Ultra Scientist Vol. 27(3)B, 167-174 (2015).

An efficient synthesis of thiophene containing 1,8-Naphthyridine derivatives

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(Acceptance Date 16th November, 2015)

Abstract

A new family of Naphthyridines heterocycle have been synthesized by the condensation of various N-Substituted-Naphthyridines with tributyl(5-substitutedthiophen-2-yl)stannanes. All title compounds were thoroughly characterized by ^{1}H, ^{13}C NMR and Mass spectra.

Key words : Naphthyridines, stannanes, Thiophene, butyloxycarbonyl (Boc) and spectral analysis.

Introduction

Naphthyridine derivatives continued to be of great interest due to a wide spectrum of their biological activity. Antibiotics of this group are being widely used for the diagnostics and chemotherapy of infectious diseases of humans. 1,8-naphthyridine derivatives have also attracted considerable attention because of the it's skeleton is present in many compounds which have been isolated from natural sources, with various biological activities. Nalidixic acid, for example, possesses strong antibacterial activity and used mainly for the treatment of urinary tract infections with gram negative pathogens[1] and Gemifloxacin has antimicrobial and antibacterial activities.[2] It is known that (E)- and (Z)-o-(diethylamino)ethyl oximes of 1,8-naphthyridine series **(A)** are potential drugs for local anesthesia,[3] and 1-(2-fluorobenzyl)-3-(2-tolyl)- 1,8-naphthyridin-2(1H)-one is used for the treatment of memory disorders, in particular, Alzheimer's disease.[4]

There is evidence that antitumour activity is due to the intercalation between the base pairs of DNA and interference with normal functioning of the enzyme topoisomerase II which is involved in the breaking and releasing of DNA strands.[5] Binding of these

drugs is due to the presence of planar linearly fused tetracyclic heterocyclic system. This finding has stimulated the research in finding new antitumour drugs containing planar fused ring system. In recent years various fused systems such as thiophene,[6] furan and pyridine analogues of ellipticine[7] and benzothiazoloquinolines[8] have been studied for their properties. Recently, Cao and He15 studied DNA affinity properties of safranine-T which features a planar phenazine ring and have shown that electrostatic binding plays an important role in the intercalation of safranine-T. The results of these various binding studies have been useful in designing new and promising anticancer agents for clinical use.[9-11] In view of medical importance of 1,8-Naphthyridine and thiophene and in continuation of our work in organic synthesis of condensed heterocycles,[12-15] we have investigated a new, simple method for the preparation of title compounds.

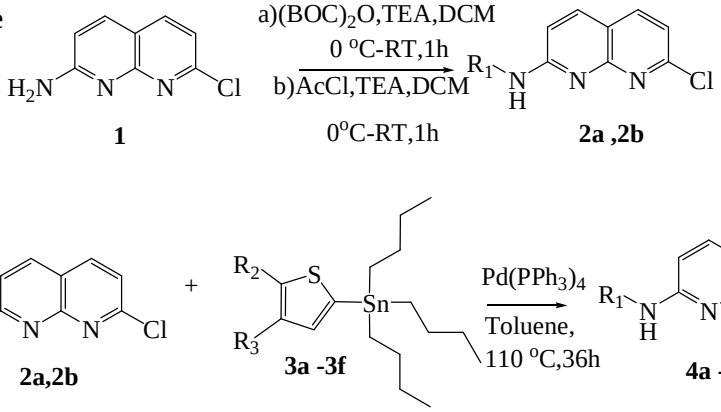
Experimental

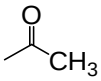
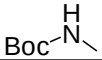
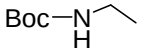
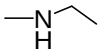
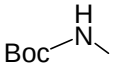
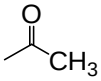
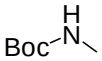
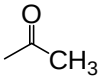
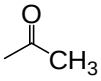
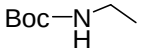
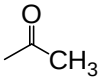
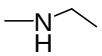
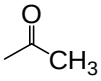
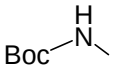
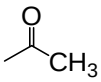
Chemicals and solvents were reagent grade and used without further purification. The ^{1}H NMR was recorded in the indicated solvent on a Varian 400 MHz and ^{13}C NMR on a Varian 100 MHz spectrometer with TMS as internal standard. All chemical shifts (δ) were reported in ppm from internal TMS. Mass spectra were measured on a Jeol JMS D-300 spectrometer. The homogeneity of the compounds was checked using pre-coated TLC plates (E.Merk Kieselgel 60 F_{254}).

