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Solvent-free synthesis of 1-(benzothiazolylamino) methyl-2-naphthols with maltose as green catalyst

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Abstract We report an efficient and entirely green procedure for preparation of 1-(benzothiazolylamino)methyl-2-naphthol derivatives involving multi-component, one-pot condensation reaction of 2-naphthol, 2-aminobenzothiazole, and aromatic aldehydes in the presence of maltose under solvent-free conditions. This method has several advantages, including mild conditions, high yields, clean reaction profile, simple operation, and environmentally benign and simple work-up procedures.

Keywords Green procedure · 1-(Benzothiazolylamino)methyl-2-naphthol · Multi-component reaction · Maltose · Solvent-free conditions

Introduction

Multi-component reactions (MCR) are important in combinatorial chemistry because of their suitability for synthesis of small drug-like molecules with structural diversity. These reactions enable compound synthesis in few steps, usually in one pot [1]. Among the advantages of these reactions are that they are inexpensive, simple, and time and energy-saving with high bond-forming efficiency [2]. Therefore finding and designing new MCR has been the subject of extensive research.

In modern organic synthesis, biologically active benzothiazoles are regarded as valuable building blocks. 2-Aminobenzothiazoles are unique structures that are widely used in medicinal and biological chemistry [3]. Their diverse functions range from facilitation of electron transfer in the firefly luciferine cycle [4], and, in pharmaceutical chemistry, antitumor [5] and antidiabetic activity [6], use as

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indicators of Alzheimer's disease [7], and anticancer activity [8]. Benzothiazoles are also commercially important as reactive dyes [9], hair dyes [10] agrochemical fungicides, insecticides, acaricides, herbicides, plant desiccants, and defoliants [11].

Because of the biological activity of 2-aminobenzothiazoles, and as part of our ongoing program on MCR [12, 13], herein we report an eco-friendly, simple, and efficient method for synthesis of 1-(benzothiazolylamino)methyl-2-naphthols by one-pot three-component reaction of 2-naphthol, 2-aminobenzothiazole, and aromatic aldehydes in the presence of maltose under solvent-free conditions (Scheme 1).

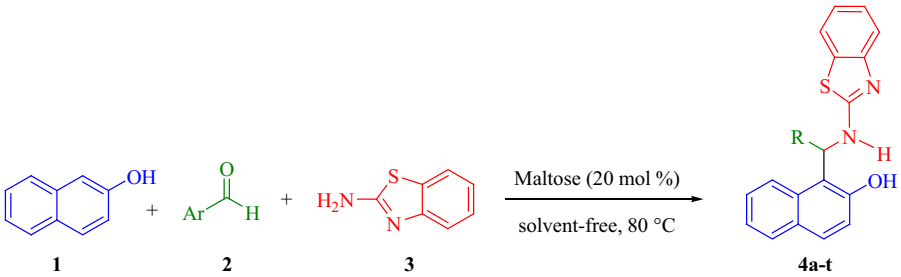
The principles of green chemistry were introduced to eliminate or reduce the use of hazardous materials such as H_2SO_4 or H_3PO_4 in chemical processes. Cleaner techniques make use of such environmentally benign materials as carbohydrates, which are also inexpensive and safe [14]. For this reason, we focused our investigation on developing new, environmentally benign synthetic methods for preparation of 1-(benzothiazolylamino)methyl-2-naphthols. This one-pot reaction under solvent-free conditions is not only operationally simple, clean, and efficient but also consistently gives the corresponding products in good to excellent yields.

Results and discussion

We performed a set of preliminary experiments with 2-naphthol, 2-aminobenzothiazole, and benzaldehyde in the presence of different catalysts as model reaction. Initially, different carbohydrates were screened as catalyst in the model reaction. The reaction did not progress even after 24 h in the absence of catalyst. When the reaction was performed in the presence of sucrose, xylose, or lactose the product was obtained in moderate yields. However, 20 mol% maltose proved an efficient catalyst giving high yields. As shown in Table 1, the shortest reaction time and best yield were achieved at 80 °C. Increasing the amount of catalyst or the temperature did not improve the results.

