Chapter 4

Synthesis of indole derivatives containing naphthofuran moiety via Mannich base
4.1 Introduction

Isatin, possessing an indole nucleus containing both the keto and lactam moiety has aroused tremendous curiosity due to its diverse biological and pharmacological studies and also for the synthesis of numerous heterocyclic compounds. From literature survey it is well known that isatin heterocycles exhibit manifold importance in the field of medicinal chemistry as a potent chemotherapeutic agent. Recently Islam et al.,\textsuperscript{2,3} in collaboration with National Cancer Institute (NCI) of USA, have observed and reported that acylated $\Delta^2$-1,3,4 thiadiazoline derivatives of isatin which have shown effective anticancer activity against a number of cancer cells especially for breast cancer.

4.2 Methods for the synthesis of mannich base derivatives of isatin

Md. Rabiul et al.,\textsuperscript{4} reported the synthesis of isatin 3-carbohydrazone by reaction of carbohydrazide and isatin in glacial acetic acid.

\[
\begin{array}{c}
\text{O} \\
\text{X} \\
\text{N} \\
\text{H} \\
\text{NH}_2 \\
\end{array}
\]

\[X = \text{H, Cl, Br, CH}_3\]

Selvam et al.,\textsuperscript{5} synthesized a series of novel isatine-sulphonamide derivatives by combining isatin derivatives with sulphonamides. Investigation of anti-HIV activity was done against HIV-1(III B) in MT-4 cells and HIV integrase.

Where \(R = \text{H, Cl, Br, CH}_3\)
\(R' = \text{H, COCH}_3, \text{COC}_6\text{H}_5\)
\(R'' = \text{H, 4,5-dimethyl-2-isoxazolyl, 4,6-dimethyl-2-pyrimidinyl}\)
K.M. Khan et al., reported bis-schiff's bases from commercially available isatins by refluxing with hydrazine hydrate and then condensing with different aromatic aldehydes using methanol in high yields, showed an excellent anti-glycation activity better than the standard.

\[ R_1 = 2\text{-OH-C}_6\text{H}_5, 3\text{-OH-C}_6\text{H}_5, 4\text{-OH-C}_6\text{H}_5, 2,4\text{-OH-C}_6\text{H}_4, 2\text{-OMe-C}_6\text{H}_5, 2\text{-OH,3-OMe-C}_6\text{H}_4 \\
2\text{-OH,5-Cl-C}_6\text{H}_4, 2\text{-OH,5-Me-C}_6\text{H}_4, 3\text{-Cl-C}_6\text{H}_4, 3,4\text{-Cl-C}_6\text{H}_4, 2\text{-F-C}_6\text{H}_5, 4\text{-F-C}_6\text{H}_5 \\
2\text{-NO}_2\text{-C}_6\text{H}_4, 4\text{-NO}_2\text{-C}_6\text{H}_5, 2\text{-OEt-C}_6\text{H}_5, 4\text{-OEt-C}_6\text{H}_5 \]

S.N. Pandeya et al., reported some mannich bases of isatin and screened them for anti-microbial and anti-HIV active showed good activity.
S.K. Sridhar et al.,11-13 reported the Schiff’s bases and phenyl hydrazone of isatin by reacting isatin and appropriate aromatic primary amine/hydrazines. A new series of the corresponding N-mannich bases were synthesized by reacting them with formaldehyde and diphenylamine. The compounds were screened for analgesic, anti-inflammatory and anti-pyretic activity and some compounds showed enhanced activity.

R= H, Cl, Br

R'=

R''= H, -CH2-N C6H5
N. Karali\textsuperscript{14}, reported the synthesis, structural determination and primary cytotoxicity evaluation of 5-nitroindole-2,3-dione-3-thiosemicarbazone derivatives.

\[
\begin{align*}
\text{O}_2\text{N} & \quad \text{X} = \text{O, CH}_2 \\
\text{N} & \quad \text{R} = \text{CH}_3, \text{C}_6\text{H}_5, \text{CH}_2\text{CH} = \text{CH}_2, \\
\text{N} & \quad \text{cycl-C}_6\text{H}_5, \text{C}_6\text{H}_4\text{CH}_3(4-), \text{C}_6\text{H}_4\text{Cl}(4-) \\
\end{align*}
\]

P. Yogeeswari \textit{et al.},\textsuperscript{15} reported mannich bases of gatifloxacin were synthesized by reacting them with formaldehyde with several isatin derivatives. The compounds were tested \textit{in vitro} against a panel of 58 human tumor cell lines derived from nine neoplastic diseases emerged as a potent anti-cancer agent being more active than standard DNA topoisomerase II inhibitor.

