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Design, synthesis and antibacterial activity of 9-aryl-6-(2-naphthyl) [1,2,4]triazolo[4,3-*a*][1,8]naphthyridines

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A concise and highly efficient procedure has been described for the synthesis of 9-aryl-6-(2-naphthyl)[1,2,4] triazolo[4,3-*a*][1,8]naphthyridines**8** by the oxidation of the corresponding aryl aldehyde1-[3-(2-naphthyl) [1,8]naphthyridin-2-yl]hydrazones **7** using silica gel supported ferric chloride (SiO_2-$FeCl_3$) in solvent-free conditions under microwave irradiation. The desired products are obtained in very good yields and in a state of high purity. The structure of compounds **3-8** have been confirmed by their spectroscopic (IR, ^{1}H NMR and MS) and analytical data. The compounds **8a-j** have been screened for their antibacterial activity.

Keywords: 1,8-Naphthyridine, 1,2,4-triazole, SiO_2-$FeCl_3$, microwave irradiation, antibacterial activity

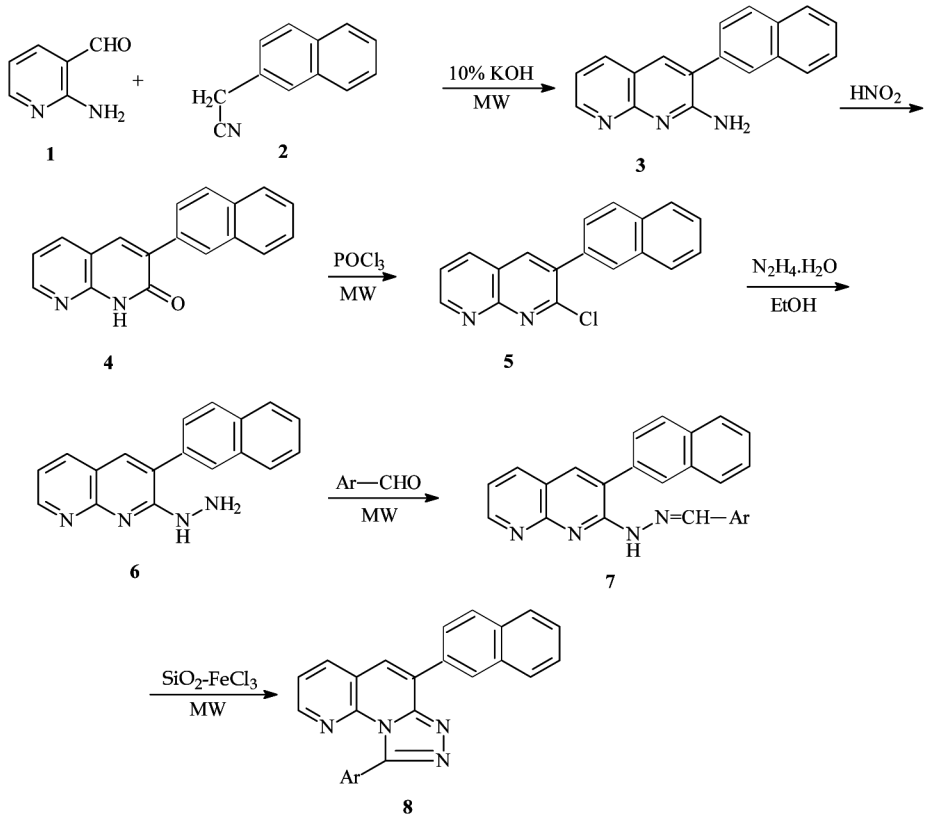
1,8-Naphthyridines are an important group of heterocyclic compounds possessing a variety of biological activities[1-3]. Fused 1,2,4-triazoles are of interest to many researchers in diverse fields[4-6]. Though various routes for the synthesis of these compounds are known[7-12], the majority of them involve longer preparation time, high reaction temperature, low yields and usage of toxic reagents. Therefore, development and introduction of convenient and efficient method for the preparation of fused 1,2,4-triazoles are pharmaceutical importance and is still in demand. In recent years, microwave irradiation, as a high energy technique, has been used in organic synthesis[13-15]. The application of microwave (MW) heating under solvent-free reaction conditions[14] and on inorganic solid support[16,17] is promising alternative to polluting reactions and has been a current field of interest. In view of this and in continuation of our interest in the microwave-assisted organic transformations of 1,8-naphthyridine derivatives[18-20], we report herein a convenient, efficient and high yielding protocol for the synthesis of 9-aryl-6-(2-naphthyl)[1,2,4]triazolo [4,3-*a*] [1,8]naphthyridines using silicagel supported ferric chloride (SiO_2-$FeCl_3$) in solvent-free conditions under microwave irradiation.

