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(*Z*)-3-(4-Hydroxyphenethylamino)-2-methyl-1-phenylprop-2-en-1-one

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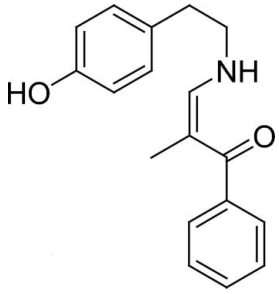
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.074; wR factor = 0.157; data-to-parameter ratio = 13.8.

The title compound, $C_{18}H_{19}NO_2$, was synthesized by the substituted one-carbon unit transfer reaction of 4-(2-aminoethyl)phenol with an imidazolidine derivative. In the crystal structure, the molecules are packed together by intermolecular O—H···O and N—H···O hydrogen bonds into layers. The different layers are stacked together by π–π interactions with a centroid-to-centroid distance of 5.836 Å between 4-hydroxyphene groups in adjacent layers.

Related literature

For related literature, see: Duthaler (2003); Elassar & Ei-Khair (2003); Fan *et al.* (2005); Li *et al.* (2004); Stanovnik & Svete (2004).



Experimental

Crystal data

$C_{18}H_{19}NO_2$
M_r = 282.35
Monoclinic, $P2_1/n$
a = 9.8035 (17) Å
b = 9.4636 (16) Å
c = 16.457 (3) Å
β = 100.064 (2)°
V = 1503.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
0.30 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
7096 measured reflections
2657 independent reflections
2085 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.157
S = 1.01
2657 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.91	2.728 (3)	178
N1—H1A···O2^{ii}	0.86	2.20	3.014 (3)	158

Symmetry codes: (i) $-x+2, -y, -z$; (ii) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2021).

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supplementary materials

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(*Z*)-3-(4-Hydroxyphenethylamino)-2-methyl-1-phenylprop-2-en-1-one

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Comment

Secondary enamines have attracted a great deal of attention in recent years because of their range of applications (Duthaler, 2003; Stanovnik & Svete, 2004; Elassar & Ei-Khair, 2003). Moreover, functionalize secondary enamine derives may enable chemical and biological studies on these derivatives, which will be used in pharmaceutical reseach. Here we carried out the synthesis of secondary enamine derivative according to early papers (Fan *et al.*, 2005; Li *et al.*, 2004).

The molecular geometry of compound (I) including C=N is an E configuration (Fig. 1). The bond distance of C10—C12 and C12—C13 are shorter than normal case. These bond distances suggest delocalization of π electrons, and there is a great interaction between π bond of the C9—C10 and N lone-pair electrons.

The O—H···O and N—H···O hydrogen bonds were responsible for packing of molecules (see Fig. 2). two parallel benzen groups attached to carbonyl located in one unit which was formed by the intermolecular hydrogen bonds O1- H1— O2. The units are linked together by intermolecular hydrogen bonds N1- H1A—O2.

In the crystal sructure,the molecules are linked together into layers by a combination of O1- H1— O2 and N1- H1A—O2 hydrogen bonds. The different layers were stacked together by π -π interactions between 4-hydroxyphene groups in adjacent layers (see Fig.3).

Experimental

Into a 50 ml 3-necked roundbottom flask, was placed a solution of 2-(3,4-dimethyl-1-tosylimidazolidin-2-yl)-1-phenylpropan-1-one (0.387 g, 1 mmol) in anhydrous acetonitrile (10 ml). To this was added 4-(2-aminoethyl)phenol (0.137 g, 1 mmol). The resulting solution was allowed to react, with stirring, for 2 h while the temperature was maintained at reflux in a bath of oil. The reaction progress was monitored by TLC (EtOAc/PE = 1:1). The residue was purified by eluting through a column with a 1:10 EA/PE solvent system This resulted in 0.253 g (90%) of (*Z*)-3-(4-hydroxyphenethylamino)-2-methyl-1-phenylprop-2-en-1-one as a white solid. Crystals of the compound were obtained by slow evaporation of an ethanol solution. m.p.=189–190oC.1*H*-NMR (300 MHz, ? p.p.m., DMSO-d6): 1.68 (s, 3H), 2.68 (t, 2H), 3.22 (m, 2H), 6.59–6.71 (m, 4H), 6.93–7.05 (m, 3H), 7.30–7.35 (m, 3H), 9.24 (br, 1H),10.86 (br, 1H).

