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ORIGINAL ARTICLE

Synthesis of enantiopure aminonaphthol derivatives under conventional/ultrasonic technique and their ring-closure reaction

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KEYWORDS

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Abstract New optically active aminoalkylnaphthols were obtained by condensation of 2-naphthol, substituted aromatic and heteroaromatic aldehydes and (*R*)-(+)-1-phenylethylamine or (*S*)-(−)-1-phenylethylamine under conventional methods and ultrasonic irradiation. The enantiopure aminoalkylnaphthol derivatives were converted in ring-closure reaction with formaldehyde to the corresponding naphthoxazine derivatives.

1. Introduction

The Mannich reaction is one of the most important carbon–carbon bond forming reactions in organic synthesis (Kitamura et al., 1995, 2004; Rijnberg et al., 1997; Kobayashi and Ishitani, 1999). The aminoalkylation of the electron rich aromatic substrates by the Mannich Reaction affords synthetically and biologically important compounds which are useful intermediates for the formation of various nitrogen-containing natural products and pharmaceuticals (Tramontini and Angiolini, 1990, 1994; Müller et al., 1999; Chi et al., 1999; Pu and Yu, 2001; Turgut et al., 2007; Szatmari and Fülöp, 2013). Betti has prepared the aminoalkylnaphthols for the first time at the beginning of the 20th century (Betti 1941). The development of novel stereoselective aminoalkylation of electron-rich aromatic compounds ais recently of major interest. Particularly new direct approaches that are stereoselective and mild enough to permit the preparation of single diastereomers are continuously attracting interest (Palmieri, 1999; Cimarelli et al., 2001, 2002; Cimarelli and Palmieri, 2009; Cardellicchio et al., 1999, 2010; Dong et al., 2004; Saidi et al., 2001, 2003; Wei et al., 2011). Palmieri et al. and Wang et al. developed a practical method for the stereoselective synthesis of aminoalkylnaphthols from 2-naphthol, aromatic aldehydes and commercially available (*R*)-1-phenylethylamine. These compounds exhibited fairly good enantioselectivities in the asymmetric addition of diethylzinc to aromatic aldehydes (Cimarelli et al., 2001; Liu et al., 2001).

Sonochemistry is the application of ultrasound to chemical reactions and processes. Ultrasound irradiation, due to the cavitational collapse, is able to activate many organic reactions. In recent years, ultrasound irradiation has gained increased attention as a clean and useful technique in organic synthesis (Mason and Peters, 2003; Doble and Kumar, 2007; Li et al., 2002). A broad range of the organic synthesis can

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be performed in higher yields, higher selectivities, shorter reaction times and milder reaction conditions under ultrasonic irradiation (Zeng et al., 2009; Chen and Li, 2009; Li et al., 2011, 2005; Luche, 1998; Javanshir et al., 2011).

The development of new 1,3-oxazine derivatives has attracted considerable attention due to their biological properties such as analgesic, anticonvulsant, antitubercular, antibacterial, antifungal and anticancer activities (Kurz, 2005; Zhao et al., 2007; Tabuchi et al., 2009; Wang et al., 2008). Also in recent years, these compounds are used in the treatment of AIDS and Parkinson's disease (Joyce et al., 2003; Kerdesky, 2005). Furthermore 1,3-oxazines are useful synthetic intermediates in organic synthesis.

Herein, we report on the preparation of aminoalkylnaphthols under conventional conditions and ultrasonic conditions and the ring-closure reaction of these compounds.

2. Material and methods

2.1. General methods

^{1}H and ^{13}C NMR spectra were recorded on Varian-INOVA and Mercury-VX spectrometers at 500 or 400 MHz and 125 or 100 MHz, respectively. Chemical shifts are given in ppm downfield from Me_4Si in $CDCl_3$ solution. Coupling constants are given in Hz. The FTIR spectra were recorded on a Perkin-Elmer FT-IR spectrometer (KBr). MS spectra were recorded on Thermo Elemental X Series ICP-MS or VG 2AB-HFQ double sector MS. The X-ray analysis was recorded on Rigaku *R*-Axis Rapid-S X-ray Single Cristal Diffractometer. Optical rotations were measured with Bellingham Stanley ADP-410 Polarimeter. Ultrasonication was performed in Intersonik cleaner with a frequency of 60 kHz and a power of 300 W. The reaction flasks were suspended at the centre of the bath, and the addition or removal of water controlled the temperature of the water bath. Melting points were measured on a Gallenkamp melting-point apparatus. Silica gel 60 (Merck) was used for column separations. TLC was conducted on standard conversion aluminium sheets pre-coated with a 0.2-mm layer of silica gel. Elemental analyses were measured with Flash EA 1112 Series apparatus and were in good agreement (±0.2%) with the calculated values. All reagents were commercially available. THF was distilled and stored on sodium wire before use. Commercial (*R*)-(+)-1-phenylethylamine and (*S*)-(−)-1-phenylethylamine were used.

2.1.1. General procedure for the synthesis of aminoalkylnapthols (**1a–h** and **2a–e**)

A mixture of 2-naphthol (5.00 mmol), aromatic aldehyde (6.00 mmol) and (*R*)-(+)-1-phenylethylamine or (*S*)-(−)-1-phenylethylamine (5.25 mmol) was stirred at 60 °C for 8–30 h under nitrogen atmosphere. Aminoalkylnaphthols (**1a–h** and **2a–e**) were purified by column chromatography (hexane/EtOAc) directly from the reaction mixture, without any work-up.

2.1.2. General procedure for the synthesis of aminoalkylnapthols under ultrasonic irradiation (**1a–h** and **2a–e**)

For the ultrasound-assisted method a mixture of 2-naphthol (5.00 mmol), aromatic aldehyde (6.00 mmol) and (*R*)-(+)-1-phenylethylamine or (*S*)-(−)-1-phenylethylamine (5.25 mmol) was sonicated at 60 °C under nitrogen atmosphere at the centre of an ultrasound cleaner bath for the period of time as indicated in Tables 1 and 2. Aminoalkylnaphthols (**1a–h** and **2a–e**) were purified by column chromatography (hexane/EtOAc) directly from the reaction mixture, without any work-up.

