## Friedel–Crafts Reactions of 2-Naphthol with α-Amido Sulfones and Conversion of the Products with Nucleophiles<sup>1</sup>

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**Abstract:** 2-Naphthol underwent Friedel–Crafts reaction with *N*-benzyloxycarbonylamino sulfones in the presence of  $InCl_3$  at room temperature to form the corresponding sulfonyl derivatives in high yields. The products were subsequently reacted with nucleophiles such as allyltributyltin and anisole to replace the sulfonyl group.

Key words: 2-naphthol,  $\alpha$ -amido sulfone, InCl<sub>3</sub>, Friedel–Crafts reaction, nucleophile

2-Naphthol derivatives exhibit various important biological properties including hypotensive and bradycardiac effects.<sup>2</sup> Many of these compounds have recently been prepared by us<sup>3</sup> as well as by others.<sup>2,4</sup> In continuation of our work<sup>5</sup> on the development of useful synthetic methodologies applying an  $\alpha$ -amido sulfone we have observed that 2-naphthol can easily undergo Fridel–Crafts reaction with this reagent in the presence of catalytic amount of InCl<sub>3</sub> at room temperature (Scheme 1). The products (sulfone derivatives **3**) were formed in high yields (81–93%).





*N*-Benzyloxycarbonylamino sulfones **2** used in the present conversion can be conveniently prepared<sup>6</sup> from aldehydes and are generally stable. Both the aromatic and aliphatic aldehydes were employed to prepare these sulfone derivatives. The aromatic aldehydes contained electron-donating as well as electron-withdrawing groups. *N*-Benzyloxycarbonylamino sulfones were used<sup>6</sup> earlier by us in the presence of InCl<sub>3</sub> in various synthetic work. In the present conversion, initially we treated 2-naphthol with the sulfone **2a** (R = Ph) using different Lewis acids (Table 1). Considering the reaction time and yield, InCl<sub>3</sub> was found to be most efficient. Thus, subsequently it was used to prepare a series of sulfonyl derivatives **3** from *N*benzyloxycarbonylamino sulfones **2** (Table 2). Different functionalities such as halogen, ether, and nitro groups re-

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Table 1	Evaluation of Catalytic Activity of Different Catalysts for
the Prepar	ation of Sulfone Derivative <b>3a</b> <sup>a</sup>

Entry	Catalyst	Yield (%) <sup>b</sup>	Time (h)	
1	InCl <sub>3</sub>	93	5	
2	$BF_3 \cdot OEt_2$	91	5.5	
3	FeCl <sub>3</sub>	87	8	
4	ZnCl <sub>2</sub>	84	10	
5	$ZrCl_4$	85	9	
6	I <sub>2</sub>	81	12	
7	Cu(OTf) <sub>2</sub>	86	10	
8	Sc(OTf) <sub>3</sub>	83	11	
9	Ba(NO <sub>3</sub> ) <sub>2</sub>	81	16	

<sup>a</sup> Reaction conditions: *N*-benzyloxycarbonylamino sulfone **2a** (R = Ph, 1 mmol), 2-naphthol (**1**; 1 mmol) and catalyst (10 mol%) were stirred at r.t.

<sup>b</sup> Isolated yields after purification.

mained intact. The structures of the products were established from their spectral (IR, <sup>1</sup>H and <sup>13</sup>C NMR, ESI-MS, and HR-ESI-MS) and analytical data.

It is known<sup>7</sup> that  $\alpha$ -amido sulfones are converted into the corresponding protected imines (*N*-acyliminium ions) on treatment with a Lewis acid (Scheme 2). These imine derivatives are highly suitable for nucleophilic addition. 2-Naphthol (1) reacts first with these ions and next the amine moiety of the resulting intermediate is displaced by a sulfonyl group in the presence of the catalyst to form the products **3**.

