

Supporting Information

Synthesis of Chiral Seven-membered Cyclic Sulfonamides through Pd-catalyzed Arylation of Cyclic Imines

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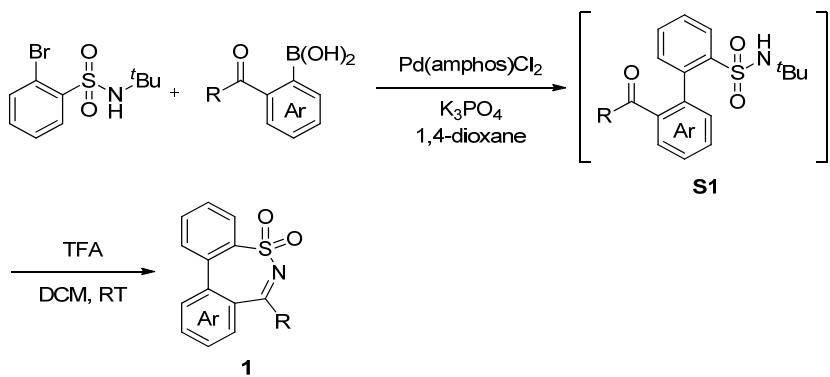
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1. General

All reactions were carried out under an atmosphere of nitrogen using the standard Schlenk techniques, unless otherwise noted. Commercially available reagents were used without further purification. Solvents were treated prior to use according to the standard methods. ^1H NMR, ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz with the Brucker spectrometer. ^{19}F was recorded at 376 MHz with Brucker spectrometer. Chemical shifts are reported in ppm using tetramethylsilane as internal standard when using CDCl_3 as solvent for ^1H NMR spectra. The following abbreviations were used to symbolize the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Flash column chromatography was performed on silica gel (200-300 mesh). All reactions were monitored by TLC analysis. Optical rotations were measured by the polarimeter. Enantiomeric excess was determined by HPLC analysis using chiral column described below in detail.

2. Synthesis of Seven-membered Cyclic *N*-Sulfonyl Imines

Seven-membered cyclic *N*-sulfonylimines **1** can be conveniently prepared by slightly modified procedure from readily available 2-bromobenzenesulfonamide and 2-acylphenylboronic acids according to the known literature procedure.¹⁻³ The detailed synthetic steps include palladium-catalyzed Suzuki coupling reaction and trifluoroacetic acid promoted intramolecular cyclization.



General Procedure: 2-Formylbenzeneboronic acid (3.000 g, 20 mmol), $\text{Pd}(\text{amphos})\text{Cl}_2$ (0.212 g, 0.3 mmol), 2-bromo-*N*-(*tert*-butyl)benzenesulfonamide (2.922 g, 10.0 mmol) and potassium phosphate (6.368 g, 30.0 mmol) in 1,4-dioxane (50 mL) and water (30 mL) was stirred at reflux for 16 h, then cooled to ambient temperature, the solvent was removed under the reduced pressure, diluted with water (10 mL), then extracted with dichloromethane (30 mL \times 3). The combined organic layer was dried over anhydrous sodium sulfate. After filtration, the solvent was concentrated in *vacuo*, and the residue was purified by flash chromatography to give products **S1**.

Subsequently, the above products (0.952 g, 3.0 mmol) and trifluoroacetic acid (7 mL/g of coupling product) in dichloromethane (45 mL) was stirred at ambient temperature for 20 min, then the mixture pH adjusted to 7 with saturated sodium bicarbonate solution. The organic layer was separated and the aqueous layer extracted with dichloromethane (30 mL \times 3). The combined organic layer was dried over anhydrous sodium sulfate. After filtration, the solvent was removed under reduced pressure and the crude product was recrystallized with ethyl acetate to give the cyclic *N*-sulfonyl imines **1**.

Dibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (1a): 1.220 g, 41% yield (two steps), white solid, mp 268-269 °C, new compound, R_f = 0.45 (hexanes/ethyl acetate 3/1), ^1H NMR (400 MHz, CDCl_3) δ



8.87 (s, 1H), 8.31 (dd, $J = 7.9, 1.0$ Hz, 1H), 7.92 (d, $J = 7.8$ Hz, 1H), 7.83-7.75 (m, 3H), 7.75-7.69 (m, 2H), 7.66 (td, $J = 7.6, 1.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 172.8, 140.6, 138.9, 135.6, 133.8, 133.4, 132.9, 131.4, 131.3, 130.1, 129.7, 128.9, 128.2. HRMS Calculated for $\text{C}_{13}\text{H}_{10}\text{NO}_2\text{S} [\text{M}+\text{H}]^+$ 244.0427, found: 244.0426.

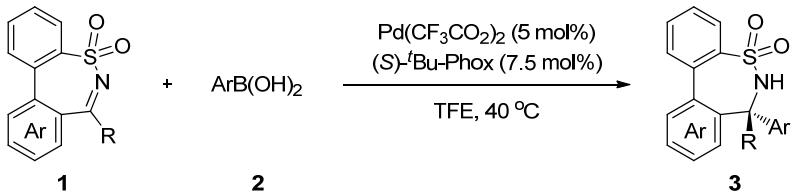
9-Methoxydibenzo[d,f][1,2]thiazepine 5,5-dioxide (1b): 0.479 g, 50% yield (two steps), yellow solid, mp 232-233 °C, new compound, $R_f = 0.40$ (hexanes/ethyl acetate 3/1), ^1H NMR (400 MHz, CDCl_3) δ 8.80 (s, 1H), 8.27 (d, $J = 7.6$ Hz, 1H), 7.84 (d, $J = 8.6$ Hz, 1H), 7.80-7.55 (m, 3H), 7.30 (d, $J = 8.7$ Hz, 2H), 3.94 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 172.4, 159.5, 138.0, 135.6, 134.1, 133.7, 133.1, 131.7, 131.0, 129.0, 128.2, 120.0, 115.0, 55.8. HRMS Calculated for $\text{C}_{14}\text{H}_{12}\text{NO}_3\text{S} [\text{M}+\text{H}]^+$ 274.0532, found: 274.0531.

10-Chlorodibenzo[d,f][1,2]thiazepine 5,5-dioxide (1c): 1.135 g, 41% yield (two steps), white solid, mp 256-257 °C, new compound, $R_f = 0.45$ (hexanes/ethyl acetate 3/1), ^1H NMR (400 MHz, CDCl_3) δ 8.82 (s, 1H), 8.30 (d, $J = 7.9$ Hz, 1H), 7.90 (d, $J = 1.6$ Hz, 1H), 7.83-7.77 (m, 1H), 7.76-7.71 (m, 3H), 7.62 (dd, $J = 8.3, 1.8$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 171.7, 142.2, 139.9, 138.9, 134.3, 134.0, 132.7, 131.3, 131.3, 130.3, 130.1, 129.1, 128.3. HRMS Calculated for $\text{C}_{13}\text{H}_9\text{ClNO}_2\text{S} [\text{M}+\text{H}]^+$ 278.0037, found: 278.0034.

9-Chlorodibenzo[d,f][1,2]thiazepine 5,5-dioxide (1d): 0.801 g, 31% yield (two steps), white solid, mp 235-236 °C, new compound, $R_f = 0.50$ (hexanes/ethyl acetate 3/1), ^1H NMR (400 MHz, CDCl_3) δ 8.78 (s, 1H), 8.29 (d, $J = 7.6$ Hz, 1H), 7.84 (d, $J = 8.5$ Hz, 1H), 7.81-7.74 (m, 2H), 7.74-7.66 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 171.2, 138.8, 138.7, 135.0, 134.7, 134.1, 134.0, 133.3, 131.5, 131.2, 130.6, 130.0, 128.5. HRMS Calculated for $\text{C}_{13}\text{H}_9\text{ClNO}_2\text{S} [\text{M}+\text{H}]^+$ 278.0037, found: 278.0036.

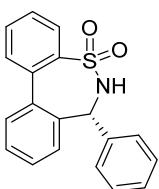
7-Methyldibenzo[d,f][1,2]thiazepine 5,5-dioxide (1e): 0.106 g, 7% yield (two steps), yellow solid, mp 192-193 °C, new compound, $R_f = 0.25$ (hexanes/ethyl acetate 5/1), ^1H NMR (400 MHz, CDCl_3) δ 8.23 (d, $J = 7.8$ Hz, 1H), 7.80-7.61 (m, 6H), 7.57 (t, $J = 7.6$ Hz, 1H), 2.65 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 183.1, 140.2, 138.6, 136.3, 135.9, 133.5, 132.1, 130.9, 130.4, 129.0, 128.6, 128.3, 127.5, 29.1. HRMS Calculated for $\text{C}_{14}\text{H}_{12}\text{NO}_2\text{S} [\text{M}+\text{H}]^+$ 258.0583, found: 258.0585.

3. Pd-catalyzed Asymmetric Arylation of Cyclic N-Sulfonyl Imines



Ligand *(S)*-*t*Bu-Phox (5.8 mg, 0.015 mmol) and Pd(CF_3CO_2)₂ (3.3 mg, 0.01 mmol) were placed in a dried Schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone (1 mL) was added. The mixture was stirred at room temperature for 1 hour. The solvent was removed under vacuum to give the catalyst. Under nitrogen atmosphere, to the mixture of seven-membered cyclic *N*-sulfonyl imines (**1a-1d**, 0.20 mmol) and arylboronic acids (0.60 mmol) was added the above catalyst with 2,2,2-trifluoroethanol (3.0 mL). The solution was stirred at 40 °C for 4-72 h. The reaction mixture was cooled to room temperature, and the solvent was removed under reduced pressure. Flash chromatography on silica gel using hexanes/ethyl acetate as the eluent gave the chiral products **3**.

(R)-(+)7-Phenyl-6,7-dihydronbenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3aa): 63 mg, 98% yield, white solid, mp 204-205 °C, new compound, $R_f = 0.50$ (hexanes/ethyl acetate 3/1), 99% ee,



$[\alpha]^{20}_D = 51.35$ (*c* 1.26, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, *J* = 7.7 Hz, 1H), 7.80-7.70 (m, 1H), 7.69-7.54 (m, 2H), 7.50-7.34 (m, 6H), 7.31-7.23 (m, 2H), 6.84-6.71 (m, 1H), 5.35 (brs, 1H), 5.30 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.1, 139.0, 138.0, 137.1, 136.2, 133.5, 130.2, 129.5, 129.2, 129.1, 129.0, 128.8, 128.7, 128.5, 128.1, 125.9, 60.5. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 12.7 min and 18.3 min (major). HRMS Calculated for $\text{C}_{19}\text{H}_{16}\text{NO}_2\text{S}$ [M+H]⁺ 322.0896, found: 322.0895.

(+)-7-(4-Methoxyphenyl)-6,7-dihydronbenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3ab): 69 mg, 98% yield, white solid, mp 114-115 °C, new compound, $R_f = 0.60$ (hexanes/ethyl acetate 3/1), 95% ee, $[\alpha]^{20}_D = 52.08$ (*c* 0.72, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.07 (d, *J* = 7.6 Hz, 1H), 7.80-7.72 (m, 1H), 7.68-7.56 (m, 2H), 7.43-7.40 (m, 2H), 7.38-7.25 (m, 3H), 6.92 (d, *J* = 8.1 Hz, 2H), 6.84-6.77 (m, 1H), 5.31 (brs, 1H), 5.26 (s, 1H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.0, 139.9, 139.0, 137.1, 136.4, 133.4, 130.2, 129.8, 129.4, 129.2, 129.1, 129.0, 128.6, 125.9, 114.3, 60.1, 55.4. HPLC: Chiracel OD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 20.0 min and 23.5 min (major). HRMS Calculated for $\text{C}_{20}\text{H}_{18}\text{NO}_3\text{S}$ [M+H]⁺ 352.1002, found: 352.1006.

(+)-7-(*p*-Tolyl)-6,7-dihydronbenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3ac): 67 mg, 99% yield, white solid, mp 110-111 °C, new compound, $R_f = 0.80$ (hexanes/ethyl acetate 3/1), 97% ee, $[\alpha]^{20}_D = 27.65$ (*c* 1.28, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, *J* = 7.7 Hz, 1H), 7.78-7.71 (m, 1H), 7.68-7.54 (m, 2H), 7.43-7.38 (m, 2H), 7.33-7.27 (m, 3H), 7.21-7.13 (m, 2H), 6.84-6.74 (m, 1H), 5.31 (brs, 1H), 5.26 (s, 1H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.0, 139.0, 138.4, 136.3, 134.9, 133.4, 130.2, 129.5, 129.5, 129.1, 129.0, 128.6, 128.0, 125.9, 60.4, 21.2. HPLC:

Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 13.4 min and 21.8 min (major). HRMS Calculated for $\text{C}_{20}\text{H}_{18}\text{NO}_2\text{S}$

$[M+H]^+$ 336.1053, found: 336.1049.

(+)-7-(4-(tert-Butyl)phenyl)-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3ad): 75 mg, 99% yield, white solid, mp 125-126 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 3/1), 96% ee, $[\alpha]^{20}_D$ = 50.12 (*c* 1.56, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 7.7 Hz, 1H), 7.71-7.63 (m, 1H), 7.61-7.55 (m, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.40-7.18 (m, 7H), 6.82-6.70 (m, 1H), 5.24 (brs, 1H), 5.19 (s, 1H), 1.26 (s, 9H) ¹³C NMR (100 MHz, CDCl₃) δ 151.6, 140.0, 139.1, 137.1, 136.3, 134.8, 133.4, 130.2, 129.5, 129.1, 129.0, 128.6, 127.8, 125.9, 125.8, 60.4, 34.7, 31.4. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 13.1 min and 24.4 min (major). HRMS Calculated for C₂₃H₂₄NO₂S [M+H]⁺ 378.1522, found: 378.1524.

(+)-7-(*m*-Tolyl)-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3ae): 64 mg, 95% yield, white solid, mp 179-180 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 3/1), 98% ee, $[\alpha]^{20}_D$ = 47.26 (*c* 1.28, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 7.7 Hz, 1H), 7.79-7.72 (m, 1H), 7.68-7.56 (m, 2H), 7.47-7.40 (m, 2H), 7.31-7.15 (m, 5H), 6.86-6.74 (m, 1H), 5.33 (brs, 1H), 5.26 (s, 1H), 2.34 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 140.0, 139.0, 138.6, 137.9, 137.1, 136.2, 133.4, 130.2, 129.5, 129.2, 129.0, 128.7, 128.7, 125.9, 125.2, 60.5, 21.4. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 12.0 min and 13.4 min (major). HRMS Calculated for C₂₀H₁₈NO₂S [M+H]⁺ 336.1053, found: 336.1056.

(+)-7-(*o*-Tolyl)-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3af): 65 mg, 97% yield, white solid, mp 227-228 °C, new compound, R_f = 0.70 (hexanes/ethyl acetate 3/1), 95% ee, $[\alpha]^{20}_D$ = 2.27 (*c* 0.75, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 7.8 Hz, 1H), 7.85 (d, *J* = 7.4 Hz, 1H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.43 (d, *J* = 4.1 Hz, 2H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.30-7.23 (m, 2H), 7.11 (d, *J* = 7.4 Hz, 1H), 6.70 (d, *J* = 7.8 Hz, 1H), 5.35 (brs, 1H), 5.25 (s, 1H), 1.82 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 140.3, 138.9, 136.9, 136.2, 135.6, 134.9, 133.5, 131.0, 129.8, 129.3, 129.2, 128.9, 128.8, 128.7, 128.3, 127.6, 126.5, 126.1, 57.3, 18.9. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 9.8 min and 12.1 min (major). HRMS Calculated for C₂₀H₁₈NO₂S [M+H]⁺ 336.1053, found: 336.1052.

(+)-7-(4-Fluorophenyl)-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxidee (3ag): 67 mg, 98% yield, white solid, mp 229-230 °C, new compound, R_f = 0.80 (hexanes/ethyl acetate 3/1), 98% ee, $[\alpha]^{20}_D$ = 44.38 (*c* 1.53, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, *J* = 7.7 Hz, 1H), 7.79-7.72 (m, 1H), 7.68-7.55 (m, 2H), 7.52-7.36 (m, 4H), 7.30 (t, *J* = 7.0 Hz, 1H), 7.17-6.99 (m, 2H), 6.82-6.68 (m, 1H), 5.36 (brs, 1H), 5.29 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 162.6 (d, *J* = 246.0 Hz), 140.0, 138.9, 137.0, 136.0, 133.8, 133.5, 130.2, 129.8, 129.3, 129.2, 129.1, 128.7, 125.9, 115.8 (d, *J* = 22.0 Hz), 59.9. ¹⁹F NMR (376 MHz, CDCl₃) δ -113.0. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 12.5 min and 18.5 min (major). HRMS Calculated for C₁₉H₁₅FNO₂S [M+H]⁺ 340.0802, found: 340.0800.

(+)-7-(4-Chlorophenyl)-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (3ah): 69 mg,

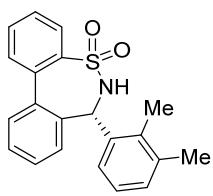
97% yield, white solid, mp 191-192 °C, new compound, R_f = 0.60 (hexanes/ ethyl acetate 3/1), 99% ee, $[\alpha]^{20}_D$ = 66.19 (*c* 0.63, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.03 (d, *J* = 7.7 Hz, 1H), 7.80-7.71 (m, 1H), 7.70-7.58 (m, 2H), 7.53-7.25 (m, 7H), 6.84-6.62 (m, 1H), 5.41 (brs, 1H), 5.28 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 140.0, 138.9, 136.6, 135.7, 134.4, 133.5, 130.2, 129.4, 129.3, 129.2, 129.1, 129.1, 128.8, 125.9, 60.0. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 13.3 min and 19.1 min (major). HRMS Calculated for C₁₉H₁₅ClNO₂S [M+H]⁺ 356.0507, found: 356.0510.

(+)-7-(4-Bromophenyl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ai): 76 mg, 95% yield, white solid, mp 211-212 °C, new compound, R_f = 0.60 (hexanes/ethyl acetate 3/1), 99% ee, $[\alpha]^{20}_D$ = 66.20 (*c* 1.16, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 4.7 Hz, 1H), 7.80-7.71 (m, 1H), 7.68-7.55 (m, 2H), 7.54-7.46 (m, 2H), 7.45-7.39 (m, 2H), 7.37-7.24 (m, 3H), 6.82-6.62 (m, 1H), 5.44 (brs, 1H), 5.26 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 140.0, 138.8, 137.1, 135.6, 133.5, 132.0, 130.2, 129.8, 129.4, 129.2, 129.1, 128.8, 125.9, 122.5, 60.00. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 14.1 min and 20.4 min (major). HRMS Calculated for C₁₉H₁₅BrNO₂S [M+H]⁺ 400.0001, found: 400.0006.

(+)-7-(4-(Trifluoromethyl)phenyl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3aj): 73 mg, 94% yield, white solid, mp 223-224 °C, new compound, R_f = 0.60 (hexanes/ethyl acetate 3/1), 98% ee, $[\alpha]^{20}_D$ = 46.00 (*c* 1.80, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 7.6 Hz, 1H), 7.81-7.72 (m, 1H), 7.71-7.37 (m, 8H), 7.35-7.28 (m, 1H), 6.78-6.60 (m, 1H), 5.45 (brs, 1H), 5.37 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 142.2, 140.1, 138.8, 137.2, 135.4, 133.6, 130.2, 129.5, 129.3, 129.1, 128.8, 128.6, 126.0, 125.8, 123.9 (q, *J* = 271.0 Hz), 60.1. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.6. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 10.5 min and 15.7 min (major). HRMS Calculated for C₂₀H₁₅F₃NO₂S [M+H]⁺ 390.0770, found: 390.0774.

(+)-7-(Naphthalen-2-yl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ak): 65 mg, 87% yield, white solid, mp 140-141 °C, new compound, R_f = 0.55 (hexanes/ethyl acetate 3/1), 95% ee, $[\alpha]^{20}_D$ = 63.58 (*c* 1.70, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 7.7 Hz, 1H), 8.01 (s, 1H), 7.90-7.57 (m, 6H), 7.55-7.49 (m, 2H), 7.47-7.33 (m, 3H), 7.25-7.17 (m, 1H), 6.82-6.70 (m, 1H), 5.50 (brs, 1H), 5.45 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 140.1, 139.0, 137.2, 136.0, 135.4, 133.5, 133.2, 130.2, 129.7, 129.3, 129.1, 129.0, 128.7, 128.6, 128.2, 127.8, 126.9, 126.7, 126.6, 126.0, 125.9, 60.5. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 18.2 min and 27.2 min (major). HRMS Calculated for C₂₃H₁₈NO₂S [M+H]⁺ 372.1053, found: 372.1050.

(+)-7-(2,3-Dimethylphenyl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ai): 67 mg, 96% yield, white solid, mp 285-286 °C, new compound, R_f = 0.70 (hexanes/ethyl acetate 3/1), 91% ee, $[\alpha]^{20}_D$ = 18.77 (*c* 1.30, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.11 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.82 (td, *J* = 7.6, 1.3 Hz, 1H), 7.77-7.71 (m, *J* = 2H), 7.65 (td, *J* = 7.7, 1.3 Hz, 1H), 7.47 (d, *J* = 4.1 Hz, 2H), 7.34-7.24 (m, 2H), 7.23-7.17 (m, 1H), 6.76 (d, *J* = 7.8 Hz, 1H), 5.43 (d, *J* = 2.6 Hz,



1H), 5.25 (d, $J = 2.4$ Hz, 1H), 2.25 (s, 3H), 1.75 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.1, 139.0, 137.6, 137.0, 136.0, 135.2, 134.1, 133.5, 130.0, 129.8, 129.3, 129.2, 128.9, 128.9, 128.7, 126.1, 125.9, 125.4, 57.7, 20.5, 14.6. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 9.5 min and 15.2 min (major). HRMS Calculated for $\text{C}_{21}\text{H}_{20}\text{NO}_2\text{S} [\text{M}+\text{H}]^+$ 350.1209, found: 350.1212.

(+)-7-(2,4-Dimethylphenyl)-6,7-dihydrodibenzodifluorothiazepine 5,5-dioxide (3am): 69 mg, 99% yield, white solid, mp 214-215 °C, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 3/1), 95% ee, $[\alpha]^{20}_D = 9.08$ (c 1.30, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.00 (dd, $J = 7.8, 1.1$ Hz, 1H), 7.74-7.60 (m, 3H), 7.54 (td, $J = 7.7, 1.3$ Hz, 1H), 7.36 (d, $J = 4.0$ Hz, 2H), 7.22-7.14 (m, 1H), 7.09 (d, $J = 7.8$ Hz, 1H), 6.88 (s, 1H), 6.66 (d, $J = 7.8$ Hz, 1H), 5.26 (d, $J = 2.5$ Hz, 1H), 5.14 (d, $J = 2.4$ Hz, 1H), 2.27 (s, 3H), 1.72 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.2, 139.0, 138.0, 136.9, 135.4, 135.1, 133.5, 133.1, 131.8, 129.8, 129.3, 129.2, 128.9, 128.7, 127.5, 127.1, 126.1, 57.2, 21.0, 18.8. HPLC: Chiracel AD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 10.1 min and 12.7 min (major). HRMS Calculated for $\text{C}_{21}\text{H}_{20}\text{NO}_2\text{S} [\text{M}+\text{H}]^+$ 350.1209, found: 350.1204.

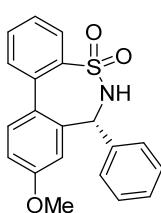
(+)-7-(3,5-Dimethylphenyl)-6,7-dihydrodibenzodifluorothiazepine 5,5-dioxide (3an): 66 mg, 94% yield, white solid, mp 202-203 °C, new compound, $R_f = 0.80$ (hexanes/ethyl acetate 3/1), 95% ee, $[\alpha]^{20}_D = 55.23$ (c 1.28, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.09 (dd, $J = 7.8, 1.0$ Hz, 1H), 7.79 (t, $J = 7.3$ Hz, 1H), 7.73-7.59 (m, 2H), 7.53-7.41 (m, 2H), 7.38-7.29 (m, 1H), 7.14-6.96 (m, 3H), 6.92-6.80 (m, 1H), 5.33 (brs, 1H), 5.25 (s, 1H), 2.33 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.0, 139.1, 138.5, 137.8, 137.2, 136.3, 133.3, 130.2, 130.1, 129.5, 129.1, 129.0, 128.6, 125.9, 60.5, 21.3. HPLC: Chiracel OD-H column, 220 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 0.8 mL/min, retention time 12.1 min and 14.1 min (major). HRMS Calculated for $\text{C}_{21}\text{H}_{20}\text{NO}_2\text{S} [\text{M}+\text{H}]^+$ 350.1209, found: 350.1212.

(+)-7-(furan-3-yl)-6,7-dihydrodibenzodifluorothiazepine 5,5-dioxide (3ao): 40 mg, 64% yield, white solid, mp 107-108 °C, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 3/1), 98% ee, $[\alpha]^{20}_D = 19.30$ (c 0.72, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 7.7$ Hz, 1H), 7.77-7.70 (m, 1H), 7.69-7.51 (m, 3H), 7.49-7.34 (m, 4H), 7.15 (s, 1H), 6.33 (brs, 1H), 5.46 (s, 1H), 5.26 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 141.0, 140.0, 138.9, 135.1, 133.5, 130.2, 129.5, 129.2, 128.9, 128.7, 125.9, 123.5, 110.0, 53.7. HPLC: Chiracel AD-H column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 13.5 min and 14.6 min (major). HRMS Calculated for $\text{C}_{17}\text{H}_{13}\text{NNaO}_3\text{S} [\text{M}+\text{Na}]^+$ 334.0508, found: 334.0516.

(+)-7-(benzofuran-2-yl)-6,7-dihydrodibenzodifluorothiazepine 5,5-dioxide (3ap): 19 mg, 26% yield, white solid, mp 137-138 °C, new compound, $R_f = 0.50$ (hexanes/ethyl acetate 4/1), 97% ee, $[\alpha]^{20}_D = 29.09$ (c 0.44, CHCl_3). ^1H NMR (400 MHz, MeOD) δ 7.91 (d, $J = 7.7$ Hz, 1H), 7.82-7.44 (m, 3H), 7.43-7.28 (m, 3H), 7.28-6.48 (m, 6H), 5.35 (s, 1H). ^{13}C NMR (100 MHz, MeOD) δ 154.9, 154.8, 140.3, 138.7, 134.4, 133.2, 130.0, 129.2, 128.6, 128.4, 127.9, 125.1, 124.1, 122.7, 120.7, 110.6, 105.0, 55.5. HPLC: Chiracel AD-H column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH

= 80/20, flow = 1.0 mL/min, retention time 15.8 min and 19.8 min (major). HRMS Calculated for C₂₁H₁₆NO₃S [M+H]⁺ 362.0845, found: 362.0856.

(-)9-Methoxy-7-phenyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ba): 66 mg, 94% yield, white solid, mp 193-194 °C, new compound, R_f = 0.55 (hexanes/ethyl acetate 3/1),



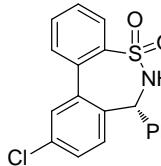
99% ee, [α]²⁰_D = -56.42 (c 1.26, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, J = 7.2 Hz, 1H), 7.76-7.69 (m, 1H), 7.68-7.51 (m, 2H), 7.47-7.29 (m, 6H), 6.95 (dd, J = 8.5, 2.6 Hz, 1H), 6.39-6.27 (m, 1H), 5.34 (brs, 1H), 5.29 (s, 1H), 3.68 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 160.0, 138.9, 137.9, 137.7, 133.4, 132.4, 130.3,

130.1, 128.9, 128.6, 128.1, 128.0, 125.9, 116.4, 113.6, 60.6, 55.2. HPLC:

Chiracel IC column, 220 nm, 30 °C, n-Hexane/i-PrOH = 80/20, flow = 1.0 mL/min, retention time 49.1 min (major) and 53.0 min. HRMS Calculated for C₂₀H₁₈NO₃S [M+H]⁺ 352.1002, found: 352.1003.

(+)-10-Chloro-7-phenyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ca): 67 mg, 94% yield, white solid, mp 236-237 °C, new compound, R_f = 0.80 (hexanes/ethyl acetate 3/1),



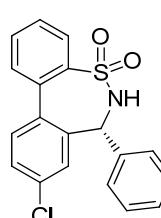
98% ee, [α]²⁰_D = 50.53 (c 1.30, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 7.6 Hz, 1H), 7.75-7.65 (m, 1H), 7.62-7.50 (m, 2H), 7.48-7.20 (m, 6H), 7.17-7.13 (m, 1H), 6.72-6.50 (m, 1H), 5.29 (brs, 1H), 5.16 (s, 1H). ¹³C NMR

(100 MHz, CDCl₃) δ 141.6, 137.6, 137.6, 135.1, 134.8, 133.6, 130.9, 130.1,

129.3, 129.0, 128.9, 128.8, 128.0, 126.1, 60.0. HPLC: Chiracel AD-H column,

220 nm, 30 °C, n-Hexane/i-PrOH = 80/20, flow = 0.8 mL/min, retention time 16.9 min and 18.4 min (major). HRMS Calculated for C₁₉H₁₅ClNO₂S [M+H]⁺ 356.0507, found: 356.0510.

(-)9-Chloro-7-phenyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3da): 68 mg, 96% yield, white solid, mp 227-228 °C, new compound, R_f = 0.60 (hexanes/ethyl acetate 3/1),



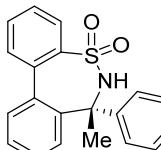
99% ee, [α]²⁰_D = -11.29 (c 1.16, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, J = 7.5 Hz, 1H), 7.82-7.71 (m, 1H), 7.68-7.56 (m, 2H), 7.48-7.32 (m, 7H), 6.82-6.60 (m, 1H), 5.38 (brs, 1H), 5.24 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ

138.5, 138.0, 137.9, 137.2, 135.2, 133.5, 130.3, 130.1, 129.7, 129.4, 129.1,

129.0, 128.9, 128.0, 126.0, 60.3. HPLC: Chiracel AD-H column, 220 nm, 30 °C, n-Hexane/i-PrOH = 80/20, flow = 1.0 mL/min, retention time 13.2 min and 15.0

min (major). HRMS Calculated for C₁₉H₁₅ClNO₂S [M+H]⁺ 356.0507, found: 356.0502.

(+)-7-Methyl-7-phenyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ea): 28 mg, 84% yield, white solid, mp 239-240 °C, new compound, R_f = 0.70 (hexanes/ethyl acetate 3/1),



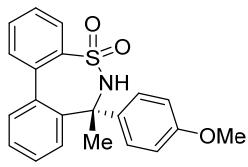
99% ee, [α]²⁰_D = 68.61 (c 0.72, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.76 (dd, J = 13.5, 7.6 Hz, 2H), 7.53-7.41 (m, 2H), 7.25-7.11 (m, 3H), 6.82 (m, 3H), 6.74

(d, J = 7.3 Hz, 1H), 6.63 (d, J = 3.0 Hz, 2H), 5.94 (brs, 1H), 1.82 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 145.6, 140.7, 140.1, 138.0, 132.5, 130.9, 129.9,

129.2, 128.6, 127.6, 127.4, 127.0, 126.1, 124.6, 124.3, 63.2, 33.5. HPLC: Chiracel AD-H column,

220 nm, 30 °C, n-Hexane/i-PrOH = 80/20, flow = 1.0 mL/min, retention time 10.2 min (major) and 15.1 min. HRMS Calculated for C₂₀H₁₈NO₂S [M+H]⁺ 336.1053, found: 336.1050.

(+)-7-(4-Methoxyphenyl)-7-methyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine-5,5-dioxide (3eb): 32 mg, 88% yield, white solid, mp 164-165 °C, new compound, R_f = 0.55 (hexanes/ethyl acetate 3/1), 99% ee, [α]²⁰_D = 47.85 (c 0.56, CHCl₃). ¹H NMR (400 MHz, CDCl₃) ¹H NMR (400 MHz, CDCl₃) δ 7.86-7.76 (m, 2H), 7.58-7.46 (m, 3H), 7.30-7.22 (m, 2H), 6.89-6.80 (m, 1H), 6.61



(d, $J = 8.0$ Hz, 2H), 6.41 (d, $J = 8.0$ Hz, 2H), 6.02 (brs, 1H), 3.63 (s, 3H), 1.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 157.6, 140.7, 140.3, 140.1, 138.0, 137.7, 132.5, 130.9, 129.8, 129.1, 128.5, 127.4, 126.9, 125.9, 124.3, 112.9, 62.8, 55.2, 33.6. HPLC: Chiracel IA column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 11.6 min and 12.6 min (major). HRMS Calculated for C₂₁H₂₀NO₃S [M+H]⁺ 366.1158, found: 366.1162.

(+)-7-(4-(tert-Butyl)phenyl)-7-methyl-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ec): 35 mg, 90% yield, white solid, mp 257-258 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 3/1), 99% ee, [α]²⁰_D = 56.53 (c 0.52, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.88-7.74 (m, 2H), 7.58-7.46 (m, 2H), 7.25-7.07 (m, 3H), 6.87 (d, J = 7.9 Hz, 2H), 6.78-6.72 (m, 1H), 6.60 (d, J = 7.9 Hz, 2H), 6.10 (brs, 1H), 1.91 (s, 3H), 1.15 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 148.8, 142.5, 140.8, 140.5, 140.2, 138.1, 132.2, 130.7, 129.9, 129.1, 128.5, 127.2, 126.9, 124.6, 124.4, 124.3, 63.0, 34.2, 33.1, 31.1. HPLC: Chiracel IA column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 8.9 min and 10.2 min (major). HRMS Calculated for C₂₄H₂₆NO₂S [M+H]⁺ 392.1679, found: 392.1670.

