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Hypervalent iodine mediated solid state synthesis of 2-aryl-5-[2-(3,4,5-trimethoxyphenyl)[1,8]naphthyridin- 3-yl]-1,3,4-oxadiazoles

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The hydrazones **5** on oxidative cyclization of with [$PhI(OAc)_2$] in the solid state at room temperature under grinding conditions has furnished the respective 2-aryl-5-[2-(3,4,5-trimethoxyphenyl)[1,8]naphthyridin-3-yl]-1,3,4-oxadiazoles **6** in good yields without any undesirable byproducts. The purity of the products is high. The method is preoperatively convenient, useful and environmentally benign. The experimental procedure is very simple and avoids sophistication. The most probable mechanism of above transformation is suggested**.** The structure of compounds **5** and **6** has been confirmed by their spectroscopic(IR, ^{1}H NMR and MS) and analytical data.

Keywords: 1,8-Naphthyridine, $PhI(OAc)_2$, SiO_2-$FeCl_3$, Oxadiazoles

1,8-Naphthyridines are an important group of heterocyclic compounds possessing a variety of biological activities[1-3]. Fused systems are of interest to many researchers in diverse fields[4-6] and various routes for the synthesis of these compounds are already known[7-12]. 1,3,4-Oxadiazole is considered as an important chemical moiety because of its various biological and pharmacological activities, such as antimicrobial,[13] antiinflamatory,[14] antiproliferative[15] and antitubercular[16] activities. On the other hand compounds possessing 1,8-naphthyridine moieties are endowed with antibacterial,[17] antiinflamatory,[18] antitumer[19] and antimicobacterial[20] activities. The combination of structural features of both these heterocycles into a single scaffold is expected to provide new chemical entities with noteworthy biological and pharmacological properties. Iodobenzene diacetate [$PhI(OAc)_2$] is a very versatile oxidizing agent and is of much importance in its synthetic utility. [21-23]

To protect the natural resources, the development of environmentally sustainable processes and the optimization of energy consumption are required. Organic solid state (solvent-free) reactions have attracted considerable interest due to increasing awareness about environmental problems in chemical research and industry. The solvent used in organic synthesis are high on the list of environmental pollutants, because they are employed in large amounts and usually are volatile liquids. In recent years, organic reactions in the solid state by grinding have been attracted the synthetic organic chemists because of their simplicity and synthetic value.[24-26] The majority of them involve longer preparation time, high reaction temperature, low yields and usage of toxic reagents.

Inspired by these facts, we describe herein an efficient, practical and eco-friendly method for the synthesis of 2-aryl-5-[2-(3,4,5-trimethoxy-phenyl-[1,8]naphthyridin-3-yl]-1,3,4-oxadiazoles using iodobenzene diacetate [$PhI(OAc)_2$] in the solid state at room temperature (RT). The structural assignment of compounds **5, 6** were based on their elemental analyses and spectral (IR, ^{1}H NMR and MS) data. 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.

Experimental Details

Melting points were measured in open capillaries on a Cintex melting point apparatus and are uncorrected. Purity of the compounds were 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 (chemical shifts in δ ppm) and mass

spectra on a Finnigan MAT-1020, automated GC-MS and VG Auto Spec-M instruments. Elemental analyses were performed on a Perkin-Elmer 240 CHN elemental analyzer. The ethyl 3,4,5-trimethoxy-benzoyl acetate **2** and iodobenzene diacetate [$PhI(OAc)_2$] were purchased from Aldrich Chemical Company.

A series of stepwise synthesis of RT solid state mediated compounds **1-6** displayed in Scheme 1. All the synthesized compounds were further confirmed by spectral analysis.

