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Synthesis, spectroscopic and X-ray crystallographic analysis of *N*-(2-(2-(4-chlorophenoxy)acetamido)phenyl)-1*H*-indole-2-carboxamide

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ABSTRACT

Medicinal chemistry of indole analogs constitutes important therapeutic agents with anti-oxidant, anti-HIV and anti-cancer activities. Indole nucleus is frequently found in synthetic and natural products, pharmaceuticals, functional materials, agrochemicals, etc. The title compound, *N*-(2-(2-(4-chlorophenoxy)acetamido)phenyl)-1*H*-indole-2-carboxamide (5), has been synthesized in good yield by stirring the compound *N*-(2-aminophenyl)-2-(4-chlorophenoxy)acetamide (3) with 1*H*-indole-2-carboxylic acid (4), in dry dichloromethane followed by the addition of 2,6-lutidine, and *o*-(benzotriazol-1-yl)-*N*,*N*,*N*',*N*'-tetramethyl uraniumtetrafluoroborate in cooled condition. Compound 5 was synthesized and characterized by the conventional spectroscopic techniques (^{1}H NMR, ^{13}C NMR and LC-MS) and the three-dimensional structure was elucidated by using single crystal X-ray diffraction methods. It crystallizes in the monoclinic crystal system with space group $P2_1/c$. The structure was solved by direct methods and refined by full matrix least square procedure to a final R value of 0.043 for 2490 observed reflections. Three intra-molecular interactions of the type N-H···N and C-H···N were observed. The packing of molecules in the unit cell is governed by N-H···O and C-H···O intermolecular H-boned interactions which leads to the formation of infinite staking chain along [001] direction. In addition, two weak C-H···π interactions also contribute to molecular packing.

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1. Introduction

In drug discovery, medicinal chemists use the privileged structures to synthesize a novel compounds based on a central scaffold and to screen them against different receptors involved in various pathways, in other cases producing biologically active compounds. Indole nucleus is often found in medicinal chemistry world with unique properties due to the presence of a rich-in-electron at pyrrole moiety [1] that can use non-covalent interactions with other molecules by formation of hydrogen bonding in the NH moiety by π-π system [2] and is considered as privileged structures [3,4]. Due to this unique property, indole and its derivatives are used broadly for new drugs therapy development [5]. Therefore, due and considerable attention has been given to the development of synthetic methods for the preparation of such materials and their structure elucidation for its possible pharmaceutical properties, *viz*. anti-histaminic, anti-inflammatory [6,7], anti-oxidant [8], anti-rheumatoid, anti-HIV [9,10] and anti-cancer activity [11-14]. Indole and its derivatives have also played a vital role in the field of immunology [15,16]. Furthermore, it is considered as the most potent scavenger of free radicals [17]. Different studies involving *in vitro* and *in vivo* inhibition activity have shown that compounds with indole moiety can effectively inhibit the diabetic activity [18,19]. The design and selective functioning of indoles have been the center of the current investigation over the years [20-24]. In view of their broad spectrum of biological properties and as a part of our ongoing work on synthesis and characterization of indole derivatives [25,26], the synthesis, spectroscopic and X-ray crystallographic analy-

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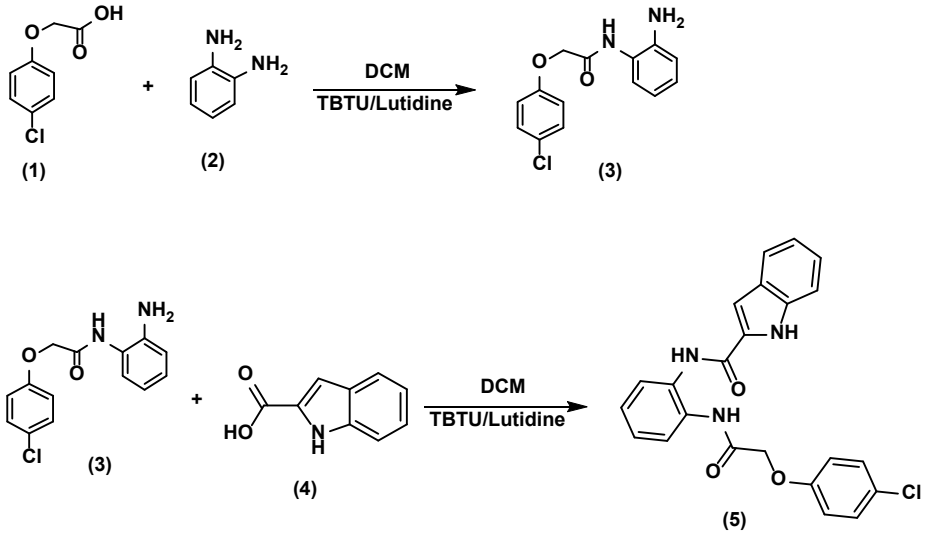