Results and Discussions

The synthesis of thiophene containing 1,8-Naphthyridine derivatives (4a-l) is presented in scheme and they were characterized by ^{1}H-NMR, ^{13}C-NMR and Mass spectral data. The synthesis compounds 4a-l was carried out by utilizing the commercially available 7-chloro-1,8-naphthyridin-2-amine(1). Boc protection of amine in compound 1 was done in presence of dichloromethane, triethylamine and Boc-anhydride at 0 °C to rt for 1h. N-acetyl protection of compound 1 was done in presence of dichloromethane, triethylamine and acetyl chloride at 0 °C to rt for 1 h. Thiophene containing naphthyridines heterocycle have been synthesized by the condensation of various N-substituted -Naphthyridines (2a,2b) with tributyl(5-substituted thiophene-2-yl) stannanes (3a-f) in dry toluene and $Pd(pph_3)_4$ under argon at 110 °C for 36 h.

Scheme



	R1	R2	R3
2a	—Boc	-	-
2b		-	-
4a	—Boc		H
4b	—Boc		H
4c	—Boc		H
4d	—Boc		H
4e	—Boc		-CH_3
4f	—Boc		-CH_3
4g			H
4h			H
4i			H
4j			H
4k			-CH_3
4l			-CH_3

tert-butyl (7-chloro-1,8-naphthyridin-2-yl)carbamate (2a) *:*

To a stirred solution of 7-chloro-1,8-naphthyridin-2-amine **1**(3g, 16.7mmol) in DCM(30ml) was added to triethylamine(2.78g, 20.0mmol) at 0 °C. Then reaction stirred for 5 min. Then boc-anhydride (3.65g,16.7mmol) was added at 0 °C dropwise. Reaction stirred at rt for 1h.Completion of the reaction was monitered by TLC. Water(30ml) was added to the reaction mixture, then extracted with DCM (2x30ml). Combined organic layers were dried over sodium sulphate and solvent was evaporated in rotavapour to gave the crude product. Crude product was purified by column chromatography gave the pure compound(3.8g, yield 81.3%) **2a**.

^{1}H NMR (DMSO-d_6, 400MHz) : 1.51 (s, 9H), 8.18 (d, 1H, *J*=7.8Hz), 8.22 (d, 1H), 8.42 (m, 2H), 11.21 (brs, 1H). Mass: m/z 280.1 $[M+H]^+$.

N-(7-chloro-1,8-naphthyridin-2-yl) acetamide (2b)

To a stirred solution of 7-chloro-1,8-naphthyridin-2-amine **1**(3g, 16.7mmol) in DCM(30ml) was added to triethylamine(2.78g, 20.0mmol) at 0 °C. Then reaction stirred for 5 min. Then acetyl chloride(1.44g, 18.4mmol) was added at 0 °C dropwise. Reaction stirred at rt for 1h. Completion of the reaction was monitered by TLC. Water (30ml) was added to the reaction mixture, then extracted with DCM (2x30ml). Combined organic layers were dried over sodium sulphate and solvent was evaporated in rotavapour to gave the crude product. Crude product was purified by column chromatography gave the pure compound (3.0g, yield 81.08%) **2b.**

^{1}H NMR (DMSO-d_6, 400MHz) : 2.18 (s, 3H), 8.10 (d, 1H, *J*=7.8Hz), 8.20 (d, 1H, *J*=8.2Hz), 8.43 (d, 1H, *J*=7.8Hz), 8.59 (d, 1H, *J*=8.2Hz), 11.18, (brs, 1H). Mass: m/z 222.1 $[M+H]^+$.

tert-butyl (5-(tributylstannyl)thiophen-2-yl) carbamate (3a)

To a stirred 2M solution of LDA (2.5 ml, 5.04 mmol) at –78°C under an N2 atmosphere was added dropwise a solution of 1-(thiophen-2-yl)carbamate (5 mmol) in dry THF (3 ml). After 1 hour, a solution of TBTC (1.5 ml, 4.62 mmol) in dry THF (4 ml) was added dropwise. After being stirred at -78 °C for further 2 h, the mixture was allowed to warm to rt, and the stirring was continued overnight. The resulting mixture was evaporated under reduced pressure, then diluted with DCM, and washed twice with water. After drying over sodium sulfate and evaporation of the solvent, the residue was purified by distillation (142-150 °C, 0.1 mmHg) to give the desired compound as a pale yellow oil.

