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THREE STEP SYNTHESIS OF SERIES OF 7-(4-NITROPHENYL)-1-((1*H*-1,2,3-TRIAZOL-4-YL))-*1H*-IMIDAZO[4,5-b][1,8]NAPHTHYRIDINES

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ABSTRACT: Herein, we report the heterocycles synthesis of 7-(4-nitrophenyl)-1-((1*H*-1,2,3-triazol-4-yl))-1*H*-imidazo[4,5-b][1,8]naphthyridines (4a-l) through 1,3-dipolar cycloaddition in between various azides and terminal alkynes such as 7-(4-nitrophenyl)-1-(prop-2-yn-1-yl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (3) by means of CuI/DMF solvent at 80 ^{o}C temperature, during 10-12h to give desired1,4-substituted regio triazoles with promising yileds. Compound (3) is formed *N*-propargylation of 7-(4-nitrophenyl)-1*H*-imidazo[4,5-b] [1,8] naphthyridine (2) by using Propargyl bromide, Cs_2CO_3, DMF. Furthermore, structures are confirmed by spectral analysis.

KEYWORDS: 1,2,3-Triazole, Propargylation, Imidazole, [1,8] naphthyridines, CuI

INTRODUCTION:

The benzimidazole ring system, a common substructure in heterocyclic pharmacophores, is privileged due to their frequent presence in bioactive compounds, with primary interest in their biological activities like anti-cancer, fungicidal, analgesic, anti-viral properties, cardiovascular and HIV infectivity [i-x].

We are going to report 3 step synthesis of 7-(4-nitrophenyl)-1-((1*H*-1,2,3-triazol-4-yl))-1*H*-imidazo[4,5-b][1,8] naphthyridines(4a-I) depicted in Figure 1 below.

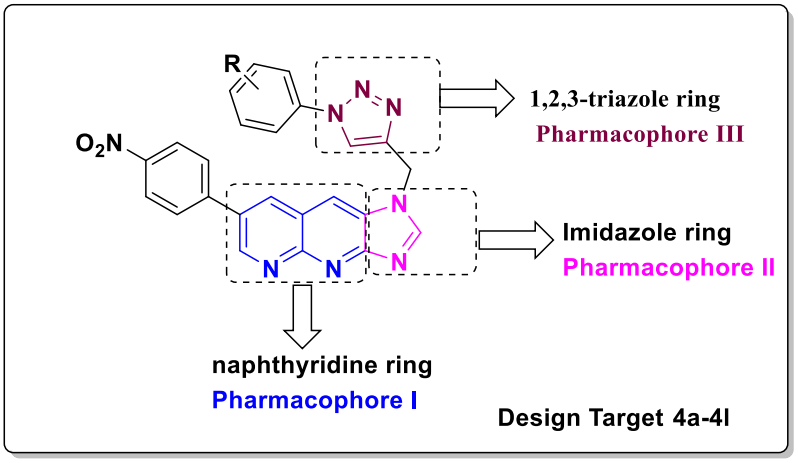


Figure 1

EXPERIMENTAL:

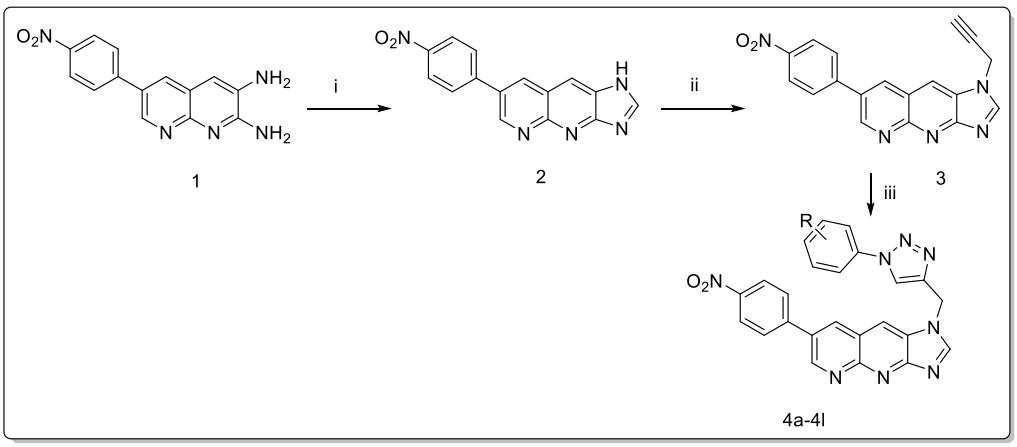
The progress of the reaction was monitored by thin layer chromatography on silica gel coated aluminum plates (Merck) as adsorbent. The purity of the compounds was monitored by TLC. ^{1}H-NMR spectra were recorded on Varian 500 MHz NMR spectrophotometer using DMSO-d_6 ^{13}C-NMR spectra were recorded on Varian 125 MHz NMR spectrophotometer using, DMSO-d_6 as solvent and TMS as an internal standard (chemical shifts in δ ppm). mass spectra were recorded on Low-resolution mass spectra (LRMS) data were measured on GCMS-QP2010 Ultra.