To evaluate the generality of the process, use of the method for synthesis of other 1-(benzothiazolylamino)methyl-2-naphthols (**4**) was studied (Table 2). Reaction of 2-naphthol with a variety of aromatic aldehydes and 2-aminobenzothiazole was performed in the presence of 20 mol% maltose at 80 °C. In all the reactions, good to excellent yields were obtained in short reaction times (5–15 min). Using these optimized reaction conditions, use of several types of aldehyde was investigated. As shown in Table 2, the direct three-component reactions worked well with a variety of aryl aldehydes, including those bearing electron-withdrawing and electron-donating groups, and the desired products were obtained in good yields, although product yield was lower for aryl aldehydes containing electron-withdrawing substituents.

A suggested mechanism for this transformation is proposed in Scheme 2. As reported in the literature [15–19], reaction of 2-naphthol with aldehydes in the presence of catalyst is known to give *ortho*-quinone methides (*o*-QMs). The same *o*-QMs, generated in situ, have been reacted with 2-aminobenzothiazole via conjugate



Scheme 1 Maltose-catalyzed synthesis of 1-(benzothiazolylamino)methyl-2-naphthols **4a–t**

Table 1 Optimization of the catalyst for synthesis of 1-(benzothiazolylamino)methyl-2-naphthol[a]

Entry	Catalyst (mol%)	Temperature (°C)	Time (min)	Isolated Yield (%)
1	Lactose (10)	80	20	45
2	Sucrose (10)	80	14	60
3	Xylose (10)	80	30	25
4	Maltose (10)	80	14	80
5	Maltose (15)	80	10	85
6	Maltose (20)	80	10	93
7	Maltose (30)	80	8	87
8	Maltose (20)	60	20	35
9	Maltose (20)	25	90	–
9	Maltose (20)	50	90	10
9	Maltose (20)	100	8	55

[a] Reaction conditions: 2-naphthol (1.0 mmol), 2-aminobenzothiazole (1.0 mmol), and benzaldehyde (1.0 mmol) in the presence of different catalysts at different temperatures

addition to form 1-(benzothiazolylamino)methyl-2-naphthols **4a–t**. There is an alternative pathway, a Mannich-type reaction (Scheme 3).

The products were characterized by IR, ^{1}H NMR, and ^{13}C NMR spectroscopy. The ^{1}H NMR spectrum of **4s** contained a multiplet at $\delta = 7.15$–7.45 for the seven aromatic hydrogens and an aliphatic CH, and two singlets at $\delta = 8.61$ and $\delta = 9.92$ for the NH and OH groups, respectively.

Experimental

General

Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a Jasco FT/IR- 460 plus spectrometer, respectively. ^{1}H and ^{13}C NMR spectra were obtained on Bruker DRX-400 Avance instruments with DMSO as a solvent.

Table 2 Synthesis of 1-(benzothiazolylamino)methyl-2-naphthol derivatives

Entry	Ar	Time (min)	Yield (%)[a]	Product	M.p. (lit. m.p.) (°C) [Ref.]
1	4-O_2N-C_6H_4	15	45	**4a**	188–190 (189–191) [15]
2	4-Cl-C_6H_4	5	89	**4b**	208–210 (209–210) [16]
3	3- O_2N-C_6H_4	6	50	**4c**	190–192 (191-194) [17]
4	2,4-$Cl_2C_6H_3$	6	82	**4d**	204–206 (206–207) [15]
5	3-MeO-C_6H_4	7	89	**4e**	185–187 (184–186) [17]
6	4-Me-C_6H_4	12	90	**4f**	183–185 (182–183) [16]
7	2-Cl-C_6H_4	6	88	**4g**	187–189 (189–190) [15]
8	2,4-$(MeO)_2$-C_6H_3	10	89	**4h**	162–164 (161–163) [17]
9	4-MeO-C_6H_4	9	92	**4i**	173–175 (175–176) [16]
10	2- O_2N-C_6H_4	15	58	**4j**	212–214 (215–216) [15]
11	2,6-Cl_2-C_6H_3	8	86	**4k**	194–196 (193–195) [20]
12	3-Br-C_6H_4	5	95	**4l**	200–202 (202–204) [17]
13	4-Br-C_6H_4	5	92	**4m**	200–202 (200–202) [20]
14	4-F-C_6H_4	13	82	**4n**	175–177 (176–178) [17]
15	5-Br,2-HO-C_6H_3	9	90	**4o**	181–183 (183–185) [20]
16	C_4H_3S	10	90	**4p**	190–192 (191–193) [20]
17	2,3-$(MeO)_2$-C_6H_3	12	93	**4q**	200–202 (201–203) [20]
18	Ph	10	89	**4r**	202–204 (202–203) [15]
19	2,5-$(MeO)_2$-C_6H_3	12	90	**4s**	209–211[b]
20	5- MeO, 2-HO-C_6H_3	8	92	**4t**	200–202[b]