Refinement

H atoms bonded to C atoms were treated as riding atoms, with C–H = 0.93–0.97 Å, their U_{iso} values were set at 1.2 U_{eq} (C atom); The amine H atom were refined freely in isotropic approximation; The H atom bonded to N3 was permitted to ride at the distance deduced from difference maps (0.88 A Ê), with U_{iso}(H) = 1.2Ueq(N).

Figures

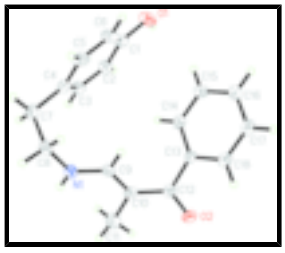

Fig. 1. The structure of the compound (I) with the atom-numbering scheme, showing 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitary radii

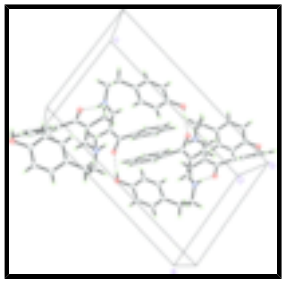

Fig. 2. Packing diagram of (I), showing the hydrogen bond interactions (dashed lines) and hydrogen bonds are indicated by dashed lines

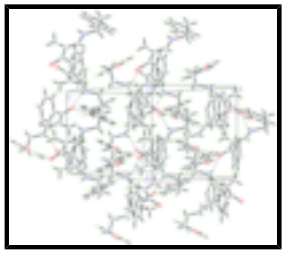

Fig. 3. A view along the *b* axis showing the stacking of the layers. with small black spheres representing H atoms.

(*Z*)-3-(4-Hydroxyphenethylamino)-2-methyl-1-phenylprop-2-en-1-one

Crystal data

$C_{18}H_{19}NO_2$	F_{000} = 604
M_r = 282.35	D_x = 1.248 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -p 2yn	Cell parameters from 1527 reflections
a = 9.8035 (17) Å	θ = 2.5–21.9º
b = 9.4636 (16) Å	μ = 0.08 mm^{-1}
c = 16.457 (3) Å	T = 294 (2) K
β = 100.064 (2)º	Block, colorless
V = 1503.3 (4) Å^3	0.30 × 0.30 × 0.30 mm
Z = 4	

Data collection

Bruker SMART CCD area detector diffractometer	2085 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
Monochromator: graphite	θ_{max} = 25.0º

T = 566(2) K
φ and ω scans
Absorption correction: none
7096 measured reflections
2657 independent reflections

θ_{min} = 2.3°
h = −11→11
k = −11→9
l = −11→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.157
S = 1.01
2657 reflections
192 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 1.2494P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	1.0477 (2)	−0.0208 (3)	0.30663 (14)	0.0409 (6)
H1A	1.1270	−0.0581	0.3253	0.061*
O1	0.6077 (2)	−0.1801 (3)	−0.01827 (14)	0.0591 (7)
H1	0.6602	−0.2100	−0.0480	0.089*
O2	1.2241 (2)	0.2788 (2)	0.12128 (13)	0.0486 (6)
C1	0.6726 (3)	−0.1854 (3)	0.06243 (19)	0.0431 (8)
C2	0.8010 (3)	−0.2461 (3)	0.0859 (2)	0.0440 (8)
H2	0.8478	−0.2840	0.0464	0.053*
C3	0.8598 (3)	−0.2502 (3)	0.1681 (2)	0.0436 (8)
H3	0.9459	−0.2931	0.1832	0.052*
C4	0.7964 (3)	−0.1934 (3)	0.22892 (19)	0.0388 (7)
C5	0.6678 (3)	−0.1302 (3)	0.2033 (2)	0.0481 (8)
H5	0.6217	−0.0903	0.2425	0.058*

