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(3*R**,1′*S**,3′*R**)-3-(3′-Hydroxy-1′*H*,3′*H*-benzo[*c*]furan-1′-yl)-2-(2″-hydroxyethyl)-2,3-dihydro-1*H*-benzo[*c*]pyrrol-1-one

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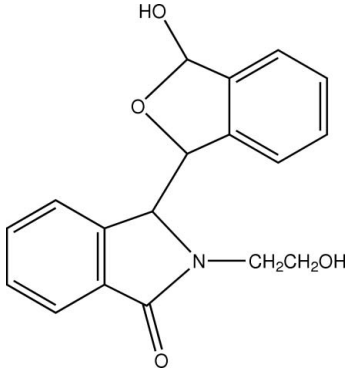
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.069; data-to-parameter ratio = 8.0.

The title compound, $C_{18}H_{17}NO_4$, is the first example of a molecular structure where the isobenzofuran and isoindoline groups are directly bonded. In the crystal structure, molecules are linked by O—H···O hydrogen bonds into sheets parallel to (001). Weaker interactions are also present, with C—H···O and π–π stacking interactions between the substituted furan and substituted pyrrole aromatic rings [centroid-to-centroid distance 3.3172 (13) Å].

Related literature

For the structure of a closely related compound isolated from the same reaction, see: Urban *et al.* (2007). For related literature, see: Becker & Coppens (1974); Zuman (2004). For an article on the biological activity of isoindolines, see: Mukherjee *et al.* (2000).



Experimental

Crystal data

$C_{18}H_{17}NO_4$
M_r = 311.3
Orthorhombic, $P2_12_12_1$
a = 7.0292 (1) Å
b = 11.9861 (5) Å
c = 18.0014 (7) Å
V = 1516.67 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 (2) K
0.2 × 0.08 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
18223 measured reflections
1737 independent reflections
1482 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.069
S = 1.65
1737 reflections
217 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Geometry of hydrogen bonds and D—H···π-ring interactions from *PLATON* (Spek, 2003).

*Cg*1 and *Cg*2 are the centroids of the rings C4′a–C4′ and C4–C5, respectively.

D—H···*A*/*Cg*	*D*—H	H···*A*/*Cg*	*D*···*A*/*Cg*	*D*—H···*A*/*Cg*
O4—H4O···O3	0.82 (2)	2.03 (2)	2.841 (2)	170 (2)
O3—H3O···O1	0.82 (2)	1.87 (2)	2.690 (2)	172 (2)
C1″—H1″a···O1	0.97	2.49	2.876 (2)	104
C1″—H1″a···O4	0.97	2.59	3.488 (3)	154
C3—H3···O3	0.98	2.49	3.244 (2)	133
C7—H7···O2′	0.93	2.50	3.408 (2)	165
C4—H4···*Cg*1	0.93	2.85	3.591 (2)	137
C1′—H1′···*Cg*2^{v}	0.98	2.81	3.645 (2)	144

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

Data collection: *COLLECT* (Nonius, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *JANA2000* (Petříček *et al.*, 2000); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *JANA2000*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2023).

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supplementary materials

Acta Cryst. (2007). E**63**, o4139-o4140 [**doi:10.1107/S1600536807045308**]

(3*R**,1'*S**,3'*R**)-3-(3'-Hydroxy-1'*H*,3'*H*-benzo[*c*]furan-1'-yl)-2-(2''-hydroxyethyl)-2,3-dihydro-1*H*-benzo[*c*]pyrrol-1-one

J. Urban, J. Fábry, P. Zuman, J. Ludvík and I. Císarová

Comment

Our research project deals with chemical and electrochemical properties of diketones (Zuman, 2004). As a part of this study, we have observed that a main product of the reaction of orthophthalaldehyde with amines in low concentrations about 10^{-3} mol/l is reducible about 0.5 V more negatively than the parent dialdehyde. In order to study this reaction as well as in order to identify the product (isoindoline derivative is expected to be formed) the reaction of phthalaldehyde with kolamine (2-aminoethanol) was performed.

