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Synthesis and X-ray Crystallography of 2-(2-(4-Methoxyphenyl)-4,5-diphenyl-1H-imidazol-1-yl)-5-methylpyridine

Amit Kumar[1], A. Jayashree[2], B. Narayana[2], B. K. Sarojini[3], Sumati Anthal[1], Rajni Kant[1*]

[1]*X-ray Crystallography Laboratory, Post-Graduate Department of Physics & Electronics, University of Jammu, India*
[2]*Department of Studies in Chemistry, Mangalore University, Mangalagangotri-574 199, India*
[3]*Department of Industrial chemistry, Chemistry, Mangalore University, Mangalagangotri-574 199, India; Mangalore 574 153, India*

Abstract:*2-(2-(4-Methoxyphenyl)-4,5-diphenyl-1H-imidazol-1-yl)-5-methylpyridine, crystallizes in the orthorhombic space group $Pca2_1$ with unit cell parameters: a= 19.0002(15), b= 6.0827(4), c= 18.6173(14). The number of molecules per unit cell (Z)=4. The crystal structure has been solved by direct methods and refined by full matrix least squares procedures to a final R value of 0.0425 for 2392 observed reflections. Central imidazole ring in the structure is planar. The methylpyridine and methoxyphenyl rings are in synperiplanar conformation. No classic Hydrogen bonds were observed.*

Keywords: *Crystal structure, Direct methods, Hydrogen bond, Imidazole.*

I. Introduction

Pyridine scaffold possess potential biological activities like antimalarial, antioxidant, anticonvulsant, anesthetic, antioxidant, antibacterial and antiparasitic properties [1-3]. It is reported that some important anticancer drugs possess pyridine nucleus [4]. Tetrasubstituted imidazoles are important heterocycles with numerous biological applications and it is most active constituent in many biological systems and drug molecules [5]. So, promising biological activities can be expected for pyridines containing imidazole moiety. It has been reported that compounds containing pyridine and imidazole scaffolds display improved efficacy and decrease the adverse effect and are potential in developing new anticancer drugs [4]. In view of the numerous biological and pharmacological applications associated with pyridine containing imidazole moiety, we report the synthesis and crystal structure of the 2-(2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazol-1-yl)-5-methylpyridine.

II. Experimental

2.1. Synthesis of 2-(2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazol-1-yl)-5-methylpyridine

A mixture of benzil (0.210 g, 1 mmol), 2-amino-5-methylpyridine (0.108 g, 1 mmol), *p*-anisaldehyde (0.136 g, 1 mmol), ammonium acetate (0.77 g, 1 mmol) and ZnO nanoparticles (0.008g, 0.1 mmol) in 25 ml glacial acetic acid was heated at 60 ^{o}C under stirring for around 2 hrs. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature and then poured into cold water with constant stirring. The solid separated was filtered by suction to afford crude product. The pure product was obtained by further recrystallization from ethanol. Single crystal of the purified product suitable for X-ray analysis was developed from ethanol by slow evaporation method (M.P.: 473-475 K). The synthetic route of the compound is shown in Scheme 1.

2.2. X-ray data collection and structure refinement

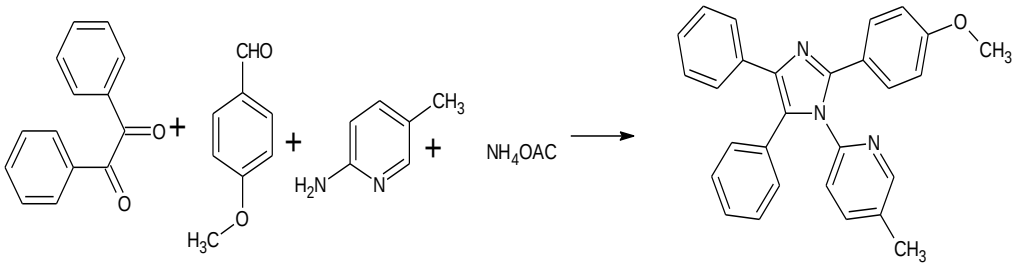
A crystal of dimensions 0.30x0.20x0.20 mm was used for data collection on X'calibur CCD area-detector single crystal X-ray diffractometer equipped with graphite monochromated MoKα radiation (λ=0.71073 Å). X-ray intensity data consisting of 5300 reflections were collected at 293(2) K and out of these reflections 2980 were found to be unique. The intensities were measured by ω–scan mode for θ ranging between 3.68 to 26.00°. A total number of 2392 reflections were treated as observed [I>2σ(I)]. Data were corrected for Lorentz-polarization and absorption factors. The structure was solved by direct methods using SHELXS-97 [6]. All non-hydrogen atoms of the molecule were located in the best E-map. All the hydrogen atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms with C-H= 0.93-0.96 Å and U_{iso}= 1.2 U_{eq}(C), except for the methyl groups where U_{iso}(H) = 1.5U_{eq}(C). The final refinement cycles converges to an R- factor of 0.0425 (wR(F2) = 0.0968) for 2392 observed reflections. Residual electron density ranges from -0.164 to 0.138 eÅ^{-3}.Geometrical calculations of the molecule was done using WinGX [7], PARST

