 (3)	−0.0014 (3)	0.0015 (3)	−0.0008 (3)
C15A	0.0160 (4)	0.0162 (4)	0.0195 (4)	−0.0051 (3)	0.0072 (3)	0.0022 (3)
C16A	0.0203 (5)	0.0245 (5)	0.0190 (4)	−0.0016 (4)	0.0066 (4)	0.0024 (4)
C17A	0.0086 (3)	0.0089 (3)	0.0130 (4)	0.0004 (3)	0.0014 (3)	−0.0009 (3)
C18A	0.0124 (4)	0.0098 (4)	0.0136 (4)	−0.0019 (3)	0.0020 (3)	−0.0013 (3)
C19A	0.0124 (4)	0.0120 (4)	0.0135 (4)	−0.0022 (3)	0.0024 (3)	−0.0008 (3)
C20A	0.0104 (4)	0.0123 (4)	0.0149 (4)	−0.0013 (3)	0.0015 (3)	−0.0040 (3)
C21A	0.0100 (4)	0.0082 (3)	0.0185 (4)	−0.0016 (3)	0.0017 (3)	−0.0027 (3)
C22A	0.0107 (3)	0.0091 (3)	0.0150 (4)	−0.0004 (3)	0.0023 (3)	−0.0003 (3)
C23A	0.0280 (5)	0.0149 (4)	0.0177 (4)	−0.0065 (4)	0.0052 (4)	0.0013 (3)
C24A	0.0219 (5)	0.0102 (4)	0.0281 (5)	0.0000 (3)	0.0039 (4)	−0.0003 (3)
O1B	0.0299 (4)	0.0093 (3)	0.0169 (3)	−0.0001 (3)	0.0023 (3)	−0.0026 (2)

O2B	0.0220 (4)	0.0223 (4)	0.0258 (4)	0.0014 (3)	0.0142 (3)	0.0004 (3)
O3B	0.0148 (3)	0.0159 (3)	0.0229 (3)	−0.0016 (2)	0.0082 (3)	0.0013 (3)
O4B	0.0143 (3)	0.0165 (3)	0.0203 (3)	0.0059 (3)	0.0001 (3)	0.0028 (3)
O5B	0.0112 (3)	0.0149 (3)	0.0272 (4)	0.0005 (2)	−0.0007 (3)	−0.0011 (3)
O6B	0.0181 (3)	0.0111 (3)	0.0256 (4)	0.0029 (2)	0.0060 (3)	0.0060 (3)
N1B	0.0151 (3)	0.0098 (3)	0.0137 (3)	0.0001 (3)	0.0038 (3)	−0.0031 (3)
C2B	0.0121 (4)	0.0147 (4)	0.0128 (4)	0.0025 (3)	0.0027 (3)	−0.0008 (3)
C3B	0.0101 (4)	0.0136 (4)	0.0129 (4)	0.0022 (3)	0.0026 (3)	0.0010 (3)
C4B	0.0107 (4)	0.0095 (3)	0.0123 (4)	0.0011 (3)	0.0017 (3)	0.0001 (3)
C5B	0.0105 (3)	0.0094 (3)	0.0119 (4)	0.0012 (3)	0.0014 (3)	−0.0003 (3)
C6B	0.0113 (4)	0.0099 (4)	0.0128 (4)	0.0016 (3)	0.0001 (3)	−0.0009 (3)
C7B	0.0147 (4)	0.0114 (4)	0.0117 (4)	−0.0004 (3)	0.0017 (3)	−0.0008 (3)
C8B	0.0127 (4)	0.0122 (4)	0.0131 (4)	−0.0004 (3)	0.0032 (3)	−0.0011 (3)
C9B	0.0143 (4)	0.0093 (4)	0.0147 (4)	−0.0006 (3)	0.0037 (3)	−0.0013 (3)
C10B	0.0098 (3)	0.0107 (4)	0.0122 (4)	0.0015 (3)	0.0014 (3)	−0.0011 (3)
C11B	0.0133 (4)	0.0205 (4)	0.0197 (4)	0.0007 (3)	0.0032 (3)	−0.0036 (3)
C12B	0.0230 (5)	0.0174 (4)	0.0177 (4)	−0.0045 (4)	0.0061 (4)	0.0020 (3)
C13B	0.0250 (5)	0.0196 (5)	0.0191 (4)	0.0027 (4)	0.0105 (4)	−0.0044 (4)
C14B	0.0122 (4)	0.0162 (4)	0.0162 (4)	0.0020 (3)	0.0030 (3)	0.0030 (3)
C15B	0.0148 (4)	0.0195 (4)	0.0252 (5)	−0.0026 (3)	0.0082 (4)	0.0042 (4)
C16B	0.0210 (5)	0.0193 (5)	0.0244 (5)	−0.0038 (4)	0.0046 (4)	0.0020 (4)
C17B	0.0117 (4)	0.0108 (4)	0.0116 (4)	0.0020 (3)	0.0023 (3)	−0.0005 (3)
C18B	0.0131 (4)	0.0106 (4)	0.0152 (4)	0.0014 (3)	0.0012 (3)	−0.0006 (3)
C19B	0.0118 (4)	0.0138 (4)	0.0140 (4)	0.0009 (3)	0.0012 (3)	−0.0016 (3)
C20B	0.0138 (4)	0.0139 (4)	0.0119 (4)	0.0046 (3)	0.0021 (3)	0.0008 (3)
C21B	0.0166 (4)	0.0107 (4)	0.0126 (4)	0.0028 (3)	0.0049 (3)	0.0016 (3)
C22B	0.0135 (4)	0.0120 (4)	0.0134 (4)	0.0015 (3)	0.0041 (3)	0.0004 (3)
C23B	0.0156 (4)	0.0162 (4)	0.0307 (5)	−0.0016 (3)	0.0025 (4)	−0.0033 (4)
C24B	0.0213 (5)	0.0145 (4)	0.0243 (5)	−0.0009 (3)	0.0078 (4)	0.0041 (3)

