

# Asymmetric synthesis of corsifuran A by an enantioselective reduction

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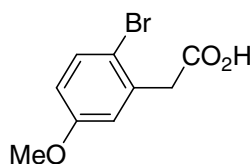
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## Supplementary Information

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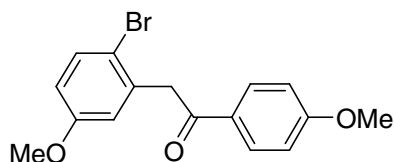
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### Preparation of 2-bromo-5-methoxyphenylacetic acid **4**<sup>1</sup>



Bromine (4.31 g, 1.39 mL, 27 mmol) was added at 0 °C to a solution of 3-methoxyphenylacetic acid (4.50 g, 27 mmol) in dichloromethane (25 mL) and stirred at room temperature for 24 hrs. The dark red solution was discoloured with sodium thiosulfate solution (1M, 15 mL), washed with water (20 mL) and separated. The aqueous layer was extracted into dichloromethane (2 × 25 mL) and the combined organic layers dried over MgSO<sub>4</sub>, filtered and evaporated to dryness to give compound **4** (6.62 g, 100%) as a white solid. mp 114 °C (lit.<sup>1</sup> 114 – 115 °C); IR 2974 (brd), 1690, 1596, 1571, 1473 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>) δ<sub>H</sub> 7.75 – 8.30 (1H, brd), 7.45 (1H, d, *J* 9.0), 6.83 (1H, d, *J* 3.0), 6.70 (1H, dd, *J* 9.0, 3.0), 3.55 (3H, s), 3.50 (2H, s); <sup>13</sup>C NMR (63 MHz; CDCl<sub>3</sub>) δ<sub>C</sub> 177.1, 158.9, 134.3, 133.4, 117.3, 115.1, 114.8, 55.5, 41.6; HRMS calcd for C<sub>9</sub>H<sub>9</sub>O<sub>3</sub><sup>79</sup>Br [M<sup>+</sup>] 243.9735, found 243.9739; MS (EI) 246 (25), 244 (25), 201 (20), 199 (20), 166 (18), 165 (100), 121 (20), 109 (15).

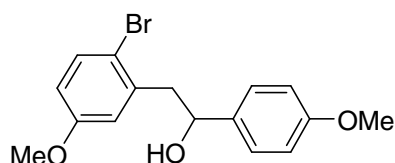
### Preparation of 1-(4-methoxyphenyl)-2-(2-bromo-4-methoxyphenyl)ethanone **3**<sup>2</sup>



2-Bromo-5-methoxyphenyl acetic acid **4** (312 mg, 1.3 mmol) and thionyl chloride (155 mg, 1.3 mmol) were combined in a round-bottomed flask and refluxed for 30 mins. The reaction was then evaporated under a reduced pressure to dryness, re-dissolved in dichloromethane (10 mL) and cooled to -5 °C. Anisole (140 mg, 1.3 mmol) was added followed by the addition of powdered aluminium trichloride (173 mg, 1.3 mmol) portion-wise. The reaction was stirred for 30 mins and then poured into a small beaker of ice, the organic layer separated and the aqueous layer extracted with dichloromethane (2 × 10 mL). The combined organic fractions were washed with 1M HCl (30 mL) and dried over MgSO<sub>4</sub>. After purification by chromatography on silica gel, eluting with 20% EtOAc / 80% petroleum ether 40 – 60), compound **3** was obtained as an off white solid (0.380 g; 89%); mp 113 – 114 °C (lit.<sup>2</sup> 114 °C); IR 1672, 1596, 1574, 1459 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>) δ<sub>H</sub> 8.08 – 8.00 (2H, m),

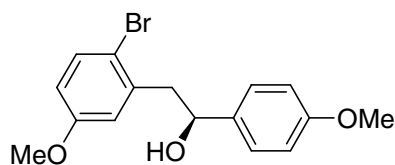
7.48 (1H, d, *J* 8.8), 6.99 – 6.93 (2H, m) 6.78 (1H, d, *J* 2.8), 6.41 (1H, dd, *J* 8.0, 2.8), 4.38 (2H, s), 3.90 (3H, s) 3.78 (3H, s); <sup>13</sup>C NMR (63 MHz; CDCl<sub>3</sub>) δ<sub>C</sub> 194.9, 163.7, 158.9, 136.2, 133.2, 130.7, 129.6, 117.2, 115.4, 114.5, 113.9, 55.5, 55.4, 45.6; HRMS calcd for C<sub>16</sub>H<sub>15</sub>O<sub>3</sub><sup>79</sup>Br [M<sup>+</sup>] 334.0205, found 334.0207; MS (EI) 336 (1), 334 (1), 255 (32), 135 (100), 107 (6), 92 (15), 77 (24), 64 (6).

### Preparation of racemic 1-(2-bromo-5-methoxy-phenyl)-2-(3-methoxy-phenyl)-ethanol **2**



Sodium borohydride (0.33 g, 8.9 mmol) was added to ethanol (25 mL) and stirred at room temperature. Ketone **3** (1.50 g, 4.4 mmol) was added drop-wise with stirring and the reaction was left for 1 h at room temperature. Water (10 mL) was added, the mixture extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 30 cm<sup>3</sup>), the organic layer washed with water (2 × 200 mL) and dried over MgSO<sub>4</sub>. The solution was filtered and concentrated under reduced pressure to provide the desired product **2** (1.38 g, 92%); Analytical data was as reported below.

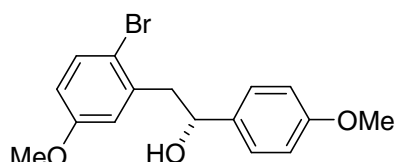
### Preparation of 2*S*-(2-bromo-5-methoxy-phenyl)-1-(4-methoxy-phenyl)-ethanol **2**



Trimethyl borate (0.25 mL, 0.2 mmol) was added to a solution of the (1*R*, 2*S*)-1-amino-indan-2-ol (3 mg, 0.2 mmol) in THF (3 mL) and stirred at room temperature under a nitrogen atmosphere for 30 min. BH<sub>3</sub>.DMS complex (0.17 mL, 2.0 mmol) was added, the reaction stirred for 30 min, then ketone **3** (0.737 g, 2.19 mmol) in THF (2 mL) added via cannula. The reaction mixture was stirred for a further 30 min at room temperature then quenched with methanol (5 mL). Water (10 mL) was added and the solvent removed under reduced pressure. The mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL), the organic phase washed with 1M HCl (30 mL), water (30 mL) and dried over MgSO<sub>4</sub>. Filtration, and removal of the solvent under reduced pressure

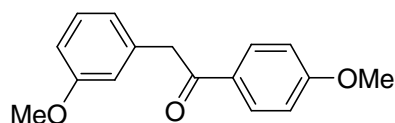
produced the crude product (*S*)-**2** in 76% ee. The alcohol was recrystallised to 99% ee from petroleum ether (40 – 60) / CH<sub>2</sub>Cl<sub>2</sub>; mp 76-77 °C, [ $\alpha$ ]<sub>D</sub> -11.2 (*c* 1, CHCl<sub>3</sub>); ee 99% [Kromasil 3-CelluCoat 8% iso-propanol in hexane @ 1.00 mL / min, 10.04 and 15.36 mins (major)]; IR 3361, 2931, 1614, 1571, 1514, 1454 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>)  $\delta$ <sub>H</sub> 7.45 (1H, d, *J* 9.1), 7.36 – 7.29 (2H, m), 6.90 – 6.82 (2H, m), 6.71 (1H, d, *J* 2.8), 6.67 (1H, dd, *J* 9.1, 2.8), 4.95 (1H, dd, *J* 4.9, 8.5), 3.80 (3H, s), 3.72 (3H, s), 3.13 (1H, dd, *J* 4.9, 13.7), 3.02 (1H, dd, *J* 8.5, 13.7), 1.95 (1H, brd s); <sup>13</sup>C NMR (125 MHz; CDCl<sub>3</sub>)  $\delta$ <sub>C</sub> 159.1, 158.7, 138.6, 136.0, 133.3, 127.0, 117.4, 115.2, 114.1, 113.8, 73.1, 55.4, 55.3, 46.4; HRMS calcd for C<sub>16</sub>H<sub>17</sub>O<sub>3</sub><sup>79</sup>Br [M<sup>+</sup>] 336.0361, found 336.0367; MS (EI) 338 (1), 336 (1), 202 (13), 200 (14), 137 (100), 109 (20), 94 (15), 77 (30).

#### Preparation of 2*R*-(2-bromo-5-methoxy-phenyl)-1-(4-methoxy-phenyl)-ethanol **2**.



Using the above method with (1*S*, 2*R*)-catalyst with ketone **3** (2.66 g, 7.92 mmol), gave alcohol (*R*)-**2** (2.38 g, 89%) in 78% ee. The ee was increased to 99% by repeated recrystallisation from petroleum ether (40 – 60) / CH<sub>2</sub>Cl<sub>2</sub>; mp 77-78 °C, [ $\alpha$ ]<sub>D</sub> +15.9 (*c* 1, CHCl<sub>3</sub>); ee 99% [Kromasil 3-CelluCoat 8% iso-propanol in hexane @ 1.00 mL / min, 9.90 (major) and 15.84 mins]; remainder of analytical data the same as for (*S*)-**2**.

