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CHLORAMINE-T MEDIATED SYNTHESIS OF 9-ARYL-6-(3-METHOXYPHENYL) [1,2,4] TRIAZOLO [4,3-*a*] [1,8] NAPHTHYRIDINES UNDER MICROWAVE IRRADIATION

D. Praveena[1,*], E. Laxminarayana[2] and M. Ramesh[3]

[1]Department of Chemistry, S. R. Engineering College,
Warangal Urban-506 371, Telangana, India.
[2]Sreenidhi Institute of Science and Technology (Autonomous) Yannampet, Ghatkesar,
Hyderabad-501301, Telangana, India.
[3]Jawaharlal Nehru Technological University Kakinada, Kakinada, Andhra Pradesh, 533003,India
*E-mail: praveenadugyala@gmail.com

ABSTRACT

Hetero cyclic compounds fused on 1,2,4-triazoles have become attractive targets in organic synthesis due to their significant biological properties. We herein report Chloramine-T mediated green synthesis of some Naphthyridines.

Keywords: Fused 1,2,4-triazoles, 1,8- Naphthyridines, Chloramine-T, Microwave Irradiation, Non-toxic (Green)

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INTRODUCTION

Fused 1,2,4-triazoles are the biologically active molecules and their chemistry has received considerable interest.[1-4] These hetero drugs physical properties are found by the methods.[5-9] The synthesis of a fused 1,2,4-triazole can be possible by two distinct routes either by treatment of a suitably substituted 1,2,4-triazole with an appropriate reagents to give either the fused 1,2,4-triazole system or an intermediary product which may be cyclized subsequently or more conventionally by starting from a suitable α-hydrazino heterocyclic compound and creating the triazole unit thereon. The latter method for the formation of fused 1,2,4-triazoles has been discussed in a review and is the one more frequently employed method for the synthesis. The wide applicability of this approach was recognized by a number of workers and a variety of fused 1,2,4-triazoles were prepared by a proper choice of conditions and reagents.[10-14] However, these methods do not fall down under very satisfactory due to drawbacks such as low yields, expensive reagents, longer reaction time at higher reaction temperature and tedious work-up procedures. Therefore, the development of new methods with greater efficiency, straight forward procedures and better yields still are desirable. The 1,8-naphthyridine nucleus has the ubiquitous future of various compounds possessing many pharmacological and biological activities and therefore, they are useful materials. In recent years, chloramine-T (*p*-CH_3 $C_6H_4SO_2N^-$-$ClNa^+$ $3H_2O$ or CAT) has emerged as a potential oxidizing agent in different areas of organic synthesis because it is non-toxic, easy to handle and readily available.

Microwave (MW) irradiation has become the most popular and very useful technology in synthetic organic chemistry. So many methods have been developed for performing reactions with Microwave irradiation in solutions and under solvent-free conditions, but a homogeneous mixture is preferable to get uniform heating. The solvents with higher dielectric constants are superheated and reactions take place more rapidly. Encouraged by the facts, here it is reported a convenient and most efficient method for the synthesis of 9-aryl-6-(3-methoxy phenyl) [1,2,4]- triazolo [4,3-*a*] [1,8] naphthyridines with chloramine-T in ethanol by the MW activation.

EXPERIMENTAL

Thin layer chromatography was run on silica gel-G and visualization was done by using iodine or by UV light. NMR spectra were recorded on a Varian Gemini 300M HZ instrument with the help of

