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Microwave-assisted Synthesis of 2-Substituted Naphtho[1,2-*d*][1,3]oxazoles by Reacting 1-Nitroso-2-naphthol with Allyl Bromides and Benzyl Bromides using $FeCl_3$ as Catalyst

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Efficient and improved preparation of 2-substituted[1,2-*d*][1,3]oxazoles by the reaction of 1-nitroso-2-naphthol and allyl bromides, benzyl bromides under microwave condition utilizing $FeCl_3$ as a catalyst with yield ranging from 32% to 72%. Reaction with bromo acetonitrile yields the corresponding 2-cyanonaphthoxazole with 58% yield.

Keywords: Microwave, Allyl bromides, Benzyl bromides, 2-Substituted naphthoxazoles, 1-Nitroso-2-naphthol.

INTRODUCTION

Heterocyclic compounds containing oxygen and nitrogen as heteroatoms, such as annulated oxazoles [1-3] form the key unit in a wide variety of natural products and biologically active compounds that have pharmacological properties [4]. Representative examples include the anticancer agent NCS-693638 [5], the estrogen receptor-β agonist ERB-041 [6], the HIV reverse transcriptase inhibitor L-697661 [7], the HT_3 receptor agonist [8], cathepsin S inhibitor [9], UK-1 [10], salvianen [11], AJ9561 [12] and pseudopteroxazole [13]. Naphthoxazoles are known for their significant interest in material sciences as they are known for their exceptional fluorescent properties [14-17] (Fig. 1).

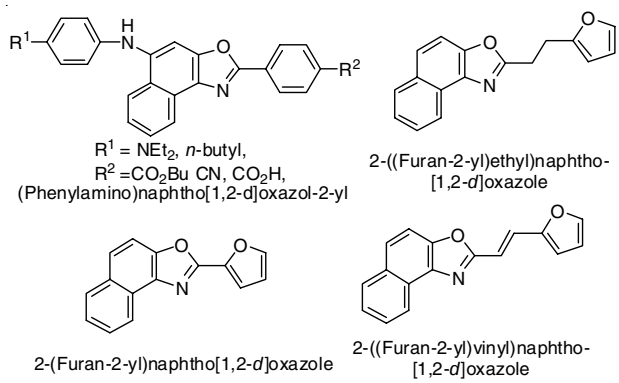


Fig. 1 Synthetic naphthoxazoles with significant fluorescent properties

On that account, there is a great deal of effort in the development of new synthetic approaches to the naphthoxazoles skeleton. Most of them are based on the reaction of oximes with allyl bromide, 4-nitrobenzyl bromide, dimethyl sulfate and ethyl iodide [18], oxime or 5-*N*,*N*-diethylamino-2-nitrosophenol with benzyl bromides [19], 1,2-naphthalenedione 1-oxime with benzyl bromides in the presence of Et_3N [20] and the reaction of 1-nitroso-2-naphthols and α-functionalized ketones [21]. Other methods of preparation include the condensation of 1-amino-2-naphthol with aromatic aldehydes [22,23], Pd/C-catalyzed reaction of 1-nitronaphthalen-2-yl benzoate with a 1,4-dihydropyridine [24] and the decomposition of the corresponding benzo- and naphthoxazinones with potassium hydroxide [25]. In view of this, a new and straight-forward approach for the synthesis of 2-substituted napthoxazoles derivatives is divulged, which relied on the $FeCl_3$ catalyzed the reaction between 1-nitroso-2-naphthol with allyl bromides and benzyl bromides under microwave condition.

EXPERIMENTAL

All the starting materials and reagents are available commercially and used as received. Glassware was dried for 24 h at 100 ºC. Solvents used in reactions were distilled over suitable drying agents prior to use. Microwave-assisted reactions were performed using a Biotage single-mode cavity microwave synthesizer producing continuous microwave irradiation at 2450 MHz. Compounds were visualized with UV light (λ = 254 nm). Products were purified by flash chromatography on silica gel 0.04-0.063 mm. Melting points were obtained on a melting point apparatus with open capillary tubes and are uncorrected. ^{1}H (^{13}C) NMR spectra were recorded at 300 (75) and 400 (100) MHz using $CDCl_3$ as the solvent. The ^{1}H and ^{13}C chemical shifts were referenced to residual solvent signals at δ H/C 7.26/77.0 ($CDCl_3$). Coupling constants *J* [Hz] were directly taken from the spectra and are not averaged.