^{1}H NMR ($CDCl_3$, 400MHz) : 0.89 (t, 9H), 1.18(m, 6H), 1.34 (m, 8H), 1.59 (s, 9H), 1.63 (m, 4H), 4.60 (brs, 1H), 7.01 (d, 1H), 7.10 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz) : 3.21, 10.75, 13.63, 15.99, 17.51, 125.08, 126.47, 134.89, 136.70, 146.16, 165.20, 170.51. Mass: m/z 489.9 $[M+H]^+$.

N-methyl-5-(tributylstannyl)thiophen-2-amine (3b)

^{1}H NMR ($CDCl_3$, 400MHz) : 0.88 (t,

9H), 1.10(m, 6H), 1.35 (m, 8H), 1.63 (m, 4H), 2.78 (s, 3H), 4.58 (brs, 1H), 7.01 (d, 1H), 7.08 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz) : 3.21, 10.75, 13.63, 15.99, 24.51, 126.08, 126.48, 134.88, 136.70, 146.16, 165.20. Mass: m/z 404.1 $[M+H]^+$.

tert-butyl ((5-(tributylstannyl)thiophen-2-yl)methyl)carbamate (3c)

^{1}H NMR ($CDCl_3$, 400MHz) : 0.88 (t, 9H), 1.08(m, 6H), 1.35 (m, 8H), 1.51 (s, 9H), 1.63 (m, 4H), 2.75 (s, 3H), 4.18 (s, 2H), 7.01 (d, 1H), 7.10 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz): 3.21, 10.75, 13.63, 15.99, 22.23, 25.51, 35.20, 126.07, 126.48, 134.88, 136.70, 146.17, 164.20, 170.02. Mass: m/z 504 $[M+H]^+$.

N-methyl-1-(5-(tributylstannyl)thiophen-2-yl) methanamine (3d)

^{1}H NMR ($CDCl_3$, 400MHz) : 0.89 (t, 9H), 1.07(m, 6H), 1.36 (m, 8H), 1.63 (m, 4H), 2.71 (s, 3H), 4.15 (s, 2H), 7.01 (d, 1H), 7.08 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz) : 3.21, 10.75, 13.63, 22.23, 25.51, 35.20, 126.07, 126.48, 134.88, 136.71, 146.27, 163.20. Mass: m/z 418 $[M+H]^+$.

tert-butyl (3-methyl-5-(tributylstannyl) thiophen-2-yl)carbamate (3e)

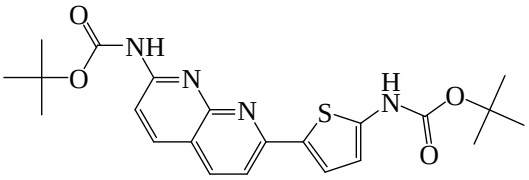
^{1}H NMR ($CDCl_3$, 400MHz) : 0.89 (t, 9H), 1.08(m, 6H), 1.33 (m, 8H), 1.59 (s, 9H), 1.63 (m, 4H), 2.36 (s, 3H), 7.01 (d, 1H), 7.09 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz) : 3.21, 10.75, 13.64, 15.89, 17.51, 24.32, 125.08, 126.47, 134.89, 136.70, 146.16, 164.20, 170.48. Mass: m/z 504 $[M+H]^+$.

N,3-dimethyl-5-(tributylstannyl)thiophen-2-amine (3f)

^{1}H NMR ($CDCl_3$, 400MHz) : 0.88 (t, 9H), 1.08(m, 6H), 1.35 (m, 8H), 1.63 (m, 4H), 2.33, 2.75 (s, 3H), 7.01 (d, 1H), 7.10 (d, 1H); ^{13}C NMR ($CDCl_3$, 100MHz) : 3.21, 10.75, 13.63, 15.99, 24.51, 126.08, 126.48, 134.88, 136.70, 146.16, 164.20. Mass: m/z 418 $[M+H]^+$.