In ethanol, however, the reaction results in a mixture of non-separable, viscous, probably polymeric compounds. On the other hand, the reaction in acetonitrile leads to two minor products together with formation of a non-separable mixture. The minor products were isolated, purified, crystallized and analyzed by NMR and single-crystal X-ray diffraction.

One of these compounds was identified as 2-(2''-hydroxyethyl)-3-(3'-hydroxy-1'*H*,3'*H*-benzo[*c*]furan-1'-yl-1H,3*H*-benzo[*c*]pyrrol-1-one (here reported as a title structure) while the second compound was characterized as 2-(2-hydroxyethyl)-1H,3H benzo[*c*]pyrrol-1-one (Urban *et al.*, 2007) as described in the preceeding article.

For more details, see Urban *et al.* (2007).

Two kinds of the O—H···O hydrogen bonds bind the title molecules into layers parallel to (001). The graph set motifs are $C_1^1(9)$ and $C_1^1(10)$ for the O1···O3 and O4···O3 hydrogen bonds, respectively.

The atoms in the five-membered ring N2–C1–C7a–C4a–C3 (substituted pyrrole) form a fair plane with a maximal deviation of C4a from the mean plane that is 0.010 (2) Å. On the other hand, the atoms in the five-membered ring O2'–C3'–C4'a–C7'a–C1' (substituted furan) are situated approximately in the plane with a maximal deviation from the mean plane for C3' that is 0.082 (3) Å.

The dihedral angle between the pyrrole and the attached phenyl ring is 1.19 (8)° while the dihedral angle between the substituted furan and the attached phenyl ring is 6.97 (8)°. These values also indicate a lesser aromaticity of the isobenzofuran ring in comparison with the isoindoline ring.

There is also a short π–π electron interaction between the aromatic rings O2'–C3'–C4'a–C7'a–C1' and N2–C1–C7a–C4a–C3 as indicates the distance between the respective centroids being 3.3172 (13) Å.

Many isoindoline derivatives display biological as well as pharmaceutical activity (Mukherjee *et al.*, 2000).

Experimental

The reaction has been described in the preceeding paper (Urban *et al.*, 2007). At least two products were produced by the reaction. Column chromatography afforded 256 mg of the title compound (I), 150 mg after its recrystallization from $CHCl_3$:n–C_6H_{14}.

Refinement

In the absence of significant anomalous scattering effects 1249 Friedel pairs have been merged (R_{int} = 0.022). All of the H atoms could be discerned in the difference Fourier maps, nevertheless, all of the H attached to the C atoms were constrained in a riding motion approximation while the hydroxyl H atoms were restrained (0.820 (1) Å) and their isotropic displacement parameters were freely refined, C_{aryl}—H = 0.93, $C_{methylene}$—H = 0.97, $C_{methine}$—H = 0.98 Å while U_{iso}H = 1.2U_{eq}C. The absolute configuration has not been determined.

Figures

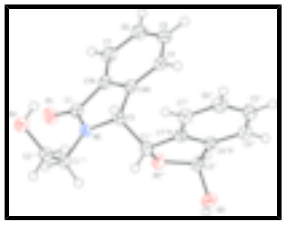

Fig. 1. The molecules of the title structure (I) with displacement parameters shown at 50% probability level.

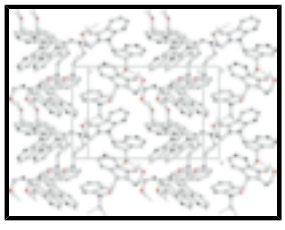

Fig. 2. A view of the title structure (I) with O—H···O hydrogen bonds.

