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Synthesis and Characterization of Diethyl 4-Amino-1-(3-Chlorophenyl)-1H-Pyrazole-3,5 Dicarboxylate

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***Abstract*—** In the present study, Diethyl 4-amino-1-(3-chlorophenyl) -1H-pyrazole-3,5 dicarboxylate (D4A1(3-cl)1HP35D) was synthesized. The structure of newly synthesized compounds of Diethyl 4-amino-1-(3-chlorophenyl -1H-pyrazole-3,5 dicarboxylate was characterized by x-ray diffraction (XRD), Fourier transforms infrared spectroscopy (FTIR) and Transmission electron microscopy (TEM). The compound has been prepared by the chemical root method and crystal structure was determined by x-ray diffraction technique. The XRD pattern of (D4A1(3-cl)1HP35D) confirms the crystalline nature and (h k l) values are indexed. From the experimental analysis, the presence of functional groups in the synthesized compound and particle size of the compound has been estimated by FTIR and TEM spectroscopy.

Keywords— *Pyrazole, Synthesis, XRD, FTIR, TEM spectroscopy.*

I. INTRODUCTION

Our interest in compound with pyrazole based compounds has lasted more than a decade, due to their attractive physicochemical characteristic [1-3]. The pyrazole compounds are the important class of nitrogen-containing heterocyclic compounds. Heterocyclic compounds are generally known for their significance in the pharmaceutical industry [4]. Many pyrazole derivatives exhibit biological properties such as antitumor, anticoagulant, antihyperglycemic, analgesic, anti-inflammatory, antipyretic, antibacterial, hypoglycemic and sedative-hypnotic activity [5-6].

In our paper, we present the synthesis of Diethyl4-amino-1-(3-chlorophenyl)-1H-pyrazole-3,5 dicarboxylate (D4A1(3-cl)1HP35D). Powder x-ray diffraction parameters suggested a monoclinic system for the compound. FT-IR spectroscopy provides information about identifying unknown materials, determine the quality or consistency of a sample and determine the number of components in a mixture. And the particle size of the samples calculated from TEM images. The synthesis was done at School Of Chemical Science, DAVV, Indore and All spectra have been recorded at UGC-DAE-CSR, Indore, India.

II. EXPERIMENTAL DETAILS

The compound (D4A1(3-cl)1HP35D) was prepared by the chemical root method. The X-ray diffraction pattern of the title compound has been recorded using Rigaku RINT-2000 X-ray diffractometer with a rotating anode with a tube voltage of 40 kV and a current of 100 mA. Copper target was used as the source of X-rays at wavelength λ=1.54Å. The infrared absorption spectra of the compound were measured at room temperature, in the wavenumber range 4000 to 400 cm-1 on the Jasco FTIR-300 spectrometer using the KBr pellet technique. TEM images have been obtained on Tecnai (300 kV), FEI, Holland. It has a LaB6 an electron gun, which can be operated between 50 to 300 kV.

III. RESULTS AND DISCUSSION

A. Synthesis

The Diethyl 4-amino-1-(3-chlorophenyl -1H-pyrazole-3,5 dicarboxylate (D4A1(3-cl)1HP35D) were synthesized by the chemical root method. Pertinent aniline derivatives were dissolving in distilled water and kept at freezing temperature in the refrigerator. To this, an aqueous solution of sodium nitrite in distilled water was added with continuous stirring keeping the temperature in the vicinity of 0 - 5ºC. Meanwhile, in another beaker ethyl cyanoacetate, sodium acetate and distilled water were taken and cooled in an ice bath. The reaction mixture was kept for overnight period, filtered through suction, washed with water and dried in vacuum. Fine crystals of the compound were obtained, which were recrystallized from ethyl alcohol. To a solution of couple product, Triethylamine and Ethyl Bromo Acetate were added. The reaction mixture was then refluxed, and

then poured onto ice-cold water and HCl, the solid product so formed was filtered off and crystallized from ethanol [7].

B. X-Ray Diffraction

The x-ray powder diffraction data of the Diethyl 4-amino-1-(3-chlorophenyl) -1H-pyrazole-3, 5 dicarboxylate (D4A1(3-cl)1HP35D) compound was recorded at room temperature using Cu Kα radiation. The XRD analysis of the compound was carried out to determine the type of crystal system, lattice parameters and the cell volume. As shown in figure 1 the XRD pattern indicates a crystalline nature for the compound. The diffraction data of the title compound are listed in the table. The diffraction patterns of samples recorded between 2θ ranging from 0° to 40°. The compound has monoclinic crystal lattice with unit cell volume V = 892.10 × 10-8 cm3. The unit cell parameters are a = 13.92 Å, b = 6.68 Å, c = 9.64 Å, and β= 94.60˚ [8]. Inter-planar d spacing and unit cell volume of the synthesized compound was calculated by the formula:

$$\frac{1}{d^2} = \frac{1}{\sin^2\beta}\left[\frac{h^2}{a^2} + \frac{k^2\sin^2\beta}{b^2} + \frac{l^2}{c^2} + \frac{2hl\cos\beta}{ac}\right]$$