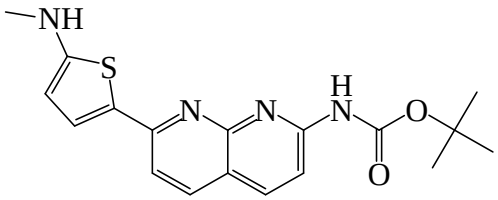
tert-butyl (5-(7-((tert-butoxycarbonyl) amino)-1,8-naphthyridin-2-yl)thiophen-2-yl)carbamate (4a)

2 (2.0 g, 1.8 mmol), **3** (2 mL, 6.1 mmol) and $Pd(PPh_3)_4$ (50 mg) were dissolved in dry toluene (80 mL) under Argon, and the mixture was heated at 110 °C for 36 h. After cooling to room temperature, the reaction mixture was evaporated to dryness to give a crude product. The crude product was purified by column chromatography gave pure product **4a-l**. (70%-80%)



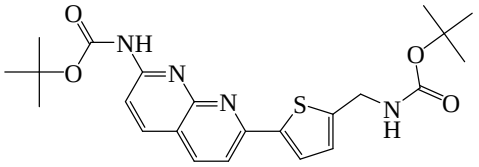
^{1}H NMR (DMSO-d6, 400MHz) : 1.48 (s, 9H), 1.51(s, 9H), 7.18 (s, 1H), 7.60 (s, 1H) 7.85 (d, 1H, *J*=7.8Hz), 7.91 (s, 1H), 8.04 (d, 1H, *J*=8.4Hz), 8.32 - 8.41 (m, 3H), ^{13}C NMR (DMSO-d6, 100MHz) : 21.58, 24.83, 113.88, 116.87, 119.28, 127.15, 127.91, 138.10, 139.61, 143.86, 154.86, 155.02, 156.27, 169.29, 170.50, Mass: m/z 443 $[M+H]^+$.

tert-butyl (7-(5-(methylamino)thiophen-2-yl)-1,8-naphthyridin-2-yl)carbamate (4b)



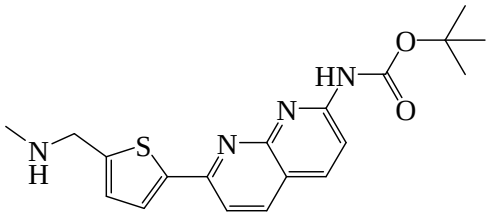
^{1}H NMR (DMSO-d$_6$, 400MHz) : 1.50 (s, 9H), 2.77(s, 3H), 7.12 (s, 1H), 7.60 (m, 1H), 7.91 (s, 1H), 8.03 (d, 1H), 8.31 - 8.41 (m, 3H); ^{13}C NMR (DMSO-d$_6$, 100MHz) : 21.82, 35.22, 114.29, 116.83, 119.25, 127.10, 127.92, 138.12, 139.61, 145.86, 154.89, 155.02, 155.25, 170.43.
Mass: m/z 357 $[M+H]^+$.

tert-butyl (5-(7-((tert-butoxycarbonyl) amino)-1,8-naphthyridin-2-yl)thiophen-2-yl-methyl) carbamate (4c) :



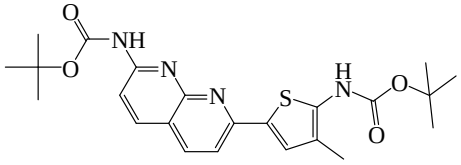
^{1}H NMR (DMSO-d$_6$, 400MHz) : 1.48 (s, 9H), 1.51(s, 9H), 4.22 (s, 2H), 7.13 (s, 1H), 7.65 (s, 1H), 7.81 (d, 1H, *J*=7.6Hz), 7.95 (s, 1H), 8.04 (d, 1H, *J*=8.2Hz), 8.30-8.41 (m, 3H); ^{13}C NMR (DMSO-d6, 100MHz) : 21.58, 21.82, 49.72, 113.81, 116.85, 119.22, 127.10, 127.92, 138.10, 139.61, 143.85, 154.82, 155.02, 155.27, 169.20, 170.50.
Mass: m/z 457 $[M+H]^+$.

tert-butyl 2-(5-((methylamino)methyl) thiophen-2-yl)-1,8-naphthyridin-7-ylcarbamate (4d)



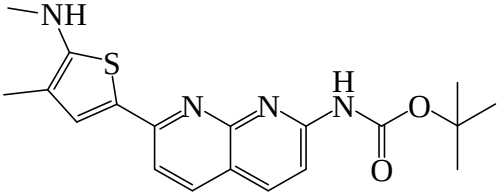
^{1}H NMR (DMSO-d$_6$, 400MHz) : 1.51 (s, 9H), 2.48(s, 3H), 4.44 (s, 2H), 7.14 (s, 1H), 7.62 (d, 1H, *J*=7.8Hz), 7.75 (d, 1H), 7.91 (s, 1H), 8.04 (d, 1H, *J*=8.4Hz), 8.30 - 8.41 (m, 3H), ^{13}C NMR (DMSO-d$_6$, 100MHz) : 21.83, 35.57, 49.76, 114.28, 116.84, 119.26, 127.10, 127.92, 138.11, 139.61, 143.85, 154.88, 155.01, 155.29, 170.52.
Mass: m/z 371 $[M+H]^+$.