(3*R**,1'*S**,3'*R**)-3-(3'-Hydroxy-1'*H*,3'*H*- benzo[*c*]furan-1'-yl)-2-(2''-hydroxyethyl)-2,3-dihydro-1*H*-benzo[*c*]pyrrol-1-one

Crystal data

$C_{18}H_{17}N_1O_4$	F_{000} = 656
M_r = 311.3	D_x = 1.363 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 1756 reflections
a = 7.0292 (1) Å	θ = 1–26.0º
b = 11.9861 (5) Å	μ = 0.10 mm^{-1}
c = 18.0014 (7) Å	T = 150 (2) K
V = 1516.67 (9) Å^3	Prism, colourless
Z = 4	0.2 × 0.08 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1737 independent reflections
Radiation source: fine-focus sealed tube	1482 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.058$
Detector resolution: 9.091 pixels mm^{-1}	$\theta_{max} = 26.0^{o}$
$T = 150(2)$ K	$\theta_{min} = 2.0^{o}$
φ and ω scans	$h = -8\rightarrow8$
Absorption correction: none	$k = -14\rightarrow14$
18223 measured reflections	$l = -22\rightarrow22$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.037$	Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$wR(F^2) = 0.069$	$(\Delta/\sigma)_{max} = 0.006$
$S = 1.65$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
1737 reflections	$\Delta\rho_{min} = -0.17$ e Å^{-3}
217 parameters	Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
2 restraints	Extinction coefficient: 1.57 (16)
60 constraints	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6319 (3)	0.35574 (19)	0.31944 (13)	0.0247 (7)
O1	0.4921 (2)	0.36611 (15)	0.36055 (9)	0.0344 (6)
N2	0.7803 (2)	0.28509 (16)	0.33170 (10)	0.0228 (6)
C3	0.9250 (3)	0.28969 (18)	0.27316 (12)	0.0223 (7)
H3	1.049523	0.313389	0.291876	0.0268*
C4a	0.8429 (3)	0.37409 (19)	0.22012 (12)	0.0229 (7)
C7a	0.6721 (3)	0.41363 (19)	0.24872 (13)	0.0243 (7)
C4	0.9105 (3)	0.41434 (19)	0.15273 (12)	0.0254 (8)
H4	1.025186	0.389264	0.133152	0.0305*
C5	0.8021 (3)	0.49305 (18)	0.11539 (13)	0.0286 (8)
H5	0.843911	0.519653	0.069689	0.0344*
C6	0.6330 (3)	0.5332 (2)	0.14428 (13)	0.0304 (8)
H6	0.564561	0.58684	0.118234	0.0364*
C7	0.5651 (3)	0.49403 (19)	0.21169 (13)	0.0300 (8)
H7	0.451645	0.520785	0.231403	0.036*
O2'	0.8001 (2)	0.13188 (13)	0.20416 (8)	0.0284 (5)
C3'	0.8425 (3)	0.09010 (18)	0.13168 (13)	0.0256 (7)
H3'	0.742632	0.110399	0.096186	0.0307*

