

Synthesis of (*E*)-1-benzyl-4-benzylidenepyrrolidine-2,3-dione: Spectroscopic Characterization and X-ray Structure Determination

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Abstract: The compound (*E*)-1-benzyl-4-benzylidenepyrrolidine-2,3-dione was synthesized using Ethyl 1-benzyl-4,5-dioxopyrrolidine-3-carboxylate, and benzaldehyde in Ethanol/HCl medium. The isolated compound was characterized by elemental analyses, ¹H and ¹³C NMR and FTIR spectroscopy. Suitable crystal for X-ray diffraction was obtained from slow evaporation of a solution of the compound in ethyl acetate. The compound crystallizes in the centrosymmetric space group P-1 of the triclinic system with the following unit cell parameters $a = 6.4367(4) \text{ \AA}$, $b = 7.4998(5) \text{ \AA}$, $c = 15.3455(5) \text{ \AA}$, $\alpha = 86.448(4)^\circ$, $\beta = 78.732(4)^\circ$, $\gamma = 83.943(5)^\circ$, $V = 721.80(7) \text{ \AA}^3$, $Z = 2$, $R_1 = 0.049$ and $wR_2 = 0.135$. In the title molecule, C₁₈H₁₅NO₂, the pyrrolidine-2,3-dione ring is almost planar (r.m.s. deviation = 0.0081 Å). The pyrrolidine ring has an envelope conformation with a methylene carbon as the flap. The two phenyl rings are severely twisted with dihedral angle of 72.234(5)°. The pyrrolidine ring is quite coplanar with the C13-C18 phenyl ring and severely twisted from the C1-C6 phenyl ring with dihedral angles of 0.762(5)° and 72.750(5)°. The molecule adopts an *E* configuration with respect to the ethylenic moiety. Unclassical C—H...O hydrogen bonding link the molecules, forming layers in the *bc* plane.

Keywords: Pyrrolidine, Benzaldehyde, FTIR, NMR, Crystal, X-ray Diffraction

1. Introduction

Five- or six-membered heterocycles containing the nitrogen atom are scaffolds very often present in compounds for therapeutic purposes [1–4]. Five-membered heterocycles containing one or more nitrogen atoms are the most prevalent in bioactive molecules [5]. The synthesis of this type of molecule remains a challenge given their strong economic potential for the pharmaceutical industry [6–9]. The method of multicomponent synthesis which uses a sequence of simple reactions of short duration is quite widespread [5, 10–12]. Mannich reactions are widely used in the synthesis of heterocycles containing functionalized pyrrolidine rings with important biological properties [13–16]. Compounds with antimicrobial [17, 18], anticancer [19–21], or antiviral [22, 23] properties are reported. Given the great importance of compounds containing a pyrrolidine ring in the synthesis of drugs, it is useful to

precisely know the information on the structural conformation to better understand the reactivity of these molecules. X-ray analysis achieves these goals. The present work presents the synthesis of the compound (*E*)-1-benzyl-4-benzylidenepyrrolidine-2,3-dione and the determination of its structure by spectroscopic analysis and by X-ray diffraction.

2. Experimental

2.1. Starting Materials and Instrumentations

Ethyl 1-benzyl-4,5-dioxopyrrolidine-3-carboxylate and benzaldehyde were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on

an FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000–400 cm^{-1} region. The ^1H and ^{13}C NMR spectra were recorded in CDCl_3 on a BRUKER 300.13 MHz spectrometer at room temperature using TMS as an internal reference.