General procedure: A mixture of 1-nitroso-2-naphthol (**1a**) (1 mmol), allyl bromides or benzyl bromides **2** (1 mmol), 15 mol% $FeCl_3$ (24 mg) and *o*-dichlorobenzene (2 mL) was sealed in a 10 mL septum reaction vial and irradiated with microwaves (Biotage, 2450 MHz, 200 W, 100 ºC) for 10 min. After cooling to room temperature the reaction mixture was poured into water (25 mL) and extracted with CH_2Cl_2 (3 × 20 ml). The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated in a vacuum. The residue was purified by flash chromatography on silica gel (cyclohexane/EtOAc = 15:1) to give the desired product [21].

2-Prenylnaphtho[1,2-*d*][1,3]oxazole (3a): Pale yellow oil, 72% yield [Ref. 21]; R_f = 0.32 (cyclohexane-EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.07 (s, 3H), 2.43 (s, 3H), 6.37 (s, 1H), 7.52 (ddd, *J* = 1.2 Hz, *J* = 8.1 Hz, *J* = 8.2 Hz, 1H), 7.61-7.66 (m, 2H), 7.75 (d, *J* = 9.1 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 8.51 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.9, 27.6, 110.0, 112.0, 122.1, 125.1, 125.2, 126.3, 126.7, 128.4, 131.0, 137.2, 146.8, 149.1, 162.2.

2-Cyanonaphtho[1,2-*d*][1,3]oxazole (3b): White solid, 58% yield; R_f = 0.39 (cyclohexane-EtOAc = 5:1); m.p. 164-165 ºC (lit. m.p. 163-164 ºC) [Ref. 21]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.66 (ddd, *J* = 1.5 Hz, *J* = 7.3 Hz, *J* = 7.4 Hz, 1H), 7.72 (d, *J* = 9.0 Hz, 1H), 7.78 (ddd, *J* = 1.0 Hz, *J* = 7.0 Hz, *J* = 8.2 Hz, 1H), 7.99-8.05 (m, 2H), 8.52 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 109.4, 110.5, 122.2, 126.3, 126.9, 128.5, 128.8, 130.7, 131.6, 135.7, 136.1, 148.7.

2-Vinylnaphtho[1,2-*d*][1,3]oxazole (3c): Colourless oil , 50% yield [Ref. 18]; R_f = 0.32 (cyclohexane-EtOAc = 4:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.84 (dd, *J* = 11.1 Hz, *J* = 1.0 Hz, 1H), 6.49 (dd, *J* = 17.6 Hz, *J* = 1.0 Hz, 1H), 6.85 (dd, *J* = 17.6 Hz, *J* = 11.2 Hz, 1H), 7.54 (ddd, *J* = 1.0 Hz, *J* = 7.3 Hz, *J* = 8.0 Hz, 1H), 7.62-7.69 (m, 2H), 7.78 (d, *J* = 8.7 Hz, 1H), 7.96 (d, *J* = 8.1 Hz, 1H), 8.50 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.7, 122.0, 123.9, 124.1, 125.4, 126.4, 127.1, 128.5, 131.1, 137.2, 147.6, 161.4.

2-Phenylnaphtho[1,2-*d*][1,3]oxazole (3d): White solid, 35% yield; m.p.: 133-134 ºC (lit. m.p. 133-135 °C) [Ref. 22]; R_f = 0.52 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.50-7.60 (m, 4H), 7.68 (ddd, *J* = 1.3 Hz, *J* = 8.5 Hz, *J* = 8.1 Hz, 1H), 7.75 (d, *J* = 8.8 Hz, 1H), 7.82 (d, *J* = 8.8 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.30-8.38 (m, 2H), 8.60 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.8, 122.3, 125.5, 126.0, 126.6, 126.9, 127.3, 127.5, 128.6, 128.9, 131.1, 131.2, 137.6, 148.1, 162.4.