2.1.3. 1-((*R*)-(4-bromophenyl)((*R*)-1-phenylethylamino)methyl)naphthalen-2-ol **1a**

White crystals, mp 131–133 °C. $[\alpha]_D^{20} = -185.6$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3314, 3028, 2924, 2852, 1645, 1620, 1600, 1586, 1556, 1483, 1273, 1237, 1167, 1094, 1065, 830, 763, 701. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.40 (d, J = 6.74 Hz, 3H), 2.10 (br s, 1H), 3.78 (q, J = 6.74 Hz, 1H), 5.31 (s, 1H), 6.96 (br s, 1H), 7.06–7.31 (m, 13H), 13.43 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 21.88, 55.62, 58.62, 111.59, 119.06, 119.80, 120.90, 121.50, 125.49, 125.60, 126.97, 127.83, 127.99, 128.42, 128.94, 131.15, 131.41, 139.50, 141.93, 156.21. MS m/z (ESI) 432 (M^+), 311, 122. Anal. Calc. for $C_{25}H_{22}BrNO$ (432.35): C 69.45, H 5.13, N 3.24. Found: C 69.58, H 5.38, N 3.15%.

2.1.4. 1-((*R*)-(2,4-dimethylphenyl)((*R*)-1-phenylethylamino)methyl)naphthalen-2-ol **1b**

White crystals, mp 162–165 °C. $[\alpha]_D^{20} = -262.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3445, 3313, 3060, 3027, 2970, 2896, 1619, 1582, 1517, 1497, 1468, 1272, 1238, 1154, 1092, 1076, 1035, 831, 815, 765, 702. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.55(d, J = 6.80 Hz, 3H), 1.59 (br s, 1H), 1.93 (s, 3H), 2.21 (s, 3H), 3.91 (q, J = 6.80 Hz, 1H), 5.66 (s, 1H), 6.82 (br s, 1H), 6.92 (s, 1H), 7.15–7.39 (m, 10H), 7.74–7.77 (m, 2H), 13.79 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 18.30, 21.10, 21.86, 56.86, 56.94, 114.24, 120.29, 121.13, 122.62, 126.74, 127.43, 127.46, 127.7, 128.29, 129.02, 129.05, 129.11, 129.80, 131.79, 132.86, 134.87, 136.25, 137.88, 142.93, 158.02. MS m/z (ESI) 381 (M^+), 274, 261, 143. Anal. Calc. for $C_{27}H_{27}NO$ (381.51): C 85.00, H 7.13, N 3.67. Found: C 84.65, H 7.11, N 3.97%.

2.1.5. 1-((*S*)-(2,4-dichlorophenyl)((*R*)-1-phenylethylamino)methyl)naphthalen-2-ol **1c**

White crystals, mp 150–152 °C. $[\alpha]_D^{20} = -259.7$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3412, 3063, 3027, 2971, 2926, 1620, 1598, 1520, 1466, 1271, 1234, 1049, 815, 745, 699. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.51 (d, J = 6.83 Hz, 3H), 2.11 (br s, 1H), 3.93 (q, J = 6.83 Hz, 1H), 5.85 (s, 1H), 7.02 (br s, 1H), 7.16–7.32 (m, 11H), 7.73–7.78 (m, 2H), 13.44 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 20.66, 55.65, 56.19, 111.61, 118.96, 119.80, 121.76, 125.84, 126.29, 126.96, 127.04, 127.63, 127.83, 128.45, 129.24, 130.62, 131.33, 132.72, 133.57, 135.75, 141.04, 156.81. MS m/z (ESI) 422 (M^+), 353, 300, 283, 266, 121. Anal. Calcd. for $C_{25}H_{21}Cl_2NO$ (422.35): C 71.10, H 5.01, N 3.32. Found: C 71.25, H 5.03, N 3.44%.

2.1.6. 1-((*S*)-(2,4-difluorophenyl)((*R*)-1-phenylethylamino)methyl)naphthalen-2-ol **1d**

White crystals, mp 178–180 °C. $[\alpha]_D^{20} = -212.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3354, 3017, 3002, 2968, 2864, 1621, 1600, 1555, 1519, 1466, 1270, 1220, 1159, 1142, 1100, 818, 745, 702. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.53 (d, J = 6.80 Hz, 3H), 2.30 (br s, 1H), 3.94 (q, J = 6.80 Hz, 1H),

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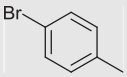
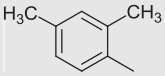
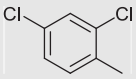
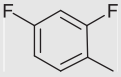
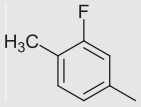
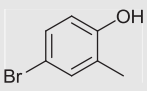
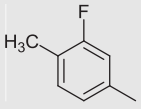
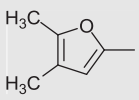
Table 1 Synthesis of aminoalkylnaphthols and naphtoxazines in the presence of (*R*)-(+)-1-phenylethylamine.

Compound	Ar	Time, (h)		Yield, (%)[a]		dr[b]		Compound	Time, (h)	Yield, (%)[a]
		(A)	(B)	(A)	(B)	(A)	(B)			
1a		8	3	74	78	60/40	55/45	**3a**	15	84
1b		12	4	67	70	66/34	62/38	**3b**	18	87
1c		12	4	66	70	74/26	70/30	**3c**	18	92
1d		15	4	69	71	68/32	64/36	**3d**	24	83
1e		18	5	62	65	65/35	60/40			
1f		30	5	42	40	59/41	53/47			
1g		18	5	41	43	72/28	70/30			
1h		18	5	51	58	77/23	73/27			

(A) stirring without ultrasound; and (B) under ultrasound irradiation.
[a] Yields of the pure isolated major diastereomer.
[b] The dr values were determined by ^{1}H NMR of the reaction mixture.

