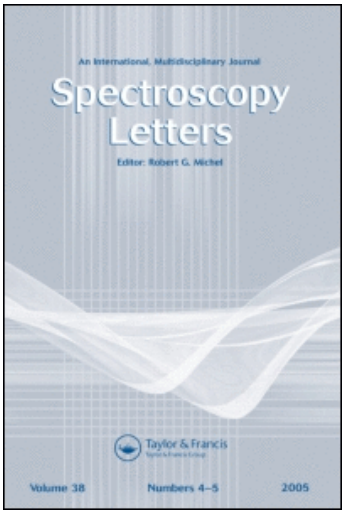

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X-ray Analysis of a Dipyrrolic Dimer: Molecular Geometry Induced by Intramolecular Hydrogen Bonding

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X-RAY ANALYSIS OF A DIPYRROLIC DIMER: MOLECULAR GEOMETRY INDUCED BY INTRAMOLECULAR HYDROGEN BONDING

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ABSTRACT

X-ray structure analysis is discussed in order to probe the information about the conformational behaviour of compound 1.

INTRODUCTION

Recently[1] we have presented the structure and conformation of 1-phenyl-1- (1, 2-dimethyl-5-pyrrolyl)-2-(2-methyl)-5-benzoyl-1-pyrrolyl-1-) ethanol (1), which bears some characteristics of molecular propellers. Our finding is supported by ^{1}H and ^{13}C NMR data, in particular by n.O.e. difference measurments and 2D NMR spectroscopies.
The NMR signals observed in $CDCl_3$, acetone-d_6 and DMSO-d_6 solutions at room temperature lead to the conclusion that compound 1 exists in one conformation only, with an intramolecular hydrogen bond involving a macrocycle formed by the carbonyl group of the benzoyl moiety (C8) and the hydroxyl group on C8'.

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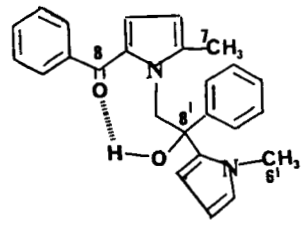

RESULTS AND DISCUSSION

In order to support these findings as an extension of our previous work[1], we now report that this analysis is nicely confirmed by the crystal structure depicted in Fig. 1 where a parallel conformation is adopted in solution and in the crystal.

As previously described,[1]HNMR analysis of 1 shows that the molecule adopts a distorted twist conformation with a shape of a molecular propeller.

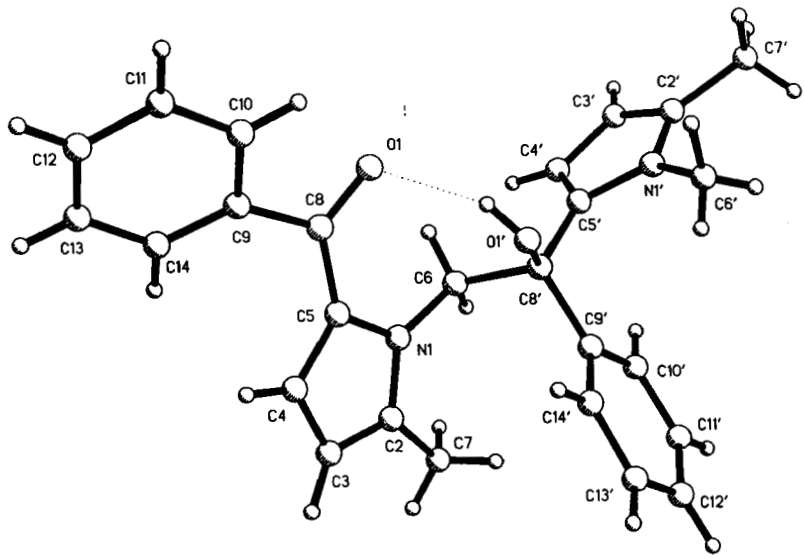

Fig. 1.- Perspective drawing of 1 showing atom labeling.

Table 1.

Structure Determination Summary.

Crystal Data

Empirical Formula	$C_{26} H_{26} N_2 O_2$
Color; Habit	Colorless, prism
Crystal size (mm)	0.12 x 0.23 x 0.38
Crystal System	Monoclinic
Space Group	$P2_1/n$
Unit Cell Dimensions	a = 5.768(2) A
	b = 12.936(3) A
	c = 29.023(6) A
	beta = 90.11(3)°
Volume	2165.4(11) A^3
Z	4
Formula weight	398.5
Density(calc.)	1.222 Mg/m^3
Absorption Coefficient	0.611 mm^{-1}
F(000)	848

Data Collection

Diffractometer Used	Siemens P3/PC
Radiation	CuKa (lambda = 1.54178 A)
Temperature (K)	293
Monochromator	Ni-filter

(continued)

Table 1. Continued.