(+)-7-methyl-7-(*p*-tolyl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ed): 33 mg, 95% yield, white solid, mp 106-107 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 3/1), 99% ee, [α]²⁰_D = 44.54 (c 0.66, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, J = 14.6, 7.2 Hz, 2H), 7.59-7.46 (m, 2H), 7.32-7.25 (m, 2H), 7.24-7.19 (m, 1H), 6.82 (d, J = 6.8 Hz, 1H), 6.68 (d, J = 7.5 Hz, 2H), 6.57 (d, J = 7.6 Hz, 2H), 6.08 (brs, 1H), 2.12 (s, 3H), 1.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 142.7, 140.7, 140.2, 140.1, 138.1, 135.6, 132.4, 130.9, 129.9, 129.1, 128.5, 128.2, 127.4, 127.0, 124.6, 124.3, 63.1, 33.6, 20.7. HPLC: Chiracel IA column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 9.9 min and 11.8 min (major). HRMS Calculated for C₂₁H₂₀NO₂S [M+H]⁺ 350.1209, found: 350.1223.

(+)-7-methyl-7-(*m*-tolyl)-6,7-dihydrodibenzo[d,f][1,2]thiazepine 5,5-dioxide (3ee): 32 mg, 92% yield, white solid, mp 178-179 °C, new compound, R_f = 0.50 (hexanes/ethyl acetate 3/1), 99% ee, [α]²⁰_D = 90.29 (c 0.68, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, J = 7.3 Hz, 2H), 7.59-7.47 (m, 2H), 7.31-7.26 (m, 2H), 7.22-7.16 (m, 1H), 6.83-6.75 (m, 2H), 6.73-6.67 (m, 1H), 6.51 (s, 1H), 6.46-6.40 (m, 1H), 5.98 (brs, 1H), 2.04 (s, 3H), 1.88 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 145.3, 140.7, 140.1, 140.1, 138.2, 137.0, 132.5, 130.8, 129.9, 129.1, 128.5, 127.6, 127.3, 126.9, 126.7, 125.2, 124.2, 122.3, 63.2, 33.3, 21.1. HPLC: Chiracel IA column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 1.0 mL/min, retention time 7.5 min (major) and 9.9 min. HRMS Calculated for C₂₁H₁₉NNaO₂S [M+Na]⁺ 372.1029, found: 372.1041.

4. Determination of Absolute Configuration of Products

To determine the absolute configuration of (+)-7-Phenyl-6,7-dihydrodibenzo[*d,f*][1,2]thiazepine 5,5-dioxide (+)-**3aa** (99% ee), firstly, (+)-**3aa** was upgraded to >99% ee by recrystallization with *n*-hexane/ethyl acetate. Then, *n*-hexane was added into the solution of (+)-**3aa** (>99% ee) in dichloromethane, then the solution was slowly evaporated and single crystal of (+)-**3aa** was obtained after 1 days. The crystal was grown from the solution, which is suitable for X-ray diffraction analysis. The structure in **Figure S1** showed that the absolute configuration of (+)-**3aa** is *R* [CCDC 1877280] contains the structure and supplementary crystallographic data for (*R*)-(+)-**3aa**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk.

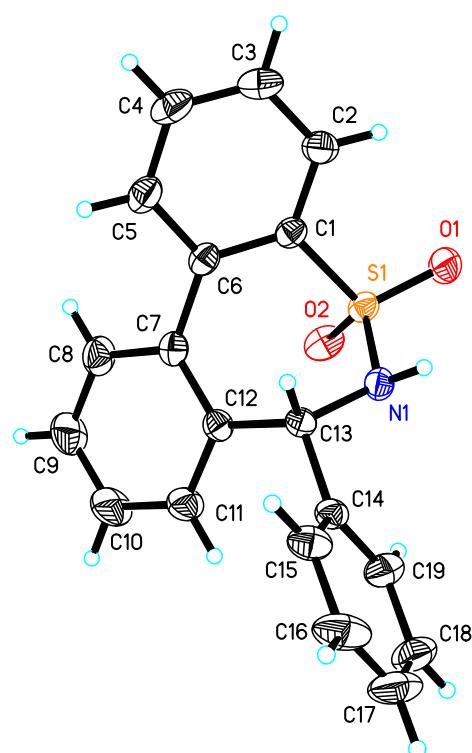
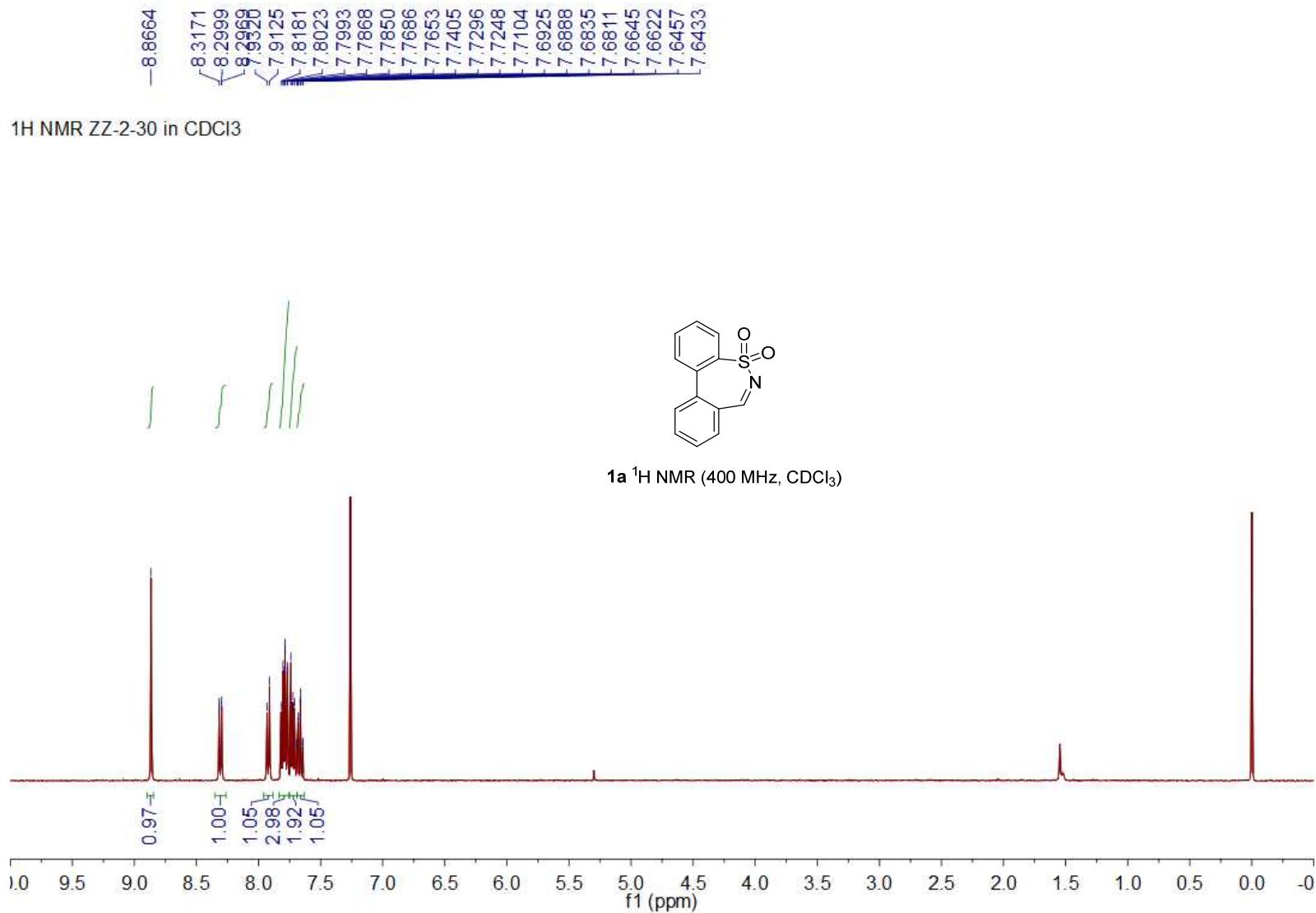


Figure S1. X-ray crystallographic analysis of (*R*)-(+)-**3aa**

5. References

1. Burlein, C.; Wang, C.; Xu, M.; Bhatt, T.; Stahlhut, M.; Ou, Y.; Adam, G. C.; Heath, J.; Klein, D. J.; Sanders, J.; Narayan, K.; Abeywickrema, P.; Heo, M. R.; Carroll, S. S.; Grobler, J. A.; Sharma, S.; Diamond, T. L.; Converso, A.; Krosky, D. J. *ACS Chem. Biol.* **2017**, *12*, 2858.
2. France, S. P.; Aleku, G. A.; Sharma, M.; Mangas-Sanchez, J.; Howard, R. M.; Steflik, J.; Kumar, R.; Adams, R. W.; Slabu, I.; Crook, R.; Grogan, G.; Wallace, T. W.; Turner, N. J. *Angew. Chem. Int. Ed.* **2017**, *56*, 15589.
3. Ni, C.; Zha, D.; Ye, H.; Hai, Y.; Zhou, Y.; Anslyn, E. V.; You, L. *Angew. Chem. Int. Ed.* **2018**, *57*, 1300.

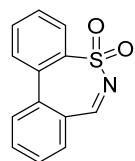
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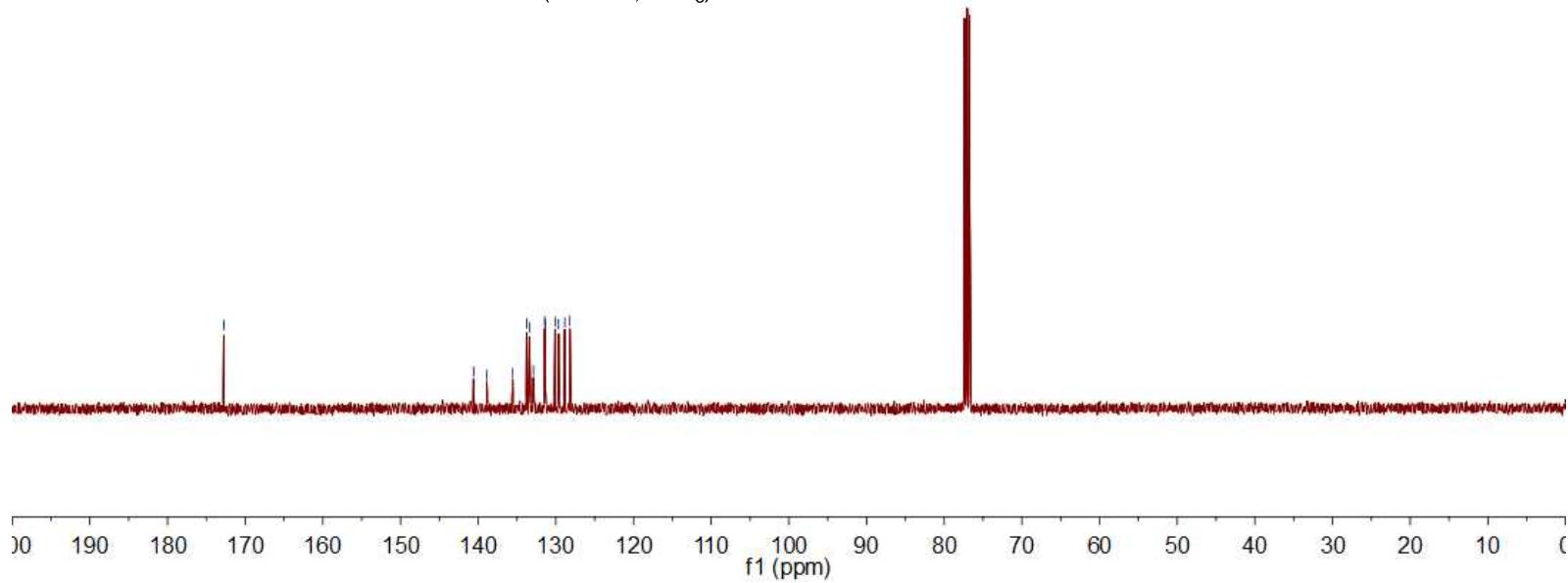
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128.20

^{13}C NMR ZZ-2-30 in CDCl_3

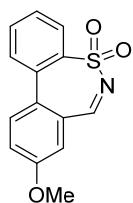


1a ^{13}C NMR (100 MHz, CDCl_3)

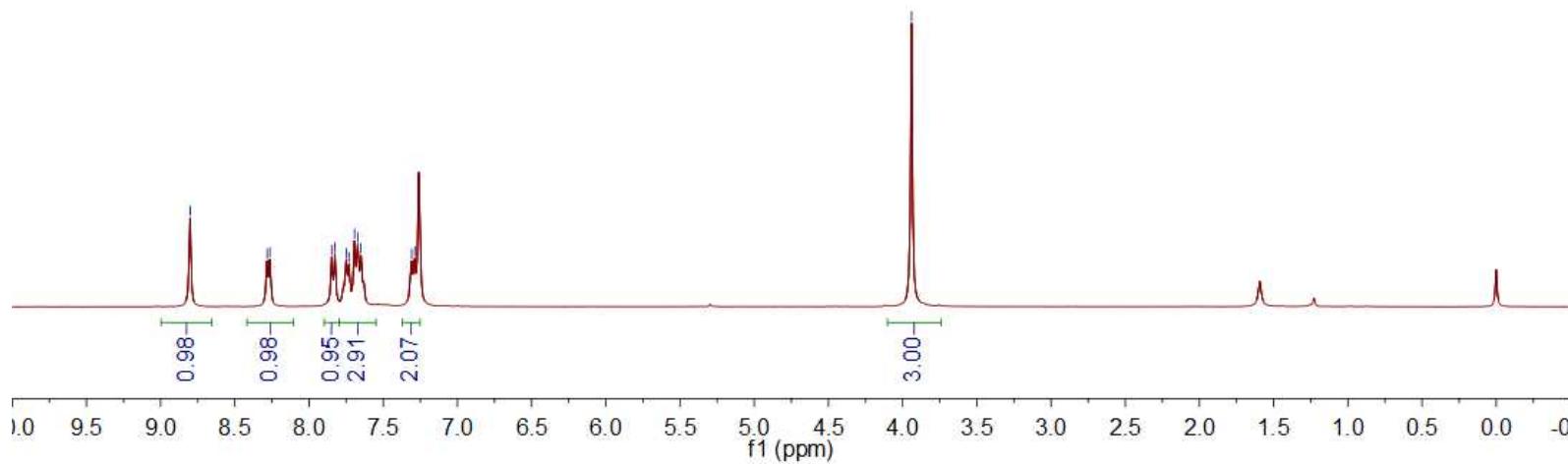


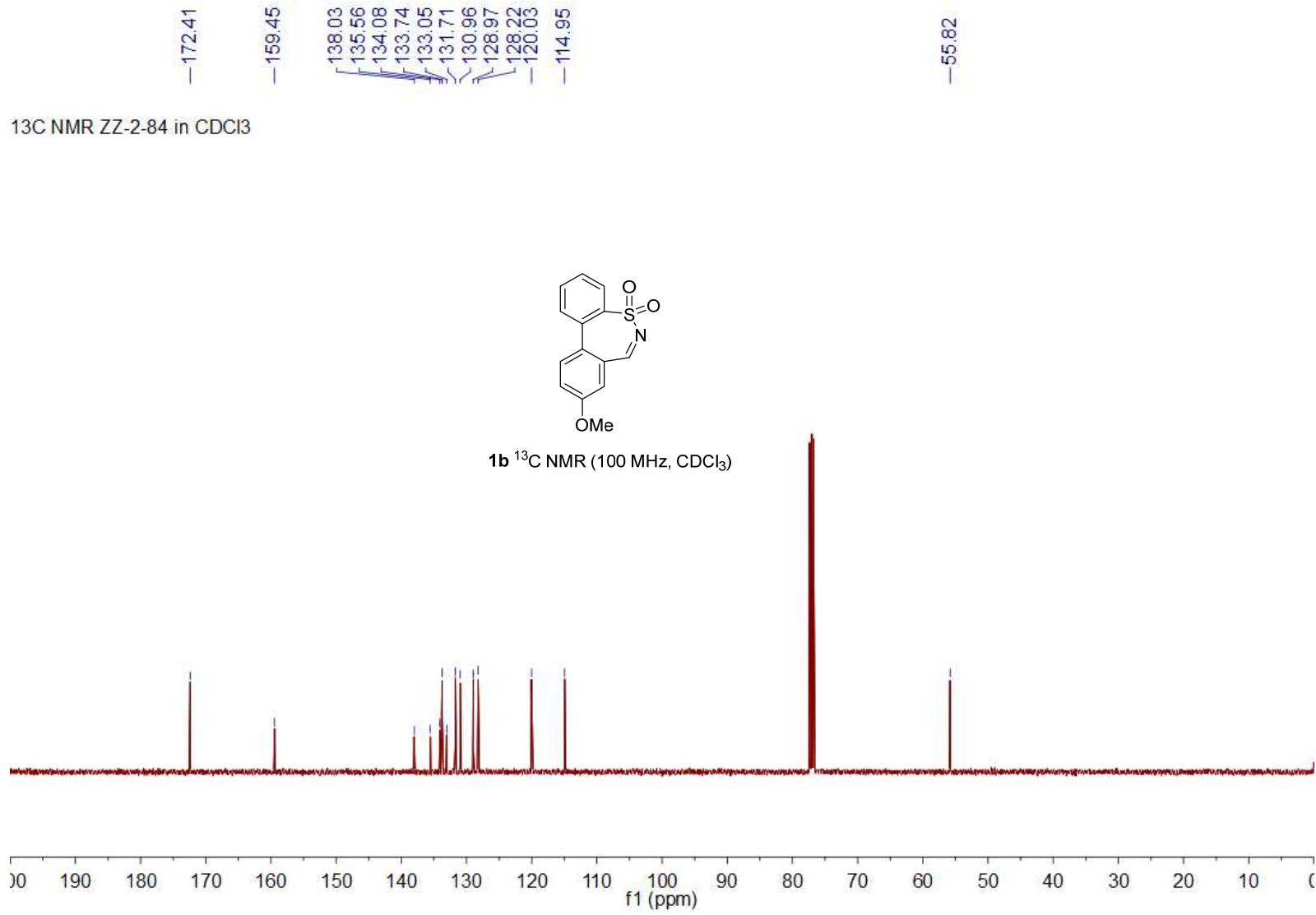
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^1H NMR ZZ-2-84 in CDCl_3



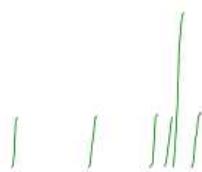
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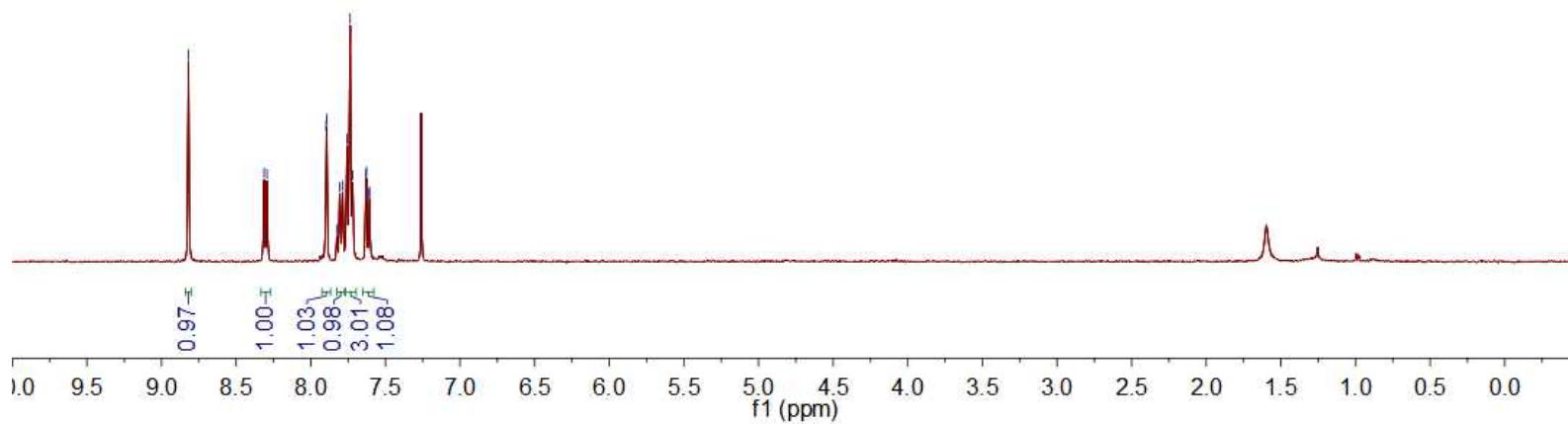


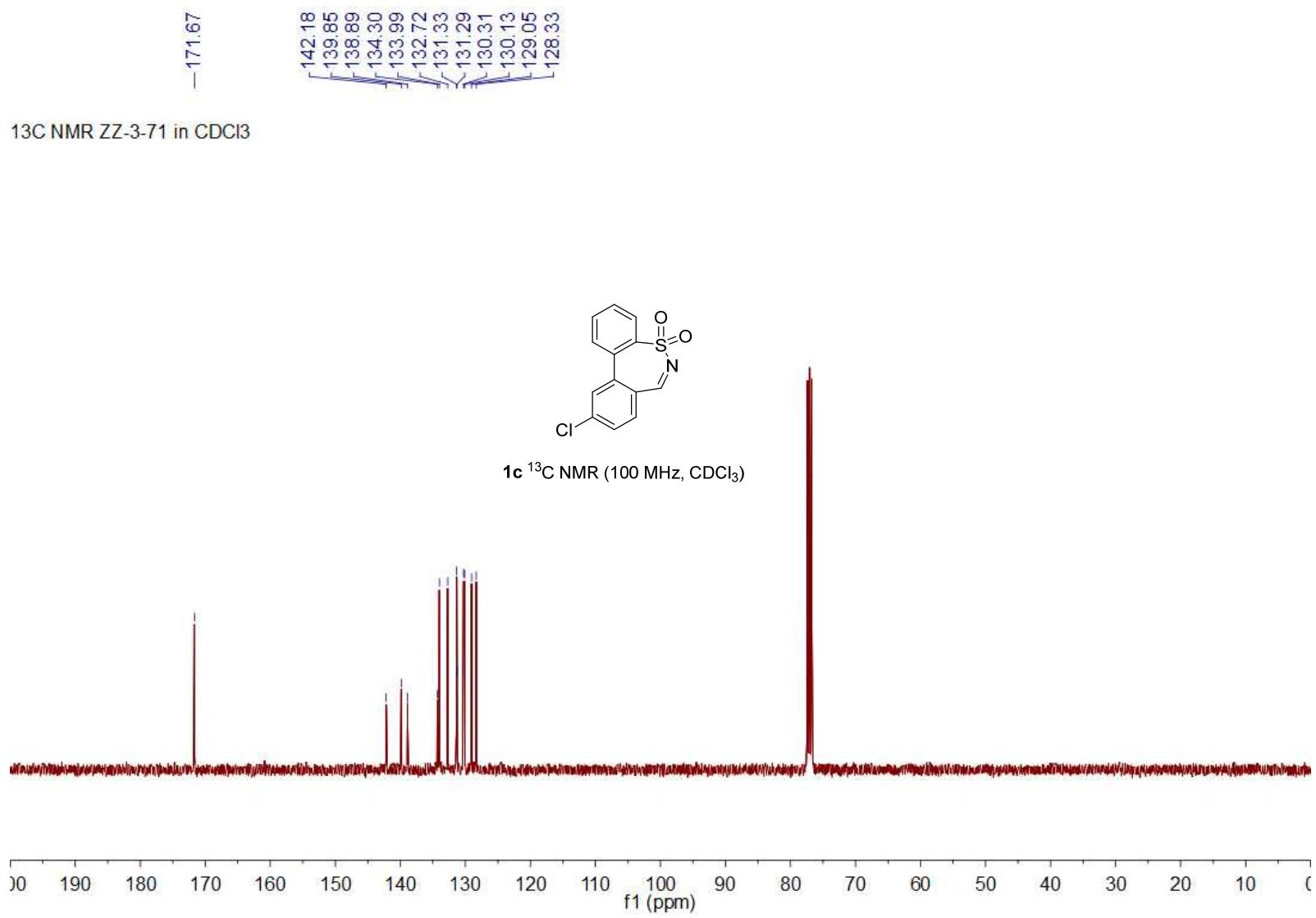
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^1H NMR ZZ-3-71 in CDCl_3



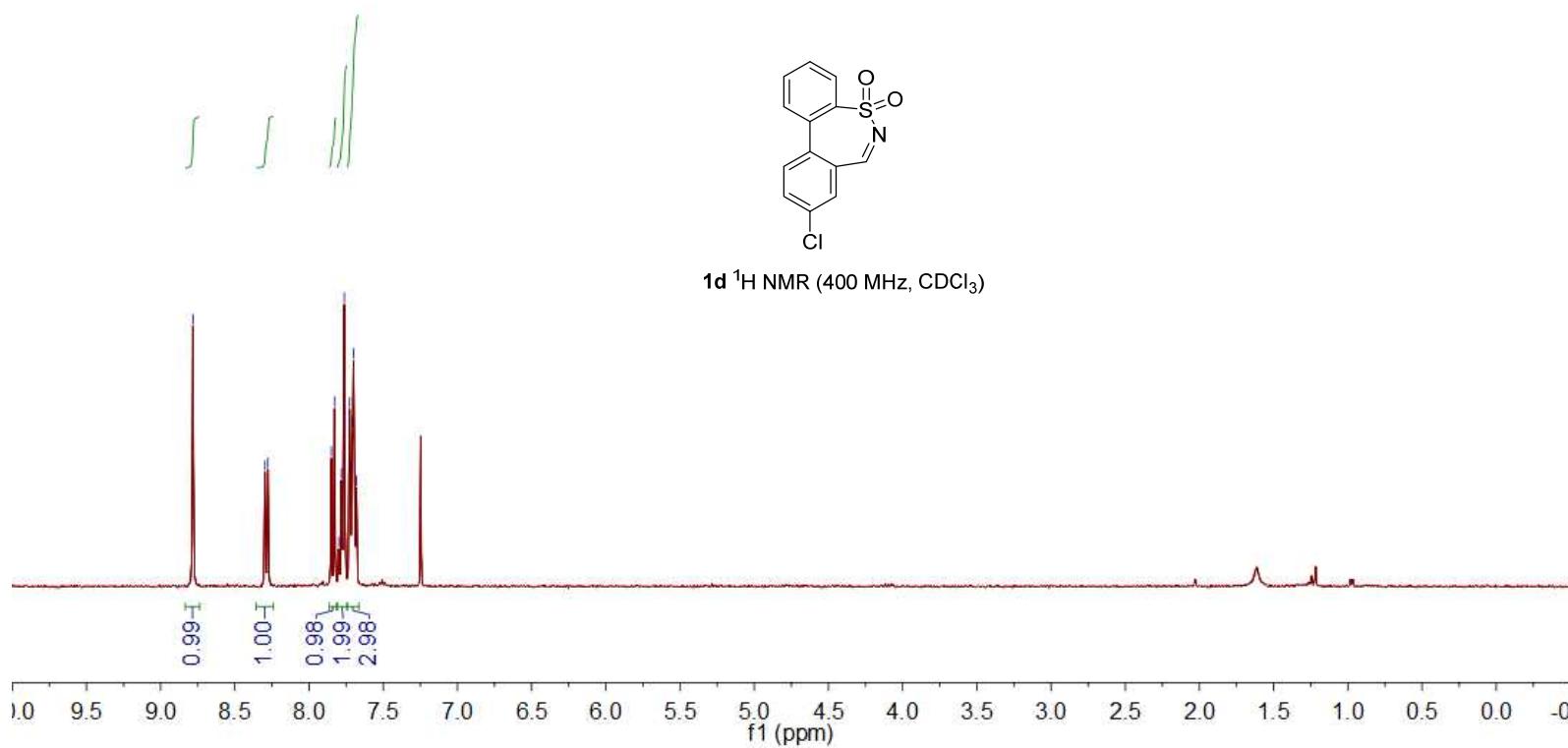
1c ^1H NMR (400 MHz, CDCl_3)