Geometric parameters (Å, º)

O1A—C6A	1.2412 (10)	O1B—C6B	1.2355 (11)
O2A—C14A	1.2159 (11)	O2B—C14B	1.2156 (12)
O3A—C14A	1.3583 (11)	O3B—C14B	1.3593 (12)
O3A—C15A	1.4529 (11)	O3B—C15B	1.4530 (11)
O4A—C20A	1.3587 (11)	O4B—C20B	1.3713 (11)
O5A—C19A	1.3661 (11)	O5B—C19B	1.3717 (11)
O5A—C23A	1.4206 (12)	O5B—C23B	1.4257 (12)
O6A—C21A	1.3837 (10)	O6B—C21B	1.3690 (11)
O6A—C24A	1.4341 (12)	O6B—C24B	1.4245 (12)
N1A—C2A	1.3874 (11)	N1B—C2B	1.3913 (12)
N1A—C10A	1.3683 (11)	N1B—C10B	1.3733 (11)
C2A—C3A	1.3617 (12)	C2B—C3B	1.3589 (13)
C2A—C13A	1.5006 (12)	C2B—C13B	1.4979 (13)
C3A—C4A	1.5275 (12)	C3B—C4B	1.5280 (12)
C3A—C14A	1.4684 (12)	C3B—C14B	1.4725 (13)
C4A—C5A	1.5112 (11)	C4B—C5B	1.5134 (12)

C4A—C17A	1.5344 (12)	C4B—C17B	1.5337 (12)
C5A—C6A	1.4437 (11)	C5B—C6B	1.4479 (12)
C5A—C10A	1.3628 (11)	C5B—C10B	1.3604 (12)
C6A—C7A	1.5134 (12)	C6B—C7B	1.5062 (12)
C7A—C8A	1.5371 (12)	C7B—C8B	1.5314 (12)
C8A—C9A	1.5346 (12)	C8B—C9B	1.5350 (12)
C8A—C11A	1.5342 (12)	C8B—C11B	1.5374 (13)
C8A—C12A	1.5313 (12)	C8B—C12B	1.5313 (13)
C9A—C10A	1.4998 (11)	C9B—C10B	1.5027 (12)
C15A—C16A	1.5111 (14)	C15B—C16B	1.5088 (15)
C17A—C18A	1.4034 (12)	C17B—C18B	1.3972 (12)
C17A—C22A	1.3895 (11)	C17B—C22B	1.3987 (12)
C18A—C19A	1.3906 (12)	C18B—C19B	1.3896 (12)
C19A—C20A	1.4082 (12)	C19B—C20B	1.3932 (13)
C20A—C21A	1.3904 (13)	C20B—C21B	1.3931 (13)
C21A—C22A	1.3981 (12)	C21B—C22B	1.3994 (12)

C14A—O3A—C15A	115.43 (7)	C14B—O3B—C15B	115.02 (7)
C19A—O5A—C23A	117.08 (7)	C19B—O5B—C23B	117.82 (7)
C21A—O6A—C24A	113.00 (7)	C21B—O6B—C24B	118.16 (7)
C10A—N1A—C2A	121.78 (7)	C10B—N1B—C2B	121.19 (8)
N1A—C2A—C13A	113.36 (7)	N1B—C2B—C13B	112.51 (8)
C3A—C2A—N1A	119.23 (8)	C3B—C2B—N1B	119.59 (8)
C3A—C2A—C13A	127.37 (8)	C3B—C2B—C13B	127.86 (9)
C2A—C3A—C4A	120.17 (7)	C2B—C3B—C4B	120.50 (8)
C2A—C3A—C14A	119.84 (8)	C2B—C3B—C14B	119.94 (8)
C14A—C3A—C4A	119.99 (7)	C14B—C3B—C4B	119.50 (8)
C3A—C4A—C17A	111.17 (7)	C3B—C4B—C17B	112.57 (7)
C5A—C4A—C3A	109.20 (7)	C5B—C4B—C3B	109.09 (7)
C5A—C4A—C17A	112.05 (7)	C5B—C4B—C17B	111.19 (7)
C6A—C5A—C4A	120.12 (7)	C6B—C5B—C4B	120.05 (7)
C10A—C5A—C4A	120.36 (7)	C10B—C5B—C4B	121.01 (8)
C10A—C5A—C6A	119.51 (7)	C10B—C5B—C6B	118.93 (8)
O1A—C6A—C5A	120.93 (8)	O1B—C6B—C5B	122.00 (8)
O1A—C6A—C7A	120.49 (8)	O1B—C6B—C7B	119.74 (8)
C5A—C6A—C7A	118.54 (7)	C5B—C6B—C7B	118.21 (7)
C6A—C7A—C8A	113.41 (7)	C6B—C7B—C8B	113.78 (7)
C9A—C8A—C7A	108.72 (7)	C7B—C8B—C9B	108.25 (7)
C11A—C8A—C7A	109.30 (7)	C7B—C8B—C11B	109.50 (7)
C11A—C8A—C9A	111.15 (7)	C9B—C8B—C11B	110.73 (7)
C12A—C8A—C7A	109.71 (7)	C12B—C8B—C7B	109.08 (7)
C12A—C8A—C9A	109.08 (7)	C12B—C8B—C9B	109.31 (7)
C12A—C8A—C11A	108.87 (7)	C12B—C8B—C11B	109.94 (8)
C10A—C9A—C8A	113.17 (7)	C10B—C9B—C8B	113.28 (7)
N1A—C10A—C9A	115.91 (7)	N1B—C10B—C9B	115.54 (7)
C5A—C10A—N1A	119.81 (8)	C5B—C10B—N1B	119.61 (8)
C5A—C10A—C9A	124.26 (7)	C5B—C10B—C9B	124.85 (8)
O2A—C14A—O3A	121.80 (8)	O2B—C14B—O3B	121.11 (9)