\[
\begin{align*}
\text{R} & = \text{H, Cl, Br, F} \\
\text{R'} & = \text{O, NNHCO} \\
\end{align*}
\]
D. Sriram et al., synthesized mannich bases by the reaction of ciprofloxacin, and isatin derivatives, with 37% formalin in ethanol. The synthesized compounds are evaluated for in vitro and in vivo anti-mycobacterial activity against Mycobacterium tuberculosis and for inhibition of the super coiling activity of DNA gyrase from Mycobacterium smegmatis and were considered to be moderately active in reducing bacterial count in spleen.

V.A. Muthukumar et al., reported some novel Mannich base isatin derivatives were synthesized by reacting 1-(5-methyl-2-oxoindolin-3-ylidene)-4-(substitutedpyridin-2-yl)thiosemicarbazide with formaldehyde and various secondary amines. These synthesized compounds showed significant anti-inflammatory and analgesic activity.
Olcay Bekircan and Hakan Bektas reported general and convenient method for the synthesis of new heterocyclic compounds bearing two rings.

4.3 Present work

The survey of literature revealed that, the derivatives of naphtho[1,2-\textit{b}] contains isatin is not yet reported. Hence, our interest towards synthesis, characterization and pharmacological investigation of biheterocycles of naphthofuran contain isatin. In the present work, we have interested in synthesis of novel Indole derivatives containing naphthofuran moiety via Mannich base.

The research work carried out during the present investigation has been described in Scheme 1. The targeted compounds NF(26-35) are synthesized by the reaction of mannich base product of mono hydrazone of isatin (3) and naphtho[2,1-\textit{b}]furan-2-carboxylate in 20 mL of ethanol in presence of glacial acetic acid and then it was refluxed for about 2-3 h on water bath and cooled to room temperature. The solid that separated was filtered and recrystallized from ethanol.
Scheme 1:

All the newly synthesized Mannich bases were screened for *in vitro* anti-microbial, DNA Cleavage and *in vivo* pharmacological activities. Screening results have been discussed in Chapter 9.

### 4.4 Materials and methods

The homogeneity of the compounds was described by TLC on aluminum silica gel 60 F254 (Merck) detected by U.V light (254 nm) and iodine vapors. The melting points of the products were determined by open capillaries on a Buchi apparatus and are uncorrected. The IR spectra were recorded on a *Nicolet Impact-410 FT-IR* Spectrophotometer, using KBr pellets. $^1$H NMR and $^{13}$C NMR spectra were recorded on a *Bruker Avance-400F* 400 MHz spectrometer in DMSO using TMS as an internal standard with $^1$H resonant frequency of 400 MHz and $^{13}$C resonant frequency of 100 MHz. The Mass spectra were recorded on a GC-MS (*Shimadzu QP2010S*, Japan). The elemental analysis was carried out by using *Heraus CHN* rapid analyzer. All the compounds gave C, H and N analysis within ± 0.5 % of the theoretical values.
4.5 Experimental procedure

Synthesis of Mannich bases of isatin\(^{11}\) (2)

Equimolar quantity (0.01 mol) of secondary amines in 25 ml of ethanol was added to slurry containing appropriate isatin (0.01 mol) (1) and 1 ml of formaldehyde solution (37% v/v) in 20 ml of ethanol. The reaction mixture was stirred for 1 h at room temperature and refrigerated for 48 h. The products were separated by suction filtration, vacuum dried and recrystallized from ethanol.

Synthesis of Isatin hydrazones\(^{20}\) (3)

The preparation of hydrazone's (3) was carried out by refluxing a mixture of isatin derivatives (2) (1 g) and hydrazine hydrate (10 mL) in methanol on water bath for 3-4 h and cooled. The solid that separated was filtered and recrystallized from methanol.

General synthesis\(^{19}\) of novel indole derivatives containing naphthofuran moiety via Mannich base (NF26-35)

The equimolar ratio of naphtho-[2,1-b]furan-2-carboxylate (5) (0.01 mole) is dissolved in 20 mL of ethanol containing (0.01 mol) of mono hydrazone's of isatin derivative (3) containing catalytic amount of glacial acetic acid and then it was refluxed for about 2-3 h on water bath and cooled to room temperature. The solid that separated was filtered and recrystallized from ethanol.