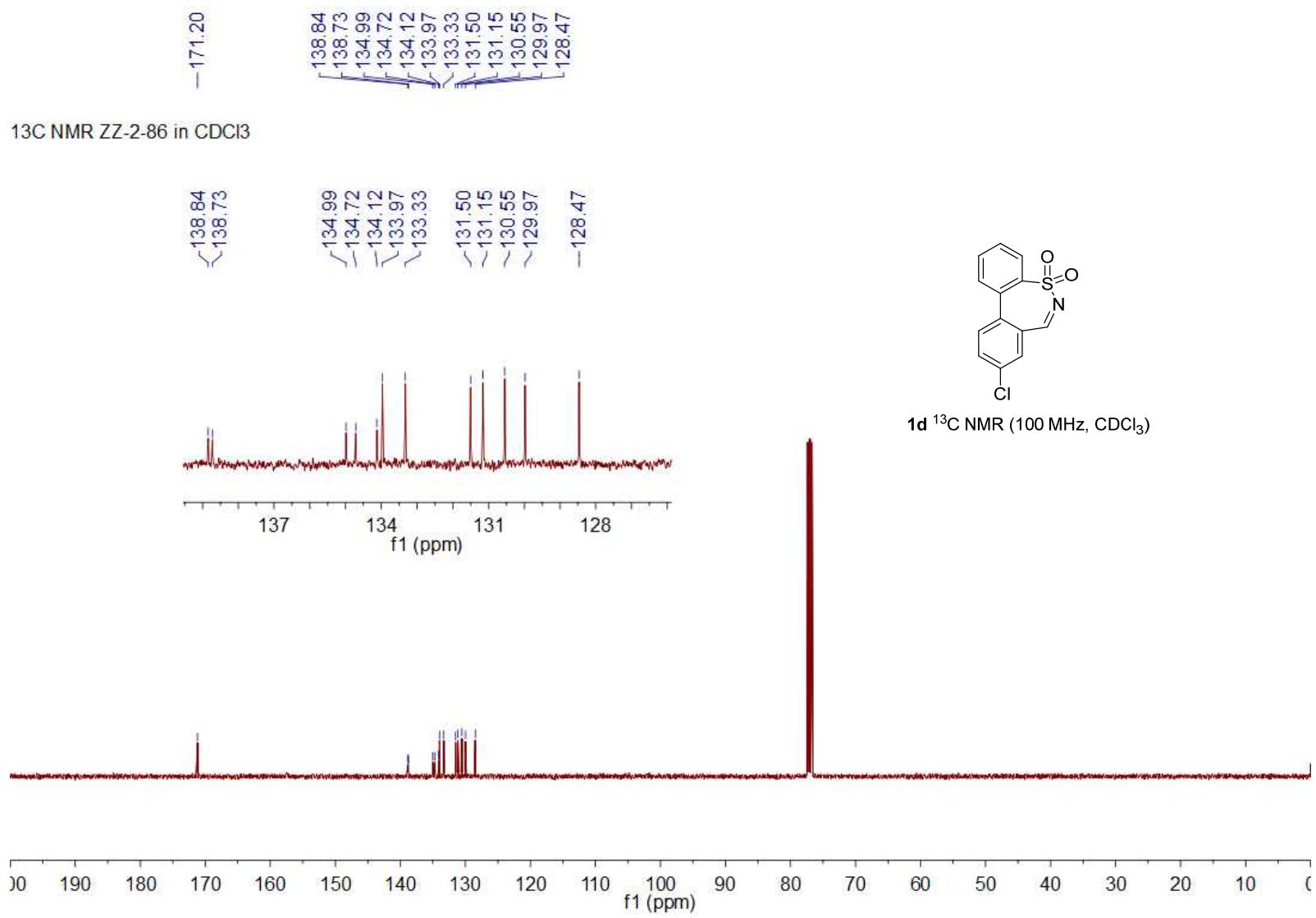


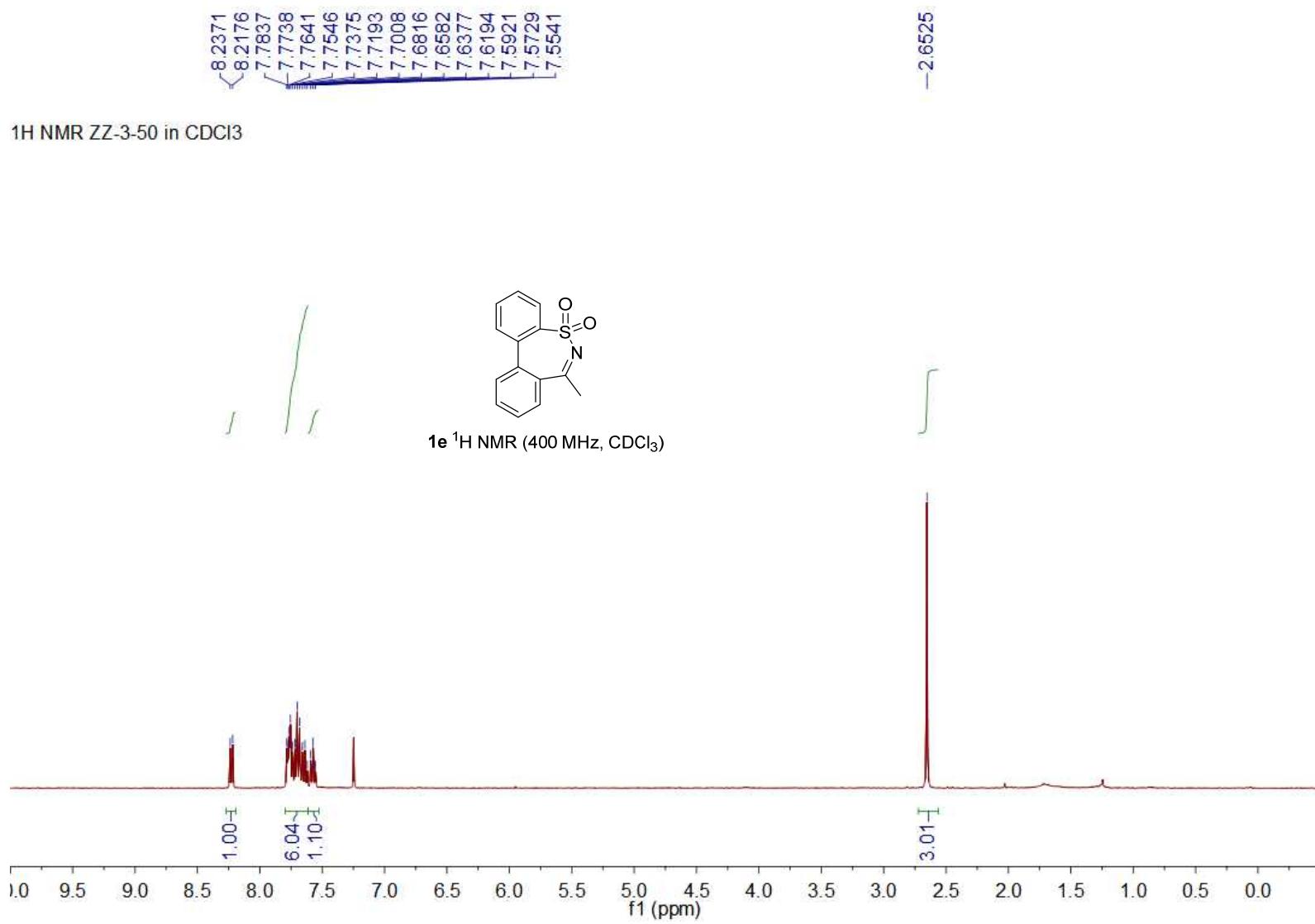


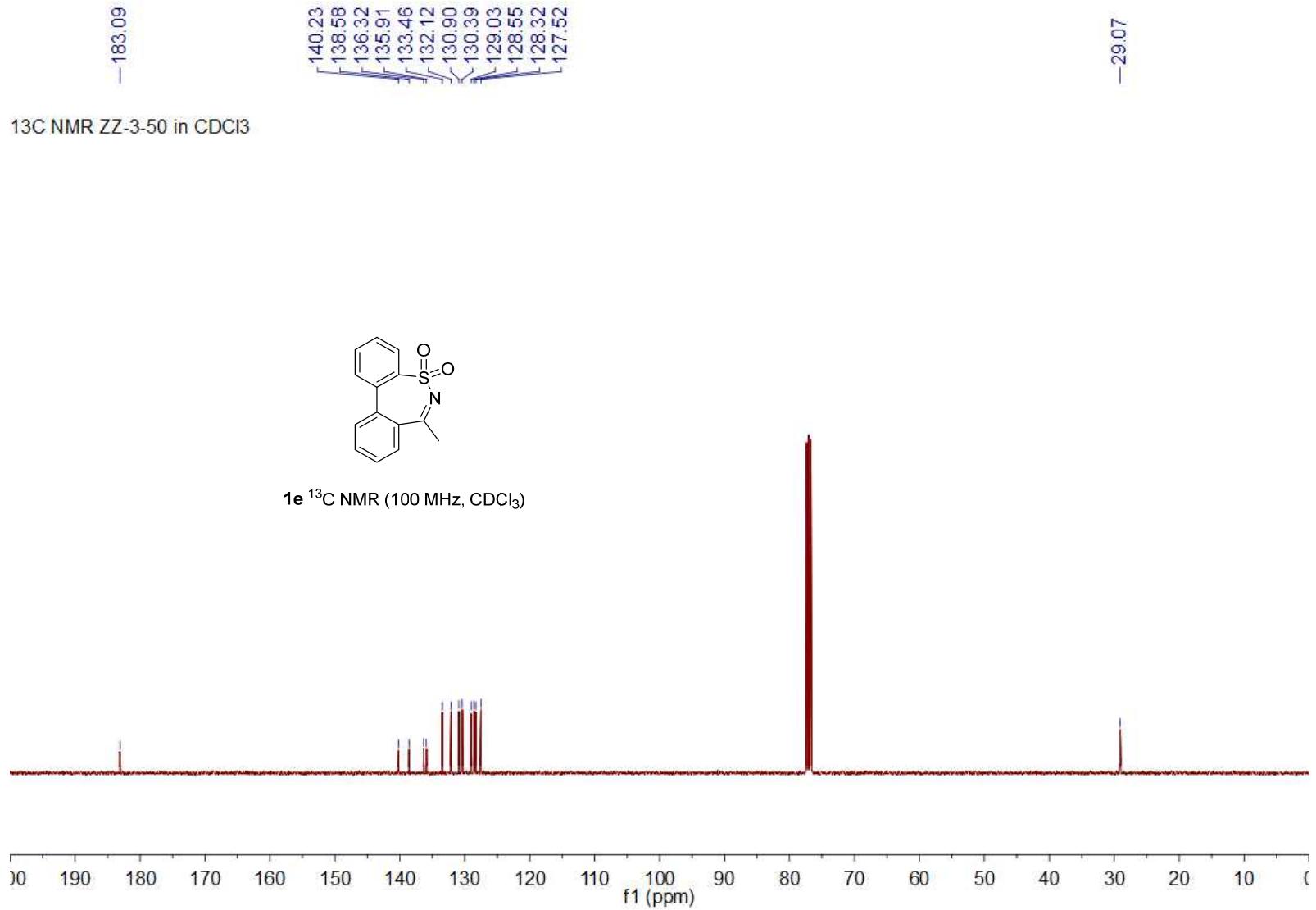
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-7.8492
-7.8280
-7.8000
-7.7816
-7.7621
-7.7265
-7.7205
-7.7082
-7.7003
-7.6815

^1H NMR ZZ-2-86 in CDCl_3





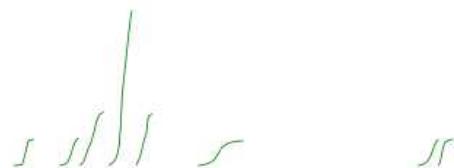




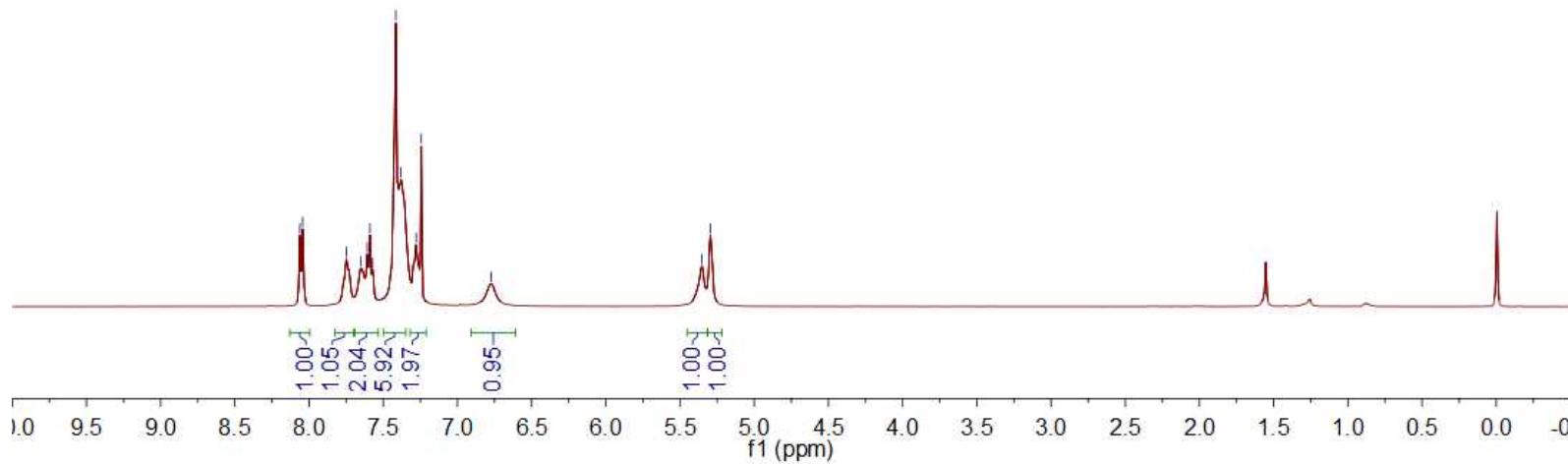
8.0622
8.0430
7.7479
7.6502
7.6083
7.5896
7.5714
7.4154
7.3799
7.2782
7.2439
-6.7730

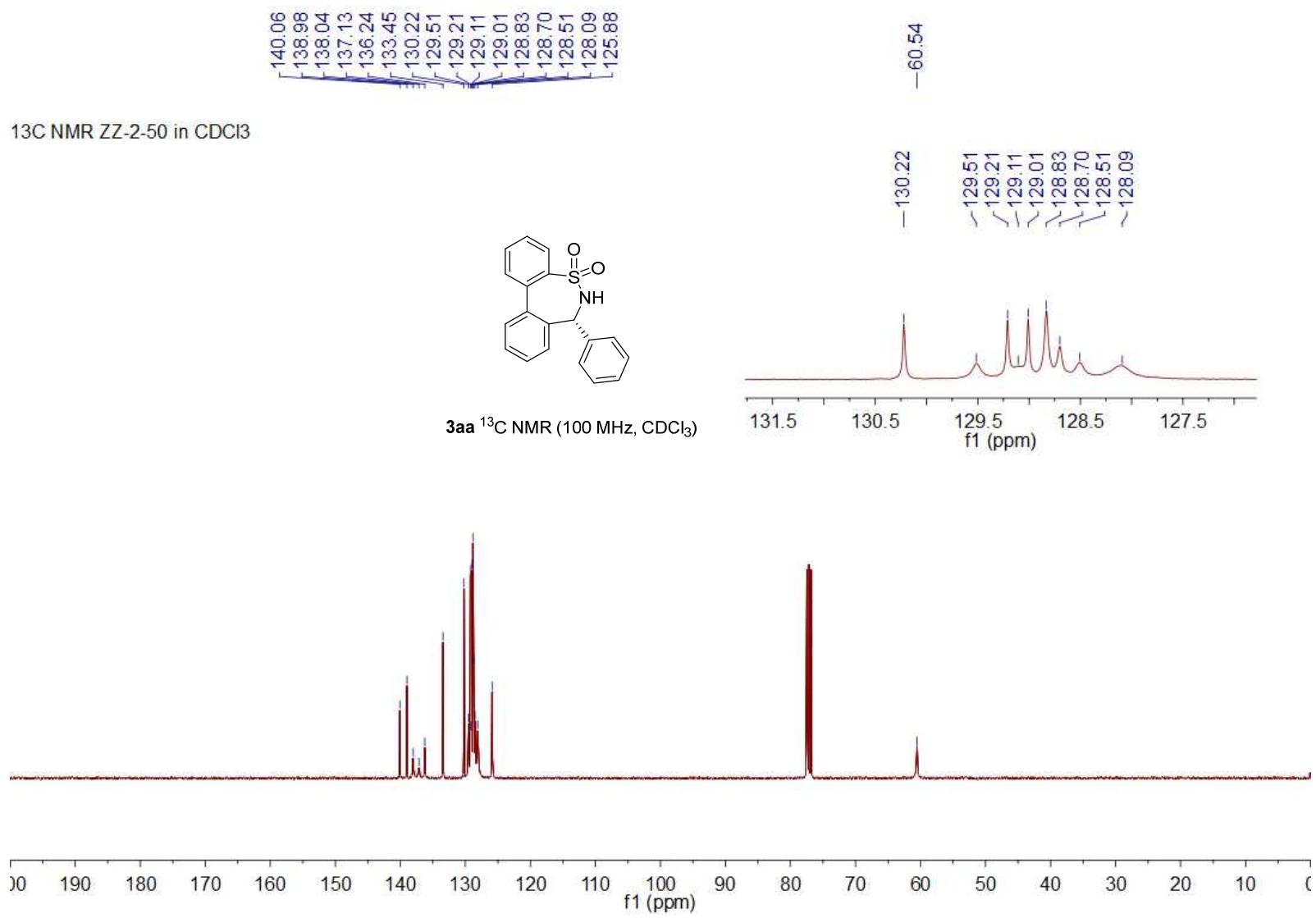
5.3531
5.2953

^1H NMR ZZ-2-50 in CDCl_3



3aa ^1H NMR (400 MHz, CDCl_3)



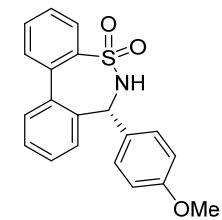
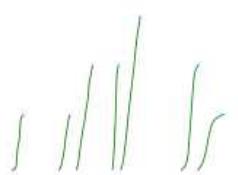


8.0746
8.0555
7.7749
7.7577
7.7382
7.6609
7.6199
7.6007
7.3517
7.3130
7.2927
6.9315
6.9113
6.8148

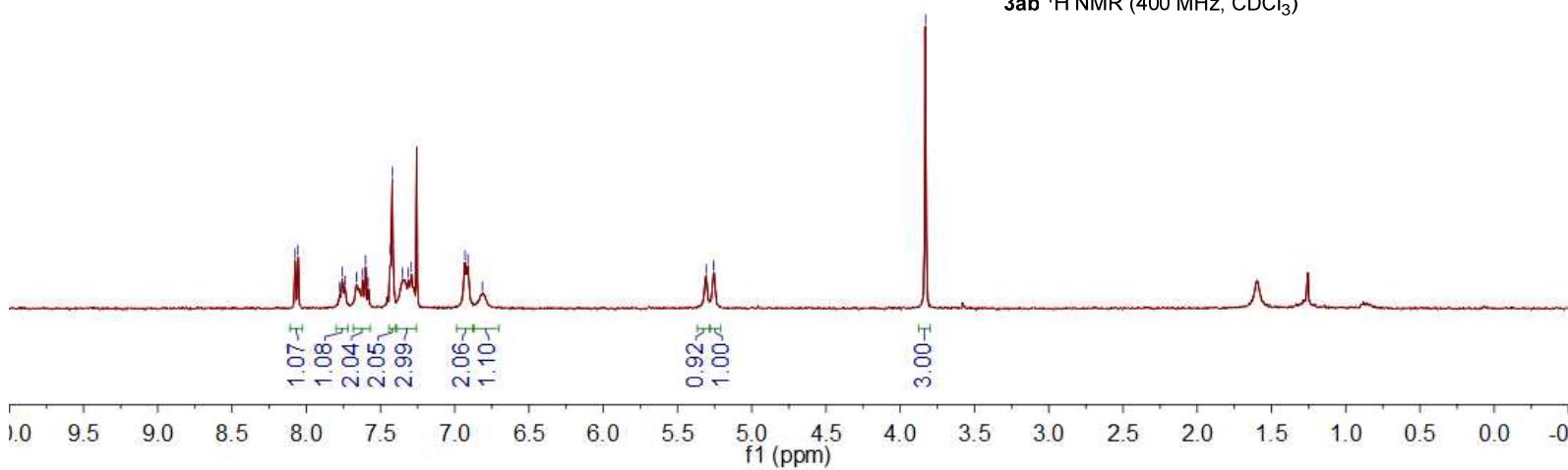
5.3070
5.2563

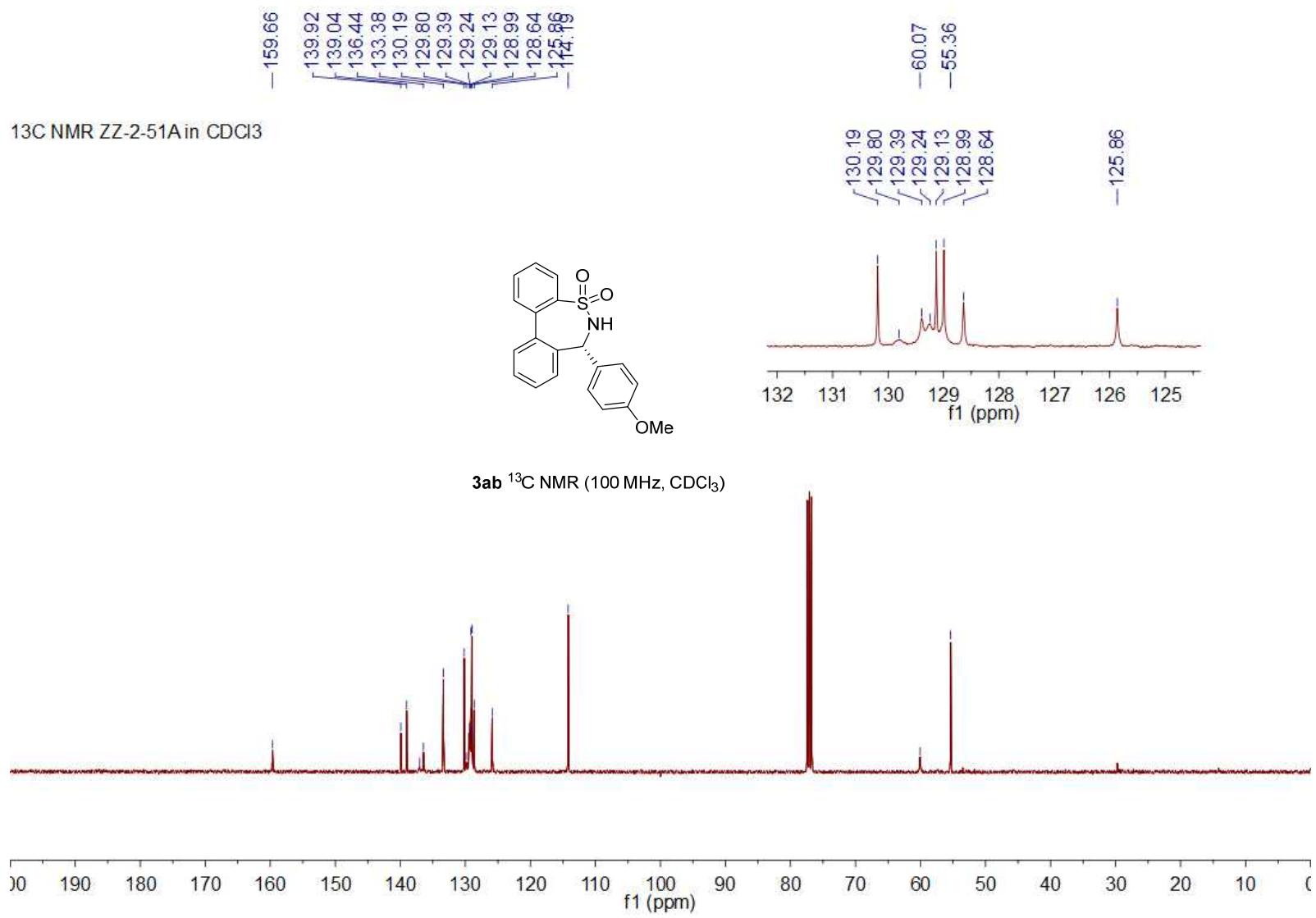
-3.8299

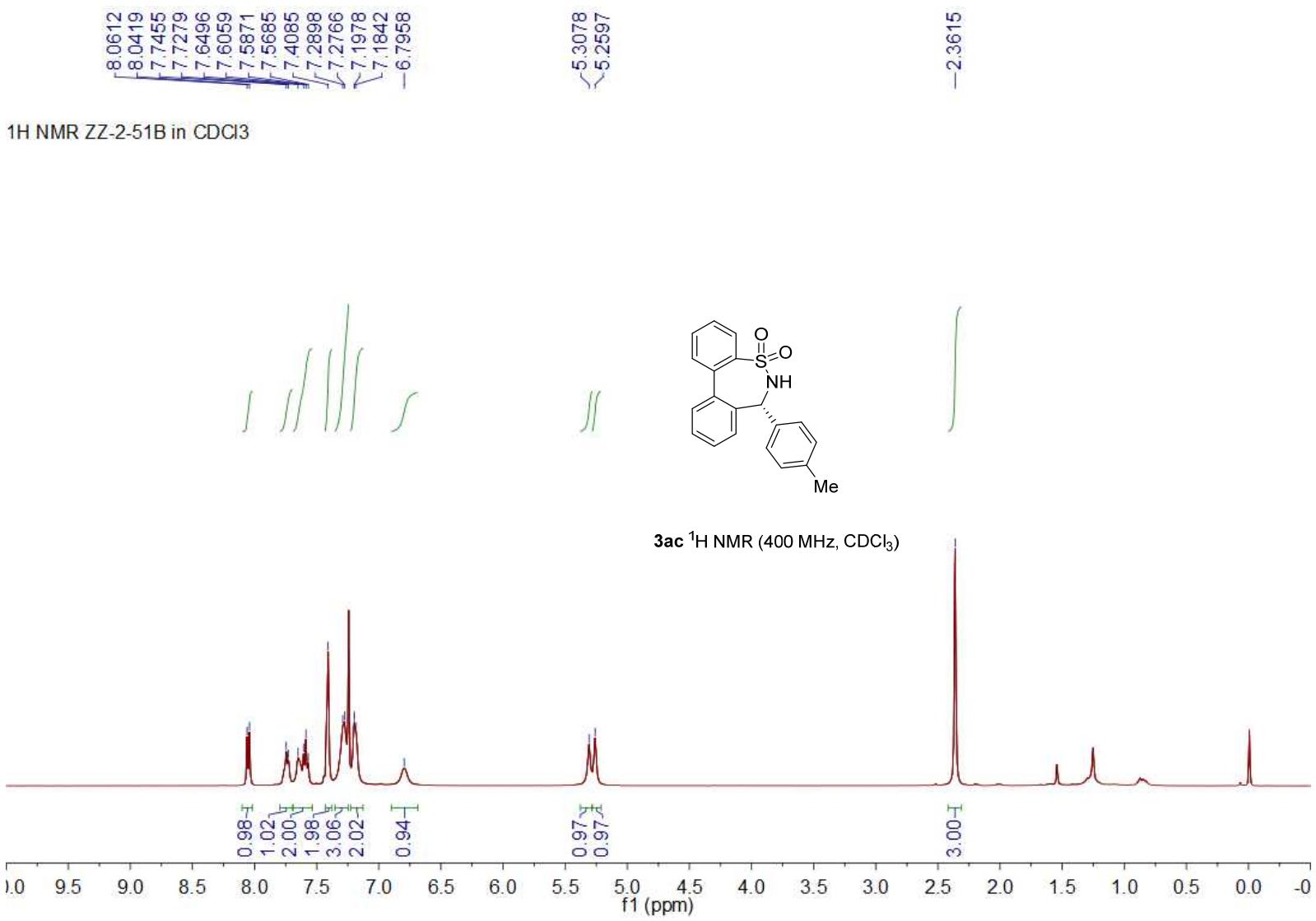
^1H NMR ZZ-2-51A in CDCl_3

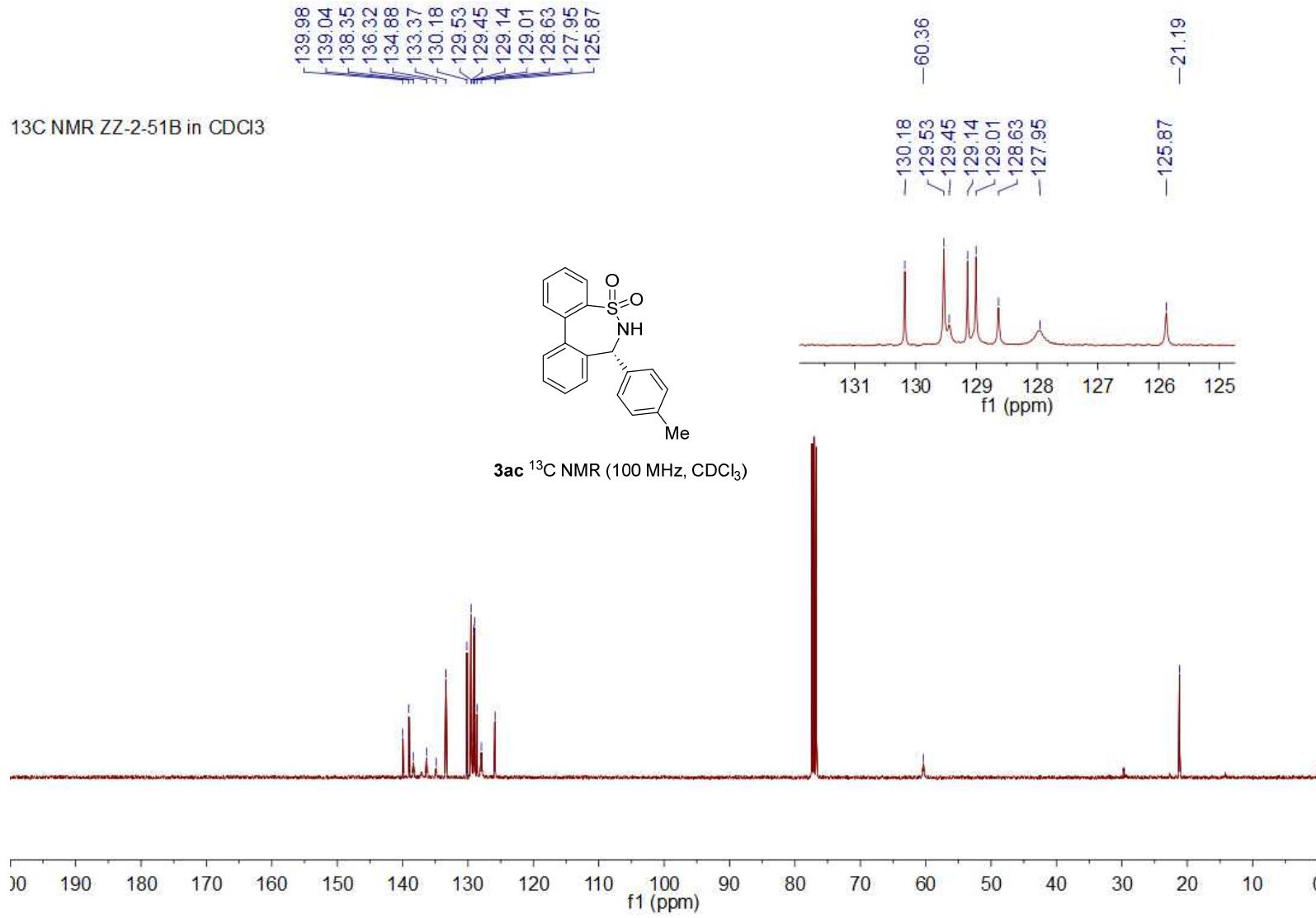


3ab ^1H NMR (400 MHz, CDCl_3)







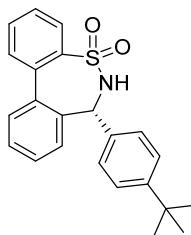


7.9804
7.9611
7.6728
7.5830
7.5295
7.5105
7.4918
7.3502
7.3440
7.3369
7.3253
7.3176
7.2629
7.2334
7.2256
7.2183
7.1947