O2A—C14A—C3A	126.64 (8)	O2B—C14B—C3B	126.85 (9)
O3A—C14A—C3A	111.55 (7)	O3B—C14B—C3B	112.03 (8)
O3A—C15A—C16A	110.04 (8)	O3B—C15B—C16B	108.38 (8)
C18A—C17A—C4A	120.65 (7)	C18B—C17B—C4B	119.57 (8)
C22A—C17A—C4A	120.45 (8)	C18B—C17B—C22B	119.86 (8)
C22A—C17A—C18A	118.83 (8)	C22B—C17B—C4B	120.56 (8)
C19A—C18A—C17A	120.88 (8)	C19B—C18B—C17B	119.78 (8)
O5A—C19A—C18A	125.29 (8)	O5B—C19B—C18B	125.90 (8)
O5A—C19A—C20A	114.41 (8)	O5B—C19B—C20B	113.51 (8)
C18A—C19A—C20A	120.30 (8)	C18B—C19B—C20B	120.59 (8)
O4A—C20A—C19A	117.75 (8)	O4B—C20B—C19B	120.77 (8)
O4A—C20A—C21A	123.89 (8)	O4B—C20B—C21B	119.34 (8)
C21A—C20A—C19A	118.35 (8)	C21B—C20B—C19B	119.89 (8)
O6A—C21A—C20A	118.27 (8)	O6B—C21B—C20B	114.60 (8)
O6A—C21A—C22A	120.27 (8)	O6B—C21B—C22B	125.54 (8)
C20A—C21A—C22A	121.37 (8)	C20B—C21B—C22B	119.85 (8)
C17A—C22A—C21A	120.23 (8)	C17B—C22B—C21B	120.02 (8)
O1A—C6A—C7A—C8A	−151.01 (8)	O1B—C6B—C7B—C8B	−147.46 (8)
O4A—C20A—C21A—O6A	3.22 (13)	O4B—C20B—C21B—O6B	−0.68 (12)
O4A—C20A—C21A—C22A	179.97 (8)	O4B—C20B—C21B—C22B	−179.08 (8)
O5A—C19A—C20A—O4A	0.60 (12)	O5B—C19B—C20B—O4B	−0.23 (12)
O5A—C19A—C20A—C21A	−178.29 (8)	O5B—C19B—C20B—C21B	179.96 (8)
O6A—C21A—C22A—C17A	176.30 (8)	O6B—C21B—C22B—C17B	−178.23 (8)
N1A—C2A—C3A—C4A	8.37 (12)	N1B—C2B—C3B—C4B	5.35 (13)
N1A—C2A—C3A—C14A	−170.77 (8)	N1B—C2B—C3B—C14B	−171.69 (8)
C2A—N1A—C10A—C5A	−16.97 (12)	C2B—N1B—C10B—C5B	−17.81 (13)
C2A—N1A—C10A—C9A	161.69 (8)	C2B—N1B—C10B—C9B	161.71 (8)
C2A—C3A—C4A—C5A	−29.30 (10)	C2B—C3B—C4B—C5B	−26.83 (11)
C2A—C3A—C4A—C17A	94.83 (9)	C2B—C3B—C4B—C17B	97.09 (10)
C2A—C3A—C14A—O2A	−2.27 (14)	C2B—C3B—C14B—O2B	3.16 (15)
C2A—C3A—C14A—O3A	176.45 (8)	C2B—C3B—C14B—O3B	−178.35 (8)
C3A—C4A—C5A—C6A	−149.71 (8)	C3B—C4B—C5B—C6B	−153.27 (8)
C3A—C4A—C5A—C10A	29.31 (10)	C3B—C4B—C5B—C10B	28.25 (11)
C3A—C4A—C17A—C18A	−83.76 (9)	C3B—C4B—C17B—C18B	−84.89 (10)
C3A—C4A—C17A—C22A	93.08 (9)	C3B—C4B—C17B—C22B	96.04 (9)
C4A—C3A—C14A—O2A	178.59 (8)	C4B—C3B—C14B—O2B	−173.92 (9)
C4A—C3A—C14A—O3A	−2.69 (11)	C4B—C3B—C14B—O3B	4.58 (11)
C4A—C5A—C6A—O1A	1.72 (12)	C4B—C5B—C6B—O1B	0.45 (13)
C4A—C5A—C6A—C7A	179.35 (7)	C4B—C5B—C6B—C7B	177.94 (7)
C4A—C5A—C10A—N1A	−8.32 (12)	C4B—C5B—C10B—N1B	−7.91 (12)
C4A—C5A—C10A—C9A	173.14 (7)	C4B—C5B—C10B—C9B	172.62 (8)
C4A—C17A—C18A—C19A	176.22 (8)	C4B—C17B—C18B—C19B	−178.18 (8)
C4A—C17A—C22A—C21A	−175.56 (8)	C4B—C17B—C22B—C21B	178.27 (8)
C5A—C4A—C17A—C18A	38.74 (10)	C5B—C4B—C17B—C18B	37.86 (11)
C5A—C4A—C17A—C22A	−144.43 (8)	C5B—C4B—C17B—C22B	−141.21 (8)
C5A—C6A—C7A—C8A	31.35 (11)	C5B—C6B—C7B—C8B	34.99 (11)
C6A—C5A—C10A—N1A	170.71 (8)	C6B—C5B—C10B—N1B	173.59 (8)