2.2. Synthesis of (E)-1-benzyl-4-benzylidenepyrrolidine-2,3-dione

Ethyl 1-benzyl-4,5-dioxopyrrolidine-3-carboxylate (783 mg, 3 mmol) and benzaldehyde (319 mg, 3 mmol) were solubilized in a mixture of ethanol (6 mL) and HCl_{aq} (20%, 15 mL) then heated under reflux for 4 hours. After cooling to room temperature, the aqueous phase was removed. The solid obtained is collected and then recrystallized with ethyl acetate (AcOEt). Yellow crystals suitable for X-ray analyses were collected after one week. M.p. 189.1°C. Yield 32%. Analysis calculated for $[\text{C}_{18}\text{H}_{15}\text{NO}_2]$ C, 77.96; H, 5.45; N, 5.05. Found: C, 77.94; H, 5.43; N, 5.03. IR (cm^{-1}): 3028, 1720, 1699, 1622, 1574, 1494, 1480, 754, 700, 683, 634, 596, 547, 517, 471. CCM (DCM/AcOEt 95/5) $R_f = 0.34$ (UV, *p*-anisaldéhyde). NMR- ^1H (400 MHz, CDCl_3 , δ (ppm)): 7.70 (s, 1H), 7.48–7.32 (m, 10H), 4.81 (s, 2H), 4.42 (d, $J = 1.9$ Hz, 2H). RMN- ^{13}C

(101 MHz, CDCl_3) δ (ppm) 186.56 (C=O), 160.67 (C=O), 138.31 (CH), 134.78 (C), 133.51 (C), 131.64 (CH), 131.38 (2CH), 129.50 (2CH), 129.26 (2CH), 128.63 (2CH), 128.54 (CH), 124.85 (C), 48.24 (CH_2), 46.54 (CH_2).

2.3. Crystal Structure Determination

Crystals suitable for single-crystal X-ray diffraction, of the reported compound, were grown by slow evaporation of its ethyl acetate solution. Details of the crystal structure solution and refinement are given in Table 1. Diffraction data were collected using a Bruker APEX-II CCD diffractometer with graphite monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 1.54178 \text{ \AA}$). All data were corrected for Lorentz and polarization effects. The structure was solved and refined using the Bruker SHELXTL Software Package [24]. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms [25]. The hydrogen atoms (CH and CH_2 groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP [26].

Table 1. Crystallographic data and refinement parameters for compound.

Chemical formula	$\text{C}_{18}\text{H}_{15}\text{NO}_2$
Mr	277.31
Crystal system	Triclinic
Space group	P-1
Temperature (K)	295
<i>a</i> (Å)	6.4367 (4)
<i>b</i> (Å)	7.4998 (5)
<i>c</i> (Å)	15.3455 (5)
α (°)	86.448 (4)
β (°)	78.732 (4)
γ (°)	83.943 (5)
<i>V</i> (Å ³)	721.80 (7)
<i>Z</i>	2
ρ_{calc} (g/cm ³)	1.276
μ (mm ⁻¹)	0.67
<i>F</i> (000)	292
Crystal size (mm ³)	0.38 × 0.12 × 0.05
$\text{CuK}\alpha$ (Å)	1.54178
Θ_{min} , Θ_{max} (°)	2.9140, 72.3430
<i>h k l</i> ranges	-7 ≤ <i>h</i> ≤ 7; -9 ≤ <i>k</i> ≤ 8; -18 ≤ <i>l</i> ≤ 18
<i>T</i> _{min} , <i>T</i> _{max}	0.630, 1.000
No. of measured	12572
Independent reflections	2806
Observed [<i>I</i> > 2σ (<i>I</i>)] reflections	2446
<i>R</i> _{int}	0.032
$R[F^2 > 2\sigma(F^2)]$	0.049
<i>wR</i> (F^2)	0.135
Final <i>R</i> indexes [all data]	0.054, 0.129
Goodness-of-fit (GOF)	1.03
No. of reflections	2806
Parameters / restraints	190 / 36
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.17, -0.23