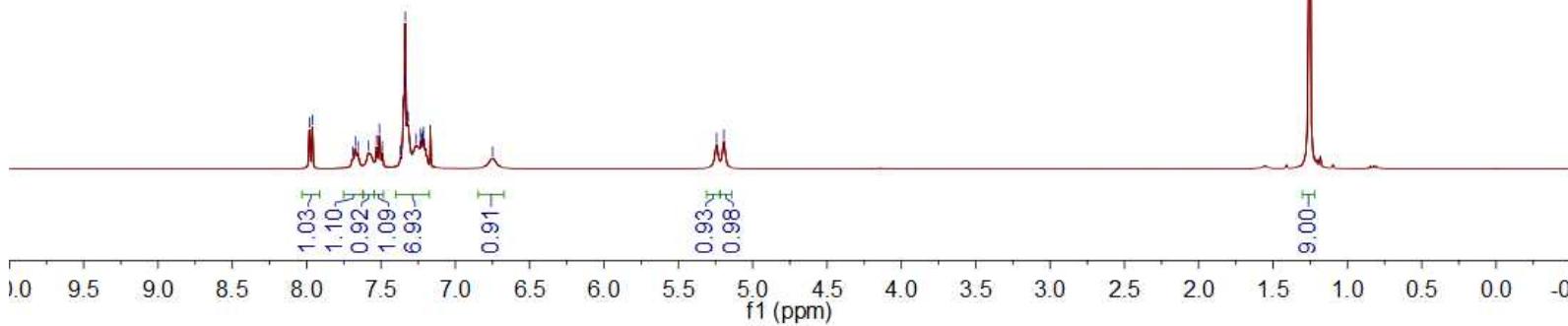
1H NMR ZZ-2-94 in CDCl₃

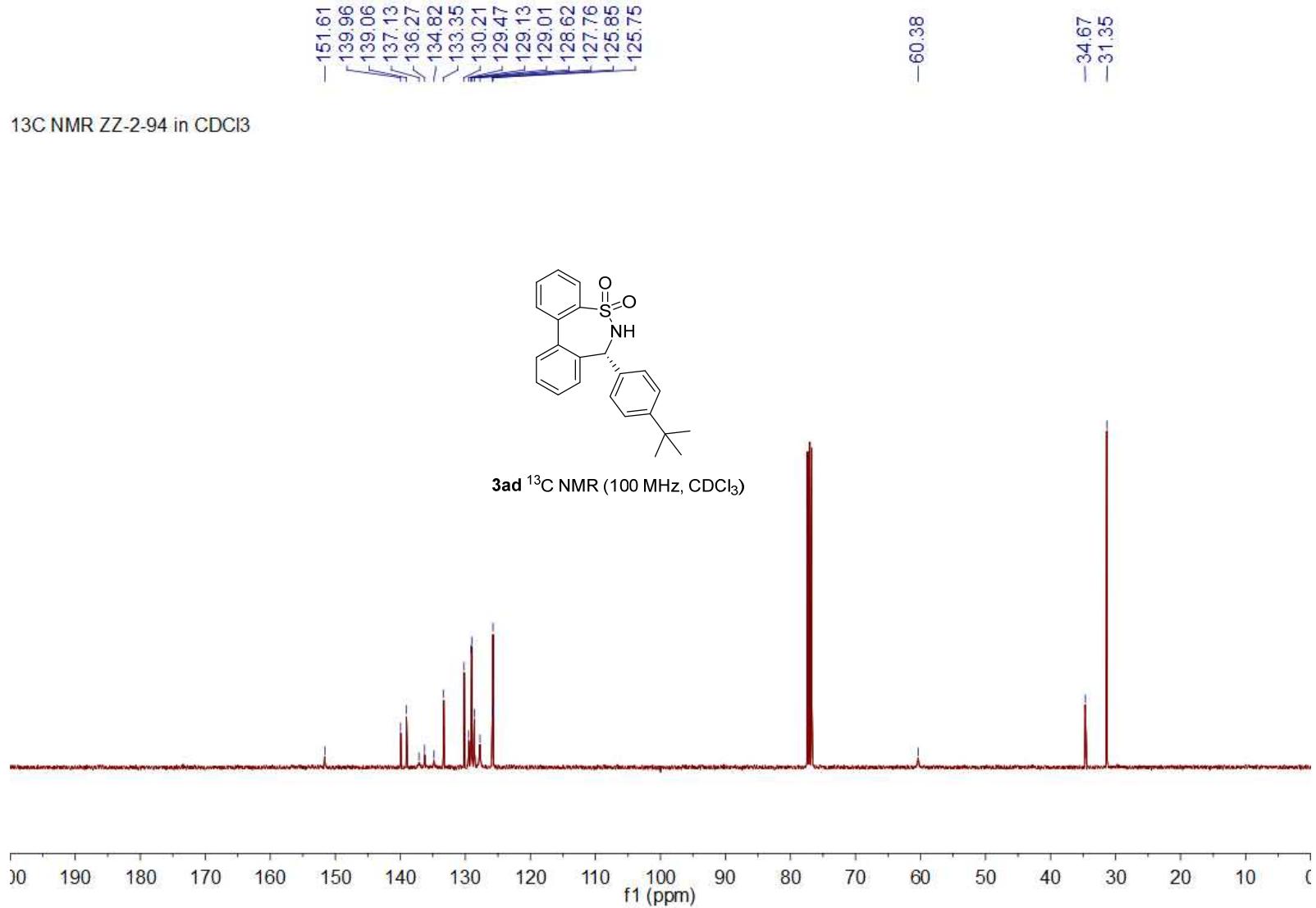
5.2432
5.1941

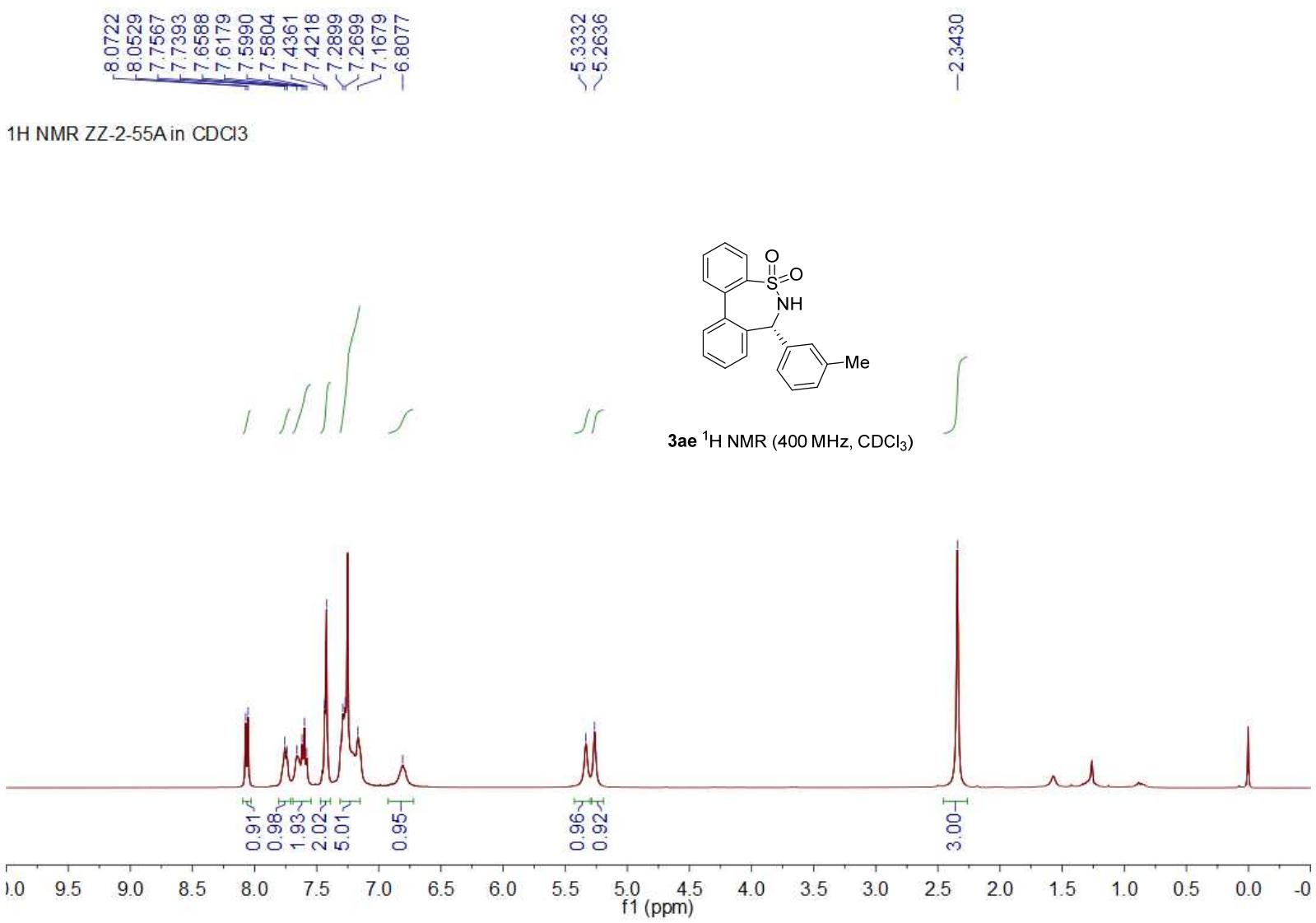
-1.2552

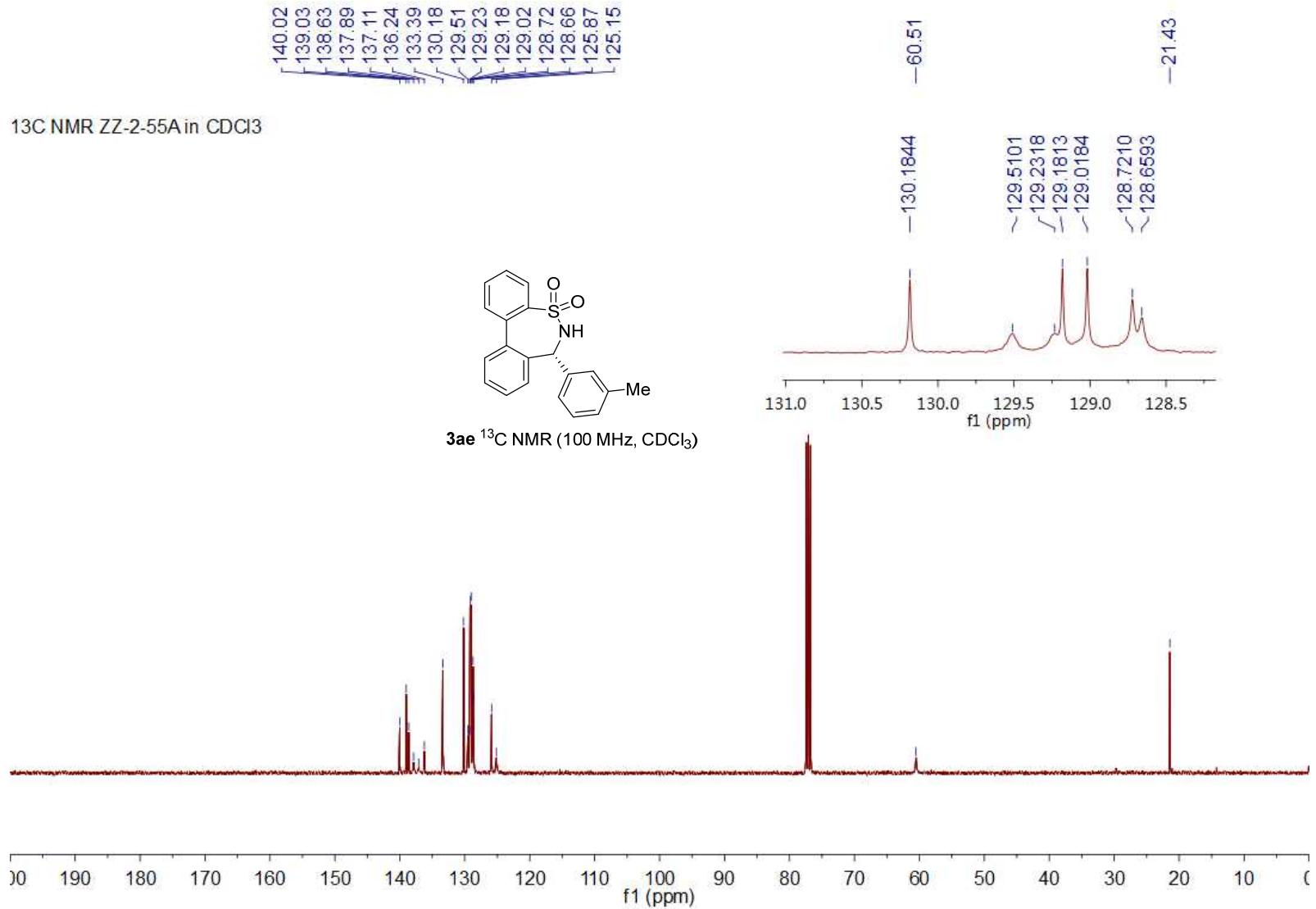


3ad ¹H NMR (400 MHz, CDCl₃)



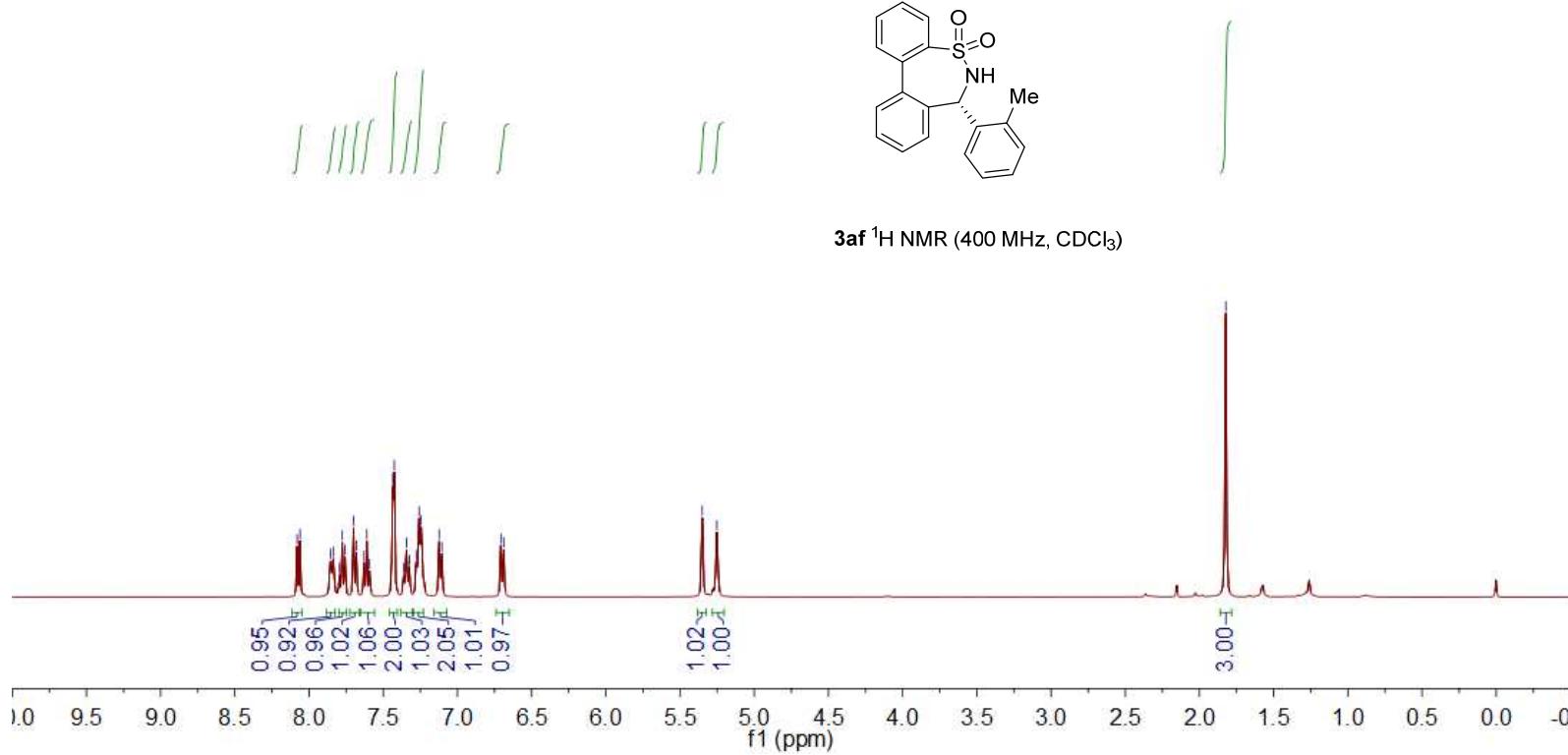








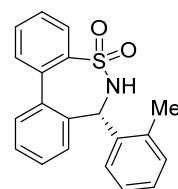
^1H NMR ZZ-2-55B in CDCl_3



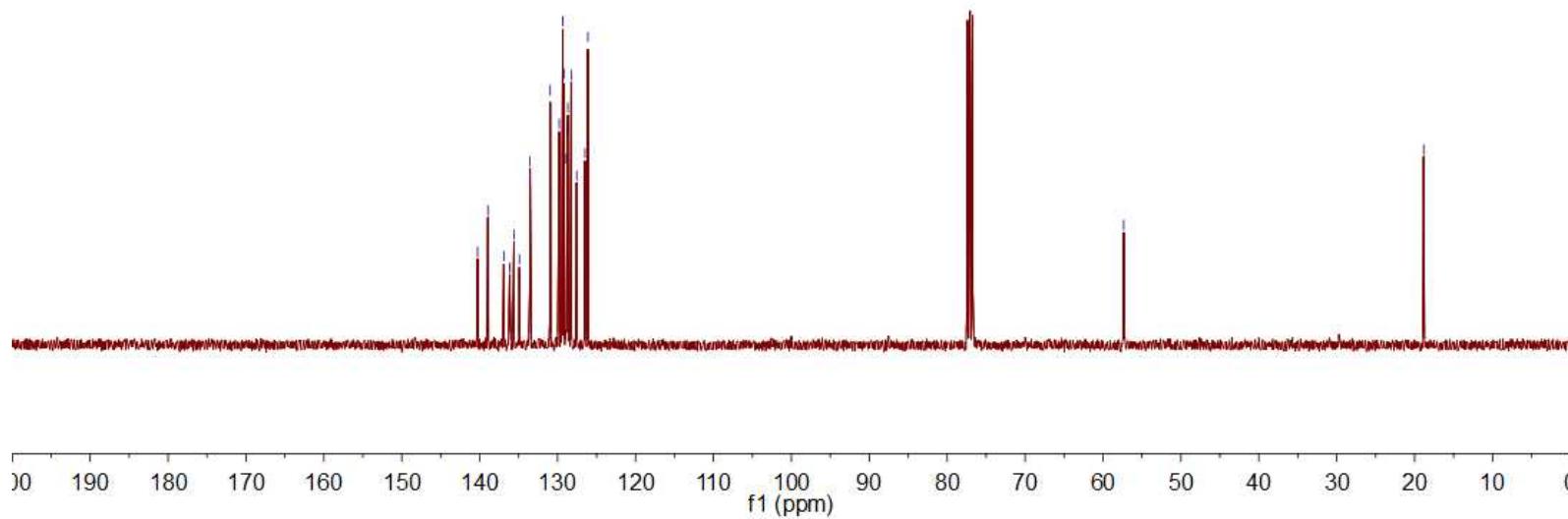
3af ^1H NMR (400 MHz, CDCl_3)



¹³C NMR ZZ-2-55B in CDCl₃



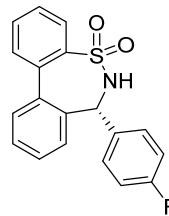
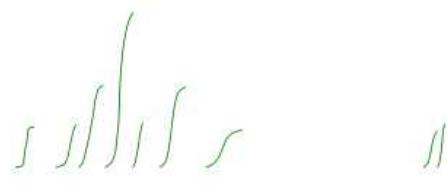
3af ¹³C NMR (100 MHz, CDCl₃)



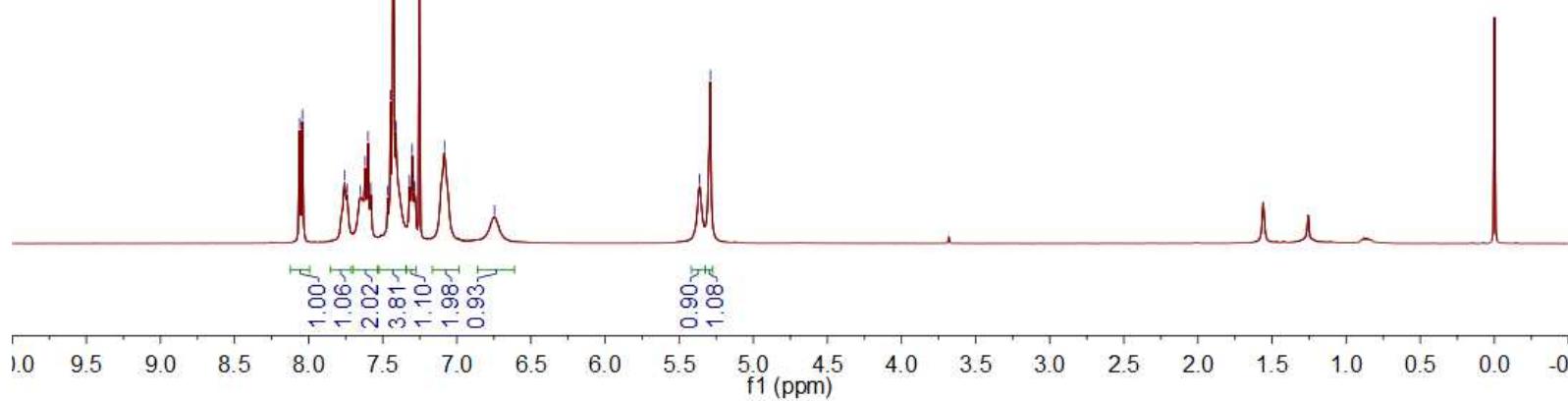
8.0610
8.0417
7.7585
7.7411
7.6501
7.6186
7.5994
7.5807
7.4661
7.4474
7.4303
7.4120
7.3197
7.3011
7.2849
7.0840
6.7468

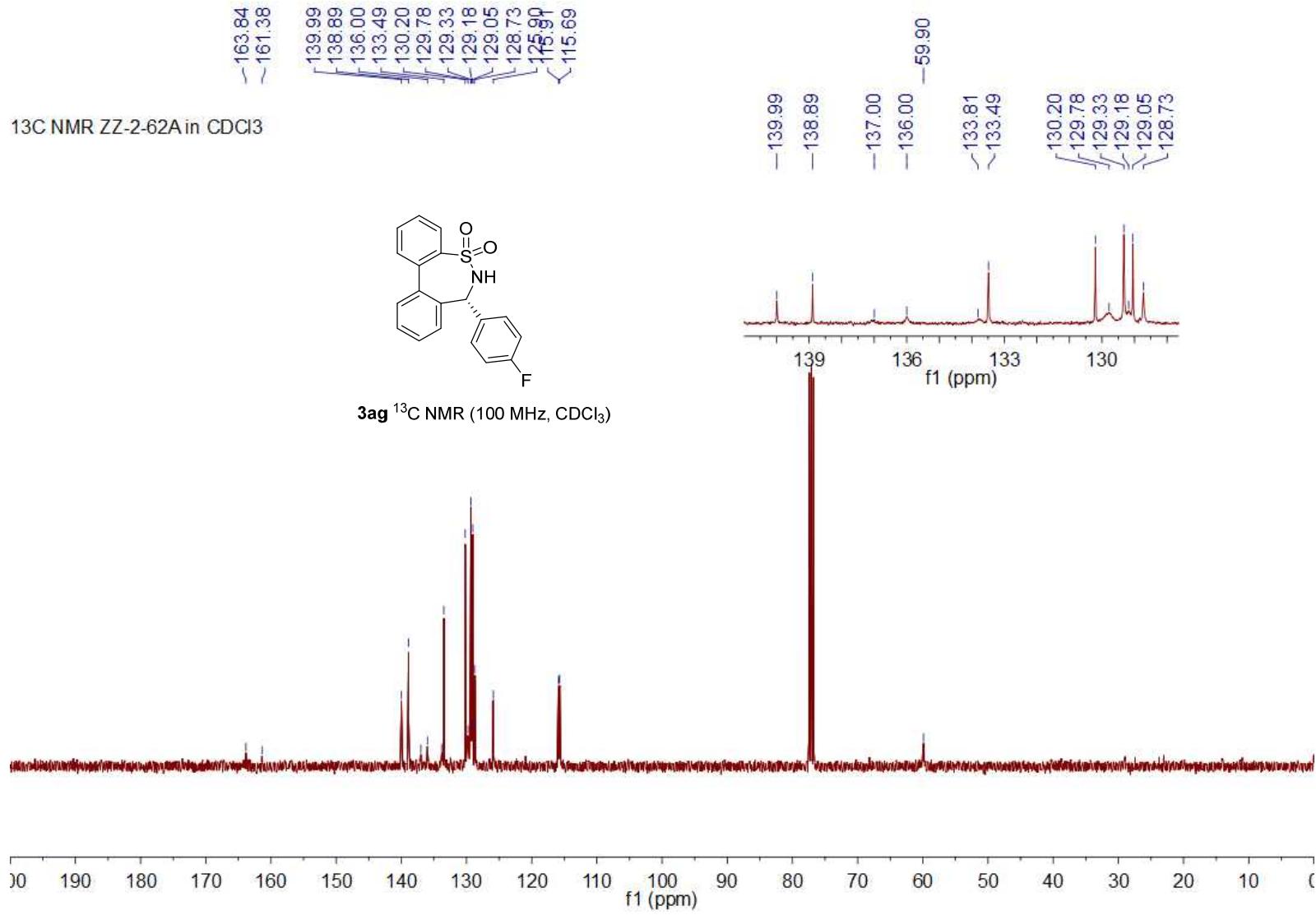
~5.3628
~5.2894

^1H NMR ZZ-2-62A in CDCl_3

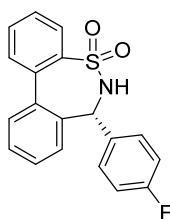


3ag ^1H NMR (400 MHz, CDCl_3)

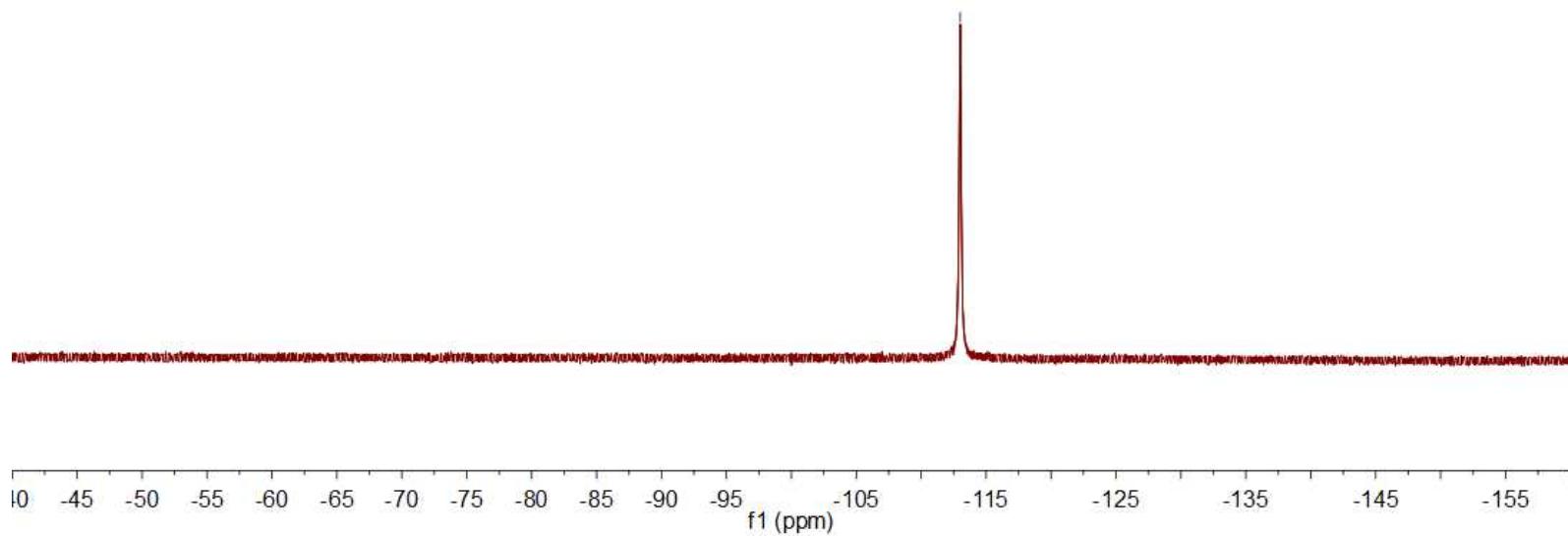


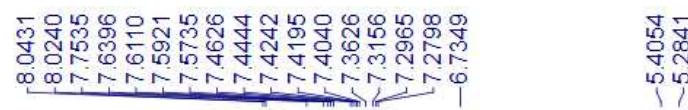


¹⁹F NMR ZZ-2-62A in CDCl₃

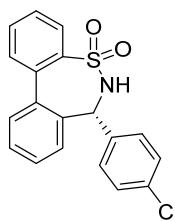


3ag ¹⁹F NMR (376 MHz, CDCl₃)

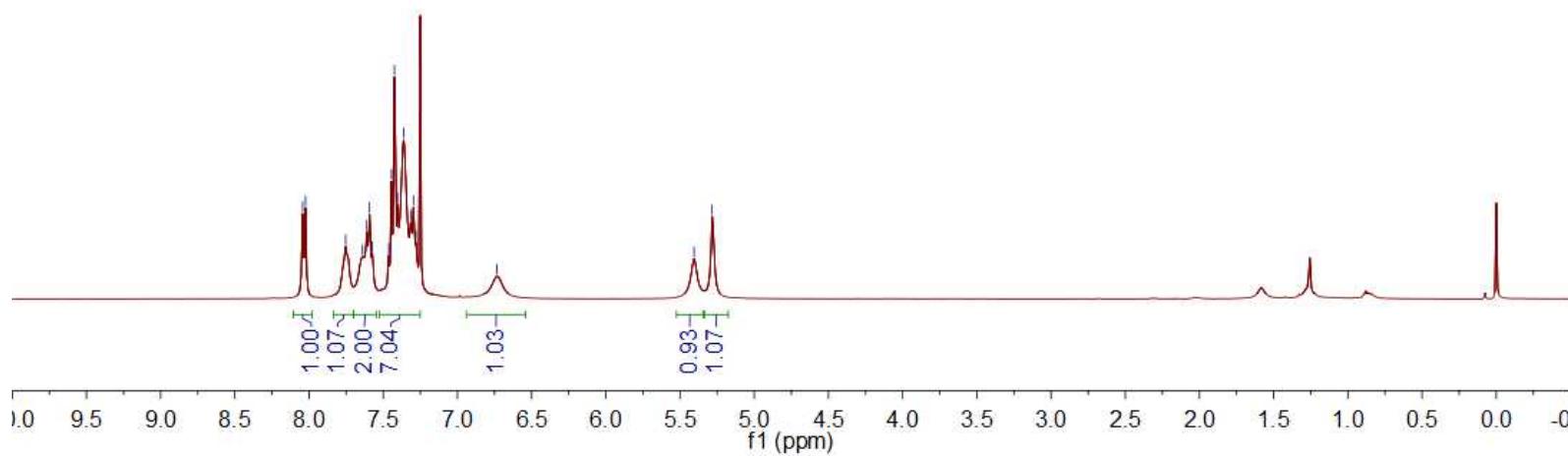


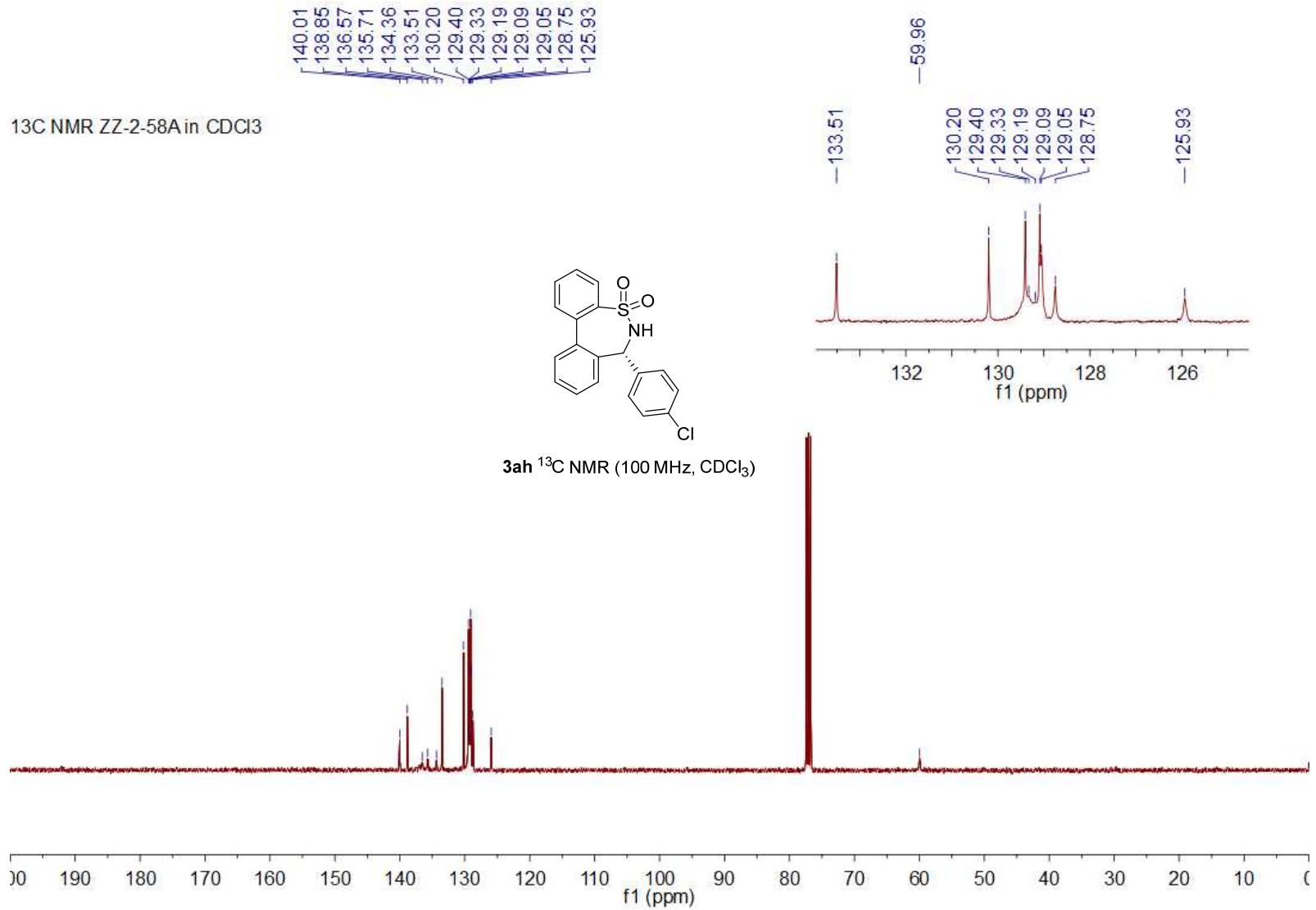


^1H NMR ZZ-2-58A in CDCl_3



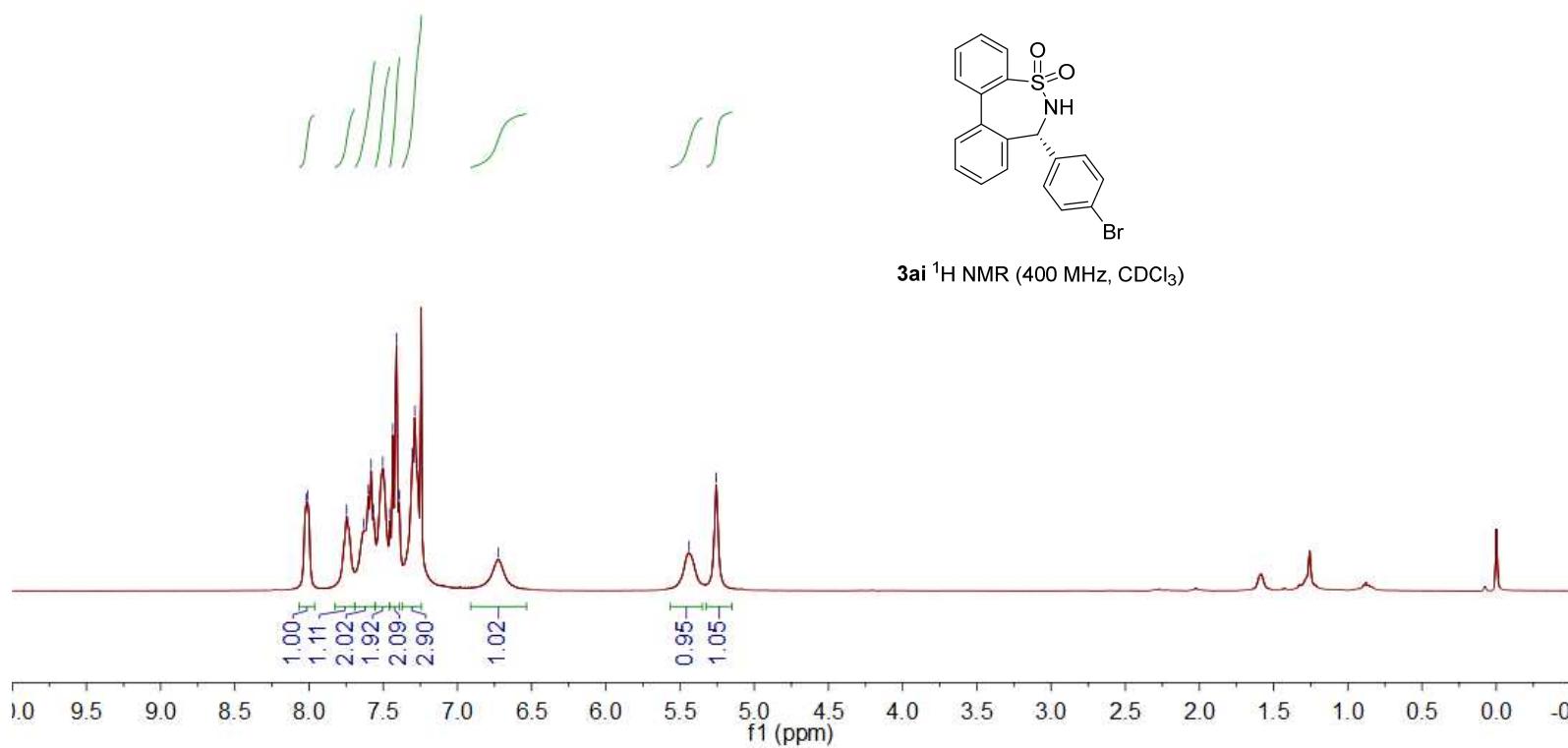
3ah ^1H NMR (400 MHz, CDCl_3)



