Research on Chemical Intermediates (2018) 44:7449–7458
https://doi.org/10.1007/s11164-018-3566-y

Synthesis of 3-aminoisoxazolmethylnaphthols via one-pot three-component reaction under solvent-free conditions

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Received: 5 May 2018 / Accepted: 16 August 2018 / Published online: 5 September 2018

Abstract
A one-pot three-component condensation reaction of 3-amino-5-methylisoxazole, aryl aldehyde and 2-naphthol to afford the corresponding 3-amino isoxazol-methylnaphthols in good to excellent yields. The remarkable features of this new procedure are high conversions, clean reaction condition, short reaction time, nonhazardous and environmentally friendly reaction condition, inexpensive and easily commercially availability of the catalyst and simple work-up procedures.

Keywords 3-Amino-5-methylisoxazole · β-Naphthol · Aryl aldehyde · Solvent-free conditions

Introduction

Multi-component reactions (MCRs) are an effective strategy for the synthesis of complex structures and research into new processes can lead to the discovery of unreported reactions. [1–4]. Since they are one-pot processes in which three or more accessible components react to form a single product, the results of them can be achieved in an expedient manner without isolation of any intermediate [5, 6]. Biginelli [7, 8], Betti [9], Passerini [10, 11], and Mannich [12–14] reactions are some examples of MCRs. Nevertheless, development and discovery of new MCRs is still in demand. Recently, this strategy became important in drug discovery in the context of synthesis of biologically active compounds. This method increases the efficiency of the reactions by reducing the reaction time and increases the yield of products in comparison with normal multistep methods [15, 16]. Furthermore,

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isoxazole derivatives, especially 5-methylisoxazole, represent an interesting class of heterocycles possessing a wide range of biological activity [17–22].

A large number of isoxazole derivatives exhibited antibacterial [23], antifungal [24], anticonvulsant [25], analgesic and anticancer [26] activity (Fig. 1).

In continuation of our work on one-pot multi-component reactions (MCRs) [27–31], we embarked on the synthesis of a novel compound possessing 2-amino-5-methylisoxazole, aromatic aldehydes and 2-naphthol moieties embedded in a fused molecular framework via a three-component reaction under solvent-free conditions (Scheme 1).

Experimental section

General

Infrared (IR) spectra and melting points of all compounds were determined using an Electrothermal 9100 apparatus and an FT-IR-JASCO-460 plus spectrometer. The ^{1}H and ^{13}C NMR spectra of compounds were recorded on a Bruker DRX-300 Avance instrument in DMSO at 300 MHz. Mass spectra were obtained on Agilent-HP and

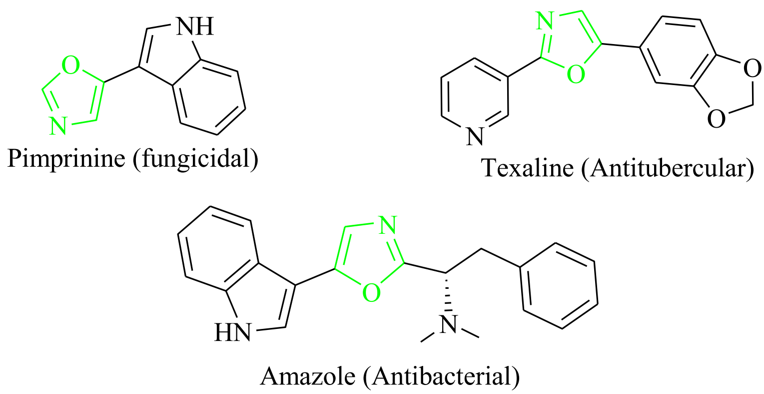
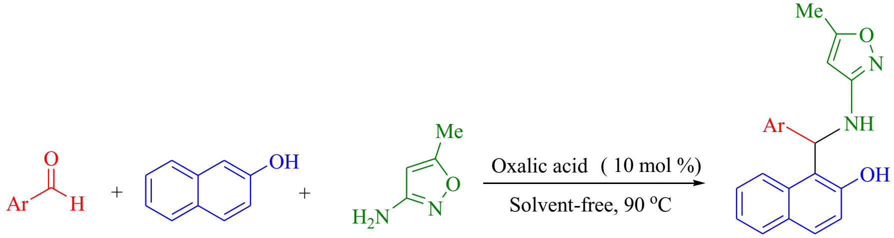


Fig. 1 A few selected drugs and biologically active compounds containing an oxazole ring



1 2 3 4

Scheme 1 Synthesis of 3-aminoisoxazolmethylnaphthols

Sciex-3200 Technology spectrometers operating at an ionization potential of 70 eV. We purchased all the chemicals from chemical producers Merck and Fluka and they were used without further purification.