3. Result and Discussion

3.1. General Study

The compound obtained after the reaction between Ethyl

1-benzyl-4,5-dioxopyrrolidine-3-carboxylate, and benzaldehyde (Figure 1) is characterized by physico-chemical and spectroscopic analyses. Elemental analysis of the compound confirms the formation of the compound with the chemical formula $\text{C}_{18}\text{H}_{15}\text{NO}_2$. The FTIR spectrum of the compound reveals stretching vibration bands of the carbonyl

groups pointed at 1720 and 1699 cm^{-1} [27]. The band due to the ethylenic moiety is pointed at 1622 cm^{-1} . The aromatic $\text{C}_{\text{Ar}}=\text{C}_{\text{Ar}}$ stretching vibration bands are pointed at 1480, 1494 and 1574 cm^{-1} . Bands at 3028 cm^{-1} is indicative of the presence of $\text{Csp}^2\text{—H}$. Additional strong bands due to the $\text{C}_{\text{Ar}}\text{—H}$ pointed at 754 and 700 cm^{-1} are indicative of the presence of mono substituted benzene ring. The ^1H NMR spectrum (Figure 2) presents signal in the range 7.48-7.32 ppm which are attributed to the protons of the two monosubstituted benzene rings. The signal pointed at 7.70 ppm is attributed to the H—C=C proton. The signal pointed at 4.81 ppm as singlet is due to the methylene of the benzyl group. The signal at 4.42 ppm, which appears as a doublet with coupling constant J of 1.9 Hz, is assigned to the methylene group of the pyrrolidine ring. The ^{13}C NMR spectra (Figures 3 and 4) of compound exhibits weak-field signal of carbonyl atom at 186.56 ppm attributed to the strongly conjugated carbonyl carbon atom. The second carbonyl carbon atom is pointed at 160.67 ppm. The DEPT 135 spectroscopic (Figure 4) data makes it possible to attribute the signal of signal of the C_{ipso} of the phenyl groups [133.51 and 124.85 ppm]. The signals due to the carbon atoms of the methylene moieties are pointed at 48.24 ppm [pyrrolidine] and 46.54 ppm [benzyl]. The signals pointed in the range [131.64-128.54 ppm] are attributed to the unsubstituted carbon atoms [$\text{C}_{\text{Ar}}\text{—H}$] of the phenyl rings. The signals at

138.31 ppm and 134.78 ppm are, respectively, assigned to the trisubstituted and tetrasubstituted ethylenic carbon atoms.

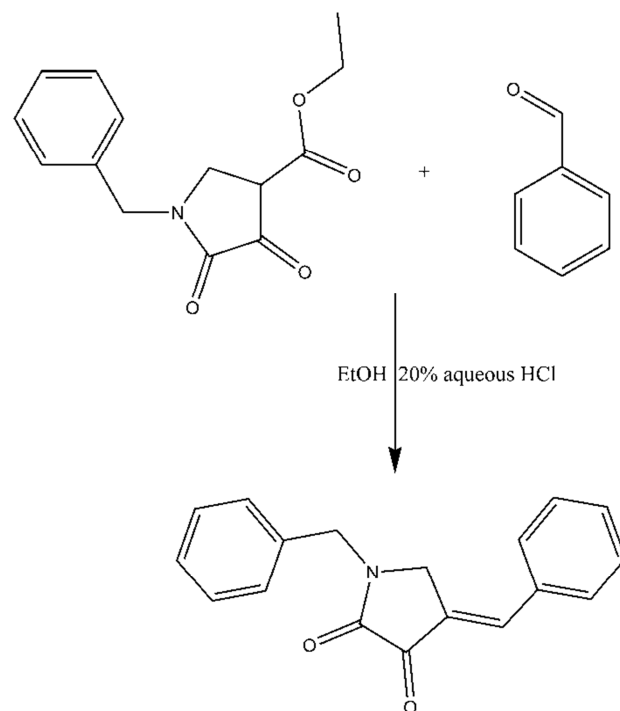


Figure 1. Synthetic scheme for the compound.