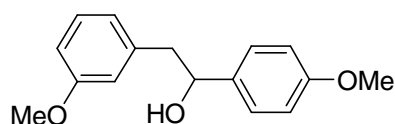
#### Preparation of 3, 4-dimethoxydeoxybenzoin **8**<sup>3</sup>



3-Methoxyphenylacetic acid (4.00 g, 24 mmol) and thionyl chloride (2.86 g, 23 mmol) were combined in a round-bottomed flask and refluxed for 30 mins. The reaction was then evaporated under a reduced pressure to dryness, re-dissolved in dichloromethane (40 mL) and cooled to -5 °C. Anisole (2.60 g, 24 mmol) was added followed by the addition of powdered aluminium trichloride (3.20 g, 24 mmol) portion-wise. The reaction was stirred for 30 mins and then poured into a small beaker of ice, the organic layer separated and the aqueous layer extracted with

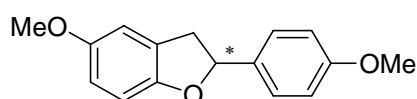
dichloromethane (2 × 40 mL). The combined organic fractions were washed with 1M HCl (40 mL) and dried over MgSO<sub>4</sub>. After purification by chromatography on silica gel, eluting with 20% EtOAc / 80% petroleum ether 40 – 60), compound **8** (3.90 g; 65%) was obtained as an off white solid; mp 44 – 45 °C (lit.<sup>3</sup> 38 – 40 °C); IR 1672, 1592, 1492 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>) δ<sub>H</sub> 8.02 – 7.95 (2H, m), 7.25 – 7.17 (1H, m), 6.95 – 6.74 (5H, m), 4.22 (2H, s), 3.85 (3H, s) 3.80 (3H, s); <sup>13</sup>C NMR (125 MHz; CDCl<sub>3</sub>) δ<sub>C</sub> 195.9, 163.3, 159.6, 136.3, 130.8, 129.4, 121.6, 114.9, 113.6, 112.1, 55.2, 54.9, 45.1; HRMS calcd for C<sub>16</sub>H<sub>16</sub>O<sub>3</sub> [M<sup>+</sup>], 256.1099, found 256.1093; MS (EI) 256 (7), 136 (11), 135 (100), 121 (3), 107 (6), 92 (11), 77 (18).

### Preparation of 1-(4-methoxy-phenyl)-2-(3-methoxy-phenyl)-ethanol **7**



Sodium borohydride (0.73 g, 19 mmol) was added to ethanol (30 mL) and stirred at room temperature. Ketone **9** (2.50 g, 9.7 mmol) was added drop-wise with stirring and the reaction was left for 1 h at room temperature. Water (10 mL) was added, the mixture extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 30 mL), the organic layer washed with water (2 × 200 mL) and dried over MgSO<sub>4</sub>. The solution was filtered and concentrated under reduced pressure to provide the desired product **7** (2.20 g, 88%); Found: C, 74.03; H, 7.16%. C<sub>16</sub>H<sub>18</sub>O<sub>3</sub> requires C, 64.39, H, 7.02; IR 3425, 2936, 1610, 1584, 1511 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>) δ<sub>H</sub> 7.35 – 7.21 (3H, m), 6.95 – 6.85 (2H, m), 6.81 – 6.72 (4H, m), 4.85 (1H, td, *J* 4.7, 2.1), 3.80 (3H, s), 3.75 (3H, s), 3.05 – 2.91 (2H, m), 2.63 (1H, d, *J* 2.1); <sup>13</sup>C NMR (63 MHz, CDCl<sub>3</sub>) δ<sub>C</sub> 159.7, 159.1, 139.8, 136.0, 129.5, 127.2, 121.8, 115.1, 113.8, 112.1, 74.9, 55.3, 55.2, 46.0; HRMS calcd for C<sub>16</sub>H<sub>18</sub>O<sub>3</sub> [M<sup>+</sup>] 258.1256, found 258.1252; MS (EI) 258 (4), 241 (15), 240 (23), 165 (10), 122 (100), 121 (55), 109 (55), 94 (47), 91 (52), 79 (66), 77 (95).

### General procedure for preparation of Corsifuran A **1** by palladium-catalyzed intramolecular C-O bond formation



2-Bromo-5-methoxy-phenyl-1-(4-methoxy-phenyl)-ethanol **2** (400 mg, 1.8 mmol) and base (1.5 equiv.), in dry toluene (5 mL) were added to a flame dried 50 cm<sup>3</sup> two-necked round bottomed flask fitted with a condenser under a N<sub>2</sub> atmosphere, containing the palladium source (3 mol %) and ligand (3.5 mol %) in toluene (5 mL). The reaction was refluxed for 24 h then cooled to room temperature, diluted with diethyl ether (15 mL) and filtered through Celite. The solvent was evaporated under a reduced atmosphere to give a dark yellow solution. Column chromatography on silica gel eluting with 20 % EtOAc / 80 % petroleum ether 40 – 60 gave natural product **1** as the first fraction, inseparable alcohols **2** and **7** as the second fraction, and inseparable ketones **3** and **8** as the third fraction. Conversion and yields were as reported in the manuscript table. Analytical data was as reported below.

#### **Preparation of (*R*)-Corsifuran A **1** by copper-catalyzed intramolecular C-O bond formation**

(*R*)-2-Bromo-5-methoxy-phenyl-1-(4-methoxy-phenyl)-ethanol **2** (500 mg, 1.48 mmol, 100% ee) was added to a stirred solution of sodium hydride (60% dispersion in mineral oil; pre-washed with petroleum ether; 44 mg, 1.85 mmol) in toluene (2 mL). The solution was heated to 40 °C for 15 mins and cooled back to room temperature. Copper<sup>I</sup> chloride<sup>1</sup> (7 mg, 0.074 mmol) and ethyl acetate (1-2 drops) in toluene (2 mL) were added and the reaction was heated at reflux for 24 h. After cooling to room temperature, the solution was quenched with water (20 mL) and filtered through Celite with ether washings (2 × 10 mL). The organic layer was separated, the aqueous layer extracted with ether (2 × 20 mL) and the combined organic fractions washed sequentially with 1 M HCl (30 mL), saturated NaHCO<sub>3</sub> (30 mL), saturated NaCl (30 mL) and water (30 mL) and dried over NaSO<sub>4</sub>. After flash chromatography on silica gel, eluting with 15 % EtOAc / 75 % petroleum ether 40-60, (*R*)-corsifuran A **1** was obtained in a 0.288 g, 76% yield, >99 % ee; (5 m octakis(2, 6-O-Me-3-O-Pe)-gamma-cyclodextrin capillary, hydrogen as the carrier gas at 2 mL per minute; Injector temp 250 °C, flame ionisation detector at 250 °C, isothermal at 160 °C; mp 61 °C; [α]<sub>D</sub> -11.5 (*c* 1, CHCl<sub>3</sub>) [lit.<sup>4</sup> -11.1 (*c* 0.1 CDCl<sub>3</sub>)]; IR 2937, 1590, 1486 cm<sup>-1</sup>; <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>) δ<sub>H</sub> 7.33 – 7.22 (2H, m), 6.86 – 6.78 (2H, m), 6.73 – 6.56 (4H, m), 5.62 (1H, app t, *J* 8.8), 3.75 (3H, s), 3.70 (3H, s), 3.45 (1H, dd, *J* 8.8, 16.5), 3.10 (1H, dd, *J*

<sup>1</sup> Purified reagent plus, greater than or equal to 99%, Sigma Aldrich.

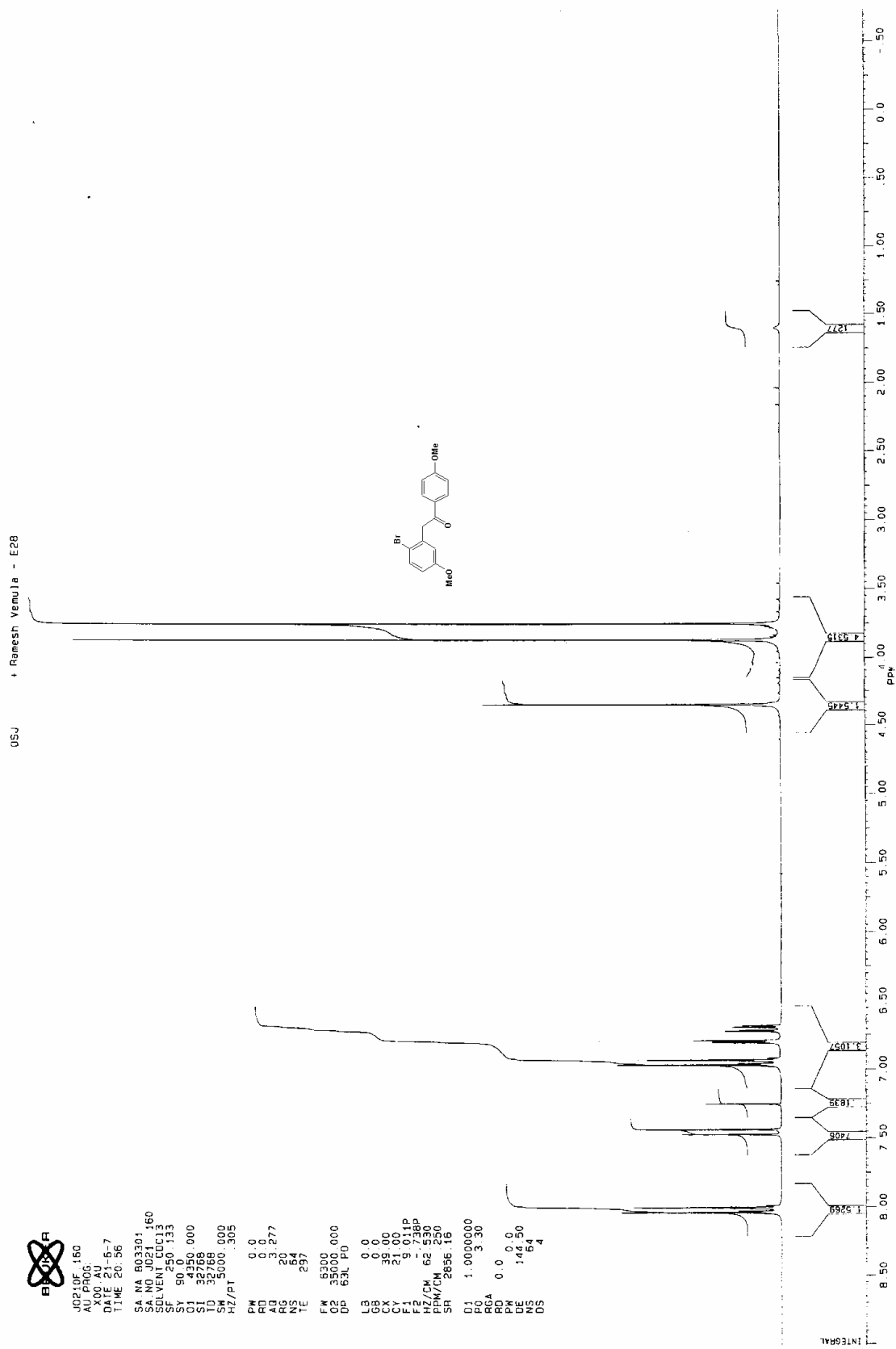
8.8, 16.5);  $^{13}\text{C}$  NMR (63 MHz;  $\text{CDCl}_3$ )  $\delta_{\text{C}}$  159.4, 154.2, 153.7, 133.9, 127.7, 127.3, 113.9, 113.0, 111.1, 109.2, 84.2, 56.0, 55.3, 38.7; HRMS calcd for  $\text{C}_{16}\text{H}_{16}\text{O}_3$  [ $\text{M}^+$ ] 256.1099, found 256.1103; MS (EI) 256 (26), 241 (6), 213 (6), 198 (7), 181 (11), 152 (16), 115 (26).