C4'a	1.0382 (3)	0.13391 (19)	0.11551 (12)	0.0221 (7)
C7'a	1.1135 (3)	0.18025 (19)	0.17942 (13)	0.0222 (7)
C1'	0.9675 (3)	0.17481 (19)	0.23995 (12)	0.0239 (7)
H1'	1.008275	0.125368	0.280284	0.0286*
C4'	1.1405 (3)	0.13336 (19)	0.04959 (13)	0.0297 (8)
H4'	1.091314	0.100496	0.006917	0.0356*
C5'	1.3188 (3)	0.18345 (19)	0.04942 (14)	0.0322 (8)
H5'	1.388937	0.18607	0.005654	0.0387*
C6'	1.3937 (3)	0.2297 (2)	0.11368 (14)	0.0310 (8)
H6'	1.513096	0.262987	0.112345	0.0372*
C7'	1.2934 (3)	0.22706 (19)	0.17991 (14)	0.0271 (8)
H7'	1.345299	0.255859	0.223395	0.0325*
O3	0.8433 (2)	−0.02678 (14)	0.13010 (10)	0.0316 (6)
H3O	0.737 (2)	−0.055 (3)	0.131 (2)	0.107 (14)*
C1"	0.7943 (3)	0.21383 (19)	0.39678 (12)	0.0254 (7)
H1"a	0.675818	0.216763	0.424362	0.0304*
H1"b	0.807196	0.136731	0.381209	0.0304*
C2"	0.9573 (3)	0.24420 (19)	0.44749 (13)	0.0278 (8)
H2"a	0.963299	0.191412	0.488262	0.0334*
H2"b	1.076237	0.238248	0.420462	0.0334*
O4	0.9379 (3)	0.35389 (15)	0.47616 (10)	0.0349 (6)
H4O	0.993 (4)	0.395 (2)	0.4465 (14)	0.086 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0259 (12)	0.0258 (14)	0.0224 (13)	−0.0003 (11)	0.0000 (11)	−0.0029 (11)
O1	0.0323 (9)	0.0402 (11)	0.0308 (10)	0.0089 (8)	0.0089 (8)	0.0042 (9)
N2	0.0266 (10)	0.0256 (11)	0.0162 (10)	0.0031 (8)	0.0011 (8)	0.0006 (9)
C3	0.0205 (11)	0.0269 (13)	0.0195 (13)	0.0011 (10)	0.0018 (9)	0.0005 (11)
C4a	0.0270 (11)	0.0219 (12)	0.0199 (12)	−0.0033 (10)	−0.0029 (10)	−0.0029 (11)
C7a	0.0284 (11)	0.0223 (12)	0.0222 (13)	−0.0014 (10)	−0.0027 (10)	−0.0026 (10)
C4	0.0291 (12)	0.0221 (13)	0.0249 (14)	−0.0007 (10)	0.0011 (10)	−0.0029 (11)
C5	0.0361 (14)	0.0242 (14)	0.0256 (14)	−0.0039 (11)	−0.0021 (11)	0.0011 (11)
C6	0.0363 (13)	0.0262 (14)	0.0286 (14)	0.0022 (11)	−0.0081 (11)	0.0025 (12)
C7	0.0305 (13)	0.0300 (14)	0.0295 (14)	0.0066 (12)	−0.0029 (11)	−0.0018 (12)
O2'	0.0248 (8)	0.0306 (9)	0.0298 (10)	−0.0061 (7)	0.0054 (7)	−0.0079 (8)
C3'	0.0273 (12)	0.0239 (13)	0.0254 (14)	0.0016 (10)	0.0045 (11)	−0.0027 (11)
C4'a	0.0227 (11)	0.0196 (12)	0.0241 (13)	0.0025 (10)	0.0008 (10)	0.0006 (11)
C7'a	0.0220 (11)	0.0210 (13)	0.0237 (13)	0.0045 (9)	0.0020 (10)	0.0023 (10)
C1'	0.0228 (11)	0.0266 (13)	0.0222 (13)	0.0004 (10)	−0.0006 (10)	−0.0005 (11)
C4'	0.0347 (13)	0.0268 (13)	0.0276 (14)	−0.0003 (11)	0.0019 (11)	−0.0014 (12)
C5'	0.0311 (12)	0.0331 (14)	0.0324 (15)	−0.0028 (12)	0.0122 (12)	−0.0018 (13)
C6'	0.0229 (11)	0.0315 (15)	0.0387 (16)	−0.0015 (10)	0.0053 (11)	−0.0001 (12)
C7'	0.0225 (11)	0.0291 (15)	0.0296 (15)	0.0002 (10)	−0.0011 (11)	−0.0019 (12)
O3	0.0296 (9)	0.0230 (10)	0.0422 (11)	−0.0031 (7)	0.0046 (8)	−0.0034 (8)
C1"	0.0302 (12)	0.0264 (14)	0.0195 (13)	0.0006 (10)	0.0033 (10)	0.0022 (11)
C2"	0.0356 (13)	0.0255 (13)	0.0224 (13)	−0.0007 (11)	−0.0002 (11)	0.0017 (11)