The sulfone derivatives of type **3** were subsequently treated with the nucleophiles such as allyltributyltin and anisole in the presence of BF<sub>3</sub>·OEt<sub>2</sub> at -10 °C (Scheme 3).

The conversion of **3** with allyltributyltin required one hour while with anisole the reaction time was 45 minutes. The products **4** and **5** were formed by replacement of the sulfonyl group of **3** by the nucleophiles (Table 3) in high yields (81–89%). Thus, the present method has been utilized for the preparation of 4,4-diarylbut-1-ene and triarylmethanes starting from 2-naphthol (**1**). The interesting point is that the conversions of **3** into **4** and **5** did not proceed in the presence of  $InCl_3$ .

Table 2 Synthesis of Sulfone Derivatives 3<sup>a</sup>

Sulfone 2	R	Time (h)	Product <sup>b</sup>	Yield (%)
2a	Ph	5	<b>3</b> a	93
2b	$4-\text{MeC}_6\text{H}_4$	4	3b	91
2c	4- <i>i</i> -PrC <sub>6</sub> H <sub>4</sub>	4.5	3c	92
2d	$3-FC_6H_4$	7	3d	90
2e	$4-ClC_6H_4$	6	3e	89
2f	2-Cl-4-FC <sub>6</sub> H <sub>3</sub>	8.5	3f	83
2g	3,4-(OCH <sub>2</sub> O)C <sub>6</sub> H <sub>3</sub>	6	3g	86
2h	3,4,5-(MeO) <sub>3</sub> C <sub>6</sub> H <sub>2</sub>	8.5	3h	81
2i	$4-O_2NC_6H_4$	9	3i	82
2j	2-naphthyl	6.5	3ј	88
2k	<i>n</i> -Pr	9	3k	85
21	<i>i</i> -Pr	8	31	89

<sup>a</sup> Reaction conditions: *N*-benzyloxycarbonylamino sulfone **2** (1 mmol), 2-naphthol (**1**; 1 mmol), and InCl<sub>3</sub> (10 mol%), r.t.

<sup>b</sup> The structures of the products were established from their spectral (IR,<sup>1</sup>H and <sup>13</sup>C NMR, ESI-MS, and HR-ESI-MS) and analytical data. <sup>c</sup> Isolated yields after purification.



Scheme 2



Scheme 3

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Table 3Synthesis of 4 and 5 from Sulfone Derivatives

Sulfone	R	Time (h)	Product <sup>b</sup>	Yield (%) <sup>c</sup>
3a	Ph	1	$4a^{d}$	89
3a	Ph	0.75	5a <sup>e</sup>	83
3b	4-MeC <sub>6</sub> H <sub>4</sub>	1	$\mathbf{4b}^{d}$	88
3c	4- <i>i</i> -PrC <sub>6</sub> H <sub>4</sub>	0.75	5b <sup>e</sup>	81

<sup>a</sup> Reaction conditions: sulfone derivative **3a–c** (1 mmol), nucleophile (1.5 mmol), and BF<sub>3</sub>×OEt<sub>2</sub> (20 mol%), -10 °C, N<sub>2</sub> atmosphere.
<sup>b</sup> The structures of the products were established from their spectral (IR, <sup>1</sup>H, <sup>13</sup>C NMR and ESI-MS) and analytical data.

<sup>°</sup> Isolated yields after purification.

<sup>d</sup> Derived from allyltributyltin.

<sup>e</sup> Derived from anisole.

Derived from anisole.

In conclusion, we have demonstrated the synthesis of various 2-naphthol derivatives **3** by Friedel–Crafts reaction of 2-naphthol (**1**) with *N*-benzyloxylcarbonylamino sulfones **2** in the presence of InCl<sub>3</sub> and by subsequent treatment of the products with nucleophiles using  $BF_3 \cdot OEt_2$  as a catalyst.