(*S*)-Corsifuran A **1**; >99% ee, (5 m octakis(2, 6-O-Me-3-O-Pe)-gamma-cyclodextrin capillary, hydrogen as the carrier gas at 2 mL per minute; Injector temp 250 °C, flame ionisation detector at 250 °C, isothermal at 160 °C; mp 61 – 62 °C;  $[\alpha]_{\text{D}}$  +15.5 (*c* 1,  $\text{CHCl}_3$ ).

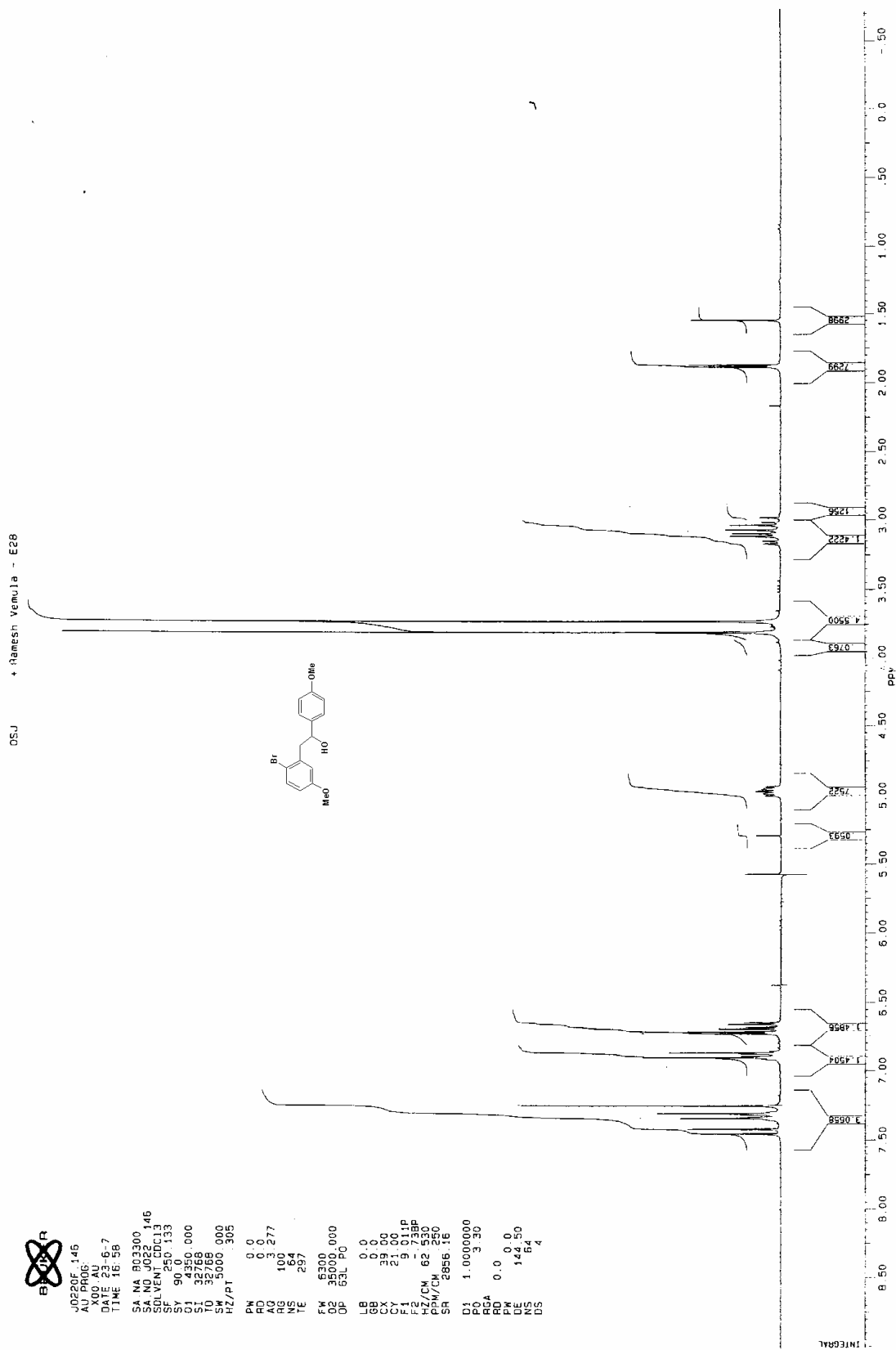
Racemic corsifuran was prepared in an identical manner starting from racemic alcohol **2**.



# <sup>1</sup>H NMR Spectrum of compound 3

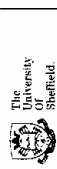


# <sup>1</sup>H NMR Spectrum of compound 2

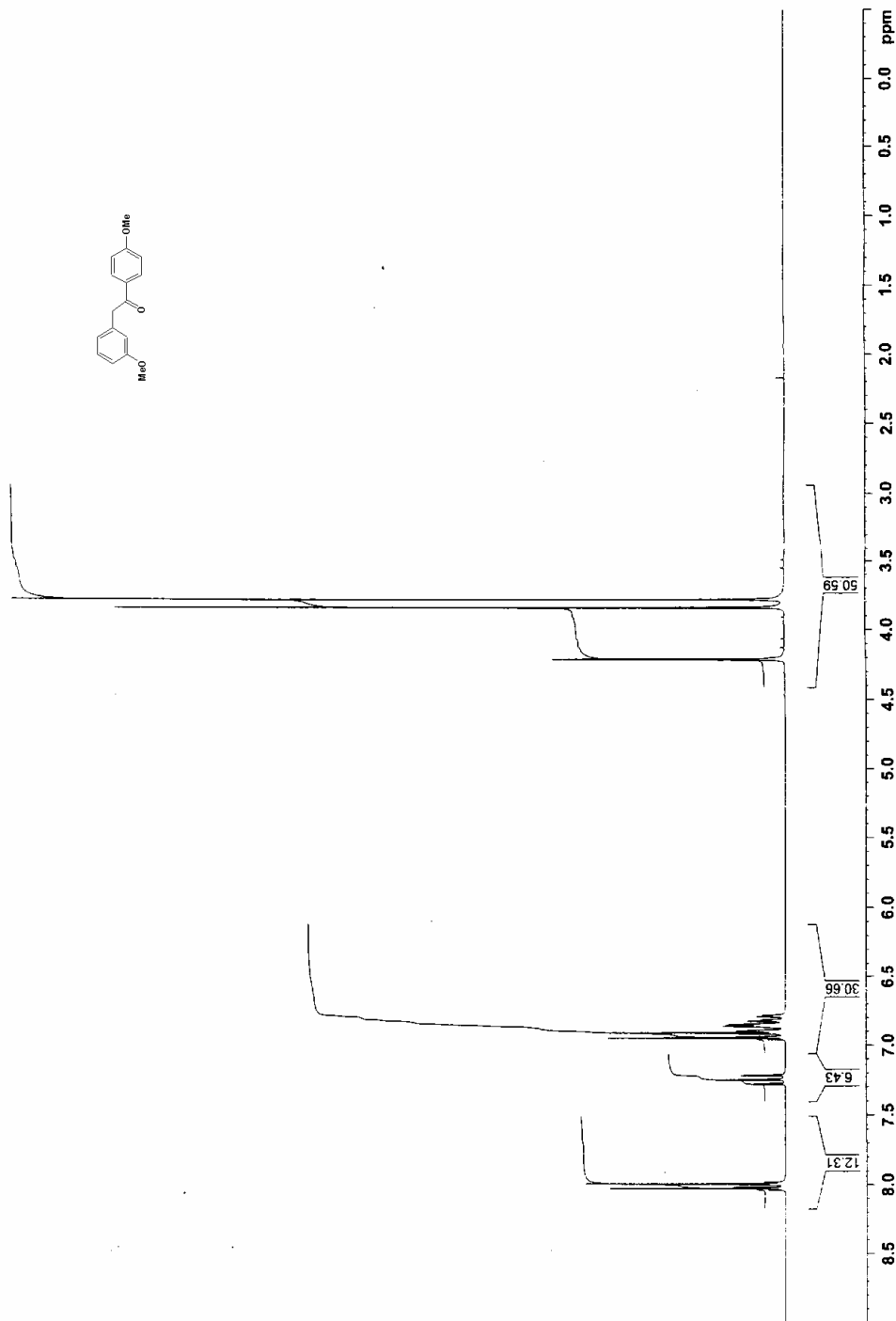
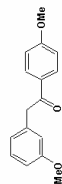


