

***Supporting Information***

**Efficient Synthesis of Chiral  $\beta$ -Hydroxy Sulfones via Iridium-Catalyzed  
Hydrogenation**

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**Contents**

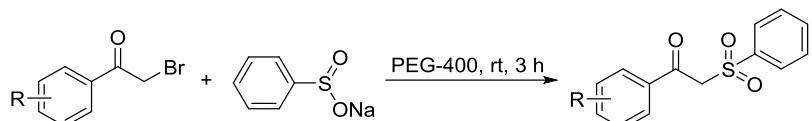
|                                                                    |    |
|--------------------------------------------------------------------|----|
| 1. General remarks .....                                           | 2  |
| 2. General procedure for synthesis of $\beta$ -keto sulfones ..... | 2  |
| 3. General procedure for asymmetric hydrogenation .....            | 3  |
| 4. NMR spectra .....                                               | 12 |
| 5. HPLC spectra .....                                              | 31 |
| 6. Reference .....                                                 | 67 |

## 1. General remarks

All reactions and manipulations which are sensitive to moisture or air were performed in an argon-filled glovebox or using standard Schlenk techniques. Hydrogen gas (99.999%) was purchased from Shanghai Regulator Factory Co., Ltd. Anhydrous hexane, THF, 1,4-dioxane and toluene was distilled from sodium benzophenone ketyl. Anhydrous *i*-PrOH, DCE, CHCl<sub>3</sub>, CH<sub>2</sub>Cl<sub>2</sub> were freshly distilled from calcium hydride. Anhydrous MeOH and EtOH were freshly distilled from Mg. Solvents were transferred by syringe. [Ir(COD)Cl]<sub>2</sub> was prepared according to the literature. <sup>1</sup>H and <sup>13</sup>C spectra were recorded with a Bruker ADVANCE III (400 MHz) spectrometer with CDCl<sub>3</sub> as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in parts per million (ppm,  $\delta$  scale) downfield from TMS at 0.00 ppm and referenced to the CDCl<sub>3</sub> at 7.26 ppm (for <sup>1</sup>H NMR) or 77.00 ppm (for <sup>13</sup>C NMR). Data are reported as: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant in hertz (Hz) and signal area integration in natural numbers. <sup>13</sup>C NMR analyses were run with decoupling. Optical rotations  $[\alpha]_D$  were determined using a PERKIN ELMER polarimeter 343 instrument. HPLC analyses were performed using Daicel chiral column on an Agilent 1260 Series HPLC instrument.

## 2. General procedure for synthesis of $\beta$ -keto sulfones

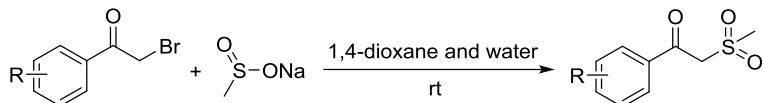
### Method A:



In a 150 mL flask, bromoacetophenone (3.0 mmol) and sodium phenylsulfite (3.3 mmol) were added to the flask, and then 30 mL polyethylene glycol 400 was poured into the flask and stirred for 3 h at room temperature. The reaction system was extracted with ethyl acetate three times, then washed with water to remove polyethylene glycol three times, and then dried with anhydrous sodium sulfate. Then the reaction mixture was concentrated under reduced pressure, and the resulting residue was separated by column chromatography (petroleum ether:EtOAc = 6:1) to give the desired products.

The product can be further purified by recrystallization from dichloromethane.

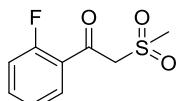
**Method B:**



Bromoacetophenone (3.0 mmol) was added to a 150 mL flask, and then 20 mL 1,4-dioxane was added to dissolve it. Then 20 mL water solution of sodium methylene sulfite was added to the flask, and the reaction system was reacted at room temperature for 3 h. 1,4-Dioxane in the reaction system was removed by rotary evaporator, then a small amount of water was added, and then dichloromethane was used to extract the reaction system. Then the reaction mixture was concentrated under reduced pressure, and the resulting residue was separated by column chromatography (petroleum ether:EtOAc = 6:1) to give the desired products. The product can be further purified by recrystallization from dichloromethane.

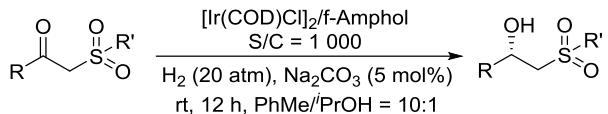
Substrates **1a-1j**, **1q-1r** were prepared through method A.<sup>[1]</sup> Substrates **1k-1p** were prepared through method B.<sup>[2]</sup> The absolute configuration of product **2a** was determined by comparison of analytical data (optical rotation) with the literature.<sup>[3-4]</sup> The absolute configuration of others were assigned by analogy.

**1-(2-fluorophenyl)-2-(methylsulfonyl)ethanone **1l****



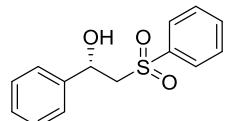
White solid, 476.3 mg, 73% yield; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.94 (td, *J* = 7.7, 1.9 Hz, 1H), 7.67-7.61 (m, 1H), 7.32-7.28 (m, 1H), 7.23-7.18 (m, 1H), 4.68 (s, 2H), 3.18 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 187.1 (d, *J* = 3.0 Hz), 162.1 (d, *J* = 254.0 Hz), 136.6 (d, *J* = 10.0 Hz), 131.1 (d, *J* = 1.0 Hz), 125.0 (d, *J* = 4.0 Hz), 124.2 (d, *J* = 11.0 Hz), 117.0 (d, *J* = 24.0 Hz), 64.9 (d, *J* = 9.0 Hz), 42.3.

**3. General procedure for asymmetric hydrogenation**



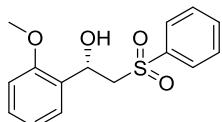
General procedure (at S/C = 1 000): To a 4.0 mL vial was added the catalyst precursor  $[\text{Ir}(\text{COD})\text{Cl}]_2$  (1.4 mg,  $2.0 \times 10^{-3}$  mmol), ligand **L4** (3.3 mg,  $4.2 \times 10^{-3}$  mmol) and anhydrous isopropanol (2.0 mL) in the argon-filled glovebox. The mixture was stirred for 2.0 h at 25 °C giving orange red solution. And then 0.2 mmol  $\beta$ -keto sulfones,  $\text{Na}_2\text{CO}_3$  (1.06 mg, 0.01 mmol) were added into a 5 mL hydrogenation vessel. 1.0 mL anhydrous toluene was added as solvent and a solution of Ir/f-Amphol **L4** in anhydrous isopropanol (100  $\mu\text{L}$ ) was added via an injection port. Then the vessel was placed in an autoclave, closed it and moved it out from glovebox. The autoclave quickly purged with hydrogen gas for three times, then pressurized to 20 atm  $\text{H}_2$ . The reaction solution was stirred at room temperature until for 12 h, then released pressure carefully. The solution of reaction mixture was purified by a flash chromatography on a silical gel with ethyl acetate and the solvent was removed under reduced pressure. The product was analyzed by NMR spectroscopy for conversion and chiral HPLC for ee values.

**(S)-1-phenyl-2-(phenylsulfonyl)ethan-1-ol **2a****



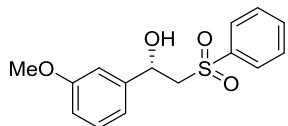
White solid, 99% yield, 51.9 mg; 95% ee;  $[\alpha]_D^{25} = +27.2$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (minor) = 31.6 min,  $t_R$  (major) = 33.6 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99–7.96 (m, 2H), 7.72–7.68 (m, 1H), 7.63–7.58 (m, 2H), 7.35–7.28 (m, 5H), 5.30–5.27 (m, 1H), 3.68 (d,  $J = 2.1$  Hz, 1H), 3.54–3.33 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  140.6, 139.1, 134.1, 129.5, 128.8, 128.4, 128.0, 125.6, 68.4, 63.9.

**(S)-1-(2-methoxyphenyl)-2-(phenylsulfonyl)ethan-1-ol **2b****



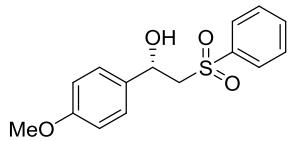
S/C = 500, 0.1 mmol substrate,  $\text{Na}_2\text{CO}_3$  (10 mol%). White solid, 99% yield, 28.9 mg; >99% ee;  $[\alpha]_D^{25} = +24.3$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chirapak AD-H column, 210 nm, 20 °C, *n*-hexane: *i*-PrOH = 90:10; flow 1.0 mL/min;  $t_R$  (minor) = 34.6 min,  $t_R$  (major) = 41.0 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97–7.95 (m, 2H), 7.70–7.66 (m, 1H), 7.61–7.57 (m, 2H), 7.49–7.46 (m, 1H), 7.25–7.21 (m, 1H), 6.99–6.95 (m, 1H), 6.76–6.73 (m, 1H), 5.35–5.32 (m, 1H), 3.70 (d,  $J = 4.0$  Hz, 1H), 3.60 (s, 3H), 3.58–3.40 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.2, 139.2, 133.7, 129.2, 129.0, 128.3, 128.1, 126.4, 120.9, 110.1, 64.4, 61.9, 55.0.

**(S)-1-(3-methoxyphenyl)-2-(phenylsulfonyl)ethan-1-ol 2c**



White solid, 98% yield, 57.3 mg; 96% ee;  $[\alpha]_D^{25} = +17.4$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (minor) = 34.5 min,  $t_R$  (major) = 43.9 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98–7.95 (m, 2H), 7.72–7.68 (m, 1H), 7.62–7.58 (m, 2H), 7.23 (t,  $J = 7.9$  Hz, 1H), 6.88–6.79 (m, 3H), 5.27–5.25 (m, 1H), 3.78 (s, 3H), 3.67 (d,  $J = 2.2$  Hz, 1H), 3.53–3.32 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.9, 142.2, 139.1, 134.1, 129.8, 129.5, 128.0, 117.8, 113.9, 111.1, 68.3, 63.9, 55.3.

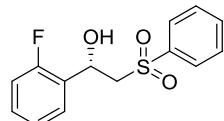
**(S)-1-(4-methoxyphenyl)-2-(phenylsulfonyl)ethan-1-ol 2d**



White solid, 99% yield, 57.8 mg; 98% ee;  $[\alpha]_D^{25} = +13.9$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel AD-H column, 210 nm, 20

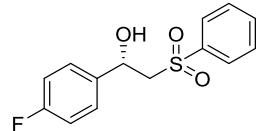
°C, *n*-hexane: *i*-PrOH = 90:10; flow 0.5 mL/min; *t*<sub>R</sub> (major) = 132.5 min, *t*<sub>R</sub> (minor) = 141.1 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.97–7.95 (m, 2H), 7.71–7.67 (m, 1H), 7.62–7.57 (m, 2H), 7.23–7.20 (m, 2H), 6.86–6.83 (m, 2H), 5.25–5.21 (m, 1H), 3.78 (s, 3H), 3.61 (d, *J* = 2.0 Hz, 1H), 3.54–3.30 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 159.5, 139.2, 134.1, 132.7, 129.4, 128.0, 127.0, 114.1, 68.1, 63.9, 55.3.

**(*S*)-1-(2-fluorophenyl)-2-(phenylsulfonyl)ethan-1-ol **2e****



White solid, 97% yield, 54.4 mg; 90% ee; [α]<sub>D</sub><sup>25</sup> = +6.6 (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; *t*<sub>R</sub> (major) = 25.3 min, *t*<sub>R</sub> (minor) = 31.4 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98–7.95 (m, 2H), 7.72–7.68 (m, 1H), 7.62–7.53 (m, 3H), 7.26–7.24 (m, 1H), 7.18–7.14 (m, 1H), 6.97–6.92 (m, 1H), 5.46–5.45 (m, 1H), 3.84 (d, *J* = 2.7 Hz, 1H), 3.50–3.48 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 159.0 (d, *J* = 244.0 Hz), 138.8, 134.1, 129.8 (d, *J* = 8.0 Hz), 129.4, 128.0, 127.5 (d, *J* = 12.0 Hz), 127.3 (d, *J* = 4.0 Hz), 124.6 (d, *J* = 4.0 Hz), 115.3 (d, *J* = 21.0 Hz), 63.0 (d, *J* = 3.0 Hz), 62.2 (d, *J* = 1.0 Hz).

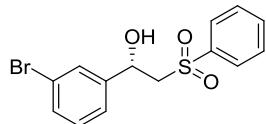
**(*S*)-1-(4-fluorophenyl)-2-(phenylsulfonyl)ethan-1-ol **2f****



White solid, 98% yield, 54.9 mg; 95% ee; [α]<sub>D</sub><sup>25</sup> = +24.8 (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; *t*<sub>R</sub> (minor) = 27.3 min, *t*<sub>R</sub> (major) = 33.3 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98–7.95 (m, 2H), 7.73–7.68 (m, 1H), 7.63–7.58 (m, 2H), 7.30–7.26 (m, 2H), 7.03–6.99 (m, 2H), 5.29–5.27 (m, 1H), 3.76 (d, *J* = 2.1 Hz, 1H), 3.52–3.29 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 162.5 (d, *J* = 246.0 Hz),

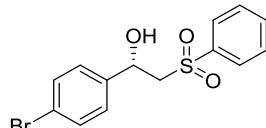
139.0, 136.4 (d,  $J = 4.0$  Hz), 134.2, 129.5, 127.9, 127.4 (d,  $J = 8.0$  Hz), 115.7 (d,  $J = 22.0$  Hz), 67.8, 63.9.

*(S)*-1-(3-bromophenyl)-2-(phenylsulfonyl)ethan-1-ol **2g**



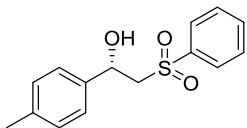
White solid, 96% yield, 65.5 mg; 94% ee;  $[\alpha]_D^{25} = +21.5$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chirapak AD-H column, 210 nm, 20 °C, *n*-hexane: *i*-PrOH = 90:10; flow 1.0 mL/min;  $t_R$  (minor) = 28.4 min,  $t_R$  (major) = 34.9 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97–7.95 (m, 2H), 7.73–7.69 (m, 1H), 7.63–7.59 (m, 2H), 7.42–7.39 (m, 1H), 7.24–7.17 (m, 2H), 5.29–5.25 (m, 1H), 3.81 (d,  $J = 2.1$  Hz, 1H), 3.50–3.31 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  142.8, 138.9, 134.3, 131.4, 130.3, 129.5, 128.8, 127.9, 124.3, 122.8, 67.8, 63.7.

*(S)*-1-(4-bromophenyl)-2-(phenylsulfonyl)ethan-1-ol **2h**



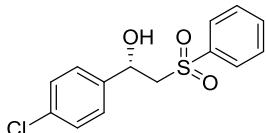
White solid, 99% yield, 67.6 mg; 94% ee;  $[\alpha]_D^{25} = +24.5$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (minor) = 39.7 min,  $t_R$  (major) = 54.8 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96–7.94 (m, 2H), 7.73–7.69 (m, 1H), 7.63–7.58 (m, 2H), 7.47–7.43 (m, 2H), 7.20–7.17 (m, 2H), 5.28–5.24 (m, 1H), 3.78 (d,  $J = 2.2$  Hz, 1H), 3.49–3.29 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  139.6, 138.9, 134.2, 131.9, 129.5, 127.9, 127.4, 122.2, 67.8, 63.7.

*(S)*-2-(phenylsulfonyl)-1-(*p*-tolyl)ethan-1-ol **2i**



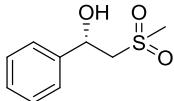
White solid, 99% yield, 54.7 mg; 96% ee;  $[\alpha]_D^{25} = +18.5$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (minor) = 23.6 min, t<sub>R</sub> (major) = 28.2 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98–7.95 (m, 2H), 7.71–7.67 (m, 1H), 7.62–7.57 (m, 2H), 7.19–7.12 (m, 4H), 5.25–5.22 (m, 1H), 3.61 (d, *J* = 2.1 Hz, 1H), 3.54–3.31 (m, 2H), 2.31 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 139.2, 138.2, 137.6, 134.1, 129.4, 129.4, 128.0, 125.6, 68.3, 63.9, 21.1.

#### (S)-1-(4-chlorophenyl)-2-(phenylsulfonyl)ethan-1-ol **2j**



White solid, 98% yield, 58.5 mg; 94% ee;  $[\alpha]_D^{25} = +19.0$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (minor) = 32.8 min, t<sub>R</sub> (major) = 42.9 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.97–7.94 (m, 2H), 7.73–7.69 (m, 1H), 7.63–7.58 (m, 2H), 7.31–7.28 (m, 2H), 7.26–7.22 (m, 2H), 5.29–5.26 (m, 1H), 3.78 (d, *J* = 2.2 Hz, 1H), 3.49–3.29 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 139.0, 138.9, 134.2, 134.1, 129.5, 128.9, 127.9, 127.0, 67.8, 63.8.

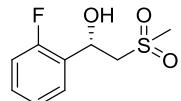
#### (S)-2-(methylsulfonyl)-1-phenylethan-1-ol **2k**



S/C = 500, 0.2 mmol substrate, Na<sub>2</sub>CO<sub>3</sub> (5 mol%). White solid, 99% yield, 39.6 mg; 97% ee;  $[\alpha]_D^{25} = +49.9$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 210 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (major) = 20.4 min, t<sub>R</sub> (minor) = 30.9 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

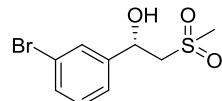
$\delta$  7.42–7.31 (m, 5H), 5.36–5.32 (m, 1H), 3.45 (dd,  $J$  = 14.7, 10.3 Hz, 1H), 3.19–3.14 (m, 1H), 3.05 (s, 3H), 3.05–3.02 (m, 1H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  141.0, 129.0, 128.7, 125.6, 69.3, 62.4, 42.8.

**(S)-1-(2-fluorophenyl)-2-(methylsulfonyl)ethan-1-ol **2l****



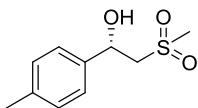
White solid, 99% yield, 43.2 mg; 95% ee;  $[\alpha]_D^{25} = +32.1$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (major) = 13.1 min,  $t_R$  (minor) = 17.6 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57–7.53 (m, 1H), 7.35–7.30 (m, 1H), 7.23–7.19 (m, 1H), 7.10–7.05 (m, 1H), 5.61–5.58 (m, 1H), 3.48–3.40 (m, 2H), 3.34–3.29 (m, 1H), 3.07 (s, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.2 (d,  $J$  = 244.0 Hz), 130.1 (d,  $J$  = 9.0 Hz), 127.8 (d,  $J$  = 13.0 Hz), 127.2 (d,  $J$  = 4.0 Hz), 124.8 (d,  $J$  = 4.0 Hz), 115.6 (d,  $J$  = 21.0 Hz), 63.8 (d,  $J$  = 3.0 Hz), 60.8 (d,  $J$  = 1.0 Hz), 42.6

**(S)-1-(3-bromophenyl)-2-(methylsulfonyl)ethan-1-ol **2m****



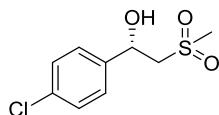
S/C = 500, 0.1 mmol substrate,  $\text{Na}_2\text{CO}_3$  (10 mol%). White solid, 99% yield, 27.6 mg; 97% ee;  $[\alpha]_D^{25} = +32.9$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 210 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (minor) = 32.3 min,  $t_R$  (major) = 34.2 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (t,  $J$  = 1.8 Hz, 1H), 7.48–7.46 (m, 1H), 7.32–7.24 (m, 2H), 5.35–5.32 (m, 1H), 3.45–3.38 (m, 1H), 3.18–3.14 (m, 2H), 3.07 (s, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  143.1, 131.7, 130.5, 128.8, 124.2, 123.1, 68.6, 62.2, 42.9.

**(S)-2-(methylsulfonyl)-1-(*p*-tolyl)ethan-1-ol **2n****



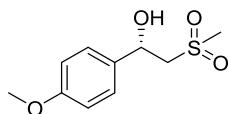
White solid, 98% yield, 50.0 mg; 98% ee;  $[\alpha]_D^{25} = +34.1$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OD-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (major) = 11.4 min, t<sub>R</sub> (minor) = 13.4 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.28–7.26 (m, 2H), 7.20–7.18 (m, 2H), 5.32–5.28 (m, 1H), 3.48–3.42 (m, 1H), 3.17–3.12 (m, 1H), 3.04 (s, 3H), 2.94–2.93 (m, 1H), 2.35 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 138.5, 138.0, 129.6, 125.5, 69.2, 62.4, 42.8, 21.1.

**(S)-1-(4-chlorophenyl)-2-(methylsulfonyl)ethan-1-ol 2o**



White solid, 99% yield, 46.5 mg; 99% ee;  $[\alpha]_D^{25} = +43.8$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (major) = 27.7 min, t<sub>R</sub> (minor) = 31.9 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.39–7.31 (m, 4H), 5.36–5.32 (m, 1H), 3.42 (dd, *J* = 14.7, 10.3 Hz, 1H), 3.16–3.12 (m, 2H), 3.07 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 139.4, 134.4, 129.1, 127.0, 68.6, 62.3, 42.9.

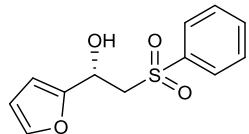
**(S)-1-(4-methoxyphenyl)-2-(methylsulfonyl)ethan-1-ol 2p**



White solid, 99% yield, 45.6 mg; 99% ee;  $[\alpha]_D^{25} = +46.3$  (c = 1.50, CHCl<sub>3</sub>). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min; t<sub>R</sub> (major) = 42.2 min, t<sub>R</sub> (minor) = 60.0 min. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.32–7.28 (m, 2H), 6.93–6.89 (m, 2H), 5.30–5.27 (m, 1H), 3.81 (s, 3H), 3.49–3.42 (m, 1H), 3.14 (dd, *J* = 14.7, 2.0 Hz, 1H), 3.04 (s, 3H), 2.96–2.95 (m, 1H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 159.8, 133.1, 127.0, 114.3,

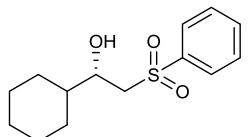
68.9, 62.4, 55.3, 42.8.

*(S)*-1-(furan-2-yl)-2-(phenylsulfonyl)ethanol **2q**



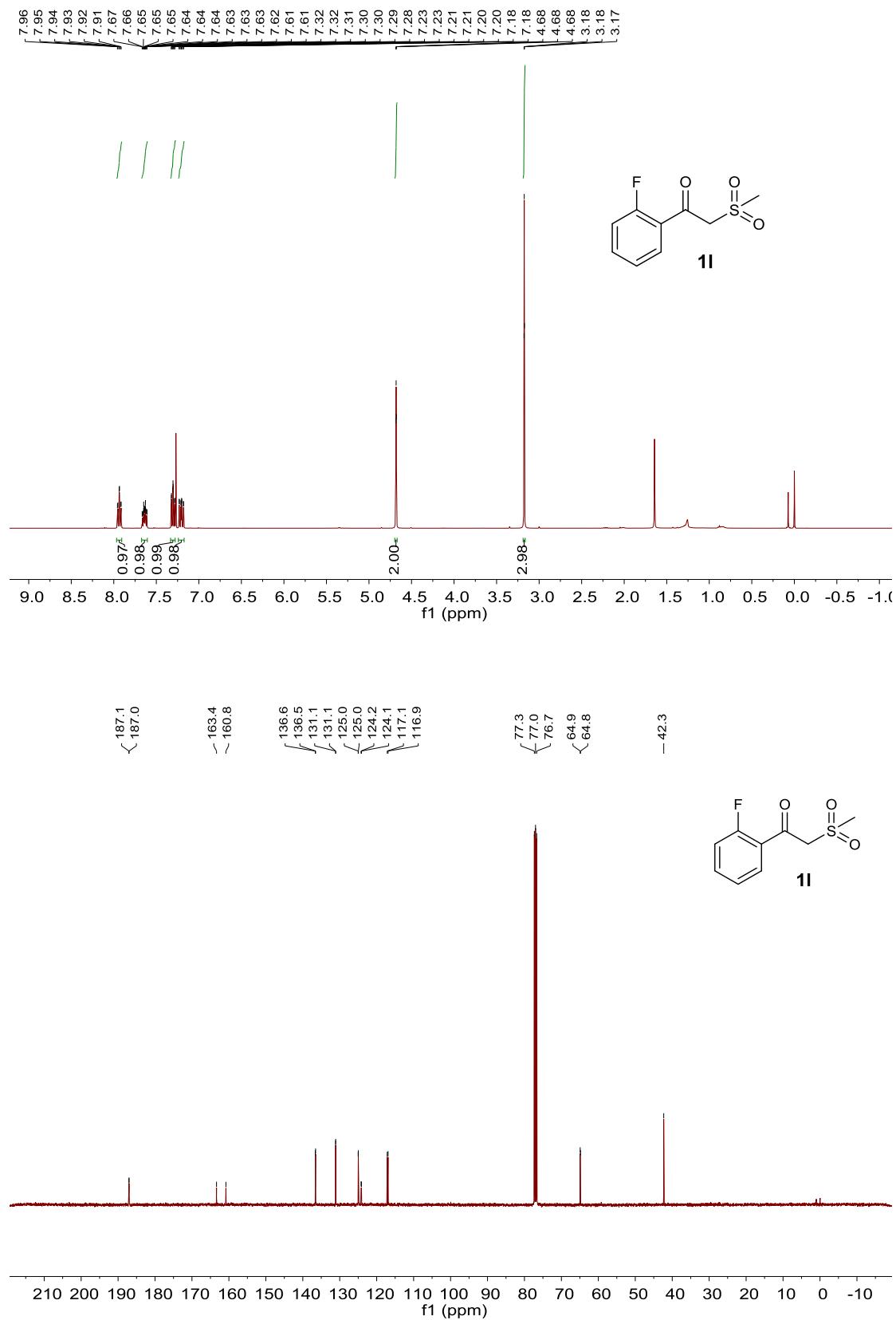
S/C = 500, 0.2 mmol substrate,  $\text{Na}_2\text{CO}_3$  (5 mol%). White solid, 98% yield, 49.4 mg; 86% ee;  $[\alpha]_D^{25} = +8.0$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chiracel OJ-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 1.0 mL/min;  $t_R$  (major) = 38.1 min,  $t_R$  (minor) = 36.0 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96-7.93 (m, 2H), 7.71-7.66 (m, 1H), 7.61-7.56 (m, 2H), 7.30 (dd,  $J = 1.7, 1.0$  Hz, 1H), 6.31-6.29 (m, 2H), 5.30-5.26 (m, 1H), 3.68 (dd,  $J = 14.4, 9.4$  Hz, 1H), 3.56-3.49 (m, 2H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  152.4, 142.6, 139.0, 134.1, 129.4, 128.0, 110.4, 107.4, 62.7, 60.5.