2Theta Range	3.0 to 100.0°
Scan Type	omega
Scan Speed	Variable; 4.00 to 29.30°/min. in omega
Scan Range (omega)	1.2°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 100 reflections
Index Ranges	$0 \leq h \leq 5$, $0 \leq k \leq 12$ $-28 \leq l \leq 28$
Reflections Collected	2507
Independent Reflections	2223 (R_{int} = 3.03%)
Observed Reflections	1758 (F > 4.0 sigma(F))
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\text{Sum}[w(F_o - F_c)^2]$
Absolute Structure	N/A
Extinction Correction	x = 0.0061(14), where $F^* = F\,[\,1 + 0.002xF^2/\sin(2\text{Theta})\,]^{-1/4}$

Table 1. Continued.

Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = sigma^2(F) + 0.0041F^2$
Number of Parameters Refined	302
Final R Indices (obs. data)	R = 5.57 %, wR = 8.37 %
R Indices (all data)	R = 6.79 %, wR = 9.20 %
Goodness-of-Fit	1.15
Largest and Mean Delta/sigma	0.185, 0.010
Data-to-Parameter Ratio	5.8:1
Largest Difference Peak	0.19 eA^{-3}
Largest Difference Hole	-0.22 eA^{-3}

This conformation in solution is indicated by the combination of the highly shielded nature of N-Me (6') as well as the C7 methyl group, resulting from their respective locations above and below the aromatic benzylic ring and supported by nuclear Overhauser effect measurements. The close proximity of both methyls to the faces of the benzylic aromatic ring initially suggested that 1 was stabilized by an attractive, through-space hydrogen bond.

The importance of hydrogen bonding on the conformation of compound **1** is supported by the following considerations. The H-bonded conformation is favoured in nonpolar solvent because in such solvent strong solvent-solute hydrogen bonds cannot formed.. Consequently we observed in ^1H NMR a strongly deshielded OH proton even in $CDCl_3$.The x-ray crystal structure of compound 1 is depicted in Fig 1 and salient torsion angles of the structure presented here are grouped in table 5.

If an effective intramolecular H-bonding is to occur, the donor and the acceptor groups must bear a close spatial relationship with each other, conferings an appreciable amount of stability to this conformer.

Typical hydrogen bonds can be found in proteins due to the peptidic bonds that links together aminoacid residues. Because there are so

Table 2.

Atomic coordinates (x 10^4) and equivalent isotropic thermal parameters (Å x 10^3) for non-H atoms with e.s.d. in parentheses.

	x	y	z	U(eq)
N(1)	2935(5)	3993(2)	1155(1)	52(1)
C(2)	3922(7)	4565(3)	818(1)	61(1)
C(3)	2744(8)	5490(3)	789(2)	73(2)
C(4)	1056(8)	5491(3)	1120(1)	66(2)
C(5)	1142(7)	4563(3)	1359(1)	58(1)
C(6)	3683(7)	2955(3)	1288(1)	58(1)
C(7)	5989(9)	4236(4)	547(2)	73(2)
C(8)	-227(7)	4252(3)	1745(1)	61(2)
O(1)	-267(6)	3356(2)	1891(1)	87(1)
C(9)	-1725(7)	5017(3)	1987(1)	60(2)
C(10)	-3793(8)	4645(4)	2170(1)	70(2)
C(11)	-5262(9)	5294(5)	2407(2)	86(2)
C(12)	-4689(10)	6306(5)	2468(2)	88(2)
C(13)	-2655(10)	6680(4)	2300(2)	84(2)
C(14)	-1156(8)	6036(3)	2060(1)	68(2)
N(1')	1970(6)	145(3)	1210(1)	64(1)
C(2')	3001(9)	-613(3)	1467(2)	72(2)
C(3')	4756(9)	-175(3)	1708(2)	75(2)
C(4')	4779(7)	884(3)	1604(1)	66(2)
C(5')	3052(7)	1077(3)	1295(1)	56(1)
C(6')	6(10)	-24(4)	914(2)	80(2)
C(7')	2154(14)	-1697(4)	1471(3)	110(3)
C(8')	2338(6)	2054(3)	1057(1)	51(1)
O(1')	-112(4)	2235(2)	1111(1)	62(1)
C(9')	2776(6)	2034(3)	540(1)	52(1)
C(10')	4660(8)	1504(3)	366(2)	71(2)
C(11')	5136(9)	1506(4)	-99(2)	86(2)
C(12')	3723(10)	2031(4)	-400(2)	89(2)
C(13')	1877(9)	2568(4)	-229(2)	88(2)
C(14')	1366(8)	2560(4)	233(2)	73(2)

• Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 3.

Bond lenghts (Å) of compound 1.