# <sup>1</sup>H NMR Spectrum of compound 8

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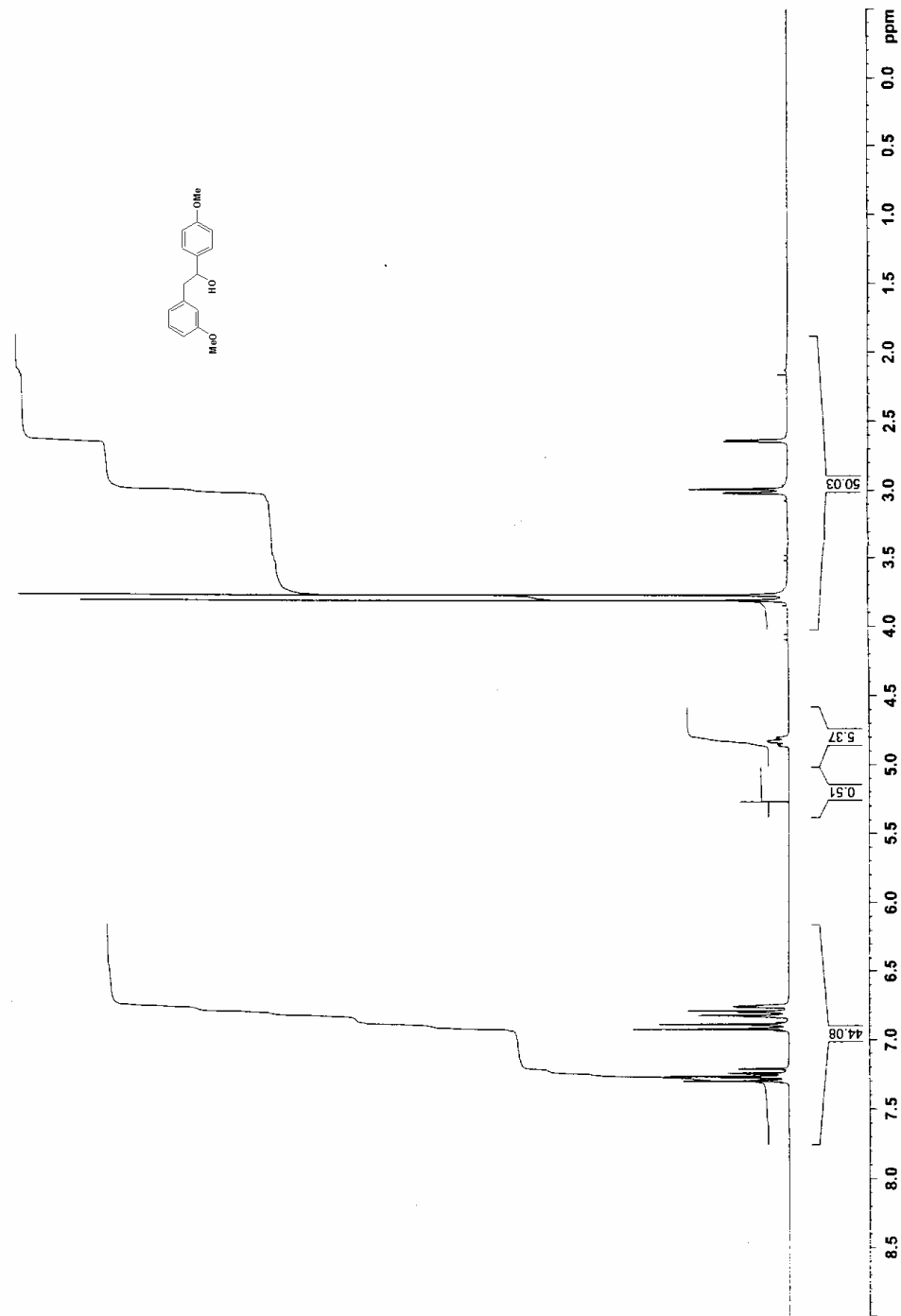


# <sup>1</sup>H NMR Spectrum of compound 7

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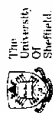


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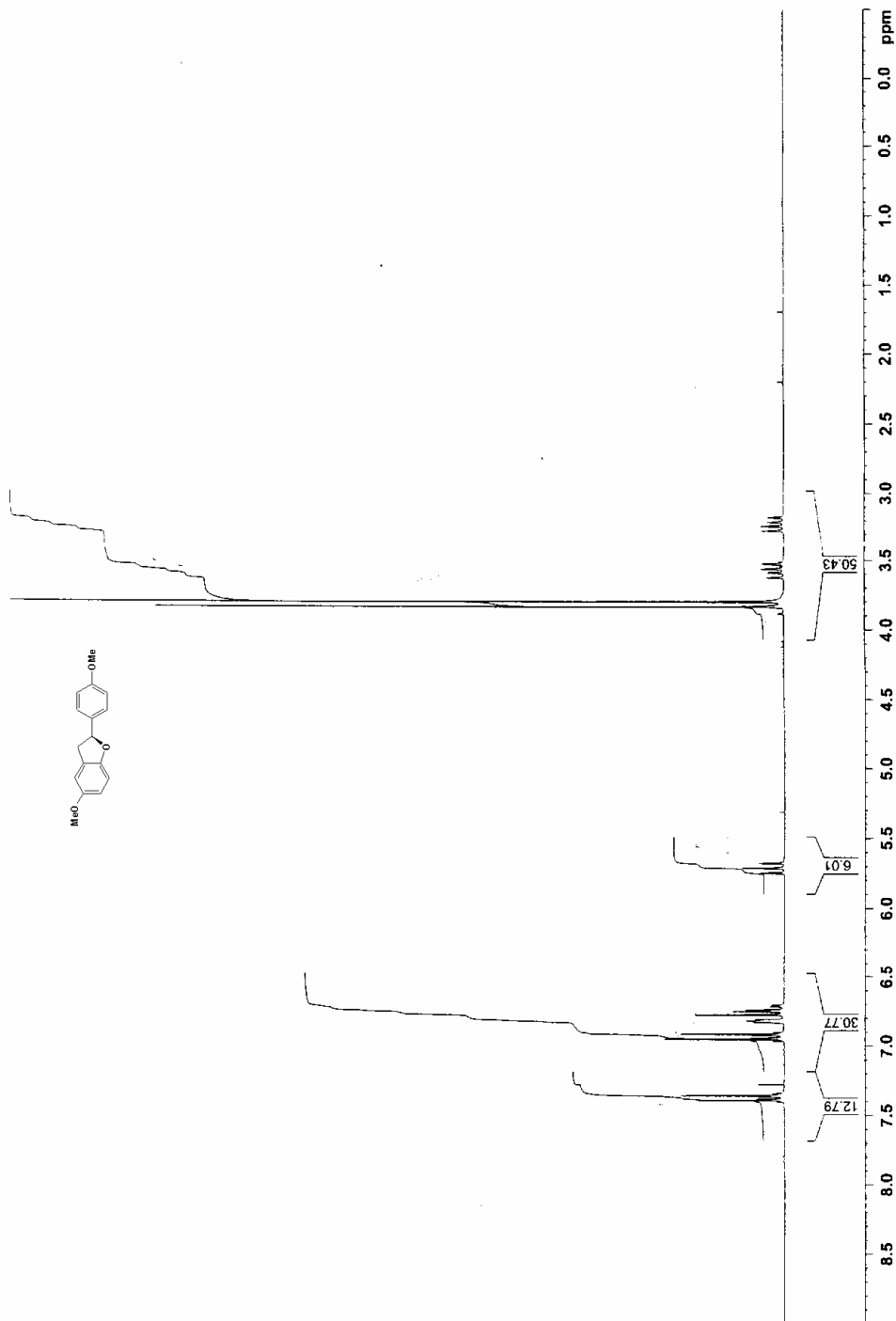
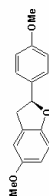


# <sup>1</sup>H NMR Spectrum of compound 1

RV171627 (Pure)  
 PRO CDCl3 (C:\NMRData) ch3sj 2

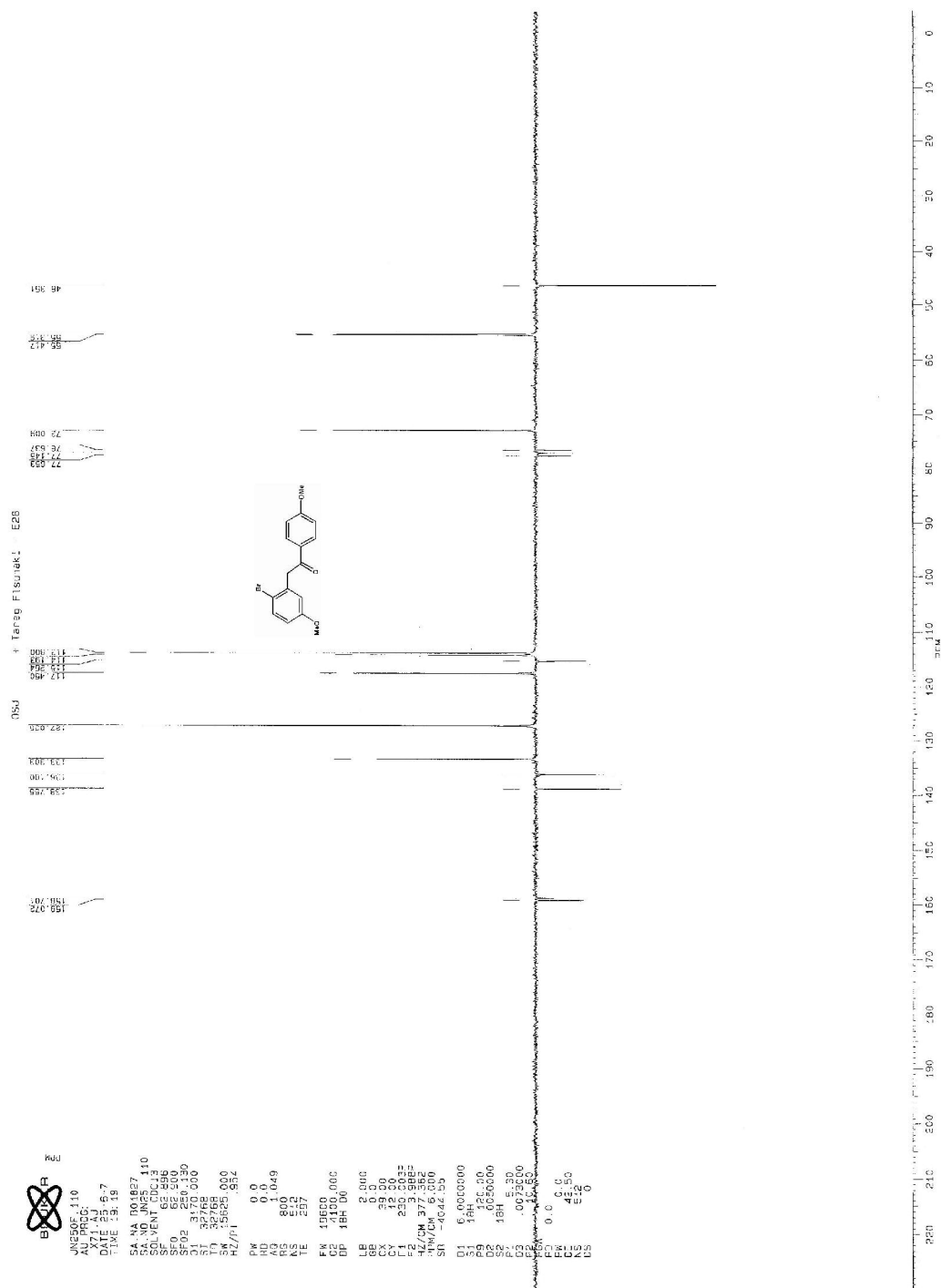


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 PROCNO 1  
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 Time 11.10  
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 US 4  
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 =====  
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 RDW 40  
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 GB 0  
 CB 0.00 Hz  
 PC 1.00