Scheme 1. Reaction pathway for the synthesis of compound **5**.

sis of *N*-(2-(2-(4-chlorophenoxy)acetamido)phenyl)-1*H*-indole-2-carboxamide is reported in this paper.

2. Experimental

2.1. Materials and instrumentations

Chemicals, solvents and agents were purchased from Sigma Aldrich. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel plates (Merck 60 F_{254}) by using solvent system using hexane: ethyl acetate (9:3, *v*:*v*). Melting point was determination using the Chemi Line CL725 Micro Controller Based melting point apparatus with a digital thermometer. The NMR spectrum (^{1}H NMR and ^{13}C NMR) was recorded on a VNMRS-400 MHz Agilent-NMR spectrophotometer in DMSO-d_6. Mass spectra was obtained with a VG70-70H spectrometer. Elemental analysis (Elementar Vario EL III elemental analyzer) results are within 0.5% range of the calculated values. The three dimensional molecular structure of the compound was confirmed by single crystal X-ray diffraction technique.

2.2. Synthesis of N-(2-aminophenyl)-2-(4-chlorophenoxy) acetamide (3)

The title compound *N*-(2-aminophenyl)-2-(4-chloro phenoxy)acetamide (**3**) was obtained by the synthetic procedure as shown in Scheme 1. To compound **1** (0.8 g, 0.009 mol), in dry DCM (10 mL), 2,6-lutidine (1.3 vol.) was added at 25-30 °C, followed by the addition of 1,2-diaminobenzene **2** (1.5 g, 0.009 mol), the reaction mixture was stirred at 25-30 °C for 25 min. The reaction was cooled to 0-5 °C and 2-(1*H*-benzotriazole-1-yl)-1, 1, 3, 3-tetramethylaminium tetrafluoro borate (TBTU) (4.5 g, 0.02 mol) was added over a period of 30 minutes while maintaining the temperature below 5 °C. The reaction was stirred overnight and monitored by TLC using mobile phase system (hexane: ethyl acetate, 9:3, *v*:*v*). The reaction mixture was diluted with 25 mL of DCM and treated with 2.0 N HCl solution (20 mL). The organic layer was washed with water (3 × 25 mL) and brine (3 × 25 mL). Finally, the organic layer was dried over anhydrous sodium sulfate and concentrated to afford compound **3** [27,28].

2.3. Synthesis of N-(2-(2-(4-chlorophenoxy)acetamido) phenyl)-1H-indole-2-carboxamide (5)

To the compound of *N*-(2-aminophenyl)-2-(4-chloro phenoxy)acetamide **3** (0.5 g, 0.002 mol), in dry DCM (10 mL), 1*H*-indole-2-carboxylic acid **4** (0.3 g, 0.002 mol), was added at 25-30 °C, followed by the addition of 2,6-lutidine (0.001 mol). The reaction mixture was stirred at 25-30 °C for 30 minutes. The reaction was cooled to 0-5 °C, TBTU (0.9 g, 0.003 mol) was added over a period of 30 min while maintaining the temperature below 5 °C. The reaction was stirred overnight and monitored by TLC mobile phase system (hexane: ethyl acetate, 9:3, *v*:*v*). The reaction mixture was diluted with (25 mL) of DCM and treated with 10% of sodium bicarbonate solution (3×25 mL). The organic layer was washed with water (3×25 mL), dried over anhydrous sodium sulfate and concentrated to yield compound **5** [29,30]. The crude product upon re-crystallization with ethanol afforded the title compound **5** as colorless rectangular block shape crystals and then confirmed by ^{1}H NMR, ^{13}C NMR, LC-MS spectra. The schematic diagram of the synthesized compound is shown in Scheme 1.