2-Aminonicotinaldehyde **1** on condensation with 2-naphthylacetonitrile **2** in the presence of 10% KOH without any solvent under MW irradiation afforded 3-(2-naphthyl)[1,8]naphthyridin-2-amine **3,** which is converted into 3-(2-naphthyl)-1,2-dihydro[1,8] naphthyridin-2-one **4** by reaction with HNO_2. Treatment of **4** with $POCl_3$ under MW irradiation furnished 2-chloro-3-(2-naphthyl)[1,8] naphthyridine **5**, which on hydrazinolysis with refluxing hydrazine hydrate resulted in the formation of 1-[3-(2-naphthyl)[1,8]naphthyridin-2-yl]hydrazine **6**.

Condensation of **6** with various aromatic aldehydes in the presence of catalytic amount of DMF under MW irradiation afforded the corresponding aryl aldehyde 1-[3-(2-naphthyl)[1,8]naphthyridin-2-yl] hydrazones **7** in excellent yields.

The hydrazones **7** on oxidative cyclization with silica gel supported ferric chloride (SiO_2-$FeCl_3$) in solvent-free conditions under MW irradiation furnished the respective 9-aryl-6-(2-naphthyl) [1,2,4] triazolo[4,3-*a*][1,8]naphthyridines **8a-j** (Scheme I) in very good yields (88-94%) with short reaction time period (3.5-5.0 min). The oxidative transformation is simple, rapid and efficient and is devoid of any side products. The process is environmentally benign. The experimental procedure is very simple.

In a typical experiment, **7a** (Ar = C_6H_5) was mixed with SiO_2-$FeCl_3$ and the mixture was exposed to MW irradiation at 400 W intermittently at 30 sec intervals for 4.0 min. The reaction mixture was cooled to RT and digested with methanol. After work-up 9-phenyl-6-(2-naphthyl)[1,2,4]triazolo[4,3-*a*][1,8]naphthyridines **8a-j**



Scheme I

(Ar = C_6H_5) was obtained in 90% yield. The generality of the above transformation was checked by treating other hydrazones **7** with SiO_2-$FeCl_3$ under MW irradiation and in all cases respective 9-aryl-6-(2-naphthyl)[1,2,4]triazolo[4,3-*a*][1,8]naphthyridines**8** were obtained in 88-94% yields in Table I.

The structural assignment of compounds **3-8** were based on their elemental analyses and spectral (IR, ^{1}H NMR, ^{13}CNMR and MS) data in Table II. High yields of the products, excellent purity, short reaction times; simple operation, inexpensive and non-toxicity of the reagent are noteworthy advantages of this method.

Antibacterial activity

The antibacterial activity of the title compounds **8a-j** were examined against the bacteria *Escherichia coli* (gram-negative) and *Bacillus substilis* (gram-positive) by filter paper disc technique of Vincent and Vincent[21] at 250 and 500 µg/disc concentrations. Standard antibacterial Gentamycin was also screened under similar conditions for comparison. The results are presented in Table III.