N(1)-C(2)	1.352 (5)	N(1)-C(5)	1.402 (5)
N(1)-C(6)	1.462 (5)	C(2)-C(3)	1.379 (6)
C(2)-C(7)	1.492 (7)	C(3)-C(4)	1.370 (6)
C(4)-C(5)	1.387 (6)	C(5)-C(8)	1.430 (6)
C(6)-C(8')	1.552 (5)	C(8)-O(1)	1.235 (5)
C(8)-C(9)	1.491 (6)	C(9)-C(10)	1.392 (6)
C(9)-C(14)	1.376 (6)	C(10)-C(11)	1.379 (7)
C(11)-C(12)	1.362 (9)	C(12)-C(13)	1.361 (8)
C(13)-C(14)	1.389 (7)	N(1')-C(2')	1.369 (6)
N(1')-C(5')	1.380 (5)	N(1')-C(6')	1.437 (7)
C(2')-C(3')	1.354 (7)	C(2')-C(7')	1.485 (8)
C(3')-C(4')	1.402 (6)	C(4')-C(5')	1.363 (6)
C(5')-C(8')	1.498 (5)	C(8')-O(1')	1.441 (4)
C(8')-C(9')	1.522 (5)	C(9')-C(10')	1.382 (6)
C(9')-C(14')	1.385 (6)	C(10')-C(11')	1.378 (7)
C(11')-C(12')	1.374 (7)	C(12')-C(13')	1.364 (8)
C(13')-C(14')	1.375 (7)		

Table 4.

Bond angles (°) for structure 1.

C(2)-N(1)-C(5)	109.2(3)	C(2)-N(1)-C(6)	124.7(3)
C(5)-N(1)-C(6)	126.1(3)	N(1)-C(2)-C(3)	108.2(4)
N(1)-C(2)-C(7)	124.3(4)	C(3)-C(2)-C(7)	127.5(4)
C(2)-C(3)-C(4)	108.0(4)	C(3)-C(4)-C(5)	108.9(4)
N(1)-C(5)-C(4)	105.7(3)	N(1)-C(5)-C(8)	126.3(3)
C(4)-C(5)-C(8)	128.0(4)	N(1)-C(6)-C(8')	115.4(3)
C(5)-C(8)-O(1)	123.0(4)	C(5)-C(8)-C(9)	120.3(4)
O(1)-C(8)-C(9)	116.7(4)	C(8)-C(9)-C(10)	116.6(4)
C(8)-C(9)-C(14)	124.8(4)	C(10)-C(9)-C(14)	118.5(4)
C(9)-C(10)-C(11)	120.5(4)	C(10)-C(11)-C(12)	120.1(5)
C(11)-C(12)-C(13)	120.3(5)	C(12)-C(13)-C(14)	120.3(5)
C(9)-C(14)-C(13)	120.2(4)	C(2')-N(1')-C(5')	109.4(3)
C(2')-N(1')-C(6')	123.9(4)	C(5')-N(1')-C(6')	126.6(4)
N(1')-C(2')-C(3')	107.8(4)	N(1')-C(2')-C(7')	122.6(5)
C(3')-C(2')-C(7')	129.6(5)	C(2')-C(3')-C(4')	107.8(4)
C(3')-C(4')-C(5')	108.3(4)	N(1')-C(5')-C(4')	106.7(3)
N(1')-C(5')-C(8')	122.0(3)	C(4')-C(5')-C(8')	131.2(4)
C(6)-C(8')-C(5')	107.3(3)	C(6)-C(8')-O(1')	108.7(3)
C(5')-C(8')-O(1')	110.9(3)	C(6)-C(8')-C(9')	110.7(3)
C(5')-C(8')-C(9')	113.2(3)	O(1')-C(8')-C(9')	105.9(3)
C(8')-C(9')-C(10')	120.1(3)	C(8')-C(9')-C(14')	121.9(3)
C(10')-C(9')-C(14')	118.0(4)	C(9')-C(10')-C(11')	121.0(4)
C(10')-C(11')-C(12')	120.3(5)	C(11')-C(12')-C(13')	119.0(5)
C(12')-C(13')-C(14')	121.2(5)	C(9')-C(14')-C(13')	120.4(4)

Table 5.

Torsion angles (°) for compound 1.