O4	0.0498 (10)	0.0271 (11)	0.0279 (11)	−0.0029 (9)	0.0007 (8)	−0.0038 (9)

Geometric parameters (Å, °)

C1—O1	1.237 (3)	C5'—C6'	1.387 (3)
C1—N2	1.362 (3)	C6'—C7'	1.385 (3)
C1—C7a	1.477 (3)	C1"—C2"	1.510 (3)
N2—C3	1.466 (3)	C2"—O4	1.419 (3)
N2—C1"	1.453 (3)	C3—H3	0.98
C3—C4a	1.506 (3)	C4—H4	0.93
C3—C1'	1.531 (3)	C5—H5	0.93
C4a—C7a	1.390 (3)	C6—H6	0.93
C4a—C4	1.389 (3)	C7—H7	0.93
C7a—C7	1.392 (3)	C3'—H3'	0.98
C4—C5	1.387 (3)	C1'—H1'	0.98
C5—C6	1.384 (3)	C4'—H4'	0.93
C6—C7	1.386 (3)	C5'—H5'	0.93
O2'—C3'	1.429 (3)	C6'—H6'	0.93
O2'—C1'	1.437 (3)	C7'—H7'	0.93
C3'—C4'a	1.501 (3)	O3—H3O	0.82 (2)
C3'—O3	1.401 (3)	C1"—H1"a	0.97
C4'a—C7'a	1.383 (3)	C1"—H1"b	0.97
C4'a—C4'	1.388 (3)	C2"—H2"a	0.97
C7'a—C1'	1.499 (3)	C2"—H2"b	0.97
C7'a—C7'	1.383 (3)	O4—H4O	0.82 (3)
C4'—C5'	1.389 (3)		
O1—C1—N2	125.1 (2)	O2'—C3'—C4'a	104.22 (17)
O1—C1—C7a	128.4 (2)	O2'—C3'—O3	111.69 (18)
N2—C1—C7a	106.57 (18)	C4'a—C3'—O3	110.02 (18)
C1—N2—C3	113.06 (18)	C3'—C4'a—C7'a	109.29 (19)
C1—N2—C1"	123.23 (18)	C3'—C4'a—C4'	129.7 (2)
C3—N2—C1"	123.70 (17)	C7'a—C4'a—C4'	120.97 (19)
N2—C3—C4a	102.43 (16)	C4'a—C7'a—C1'	108.97 (17)
N2—C3—C1'	112.48 (17)	C4'a—C7'a—C7'	121.2 (2)
C4a—C3—C1'	115.56 (18)	C1'—C7'a—C7'	129.7 (2)
C3—C4a—C7a	108.98 (18)	C3—C1'—O2'	109.73 (16)
C3—C4a—C4	130.99 (19)	C3—C1'—C7'a	112.24 (18)
C7a—C4a—C4	120.0 (2)	O2'—C1'—C7'a	104.50 (17)
C1—C7a—C4a	108.93 (19)	C4'a—C4'—C5'	117.9 (2)
C1—C7a—C7	129.4 (2)	C4'—C5'—C6'	120.9 (2)
C4a—C7a—C7	121.7 (2)	C5'—C6'—C7'	121.1 (2)
C4a—C4—C5	118.2 (2)	C7'a—C7'—C6'	117.9 (2)
C4—C5—C6	121.8 (2)	N2—C1"—C2"	113.39 (18)
C5—C6—C7	120.5 (2)	C1"—C2"—O4	111.75 (19)
C7a—C7—C6	117.9 (2)	H1"a—C1"—H1"b	105.2
C3'—O2'—C1'	111.36 (15)	H2"a—C2"—H2"b	107.1

Hydrogen bonds and D—H···π-ring interactions from PLATON (Spek, 2003). Cg1 and Cg2 are the aromatic C_6 ring centroids of C4'a–C4' and C4–C5, respectively (Fig. 1).