Synthesis of Ethyl 2-(3,4,5-trimethoxyphenyl)[1,8]naphthyridine-3-carboxylate 3

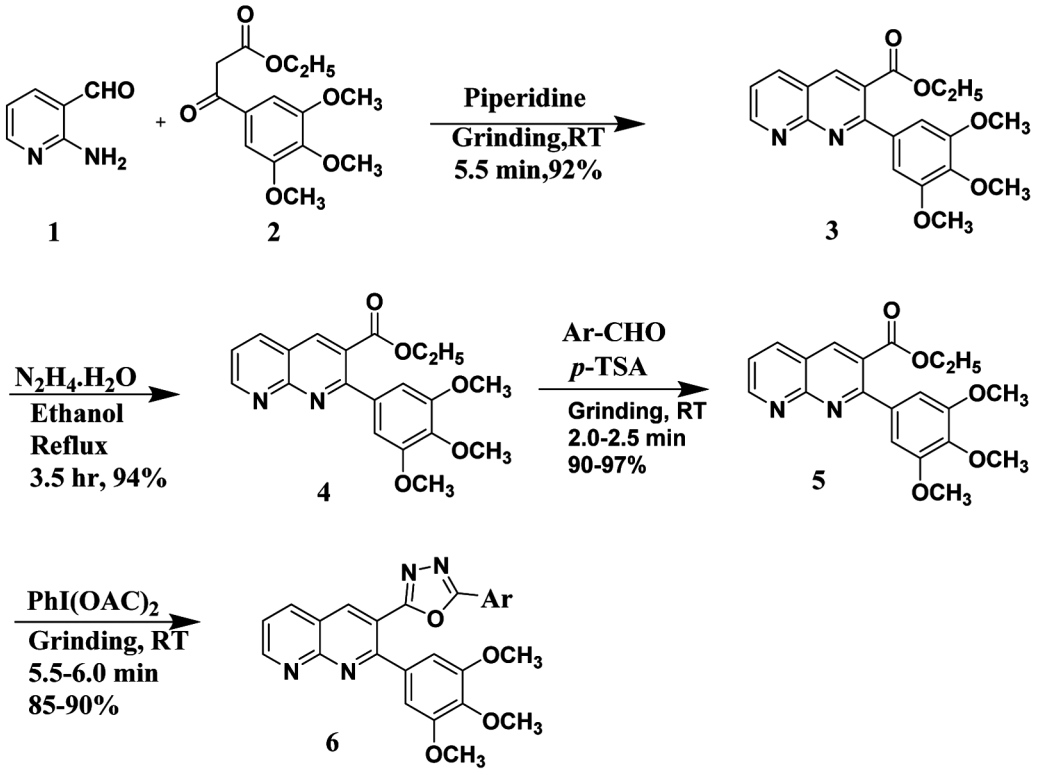
A mixture of 2-aminonicotinaldehyde **1** (0.01 mol), ethyl 3,4,5-trimethoxybenzoyl acetate **2** (0.01 mol) and piperidine (0.01 mol) was ground by pestle and mortar at RT for 5.5 min. The solid separated was treated with water, filtered and purified by recrystallization from ethanol to obtain **3**, m.p. 135 °C, yield 92%.

Synthesis of 2-(3,4,5-Trimethoxyphenyl)[1,8]naphthyridine-3-carbohydrazide 4

A mixture of **3** (0.01 mol) and hydrazine hydrate (0.015 mol) in ethanol (30 mL) was refluxed on a water-bath for 3.5 h. The reaction mixture was cooled, the solid thus obtained was filtered and purified by recrystallization from ethanol to give **4**, m.p. 196 °C, yield 94%.

Synthesis of *N*'-3-(1-arylmethylidene)-2-(3,4,5-trimethoxyphenyl)[1,8]naphthyridine-3-carbohydrazides 5

A mixture of **4** (0.01 mol), aromatic aldehyde (0.01 mol) and PTSA (0.01 mol) was ground by pestle and mortar at RT for specified time. On completion of the reaction (monitored by TLC), the reaction mixture was treated with cold water. The



Scheme 1 — Synthesis of 2-aryl-5-[2-(3,4,5-trimethoxyphenyl)[1,8]naphthyridin-3-yl]-1,3,4-oxadiazoles

precipitated solid was filtered, washed with water and purified by recrystallization from ethanol to furnish **5**.

Synthesis of 2-aryl-5-[2-(3,4,5-trimethoxyphenyl)[1,8] naphthyridin-3-yl]-1,3,4-oxadiazoles 6

A mixture of appropriate hydrazone **5** (0.01 mol) and [$PhI(OAc)_2$] (0.015 mol) was ground in a mortar by pestle at RT for the period indicated. After complete conversion as indicated by TLC, the reaction mixture was digested with cold water. The solid separated was filtered, washed with water and purified by recrystallization from ethanol to afford **6**.