C5	N1	C2	C3	-2.0(0.4)	C5	N1	C2	C7	175.8(0.4)	C6	N1	C2	C3	179.5(0.3)
C6	N1	C2	C7	-2.7(0.6)	C2	N1	C5	C4	1.7(0.4)	C2	N1	C5	C8	-176.0(0.4)
C6	N1	C5	C4	-179.8(0.3)	C6	N1	C5	C8	2.5(0.6)	C2	N1	C6	H6A	25.7(0.4)
C2	N1	C6	H6B	144.0(0.3)	C2	N1	C6	C8'	-95.2(0.4)	C5	N1	C6	H6A	-152.6(0.3)
C5	N1	C6	H6B	-34.3(0.4)	C5	N1	C6	C8'	86.6(0.4)	N1	C2	C3	H3	-178.4(0.2)
N1	C2	C3	C4	1.6(0.5)	C7	C2	C3	H3	3.8(0.6)	C7	C2	C3	C4	-176.1(0.4)
N1	C2	C7	H7A	-164.5(2.5)	N1	C2	C7	H7B	65.2(2.4)	N1	C2	C7	H7C	-43.7(2.4)
C3	C2	C7	H7A	12.9(2.6)	C3	C2	C7	H7B	-117.4(2.4)	C3	C2	C7	H7C	133.7(2.4)
C2	C3	C4	H4	179.5(0.2)	C2	C3	C4	C5	-0.5(0.5)	H3	C3	C4	H4	-0.5(0.4)
H3	C3	C4	C5	179.5(0.2)	C3	C4	C5	N1	-0.7(0.5)	C3	C4	C5	C8	177.0(0.4)
H4	C4	C5	N1	179.3(0.2)	H4	C4	C5	C8	-3.0(0.5)	N1	C5	C8	O1	-13.9(0.6)
N1	C5	C8	C9	167.1(0.4)	C4	C5	C8	O1	168.9(0.4)	C4	C5	C8	C9	-10.1(0.6)
N1	C6	C8'	C5'	-170.2(0.3)	N1	C6	C8'	O1'	-50.2(0.4)	N1	C6	C8'	C9'	65.7(0.4)
H6A	C6	C8'	C5'	68.9(0.3)	H6A	C6	C8'	O1'	-171.1(0.2)	H6A	C6	C8'	C9'	-55.1(0.3)
H6B	C6	C8'	C5'	-49.4(0.3)	H6B	C6	C8'	O1'	70.6(0.3)	H6B	C6	C8'	C9'	-173.4(0.2)
C5	C8	C9	C10	146.7(0.4)	C5	C8	C9	C14	-36.7(0.6)	O1	C8	C9	C10	-32.4(0.5)
O1	C8	C9	C14	144.2(0.4)	C8	C9	C10	H10	-1.3(0.4)	C8	C9	C10	C11	178.7(0.4)
C14	C9	C10	H10	-178.1(0.3)	C14	C9	C10	C11	1.9(0.6)	C8	C9	C14	C13	-178.4(0.4)
C8	C9	C14	H14	1.6(0.5)	C10	C9	C14	C13	-1.8(0.6)	C10	C9	C14	H14	178.1(0.3)
C9	C10	C11	H11	179.4(0.3)	C9	C10	C11	C12	-0.6(0.7)	H10	C10	C11	H11	-0.6(0.3)
H10	C10	C11	C12	179.4(0.3)	C10	C11	C12	H12	179.3(0.3)	C10	C11	C12	C13	-0.7(0.8)
H11	C11	C12	H12	-0.7(0.3)	H11	C11	C12	C13	179.3(0.3)	C11	C12	C13	H13	-179.3(0.3)
C11	C12	C13	C14	0.7(0.8)	H12	C12	C13	H13	0.7(0.3)	H12	C12	C13	C14	-179.3(0.3)
C12	C13	C14	C9	0.6(0.7)	C12	C13	C14	H14	-179.4(0.3)	H13	C13	C14	C9	-179.4(0.3)
H13	C13	C14	H14	0.6(0.3)	C5'	N1'	C2'	C3'	-1.2(0.5)	C5'	N1'	C2'	C7'	176.7(0.5)
C6'	N1'	C2'	C3'	-178.9(0.4)	C6'	N1'	C2'	C7'	-1.0(0.8)	C2'	N1'	C5'	C4'	0.6(0.5)
C2'	N1'	C5'	C8'	178.9(0.4)	C6'	N1'	C5'	C4'	178.3(0.4)	C6'	N1'	C5'	C8'	-3.5(0.6)
C2'	N1'	C6'	H6'A	-151.3(3.0)	C2'	N1'	C6'	H6'B	77.0(2.0)	C2'	N1'	C6'	H6'C	-52.3(3.0)
C5'	N1'	C6'	H6'A	31.4(3.0)	C5'	N1'	C6'	H6'B	-100.3(2.0)	C5'	N1'	C6'	H6'C	130.4(2.9)
N1'	C2'	C3'	H3'	-178.8(0.2)	N1'	C2'	C3'	C4'	1.2(0.5)	C7'	C2'	C3'	H3'	3.5(0.7)
C7'	C2'	C3'	C4'	-176.5(0.6)	N1'	C2'	C7'	H7'A	179.8(2.7)	N1'	C2'	C7'	H7'B	60.0(2.5)
N1'	C2'	C7'	H7'C	-73.7(2.8)	C3'	C2'	C7'	H7'A	-2.8(2.9)	C3'	C2'	C7'	H7'B	-122.6(2.5)
C3'	C2'	C7'	H7'C	103.7(2.8)	C2'	C3'	C4'	H4'	179.2(0.3)	C2'	C3'	C4'	C5'	-0.8(0.5)
H3'	C3'	C4'	H4'	-0.8(0.4)	H3'	C3'	C4'	C5'	179.2(0.2)	C3'	C4'	C5'	N1'	0.1(0.5)
C3'	C4'	C5'	C8'	-177.9(0.4)	H4'	C4'	C5'	N1'	-179.9(0.2)	H4'	C4'	C5'	C8'	2.1(0.6)
N1'	C5'	C8'	C6	172.1(0.3)	N1'	C5'	C8'	O1'	53.5(0.5)	N1'	C5'	C8'	C9'	-65.3(0.5)
C4'	C5'	C8'	C6	-10.1(0.6)	C4'	C5'	C8'	O1'	-128.7(0.4)	C4'	C5'	C8'	C9'	112.4(0.5)
C6	C8'	O1'	H1'	-44.8(2.8)	C5'	C8'	O1'	H1'	73.0(2.8)	C9'	C8'	O1'	H1'	-163.8(2.8)
C6	C8'	C9'	C10'	87.2(0.4)	C6	C8'	C9'	C14'	-91.1(0.4)	C5'	C8'	C9'	C10'	-33.4(0.5)
C5'	C8'	C9'	C14'	148.4(0.4)	O1'	C8'	C9'	C10'	-155.1(0.3)	O1'	C8'	C9'	C14'	26.7(0.5)
C8'	C9'	C10'	H10'	2.3(0.4)	C8'	C9'	C10'	C11'	-177.7(0.4)	C14'	C9'	C10'	H10'	-179.4(0.3)
C14'	C9'	C10'	C11'	0.6(0.6)	C8'	C9'	C14'	C13'	176.8(0.4)	C8'	C9'	C14'	H14'	-3.2(0.4)
C10'	C9'	C14'	C13'	-1.5(0.6)	C10'	C9'	C14'	H14'	178.5(0.3)	C9'	C10'	C11'	H11'	179.4(0.3)
C9'	C10'	C11'	C12'	-0.6(0.7)	H10'	C10'	C11'	H11'	-0.6(0.3)	H10'	C10'	C11'	C12'	179.4(0.4)
C10'	C11'	C12'	H12'	-178.6(0.3)	C10'	C11'	C12'	C13'	1.4(0.8)	H11'	C11'	C12'	H12'	1.4(0.4)
H11'	C11'	C12'	C13'	-178.6(0.4)	C11'	C12'	C13'	H13'	177.6(0.4)	C11'	C12'	C13'	C14'	-2.4(0.8)
H12'	C12'	C13'	H13'	-2.4(0.4)	H12'	C12'	C13'	C14'	177.6(0.3)	C12'	C13'	C14'	C9'	2.4(0.8)
C12'	C13'	C14'	H14'	-177.6(0.4)	H13'	C13'	C14'	C9'	-177.6(0.3)	H13'	C13'	C14'	H14'	2.4(0.3)

