# Supporting Information

# A direct approach for the expedient synthesis of unsymmetrical ethers by employing bromodimethylsulfonium bromide (BDMS) mediated C-S bond cleavage of naphthalene-2-ol sulfides

Kobirul Islam,<sup>a</sup> R. Sidick Basha,<sup>†a</sup> Ajaz A. Dar,<sup>†a</sup> Deb K. Das<sup>†a</sup> and Abu T. Khan<sup>\*a,b</sup>

<sup>a</sup> Department of Chemistry, Indian Institute of Technology Guwahati, Guwahati 781 039, India

<sup>b</sup> Vice-Chancellor, Aliah University, IIA/27, New Town, Kolkata-700 156, West Bengal, India.Tel.: +91

*361 2582305; fax: +91 361 2582349* 

Email: atk@iitg.ernet.in (A. T. Khan)

<sup>†</sup>These authors contributed equally

### List of Contents

1.	Title page	1
2.	General Information	2
3.	Table SI-1	2
6.	Spectroscopic Data	3-11
8.	Crystallographic description	11-12
9.	Table SI-2, SI-3, SI-4	12-13
10.	Copy of spectra ( <sup>1</sup> H and <sup>13</sup> C NMR) of all compounds	14-87

#### I. General Information:

Melting points were determined on a melting point apparatus and are uncorrected. IR spectra were recorded on IR spectrophotometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on NMR spectrometer TMS as internal reference; chemical shifts ( $\delta$  scale) are reported in parts per million (ppm). <sup>1</sup>H NMR Spectra are reported in the order: multiplicity, coupling constant (*J* value) in hertz (Hz) and number of protons; signals were characterized as s (singlet), d (doublet), t (triplet), q (quartet), sext (sextet), m (multiplet), and dd (doublet of doublet) and bs (broad singlet). Elemental analyses were carried out using CHNS/O analyzer. The X-ray crystallographic data were collected using a single XRD diffractometer.

Table SI-1. Synthesis of 1-[(alky/aryllthio)(phenyl)methyl]naphthalene-2-ol (1a-i)<sup>a</sup>

				Ar	∕S−R¹
	Ar-CHO +	OH + R <sup>1</sup> -SH	10 mol% BDMS CH <sub>3</sub> CN, rt		ОН
	5			1	
Entry	Ar	$R^1$	Product	Time/h	%Yield <sup>b</sup>
1	C <sub>6</sub> H <sub>5</sub>	4-Me-C <sub>6</sub> H <sub>4</sub>	<b>1</b> a	6	78
2	$4-NO_2-C_6H_4$	4-Me-C <sub>6</sub> H <sub>4</sub>	1b	6	80
3	$4-F-C_6H_4$	$4-\text{Me-C}_6\text{H}_4$	1c	6	78
4	$3-Br-C_6H_4$	$4-\text{Me-C}_6\text{H}_4$	1d	6	71 <sup>[c]</sup>
5	2-Naphthyl	$4-\text{Me-C}_6\text{H}_4$	<b>1e</b>	6	76 <sup>[c]</sup>
6	$4-Cl-C_6H_4$	$4-\text{Me-C}_6\text{H}_4$	<b>1f</b>	5	74
7	$4-\text{Me-C}_6\text{H}_4$	$4-Cl-C_6H_4$	1g	6	72
8	$C_6H_5$	$C_2H_5$	1 <b>h</b>	6	74 <sup>[c]</sup>
9	$4-NO_2-C_6H_4$	$C_3H_7$	<b>1i</b>	6	80 <sup>[c]</sup>

<sup>a</sup>The reactions were carried out in 2 mmol scale. <sup>b</sup>Isolated yield. <sup>c</sup>Reference No. 8a.

# II. General procedure for the preparation of 1-[aryl(alkyl/arylthio)methyl)-naphthalene-2-ol deri vatives (1)

Bromodimethylsulfonium bromide BDMS (0.2 mmol) was added to a mixture of aromatic aldehyde (2.0 mmol) and 2-naphthol (2.0 mmol) in 5 mL of acetonitrile and the reaction mixture was kept for stirring at room temperature. Then, the corresponding thiol (2.0 mmol) was added into it and the progress of the reaction was monitored by TLC. After completion of the reaction, reaction mixture was evaporated in rotary evaporator and DCM (20 mL) was added into it and the organic layer was washed with 20 mL of water. The water layer was further extracted using DCM (2 x 10 mL). The combined organic layer was dried over anhydrous sodium sulfate and it was concentrated in a rotary evaporator. The crude mixture was purified through silica gel column chromatography and the desired products (1) were obtained by eluting with ethyl acetate and hexane (1:99) mixture.

The sulfides **1d**, **1e**, **1h** and **1i** were prepared by following reported procedure<sup>[8a]</sup> and spectroscopic data are available there.

Note: Ethanethiol and propanethiol were handled in a well-ventilated fume hood using airtight syring to avoide nuisance odour.

#### 1-(Phenyl(p-tolylthio)methyl)naphthalen-2-ol (1a)

Gummy liquid (0.555 g, 78%);  $R_f$  (2% ethyl acetate/hexane) 0.40; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.36 (s, 1H), 7.65 (d, J = 8.8 Hz, 1H), 7.52-7.49 (m, 2H), 7.37 (d, J = 7.6 Hz, 2H), 7.18-7.00 (m, 8H), 6.71 (d, J = 8.0 Hz, 2H), 6.45 (s, 1H), 1.94 (s, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  154.3, 138.5, 138.1, 133.0, 131.6, 130.3, 130.1, 129.9 (2C), 129.4, 129.0 (3C), 129.5 (3C), 127.8, 126.9, 123.2, 122.2, 120.0, 115.2, 51.4, 21.1 ppm; IR (KBr)  $v_{max}$  3434 (-OH) cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>20</sub>OS (356.48): C, 80.86; H, 5.65. Found: C, 80.98; H, 5.74.

#### 1-((4-Nitrophenyl)(p-tolylthio)methyl)naphthalen-2-ol (1b)

Gummy liquid (0.642 g, 80%);  $R_f$  (2% ethyl acetate/hexane) 0.18; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.07 (d, J = 9.2 Hz, 2H), 8.06-7.66 (m, 5H), 7.40-7.26 (m, 4H), 7.20 (d, J = 8.8 Hz, 1H), 6.99 (d, J = 8.0 Hz, 2H), 6.66 (s, 1H), 2.21 (s, 3H) ppm; <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>):  $\delta$  153.3, 147.2, 147.1, 138.5, 132.5, 131.9 (2C), 130.8, 130.1 (2C), 129.7, 129.4 (2C), 129.2 (2C), 127.2, 123.9 (2C), 123.6, 122.5, 119.3, 115.7, 50.4, 21.2 ppm; IR (KBr)  $v_{max}$  3433 (-OH), 1588 (NO<sub>2</sub>), 1322 (NO<sub>2</sub>) cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>19</sub>NO<sub>3</sub>S (401.47): C, 71.80; H, 4.77. Found: C, 71.94; H, 4.84.