[8] and PLATON [9] softwares.Crystallographic information has been deposited to Cambridge Crystallographic Data Centre with CCDC number 1505844. This data can be obtained free of charge at Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

III. Results and Discussion

The crystallographic data so obtained after performing the single crystal XRD analysis is summarized in Table 1. The molecule containing atomic labelling is shown in Fig 1. (ORTEP) [10] and the packing diagram as generated using PLATON is shown in Fig 2. Selected bond lengths, bond angles and torsion angles of the titled compound are enlisted in Table 2. The structural parameters including bond distances and bond angles show normal geometry [11]. The molecule consists of two phenyl rings, one methoxyphenyl ring, one methylpyridine ring and the central imidazole ring. The values for exocyclic and endocyclic bond angles at C8, C9, N2 and C16 are in agreement with those existing in the literature [12-13]. The dihedral angle between the central imidazole ring with phenyl ring A (C10-C15) and ring B (C17-C22) is 31.16(1)° and 71.18(1)°, respectively, while it is 77.25(1)° with methylpyridine and 25.44(1)° with methoxyphenyl ring. These values differ significantly in case of some analogous structures [12-13]. However, the remaining bond distances and bond angles are in agreement with the values observed for some analogous imidazole derivatives [12-13]. All the five rings are planar with a maximum deviation of -0.0225(3) Å observed for the C2. The methylpyridine and methoxyphenyl rings are in *synperiplanar* conformation as indicated by the torsion angle of -15.4(4)° about the atoms C23-N2-C8-C5. Molecular packing in the unit cell as viewed down the b-axis is shown in Fig 2. No classical hydrogen bonds were observed and only van der Walls forces stabilize the crystal packing.

IV. Figures and Tables



Scheme 1. Reaction Scheme shows the synthesis of 2-(2-(4-MethoxyphenPhenyl)-4,5-diphenyl-1H-imidazol-1-yl)-5-methylpyridine.

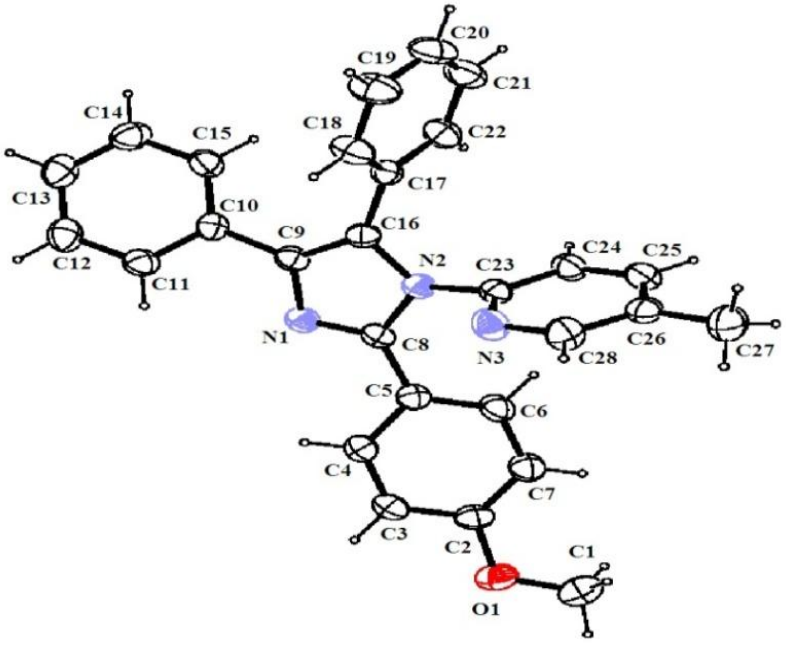


Figure 1. ORTEP view of molecules with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii.