many peptidic bonds in a protein molecule, the importance of these weak forces is considerable, and they play a special role in determining specific biological activity and active sites of proteins.

Regardless of the particular aminoacid involved in a peptidic linkage, a similarity between of the peptide carbonyl group dimensions[2] of 1.24 Å with that of compound **1**, $-C_8= O_1$ group 1.235 Å , is observed. On the other hand, it has been described[3,4] that the O-H bond length in O-H·····O=C- bond increase from 0.97Å to 1.07Å as the O·····O distance decreases from 2.85Å to- 2.50Å. For compound **1**, the internuclear distances of O_1·····$O_{1'}$ 2.69 Å,(σ=0.004) and $O_{1'}$-$H_{1'}$ 0.84 Å (σ=0.004) were found.

Table 6.

Non bonded distances for compound 1.

Second atoms generated by transformation:						**0.00000 +X**		**0.00000 +Y**		**0.00000 +Z**	
H6'C-H6'A	1.167	H7'C-H7'A	1.340	H7C -H7B	1.478	H7'B-H7'A	1.522	H6B -H6A	1.568	H7C -H7A	1.575
H6'C-H6'B	1.597	H7'C-H7'B	1.605	H7B -H7A	1.623	H6'B-H6'A	1.646	H1' -C8'	1.828	H1' -O1	1.898
H6'C-N1'	1.959	H7'C-C2'	1.963	H7A -C2	1.967	H6'A-N1'	1.971	H6B -N1	1.981	H6A -N1	1.981
H6'B-N1'	1.987	H7'A-C2'	1.992	H13'-C12'	2.017	H13 -C12	2.018	C13 -H12	2.018	H12 -C11	2.019
C12 -H11	2.020	H7'B-C2'	2.024	C14'-H13'	2.026	C13'-H12'	2.028	C11'-H10'	2.030	C12'-H11'	2.030
H14'-C13'	2.031	H14 -C9	2.032	C11 -H10	2.034	H11'-C10'	2.034	H10'-C9'	2.034	H11 -C10	2.036
H12'-C11'	2.037	H14'-C9'	2.040	C14 -H13	2.044	H14 -C13	2.044	H10 -C9	2.046	C8' -H6A	2.061
C8' -H6B	2.061	H7B -C2	2.070	H3' -C2'	2.070	H7C -C2	2.071	C5' -H4'	2.076	H4 -C3	2.080
C4 -H3	2.084	H3 -C2	2.093	C5 -H4	2.096	H4' -C3'	2.113	C4' -H3'	2.116	H7C -H6A	2.196
C3' -N1'	2.200	C4' -N1'	2.201	C3 -N1	2.212	C4 -N1	2.223	C4 -C2	2.223	C4' -C2'	2.227
C5' -C3'	2.241	C5' -C2'	2.243	C5 -C3	2.244	C5 -C2	2.245	H14 -H4	2.257	H13 -H12	2.316
H12 -H11	2.319	H13'-H12'	2.323	H14'-H13'	2.323	C9 -O1	2.325	H11'-H10'	2.328	H1' -H6B	2.333
H11 -H10	2.334	H12'-H11'	2.339	O1 -C5	2.343	H14 -H13	2.345	C13'-C11'	2.359	C13 -C11	2.361
C9' -O1'	2.365	C14'-C10'	2.372	C12 -C10	2.375	C14 -C10	2.379	H1' -C6	2.380	C14 -C12	2.385
C14'-C12'	2.387	C12'-C10'	2.387	H4' -H6B	2.387	C13'-C9'	2.395	O1 -H6B	2.396	C13 -C9	2.397
H14'-O1'	2.400	C11'-C9'	2.402	C11 -C9	2.406	O1' -C5'	2.420	O1' -C6	2.433	C10 -C8	2.454
C5' -C6	2.456	C6' -C2'	2.476	H4' -H6A	2.478	H10 -O1	2.487	H4 -H3	2.492	C6 -C2	2.493
C7' -N1'	2.503	C7 -N1	2.515	C10'-C8'	2.516	C6' -C5'	2.517	C8' -N1'	2.518	C9' -C5'	2.521
C8 -N1	2.526	C9' -C6	2.529	H4' -H3'	2.531	C8 -C4	2.532	C9 -C5	2.533	C5' -H6B	2.538