GENERAL PROCEDURE:

RESULTS AND DISCUSSION:

Synthesis of 7-(4-nitrophenyl)-1*H*-imidazo[4,5-b][1,8] naphthyridine (2)

Half gram (0.0046 mol) of 6-(4-nitrophenyl)-1,8-naphthyridine-2,3-diamine (1) and 0.32 g (3 ml, 0.062 mol) of 90% formic acid was added in Erlenmeyer flask and the reaction mixture was homogeneously mixed. The mixture was heated at 20% for 80 s, the flask was then removed, cooled and 10% sodium hydroxide solution was added slowly, with constant stirring, until the mixture was just alkaline to litmus. The crude imidazo naphthyridine (2) was filtered off and washed thrice with ice-cold water and dried. The crude product obtained was dissolved in boiling water, 0.2 g of decolorizing carbon was added and digested for 10 min then filtered rapidly at 10 °C. The product was filtered off, washed with cold water and dried at 100 °C. Recrystallized the product with methanol and weighed [xi].



Scheme 1: *Reagents & conditions*. (i) Formic acid, heat; (ii) Propargyl bromide, Cs_2CO_3, DMF, 80 °C, 8h;(iii) Ar-N_3, CuI, DMF, 80 °C, 10-12h.

Synthesis of 7-(4-nitrophenyl)-1-(prop-2-yn-1-yl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (3)

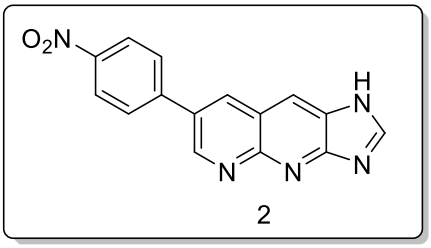
The reaction of 7-(4-nitrophenyl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (2) Propargyl bromide(1 mmol), Cs_2CO_3, DMF, reaction to examine the feasibility of N-propargylation under cited conditions, 80 ^oC, temperatures for an appropriate time 8h, (Scheme 1)on *N*-propargylation to yield desired product 7-(4-nitrophenyl)-1-(prop-2-yn-1-yl)-1H-imidazo[4,5-b][1,8]naphthyridine (3).

Synthesis of 7-(4-nitrophenyl)-1-((1-phenyl-1*H*-1,2,3-triazol-4-yl)methyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4a)

7-(4-nitrophenyl)-1-(prop-2-yn-1-yl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (3) is treated with aryl azides, followed by CuI, by means of solvent DMF, at 80 ^oC, to 10-12h to yield desired product 4a, via 3+2 1,3- dipolar cyclo addition viz quick protocols. By using various azides 4b to 4l different triazoles are obtained is shown in Scheme 1[xii] .

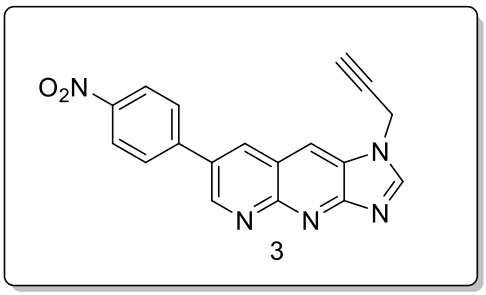
Characterization data

7-(4-nitrophenyl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (2): ^{1}H-NMR (500 MHz, DMSO-d_6): δ 9.20 (s, 1H), 8.46 (s, 1H), 8.32 (d, *J* = 8.0 Hz, 2H), 8.15 (s, 1H), 7.99 (d, *J*=8.0 Hz, 2H), 7.85(s, 1H), 6.72 (brs, 1H, -NH) ;^{13}C-NMR (125 MHz, DMSO-d_6): δ 156.34, 149.99 (2C), 149.74 , 147.84, 143.91, 143.32 , 133.66, 133.44 , 129.46, 126.48 , 124.44 (2C), 120.10 (2C), ESI-MS (m/z): Cal for $C_{15}H_9N_5O_2$: $[M]^+$: 291, found: 292 $[M+H]^+$.