C6A—C5A—C10A—C9A	−7.84 (12)	C6B—C5B—C10B—C9B	−5.88 (13)
C6A—C7A—C8A—C9A	−53.22 (9)	C6B—C7B—C8B—C9B	−54.30 (10)
C6A—C7A—C8A—C11A	68.27 (9)	C6B—C7B—C8B—C11B	66.51 (10)
C6A—C7A—C8A—C12A	−172.43 (7)	C6B—C7B—C8B—C12B	−173.14 (8)
C7A—C8A—C9A—C10A	45.78 (9)	C7B—C8B—C9B—C10B	44.60 (10)
C8A—C9A—C10A—N1A	164.48 (7)	C8B—C9B—C10B—N1B	164.10 (8)
C8A—C9A—C10A—C5A	−16.93 (12)	C8B—C9B—C10B—C5B	−16.41 (12)
C10A—N1A—C2A—C3A	16.82 (12)	C10B—N1B—C2B—C3B	19.06 (13)
C10A—N1A—C2A—C13A	−161.19 (8)	C10B—N1B—C2B—C13B	−158.87 (8)
C10A—C5A—C6A—O1A	−177.31 (8)	C10B—C5B—C6B—O1B	178.96 (9)
C10A—C5A—C6A—C7A	0.32 (12)	C10B—C5B—C6B—C7B	−3.55 (12)
C11A—C8A—C9A—C10A	−74.57 (9)	C11B—C8B—C9B—C10B	−75.44 (9)
C12A—C8A—C9A—C10A	165.38 (7)	C12B—C8B—C9B—C10B	163.30 (8)
C13A—C2A—C3A—C4A	−173.92 (8)	C13B—C2B—C3B—C4B	−177.07 (9)
C13A—C2A—C3A—C14A	6.94 (13)	C13B—C2B—C3B—C14B	5.89 (14)
C14A—O3A—C15A—C16A	77.56 (10)	C14B—O3B—C15B—C16B	−173.74 (8)
C14A—C3A—C4A—C5A	149.84 (7)	C14B—C3B—C4B—C5B	150.23 (8)
C14A—C3A—C4A—C17A	−86.04 (9)	C14B—C3B—C4B—C17B	−85.86 (10)
C15A—O3A—C14A—O2A	8.56 (12)	C15B—O3B—C14B—O2B	3.78 (13)
C15A—O3A—C14A—C3A	−170.22 (7)	C15B—O3B—C14B—C3B	−174.82 (8)
C17A—C4A—C5A—C6A	86.68 (9)	C17B—C4B—C5B—C6B	82.01 (10)
C17A—C4A—C5A—C10A	−94.30 (9)	C17B—C4B—C5B—C10B	−96.48 (9)
C17A—C18A—C19A—O5A	179.24 (8)	C17B—C18B—C19B—O5B	179.14 (8)
C17A—C18A—C19A—C20A	−0.95 (13)	C17B—C18B—C19B—C20B	−0.19 (13)
C18A—C17A—C22A—C21A	1.33 (13)	C18B—C17B—C22B—C21B	−0.80 (13)
C18A—C19A—C20A—O4A	−179.24 (8)	C18B—C19B—C20B—O4B	179.18 (8)
C18A—C19A—C20A—C21A	1.87 (13)	C18B—C19B—C20B—C21B	−0.63 (13)
C19A—C20A—C21A—O6A	−177.97 (8)	C19B—C20B—C21B—O6B	179.13 (8)
C19A—C20A—C21A—C22A	−1.22 (13)	C19B—C20B—C21B—C22B	0.72 (13)
C20A—C21A—C22A—C17A	−0.39 (13)	C20B—C21B—C22B—C17B	−0.02 (13)
C22A—C17A—C18A—C19A	−0.67 (13)	C22B—C17B—C18B—C19B	0.89 (13)
C23A—O5A—C19A—C18A	−0.11 (14)	C23B—O5B—C19B—C18B	8.86 (14)
C23A—O5A—C19A—C20A	−179.94 (8)	C23B—O5B—C19B—C20B	−171.77 (9)
C24A—O6A—C21A—C20A	−93.57 (10)	C24B—O6B—C21B—C20B	171.90 (8)
C24A—O6A—C21A—C22A	89.64 (10)	C24B—O6B—C21B—C22B	−9.80 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4*A*—H4*C*···O1*A*^{i}	0.848 (17)	1.937 (17)	2.6948 (9)	148.0 (16)
N1*A*—H1*A*···O1*B*^{ii}	0.880 (15)	1.890 (15)	2.7666 (10)	174.1 (13)
C7*A*—H7*C*···O6*B*^{ii}	0.99	2.67	3.4510 (12)	136
C12*A*—H12*D*···O2*A*^{iii}	0.98	2.60	3.5237 (12)	157
C13*A*—H13*D*···O1*B*^{ii}	0.98	2.59	3.3590 (12)	136
C16*A*—H16*D*···O4*A*^{iv}	0.98	2.65	3.3136 (13)	126
C24*A*—H24*E*···O4*B*^{v}	0.98	2.43	3.3105 (13)	149
N1*B*—H1···O1*A*^{i}	0.888 (15)	2.166 (15)	2.9479 (10)	146.6 (12)
C7*B*—H7*B*···O2*A*	0.99	2.69	3.4992 (11)	139