# <sup>13</sup>C NMR Spectrum of compound 3



# <sup>13</sup>C NMR Spectrum of compound 2

JMOD250PPM CDCl3 {C:\NMR\data\ ch3sj 44

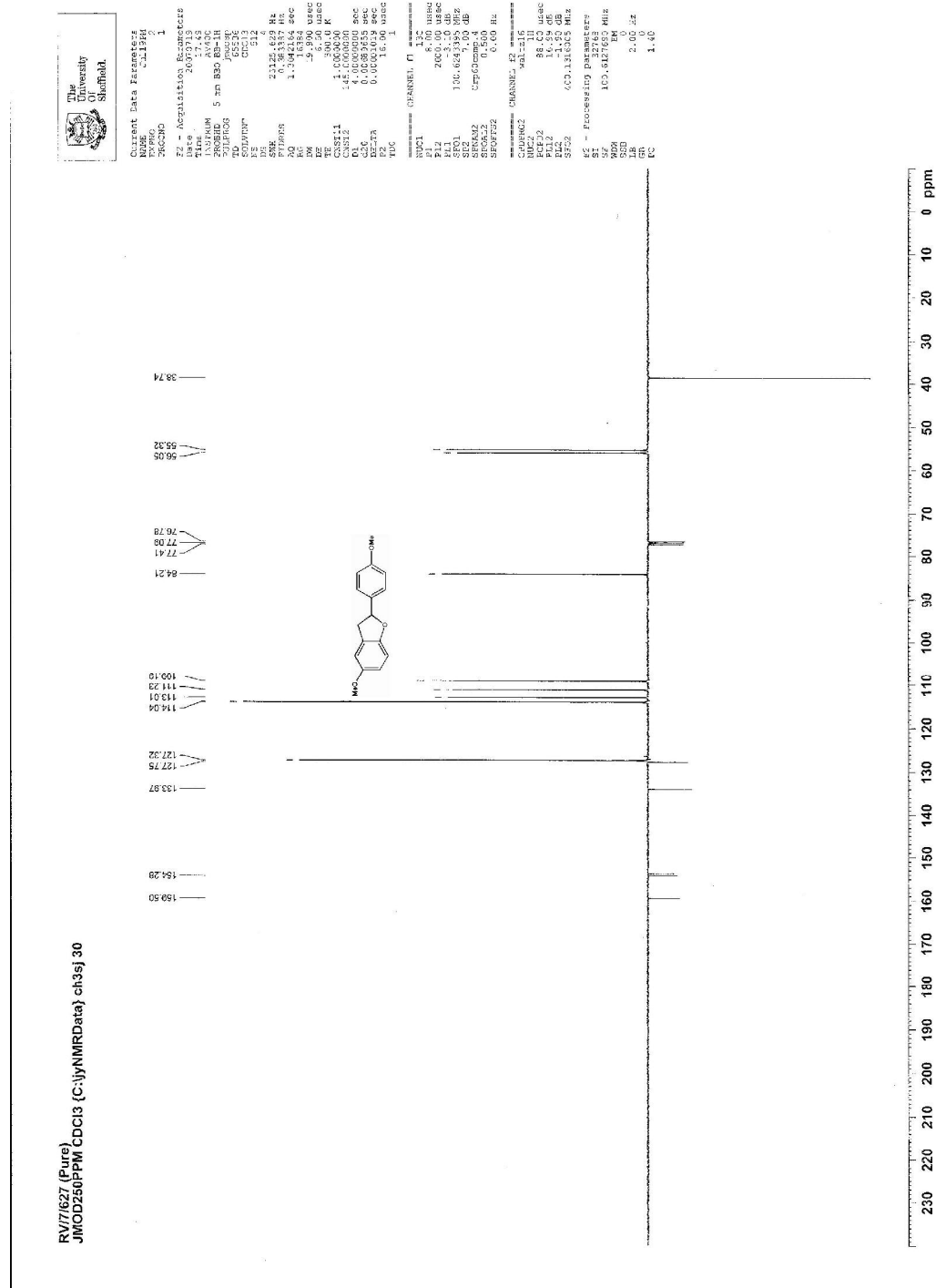


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 PD46: 0.00  
 PR46: 1.00  
 SFO46: 125.761350 MHz  
 NUC46: 13C  
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 PD47: 0.00  
 PR47: 1.00  
 SFO47: 125.761350 MHz  
 NUC47: 13C  
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 PD48: 0.00  
 PR48: 1.00  
 SFO48: 125.761350 MHz  
 NUC48: 13C  
 P49: 12.00  
 PD49: 0.00  
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 SFO49: 125.761350 MHz  
 NUC49: 13C  
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 PD50: 0.00  
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 P52: 12.00  
 PD52: 0.00  
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 SFO52: 125.761350 MHz  
 NUC52: 13C  
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 SFO53: 125.761350 MHz  
 NUC53: 13C  
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 SFO54: 125.761350 MHz  
 NUC54: 13C  
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 PD55: 0.00  
 PR55: 1.00  
 SFO55: 125.761350 MHz  
 NUC55: 13C  
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 PD56: 0.00  
 PR56: 1.00  
 SFO56: 125.761350 MHz  
 NUC56: 13C  
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 PD57: 0.00  
 PR57: 1.00  
 SFO57: 125.761350 MHz  
 NUC57: 13C  
 P58: 12.00  
 PD58: 0.00  
 PR58: 1.00  
 SFO58: 125.761350 MHz  
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 PD59: 0.00  
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 NUC59: 13C  
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 PD62: 0.00  
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 PD63: 0.00  
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 SFO63: 125.761350 MHz  
 NUC63: 13C  
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 PD64: 0.00  
 PR64: 1.00  
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 NUC64: 13C  
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 PD65: 0.00  
 PR65: 1.00  
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 PD66: 0.00  
 PR66: 1.00  
 SFO66: 125.761350 MHz  
 NUC66: 13C  
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 PD67: 0.00  
 PR67: 1.00  
 SFO67: 125.761350 MHz  
 NUC67: 13C  
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 PD68: 0.00  
 PR68: 1.00  
 SFO68: 125.761350 MHz  
 NUC68: 13C  
 P69: 12.00  
 PD69: 0.00  
 PR69: 1.00  
 SFO69: 125.761350 MHz  
 NUC69: 13C  
 P70: 12.00  
 PD70: 0.00  
 PR70: 1.00  
 SFO70: 125.761350 MHz  
 NUC70: 13C  
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 PD71: 0.00  
 PR71: 1.00  
 SFO71: 125.761350 MHz  
 NUC71: 13C  
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 PD72: 0.00  
 PR72: 1.00  
 SFO72: 125.761350 MHz  
 NUC72: 13C  
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 PD73: 0.00  
 PR73: 1.00  
 SFO73: 125.761350 MHz  
 NUC73: 13C  
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 PD74: 0.00  
 PR74: 1.00  
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 NUC74: 13C  
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 PD75: 0.00  
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 PD80: 0.00  
 PR80: 1.00  
 SFO80: 125.761350 MHz  
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 PD81: 0.00  
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 PD82: 0.00  
 PR82: 1.00  
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 NUC82: 13C  
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 PD83: 0.00  
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 SFO83: 125.761350 MHz  
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 PD85: 0.00  
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 PD86: 0.00  
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 PD87: 0.00  
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 PD88: 0.00  
 PR88: 1.00  
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 NUC88: 13C  
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 PD89: 0.00  
 PR89: 1.00  
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 PD90: 0.00  
 PR90: 1.00  
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 PD91: 0.00  
 PR91: 1.00  
 SFO91: 125.761350 MHz  
 NUC91: 13C  
 P92: 12.00  
 PD92: 0.00  
 PR92: 1.00  
 SFO92: 125.761350 MHz  
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 P93: 12.00  
 PD93: 0.00  
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 SFO93: 125.761350 MHz  
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 PD95: 0.00  
 PR95: 1.00  
 SFO95: 125.761350 MHz  
 NUC95: 13C  
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 PD96: 0.00  
 PR96: 1.00  
 SFO96: 125.761350 MHz  
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 PD97: 0.00  
 PR97: 1.00  
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 PD98: 0.00  
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 PR100: 1.00  
 SFO100: 125.761350 MHz  
 NUC100: 13C  
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 PD101: 0.00  
 PR101: 1.00  
 SFO101: 125.761350 MHz  
 NUC101: 13C  
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 PD102: 0.00  
 PR102: 1.00  
 SFO102: 125.761350 MHz  
 NUC102: 13C  
 P103: 12.00  
 PD103: 0.00  
 PR103: 1.00  
 SFO103: 125.761350 MHz  
 NUC103: 13C  
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 PD104: 0.00  
 PR104: 1.00  
 SFO104: 125.761350 MHz  
 NUC104: 13C  
 P105: 12.00  
 PD105: 0.00  
 PR105: 1.00  
 SFO105: 125.761350 MHz  
 NUC105: 13C  
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 PD106: 0.00  
 PR106: 1.00  
 SFO106: 125.761350 MHz  
 NUC106: 13C  
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 PD107: 0.00  
 PR107: 1.00  
 SFO107: 125.761350 MHz  
 NUC107: 13C  
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 PD108: 0.00  
 PR108: 1.00  
 SFO108: 125.761350 MHz  
 NUC108: 13C  
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 PD109: 0.00  
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 SFO109: 125.761350 MHz  
 NUC109: 13C  
 P110: 12.00  
 PD110: 0.00  
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 SFO110: 125.761350 MHz  
 NUC110: 13C  
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 PD111: 0.00  
 PR111: 1.00  
 SFO111: 125.761350 MHz  
 NUC111: 13C  
 P112: 12.00  
 PD112: 0.00  
 PR112: 1.00  
 SFO112: 125.761350 MHz  
 NUC112: 13C  
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 PD113: 0.00  
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 SFO113: 125.761350 MHz  
 NUC113: 13C  
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 PD115: 0.00  
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 NUC115: 13C  
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 PR116: 1.00  
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 PD118: 0.00  
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 SFO143: 125.761350 MHz  
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 PD144: 0.00  
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 SFO144: 125.761350 MHz  
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 PD145: 0.00  
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 SFO159: 125.761350 MHz  
 NUC159: 13C  
 P160: 12.00  
 PD160: 0.00