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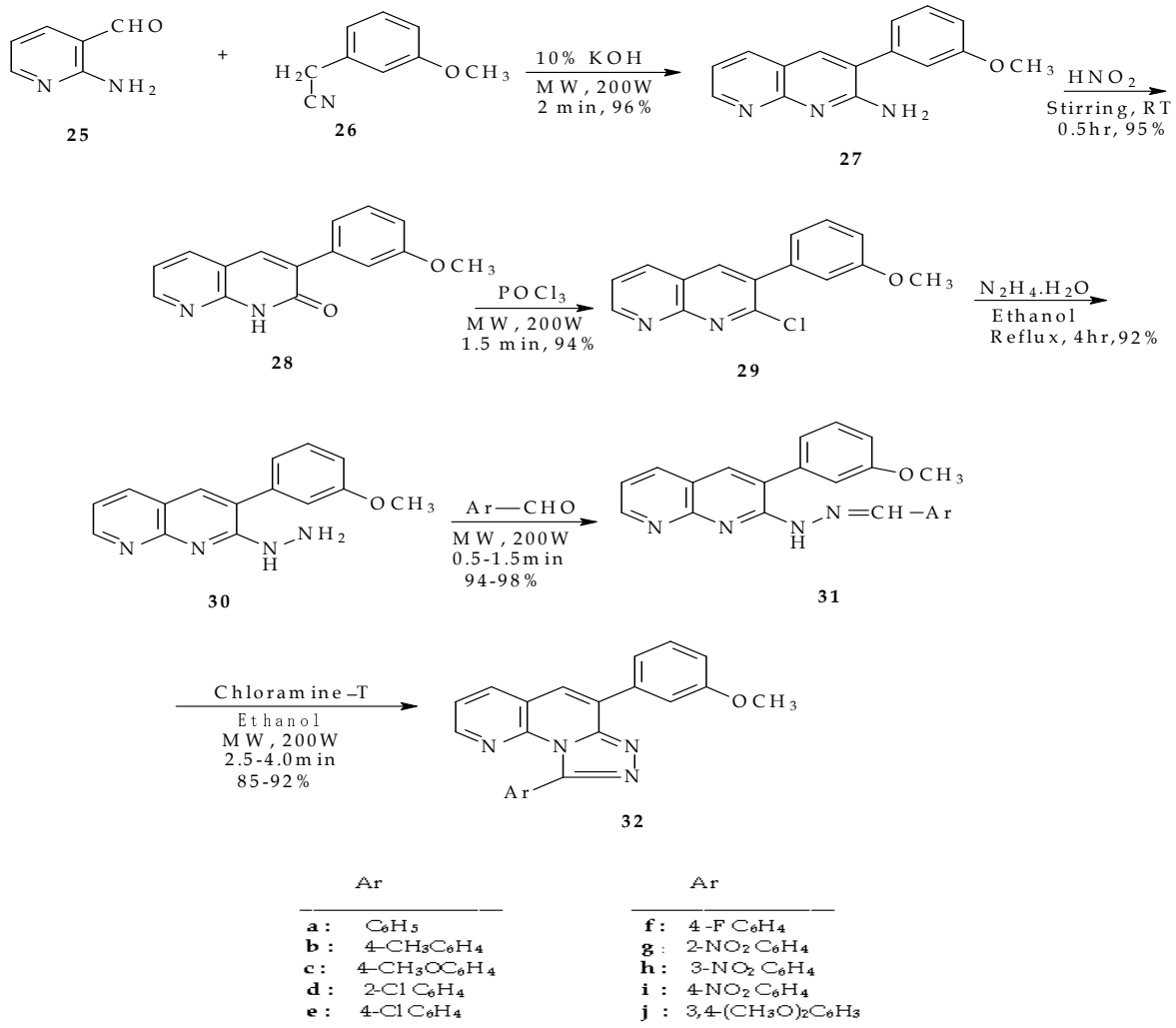
tetramethylsilane as an internal standard in DMSO-d_6. chemical shifts were expressed in ppm. Mass spectra were recorded on an Agilent-LCMS instrument**.**



Scheme-1

2-Aminonicotinaldehyde (1)

The Mixture of nicotinamide (36.5 g) and ammonium sulphamate (52 g) were heated in an oil bath at 150°C. When a clear melt was obtained, the temperature was raised slowly to 200°C. The mixture kept for 6 hrs at this temperature, when the contents of the flask had completely solidified. Water was added, the formed precipitate was collected and washed with ether to remove nicotinonitrile. The solid material thus obtained was refluxed in 2 N HCl for about 4 hrs, made alkaline and extracted with ether. The resulting ether solution was dried (K_2CO_3) and evaporated, which gave pure 2-aminonicotinaldehyde **1,** m.p. 98°C (lit[28], m.p. 98°C).

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

The Mixture of 2-aminonicotinaldehyde **1**(0.01 mole), 3-methoxyphenylacetonitrile **2** (0.01 mole) and 10% KOH (5 drops) were subjected to micro wave irradiation at 200 W intermittently at 30-sec intervals for 2.0 min. On the completion of the reaction, as monitored by thin layer chromatography, the reaction mixture was subjected for cooling. The solid precipitate thus deposited was filtered, washed with the help of water and purified by recrystallization from methanol obtained **3,** yield 96%; m.p. 222°C. analysis calculated for $C_{15}H_{13}N_3O$: C, 71.70; H, 5.21; N, 16.72. Found: C, 71.82; H, 5.23; N, 16.77%.