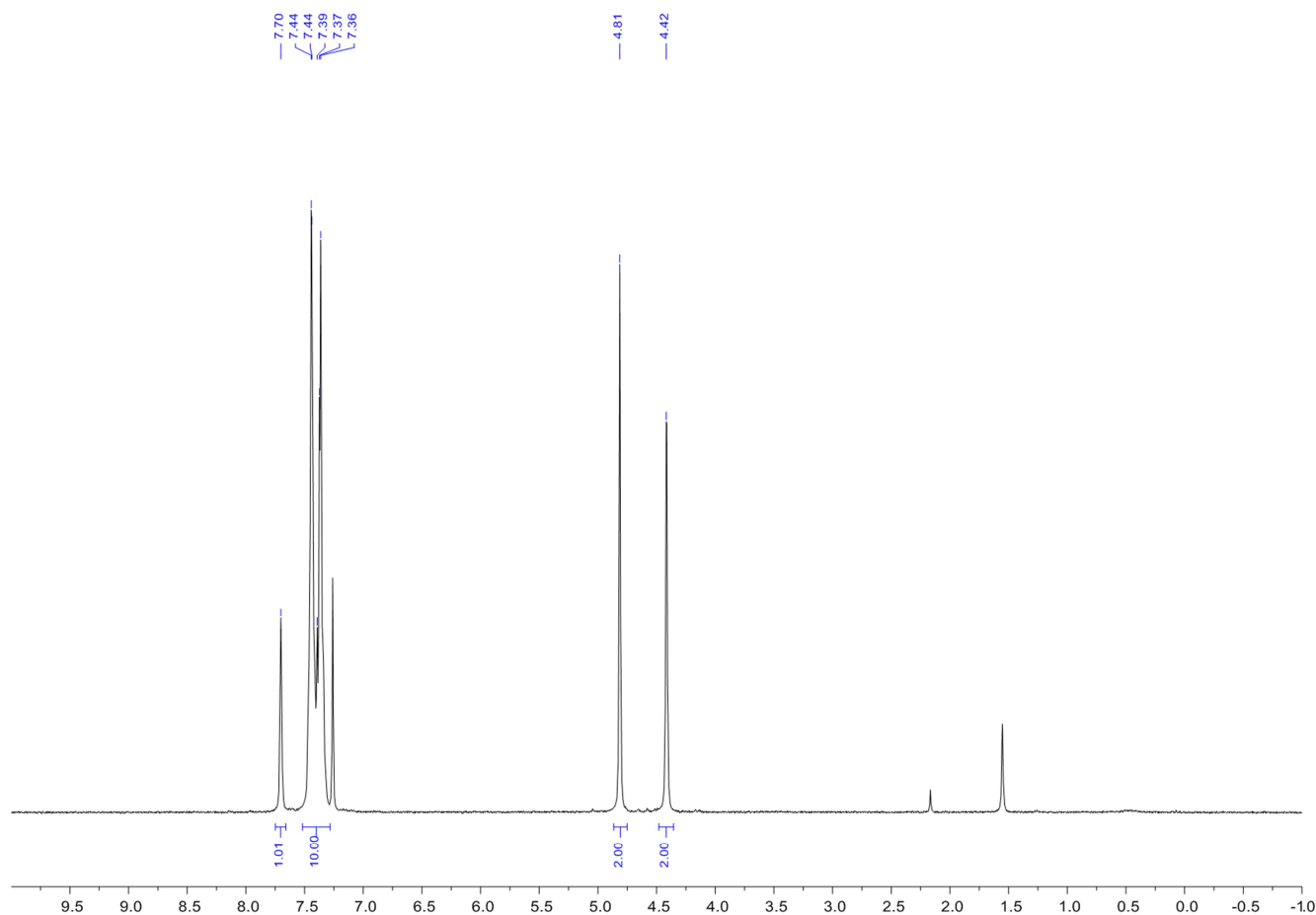


Figure 2. ^1H NMR of the compound.