General procedure for the preparation of 3-aminoisoxazolmethylnaphthols

To a mixture of aldehyde (1.0 mmol), 2-naphthol (1.0 mmol) and 3-amino-5-methylisoxazole (1.0 mmol) oxalic acid (10 mol%) were added. The mixture was stirred at 90 °C in an oil bath and the reaction was followed by thin layer chromatography (TLC). After completion of the reaction, the mixture was cooled to room temperature and washed with water to remove oxalic acid from the reaction mixture. The solid obtained was recrystallized from absolute EtOH to furnish the desired pure product.

Spectral data for the selected compounds

1-[(5-Methyl-isoxazol-3-ylamino)-phenyl-methyl]-naphthalen-2-ol (4a) White powder, IR (KBr ν_{max}, cm^{-1}): 3403, 3058, 2925, 1628, 1600, 1581, 1541, 1516, 1493; ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.90 (1H, s, CHNH), 6.74–6.85 (2H, dd, H_{aromat}), 7.14–7.41 (8H, m, H_{aromat}), 7.76–7.83 (2H, m, H_{aromat}), 8.01–8.04 (1H, d, NH), 10.10 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 53.0, 94.5, 118.9, 120.4, 122.8, 124.1, 126.4, 126.6, 128.3, 128.9, 129.0, 129.5, 132.6, 143.6, 153.2, 165.0, 167.7; MS *m/z* (%):77.1 (5), 98.1 (7), 115.2 (8), 202.2 (23), 215.2 (8), 231.2 (100), 246.2 (4), 330.2 (M^+, 3).

1-[(5-Methyl-isoxazol-3-ylamino)-p-tolyl-methyl]-naphthalen-2-ol (4b) White powder, IR (KBr ν_{max}, cm^{-1}): 3393, 3060, 1625, 1581, 1538, 1535, 1511, 1478, 1437; ^{1}H NMR (300 MHz, DMSO-d_6): 2.22, 2.24 (6H, s, $2CH_3$), 5.88 (1H, s, CHNH), 6.67–6.80 (2H, dd, H_{aromat}), 7.05–7.08 (2H, d, H_{aromat}), 7.19–7.29 (4H, m, H_{aromat}), 7.34–7.39 (1H, t, H_{aromat}), 7.74–7.82 (2H, m, H_{aromat}), 7.99–8.02 (1H, d, NH), 10.06 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 21.0, 52.9, 94.4, 118.9, 120.5, 122.7, 124.2, 126.6, 128.9, 129.0, 129.4, 132.6, 135.3, 140.6, 153.1, 164.9, 167.7. MS *m/z* (%): 98.2 (10), 115.5 (8), 202.2 (20), 216.2 (9), 231.2 (100), 245.2 (46), 344.3 (M^+, 2).

1-[(4-Methoxy-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4c) White powder, IR (KBr ν_{max}, cm^{-1}): 3380, 2962, 1626, 1579, 1556, 1509, 1475, 1436; ^{1}H NMR (300 MHz, DMSO-d_6): 2.21 (3H, s, CH_3), 3.69 (3H, s, OCH_3), 5.86 (1H, s, CHNH), 6.62–6.64 (1H, d, H_{aromat}), 6.76–6.85 (3H, m, H_{aromat}), 7.20–7.39 (4H, m, H_{aromat}), 7.73–7.81 (2H, m, H_{aromat}), 7.99–8.02 (1H, d, NH), 10.06 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.7, 55.4, 94.4, 113.8, 118.9, 120.4, 122.7, 124.1, 126.5, 127.8, 128.9, 129.0, 129.4, 132.6, 135.4, 153.1, 158.0, 164.9, 167.7; MS *m/z* (%): 98.2 (20), 115.2(5), 165.2(10), 189.2 (45), 202.2 (10), 218.2 (30), 231.2 (95), 261.1 (100), 360.3 (M^+, 2).