#### 1-((4-Fluorophenyl)(p-tolylthio)methyl)naphthalen-2-ol (1c)

Gummy liquid (0.583 g, 78%);  $R_f$  (2% ethyl acetate/hexane) 0.42; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.41 (s, 1H), 7.73 (d, J = 8.8 Hz, 1H), 7.68-7.66 (m, 2H), 7.46-7.42 (m, 2H), 7.33-7.20 (m, 4H), 7.14 (d, J = 8.8 Hz, 1H), 6.96-6.87 (m, 4H), 6.52 (s, 1H), 2.12 (s, 3H) ppm; <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>):  $\delta$  163.2, 161.5, 154.4, 138.5, 134.2, 133.0, 131.9 (3C), 130.5, 130.4, 130.3, 130.1 (2C), 129.5, 129.1, 127.1, 123.4, 122.0, 120.2, 116.0, 115.8, 50.9, 21.3 ppm; IR (KBr)  $v_{max}$  3430 (-OH) cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>19</sub>FOS (374.48): C, 76.98; H, 5.11. Found: C, 77.16; H, 5.18.

1-((4-Chlorophenyl)(p-tolylthio)methyl)naphthalen-2-ol (1f)

Gummy liquid (0.577 g, 74%);  $R_f$  (2% ethyl acetate/hexane) 0.50; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.06 (s, 1H), 7.46 (d, J = 8.8 Hz, 3H), 7.16 (d, J = 8.4 Hz, 2H), 7.09 (t, J = 8.4 Hz, 2H), 7.03-6.99 (m, 5H), 6.90 (d , J = 8.8 Hz, 1H ), 6.68 (d , J = 8.0 Hz, 1H ), 6.24 (s, 1H), 1.94 (s, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  154.0, 138.2, 137.3, 133.6, 132.8, 131.7 (2C), 130.5, 130.0 (2C), 129.9 (2C), 129.5,

129.0 (3C), 127.0, 123.6, 123.4, 122.2, 119.8, 115.2, 50.6, 21.1 ppm; IR (KBr) *v*<sub>max</sub> 3436 (-OH), 1622, 1467, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>19</sub>ClOS (390.93): C, 73.74; H, 4.90. Found: C, 73.92; H, 4.96.

#### 1-(((4-Chlorophenyl)thio)(p-tolyl)methyl)naphthalen-2-ol (1g)

Gummy liquid (0.562 g, 72%);  $R_f$  (2% ethyl acetate/hexane) 0.54; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.01 (s, 1H), 7.81 (d, J = 8.4 Hz, 1H), 7.73 (t, J = 8.8 Hz, 2H), 7.41-7.33 (m, 4H), 7.31-7.25 (m, 3H), 7.12 (t, J = 7.2 Hz, 2H), 7.08 (d, J = 6.8 Hz, 2H), 6.59 (s, 1H), 2.31 (s, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  153.8, 137.7, 135.0, 133.7, 132.8, 132.7, 132.3 (2C), 130.5, 129.7 (2C), 129.5, 129.2 (2C), 129.0 (2C), 128.3 (2C), 127.1, 123.4, 122.3, 119.6, 50.4, 21.2 ppm; IR (KBr)  $v_{max}$  3302 (-OH), 1622, 1467, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>19</sub>CIOS (390.93): C, 73.74; H, 4.90. Found: C, 73.86; H, 4.98.

#### 1-(Ethoxy (phenyl) methyl) naphthalen-2-ol (3a)

Gummy liquid (0.261 g, 94%);  $R_f$  (2% ethyl acetate/hexane) 0.50; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  9.38 (s, 1H), 7.80-7.75 (m, 2H), 7.73 (d, J = 8.8 Hz, 1H), 7.42-7.39 (m, 3H), 7.33-7.25 (m, 4H), 7.20 (d, J = 8.8 Hz, 1H), 6.34 (s, 1H), 3.80-3.73 (m, 2H), 1.36 (t, J = 7.2 Hz, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  154.6, 139.9, 132.4, 130.3, 129.0 (2C), 128.8 (2C), 128.5, 127.8 (2C), 127.0, 123.2, 121.5, 119.9, 114.9, 81.7, 66.3, 15.4 ppm; IR (KBr)  $v_{max}$  3302 (-OH), 1622, 1467, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>19</sub>H<sub>18</sub>O<sub>2</sub> (278.35): C, 81.99; H, 6.52. Found: C, 82.14; H, 6.60.

#### 1-(Methoxy (phenyl) methyl) naphthalen-2-ol (3b)

White solid (0.237 g, 90%);  $R_f$  (2% ethyl acetate/hexane) 0.65; mp 74-75°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  9.12 (s, 1 H), 7.78-7.73 (m, 2H), 7.71 (d, J = 8.4 Hz, 1H), 7.40-7.36 (m, 3H), 7.31-7.23 (m, 4H), 7.18 (d, J = 8.4 Hz, 1H), 6.20 (s, 1H), 3.55 (s, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  154.3, 139.5, 132.4, 130.4, 129.0 (2C), 128.8 (2C), 128.5, 127.8 (2C), 127.0, 123.2, 121.4, 119.8, 114.4, 83.7, 57.9 ppm; IR (KBr)  $v_{max}$  3302(-OH), 1621, 1467, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>18</sub>H<sub>16</sub>O<sub>2</sub> (264.32): C, 81.79; H, 6.10. Found: C, 81.94; H, 6.01; MS (ESI) calcd for C<sub>18</sub>H<sub>15</sub>O<sub>2</sub> (M - H<sup>+</sup>) 263.1150, found 263.0648.

#### 1-(Phenyl (propoxy) methyl) naphthalen-2-ol (3c)

White solid (0.268 g, 92%); mp 72-73 °C;  $R_f$  (2% ethyl acetate/hexane) 0.54; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  9.32 (s, 1H), 7.78-7.70 (m, 3H), 7.41-7.36 (m, 3H), 7.30-7.22 (m, 4H), 7.18 (d, J = 8.8 Hz, 1 H), 6.31 (s, 1H), 3.70-3.57 (m, 2H), 1.78-1.68 (m, 2H), 0.97 (t, J = 7.6 Hz, 3H) ppm; <sup>13</sup>C NMR (100

MHz, CDCl<sub>3</sub>):  $\delta$  154.5, 139.9, 132.4, 130.2, 129.0, 128.9, 128.7 (2C), 128.4, 127.7 (2C), 126.9, 123.1, 121.5, 119.9, 115.0, 81.8, 72.4, 23.0, 10.8 ppm; IR (KBr)  $v_{\text{max}}$  3302 (-OH), 1601, 1467, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>20</sub>H<sub>20</sub>O<sub>2</sub> (292.368): C, 82.16; H, 6.89. Found: C, 82.02; H, 6.96; MS (ESI) calcd for C<sub>20</sub>H<sub>19</sub>O<sub>2</sub> (M - H<sup>+</sup>) 291.1385, found 291.1381.

