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Crystal structure analysis of 2-Benzyl-4-(9*H*-fluoren-9-ylidene)-6,7-dimethoxy-3-(naphthalen-1-yl)-1,2,3,4-tetrahydroisoquinoline

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Abstract : In the asymmetric unit of the title compound, $C_{41}H_{33}N_1O_2$, the piperidine adopts a half chair conformation, with the N-C bond in an equatorial orientation in both molecules. The dihedral angle between the fluorine and piperidine ring is 47.51(2)° for molecule A and the corresponding angle in molecule B is 47.1(2)°. The molecular packing is C--H...O hydrogen bonds that lead to a twisted supramolecular chain along b-axis direction. The adjacent molecular packing is further connected by C–H…π and offset π…πinteractions. Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct method and refined on F^2 by full-matrix least-squares procedure to the final R_1 of 0.0917 using SHELXL programs.
Key Words: Fluoren, Dimethoxy, Naphthalene, Tetrahydroisoquinoline and Crystal structure.

Introduction

Naphthalene derivatives has been identified as new range of potent antimicrobials effective against wide range of human pathogens and have diverse and interesting antibiotic properties with minimum toxicity[1,4]. Thiopheneand thiazole derivatives are known to possess interesting biological properties like anticancer[7].

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Experimental

X-ray Structure Determination

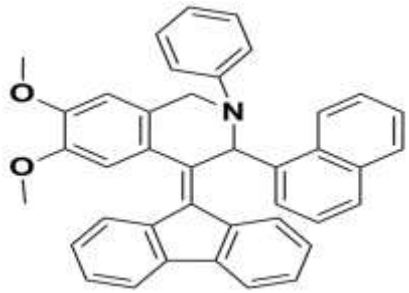
Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a Bruker[2]SMART APEX CCD diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å). The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[5]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[6]. The crystallographic data for the compound are listed in Table 1.

Table 1: Crystal data and structure refinement of the titled compound

Compound	**Parameters**
Empirical formula	C_{41} H_{33} N O_2
Formula weight	571.68
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 9.8986(8) Å; alpha = 86.497(5)º
	b = 9.9772(8) Å; beta = 89.511(5)º
	c = 32.254(3) Å; gamma = 73.341(5)º
Volume	3045.8(4)A^3
Z, Calculated density	4, 1.247Mg/m^3
Absorption coefficient	0.076mm^{-1}
F(000)	1208
Crystal size	0.35 x 0.30 x 0.20 mm
Theta range for data collection	0.632 to 26.629º.
Limiting indices	-12<=h<=12, -12<=k<=12, -40<=l<=40
Reflections collected / unique	43528 / 12705 [R(int) = 0.0823]
Completeness to theta = 25.00	99.9%
Max. and min. transmission	0.1774 and 0.0905
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12705 / 0 / 798
Goodness-of-fit on F2	1.076
Final R indices [I>2sigma(I)]	R1 = 0.0917, wR2 = 0.2665
R indices (all data)	R1 = 0.2149, wR2 = 0.3195
Largest diff. peak and hole	0.328 and -0.357e. $Å^{-3}$

Synthesis of the compound

To a clean, dry, two neck round-bottomed flask containing $Pd(PPh_3)_4$(0.03 mmol, 10 mol%) and K_2CO_3 (0.15 mmol) under N_2 atmosphere, was added a solution of 3-([1,1'-Biphenyl]-2-yl)-N-benzyl-N-(2-bromo-4,5-dimethoxybenzyl)-1-(naphthalen-1-yl)prop-2-yn-1-amine (0.3 mmol) in DMF (4 ml). The reaction mixture was stirred at 100% for 2 hour. The reaction mixture was cooled to room temperature, diluted with water (50 ml) and extracted with ethyl acetate. The combined organic layer was washed with saturated NaCl solution, dried over anhydrous $Na_2SO_4$and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (5-40%) to afford product I. The single crystal of the title compound (I) have been grown in an ethanol solution by slow evaporation of the solvent at room temperature and suitable for single crystal X-ray diffraction studies.