[a] Isolated yield

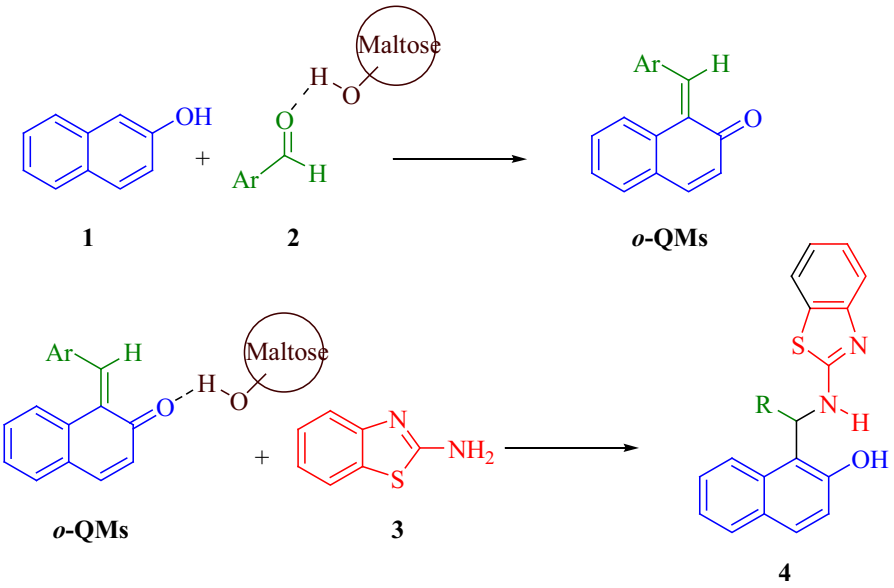
[b] New compounds synthesized in this work

All reagents and solvents were obtained from Fluka and Merck and used without further purification.

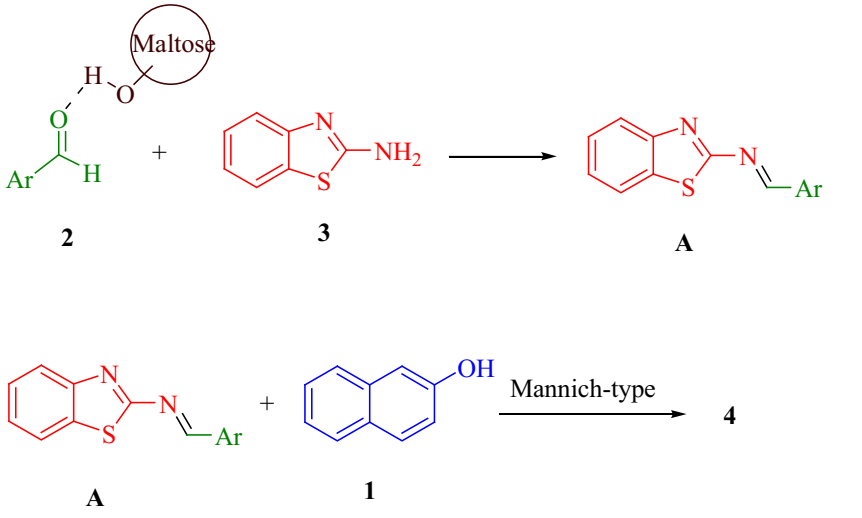
Typical procedure for the synthesis of 1-amidoalkyl -2-naphthols (**4a**–**t**)