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

To the cold solution of **3** (0.01 mole) in presence of 2 M HCl (25 mL), $NaNO_2$ solution was added (0.01 mole in 25 mL water), the total reaction mixture was stirred continuously at RT for about 0.5 hr and added cold water. The solid precipitated was filtered, washed with the help of water and purified by recrystallization from methanol gave **4,** yield 95%; m.p. 174°C. Anal. calcd for $C_{15}H_{12}N_2O_2$: C, 71.42; H, 4.79; N, 11.10. Found: C, 71.55; H, 4.80; N, 11.14%.

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

The mixture of **4** (0.01 mole) and $POCl_3$ (10 mL) was subjected for MW activation at 200 W intermittently at 10 seconds interval for about 1.5 min. After the reaction completed, as indicated by thin layer chromatography, the total reaction mixture was subjected for cooling and poured the reaction mixture on to the mixture of crushed ice and $NaHCO_3$. The product that formed and separated was filtered, washed with the help of water and purified by recrystallization from ethanol obtained **5,** yield 94%; m.p. 127°C. Analysis calculated for $C_{15}H_{11}ClN_2O$: C, 66.55; H, 4.10; N, 10.35. Found : C, 66.67; H, 4.12; N, 10.39%.

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

The mixture of **5** (0.01 mole) and hydrazine hydrate (0.015 moles) in ethanol (20 mL) was subjected for reflux on a water bath for about 4.0 hrs. The total reaction mixture was subjected for cooling after that it was poured into ice-cold water. The precipitated solid was separated and filtered, washed with the help of water and then purified by recrystallization from ethanol gave **6,** yield: 92%; m.p. 185°C. Analysis calculated for the compound $C_{15}H_{14}N_4O$: C, 67.65; H, 5.30; N, 21.04. Found: C, 67.78; H, 5.32; N, 21.09%.

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

The mixture of **6** (0.01 mole), aromatic aldehyde (0.01 mole) and di methyl formamide (5 drops) was subjected for MWactivation at 200 W intermittently at 10 seconds interval with the specified time (Table-5). After the reaction completed, as monitored by thin layer chromatography, the reaction mixture was subjected for cooling and digested with cold water. The resultant product was filtered, applied for washing with water and subjected for the purification by recrystallization from ethanol to obtain compound **7** (Table-5).

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

To a solution of suitable hydrazone **7** (0.01 mole) with ethanol (10 mL), added chloramine-T as (0.01 mole). The total reaction mixture then subjected for MW activation at 200 W intermittently at 20 seconds interval with the specified time (Table-6). After the reaction completed as indicated by thin layer chromatography, the reaction mixture was subjected for cooling and digested with the cold water. The solid thus obtained subjected to filtration, washed with the help of water and applied the purification process by recrystallization with ethanol gave compound **8** (Table-**6**).

Table-1: IR and Mass Spectral Data of Phenyl aldehyde 1-[3-(3-methoxy-phenyl) [1,8] naphthyridin-2-yl] hydrazones 7

Compd	Ar	ν_{max} in cm^{-1}		MS (LC-MSD) $[M+H]^+ m/z$
		NH	C=N	
7a	C_6H_5	3351	1623	355.3
7b	4-$CH_3C_6H_4$	3351	1622	369.3
7c	4-$CH_3OC_6H_4$	3350	1622	385.3
7d	2-ClC_6H_4	3352	1621	389.2
7e	4-ClC_6H_4	3351	1622	389.3
7f	4-FC_6H_4	3353	1624	373.3
7g	2-$NO_2C_6H_4$	3353	1621	400.3

7h	3-$NO_2C_6H_4$	3351	1623	400.3
7i	4-$NO_2C_6H_4$	3352	1622	400.3
7j	3,4-$(CH_3O)_2C_6H_3$	3353	1623	415.3

Mass Spectra

The LC-MSD mass spectra of phenyl aldehyde 1-[3-(3-methoxy phenyl) [1,8] naphthyridin-2-yl] hydrazones 7 exhibited strong $[M+H]^+$ ions (Table-1).

H^1 NMR Spectra

The ^1H NMR (300 MHz) spectra of phenyl aldehyde 1-[3-(3-methoxy phenyl) [1,8] naphthyridin-2-yl] hydrazones 7 were measured in $CDCl_3$ and the data is listed in Table-2.