#### 1-(Isopropoxy(phenyl)methyl)naphthalen-2-ol (3d)

Light yellow solid (0.239 g, 82%); mp 43-45°C;  $R_f$  (2% ethyl acetate/hexane) 0.55; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.52 (s, 1H), 7.86-7.81 (m, 3H), 7.51-7.45 (m, 3H), 7.40-7.31 (m, 4H), 7.28 (d, J = 8.8 Hz, 1H), 6.55 (s, 1H), 4.04-3.98 (m, 1H), 1.42 (d, J = 6.0 Hz, 3H), 1.36 (d, J = 6.4 Hz, 3 H ) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.9, 140.3, 132.4, 130.2, 129.0, 128.8, 128.7 (2C), 128.2, 127.7 (2C), 127.0, 123.1, 121.3, 120.0, 115.5, 78.2, 71.7, 22.6, 22.0 ppm; IR (KBr)  $v_{max}$  3267, 1622, 1467, 1227 cm<sup>-1</sup>; Anal. Calcd for C<sub>20</sub>H<sub>20</sub>O<sub>2</sub> (292.37): C, 82.16; H, 6.89. Found: C, 81.94; H, 6.98.

#### 1-(Butoxy(phenyl)methyl)naphthalen-2-ol (3e)

Gummy liquid (0.269 g, 88%);  $R_f$  (2% ethyl acetate/hexane) 0.60; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.31 (s, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.72 (d, J = 8.8 Hz, 1H), 7.41-7.37 (m, 3H), 7.31-7.25 (m, 4H), 7.17 (d, J = 8.8 Hz, 1H), 6.31 (s, 1H), 3.74-3.62 (m, 2H), 1.70 (q, J = 6.8 Hz, 2H), 1.43 (sex, J = 7.2 Hz, 2H ), 0.93 (t, J = 8.0 Hz, 3H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  154.5, 139.9, 132.5, 130.3, 129.0 (2C), 128.8 (2C), 128.4, 127.8 (2C), 127.0, 123.2, 121.5, 120.0, 115.0, 81.9, 70.6, 31.9, 19.6, 14.0 ppm; IR (KBr)  $v_{\text{max}}$  3302, 1622, 1463, 1226 (C-O) cm<sup>-1</sup>; Anal. Calcd for C<sub>21</sub>H<sub>22</sub>O<sub>2</sub> (306.40): C, 82.32; H, 7.24. Found: C, 82.48; H, 7.34.

#### 1-((Benzyloxy)(phenyl)methyl)naphthalen-2-ol (3f)

grey solid (0.285 g, 84%); mp 104-106 °C;  $R_f$  (2% ethyl acetate/hexane) 0.44; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.02 (s, 1H), 7.72-7.69 (m, 2H), 7.57 (d, J = 8.8 Hz, 1H), 7.30-7.12 (m, 13H), 6.35 (s, 1H), 4.69 (d, J = 11.6 Hz, 1H), 4.55 (d, J = 11.6 Hz, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 150 MHz)  $\delta$  154.6, 139.5, 136.7, 132.6, 130.5, 129.1, 129.0, 128.9 (2C), 128.8 (3C), 128.7, 128.6, 128.5, 127.8 (2C), 127.1, 123.3, 121.4, 119.9, 114.5, 80.0, 71.9 ppm; IR (KBr)  $v_{max}$  3320, 1618, 1466, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>24</sub>H<sub>20</sub>O<sub>2</sub> (340.41): C, 84.68; H, 5.92 Found: C, 84.80; H, 6.01.

#### 1-((Allyloxy)(phenyl)methyl)naphthalen-2-ol (3g)

white solid (0.238 g, 82%); mp 60-62 °C;  $R_f$  (2% ethyl acetate/hexane) 0.44; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.14 (s, 1H), 7.77-7.30 (m, 2H), 7.70 (d, J = 8.4 Hz, 1H), 7.40-7.36 (m, 3H), 7.30-7.23 (m, 4H),

7.18 (d, J = 8.8 Hz, 1H), 6.40 (s, 1H), 6.05-5.94 (m, 1H), 5.34-5.24 (m, 2H), 4.26-4.20 (m, 1H), 4.16-4.10 (m, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.5, 139.6, 133.4, 132.4, 130.4, 129.0, 128.9, 128.7 (2C), 128.4, 127.8 (2C), 127.0, 123.1, 121.4, 119.9, 118.9, 114.6, 80.3, 70.8 ppm; IR (KBr)  $v_{max}$  3312, 1621, 1600, 1467, 1225 cm<sup>-1</sup>; Anal. Calcd for C<sub>20</sub>H<sub>18</sub>O<sub>2</sub> (290.36): C, 82.73; H, 6.25. Found: C, 82.88; H, 6.34; MS (ESI) calcd for C<sub>20</sub>H<sub>17</sub>O<sub>2</sub> (M - H<sup>+</sup>) 289.1229, found 289.0776.

#### 1-(Phenyl(prop-2-yn-1-yloxy)methyl)naphthalen-2-ol (**3h**)

reddish liquid (0.230 g, 80%);  $R_f$  (2% ethyl acetate/hexane) 0.35; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.59 (s, 1H), 7.80-7.74 (m, 3H), 7.44-7.39 (m, 3H), 7.34-7.27 (m, 4H), 7.19 (d, J = 8.8 Hz, 1H), 6.70 (s, 1H), 4.39 (dd, J = 2.4, 15.6 Hz, 1H), 4.29 (dd, J = 2.4, 18.0 Hz, 1H), 2.57 (t, J = 2.4 Hz, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.5, 138.8, 132.6, 130.7, 129.0 (2C), 128.8 (2C), 128.7, 128.1 (2C), 127.1, 123.3, 121.5, 119.8, 113.7, 79.4, 78.6, 76.3, 56.8 ppm; IR (KBr)  $\nu_{max}$  3350, 3290, 2118, 1622, 1468, 1224 cm<sup>-1</sup>; Anal. Calcd for C<sub>20</sub>H<sub>16</sub>O<sub>2</sub> (288.34): C, 83.31; H, 5.59. Found: C, 83.46; H, 5.68.