C9*B*—H9*B*···O6*A*	0.99	2.59	3.5751 (11)	172
C15*B*—H15*A*···O5*B*^{iii}	0.99	2.60	3.4993 (12)	151
C23*B*—H23*B*···O2*B*^{vi}	0.98	2.55	3.4277 (13)	149

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) *x*+1/2, −*y*+3/2, *z*−1/2; (v) −*x*+3/2, *y*+1/2, −*z*+3/2; (vi) *x*+1, *y*, *z*.

Ethyl 4-(anthracen-9-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (III)

Crystal data

$C_{29}H_{29}NO_3$
M_r = 439.53
Monoclinic, $P2_1/n$
a = 11.6527 (3) Å
b = 18.1986 (4) Å
c = 12.3435 (3) Å
β = 114.8758 (12)°
V = 2374.74 (10) Å^3
Z = 4
F(000) = 936
D_x = 1.229 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9872 reflections
θ = 2.2–27.6°
μ = 0.08 mm^{-1}
T = 100 K
Prism, yellow
0.45 × 0.12 × 0.11 mm

Data collection

Bruker SMART BREEZE CCD diffractometer
φ and *ω* scans
72579 measured reflections
5902 independent reflections
4515 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −15→15
k = −24→24
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.135
S = 1.04
5902 reflections
306 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 1.0544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.34009 (10)	0.80683 (6)	0.16928 (9)	0.0235 (2)
O2	0.31457 (11)	0.54998 (7)	0.51900 (11)	0.0331 (3)
O3	0.19449 (10)	0.64051 (6)	0.40339 (10)	0.0256 (2)
N1	0.61943 (12)	0.65569 (7)	0.47273 (11)	0.0182 (3)
H1	0.6990 (19)	0.6551 (11)	0.5295 (18)	0.032 (5)*