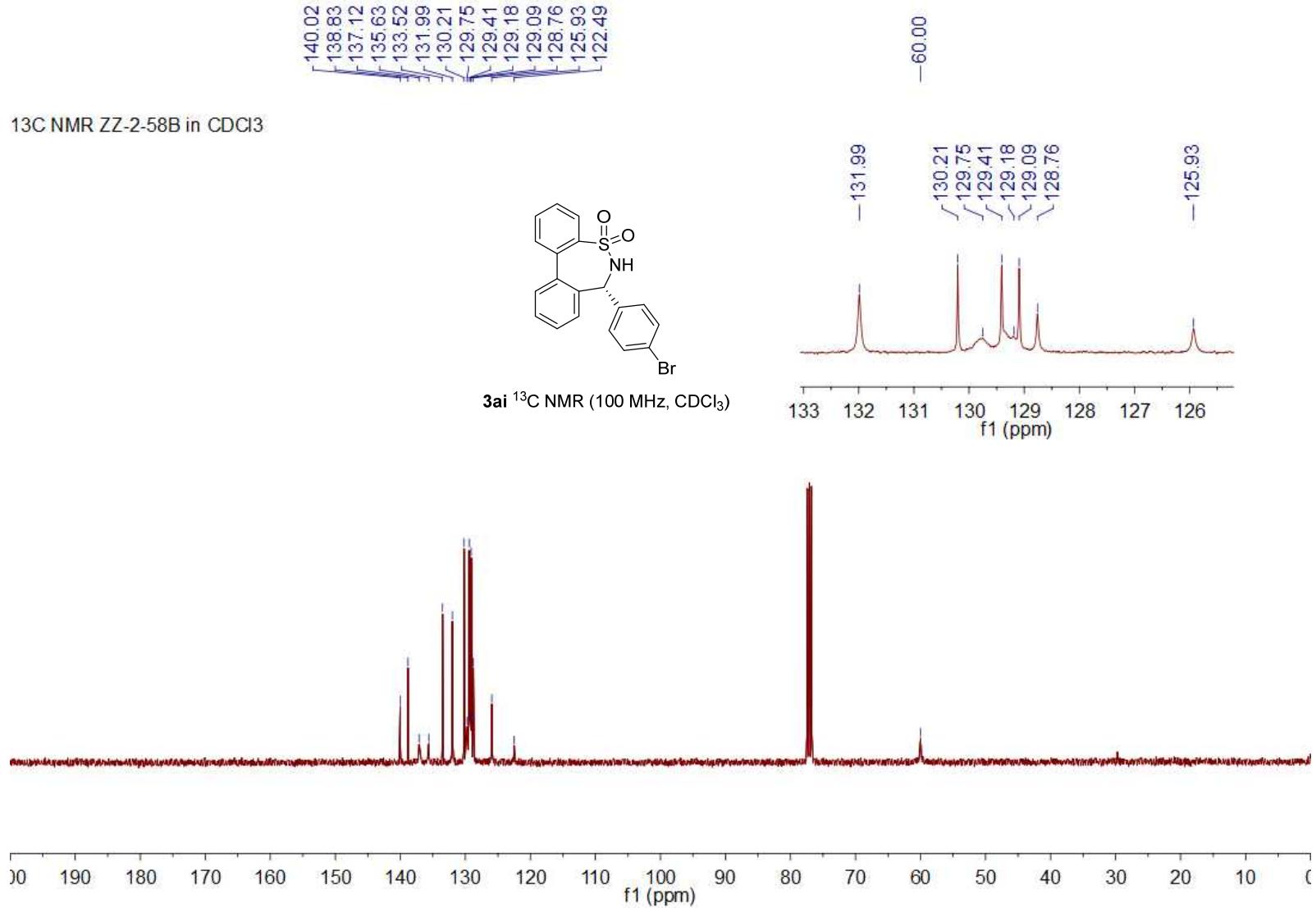


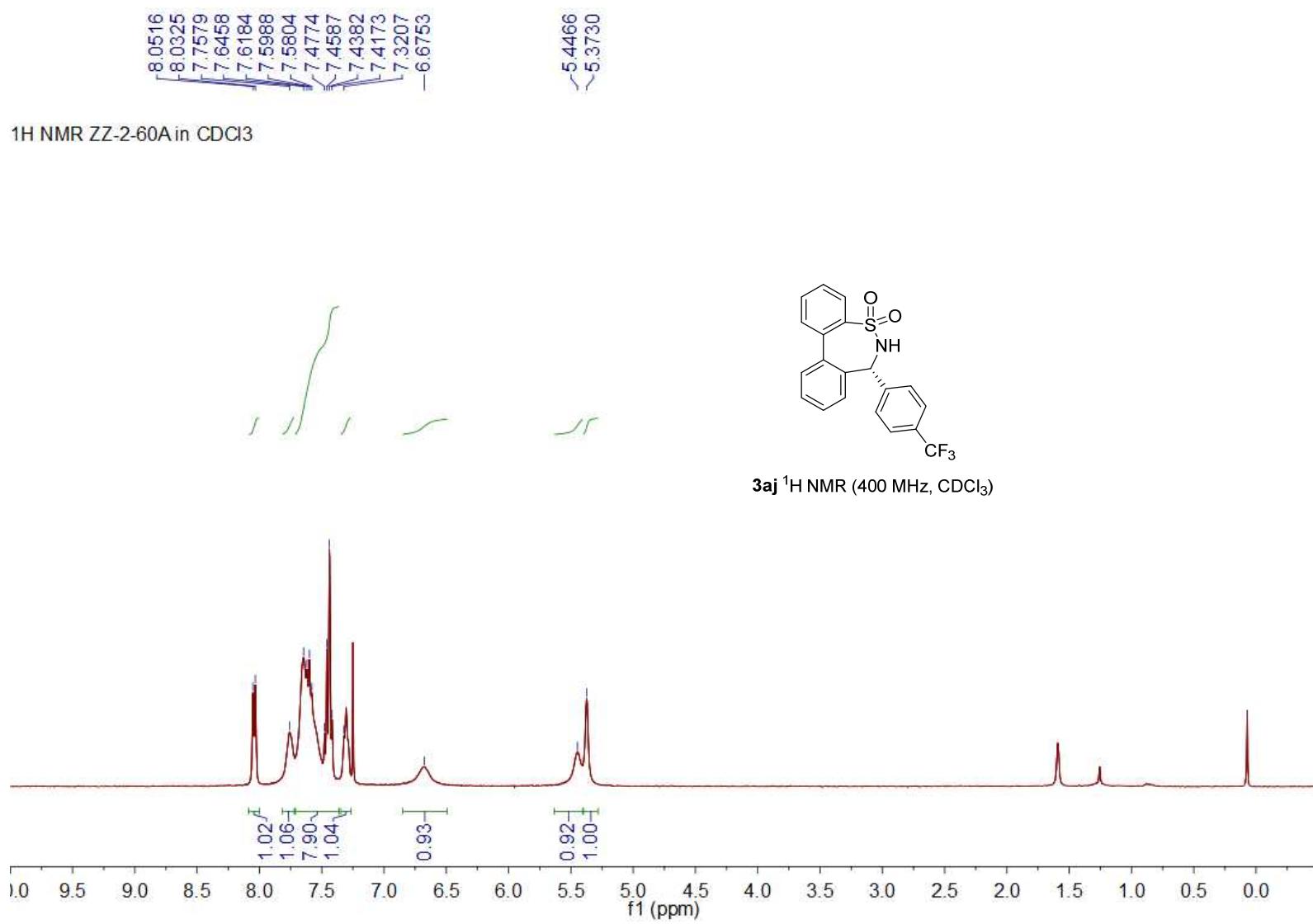
8.0204
8.0086
7.7460
7.6323
7.6004
7.5820
7.5641
7.5035
7.4542
7.4355
7.4099
7.3920
7.3041
7.2880
7.2726
-6.7251

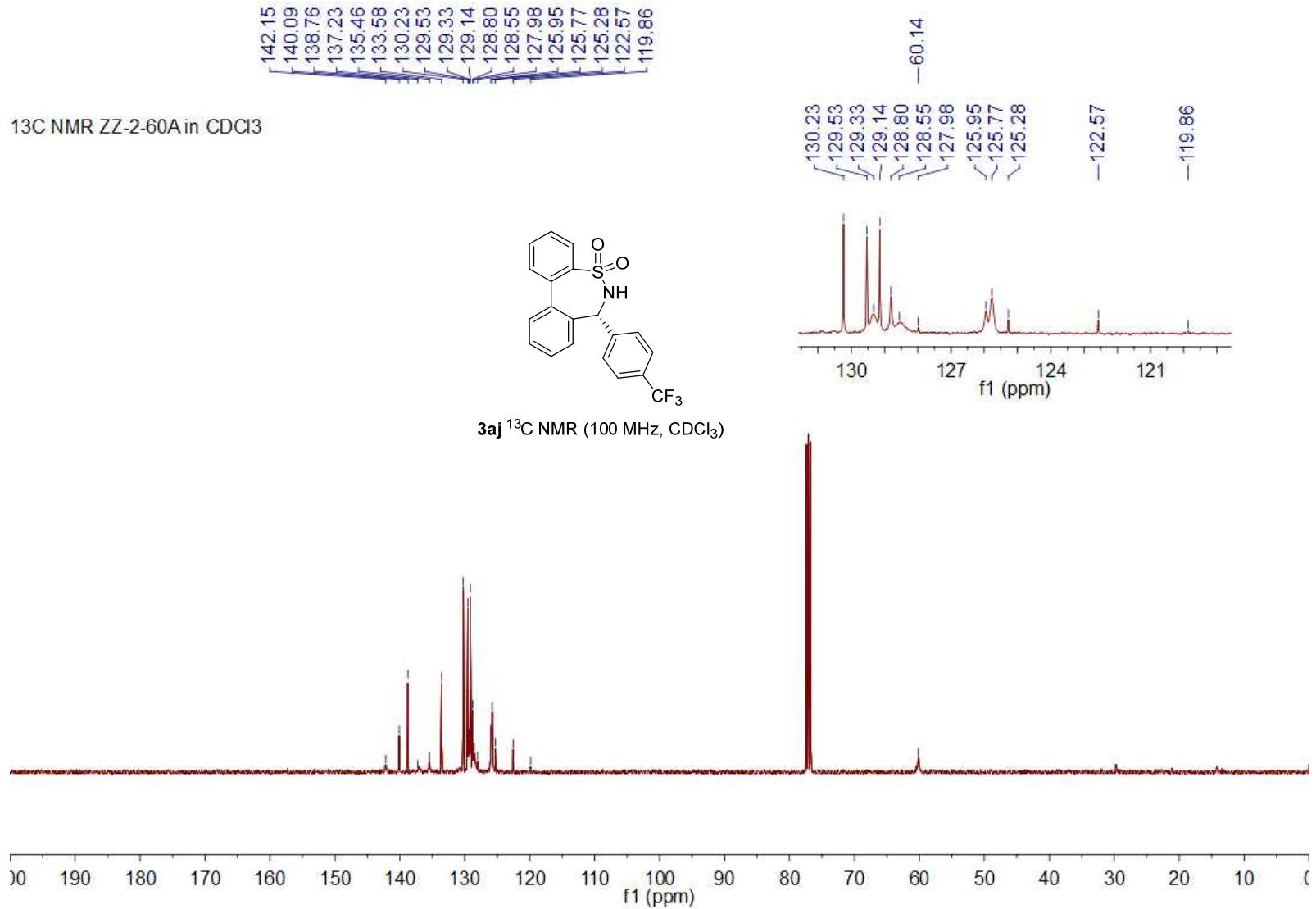
¹H NMR ZZ-2-58B in CDCl₃



3ai ¹H NMR (400 MHz, CDCl₃)

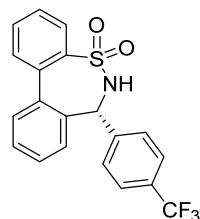




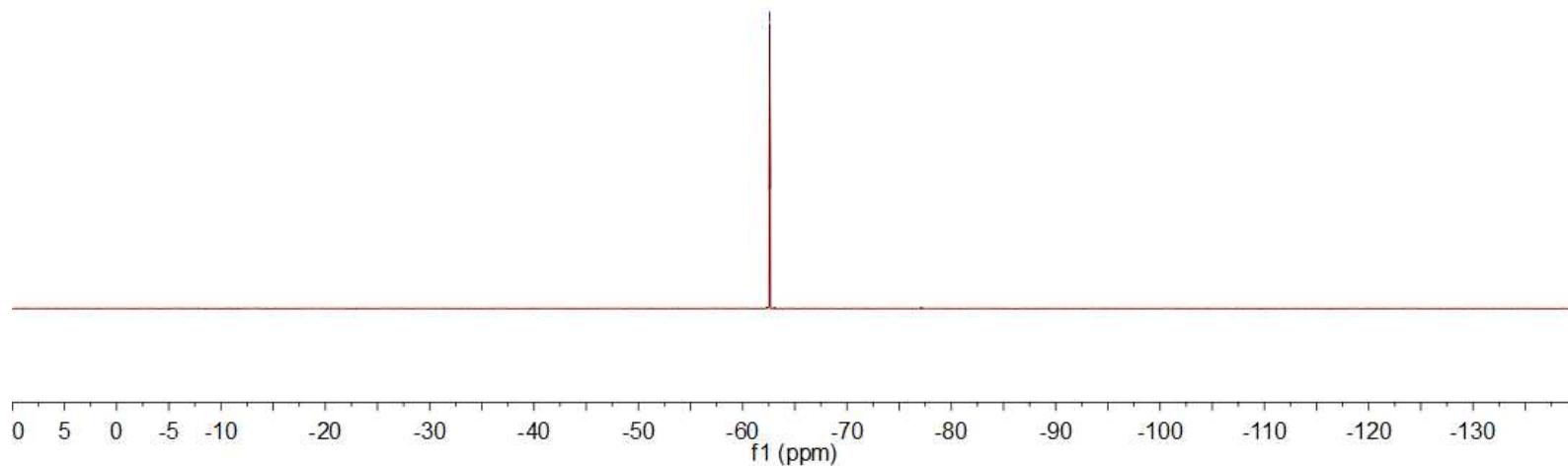


—
62.59

¹⁹F NMR ZZ-2-60 in CDCl₃



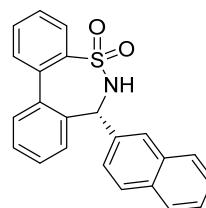
3aj ¹⁹F NMR (376 MHz, CDCl₃)



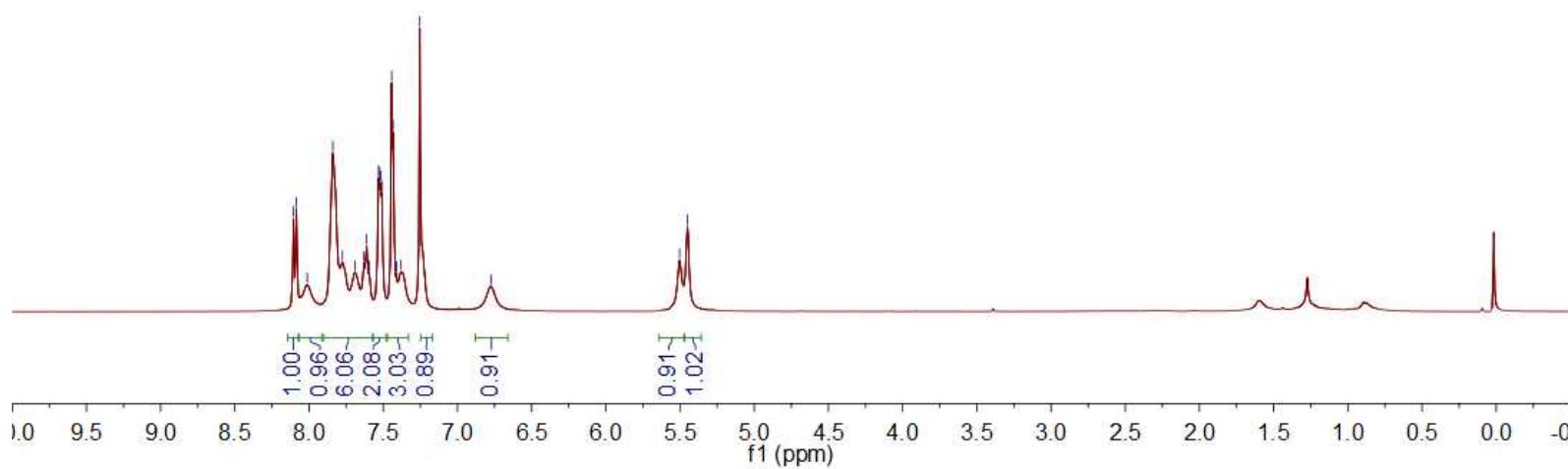
8.1046
8.0853
7.8397
7.7749
7.6904
7.6306
7.6125
7.5950
7.5313
7.5236
7.5167
7.5093
7.4430
7.4340
7.4109
7.3795
7.2538

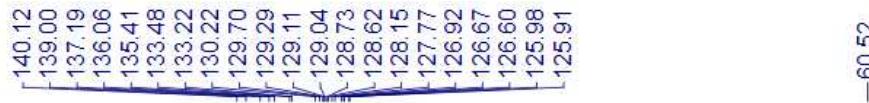
^1H NMR ZZ-2-61 in CDCl_3

5.5026
5.4498

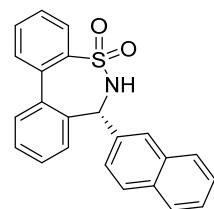


3ak ^1H NMR (400 MHz, CDCl_3)

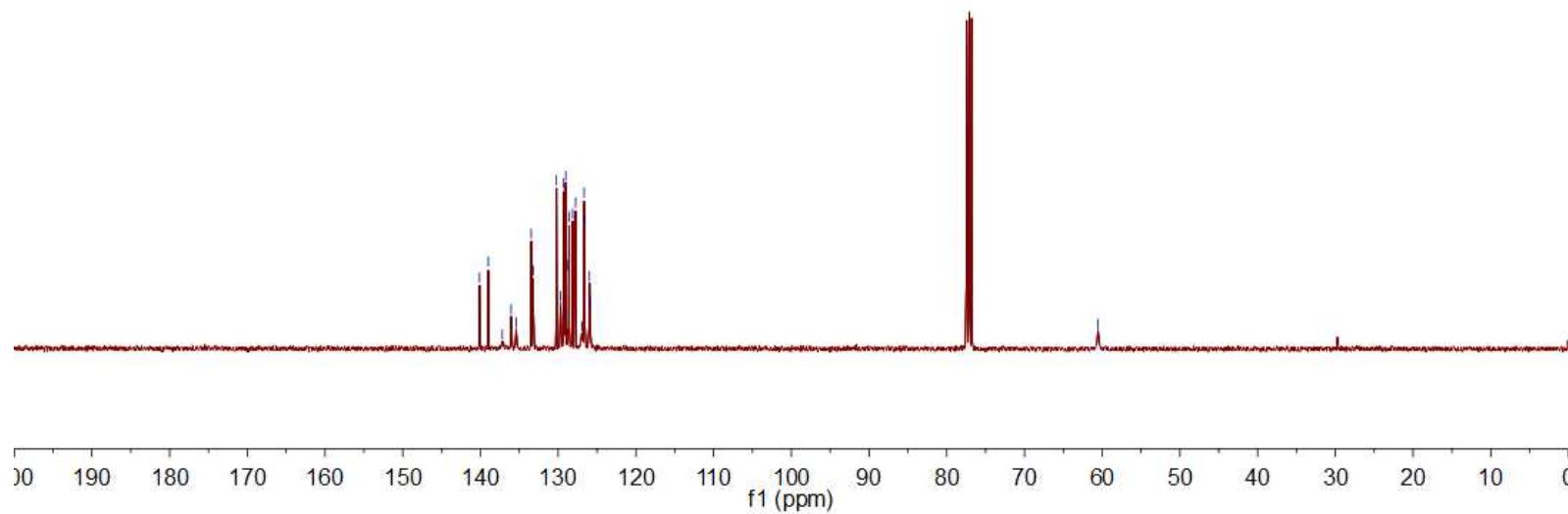


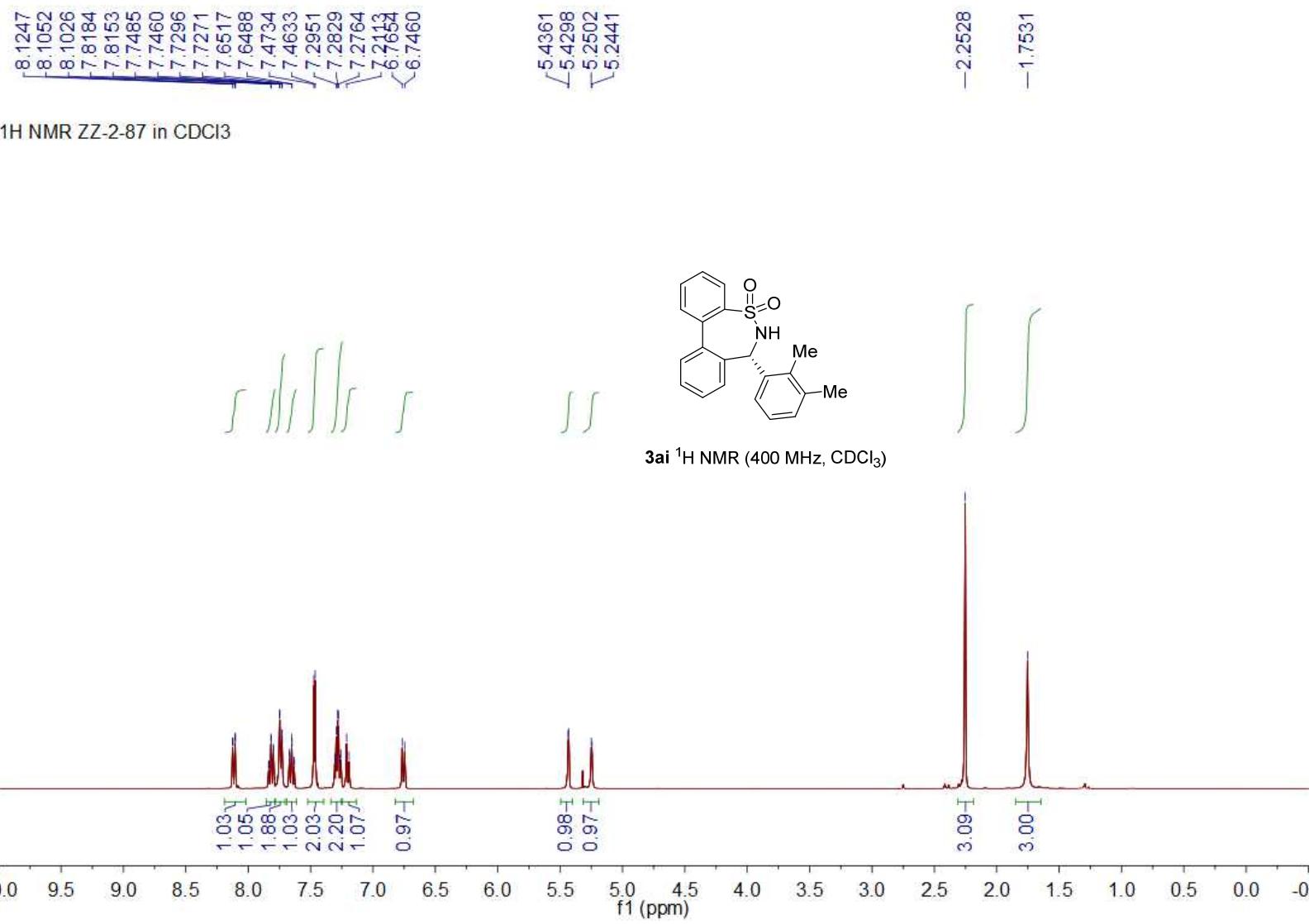


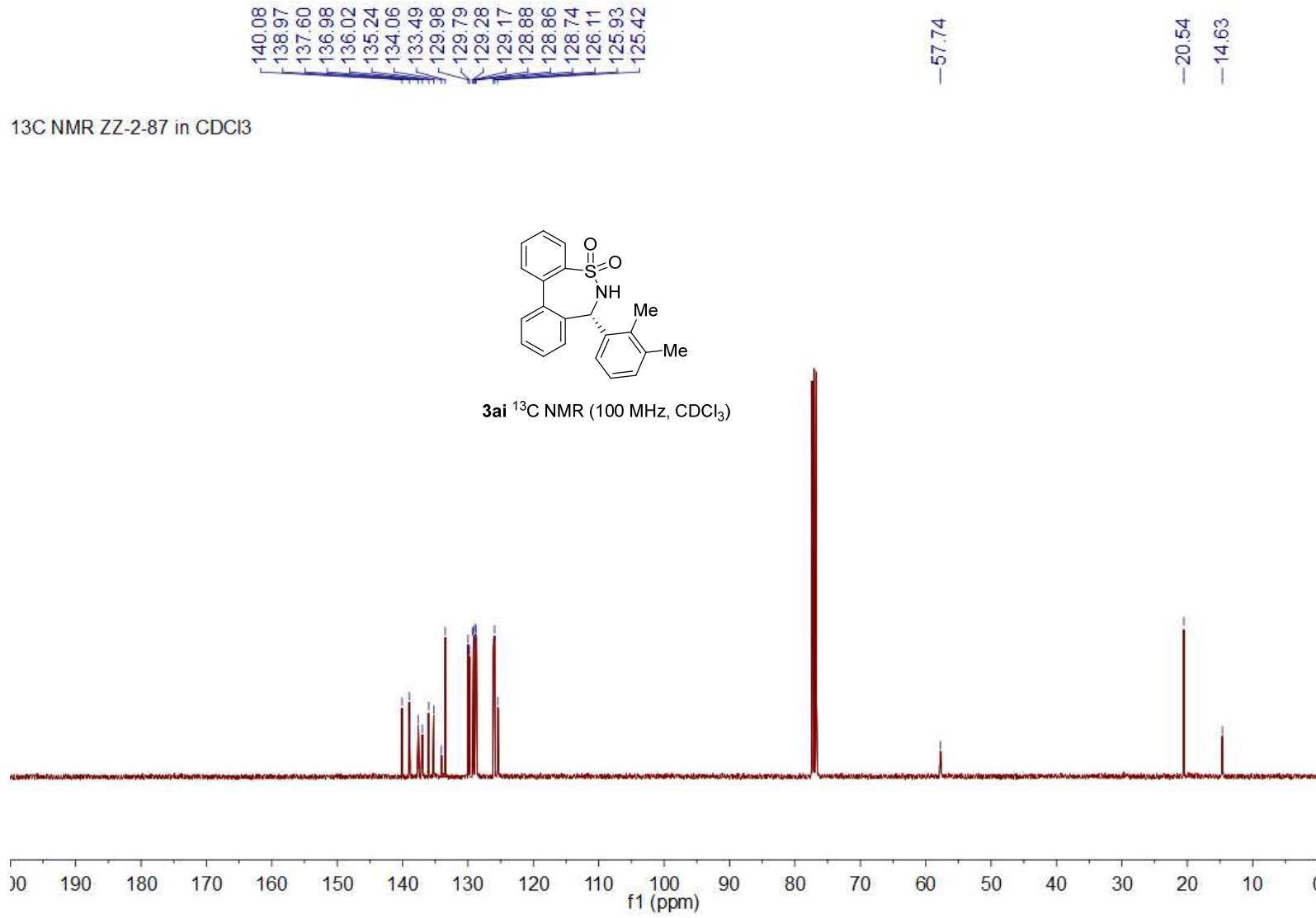
^{13}C NMR ZZ-2-61A in CDCl_3

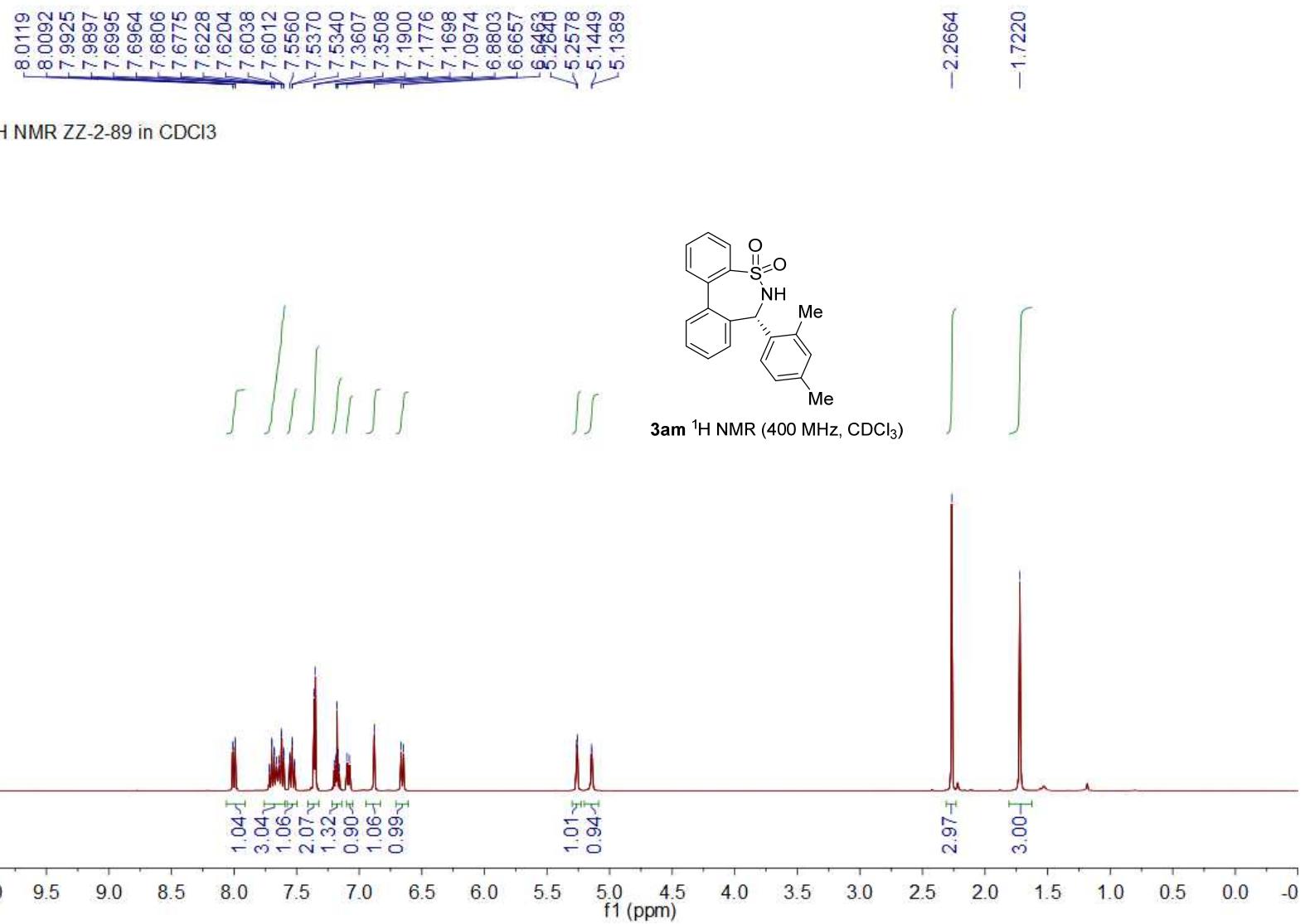


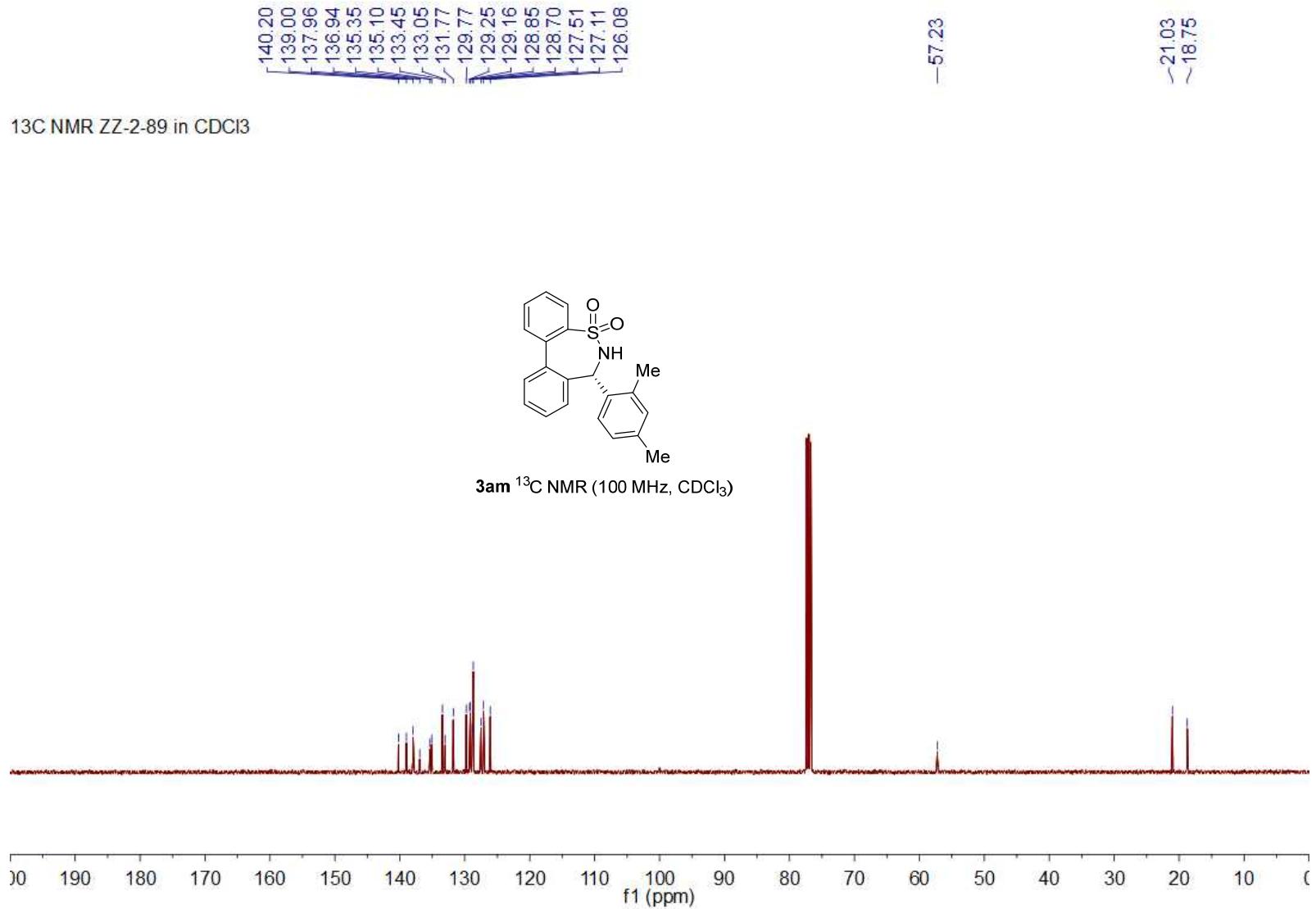
3ak ^{13}C NMR (100 MHz, CDCl_3)