C14 -C8	2.541	C14'-C8'	2.542	C8' -N1	2.547	C6 -C5	2.553	C7' -C3'	2.569	H6A -C2	2.574
C7 -C3	2.576	H1' -C5'	2.579	H10'-C5'	2.581	H10 -C8	2.585	H1' -C8	2.590	H7'B-H6'C	2.592
H7A -H3	2.605	C8' -C4'	2.606	H7A -C3	2.613	C7 -H6A	2.628	C14 -H4	2.645	H14 -C4	2.653
C9' -H6A	2.659	H10'-C8'	2.661	H6B -C5	2.662	H7'A-C3'	2.676	C5' -H6A	2.683	O1' -H6B	2.685
O1' -O1	2.690	H6'A-C5'	2.698	C4' -H6B	2.699	O1' -H6'A	2.701	H4' -C6	2.701	H14'-C8'	2.706
C9' -H7B	2.717	C14'-O1'	2.722	C9' -H6'A	2.727	H6'C-C2'	2.727	C13'-C10'	2.730	H7C -N1	2.731
H14 -C8	2.733	C13 -C10	2.739	C14'-C11'	2.743	C14 -C11	2.749	H7'B-N1'	2.752	C10 -O1	2.753
C12 -C9	2.769	C12'-C9'	2.783	C8' -H6'A	2.796	H7'C-N1'	2.796	C8 -H6B	2.807	H6'B-C2'	2.813
C10'-H7B	2.818	H1' -N1	2.820	C8 -H4	2.846	C4' -H6A	2.848	H7B -N1	2.849	H7C -C6	2.856
H14 -C5	2.866	C14'-H7B	2.873	C9 -H4	2.874	O1' -N1	2.876	C7 -H3	2.882	H1' -C5	2.889
C7' -H3'	2.890	C4' -C6	2.902	C10'-C5'	2.905	C7' -H6'C	2.910	H7'B-C6'	2.920	O1 -C6	2.922
C8' -H4'	2.933	O1 -N1	2.945	C7' -C6'	2.970	O1' -N1'	2.972	O1' -C6'	2.979	H6'B-C5'	3.011
C7 -C6	3.025	H7'C-C3'	3.028	C8' -C6'	3.034	C9 -C4	3.048	C10'-H6A	3.049	H10'-C4'	3.054
C9' -H1'	3.054	C11'-H7B	3.069	H7B -C6	3.070	H7'C-C6'	3.075	C14 -C5	3.089	C14 -C4	3.095
C9' -N1	3.099	C8 -C6	3.110	C9' -N1'	3.156	O1' -C5	3.179	O1' -C8	3.193	C9' -C6'	3.290
C8' -O1	3.311	C10'-C6	3.317	C14'-N1	3.377	C14'-C6	3.378	C10'-N1'	3.391	C9' -C7	3.398
C14'-C2	3.431	C8' -C5	3.432	C9' -C2	3.435	C8' -C2	3.445				
Second atoms generated by transformation:						**1.00000 +X**		**0.00000 +Y**		**0.00000 +Z**	
H7C -C5	3.001	H10'-C6'	3.082								
Second atoms generated by transformation:						**-1.00000 +X**		**0.00000 +Y**		**0.00000 +Z**	
H14'-H7C	2.394	H10 -H6B	2.405	H6'B-C3'	2.767	O1' -H6A	2.796	H6'B-C4'	2.861	O1' -H7C	2.883
H14'-C7	2.943	H10 -C6	3.008								
Second atoms generated by transformation:						**0.00000 -X**		**1.00000 -Y**		**0.00000 -Z**	
H13'-H4	2.561	H13'-C4	3.040	H13'-C3	3.086						
Second atoms generated by transformation:						**1.00000 -X**		**0.00000 -Y**		**0.00000 -Z**	
H12'-H7'B	2.507	H11'-H6'C	2.600								
Second atoms generated by transformation:						**-0.50000 -X**		**0.50000 +Y**		**0.50000 -Z**	
H12 -O1	2.554	H13 -H10	2.636	H13 -O1	2.914	C12 -O1	3.238	C13 -O1	3.414		
Second atoms generated by transformation:						**-0.50000 -X**		**-0.50000 +Y**		**0.50000 -Z**	
C3' -H11	2.847										
Second atoms generated by transformation:						**0.50000 -X**		**-0.50000 +Y**		**0.50000 -Z**	
H4' -C13	3.027										

