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Ethyl *N*-[2-(2-hydroxymethyl-1,3-dioxolan-2-yl)phenyl]carbamate

Simon J. Garden,[a] Marilza B. Corrêa,[a] Angelo C. Pinto,[a] James L. Wardell,[a] John N. Low[b] and Christopher Glidewell[c]*

[a]Universidade Federal do Rio de Janeiro, Instituto de Química, Cidade Universitária, CT, Bloco A, Ilha do Fundão, Rio de Janeiro, RJ, CEP 21941-909, Brazil, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland
Correspondence e-mail: cg@st-andrews.ac.uk

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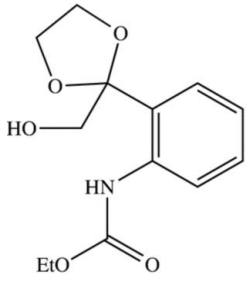
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.108; data-to-parameter ratio = 16.4.

The title compound, $C_{13}H_{17}NO_5$, (I), is a precursor of ethyl *N*-[2-(hydroxyacetyl)phenyl]carbamate, (II), whose structure we reported recently [Garden, Côrrea, Pinto, Wardell, Low & Glidewell (2007). *Acta Cryst.* C**63**, o234–o238]. In compound (I), the 1,3-dioxolane ring adopts a twisted conformation and the carbamate ester side chain adopts an almost planar all-*trans* conformation. Pairs of molecules are linked by O—H···O hydrogen bonds into a cyclic centrosymmetric $R_2^2(18)$ dimer to which are fused two $S(6)$ rings generated by an intramolecular N—H···O hydrogen bond.

Related literature

In the precursor compound (II) (Garden *et al.*, 2007) all of the non-H atoms lie on a mirror plane in space group *Pnma* and the molecules are linked by a single C—H···O hydrogen bond into a simple $C(6)$ chain.

For related literature, see: Garden *et al.* (2003).



Experimental

Crystal data

$C_{13}H_{17}NO_5$
M_r = 267.28
Monoclinic, $P2_1/n$
a = 7.4469 (2) Å
b = 20.2814 (6) Å
c = 8.3443 (3) Å
β = 94.1400 (17)°
V = 1256.98 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 (2) K
0.40 × 0.35 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.966, T_{max} = 0.991
15419 measured reflections
2859 independent reflections
2351 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.06
2859 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected torsion angles (°).

C2—C1—N1—C11	−158.84 (13)	C1—C2—C21—O21	−165.86 (11)
C1—N1—C11—O12	176.87 (12)	C1—C2—C21—O23	−48.29 (16)
N1—C11—O12—C12	177.10 (11)	C1—C2—C21—C22	71.59 (15)
C11—O12—C12—C13	175.90 (11)	C2—C21—C22—O22	52.33 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O23	0.91	2.04	2.7451 (14)	134
O22—H22···O11^{i}	0.84	2.05	2.8324 (14)	155

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JLW thanks CNPq and FAPERJ for financial support; SJG and ACP thank CNPq for financial support, and CAPES for a grant for MBC.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2361).

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supplementary materials

Acta Cryst. (2007). E**63**, o2453-o2454 [**doi:10.1107/S1600536807016467**]

Ethyl *N*-[2-(2-hydroxymethyl-1,3-dioxolan-2-yl)phenyl]carbamate

S. J. Garden, M. B. Corrêa, A. C. Pinto, J. L. Wardell, J. N. Low and C. Glidewell

Comment

We report here the structure of the title compound (I) (Fig. 2), which is a precursor (Garden *et al.*, 2003) of ethyl *N*-[2-(hydroxyacetyl)phenyl]carbamate (II), whose structure we reported recently (Garden *et al.*, 2007).

The 1,3-dioxolane ring in (I) adopts a twisted conformation with ring-puckering parameters (Cremer & Pople, 1975) for the atom sequence (O21, C21, O23, C24, C23) of Q_2 0.360 (2) Å and φ_2 347.3 (2)°, with atom displacements from the mean plane indicative of a conformation twisted about the line joining atom O23 to the mid-point of the O21—C23 bond. The carbamate ester side chain adopts an almost planar, all-*trans* conformation, as indicated by the relevant torsional angles (Table 1). The conformation about the C2—C21 bond is such that atom O21 is close to the plane of the aryl ring: hence it seems unlikely that this conformation is materially influenced by the intramolecular N—H···O hydrogen bond (Table 2).