C6	0.6073 (3)	−0.1253 (4)	0.1214 (2)	0.0498 (9)
H6	0.5220	−0.0813	0.1058	0.060*
C7	0.8627 (3)	−0.1961 (3)	0.31844 (19)	0.0447 (8)
H7A	0.9313	−0.2709	0.3271	0.054*
H7B	0.7925	−0.2175	0.3515	0.054*
C8	0.9321 (3)	−0.0567 (3)	0.34762 (18)	0.0425 (8)
H8A	0.8637	0.0183	0.3379	0.051*
H8B	0.9653	−0.0621	0.4066	0.051*
C9	1.0387 (3)	0.0649 (3)	0.24285 (17)	0.0366 (7)
H9	0.9512	0.0994	0.2209	0.044*
C10	1.1465 (3)	0.1080 (3)	0.20575 (18)	0.0396 (7)
C11	1.2915 (3)	0.0590 (4)	0.2396 (2)	0.0673 (11)
H11A	1.3160	0.0869	0.2963	0.101*
H11B	1.3550	0.1008	0.2083	0.101*
H11C	1.2960	−0.0421	0.2356	0.101*
C12	1.1256 (3)	0.2083 (3)	0.14051 (18)	0.0382 (7)
C13	0.9861 (3)	0.2319 (3)	0.08866 (18)	0.0345 (7)
C14	0.9037 (3)	0.1187 (3)	0.05629 (19)	0.0449 (8)
H14	0.9292	0.0272	0.0730	0.054*
C15	0.7844 (3)	0.1412 (3)	−0.0005 (2)	0.0544 (9)
H15	0.7305	0.0646	−0.0221	0.065*
C16	0.7448 (4)	0.2756 (4)	−0.0251 (2)	0.0555 (9)
H16	0.6647	0.2901	−0.0637	0.067*
C17	0.8237 (3)	0.3888 (3)	0.0073 (2)	0.0524 (9)
H17	0.7963	0.4803	−0.0085	0.063*
C18	0.9440 (3)	0.3665 (3)	0.0634 (2)	0.0452 (8)
H18	0.9976	0.4435	0.0846	0.054*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0348 (14)	0.0486 (15)	0.0379 (15)	0.0017 (11)	0.0024 (11)	0.0086 (12)
O1	0.0525 (14)	0.0759 (17)	0.0436 (14)	−0.0026 (12)	−0.0062 (11)	−0.0095 (13)
O2	0.0433 (13)	0.0539 (13)	0.0481 (13)	−0.0179 (10)	0.0070 (10)	−0.0008 (11)
C1	0.0408 (18)	0.0431 (18)	0.0423 (19)	−0.0109 (14)	−0.0013 (15)	−0.0055 (15)
C2	0.0480 (19)	0.0398 (17)	0.0452 (19)	−0.0014 (14)	0.0115 (16)	−0.0058 (15)
C3	0.0382 (17)	0.0417 (18)	0.050 (2)	0.0064 (13)	0.0050 (15)	0.0069 (15)
C4	0.0362 (17)	0.0376 (16)	0.0436 (18)	−0.0046 (13)	0.0091 (14)	0.0001 (14)
C5	0.0380 (18)	0.058 (2)	0.050 (2)	−0.0032 (15)	0.0128 (15)	−0.0105 (17)
C6	0.0320 (17)	0.059 (2)	0.055 (2)	0.0067 (15)	−0.0010 (15)	−0.0119 (18)
C7	0.0425 (18)	0.0486 (19)	0.0440 (19)	−0.0009 (14)	0.0106 (15)	0.0131 (15)
C8	0.0435 (18)	0.0498 (18)	0.0344 (17)	−0.0014 (14)	0.0074 (14)	0.0032 (15)
C9	0.0357 (16)	0.0376 (16)	0.0328 (16)	−0.0007 (13)	−0.0041 (13)	−0.0014 (14)
C10	0.0321 (16)	0.0445 (18)	0.0413 (18)	−0.0070 (13)	0.0042 (14)	0.0008 (15)
C11	0.0360 (19)	0.089 (3)	0.075 (3)	−0.0027 (18)	0.0047 (18)	0.028 (2)
C12	0.0409 (17)	0.0366 (16)	0.0378 (17)	−0.0068 (14)	0.0089 (14)	−0.0049 (14)
C13	0.0398 (16)	0.0320 (15)	0.0327 (16)	−0.0077 (12)	0.0089 (13)	0.0007 (13)
C14	0.0534 (19)	0.0307 (16)	0.0477 (19)	−0.0045 (14)	0.0009 (16)	0.0049 (14)

C15	0.053 (2)	0.0450 (19)	0.057 (2)	−0.0135 (16)	−0.0116 (17)	0.0025 (17)
C16	0.053 (2)	0.054 (2)	0.054 (2)	−0.0042 (16)	−0.0061 (17)	0.0120 (17)
C17	0.055 (2)	0.0383 (18)	0.061 (2)	0.0016 (15)	0.0029 (18)	0.0160 (16)
C18	0.0485 (19)	0.0331 (16)	0.053 (2)	−0.0068 (14)	0.0044 (16)	0.0008 (15)