tert-butyl-(5-(7-((tert-butoxycarbonyl) amino)-1,8-naphthyridin-2-yl)-3-methyl thiophen-2-yl)carbamate (4e)



^{1}H NMR (DMSO-d$_6$, 400MHz) : 1.49 (s, 9H), 1.51 (s, 9H), 2.36 (s, 3H), 7.13 (s, 1H), 7.85 (d, 1H, *J*=7.8Hz), 7.63 (s, 1H), 8.02 (d, 1H, *J*=8.0Hz), 8.30 - 8.40 (m, 3H), ^{13}C NMR (DMSO-d$_6$, 100MHz) : 21.58, 21.82, 28.86, 113.89, 116.87, 119.28, 127.15, 127.91, 138.10, 139.69, 144.88, 154.86, 154.82, 156.21, 169.85, 170.50,
Mass: m/z 457 $[M+H]^+$.

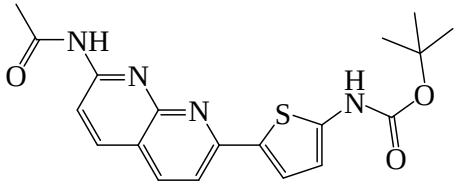
tert-butyl-(7-(4-methyl-5-(methylamino) thiophen-2-yl)-1,8-naphthyridin-2-yl) carbamate (4f) :



^{1}H NMR (DMSO-d$_6$, 400MHz) : 1.51 (s, 9H), 2.35 (s, 3H), 2.78(s, 3H), 7.14 (s, 1H), 7.86 (d, 1H, *J*=7.8Hz), 7.95 (s, 1H), 8.02 (d,

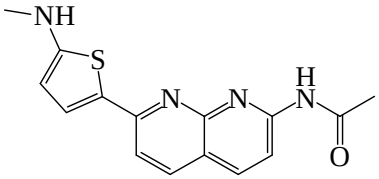
1H, *J*=8.0Hz), 8.30 - 8.41 (m, 3H), ^{13}C NMR (DMSO-d_6, 100MHz) : 21.50, 28.92, 114.21, 116.84, 119.28, 127.11, 127.93, 138.11, 139.61, 143.85, 154.81, 155.02, 155.28, 169.53, 170.58. Mass: m/z 371 $[M+H]^+$.

tert-butyl (5-(2-acetamido-1,8-naphthyridin-7-yl) thiophen-2-yl)carbamate (4g)



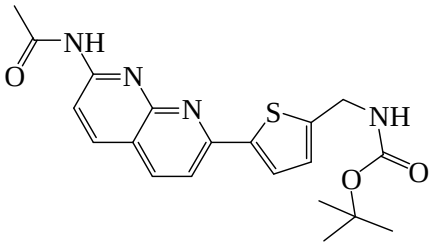
^{1}H NMR (DMSO-d_6, 400MHz) : 1.49 (s, 9H), 2.18(s, 3H), 7.11 (s, 1H), 7.62 (d, 1H), 7.85 (d, 1H, *J*=7.6Hz), 7.92 (s, 1H), 8.02 (d, 1H, *J*=8.2Hz), 8.32-8.40 (m, 3H); ^{13}C NMR (DMSO-d6, 100MHz) : 21.58, 24.83, 113.89, 116.87, 119.28, 127.15, 127.91, 138.10, 139.61, 143.86, 154.86, 155.02, 156.27, 170.51. Mass: m/z 385 $[M+H]^+$.

N-(7-(5-(methylamino)thiophen-2-yl)-1,8-naphthyridin-2-yl)acetamide (4h)



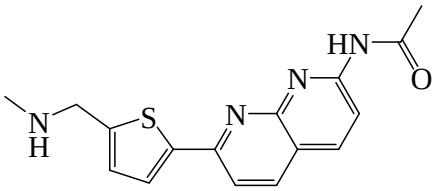
^{1}H NMR (DMSO-d_6, 400MHz) : 2.19 (s, 3H), 2.78 (s, 3H), 7.13 (s, 1H), 7.65 (d, 1H), 7.86 (d, 1H, *J*=7.6Hz), 7.90 (s, 1H), 8.04 (d, 1H, *J*=8.2Hz), 8.31 - 8.40 (m, 3H); ^{13}C NMR (DMSO-d_6, 100MHz) : 24.57, 35.24, 114.29, 116.83, 119.26, 127.10, 127.92, 138.11, 139.61, 143.85, 154.88, 155.01, 155.27, 170.53. Mass: m/z 299 $[M+H]^+$.

