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ORIGINAL PAPER

Spectroscopic Studies and Crystal Structure of 1-(Morpholino (Phenyl) Methyl) Pyrrolidine-2,5-Dione

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Abstract A new mannich base 1-(morpholino(phenyl) methyl)pyrrolidine-2,5-dione formed by the direct condensation of morpholine, succinimide and benzaldehyde has been synthesized. The structure of this mannich base has been elucidated on the basis of micro elemental analysis, IR, ^{1}H NMR, ^{13}C NMR, UV–Visible Techniques and Mass. The crystal structure of the title compound C_{15} H_{18} N_2 O_3 was determined. It crystallizes in the triclinic system, space group $P-1$, with $a = 8.8122(13)$ Å, $b = 9.2794(14)$ Å, $c = 9.814(3)$ Å, $\alpha = 107.154(9)°$, $\beta = 97.936(9)°$, $\gamma = 110.197(7)°$, $V = 693.4(2)$ Å^3, $D_x = 1.314$ Mg/m^3. The structure was solved by the full-matrix least squares on F^2 and had a refined R value of 0.0486 for 8567 observed reflections. The six membered heterocyclic ring of the morpholino moiety adopts a chair conformation. The crystal structure is stabilized by weak intermolecular C–H···O interactions that link the molecules into inversion dimers.

Keywords Mannich base · Spectroscopy · Crystal structure · Triclinic 1-(morpholino(phenyl)methyl) pyrrolidine-2,5-dione

Introduction

Morpholine is a multipurpose chemical which is used as a solvent for resins, dyes and waxes. One of its most important use is as a chemical intermediate in the preparation of pesticides [1]. Morpholine has many important applications. It is used as an intermediate in the manufacture of rubber chemicals and optical brighteners. It is also used extensively as a corrosion inhibitor in steam boiler systems. Fatty acid derivatives of morpholine are used as emulsifiers in the manufacture of waxes and polishes.

Drugs containing a morpholine ring have established activities that include the reduction of blood sugar and lipid levels [2] and amelioration of obesity and insulin resistance [3]. The physiological activity of the morpholine nucleus is attested by the number of pharmaceutical applications which have been found for it. The hydroperiodide is suitable for incorporation in ointments for the treatment of skin disorders, such as athlete's foot. A number of morpholine derivatives have been described as analgesics and local anesthetics. Several morpholine-derived chemicals are useful as respiratory and vasomotor stimulants. Other pharmaceutical fields in which morpholine has found application include choleretics, antispasmodics, analeptics, and antimalarials. In addition, the use of morpholine as a peptizing agent for preparing aqueous dispersions of phenothiazines for anthelmintic purposes has been claimed.

Owing to their pharmacological activities these compounds have received a great deal of attention in respect of their synthesis and conformation. A number of morpholine derivatives have been shown to possess bactericidal activity. For example, morpholinium salts of certain acylated sulfonamides possess strong bacteriostatic or bactericidal properties, and morpholine hydroperiodide has been used as a water disinfectant. The reaction of morpholine with 3,4,5-trichloro-2,6-pyridinedicarbonitrile yields a product which is useful in the control of fungi. Morpholine is used in preparing compounds that are excellent herbicides and that can be applied either to the soil before the

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weeds emerge or to the growing plants. The morpholinomethyl derivative of pyrizinamide (morphozinamide) has been found to be more effective in the treatment of tuberculosis than pyrizinamide [4].

Succinimide is a diketopyrrolidine that is prepared by heating succinic anhydride with aqueous ammonia, followed by rapid distillation of ammonium succinate [5, 6]. Succinimide and other cyclic carboximides are used as starting materials in organic synthesis. Their nitranions are also important intermediates in many chemical reactions [5–8]. Derivatives of succinimide are of important biological and pharmacological interest. In addition, succinimide itself is a hypooxaluric agent [9]. Succinimide oxidises in various substrates in different media [10–15]. Extensive work has been reported on the kinetics of oxidation of amino acids and peptides with various metal ions and several other oxidants [15–17]. The comparative studies on the kinetics of oxidation of amino acids and tetrapeptides by *N*-bromosuccinimide have been reported [18]. The crystal structures of several succinimide derivatives have been reported with respect to their biological properties, e.g. antiepileptic and anticonvulsive [19–22], fungicidal [19] and other pharmacological [23] activities. In each of these compounds, the ring nitrogen is substituted either by methyl group, or differently substituted phenyl and pyridine rings or morpholinomethyl moiety [24]. The succinimide derivatives are useful as anti-anxiety drugs.