4.6 Results and discussion

The required starting material to accomplish the targeted compound was achieved by the literature methods.\(^{11,19}\) Reaction of naphtho-[2,1-b]furan-2-carbohydrazide (3) with different mannich bases of hydrazone of isatin in ethanol using acid catalyst at reflux temperature for 4-5 h yielded the corresponding (E)-N'-{(substituted)-2-oxoindolin-3-ylidene}naphtho[2,1-b]furan-2-carbohydrazide derivatives NF(26-35) in good yield Scheme 1.

These compounds NF(26-35) were confirmed from their spectral data. IR spectra showed bands at 3248, 2917, 2848, 1675 for C=O of isatin, 1620 for C=O of furan and 1594 for imine group for the compound NF26. \(^1\)H NMR spectrum of
NF26 in DMSO-$d_6$ revealed signals at 2.56, 3.55 (CH$_2$, morpholine ring) and 4.39 (N-CH$_2$-N), and 7.0-8.59 ppm (Ar-H, isatin and naphthofuran ring). Further, $^{13}$C NMR spectrum of NF26 in DMSO-$d_6$ shows signals at 70 and 134 ppm, indicates the formation of methyl bridged carbon and C=N of isatin ring. The formations of naphthofuran derivatives were further confirmed by the melting point of the starting material and the obtained product, as well as by the spectral data (IR, NMR and Mass). The X-ray analysis of the compound(s) is under progress.

$N$-(1-(morpholinomethyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF26): Light orange: mp. 121-123 °C (80%), IR (KBr): $v$ (cm$^{-1}$), 3241 (CH of naphtho), 947 (CO of naphtho), 1672 (CO), 1622 (CO of isatin), 1594 (C=N); $^1$H NMR (400 MHz, DMSO, $\delta$ ppm): 2.49 (4H, t, $J = 7$, CH$_2$-O- CH$_2$), 3.55 (4H, t, $J = 8.4$, CH$_2$-N-CH$_2$), 4.40 (2H, s, CH$_2$), 7.07 (1H, s, NH), 6.856 (1H, t, $J = 7.6$, Ar-H), 6.918 (1H, s, Ar-H), 6.95 (1H, t, $J = 6.8$, Ar-H), 7.139 (1H, t, $J = 6.9$, Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, $J = 6.8$, Ar-H), 7.884 (1H, d, $J = 8.8$, Ar-H), 8.020 (1H, d, $J = 9$, Ar-H), 8.084 (1H, d, $J = 8$, Ar-H), 8.455 (2H, t, $J = 6$, Ar-H); $^{13}$C NMR (100 MHz, DMSO, $\delta$ ppm): 51.17, 61.06, 70.0, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.07, 138.60, 142.23, 145.16, 153.27, 158.57, 163.36; GC-MS: 453 [M$^+$-1]; Anal.Calcd. for C$_{26}$H$_{22}$N$_4$O$_4$: C, 68.71; H, 4.88; N, 12.33 %. Found: C, 68.68; H, 4.85; N, 12.29 %.
**N-(2-oxo-1-(piperidin-1-ylmethyl)indolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF27):** Buff color: mp. 113-115 °C (80%), IR (KBr): ν (cm⁻¹), 3273 (CH of naphtho), 948 (CO of naphtho), 1725 (CO), 1618 (CO of isatin), 1589 (C=N); \(^1\)H NMR (400 MHz, DMSO, δ ppm): 1.6 (6H, m, CH₂), 2.34 (4H, t, J = 8.4, CH₂-N-CH₂), 6.95 (1H, t, J = 6.8, Ar-H), 6.92 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.2 (1H, t, J = 6, Ar-H), 7.354 (1H, s, furan CH), 7.6 (1H, d, J = 8, Ar-H), 7.884 (1H, d, J = 8.8, Ar-H), 8.022 (1H, d, J = 9, Ar-H), 8.084 (1H, d, J = 8, Ar-H), 8.456 (2H, t, J = 6, Ar-H); \(^13\)C NMR (100 MHz, DMSO, δ ppm): 25.7, 30.02, 51.15, 61.04, 70.1, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.03, 138.49, 142.23, 145.16, 153.17, 158.57, 163.35; GC-MS : 451 [M⁺⁻¹]; Anal.Calcd. for C₂₇H₂₄N₄O₃: C, 71.67; H, 5.35; N, 12.38 %. Found: C, 71.62; H, 5.32; N, 12.36 %.