N-(2-(2-(4-chlorophenoxy)acetamido)phenyl)-1H-indole-2-carboxamide (**5**): Color: White. Yield: 85%. M.p.: 205-207 °C. FT-IR (KBr, ν_{max}, cm^{-1}): 1633 (amide, C=O), 3210-3320 (amide CO-NH). ^{1}H NMR (400 MHz, DMSO-d_6, δ, ppm): 4.71 (s, 2H, OCH_2), 6.89-7.77 (s, 13H, Ar-H), 9.65 (s, 1H, NH), 10.18 (s, 1H, NH), 11.83 (s, 1H, NH indole). ^{13}C NMR (100 MHz, DMSO-d_6, δ, ppm): 166.96, 160.65, 156.44, 137.40, 131.51, 131.10, 130.10, 129.67, 129.48, 127.49, 126.43, 126.37, 125.83, 125.55, 124.44, 122.25, 120.48, 116.87, 116.63, 112.90, 104.70, 67.76. LC-MS (*m/z*): 420 [M+], 422 [M+2].

2.4. X-ray intensity data collection, structure solution and refinement

A block-shaped single crystal with good surface morphology was chosen for intensity data collection. The data were collected on Bruker Kappa *APEX*-II four circle-CCD diffractrometer [31] using graphite monochromated MoK_α radiation (λ = 0.71071 Å). A total of 26028 reflections were collected at 296 K out of which 4086 reflections were found unique. The structure solution was carried out by Direct Methods using SHELXS [32] and the non-H atoms were located in the best E-map. Full matrix least square refinement procedure resulted the structure to converge to a final R-value of 0.043 for 2490 observed reflections (I>2σ(I)) using SHELXL [33].

Table 1. Crystal data and experimental details:

Crystal data	
Chemical formula	$C_{23}H_{18}ClN_3O_3$
M_r	419.85
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	11.171 (2), 21.929 (5), 9.307 (2)
β (°)	114.257 (14)
V (Å^3)	2078.7 (9)
Z	4
Radiation type	Mo K_α
μ (mm^{-1})	0.21
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker *APEX*-II CCD
Absorption correction	Multi-scan *SADABS* (Sheldrick, 1996)
T_{min}, T_{max}	0.885, 0.959
No. of measured, independent and observed [I> 2σ(I)] reflections	26028, 4086, 2490
R_{int}	0.050
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2> 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.118, 1.00
No. of reflections	4086
No. of parameters	271
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.28

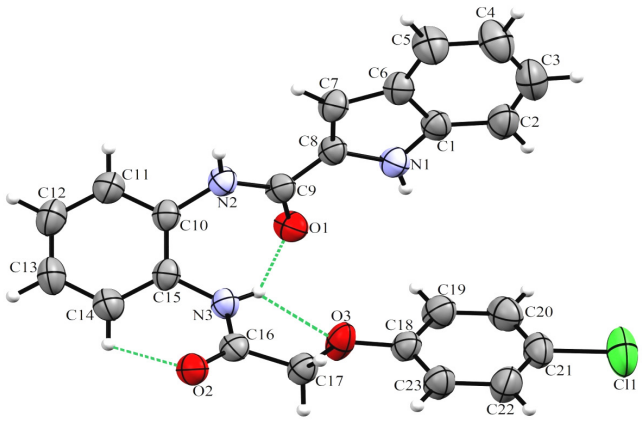

Figure 1. ORTEP view of the molecule with the ellipsoid probability at 40% (dashed lines represents intra-molecular H-bonded interactions).

All the hydrogen atoms were fixed geometrically and allowed to ride on their corresponding non-H atoms with U_{iso}(H) = 1.2 U_{eq}(N) and 1.2 U_{eq}(C) (N-H= 0.86 Å, C-H= 0.93 Å). The atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol C, Tables 4.2.6.8 and 6.1.1.4). The publication materials of the structure of the compound have been prepared using WingX [34], PLATON [35] and PARST [36] software programs.