Table-2: ^1H NMR Spectral Data of Phenyl aldehyde 1-[3-(3-methoxy phenyl)-[1,8]-naphthyridin-2-yl] hydrazones 7

Compd	Ar	^1H NMR (300 MHz, $CDCl_3$) (δ, ppm)
7a	C_6H_5-	3.84 (s, 3H, OCH_3), 7.65 (m, 1H, C_6-Hydrogen), 7.70 (m, 2H, C_4-H, C_5-H), 8.26 (m, 1H, C_7-H), 8.38 (s, 1H, N=CH), 6.85-7.60 (m, 9H, Ar-H), 10.15 (s, 1H, NH).
7b	4-$CH_3C_6H_4$	2.40 (s, 3H, CH_3), 3.85 (s, 3H's, OCH_3), 7.70 (m, 3H, C_4-H, C_5-H, C_6-Hydrogen), 8.30 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 6.85-7.40 (m, 8H, Ar-H), 10.18 (s, 1H, NH).
7c	4-$CH_3OC_6H_4$	3.84 (s, 6H, 2XOCH_3), 7.63 (multiplet, 1H, C_6-H), 7.78 (m, 2H, C_4-H, C_5-Hydrogen), 8.28 (m, 1H, C_7-H), 8.42 (s, 1H, N=CH), 6.85-7.40 (m, 8H, Ar-H), 10.22 (s, 1H, NH).
7d	2-ClC_6H_4	3.85 (s, 3H's, OCH_3), 7.68 (multiplet, 2H's, C_4-H, C_6-H), 8.24 (multiplet, 1H, C_5-H), 8.35 (multiplet, 1H, C_7-H), 8.85 (singlet, 1H, N=CH), 6.92-7.40 (multiplet, 8H, Ar-H), 10.25 (singlet, 1H, NH).
7e	4-ClC_6H_4	3.83 (s, 3H's, OCH_3), 7.75 (multiplet, 2H, C_4-H, C_6-H), 8.02 (multiplet, 1H, C_5-H), 8.37 (multiplet, 1H, C_7-H), 8.42 (singlet, 1H, N=CH), 6.90-7.44 (multiplet, 8H, Ar-Hydrogen), 10.24 (s, 1H, NH).
7f	4-FC_6H_4	3.86 (s, 3H's, OCH_3), 7.67 (multiplet, 1H, C_6-Hydrogen), 7.80 (multiplet, 2H's, C_4-Hydrogen, C_5-Hydrogen), 8.34 (multiplet, 1H, C_7-Hydrogen), 8.44 (singlet, 1H, N=CH), 6.94-7.40 (multiplet, 8H, Ar-Hydrogen), 10.20 (singlet, 1H, NH).
7g	2-$NO_2C_6H_4$	3.84 (s, 3H, OCH_3), 7.70 (multiplet, 1H, C_6-H), 8.22 (m, 2H, C_4-Hydrogen, C_5-Hydrogen), 8.40 (multiplet, 1H, C_7-Hydrogen), 8.56 (singlet, 1H, N=CH), 6.98-7.65 (multiplet, 8H's, Ar-Hydrogen), 10.26 (s, 1H, NH).
7h	3-$NO_2C_6H_4$	3.86 (s, 3H's, OCH_3), 7.75 (m, 1H, C_6-Hydrogen), 8.20 (m, 2H's, C_4-Hydrogen, C_5-Hydrogen), 8.38 (m, 1H, C_7-Hydrogen), 8.60 (s, 1H, N=CH), 6.95-7.62 (m, 8H's, Ar-Hydrogen), 10.28 (s, 1H, NH).
7i	4-$NO_2C_6H_4$	3.85 (s, 3H's, OCH_3), 7.72 (m, 1H, C_6-Hydrogen), 8.18 (m, 2H's, C_4-Hydrogen, C_5-Hydrogen), 8.42 (m, 1H, C_7-Hydrogen), 8.58 (s, 1H, N=CH), 7.00-7.67 (m, 8H's, Ar-Hydrogen), 10.24 (s, 1H, NH).
7j	3,4-$(CH_3O)_2C_6H_3$	3.84 (s, 3H, OCH_3), 3.92 (s, 3H's, OCH_3), 3.98 (s, 3H's, OCH_3), 7.38 (m, 1H, C_6-Hydrogen), 7.65 (m, 1H, C_5-Hydrogen), 7.45 (s, 1H, C_4-Hydrogen), 8.30 (m, 1H, C_7-Hydrogen), 8.42 (s, 1H, N=CH), 6.84-7.32 (m, 7H, Ar-Hydrogen), 10.18 (singlet, 1H, NH).