1-[(4-Chloro-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4d) White powder, IR (KBr ν_{max}, cm^{-1}): 3389, 3064, 1624, 1580, 1572, 1532, 1516, 1488; ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.88 (1H, s, CHNH), 6.69–6.87 (2H, dd, H_{aromat}), 7.25–7.42 (7H, m, H_{aromat}), 7.77–7.83 (2H, t, H_{aromat}), 7.95–7.98 (1H, d, NH), 10.16 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.5, 94.4, 118.8, 119.9, 122.9, 124.0, 126.8, 128.3, 128.5, 129.0, 129.8, 130.9, 132.5, 142.8, 153.2, 164.9, 167.8; MS *m/z* (%): 98.2 (8), 115.2 (9), 202.2 (32), 231.2 (100), 265.1 (58), 364.2 (M^+, 1).

1-[(3-Chloro-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4e) White powder, IR (KBr v_{max}, cm^{-1}): 3414, 3066, 1629, 1592, 1551, 1537, 1516, 1473, 1437; ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.88 (1H, s, CHNH), 6.70–6.91 (2H, dd, H_{aromat}), 7.19–7.34 (6H, m, H_{aromat}), 7.39–7.44 (1H, t, H_{aromat}), 7.78–7.84 (1H, t, H_{aromat}), 7.97–8.00 (1H, d, NH), 10.16 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.6, 94.4, 118.8, 119.8, 122.9, 123.8, 125.4, 126.3, 126.4, 126.9, 129.0, 129.0, 129.9, 130.3, 132.4, 133.2, 146.5, 153.2, 164.8, 167.9; MS *m/z* (%): 98.2 (10), 115.2 (13), 202.2 (32), 231.2 (100), 265.1 (48), 364.2 (M^+, 3).

1-[(2-2.3.6.1-[(3-Chloro-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4f) White powder, IR (KBr v_{max}, cm^{-1}): 3411, 3057, 2924, 1629, 1583, 1544, 1515, 1468, 1438; ^{1}H NMR (300 MHz, DMSO-d_6): 2.22 (3H, s, CH_3), 5.78 (1H, s, CHNH), 6.72–6.89 (2H, dd, H_{aromat}), 7.16–7.42 (6H, m, H_{aromat}), 7.69–7.82 (2H, m, H_{aromat}), 8.04–8.07 (1H, d, NH), 9.94 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.5, 94.4, 118.6, 120.0, 122.9, 123.6, 125.4, 126.5, 126.4, 126.9, 129.2, 129.5, 129.9, 130.3, 132.4, 134.2, 146.5, 154.2, 163.6, 168.2; MS *m/z* (%): 98.2 (5), 115.2 (7), 202.2 (20), 231.2 (100), 265.1 (8), 364.2 (M^+, 1).

1-[(4-Bromo-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4 g) White powder, IR (KBr v_{max}, cm^{-1}): 3391, 3064, 1624, 1580, 1533, 1516, 1486, 1436. ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.88 (1H, s, CHNH), 6.67–6.87 (2H,dd, H_{aromat}), 7.23–7.48 (7H, m, H_{aromat}), 7.77–7.83 (2H,t, H_{aromat}), 7.95–7.98 (1H, d, NH), 10.16 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.6, 94.4, 118.8, 119.4, 119.9, 122.9, 124.0, 126.8, 128.9, 129.0, 129.8, 131.2, 132.9, 143.3, 153.2, 164.9, 167.8; MS *m/z* (%): 98.2 (8), 101.2 (9), 115.3 (9), 202.2 (35), 215.2 (4), 231.2 (100), 311.2 (23), 408.2 (M^+, 1).

1-[(3-Bromo-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4h) White powder, IR (KBr v_{max}, cm^{-1}): 3417, 3065, 1629, 1586, 1555, 1516, 1504, 1474, 1437; ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.88 (1H, s, CHNH), 6.70–6.91 (2H, dd, H_{aromat}), 7.19–7.49 (7H, m, H_{aromat}), 7.78–7.84 (2H, t, H_{aromat}), 7.98–8.00 (1H, d, NH), 10.14 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.6, 94.4, 118.8, 119.8, 121.9, 122.9, 123.8, 125.8, 126.9, 129.0, 129.0, 129.1, 129.3, 129.9, 130.6, 132.4, 146.7, 153.2, 164.8, 167.9; MS *m/z* (%): 98.2 (10), 101.2 (9), 115.3 (12), 202.2 (32), 231.2 (100), 311.1 (25), 408.2 (M^+, 1).