C2	0.52536 (13)	0.62310 (7)	0.49757 (12)	0.0177 (3)
C3	0.40249 (13)	0.63374 (7)	0.42341 (12)	0.0169 (3)
C4	0.36056 (13)	0.68248 (7)	0.31263 (12)	0.0153 (3)
H4	0.304711	0.721153	0.322453	0.018*
C5	0.47314 (13)	0.72274 (7)	0.30848 (12)	0.0152 (3)
C6	0.44911 (13)	0.78495 (7)	0.22944 (12)	0.0170 (3)
C7	0.55990 (14)	0.82620 (8)	0.22492 (13)	0.0202 (3)
H7A	0.575271	0.870831	0.274940	0.024*
H7B	0.536301	0.842425	0.141710	0.024*
C8	0.68311 (14)	0.78268 (8)	0.26676 (13)	0.0199 (3)
C9	0.70717 (13)	0.74830 (8)	0.38790 (13)	0.0188 (3)
H9A	0.778956	0.713567	0.410903	0.023*
H9B	0.731471	0.787529	0.449080	0.023*
C10	0.59379 (13)	0.70822 (7)	0.38663 (12)	0.0158 (3)
C11	0.67348 (15)	0.72310 (9)	0.17535 (14)	0.0260 (3)
H11A	0.602860	0.690143	0.164169	0.039*
H11B	0.752376	0.694870	0.204489	0.039*
H11C	0.659039	0.746243	0.098998	0.039*
C12	0.79290 (15)	0.83464 (9)	0.28370 (15)	0.0262 (3)
H12A	0.872397	0.806942	0.314765	0.039*
H12B	0.797430	0.873461	0.340391	0.039*
H12C	0.779071	0.856745	0.206789	0.039*
C13	0.57721 (15)	0.58079 (8)	0.61266 (13)	0.0240 (3)
H13A	0.663101	0.597854	0.662873	0.036*
H13B	0.579314	0.528329	0.595650	0.036*
H13C	0.523073	0.588557	0.654582	0.036*
C14	0.30380 (14)	0.60270 (8)	0.45520 (13)	0.0215 (3)
C15	0.08506 (16)	0.61002 (11)	0.41441 (16)	0.0340 (4)
H15A	0.023216	0.649703	0.404172	0.041*
H15B	0.111372	0.589143	0.495409	0.041*
C16	0.0228 (2)	0.55080 (12)	0.32261 (18)	0.0466 (5)
H16A	0.080715	0.509048	0.338088	0.070*
H16B	0.002429	0.570354	0.242627	0.070*
H16C	−0.055015	0.534573	0.327545	0.070*
C17	0.27883 (12)	0.64059 (7)	0.19723 (12)	0.0149 (3)
C18	0.33088 (13)	0.58220 (7)	0.15614 (12)	0.0151 (3)
C19	0.46066 (13)	0.55974 (8)	0.21467 (12)	0.0171 (3)
H19	0.515995	0.585501	0.283915	0.021*
C20	0.50692 (14)	0.50254 (8)	0.17387 (13)	0.0199 (3)
H20	0.593432	0.489206	0.215031	0.024*
C21	0.42774 (15)	0.46271 (8)	0.07091 (14)	0.0223 (3)
H21	0.460922	0.422798	0.043432	0.027*
C22	0.30435 (14)	0.48181 (8)	0.01176 (13)	0.0206 (3)
H22	0.251852	0.455228	−0.057734	0.025*
C23	0.25166 (13)	0.54115 (8)	0.05188 (12)	0.0174 (3)
C24	0.12363 (14)	0.55804 (8)	−0.00657 (12)	0.0192 (3)
H24	0.071627	0.530269	−0.074803	0.023*
C25	0.06980 (13)	0.61454 (8)	0.03234 (12)	0.0178 (3)

C26	0.14856 (13)	0.65780 (7)	0.13450 (12)	0.0162 (3)
C27	0.08637 (13)	0.71596 (8)	0.16756 (13)	0.0191 (3)
H27	0.135306	0.747691	0.231472	0.023*
C28	−0.04097 (14)	0.72696 (8)	0.10984 (14)	0.0220 (3)
H28	−0.078874	0.765462	0.135365	0.026*
C29	−0.11792 (14)	0.68199 (8)	0.01238 (14)	0.0228 (3)
H29	−0.206831	0.689377	−0.025495	0.027*
C30	−0.06317 (14)	0.62827 (8)	−0.02621 (13)	0.0210 (3)
H30	−0.114157	0.599310	−0.093293	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0196 (5)	0.0252 (5)	0.0203 (5)	0.0012 (4)	0.0030 (4)	0.0046 (4)
O2	0.0326 (7)	0.0324 (6)	0.0380 (7)	−0.0054 (5)	0.0184 (6)	0.0102 (5)
O3	0.0203 (5)	0.0324 (6)	0.0278 (6)	−0.0043 (4)	0.0139 (5)	0.0013 (5)
N1	0.0150 (6)	0.0202 (6)	0.0164 (6)	−0.0014 (5)	0.0038 (5)	0.0016 (5)
C2	0.0210 (7)	0.0160 (6)	0.0158 (6)	−0.0025 (5)	0.0074 (6)	−0.0012 (5)
C3	0.0198 (7)	0.0171 (6)	0.0147 (6)	−0.0025 (5)	0.0082 (5)	−0.0011 (5)
C4	0.0146 (6)	0.0163 (6)	0.0147 (6)	−0.0009 (5)	0.0059 (5)	−0.0005 (5)
C5	0.0156 (6)	0.0164 (6)	0.0140 (6)	−0.0020 (5)	0.0065 (5)	−0.0020 (5)
C6	0.0187 (7)	0.0178 (6)	0.0135 (6)	−0.0011 (5)	0.0058 (5)	−0.0016 (5)
C7	0.0231 (7)	0.0202 (7)	0.0173 (7)	−0.0036 (6)	0.0084 (6)	0.0021 (5)
C8	0.0196 (7)	0.0222 (7)	0.0195 (7)	−0.0046 (6)	0.0099 (6)	−0.0007 (5)
C9	0.0146 (7)	0.0228 (7)	0.0179 (7)	−0.0016 (5)	0.0058 (5)	0.0004 (5)
C10	0.0169 (7)	0.0162 (6)	0.0146 (6)	−0.0012 (5)	0.0070 (5)	−0.0014 (5)
C11	0.0274 (8)	0.0308 (8)	0.0247 (8)	−0.0053 (6)	0.0156 (7)	−0.0049 (6)
C12	0.0241 (8)	0.0297 (8)	0.0277 (8)	−0.0080 (6)	0.0137 (7)	0.0003 (6)
C13	0.0268 (8)	0.0236 (7)	0.0180 (7)	−0.0033 (6)	0.0061 (6)	0.0033 (6)
C14	0.0225 (7)	0.0266 (7)	0.0166 (7)	−0.0058 (6)	0.0092 (6)	−0.0055 (6)
C15	0.0261 (8)	0.0496 (11)	0.0316 (9)	−0.0113 (8)	0.0174 (7)	−0.0048 (8)
C16	0.0397 (11)	0.0598 (13)	0.0417 (11)	−0.0258 (10)	0.0186 (9)	−0.0104 (10)
C17	0.0157 (6)	0.0156 (6)	0.0140 (6)	−0.0022 (5)	0.0067 (5)	0.0004 (5)
C18	0.0168 (6)	0.0162 (6)	0.0141 (6)	−0.0022 (5)	0.0083 (5)	0.0013 (5)
C19	0.0172 (7)	0.0182 (6)	0.0164 (7)	−0.0014 (5)	0.0076 (5)	0.0013 (5)
C20	0.0200 (7)	0.0205 (7)	0.0220 (7)	0.0022 (5)	0.0115 (6)	0.0026 (5)
C21	0.0301 (8)	0.0174 (7)	0.0253 (8)	0.0001 (6)	0.0175 (7)	−0.0008 (6)
C22	0.0260 (8)	0.0188 (7)	0.0199 (7)	−0.0070 (6)	0.0125 (6)	−0.0046 (5)
C23	0.0207 (7)	0.0170 (6)	0.0162 (7)	−0.0041 (5)	0.0094 (6)	0.0005 (5)
C24	0.0207 (7)	0.0198 (7)	0.0152 (6)	−0.0056 (5)	0.0057 (6)	−0.0006 (5)
C25	0.0166 (7)	0.0189 (7)	0.0169 (7)	−0.0043 (5)	0.0062 (5)	0.0029 (5)
C26	0.0154 (6)	0.0170 (6)	0.0168 (6)	−0.0027 (5)	0.0074 (5)	0.0023 (5)
C27	0.0176 (7)	0.0195 (7)	0.0209 (7)	−0.0006 (5)	0.0087 (6)	0.0014 (5)
C28	0.0193 (7)	0.0220 (7)	0.0275 (8)	0.0017 (6)	0.0125 (6)	0.0055 (6)
C29	0.0144 (7)	0.0262 (7)	0.0260 (8)	−0.0014 (6)	0.0067 (6)	0.0094 (6)
C30	0.0175 (7)	0.0230 (7)	0.0193 (7)	−0.0060 (5)	0.0046 (6)	0.0041 (6)