5.82 (s, 1H), 6.79 (br s, 1H), 7.12–7.46 (m, 10H), 7.68–7.81 (m, 2H), 13.51 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 22.67, 52.60, 56.70, 103.80, 111.90, 119.90, 120.70, 122.70, 123.80, 123.90, 126.70, 126.80, 127.90, 128.70, 130.19, 131.26, 132.32, 142.37, 157.76, 158.90, 161.30, 163.80. MS *m*/*z* (ESI) 389 (M^+), 269, 249, 122. Anal. Calc. for $C_{25}H_{21}F_2NO$ (389.44): C 77.10, H 5.44, N 3.60. Found: C 76.90, H 5.50, N 3.80%.

2.1.7. 1-((R)-(3-fluoro-4-methylphenyl)((i)-1-phenylethylamino)methyl) naphthalen-2-ol ***1e***

White crystals, mp 111–113 °C. $[\alpha]_D^{20} = -180.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3315, 3279, 3058, 3025, 2963, 1621, 1600, 1582, 1467, 1413, 1269, 1237, 1158, 1115, 1077, 815, 756, 699. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.51 (d, J = 6.80 Hz, 3H), 2.17 (s, 3H), 2.24 (br s, 1H), 3.88 (q, J = 6.80 Hz, 1H), 5.42 (s, 1H), 6.84–6.87 (m, 2H), 7.02–7.05 (m, 1H), 7.17–7.26 (m, 5H), 7.33–7.42 (m, 4H), 7.73–7.75 (m, 2H), 13.56 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 13.15, 21.91, 55.62, 58.62, 111.78, 113.18, 119.09, 119.92, 121.43, 121.91, 123.62, 125.44, 125.63, 126.93, 127.80, 127.98, 128.85, 130.97, 131.52, 140.25, 142.01, 156.24, 159.47, 161.42. MS *m*/*z* (ESI) 385 (M^+), 265, 247. Anal. Calc. for $C_{26}H_{24}FNO$ (385.47): C 81.01, H 6.28, N 3.63. Found: C 80.90, H 6.40, N 3.40%.

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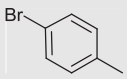
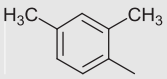
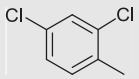
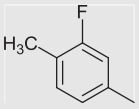
Table 2 Synthesis of aminoalkylnaphthols and naphtoxazines in the presence of (*S*)-(−)-1-phenylethylamine.

Compound	Ar	Time, (h)		Yield, (%)[a]		dr[b]		Compound	Time, (h)	Yield, (%)[a]
		(A)	(B)	(A)	(B)	(A)	(B)			
2a		8	3	73	77	58/42	53/47	**4a**	16	86
2b		14	5	65	68	63/37	60/40	**4b**	18	90
2c		14	5	68	72	72/28	67/33	**4c**	18	90
2d		16	5	72	74	63/37	59/41			
2e		18	5	45	42	75/25	70/30			

(A) stirring without ultrasound; and (B) under ultrasound irradiation.
[a] Yields of the pure isolated major diastereomer.
[b] The dr values were determined by ^{1}H NMR of the reaction mixture.

2.1.8. 1-((R)-(5-bromo-2-hydroxyphenyl)((R)-1-phenylethylamino)methyl)naphthalen-2-ol ***1f***

Yellow crystals, mp 129–130 °C. $[\alpha]_D^{20} = -143.4$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3472, 3414, 3083, 3023, 2983, 2937, 1630, 1603, 1570, 1473, 1367, 1275, 1181, 1088, 820, 768, 700. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.30 (br s, 1H), 1.62 (d, J = 6.35 Hz, 3H), 4.56 (q, J = 6.35 Hz, 1H), 5.20 (s, 1H), 6.85 (br s, 1H), 7.25–7.37 (m, 5H), 8.30 (br s, 1H), 13.54 (br s, 1H).^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 23.64, 57.43, 67.36, 109.02, 118.02, 119.24, 125.42, 126.45, 127.79, 132.51, 133.92, 142.31, 159.23, 161.20. MS m/z (ESI) 448 (M^+), 365, 327, 303, 255, 169. Anal. Calc. for $C_{25}H_{22}BrNO_2$ (448.35): C 66.97, H 4.95, N 3.12. Found: C 65.85, H 5.05, N 3.32%.

2.1.9. 1-((S)-(3-methylthiophen-2-yl)((R)-1-phenylethylamino)methyl)naphthalen-2-ol ***1g***

White crystals, mp 153–155 °C. $[\alpha]_D^{20} = -274.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3449, 3297, 3083, 3029, 2970, 2930, 1621, 1599, 1589, 1469, 1237, 1174, 1081, 1056, 823, 751, 703. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.55 (d, J = 6.84 Hz, 3H), 1.89 (s, 3H), 2.17 (br s, 1H), 3.87 (q, J = 6.84 Hz, 1H), 5.75 (s, 1H), 6.70 (d, J = 5.09 Hz, 1H), 7.00(d, J = 5.09 Hz, 1H), 7.10–7.45 (m, 9H), 7.70–7.80 (m, 2H), 13.60 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 13.23, 22.26, 52.89, 56.11, 114.12, 120.22, 120.64, 122.50, 124.43, 126.59, 126.96, 128.10, 128.62, 128.92, 129.63, 129.99, 132.43, 133.50, 139.06, 142.81, 156.97. MS m/z (ESI) 373 (M^+), 359, 343, 239. Anal. Calc. for $C_{25}H_{23}NOS$ (373.51): C 77.18, H 6.21, N 3.75. Found: C 76.85, H 6.45, N 3.95%.