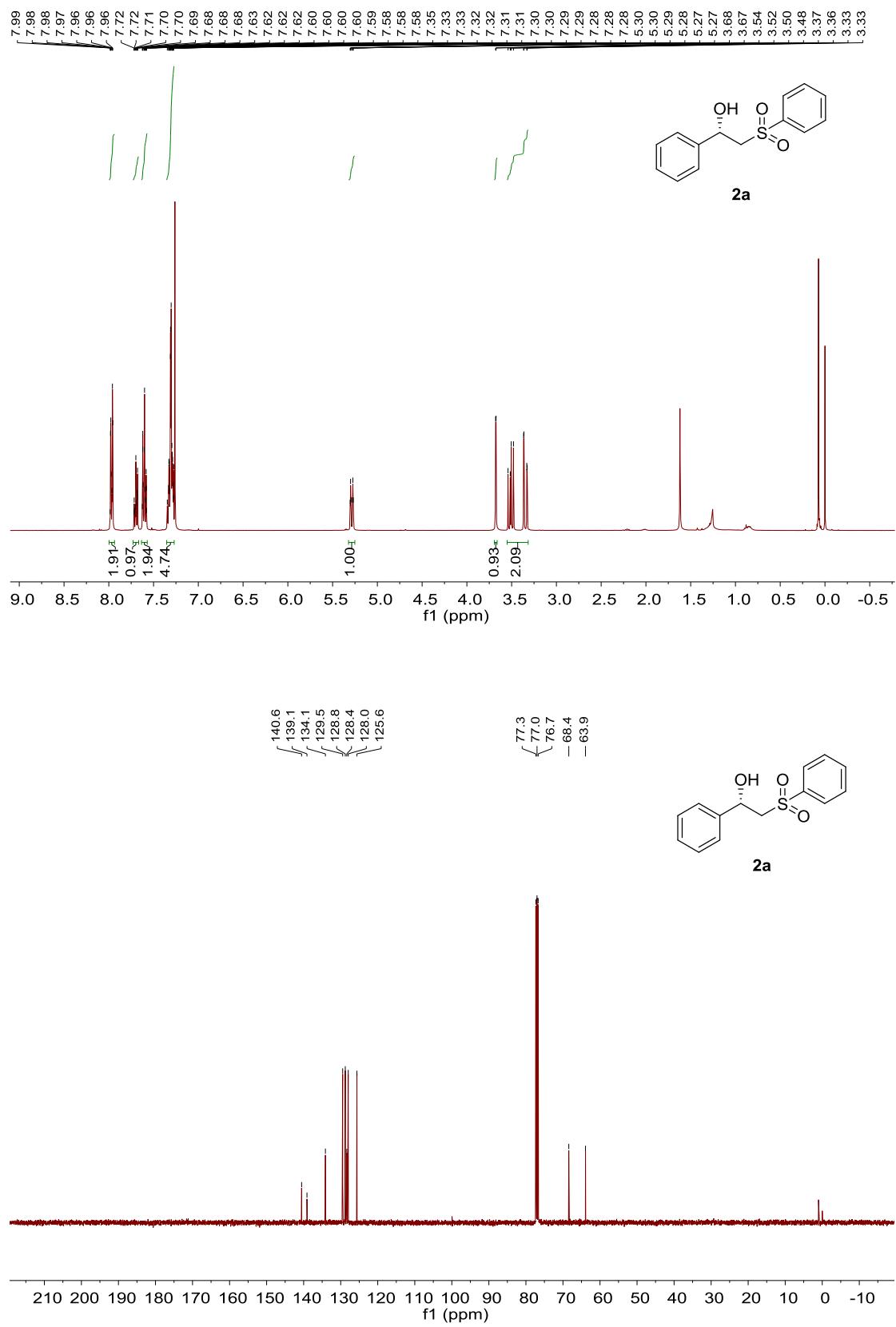
*(S)*-1-cyclohexyl-2-(phenylsulfonyl)ethan-1-ol **2r**

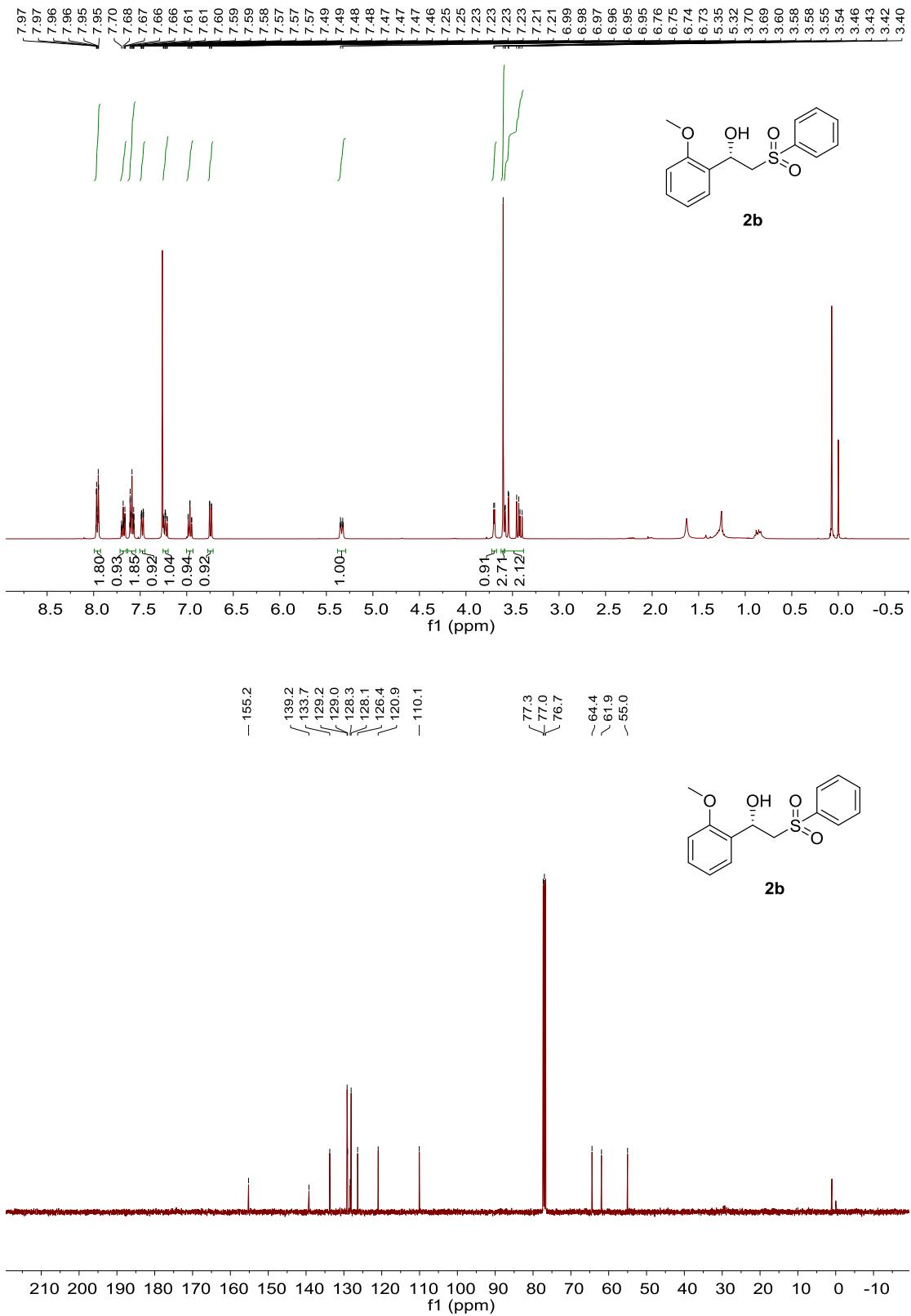


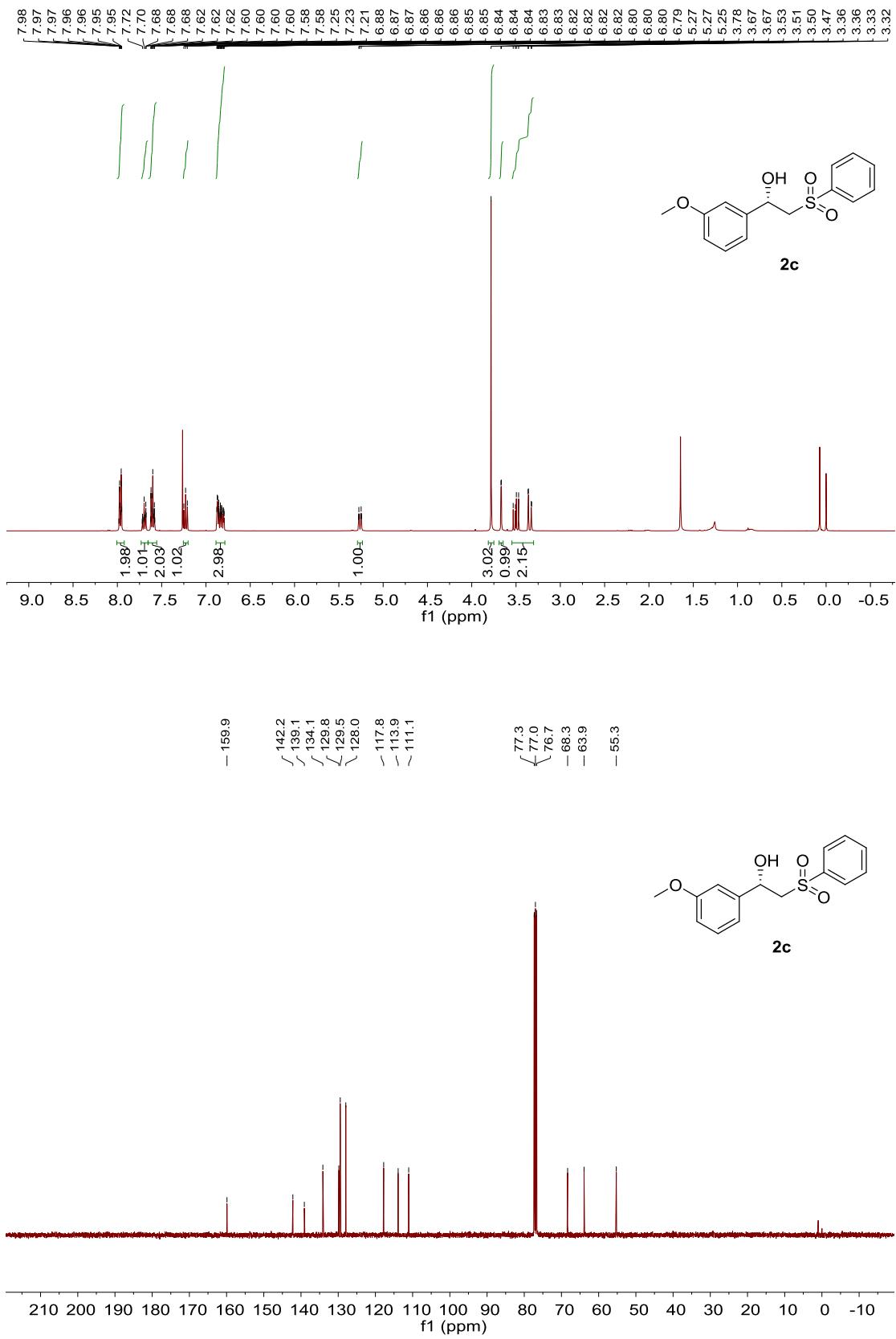
S/C = 500, 0.2 mmol substrate,  $\text{Na}_2\text{CO}_3$  (5 mol%). Colorless liquid, 35% yield, 18.9 mg; 90% ee;  $[\alpha]_D^{25} = +10.6$  ( $c = 1.50$ ,  $\text{CHCl}_3$ ). The enantiomeric excess was determined by HPLC on Chirapak AD-H column, 220 nm, 20 °C, *n*-hexane: *i*-PrOH = 80:20; flow 0.7 mL/min;  $t_R$  (minor) = 18.9 min,  $t_R$  (major) = 22.1 min.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 (dd,  $J = 8.3, 1.3$  Hz, 2H), 7.69-7.67 (m, 1H), 7.62-7.58 (m, 2H), 3.96-3.95 (m, 1H), 3.26 (d,  $J = 2.6$  Hz, 1H), 3.26-3.22 (m, 2H), 1.74-1.63 (m, 5H), 1.46-1.36 (m, 1H), 1.27-0.99 (m, 5H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  139.2, 134.0, 129.4, 127.9, 69.7, 60.2, 43.1, 28.4, 27.5, 26.2, 25.9, 25.8.

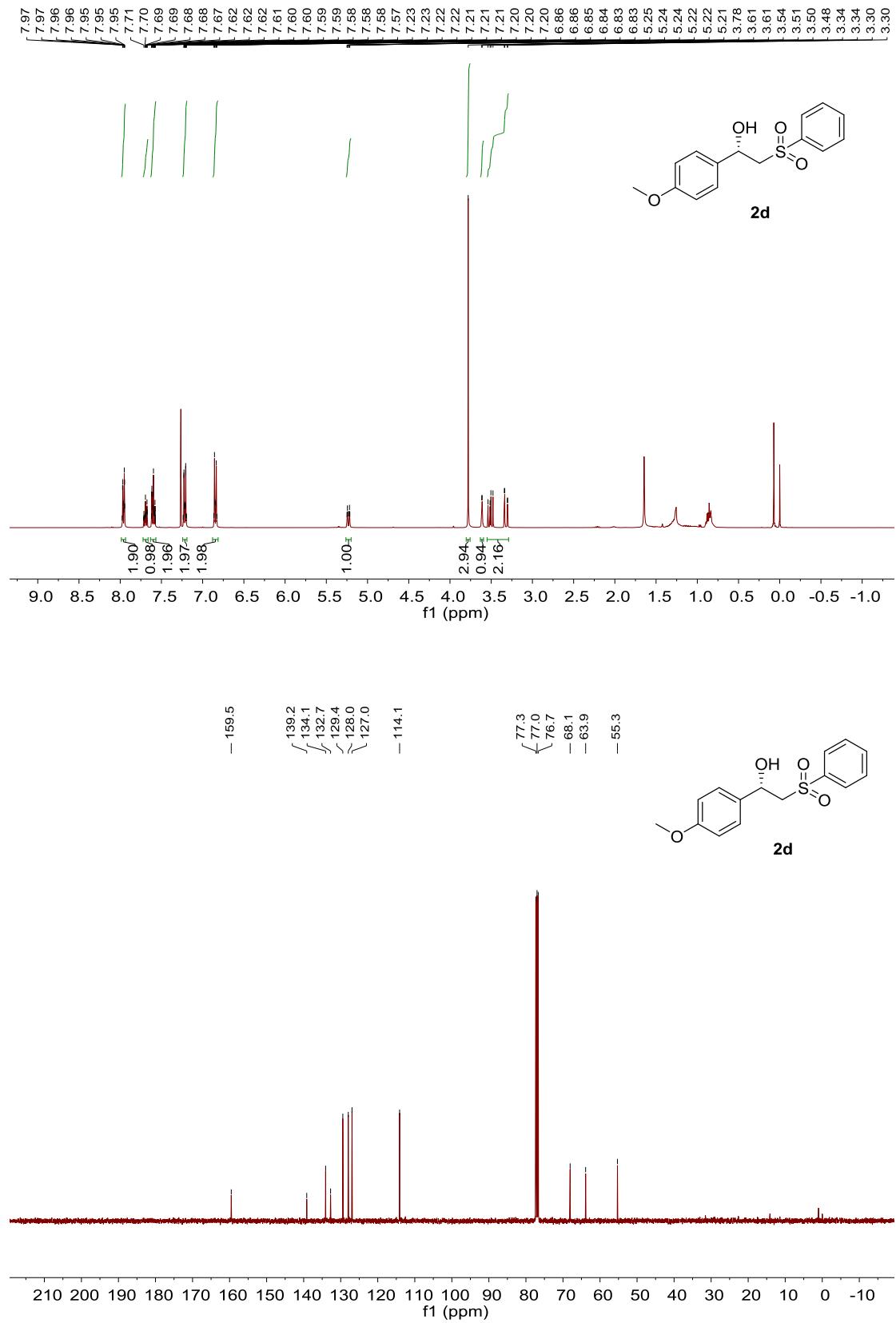
## 4. NMR spectra

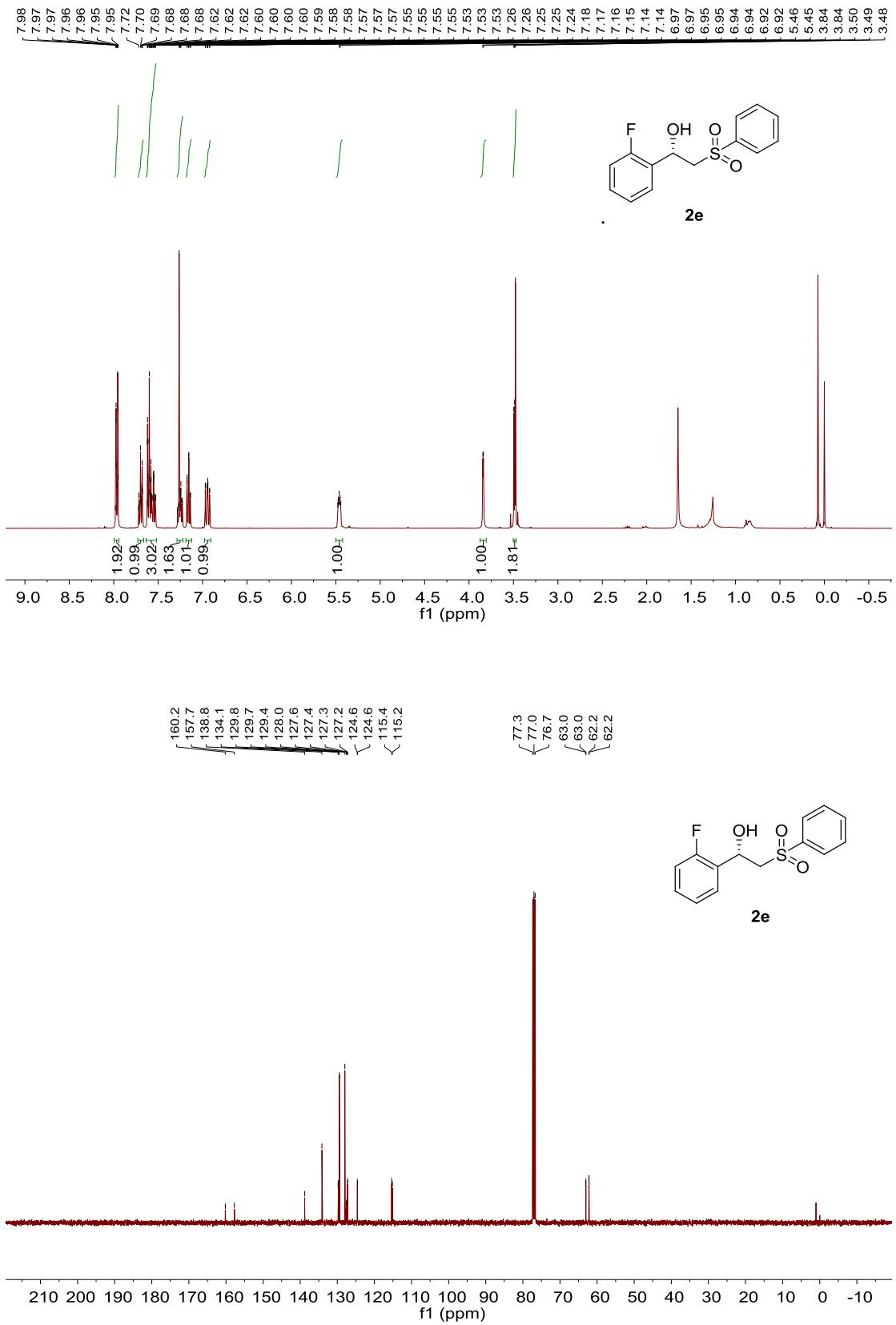


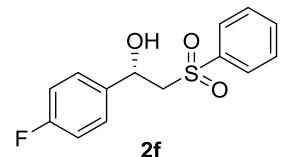
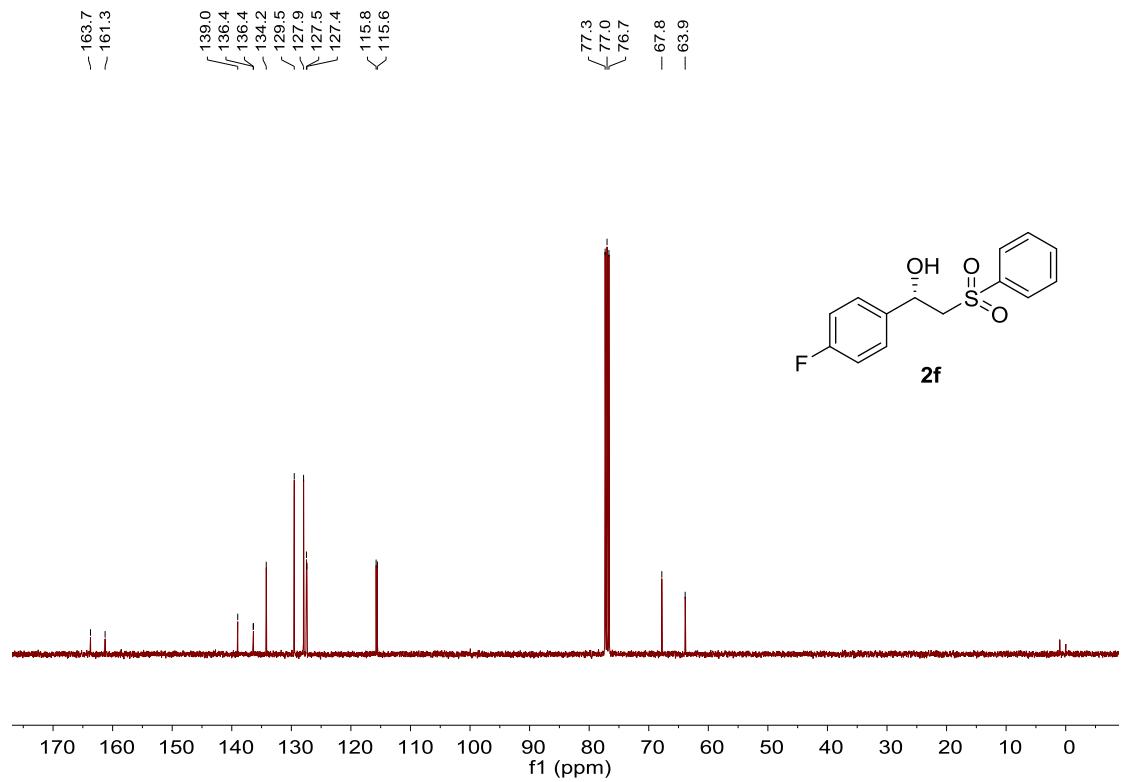
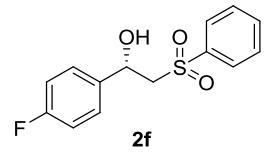
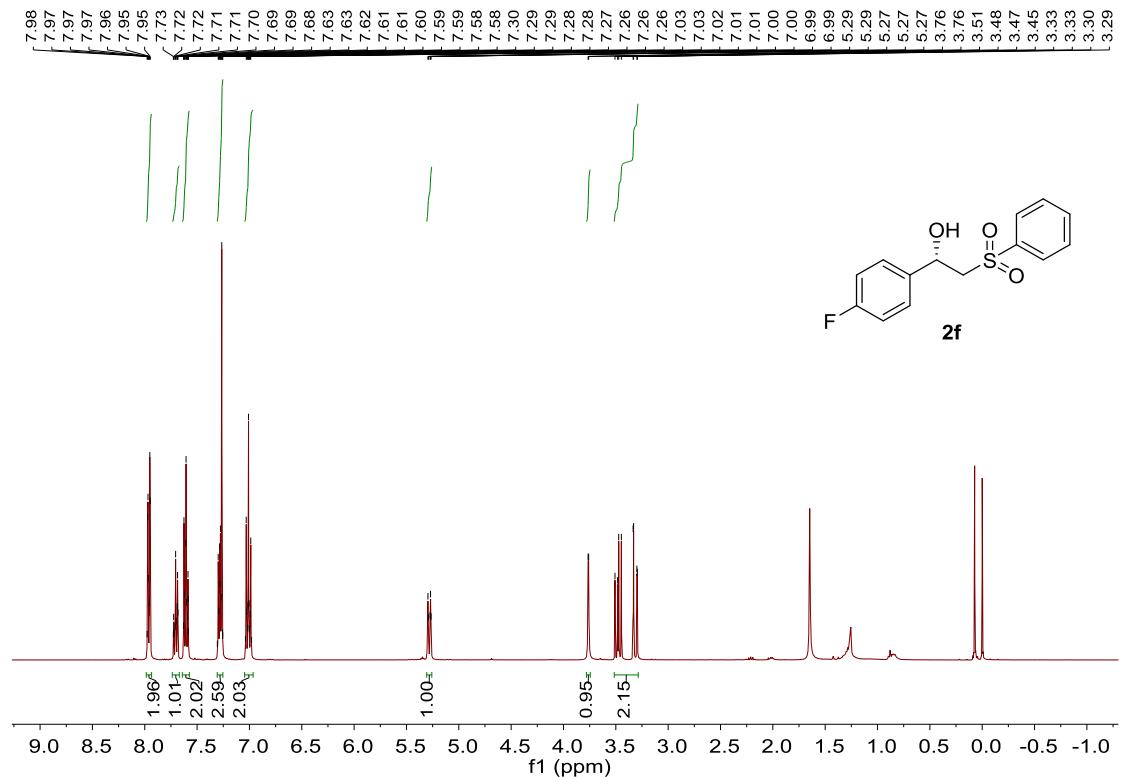