Results and Discussion

Fig. 1 shows the asymmetric unit consisting of the two independent molecules (A and B) of the title compound. The two molecules have the same geometrical parameters within the precision of the experiment. The piperidine ring adopts a half chair conformation with puckering parameters Q = 0.525(5)Å, Q = 55.9(5)Å and f = 313.3(7)°. The dihedral angle between the quinoline andnaphtahlane ring is 79.21(2)°(A) and 78.38(2)° (B). In molecule A, the methoxy groupassumes an extended conformation as can be seen from the C19-C18-O1-C40 and C16-C17-O2-41 torsion angles of -24.7(8)° and 6.8(7)° [In B, C57-C58-O3-C81 = -1.6(7)° and C60-C59-O4-82 = 26.1(2)°]. In the crystal, the A and B molecules are linked via pairs of C11-H11...O2 hydrogen bonds, forming chains. The chains are linked via C45-H45...O3 hydrogen bonds, forming slabs lying parallel to (010) (Table 2, Fig.1).The selected bond lengths and angles are listed in table 3.

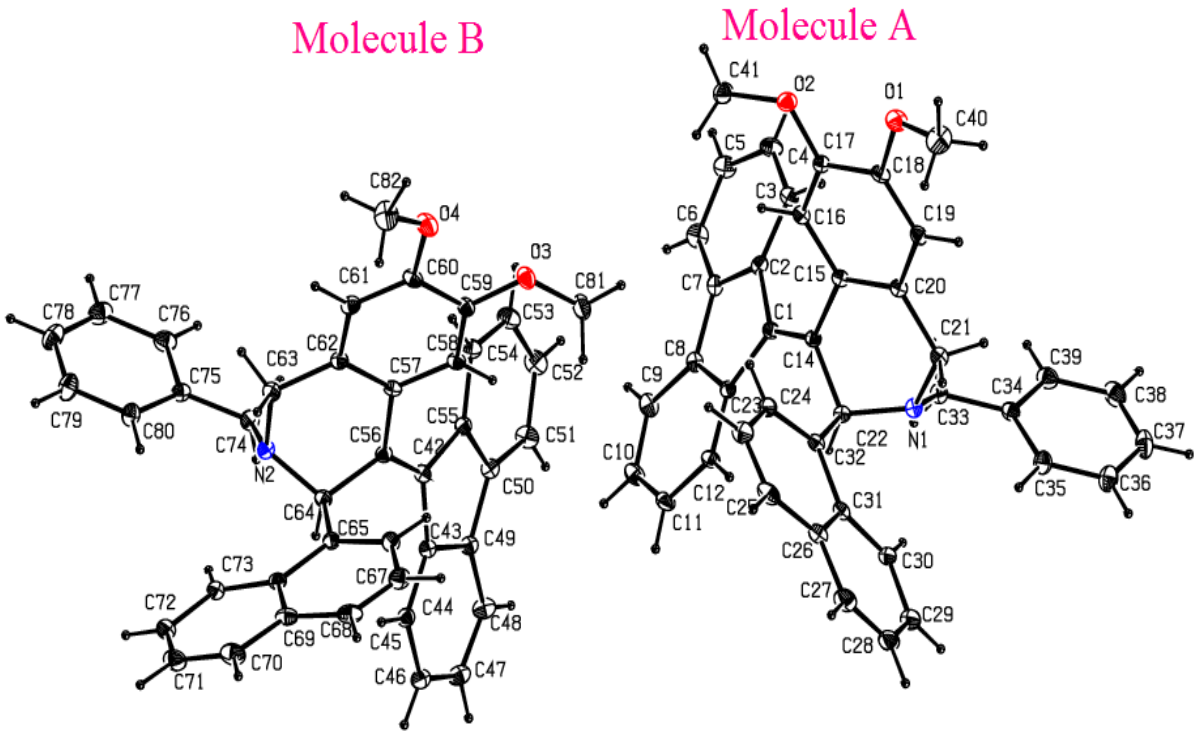


Fig.1. The molecular structure of the title compound with displacementellipsoids is drawn at the 10% probability level. The H atoms not involved in the hydrogen bonding have been excluded for clarity.