Results and Discussion

Physical and analytical data of compound **5** and **6** are given in Table 1 and 2, respectively.

Spectral data of compounds 5 and 6

5a: IR ν_{max} in cm^{-1} NH 3344, C=O 1663, C=N 1620, C_6H_5 3.67 (s, 6H, $2XOCH_3$), 3.80 (s, 3H, OCH_3), 7.82 (m, 1H, C_6-H), 8.33 (m, 1H, C_5-H), 8.45 (s, 1H, C_4-H), 9.20(m, 1H, C_7-H), 8.66 (s, 1H, N=CH),7.10 (s, 2H, Ar-H), 7.20 -7.43(m, 5H, Ar-H), 9.48 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 443.25.

5b: IR ν_{max} in cm^{-1} NH 3357, C=O 1664, C=N 1622, 4-$CH_3C_6H_4$ 2.36 (s, 3H, CH_3), 3.78 (s, 6H, $2XOCH_3$), 3.82 (s, 3H, OCH_3), 7.83 (m, 1H, C_6-H), 8.36 (m, 1H, C_5-H), 8.48 (s, 1H, C_4-H), 9.18 (m, 1H, C_7-H), 8.67 (s, 1H, N=CH), 7.13 (s, 2H, Ar-H), 7.18 - 7.45 (m, 4H, Ar-H), 9.54 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 457.20.

5c: IR ν_{max} in cm^{-1} NH 3351, C=O 1663, C=N 1620, 4-$CH_3OC_6H_4$ 3.80 (s, 6H, $2XOCH_3$), 3.85 (s, 6H, $2XOCH_3$), 7.85 (m, 1H, C_6-H), 8.37 (m, 1H, C_5-H), 8.50 (s, 1H, C_4-H), 9.22(m, 1H, C_7-H), 8.70 (s, 1H, N=CH), 7.15 (s, 2H, Ar-H), 7.22 -7.47(m, 4H, Ar-H), 9.56 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 473.26.

5d: IR ν_{max} in cm^{-1} NH 3337, C=O1666,C=N 1623, 2-ClC_6H_4 3.82 (s, 6H, $2XOCH_3$), 3.87 (s, 3H, OCH_3),7.87 (m, 1H, C_6-H), 8.36 (m, 1H, C_5-H), 8.47 (s, 1H, C_4-H), 9.20(m, 1H, C_7-H), 8.66 (s, 1H, N=CH),7.13 (s, 2H, Ar-H), 7.20-7.42(m, 4H, Ar-H), 9.52 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z*477.10.

5e: IR ν_{max} in cm^{-1} NH 3348, C=O 1663, C=N 1621, 4-ClC_6H_4 3.84 (s, 6H, $2XOCH_3$), 3.88 (s, 3H,

Table 1 — Physical and analytical data of *N'* 3-(1-Arylmethylidene)-2-(3,4,5-trimethoxyphenyl) [1,8]naphthyridine-3-carbohydrazides **5**

Compd	Ar	Reaction time (min)	m.p. °C	Yield (%)	Mol. formula	Found (%) (Cacld)		
						C	H	N
5a	C_6H_5	2.5	182	92	$C_{25}H_{22}N_4O_4$	67.98 (67.86)	5.03 (5.01)	12.70 (12.66)
5b	4-$CH_3C_6H_4$	2.0	175	96	$C_{26}H_{24}N_4O_4$	68.52 (68.41)	5.31 (5.30)	12.32 (12.27)
5c	4-$CH_3OC_6H_4$	2.5	190	95	$C_{26}H_{24}N_4O_5$	66.22 (66.09)	5.14 (5.12)	11.91 (11.86)
5d	2-ClC_6H_4	2.5	236	94	$C_{25}H_{21}ClN_4O_4$	63.07 (62.96)	4.45 (4.44)	11.79 (11.75)
5e	4-ClC_6H_4	2.0	223	97	$C_{25}H_{21}ClN_4O_4$	67.06 (62.96)	4.46 (4.44)	11.80 (11.75)
5f	4-FC_6H_4	2.0	215	96	$C_{25}H_{21}FN_4O_4$	65.33 (65.21)	4.61 (4.60)	12.21 (12.17)
5g	3-$NO_2C_6H_4$	2.0	270	90	$C_{25}H_{21}N_5O_6$	61.71 (61.60)	4.35 (4.34)	14.42 (14.37)
5h	4-$NO_2C_6H_4$	2.0	257	93	$C_{25}H_{21}N_5O_6$	61.72 (61.60)	4.36 (4.34)	14.41 (14.37)
5i	3,4-$(CH_3O)_2C_6H_3$	2.5	193	95	$C_{27}H_{26}N_4O_6$	64.66 (64.53)	5.23 (5.21)	11.20 (11.15)