Experimental Section

Melting points were measured in open capillaries on a Cintex melting point apparatus and are uncorrected. Purity of the compounds was checked using precoated TLC plates (Merk, 60F-254). IR spectra (KBr) were recorded on a Perking-Elmer spectrum BX series FT-IR spectrophotometer, ^{1}H NMR spectra on a Varian Gemini 300 MHz spectrometer, ^{13}CNMR 100 MHz spectrometer, (chemical shifts in δ ppm) and mass spectra on a Finnigan MAT-1020, automated GC-MS and VG Auto Spec-M instruments. Elemental analyses

Table I — Table showing Physical and analytical data of compounds **7** and **8**

Compd	Reaction time (min)	m.p. (°C)	Yield (%)	Mol. Formula	Found (%) (Calcd)		
					C	H	N
7a	0.5	184	95	$C_{25}H_{18}N_4$	80.32 (80.19	4.87 4.85	15.01 14.96)
7b	1.0	194	97	$C_{26}H_{20}N_4$	80.53 (80.39	5.23 5.19	14.46 14.42)
7c	1.0	171	94	$C_{26}H_{20}N_4O$	77.33 (77.21	5.00 4.98	13.90 13.85)
7d	0.5	165	95	$C_{25}H_{17}N_4Cl$	73.57 (73.44	4.22 4.19	13.74 13.70)
7e	1.0	186	98	$C_{25}H_{17}N_4Cl$	73.56 (73.44	4.21 4.19	13.76 13.70)
7f	0.5	188	96	$C_{25}H_{17}N_4F$	76.65 (76.52	4.40 4.37	14.32 14.28)
7g	0.5	183	95	$C_{25}H_{17}N_5O_2$	71.74 (71.59	4.10 4.09	16.76 16.70)
7h	0.5	175	95	$C_{25}H_{17}N_5O_2$	71.72 (71.59	4.12 4.09	16.75 16.70)
7i	0.5	145	97	$C_{25}H_{17}N_5O_2$	71.73 (71.59	4.11 4.09	16.74 16.70)
7j	1.0	196	96	$C_{27}H_{22}N_4O_2$	74.78 (74.64	5.12 5.10	12.94 12.89)
8a	4.0	224	90	$C_{25}H_{16}N_4$	80.75 (80.63	4.36 4.33	15.08 15.04)
8b	4.0	210	92	$C_{26}H_{18}N_4$	80.94 (80.81	4.71 4.69	14.55 14.50)
8c	4.5	255	90	$C_{26}H_{18}N_4O$	77.73 (77.60	4.53 4.51	13.97 13.92)
8d	4.0	235	90	$C_{25}H_{15}N_4Cl$	73.92 (73.80	375 3.72	13.82 13.77)
8e	3.5	248	94	$C_{25}H_{15}N_4Cl$	73.94 (73.80	3.74 3.72	13.81 13.77)
8f	4.0	242	92	$C_{25}H_{15}N_4F$	77.03 (76.91	3.89 3.87	14.40 14.35)
8g	4.0	252	88	$C_{25}H_{15}N_5O_2$	72.08 (71.94	3.63 3.62	16.83 16.78)
8h	4.5	245	88	$C_{25}H_{15}N_5O_2$	72.07 (71.94	3.64 3.62	16.82 16.78)
8i	5.0	258	90	$C_{25}H_{15}N_5O_2$	72.06 (71.94	3.65 3.62	16.83 16.78)
8j	4.0	208	91	$C_{27}H_{20}N_4O_2$	75.12 (74.99	4.69 4.66	12.99 12.95)

were performed on a Perkin-Elmer 240 CHN elemental analyser. MWI was carried out microwave oven (LGMG 556P, 2450 MHz). The 2-naphthylacetonitrile **2** was purchased from Aldrich Chemical Company.

3-(2-Naphthyl)[1,8]naphthyridin-2-amine 3

A mixture of 2-aminonicotinaldehyde **1** (0.01 mol), 2-naphthylacetonitrile **2** (0.01 mol) and 10% KOH (5 drops) was exposed to MW irradiation at 200 W intermittently at 30 sec for 2.5 min. On completion of the reaction, as monitored by TLC, the reaction mixture was cooled, and filtered, washed with water and purified by recrystallization from methanol to afford **3,** yield 97%; m.p. 310°C.