# <sup>13</sup>C NMR Spectrum of compound 1



# HPLC Chromatogram of (S)-2 crude reaction mixture

27/06/2007 16:16  
 Created : 27/06/2007 14:59:56  
 Project : Work3  
 Sample ID : RV77612(rc)  
 Calibration : RV-7-612(rc)

Method : FMG  
 Description :  
 Created : 24/08/2004 16:25

Column : Kromasil 3-CelluCoat  
 Mobile Phase : 8% IPA : Hex  
 Flow Rate : 1.0 mL/min  
 Note :

Autostop : 20.00, min  
 Detector 1 : Signal 1  
 Subtraction chromatogram : (None)

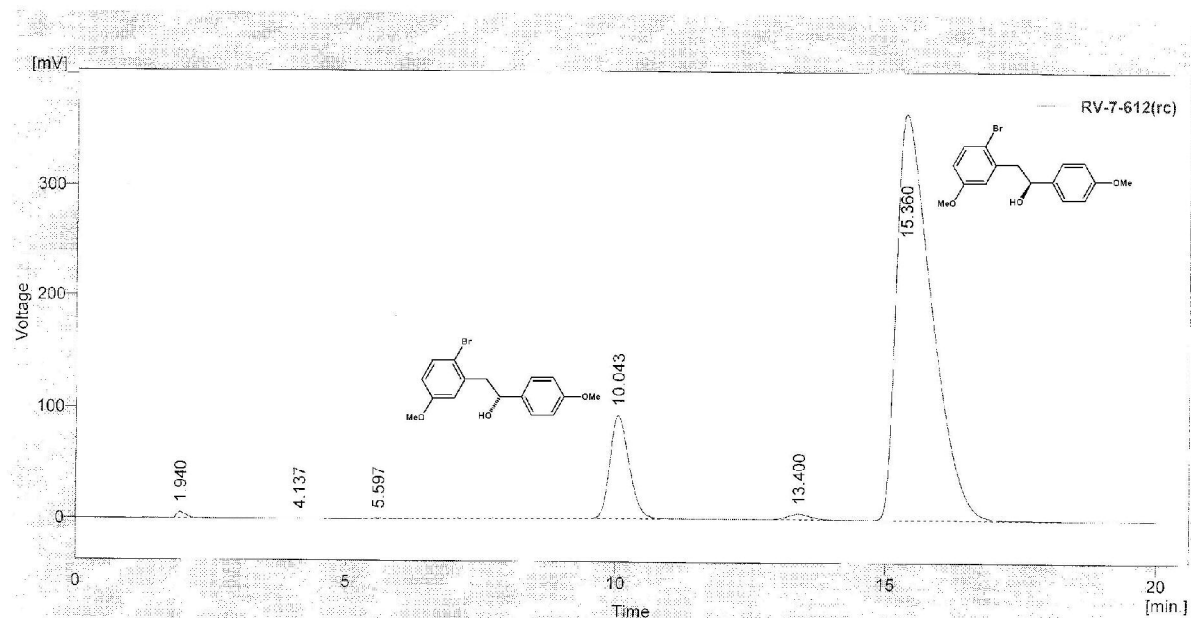
Chromatogram RV-7-612(rc) Page 1 of 1

By : Clarity  
 Report Style : Full  
 Sample : (1S)(2R) BOMe, (rc)  
 Chromatogram : DATA\RV-7-612(rc)

By : Clarity  
 Modified : 27/06/2007 14:59

Detection : UV 254nm  
 Temperature : RT  
 Pressure :

External Start : Start Only, Down  
 Range 1 : Bipolar, 10000 mV, 10 Samp. per Sec.  
 Matching : No Change



Result Table - Calculation Method Uncal

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	1.940	59.221	5.386	0.3	1.1	0.19
2	4.137	8.736	0.212	4.6e-02	4.5e-02	0.18
3	5.597	11.128	0.604	0.1	0.1	0.29
4	10.043	2370.023	92.884	12.4	19.8	0.38
5	13.400	154.470	5.090	0.8	1.1	0.46
6	15.360	16535.640	365.795	86.4	77.8	0.70
	Total	19139.219	469.971	100.0	100.0	

# HPLC Chromatogram of (R)-2 crude reaction mixture

26/06/2007 16:12

Chromatogram RV-7-612

Page 1 of 1

Created : 26/06/2007 15:59:37  
 Project : Work3  
 Sample ID : RV/7/612  
 Calibration : RV-7-612

By : Clarity  
 Report Style : Full  
 Sample : (1S)-(2R) B-OMe, crude  
 Chromatogram : DATA\RV-7-612

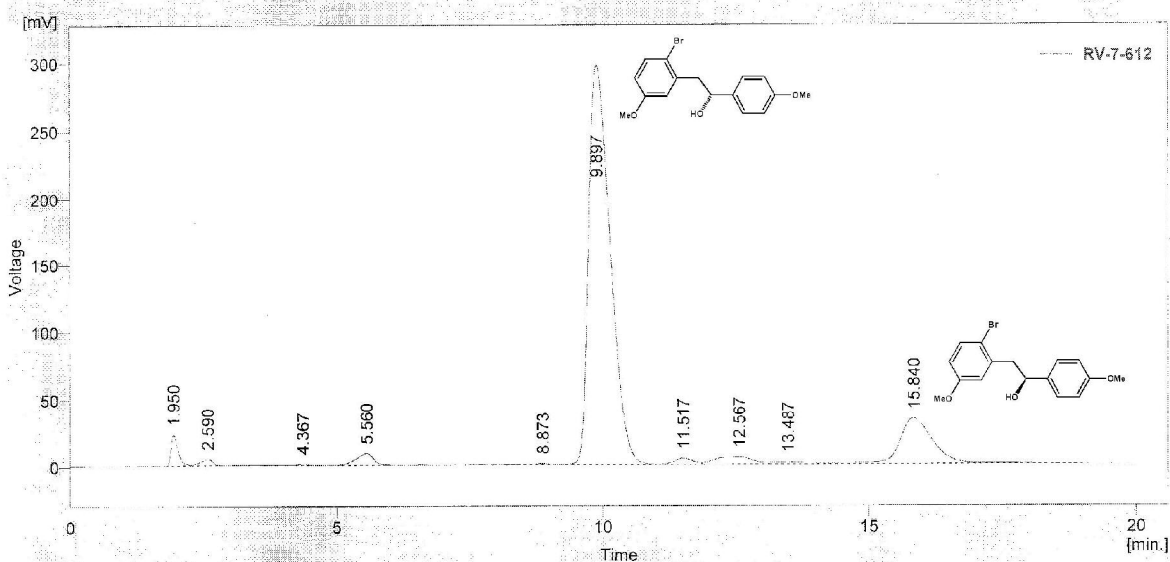
Method : FMG  
 Description :  
 Created : 24/08/2004 16:25  
 Column : Kromasil 3-CelluCoat  
 Mobile Phase : 8%IPA : Hex  
 Flow Rate : 1.0 mL/min  
 Note :

By : Clarity  
 Modified : 26/06/2007 15:59

Detection : UV 254nm  
 Temperature : RT  
 Pressure :

Autostop : 20.00, min  
 Detector 1 : Signal 1  
 Subtraction chromatogram : (None)

External Start : Start Only, Down  
 Range 1 : Bipolar, 10000 mV, 10 Samp. per Sec.  
 Matching : No Change



Result Table - Calculation Method Uncal

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	1.950	209.153	22.952	1.9	6.0	0.14
2	2.590	75.363	4.689	0.7	1.2	0.26
3	4.367	12.546	1.097	0.1	0.3	0.17
4	5.560	200.450	8.679	1.8	2.3	0.36
5	8.873	25.522	0.969	0.2	0.3	0.34
6	9.897	8331.482	296.745	76.8	77.9	0.45
7	11.517	116.325	4.587	1.1	1.2	0.40
8	12.567	271.861	5.559	2.5	1.5	0.81
9	13.487	87.075	1.749	0.8	0.5	0.92
10	15.840	1520.793	33.914	14.0	8.9	0.62
	Total	10850.569	380.939	100.0	100.0	