2.1.10. 1-((S)-(4,5-dimethylfuran-2-yl)((R)-1-phenylethylamino)methyl)naphthalen-2-ol ***1h***

White crystals, mp 145–147 °C. $[\alpha]_D^{20} = -128.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3355, 3017, 3002, 2969, 2918, 1621, 1600, 1519, 1468, 1270, 1238, 1155, 1102, 1082, 1046, 834, 736, 703. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.45(d, J = 6.80 Hz, 3H), 1.90 (s, 3H), 2.20 (s, 3H), 2.50 (br s, 1H), 3.87 (q, J = 6.80 Hz, 1H), 5.50 (s, 1H), 7.00–7.80 (m, 12H), 13.10 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 9.70, 11.45, 23.31, 53.29, 55.61, 111.16, 111.61, 114.89, 119.95, 121.09, 122.41, 126.44, 126.66, 127.70, 128.46, 128.65, 128.89, 129.85, 132.77, 142.87, 147.21, 150.06, 157.39. MS m/z (ESI) 371 (M^+), 355, 266, 251. Anal. Calc. for $C_{25}H_{25}NO_2$ (371.47): C 80.83, H 6.78, N 3.77. Found: C 80.50, H 6.58, N 3.90%.

2.1.11. 1-((S)-(4-bromophenyl)((S)-1-phenylethylamino)methyl)naphthalen-2-ol ***2a***

White crystals. mp 124–126 °C. $[\alpha]_D^{20} = +100.8$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3446, 3313, 3065, 3018, 2968, 1620, 1599, 1464, 1271, 1234, 1156, 1095, 846, 829, 752. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.50 (d, J = 6.60 Hz, 3H), 2.20 (br s, 1H), 3.90 (q, J = 6.60 Hz, 1H), 5.41 (s, 1H), 7.01–7.81 (m,

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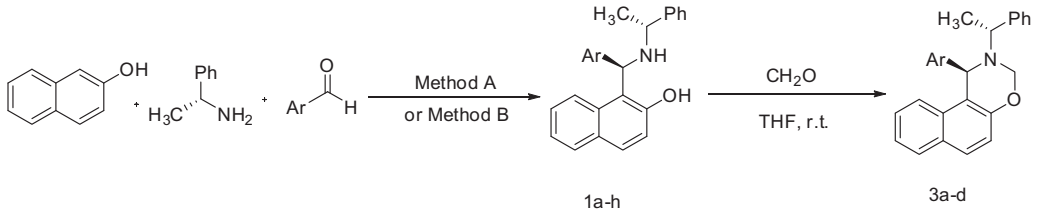
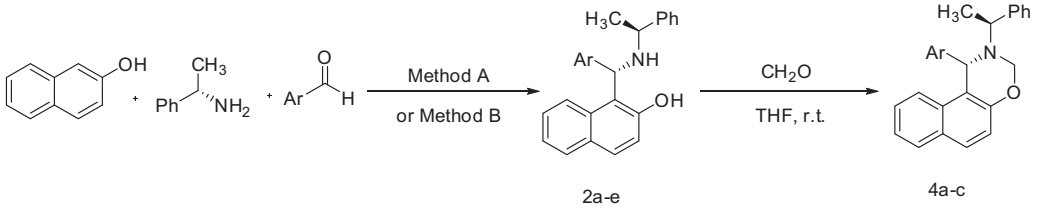


Method A: stirring without ultrasound at 60 °C under N_2 atmosphere
Method B: ultrasound irradiation at 60 °C under N_2 atmosphere

Scheme 1 Synthesis of enantiopure aminoalkylnaphthols (**1a**–**h**) and naphthoxazines (**3a**–**d**).



Method A: stirring without ultrasound at 60 °C under N_2 atmosphere
Method B: ultrasound irradiation at 60 °C under N_2 atmosphere

Scheme 2 Synthesis of enantiopure aminoalkylnaphthols (**2a**–**e**) and naphtoxazines (**4a**–**c**).

15H), 13.51 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 22.95, 56.62, 59.62, 112.57, 120.08, 120.83, 121.94, 122.54, 126.53, 126.63, 128.02, 128.80, 129.03, 129.46, 129.98, 132.19, 132.40, 140.48, 142.92, 157.21. MS *m*/*z* (ESI) 432 (M^+), 311, 232, 122. Anal. Calc. for $C_{25}H_{22}BrNO$ (432.35): C 69.45, H 5.13, N 3.24. Found: C 69.60, H 5.35, N 3.18%.

2.1.12. 1-((S)-(2,4-dimethylphenyl)((S)-1-phenylethylamino)methyl)naphthalen-2-ol **2b**

White crystals, mp 174–177 °C. $[\alpha]_D^{20} = +257.8$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3314, 3053, 3022, 2971, 1620, 1582, 1517, 1454, 1272, 1238, 1154, 1092, 1075, 831, 764, 702. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.52 (d, J = 6.80 Hz, 3H), 1.60 (br s, 1H), 1.90 (s, 3H), 2.25 (s, 3H), 3.90 (q, J = 6.80 Hz, 1H), 5.65 (s, 1H), 6.78–6.94 (m, 2H), 7.12–7.44 (m, 10H), 7.72–7.82 (m, 2H), 13.85 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 18.07, 20.89, 21.62, 56.57, 56.66, 113.98, 120.04, 120.87, 122.40, 126.52, 127.20, 127.53, 128.07, 128.76, 128.82, 128.86, 129.57, 131.56, 132.60, 132.63, 134.64, 135.97, 137.66, 142.65, 157.76. MS *m*/*z* (ESI) 381 (M^+), 216. Anal. Calc. for $C_{27}H_{27}NO$ (381.51): C 85.00, H 7.13, N 3.67. Found: C 84.75, H 7.15, N 3.94%.