A single O—H···O hydrogen bond (Table 2) links pairs of molecules into cyclic dimers: the hydroxyl atom O22 at (*x*, *y*, *z*) acts as hydrogen-bond donor to the carbonyl atom O11 in the molecule at (1 - *x*, 1 - *y*, 1 - *z*), so generating by inversion an $R^2_2(18)$ ring (Bernstein *et al.*, 1995) centred at (1/2, 1/2, 1/2) to which are fused two S(6) rings generated by the intramolecular N—H···O hydrogen bond (Fig. 3). Two dimers of this type lie in each unit cell, but there are no direction-specific interactions between the dimer units.

Experimental

Compound (I) was prepared as previously reported (Garden *et al.*, 2003). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in hexane-dichloromethane (1:1, *v*/*v*).

Refinement

All the H atoms were located in difference maps, relocated in idealized positions, and treated as riding atoms with distances C—H 0.95 Å (aromatic), 0.98 Å (CH_3) or 0.99 Å (CH_2), N—H 0.91 Å and O—H 0.84 Å, and with $U_{iso}(H) = kU_{eq}$(carrier), where k = 1.5 fo the hydroxyl and methyl H atoms and 1.2 for all other H atoms.

Figures

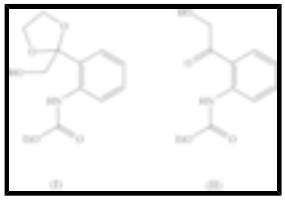


Fig. 1. Scheme showing (I) and (II).

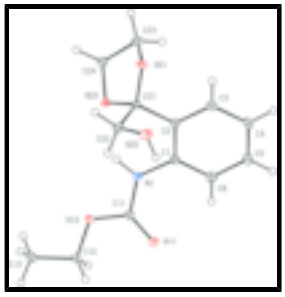


Fig. 2. A molecule of compound (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

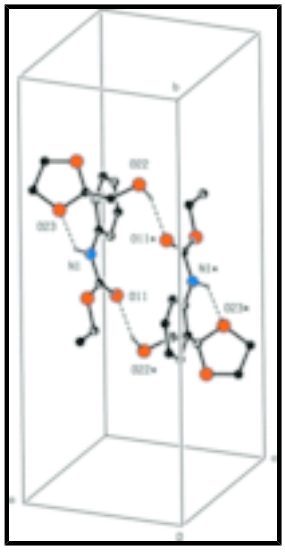


Fig. 3. Part of the crystal structure of compound (I) showing the formation of a hydrogen-bonded dimer centred at (1/2, 1/2, 1/2) and containing one $R^2_2(18)$ ring and two S(6) rings. For the sake of clarity, the H atoms not involved in the motifs shown have been omitted. The atoms marked with an asterisk (*) are at the symmetry position (1 - *x*, 1 - *y*, 1 - *z*).

Ethyl *N*-[2-(2-hydroxymethyl-1,3-dioxolan-2-yl)phenyl]carbamate

Crystal data

$C_{13}H_{17}NO_5$	F_{000} = 568
M_r = 267.28	D_x = 1.412 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 7223 reflections
a = 7.4469 (2) Å	θ = 2.9–27.5º
b = 20.2814 (6) Å	μ = 0.11 mm^{-1}
c = 8.3443 (3) Å	*T* = 120 (2) K
β = 94.1400 (17)º	Plate, colourless
V = 1256.98 (7) Å^3	0.40 × 0.35 × 0.08 mm
Z = 4	

Data collection

Bruker–Nonius KappaCCD diffractometer	2859 independent reflections
Radiation source: Bruker-Nonius FR591 rotating anode	2351 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.043

Detector resolution: 9.091 pixels mm^{-1}

T = 120(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2003)

T_{min} = 0.966, T_{max} = 0.991

15419 measured reflections

θ_{max} = 27.5º

θ_{min} = 2.9º

h = −9→9

k = −26→25

l = −10→10

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.040

$wR(F^2)$ = 0.108

S = 1.06

2859 reflections

174 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.3663P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.25 e Å^{-3}