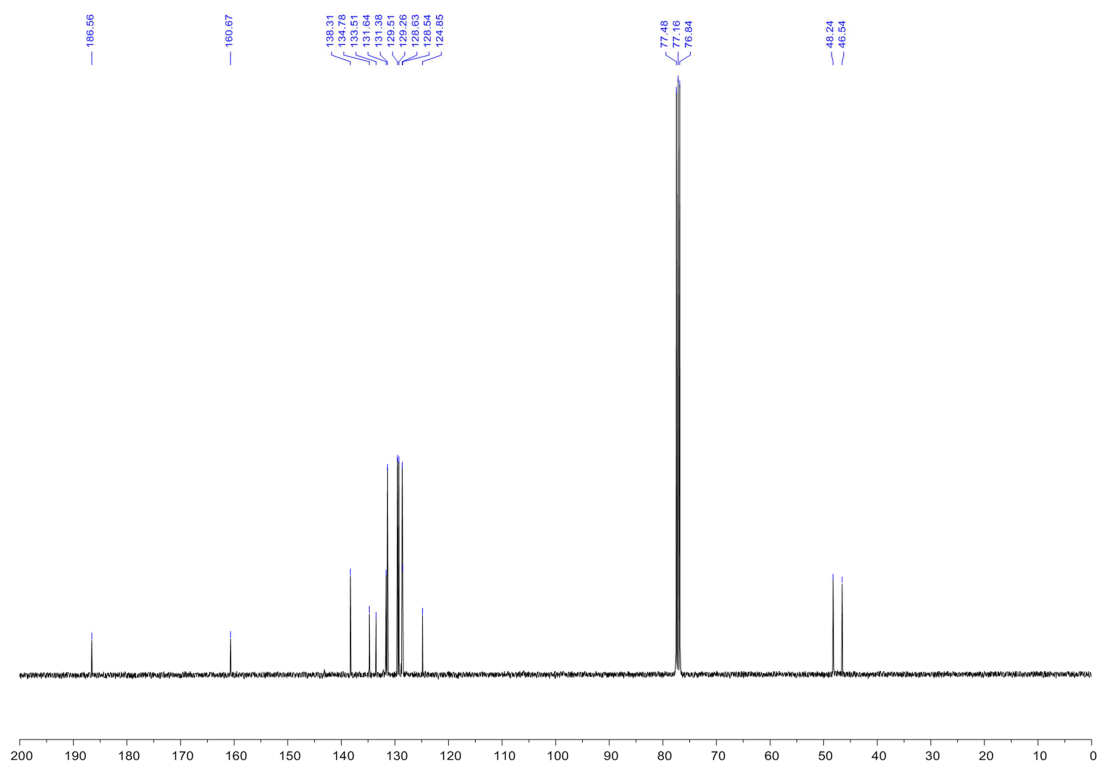


Figure 3. ^{13}C NMR of the compound.