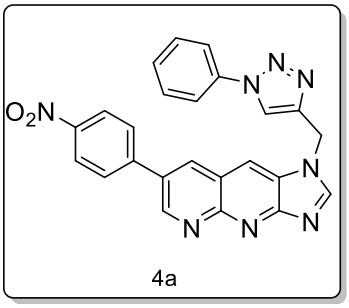
2

7-(4-nitrophenyl)-1-(prop-2-yn-1-yl)-1*H*-imidazo[4,5-b][1,8]naphthyridine (**3**): ^{1}H-NMR (500 MHz, DMSO-d_6): δ 9.18 (s, 1H), 8.45 (s, 1H), 8.33 (d, *J* = 8.0 Hz, 2H), 8.13 (s, 1H), 7.98 (d, *J*=8.0 Hz, 2H), 7.83(s, 1H), 3.73 (d, J=4.0 Hz, 2H, N-CH2), 2.02(t, J=4.0Hz, 1H, CH-alkyne) ;^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99 , 148.39 (2C), 147.84 (2C), 143.32, 133.75 , 133.13 , 129.46 , 126.50 , 124.43 (2C), 121.38 , 73.39 , 70.16 , 37.21 , ESI-MS (m/z): Cal for $C_{18}H_{11}N_5O_2$: $[M]^+$: 329, found: 330 $[M+H]^+$.



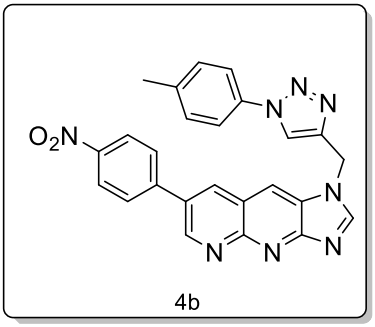
3

7-(4-nitrophenyl)-1-((1-phenyl-1*H*-1,2,3-triazol-4-yl)methyl)-1H-imidazo[4,5-b][1,8]naphthyridine 4a: ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (s, 1H), 8.61 (s, 1H), 8.32 (d, *J* = 7.5 Hz, 2H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.46 (d, *J* = 7.6 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 1H), 5.96 (s, 1H), 5.76 (s, 1H) ;^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99(2C), 148.08(2C), 147.08,143.32, 139.31, 137.08 , 133.75, 133.13, 129.46(4C),127.60(2C), 126.50, 126.33, 124.43 (2C), 121.38(2C) , 41.43,ESI-MS (m/z): Cal for $C_{24}H_{16}N_8O_2$: $[M]^+$: 448, found: 449 $[M+H]^+$.



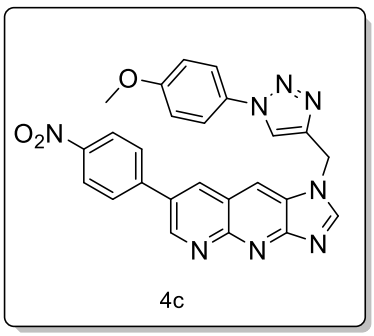
4a

7-(4-nitrophenyl)-1-((1-(p-tolyl)-1*H*-1,2,3-triazol-4-yl)methyl)-1H-imidazo[4,5-b][1,8]naphthyridine 4b: ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.70 (s, 1H), 8.61 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.43 (d, *J* = 7.5 Hz, 2H), 7.06 (d, *J* = 7.5 Hz, 2H), 5.95 (s, 1H), 5.76 (s, 1H), 2.32 (s, 3H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 147.84 , 143.32, 139.31, 137.81, 136.90, 133.75, 133.13, 129.46, 129.04, 126.50, 124.43, 124.16, 121.38 , 41.43, 21.12, ESI-MS (m/z): Cal for $C_{25}H_{18}N_8O_2$: $[M]^+$: 462, found: 463 $[M+H]^+$.