A group of phenylsuccinimides [25, 26] proved to have strong anticonvulsive activity. A search through the literature firmly revealed that no work has been carried out on the synthesis of Mannich bases employing succinimide, morpholine and benzaldehyde as the reactants for condensation. Therefore it was thought worthwhile to synthesize this condensation product through Mannich condensation reaction and investigate its molecular structure. Mannich reaction is a three component condensation reaction consisting of a compound with active hydrogen, an aldehyde (generally formaldehyde) and a secondary amine [27, 28]. In continuation with the earlier reported work on the synthesis of 1-[(2,5-dioxopyrrolidin-1-yl)(phenyl)methyl] urea using urea, succinimide and benzaldehyde [29], we herein report the synthesis, spectroscopic studies and crystal structure of the title compound (Fig. 1).

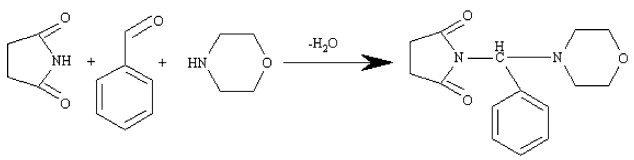

Fig. 1 The Mannich reaction scheme

Experimental

Reagents and Techniques

All the reagents used for synthesizing the title compound were of A.R Grade and the solvents used were commercial products of the highest available purity. The micro elemental analysis was carried out with a Carlo Erba 1108 Elemental Analyzer. Infrared spectrum was recorded in KBr medium on a Spectrum-One Perkin Elmer FTIR instrument. ^{1}H NMR spectrum was recorded in DMSO-d_6 and ^{13}C NMR spectrum was recorded in $CDCl_3$ using a Bruker 300 MHz instrument. UV–Visible spectrum was recorded in DMF on a EZ301 Perkin Elmer spectrophotometer. FAB Mass spectrum was recorded on a JEOL SX 102 Mass Spectrometer.

Synthesis

Succinimide (19.8 g, 0.2 M), morpholine (18 mL, 0.2 M) and benzaldehyde (22 mL, 0.2 M) were taken in equimolar ratio. Morpholine was added slowly to succinimide taken in a 250 mL beaker. Sufficient amount of ethanol was added to make the contents a homogeneous solution. Benzaldehyde was added slowly in drops with continuos stirring of the solution. A yellowish white powdery substance was formed immediately. After 10 days the product was washed several times with distilled water. The product was dried in the air oven at 60 °C and recrystallised from ethanol by slow evaporation. Colorless crystals suitable for X-ray diffraction study were obtained m.p. 100–105 °C, yield 85.4%. Found: C, 63.68; H, 6.52; N, 10.43. Calc. For $C_{15}H_{18}N_2O_3$; C, 65.69; H, 6.57; N, 10.22%.

Crystallography

A crystal of dimensions 0.40 × 0.28 × 0.22 mm was used for collection of intensity data on a "Bruker APEXII CCD" area detector diffractometer with graphite monochromated MoKα radiation (50 kV, 30 mA) using the APEX2 [30] data collection software. The collection method involved phi and ω-scans of width 0.5° with an exposure time of 10 s/frame. Data reduction was carried out using the program SAINT+ [31]. The crystal structure was solved by direct methods using SHELXTL [32]. Non-hydrogen atoms were first refined isotropically followed by anisotropic refinement by full matrix least-squares calculations based on F^2 using SHELXTL. Hydrogen atoms were first located in the difference map then positioned geometrically and allowed to ride on their respective parent atoms. Hydrogen atoms involved in hydrogen-bonding were located in the difference map and refined freely. Diagrams and publication material were generated using