#### 1-((Pent-4-en-1-yloxy)(phenyl)methyl)naphthalen-2-ol (3i)

Off white (0.267 g, 84%) 58-60°C;  $R_f$  (2% ethyl acetate/hexane) 0.48; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.24 (s, 1H), 7.74-7.68 (m, 3H), 7.38-7.33 (m, 3H), 7.27-7.16 (m, 5H), 6.28 (s, 1H), 5.81-5.70 (m, 1H), 5.03-5.01 (m, 1H), 4.98-4.93 (m, 1H), 3.70-3.58 (m, 2H), 2.16-2.11 (m, 2H), 1.82-1.72 (m, 2H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.4, 139.8, 137.8, 132.4, 130.3, 129.0, 128.9, 128.7 (2C), 128.4, 127.7 (2C), 127.0, 123.1, 121.4, 119.9, 115.5, 114.9, 81.8, 70.0, 30.4, 28.9 ppm; IR (KBr)  $\nu_{max}$  3299, 1622, 1600, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>22</sub>H<sub>22</sub>O<sub>2</sub> (318.41): C, 82.99; H, 6.96. Found: C, 83.16; H, 7.04; MS (ESI) calcd for C<sub>22</sub>H<sub>21</sub>O<sub>2</sub> (M - H<sup>+</sup>) 317.1542, found 317.1018.

#### 1-(Phenyl(4-phenylbutoxy)methyl)naphthalen-2-ol (3j)

yellowish liquid (0.336 g, 88 %);  $R_f$  (2% ethyl acetate/hexane) 0.43; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.24 (s, 1H), 7.74 (t, J = 8.4 Hz, 2H), 7.69 (d, J = 8.8 Hz, 1H), 7.40-7.34 (m, 3H), 7.30-7.23 (m, 5H), 7.21-7.12 (m, 5H), 6.28 (s, 1H), 3.72-3.61 (m, 2H), 2.60 (t, J = 7.2 Hz, 2H), 1.74-1.69 (m, 4H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 150 MHz)  $\delta$  154.4, 142.2, 139.8, 132.4, 130.3, 129.0, 128.9, 128.8 (2C), 128.6 (2C), 128.5 (2C), 128.4, 127.8 (2C), 127.0, 126.0, 123.2, 121.4, 119.9, 114.9, 81.8, 70.5, 35.7, 29.3, 28.0 ppm; IR (KBr)  $v_{max}$  3285, 1621, 1600, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>27</sub>H<sub>26</sub>O<sub>2</sub> (382.49): C, 84.78; H, 6.85. Found: C, 84.90; H, 6.93; MS (ESI) calcd for C<sub>27</sub>H<sub>25</sub>O<sub>2</sub> (M - H<sup>+</sup>) 381.1855, found 381.1700.

gummy liquid (0.238 g, 81%);  $R_f$  (10% ethyl acetate/hexane) 0.13; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.97 (s, 1H), 7.78-7.73 (m, 2H), 7.70 (d, J = 8.4 Hz, 1H), 7.41-7.36 (m, 3H), 7.31-7.25 (m, 4H), 7. 17 (d, J = 8.8 Hz, 1H), 6.38 (s, 1H), 3.83-3.74 (m, 4H), 2.16 (bs, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.3, 139.5, 132.4, 130.6, 129.1 (2C), 129.0 (2C), 128.7, 127.9 (2C), 127.1, 123.4, 121.5, 119.9, 114.5, 82.3, 71.9, 62.0 ppm; IR (KBr)  $\nu_{max}$  3317, 1621, 1600, 1467, 1224 cm<sup>-1</sup>; Anal. Calcd for C<sub>19</sub>H<sub>18</sub>O<sub>3</sub> (294.34): C, 77.53; H, 6.16. Found: C, 77.71; H, 6.22.

#### 1-((2-Acetoxyethoxy)(phenyl)methyl)naphthalen-2-yl acetate (3k')

The compound 3k (0.5 mmol) was added to a mixture of 1 mL of acetic anhydride and 1 mL of pyridine and kept for stirring overnight at room temperature. After completion of reaction, pyridine and unreacted acetic anhydride were removed by co-evaporation using toluene (1 x 2 mL) in a rotary evaporator. The resulting crude residue was purified through silica gel column to obtain pure product 3k' in 94% yield as a gummy liquid. The product was obtained 0.138 g after purification and it was eluted with ethyl acetate and hexane (1:3).

*R<sub>f</sub>*: 0.5 (2% ethyl acetate/hexane); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.15 (d, *J* = 8.4 Hz, 1H), 7. 77 (d, *J* = 9.2 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.34-7.23 (m, 4H), 7.20-7.09 (m, 4H), 6.16 (s, 1H), 4.20-4.10 (m, 2H), 3.62-3.57 (m, 1H), 3.50-3.44 (m, 1H), 2.25 (s, 3H), 1.88 (s, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  171.2, 170.0, 148.1, 141.7, 132.8, 132.1, 130.5, 128.7, 128.3 (2C), 127.1, 126.5 (2C), 126.4, 126.0 (2C), 125.7, 121.6, 76.3, 67.2, 63.6, 21.1, 21.0 ppm; IR (KBr)  $\nu_{max}$  3058, 3022, 2928, 1766, 1730 cm<sup>-1</sup>; Anal. Calcd for C<sub>23</sub>H<sub>22</sub>O<sub>5</sub> (378.42): C, 73.01; H, 5.86. Found: C, 73.17; H, 5.94.

#### 1-(((8-Hydroxyoctyl)oxy)(phenyl)methyl)naphthalen-2-ol (31)

gummy liquid (0.309 g, 82%);  $R_f$  (10% ethyl acetate/hexane) 0.26; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.24 (s, 1H), 7.67 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 7.6 Hz, 1H), 7.31-7.28 (m, 3H), 7.22-7.13 (m, 4H), 7.09 (d, J = 8.8 Hz, 1H), 6.21 (s, 1H), 3.59-3.49 (m, 5H), 1.63-1.57 (m, 2H), 1.46-1.42 (m, 2H), 1.35-1.21 (m, 8H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.3, 139.9, 132.3, 130.2, 128.9, 128.9, 128.7 (2C), 128.3, 127.7 (2C), 126.9, 123.1, 121.5, 119.8, 115.0, 81.7, 70.7, 62.9, 32.8, 29.7, 29.3 (2C), 26.1, 25.7 ppm; IR (KBr)  $v_{\text{max}}$  3303, 1622, 1601, 1463, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>25</sub>H<sub>30</sub>O<sub>3</sub> (378.50): C, 79.33; H, 7.99. Found: C, 79.48; H, 8.06.