2-(4-Cyanophenyl)naphtho[1,2-*d*][1,3]oxazole (3e): White solid, 45% yield; m.p. 220-222 ºC (lit. m.p. 221-222 ºC) [Ref. 21]; R_f = 0.30 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58 (ddd, *J* = 1.2 Hz, *J* = 7.9 Hz, *J* = 8.0 Hz, 1H), 7.70 (ddd, *J* = 1.0 Hz, *J* = 7.9 Hz, *J* = 7.9 Hz, 1H), 7.75 (d, *J* = 9.1 Hz, 2H), 7.80 (d, *J* = 8.7 Hz, 1H), 7.85 (d, *J* = 8.7 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.40 (d, *J* = 8.0 Hz, 2H), 8.56 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.7, 114.1, 118.2, 122.1, 125.7, 126.5, 127.2, 127.3, 127.5, 128.6, 131.31, 131.34, 132.6, 137.6, 148.3, 160.1.

2-(4-Fluorophenyl)naphtho[1,2-*d*][1,3]oxazole (3f): White solid, 42% yield; m.p. 169-170 ºC (lit. m.p. 168-169 ºC) [Ref. 21]; R_f = 0.60 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26-7.32 (m, 2H), 7.61 (ddd, *J* = 1.3 Hz, *J* = 7.1 Hz, *J* = 7.1 Hz, 1H), 7.72 (d, *J* = 7.4 Hz, 1H), 7.77 (d, *J* = 8.7 Hz, 1H), 7.86 (d, *J* = 8.7 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 8.33-8.42 (m, 2H), 8.63(d, *J* = 8.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.7, 116.1 (d, 22.0 Hz), 122.1, 123.8 (d, 3.1 Hz), 125.4, 126.0, 126.5, 126.9, 128.5, 129.4 (d, 8.4 Hz), 131.2, 137.5, 148.0, 161.4, 164.6 (d, 251 Hz).

2-(4-Chlorophenyl)naphtho[1,2-*d*][1,3]oxazole (3g): White solid, 40% yield, m.p. 188-190 ºC (lit. m.p. 189-190 ºC) [Ref. 21] R_f = 0.46 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.2 Hz, 2H), 7.57 (dd, *J* = 1.3 Hz *J* = 7.6 Hz, 1H), 7.67 (d, *J* = 7.4 Hz, 1H), 7.72 (d, *J* = 9.1 Hz, 1H), 7.82 (d, *J* = 9.1 Hz, 1H), 7.98 (d, *J* = 8.6 Hz, 1H), 8.26 (d, *J* = 8.2 Hz, 2H), 8.57 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.7, 122.1, 125.4, 126.0, 126.2, 126.5, 127.0, 128.4, 128.5, 129.2, 131.2, 137.1, 137.6, 148.2, 161.3.

2-(3-Bromophenyl)naphtho[1,2-*d*][1,3]oxazole (3h): White solid, 39% yield; m.p. 197-199 ºC (lit. m.p. 199 ºC) [Ref. 22]; R_f = 0.43 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.42 (t, *J* = 7.9 Hz, 1H), 7.57 (ddd, *J* = 1.2 Hz, *J* = 7.5 Hz, *J* = 7.5 Hz, 1H), 7.62-7.70 (m, 2H), 7.73 (d, *J* = 8.9 Hz, 1H), 7.83 (d, *J* = 9.3 Hz, 1H), 7.98 (d, *J* = 7.8 Hz, 1H), 8.27 (d, *J* = 7.8 Hz, 1H), 8.48 (s, 1H), 8.58 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 110.7, 122.2, 123.0, 125.5, 125.7, 126.52, 126.56, 127.1, 128.6, 129.4, 130.1, 130.4, 131.2, 133.9, 137.5, 148.1, 160.7.