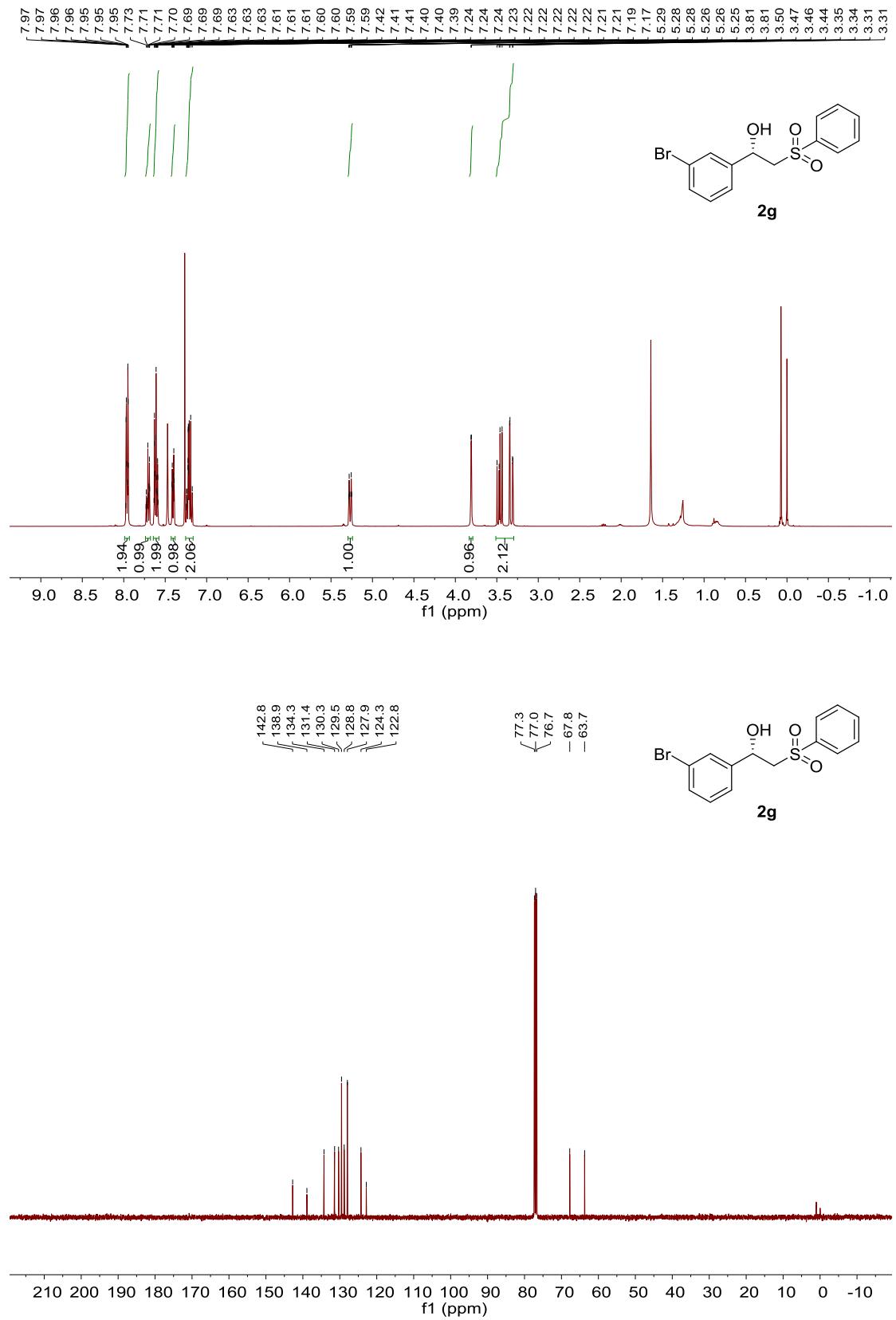


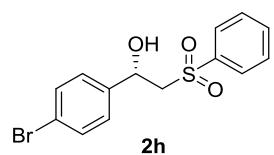
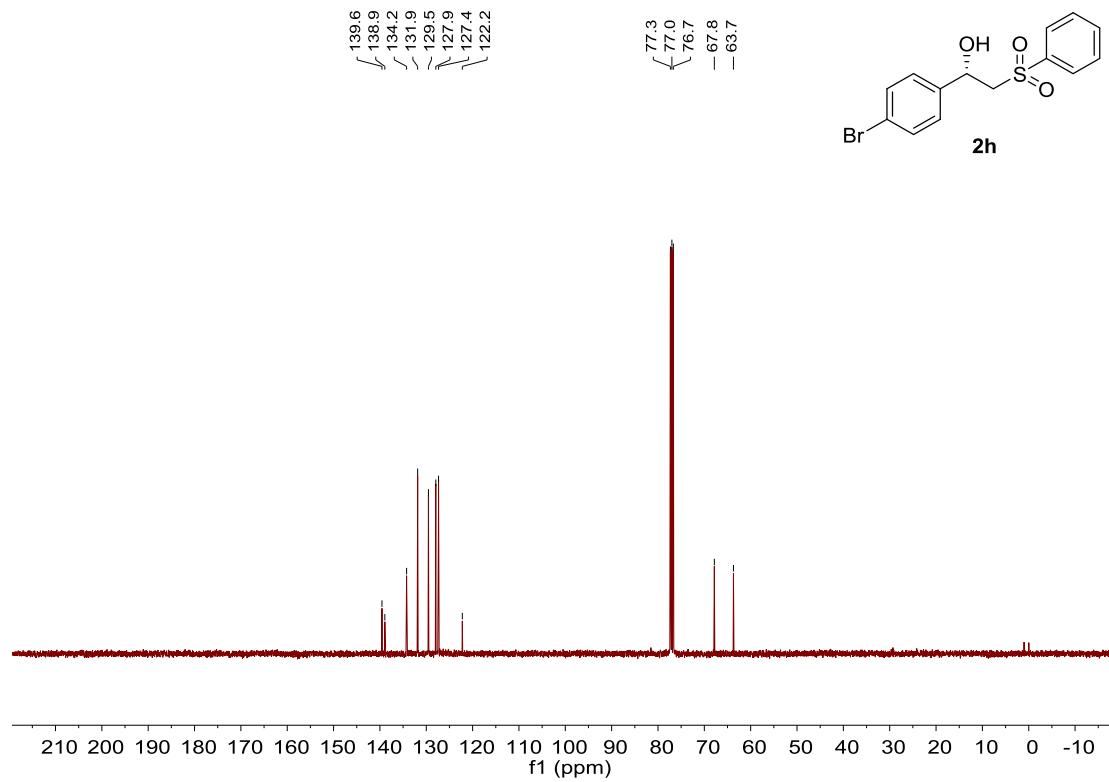
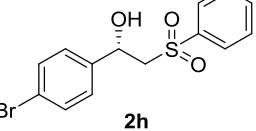
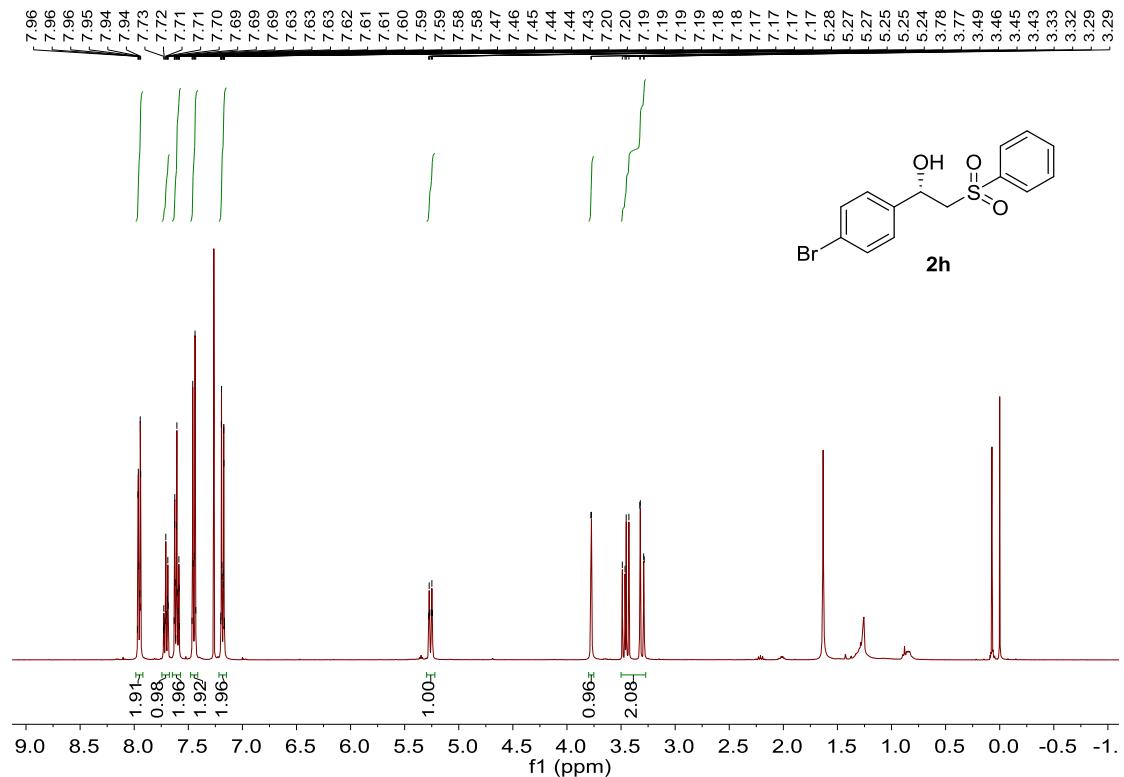


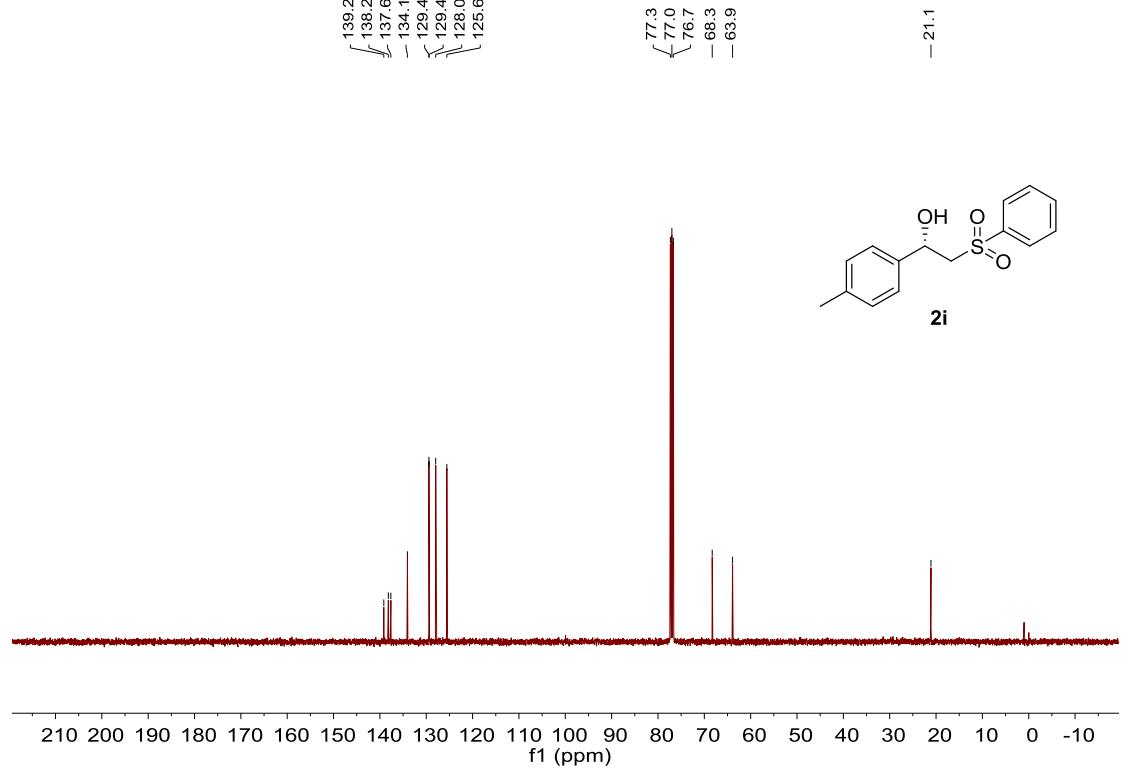
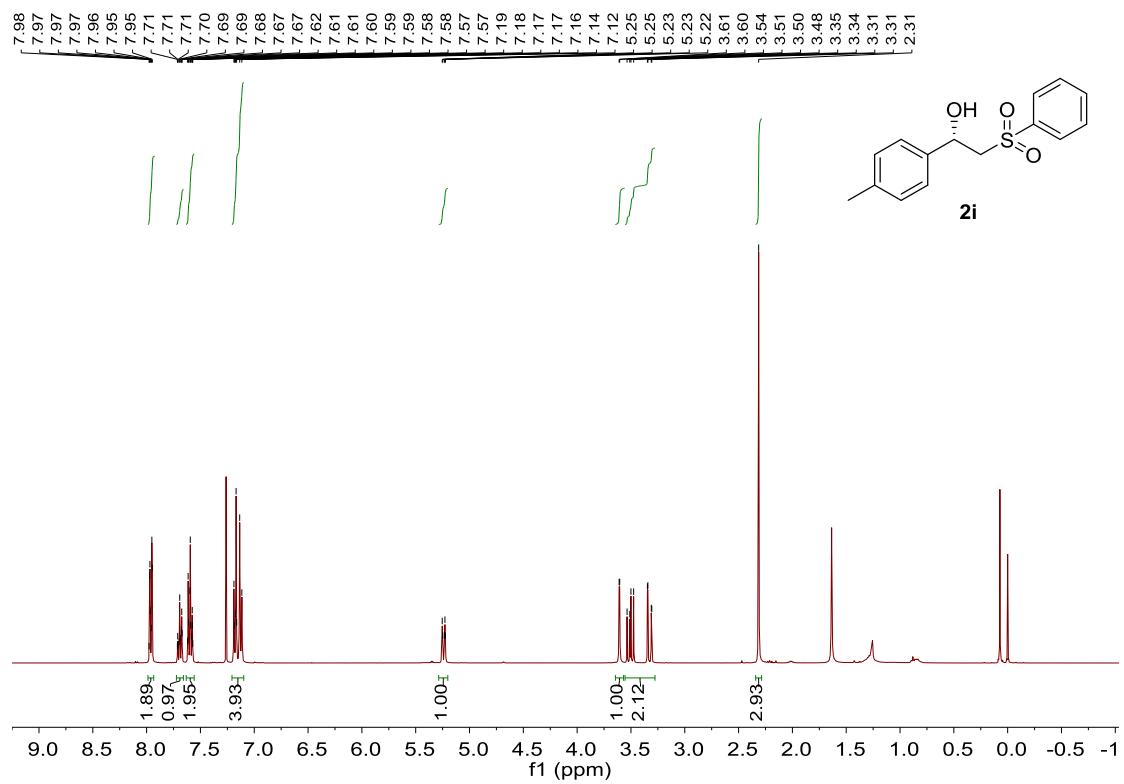


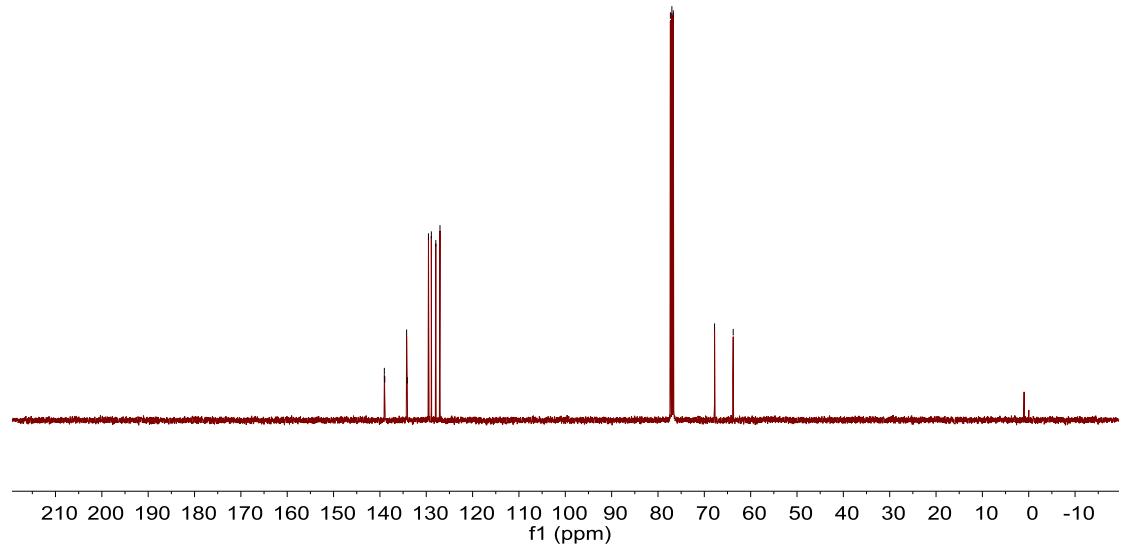
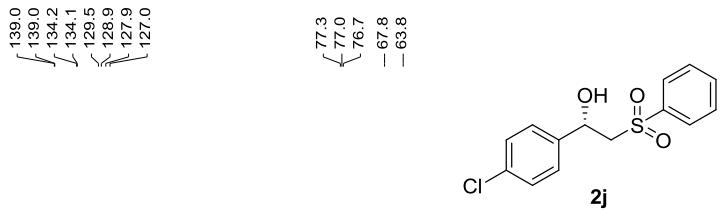
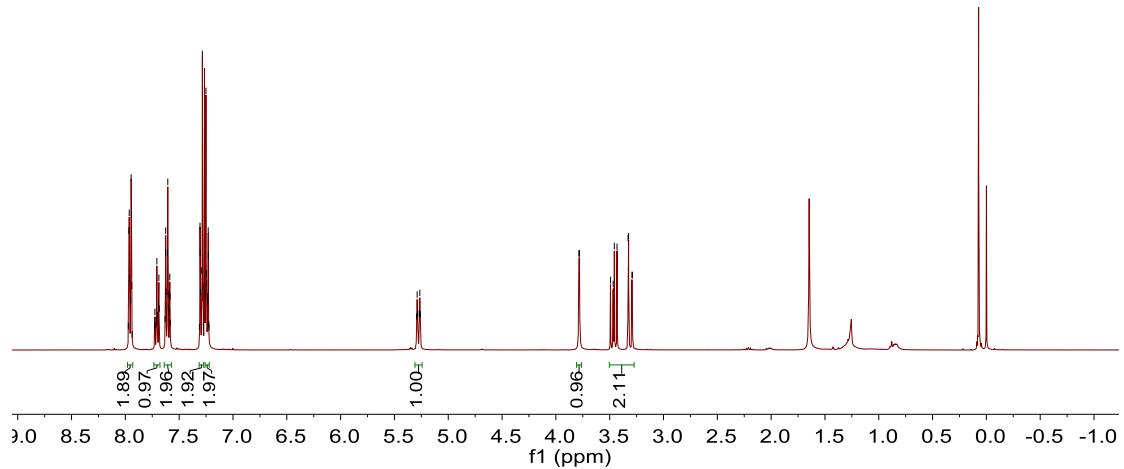
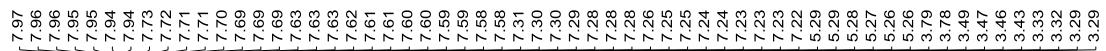