Maltose (20 mol%, 0.068 g) was added to a mixture of benzaldehyde (1.0 mmol), 2-naphthol (1.0 mmol), and 2-aminobenzothiazole (1.0 mmol), then the reaction mixture was heated to 80 °C and maintained for the appropriate time (Table 1). After completion of the reaction (monitored by TLC), the reaction mixture was washed with H_2O (3 × 10 mL). The catalyst is soluble in water and was removed from the reaction mixture. The residue was then recrystallized from EtOH to furnish the pure product. Analytical and spectral data for the products are listed below:

1-((benzo[*d*]thiazol-2-ylamino)(4-chlorophenyl)methyl)naphthalen-2-ol (**4b**) Yield: 89 %; m.p. 208–210 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.81$ (s, 1H, $H_{aromatic}$), 6.89–6.91 (m, 1H, $H_{aromatic}$), 7.00–7.04 (m, 1H, $H_{aromatic}$), 7.19–7.30 (m, 3H, $H_{aromatic}$), 7.33 (d, 1H, $J = 5.2$ Hz, $H_{aromatic}$), 7.39 (d, 2H, $J = 8$ Hz, $H_{aromatic}$), 7.46(s, 1H, $H_{aromatic}$), 7.67 (d, 1H, $J = 7.6$ Hz, $H_{aromatic}$), 7.80 (t, 2H, $J = 7.6$ Hz, $H_{aromatic}$), 8.00 (d, 1H, $J = 8$ Hz, $H_{aromatic}$),8.94 (s, 1H, NH), 10.27 (s, 1H, OH).



Scheme 2 Suggested mechanism for synthesis of 1-(benzothiazolylamino)methyl-2-naphthols based on *o*-QMs



Scheme 3 Suggested mechanism for synthesis of 1-(benzothiazolylamino)methyl-2-naphthols based on the Mannich-type reaction

1-((benzo[*d*]thiazol-2-ylamino)(2,4-dichlorophenyl)methyl)naphthalen-2-ol (**4d**) Yield: 84 %; m.p. 204–206 °C; ^{1}H-NMR (400 MHz, DMSO-d6): $\delta = 7.01$ (t, $J = 7.2$ Hz, 1H), 7.13–7.21 (m, 3H), 7.27 (t, $J = 7.6$ Hz, 1H), 7.37–7.42 (m, 3H), 7.50 (s,1H),