3-(2-Naphthyl)-1,2-dihydro[1,8]naphthyridin-2-one 4

To a cold solution of **3**(0.01 mol) in 2 M HCl (25 mL) was added $NaNO_2$ solution (0.01 mole in 25 mL water) and the reaction mixture was stirred at room temperature for 0.5 hr and treated with chilled water. The solid that separated was filtered, washed with water and purified by recrystallization from methanol to give **4,** yield 95%; m.p. 183°C.

2-Chloro-3-(2-naphthyl)[1,8]naphthyridine 5

A mixture of **4** (0.01 mol) and $POCl_3$ (10 mL) was subjected to MW irradiation at 200 W intermittently at 30 sec intervals for 2.0 min. After completion of reaction, as indicated by TLC, the reaction mixture

Table II — IR, ^{1}H, ^{13}C NMR and MS data of compounds **7** and **8**

7a: IR (KBr): 3286 (NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.88 (m, 2H, C_5-H C_6-H), 8.15 (s, 1H, C_4-H), 8.34 (m, 1H, C_7-H), 8.45 (m, 1H, N=CH), 7.00-7.83 (m, 12H, Ar-H), 10.32 (s, 1H, NH); ESI-MS : *m/z* 375 $[M+H]^+$.

7b: IR (KBr): 3390(NH),1624 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 2.40 (s, 3H, CH_3), 7.85 (m, 2H, C_5-H, C_6-H), 8.14 (s, 1H, C_4-H), 8.36 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 6.98-7.72 (m, 11H, Ar-H), 10.30 (s, 1H, NH); ESI-MS : *m/z* 389 $[M+H]^+$.

7c: IR (KBr): 3395(NH), 1625 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 3.85 (s, 3H, OCH_3), 7.84 (m, 2H, C_5-H, C_6-H), 8.15 (s, 1H, C_4-H), 8.35 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 6.90-7.80 (m, 11H, Ar-H), 10.24 (s, 1H, NH); ESI-MS : *m/z* 405 $[M+H]^+$.

7d: IR (KBr): 3393(NH),1620 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.90 (m, 2H, C_5-H, C_6-H), 8.15 (s, 1H, C_4-H), 8.38 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 7.02-7.83 (m, 11H, Ar-H), 10.28 (s, 1H, NH); ESI-MS : *m/z* 409 $[M+H]^+$.

7e: IR (KBr): 3422(NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.85 (m, 1H, C_6-H), 8.13 (s, 1H, C_4-H), 8.37 (m, 1H, C_7-H), 8.40 (s, 1H, N=CH), 7.00-7.80 (m, 11H, Ar-H), 10.30 (s, 1H, NH); ESI-MS : *m/z* 409 $[M+H]^+$.

7f: IR (KBr): 3395(NH), 1625 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.88 (m, 2H, C_5-H, C_6-H), 8.16 (s, 1H, C_4-H), 8.27 (m, 1H, C_7-H), 8.38 (s, 1H, N=CH), 7.02-7.83 (m, 11H, Ar-H), 10.28 (s, 1H, NH); ESI-MS : *m/z* 393 $[M+H]^+$.

7g: IR (KBr): 3390(NH),1624 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.90 (m, 2H, C_5-H, C_6-H), 8.15 (s, 1H, C_4-H), 8.32 (m, 1H, C_7-H), 8.45 (s, 1H, N=CH), 7.00-7.82 (m, 11H, Ar-H), 10.28 (s, 1H, NH); ESI-MS : *m/z*420 $[M+H]^+$.

7h: IR (KBr): 3432(NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.86 (m, 2H, C_5-H, C_6-H), 8.17 (s, 1H, C_4-H), 8.36 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 6.98-7.75 (m, 11H, Ar-H), 10.32 (s, 1H, NH); ESI-MS : *m/z*420 $[M+H]^+$.