$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.80896 (17)	0.55854 (6)	0.58422 (16)	0.0149 (3)
N1	0.78526 (15)	0.52817 (5)	0.43164 (13)	0.0162 (3)
C11	0.70954 (17)	0.46878 (6)	0.39499 (16)	0.0150 (3)
O11	0.65376 (13)	0.42894 (5)	0.48877 (12)	0.0216 (2)
O12	0.70481 (13)	0.45971 (5)	0.23489 (11)	0.0185 (2)
C12	0.63510 (19)	0.39661 (6)	0.17684 (16)	0.0181 (3)
C13	0.6294 (2)	0.39934 (7)	−0.00381 (17)	0.0236 (3)
C2	0.83290 (16)	0.62743 (6)	0.59031 (16)	0.0142 (3)
C21	0.81496 (17)	0.67041 (6)	0.43921 (16)	0.0150 (3)
O21	0.88614 (12)	0.73417 (4)	0.46946 (12)	0.0179 (2)
C23	1.07494 (19)	0.72825 (7)	0.44691 (18)	0.0204 (3)
C24	1.07660 (18)	0.68280 (7)	0.30267 (17)	0.0195 (3)
O23	0.91580 (13)	0.64355 (4)	0.31326 (11)	0.0189 (2)
C22	0.62001 (18)	0.67700 (7)	0.36960 (17)	0.0191 (3)
O22	0.50165 (13)	0.69769 (5)	0.48450 (12)	0.0220 (2)
C3	0.86213 (17)	0.65753 (7)	0.74049 (16)	0.0168 (3)
C4	0.86634 (18)	0.62115 (7)	0.88192 (16)	0.0187 (3)
C5	0.84013 (18)	0.55357 (7)	0.87435 (16)	0.0197 (3)
C6	0.81261 (19)	0.52213 (7)	0.72655 (17)	0.0185 (3)
H1	0.8325	0.5492	0.3486	0.019*
H12A	0.5129	0.3891	0.2128	0.022*

H12B	0.7145	0.3603	0.2181	0.022*
H13A	0.5472	0.4345	−0.0431	0.035*
H13C	0.5869	0.3570	−0.0485	0.035*
H13B	0.7504	0.4084	−0.0374	0.035*
H23A	1.1410	0.7086	0.5425	0.024*
H23B	1.1285	0.7716	0.4242	0.024*
H24A	1.0717	0.7082	0.2012	0.023*
H24B	1.1856	0.6547	0.3092	0.023*
H22A	0.6152	0.7092	0.2802	0.023*
H22B	0.5788	0.6339	0.3250	0.023*
H22	0.4503	0.6647	0.5212	0.033*
H3	0.8795	0.7039	0.7461	0.020*
H4	0.8870	0.6425	0.9829	0.022*
H5	0.8410	0.5286	0.9707	0.024*
H6	0.7962	0.4757	0.7223	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0136 (6)	0.0167 (6)	0.0143 (6)	0.0003 (5)	0.0012 (5)	−0.0023 (5)
N1	0.0210 (6)	0.0147 (5)	0.0132 (5)	−0.0031 (4)	0.0033 (4)	−0.0009 (4)
C11	0.0135 (6)	0.0155 (6)	0.0159 (7)	0.0004 (5)	0.0007 (5)	−0.0013 (5)
O11	0.0279 (5)	0.0186 (5)	0.0185 (5)	−0.0070 (4)	0.0027 (4)	0.0014 (4)
O12	0.0260 (5)	0.0147 (5)	0.0148 (5)	−0.0055 (4)	0.0018 (4)	−0.0027 (4)
C12	0.0215 (7)	0.0138 (6)	0.0188 (7)	−0.0044 (5)	−0.0002 (5)	−0.0023 (5)
C13	0.0338 (8)	0.0185 (7)	0.0181 (7)	−0.0046 (6)	−0.0007 (6)	−0.0020 (5)
C2	0.0100 (6)	0.0164 (6)	0.0164 (7)	0.0004 (5)	0.0023 (5)	−0.0009 (5)
C21	0.0164 (6)	0.0123 (6)	0.0168 (6)	−0.0015 (5)	0.0038 (5)	−0.0023 (5)
O21	0.0173 (5)	0.0125 (5)	0.0246 (5)	−0.0029 (4)	0.0055 (4)	−0.0031 (4)
C23	0.0173 (7)	0.0192 (7)	0.0255 (8)	−0.0047 (5)	0.0072 (6)	−0.0022 (6)
C24	0.0180 (7)	0.0215 (7)	0.0195 (7)	−0.0038 (5)	0.0053 (5)	−0.0009 (5)
O23	0.0229 (5)	0.0168 (5)	0.0178 (5)	−0.0056 (4)	0.0080 (4)	−0.0036 (4)
C22	0.0188 (7)	0.0193 (7)	0.0188 (7)	−0.0017 (5)	−0.0008 (5)	0.0028 (5)
O22	0.0168 (5)	0.0209 (5)	0.0289 (6)	0.0002 (4)	0.0041 (4)	0.0017 (4)
C3	0.0129 (6)	0.0180 (6)	0.0196 (7)	0.0001 (5)	0.0020 (5)	−0.0042 (5)
C4	0.0164 (6)	0.0247 (7)	0.0146 (7)	0.0008 (5)	−0.0019 (5)	−0.0043 (5)
C5	0.0198 (7)	0.0240 (7)	0.0149 (7)	0.0035 (5)	−0.0012 (5)	0.0018 (5)
C6	0.0203 (7)	0.0163 (6)	0.0190 (7)	0.0016 (5)	0.0017 (5)	0.0009 (5)

