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CRYSTAL STRUCTURE OF 4-METHYL-3-PHENYL-6-(4-PHENYL-5*H*-INDENO[1,2-*B*]PYRIDIN-2-YL)COUMARIN CHLOROFORM MONOSOLVATE

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ABSTRACT

The title compound, 4-Methyl-3-phenyl-6-(4-phenyl-5*H*-indeno[1,2-*b*]pyridin-2-yl)coumarin chloroform monosolvate, $C_{34}H_{23}NO_2.CHCl_3$, was synthesized and characterized by spectroscopic techniques such as 1H-NMR, 13C-NMR, IR and mass spectroscopy. In addition to that the title compound was also characterized by single crystal x-ray diffraction method. The compound crystallizes in the monoclinic space group *P*21/*n* with unit-cell parameters: a = 9.2340(2), b = 14.1626(3), c = 22.2540(5)Å, α = 90, β = 100.121(2), γ = 90˚, Z = 4. The crystal structure was solved by direct methods using single-crystal X-ray diffraction data, and refined by full-matrix least-squares procedures to a final *R*-value of 0.0695 for 4979 observed reflections.

KEYWORDS: Indeno[1,2-*b*]pyridine, coumarins, crystal structure, antimicrobial activity

INTRODUCTION

For decades, various heterocyclic bearing coumarin derivatives have been exploited by medicinal chemists in order to achieve desirable biological properties. Among the heterocyclic substituted coumarins, pyridine substituted coumarins constitute an elite class of compounds as they exhibit important biological activities such as CNS depressant[1], antifungal[2], antibacterial[3] etc. On the other hand, there are pronounced reports expressing that heterocycles bearing modified pyridine moieties such as indeno[1,2-*b*]pyridine display

important biological properties such as cytotoxicity[4], calcium antagonistic[5], inhibition of bovine liver glutathione S-transferase[6], antimicrobial[7] etc. Hence, it was encouraging to synthesize molecules having both the aforementioned moieties in anticipation of synergistic biological response. Thus, various indeno[1,2-*b*]pyridinylcoumarins were synthesized and characterized by various spectroscopic techniques and were also screened for their antimicrobial activity.[8]

In this study the x-ray single crystal structure of 4-methyl-3-phenyl-6-(4-phenyl-5*H*-indeno[1,2-*b*]pyridin-2-yl)coumarin chloroform monosolvate was analysed.

EXPERIMENTAL

Synthesis. The title compound (**Figure 1**), $C_{34}H_{23}NO_2.CHCl_3$, was synthesized by our laboratory using reported method.[8]

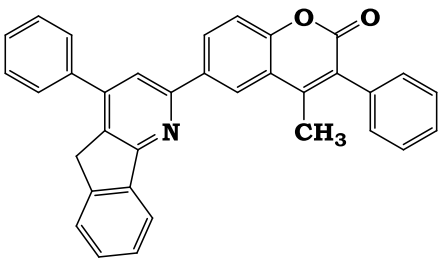

Figure 1: Molecular structure of title compound.

X-ray diffraction study. Colorless crystals of suitable size of the title compound were obtained by slow evaporation of chloroform solution. Single crystal X-ray diffraction intensity data of the compound was collected at 293 K using Oxford X-CALIBUR-S CCD diffractometer with Cu-Kα radiation (λ = 1.54184 Å) at 293 K. Data reduction was carried out using the program CrysAlisPro, Agilent Technologies, Version 1.171.35.19. An absorption correction based on multi-scan method[9] was applied. The structures were solved by direct methods and refined by the full-matrix least-square technique on F^2 using the programs SHELXS-97 and SHELXL-97[10] respectively. All calculations were carried out using WinGX system Ver-1.64.[11] All hydrogen atoms were located from difference Fourier map and treated as riding. All non hydrogen atoms were refined with anisotropic displacement coefficients.

RESULTS AND DISCUSSIONS

The details of data collection and some important features of the refinement for the compound are given in **Table 1**.