Geometric parameters (Å, º)

O1—C6	1.2370 (17)	C9—C10	1.5036 (19)
O2—C14	1.2138 (19)	C15—C16	1.510 (3)
O3—C14	1.3489 (19)	C17—C18	1.4188 (19)
O3—C15	1.4486 (19)	C17—C26	1.4188 (19)
N1—C2	1.3893 (18)	C18—C19	1.4342 (19)
N1—C10	1.3658 (18)	C18—C23	1.4377 (19)
C2—C3	1.350 (2)	C19—C20	1.362 (2)
C2—C13	1.5013 (19)	C20—C21	1.416 (2)
C3—C4	1.5275 (18)	C21—C22	1.356 (2)
C3—C14	1.4749 (19)	C22—C23	1.430 (2)
C4—C5	1.5220 (18)	C23—C24	1.391 (2)
C4—C17	1.5413 (18)	C24—C25	1.391 (2)
C5—C6	1.4430 (19)	C25—C26	1.4417 (19)
C5—C10	1.3550 (19)	C25—C30	1.430 (2)
C6—C7	1.5146 (19)	C26—C27	1.4356 (19)
C7—C8	1.527 (2)	C27—C28	1.364 (2)
C8—C9	1.534 (2)	C28—C29	1.419 (2)
C8—C11	1.534 (2)	C29—C30	1.358 (2)
C8—C12	1.533 (2)		
C14—O3—C15	116.95 (13)	O2—C14—O3	122.01 (14)
C10—N1—C2	122.41 (12)	O2—C14—C3	126.59 (14)
N1—C2—C13	112.76 (12)	O3—C14—C3	111.41 (12)
C3—C2—N1	119.95 (12)	O3—C15—C16	111.51 (14)
C3—C2—C13	127.23 (13)	C18—C17—C4	120.66 (12)
C2—C3—C4	122.61 (12)	C18—C17—C26	119.36 (12)
C2—C3—C14	119.20 (13)	C26—C17—C4	119.90 (12)
C14—C3—C4	117.97 (12)	C17—C18—C19	123.51 (12)
C3—C4—C17	112.38 (11)	C17—C18—C23	119.94 (12)
C5—C4—C3	110.73 (11)	C19—C18—C23	116.53 (12)
C5—C4—C17	114.41 (11)	C20—C19—C18	121.93 (13)
C6—C5—C4	118.40 (12)	C19—C20—C21	120.87 (14)
C10—C5—C4	122.21 (12)	C22—C21—C20	119.68 (13)
C10—C5—C6	118.85 (12)	C21—C22—C23	121.31 (13)
O1—C6—C5	121.10 (13)	C22—C23—C18	119.67 (13)
O1—C6—C7	119.63 (12)	C24—C23—C18	119.61 (13)
C5—C6—C7	119.20 (12)	C24—C23—C22	120.69 (13)
C6—C7—C8	115.17 (12)	C25—C24—C23	121.68 (13)
C7—C8—C9	107.55 (11)	C24—C25—C26	119.48 (13)
C7—C8—C11	110.50 (12)	C24—C25—C30	120.32 (13)
C7—C8—C12	109.77 (12)	C30—C25—C26	120.17 (13)
C9—C8—C11	110.82 (12)	C17—C26—C25	119.88 (12)
C12—C8—C9	108.59 (12)	C17—C26—C27	124.04 (13)
C12—C8—C11	109.57 (12)	C27—C26—C25	116.07 (12)
C10—C9—C8	112.84 (11)	C28—C27—C26	121.87 (14)
N1—C10—C9	115.33 (12)	C27—C28—C29	121.26 (14)