2.1.13. 1-((R)-(2,4-dichlorophenyl)((S)-1-phenylethylamino)methyl)naphthalen-2-ol **2c**

White crystals, mp 145–147 °C. $[\alpha]_D^{20} = +288.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3311, 3058, 3022, 2980, 1629, 1580, 1469, 1 1273, 1181, 1089, 842, 767, 703. ^{1}H NMR δ_H ($CDCl_3$, 400 MHz) 1.52 (d, J = 6.80 Hz, 3H), 2.13 (br s, 1H), 3.95 (q, J = 6.80 Hz, 1H), 5.86 (s, 1H), 7.01–7.35 (m, 12H), 7.75–7.80 (m, 2H), 13.53 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 100 MHz) 21.93, 56.76, 57.33, 112.80, 120.20, 121.04, 123.04, 127.13, 127.57, 128.30, 128.89, 129.00, 129.10, 129.68, 130.51, 131.86, 132.52, 133.95, 134.79, 136.88, 142.18, 158.01. MS *m*/*z* (ESI) 422 (M^+), 300, 266, 122. Anal. Calc. for $C_{25}H_{21}Cl_2NO$ (422.35): C 71.10, H 5.01, N 3.32. Found: C 71.27, H 5.05, N 3.40%.

Table 3 The reaction of 2-naphthol, 4-bromobenzaldehyde and (*R*)-(+)-1-phenylethylamine[a]: effects of solvents (3 ml were used).

Entry	Solvent	Time, (h)	Temperature (°C)	Yield, (%)[b]
1	$PhCH_3$	3	25	–
2	$PhCH_3$	3	60	Trace
3	CH_2Cl_2	3	25	Trace
4	CH_2Cl_2	3	60	10
5	CH_3CN	3	25	Trace
6	CH_3CN	3	60	23
7	C_2H_5OH	3	25	17
8	C_2H_5OH	3	60	50
9	None	3	25	21
10	None	3	60	78

[a] Reactions were carried out under ultrasonic irradiation.
[b] Yields of the pure isolated major diastereomer.

2.1.14. 1-((S)-(3-fluoro-4-methylphenyl)((S)-1-phenylethylamino)methyl)naphthalen-2-ol **2d**

White crystals, mp 117–119 °C. $[\alpha]_D^{20} = +160.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3436, 3271, 3062, 2965, 1623, 1580, 1470, 1414, 1267, 1162, 1131, 1077, 817, 746, 702. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.41 (d, J = 6.80 Hz, 3H), 2.06 (s, 3H), 2.15 (br s, 1H), 3.78 (q, J = 6.80 Hz, 1H), 5.32 (s, 1H), 6.74–6.77 (m, 2H), 6.92–7.16 (m, 6H), 7.23–7.32 (m, 4H),

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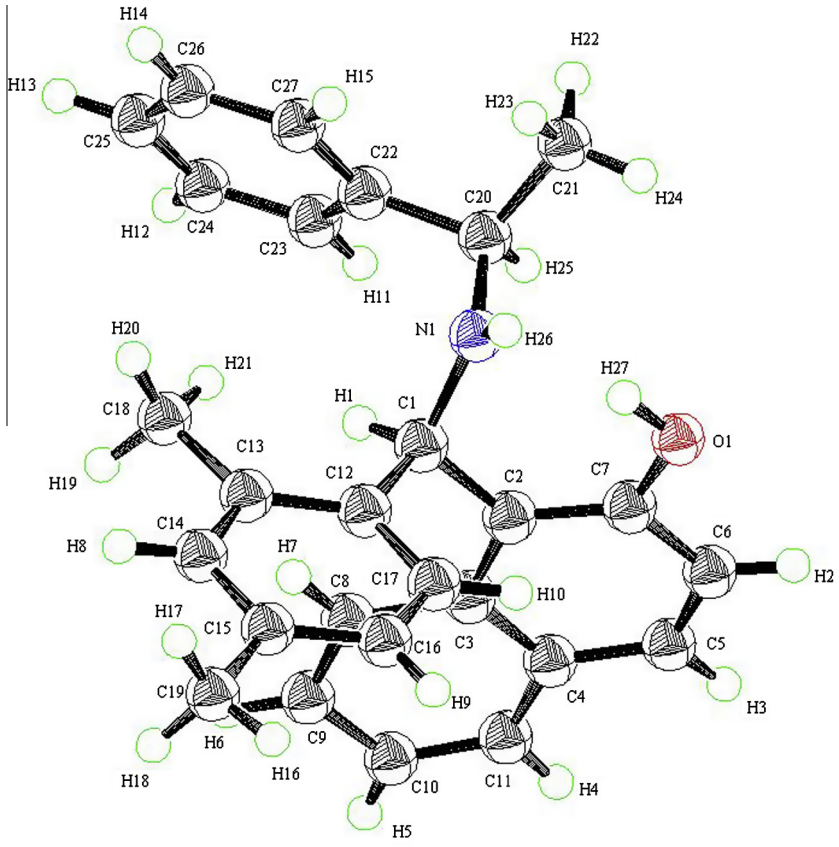

Figure 1 Molecular structure of compound **1b**.

7.61–7.65 (m, 2H), 13.46 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 13.14, 21.90, 55.61, 58.62, 111.77, 113.17, 119.08, 119.91, 121.90, 123.60, 125.44, 125.62, 126.93, 127.72, 127.79, 127.98, 128.85, 130.96, 131.51, 140.25, 141.99, 156.24, 159.46, 161.41. MS *m/z* (ESI) 385 (M^+), 280, 265, 247, 122. Anal. Calc. for $C_{26}H_{24}FNO$ (385.47): C 81.01, H 6.28, N 3.63. Found: C 80.92, H 6.43, N 3.45%.