1-[(4-Fluoro-phenyl)-(5-methyl-isoxazol-3-ylamino)-methyl]-naphthalen-2-ol (4i) White powder, IR (KBr v_{max}, cm^{-1}): 3400, 3061, 1626, 1604, 1582, 1539,1507, 1470, 1437; ^{1}H NMR (300 MHz, DMSO-d_6): 2.23 (3H, s, CH_3), 5.87 (1H, s, CHNH), 6.68–6.86 (2H, dd, H_{aromat}), 7.06–7012 (2H, t, H_{aromat}), 7.25–7.33 (4H, m, H_{aromat}), 7.36–7.41 (1H, t, H_{aromat}), 7.76–7.83 (2H, m, H_{aromat}), 7.97–7.99 (1H, d, NH), 10.11 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.5, 94.4, 114.9, 115.1, 118.8, 120.1, 122.8, 124.0, 126.7, 128.4, 128.5, 129.0, 129.7, 132.5, 139.7, 139.7, 153.1, 159.5, 162.7, 164.9, 167.8; MS *m/z* (%): 98.2 (10), 115.2 (8), 202.2 (8), 220.2 (21), 249.2 (100), 348.3 (M^+, 1).

1-[(5-Methyl-isoxazol-3-ylamino)-(4-nitro-phenyl)-methyl]-naphthalen-2-ol (4j) Yellow powder, IR (KBr ν_{max}, cm^{-1}): 3416, 3082, 1628, 1604, 1595, 1582, 1541, 1514, 1488; ^{1}H NMR (300 MHz, DMSO-d_6): 2.25 (3H, s, CH_3), 5.89 (1H, s, CHNH), 6.79–7.00 (2H, dd, H_{aromat}), 7.26–7.30 (2H, m, H_{aromat}), 7.38–7.43 (1H, t, H_{aromat}), 7.52–7.55 (2H, d, H_{aromat}), 7.80–7.85 (2H, m, H_{aromat}), 7.93–7.96 (1H, d, NH), 8.15–8.18 (2H, d, H_{aromat}), 10.25 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.9, 94.4, 96.3, 118.4, 118.8, 119.4, 123.0, 123.6, 123.8, 124.4, 127.0, 127.7, 127.8, 128.9, 129.0, 129.1, 130.0, 130.2, 132.4, 146.3, 152.2, 153.3, 164.8, 168.0, 169.8; MS *m/z* (%): 43.2 (17), 115.2 (50), 144.2 (60), 202.2 (39), 230.2 (100), 2454.2 (32), 291.2 (52), 375.3 (M^+, 1).

1-[(5-Methylisoxazol-3-ylamino)-(pyridin-4-yl)-methyl]-naphthalen-2-ol (4k) White powder, IR (KBr ν_{max}, cm^{-1}): 3375, 3140, 2924, 1629, 1604, 1576, 1560, 1512; ^{1}H NMR (300 MHz, DMSO-d_6): 2.24 (3H, s, CH_3), 5.89 (1H, s, CHNH), 6.70–6.93 (2H, dd, H_{aromat}), 7.25–7.30 (4H, m, H_{aromat}), 7.38–7.43 (1H, t, H_{aromat}), 7.79–7.84 (2H, t, H_{aromat}), 7.92–7.95 (1H, d, NH), 8.44–8.46 (2H, d, H_{aromat}), 10.2 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 52.3, 94.4, 118.8, 119.2, 121.9, 122.9, 123.8, 126.9, 129.0, 129.0, 130.1, 132.4, 149.6, 153.0, 153.4, 164.8, 168.0; MS *m/z* (%): 83.0 (9), 121.0 (9), 188.0 (13), 234.0 (15), 256.0 (30), 304.2 (20), 332.1 (100), 333.1 (14), 334.1 (M^++ H, 3).