Table 2 — Physical and analytical data of 2-Aryl-5-[2-(3,4,5-trimethoxyphenyl)[1,8]naphtyridin-3-yl]-1,3,4-oxadiazoles **6**

Compd	Ar	Reaction time (min)	m.p. °C	Yield (%)	Mol. formula	Found (%) (Cacld)		
						C	H	N
6a	C_6H_5	2.5	182	92	$C_{25}H_{20}N_4O_4$	68.28 (68.17)	4.59 (4.58)	12.77 (12.72)
6b	4-$CH_3C_6H_4$	2.0	175	96	$C_{26}H_{22}N_4O_4$	68.81 (68.71)	4.90 (4.88)	12.38 (12.33)
6c	4-$CH_3OC_6H_4$	2.5	190	95	$C_{26}H_{22}N_4O_5$	66.50 (66.38)	4.72 (4.71)	11.95 (11.91)
6d	2-ClC_6H_4	2.5	236	94	$C_{25}H_{19}ClN_4O_4$	63.35 (63.23)	4.05 (4.03)	11.85 (11.80)
6e	4-ClC_6H_4	2.0	223	97	$C_{25}H_{19}ClN_4O_4$	63.34 (63.23)	4.05 (4.03)	11.84 (11.80)
6f	4-FC_6H_4	2.0	215	96	$C_{25}H_{19}FN_4O_4$	65.62 (65.50)	4.20 (4.18)	12.27 (12.22)
6g	3-$NO_2C_6H_4$	2.0	270	90	$C_{25}H_{19}N_5O_6$	61.97 (61.85)	3.96 (3.94)	14.47 (14.43)
6h	4-$NO_2C_6H_4$	2.0	257	93	$C_{25}H_{19}N_5O_6$	61.96 (61.85)	3.96 (3.94)	14.48 (14.43)
6i	3,4-$(CH_3O)_2C_6H_3$	2.5	193	95	$C_{27}H_{24}N_4O_6$	64.91 (64.79)	4.85 (4.83)	11.23 (11.19)

OCH_3),7.86 (m, 1H, C_6-H), 8.37 (m, 1H, C_5-H), 8.48 (s, 1H, C_4-H), 9.23(m, 1H, C_7-H), 8.68 (s, 1H, N=CH),7.15 (s, 2H, Ar-H), 7.22-7.42(m, 4H, Ar-H), 9.57 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 477.10.

5f: IR ν_{max} in cm^{-1} NH 3372, C=O 1669,C=N 1626, 4-FC_6H_4 3.80 (s, 6H, $2XOCH_3$), 3.82 (s, 3H, OCH_3), 7.85 (m, 1H, C_6-H), 8.38 (m, 1H, C_5-H), 8.46 (s, 1H, C_4-H), 9.21(m, 1H, C_7-H), 8.67 (s, 1H, N=CH),7.12 (s, 2H, Ar-H), 7.18-7.40(m, 4H, Ar-H), 9.55 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 461.23.

5g: IR ν_{max} in cm^{-1} NH 3360, C=O 1665, C=N 1624, 3-$NO_2C_6H_4$ 3.82 (s, 6H, $2XOCH_3$), 3.86 (s, 3H, OCH_3),7.88 (m, 1H, C_6-H), 8.48 (m, 1H, C_5-H), 8.50 (s, 1H, C_4-H), 9.23(m, 1H, C_7-H), 8.70 (s, 1H, N=CH), 7.16 (s, 2H, Ar-H), 7.22 -7.42(m, 4H, Ar-H), 9.56 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 488.10.