The spectra were recorded with the following instruments; IR: PerkinElmer RX1 FT-IR spectrophotometer; NMR: Varian Gemini 200 MHz (<sup>1</sup>H) and 50 MHz (<sup>13</sup>C) spectrometer; ESI-MS: VG-Autospec micromass and HR-ESI-MS: QSTAR XL, Hybrid MS system. Column chromatography was carried out with silica gel (BDH 100–200 mesh) and TLC with silica gel GF<sub>254</sub> precoated plates.

# Reaction of 2-Naphthol (1) with *N*-Benzyloxycarbonylamino Sulfones 2; General Procedure

InCl<sub>3</sub> (10 mol%) was added to a solution of an  $\alpha$ -amido sulfone 2 (1 mmol) and 2-naphthol (1; 144 mg, 1 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (5 mL) under N<sub>2</sub>. The mixture was stirred at r.t. and the reaction was monitored by TLC. After completion, the reaction mixture was concentrated and H<sub>2</sub>O (10 mL) and EtOAc (10 mL) were added. The organic layer was separated and concentrated. The residue was subjected to column chromatography (silica gel, hexane–EtOAc) to obtain the pure product.

#### 1-[(Phenyl)(p-tosyl)methyl]-2-naphthol (3a)

Yellow oil.

IR (KBr): 3377, 1624, 1598, 1442, 1280 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.74 (1 H, br s), 7.60–7.51 (7 H, m), 7.32–7.11 (6 H, m), 7.01 (2 H, d, *J* = 8.0 Hz), 6.41 (1 H, s), 2.22 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 156.4, 144.8, 135.1, 131.9, 130.6, 129.8, 129.5, 129.2, 129.0, 127.3, 123.5, 121.2, 120.8, 70.2, 21.3.

ESI-MS:  $m/z = 389 [M + H]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>20</sub>O<sub>3</sub>S + Na: 411.1030; found: 411.1013.

### 1-[(p-Tolyl)(p-tosyl)methyl]-2-naphthol (3b)

Yellow oil.

IR (KBr): 3320, 1595, 1512, 1403, 1248 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 8.79$  (1 H, br s), 7.68–7.55 (5 H, m), 7.54 (2 H, d, J = 8.0 Hz), 7.30–7.07 (5 H, m), 7.01 (2 H, d, J = 8.0 MHz), 6.39 (1 H, s), 2.31 (3 H, s), 2.20 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 154.8, 145.1, 136.1, 131.8, 130.0, 129.9, 129.7, 129.5, 129.0, 127.9, 127.1, 123.5, 121.4, 120.2, 70.0, 21.5, 21.1.

ES-IMS:  $m/z = 425 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>27</sub>H<sub>26</sub>O<sub>6</sub>S + Na: 501.1187; found: 425.1169.

#### 1-[(4-Isopropylphenyl)(p-tosyl)methyl]-2-naphthol (3c) Yellow oil.

IR (KBr): 3369, 1624, 1592, 1513, 1281 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.86 (1 H, br s), 7.70–7.51 (7 H, m), 7.29–7.11 (5 H, m), 6.99 (2 H, d, J = 8.0 Hz), 6.36 (1 H, s), 2.84 (1 H, m), 2.20 (3 H, s), 1.20 (6 H, d, J = 7.0 Hz).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 154.4, 149.1, 144.7, 134.8, 133.1, 131.9, 130.0, 129.2, 129.1, 128.7, 127.0, 126.9, 123.1, 121.0, 120.5, 96.3, 69.9, 33.7, 23.5, 21.2.

ESI-MS:  $m/z = 453 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>27</sub>H<sub>26</sub>O<sub>3</sub>S + Na: 453.1500; found: 453.1512.