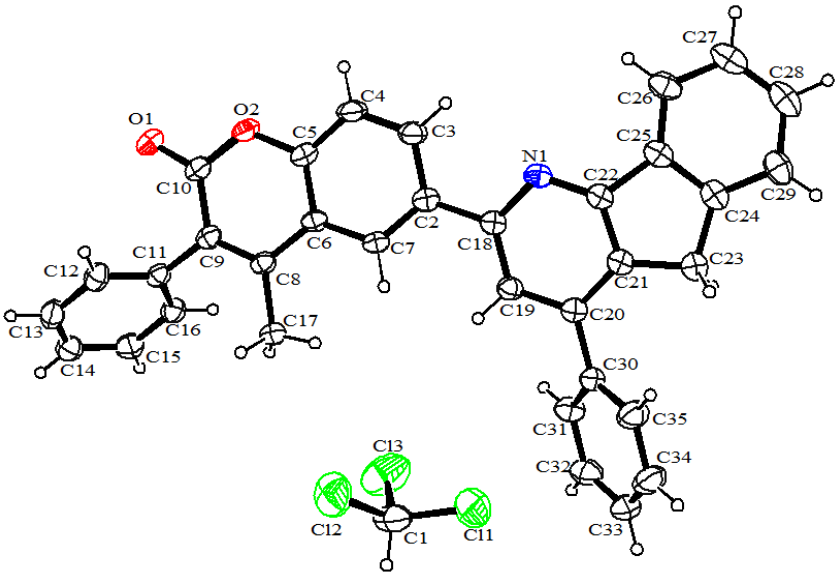

Figure 2: ORTEP diagram of title compound with 30% probability factor for the thermal ellipsoids.

Table 1: Crystal and experimental data.

Empirical formula	$C_{35}H_{24}NCl_3O_2.CHCl_3$
Formula weight	596.90
Temperature (K)	293(2)
Wavelength (Å)	1.54184
Crystal system	Monoclinic
Space group	$P2_1/C$
a (Å)	9.2340(2)
b (Å)	14.1626(3)
c (Å)	22.2540(5)
α(°)	90
β(°)	100.121(2)
γ(°)	90
Volume (Å^3)	2865.03(11)
Z	4
Density (mg/m^3)	1.384
Absorption coefficient (mm^{-1})	3.164
F(000)	1232
Theta range for data collection (°)	4.8670 to 73.1090
Index ranges	$-11\leq h\leq 11$, $-17\leq k\leq 11$, $-25\leq l\leq 27$
Reflections collected	17693

Independent reflections	5722 [R(int) = 0.0246]
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.642 and 0.510
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5722 / 0 / 370
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0695, wR2 = 0.1980
R indices (all data)	R1 = 0.0759, wR2 = 0.2052
Largest diff. peak and hole (eA^{-3})	0.534 and -0.598
CCDC deposition number	CRM:0001000199442

Some selected bond lengths and angles are given in **Table 2**. The bond lengths and bond angles observed in the structure are comparable with those reported for other coumarin derivatives. The aromatic C-C bond lengths in the phenyl rings ranges between 1.360(5)-1.405(3) Å. The values of the two C-O bonds, (O_2-C_5 = 1.375(3); O_2-C_{10} = 1.369(3) Å) in the pyrone ring is in agreement with the C(sp2)-O distance. The double bond character of O_1-C_{10} is confirmed by the distance 1.209(3) Å. The bond angles, O_2-C_5-C_4 and C_7-C_6-C_8, at the junction of the pyrone and benzene rings are, respectively, smaller and greater than 120˚. This phenomenon has also been observed in some analogous coumarin derivatives. The widening of the angle O_1-C_{10}-C_9 (125.7(3) Å) is attributed to lone-pair interactions between O_1 andO_2. This is also indicated by slight lengthening of the O_1=C_{10} double bond.

Table 2: Selected bond distances (Å), bond angles (˚) and torsion angles (˚).

Bond	**(Å)**	**Bond**	**(Å)**
O2-C5	1.375(3)	C8-C17	1.502(3)
O2-C10	1.369(3)	C15-C14	1.384(5)
C6-C8	1.454(3)	C9-C10	1.463(3)
O1-C10	1.209(3)	C23-C21	1.507(4)
N1-C18	1.354(3)	C24-C29	1.388(4)
Bond Angle	**(˚)**	**Bond Angle**	**(˚)**
O2-C5-C4	117.4(2)	C24-C23-C21	102.6(2)
C7-C6-C8	123.9(2)	N1-C22-C25	125.7(2)
O1-C10-C9	125.7(3)	O1-C10-O2	116.1(2)
C22-C1-C18	115.8(2)	C15-C16-C11	120.5(3)
N1-C18-C2	116.2(2)	C19-C20-C30	121.5(2)
Torsion angle	**(˚)**	**Torsion angle**	**(˚)**
C12-C11-C9-C8	119.8(3)	C8-C6-C7-C2	177.0(2)
N1-C18-C2-C7	-177.3(2)	C26-C25-C22-C21	-178.2(3)
C23-C24-C29-C28	-176.7(3)	C31-C30-C20-C19	46.4(4)
C35-C30-C20-C19	-134.3(3)	C19-C18-C2-C7	4.1(4)
C11-C9-C8-C17	-8.8(4)	C10-C9-C8-C6	-7.8(3)

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