5h: IR ν_{max} in cm^{-1} NH 3370, C=O 1672, C=N 1626, 4-$NO_2C_6H_4$ 3.84 (s, 6H, $2XOCH_3$), 3.86 (s, 3H, OCH_3),7.87 (m, 1H, C_6-H), 8.40 (m, 1H, C_5-H), 8.48 (s, 1H, C_4-H), 9.25(m, 1H, C_7-H), 8.72 (s, 1H, N=CH),7.18 (s, 2H, Ar-H), 7.25-7.44(m, 4H, Ar-H), 9.58 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 488.10.

5i: IR ν_{max} in cm^{-1} NH 3354, C=O 1670, C=N 1624, 3,4-$(CH_3O)_2$ C_6H_3 3.75 (s, 9H, $3XOCH_3$), 3.80 (s, 6H, $2XOCH_3$),7.84 (m, 1H, C_6-H), 8.37 (m, 1H, C_5-H), 8.46 (s, 1H, C_4-H), 9.22(m, 1H, C_7-H), 8.70 (s, 1H, N=CH),7.14 (s, 2H, Ar-H), 7.20 -7.40(m, 3H, Ar-H), 9.25 (s, 1H, CONH). MS (ESI) $[M+H]^+$ *m/z* 503.24.

6a: IR ν_{max} in cm^{-1} C=N 1601. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm)C_6H_5 3.80 (s, 6H, $2XOCH_3$), 3.90 (s, 3H, OCH_3), 7.72 (m, 1H, C_6-H), 8.19 (m, 2H, C_4-H, C_5-H), 8.80 (m, 1H, C_7-H), 7.17 (s, 2H, Ar-H), 7.40-7.65 (m, 5H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 441.20.

6b: IR ν_{max} in cm^{-1} C=N 1602. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm) 4-$CH_3C_6H_4$ 2.38(s, 3H, CH_3), 3.82 (s, 6H, $2XOCH_3$), 3.92 (s, 3H, OCH_3), 7.76 (m, 1H, C_6-H), 8.22 (m, 2H, C_4-H, C_5-H), 8.83 (m, 1H, C_7-H), 7.20 (s, 2H, Ar-H), 7.38-7.62 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 455.24.

6c: IR ν_{max} in cm^{-1} C=N 1605. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm) 4-$CH_3OC_6H_4$ 3.84 (s, 6H, $2XOCH_3$), 3.93 (s, 6H, $2XOCH_3$), 7.78 (m, 1H, C_6-H), 8.20 (m, 2H, C_4-H, C_5-H), 8.84 (m, 1H, C_7-H), 7.18 (s, 2H, Ar-H), 7.42-7.66 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 471.22.

6d: IR ν_{max} in cm^{-1} C=N1600. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm) 2-ClC_6H_4 3.83 (s, 6H, $2XOCH_3$), 3.92 (s, 3H, OCH_3), 7.77 (m, 1H, C_6-H), 8.19 (m, 2H, C_4-H, C_5-H), 8.82 (m, 1H, C_7-H), 7.20 (s, 2H, Ar-H), 7.40-7.64 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 475.15.

6e: IR ν_{max} in cm^{-1} C=N 1604. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm)4-ClC_6H_4 3.86 (s, 6H, $2XOCH_3$), 3.94 (s, 3H, OCH_3), 7.76 (m, 1H, C_6-H), 8.20 (m, 2H, C_4-H, C_5-H), 8.85 (m, 1H, C_7-H), 7.20 (s, 2H, Ar-H), 7.42-7.65 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 475.17.