Table - 3: IR and mass spectral data for 9-Aryl-6-(3-methoxyphenyl) [1,2,4] triazolo [4,3-*a*] [1,8] naphthyridines 8

Compd	Ar	ν_{max} in cm^{-1}	MS (LC-MSD)
		C=N	$[M+H]^+$ *m/z*
8a	C_6H_5	1603	353.2
8b	4-$CH_3C_6H_4$	1606	367.3
8c	4-$CH_3OC_6H_4$	1611	383.3
8d	2-ClC_6H_4	1611	387.2
8e	4-ClC_6H_4	1597	387.3
8f	4-FC_6H_4	1609	371.3

8g	2-$NO_2C_6H_4$	1612	398.2
8h	3-$NO_2C_6H_4$	1613	398.3
8i	4-$NO_2C_6H_4$	1611	398.3
8j	3,4-$(CH_3O)_2C_6H_3$	1607	413.3

^{1}H NMR Spectra

The ^{1}H NMR (300 MHz) spectra for 9-aryl-6-(3-methoxyphenyl) [1,2,4] triazolo [4,3-*a*] [1,8] naphthyridines 8 were recorded in $CDCl_3$ and the data are presented in Table-4.

Table-4: ^{1}H NMR spectral data for 9-Aryl-6-(3-methoxyphenyl) [1,2,4] triazolo [4,3-*a*] [1,8] naphthyridines 8

Compd	Ar	H^1 NMR (300 MHz, $CDCl_3$) (δ, ppm)
8a	C_6H_5	3.90 (s, 3H's, OCH_3), 7.90 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.16 (multiplet, 1H, C_4-Hydrogen), 8.42 (multiplet, 1H, C_2-Hydrogen), 7.00-7.75 (multiplet, 9H's, Ar-Hydrogen).
8b	4-$CH_3C_6H_4$	2.45 (s, 3H's, CH_3), 3.92 (s, 3H's, OCH_3), 7.80 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.18 (multiplet, 1H, C_4-Hydrogen), 8.45 (multiplet, 1H, C_2-Hydrogen), 7.01-7.67 (multiplet, 8H, Ar-Hydrogen).
8c	4-$CH_3OC_6H_4$	3.90 (s, 6H, 2×OCH_3), 7.86 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.17 (multiplet, 1H, C_4-Hydrogen), 8.43 (multiplet, 1H, C_2-Hydrogen), 7.00-7.75 (multiplet, 8H, Ar-Hydrogen).
8d	2-ClC_6H_4	3.93 (s, 3H, OCH_3), 7.68 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.15 (multiplet, 1H, C_4-Hydrogen), 8.38 (multiplet, 1H, C_2-Hydrogen), 7.02-7.58 (multiplet, 8H, Ar-Hydrogen).
8e	4-ClC_6H_4	3.95 (s, 3H, OCH_3), 7.70 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.16 (multiplet, 1H, C_4-Hydrogen), 8.37 (multiplet, 1H, C_2-Hydrogen), 7.00-7.56 (multiplet, 8H, Ar-Hydrogen).
8f	4-FC_6H_4	3.94(s, 3H's, OCH_3), 7.88 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.20 (multiplet, 1H, C_4-Hydrogen), 8.42 (multiplet, 1H, C_2-Hydrogen), 7.02-7.74 (multiplet, 8H, Ar-Hydrogen).
8g	2-$NO_2C_6H_4$	3.92 (s, 3H's, OCH_3), 7.72 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.30 (multiplet, 1H, C_4-Hydrogen), 8.45 (multiplet, 1H, C_2-Hydrogen), 7.00-7.62 (multiplet, 8H, Ar-Hydrogen).
8h	3-$NO_2C_6H_4$	3.95 (s, 3H, OCH_3), 7.70 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.26 (multiplet, 1H, C_4-Hydrogen), 8.43 (multiplet, 1H, C_2-Hydrogen), 7.03-7.60 (multiplet, 8H, Ar-Hydrogen).
8i	4-$NO_2C_6H_4$	3.96 (s, 3H, OCH_3), 7.75 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.24 (multiplet, 1H, C_4-Hydrogen), 8.46 (multiplet, 1H, C_2-Hydrogen), 7.02-7.64 (multiplet, 8H, Ar-Hydrogen).
8j	3,4-$(CH_3O)_2C_6H_3$	3.92 (s, 6H, 2xOCH_3), 4.00 (s, 3H, OCH_3), 7. 65 (multiplet, 2H's, C_3-Hydrogen, C_5-Hydrogen), 8.18 (multiplet, 1H, C_4-Hydrogen), 8.48 (multiplet, 1H, C_2-Hydrogen), 6.98-7.56 (multiplet, 7H, Ar-Hydrogen).