yellow solid (0.290 g, 94%); mp 101-103°C;  $R_f$  (2% ethyl acetate/hexane) 0.24; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.62 (s, 1H), 8.11 (d, J = 8.8 Hz, 2H), 7.80 (d, J = 7.2 Hz, 1H), 7.79 (d, J = 8.8 Hz, 1H), 7.74 (d, J = 8.8 Hz, 1H), 7.52 (d, J = 9.2 Hz, 2H), 7.46 (td, J = 8.4, 1.2 Hz, 1H), 7.34 (t, J = 8.0 Hz, 1H), 7.17 (d, J = 8.8 Hz, 1H), 6.32 (s, 1H), 3.60 (s, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.4, 147.8, 147.0, 132.5, 131.1, 129.3, 129.1, 128.2 (2C), 127.5, 123.9 (2C), 123.58, 121.1, 119.8, 113.6, 81.4, 58.2 ppm; IR (KBr)  $v_{\text{max}}$  3327, 1599, 1520, 1467, 1320, 1224 cm<sup>-1</sup>; Anal. Calcd for C<sub>18</sub>H<sub>15</sub>NO<sub>4</sub> (309.32): C, 69.89; H, 4.89; N, 4.53. Found: C, 70.07; H, 4.96; N, 4.64.

#### 1-(Butoxy(4-nitrophenyl)methyl)naphthalen-2-ol (3n)

semi solid (0.308 g, 88%);  $R_f$  (2% ethyl acetate/hexane) 0.26; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.82 (s, 1H), 8.11 (d, J = 9.2 Hz, 2H), 7.81-7.76 (m, 2H), 7.74 (d, J = 8.8 Hz, 1H), 7.52 (d, J = 8.8 Hz, 2H), 7.45 (td, J = 7.2, 1.2 Hz, 1H), 7.33 (t, J = 8.0 Hz, 1H), 7.16 (d, J = 8.8 Hz, 1H), 6.40 (s, 1H), 3.78-3.66 (m, 2H), 1.75-1.65 (m, 2H), 1.47-1.39 (m, 2H), 0.93 (t, J = 7.6 Hz, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.6, 147.8, 147.3, 132.4, 131.0, 129.3, 129.1, 128.2 (2C), 127.5, 123.9 (2C), 123.5, 121.3, 119.9, 114.2, 79.8, 70.9, 31.8, 19.5, 14.0 ppm; IR (KBr)  $v_{max}$  3313, 1623, 1520, 1467, 1347, 1225 cm<sup>-1</sup>; Anal. Calcd for C<sub>21</sub>H<sub>21</sub>NO<sub>4</sub> (351.40): C, 71.78; H, 6.02; N, 3.99. Found: C, 71.92; H, 5.92, N, 4.07.

#### 1-(Ethoxy(4-fluorophenyl)methyl)naphthalen-2-ol (30)

gummy liquid (0.278 g, 94%);  $R_f$  (2% ethyl acetate/hexane) 0.53; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.26 (s, 1H), 7.74-7.70 (m, 2H), 7.65 (d, J = 8.8 Hz, 1H), 7.38-7.24 (m, 4H), 7.18 (d, J = 8.8 Hz, 1H), 6.92 (t, J = 8.8 Hz, 2H), 6.26 (s, 1H), 3.71-3.65 (m, 2H), 1.29 (t, J = 7.2 Hz, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.5, 135.7, 132.3, 130.4, 129.6 (2C), 129.0 (2C), 128.9, 127.0, 123.2, 121.3, 119.8, 115.6, 115.4, 114.6, 80.8, 66.2, 15.3 ppm; IR (KBr)  $v_{max}$  3297, 1622, 1601, 1508, 1463, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>19</sub>H<sub>17</sub>FO<sub>2</sub> (296.34): C, 77.01; H, 5.78. Found: C, 77.12; H, 5.84.

#### *1*-((3-Bromophenyl)(methoxy)methyl)naphthalen-2-ol (3p)

white solid (0.223 g, 92%); mp 90-92°C;  $R_f$  (2% ethyl acetate/hexane) 0.45; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.89 (s, 1H), 7.76-7.71 (m, 2H), 7.66 (d, J = 8.4 Hz, 1H), 7.60-7.50 (m, 1H), 7.41-7.33 (m, 2H), 7.30-7.26 (m, 1H), 7.21-7.14 (m, 2H), 7.08-7.03 (m, 1H), 6.14 (s, 1H), 3.49 (s, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 150 MHz)  $\delta$  154.4, 141.9, 132.4, 131.6, 130.7, 130.4, 129.1, 129.0, 127.2, 126.3, 123.4, 122.9, 121.2, 119.9 (2C), 113.7, 82.6, 58.1 ppm; IR (KBr)  $v_{max}$  3318, 1622, 1599, 1468, 1224 cm<sup>-1</sup>; Anal. Calcd for

 $C_{18}H_{15}BrO_2$  (343.21): C, 62.99; H, 4.41. Found: C, 63.14; H, 4.34; MS (ESI) calcd for  $C_{18}H_{14}BrO_2$  (M - H<sup>+</sup>) 341.0177, found 341.0165.

#### 1-(Methoxy(naphthalen-2-yl)methyl)naphthalen-2-ol (3q)

White solid (0.276 g, 88%); mp 98-100°C;  $R_f$  (2% ethyl acetate/hexane) 0.40; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.20 (s, 1H), 7.79-7.73 (m, 8H), 7.56 (dd, J = 8.8, 1.6 Hz, 1H), 7.44-7.36 (m, 2H), 7.28 (t, J = 8.0 Hz, 1H), 7.22 (d, J = 9.2 Hz, 1H), 6.36 (s, 1H), 3.60 (s, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.5, 136.8, 133.3, 130.5 (2C), 129.0, 128.8, 128.5, 127.8 (2C), 127.0, 126.9, 126.5, 126.4, 125.7, 123.2, 121.5, 120.0, 114.3, 102.0, 84.0, 58.1 ppm; IR (KBr)  $\nu_{max}$  3300, 1622, 1599, 1466, 1225 cm<sup>-1</sup>; Anal. Calcd for C<sub>22</sub>H<sub>18</sub>O<sub>2</sub> (314.38): C, 84.05; H, 5.77. Found: C, 84.22; H, 5.90.

#### 1-(Ethoxy(p-tolyl)methyl)naphthalen-2-ol (8)

yellow liquid (0.262 g, 90%);  $R_f$  (2% ethyl acetate/hexane) 0.50; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.41 (s, 1 H), 7.76-7.71 (m, 2H), 7.69 (d, J = 8.4 Hz, 1H), 7.36 (td, J = 6.8, 1.6 Hz, 1H), 7.28-7.25 (m, 3H), 7.17 (d, J = 8.8 Hz, 1H), 7.09 (d, J = 8.0 Hz, 2H), 6.27 (s, 1H), 3.73-3.71 (m, 2H), 2.27 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.4, 138.1, 136.9, 132.3, 130.1, 129.4 (3C), 128.9, 127.7 (2C), 126.8, 123.0, 121.4, 119.8, 115.0, 81.6, 65.9, 21.1, 15.2 ppm; IR (KBr)  $v_{max}$  3287, 1622, 1600, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>20</sub>H<sub>20</sub>O<sub>2</sub> (292.37): C, 82.16; H, 6.89. Found: C, 82.34; H, 6.80.