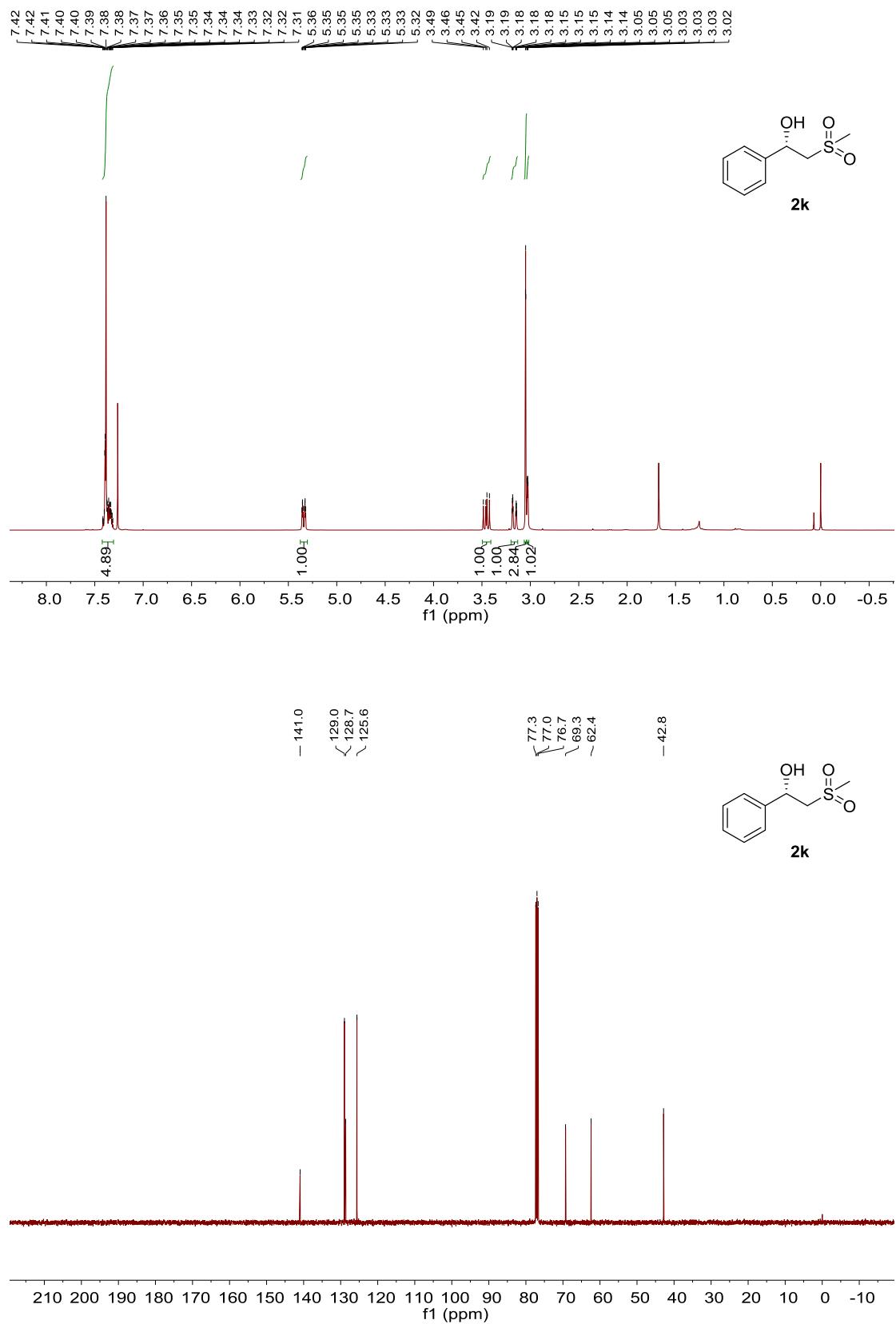


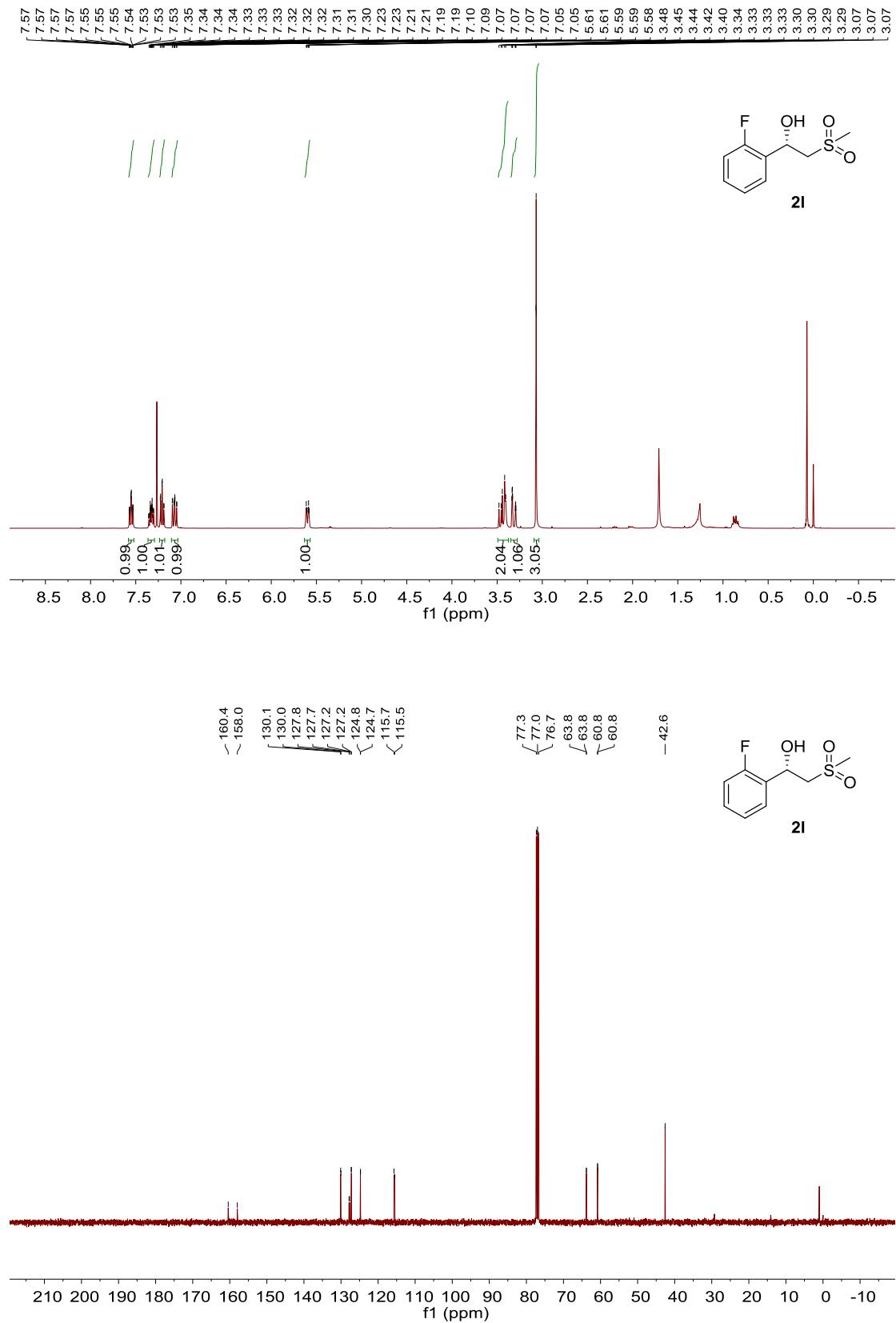


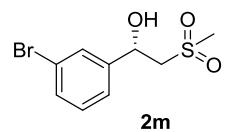
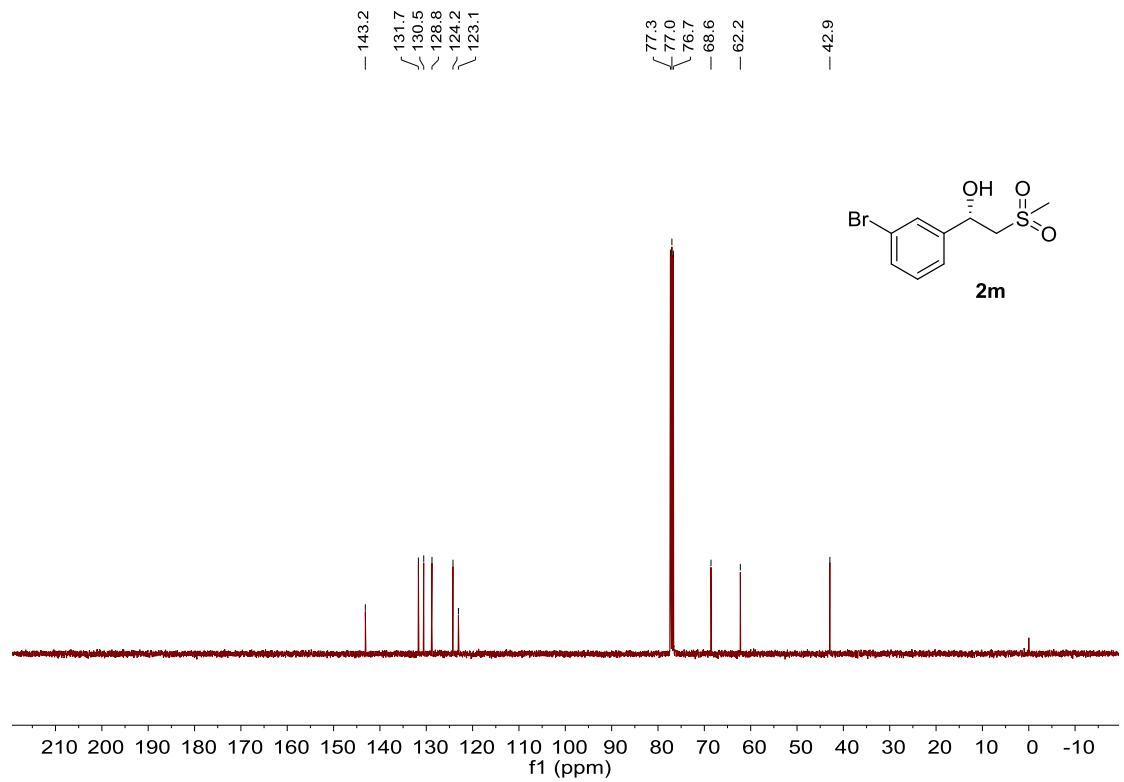
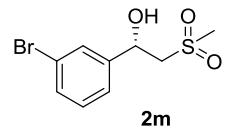
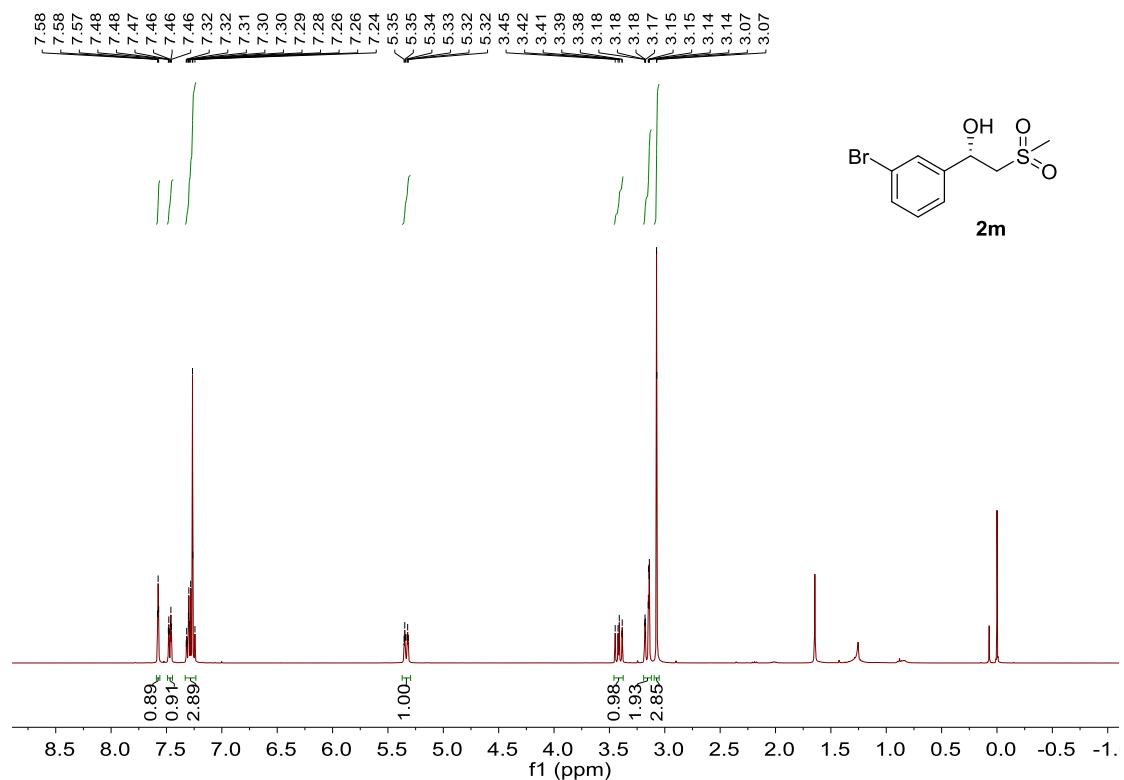


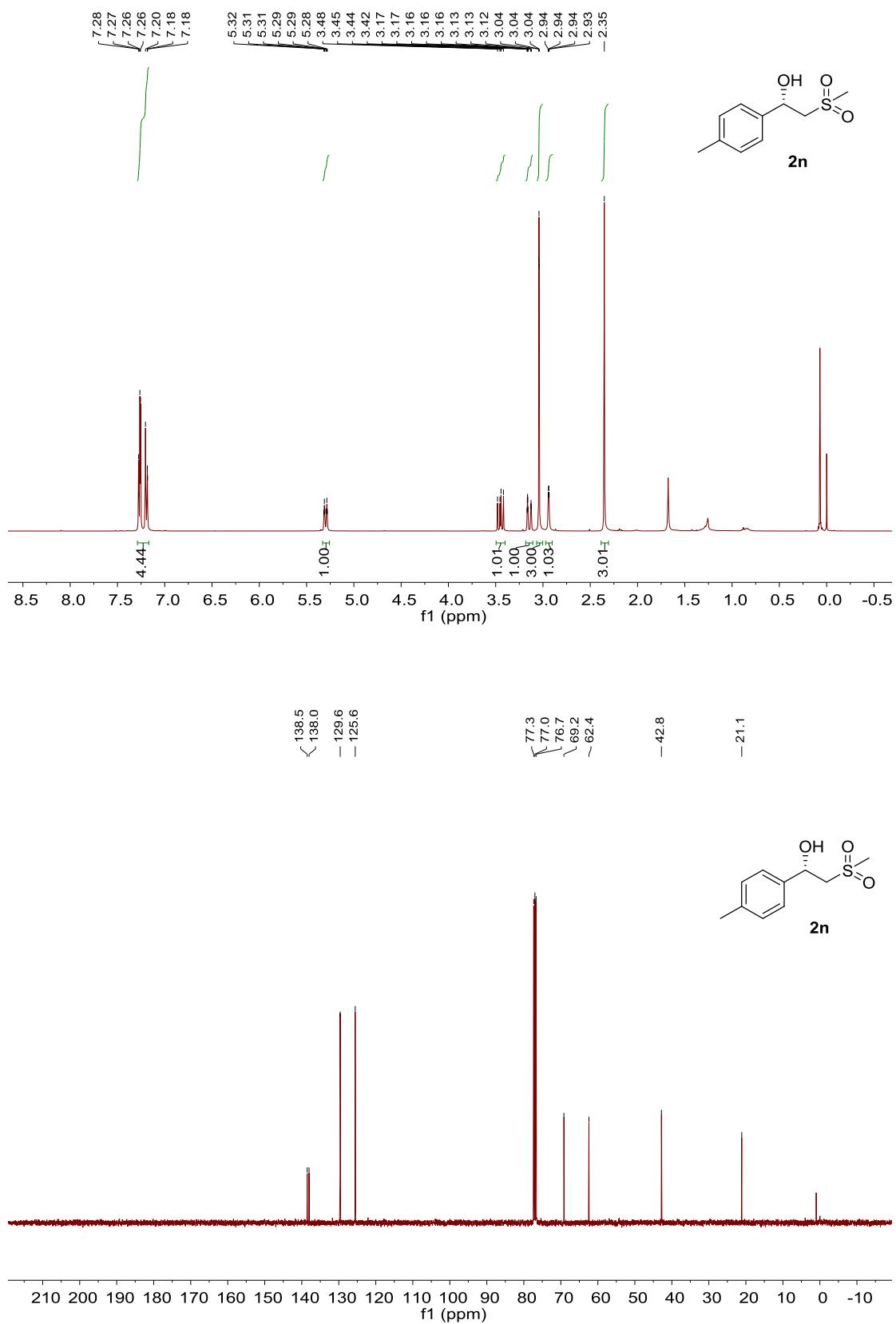


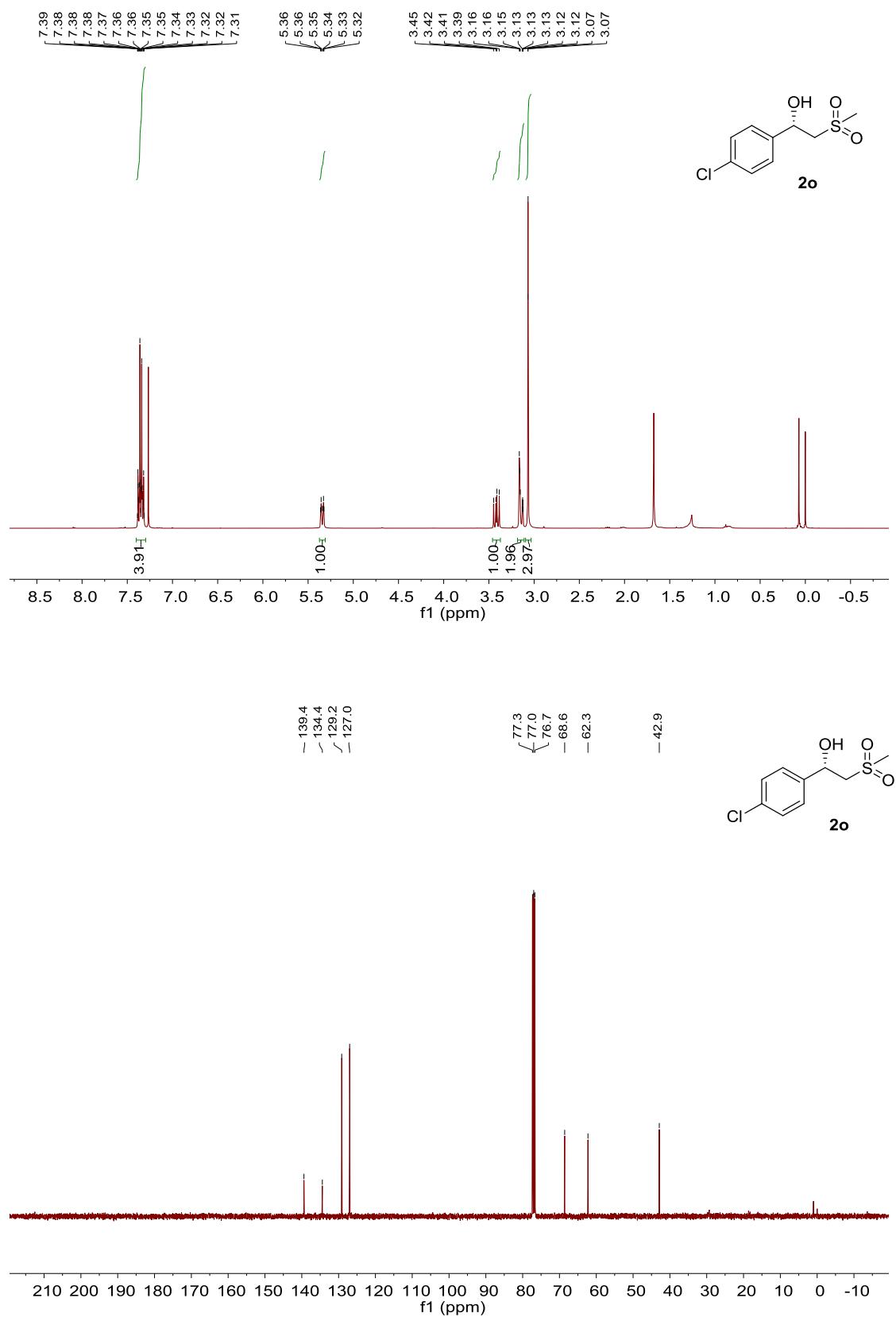


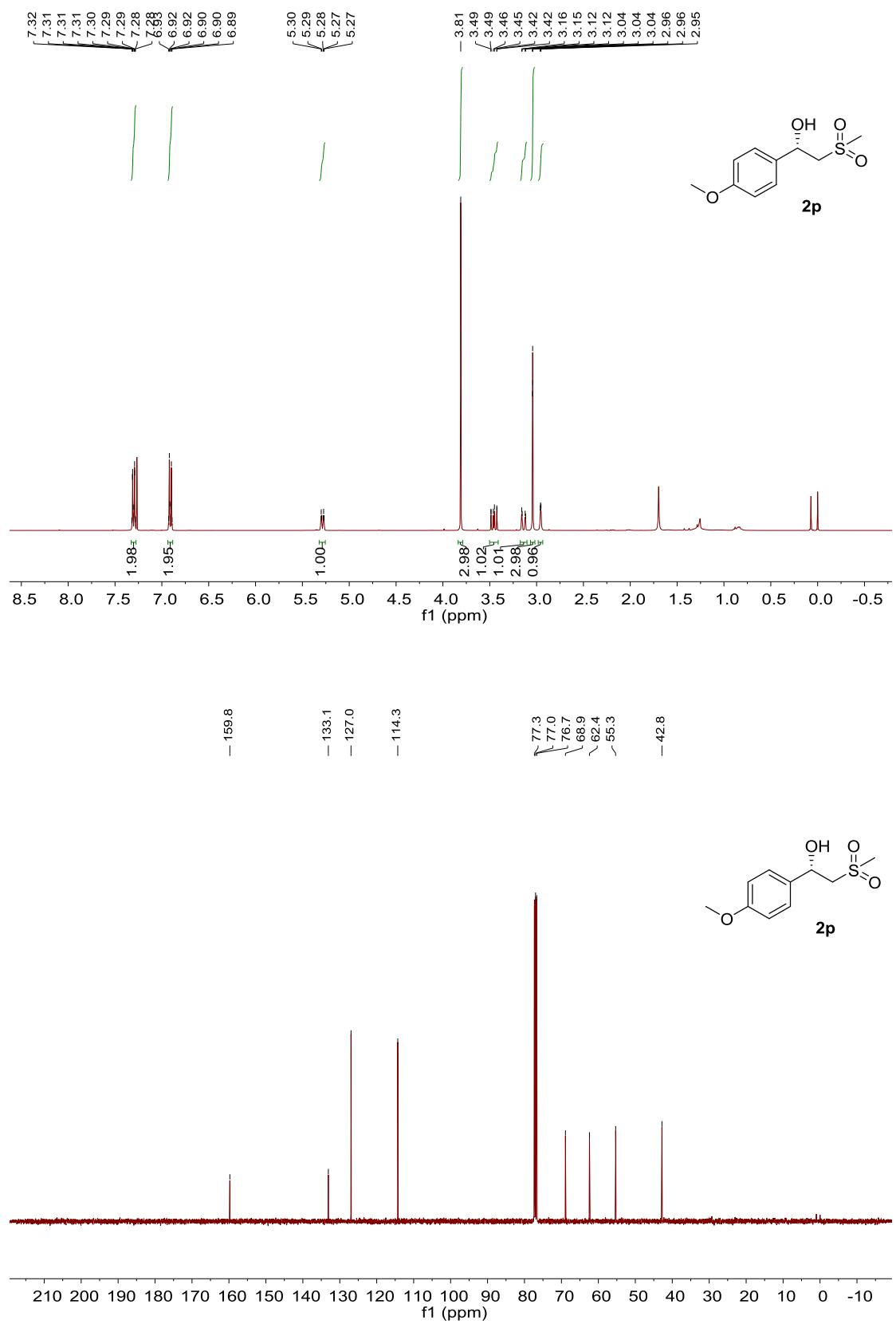


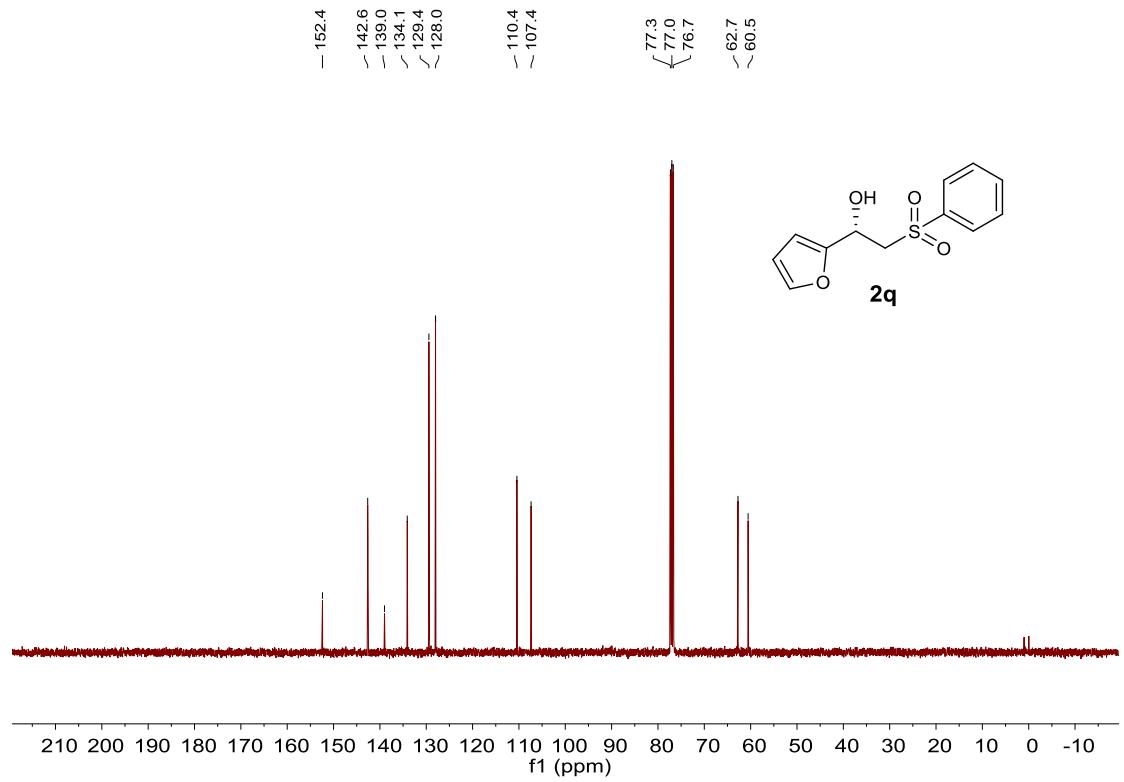
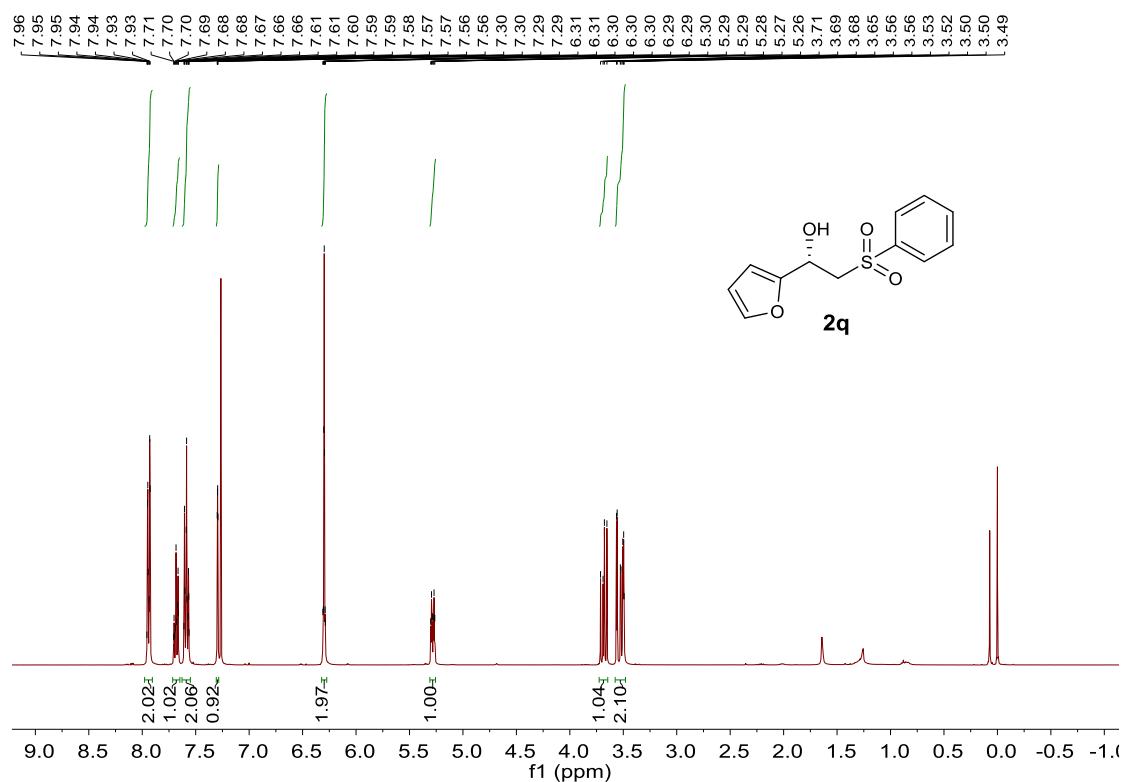


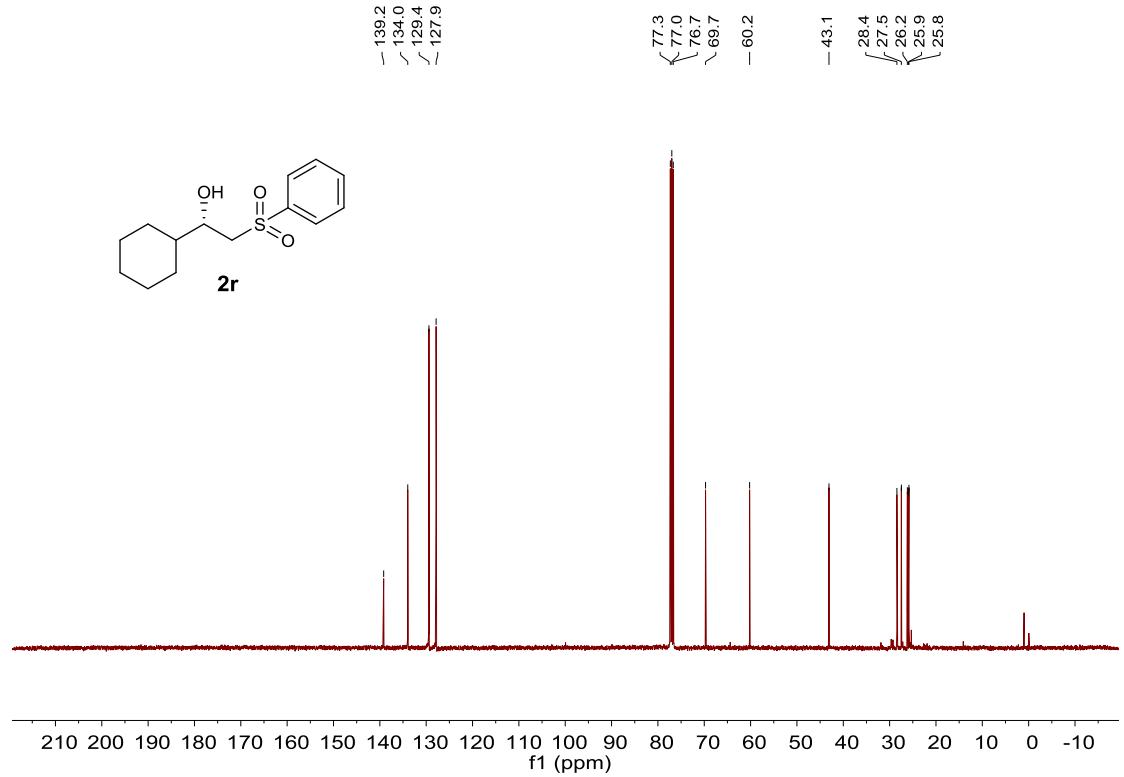
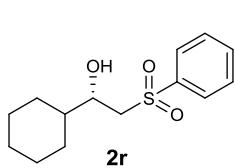
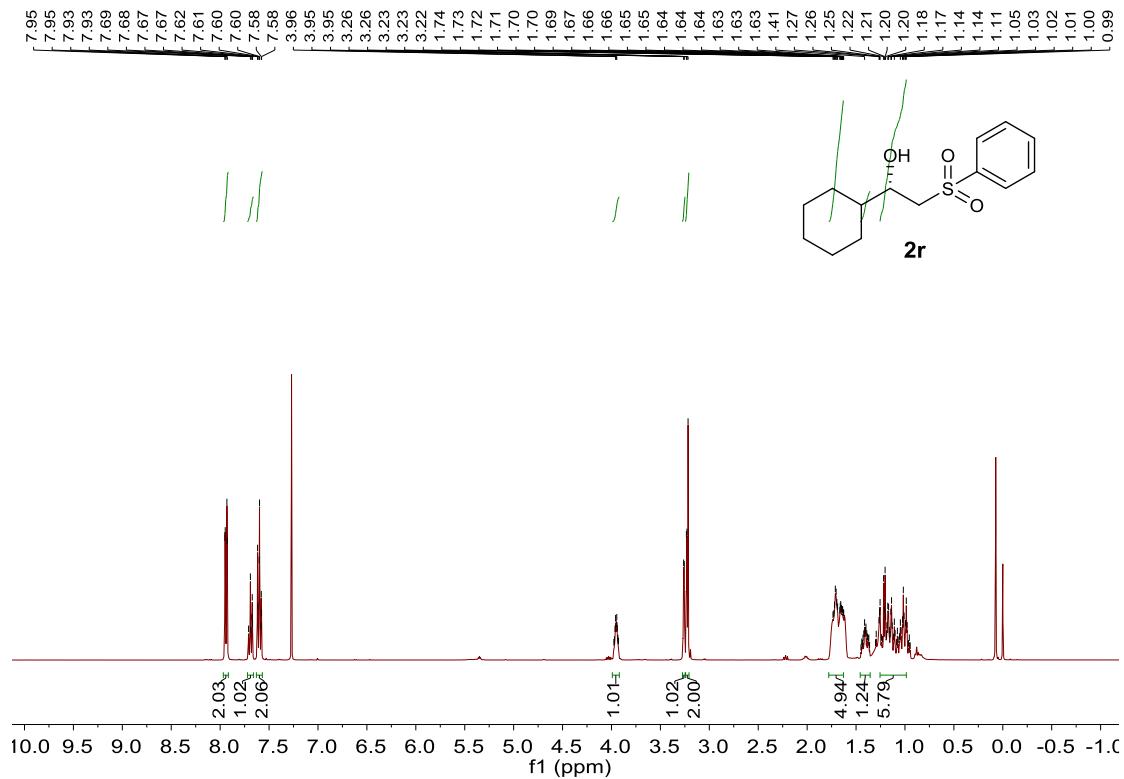