7i: IR (KBr): 3427(NH), 1620 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 7.88 (m, 2H, C_5-H, C_6-H), 8.18 (s, 1H, C_4-H), 8.35 (m, 1H, C_7-H), 8.44 (s, 1H, N=CH), 7.05-7.86 (m, 11H, Ar-H), 10.30 (s, 1H, NH); ESI-MS : *m/z* 420 $[M+H]^+$.

7j: IR (KBr): 3393(NH),1625 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 3.86 (s, 3H, OCH_3), 3.96 (s, 3H, OCH_3), 7.85 (m, 2H, C_5-H, C_6-H), 8.18 (s, 1H, C_4-H), 8.37 (m, 1H, C_7-H), 8.52 (s, 1H, N=CH), 7.05-7.78 (m, 10H, Ar-H), 10.30 (s, 1H, NH); ESI-MS : *m/z*435$[M+H]^+$.

8a: IR (KBr): 3286 (NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.20 (m, 2H, C_3-H, C_5-H), 8.45 (m, 1H, C_4-H), 8.80 (m, 1H, C_2-H), 7.23-8.02 (m, 12H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 156.11,152.89,150.29, 140.23, 132.70,126.56,121.21;ESI-MS: *m/z* 373 $[M+H]^+$.

8b: IR (KBr): 3390(NH),1624 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 2.48 (s, 3H, CH_3), 8.18 (m, 2H, C_3-H, C_5-H), 8.45 (m, 1H, C_4-H), 8.78 (m, 1H, C_2-H), 7.25-8.00 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 160.41, 152.89, 148.37,129.23, 127.72, 130.70,118.61; ESI-MS: *m/z* 387$[M+H^]^+$.

8c: IR (KBr): 3395(NH), 1625 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 3.92 (s, 3H, OCH_3), 8.17 (m, 2H, C_3-H, C_5-H), 8.40 (m, 1H, C_4-H), 8.76 (m, 1H, C_2-H), 7.04-8.02 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 160.41, 156.11, 150.29, 140.23, 133.66 126.99, 114.85, 114.85; ESI-MS: *m/z* 403 $[M+H]^+$.

8d: IR (KBr): 3393(NH),1620 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.22 (m, 2H, C_3-H, C_5-H), 8.45 (m, 1H, C_4-H), 8.74 (m, 1H, C_2-H), 7.18-8.04 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 156.11, 140.23, 133.66, 128.3, 124.24, 126.56, 119.11, 114.8; ESI-MS: *m/z* 407 $[M+H]^+$.

8e: IR (KBr): 3422(NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.20 (m, 3H, C_3-H, C_5-H), 8.47 (m, 1H, C_4-H), 8.78 (m, 1H, C_2-H), 7.25-8.02 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 156.11, 150.29, 140.23,132.70, 126.56, 124.24, 118.62; ESI-MS: *m/z* 407 $[M+H]^+$.

8f IR (KBr): 3427(NH), 1622 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.22 (m, 2H, C_3-H, C_5-H), 8.46 (m, 1H, C_4-H), 8.76 (m, 1H, C_2-H), 7.22-8.04 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ163.35, 156.11, 150.29, 148.37,136.39,126.56, 123.86; ESI-MS: *m/z* 391 $[M+H]^+$.

8g: IR (KBr): 3390(NH),1624 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.19 (m, 2H, C_3-H, C_4-H), 8.45 (m, 1H, C_5-H), 8.78 (m, 1H, C_2-H), 7.24-8.03 (m, 11H, Ar-H); ^{13}CNMR($CDCl_3$): δ 152,11,148.11, 135.22,134.21,133.41,131.67, 129.67, 128.11, 126.21, 125.76,124.51,122.11, 121.11; ESI-MS: *m/z* 418 $[M+H]^+$.