**N-(2-methylpiperidin-1-yl)methyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF28):** Dark buff color: mp. 210-212 °C (80%), IR (KBr): ν (cm⁻¹), 3275 (CH of naphtho), 944 (CO of naphtho), 1658 (CO), 1620 (CO of isatin), 1594 (C=N); \(^1\)H NMR (400 MHz, DMSO, δ ppm): 1.18 (3H, d, J = 8, CH₃), 1.59 (6H, m, CH₂), 2.2 (2H, d, J = 6, CH₂), 2.49 (1H, d, J = 7, CH), 4.40 (2H, s, CH₂), 7.07 (1H, s, NH), 6.856 (1H, t, J = 7, Ar-H), 6.92 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.14 (1H, t, J = 6, Ar-H), 7.36 (1H, s, furan CH), 7.59 (1H, d, J = 8, Ar-H), 7.879 (1H, d, J = 8, Ar-H), 8.020 (1H, d, J = 9, Ar-H), 8.084 (1H, d, J = 8, Ar-H), 8.449 (2H, t, J = 6, Ar-H); \(^13\)C NMR (100 MHz, DMSO, δ ppm): 18.6, 24.9, 26.2, 30.4, 49.2, 51.17, 61.06, 70.0, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.07, 138.60, 142.23, 145.16, 153.27, 158.57, 163.36; GC-MS : 465 [M⁺⁻¹]; Anal.Calcd. for C₂₈H₂₆N₄O₃: C, 72.09; H, 5.62; N, 12.01 %. Found: C, 72.05; H, 5.59; N, 11.99 %.
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\[ N-(1-((3\text{-methylpiperidin-1-yl})methyl)-2\text{-oxoindolin-3-ylidene})\text{naphtho}[2,1-b]furan-2\text{-carbohydrazide (NF29)}: \]

Dark brown color: mp. 108-110 °C (80%),
IR (KBr): \( v \) (cm\(^{-1}\)), 3267 (CH of naphtho), 948 (CO of naphtho), 1726 (CO), 1618 (CO of isatin), 1589 (C=\( N \)); \( ^1H \) NMR (400 MHz, DMSO, \( \delta \) ppm): 1.02 (3H, d, \( J = 7 \), CH\(_3\)), 1.45-1.56 (4H, m, CH\(_2\)), 1.63(1H, m, CH), 2.34-2.41 (4H, m, CH\(_2\)), 4.40 (2H, s, CH\(_2\)), 7.07 (1H, s, NH), 6.856 (1H, t, \( J = 7 \), Ar-H), 6.918 (1H, s, Ar-H), 6.95 (1H, t, \( J = 6 \), Ar-H), 7.139 (1H, t, \( J = 6 \), Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, \( J = 8 \), Ar-H), 7.884 (1H, d, \( J = 8 \), Ar-H), 8.020 (1H, d, \( J = 9 \), Ar-H), 8.084 (1H, d, \( J = 8 \), Ar-H), 8.455 (2H, t, \( J = 6 \), Ar-H); \( ^13C \) NMR (100 MHz, DMSO, \( \delta \) ppm): 18.4, 31.8, 32.2, 49.7, 70.0, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.07, 130.13, 142.23, 145.16, 153.27, 158.0, 163.29; GC-MS: 465 [M\(^+\)]; Anal. Calcd. for C\(_{28}\)H\(_{26}\)N\(_4\)O\(_3\): C, 72.09; H, 5.62; N, 12.01 %. Found: C, 72.07; H, 5.59; N, 11.99 %.

\[ N-(1-((4\text{-methylpiperidin-1-yl})methyl)-2\text{-oxoindolin-3-ylidene})\text{naphtho}[2,1-b]furan-2\text{-carbohydrazide (NF30)}: \]