## $Bis(4-methylphenyl)disulfide (6a)^{23}$

solid; mp 44-45 °C (lit. m.p 43-46°C);  $R_f$  (2% ethyl acetate/hexane) 0.80; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.39 (d, J = 8.0 Hz, 4H), 7.11 (d, J = 8 Hz, 4H), 2.32 (s, 6H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  137.6 (2C), 134.1 (2C), 130.0 (4C), 128.7 (4C), 21.3 (2C) ppm.

#### 1-(Hydroxy(phenyl)methyl)naphthalen-2-ol (7a)

white solid (0.210 g, 84%); mp 99-101°C;  $R_f$  (10% ethyl acetate/hexane) 0.36; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.37 (s, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.67 (d, J = 8.8 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.34-7.18 (m, 7H), 7.71 (d, J = 8.4 Hz, 1H), 6.62 (s, 1H), 3.56 (s, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 150 MHz)  $\delta$  154.7, 141.5, 131.7, 130.4, 129.1 (2C), 129.0, 128.9, 128.7, 127.4 (2C), 127.0, 123.2, 121.6, 120.2, 115.9, 74.9 ppm; IR (KBr)  $v_{max}$  3373, 1623, 1600, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>17</sub>H<sub>14</sub>O<sub>2</sub> (250.29): C, 81.58; H, 5.64. Found: C, 81.35; H, 5.70; MS (ESI) calcd for C<sub>17</sub>H<sub>13</sub>O<sub>2</sub> (M - H<sup>+</sup>) 249.0916, found 249.0799.

reddish solid (0.260 g, 88%); mp 130-132°C;  $R_f$  (10% ethyl acetate/hexane) 0.15; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.54 (s, 1H), 8.10 (d, J = 8.8 Hz, 2H), 7.79 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 8.8 Hz, 1H), 7.68 (d, J = 8.8 Hz, 1H), 7.53 (d, J = 8.8 Hz, 2H), 7.45-7.41 (m, 1H), 7.36-7.31 (m, 1H), 7.13 (d, J = 8.8 Hz, 1H), 6.83 (s, 1H), 3.81 (s, 1H) ppm; <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz)  $\delta$  153.2, 152.1, 146.2, 131.6, 129.4, 128.5, 128.2, 126.8 (2C), 126.0, 123.2, 122.8 (3C), 122.4, 118.6, 68.4 ppm; IR (KBr)  $\nu_{max}$  3424, 1625, 1514, 1345 cm<sup>-1</sup>; Anal. Calcd for C<sub>17</sub>H<sub>13</sub>NO<sub>4</sub> (295.29): C, 69.15; H, 4.44; N, 4.74. Found: C, 69.37; H, 4.52; N, 4.86.

#### 1-((4-Fluorophenyl)(hydroxy)methyl)naphthalen-2-ol (7c)

white solid (0.230 g, 86%); mp 101-102 °C;  $R_f$  (10% ethyl acetate/hexane) 0.31; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.19 (s, 1H), 7.77-7.72 (m, 2H), 7.58 (d, J = 8.8 Hz, 1H), 7.39-7.34 (m, 3H), 7.31-7.25 (m, 1H), 7.15 (d, J = 8.8 Hz, 1H), 6.98 (t, J = 8.4 Hz, 2H), 6.72 (s, 1H), 3.27 (s, 1H) ppm; <sup>1</sup>H NMR (D<sub>2</sub>O, 400 MHz)  $\delta$  7.76 (d, J = 8.8 Hz, 1H), 7.73 (d, J = 9.2 Hz, 1H), 7.59 (d, J = 8.4 Hz, 1H), 7.39-7.25 (m, 4H), 7.15 (d, J = 8.8 Hz, 1H), 6.97 (t, J = 8.4 Hz, 2H), 6.71 (s, 1 H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.5, 137.3, 131.6, 131.5, 129.2 (2C), 129.2, 129.0, 129.0, 127.1, 123.4, 121.5, 120.0, 116.2, 116.0, 115.8, 73.8 ppm; IR (KBr)  $v_{max}$  3372, 1622, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>17</sub>H<sub>13</sub>FO<sub>2</sub> (268.28): C, 76.11; H, 4.88. Found: C, 75.92; H, 4.96; MS (ESI) calcd for C<sub>17</sub>H<sub>12</sub>FO<sub>2</sub> (M - H<sup>+</sup>) 267.0821, found 267.0577.

#### 1-((4-Chlorophenyl)(hydroxy)methyl)naphthalen-2-ol (7d)

white solid (0.238 g, 84%); mp 111-113 °C;  $R_f$  (10% ethyl acetate/hexane) 0.34; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  7.77 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 8.8 Hz, 1H), 7.62 (d, J = 8.4 Hz, 1H), 7.41-7.25 (m, 6H), 7.17 (d, J = 8.8 Hz, 1H), 6.77 (s, 1H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.6, 140.0, 134.4, 131.6, 130.6, 129.2 (2C), 129.1 (2C), 128.8 (2C), 127.2, 123.4, 121.5, 120.1, 116.0, 73.6 ppm; IR (KBr)  $v_{max}$  3378, 1622, 1467, 1226 cm<sup>-1</sup>; Anal. Calcd for C<sub>17</sub>H<sub>13</sub>ClO<sub>2</sub> (284.74): C, 71.71; H, 4.60. Found: C, 71.48; H, 4.70.

#### 1-(Hydroxy(p-tolyl)methyl)naphthalen-2-ol (9)

white solid (0.216 g, 82%); mp 120-121°C;  $R_f$  (10% ethyl acetate/hexane) 0.35; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  9.40 (s, 1H), 7.74 (t, J = 9.2 Hz, 2H), 7.61 (d, J = 8.4 Hz, 1H), 7.34-7.24 (m, 4H), 7.17 (d, J = 8.8 Hz, 1H), 7.12 (d, J = 7.6 Hz, 2H), 6.73 (s, 1H), 3.14 (s, 1H), 2.30 (s, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.8, 138.7, 138.6, 131.7, 130.3, 129.8 (3C), 128.9, 127.4 (2C), 126.9, 123.2, 121.7,

120.2, 115.9, 75.0, 21.3 ppm; IR (KBr)  $v_{max}$  3413, 1624, 1467, 1225 cm<sup>-1</sup>; Anal. Calcd for C<sub>18</sub>H<sub>16</sub>O<sub>2</sub> (264.32): C, 81.79; H, 6.10. Found: C, 82.02; H, 6.16; MS (ESI) calcd for C<sub>18</sub>H<sub>15</sub>O<sub>2</sub> (M - H<sup>+</sup>) 263.1072, found 263.1006.