Table 1 The crystal structure data for 1-(morpholino(phenyl) methyl)pyrrolidine-2,5-dione

CCDC no.	705027
Empirical formula	C15 H18 N2 O3
Formula weight	274.31
Temperature	298(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, $P-1$
Unit cell dimensions	$a = 8.8122(13)$ A $\alpha = 107.154(9)°$
	$b = 9.2794(14)$ A $\beta = 97.936(9)°$
	$c = 9.814(3)$ A $\gamma = 110.197(7)°$
Volume	693.4(2) A^3
Z, Calculated density	2, 1.314 Mg/m^3
Absorption coefficient	0.092 mm^{-1}
$F(000)$	292
Crystal size	0.40 × 0.28 × 0.22 mm
Theta range for data collection	2.25 to 31.87°
Limiting indices	$-7 \le h \le 12$, $-13 \le k \le 11$, $-12 \le l \le 14$
Reflections collected/unique	8,567/3,384 [R(int) = 0.0238]
Max. and min. transmission	0.9800 and 0.9640
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3,384 / 0 / 182
Goodness-of-fit on F^2	0.956
Final R indices [$I > 2\sigma(I)$] $R_1 = 0.0483$, $wR_2 = 0.1493$	
R indices (all data)	$R_1 = 0.0889$, $wR_1 = 0.1869$
Extinction coefficient	0.008(9)
Largest diff. peak and hole	0.223 and −0.198 $e.A^{-3}$

SHELXTL, PLATON [33] and ORTEP-3 [34]. Details of the data collection are given in Table 1. The atomic coordinates are given in Table 2. Selected bond distances and bond angles are listed in Table 3. Table 4 lists the hydrogen bonds. The chemical diagram of the title compound is given in Fig. 2. The molecular structure with the atom numbering scheme is shown in Fig. 3. Crystallographic data for the structure reported in this paper has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 705027.

Results and Discussion

IR, ^{1}H NMR, ^{13}C NMR, UV–Visible and Mass

The title compound shows a number of IR absorption bands and the absorption frequencies are in a slightly shifted position in comparison with those of the reactants succinimide and morpholine. This favorably indicates the substitution on succinimide and morpholine and the formation of a new compound. The aromatic ν_{C-H} stretching vibration appears at 3,091 cm^{-1}. The asymmetric and symmetric stretching vibration of ν_{CH_2} occurs at 2,945 and 2,852 cm^{-1} and the corresponding bending vibration occurs at 1,421 cm^{-1} .The $\nu_{C=O}$ stretching vibration of the amide occurs at 1,704 cm^{-1}. The ν_{C-N-C} and ν_{C-O-C} stretching vibration appears at 1,175 and 1,037 cm^{-1}. The aromatic monosubstituted vibration occurs at 699 cm^{-1}.

The ^{1}H NMR data for the title compound in DMSO-d_6 reveal the aromatic protons as multiplets at δ 7.35 ppm. The O–CH_2 and N–CH_2 protons of morpholine appear as a multiplet at δ 3.69 and δ 2.66 ppm respectively. The signal for the CH_2 protons of succinimide appear at δ 2.76 ppm while the signal for the methine CH proton appears at δ 5.88 ppm. The signals of the CH_2 protons of succinimide are shifted downfield on substitution.

The ^{13}C NMR data for the title compound in $CDCl_3$ shows that the signals for the aromatic carbon atoms appear as a multiplet at δ 138 ppm. The signal for the C=O carbon of succinimide appears at δ 178, 177 ppm. The doublet and triplet signals observed at δ 50 and δ 67 ppm is due to the N–CH_2 and O–CH_2 carbons of morpholine. The doublet signal for the CH_2 carbon of succinimide appears at δ 29 ppm while the signal for the CH carbon occurs at δ 89 ppm respectively.