2.1.15. 1-((R)-((S)-1-phenylethylamino)(thiophen-2-yl)methyl)naphthalen-2-ol ***2e***

White crystals, mp 132–135 °C. $[\alpha]_D^{20} = +140.0$ (c 0.1, $CHCl_3$). FTIR ν_{max} (KBr)/cm^{-1} 3306, 3298, 3055, 3007, 2972, 1620, 1589, 1468, 1237, 1174, 1131, 1081, 823, 746, 701. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.52 (d, J = 6.63 Hz, 3H), 2.45 (br s, 1H), 3.88 (q, J = 6.63 Hz, 1H), 5.73 (s, 1H), 6.52–7.73 (m, 13H), 8.50 (br s, 1H), 13.35 (br s, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 23.41, 54.94, 56.41, 113.85, 120.35, 121.10, 122.78, 125.35, 125.68, 126.83, 127.21, 128.19, 128.87, 129.07, 129.28, 130.26, 132.58, 143.00, 145.13, 157.00. MS *m/z* (ESI) 359 (M^+), 343, 239. Anal. Calc. for $C_{23}H_{21}NOS$ (359.48): C 76.85, H 5.89, N 3.90. Found: C 76.75, H 5.76, N 4.10%.

*2.1.16. General procedure for the preparation of the naphtoxazines (**3a–d** and **4a–c**)*

Aminonaphthols **1a–d** and **2a–c** (2 mmol) were dissolved in THF (3 mL) and 35% aqueous formaldehyde (2.2 mmol) was added. The solution was stirred for 15 h at room temperature. Solvent was removed and the residue was dried under reduced pressure. The crude oil was purified by column chromatography eluting with EtOAc/hexane.

2.1.17. (R)-1-(4-bromophenyl)-2-((R)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***3a***

White crystals, mp 107–109 °C. FTIR ν_{max} (KBr)/cm^{-1} 3061, 3025, 2923, 2852, 1622, 1599, 1484, 1231, 1157, 1147, 10.71, 1010, 812, 745, 699. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.43 (d, J = 6.50 Hz, 3H), 3.86 (q, J = 6.50 Hz, 1H), 4.75 (d, J = 9.5 Hz, 1H), 5.02 (d, J = 9.5 Hz, 2H), 5.06 (s, 1H) 6.79–6.90 (m, 2H), 7.03–7.26 (m, 10H), 7.62–7.65 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 20.44, 55.50, 58.05, 73.25, 110.49, 117.52, 120.20, 121.26, 122.21, 125.59, 126.56, 126.70, 127.54, 127.63, 127.94, 128.11, 129.80, 130.16, 131.68, 141.15, 144.13, 151.85. MS *m/z* (ESI) 444 (M^+), 339, 133. Anal. Calc. for $C_{26}H_{22}BrNO$ (444.36): C 70.28, H 4.99, N 3.15. Found: C 69.10, H 5.21, N 3.53%.

2.1.18. (R)-1-(2,4-dimethylphenyl)-2-((R)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***3b***

White crystals, mp 121–123 °C. FTIR ν_{max} (KBr)/cm^{-1} 3062, 3026, 2970 2923, 1623, 1514, 1230, 1182, 1142, 1048, 898, 809, 743. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.46 (d, J = 6.36 Hz, 3H), 1.89 (s, 3H), 2.08 (s, 3H), 3.95 (q, J = 6.36 Hz, 1H), 4.88 (dd, J_1 = 10.74 Hz, J_2 = 1.47 Hz, 1H), 4.98 (d, J = 10.74 Hz, 1H), 5.29 (s, 1H), 6.53–6.58 (m, 2H), 6.84–6.95 (m, 2H), 7.03–7.16 (m, 8H), 7.60–7.63 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 19.46, 20.15, 21.18,

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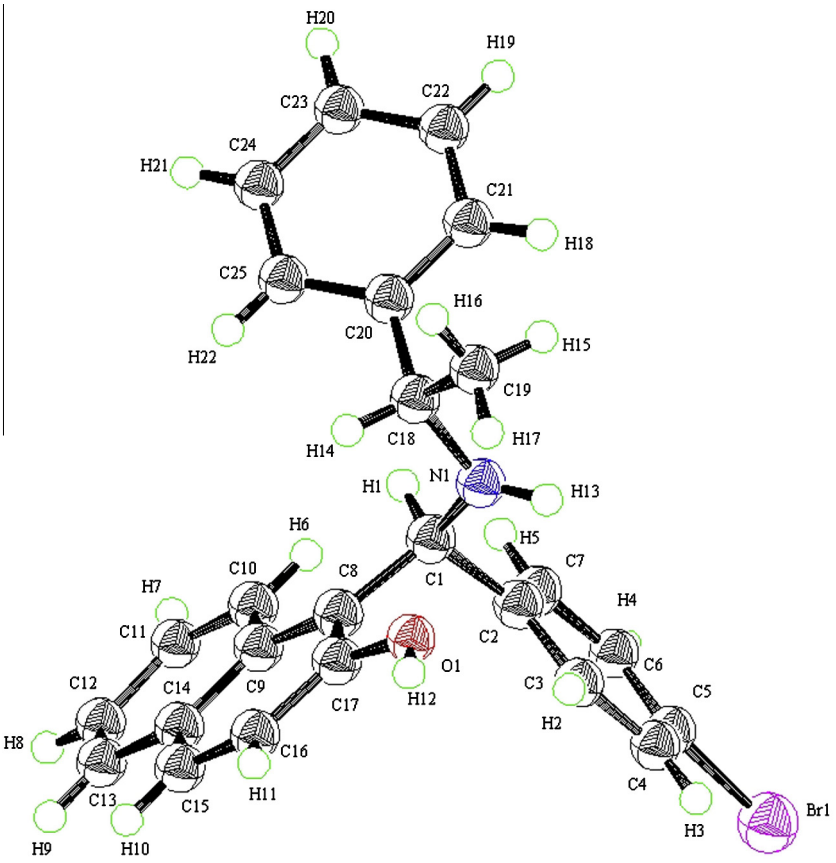

Figure 2 Molecular structure of compound **2a**.