#### 1-[(3-Fluorophenyl)(p-tosyl)methyl]-2-naphthol (3d) Yellow oil.

IR (KBr): 3375, 1591, 1485, 1441, 1250 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 8.69$  (1 H, br s), 7.69–7.59 (2 H, m), 7.51 (2 H, d, J = 8.0 Hz), 7.41–7.06 (7 H, m), 7.01–6.90 (3 H, m), 6.40 (1 H, s), 2.12 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 164.6, 154.4, 145.4, 136.1, 132.0, 130.0, 129.1, 129.0, 128.7, 127.2, 125.8, 123.2, 116.8 (d, J = 10 Hz), 115.2 (d, J = 8.0 Hz), 69.8, 21.6.

ESI-MS:  $m/z = 429 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>19</sub>ClO<sub>3</sub>S + Na: 429.0936; found: 429.0922.

#### 1-[(4-Chlorophenyl)(p-tosyl)methyl]-2-naphthol (3e) Yellow oil.

IR (KBr): 3378, 1624, 1590, 1490, 1261 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 8.72$  (1 H, br s), 7.74–7.47 (7 H, m), 7.39–7.08 (5 H, m), 6.99 (2 H, d, J = 8.0 Hz), 6.35 (1 H, br s), 2.22 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 154.8, 144.9, 133.0, 131.8, 131.2, 129.1, 128.9, 128.2, 127.0, 123.2, 121.0, 120.2, 69.1, 21.2.

ESI-MS:  $m/z = 445, 447 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>19</sub>ClO<sub>3</sub>S + Na: 445.0641; 445.0637.

### 1-[(2-Chloro-4-fluorophenyl)(p-tosyl)methyl]-2-naphthol (3f) Yellow oil.

IR (KBr): 3385, 1600, 1490, 1279 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 9.34$  (1 H, s), 8.38 (1 H, m) 7.74– 7.58 (4 H, m), 7.33-7.02 (8 H, m), 6.72 (1 H, s), 2.38 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 164.5, 154.0, 145.6, 134.8, 133.7, 132.1, 130.0, 129.9, 127.3, 123.8, 122.1, 121.8, 117.9 (d, J = 10.0 Hz), 115.1 (d, J = 10.0 Hz), 67.0, 21.5.

ESI-MS: *m*/*z* = 463, 465 [M + Na]<sup>+</sup>.

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>18</sub>ClO<sub>3</sub>S + Na: 463.0541; found: 463.0544.

1-[(1,3-Benzodioxo1-5-yl)(p-tosyl)methyl]-2-naphthol (3g) Yellow oil.

IR (KBr): 3382, 1625, 1598, 1491, 1250 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.85 (1 H, br s), 7.68–7.52 (3 H, m), 7.35–7.11 (6 H, m), 7.09–7.00 (3 H, m), 6.72 (1 H, d, J = 8.0 Hz), 6.31 (1 H, s), 5.08 (2 H, s), 2.22 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 154.8, 152.3, 148.1, 144.6, 136.7, 133.2, 131.9, 129.8, 129.7, 129.5, 128.9, 127.1, 124.2, 123.2, 119.8, 119.2, 111.0, 67.6, 21.8.

ESI-MS:  $m/z = 455 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>20</sub>O<sub>3</sub>S + Na: 455.0929; found: 455.0927.

#### 1-[(p-Tosyl)(3,4,5-trimethoxyphenyl)methyl]-2-naphthol (3h) Yellow oil.

IR (KBr): 3377, 1592, 1509, 1460, 1253 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.90 (1 H, br s), 7.75–7.52 (5 H, m), 7.32–7.11 (3 H, m), 7.02 (2 H, d, J = 8.0 Hz), 6.86 (2 H, s), 6.32 (1 H, s), 3.82 (3 H, s), 3.80 (6 H, s), 2.21 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 154.9, 153.5, 144.8, 133.1, 132.0, 129.4, 129.2, 128.6, 127.1, 123.2, 121.1, 120.9, 70.4, 61.0, 56.1, 21.5.

ESI-MS:  $m/z = 479 [M + H]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>27</sub>H<sub>26</sub>O<sub>6</sub>S + Na: 501.1347; found: 501.1334.