1-[(5-Methylisoxazol-3-ylamino)-(thiophen-2-yl)methyl]-naphthalen-2-ol (4l) White powder, IR (KBr ν_{max}, cm^{-1}): 3394, 3113, 1624, 1582, 1537, 1530, 1516; ^{1}H NMR (300 MHz, DMSO-d_6): 2.21 (3H, s, CH_3), 5.90 (1H, s, CHNH), 6.79–6.80 (1H, d, H_{aromat}), 6.89–6.91 (2H, t, H_{aromat}), 6.97 (1H, d, H_{aromat}), 7.25–7.33 (3H, m, H_{aromat}), 7.40–7.45 (1H, t, H_{aromat}), 7.77–7.84 (2H, m, H_{aromat}),8.09–8.12 (1H, d, NH), 10.2 (1H, s, OH); ^{13}C NMR (75 MHz, DMSO-d_6): 12.5, 50.5, 94.4, 118.9, 119.6, 122.9, 123.7, 124.3, 124.7, 126.7, 126.9, 128.9, 129.0, 129.8, 132.5,148.2, 153.3, 164.5, 168.0; MS *m/z* (%):59.0 (3), 96.9 (100), 118.9 (10), 224.0 (11), 236.9 (7), 235.0 (M^+–H, 96).

Results and discussion

We compared the catalytic activity of oxalic acid with some other catalysts reported in the literature for the three-component condensation reaction of benzaldehyde, 2-naphthol and 3-amino-5-methylisoxazole in solvent-free conditions after 40 min at 80 °C (Table 1). In the absence of the catalyst, no product was formed (Table 1, entry 1). After several experiments, oxalic acid was found to the most efficient catalyst among all the other examined catalysts under these conditions.

To achieve an optimal temperature, solvent and amounts of catalyst, we used different solvents such as ethanol, tetrahydrofuran (THF), dimethylformamide (DMF), $AcOH:H_2O$(1:1) and chloroform in 70 °C, but no superior results were obtained as compared to solvent-free condition (Table 2, entries 1–8). Also, we studied the influence of temperature on the reaction time and percentage yield. Table 2 displays that 90 °C was optimum temperature for maximum conversion. Increasing the temperature above 90 °C neither improved the yield nor decreased the reaction time (Table 2, entries 9–13). In contrast, reducing the temperature is

Table 1 Effects of catalyst on the synthesis of 3-aminoisoxazolmethylnaphthols

Entry	Catalyst (10 mol%)	Yield (%)[a]
1	–	Trace
2	Fe_2O_3	30
3	$MgSO_4·3H_2O$	72
4	$ZnCl_2$	75
5	K_2HPO_4	65
6	$CoCl_2·6H_2O$	60
7	Oxalic acid	86
8	NaOAc	76

[a]Isolated yields

Table 2 Optimization of solvent, temperature and catalyst in synthesis of compound **4a**

Entry	Solvent	Temperature (°C)	Catalyst (mol%)	Time (min)	Yield (%)[a]
1	EtOH	70	10	30	55
2	H_2O: EtOH(1:1)	70	10	30	60
3	$AcOH:H_2O$(1:1)	70	10	35	66
4	AcOH	70	10	45	53
5	DMF	70	10	40	40
6	THF	70	10	45	50
7	$CHCl_3$	70	10	40	55
8	–	70	10	25	81
9	–	r.t	10	60	Trace
10	–	50	10	45	57
11	**–**	**90**	**10**	**8**	**92**
12	–	100	10	10	90
13	–	110	10	10	90
14	–	90	3	20	85
15	–	90	5	15	90
16	–	90	15	22	88

[a]Isolated yields. The bolded entry indicates the largest yield obtained.

Table 3 Synthesis of 3-aminoisoxazolmethylnaphthols using oxalic acid in solvent-free conditions