Saturated compounds containing heteroatoms such as nitrogen, oxygen etc, have non-bonding electrons in addition

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($A^2 \times 10^3$) for new. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	*y*	*z*	U(eq)
C(1)	6,340(2)	1,496(2)	6,582(2)	35(1)
C(2)	4,901(2)	1,460(2)	7,241(2)	45(1)
C(3)	4,973(3)	561(3)	8,282(3)	58(1)
C(4)	6,573(2)	316(2)	8,327(2)	42(1)
C(5)	8,839(2)	786(2)	6,920(2)	31(1)
C(6)	10,659(2)	2,347(2)	9,492(2)	44(1)
C(7)	12,449(3)	3,563(3)	10,302(2)	58(1)
C(8)	12,496(3)	4,860(2)	8,602(3)	63(1)
C(9)	10,706(2)	3,684(2)	7,750(2)	46(1)
C(10)	8,808(2)	−934(2)	6,648(2)	33(1)
C(11)	7,335(2)	−2,327(2)	5,863(2)	44(1)
C(12)	7,334(2)	−3,887(2)	5,528(2)	51(1)
C(13)	8,766(2)	−4,083(2)	5,965(2)	47(1)
C(14)	10,230(2)	−2,709(2)	6,724(2)	52(1)
C(15)	10,250(2)	−1,145(2)	7,056(2)	44(1)
N(1)	7,290(2)	880(2)	7,300(2)	34(1)
N(2)	10,385(2)	2,099(2)	7,921(1)	34(1)
O(1)	6,649(2)	1,955(2)	5,579(1)	46(1)
O(2)	7,149(2)	−279(2)	9,087(2)	61(1)
O(3)	12,783(2)	5,105(2)	10,123(2)	70(1)

to σ electrons. The UV spectrum of the title compound in DMF solution registers intense split bands centered at 280 nm and 264 nm which is presumably due to $n \rightarrow \pi^*$ transition of the carbonyl group and $\pi \rightarrow \pi^*$ transition of the carbonyl and the aromatic ring.

The FAB Mass spectrum shows a weak molecular ion peak at m/z 274 which confirms the assigned molecular mass for the title compound. Thereupon on fragmentation it records intense signals at m/z 197, 187, 99, 90, 76 which are due to $C_9H_{13}N_2O_3^+$, $C_{11}H_{10}NO_2^+$, $C_5H_9NO^+$, $C_7H_6^+$, $C_6H_5^+$, respectively.

Crystallographic Study

The title compound C_{15} H_{18} N_2 O_3 crystallizes in the triclinic system with space grouping P-1. The title compound contains three ring systems, the succinimido ring, the phenyl ring and the morpholino ring. The phenyl ring is linked by the succinimido and the morpholino ring. The C–N bond distances [C(1)–N(1) = 1.390; C(4)–N(1) = 1.395] in the succinimido ring is considerably shorter than the C–N single bond value of 1.47 Å as suggested by Pauling [35]. The C2–C3 distances are 1.505 Å. The C–C distances [C(1)–C(2) = 1.496; C(3)–C(4) = 1.500] between the carbon atoms adjacent to the carbonyl groups are found to be shorter than the normal values. The value of C=O bond distances [C(1)–O(1) = 1.210; C(4)–

Table 3 Selected bond lengths [A] and angles [°]