Dark brown color: mp. 145-147 °C (80%),
IR (KBr): \( v \) (cm\(^{-1}\)), 3274 (CH of naphtho), 949 (CO of naphtho), 1725 (CO), 1619 (CO of isatin), 1590 (C=\( N \)); \( ^1H \) NMR (400 MHz, DMSO, \( \delta \) ppm): 1.06 (3H, d, \( J = 7 \), CH\(_3\)), 1.45-1.56 (4H, m, CH\(_2\)), 1.63(1H, m, CH), 2.19-2.30 (4H, m, CH\(_2\)), 4.36 (2H, s, CH\(_2\)), 7.03 (1H, s, NH), 6.856 (1H, t, \( J = 7 \), Ar-H), 6.920 (1H, s, Ar-H), 6.99 (1H, t, \( J = 6 \), Ar-H), 7.139 (1H, t, \( J = 6 \), Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, \( J = 8 \), Ar-H), 7.884 (1H, d, \( J = 8 \), Ar-H), 8.020 (1H, d, \( J = 9 \), Ar-H), 8.084 (1H, d, \( J = 8 \), Ar-H), 8.449 (2H, t, \( J = 6 \), Ar-H); \( ^13C \) NMR (100 MHz, DMSO, \( \delta \) ppm): 20.4, 31.8, 32.2, 49.7, 70.0, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.07, 138.60, 142.23,
N-(2-oxo-1-(piperidin-1-ylmethyl)indolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF31): Dark brown (Just black): mp. 86-88 °C (80%), IR (KBr): ν (cm\(^{-1}\)), 3241 (CH of naphtho), 950 (CO of naphtho), 1724 (CO), 1618 (CO of isatin), 1590 (C=N); \(^1\)H NMR (400 MHz, DMSO, δ ppm): 2.3 (1H, s, NH), 2.49-2.70 (8H, m, CH\(_2\)), 4.03 (2H, s, CH\(_2\)), 7.05 (1H, s, NH), 6.901 (1H, t, J = 7, Ar-H), 6.920 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.140 (1H, t, J = 6, Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, J = 8, Ar-H), 7.884 (1H, d, J = 8, Ar-H), 8.018 (1H, d, J = 9, Ar-H), 8.085 (1H, d, J = 8, Ar-H), 8.449 (2H, t, J = 6, Ar-H); \(^13\)C NMR (100 MHz, DMSO, δ ppm): 46.2, 51.17, 55.2, 70.8, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 130.07, 138.60, 142.23, 145.16, 153.27, 158.57, 163.36; GC-MS: 452 [M\(^{+}\)-1]; Anal.Calcd. for C\(_{26}\)H\(_{23}\)N\(_5\)O\(_3\): C, 68.86; H, 5.11; N, 15.44 %. Found: C, 68.82; H, 5.08; N, 15.40 %.

N-(1-((4-methylpiperazin-1-yl)methyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF32): Buff brown: mp. 112-114 °C (80%), IR (KBr): ν (cm\(^{-1}\)), 3235 (CH of naphtho), 945 (CO of naphtho), 1658 (CO), 1629 (CO of isatin), 1595 (C=N); \(^1\)H NMR (400 MHz, DMSO, δ ppm): 2.27 (3H, s, CH\(_3\)), 2.47 (8H, m, CH\(_2\)), 4.02 (2H, s, CH\(_2\)), 7.04 (1H, s, NH), 6.870 (1H, t, J = 7, Ar-H), 6.920 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.140 (1H, t, J = 6, Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, J = 8, Ar-H), 7.884 (1H, d, J = 8, Ar-H), 8.020 (1H, d, J = 9, Ar-H), 8.084 (1H, d, J = 8, Ar-H), 8.455 (2H, t, J = 6, Ar-H); \(^13\)C NMR (100 MHz, DMSO, δ ppm): 43.2, 51.17, 55.3, 70.8, 109.91, 111.04, 112.61, 113.58, 117.40, 121.30, 122.37, 123.82,
125.55, 126.20, 126.98, 127.40, 129.07, 138.60, 142.23, 145.16, 153.27, 158.57, 163.36; GC-MS : 466 [M⁺-1]; Anal.Calcd. for C₂₇H₂₅N₅O₅: C, 69.36; H, 5.39; N, 14.98 %. Found: C, 69.34; H, 5.36; N, 14.96 %.

N-(1-((methyl(phenyl)amino)methyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]

**NF32**

**furan-2-carbohydrazide (NF33):** Shiny yellow orange: mp. 89-91 °C (80%), IR (KBr): ν (cm⁻¹), 3282 (CH of naphtho), 948 (CO of naphtho), 1697 (CO), 1618 (CO of isatin), 1595 (C=N); ¹H NMR (400 MHz, DMSO, δ ppm): 2.87 (3H, s, CH₃), 5.02 (2H, s, CH₂), 6.58-6.72 (5H, m, Ar-H), 7.07 (1H, s, NH), 6.856 (1H, t, J = 7, Ar-H), 6.918 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.139 (1H, t, J = 6, Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, J = 8, Ar-H), 7.884 (1H, d, J = 8, Ar-H), 8.020 (1H, d, J = 9, Ar-H), 8.084 (1H, d, J = 8, Ar-H), 8.455 (2H, t, J = 6, Ar-H); ¹³C NMR (100 MHz, DMSO, δ ppm): 35.2, 75.4, 109.91, 111.04, 112.61, 113.58, 114.3, 117.40, 118.4, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.70, 130.07, 138.60, 142.23, 145.16, 149.0, 153.27, 158.57, 163.36; GC-MS : 473 [M⁺-1]; Anal.Calcd. for C₂₉H₂₂N₄O₅: C, 73.40; H, 4.67; N, 11.81 %. Found: C, 73.36; H, 4.64; N, 11.79 %.