Geometric parameters (Å, °)

C1—C6	1.3971 (19)	O21—C23	1.4369 (17)
C1—C2	1.4090 (18)	C23—C24	1.5169 (19)
C1—N1	1.4139 (17)	C23—H23A	0.99
N1—C11	1.3555 (17)	C23—H23B	0.99
N1—H1	0.9060	C24—O23	1.4459 (16)
C11—O11	1.2185 (16)	C24—H24A	0.99
C11—O12	1.3464 (16)	C24—H24B	0.99
O12—C12	1.4512 (15)	C22—O22	1.4122 (17)

C12—C13	1.5059 (19)	C22—H22A	0.99
C12—H12A	0.99	C22—H22B	0.99
C12—H12B	0.99	O22—H22	0.84
C13—H13A	0.98	C3—C4	1.390 (2)
C13—H13C	0.98	C3—H3	0.95
C13—H13B	0.98	C4—C5	1.385 (2)
C2—C3	1.3968 (18)	C4—H4	0.95
C2—C21	1.5306 (18)	C5—C6	1.390 (2)
C21—O21	1.4133 (15)	C5—H5	0.95
C21—O23	1.4420 (15)	C6—H6	0.95
C21—C22	1.5300 (19)		
C6—C1—C2	119.93 (12)	O21—C23—C24	102.64 (11)
C6—C1—N1	121.91 (12)	O21—C23—H23A	111.2
C2—C1—N1	118.15 (11)	C24—C23—H23A	111.2
C11—N1—C1	127.82 (11)	O21—C23—H23B	111.2
C11—N1—H1	115.1	C24—C23—H23B	111.2
C1—N1—H1	117.0	H23A—C23—H23B	109.2
O11—C11—O12	124.12 (12)	O23—C24—C23	103.41 (10)
O11—C11—N1	126.86 (12)	O23—C24—H24A	111.1
O12—C11—N1	109.02 (11)	C23—C24—H24A	111.1
C11—O12—C12	115.77 (10)	O23—C24—H24B	111.1
O12—C12—C13	106.57 (11)	C23—C24—H24B	111.1
O12—C12—H12A	110.4	H24A—C24—H24B	109.0
C13—C12—H12A	110.4	C21—O23—C24	108.29 (10)
O12—C12—H12B	110.4	O22—C22—C21	112.95 (11)
C13—C12—H12B	110.4	O22—C22—H22A	109.0
H12A—C12—H12B	108.6	C21—C22—H22A	109.0
C12—C13—H13A	109.5	O22—C22—H22B	109.0
C12—C13—H13C	109.5	C21—C22—H22B	109.0
H13A—C13—H13C	109.5	H22A—C22—H22B	107.8
C12—C13—H13B	109.5	C22—O22—H22	109.5
H13A—C13—H13B	109.5	C4—C3—C2	121.47 (13)
H13C—C13—H13B	109.5	C4—C3—H3	119.3
C3—C2—C1	118.48 (12)	C2—C3—H3	119.3
C3—C2—C21	119.26 (12)	C5—C4—C3	119.47 (13)
C1—C2—C21	122.12 (11)	C5—C4—H4	120.3
O21—C21—O23	105.51 (9)	C3—C4—H4	120.3
O21—C21—C22	108.74 (10)	C4—C5—C6	120.37 (13)
O23—C21—C22	106.62 (11)	C4—C5—H5	119.8
O21—C21—C2	111.38 (11)	C6—C5—H5	119.8
O23—C21—C2	111.48 (10)	C5—C6—C1	120.27 (13)
C22—C21—C2	112.74 (10)	C5—C6—H6	119.9
C21—O21—C23	104.96 (9)	C1—C6—H6	119.9
C6—C1—N1—C11	22.4 (2)	C22—C21—O21—C23	−149.12 (11)
C2—C1—N1—C11	−158.84 (13)	C2—C21—O21—C23	86.06 (12)
C1—N1—C11—O11	−3.2 (2)	C21—O21—C23—C24	39.27 (13)
C1—N1—C11—O12	176.87 (12)	O21—C23—C24—O23	−28.36 (13)
O11—C11—O12—C12	−2.81 (18)	O21—C21—O23—C24	16.27 (14)