C5—C10—N1	120.82 (12)	C30—C29—C28	119.33 (13)
C5—C10—C9	123.84 (12)	C29—C30—C25	121.17 (14)
O1—C6—C7—C8	−160.55 (13)	C13—C2—C3—C4	175.60 (13)
N1—C2—C3—C4	−1.6 (2)	C13—C2—C3—C14	1.1 (2)
N1—C2—C3—C14	−176.08 (12)	C14—O3—C15—C16	−82.03 (19)
C2—N1—C10—C5	−10.5 (2)	C14—C3—C4—C5	167.62 (12)
C2—N1—C10—C9	168.21 (12)	C14—C3—C4—C17	−63.06 (16)
C2—C3—C4—C5	−6.93 (18)	C15—O3—C14—O2	−7.7 (2)
C2—C3—C4—C17	122.39 (14)	C15—O3—C14—C3	172.08 (12)
C2—C3—C14—O2	−25.1 (2)	C17—C4—C5—C6	67.85 (16)
C2—C3—C14—O3	155.09 (13)	C17—C4—C5—C10	−120.75 (14)
C3—C4—C5—C6	−163.92 (11)	C17—C18—C19—C20	178.82 (13)
C3—C4—C5—C10	7.48 (18)	C17—C18—C23—C22	−179.25 (12)
C3—C4—C17—C18	−65.27 (16)	C17—C18—C23—C24	−1.38 (19)
C3—C4—C17—C26	111.42 (14)	C17—C26—C27—C28	−175.04 (13)
C4—C3—C14—O2	160.16 (14)	C18—C17—C26—C25	1.94 (19)
C4—C3—C14—O3	−19.65 (17)	C18—C17—C26—C27	−179.50 (12)
C4—C5—C6—O1	2.40 (19)	C18—C19—C20—C21	0.1 (2)
C4—C5—C6—C7	179.41 (12)	C18—C23—C24—C25	0.8 (2)
C4—C5—C10—N1	0.6 (2)	C19—C18—C23—C22	−0.43 (18)
C4—C5—C10—C9	−178.06 (12)	C19—C18—C23—C24	177.45 (12)
C4—C17—C18—C19	−2.05 (19)	C19—C20—C21—C22	0.2 (2)
C4—C17—C18—C23	176.69 (12)	C20—C21—C22—C23	−0.6 (2)
C4—C17—C26—C25	−174.79 (12)	C21—C22—C23—C18	0.8 (2)
C4—C17—C26—C27	3.8 (2)	C21—C22—C23—C24	−177.10 (13)
C5—C4—C17—C18	62.12 (16)	C22—C23—C24—C25	178.66 (13)
C5—C4—C17—C26	−121.19 (13)	C23—C18—C19—C20	0.04 (19)
C5—C6—C7—C8	22.39 (18)	C23—C24—C25—C26	1.1 (2)
C6—C5—C10—N1	171.93 (12)	C23—C24—C25—C30	−177.01 (13)
C6—C5—C10—C9	−6.7 (2)	C24—C25—C26—C17	−2.52 (19)
C6—C7—C8—C9	−49.79 (16)	C24—C25—C26—C27	178.81 (12)
C6—C7—C8—C11	71.30 (15)	C24—C25—C30—C29	178.32 (13)
C6—C7—C8—C12	−167.75 (12)	C25—C26—C27—C28	3.6 (2)
C7—C8—C9—C10	49.90 (15)	C26—C17—C18—C19	−178.75 (12)
C8—C9—C10—N1	157.27 (12)	C26—C17—C18—C23	−0.01 (19)
C8—C9—C10—C5	−24.03 (19)	C26—C25—C30—C29	0.2 (2)
C10—N1—C2—C3	11.0 (2)	C26—C27—C28—C29	−1.2 (2)
C10—N1—C2—C13	−166.55 (13)	C27—C28—C29—C30	−1.8 (2)
C10—C5—C6—O1	−169.29 (13)	C28—C29—C30—C25	2.3 (2)
C10—C5—C6—C7	7.72 (19)	C30—C25—C26—C17	175.63 (12)
C11—C8—C9—C10	−70.98 (15)	C30—C25—C26—C27	−3.04 (19)
C12—C8—C9—C10	168.62 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.90 (2)	1.94 (2)	2.7776 (16)	154.2 (18)

C13—H13*B*···O2^{ii}	0.98	2.65	3.409 (2)	134
C19—H19···N1	0.95	2.48	3.4148 (19)	168

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+1.