# GC Chromatogram of Synthetic (S)-1

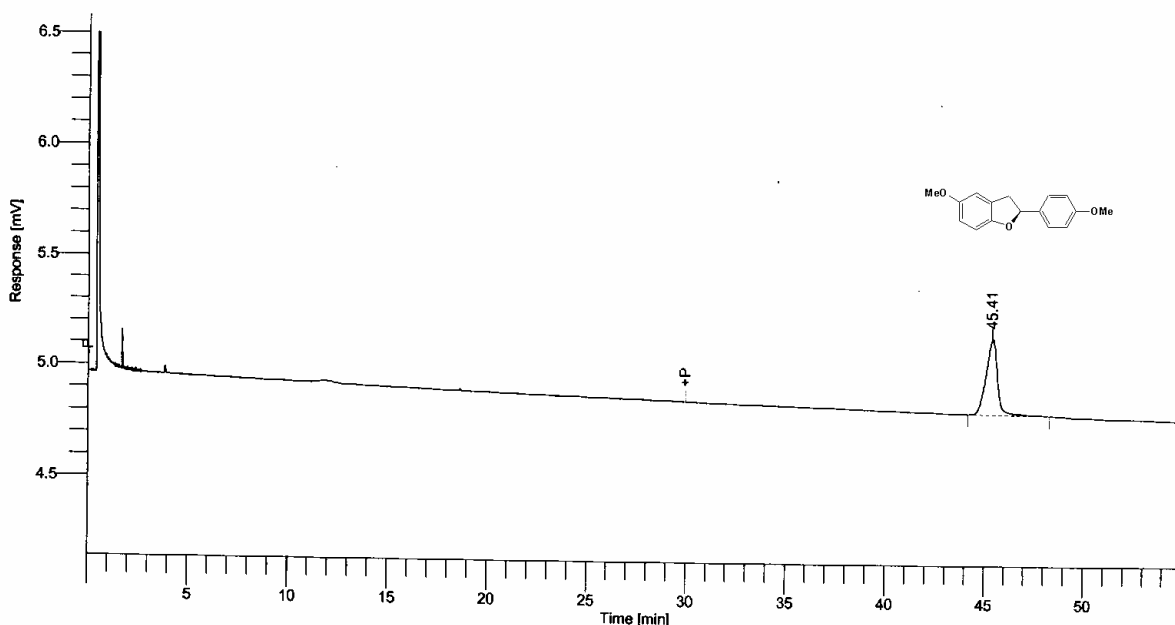
```

Software Version : 6.2.0.0.0:B27
Reprocess Number : gc_server: 13067
Operator : chp04rv
Sample Number : 002
AutoSampler : BUILT-IN
Instrument Name : AutoSystemFID
Instrument Serial # : None
Delay Time : 0.00 min
Sampling Rate : 6.2500 pts/s
Sample Volume : 1.000000
Sample Amount : 1.0000
Data Acquisition Time : 1/21/08 2:53:53 PM

Date : 1/21/08 3:49:00 PM
Sample Name : RV/8/705
Study :
Rack/Vial : 0/2
Channel : A
A/D mV Range : 1000
End Time : 55.00 min
Area Reject : 0.000000
Dilution Factor : 1.00
Cycle : 2
    
```

```

Raw Data File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.raw
Result File : \\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Inst Method : \\gc_server\TCData\AutoSystemFID\ Methods_Reports\Cyclodextrin H from
\\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.raw
Proc Method : \\gc_server\TCData\AutoSystemFID\ Methods_Reports\Cyclodextrin H from
\\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Calib Method : \\gc_server\TCData\AutoSystemFID\ Methods_Reports\Cyclodextrin H from
\\gc_server\TCData\AutoSystemFID\2008Jan\21_01_2008_chp04rv_002.rst
Report Format File : \\gc_server\TCData\AutoSystemFID\ Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID\ Sequence_Index\RVemula\RV-7-643 race,8-705,real sample.seq
    
```



## DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		45.411	13802.93	337.63	100.00	100.00			BB	0.0138	0.0138
			13802.93	337.63	100.00	100.00				0.0138	0.0138

# GC Chromatogram of (R)-1

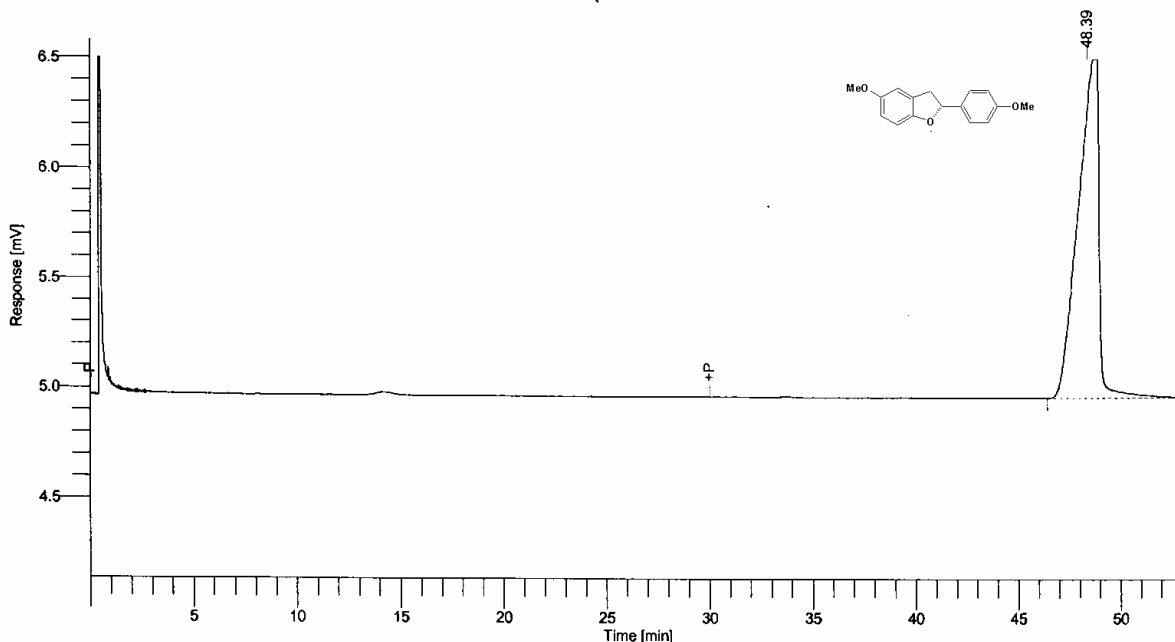
```

Software Version   : 6.2.0.0.0:B27
Reprocess Number  : gc_server: 12544
Operator          : chp04rv
Sample Number     : 003
AutoSampler       : BUILT-IN
Instrument Name    : AutoSystemFID
Instrument Serial # : None
Delay Time        : 0.00 min
Sampling Rate     : 6.2500 pts/s
Sample Volume     : 1.000000
Sample Amount     : 1.0000
Data Acquisition Time : 8/17/07 12:20:08 PM

Date              : 8/17/07 1:13:15 PM
Sample Name       : RV/7637
Study             :
Rack/Vial         : 0/12
Channel           : A
A/D mV Range     : 1000
End Time         : 53.00 min
Area Reject       : 0.000000
Dilution Factor  : 1.00
Cycle            : 2
    
```

```

Raw Data File : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.raw
Result File   : \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
Inst Method   : \\gc_server\TCData\AutoSystemFID\Methods_Reports\Cyclodextrin H from
               \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.raw
Proc Method   : \\gc_server\TCData\AutoSystemFID\Methods_Reports\Cyclodextrin H from
               \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
Calib Method  : \\gc_server\TCData\AutoSystemFID\Methods_Reports\Cyclodextrin H from
               \\gc_server\TCData\AutoSystemFID\2007Aug\17_08_2007_chp04rv_003.rst
Report Format File : \\gc_server\TCData\AutoSystemFID\Methods_Reports\Cyclodextrin H.rpt
Sequence File : \\gc_server\TCData\AutoSystemFID\Sequence_Index\RVemula\RV-7-643.seq
    
```



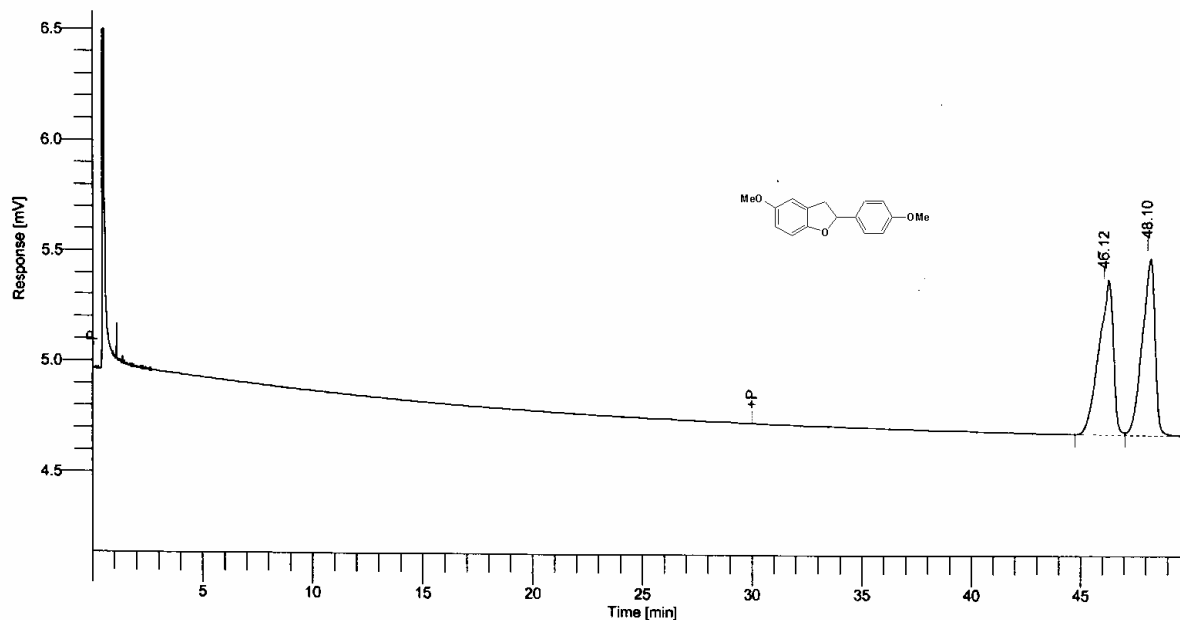
## DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		48.386	111854.09	1305.89	100.00	100.00			BB	0.1119	0.1119
			111854.09	1305.89	100.00	100.00				0.1119	0.1119