2-(4-Methoxyphenyl)naphtho[1,2-*d*][1,3]oxazole (3i):White solid, 37% yield, m.p. 117-118 ºC (lit. m.p. 116-118 ºC) [Ref. 22]; R_f = 0.31 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.90 (s, 3H), 7.01-7.09 (m, 2H), 7.53 (ddd, *J* = 1.4 Hz, *J* = 7.1 Hz, *J* = 7.2 Hz, 1H), 7.65-7.69 (m, 1H), 7.70 (d, *J* = 8.7 Hz, 1H), 7.77 (d, *J* = 8.8 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 8.23-8.30 (m, 2H), 8.59 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.4, 110.7, 114.3, 120.1, 122.2, 125.1, 125.3, 126.4, 126.7, 128.5, 129.0, 131.1, 137.6, 147.8, 161.9, 162.4.

2-(4-Methylphenyl)naphtho[1,2-*d*][1,3]oxazole (3j): White solid, 34% yield; m.p. 168-169 ºC (lit. m.p. 168-170 ºC) [Ref. 22]; R_f = 0.54 (cyclohexane/EtOAc = 5:1); ^{1}H NMR

(300 MHz, $CDCl_3$): δ 2.45 (s, 3H) , 7.35 (d, *J* = 7.9 Hz, 2H), 7.54 (ddd, *J* = 1.2 Hz, *J* = 7.6 Hz, *J* = 7.7 Hz, 1H), 7.67 (ddd, *J* = 1.4 Hz, *J* = 7.4 Hz, *J* = 7.4 Hz, 1H), 7.75 (d, *J* = 8.9 Hz, 1H), 7.82 (d, *J* = 8.8 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 2H), 8.57 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.7, 110.7, 122.2, 124.7, 125.2, 125.7, 126.5, 126.8, 127.3, 128.5, 129.6, 131.1, 137.6, 141.5, 147.8, 162.5.

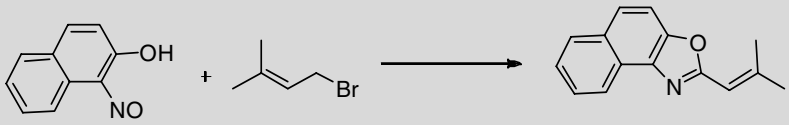
2-(Naphth-2-yl)naphtho[1,2-*d*][1,3]oxazole (3k): White solid, 32% yield; m.p. 154-155 °C (lit. m.p. 154 ºC) [Ref. 21]; R_f = 0.49 (cyclohexane/EtOAc = 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.62 (m, 3H), 7.71 (ddd, *J* = 1.1 Hz, *J* = 7.3 Hz, *J* = 7.2 Hz, 1H), 7.77 (d, *J* = 8.8 Hz, 1H), 7.83 (d, *J* = 8.8 Hz, 1H), 7.87-7.94 (m, 1H), 7.95-8.06 (m, 4H), 8.42 (dd, *J* = 1.9 Hz, *J* = 8.5 Hz, 1H), 8.64 (d, *J* = 8.3 Hz, 1H), 8.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 110.3, 122.2, 123.9, 124.8, 125.4, 126.1, 126.5, 126.8, 127.0, 127.56, 127.57, 127.9, 128.6, 128.7, 128.9, 131.2, 133.1, 134.5, 137.7, 148.1, 162.5.

RESULTS AND DISCUSSION

Initial experiment between 1-nitroso-2-naphthol (**1a**) with prenyl bromide (**2a**) using 10 mol% $FeCl_3$ as a catalyst and CH_3CN as a solvent under reflux for 3 h produced **3a** with 25% yield (Table-1, entry 1). Inspired by this result, we optimized this reaction using different solvents, temperature and amount of catalyst (Table-1). The yield of product **3a** could easily be increased to 45% by increasing the amount of catalyst to 15 mol% (Table-1, entry 2). Increasing the amount of catalyst to 20 mol% and 30 mol% lead to a decrease in the yield to 40% and 34%, respectively (Table-1, entries 3-4). Next, the influence of different solvents, such as dichloroethene, dichloromethane, *o*-dichlorobenzene, DMF, toluene and [bmin]BF_4 on the outcome of the model reaction was investigated (Table-1, entries 5-10). These experiments showed that the reaction can be performed with halogenated solvents with good yield (Table-1, entries 5-7). With DMF and toluene the yield dropped to 5% and 20%, respectively (Table-1, entries 8-9). It must be mentioned that the reaction could be performed in an ionic solvent such as [bmin]BF_4 with moderate yield (Table-1, entry 10). Without any Fe-catalyst, the formation of product **3a** didn't take place at all (Table-1, entry 11).