N-(1-((ethyl(phenyl)amino)methyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]

**NF33**

**furan-2-carbohydrazide (NF34):** Shiny brown color: mp. 135-137 °C (80%), IR (KBr): ν (cm⁻¹), 3202 (CH of naphtho), 952 (CO of naphtho), 1687 (CO), 1621 (CO of isatin), 1591 (C=N); ¹H NMR (400 MHz, DMSO, δ ppm): 1.23 (3H, t, J = 8, CH₃), 3.49(2H, m, CH₂), 5.1 (2H, s, CH₂), 6.58-6.72 (5H, m, Ar-H), 7.04 (1H, s, NH), 6.856 (1H, t, J = 7, Ar-H), 6.918 (1H, s, Ar-H), 6.95 (1H, t, J = 6, Ar-H), 7.139 (1H, t, J = 6, Ar-H), 7.354 (1H, s, furan CH), 7.610 (1H, d, J = 8, Ar-H), 7.879 (1H, d, J = 8, Ar-H), 8.020 (1H, d, J = 9, Ar-H), 8.081 (1H, d, J = 8, Ar-H), 8.446 (2H, t, J = 6, Ar-H); ¹³C NMR (100 MHz, DMSO, δ ppm): 12.2, 42.5, 72.4, 109.91, 111.04, 112.61, 113.58, 114.3,
117.40, 118.4, 121.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 129.70, 130.07, 138.60, 142.23, 145.16, 149.0, 153.27, 158.57, 163.36; GC-MS : 487 [M'+1]; Anal. Calcd. for C_{30}H_{34}N_{4}O_{3}: C, 73.76; H, 4.95; N, 11.47 %. Found: C, 73.73; H, 4.91; N, 11.44 %.

\( \text{N-(1-((diphenylamino)methyl)-2-oxoindolin-3-ylidene)naphtho[2,1-b]furan-2-carbohydrazide (NF35)} \): Buff color: mp. 88-90 °C (80%), IR (KBr): \( \nu \) (cm\(^{-1}\)), 3302 (CH of naphtho), 942 (CO of naphtho), 1689 (CO), 1616 (CO of isatin), 1516 (C=N); \(^1\)H NMR (400 MHz, DMSO, \( \delta \) ppm): 4.82(2H, s, CH\(_2\)), 7.04 (1H, s, NH), 6.43-6.87 (10H, m, Ar-H), 6.918 (1H, s, Ar-H), 6.95 (1H, t, \( J = 6 \), Ar-H), 7.139 (1H, t, \( J = 6 \), Ar-H), 7.354 (1H, s, furan CH), 7.603 (1H, d, \( J = 8 \), Ar-H), 7.884 (1H, d, \( J = 8 \), Ar-H), 8.019 (1H, d, \( J = 9 \), Ar-H), 8.085 (1H, d, \( J = 8 \), Ar-H); \(^13\)C NMR (100 MHz, DMSO, \( \delta \) ppm): 75.1, 109.91, 111.04, 112.61, 113.58, 117.40, 118.3, 119.30, 122.37, 123.82, 125.55, 126.20, 126.98, 127.40, 128.14, 129.26, 129.7, 130.07, 138.60, 142.23, 145.16, 149.4, 153.27, 158.57, 163.36; GC-MS : 535 [M'+1]; Anal. Calcd. for C_{34}H_{24}N_{4}O_{3}: C, 76.11; H, 4.51; N, 10.44 %. Found: C, 76.06; H, 4.49; N, 10.40 %.
Spectrum 1: IR Spectrum of compound NF26
Spectrum 2: $^1$H NMR Spectrum of compound NF26 in DMSO
Spectrum 3: Aromatic expansion of ¹H NMR Spectrum of compound NF26 in DMSO
Spectrum 5: GC-MS Spectrum of compound NF26
Chapter 4
References