3. Results and discussion

A precise description of the crystallographic data of the X-ray structure is given in Table 1. The molecule consists of three benzene rings [A(C1-C6), C(C10-C15) and D(C18-C23)] and a pyrrolidine ring B(C1/C6/C7/C8/N1) (Figure 1) (Mercury) [37]. Ring A and B are fused together forming indole ring system which is connected with ring C through acetamide group. Furthermore, the Ring C and D are connected through hydroxyacetamide group. The structural parameters including bond distances, bond angles and torsion angles are listed in Table 2. The bond distances of C9=O1 and C16=O2 are 1.244(3) Å and 1.234(3) Å, respectively, indicate a typical C=O double bond character. All the bond distances fall in the normal range [38], except for C18-C23 = 1.358(4) Å which is slightly shortened. This could possibly be due to the presence of oxygen atom at C18 and its involvement in hydrogen bond formation. The indole ring system is approximately planar with maximum deviation of 0.0293 Å obtained for N1 atom and the benzene rings C and D are also planar (with maximum deviation of -0.0110 Å for C11). The least square plane of indole ring (C1/C2/C3/C4/C5/C6/C7/C8/N1) makes a dihedral angle of 47.49 and 85.21°, respectively, with the least square planes of the ring C and ring D. Similarly, the least square planes of ring C and ring D make a dihedral angle of 40.83°.

The N-H···O (N3-H3A···O1, N3-H3A···O3) and C-H···O (C14-H14···O2) intra-molecular hydrogen interactions result in the formation of three virtual rings with S(7), S(6) and S(5) graph-set motif [39] as shown in the ORTEP plot (Figure 1). In the crystal structure, the adjacent molecules are linked by N-H···O (N1-H1···O1, N2-H2A···O2) and C-H···O (C19-H19···O1) intermolecular hydrogen bonded interactions forming one dimensional stacking chain along [001] direction (Figure 2) (Mercury 3.10.2) [37]. In addition, the crystal structure is also stabilized by two weak C-H···π (C23-H23···Cg2 and C23-H23···Cg5) interactions (Table 3).

4. Conclusion

The compound *N*-(2-(2-(4-chlorophenoxy)acetamido) phenyl)-1*H*-indole-2-carboxamide was synthesized and characterized by ^{1}H NMR, ^{13}C NMR and LC MS spectroscopic techniques. The molecular structure of the compound was finally confirmed by single crystal XRD technique.

Table 2. Selected bond distances, bond angles and torsion angles.