Over the last decades, vast progress has been made in using microwave irradiation in synthetic organic chemistry [26,27]. The highly effective uniform heating source in microwave leads to accelerate the reaction rate, provide better yields, greater reproducibility of reactions, help in developing cleaner synthetic methods and reduce reaction times. Due to these benefits of microwave, the model reaction was performed using a sealed vial and microwave irradiation at 100 ºC (Table-2, entries 1 and 2). Remarkably enough, the yield could be increased to 68% in a sealed vial and 72% in the microwave. In summary, the best results were achieved when 1 mmol of 1-nitroso-2-naphthol

TABLE-1
OPTIMIZATION OF THE MODEL REACTION UNDER DIFFERENT AMOUNT OF CATALYST, SOLVENTS AND TEMPERATURE[a]

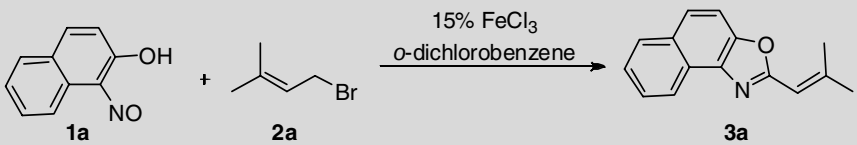


1a **2a** **3a**

Entry	$FeCl_3$ (mol%)	Solvent	Temp. (°C)	Yield **3a** (%)[b]
1	10	CH_3CN	Reflux	25
2	15	CH_3CN	Reflux	45
3	20	CH_3CN	Reflux	40
4	30	CH_3CN	Reflux	34
5	15	CH_2ClCH_2Cl	Reflux	61
6	15	CH_2Cl_2	Reflux	48
7	15	*o*-Dichlorobenzene	100	64
8	15	DMF	100	5
9	15	Toluene	100	20
10	15	[bmin]BF4	100	42
11	–	*o*-Dichlorobenzene	100	–

[a]All reactions were carried using 1 mmol **1a** and 1 mmol **2a**; [b]Isolated Yield.

TABLE-2
OPTIMIZATION OF THE MODEL REACTION UNDER DIFFERENT CONDITIONS[a]



1a **2a** **3a**

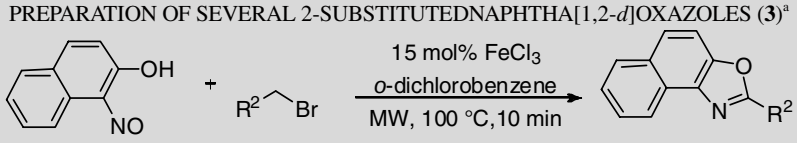
Entry	Condition	Temp. (°C)	Time	Yield **3a** (%)
1	Sealed tube	100	3 h	68
2[b]	Microwave	100	10 min	72

[a]All reactions were carried using 1 mmol **1a** and 1 mmol **2a**; [b]The reactions were performed using a microwave apparatus (200 W).