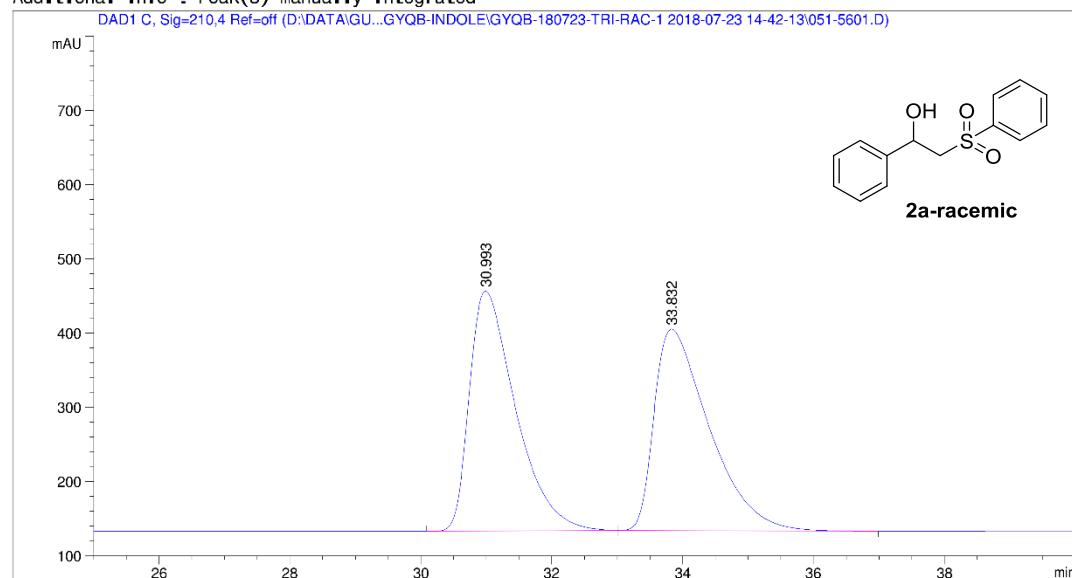




## 5. HPLC spectra

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Injection Date  : 7/25/2018 12:27:43 AM        Inj       : 1
                                                Inj Volume : 3.000 μl
Acq. Method     : D:\DATA\GUAN YUQING\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13
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Last changed    : 6/28/2018 8:52:23 PM
Analysis Method : D:\METHOD\NWD\DA-D(1-6)-90-10-0.7ML-3UL-ALL-70MIN.M
Last changed    : 8/21/2018 9:47:34 AM
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Additional Info : Peak(s) manually integrated
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=====
Area Percent Report
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Dilution      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
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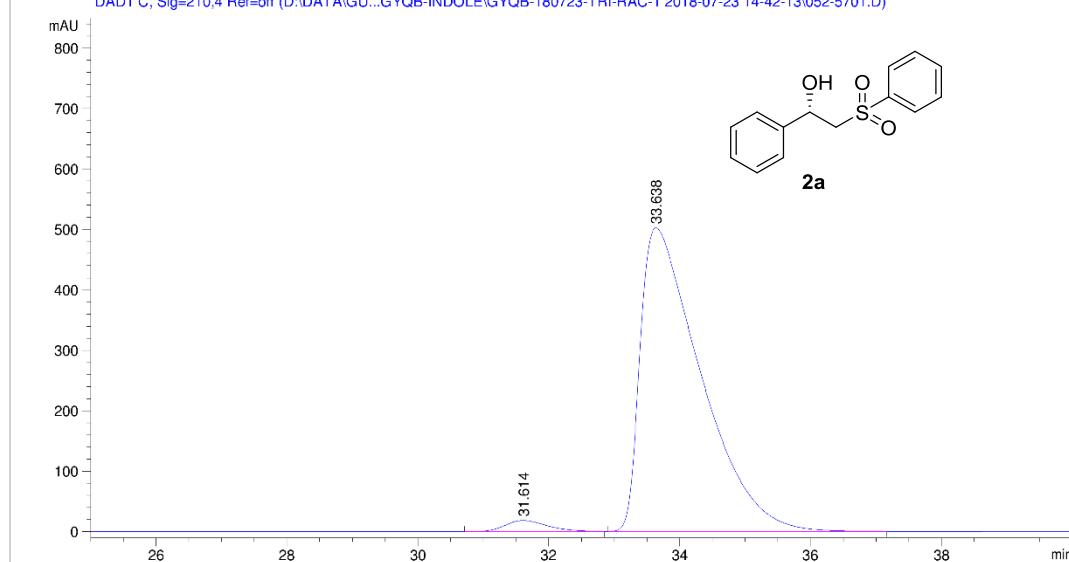
Signal 1: DAD1 C, Sig=210.4 Ref=off

| Peak #   | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|----------|---------------|------|-------------|--------------|--------------|---------|
| 1        | 30.993        | BB   | 0.7358      | 1.58370e4    | 323.13983    | 49.9975 |
| 2        | 33.832        | BB   | 0.8511      | 1.58385e4    | 271.31900    | 50.0025 |
| Totals : |               |      |             | 3.16755e4    | 594.45883    |         |

Data File D:\DATA\GU...NG\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13\052-5701.D  
Sample Name: TL-ph-ph-rac

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                                                Inj Volume : 3.000 μl
Acq. Method    : D:\DATA\GUAN YUQING\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13
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Last changed   : 6/28/2018 8:52:23 PM
Analysis Method: D:\METHOD\LWD\枉DAD-AD(1-6)-90-10-0.7ML-3UL-ALL-70MIN.M
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Additional Info : Peak(s) manually integrated
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DAD1 C, Sig=210.4 Ref=off (D:\DATA\GU...GYQB-INDOL)



## Area Percent Report

Sorted By : Signal  
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Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

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| 1      | 31.614        | BB   | 0.6241      | 828.07184    | 18.36459     | 2.5013 |
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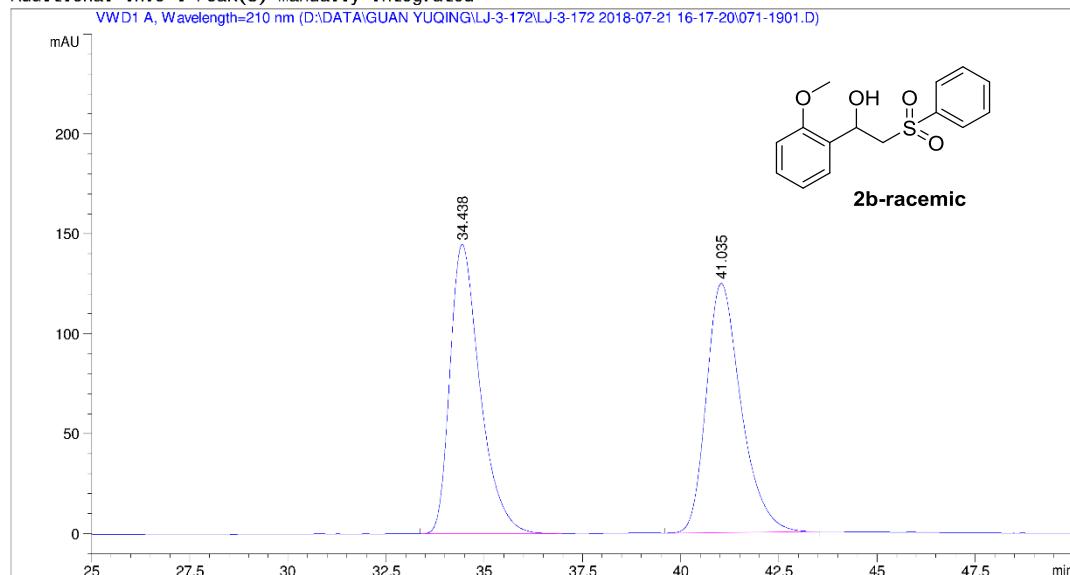
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Instrument 2 8/21/2018 9:48:37 AM

Page 1 of 2

Data File D:\DATA\GUAN YUQING\LJ-3-172\LJ-3-172 2018-07-21 16-17-20\071-1901.D  
Sample Name: TL-2-MeO-ph-rac

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Inj Volume : 3.000  $\mu$ l  
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(modified after loading)  
Additional Info : Peak(s) manually integrated



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Area Percent Report  
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Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
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| 1      | 34.438        | BB   | 0.8071      | 7703.96484   | 144.70210    | 50.0026 |
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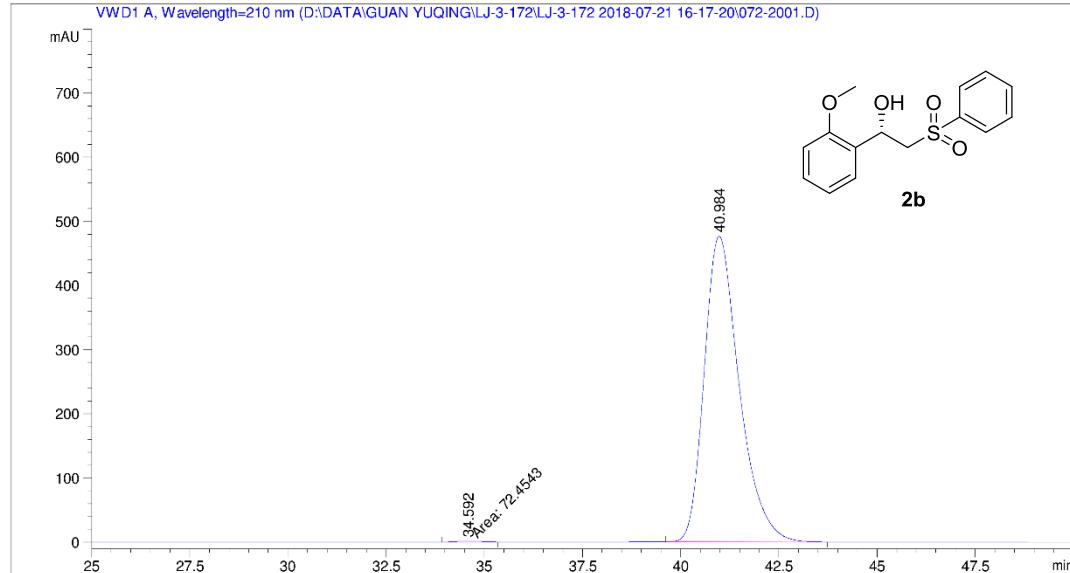
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Instrument 2 8/20/2018 9:14:32 PM

Page 1 of 2

Data File D:\DATA\GUAN YUQING\LJ-3-172\LJ-3-172 2018-07-21 16-17-20\072-2001.D  
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Injection Date : 7/22/2018 7:39:14 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\GUAN YUQING\LJ-3-172\LJ-3-172 2018-07-21 16-17-20\VWD-AD(1-2)-90-10  
-1ML-3UL-210NM-55MIN.M  
Last changed : 5/29/2018 8:00:31 PM  
Analysis Method : D:\METHOD\LWD\DA<sup>D</sup>-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:20:34 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

| # | RetTime | Type | Width  | Area      | Height    | Area %  |
|---|---------|------|--------|-----------|-----------|---------|
|   | [min]   |      | [min]  | [mAU*s]   | [mAU]     |         |
| 1 | 34.592  | MM   | 0.7966 | 72.45431  | 1.51590   | 0.2449  |
| 2 | 40.984  | BB   | 0.9404 | 2.95174e4 | 476.07034 | 99.7551 |

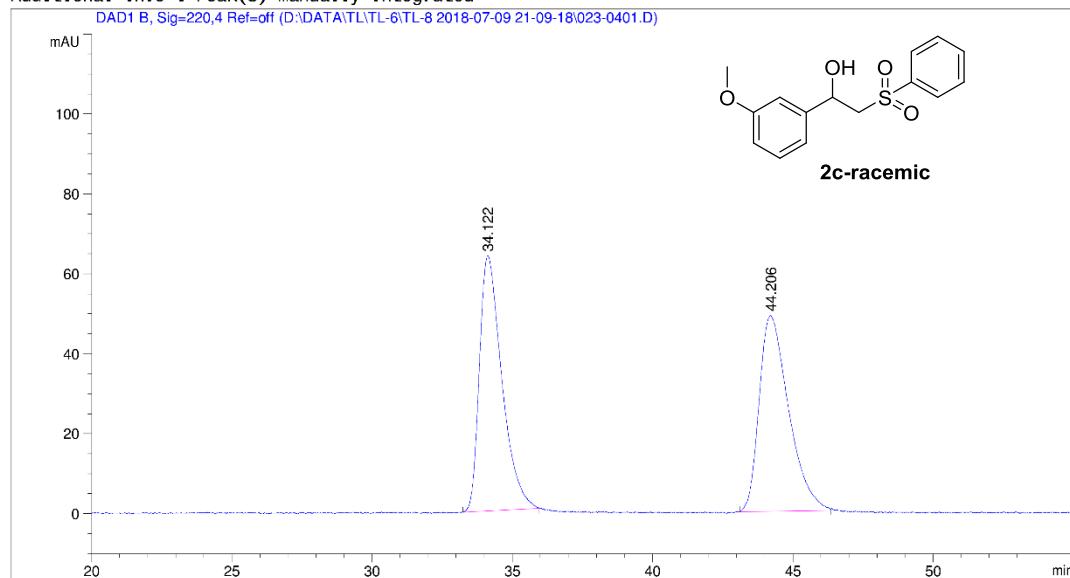
Totals : 2.95899e4 477.58624

Instrument 2 8/20/2018 9:20:39 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\023-0401.D  
Sample Name: TL-3-MeO-ph-rac

=====  
Acq. Operator : Seq. Line : 4  
Acq. Instrument : Instrument 2 Location : Vial 23  
Injection Date : 7/9/2018 10:43:17 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\DA(1-6)-80-20-1ML-3UL-ALL-  
55MIN.M  
Last changed : 7/9/2018 9:15:39 PM  
Analysis Method : D:\METHOD\NLWD\DA(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:42:06 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 34.122        | BB   | 0.6678      | 3548.98901   | 63.91422     | 49.8698 |
| 2      | 44.206        | BV   | 0.8612      | 3567.52515   | 49.01395     | 50.1302 |

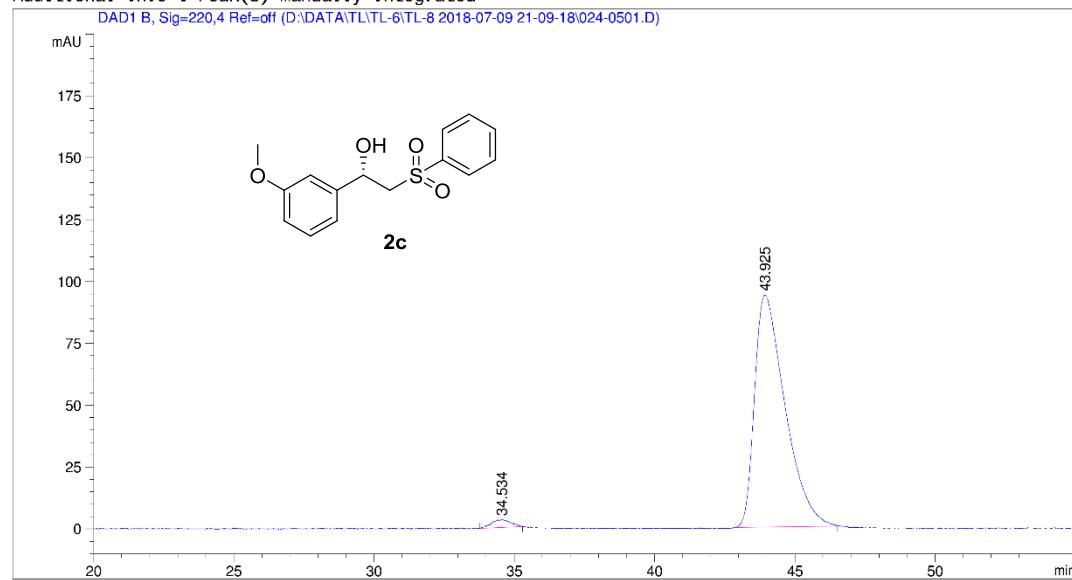
Totals : 7116.51416 112.92818

Instrument 2 8/20/2018 9:42:08 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\024-0501.D  
Sample Name: TL-3-MeO-ph-ee

=====  
Acq. Operator : Seq. Line : 5  
Acq. Instrument : Instrument 2 Location : Vial 24  
Injection Date : 7/9/2018 11:39:15 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\024-0501.D  
Last changed : 7/9/2018 9:15:39 PM  
Analysis Method : D:\METHOD\NLWD\024-0501.D  
Last changed : 8/20/2018 9:42:59 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 34.534        | BB   | 0.5624      | 149.22067    | 3.15135      | 2.0634  |
| 2      | 43.925        | BB   | 0.8869      | 7082.66650   | 93.80273     | 97.9366 |

Totals : 7231.88718 96.95408

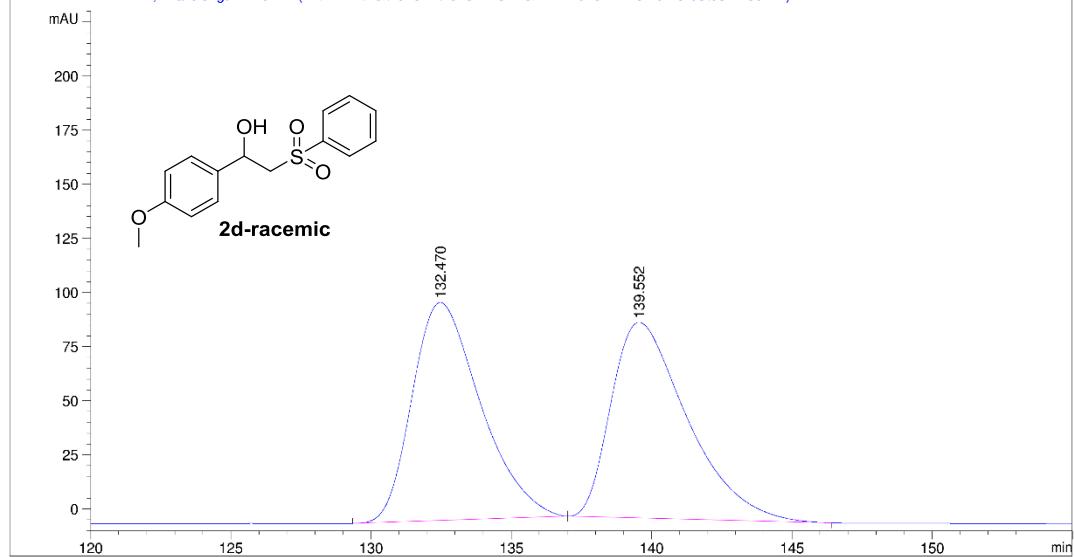
Instrument 2 8/20/2018 9:43:01 PM

Page 1 of 2

Data File D:\DATA\LG\201812\20181213-LIGAND 2018-12-13 20-18-03\031-1501.D  
Sample Name: TL-4-MeO-PH-RAC

```
=====
Acq. Operator   :                               Seq. Line : 15
Acq. Instrument : Instrument 1               Location  : Vial 31
Injection Date  : 12/14/2018 3:10:21 AM        Inj       : 1
                                                Inj Volume : 3.000 μl
Acq. Method     : D:\DATA\LG\201812\20181213-LIGAND 2018-12-13 20-18-03\VWD-AD(1-2)-90-10-0.
                           5ML-3UL-210NM-200MIN.M
Last changed    : 12/13/2018 9:25:57 PM
Analysis Method : D:\METHOD\TL\DA0-0J(1-6)-80-20-1ML-3UL-ALL-50MIN.M
Last changed    : 12/18/2018 8:01:06 PM
                           (modified after loading)
Additional Info : Peak(s) manually integrated
```

VWD1 A, Wavelength=210 nm (D:\DATA\LG\201812\20181212\20181212)



## Area Percent Report

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

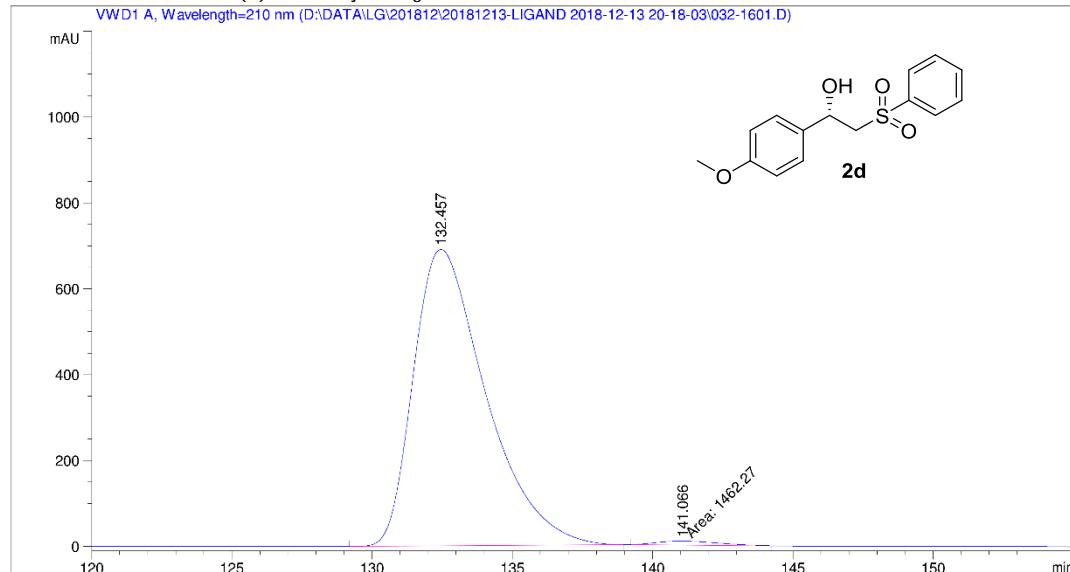
| Peak #          | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|-----------------|---------------|------|-------------|--------------|--------------|---------|
| 1               | 132.470       | BB   | 2.5083      | 1.70786e4    | 100.71841    | 50.0808 |
| 2               | 139.552       | BB   | 2.6355      | 1.70235e4    | 90.37720     | 49.9192 |
| <b>Totals :</b> |               |      |             | 3.41022e4    | 191.09560    |         |

Instrument 2 12/18/2018 8:01:12 PM

Page 1 of 2

Data File D:\DATA\LG\201812\20181213-LIGAND 2018-12-13 20-18-03\032-1601.D  
Sample Name: TL-4-MeO-PH-EE

=====  
Acq. Operator : Seq. Line : 16  
Acq. Instrument : Instrument 1 Location : Vial 32  
Injection Date : 12/14/2018 6:31:11 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\LG\201812\20181213-LIGAND 2018-12-13 20-18-03\VWD-AD(1-2)-90-10-0.  
5ML-3UL-210NM-200MIN.M  
Last changed : 12/13/2018 9:25:57 PM  
Analysis Method : D:\METHOD\TLNDAD-0J(1-6)-80-20-1ML-3UL-ALL-50MIN.M  
Last changed : 12/18/2018 8:03:44 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

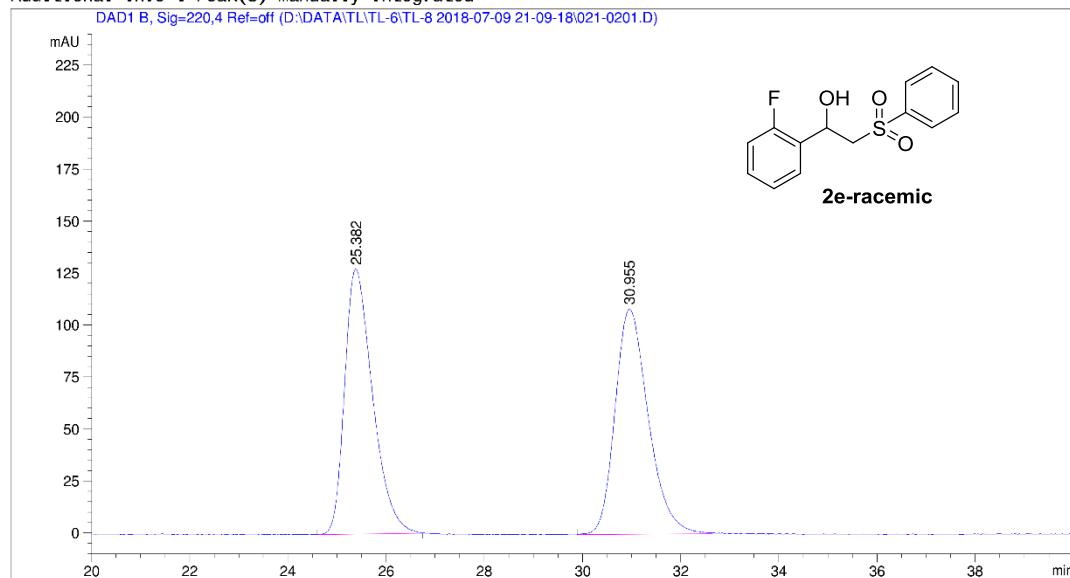
| #        | Peak RetTime | Type | Width  | Area       | Height    | Area %  |
|----------|--------------|------|--------|------------|-----------|---------|
|          | [min]        |      | [min]  | [mAU*s]    | [mAU]     |         |
| 1        | 132.457      | BB   | 2.6948 | 1.23896e5  | 690.36621 | 98.8335 |
| 2        | 141.066      | MM   | 2.5153 | 1462.26697 | 9.68918   | 1.1665  |
| Totals : |              |      |        | 1.25358e5  | 700.05539 |         |