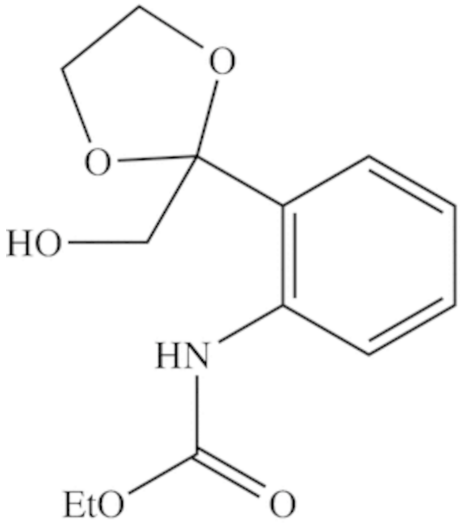
N1—C11—O12—C12	177.10 (11)	C22—C21—O23—C24	131.78 (11)
C11—O12—C12—C13	175.90 (11)	C2—C21—O23—C24	−104.78 (12)
C6—C1—C2—C3	0.77 (18)	C23—C24—O23—C21	7.74 (14)
N1—C1—C2—C3	−177.99 (11)	O21—C21—C22—O22	−71.69 (13)
C6—C1—C2—C21	−174.92 (11)	O23—C21—C22—O22	174.98 (10)
N1—C1—C2—C21	6.32 (18)	C2—C21—C22—O22	52.33 (15)
C3—C2—C21—O21	18.48 (16)	C1—C2—C3—C4	−0.55 (18)
C1—C2—C21—O21	−165.86 (11)	C21—C2—C3—C4	175.27 (12)
C3—C2—C21—O23	136.05 (12)	C2—C3—C4—C5	−0.33 (19)
C1—C2—C21—O23	−48.29 (16)	C3—C4—C5—C6	1.0 (2)
C3—C2—C21—C22	−104.07 (13)	C4—C5—C6—C1	−0.8 (2)
C1—C2—C21—C22	71.59 (15)	C2—C1—C6—C5	−0.12 (19)
O23—C21—O21—C23	−35.06 (13)	N1—C1—C6—C5	178.59 (12)

Hydrogen-bond geometry (Å, °)

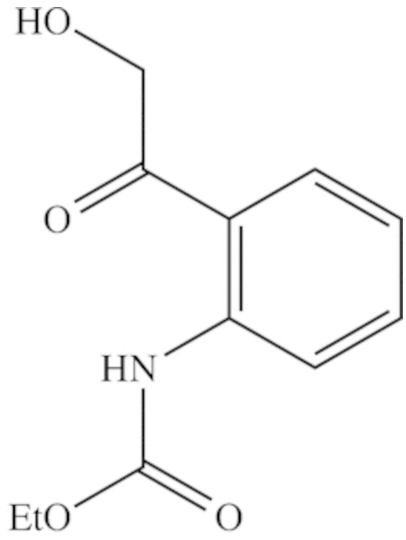
D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O23	0.91	2.04	2.7451 (14)	134
O22—H22···O11^{i}	0.84	2.05	2.8324 (14)	155