7.67(t, $J = 8$ Hz, 2H), 7.77–7.82 (m, 2H), 8.02 (d, $J = 8.4$ Hz, 1H), 8.89 (s, NH), 9.98 (broads, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(3-methoxyphenyl)methyl)naphthalen-2-ol (**4e**) Yield: 89 %; m.p. 185–187 °C; IR (KBr, cm^{-1}): ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.65$ (s, 3H, OCH_3), 6.76 (d, 1H, $J = 7.6$ Hz, $H_{aromatic}$), 6.80 (d, 1H, $J = 7.6$ Hz, $H_{aromatic}$), 6.83 (s, 1H, $H_{aromatic}$), 7.01 (t, 1H, $J = 7.2$ Hz, $H_{aromatic}$), 7.16–7.38(m, 7H, $H_{aromatic}$,1$H_{benzylic}$), 7.67 (d, 1H, $J = 7.6$ Hz, $H_{aromatic}$), 7.79 (t, 1H, $J = 7.8$ Hz, $H_{aromatic}$), 7.88 (brs, 1H, $H_{aromatic}$), 8.79 (s, 1H, NH), 10.17 (s, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(2-chlorophenyl)methyl)naphthalen-2-ol (**4g**) Yield: 88 %; m.p. 187–189 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.97$–7.01 (m, 1H, $H_{aromatic}$), 7.15–7.36 (m, 9H, $H_{aromatic}$), 7.64 (d, 2H, $J = 6.8$ Hz, $H_{aromatic}$), 7.76–7.81 (m, 2H, $H_{aromatic}$), 8.05(d, 1H, $J = 7.6$ Hz, $H_{aromatic}$), 8.85 (s, 1H, NH), 9.96 (s, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(2,4-dimethoxyphenyl)methyl)naphthalen-2-ol (**4h**) Yield: 89 %; m.p. 162–164 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.63$ and 3.70(2s, 6H, 2OCH_3), 6.44–7.75 (m, 13H), 8.19 (broads, 1H), 8.55 (s, 1H, NH), 9.92 (s, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(4-methoxyphenyl)methyl)naphthalen-2-ol (**4i**) Yield: 92 %; m.p. 173-175 °C; IR (KBr, cm^{-1}): ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.68$ (s, 3H, OCH_3), 6.83 (d, 1H, $J = 8.4$ Hz, $H_{aromatic}$), 7.00 (t, 1H, $J = 7.2$ Hz, $H_{aromatic}$), 7.15–7.36(m, 8H, $H_{aromatic}$,1$H_{benzylic}$), 7.78 (t, 2H, $J = 8.8$ Hz, $H_{aromatic}$), 7.88 (brs, 1H), 8.77 (s, 1H, NH), 10.14 (s, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(2-nitrophenyl)methyl)naphthalen-2-ol (**4j**) Yield: 58 %; m.p. 212–214 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 7.06$ (t, 2H, $J = 7.6$ Hz, $H_{aromatic}$), 7.22–7.26 (m, 2H, $H_{aromatic}$), 7.39–7.47(m, 3H, $H_{aromatic}$), 7.67–7.91 (m, 8H, $H_{aromatic}$), 8.84 (s, 1H, NH), 9.89 (brs, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(2,6-dichlorophenyl)methyl)naphthalen-2-ol (**4 k**) Yield: 86 %; m.p. 194–196 °C; ^{1}H-NMR (400 MHz, DMSO-d6): $\delta = 6.99$–7.07 (m, 2H, $H_{aromatic}$), 7.17–7.38 (m, 7H, $H_{aromatic}$, $H_{benzylic}$), 7.45 (t, $J = 7.2$ Hz, 1H, $H_{aromatic}$), 7.65 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.76 (d, $J = 8.8$ Hz, 1H, $H_{aromatic}$), 7.82 (d, $J = 8.0$ Hz, 1H, $H_{aromatic}$), 7.96 (d, $J = 8.8$ Hz, 1H, $H_{aromatic}$), 8.78 (d, $J = 6.4$ Hz, 1H, NH), 9.74 (s, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(3-bromophenyl)methyl)naphthalen-2-ol (**4 l**) Yield: 90 %; m.p. 200–202 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.86$ (s, 1H, $H_{aromatic}$), 7.00 (t, $J = 7.2$ Hz, 1H), 7.18–7.28 (m, 5H, $H_{aromatic}$, $H_{benzylic}$), 7.36–7.38 (m, 3H, $H_{aromatic}$), 7.65 (d, $J = 7.6$ Hz, 1H), 7.75–7.80 (m, 2$H_{aromatic}$), 7.99 (s, 1H, $H_{aromatic}$), 8.89 (s, 1H, NH), 10.20 (brs, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(4-bromophenyl)methyl)naphthalen-2-ol (**4m**) Yield: 90 %; m.p. 200–202 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 7.02$ (t, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.17–7.28 (m, 6H, $H_{aromatic}$, $H_{benzylic}$), 7.38 (d,

$J = 8$ Hz, 2H, $H_{aromatic}$), 7.46 (d, $J = 8.4$ Hz, 2H, $H_{aromatic}$), 7.67 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.79–7.82 (m, 2H, $H_{aromatic}$), 8.81 (brs, 1H, NH), 10.22 (brs, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(5-bromo-2-hydroxyphenyl)methyl)naphthalen-2-ol (**4o**) Yield: 90 %; m.p. 181–183 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.69$ (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 6.98 (t, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.13–7.28 (m, 6H, $H_{aromatic}$, $H_{benzylic}$), 7.34 (d, $J = 8$ Hz, 1H, $H_{aromatic}$), 7.43 (t, $J = 7.2$ Hz, 1H, $H_{aromatic}$), 7.62–7.64 (m, 2H, $H_{aromatic}$), 7.72 (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 7.77 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 8.25 (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 8.70 (s, 1H, NH), 9.62–10.28 (br, 2H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(thiophen-2-yl)methyl)naphthalen-2-ol (**4p**) Yield: 90 %; m.p. 192–194 °C; ^{1}H-NMR (400 MHz, DMSO-d6): $\delta = 7.02$ (t, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.19–7.39 (m, 10H, $H_{aromatic}$, $H_{benzylic}$), 7.68 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.79–7.82 (m, 2H, $H_{aromatic}$), 8.82 (s, 1H, NH), 10.21 (br, 1H, OH).