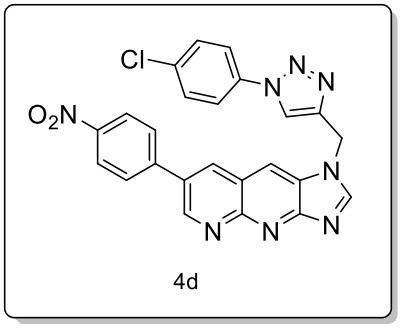
4b

1-((1-(4-methoxyphenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4c): ^{1}H-NMR (500 MHz, DMSO-d_6): δ 9.16 (s, 1H), 8.45 (s, 1H), 8.31 (d, *J* = 8.0 Hz, 2H), 8.14 (s, 1H), 8.12 (s, 1H, tri-H), 7.97 (d, *J*=8.0 Hz, 2H), 7.88 (s, 1H), 7.70 (d, J= 8.0Hz, 2H), 7.00 (d, J=8.0Hz, 2H), 5.23 (s, 2H, N-CH_2), 3.84 (s, 3H, -OCH_3); ^{13}C-NMR (125 MHz, DMSO-d_6): 159.35, 151.09, 148.81, 147.74, 146.27, 144.49, 142.80, 139.69, 135.27, 133.65, 131.17, 129.43(2C), 126.65, 125.87, 124.80(2C), 123.68, 123.07(2C), 121.47, 114.88(2C), 56.74, 42.47; ESI-MS (m/z): Cal for $C_{25}H_{18}N_8O_3$: $[M]^+$: 478, found: 479 $[M+H]^+$.



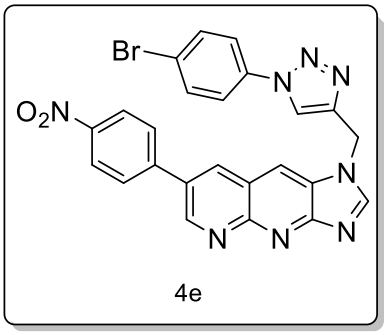
4c

1-((1-(4-chlorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4d): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (t, *J* = 1.5 Hz, 1H), 8.60 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 7.5 Hz, 2H), 5.95 (s, 1H), 5.76 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 147.84, 143.32, 139.31, 136.01, 133.75, 133.13, 132.65, 129.46(2C), 126.50 , 126.33, 124.43(2C) , 122.93(2C) , 121.38, 41.43 ; ESI-MS (m/z): Cal for $C_{24}H_{15}ClN_8O_2$: $[M]^+$: 482, found: 484 [M+2].



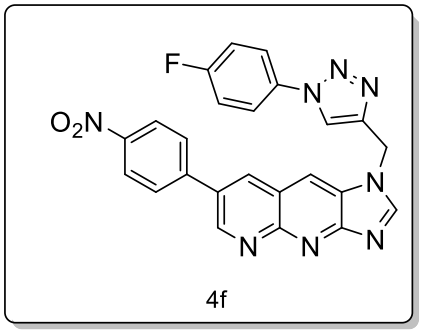
4d

1-((1-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4e): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (t, *J* = 1.5 Hz, 1H), 8.60 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 7.5 Hz, 2H), 5.95 (s, 1H), 5.76 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 147.84, 143.32, 139.31, 136.01, 133.75(2C), 132.65, 129.46, 129.01, 126.50 , 126.33, 124.43(2C) , 122.93(2C) , 121.38, 41.43 ; ESI-MS (m/z): Cal for $C_{24}H_{15}BrN_8O_2$: $[M]^+$: 526, found: 528 [M+2].



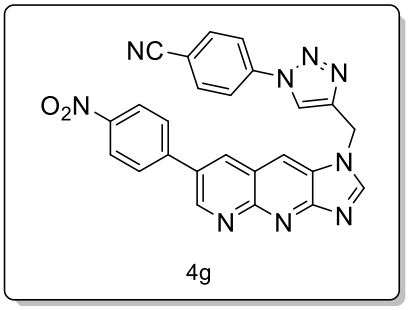
4e

1-((1-(4-fluorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4f): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (t, *J* = 1.5 Hz, 1H), 8.60 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 7.5 Hz, 2H), 5.95 (s, 1H), 5.76 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 147.84, 143.32, 139.31, 136.01, 133.75, 133.13(2C), 129.46, 129.01, 126.50, 126.33, 124.43(2C), 122.93(2C), 121.38, 41.43; ESI-MS (m/z): Cal for $C_{24}H_{15}FN_8O_2$: $[M]^+$: 466, found: 467 $[M+H]^+$.