It is well known[3] that a hydrogen bond in which the donor-acceptor angle is close to 180° is energetically more favourable compared to a bent bond. Such angle in compound 1, $O_{1'}$-$H_{1'}$···· O_1 was found to be 156 (3)° while the $H_{1'}$···· O_1 hydrogen bond distance was found to be1.90 (4) Å. This bond is comparable to the H-bond in peptides wherein is linear with a N···O distance of 2.72 Å interconnect C=O and N-H groups[5-7] along either the same chain or between two chains[8].

Table 7.

Anisotropic displacement coefficients ($Å^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	69(2)	40(2)	48(2)	-2(2)	7(2)	-1(1)
C(2)	74(3)	49(3)	59(2)	-11(2)	9(2)	-5(2)
C(3)	103(3)	49(3)	66(3)	-2(2)	18(3)	8(2)
C(4)	87(3)	51(3)	61(3)	9(2)	11(3)	2(2)
C(5)	72(3)	44(2)	56(2)	-1(2)	10(2)	-7(2)
C(6)	66(2)	53(3)	54(2)	1(2)	0(2)	-4(2)
C(7)	79(3)	67(3)	71(3)	-11(3)	16(3)	-5(3)
C(8)	82(3)	46(3)	56(2)	1(2)	9(2)	-1(2)
O(1)	124(3)	56(2)	80(2)	12(2)	45(2)	8(2)
C(9)	73(3)	60(3)	46(2)	5(2)	6(2)	-7(2)
C(10)	79(3)	77(3)	55(3)	1(2)	3(2)	-13(2)
C(11)	69(3)	117(5)	71(3)	1(3)	10(3)	-15(3)
C(12)	93(4)	96(5)	76(3)	27(3)	9(3)	-23(3)
C(13)	113(4)	65(3)	75(3)	12(3)	-2(3)	-21(3)
C(14)	86(3)	54(3)	64(3)	4(2)	9(2)	-11(2)
N(1')	72(2)	51(2)	70(2)	1(2)	9(2)	-1(2)
C(2')	84(3)	51(3)	80(3)	8(3)	24(3)	12(2)
C(3')	84(3)	66(3)	76(3)	13(3)	9(3)	21(2)
C(4')	67(3)	71(3)	62(3)	-2(2)	2(2)	9(2)
C(5')	62(2)	47(3)	58(2)	-4(2)	8(2)	-2(2)
C(6')	85(4)	65(3)	92(4)	-5(3)	10(3)	-20(3)
C(7')	134(6)	63(4)	133(6)	9(4)	41(5)	20(3)
C(8')	46(2)	53(2)	54(2)	4(2)	4(2)	-5(2)
O(1')	56(2)	62(2)	68(2)	4(1)	9(1)	-7(1)
C(9')	50(2)	47(2)	57(2)	1(2)	2(2)	-5(2)
C(10')	71(3)	74(3)	69(3)	11(2)	14(2)	-1(2)
C(11')	90(3)	85(4)	82(4)	6(3)	33(3)	-17(3)
C(12')	97(4)	114(4)	55(3)	-26(3)	12(3)	-15(3)
C(13')	88(4)	113(4)	64(3)	4(3)	-4(3)	16(3)
C(14')	64(3)	82(3)	73(3)	9(2)	-1(2)	-3(3)

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$$

Table 8.