And volume is given by V = abc

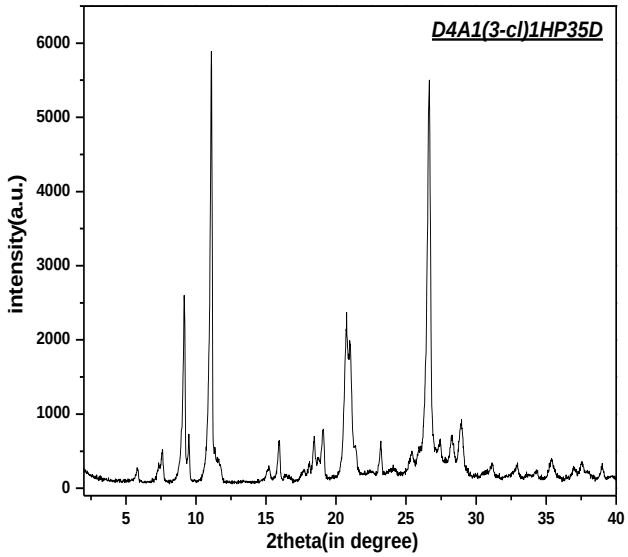

Figure 1 : X-ray diffraction pattern of D4A1(3-cl)1HP35D

Table shows the experimental data and the calculated data for the X-ray diffraction pattern of the D4A1(3-Cl)1HP35D. The prominent peaks of the compound have been identified, which are equal and in some cases nearly match the standard diffraction JCPDS CARD NO. (100754 - 441628)

2 theta (in degree)	d-spacing (in Å) observed	d-spacing (in Å) calculated	Relative intensity (%)	h	k	l
9.16	9.64	9.59	44.04	0	0	1
11.10	7.96	8.27	100	-1	0	1
15.96	4.54	5.00	11.26	1	1	1
19.08	4.64	4.61	13.59	3	0	0
20.76	4.27	3.9	40.25	0	1	2
23.19	3.83	4.41	10.41	-2	1	1
26.65	3.34	3.33	93.27	0	2	0

C. Fourier Transforms Infrared Spectroscopy (FTIR)

FTIR is the most powerful analytical tool for identifying the type of chemical bonds (functional groups) present in compounds. The IR spectra of the Diethyl 4-amino-1-(3-chlorophenyl) -1H-pyrazole-3,5 dicarboxylate (D4A1(3-cl)1HP35D) were taken in the range between 4000–400 cm^{-1} in KBr pellets. IR spectrum of the synthesized compound shown in Figure 2. By interpreting the infrared absorption spectrum of the compound shows that a band in the region 3217-3230 cm^{-1} due to (N-H stretching). Similarly band in the region 1740-1685 cm^{-1} due to (C=O stretching), 1240-1270 cm^{-1} due to (C-O stretching) and 785-775 cm^{-1} due to (C-Cl stretching) [8-9].

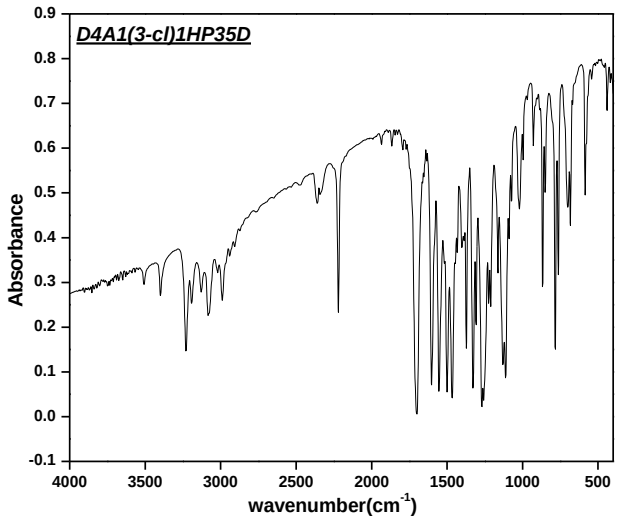

Figure 2 : FTIR spectra of D4A1(3-cl) 1HP35D

D. Transmission Electron Microscopy (TEM)

The TEM images of the samples are shown in figure 3. The structure and morphology of the compound were further confirmed by the TEM image. The values of the particle size of the samples calculated from TEM images of the compound are found to 56.03 nm**.**

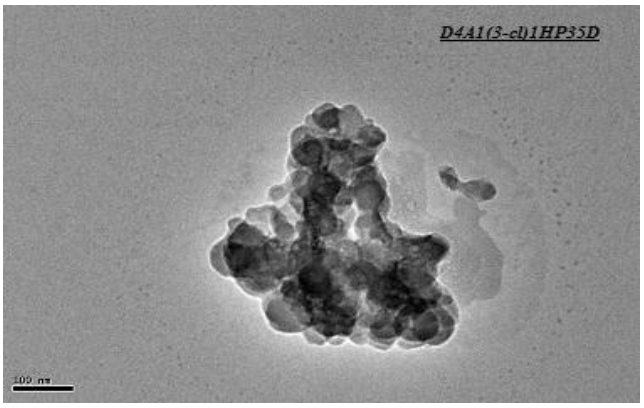

Figure 3: TEM image of D4A1(3-cl)1HP35D

IV. CONCLUSION

The synthesized compound Diethyl 4-amino-1-(3-chlorophenyl -1H-pyrazole-3,5 dicarboxylate was characterized by XRD, FTIR and TEM spectroscopy. From XRD results, it was found that the compound has monoclinic structure and the structural characteristics like lattice parameters, lattice angle and unit cell volume of the compound have been found from crystallographic data. The presence of various functional groups of the compound is identified using Fourier transform infrared spectroscopy studies. The morphology of the compound was characterized by Transmission electron microscopy.

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