Bond distance (Å)			
C1-N1	1.368 (3)	C10-N2	1.421 (3)
C6-C7	1.424 (3)	C14-C15	1.396 (3)
C7-C8	1.358 (3)	C15-N3	1.415 (3)
C8-N1	1.373 (3)	C16-O2	1.234 (3)
C8-C9	1.468 (3)	C16-N3	1.342 (3)
C9-O1	1.244 (3)	C16-C17	1.504 (3)
C9-N2	1.346 (3)	C17-O3	1.424 (3)
C10-C11	1.381 (3)	C18-O3	1.371 (3)
C10-C15	1.396 (3)	C21-Cl1	1.742 (3)
Bond angle (º)			
N1-C1-C2	130.6 (2)	C14-C15-N3	120.8 (2)
N1-C1-C6	107.5 (2)	C10-C15-N3	119.9 (2)
C2-C1-C6	121.8 (2)	O2-C16-N3	125.6 (2)
C5-C6-C1	118.6 (2)	O2-C16-C17	119.3 (2)
C5-C6-C7	134.7 (2)	N3-C16-C17	115.1 (2)
C1-C6-C7	106.7 (2)	O3-C17-C16	109.1 (2)
C8-C7-C6	107.3 (2)	C23-C18-O3	124.4 (2)
C7-C8-N1	109.2 (2)	C23-C18-C19	120.5 (2)
C7-C8-C9	132.1 (2)	O3-C18-C19	115.0 (2)
N1-C8-C9	118.5 (2)	C22-C21-Cl1	119.6 (2)
O1-C9-N2	122.2 (2)	C20-C21-Cl1	119.5 (2)
O1-C9-C8	120.92 (19)	C18-C23-C22	118.9 (3)
N2-C9-C8	116.8 (2)	C1-N1-C8	109.19 (19)
C11-C10-C15	118.9 (2)	C9-N2-C10	127.11 (19)
C11-C10-N2	118.6 (2)	C16-N3-C15	126.78 (19)
C15-C10-N2	122.3 (2)	C18-O3-C17	119.53 (19)
Torsion angle (º)			
C6-C7-C8-C9	175.1 (2)	C8-C9-N2-C10	−175.0 (2)
C7-C8-C9-O1	177.9 (2)	C11-C10-N2-C9	−131.8 (3)
N1-C8-C9-O1	−7.0 (3)	N3-C16-C17-O3	−7.9 (3)
C7-C8-C9-N2	−5.0 (4)	C14-C15-N3-C16	−39.9 (3)
N1-C8-C9-N2	170.1 (2)	C10-C15-N3-C16	140.0 (2)
N2-C10-C11-C12	−172.3 (2)	C23-C18-O3-C17	10.3 (4)
N2-C10-C15-C14	173.5 (2)	C19-C18-O3-C17	−172.1 (2)
N2-C10-C15-N3	−6.4 (3)	C16-C17-O3-C18	168.5 (2)
O2-C16-C17-O3	174.1 (2)	C2-C1-N1-C8	176.8 (3)
C15-C10-N2-C9	54.3 (3)		

Table 3. List of D-H···A and D-H···π H-bonded interactions *.

No	D-H....A	D – H (Å)	H···A (Å)	D···A (Å)	D - H···A (°)
1	N3-H3A···O1	0.86	2.06	2.7897(6)	142
2	N3-H3A···O3	0.86	2.09	2.5479(6)	113
3	C14-H14···O2	0.93	2.58	2.9874(7)	107
4	N1-H1···O1(i)	0.86	2.20	2.9702(7)	150
5	N2 -H2A···O2(ii)	0.86	2.06	2.8530(7)	153
6	C19-H19···O1(ii)	0.93	2.56	3.3155(8)	139
7	C23-H23···Cg1(iii)	0.93	2.78	3.6149(8)	150
8	C23-H23···Cg2(iii)	0.93	2.93	3.8418(9)	169

* Symmetry codes: (i) −*x*, -*y*, 1-*z*, (ii) 1-*x*,-*y*,1-*z*, (iii) *x*,*y*,-1+*z*; Cg1 represents centre of gravity of ring A and Cg2 represents centre of gravity of Indole ring system (fused structure of ring A and B).

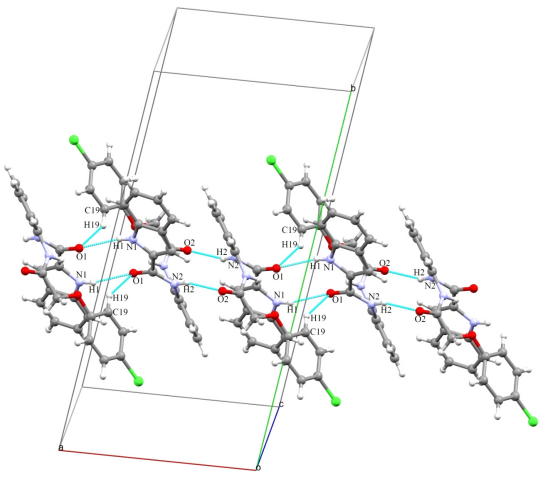

Figure 2. A fraction of molecular packing showing N-H···O and C-H···O H-bonded interaction.

The compound crystallizes in the monoclinic crystal system with space group $P2_1/c$. Crystallographic analysis of the compound reveals the existence of three intra-molecular hydrogen bonds (of the type N-H···O and C-H···O), three inter-molecular H-bonded interactions (of the type N-H···O and C-H···O) and two weak C-H···π interactions, that contributes in the stability of the crystal structure.

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Supporting information

CCDC-1893314 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
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