C(1)–O(1)	1.210(2)
C(1)–N(1)	1.390(2)
C(1)–C(2)	1.496(2)
C(2)–C(3)	1.505(3)
C(3)–C(4)	1.500(3)
C(4)–O(2)	1.204(2)
C(4)–N(1)	1.395(2)
C(5)–N(2)	1.4439(19)
C(5)–N(1)	1.4875(19)
C(5)–C(10)	1.528(2)
C(5)–H(5)	0.9800
C(6)–N(2)	1.462(2)
C(6)–C(7)	1.513(3)
C(7)–O(3)	1.427(3)
C(8)–O(3)	1.414(3)
C(8)–C(9)	1.513(3)
C(9)–N(2)	1.464(2)
C(10)–C(15)	1.375(2)
C(10)–C(11)	1.389(2)
C(11)–C(12)	1.386(3)
C(11)–H(11)	0.9300
C(12)–C(13)	1.366(3)
C(12)–H(12)	0.9300
C(13)–C(14)	1.373(3)
C(13)–H(13)	0.9300
C(14)–C(15)	1.384(2)
C(14)–H(14)	0.9300
C(15)–H(15)	0.9300
O(1)–C(1)–N(1)	124.19(15)
O(1)–C(1)–C(2)	127.20(16)
N(1)–C(1)–C(2)	108.61(15)
C(4)–C(3)–C(2)	105.84(15)
O(2)–C(4)–N(1)	125.04(17)
O(2)–C(4)–C(3)	127.13(17)
N(1)–C(4)–C(3)	107.82(15)
N(2)–C(5)–N(1)	114.70(13)
N(2)–C(5)–C(10)	113.23(13)
N(1)–C(5)–C(10)	111.10(12)
N(2)–C(5)–H(5)	105.6
N(1)–C(5)–H(5)	105.6
C(10)–C(5)–H(5)	105.6
N(2)–C(6)–C(7)	108.95(14)
O(3)–C(7)–C(6)	111.00(17)
O(3)–C(8)–C(9)	111.55(17)
N(2)–C(9)–C(8)	108.37(16)
C(15)–C(10)–C(11)	118.45(16)
C(15)–C(10)–C(5)	121.27(14)
C(11)–C(10)–C(5)	120.05(15)
C(12)–C(11)–C(10)	120.06(17)
C(12)–C(11)–H(11)	120.0

Table 3 continued

C(10)–C(11)–H(11)	120.0
C(13)–C(12)–C(11)	120.99(17)
C(13)–C(12)–H(12)	119.5
C(11)–C(12)–H(12)	119.5
C(12)–C(13)–C(14)	119.18(17)
C(12)–C(13)–H(13)	120.4
C(14)–C(13)–H(13)	120.4
C(13)–C(14)–C(15)	120.36(19)
C(13)–C(14)–H(14)	119.8
C(15)–C(14)–H(14)	119.8
C(10)–C(15)–C(14)	120.94(16)
C(10)–C(15)–H(15)	119.5
C(14)–C(15)–H(15)	119.5
C(1)–N(1)–C(4)	112.12(14)
C(1)–N(1)–C(5)	121.83(13)
C(4)–N(1)–C(5)	125.98(13)
C(5)–N(2)–C(6)	117.40(12)
C(5)–N(2)–C(9)	113.10(14)
C(6)–N(2)–C(9)	109.35(14)
C(8)–O(3)–C(7)	109.78(15)

Symmetry transformations used to generate equivalent atoms

Table 4 List of intermolecular hydrogen bonds

Donor-H···acceptor	D–H	H···A	D···A	D–H···A
C(2)–H(2B)···O(3)[a]	0.97(3)	2.46(3)	3.194(3)	132(2)

Symmetry: $a = -x, 1 - y, -z$

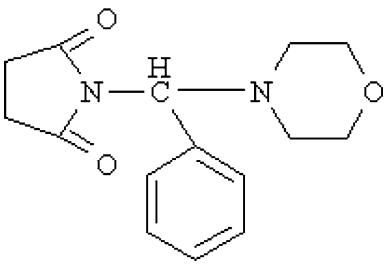

Fig. 2 The chemical diagram of the title compound

O(2) = 1.204] are found to be shorter than the C=O bond distances 1.24 and 1.26 Å found by Mason [36] in succinimide.

In the morpholine ring the C–C distances [C6–C7=C8–C9] and C–N distances [C6–N2] and [C9–N2] are 1.513 Å, 1.462 and 1.464 Å respectively. The C–O distances [C7–O3] and [C8–O3] are 1.427 and 1.414 Å respectively. The C–C, C–N and C–O distances in morpholine are found to be less than the normal values [C–C = 1.53, C–N = 1.47, C–O = 1.45 Å] reported by Alekseev [37]. This is in