1-((benzo[*d*]thiazol-2-ylamino)(2,5-dimethoxyphenyl)methyl)naphthalen-2-ol (**4s**) Yield: 90 %; m.p. 209–211 °C; IR (KBr, cm^{-1}): 3,368 (N–H), 3,060, 1,628; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.50$ and 3.64 (2s, 2OCH_3, 6H), 6.76 (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 6.84 (d, $J = 8.8$ Hz, 1H, $H_{aromatic}$), 6.98 (d, $J = 7.2$ Hz, 1H, $H_{aromatic}$), 7.15–7.45 (m, $H_{aromatic}$, 7H, 1$H_{benzylic}$), 7.63 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.71 (d, $J = 8.8$ Hz, 3H, $H_{aromatic}$), 7.77 (d, $J = 8$ Hz, 1H, $H_{aromatic}$), 8.26 (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 8.61 (brs, 1H, NH), 9.92 (s, 1H, OH); ^{13}C NMR (400 MHz, DMSO-d_6): $\delta = 50.56$, 55.69, 56.44, 111.64, 112.41, 116.22, 118.46, 118.72, 119.06, 121.22, 121.26, 122.66, 123.89, 125.82, 126.39, 128.70, 128.80, 129.51, 131.07, 131.70, 133.06, 151.37, 152.74, 153.24, 153.81, 166.14.

1-((benzo[*d*]thiazol-2-ylamino)(2-hydroxy-3-methoxyphenyl)methyl)naphthalen-2-ol (**4t**) Yield: (92 %); m.p. 200–202 °C; IR (KBr, cm^{-1}): 3,366, 3,141, 1,632; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.74$ (S, 3H, OCH_3), 6.67 (t, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 6.82 (d, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 6.96 (t, $J = 7.6$ Hz, 1H, $H_{aromatic}$), 7.01 (d, $J = 7.2$ Hz, 1H, $H_{aromatic}$), 7.14–7.40 (m, 6H, $H_{aromatic}$, $H_{benzylic}$), 7.61 (d, $J = 7.2$ Hz, 1H, $H_{aromatic}$), 7.71 (d, $J = 8$ Hz, 1H, $H_{aromatic}$), 7.76 (d, $J = 8$ Hz, 1H, $H_{aromatic}$), 8.18 (d, $J = 8.4$ Hz, 1H, $H_{aromatic}$), 8.64 and 8.79 (2brs, 2H, NH and OH), 9.95 (brs, 1H, OH); ^{13}C NMR (400 MHz, DMSO-d_6): $\delta = 50.90$, 56.12, 56.27, 110.81, 118.29, 118.53, 118.86, 119.16, 121.13, 121.24, 122.66, 123.43, 125.81, 126.42, 128.82, 129.30, 130.96, 132.17, 133.22, 144.29, 147.79, 151.68, 152.69, 153.66, 166.27.

Conclusions

In conclusion, we have developed a new method for preparation of 1-(benzothiazolylamino)methyl-2-naphthols by one-pot, three-component reaction of 2-naphthol with aromatic aldehydes and 2-aminobenzothiazole. Maltose is used as neutral organic catalyst, at 80 °C, under solvent-free conditions. The catalyst is environmentally benign, inexpensive, clean, safe, nontoxic, and readily available. This method has several other advantages including mild reaction conditions, high

yields, and simplicity; it is also clean, and performed under neutral reaction conditions. These advantages make it a useful and attractive process for synthesis of a wide variety of biologically active compounds.

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