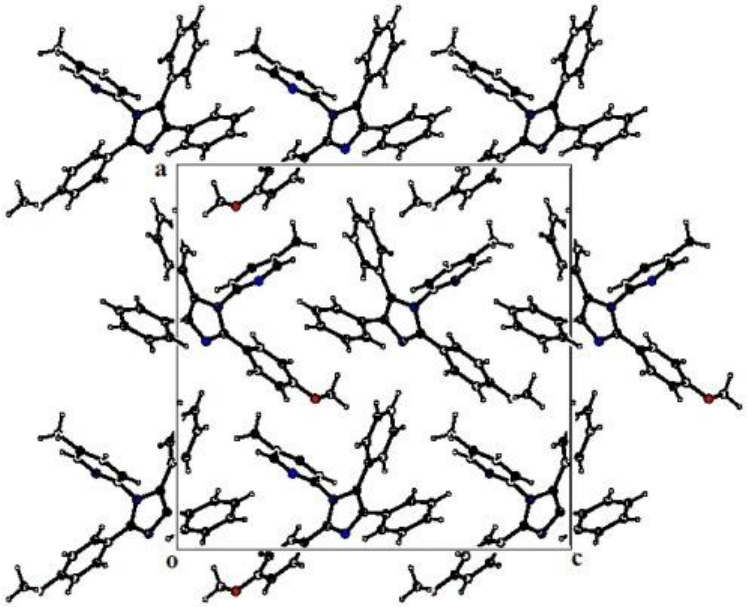


Figure2. The crystal packing viewed down the b-axis**.**

Table 1. Crystal data and other experimental details.

CCDC Number	1505844
Crystal description	block
Crystal size	0.30 x 0.20 x 0.10 mm
Empirical formula	$C_{28}H_{23}N_3O$
Formula weight	417.49
Radiation, Wavelength	Mo *K*α, 0.71073 Å
Unit cell dimensions	a = 19.0002(15)Å, b = 6.0827(4)Å, c = 18.6173(14)Å,
Crystal system, Space group	orthorhombic, $Pca2_1$
Unit cell volume	2151.6(3)$Å^3$
No. of molecules per unit cell, Z	4
Absorption coefficient	0.077 mm^{-1}
F(000)	856
θ range for entire data collection	3.68< θ < 26.00
Reflections collected / unique	5300/2980
Reflections observed (I > 2σ(I))	2392
Range of indices	*h*= -22 to 23, *k*= -7 to 6, *l*= -10 to 22
No. of parameters refined	292
Final R-factor	0.0425
wR(F2)	0.0968
R_{int}	0.0293
R_{sigma}	0.0425
Goodness-of-fit	1.096
$(\Delta/\sigma)_{max}$	0.000
Final residual electron density	-0.164 < Δ*ρ* > 0.138 $eÅ^{-3}$

Table 2. Selected bond lengths (Å), bond angles (º) and torsion angles (º) for non hydrogen atoms (e.s.d.'s are given in parentheses)

Bond lengths		**Bond angles**		**Torsion angles**	
N1-C9	1.371(4)	N2-C8-C5	124.9(3)	N1-C8-C5-C6	155.1(3)
N1-C8	1.312(4)	N1-C8-C5	124.1(3)	N2-C16-C17-C22	72.4(4)
N2-C8	1.380(4)	O1-C2-C7	125.3(3)	O1-C2-C7-C6	-178.3(3)
N2-C16	1.387(4)	O1-C2-C3	115.2(3)	C1-O1-C2-C7	6.3(5)
O1-C1	1.414(4)	C16-N2-C23	125.2(3)	N2-C8-C5-C4	154.3(3)
O1-C2	1.365(4)	C10-C9-N1	120.4 (3)	N2-C16-C17-C18	-108.5(3)
N3-C23	1.330(4)	C2-O1-C1	117.7(3)	C15-C10-C9-N1	-149.1(3)
N3-C28	1.339(4)	N2-C23-N3	115.0(2)	C28-N3-C23-N2	176.7(3)
C26-C27	1.490(5)	N2-C16-C17	121.6(3)	C11-C10-C9-C16	-148.3(3)

V. Conclusion

Synthesis of compound was achieved by reaction mixture of benzil, 2-amino-5-methylpyridine, *p*-anisaldehyde, ammonium acetate and ZnO nanoparticles. Single crystal of the purified product suitable for X-ray analysis was developed from ethanol by slow evaporation method. The molecular and crystal structure of the given compound was determined using single crystal X-ray diffraction data collected at 293(2) K with a final R value of 0.0425. No classic Hydrogen bonds were found in the structure. CCDC 1505844 contains the supplementary crystallographic data for this paper.

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