Instrument 2 12/18/2018 8:03:51 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\021-0201.D  
Sample Name: TL-2-F-ph-rac

=====  
Acq. Operator : Seq. Line : 2  
Acq. Instrument : Instrument 2 Location : Vial 21  
Injection Date : 7/9/2018 9:21:21 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\021-0201.D  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\021-0201.D  
Last changed : 8/20/2018 9:47:55 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 25.382        | BB   | 0.5636      | 5049.20996   | 127.61078    | 49.7639 |
| 2      | 30.955        | BV   | 0.6407      | 5097.11279   | 108.38634    | 50.2361 |

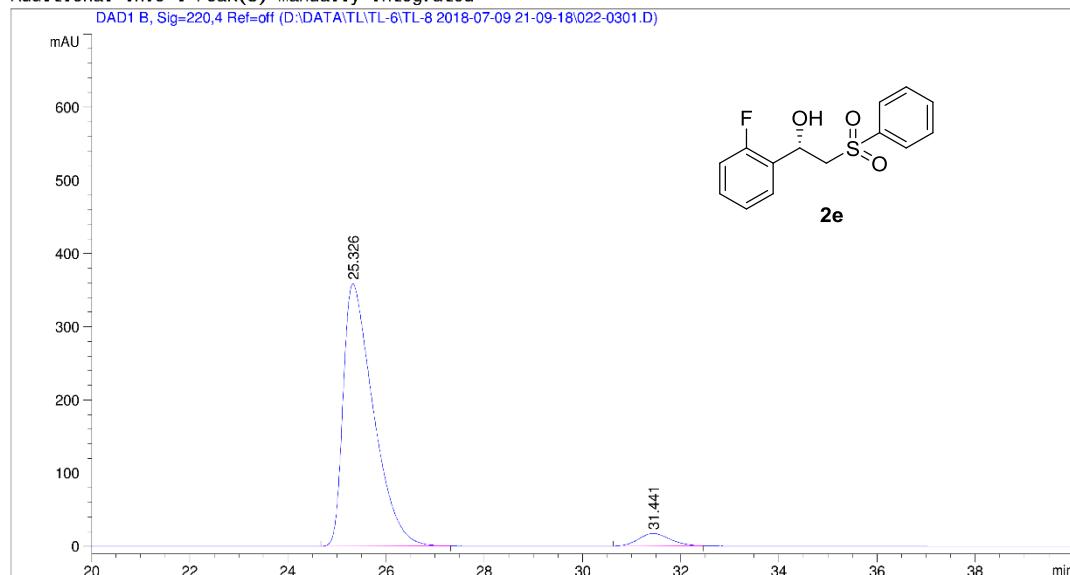
Totals : 1.01463e4 235.99712

Instrument 2 8/20/2018 9:47:58 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\022-0301.D  
Sample Name: TL-2-F-ph-ee

=====  
Acq. Operator : Seq. Line : 3  
Acq. Instrument : Instrument 2 Location : Vial 22  
Injection Date : 7/9/2018 10:02:17 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\DAD-OJ(1-6)-80-20-1ML-3UL-ALL-40MIN.M  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\NDAD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:48:53 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 25.326        | BB   | 0.6149      | 1.52677e4    | 358.47754    | 95.1553 |
| 2      | 31.441        | BV   | 0.5417      | 777.33984    | 17.12177     | 4.8447  |

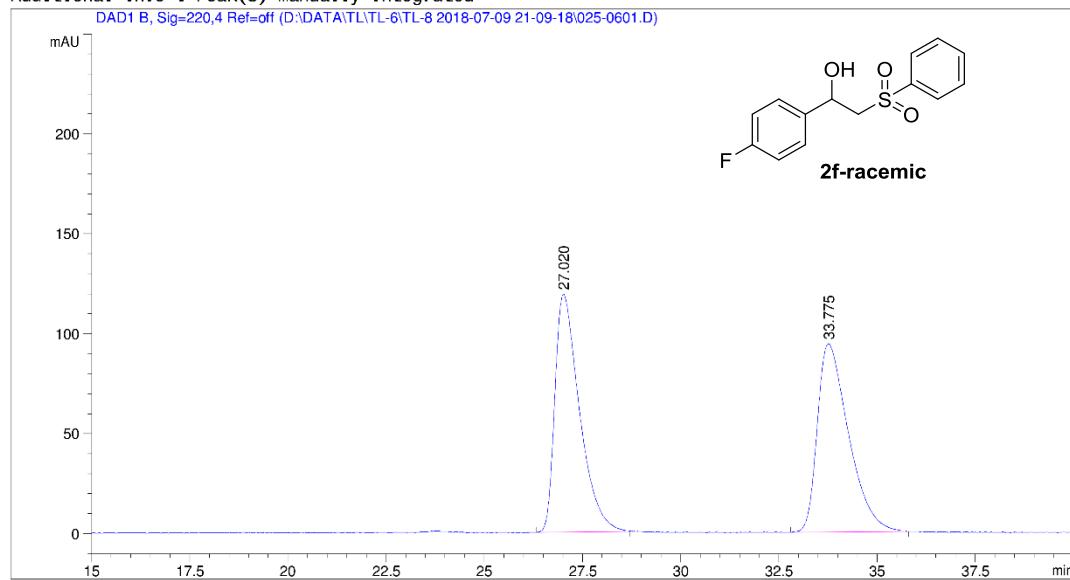
Totals : 1.60450e4 375.59930

Instrument 2 8/20/2018 9:48:56 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\025-0601.D  
Sample Name: TL-4-F-ph-rac

=====  
Acq. Operator : Seq. Line : 6  
Acq. Instrument : Instrument 2 Location : Vial 25  
Injection Date : 7/10/2018 12:35:15 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\025-0601.D  
Last changed : 7/9/2018 9:24:11 PM  
Analysis Method : D:\METHOD\NLWD\025-0601.D  
Last changed : 8/20/2018 9:44:35 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 27.020        | BV   | 0.6321      | 5188.89307   | 119.14967    | 49.8486 |
| 2      | 33.775        | BB   | 0.6662      | 5220.41846   | 94.24884     | 50.1514 |

Totals : 1.04093e4 213.39851

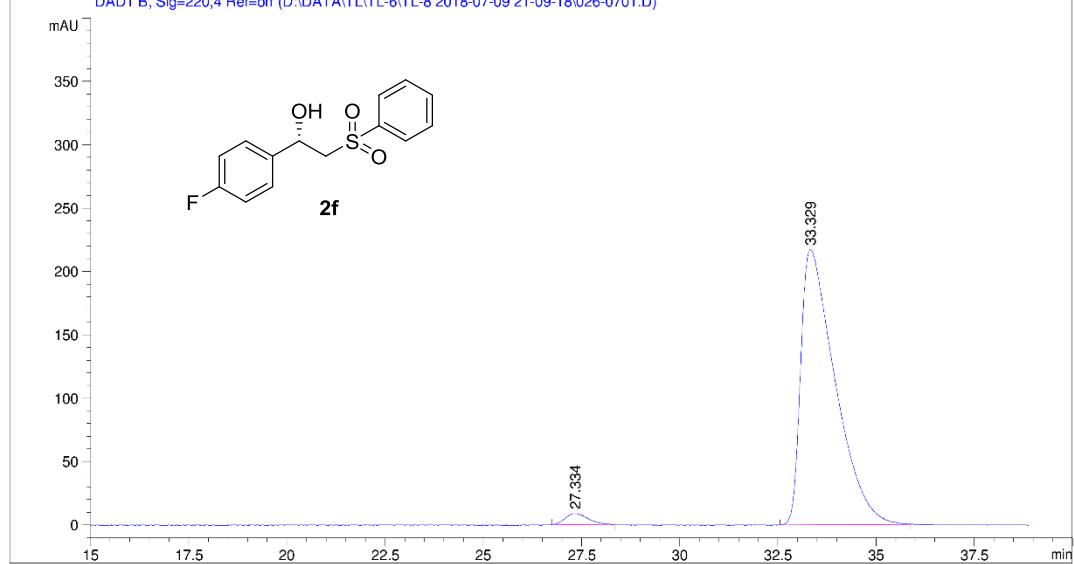
Instrument 2 8/20/2018 9:44:38 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\026-0701.D  
Sample Name: TL-4-F-ph-ree

```
=====
Acq. Operator   :                               Seq. Line : 7
Acq. Instrument : Instrument 2               Location  : Vial 26
Injection Date  : 7/10/2018 1:21:13 AM        Inj       : 1
                                                Inj Volume : 3.000 μl
Acq. Method    : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\DAD-OJ(1-6)-80-20-1ML-3UL-ALL-
                  45MIN.M
Last changed   : 7/9/2018 9:24:11 PM
Analysis Method: D:\METHOD\LWD\AD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M
Last changed   : 8/20/2018 9:46:09 PM
                  (modified after loading)
Additional Info: Peak(s) manually integrated
```

DAD1 B, Sig=220.4 Ref=off (D:\DATA\TL\TL-6\TL-8 2018-0



## Area Percent Report

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 27.334        | BV   | 0.4831      | 363.98114    | 8.86326      | 2.6803  |
| 2      | 28.260        | BV   | 0.4831      | 16.95177     | 0.45200      | 0.13610 |

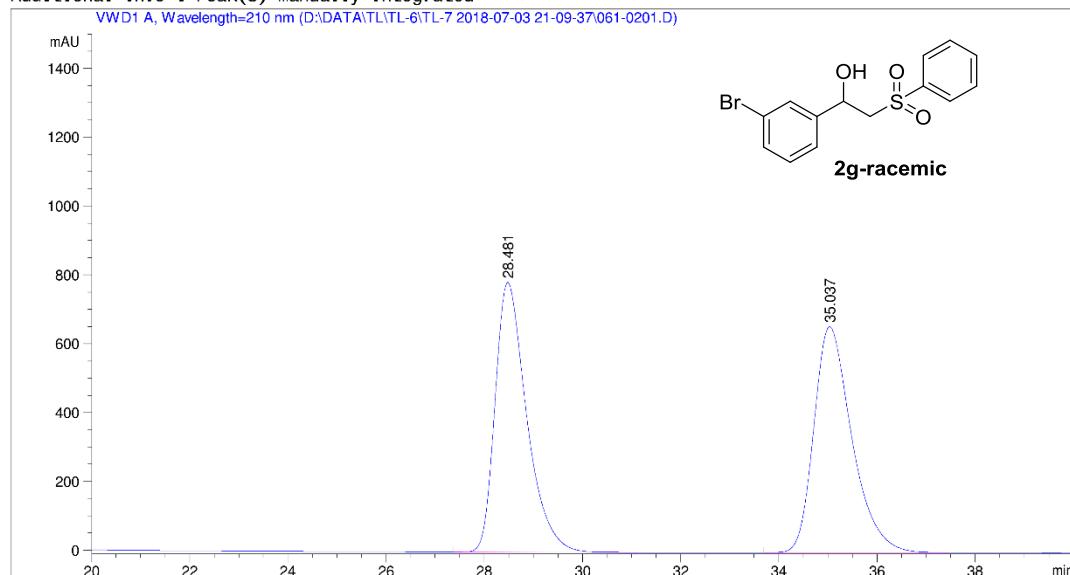
Totals : 1 357964 326 19462

Instrument 3 8/20/2018 9:46:12 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-7 2018-07-03 21-09-37\061-0201.D  
Sample Name: TL-3-Br-ph-rac

=====  
Acq. Operator : Seq. Line : 2  
Acq. Instrument : Instrument 1 Location : Vial 61  
Injection Date : 7/3/2018 9:21:26 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-7 2018-07-03 21-09-37\VWD-AD(1-2)-90-10-1ML-3UL-210NM-  
40MIN.M  
Last changed : 7/3/2018 9:05:40 PM  
Analysis Method : D:\METHOD\NLWD\AD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:35:20 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 28.481        | BB   | 0.6761      | 3.49141e4    | 783.19885    | 49.7706 |
| 2      | 35.037        | BB   | 0.8215      | 3.52360e4    | 656.15796    | 50.2294 |