tert-butyl-((5-(7-acetamido-1,8-naphthyridin-2-yl)thiophen-7-yl)methyl)carbamate (4i)



^{1}H NMR (DMSO-d_6, 400MHz) : 1.48 (s, 9H), 2.17(s, 3H), 4.12 (s, 2H), 7.12 (s, 1H), 7.63 (d, 1H), 7.84 (d, 1H, *J*=7.68Hz), 7.98 (s, 1H), 8.04 (d, 1H, *J*=8.4Hz), 8.30-8.40 (m, 3H), ^{13}C NMR (DMSO-d_6, 100MHz) : 21.58, 24.82, 49.75, 113.89, 116.83, 119.26, 127.10, 127.92, 138.10, 139.61, 143.85, 154.87, 155.02, 155.02, 155.27, 170.51. Mass: m/z 399 $[M+H]^+$.

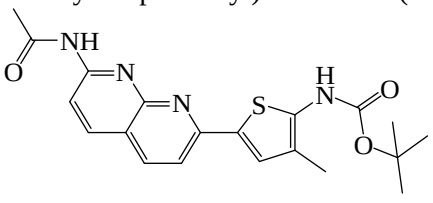
N-(7-(5-((methylamino)methyl)thiophen-2-yl)-1,8-naphthyridin-2-yl)acetamide (4j)



^{1}H NMR (DMSO-d_6, 400MHz) : 2.18 (s, 3H), 2.77(s, 3H), 4.22 (s, 2H), 7.13 (s, 1H), 7.60 (d, 1H), 7.83 (d, 1H, *J*=7.8Hz), 7.95 (s, 1H), 8.03 (d, 1H, *J*=8.4Hz), 8.31 - 8.41 (m, 3H), ^{13}C NMR (DMSO-d_6, 100MHz) : 24.57, 35.24, 49.75, 114.29, 116.83, 119.26, 127.10, 127.92, 138.11, 139.61, 143.85, 154.88, 155.01, 155.29, 170.52.

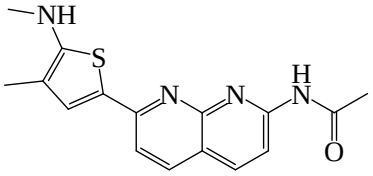
Mass: m/z 313 $[M+H]^+$.

tert-butyl-(5-(2-acetamido-1,8-naphthyridin-2-yl)-3-methylthiophen-7-yl) carbamate (4k)



^{1}H NMR (DMSO-d_6, 400MHz) : 1.51 (s, 9H), 2.18(s, 3H), 2.34 (s, 3H), 7.11 (s, 1H), 7.86 (d, 1H, *J*=7.8Hz), 7.90 (s, 1H), 8.03 (d, 1H, *J*=8.0Hz), 8.32 - 8.40 (m, 3H); ^{13}C NMR (DMSO-d_6, 100MHz) : 21.58, 24.83, 28.85, 113.89, 116.87, 119.28, 127.15, 127.91, 138.10, 139.69, 144.88, 154.86, 154.82, 156.21, 170.50. Mass: m/z 399 $[M+H]^+$.

N-(7-(4-methyl-5-(methylamino)thiophen-2-yl)-1,8-naphthyridin-2-yl)acetamide (4l)



^{1}H NMR (DMSO-d_6, 400MHz) : 2.18 (s, 3H), 2.33 (s, 3H), 2.79(s, 3H), 7.13 (s, 1H), 7.87 (d, 1H, *J*=7.8Hz), 7.92 (s, 1H), 8.04 (d, 1H, *J*=8.0Hz), 8.31 - 8.41 (m, 3H), ^{13}C NMR (DMSO-d_6, 100MHz) : 24.58, 28.92, 114.28, 116.84, 119.28, 127.11, 127.93, 138.11, 139.61, 143.85, 154.88, 155.02, 155.28, 170.58. Mass: m/z 313 $[M+H]^+$.

Conclusion

We have prepared thiophene containing 1,8-Naphthyridine derivatives which are potential drugs. The beauty of this methodology is used for the preparation of industrial scale. The attractive features of this procedure are the mild reaction conditions, high conversions, operational simplicity and inexpensive.

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