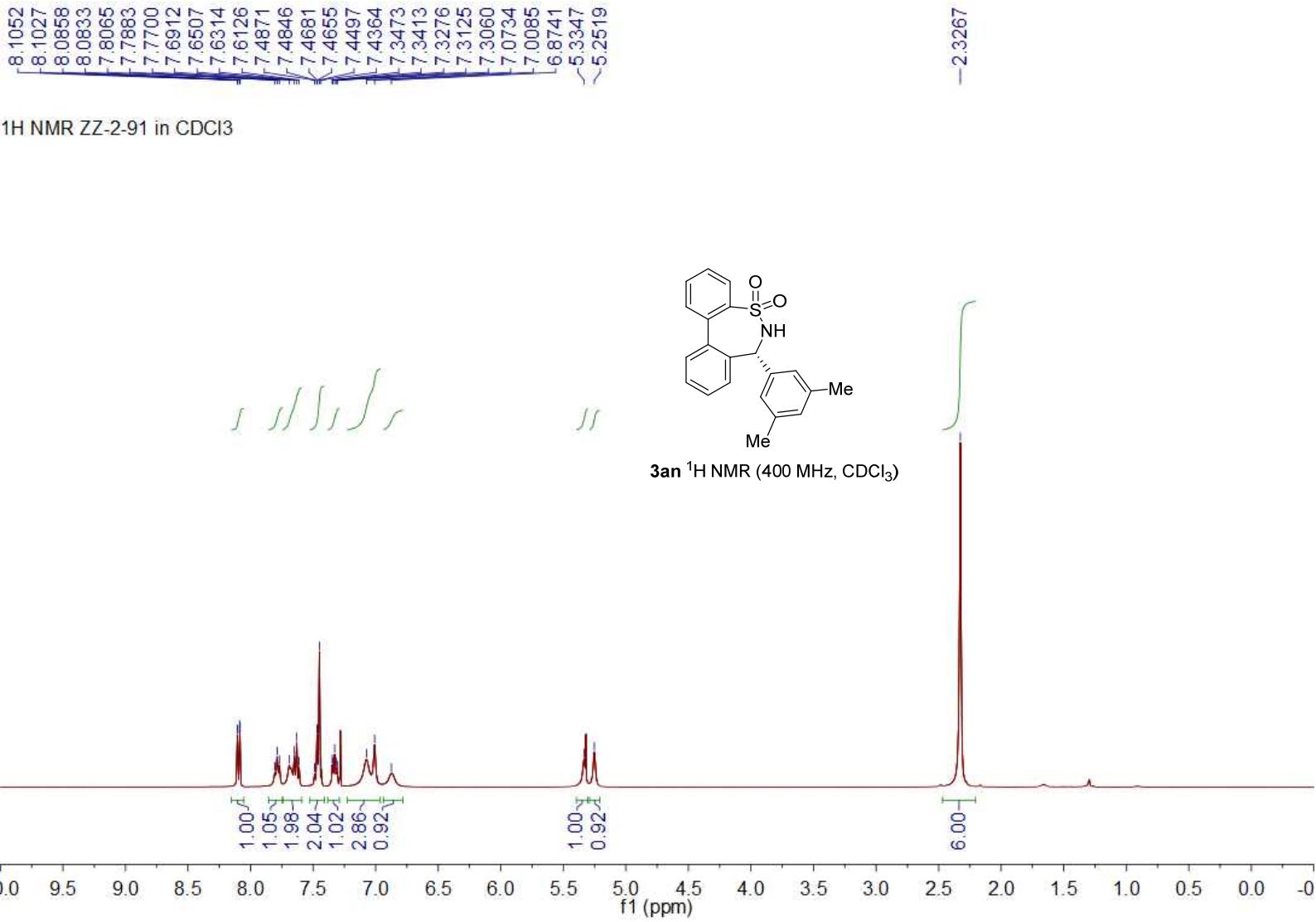


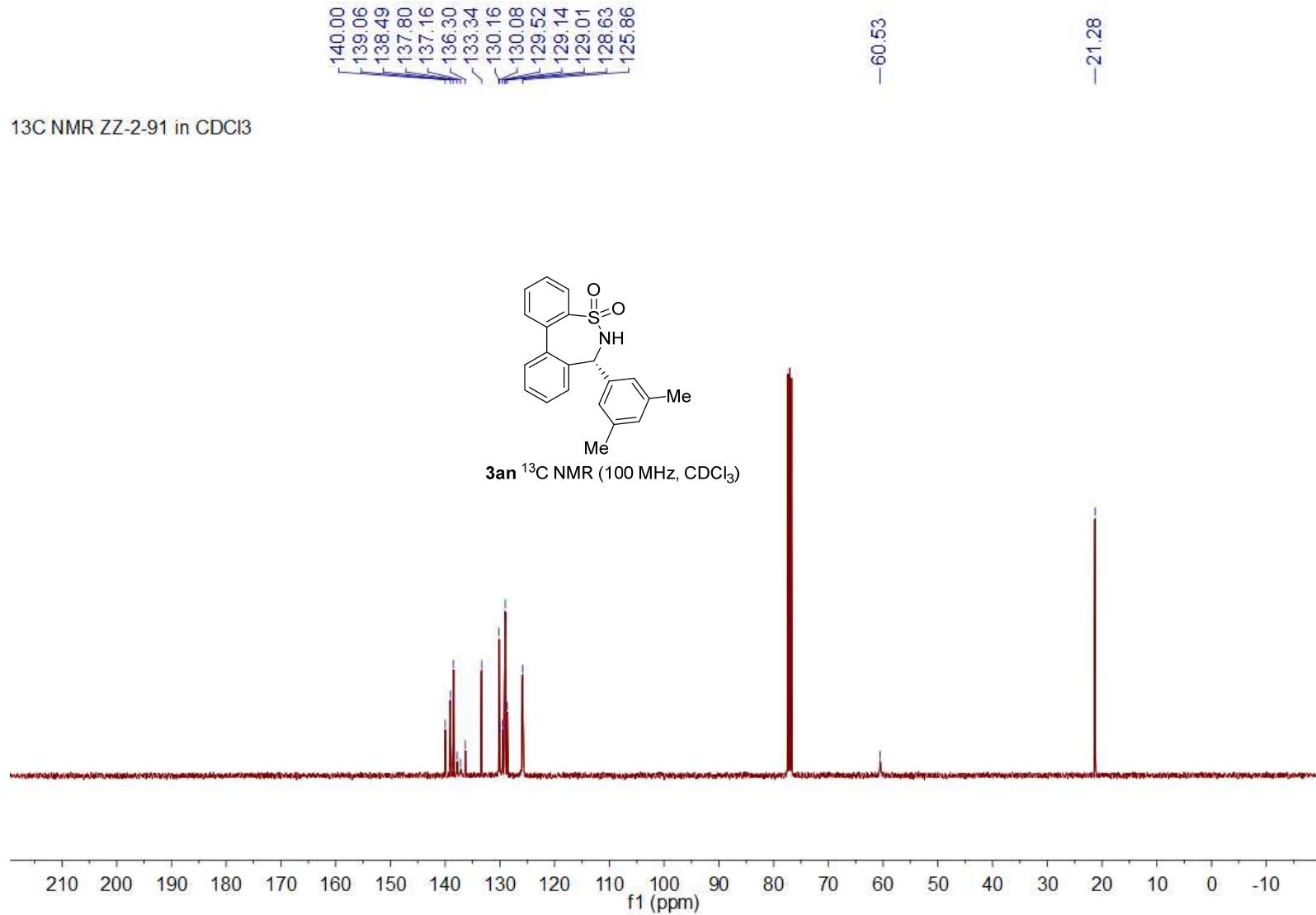


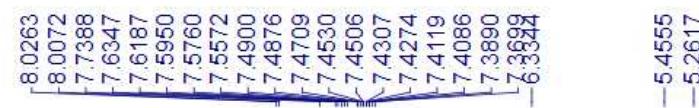




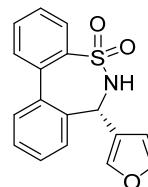




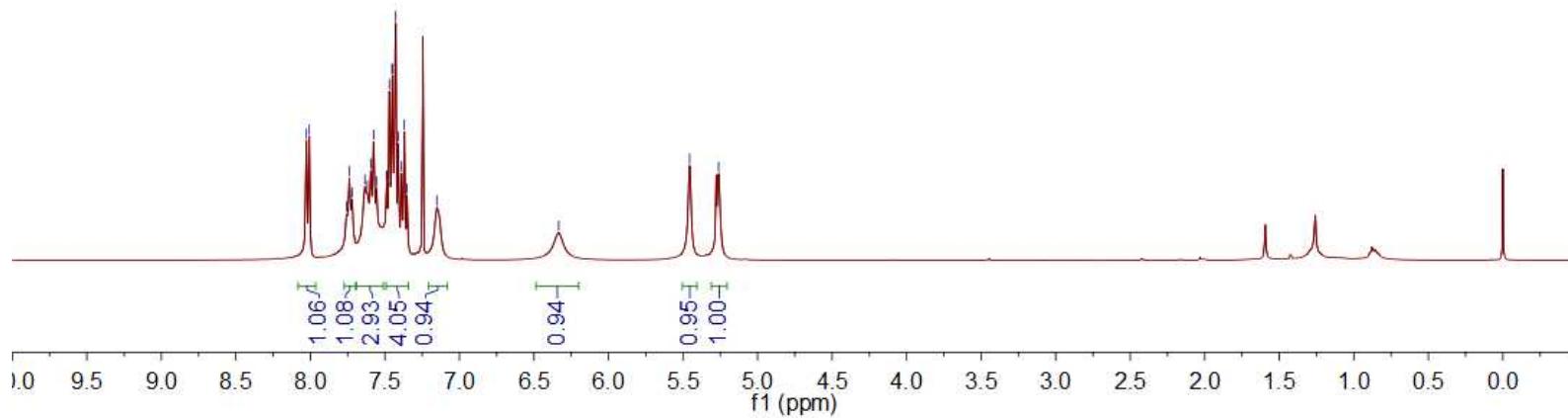


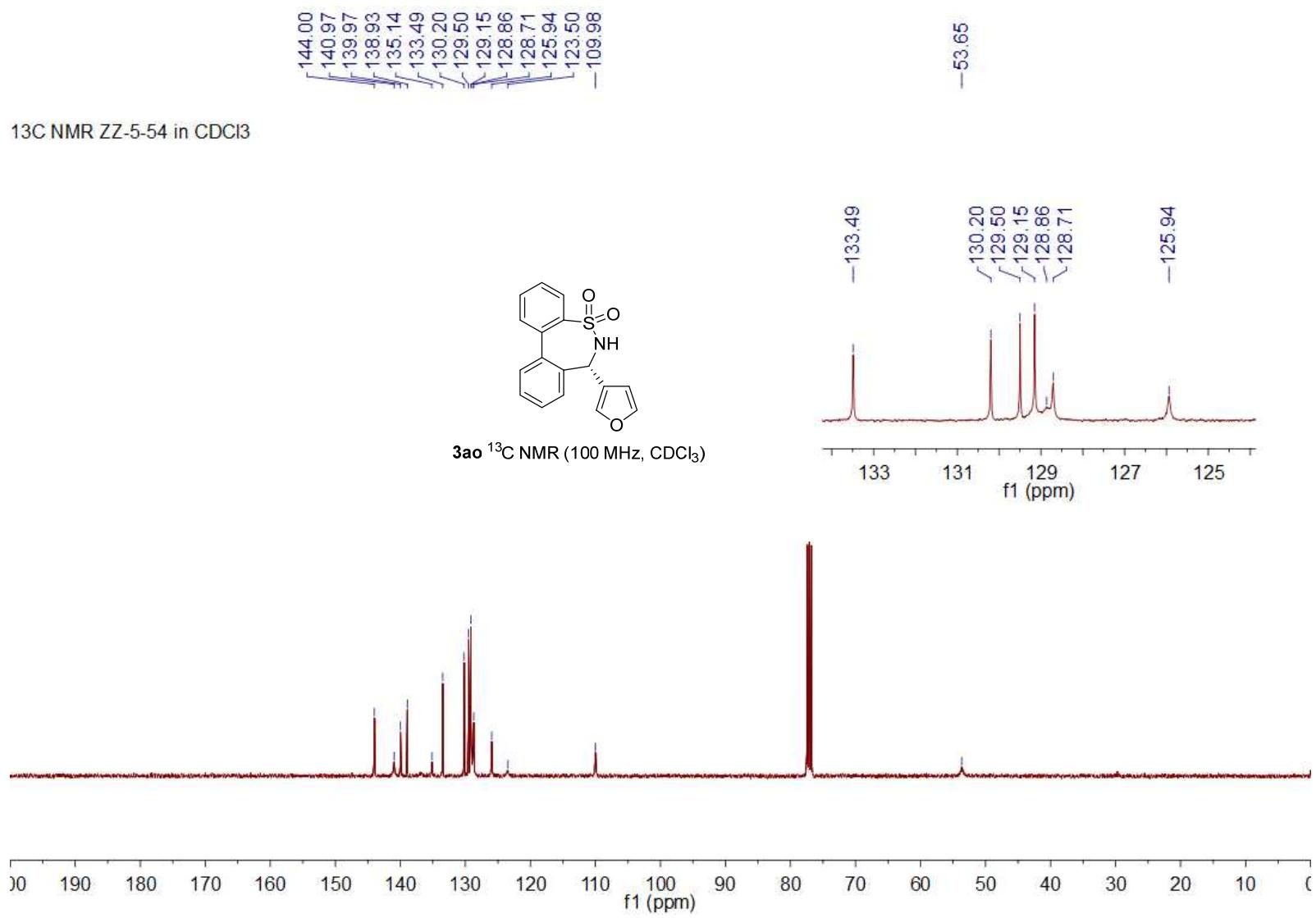


^1H NMR ZZ-5-54 in CDCl_3



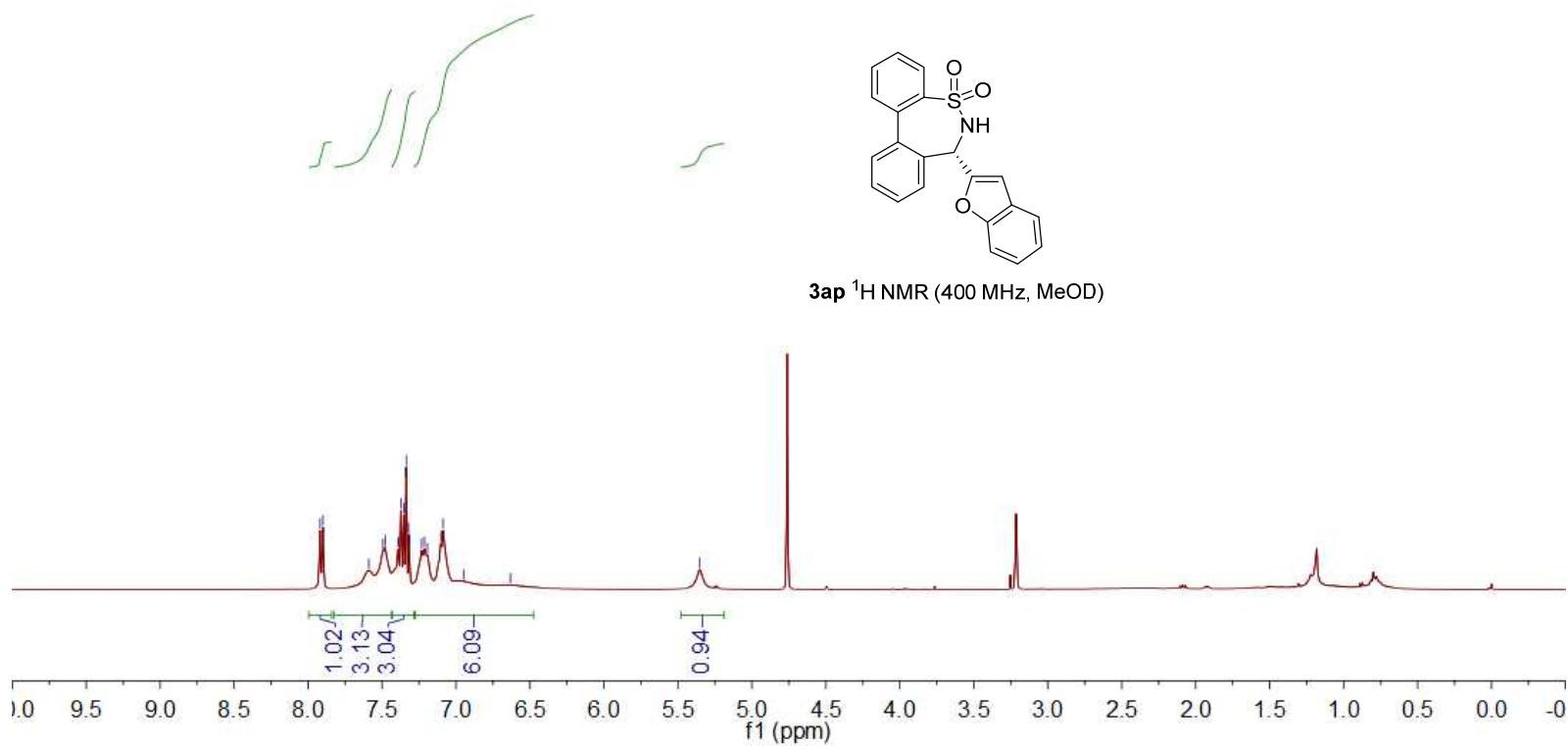
3ao ^1H NMR (400 MHz, CDCl_3)



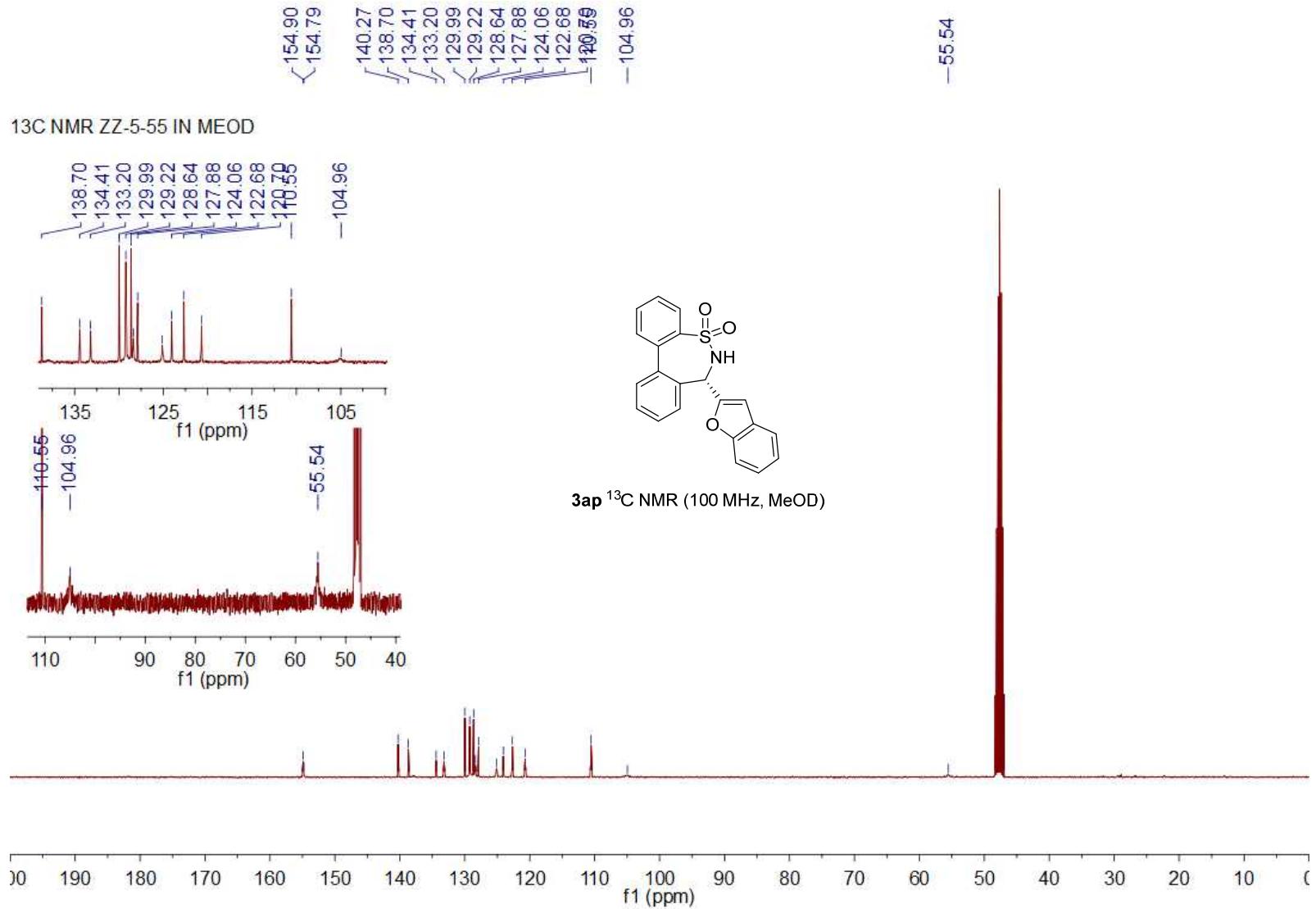


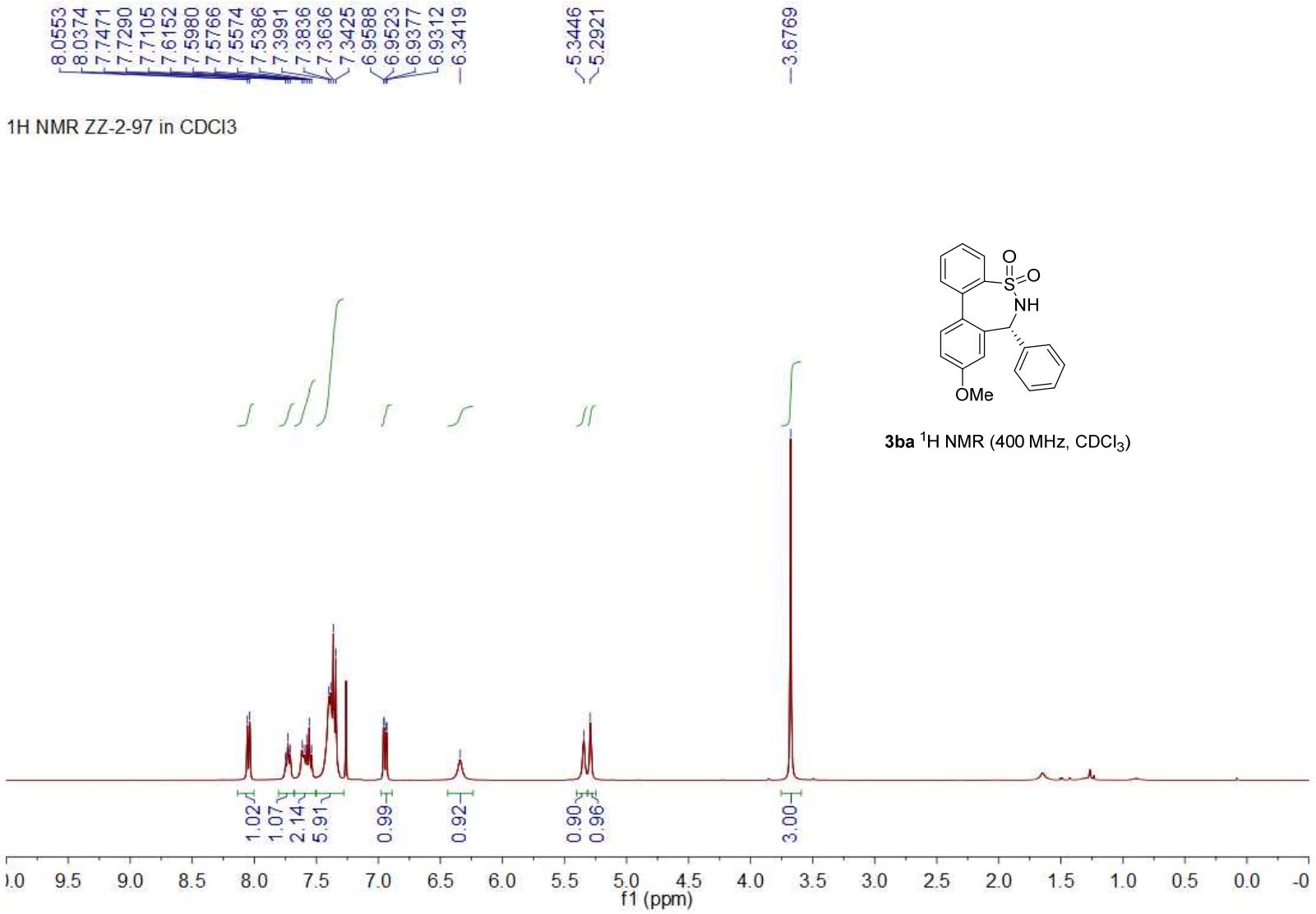


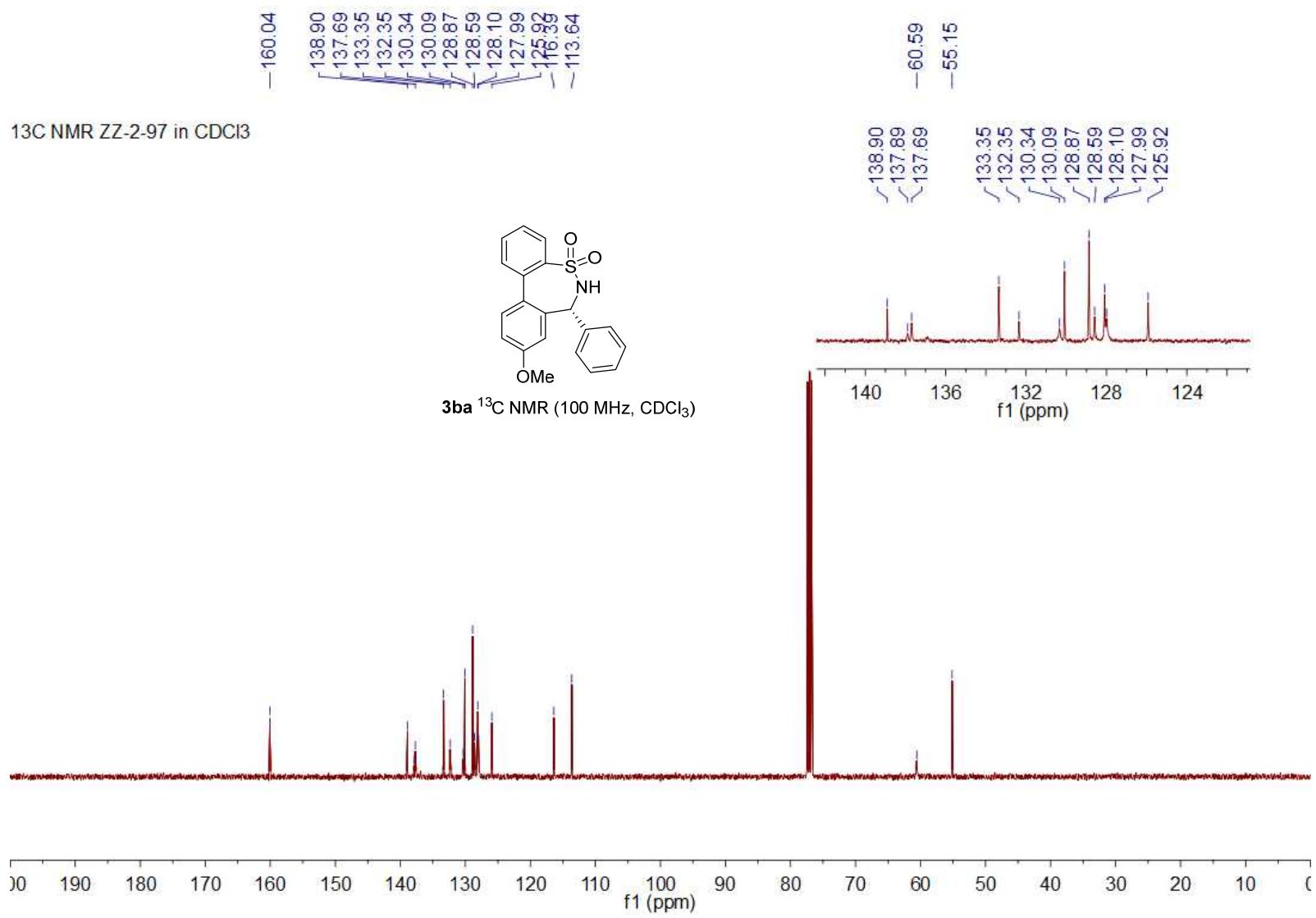
1H NMR ZZ-5-55 IN MeOD



3ap ¹H NMR (400 MHz, MeOD)