Geometric parameters (Å, °)

N1—C9	1.317 (3)	C8—H8B	0.9700
N1—C8	1.456 (4)	C9—C10	1.371 (4)
N1—H1A	0.8600	C9—H9	0.9300
O1—C1	1.370 (4)	C10—C12	1.421 (4)
O1—H1	0.8200	C10—C11	1.507 (4)
O2—C12	1.259 (3)	C11—H11A	0.9600
C1—C6	1.375 (4)	C11—H11B	0.9600
C1—C2	1.376 (4)	C11—H11C	0.9600
C2—C3	1.376 (4)	C12—C13	1.497 (4)
C2—H2	0.9300	C13—C18	1.380 (4)
C3—C4	1.376 (4)	C13—C14	1.390 (4)
C3—H3	0.9300	C14—C15	1.380 (4)
C4—C5	1.393 (4)	C14—H14	0.9300
C4—C7	1.503 (4)	C15—C16	1.370 (4)
C5—C6	1.375 (4)	C15—H15	0.9300
C5—H5	0.9300	C16—C17	1.374 (4)
C6—H6	0.9300	C16—H16	0.9300
C7—C8	1.523 (4)	C17—C18	1.381 (4)
C7—H7A	0.9700	C17—H17	0.9300
C7—H7B	0.9700	C18—H18	0.9300
C8—H8A	0.9700		
C9—N1—C8	124.3 (2)	N1—C9—C10	126.1 (3)
C9—N1—H1A	117.8	N1—C9—H9	117.0
C8—N1—H1A	117.8	C10—C9—H9	117.0
C1—O1—H1	109.5	C9—C10—C12	121.0 (3)
O1—C1—C6	118.3 (3)	C9—C10—C11	119.7 (3)
O1—C1—C2	122.3 (3)	C12—C10—C11	119.1 (3)
C6—C1—C2	119.4 (3)	C10—C11—H11A	109.5
C3—C2—C1	119.6 (3)	C10—C11—H11B	109.5
C3—C2—H2	120.2	H11A—C11—H11B	109.5
C1—C2—H2	120.2	C10—C11—H11C	109.5
C2—C3—C4	122.6 (3)	H11A—C11—H11C	109.5
C2—C3—H3	118.7	H11B—C11—H11C	109.5
C4—C3—H3	118.7	O2—C12—C10	121.9 (3)
C3—C4—C5	116.6 (3)	O2—C12—C13	116.2 (3)
C3—C4—C7	122.2 (3)	C10—C12—C13	121.8 (3)
C5—C4—C7	121.2 (3)	C18—C13—C14	118.2 (3)
C6—C5—C4	121.6 (3)	C18—C13—C12	120.3 (3)
C6—C5—H5	119.2	C14—C13—C12	121.0 (3)
C4—C5—H5	119.2	C15—C14—C13	120.5 (3)
C5—C6—C1	120.2 (3)	C15—C14—H14	119.7
C5—C6—H6	119.9	C13—C14—H14	119.7