Table-5: Physical & analytical data for Aryl aldehyde1-[3-(3-methoxyphenyl) [1,8] naphthyridin-2-yl] hydrazones 7

Compound	Ar	Reaction Time(min)	m.p. °C	Yield (%)	Molecular Formula	Found (%) (Calculated)		
						C	H	N
7a	C_6H_5	0.5	172	95	$C_{22}H_{18}N_4O$	74.70 (74.56)	5.13 5.12	15.86 (15.81)
7b	4-$CH_3C_6H_4$	1.0	144	97	$C_{23}H_{20}N_4O$	75.10 (74.98)	5.49 5.47	15.25 (15.21)
7c	4-$CH_3OC_6H_4$	1.5	138	94	$C_{23}H_{20}N_4O_2$	71.99 (71.86)	5.25 5.24	14.62 (14.57)
7d	2-ClC_6H_4	0.5	152	95	$C_{22}H_{17}ClN_4O$	68.08 (67.95)	4.43 4.41	14.46 (14.41)

7e	4-ClC_6H_4	0.5	155	98	$C_{22}H_{17}ClN_4O$	68.09 (67.95)	4.42 4.41	14.45 (14.41)
7f	4-FC_6H_4	1.0	166	96	$C_{22}H_{17}FN_4O$	71.08 (70.96)	4.62 4.60	15.08 (15.04)
7g	2-$NO_2C_6H_4$	1.0	176	94	$C_{22}H_{17}N_5O_3$	66.30 (66.16)	4.30 4.29	17.58 (17.53)
7h	3-$NO_2C_6H_4$	0.5	165	96	$C_{22}H_{17}N_5O_3$	66.28 (66.16)	4.31 4.29	17.58 (17.53)
7i	4-$NO_2C_6H_4$	0.5	182	97	$C_{22}H_{17}N_5O_3$	66.29 (66.16)	4.30 4.29	17.57 (17.53)

Table-6: Physical and analytical data for 9-Aryl-6-(3-methoxyphenyl) [1,2,4] triazolo [4,3-*a*] [1,8] naphthyridines 8

Compd	Ar	Reaction Time(min)	m.p. ^oC	Yield (%)	Mol. Formula	Found (%) (Calcd)		
						C	H	N
8a	C_6H_5	2.5	175	90	$C_{22}H_{16}N_4O$	75.11 (74.98)	4.60 4.58	15.94 (15.90)
8b	4-$CH_3C_6H_4$	2.5	165	91	$C_{23}H_{18}N_4O$	75.53 (75.39)	4.96 4.95	15.34 (15.29)
8c	4-$CH_3OC_6H_4$	3.0	159	88	$C_{23}H_{18}N_4O_2$	72.38 (72.24)	4.75 4.74	14.69 (14.65)
8d	2-ClC_6H_4	3.0	196	88	$C_{22}H_{15}ClN_4O$	68.45 (68.31)	3.92 3.91	14.53 (14.48)
8e	4-ClC_6H_4	2.5	215	92	$C_{22}H_{15}ClN_4O$	68.44 (68.31)	3.93 3.91	14.52 (14.48)
8f	4-FC_6H_4	3.0	219	90	$C_{22}H_{15}FN_4O$	71.47 (71.34)	4.09 4.08	15.18 (15.13)
8g	2-$NO_2C_6H_4$	4.0	258	85	$C_{22}H_{15}N_5O_3$	66.62 (66.49)	3.82 3.80	17.66 (17.62)
8h	3-$NO_2C_6H_4$	4.0	281	86	$C_{22}H_{15}N_5O_3$	66.63 (66.49)	3.81 3.80	17.67 (17.62)
8i	4-$NO_2C_6H_4$	3.0	276	88	$C_{22}H_{15}N_5O_3$	66.61 (66.49)	3.82 3.80	17.66 (17.62)
8j	3,4-$(CH_3O)_2C_6H_3$	3.5	188	87	$C_{24}H_{20}N_4O_3$	70.02 (69.89)	4.90 4.89	13.62 (13.58)

CONCLUSION

The conclusion from these experimental observations the drug Green Synthesis of chloramine-T mediated synthesis of 9-aryl-6-(3-methoxyphenyl) [1,2,4] triazolo [4,3-a] [1,8] naphthyridines were synthesized with the help of simple methods by applying the Micro Wave irradiation.

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