#### 1-(ethoxymethyl)naphthalen-2-ol (18)

Semi solid (0.170 g, 89%);  $R_f$  (10% ethyl acetate/hexane) 0.30; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  8.75 (s, 1H), 7.68 (d, J = 8.4 Hz, 2H), 7.58 (dd, J = 8.4, 2.4 Hz, 1H), 7.34 (td, J = 7.2, 1.2 Hz, 1H), 7.21 (t, J = 7.2 Hz, 1H), 7.02 (d, J = 9.0 Hz, 1H), 5.12 (s, 2H), 3.62 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.2 Hz, 3H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  154.7, 131.8, 129.7, 128.9, 128.8, 126.7, 123.1, 121.1, 119.4, 112.1, 69.1, 67.1, 15.1 ppm; IR (KBr)  $v_{max}$  3311, 1624, 1468, 1227 cm<sup>-1</sup>; Anal. Calcd for C<sub>13</sub>H<sub>14</sub>O<sub>2</sub> (202.25): C, 77.20; H, 6.98; Found: C, 77.34; H, 7.06.

#### **Crystallographic Description:**

The X-ray crystal structures were determined with diffractometer. Complete crystallographic data of **3p** and **7a** (CCDC no. is 913509 and 913508) for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, Copies of this information may be obtained free of charge from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-1223-336033, e-mail: deposit@ccdc.cam.ac.uk or via: www.ccdc.cam.ac.uk).The crystal structures of **3p** and **7a** were obtained by single crystal X-ray diffraction technique. The Single crystals of **3p** was obtained by slow evaporation of dichloromethane and chloroform (1:3) solution of compound. Single crystals of **7a** was obtained by slow evaporation of dichloromethane and hexane (1:3) solution of compound. The selected crystallographic data of **3p** and **7a** are given in Table S1. The crystals of all compounds were mounted on glass fiber. All geometric and intensity data for the crystals were collected at room temperature using a diffractometer equipped with a fine focus 1.75 kW sealed tube Mo K $\alpha$  ( $\lambda = 0.71073$  Å) X-ray source, with increasing  $\omega$  (width of 0.3° per frame) at a scan speed of 3 s/frame.



**Figure SI-1.** Schematic representation of **3p** self-assembled through weak C-H···O hydrogen bonding interactions viewed along *b*-axis (Left). Hydrogen-bonded 1D chain structure of **7a** forms 2D supramolecular assembly via C-H··· $\pi$  interaction viewed along *b*-axis (Right).

Compounds	<b>3</b> p	7a
Empirical formula	C <sub>18</sub> H <sub>14</sub> Br O <sub>2</sub>	C <sub>17</sub> H <sub>14</sub> O <sub>2</sub>
М	342.20	250.28
Wavelength Å	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	P2(1)/n	<i>P2(1)/c</i>
<i>a</i> , Å	13.1085(6)	4.7273(6)
b, Å	7.3865(4)	13.5885(16)
<i>c</i> , Å	16.4008(7)	20.003(3)
α	90.00°	90.00°
β	107.548(3)°	96.613(7)°
γ	90.00°	90.00°
$V/Å^3$	1514.12(13)	1276.4(3)
Ζ	4	4
$\rho/g \text{ cm}^{-3}$	1.501	1.302
μ/mm <sup>-1</sup>	2.716	0.084
Reflns collected	16477	14730
Indep reflns	2659	2208
GOF	0.949	0.969
Final R indices $[I \ge 2\sigma(I)]$	R1=0.0447	R1 = 0.0434
	wR2 = 0.0864	wR2 = 0.1036
R indices (all data)	R1=0.1202	R1 = 0.0779
	wR2=0.1092	wR2=0.1216

Table SI-2. Crystallographic data and refinement parameters<sup>a</sup>

<sup>a</sup>Refinement methods: full-matrix least-square on  $F^2$ .

3р							
01-C1	1.366(5)	C1-C2	1.392(5)	C2-C3	1.334(6)	C3-C4	1.425(6)
C5-C4	1.389(6)	C5-C6	1.358(8)	C6-C7	1.407(7)	C1-C10	1.388(5)
C7-C8	1.370(5)	C11-O2	1.428(4)	C9-C8	1.425(5)	C10-C9	1.420(5)
C9-C4	1.417(5)	C10-C11	1.523(5)	C12-C17	1.390(5)	C12-C13	1.378(5)
C11-C12	1.525(5)	C13-C14	1.389(5)	C14-C15	1.386(5)	C15-C16	1.350(6)
C16-C17	1.390(5)	Br1-C16	1.897(4)				
7a							
C9-O1	1.381(2)	C1- C2	1.374(3)	C1-C6	1.382(2)	C1-C7	1.518(2)
C2-C3	1.387(3)	C3- C4	1.355(3)	C4- C5	1.377(3)	C5-C6	1.379(3)
C7-O2	1.436(2)	C7-C8	1.520(2)	C8-C9	1.384(2)	C9-C10	1.399(3)
C8-C17	1.428(2)	C10-C11	1.349(3)	C11-C12	1.411(2)	C12-C13	1.416(3)
C13-C14	1.355(3)	C14-C15	1.397(3)	C15-C16	1.360(2)	C16-C17	1.425(2)
C17-C12	1.426(2)						

<u>**Table SI-3.** Selected bond distances (Å) of compounds **3p** and **7a 3n**</u>

Table SI-4. H-bond interactions in 7a and 3p

Tuble SI 4. If bolid interactions in 74 and 5p					
D-H···A	<i>d</i> (D-H)(Å)	$d(\mathbf{H} \cdots \mathbf{A})(\mathbf{\mathring{A}})$	$d(\mathbf{D}\cdots\mathbf{A})(\mathbf{\mathring{A}})$	$<$ DHA( $\Box$ )	
7a					
O1-H1O2	0.82	1.91	2.6235(18)	145	
O2-H2O1	0.82	2.08	2.8841(18)	167	
C2-H2AO2	0.93	2.37	2.727(2)	103	
3р					
O1-H1O2	0.82	1.96	2.669(4)	144	
C17-H17O2	0.93	2.34	2.694(4)	102	

## <sup>1</sup>H NMR spectra of 1a



<sup>13</sup>C NMR spectra of 1a



<sup>1</sup>H NMR spectra of 1b



## <sup>13</sup>C NMR spectra of 1b



<sup>1</sup>H NMR spectra of 1c



## <sup>13</sup>C NMR spectra of 1c



## <sup>1</sup>H NMR spectra of 1f



## <sup>13</sup>C NMR spectra of 1f



## <sup>1</sup>H NMR spectra of 1g



<sup>13</sup>C NMR spectra of 1g



<sup>1</sup>H NMR spectra of 1,2-di-*p*-tolyldisulfane (6a)