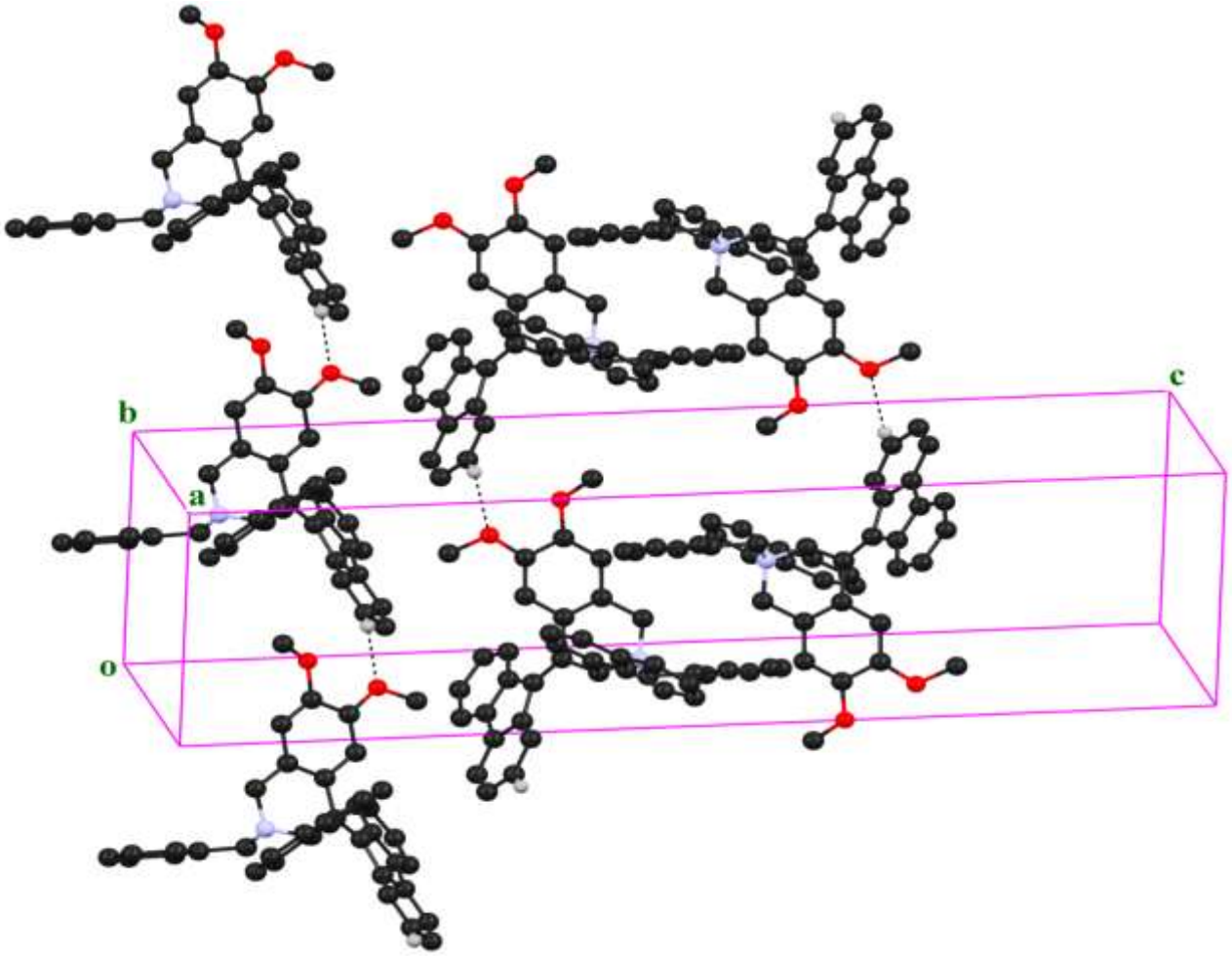


Fig.2.A packing diagram of the title compound showing a zigzag chainalong to the b axis formed via C---H...O hydrogen bonds (dashed lines).The H atoms not involved in the hydrogen bonding have been excludedfor clarity.