55.23, 59.83, 74.53, 113.73, 118.64, 122.33, 123.35, 126.25, 126.92, 127.90, 128.53, 128.87, 129.03, 129.43, 131.13, 131.97, 132.64, 137.05, 137.16, 137.95, 143.78, 153.11. MS *m*/*z* (ESI) 393 (M^+), 288. Anal. Calc. for $C_{28}H_{27}NO$ (393.52): C 85.46, H 6.92, N 3.56. Found: C 85.30, H 6.80, N 3.75%.

2.1.19. (S)-1-(2,4-dichlorophenyl)-2-((R)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***3c***

White crystals, mp 118–119 °C. FTIR ν_{max} (KBr)/cm^{-1} 3061, 3029, 2970, 1622, 1584, 1467, 1231, 1149, 1140, 1015, 813, 744, 696. ^1H NMR δ_H ($CDCl_3$, 500 MHz) 1.44 (d, J = 6.84 Hz, 3H), 4.32 (q, J = 6.84 Hz, 1H), 4.73 (dd, J_1 = 11.23 Hz, J_2 = 1.4 Hz 1H), 4.76 (dd, J_1 = 11.23 Hz, J_2 = 1.4 Hz 1H), 5.60 (s, 1H), 6.71–6.89 (m, 2H), 6.98–7.05 (m, 2H), 7.16–7.23 (m, 5H), 7.30–7.38 (m, 3H), 7.65–7.70 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 17.56, 54.27, 59.77, 72.85, 111.31, 117.62, 120.94, 122.33, 125.42, 125.87, 126.47, 127.10, 127.60, 128.13, 128.36, 128.98, 130.63, 131.34, 132.85, 133.88, 138.09, 141.55, 152.23. MS *m*/*z* (ESI) 434 (M^+), 288. Anal. Calc. for $C_{26}H_{21}Cl_2NO$ (434.36): C 71.89, H 4.87, N 3.22. Found: C 71.74, H 4.92, N 3.40%.

2.1.20. (S)-1-(2,4-fluorophenyl)-2-((R)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***3d***

White crystals, mp 153–155 °C. FTIR ν_{max} (KBr)/cm^{-1} 3138, 3063, 2985, 1623, 1463, 1234, 1160, 1141, 1098, 1074, 841, 812, 744. ^1H NMR δ_H ($CDCl_3$, 500 MHz) 1.44 (d, J = 6.90 Hz, 3H), 3.98 (q, J = 6.90 Hz, 1H,), 4.86 (d, J = 11.00 Hz, 1H), 4.96 (d, J = 11.00 Hz, 1H), 5.40 (s, 1H), 6.46–6.50 (m, 1H), 6.61–6.73 (m, 2H), 6.93–7.22 (m, 9H), 7.64–7.66 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 19.57, 50.72, 58.71, 73.21, 103.17, 109.37, 110.00, 117.48, 120.81, 122.25, 125.18, 125.26, 125.68, 126.41, 127.12, 127.60, 128.08, 128.23, 130.95, 131.35, 142.50, 152.11, 158.76, 162.40. MS *m*/*z* (ESI) 401 (M^+), 372, 297, 269, 133. Anal. Calc. for $C_{26}H_{21}F_2NO$ (401.45): C 77.79, H 5.27, N 3.49. Found: C 77.63, H 5.25, N 3.62%.

2.1.21. (S)-1-(4-bromophenyl)-2-((S)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***4a***

White crystals, mp 103–105 °C. FTIR ν_{max} (KBr)/cm^{-1} 3061, 3024, 2951, 1622, 1515, 1484, 1231, 1173, 1147, 1089, 1071, 812, 745, 699. ^1H NMR δ_H ($CDCl_3$, 500 MHz) 1.44 (d, J = 6.50 Hz, 3H), 3.87 (q, J = 6.50 Hz, 1H), 4.76 (d, J = 9.85 Hz, 1H), 5.03–5.04 (d, J = 9.85 Hz, 1H), 5.05 (s, 1H), 6.79–6.81 (m, 2H), 6.89–6.91 (m, 1H), 7.04–7.27 (m, 10H), 7.64–7.67 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 20.45, 55.51, 58.06, 73.26, 110.50, 117.53, 120.20, 121.27, 122.21, 125.60, 126.56, 126.71, 127.55, 127.64, 127.97, 128.12, 129.81, 130.17, 131.69, 141.16, 144.14, 151.86. MS *m*/*z* (ESI) 444 (M^+), 339, 311, 133. Anal. Calc. for $C_{26}H_{22}BrNO$ (444.36): C 70.28, H 4.99, N 3.15. Found: C 69.92, H 5.21, N 3.23%.

2.1.22. (S)-1-(2,4-dimethylphenyl)-2-((S)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***4b***

White crystals, mp 132–134 °C. FTIR ν_{max} (KBr)/cm^{-1} 3061, 3017, 2967, 1623, 1514, 1230, 1183, 1141, 1093, 1013, 897,

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809, 743, 698. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.49 (d, J = 6.84 Hz, 3H), 1.91 (s, 3H), 2.12 (s, 3H), 3.97 (q, J = 6.84 Hz, 1H), 4.90 (d, J = 10.73 Hz, 1H), 5.00 (d, J = 10.73 Hz, 1H), 5.31 (s, 1H), 6.55–6.63 (m, 2H), 6.87–7.07 (m, 3H), 7.10–7.28 (m, 8H), 7.37–7.40 (m, 1H), 7.64–7.68 (m, 2H), 8.02–8.03 (m, 1H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 18.10, 18.79, 19.83, 53.90, 58.51, 73.21, 112.40, 117.28, 121.00, 122.00, 124.90, 126.55, 127.18, 127.51, 127.69, 129.17, 129.79, 129.79, 130.63, 131.29, 132.66, 135.72, 135.84, 136.60, 142.44, 151.76. MS m/z (ESI) 393 (M^+), 288. Anal. Calc. for $C_{28}H_{27}NO$ (393.52): C 85.46, H 6.92, N 3.56. Found: C 85.33, H 6.85, N 3.72%.