# GC Chromatogram of racemic corsifuran 1

Software Version : 6.2.0.0.0:B27	Date : 8/17/07 11:12:33 AM
Reprocess Number : gc_server: 12542	
Operator : chp04rv	Sample Name : RV/7/643
Sample Number : 001	Study :
AutoSampler : BUILT-IN	Rack/Vial : 0/10
Instrument Name : AutoSystemFID	Channel : A
Instrument Serial # : None	A/D mV Range : 1000
Delay Time : 0.00 min	End Time : 50.00 min
Sampling Rate : 6.2500 pts/s	
Sample Volume : 1.000000	Area Reject : 0.000000
Sample Amount : 1.0000	Dilution Factor : 1.00
Data Acquisition Time : 8/17/07 10:00:52 AM	Cycle : 1

Raw Data File : \\gc\_server\TCData\AutoSystemFID\2007Aug\17\_08\_2007\_chp04rv\_001.raw  
 Result File : \\gc\_server\TCData\AutoSystemFID\2007Aug\17\_08\_2007\_chp04rv\_001-20070817-111232.rst  
 Inst Method : \\gc\_server\TCData\AutoSystemFID\Methods\_Reports\Cyclodextrin H from  
 \\gc\_server\TCData\AutoSystemFID\2007Aug\17\_08\_2007\_chp04rv\_001.raw  
 Proc Method : \\gc\_server\TCData\AutoSystemFID\Methods\_Reports\Cyclodextrin H.mth from  
 \\gc\_server\TCData\AutoSystemFID\2007Aug\17\_08\_2007\_chp04rv\_001-20070817-111232.rst  
 Calib Method : \\gc\_server\TCData\AutoSystemFID\Methods\_Reports\Cyclodextrin H.mth from  
 \\gc\_server\TCData\AutoSystemFID\2007Aug\17\_08\_2007\_chp04rv\_001-20070817-111232.rst  
 Report Format File : \\gc\_server\TCData\AutoSystemFID\Methods\_Reports\Cyclodextrin H.rpt  
 Sequence File : \\gc\_server\TCData\AutoSystemFID\Sequence\_Index\RVemula\RV-7-643-20070817-094637.idx



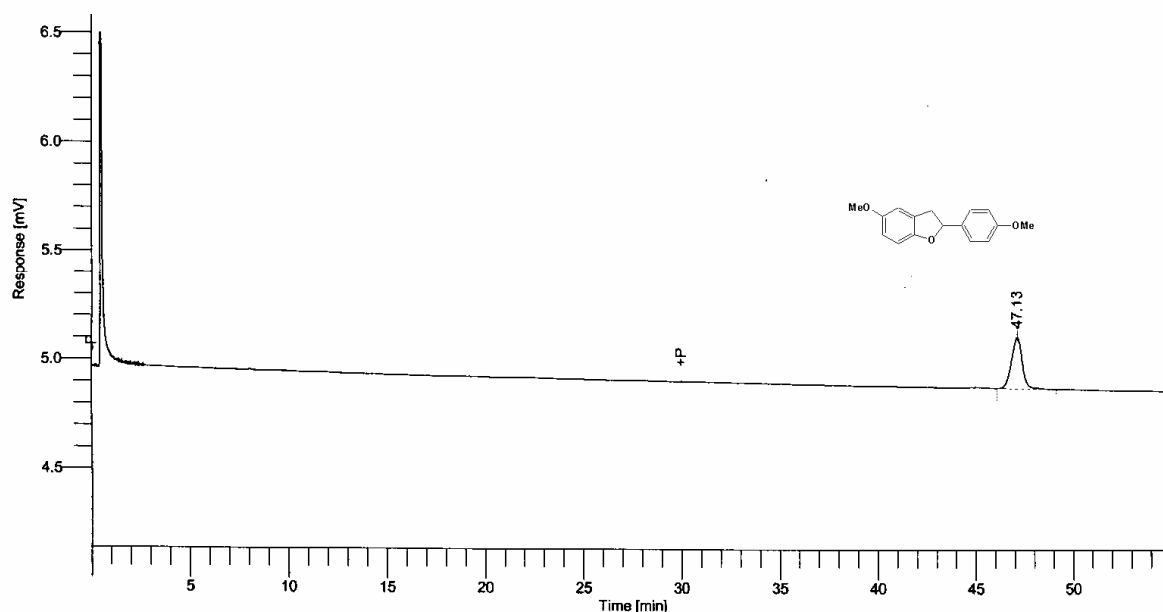
## DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		46.117	32352.73	544.61	49.53	49.53			BV	0.0324	0.0324
2		48.103	32966.69	684.56	50.47	50.47			VB	0.0330	0.0330
			65319.42	1229.17	100.00	100.00				0.0653	0.0653

# GC Chromatogram of natural corsifuran 1

Software Version : 6.2.0.0.0:B27	Date : 1/21/08 4:50:05 PM
Reprocess Number : gc_server: 13069	
Operator : chp04rv	Sample Name : RV/8/Real sample
Sample Number : 003	Study :
AutoSampler : BUILT-IN	Rack/Vial : 0/3
Instrument Name : AutoSystemFID	Channel : A
Instrument Serial # : None	A/D mV Range : 1000
Delay Time : 0.00 min	End Time : 55.00 min
Sampling Rate : 6.2500 pts/s	
Sample Volume : 1.000000	Area Reject : 0.000000
Sample Amount : 1.0000	Dilution Factor : 1.00
Data Acquisition Time : 1/21/08 3:54:59 PM	Cycle : 3

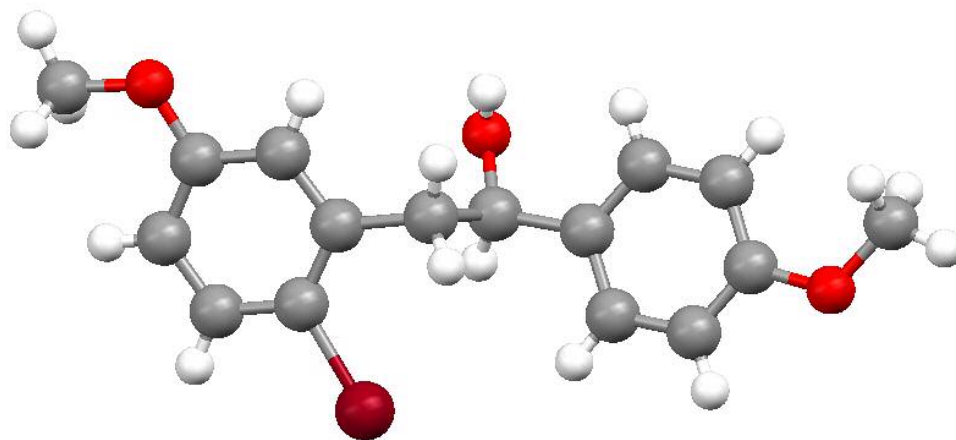
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 Result File : \\gc\_server\TCData\AutoSystemFID\2008Jan\21\_01\_2008\_chp04rv\_003.rst  
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 \\gc\_server\TCData\AutoSystemFID\2008Jan\21\_01\_2008\_chp04rv\_003.raw  
 Proc Method : \\gc\_server\TCData\AutoSystemFID\ Methods\_Reports\Cyclodextrin H from  
 \\gc\_server\TCData\AutoSystemFID\2008Jan\21\_01\_2008\_chp04rv\_003.rst  
 Calib Method : \\gc\_server\TCData\AutoSystemFID\ Methods\_Reports\Cyclodextrin H from  
 \\gc\_server\TCData\AutoSystemFID\2008Jan\21\_01\_2008\_chp04rv\_003.rst  
 Report Format File : \\gc\_server\TCData\AutoSystemFID\ Methods\_Reports\Cyclodextrin H.rpt  
 Sequence File : \\gc\_server\TCData\AutoSystemFID\ Sequence\_Index\RVemula\RV-7-643 race,8-705,real sample.seq



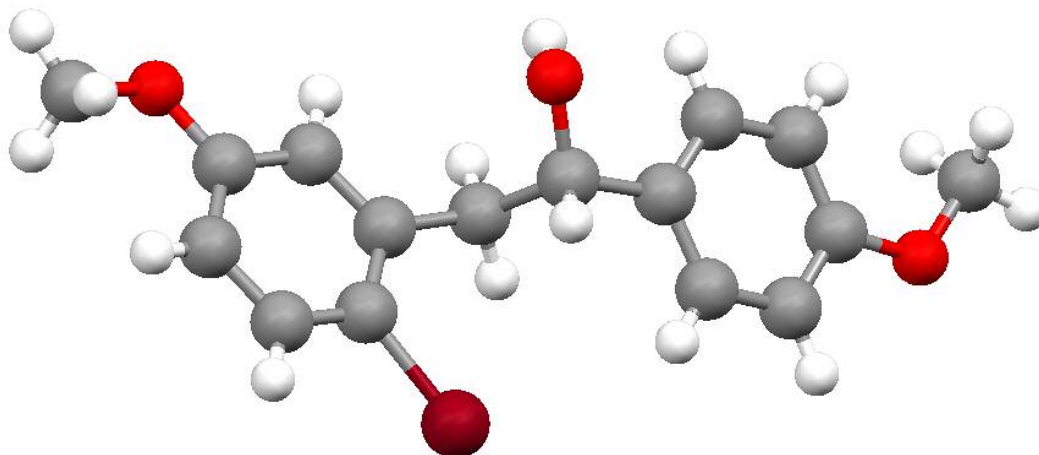
## DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		47.132	9153.96	234.26	100.00	100.00			BB	0.0092	0.0092
			9153.96	234.26	100.00	100.00				0.0092	0.0092

### X-Ray Structure of (R)-1



### X-Ray Structure of (S)-1



### References

- <sup>1</sup> Lebegue, N.; Bethegnies, G.; Berthelot, P. *Synth. Comm.* **2004**, *34*, 1041-1048.
- <sup>2</sup> Percec, V.; Zuber, M. *J. Polym. Sci., A: Polymer Chemistry* **1992**, *30*, 997-1016
- <sup>3</sup> Napolitano, E.; Ramacciotti, A.; Fiaschi, R. *Gazz. Chem. Ital.* **1988**, *118*, 101-103.
- <sup>4</sup> Personal communication; von Reuß, S. H. Institute Für Organische Chemie, Universität Hamburg. Martin-Luther-King-Platz 6, D-29146 Hamburg, Germany