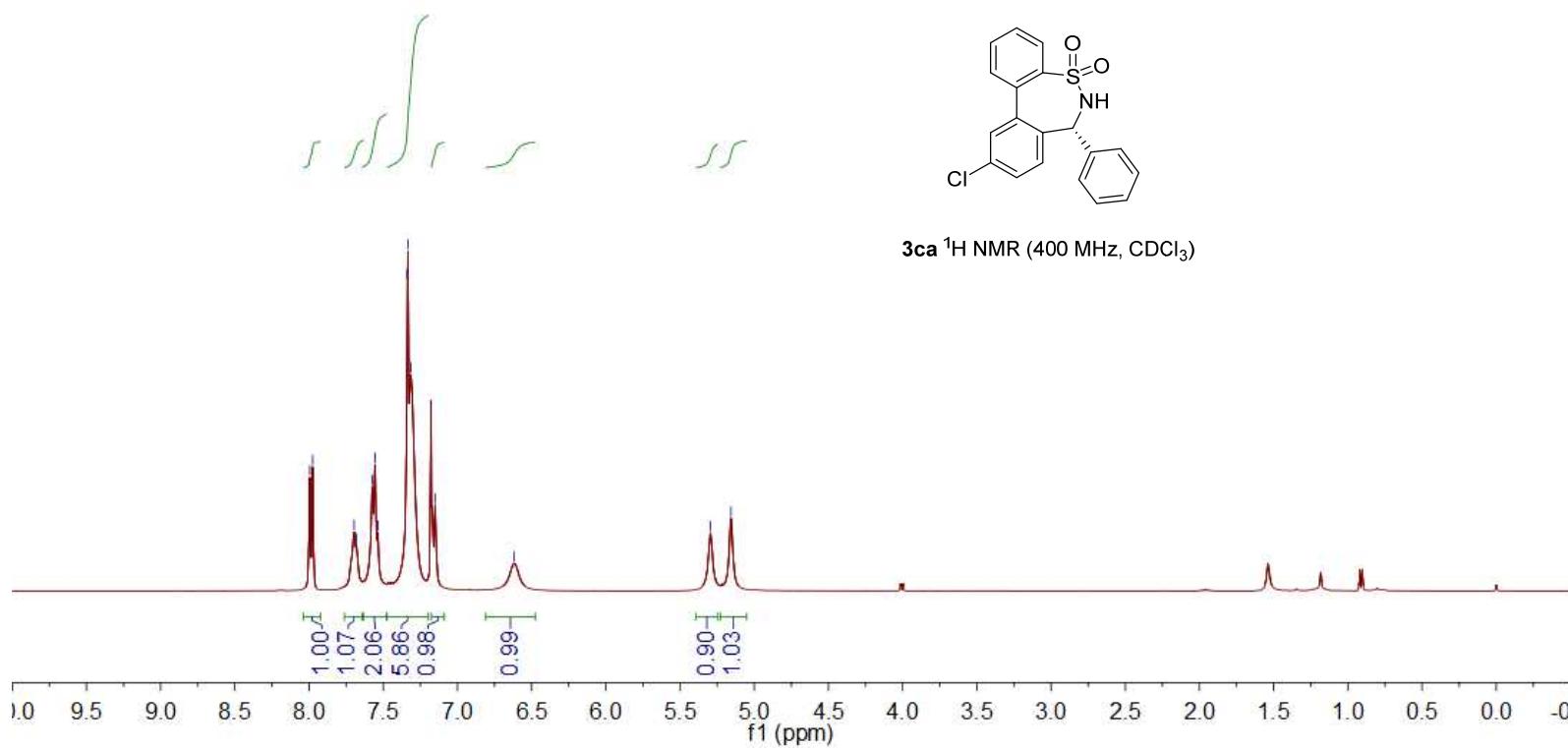


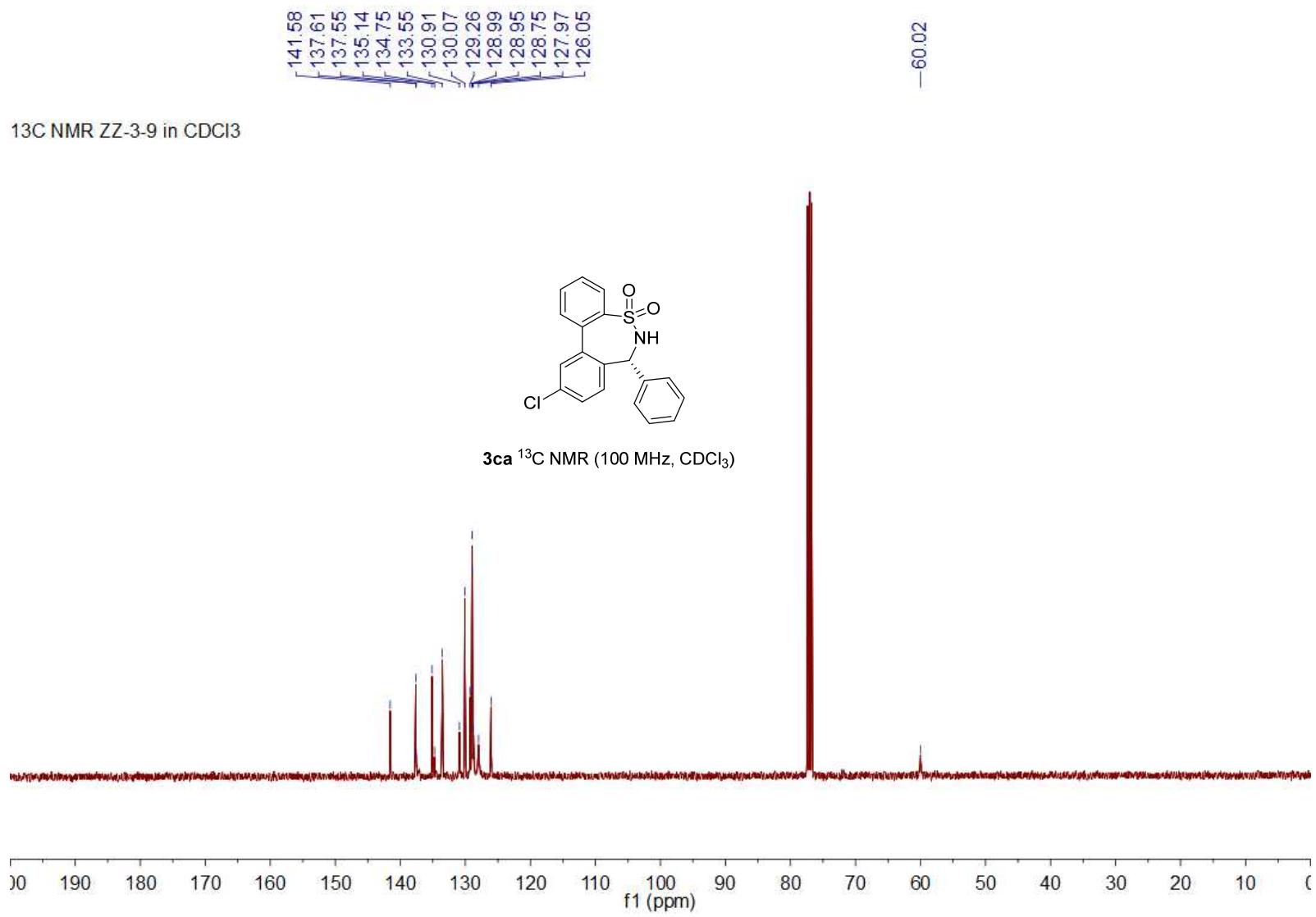


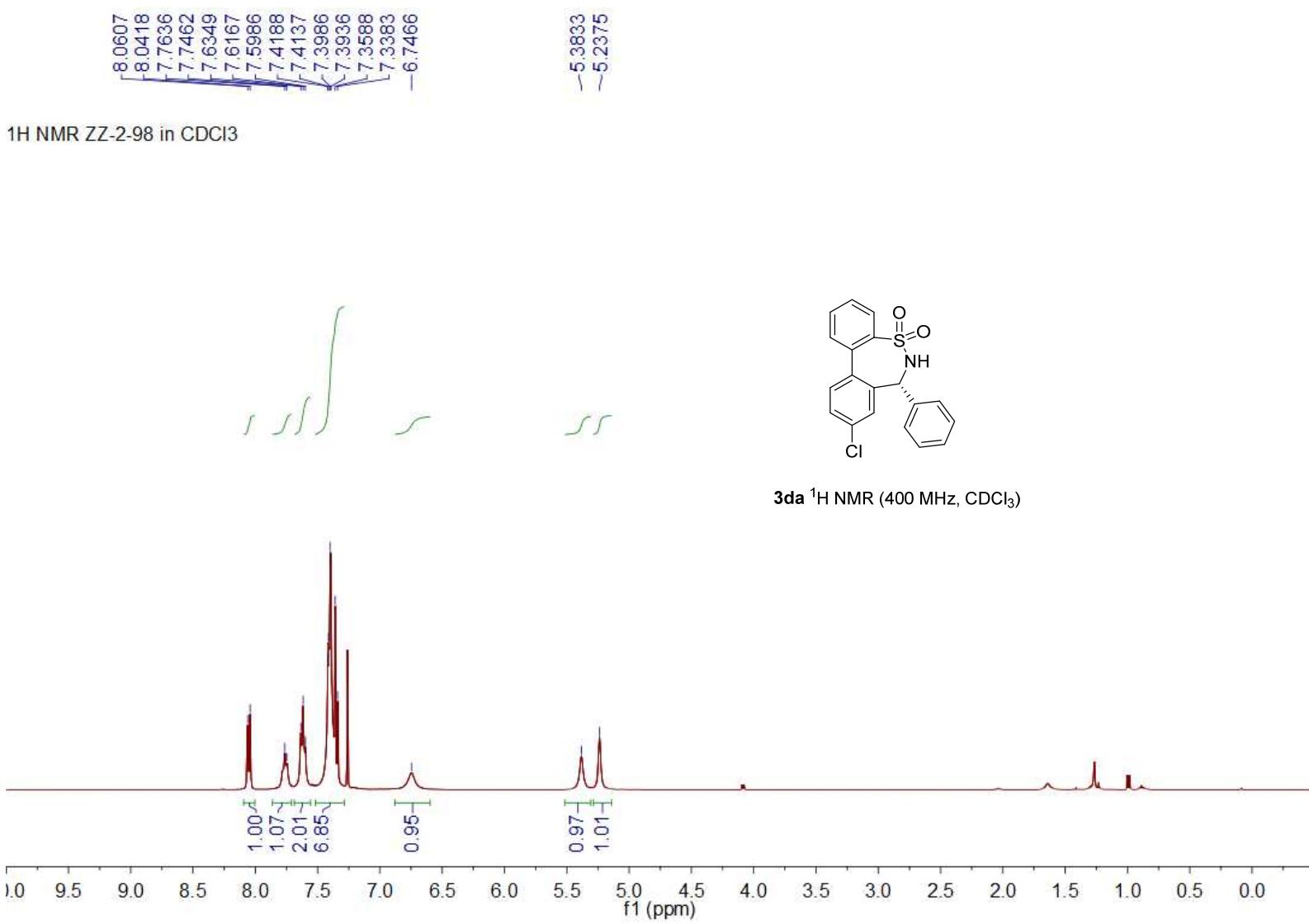
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7.9758
7.6960
7.6802
7.5737
7.5551
7.5365
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7.3339
7.3136
7.1505
-6.6171

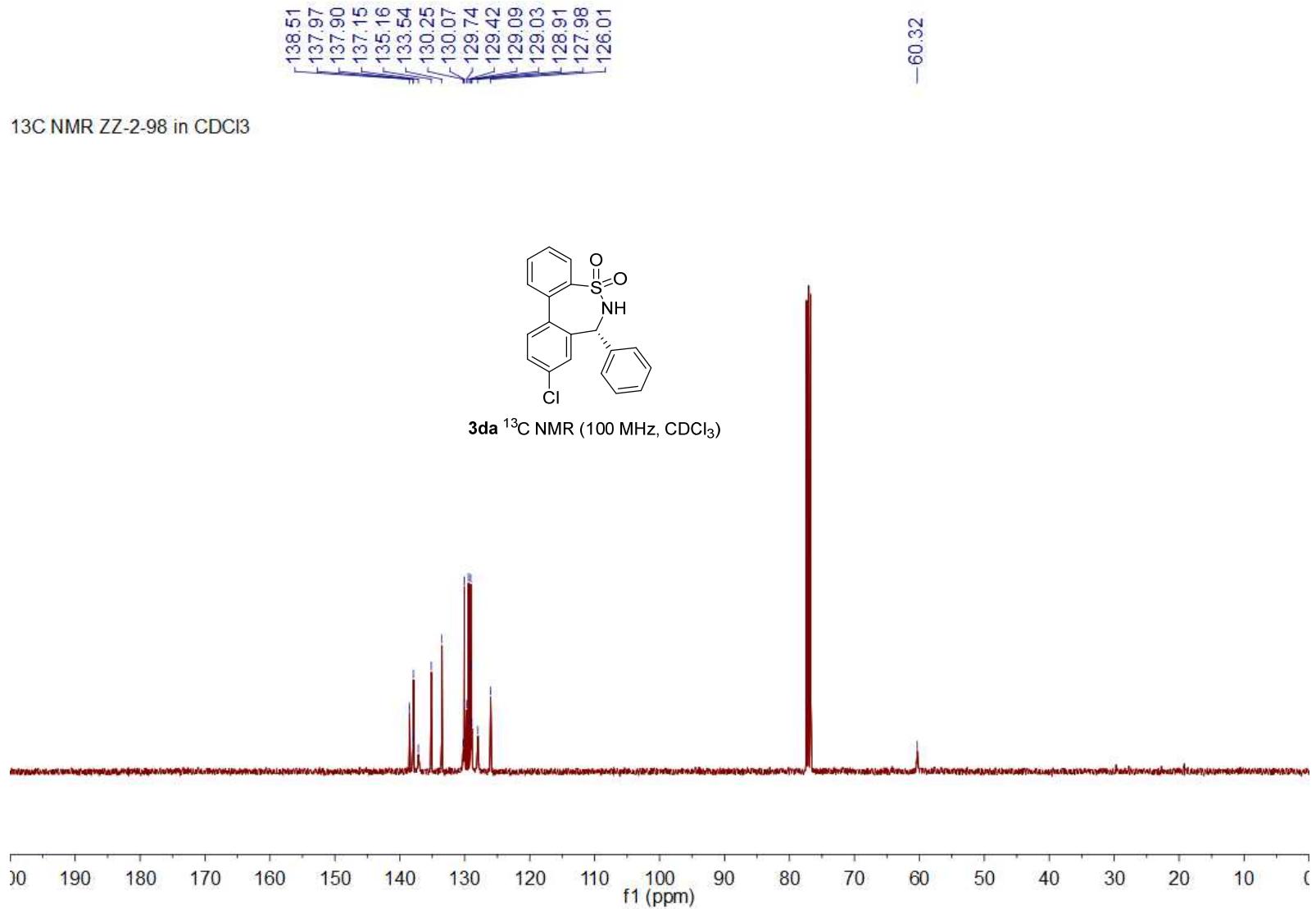
-5.2948
-5.1568

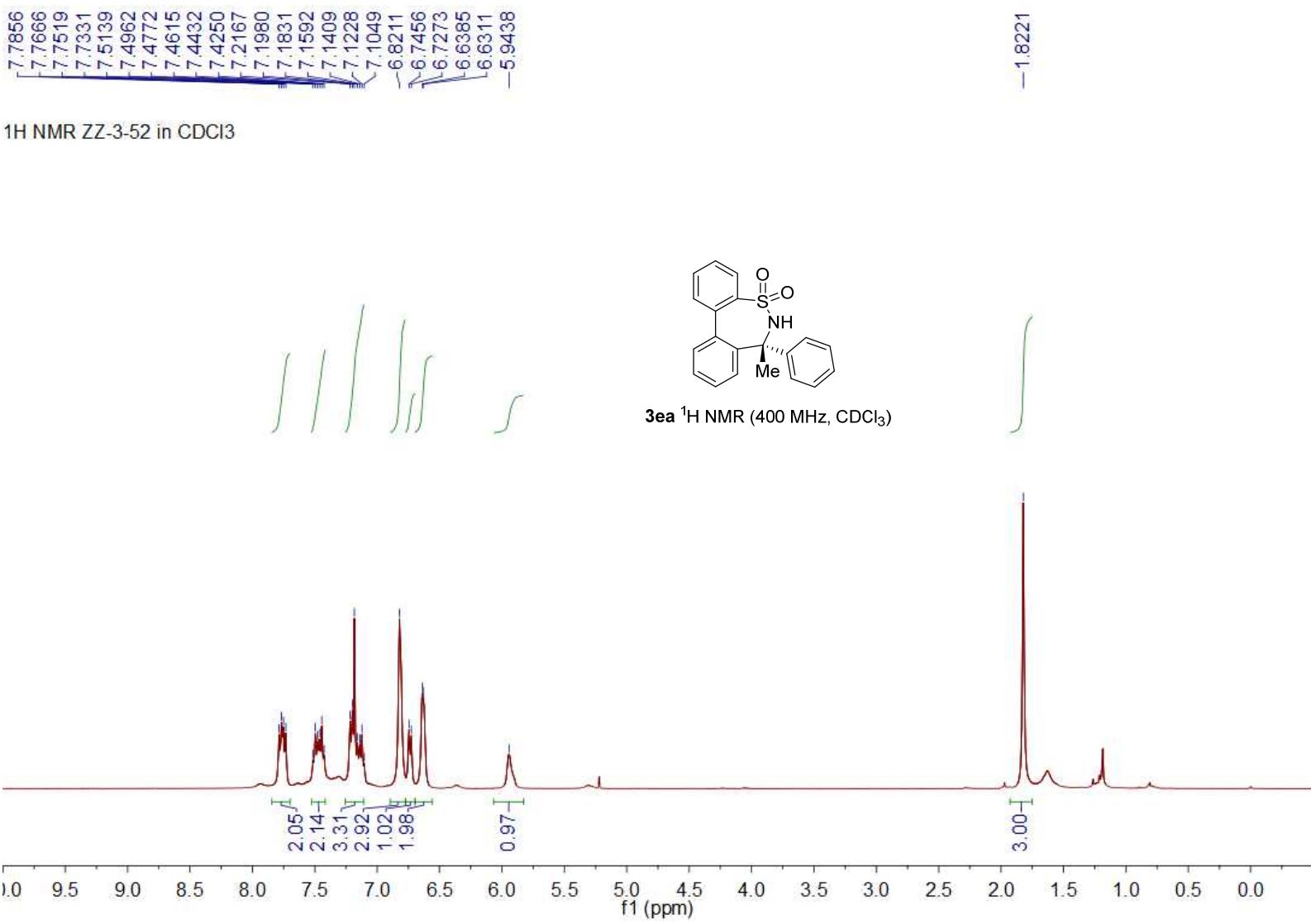
^1H NMR ZZ-3-9 in CDCl_3

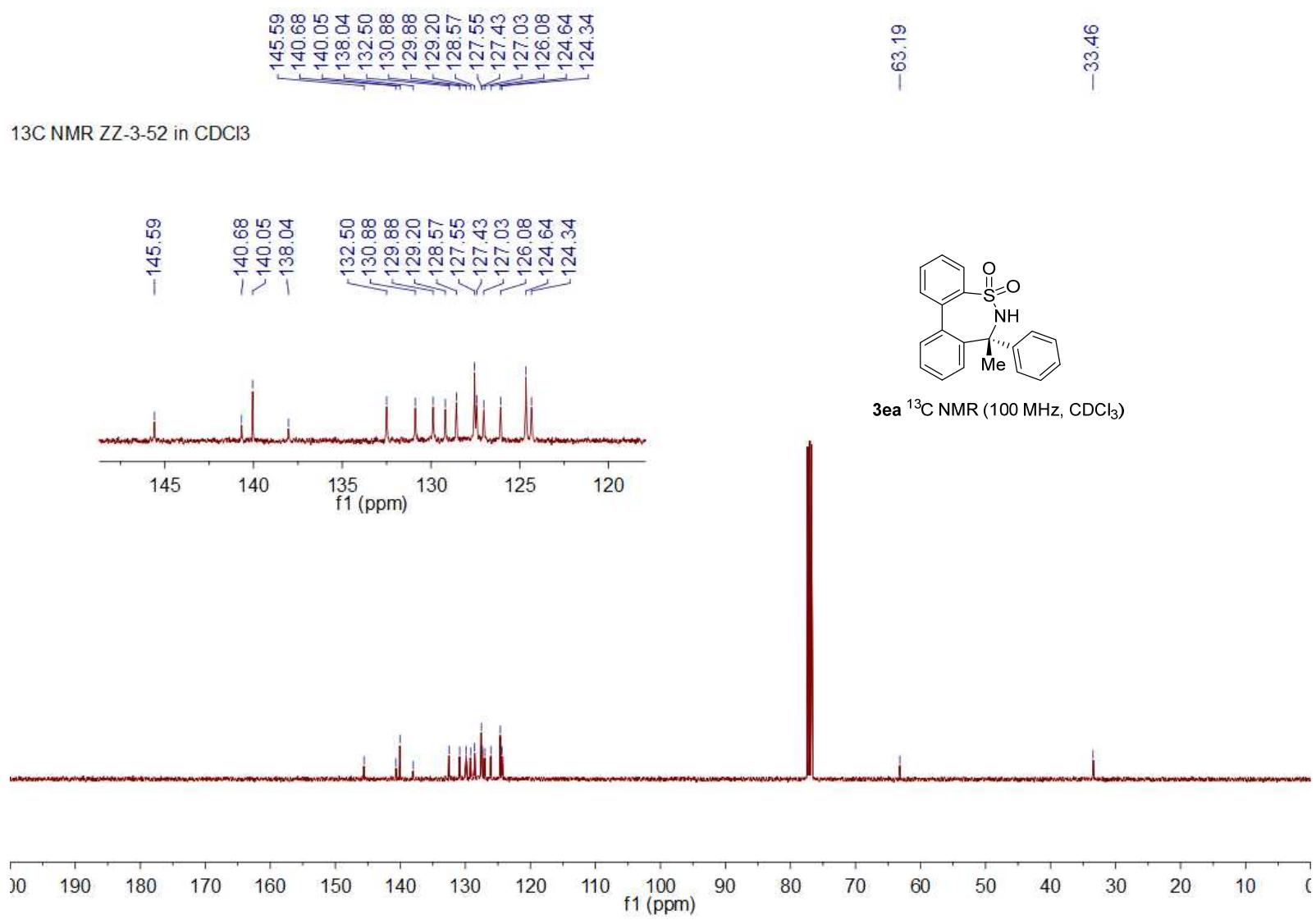


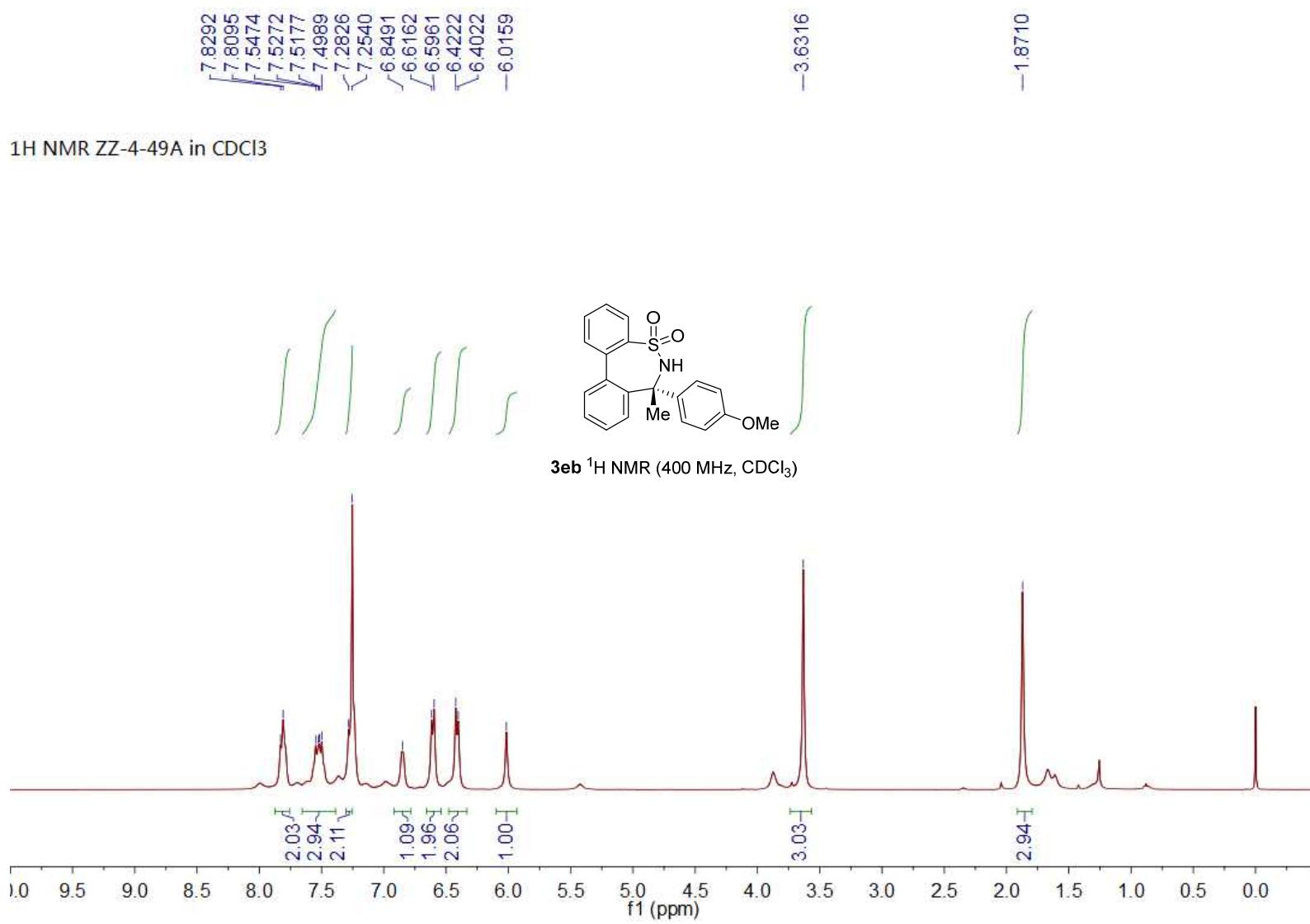


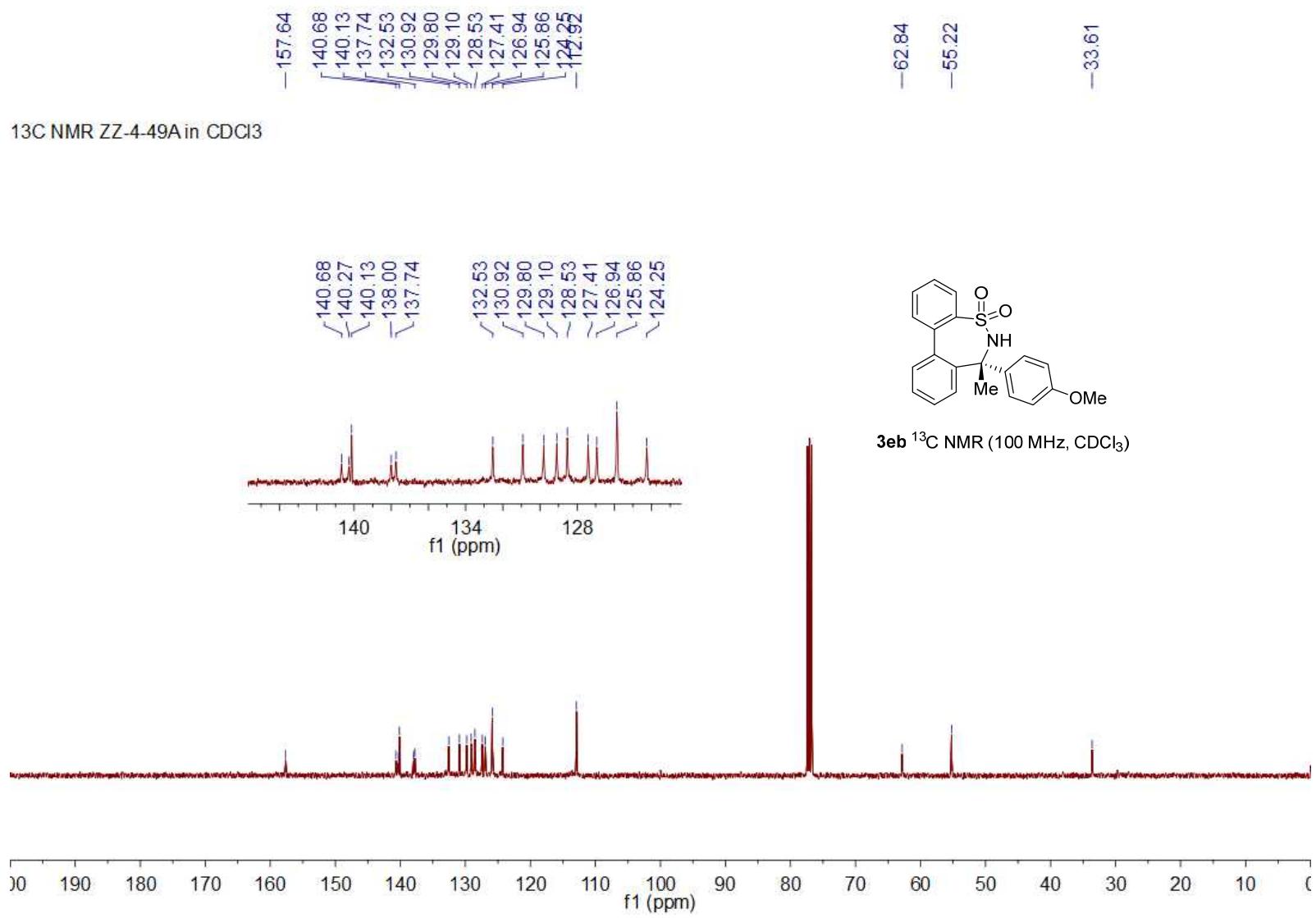






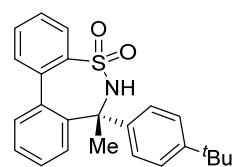




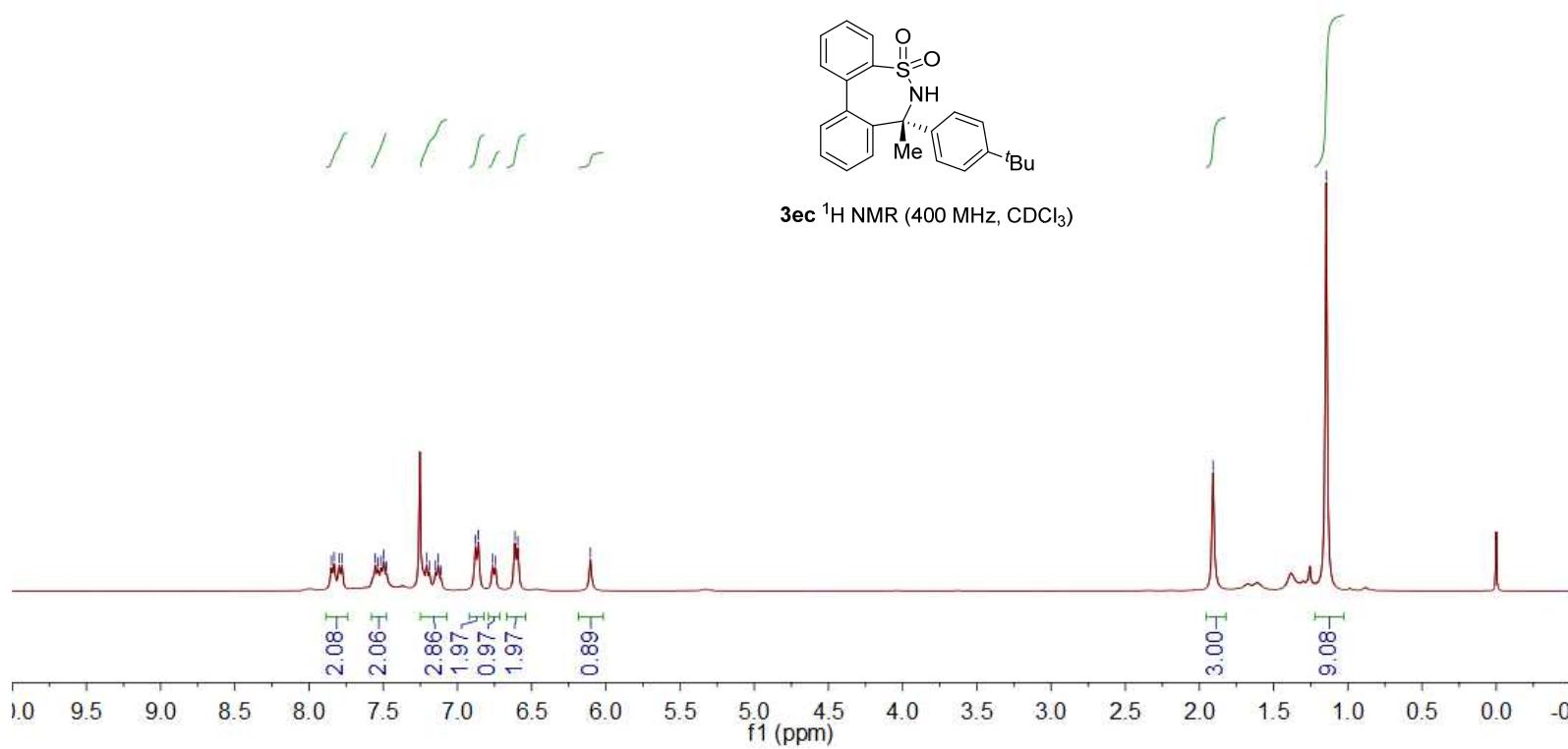


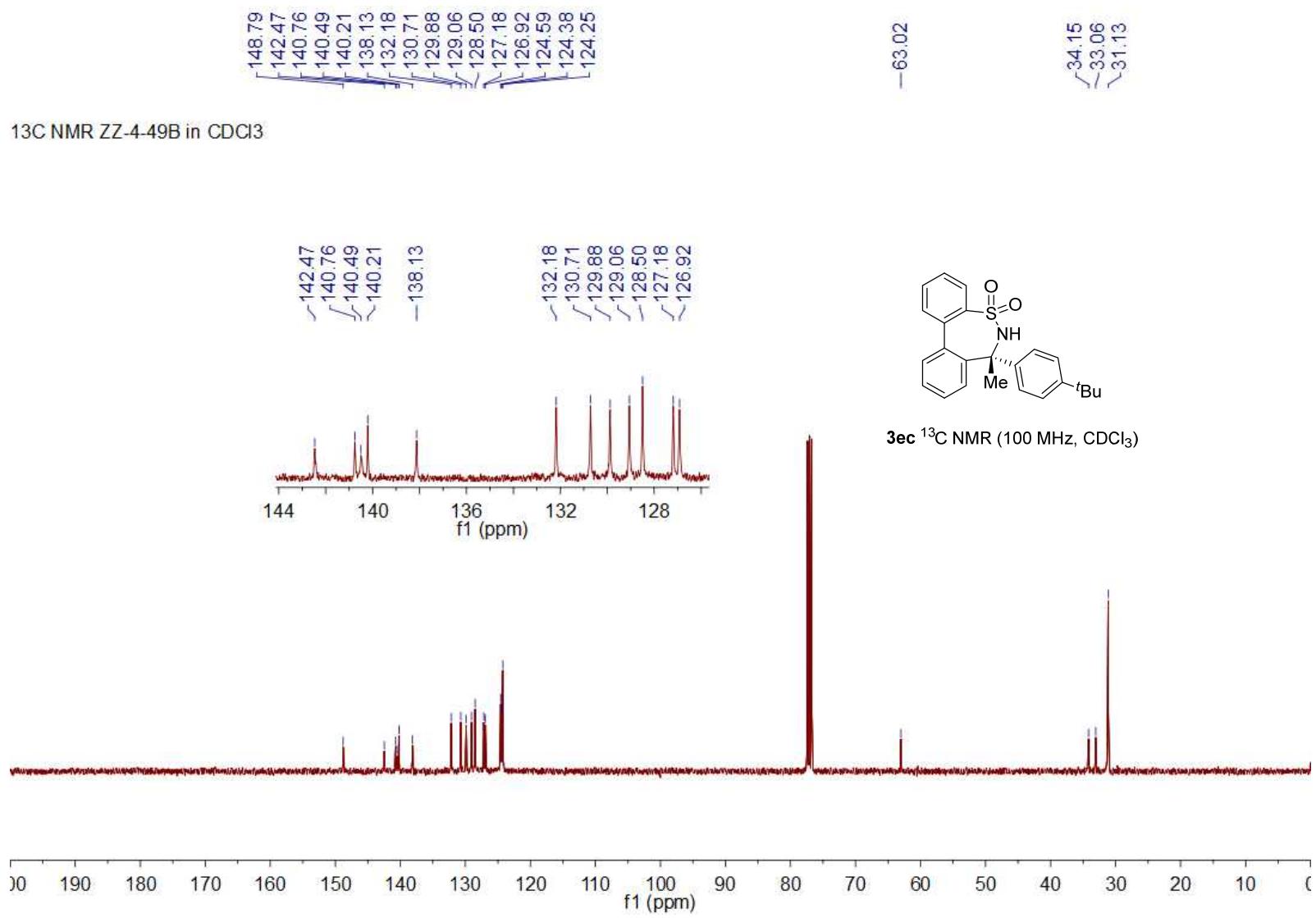
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7.1480
7.1297
7.1123
6.8791
6.8594
6.7627
6.7446
6.6130
6.5935
-6.1043