C1—C6—H6	119.9	C16—C15—C14	120.4 (3)
C4—C7—C8	112.8 (2)	C16—C15—H15	119.8
C4—C7—H7A	109.0	C14—C15—H15	119.8
C8—C7—H7A	109.0	C15—C16—C17	119.9 (3)
C4—C7—H7B	109.0	C15—C16—H16	120.1
C8—C7—H7B	109.0	C17—C16—H16	120.1
H7A—C7—H7B	107.8	C16—C17—C18	119.8 (3)
N1—C8—C7	113.4 (3)	C16—C17—H17	120.1
N1—C8—H8A	108.9	C18—C17—H17	120.1
C7—C8—H8A	108.9	C13—C18—C17	121.2 (3)
N1—C8—H8B	108.9	C13—C18—H18	119.4
C7—C8—H8B	108.9	C17—C18—H18	119.4
H8A—C8—H8B	107.7		
O1—C1—C2—C3	−178.9 (3)	C9—C10—C12—O2	159.6 (3)
C6—C1—C2—C3	2.3 (4)	C11—C10—C12—O2	−14.8 (5)
C1—C2—C3—C4	−1.2 (5)	C9—C10—C12—C13	−22.9 (4)
C2—C3—C4—C5	−0.1 (4)	C11—C10—C12—C13	162.8 (3)
C2—C3—C4—C7	−178.8 (3)	O2—C12—C13—C18	−40.9 (4)
C3—C4—C5—C6	0.2 (5)	C10—C12—C13—C18	141.4 (3)
C7—C4—C5—C6	179.0 (3)	O2—C12—C13—C14	131.0 (3)
C4—C5—C6—C1	0.9 (5)	C10—C12—C13—C14	−46.7 (4)
O1—C1—C6—C5	179.0 (3)	C18—C13—C14—C15	1.0 (5)
C2—C1—C6—C5	−2.2 (5)	C12—C13—C14—C15	−171.0 (3)
C3—C4—C7—C8	99.4 (3)	C13—C14—C15—C16	−0.6 (5)
C5—C4—C7—C8	−79.3 (4)	C14—C15—C16—C17	−0.5 (6)
C9—N1—C8—C7	97.2 (3)	C15—C16—C17—C18	1.2 (5)
C4—C7—C8—N1	−63.8 (3)	C14—C13—C18—C17	−0.3 (5)
C8—N1—C9—C10	175.8 (3)	C12—C13—C18—C17	171.8 (3)
N1—C9—C10—C12	−175.9 (3)	C16—C17—C18—C13	−0.8 (5)
N1—C9—C10—C11	−1.6 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.91	2.728 (3)	178
N1—H1A···O2^{ii}	0.86	2.20	3.014 (3)	158

Symmetry codes: (i) −*x*+2, −*y*, −*z*; (ii) −*x*+5/2, *y*−1/2, −*z*+1/2.

Fig. 1

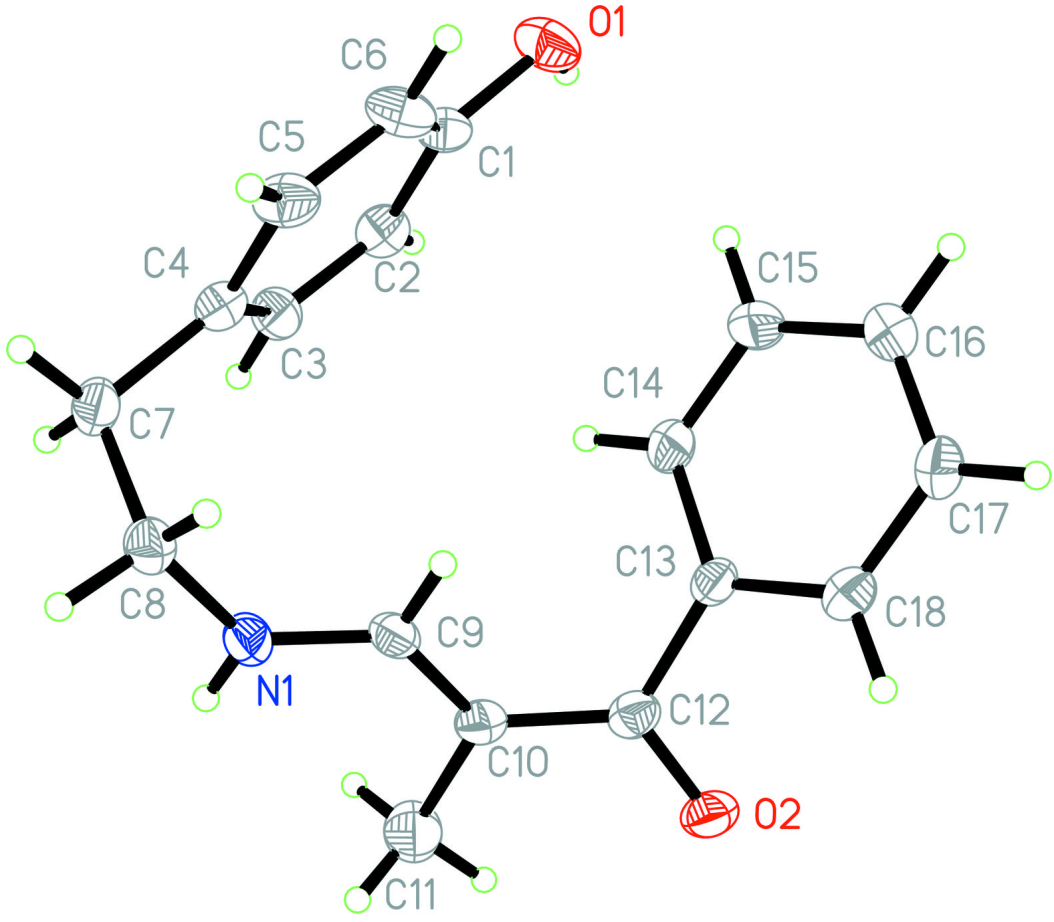
O1
C6
C5
C1
C4
C2
C3
C7
C15
C16
C14
C17
C13
C18
C8
C9
N1
C12
C10
O2
C11