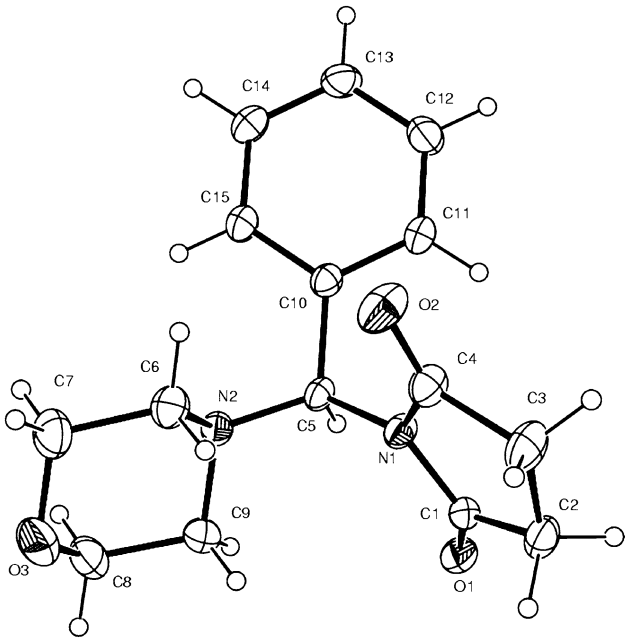

Fig. 3 The atom numbering scheme

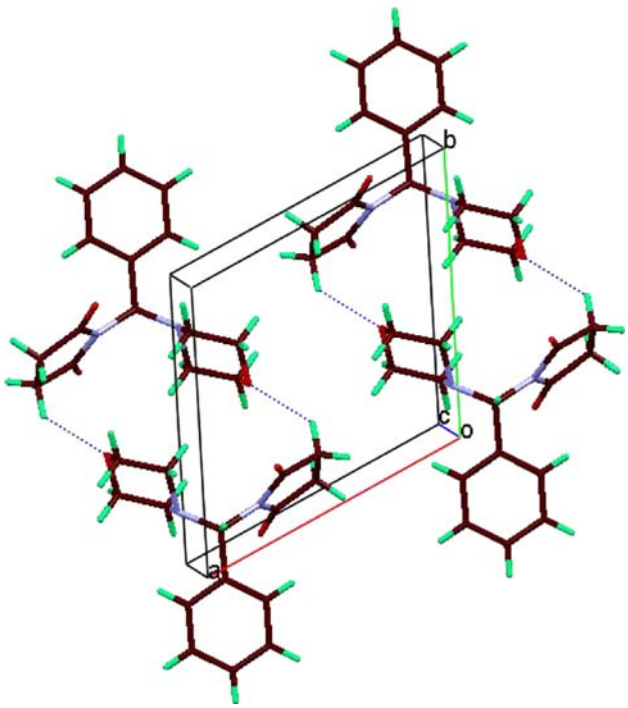

Fig. 4 Packing diagram showing C–H···O interaction

agreement with the structural data available from Version 5.14 of the Cambridge Structural Database [38]. The study of the torsion angle, asymmetry parameters and least square calculations shows that the six membered morpholine ring adopts a chair conformation with a deviation of O3 and N2 from the C6/C7/C8/C9 plane by 0.652(2) Å and

−0 .687(2) Å respectively, Q(2) = 0.019(2) Å, Φ = 8(7)°, Q_T = 0.587(2) Å, θ = 178.8(2)° [39]. The sum of the angles around N1 is 359.93° indicating sp^2 hybridization.

The [C1–N1–C5] angle between the succinimide and the phenyl moiety is 121.83° whereas the [C5–N2–C9] angle between the phenyl and morpholino moiety is 113.10°. The angle linking the three units [N1–C5–N2] is 114.70° which is less compared with the [N1–C5–N2] angle of 116.94° for 1-(Morpholin-4-ylmethyl)pyrrolidine-2,5-dione [40] and [N1–C9–N2] angle of 116.95° for 2-(Morpholin-4-ylmethyl)isoindole-1,3-dione [41]. The [N1–C5–N2] angle for the title compound may be less due to the presence of the bulky phenyl group linking the other two units.

The torsion angle of N(1)–C(5)–N(2)–C(6) is 55.34°. The structure is stabilized by weak intermolecular C–H···O interactions that link the molecule into a pair around a center (Fig. 4).

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