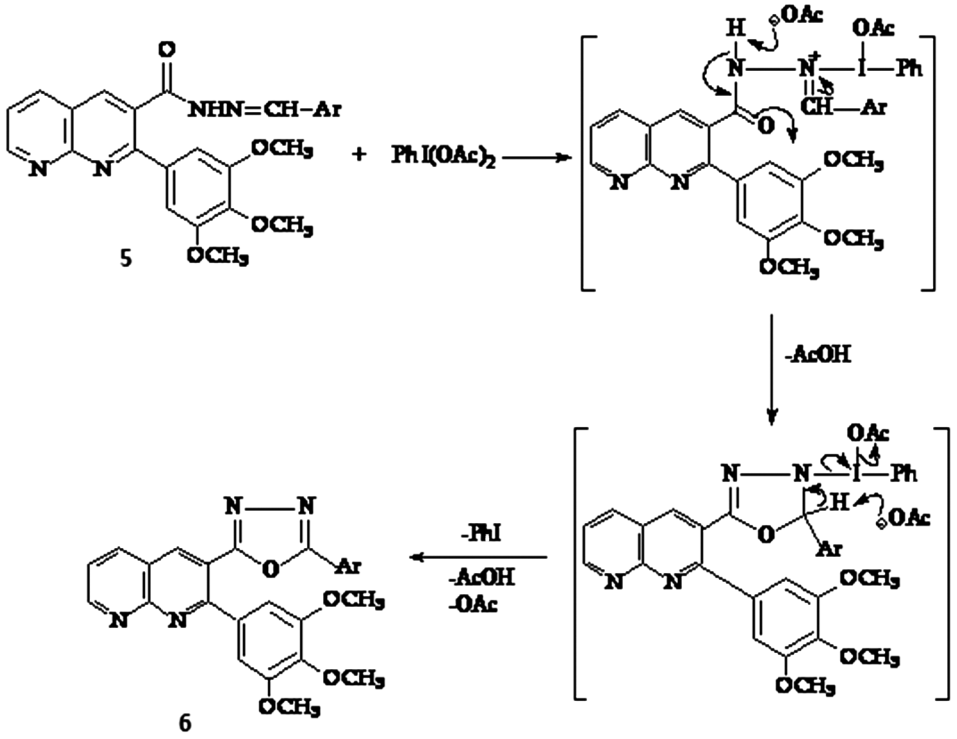
6f: IR ν_{max} in cm^{-1} C=N 1603. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm)4-FC_6H_4 3.85 (s, 6H, $2XOCH_3$), 3.93 (s, 3H, OCH_3), 7.78 (m, 1H, C_6-H), 8.22 (m, 2H, C_4-H, C_5-H), 8.87 (m, 1H, C_7-H), 7.18 (s, 2H, Ar-H), 7.38-7.60 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z*459.27.

6g: IR ν_{max} in cm^{-1} C=N 1603. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm)3-$NO_2C_6H_4$ 3.86 (s, 6H, $2XOCH_3$), 3.96 (s, 3H, OCH_3), 7.80 (m, 1H, C_6-H), 8.25 (m, 2H, C_4-H, C_5-H), 8.90 (m, 1H, C_7-H), 7.20 (s, 2H, Ar-H), 7.40-7.63 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z*486.15.

6h: IR ν_{max} in cm^{-1} C=N 1606. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm)4-$NO_2C_6H_4$ 3.88 (s, 6H, $2XOCH_3$), 3.98 (s, 3H, OCH_3), 7.82 (m, 1H, C_6-H), 8.28 (m, 2H, C_4-H, C_5-H), 8.93 (m, 1H, C_7-H), 7.20 (s, 2H, Ar-H), 7.42-7.65 (m, 4H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 486.15.

6i: IR ν_{max} in cm^{-1} C=N 1605. ^{1}H NMR(400 MHz, $CDCl_3$) (δ, ppm) 3,4-$(CH_3O)_2$ C_6H_3 3.85 (s, 9H, $3XOCH_3$), 3.95 (s, 6H, $2XOCH_3$), 7.78 (m, 1H, C_6-H), 8.22 (m, 2H, C_4-H, C_5-H), 8.84 (m, 1H, C_7-H), 7.18 (s, 2H, Ar-H), 7.40-7.62 (m, 3H, Ar-H). MS (ESI) $[M+H]^+$ *m/z* 501.22.

The most probable mechanism of above transformation is depicted in Scheme 2**.**



Scheme 2 — Plausible mechanism of formation of **6** from **5**

Conclusion

Herein, iodine mediated solid state eco-friendly synthesis of various substituted [1,8]naphthyridin- 3-yl]-1,3,4-oxadiazoles are presented. [1,8]naphthyridines containing oxadiazole as linkers can be concluded on the basis of the findings.

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