#### 1-[(4-Nitrophenyl)(p-tosyl)methyl]-2-naphthol (3i) Yellow oil.

IR (KBr): 3414, 1690, 1598, 1516, 1345, 1275 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 8.60$  (1 H, br s), 7.89–7.80 (3 H, m), 7.75-7.62 (2 H, m), 7.58 (1 H, m), 7.38 (1 H, m), 7.31-7.20 (4 H, m), 7.05–6.92 (3 H, m), 6.46 (1 H, s), 2.22 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 156.1, 146.3, 144.0, 136.0, 131.8, 131.2, 129.8, 128.2, 127.9, 127.8, 126.0, 122.9, 121.1, 118.0, 117.3, 66.5, 21.1.

ESI-MS:  $m/z = 456 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>19</sub>NO<sub>5</sub>S + Na: 456.0876; found: 456.0873.

#### 1-[(Naphthalene-2-yl)(p-tosyl)methyl]-2-naphthol (3j) Yellow oil.

IR (KBr): 3418, 1689, 1609, 1445, 1341 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.78 (1 H, br s), 7.77–7.52 (6 H, m), 7.40–7.12 (9 H, m), 6.98 (2 H, d, J = 8.0 Hz), 6.58 (1 H, s), 2.17 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 153.3, 145.2, 136.1, 133.1, 131.8, 129.7, 129.2, 128.8, 128.5, 128.2, 127.5, 127.3, 127.1, 126.8, 125.0, 123.2, 123.0, 120.4, 70.0, 21.1.

ESI-MS:  $m/z = 461 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>28</sub>H<sub>22</sub>O<sub>3</sub>S + Na: 461.1182; found: 461.1179.

### 1-[1-(p-Tosyl)butyl]-2-naphthol (3k)

Yellow oil.

IR (KBr): 3348, 1624, 1597, 1462, 1280 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta = 8.60 (1 \text{ H}, \text{ s}), 7.70-7.59 (2 \text{ H}, \text{ m}),$ 7.54 (2 H, d, J = 8.0 Hz), 7.34–7.22 (2 H, m), 7.20–7.13 (2 H, m), 7.02 (2 H, d, J = 8.0 Hz), 4.98 (1 H, dd, J = 7.0, 2.0 Hz), 2.70 (1 H, m), 2.41 (1 H, m), 2.21 (3 H, s), 1.20-1.09 (2 H, m), 0.82 (3 H, t, J = 7.0 Hz).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 155.1, 144.6, 135.1, 131.5, 129.5, 129.1, 129.0, 126.9, 123.1, 121.3, 120.6, 64.7, 26.2, 21.1, 20.7, 13.9.

ESI-MS:  $m/z = 377 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>22</sub>O<sub>3</sub>S + Na: 377.1187; found: 377.1186.

#### **1-[2-Methyl-1-(***p***-tosyl)propyl]-2-naphthol (3l)** Yellow oil.

IR (KBr): 3360, 1598, 1514, 1457, 1338, 1283 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.92 (1 H, br s), 7.51 (2 H, d, J = 8.0 Hz), 7.41–7.22 (6 H, m), 6.90 (2 H, d, J = 8.0 Hz), 5.41 (1 H, d, J = 7.0 Hz), 3.31 (1 H, m), 2.22 (3 H, s), 1.29 (6 H, d, J = 7.0 Hz).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 153.1, 143.0, 138.2, 133.2, 131.0, 130.5, 129.0, 128.7, 127.5, 126.5, 126.2, 125.2, 123.0, 122.8, 67.3, 27.5, 31.2, 14.4.

ESI-MS:  $m/z = 377 [M + Na]^+$ .

HR-ESI-MS: m/z [M + Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>22</sub>O<sub>3</sub>S + Na: 377.1187; found: 377.1189.