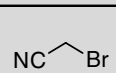
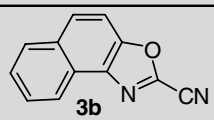
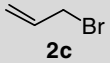
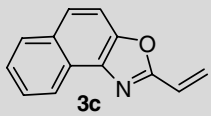
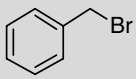
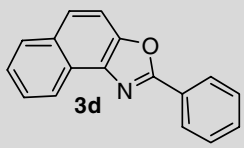
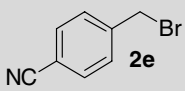
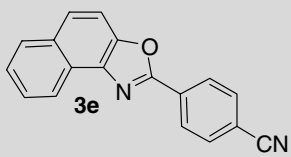
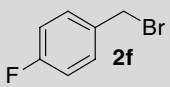
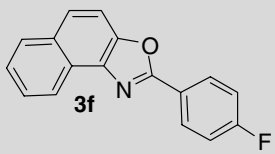
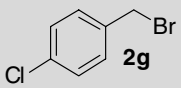
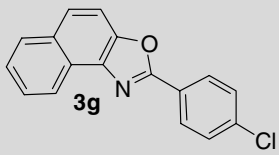
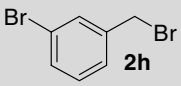
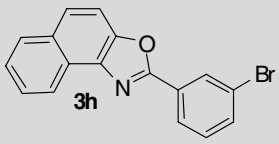
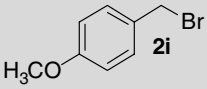
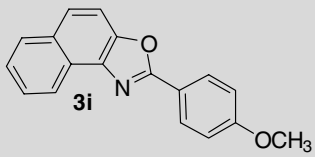
(**1a**) and 1 mmol of prenyl bromide (**2a**) in the presence of 15 mol% $FeCl_3$ in *o*-dichlorobenzene under microwave at 100 ºC for 10 min.

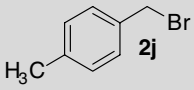
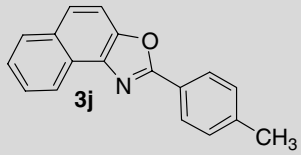
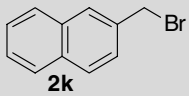
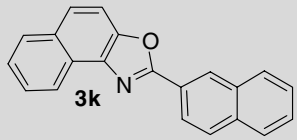
With the optimized reaction conditions, 2-substituted naphtho[1,2-*d*][1,3]oxazoles (**3b-k**) were synthesized with yields ranging from 32% to 58% by reacting 1-nitroso-2-naphthol (**1a**) with a number of allyl bromides and benzyl bromides (**2b-k**) (Table-3). The reaction with bromo acetonitrile (**2b**) and allyl bromide (**2c**) delivered 2-cyanonaphthoxazole, 2-vinyl naphthoxazole with yield 58% and 50%, respectively (Table-3, entries 1 and 2). A conversion could also be performed with a number of benzyl bromides (**2d-j**) having different substituents on the phenyl group with yield ranging from 34 to 45% (Table-3, entries 3-9). Several substituents could be employed such as cyano, fluoro, chloro, bromo, methoxy and methyl groups. It seems that the influence of the nature of the substituent on the phenyl group of 2 is only small. The yields with electron-withdrawing groups are comparable to electron-donating groups. In addition to allyl brmides and benzyl brmides the reaction was also performed with 2-(bromomethyl)naphthalene (**2k**) to yield 2-(naphth-2-yl)naphtho[1,2-*d*][1,3]oxazole (**3k**) with yield 32% (Table-3, entry 10).

TABLE-3
PREPARATION OF SEVERAL 2-SUBSTITUTEDNAPHTHA[1,2-*d*]OXAZOLES (**3**)[a]



1a **2** **3**

Entry	Substrate	Product	Yield (%)[b]
1	 **2b**	 **3b**	58
2	 **2c**	 **3c**	50
3	 **2d**	 **3d**	35
4	 **2e**	 **3e**	45
5	 **2f**	 **3f**	42
6	 **2g**	 **3g**	40
7	 **2h**	 **3h**	39
8	 **2i**	 **3i**	37

9			34
10			32

[a]All reactions were carried using 1 mmol **1** and 1 mmol **2**; [b]Isolated yield

Conclusion

In conclusion, a simple and highly efficient method for the synthesis of 2-substituted naphtho[1,2-*d*][1,3]oxazoles has been developed. Best results were accomplished when 1 mmol of compound **1a** was reacted with 1 mmol of compound **2** in the presence of 15 mol% $FeCl_3$ in *o*-dichlorobenzene solvent. Using microwave condition the time for the synthesis of 2-substituted naphthoxazoles (**3a-k**) was reduced from 3 h to 10 min with increased yield.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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