8h: IR (KBr): 3383(NH),1621 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.17 (m, 2H, C_3-H, C_4-H), 8.44 (m, 1H, C_5-H), 8.82 (m, 1H, C_2-H), 7.20-8.00 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 152.11,148.97,134.22,130.22,128.66,121.11; ESI-MS: *m/z* 418 $[M+H]^+$.

8i: IR (KBr): 3422(NH), 1625 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 8.22 (m, 2H, C_3-H, C_4-H), 8.47 (m, 1H, C_5-H), 8.80 (m, 1H, C_2-H), 7.23-8.05 (m, 11H, Ar-H); ^{13}CNMR ($CDCl_3$): δ 152.11,148.97,133.22,130.22,128.68,121.11,56.25;ESI-MS: *m/z* 418 $[M+H]^+$.

8j: IR (KBr): 3387(NH), 1620 cm^{-1}(C=N); ^{1}H NMR ($CDCl_3$): δ 3.92 (s, 3H, OCH_3), 4.00 (s, 3H, OCH_3), 8.38 (m, 2H, C_3-H, C_5-H), 8.48 (m, 1H, C_4-H), 8.77 (m, 1H, C_2-H), 7.00-8.02 (m, 10H, Ar-H); ^{13}CNMR ($CDCl_3$):δ 156.11, 152.89, 148.37, 133.66,112.24,119.11,126.80,56.15; ESI-MS: *m/z* 433 $[M+H]^+$.

was cooled and poured onto a mixture of crushed ice and $NaHCO_3$. The solid that separated was filtered, washed with water and purified by recrystallization from ethanol to afford **5,** yield 94%; m.p. 208°C.

1-[3-(2-Naphthyl)[1,8]naphthyridin-2-yl]hydrazine 6

A mixture of **5** (0.01 mol) and hydrazine hydrate (0.015 mol) in ethanol (20 mL) was refluxed on a water bath for 4.0 hr. The reaction mixture was cooled and treated with cold water. The solid that separated was filtered, washed with water and purified by recrystallization from ethanol to furnish **6,** yield: 95%; m.p. 176°C.

General procedure for the synthesis of aryl aldehyde 1-[3-(2-naphthyl)[1,8]naphthyridin-2-yl]hydrazones 7

A mixture of **6** (0.01 mol), aromatic aldehyde (0.01 mol) and DMF (5 drops) was subjected to MW irradiation at 200 W intermittently at 10 sec intervals for the specified time in Table I. On completion of the

Table III — Antibacterial activity data of compounds **8a-j**

Compd	Inhibition zone (in mm)			
	E. coli at		*B. subtilis* at	
	250 µg/disc	500 µg/disc	250 µg/disc	500 µg/disc
8a	7.0	10.5	5.0	9.5
8b	8.5	16.0	5.5	10.0
8c	8.0	14.0	5.0	9.0
8d	9.5	18.5	6.0	10.5
8e	10.5	20.0	6.5	13.5
8f	9.0	17.5	6.0	12.0
8g	6.0	9.0	4.0	6.5
8h	5.5	8.0	4.0	6.0
8i	6.5	10.0	4.5	9.0
8j	8.5	13.5	5.5	10.0
Gentamycin	12.0	22.0	8.0	15.0

reaction, as monitored by TLC, the reaction mixture was cooled and digested with cold water. The solid thus obtained was filtered, washed with water and purified by recrystallization from ethanol to afford **7** in Table I.

General procedure for the synthesis of 9-aryl-6-(2-naphthyl)[1,2,4]triazolo[4,3-*a*][1,8]naphthyridines 8

Compound **7** (0.01 mol) and silica gel supported ferric chloride (SiO_2-$FeCl_3$) (2g) are mixed thoroughly and exposed to MW irradiation at 800 W intermittently at 30 sec intervals for the specified time in Table I. After complete conversion as indicated by TLC, the reaction mixture was cooled and treated with methanol (30 mL). The methanol solution was poured into ice cold water (50 mL), the separated solid was filtered, washed with water and purified by recrystallization from ethanol to furnish **8** in Table I.

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