# Reaction of Sulfone Derivatives 3 with Nucleophiles; General Procedure

To a stirred solution of the sulfonyl derivative **3** (1 mmol) in anhyd  $CH_2Cl_2$  (4 mL) kept under  $N_2$  at -10 °C were added allyltributyltin or anisole (1.5 mmol) and  $BF_3 \cdot Et_2O$  (20 mol%) under stirring. The reaction was monitored by TLC. After completion, the reaction mixture was concentrated.  $H_2O$  (10 mL) and EtOAc (10 mL) were added. The organic layer was separated and concentrated. The residue was subjected to column chromatography (silica gel; hexane–EtOAc) to isolate the pure product.

### 1-(1-Phenylbut-3-enyl)-2-naphthol (4a)

Yellow oil.

IR (KBr): 3423, 1603, 1512, 1461, 1267 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.02 (1 H, d, *J* = 8.0 Hz), 7.72 (1 H, d, *J* = 8.0 Hz), 7.61 (1 H, d, *J* = 8.0 Hz), 7.40 (1 H, t, *J* = 8.0 Hz), 7.36–7.21 (3 H, m), 7.10 (2 H, d, *J* = 8.0 Hz), 6.95 (1 H, d, *J* = 8.0 Hz), 5.71 (1 H, m), 5.14–4.98 (2 H, m), 5.87 (1 H, d, *J* = 7.0 Hz), 3.12 (1 H, m), 2.95 (1 H, m), 2.27 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 152.2, 139.6, 136.9, 135.0, 133.4, 129.6, 128.8, 127.5, 126.3, 123.2, 122.4, 119.1, 116.4, 40.5, 36.2, 21.1.

ESI-MS:  $m/z = 289 [M + H]^+$ .

Anal. Calcd for  $C_{21}H_{20}O$ : C, 87.50; H, 6.94. Found: C, 87.61; H, 6.98.

#### **1-[(4-Methoxyphenyl)(phenyl)methyl]-2-naphthol (5a)** Yellow oil.

IR (KBr): 3481, 1610, 1508, 1460, 1249 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.91 (2 H, d, *J* = 8.0 Hz), 7.75–7.68 (2 H, m), 7.40–7.20 (7 H, m), 7.11 (2 H, d, *J* = 8.0 Hz), 7.02 (1 H, d, *J* = 8.0 Hz), 6.81 (2 H, d, *J* = 8.0 Hz), 6.30 (1 H, s), 5.11 (1 H, br s), 3.73 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 159.5, 153.2, 141.8, 133.6, 133.5, 130.0, 129.8, 129.2, 129.0, 127.3, 127.0, 123.1, 122.8, 120.2, 114.8, 55.0, 48.2.

ESI-MS:  $m/z = 329 [M + H]^+$ .

Anal. Calcd for  $C_{23}H_{20}O_2$ : C, 84.15; H, 6.10. Found: C, 84.22; H, 6.07.

#### **1-(1-***p***-Tolylbut-3-enyl)-2-naphthol (4b)** Yellow oil.

IR (KBr): 3423, 1603, 1512, 1461, 1267 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.02 (1 H, d, *J* = 8.0 Hz), 7.75 (1 H, d, *J* = 8.0 Hz), 7.61 (1 H, d, *J* = 8.0 Hz), 7.40 (1 H, t, *J* = 8.0 Hz), 7.33–7.20 (3 H, m), 7.16–7.04 (2 H, m), 6.93 (1 H, d, *J* = 8.0 Hz), 5.70 (1 H, m), 5.12–4.99 (2 H, m), 4.88 (1 H, br d, *J* = 8.0 Hz), 3.12 (1 H, m), 2.95 (1 H, m), 2.29 (3 H, s).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 152.5, 140.0, 137.2, 136.1, 133.4, 129.7, 128.9, 128.0, 126.8, 123.2, 122.3, 119.5, 116.6, 40.2, 35.9, 21.0.