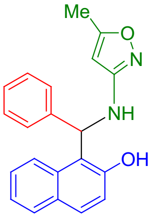
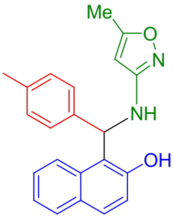
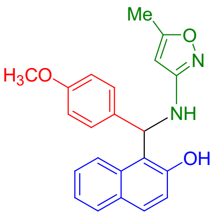
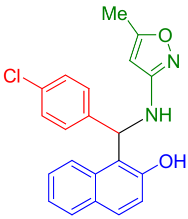
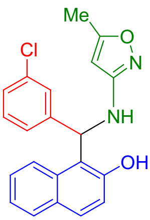
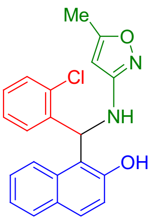
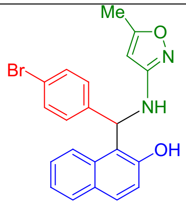
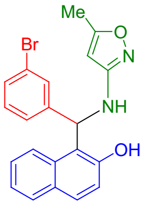
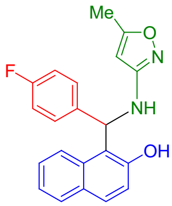
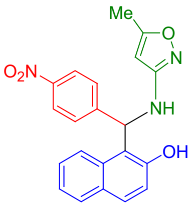
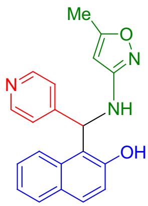
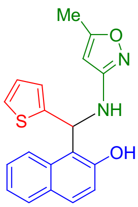
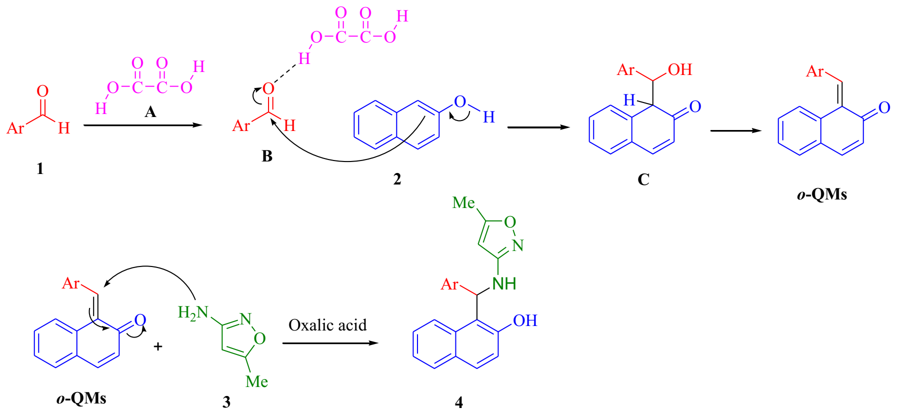
Entry	Product	Time (min)	Isolated yield (%)[a]	Product	M.p (°C)
1		8	92	**4a**	212–214
2		12	90	**4b**	202–203
3		16	88	**4c**	194–195
4		6	93	**4d**	194–195
5		10	91	**4e**	193–195
6		8	92	**4f**	185–186

Table 3 continued

Entry	Product	Time (min)	Isolated yield (%)[a]	Product	M.p (°C)
7		10	92	**4g**	200–201
8		12	90	**4h**	195–196
9		8	94	**4i**	193–194
10		10	91	**4j**	150–151
11		12	91	**4k**	200–202
12		10	92	**4l**	185–187

[a]Yields refer to those of pure isolated products characterized by IR, ^{1}H and ^{13}C NMR spectroscopic data and mass spectrometry



Scheme 2 A suggested mechanism for the three-component synthesis of 3-aminoisoxazolmethylnaphthols (**4a–k**)

detrimental to the reaction, resulting in lower yields. We also studied the model reaction catalyzed by oxalic acid with different catalyst loading (Table 2, entries 14–16). The optimal quantity of catalyst was found to be 10 mol%. Excess amount of catalyst did not increase the yields.

The reactions of 2-naphthol with various aromatic aldehydes and 3-amino-5-methylisoxazole were carried out in the presence of 10 mol% oxalic acid at 90 °C. The results are summarized in Table 3 and showed an excellent tolerance toward electron-withdrawing and electron-donating groups with similar yields and reaction times. However, aromatic aldehydes containing electron-withdrawing groups gave shorter times and higher yields than that with electron-donating groups. A suggested mechanism for this transformation is shown in Scheme 2. According to the literature [28, 32, 33], it is suggested that the reaction is performed through *o*-QMs. All the products **4a–l** are new compounds identified by IR, ^{1}H NMR, ^{13}C NMR and MS spectral data.

Conclusion

In this research, we have demonstrated a green and efficient method for the synthesis of 3-aminoisoxazolmethylnaphthols via condensation of an aldehyde, 2-naphthol and 3-amino-5-methylisoxazole using oxalic acid as the catalyst in solvent-free conditions. The catalyst is green, inexpensive and readily commercially available. Simple experimental procedure, excellent yields, shorter reaction time, easy workup, product purity, a nonhazardous nature and environmentally friendly reaction conditions are the key features of this procedure.

Acknowledgements We gratefully acknowledge financial support from the Research Council of the University of Sistan and Baluchestan.

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