2.1.23. (R)-1-(2,4-dichlorophenyl)-2-((S)-1-phenylethyl)-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine ***4c***

White crystals, mp 115–116 °C. FTIR ν_{max} (KBr)/cm^{-1} 3061, 3029, 2970, 1622, 1584, 1467, 1231, 1149, 1140, 1046, 1015, 899, 813, 744, 696. ^{1}H NMR δ_H ($CDCl_3$, 500 MHz) 1.39 (d, J = 6.83 Hz, 3H), 4.29 (q, J = 6.83 Hz, 1H), 4.69 (dd, J_1 = 10 Hz, J_2 = 1.50 Hz, 1H), 4.72 (d, J = 10 Hz, 1H), 5.57 (s, 1H), 6.65–6.79 (m, 2H), 6.96–7.01 (m, 2H), 7.11–7.17 (m, 5H), 7.26–7.33 (m, 3H), 7.59–7.63 (m, 2H). ^{13}C NMR δ_C ($CDCl_3$, 125 MHz) 17.53, 54.24, 59.73, 72.79, 111.27, 117.60, 120.89, 122.30, 125.37, 125.84, 126.43, 127.06, 127.52, 128.09, 128.32, 128.92, 130.60, 131.29, 132.80, 133.84, 138.06, 141.51, 152.21. MS m/z (ESI) 434 (M^+), 288. Anal. Calc. for $C_{26}H_{21}Cl_2NO$ (434.36): C 71.89, H 4.87, N 3.22. Found: C 71.77, H 4.95, N 3.37%. (See Schemes 1 and 2.)

3. Results and discussion

Electron-rich aromatic compounds such as 2-naphthol give 1-aminoalkylation with high yields when treated with (*R*)- or (*S*)-1-phenylethylamine and aromatic or hetero-aromatic aldehydes in solvent-free conditions (Cimarelli et al., 2001, 2002; Liu et al., 2001).

Diastereomerically pure aminoalkylnaphthols (**1a**–**h** and **2a**–**e**) were prepared under conventional conditions according to the literature method (Cimarelli et al., 2001) and under ultrasound irradiation. The reactions proceeded smoothly with aromatic or hetero-aromatic aldehydes nevertheless hetero-aromatic aldehydes gave lower yields. The ring-closure reaction of these aminoalkylnaphthols with formaldehyde gave the 1,3-oxazine derivatives **3a**–**d** and **4a**–**c**. The reaction conditions, yields and the structures of the aldehyde components are summarised in Tables 1 and 2.

In order to demonstrate the effects of ultrasonic irradiation in Mannich-type aminoalkylation reactions the synthesis of **1a** was investigated as a typical example at different temperatures and in different solvents (Table 3). It was found that increasing the temperature to 60 °C has considerably improved the yield. We also compared the efficiency of several organic solvents. The reaction worked much better with the polar solvents than the non-polar solvents. However the highest yields were obtained under solvent-free conditions. As shown in Table 1 and Table 2, applying ultrasound irradiation significantly reduced reaction times but did not bring about a noticeable improvement in the stereoselectivities compared to the conventional method. On the other hand, ultrasound irradiation has provided better product yields.

The structure of the newly generated compounds has been clarified by Fourier transform-infrared (FTIR), mass and NMR techniques. Each of the aminoalkylnaphthol compounds proved to be a pure single diastereoisomer by their NMR spectra (a single peak near 5.20–5.90 ppm for their benzyl proton in ^{1}H NMR and one group peak in ^{13}C NMR).

The characteristic absorption bands of OH and NH bands were observed at 3280–3450 cm^{-1} in the FTIR spectra of the aminoalkylnaphthol derivatives. The H-atoms of CH_3 were observed in the ranges of 1.45–1.57 ppm. NH proton signals were observed at 1.59–2.50 ppm; the H-atoms of CH, which are near the CH_3 group absorb in the ranges of 3.80–4.50 ppm. The CH protons which are next to the Ar groups were observed at 5.20–5.90 ppm, and the OH protons absorb in the ranges of 13.10–13.80 ppm. The mass spectra of all new compounds showed the expected molecular ion peak.

^{1}H NMR and X-ray single-crystal studies were carried out to determine the absolute configuration of the newly generated stereogenic centre at **1b** and **2a**. Fig. 1 clearly shows that the configuration of the newly generated centre is (*R*) for **1b** and Fig. 2 show that the configuration of the newly generated centre is (*S*) for **2a**. The X-ray study on these compounds also shows a strong intramolecular hydrogen bond between OH and N atom which gives rigidity to the molecules.

1,3-oxazine derivatives were prepared by the ring-closure reactions of aminoalkylnaphthols **1a**–**d** and **2a**–**c** with formaldehyde in THF at room temperature. The structure of the new 1,3-oxazine compounds has been clarified by Fourier transform-infrared (FTIR), mass and NMR techniques.

The CH_3 protons were observed at 1.40–1.47 ppm; CH protons which are next to CH_3 group were observed at 3.80–4.35 ppm; CH_2 protons which are between N and O atoms absorb in the ranges of 4.70–5.00 ppm. The CH protons which are near the Ar group were observed at 5.30–5.60 ppm.

4. Conclusion

In summary, we synthesised new optically active aminoalkylnaphthol compounds in moderate to good yields by the reaction of 2-naphthol with appropriate aldehydes and (*R*)- or (*S*)-1-phenylethylamine under conventional conditions and ultrasound irradiation. Compared with classical methods, ultrasound irradiation procedure provided shorter reaction times and higher yields. The ring-closure reactions of the aminoalkylnaphthols with formaldehyde gave 1,3-oxazine derivatives.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.arabjc.2014.02.017.

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