ESI-MS:  $m/z = 289 [M + H]^+$ .

Anal. Calcd for  $C_{21}H_{20}O$ : C, 87.50; H, 6.94. Found: C, 87.63; H, 6.87.

# 1-[(4-Isopropylphenyl)(4-methoxyphenyl)methyl]-2-naphthol (5b)

Yellow oil.

IR (KBr): 3483, 1615, 1509, 1462, 1250 cm<sup>-1</sup>.

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.92 (1 H, d, *J* = 8.0 Hz), 7.71 (1 H, d, *J* = 8.0 Hz), 7.65 (1 H, d, *J* = 8.0 Hz), 7.40–7.21 (2 H, m), 7.18–7.08 (6 H, m), 7.01 (1 H, d, *J* = 8.0 Hz), 6.81 (2 H, d, *J* = 8.0 Hz), 6.25 (1 H, s), 3.75 (3 H, s), 2.89 (1 H, m), 1.27 (6 H, d, *J* = 7.0 Hz).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 158.9, 153.2, 148.0, 139.5, 133.4, 130.0, 129.8, 128.9, 128.8, 127.5, 126.4, 123.0, 122.8, 120.1, 114.3, 55.6, 47.8, 30.0, 24.1.

ESI-MS:  $m/z = 405 [M + Na]^+$ .

Anal. Calcd for  $C_{27}H_{26}O_2$ : C, 84.82; H, 6.81. Found: C, 84.71; H, 6.87.

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#### References

- (1) Part 209 in the series 'Studies on Novel Synthetic Methodologies'.
- (2) Shen, A. Y.; Tsai, C. T.; Chen, C. L. Eur. J. Med Chem. 1999, 34, 877.
- (3) (a) Das, B.; Laxminarayana, K.; Ravikanth, B.; Rao, B. R. J. Mol. Catal. A: Chem. 2007, 261, 180. (b) Das, B.; Veeranjaneyulu, B.; Krishnaiah, M.; Balasubramanyam, P. Synth. Commun. 2009, 39, 1929.
- (4) (a) Khodaei, M. M.; Khosropour, A. R.; Moghanian, H. *Synlett* 2006, 916. (b) Selvam, N. P.; Perumal, P. T. *Tetrahedron Lett.* 2006, 47, 7481. (c) Srihari, G.; Nagaraju, M.; Murthy, M. M. *Helv. Chim. Acta* 2007, 90, 1497. (d) Shaterian, H. R.; Amirzadeh, A.; Khorami, E.; Ghashang, M. *Synth. Commun.* 2008, *38*, 2983. (e) Mahdavinia, G. H.; Bigdeli, M. A. *Chin. Chem. Lett.* 2009, 20, 383.
- (5) (a) Das, B.; Damodar, K.; Saritha, D.; Chowdhury, N.; Krishnaiah, M. *Tetrahedron Lett.* **2007**, *48*, 7930. (b) Das, B.; Damodar, K.; Shashi Kanth, B.; Srinivas, Y.; Kalavathi, I. *Synlett* **2008**, 3133. (c) Das, B.; Damodar, K.; Bhunia, N. *J. Org. Chem.* **2009**, *74*, 5607.
- (6) (a) Pearson, W. H.; Lindbeck, A. C.; Kampf, J. W. J. Am. Chem. Soc. 1933, 115, 2622. (b) Mecozzi, T.; Petrini, M. J. Org. Chem. 1999, 64, 8970.

(7) (a) Petrini, M. *Chem. Rev.* 2005, *105*, 3949. (b) Petrini, M.; Torregiani, E. *Synthesis* 2007, 159. (c) Thirupathi, P.; Kim, S. S. J. Org. Chem. 2009, *74*, 7755. (d) Reingruber, R.; Baumann, T.; Dabmen, S.; Bräse, S. Adv. Synth. Catal. 2009, 351, 1019.