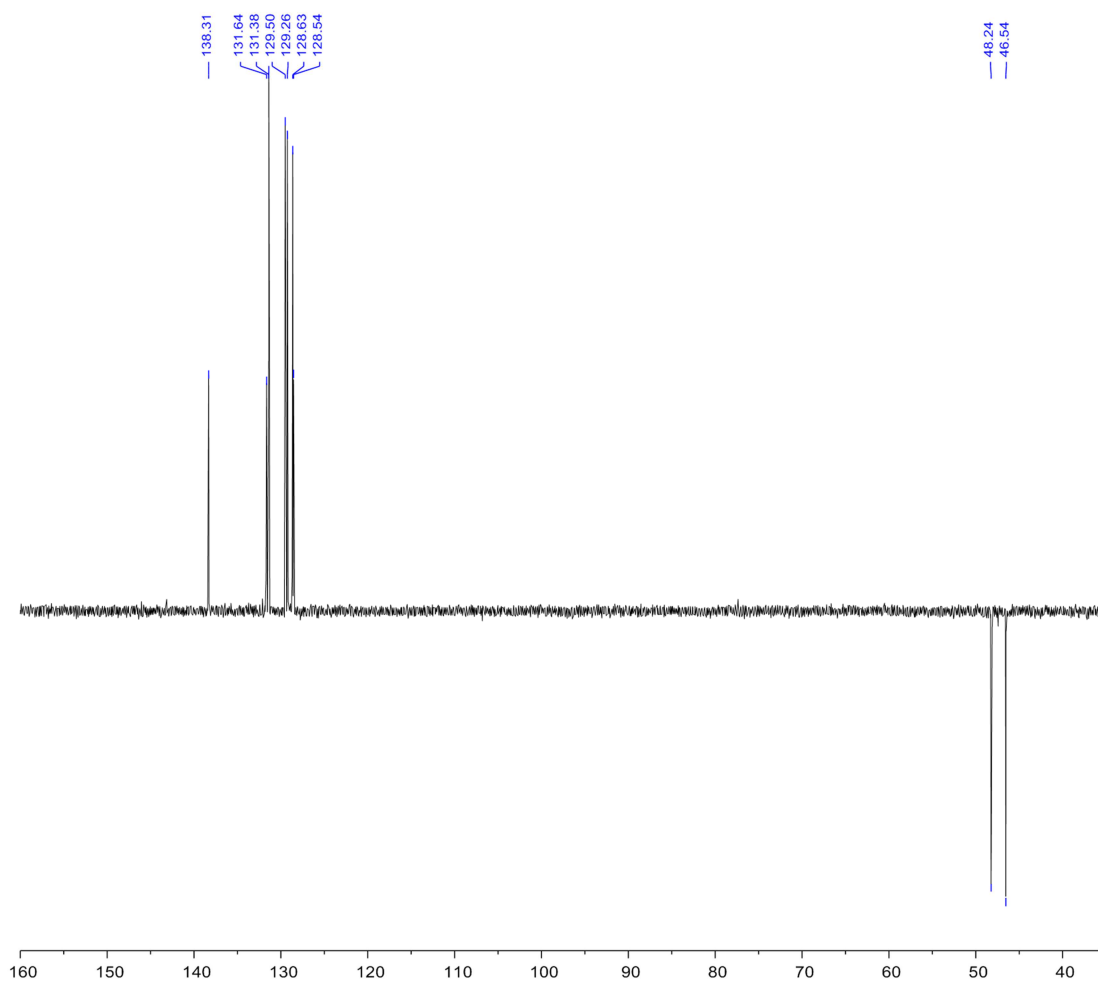


Figure 4. DEPT 135 NMR spectrum of the compound.

3.2. Crystal Structure

X-ray diffraction analysis of the compound was performed on suitable crystals obtained by slow crystallization from its ethyl acetate solution. The compound crystallizes in the centrosymmetric space group P-1 of the triclinic system. The crystallographic details are summarized in Table 1. The ORTEP diagram is shown in Figure 5. The selected bond lengths and angles are listed in Table 2. The molecule consists of a phenyl ring and a benzyl moiety connected through 4-methylenepyrrolidine-2,3-dione unit. The phenyl ring is linked to the not totally substituted methylenecarbon, while the benzyl moiety is linked to the nitrogen atom of the pyrrolidine ring through its methylene carbon atom. The molecule adopts an *E* configuration with respect to the C10=C12 which has double bond-character with bond length of 1.341 (2) Å [28]. The C8=O1 [1.2216 (19) Å] and C9=O2 [1.2147 (18) Å] bond lengths are consistent with double bond character [29]. The pyrrolidine-2,3-dione [N1/C8-C11] is planar with rms of 0.0081 Å. The pyrrolidine ring and the C13-C18 benzene ring are quite co-planar with dihedral angle of 0.762 (5)°. The mean plane of C1-C6 ring is severely twisted from the mean planes of the C13-C18 benzene ring and the pyrrolidine ring with dihedral angles of 72.234 (5)° and 72.750 (5)°, respectively.

In the pyrrolidine ring there are two different types of hybridized atoms. The angles whose vertices are C8, C9 and C10 are in the range 106.02 (12)–131.13 (12), slightly different from the ideal angle value of 120 for sp^2 -hybridized atoms. The angles whose vertex is C11 are close to the ideal angle of 109 for sp^3 -hybridized atom. The 1-benzylpyrrolidine ring has an envelope conformation with the methylene carbon atom C11 as the flap. The sum of the bond angles around N1 atom is 360.00° indicating a pyramidal geometry and sp^3 hybridization [29]. The bond lengths involving the N1 atom are respectively, 1.3458 (18) Å, 1.4562 (17) Å, 1.4596 (19) Å indicating single bond character [30]. In the crystal, unclassical C—H...O [C11—H11B...O2ⁱ; i = x+1, y, z] interactions link the molecules, forming layers in the *bc* plane (Table 3, Figure 6).