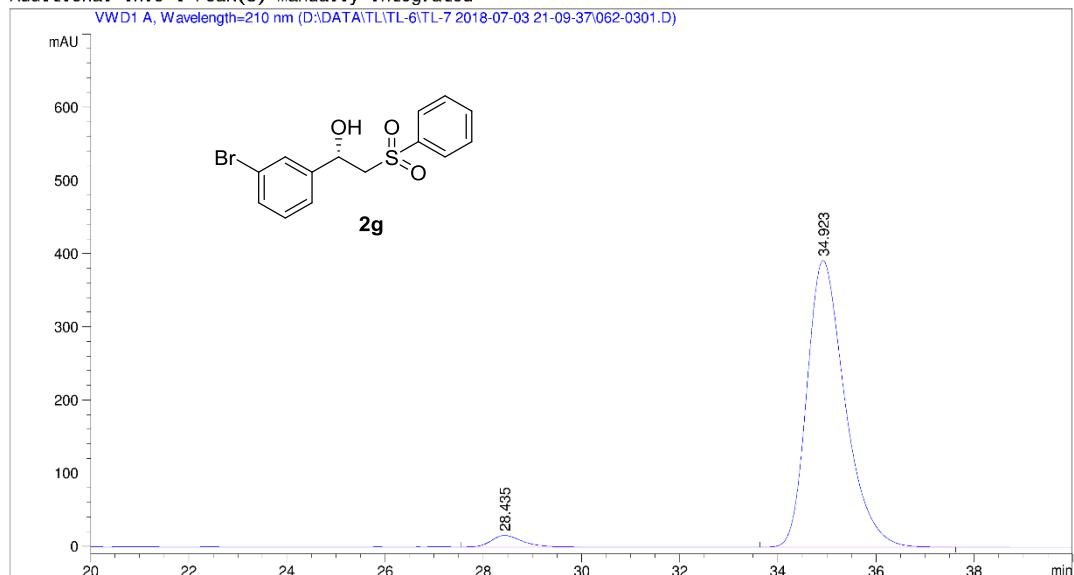
Totals : 7.01501e4 1439.35681

Instrument 2 8/20/2018 9:35:25 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-7 2018-07-03 21-09-37\062-0301.D  
Sample Name: TL-3-Br-ph-ree

```
=====
Acq. Operator   :                               Seq. Line : 3
Acq. Instrument : Instrument 1               Location : Vial 62
Injection Date  : 7/3/2018 10:02:16 PM          Inj : 1
                                                Inj Volume : 3.000 μl
Acq. Method    : D:\DATA\TL\TL-6\TL-7 2018-07-03 21-09-37\VWD-AD(1-2)-90-10-1ML-3UL-210NM-
                  40MIN.M
Last changed   : 7/3/2018 9:05:40 PM
Analysis Method : D:\METHOD\LWD\AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M
Last changed   : 8/20/2018 9:37:46 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



## Area Percent Report

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

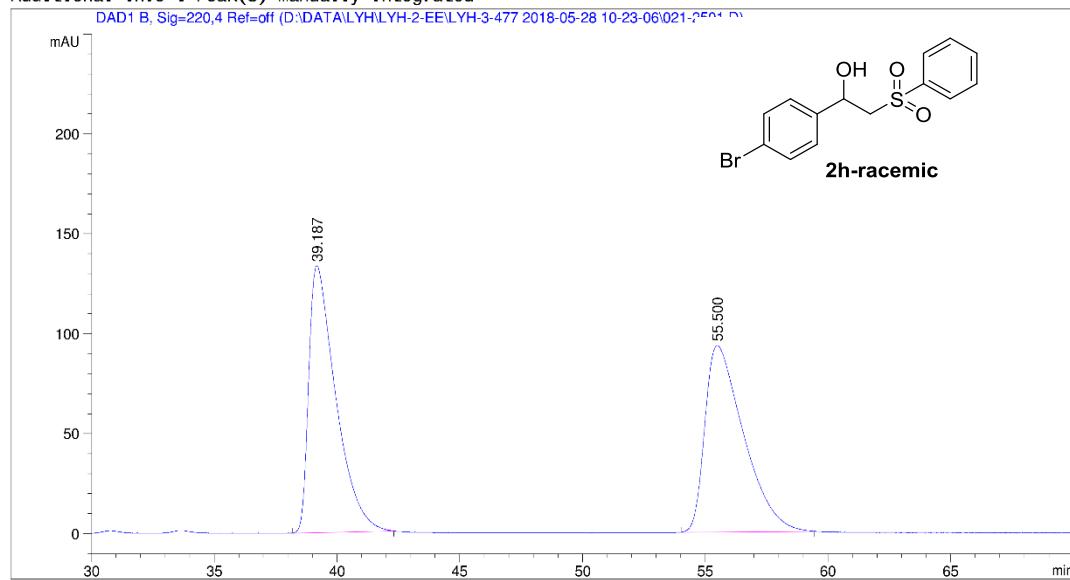
| Peak #          | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|-----------------|---------------|------|-------------|--------------|--------------|---------|
| 1               | 28.435        | BB   | 0.6577      | 694.79932    | 15.81091     | 3.2002  |
| 2               | 34.923        | BB   | 0.8195      | 2.10163e4    | 391.36459    | 96.7998 |
| <b>Totals :</b> |               |      |             | 2.17111e4    | 407.17551    |         |

Instrument 2 8/20/2018 9:37:49 PM

Page 1 of 2

Data File D:\DATA\LYH\LYH-2-EE\LYH-3-477 2018-05-28 10-23-06\021-2501.D  
Sample Name: **tl-4-Br-ph-rac**

=====  
Acq. Operator : Seq. Line : 25  
Acq. Instrument : Instrument 2 Location : Vial 21  
Injection Date : 5/29/2018 5:22:01 AM Inj : 1  
Inj Volume : 2.000  $\mu$ l  
Acq. Method : D:\DATA\LYH\LYH-2-EE\LYH-3-477 2018-05-28 10-23-06\DAD-OJ(1-6)-80-20-1ML-  
2UL-ALL-80MIN.M  
Last changed : 5/28/2018 4:24:25 PM  
Analysis Method : D:\METHOD\YCC\DA<sup>D</sup>D-OJ(1-6)-80-20-1ML-1UL-ALL-60MIN.M  
Last changed : 11/6/2018 9:03:47 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
**Area Percent Report**  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 39.187        | BB   | 0.9226      | 1.02205e4    | 133.58096    | 49.9718 |
| 2      | 55.500        | BB   | 1.2836      | 1.02320e4    | 93.43902     | 50.0282 |

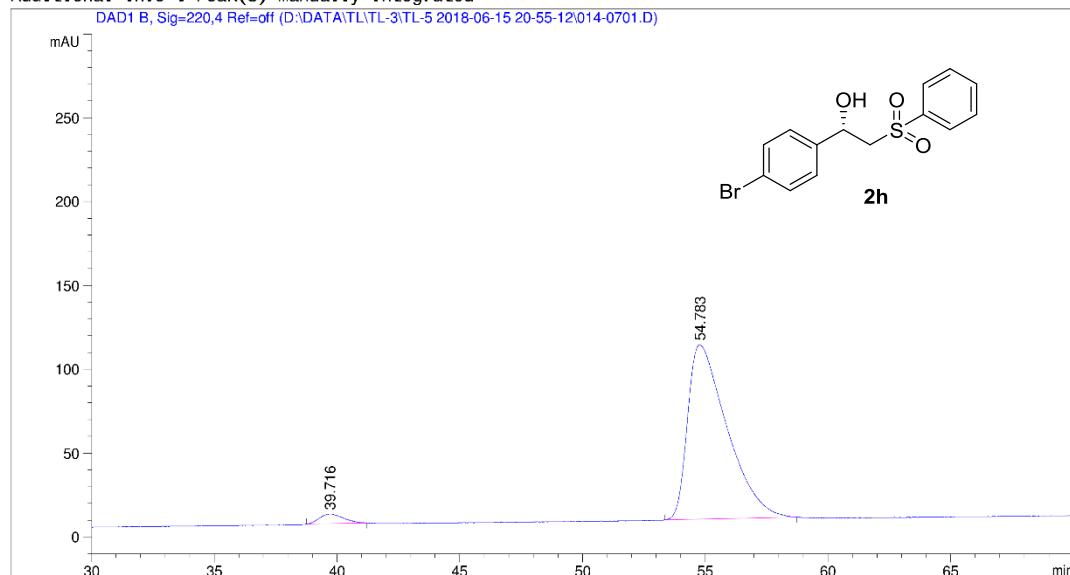
Totals : 2.04525e4 227.01998

Instrument 2 11/6/2018 9:04:03 PM

Page 1 of 2

Data File D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\014-0701.D  
Sample Name: TL-4-Br-ph-rac

=====  
Acq. Operator : Seq. Line : 7  
Acq. Instrument : Instrument 2 Location : Vial 14  
Injection Date : 6/15/2018 11:22:06 PM Inj : 1  
Inj Volume : 2.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\DAD-OJ(1-6)-80-20-1ML-2UL-ALL-70MIN.M  
Last changed : 6/15/2018 8:43:36 PM  
Analysis Method : D:\METHOD\NLWD\DA(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 8:50:57 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated

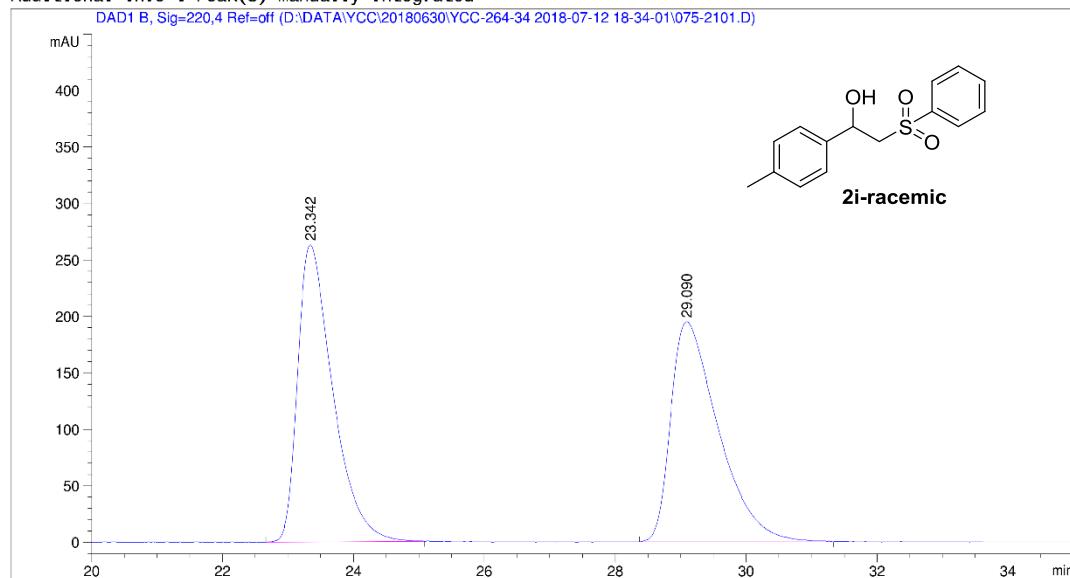


Instrument 2 8/20/2018 8:53:43 PM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\075-2101.D  
Sample Name: TL-4-Me-ph-rac

=====  
Acq. Operator : Seq. Line : 21  
Acq. Instrument : Instrument 2 Location : Vial 75  
Injection Date : 7/13/2018 4:22:03 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\075-2101.D  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\075-2101.D  
Last changed : 8/20/2018 9:50:39 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
**Area Percent Report**  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 23.342        | BB   | 0.5393      | 9802.63770   | 262.74643    | 49.9829 |
| 2      | 29.090        | BB   | 0.6898      | 9809.33984   | 194.69162    | 50.0171 |

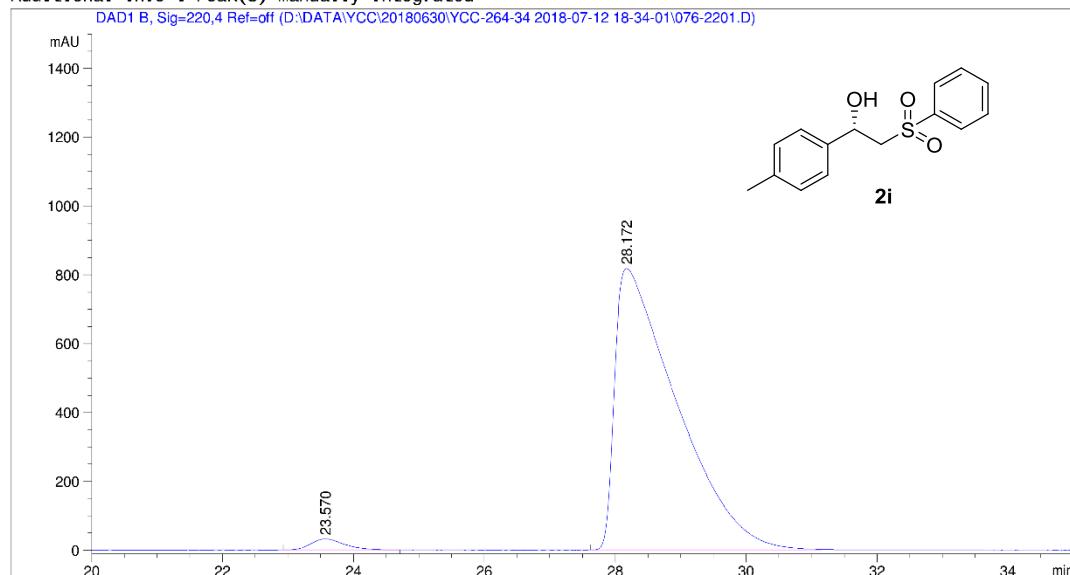
Totals : 1.96120e4 457.43805

Instrument 2 8/20/2018 9:50:42 PM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\076-2201.D  
Sample Name: TL-4-Me-ph-ee

=====  
Acq. Operator : Seq. Line : 22  
Acq. Instrument : Instrument 2 Location : Vial 76  
Injection Date : 7/13/2018 5:03:02 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\076-2201.D  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\076-2201.D  
Last changed : 8/20/2018 9:52:13 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 23.570        | BV   | 0.4595      | 1160.58228   | 32.12264     | 2.1156  |
| 2      | 28.172        | BV   | 0.8518      | 5.36976e4    | 818.01746    | 97.8844 |

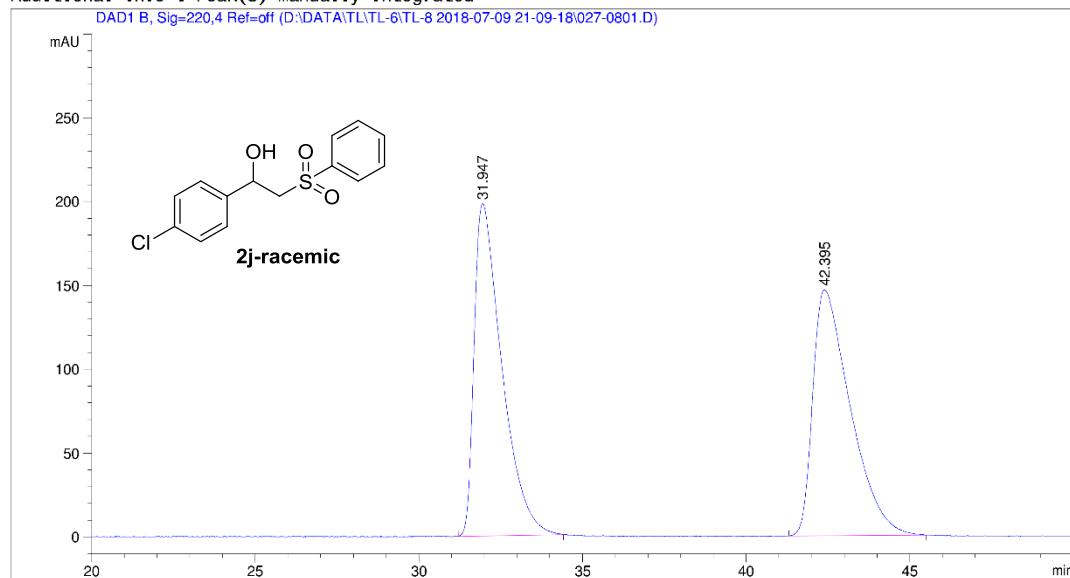
Totals : 5.48582e4 850.14009

Instrument 2 8/20/2018 9:52:18 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\027-0801.D  
Sample Name: TL-4-Cl-ph-rac

=====  
Acq. Operator : Seq. Line : 8  
Acq. Instrument : Instrument 2 Location : Vial 27  
Injection Date : 7/10/2018 2:07:14 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\027-0801.D  
Last changed : 7/9/2018 9:28:06 PM  
Analysis Method : D:\METHOD\NLW\027-0801.D  
Last changed : 8/20/2018 9:08:17 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 31.947        | BB   | 0.7134      | 1.16421e4    | 198.17842    | 49.8995 |
| 2      | 42.395        | BB   | 0.9343      | 1.16890e4    | 146.59465    | 50.1005 |

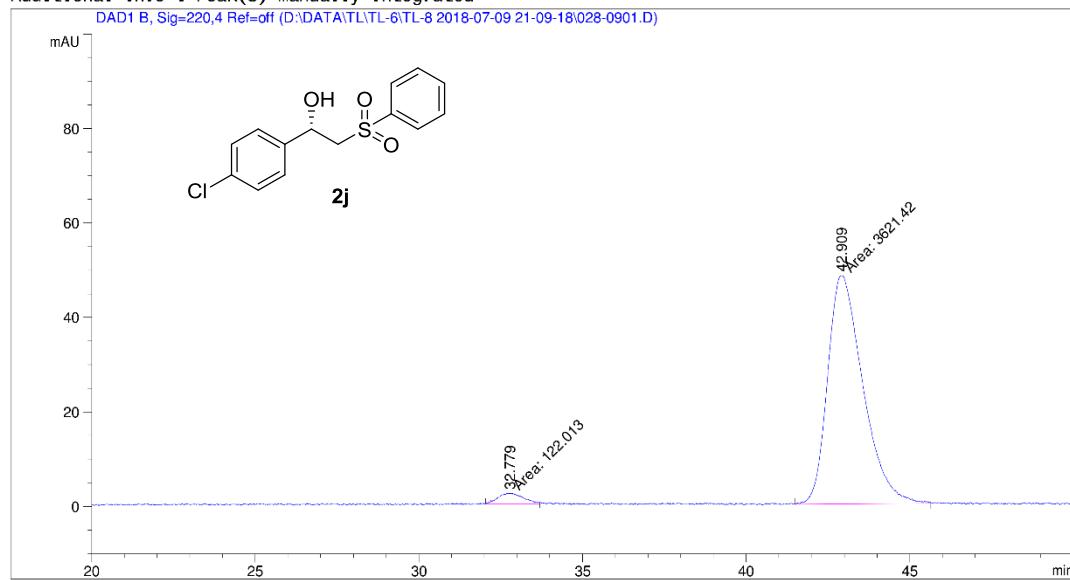
Totals : 2.33311e4 344.77307

Instrument 2 8/20/2018 9:08:19 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\028-0901.D  
Sample Name: TL-4-Cl-ph-ree

=====  
Acq. Operator : Seq. Line : 9  
Acq. Instrument : Instrument 2 Location : Vial 28  
Injection Date : 7/10/2018 2:58:11 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\028-0901.D  
Last changed : 7/9/2018 9:28:06 PM  
Analysis Method : D:\METHOD\NLWD\028-0901.D  
Last changed : 8/20/2018 9:10:55 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 32.779        | MM   | 0.9080      | 122.01331    | 2.23948      | 3.2594  |
| 2      | 42.909        | MM   | 1.2491      | 3621.41577   | 48.31980     | 96.7406 |

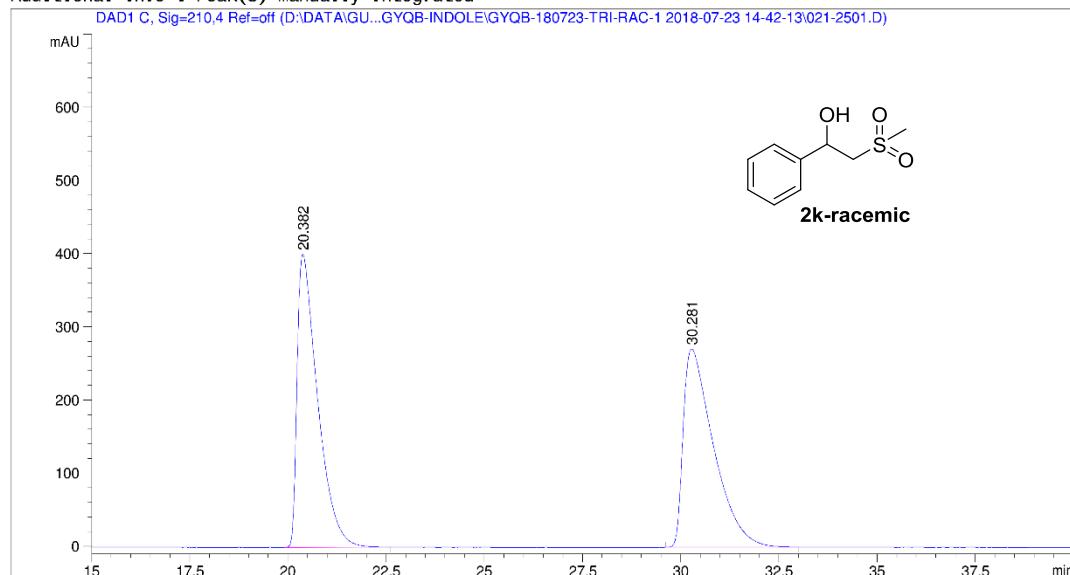
Totals : 3743.42908 50.55928

Instrument 2 8/20/2018 9:10:59 PM

Page 1 of 2

Data File D:\DATA\GU...NG\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13\021-2501.D  
Sample Name: TL-ph-Me-rac

=====  
Acq. Operator : Seq. Line : 25  
Acq. Instrument : Instrument 2 Location : Vial 21  
Injection Date : 7/24/2018 7:13:05 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\GUAN YUQING\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13  
\\DAD-OJ(1-6)-80-20-1ML-3UL-ALL-70MIN.M  
Last changed : 6/28/2018 8:52:23 PM  
Analysis Method : D:\METHOD\NLWD\DA<sup>D</sup>-AD(1-6)-90-10-0.7ML-3UL-ALL-70MIN.M  
Last changed : 8/21/2018 9:42:45 AM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,4 Ref=off

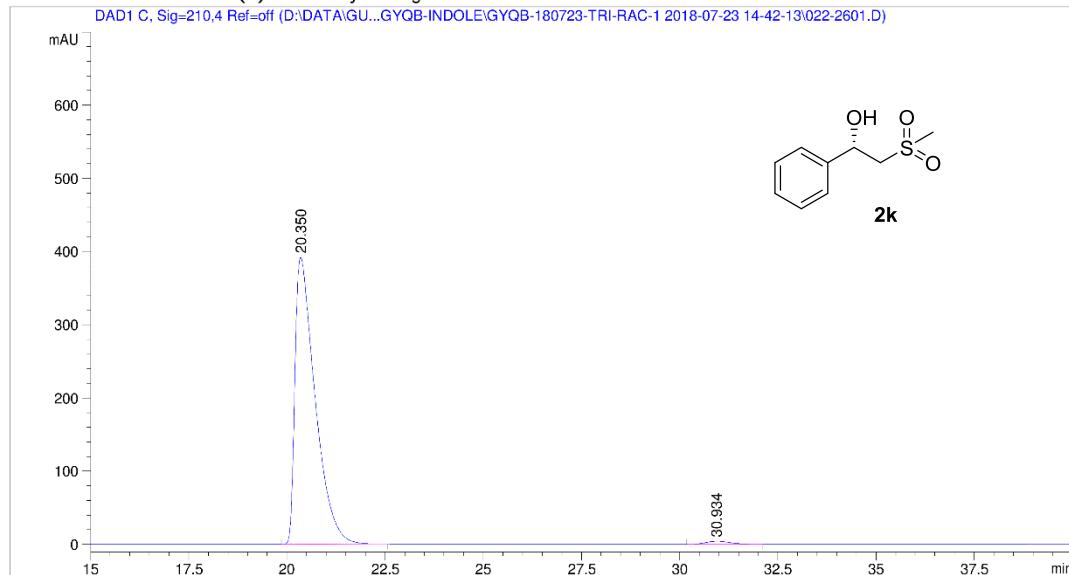
| #        | Peak RetTime | Type | Width  | Area      | Height    | Area %  |
|----------|--------------|------|--------|-----------|-----------|---------|
|          | [min]        |      | [min]  | [mAU*s]   | [mAU]     |         |
| 1        | 20.382       | BB   | 0.5401 | 1.44845e4 | 400.36893 | 49.9424 |
| 2        | 30.281       | BB   | 0.7887 | 1.45179e4 | 271.13010 | 50.0576 |
| Totals : |              |      |        | 2.90025e4 | 671.49902 |         |

Instrument 2 8/21/2018 9:42:47 AM

Page 1 of 2

Data File D:\DATA\GU...NG\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13\022-2601.D  
Sample Name: TL-ph-Me-ee

=====  
Acq. Operator : Seq. Line : 26  
Acq. Instrument : Instrument 2 Location : Vial 22  
Injection Date : 7/24/2018 8:24:03 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\GUAN YUQING\GYQB-INDOLE\GYQB-180723-TRI-RAC-1 2018-07-23 14-42-13  
\\DAD-OJ(1-6)-80-20-1ML-3UL-ALL-70MIN.M  
Last changed : 7/24/2018 9:10:19 AM  
(modified after loading)  
Analysis Method : D:\METHOD\LWD\DAE-AD(1-6)-90-10-0.7ML-3UL-ALL-70MIN.M  
Last changed : 8/21/2018 9:42:45 AM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210.4 Ref=off

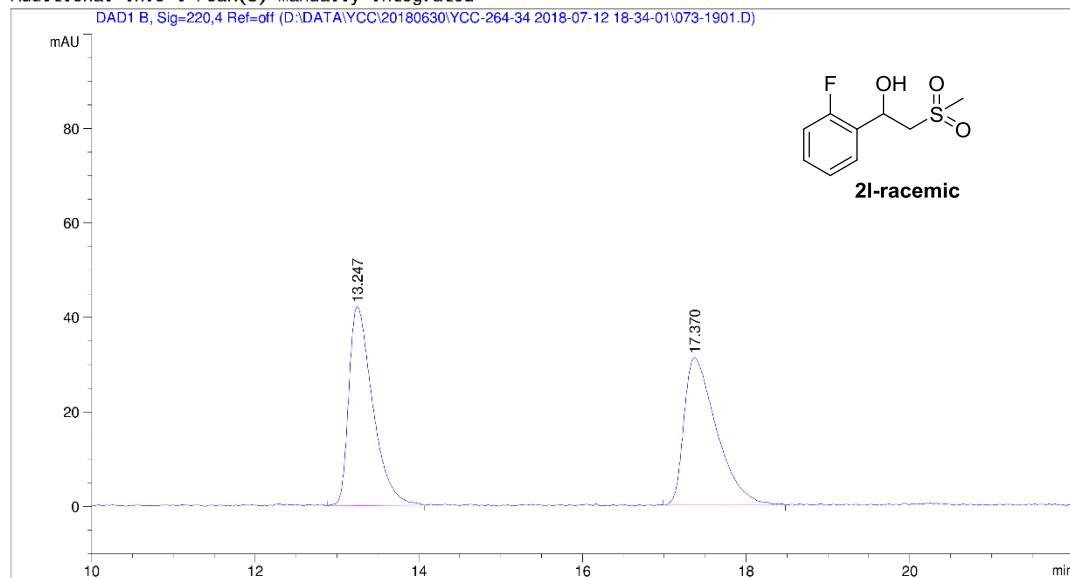
| Peak #   | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|----------|---------------|------|-------------|--------------|--------------|---------|
| 1        | 20.350        | BB   | 0.5244      | 1.39980e4    | 392.23517    | 98.6656 |
| 2        | 30.934        | BB   | 0.5349      | 189.31180    | 4.23998      | 1.3344  |
| Totals : |               |      |             | 1.41873e4    | 396.47515    |         |

Instrument 2 8/21/2018 9:43:49 AM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\073-1901.D  
Sample Name: TL-2-F-Me-rac

=====  
Acq. Operator : Seq. Line : 19  
Acq. Instrument : Instrument 2 Location : Vial 73  
Injection Date : 7/13/2018 3:29:59 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\DAD-0J(1-6)-80-20-1ML-  
3UL-ALL-25MIN.M  
Last changed : 7/12/2018 10:13:08 PM  
Analysis Method : D:\METHOD\NLWD\NDAD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 10:03:57 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 13.247        | BB   | 0.2888      | 855.37360    | 42.14367     | 49.7959 |
| 2      | 17.370        | BB   | 0.3838      | 862.38611    | 31.26658     | 50.2041 |

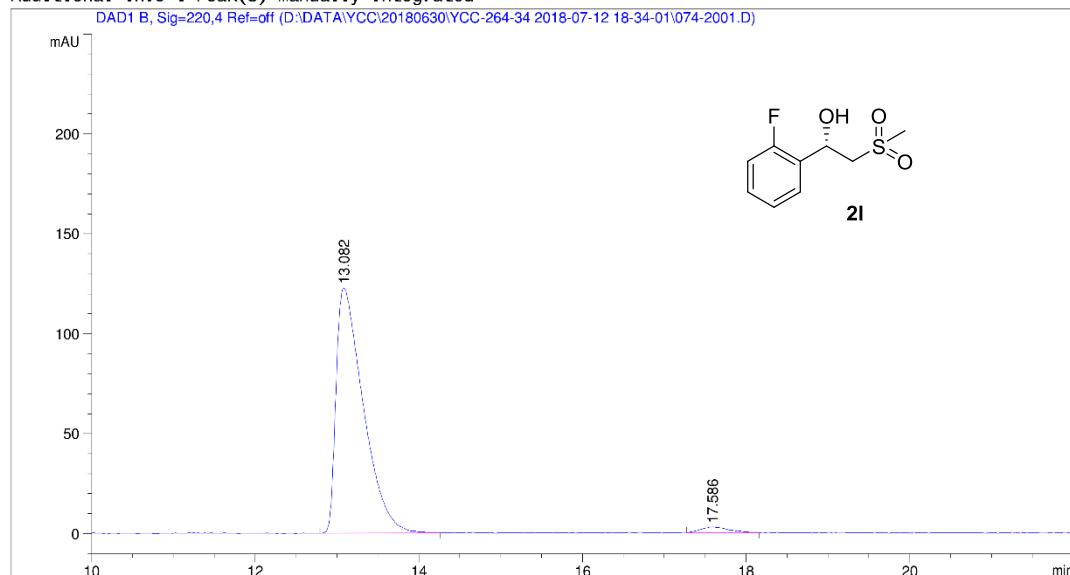
Totals : 1717.75970 73.41025

Instrument 2 8/20/2018 10:03:59 PM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\074-2001.D  
Sample Name: TL-2-F-Me-ee

=====  
Acq. Operator : Seq. Line : 20  
Acq. Instrument : Instrument 2 Location : Vial 74  
Injection Date : 7/13/2018 3:56:01 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\DAD-0J(1-6)-80-20-1ML-  
3UL-ALL-25MIN.M  
Last changed : 7/12/2018 10:13:08 PM  
Analysis Method : D:\METHOD\NLWD\NDAD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 10:04:47 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
**Area Percent Report**  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 13.082        | BB   | 0.3272      | 2833.36401   | 122.77251    | 97.4357 |
| 2      | 17.586        | VV   | 0.2947      | 74.56754     | 3.01312      | 2.5643  |

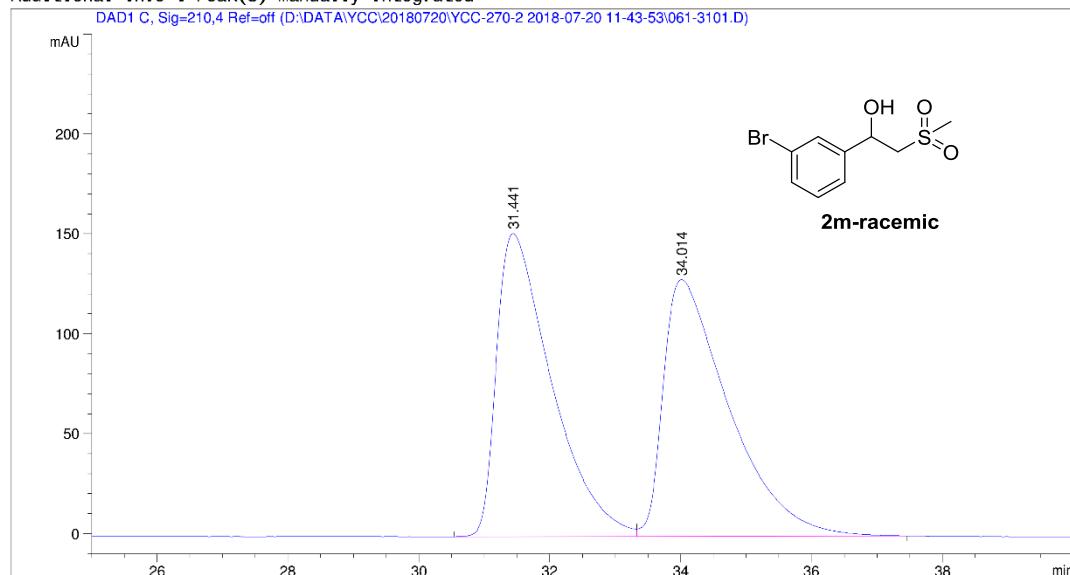
Totals : 2907.93155 125.78562

Instrument 2 8/20/2018 10:05:01 PM

Page 1 of 2

Data File D:\DATA\YCC\20180720\YCC-270-2 2018-07-20 11-43-53\061-3101.D  
Sample Name: TL-3-Br-Me-rac

=====  
Acq. Operator : Seq. Line : 31  
Acq. Instrument : Instrument 2 Location : Vial 61  
Injection Date : 7/21/2018 4:31:55 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180720\YCC-270-2 2018-07-20 11-43-53\DA(1-6)-80-20-1ML-  
3UL-ALL-40MIN.M  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\DA(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 10:07:47 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 31.441        | BV   | 0.8283      | 8846.16699   | 151.69662    | 49.7556 |
| 2      | 34.014        | VB   | 0.9737      | 8933.06934   | 128.39050    | 50.2444 |

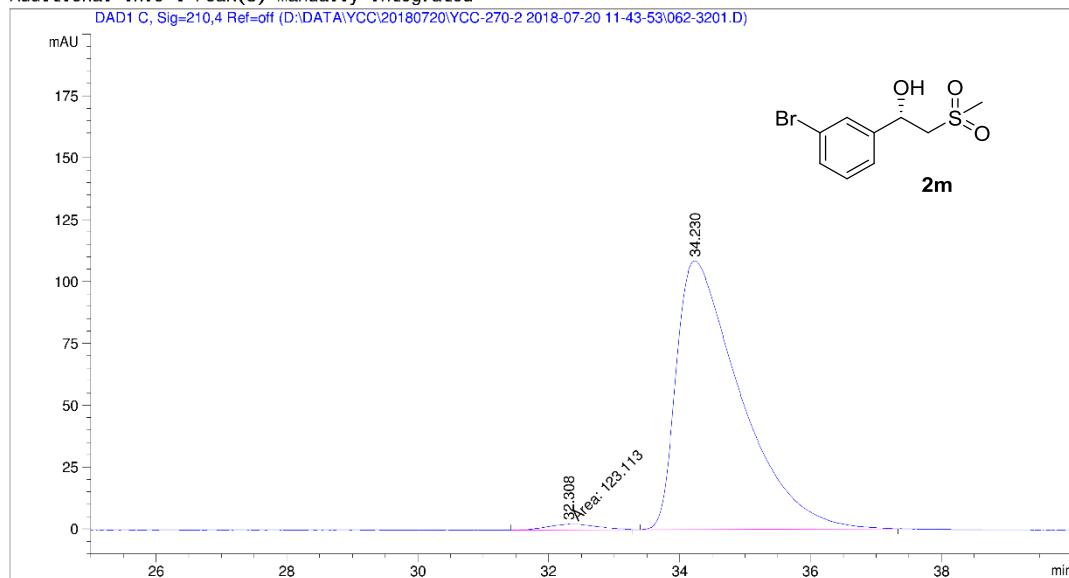
Totals : 1.77792e4 280.08713

Instrument 2 8/20/2018 10:07:54 PM

Page 1 of 2

Data File D:\DATA\YCC\20180720\YCC-270-2 2018-07-20 11-43-53\062-3201.D  
Sample Name: TL-3-Br-Me-ee

=====  
Acq. Operator : Seq. Line : 32  
Acq. Instrument : Instrument 2 Location : Vial 62  
Injection Date : 7/21/2018 5:12:55 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180720\YCC-270-2 2018-07-20 11-43-53\062-3201.D  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\062-3201.D  
Last changed : 8/20/2018 10:09:09 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 32.308        | MM   | 0.8635      | 123.11281    | 2.37613      | 1.6439  |
| 2      | 34.230        | BB   | 0.9462      | 7366.04248   | 108.48158    | 98.3561 |

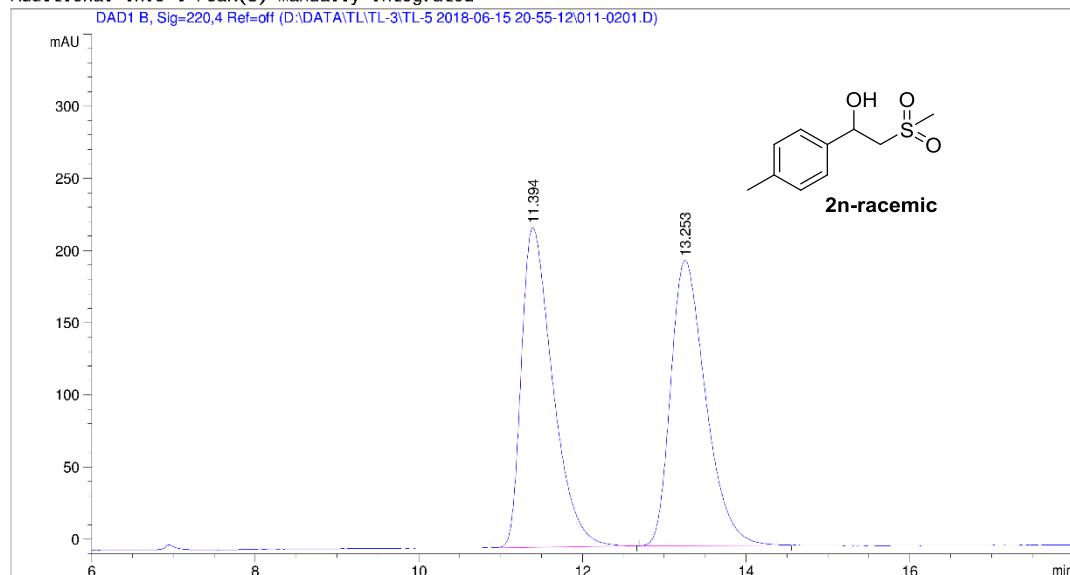
Totals : 7489.15529 110.85770

Instrument 2 8/20/2018 10:09:12 PM

Page 1 of 2

Data File D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\011-0201.D  
Sample Name: TL-4-Me-Me-rac

=====  
Acq. Operator : Seq. Line : 2  
Acq. Instrument : Instrument 2 Location : Vial 11  
Injection Date : 6/15/2018 9:07:15 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\DAD-OD(1-2)-80-20-1ML-3UL-ALL-20MIN.M  
Last changed : 6/15/2018 8:44:57 PM  
Analysis Method : D:\METHOD\NLWD\DA<sup>D</sup>-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:04:12 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| # | RetTime | Type | Width  | Area       | Height    | Area %  |
|---|---------|------|--------|------------|-----------|---------|
|   | [min]   |      | [min]  | [mAU*s]    | [mAU]     |         |
| 1 | 11.394  | BB   | 0.3997 | 5879.39258 | 221.48277 | 50.0507 |
| 2 | 13.253  | BB   | 0.4544 | 5867.47217 | 197.60023 | 49.9493 |

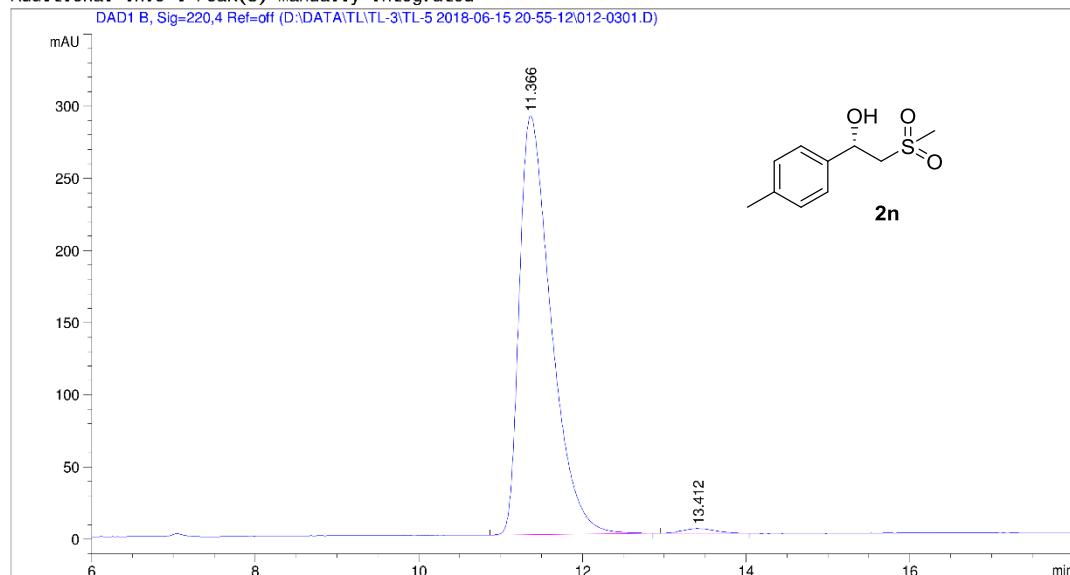
Totals : 1.17469e4 419.08301

Instrument 2 8/20/2018 9:04:25 PM

Page 1 of 2

Data File D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\012-0301.D  
Sample Name: TL-4-Me-Me-ee

=====  
Acq. Operator : Seq. Line : 3  
Acq. Instrument : Instrument 2 Location : Vial 12  
Injection Date : 6/15/2018 9:28:09 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-3\TL-5 2018-06-15 20-55-12\DAD-OD(1-2)-80-20-1ML-3UL-ALL-20MIN.M  
Last changed : 6/15/2018 8:44:57 PM  
Analysis Method : D:\METHOD\NLWD\DA<sup>D</sup>-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:04:12 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 11.366        | BB   | 0.3982      | 7818.25342   | 290.28690    | 98.8301 |
| 2      | 13.412        | BB   | 0.3304      | 92.54990     | 3.32796      | 1.1699  |

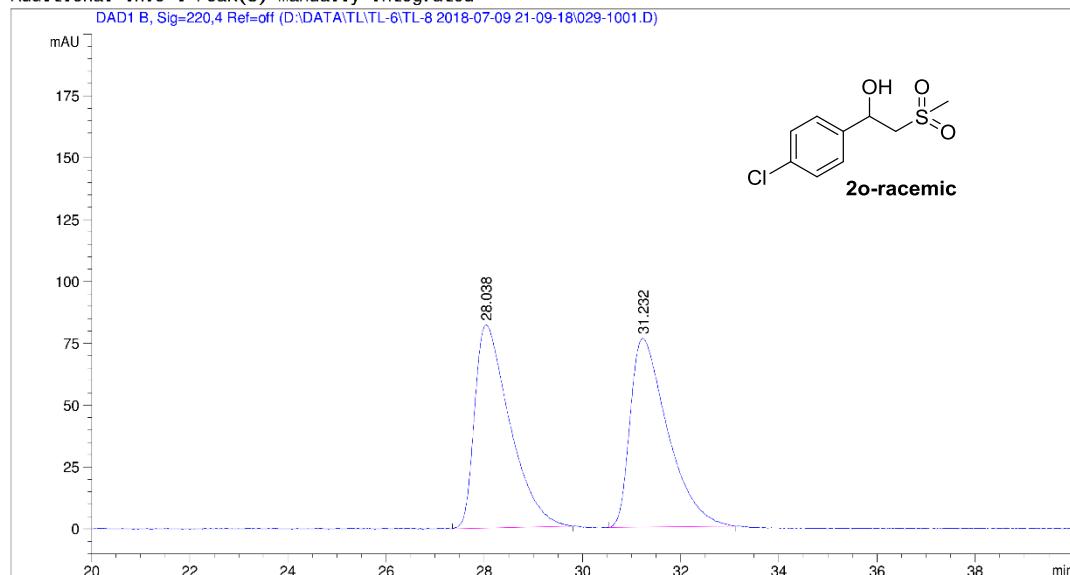
Totals : 7910.80331 293.61485

Instrument 2 8/20/2018 9:05:26 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\029-1001.D  
Sample Name: TL-4-Cl-Me-rac

=====  
Acq. Operator : Seq. Line : 10  
Acq. Instrument : Instrument 2 Location : Vial 29  
Injection Date : 7/10/2018 3:49:11 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\DA(1-6)-80-20-1ML-3UL-ALL-  
40MIN.M  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\DA(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:29:35 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 28.038        | BB   | 0.6477      | 4075.72192   | 82.03175     | 50.1508 |
| 2      | 31.232        | BB   | 0.6333      | 4051.21313   | 76.32463     | 49.8492 |

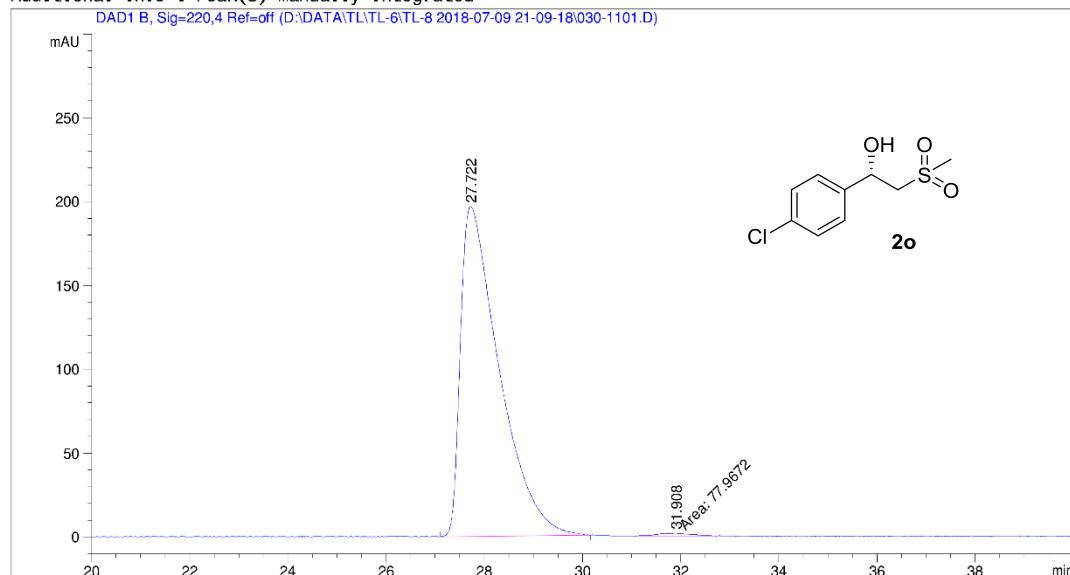
Totals : 8126.93506 158.35638

Instrument 2 8/20/2018 9:29:42 PM

Page 1 of 2

Data File D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\030-1101.D  
Sample Name: TL-4-Cl-Me-ee

=====  
Acq. Operator : Seq. Line : 11  
Acq. Instrument : Instrument 2 Location : Vial 30  
Injection Date : 7/10/2018 4:30:11 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\TL\TL-6\TL-8 2018-07-09 21-09-18\DAD-OJ(1-6)-80-20-1ML-3UL-ALL-40MIN.M  
Last changed : 7/9/2018 9:06:51 PM  
Analysis Method : D:\METHOD\NLWD\NDAD-AD(1-6)-95-5-1ML-2UL-ALL-20MIN.M  
Last changed : 8/20/2018 9:32:02 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 27.722        | BB   | 0.7384      | 1.07300e4    | 196.69144    | 99.2786 |
| 2      | 31.908        | MM   | 0.8399      | 77.96723     | 1.54711      | 0.7214  |

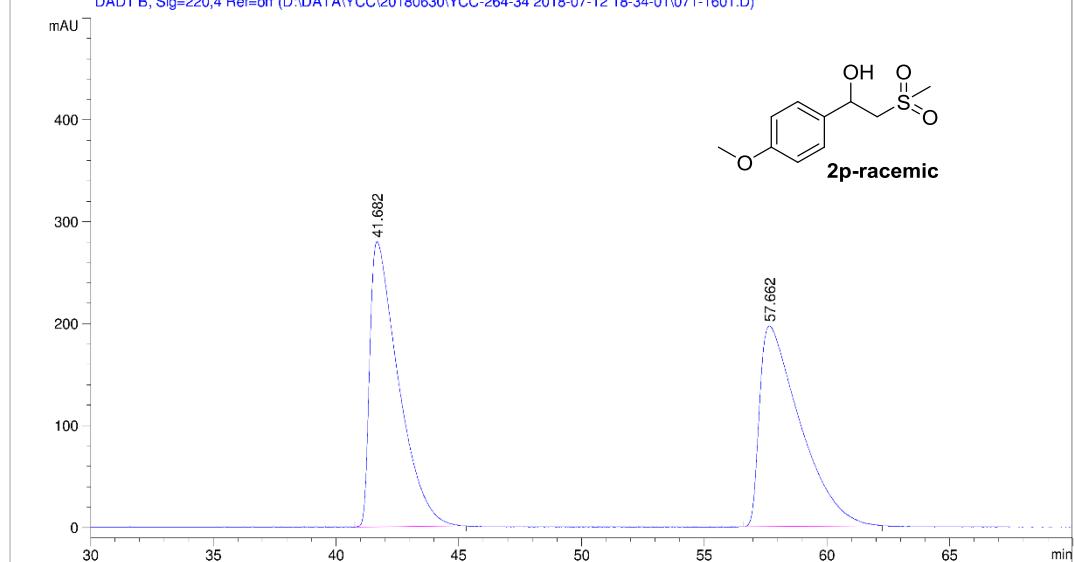
Totals : 1.08080e4 198.23854

Instrument 2 8/20/2018 9:32:04 PM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\071-1601.D  
Sample Name: TL-4-MeO-Me-rac

DAD1 B, Sig=220.4 Ref=off (D:\DATA\YCC\20180630\YCC)



## Area Percent Report

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 41.682        | BB   | 1.0604      | 2.26575e4    | 279.91312    | 50.1260 |
| 2      | 57.260        | BB   | 1.0570      | 2.05162e4    | 160.20317    | 39.3715 |

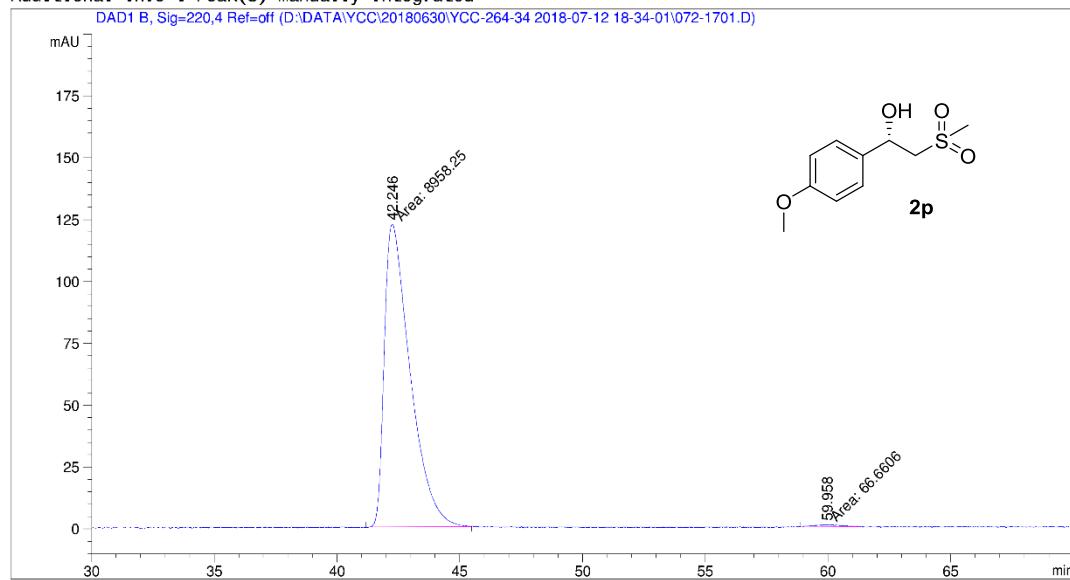
Total : 4,520,106.4 476 874.28

Instrument 3 8/20/2018 9:23:58 PM

Page 1 of 2

Data File D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\072-1701.D  
Sample Name: TL-4-MeO-Me-ee

=====  
Acq. Operator : Seq. Line : 17  
Acq. Instrument : Instrument 2 Location : Vial 72  
Injection Date : 7/13/2018 2:07:59 AM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\YCC\20180630\YCC-264-34 2018-07-12 18-34-01\072-1701.D  
Last changed : 6/28/2018 8:52:23 PM  
Analysis Method : D:\METHOD\NLWD\072-1701.D  
Last changed : 8/20/2018 9:26:49 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| # | RetTime | Type | Width  | Area       | Height     | Area %  |
|---|---------|------|--------|------------|------------|---------|
|   | [min]   |      | [min]  | [mAU*s]    | [mAU]      |         |
| 1 | 42.246  | MM   | 1.2194 | 8958.25293 | 122.44077  | 99.2614 |
| 2 | 59.958  | MM   | 1.2371 | 66.66060   | 8.98066e-1 | 0.7386  |

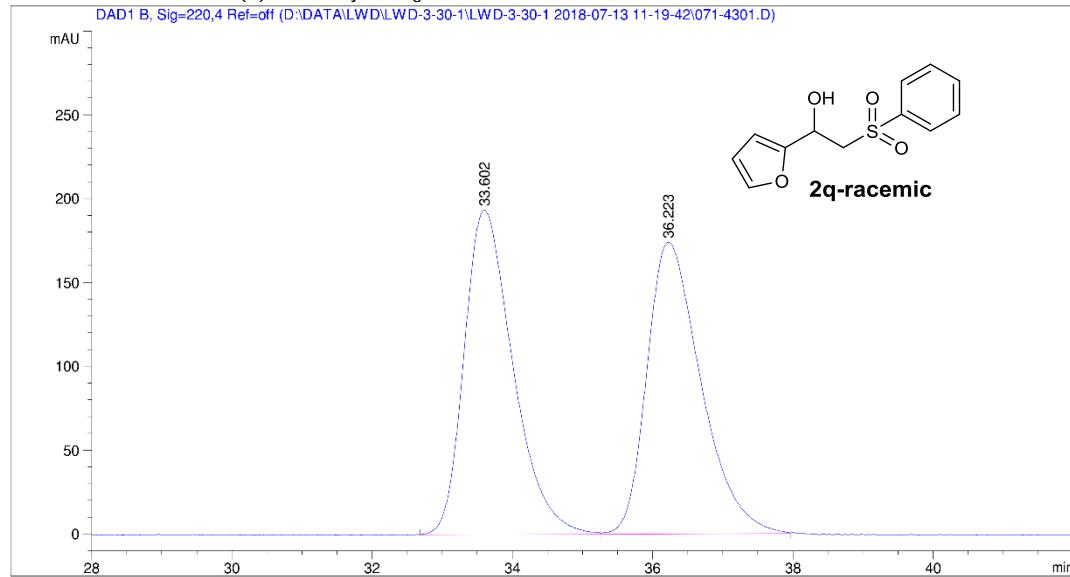
Totals : 9024.91353 123.33884

Instrument 2 8/20/2018 9:26:53 PM

Page 1 of 2

Data File D:\DATA\LWD\LWD-3-30-1\LWD-3-30-1 2018-07-13 11-19-42\071-4301.D  
Sample Name: TL-funan-ph

=====  
Acq. Operator : Seq. Line : 43  
Acq. Instrument : Instrument 2 Location : Vial 71  
Injection Date : 7/14/2018 12:41:17 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\LWD\LWD-3-30-1\LWD-3-30-1 2018-07-13 11-19-42\DAJ(1-6)-80-20-1ML-3UL-ALL-70MIN.M  
Last changed : 6/28/2018 8:52:23 PM  
Analysis Method : D:\METHOD\YCC\DAJ(1-6)-80-20-1ML-1UL-ALL-60MIN.M  
Last changed : 11/6/2018 9:10:01 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 33.602        | BV   | 0.6659      | 9356.62012   | 193.59761    | 50.1146 |
| 2      | 36.223        | VV   | 0.7561      | 9313.82422   | 174.11975    | 49.8854 |

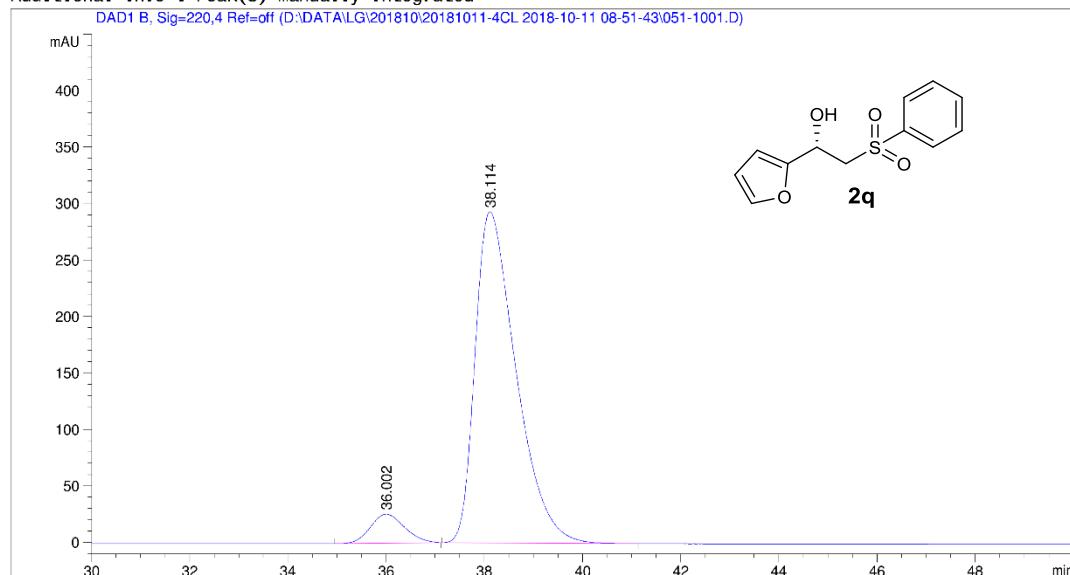
Totals : 1.86704e4 367.71736

Instrument 2 11/6/2018 9:10:15 PM

Page 1 of 2

Data File D:\DATA\LG\201810\20181011-4CL 2018-10-11 08-51-43\051-1001.D  
Sample Name: TL-funan-ph-ee

=====  
Acq. Operator : Seq. Line : 10  
Acq. Instrument : Instrument 2 Location : Vial 51  
Injection Date : 10/11/2018 1:18:47 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\LG\201810\20181011-4CL 2018-10-11 08-51-43\DA(D-OJ(1-6)-80-20-1ML-  
3UL-ALL-55MIN.M  
Last changed : 7/9/2018 9:15:39 PM  
Analysis Method : D:\METHOD\YCC\DA(D-OJ(1-6)-80-20-1ML-1UL-ALL-60MIN.M  
Last changed : 11/6/2018 9:13:59 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 36.002        | BB   | 0.7172      | 1234.81213   | 25.58069     | 6.7816  |
| 2      | 38.114        | BB   | 0.8775      | 1.69735e4    | 293.14279    | 93.2184 |

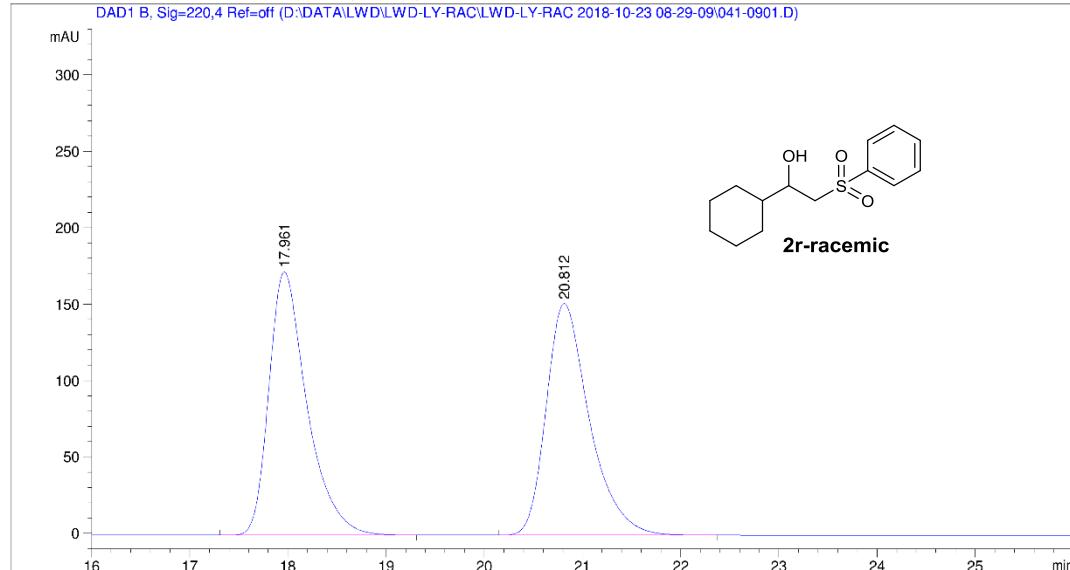
Totals : 1.82083e4 318.72349

Instrument 2 11/6/2018 9:14:21 PM

Page 1 of 2

Data File D:\DATA\LWD\LWD-LY-RAC\LWD-LY-RAC 2018-10-23 08-29-09\041-0901.D  
Sample Name: TL-C-HEX-PH-RAC

=====  
Acq. Operator : Seq. Line : 9  
Acq. Instrument : Instrument 2 Location : Vial 41  
Injection Date : 10/23/2018 12:29:02 PM Inj : 1  
Inj Volume : 3.000  $\mu$ l  
Acq. Method : D:\DATA\LWD\LWD-LY-RAC\LWD-LY-RAC 2018-10-23 08-29-09\DA(D(1-6)-80-20-0.  
7ML-3UL-ALL-60MIN.M  
Last changed : 10/23/2018 11:24:12 AM  
Analysis Method : D:\METHOD\LWD\DA(D(1-2)-80-20-1ML-3UL-ALL-10MIN.M  
Last changed : 11/17/2018 3:39:49 PM  
(modified after loading)  
Additional Info : Peak(s) manually integrated



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=off

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 17.961        | BB   | 0.4041      | 4637.86182   | 172.27362    | 50.0004 |
| 2      | 20.812        | BB   | 0.4589      | 4637.79004   | 151.59140    | 49.9996 |

Totals : 9275.65186 323.86502

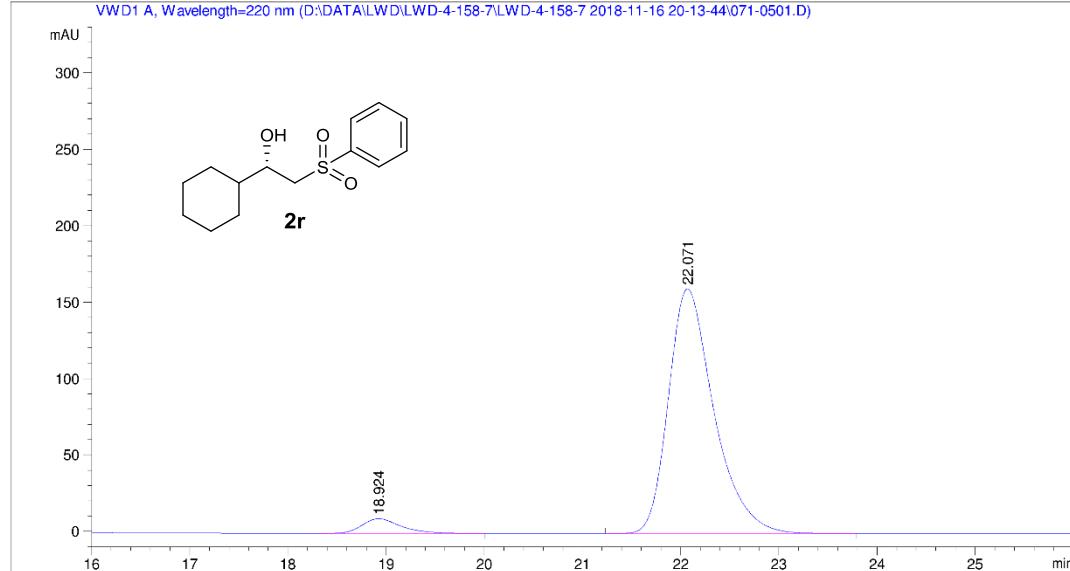
Instrument 2 11/17/2018 3:40:17 PM

Page 1 of 2

Data File D:\DATA\LWD\LWD-4-158-7\LWD-4-158-7 2018-11-16 20-13-44\071-0501.D  
Sample Name: TL-c-hex-ph-ee