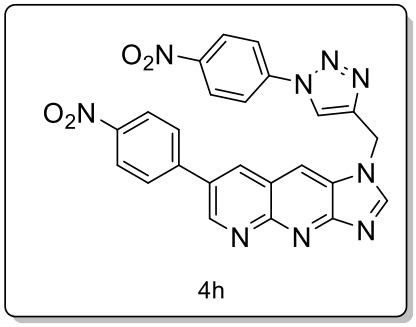
4f

4-(4-((7-(4-nitrophenyl)-1*H*-imidazo[4,5-b][1,8]naphthyridin-1-yl)methyl)-1H-1,2,3-triazol-1-yl)benzonitrile (4g): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (s, 1H), 8.60 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.25 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.68 (d, *J* = 7.5 Hz, 2H), 7.54 (s, 2H), 5.96 (s, 1H), 5.77 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97 , 149.99 , 147.84(2C) , 143.32(2C), 139.31 , 133.75 , 133.13 , 129.46(2C) , 126.50 , 126.33 , 125.38, 125.22 , 124.43(2C), 121.38 , 119.12, 116.24(2C), 41.43; ESI-MS (m/z): Cal for $C_{25}H_{15}N_9O_2$: $[M]^+$: 473, found: 474 $[M+H]^+$.



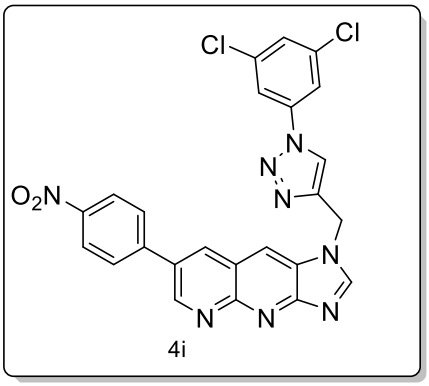
4g

7-(4-nitrophenyl)-1-((1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4h):^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.69 (s, 1H), 8.61 (s, 1H), 8.33 (s, 1H), 8.32 (s, 1H), 8.25 (s, 1H), 8.08 (s, 1H), 8.04 (s, 1H), 8.02 (s, 1H), 7.88 (s, 1H), 7.86 (s, 1H), 7.77 (s, 1H), 7.75 (s, 1H), 5.97 (s, 1H), 5.77 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 148.08, 147.84(2C), 146.86, 143.32, 140.69, 139.31, 133.75, 133.13, 129.46, 126.50(2C), 124.36 , 122.47(2C), 121.38, 41.43 ; ESI-MS (m/z): Cal for $C_{24}H_{15}N_9O_4$: $[M]^+$: 493, found: 494 $[M+H]^+$.



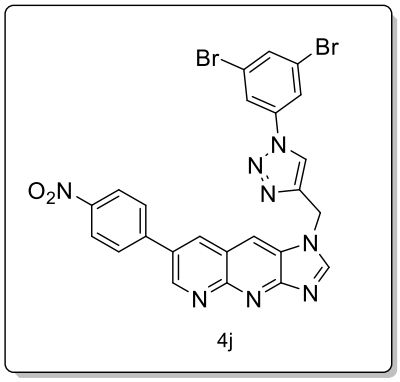
4h

1-((1-(3,5-dichlorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4i):^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.35 (s, 1H), 8.70 (d, *J* = 1.4 Hz, 1H), 8.61 (s, 1H), 8.32 (d, *J* = 7.5 Hz, 2H), 8.24 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.57 (s, 2H), 7.19 (s, 1H), 5.95 (s, 1H), 5.76 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97 , 149.99 , 147.84(2C), 143.32, 135.29, 133.75(2C) , 133.13, 129.46(2C), 126.50, 126.33 , 124.60(2C) , 121.38 , 118.71(2C) , 41.43; ESI-MS (m/z): Cal for $C_{24}H_{14}Cl_2N_8O_2$: $[M]^+$: 516, found: 518 [M+2].