H-Atom coordinates (x 10^4) and isotropic displacement coefficients ($Å^2$ x 10^3).

	x	y	z	U
H(3)	3053	6037	574	60
H(4)	-13	6045	1178	60
H(6A)	5291	2885	1209	60
H(6B)	3506	2891	1616	60
H(7A)	6549(66)	4823(32)	403(13)	60
H(7B)	5683(62)	3634(30)	356(12)	60
H(7C)	7194(66)	3926(29)	747(13)	60
H(10)	-4198	3930	2130	60
H(11)	-6691	5032	2530	60
H(12)	-5725	6757	2631	60
H(13)	-2254	7392	2348	60
H(14)	283	6304	1945	60
H(3')	5798	-526	1914	60
H(4')	5831	1386	1730	60
H(6'A)	-91(71)	387(30)	678(13)	60
H(6'B)	-1421(66)	-69(27)	1136(13)	60
H(6'C)	120(66)	-504(30)	723(13)	60
H(7'A)	3036(66)	-2093(31)	1658(13)	60
H(7'B)	2226(65)	-1976(31)	1161(12)	60
H(7'C)	860(69)	-1735(31)	1630(13)	60
H(1')	-227(67)	2416(31)	1389(13)	60
H(10')	5654	1128	573	60
H(11')	6461	1138	-213	60
H(12')	4029	2021	-724	60
H(13')	918	2959	-437	60
H(14')	23	2922	343	60

The eight membered ring formed through the intramolecular hydrogen bond ($O_{1'}$-$H_{1'}$····O_1) adopts a distorted twist conformation with a pseudo-C2 axis passing through C_8 and $C_{8'}$.

Others important features of the molecule may be described as follows. The phenyl ring of the benzoyl moiety is almost planar and is oriented towards the proximal pyrrolic moiety (10.1°). However the benzylic ring showed an "out of plane" orientation with the second (distal) pyrrolic moiety (-65.3°).

The hydroxyl group is oriented "out of plane" with respect to the benzylic ring and inclined by -155.1° as shown in Fig 1.

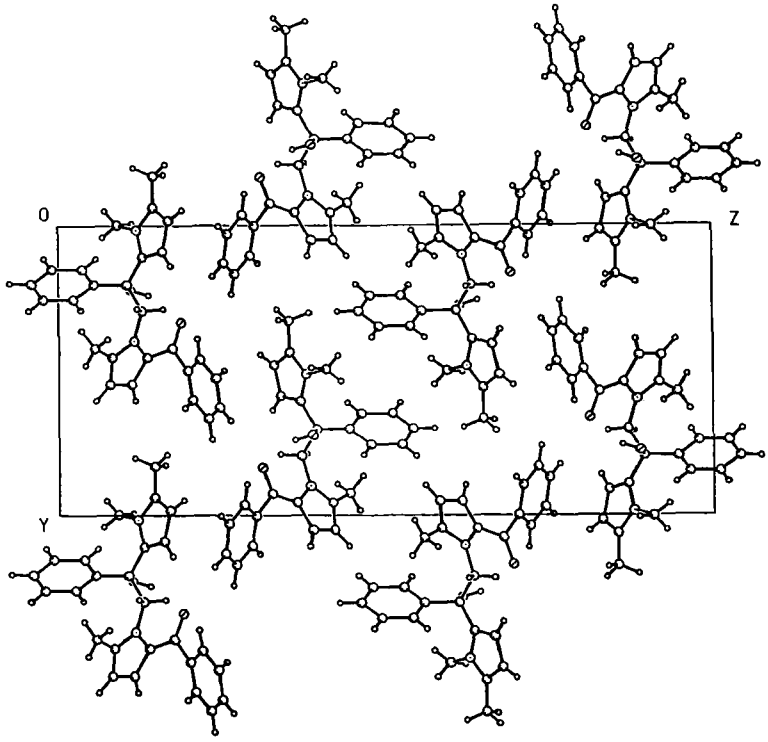

Fig. 2.- Crystal packing of the title molecule.

Concluding comments.

The propeller form observed in the structure of compound **1** confirms that intramolecular hydrogen bonding remains as a potent source of stabilization and therefore of conformation preferences.

EXPERIMENTAL

X-ray determination of **1** was carried out on a sample recrystalized from ethyl acetate. Intensities were collected on a Siemens P3/PC diffractometer using Ni-filtered CuKα radiation. The crystal structure was solved by direct methods and refined by full matrix least squares using SHELXTL program package[9] on a DEC VAX STATION II computer. (For details see Tables 1-8) With respect to the

H-atom coordinates (table 8), the hydrogens attached to carbon atoms were not refined. They were only assigned.
A diagram of the molecule with atom numbering scheme is shown in Fig 1, and a view of the unit cell is shown in Fig 2.

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