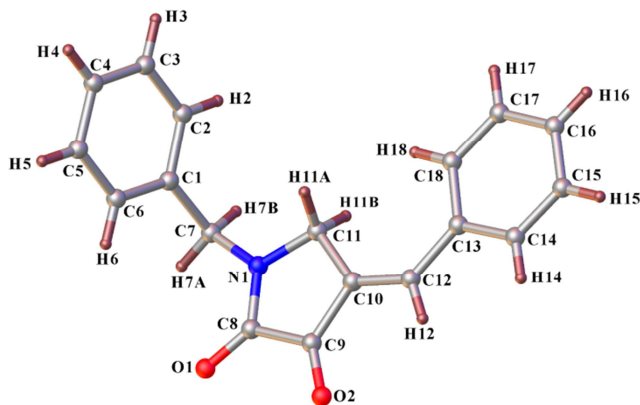


Figure 5. ORTEP plot (30% probability ellipsoids) showing the structure of the compound.

Table 2. Selected geometric parameters (Å, °).

N1—C11	1.4562 (17)	O1—C8	1.2216 (19)
N1—C7	1.4596 (19)	O2—C9	1.2147 (18)
N1—C8	1.3458 (18)	C10—C12	1.341 (2)
C11—N1—C7	120.90 (11)	N1—C8—C9	106.02 (12)
C8—N1—C11	114.97 (12)	O1—C8—N1	127.34 (17)
C8—N1—C7	124.13 (13)	O1—C8—C9	126.64 (15)
C12—C10—C11	131.13 (12)	O2—C9—C10	128.56 (17)
C12—C10—C9	121.55 (13)	O2—C9—C8	123.92 (16)
C9—C10—C11	107.32 (12)	C10—C9—C8	107.52 (12)

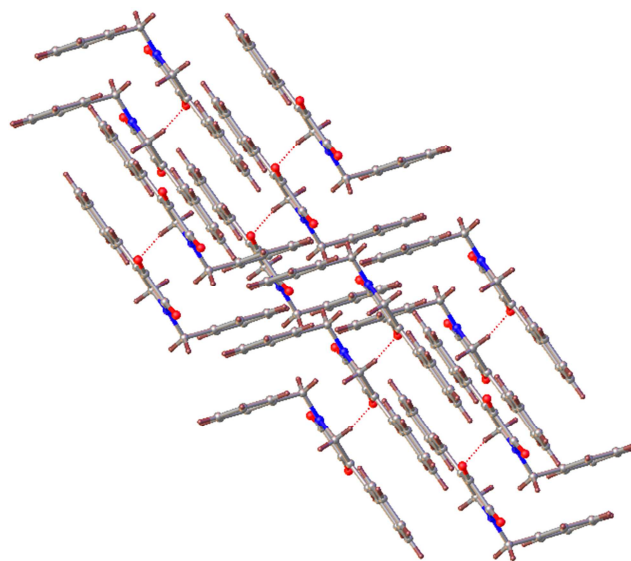


Figure 6. Layer by hydrogen bonding.

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

D—H...A	D—H	H...A	D...A	D—H...A
C11—H11B...O2 ⁱ	0.97	2.38	3.3062 (17)	159.2

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

4. Conclusion

In summary, a protocol for the preparation of (*E*)-1-benzyl-4-benzylidenepyrrolidine-2,3-dione was optimized in this work. The structure of the compound was confirmed by elemental analysis and spectroscopic techniques (FT-IR, ¹H and ¹³C NMR). Its molecular structure was determined by X-ray diffraction technique. The X-ray structure reveals that the pyrrolidine ring is planar and is quite planar with the C13-C18 benzene ring. The mean plane of C1-C6 ring is severely twisted from the mean planes of the C13-C18 benzene ring and the pyrrolidine ring.

Supplementary Materials

CCDC-2259118 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or

<http://www.ccdc.cam.ac.uk>).

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