Table 2: Hydrogen-bond geometry [Å]

Distance (Å)				**Angle (°)**
D—H…A	**D—H**	**H…A**	**D…A**	**D—H…A**
C11---H11…O2	0.93	2.53	3.330(7)	145
C45---H45…O3	0.93	2.53	3.377(7)	151

Symmetry code: *x*-1, *y*+1, *z*.

Table 3: Selected Bond lengths (Å) and Bond angles (º)

Atom	**Bond length(Å)**	**Atom**	**Bond Angle (°)**
C(1)-C(14)	1.345(6)	C(14)-C(1)-C(13)	125.6(4)
C(1)-C(13)	1.489(7)	C(14)-C(1)-C(2)	129.9(4)
C(1)-C(2)	1.497(7)	C(13)-C(1)-C(2)	104.2(4)
C(2)-C(3)	1.384(7)	C(3)-C(2)-C(7)	119.4(4)
C(2)-C(7)	1.398(6)	C(3)-C(2)-C(1)	131.4(5)
C(3)-C(4)	1.384(7)	C(7)-C(2)-C(1)	108.9(4)
C(4)-C(5)	1.379(8)	C(2)-C(3)-C(4)	119.2(5)
C(5)-C(6)	1.366(8)	C(5)-C(4)-C(3)	120.6(5)

C(6)-C(7)	1.378(7)	C(6)-C(5)-C(4)	120.7(5)
C(7)-C(8)	1.458(7)	C(5)-C(6)-C(7)	119.4(5)
C(8)-C(9)	1.391(7)	C(6)-C(7)-C(2)	120.6(5)
C(8)-C(13)	1.399(7)	C(6)-C(7)-C(8)	130.4(5)
C(9)-C(10)	1.364(8)	C(2)-C(7)-C(8)	109.0(4)
C(10)-C(11)	1.377(8)	C(9)-C(8)-C(13)	120.9(5)
C(11)-C(12)	1.382(7)	C(9)-C(8)-C(7)	130.5(5)
C(12)-C(13)	1.397(7)	C(13)-C(8)-C(7)	108.5(4)
C(14)-C(15)	1.486(6)	C(10)-C(9)-C(8)	119.2(5)
C(14)-C(22)	1.534(6)	C(9)-C(10)-C(11)	120.6(5)
C(15)-C(20)	1.376(6)	C(10)-C(11)-C(12)	121.2(5)
C(15)-C(16)	1.424(7)	C(11)-C(12)-C(13)	119.1(5)
C(16)-C(17)	1.366(6)	C(12)-C(13)-C(8)	118.7(5)
C(17)-O(2)	1.367(6)	C(12)-C(13)-C(1)	131.2(4)
C(17)-C(18)	1.393(7)	C(8)-C(13)-C(1)	109.4(4)
C(18)-O(1)	1.364(6)	C(1)-C(14)-C(15)	126.1(4)
C(18)-C(19)	1.379(7)	C(1)-C(14)-C(22)	122.4(4)
C(19)-C(20)	1.400(6)	C(15)-C(14)-C(22)	111.4(4)
C(20)-C(21)	1.505(7)	C(20)-C(15)-C(16)	118.2(4)

Conclusion

The crystal structure analysis of a novel the asymmetric unit consisting of the two independent molecules (A and B) of the title compound. The two molecules have the same geometrical parameters within the precision of the experiment. The compound was studied using X-ray diffraction method. In the crystal, the A and B molecules are linked via pairs of C11-H11...O2 hydrogen bonds, forming chains. The chains are linked via C45-H45...O3 hydrogen bonds, forming slabs lying parallel to (010).

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