Fig. 2

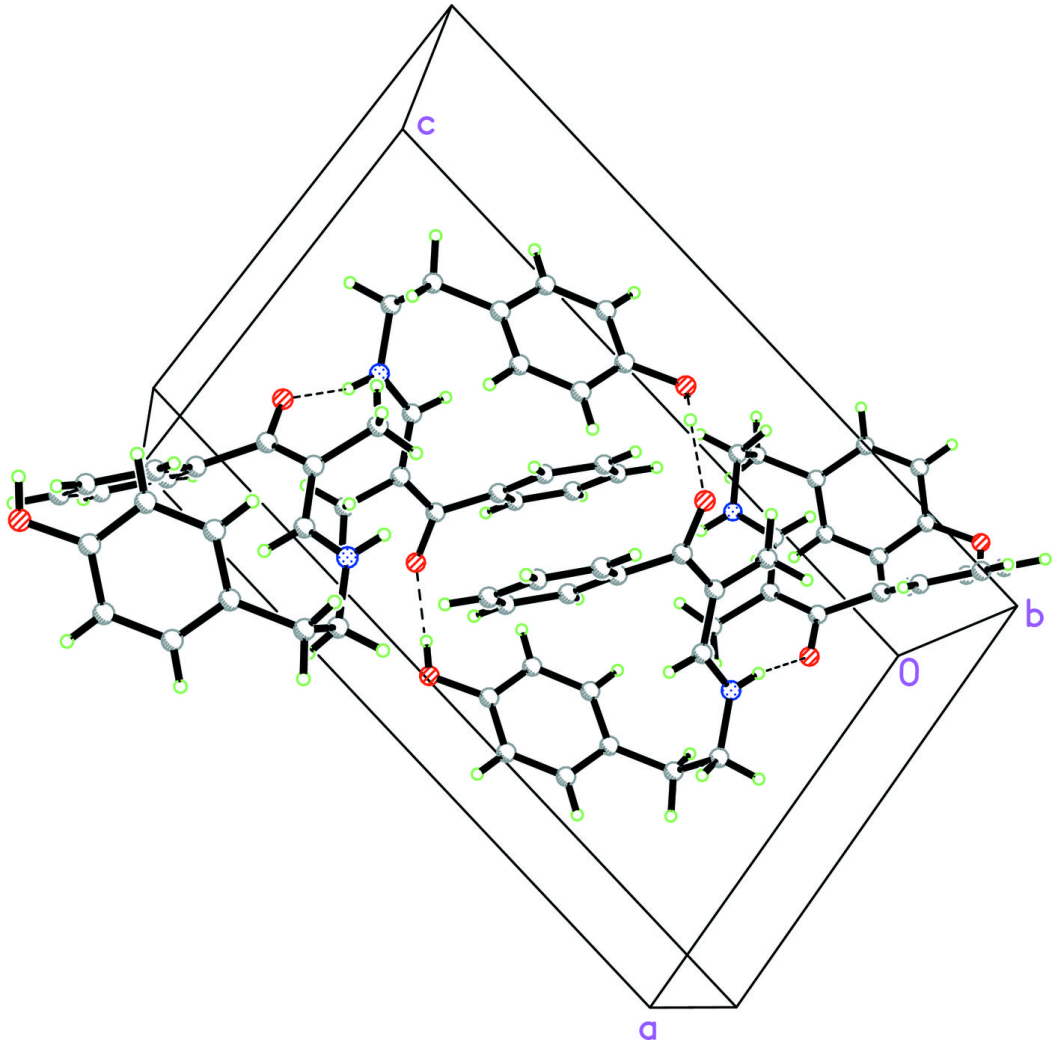

Fig. 3