## <sup>13</sup>C NMR spectra of 1,2-di-*p*-tolyldisulfane (6a)



<sup>1</sup>H NMR spectra of 3a



## <sup>13</sup>C NMR spectra of 3a



<sup>1</sup>H NMR spectra of 3b



<sup>13</sup>C NMR Spectra of 3b



# MS spectra of 3b



## <sup>1</sup> H NMR spectra of 3c



<sup>13</sup> C NMR	Spectra	of 3c
---------------------	---------	-------



MS spectra of 3c



<sup>1</sup>H NMR spectra of 3d



## <sup>13</sup>C NMR Spectra of 3d



## <sup>1</sup>H NMR spectra of 3e


# <sup>13</sup>C NMR Spectra of 3e



<sup>1</sup>H NMR spectra of 3f



#### <sup>13</sup>C NMR Spectra of 3f



$^{1}H$	<b>NMR</b>	spectra	of 3g



## <sup>13</sup>C NMR Spectra of 3g



MS spectra of 3g



<sup>1</sup>H NMR spectra of 3h



<sup>13</sup>C NMR Spectra of 3h



## <sup>1</sup>H NMR spectra of 3i



# <sup>13</sup>C NMR Spectra of 3i

KI-111-13C expl s2pul SAMPLE SPECIAL date Jun 14 2012 temp not used solvent CDC13 gain not used file exp spin not used ACOUISITION hst 0.008 sw 25125.6 pw90 18.600 at 1.199 alfa 20.000 pt 1800 11 FLAGS rb 13800 11 rn n d1 1.000 dp y nt 3000 hs nn CT 250 PROCESSING TRANSMITTER 15 tr 10556 3 sp -71.0 tr 1536.3 sp 2-71.0 tpwr 61 wp 21229.5 pw 9.300 rf1 9288.2 DECOUPLER rfp 7764.3 dof 0 1P -257.0 dmm w wc 250 dmm w wc 250 dmm w wc 250 dmm s 10 10 10 10 10 10 10 10 10 10 10 10 10								
154.426	-139.799 -137.755 -137.401 -132.401 -129.007	126.937 128.937 128.955 128.134 123.134 112.455 119.870 119.870 114.454	4-14.00 - 14.00 - 14.00					<b></b>
200 180 160	140	120	100	80	60	40 -	20	ppm

MS Spectra of 3i



## <sup>1</sup>H NMR spectra of 3j



#### <sup>13</sup>C NMR Spectra of 3j



MS Spectra of 3j



<sup>1</sup>H NMR spectra of 3k



# <sup>13</sup>C NMR Spectra of 3k





<sup>1</sup>H NMR spectra of 1-((2-acetoxyethoxy)(phenyl)methyl)naphthalen-2-yl acetate (3k')



<sup>13</sup>C NMR spectra of 1-((2-acetoxyethoxy)(phenyl)methyl)naphthalen-2-yl acetate (3k')

<sup>1</sup>H NMR spectra of 3l



<sup>13</sup>C NMR Spectra of 31



#### <sup>1</sup>H NMR spectra of 3m



<sup>13</sup> C NMR	spectra	of 3m
---------------------	---------	-------



#### <sup>1</sup>H NMR spectra of 3n



<sup>13</sup> C	NMR	spectra	of	3n
-----------------	-----	---------	----	----

220 200 180 160 140 120 100 80 60 40 20 0
-------------------------------------------

## <sup>1</sup>H NMR spectra of 30



# <sup>13</sup>C NMR spectra of 30



<sup>1</sup>H NMR spectra of 3p

Ē

KI-113   exp1 s2pul   SAMPLE SPECIAL   date Jun 16 2012 temp not is   solvent CDC13 gain not is   file spin not is   solvent CDC13 gain not is   file spin not is   solvent CDC13 gain not is   file spin not is   sw 6239.8 spin not is   at 139.8   at 1.39.8   at 1.00.0   di 1.00.0   di 1.00.0   ct 32   ct 32   TRANSMITTER b   tof 362.8   sfrq 399.853   tof 362.8   pw 3.850   cf 16   bECOUPLER rfp   dn Cl3   dof 0   dm %nn   dm %nn   dm %nn   dm %nn   dm %nn   dm %nn   dm %nn </th <th>Jsed Jsed Jsed Jsed Jsed Jsed Jsed Jsed</th> <th></th> <th></th> <th>Br O OH 3p</th> <th></th>	Jsed Jsed Jsed Jsed Jsed Jsed Jsed Jsed			Br O OH 3p	
		dal mada			
11 10	9 5.36	8 7 7.70(3.)555.98 13.536618.13.12	6 بہا 6.47	5 4 3 'ự' 18.60	2 1 ppm

# <sup>13</sup>C NMR spectra of 3p



MS Spectra of 3p



<sup>1</sup>H NMR spectra of 3q



#### <sup>13</sup>C NMR spectra of 3q



KI-122   expl s2pul   SAMPLE SPECIAL   date AuguISITION   sw 1.998   at 1.998   at 1.998   at 1.000   sw 10   at 1.998   at 1.000   sw 10   at 1.000   sw 10   at 1.000   di 1.000   sw 10   at 1.000   sw 10   at 1.000   at 10   at 1.000   at 10   at 10   at 10   at 10   at 1.000   at 11   at 11   at 10   at 11   at 10   at 10   at 10   at 10   at 10	6 5	$(\mathbf{j}, \mathbf{j}, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0$	3 2	1	pp
10 9 8 7 بن بنېېې 4.07 8.33 14. <b>36</b> .14 7.18 5.75.23	6 5 4.38	4	3 2	1 14.76	bt

<sup>13</sup>C NMR Spectra of 8



<sup>1</sup>H NMR spectra of 7a



#### <sup>13</sup>C NMR spectra of 7a



MS spectra of 7a


<sup>1</sup>H NMR spectra of 7b





<sup>1</sup> H NMR spectra of 7c
----------------------------------



<sup>13</sup>C NMR spectra of 7c



<sup>1</sup>H NMR spectra of 7c (D<sub>2</sub>O exchange)







<sup>1</sup>H NMR spectra of 7d



## <sup>13</sup>C NMR spectra of 7d



<sup>1</sup>H NMR spectra of 9







MS spectra of 9



## <sup>1</sup>H NMR spectra of 15



<sup>13</sup>C NMR spectra of 15



## <sup>1</sup>H NMR spectra of 18



## <sup>13</sup>C NMR spectra of 18