D—H···A/*Cg*	D—H	H···A/*Cg*	D···A/*Cg*	D—H···A/*Cg*
O4-H4O···O3	0.82 (2)	2.03 (2)	2.841 (2)	170 (2)
O3-H3O···O1	0.82 (2)	1.87 (2)	2.690 (2)	172 (2)
C1"-H1"a···O1	0.97	2.49	2.876 (2)	104
C1"-H1"a···O4	0.97	2.59	3.488 (3)	154
C3-H3···O3	0.98	2.49	3.244 (2)	133
C7-H7···O2'	0.93	2.50	3.408 (2)	165
C4-H4···*Cg*1	0.93	2.85	3.591 (2)	137
C1'-H1'···*Cg*2^{v}	0.98	2.81	3.645 (2)	144

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

Fig. 1

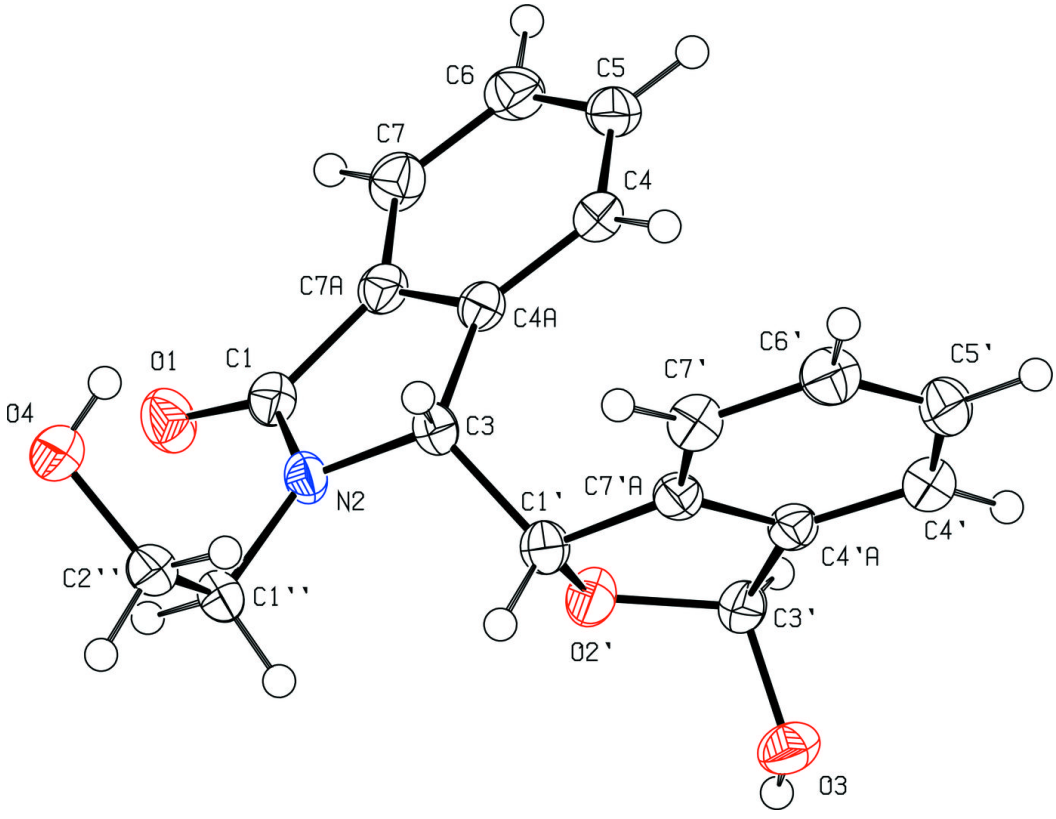
C6
C5
C7
C4
C7A
C4A
O1
C1
C6'
C5'
C7'
O4
C3
N2
C1'
C7'A
C4'
C4'A
C2''
C1''
C3'
O2'
O3

Fig. 2