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1



(I)



(II)

Fig. 2

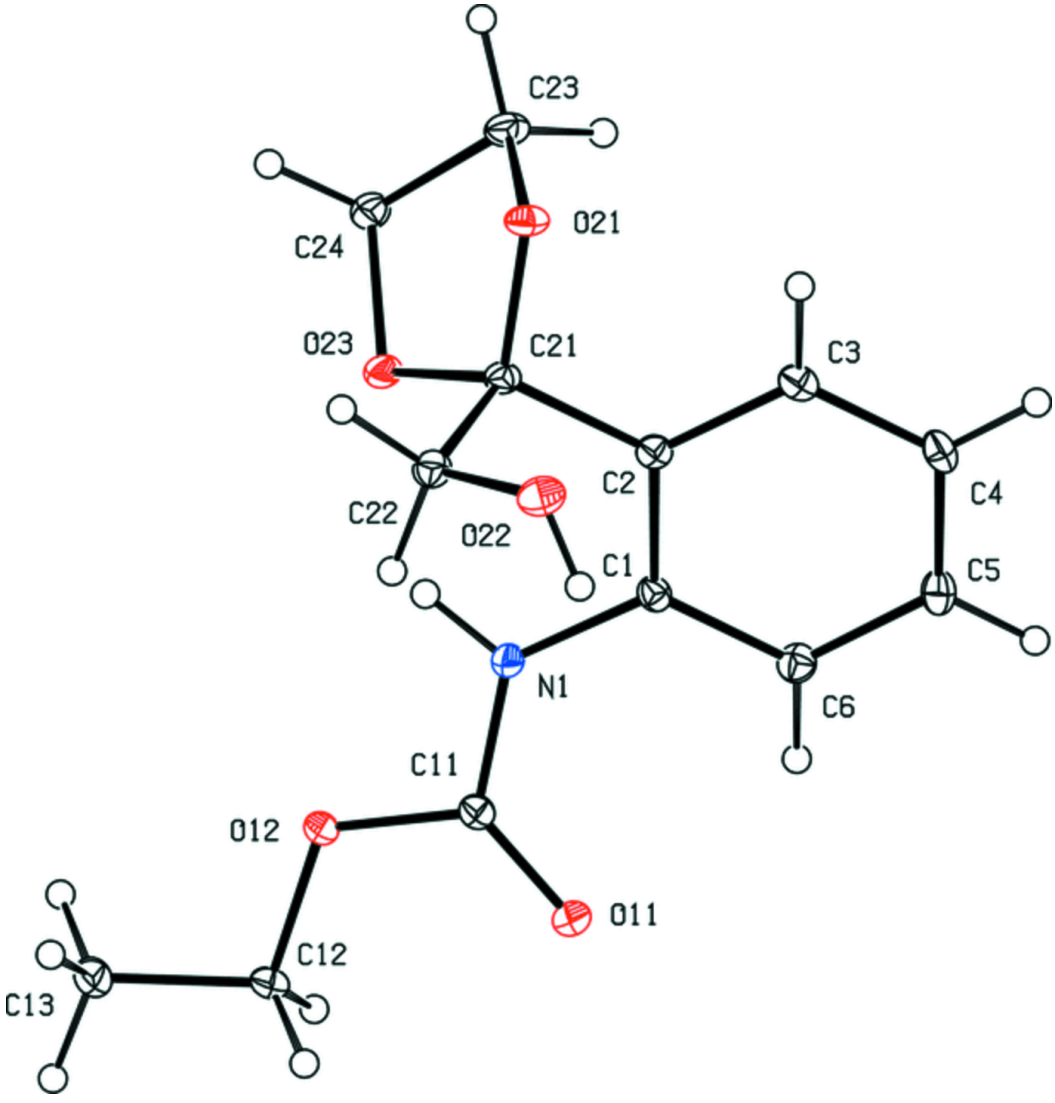

C23
O21
C24
O23
C21
C3
C2
C4
C22
O22
C1
C5
N1
C6
C11
O12
O11
C12
C13

Fig. 3