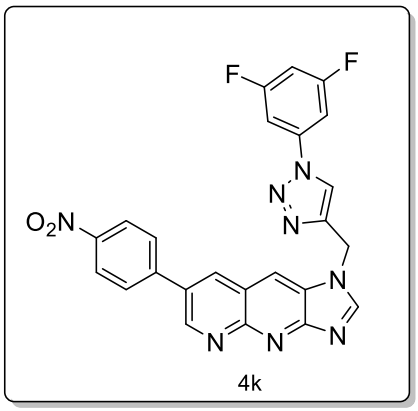
4i

1-((1-(3,5-dibromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4j):^{1}H-NMR (500 MHz, DMSO-d_6): δ 9.35 (s, 1H), 8.69 (s, 1H), 8.61 (s, 1H), 8.32 (d, *J* = 7.5 Hz, 2H), 8.25 (s, 1H), 8.08 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.75 (s, 2H), 7.52 (s, 1H), 5.95 (s, 1H), 5.76 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 152.97, 149.99, 147.84(2C), 143.32 , 139.31, 137.67, 133.75(2C), 129.46, 129.11, 126.50, 126.33, 124.43(2C), 124.12, 121.38, 41.43 ; ESI-MS (m/z): Cal for $C_{24}H_{14}Br_2N_8O_2$: $[M]^+$: 606, found: 608 [M+2].



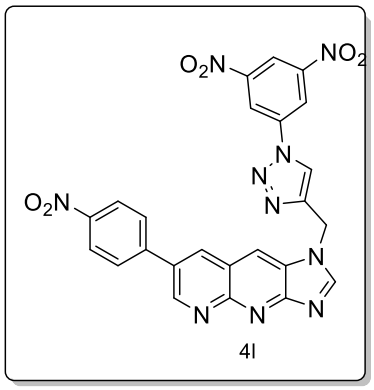
4j

1-((1-(3,5-difluorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4k): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.21 (s, 1H), 8.67 (s, 1H), 8.54 (s, 1H), 8.17 (s, 1H), 8.16 (s, 1H), 8.08 (s, 2H), 7.85 (d, *J* = 7.4 Hz, 2H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.72 (d, *J* = 8.1 Hz, 1H), 5.83 (s, 1H), 5.70 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 164.42, 162.38, 152.97, 149.99 , 148.08, 147.84 (2C), 143.32, 139.31, 135.74 , 133.75 , 133.13, 129.46 , 126.50, 126.33, 124.36(2C), 121.38, 102.63, 102.39, 41.43; ESI-MS (m/z): Cal for $C_{24}H_{14}F_2N_8O_2$: $[M]^+$: 484, found: 485 $[M+H]^+$.



4k

1-((1-(3,5-dinitrophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-7-(4-nitrophenyl)-1H-imidazo[4,5-b][1,8]naphthyridine (4l): ^{1}H-NMR (500 MHz, DMSO-d_6):δ 9.21 (s, 1H), 8.67 (s, 1H), 8.54 (s, 1H), 8.17 (s, 1H), 8.16 (s, 1H), 8.08 (s, 2H), 7.85 (d, *J* = 7.4 Hz, 2H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.72 (d, *J* = 8.1 Hz, 1H), 5.83 (s, 1H), 5.70 (s, 1H);^{13}C-NMR (125 MHz, DMSO-d_6): δ 164.42 , 162.38, 152.97, 149.99, 148.08, 147.84 (2C), 143.32, 139.31, 135.74, 133.75 , 133.13 , 129.46 , 126.50 , 126.33 (2C), 124.36 , 121.38 (2C), 102.63 , 102.39 , 41.43 ; ESI-MS (m/z): Cal for $C_{24}H_{14}N_{10}O_6$: $[M]^+$: 538, found: 539 $[M+H]^+$.



4l

CONCLUSION:

We have reported the three-step synthesis of 7-(4-nitrophenyl)-1-((1*H*-1,2,3-triazol-4-yl))-1*H*-imidazo[4,5-b][1,8] naphthyridines(4a-I) with good yields by joining three pharmacophores shown in Fig 1 and Scheme 1 under click protocols.

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