```
=====
Acq. Operator   :                               Seq. Line :  5
Acq. Instrument : Instrument 1               Location : Vial 71
Injection Date  : 11/16/2018 9:47:56 PM        Inj :  1
                                                Inj Volume : 3.000 μl
Acq. Method    : D:\DATA\LWD\LWD-4-158-7\LWD-4-158-7 2018-11-16 20-13-44\VWD-AD(1-2)-80-20-0
                  -7ML-3UL-220NM-30MIN.M
Last changed   : 11/16/2018 8:26:32 PM
Analysis Method: D:\METHOD\LWD\AD(1-2)-80-20-1ML-3UL-ALL-10MIN.M
Last changed   : 11/17/2018 3:36:36 PM
                  (modified after loading)
Additional Info: Peak(s) manually integrated
```

Additional Info : Peak(s) manually integrated



## Area Percent Report

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 18.924        | BB   | 0.4121      | 263.78177    | 9.61526      | 4.8706  |
| 2      | 23.071        | BP   | 0.4855      | 5151.99707   | 160.96725    | 95.1994 |

Totals : 5415 77884 169 68261

Instrument 3 11/17/2018 3:36:38 PM

Page 1 of 2

## 6. Reference

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4. P. Bertus, P. Phansavath, V. Ratovelomanana-Vidal, J.-P. Genêt, A. R. Touati, T. Homri, H. B. Ben, *Tetrahedron: Asymmetry*, 1999, **10**, 1369.