1H NMR ZZ-4-49B in CDCl₃



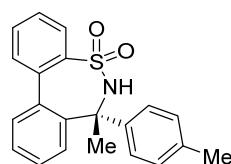
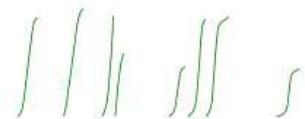
3ec ¹H NMR (400 MHz, CDCl₃)



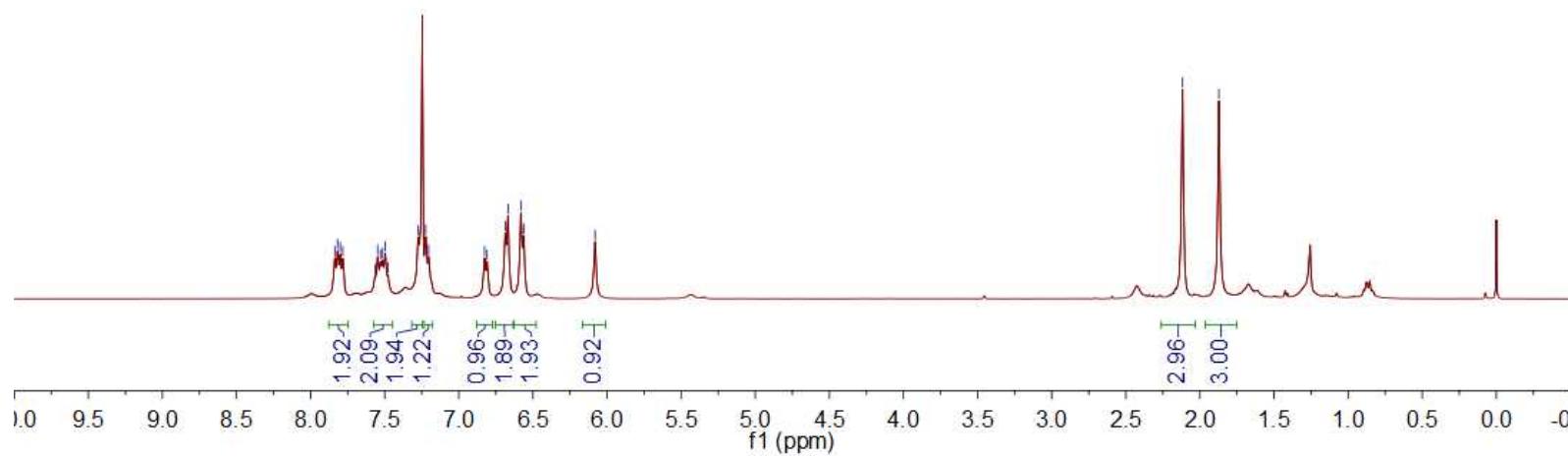


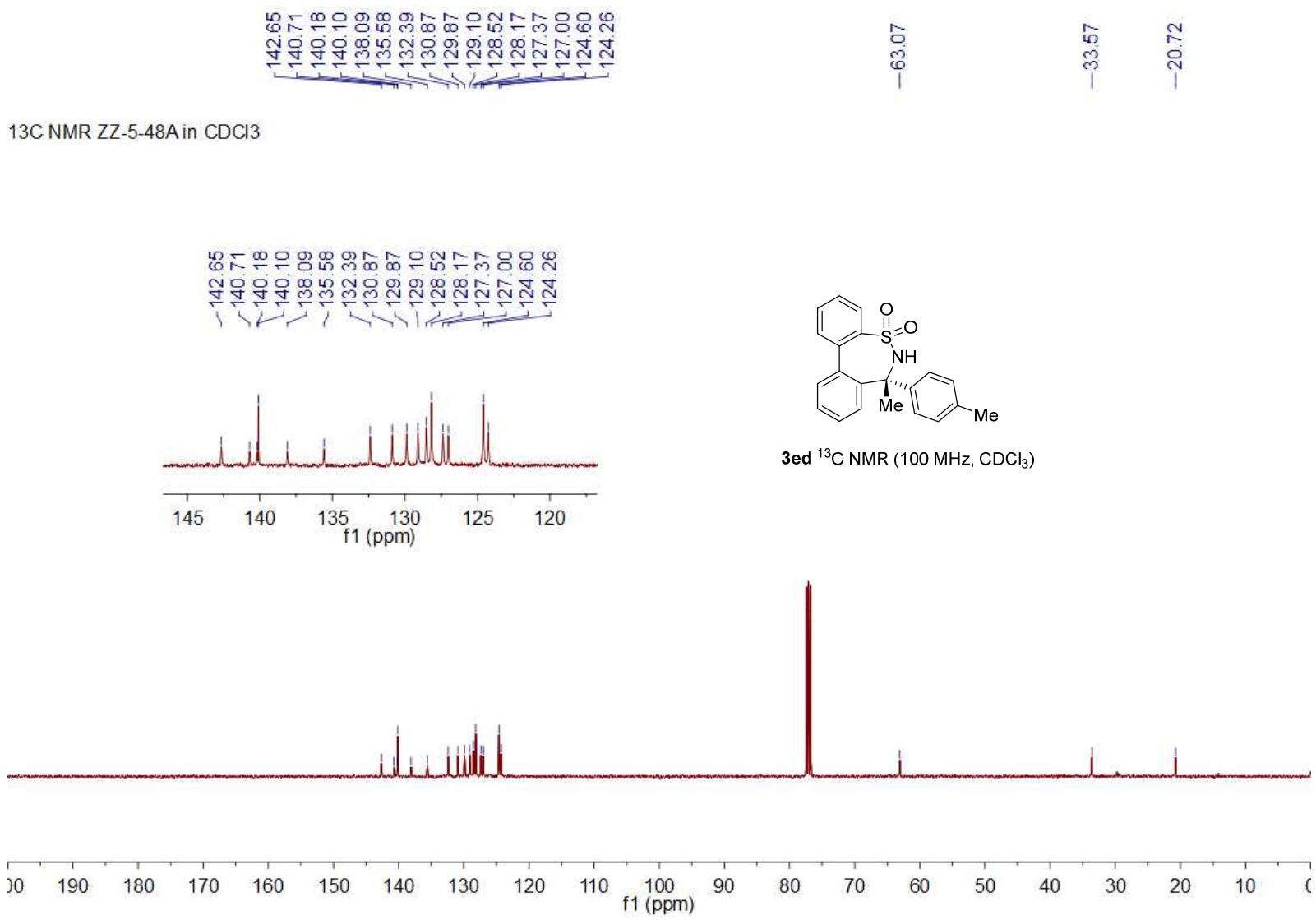


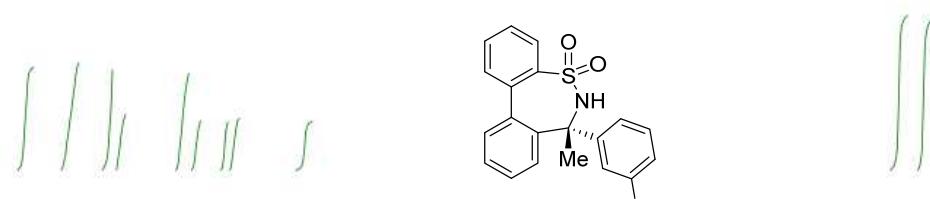
^1H NMR ZZ-5-48A in CDCl_3



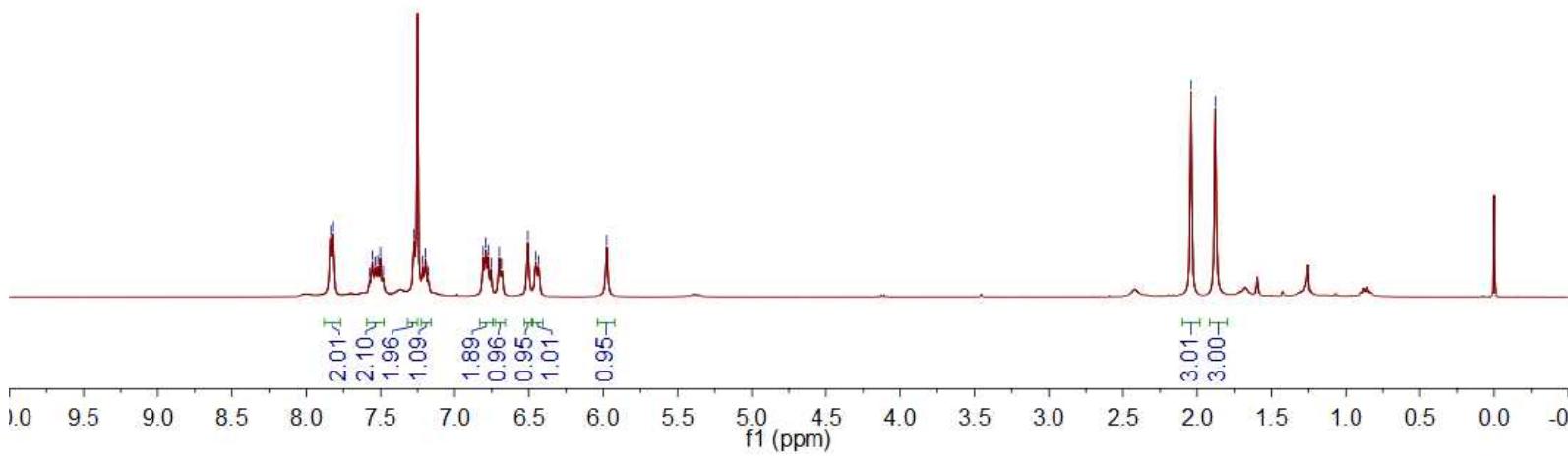
3ed ^1H NMR (400 MHz, CDCl_3)

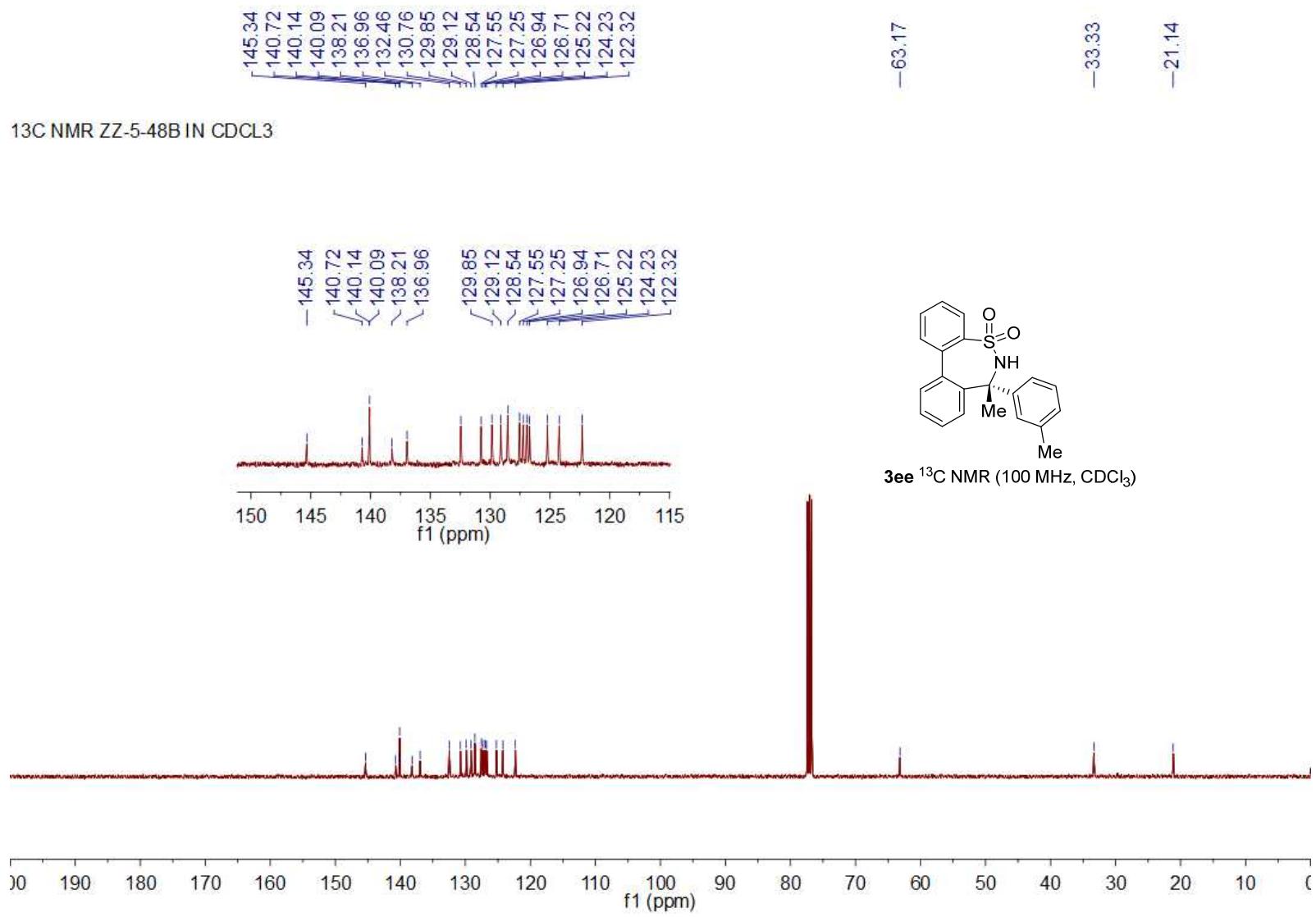






3ee ¹H NMR (400 MHz, CDCl₃)

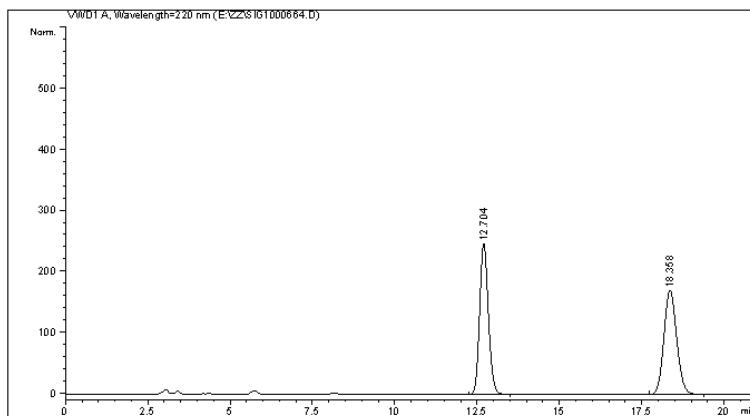




S70

Data File E:\ZZ\SIG1000664.D
Sample Name: zz-2-43(+-)

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Acq. Instrument : 仪器 1           Inj Volume : 2.000 µl
Injection Date : 3/17/2018 3:45:26 PM
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 3/17/2018 3:42:24 PM by HFE-258
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 8:58:26 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
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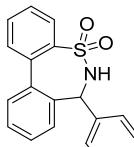
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Area Percent Report
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Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
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Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.2845	4548.92041	247.39166	49.7963
2	BB	0.4169	4586.14551	170.46342	50.2037

Totals : 9135.06592 417.85509



(+)-3aa

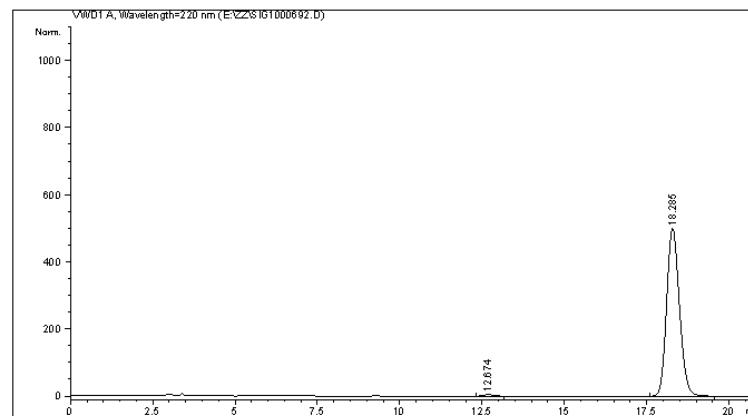
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*** End of Report ***
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Instrument 1 8/19/2018 8:58:35 PM

Page 1 of 1

Data File E:\ZZ\SIG1000692.D
Sample Name: zz-2-50

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Acq. Instrument : 仪器 1           Inj Volume : 2.000 µl
Injection Date : 3/23/2018 9:39:49 AM
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 3/23/2018 9:39:23 AM by HFE-258
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:00:44 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



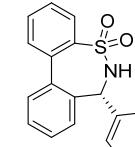
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.2936	85.69107	4.49208	0.6297
2	BB	0.4209	1.35222e4	499.44681	99.3703

Totals : 1.36079e4 503.93888



(+)-3aa

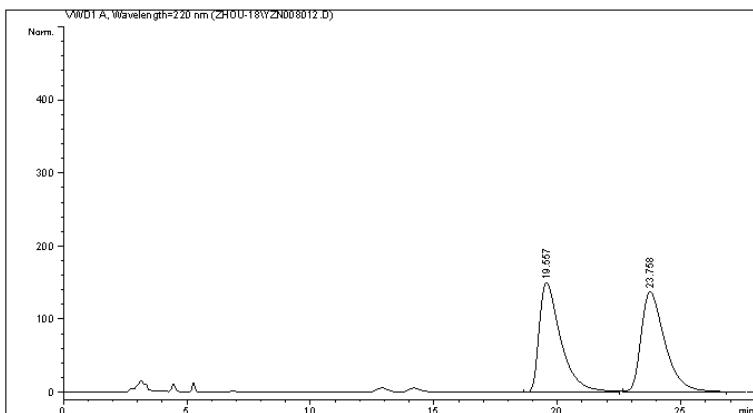
```
=====
*** End of Report ***
```

Instrument 1 8/19/2018 9:00:52 PM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008012.D
Sample Name: zz-2-51A(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/24/2018 11:02:07 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/24/2018 10:55:11 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 10:55:20 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



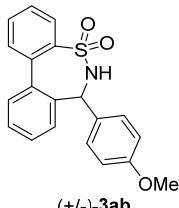
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 19.557	BB	0.9050	9129.18555	150.02177	49.9948
2 23.758	BB	1.0033	9131.06984	136.99435	50.0052

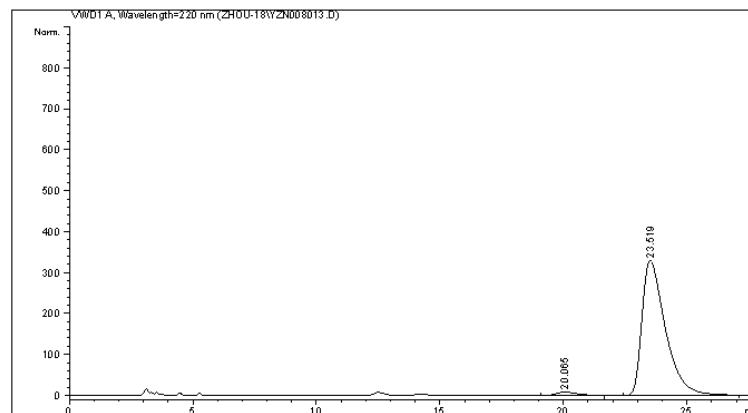
Totals : 1.82603e4 287.01613



*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008013.D
Sample Name: zz-2-51A

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/24/2018 11:43:51 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/24/2018 11:32:49 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 10:57:49 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



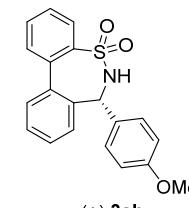
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 20.065	BB	0.8964	512.32758	8.40096	2.3077
2 23.519	BB	0.9959	2.16868e4	329.13538	97.6923

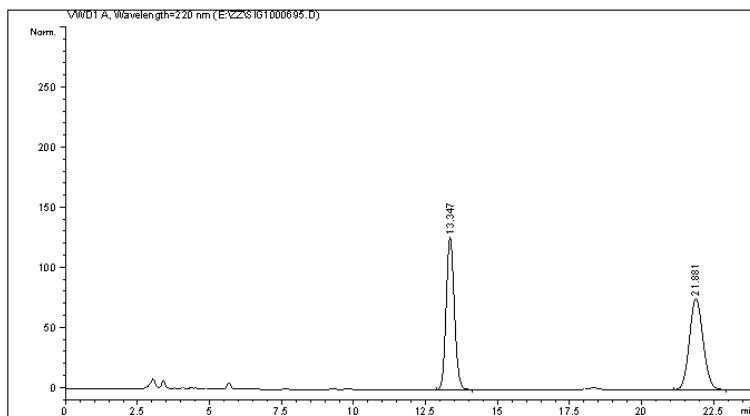
Totals : 2.22011e4 337.53634



*** End of Report ***

Data File E:\ZZ\SIG1000695.D
Sample Name: zz-2-51B(+-)

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 虹 1
Injection Date : 3/24/2018 10:07:20 AM
Inj Volume : 2.000 µl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 3/24/2018 9:26:44 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:05:09 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



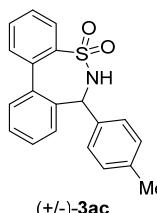
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

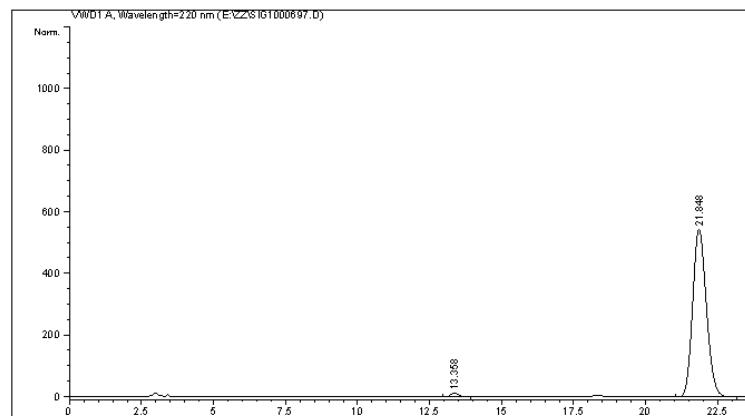
Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.3068	2504.27979	126.57328	49.9842
2	BB	0.5136	2505.86230	75.81239	50.0158

Totals : 5010.14209 202.38567



Data File E:\ZZ\SIG1000697.D
Sample Name: zz-2-51B

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 虹 1
Injection Date : 3/24/2018 11:06:34 AM
Inj Volume : 2.000 µl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 3/24/2018 11:06:08 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:07:36 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



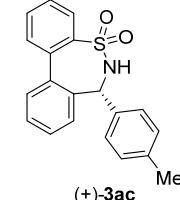
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

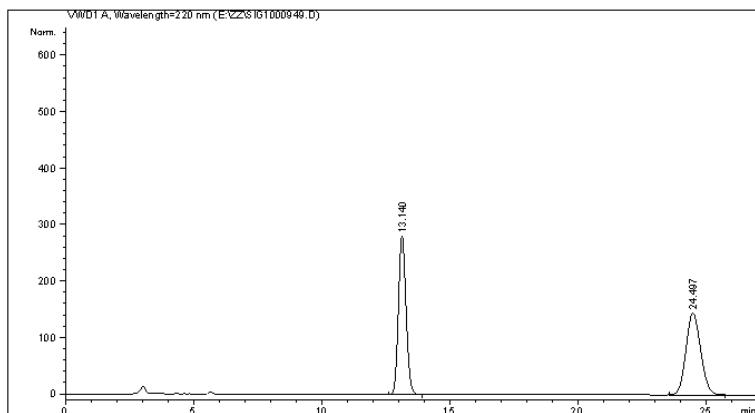
Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.3044	243.37157	12.37893	1.3269
2	BB	0.5154	1.80974e4	544.99622	98.6731

Totals : 1.83407e4 557.37514



Data File E:\ZZ\SIG1000949.D
Sample Name: zz-2-94(+-)

```
=====
Acq. Operator : zz                         Location : Vial 91
Acq. Instrument : 仪器 1                   Location : Vial 91
Injection Date : 5/9/2018 8:48:41 AM        Inj Volume : 2.000 µl
=====
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed  : 5/9/2018 8:39:38 AM by zz
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed  : 8/19/2018 9:32:04 PM
(modified after loading)
Sample Info   : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



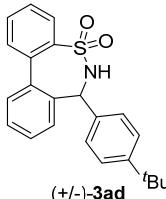
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

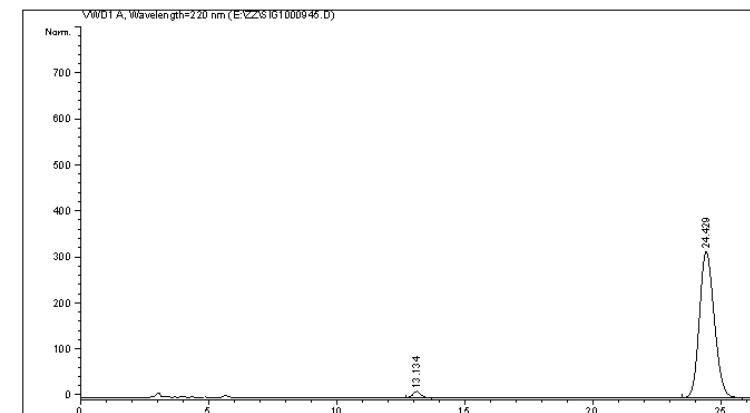
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.140	BB	0.3209	5793.94238	280.55783	50.0161
2 24.497	BB	0.6214	5790.20215	145.13426	49.9839

Totals : 1.1584e4 425.69209



Data File E:\ZZ\SIG1000945.D
Sample Name: zz-2-94

```
=====
Acq. Operator : zz                         Location : Vial 91
Acq. Instrument : 仪器 1                   Location : Vial 91
Injection Date : 5/8/2018 10:21:44 AM        Inj Volume : 2.000 µl
=====
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed  : 5/8/2018 10:18:22 AM by zz
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed  : 8/19/2018 9:33:04 PM
(modified after loading)
Sample Info   : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



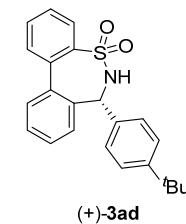
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

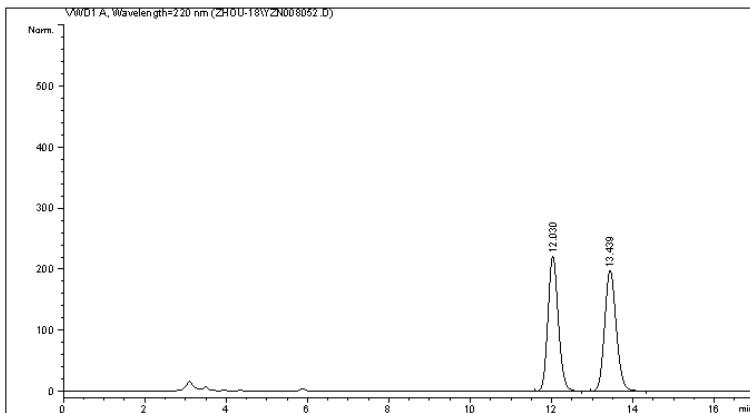
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.134	BB	0.3148	265.59509	13.08331	2.0413
2 24.429	BB	0.6243	1.27458e4	317.54691	97.9587

Totals : 1.30114e4 330.63021



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008052.D
Sample Name: zz-2-55A(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/29/2018 7:45:17 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/29/2018 7:42:46 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 10:59:31 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



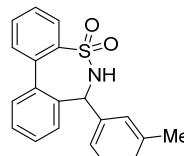
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 12.030	BB	0.2729	3892.30371	220.48262	49.8985
2 13.439	BB	0.3069	3908.13745	197.20352	50.1015

Totals : 7800.44116 417.68614



(-)-3ae

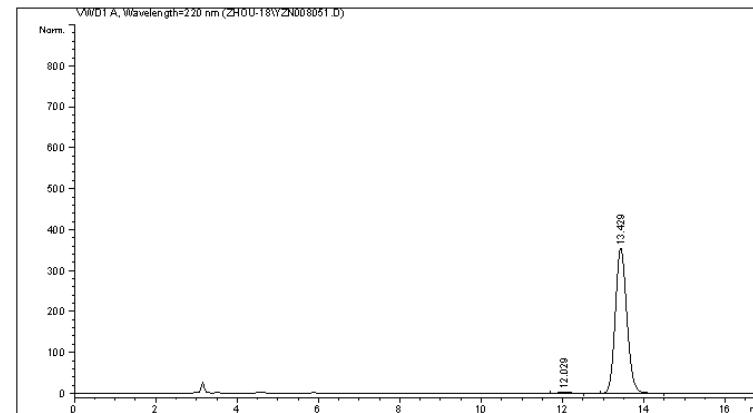
*** End of Report ***

Instrument 1 8/20/2018 10:59:39 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008051.D
Sample Name: zz-2-55A

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/29/2018 7:21:24 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/29/2018 7:19:18 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:01:05 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



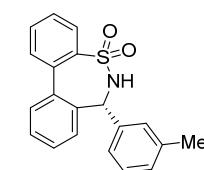
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 12.029	BB	0.2768	57.29067	3.18593	0.8163
2 13.429	BB	0.3037	6961.36963	353.99603	99.1837

Totals : 7018.66030 357.18196



(+)-3ae

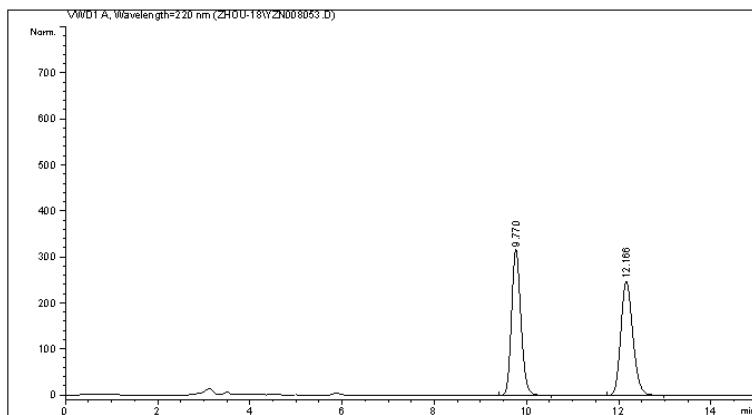
*** End of Report ***

Instrument 1 8/20/2018 11:01:14 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008053.D
Sample Name: zz-2-55B(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/29/2018 8:07:28 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/29/2018 8:04:53 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:02:29 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



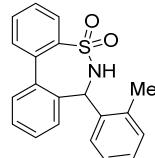
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

#	RetTime	Type	Width	Area	Height	Area
	[min]		[min]	[mAU]	*s	[mAU]
1	9.770	VB	0.2191	4479.27881	316.26987	50.0234
2	12.166	BB	0.2795	4475.06936	247.34055	49.9766

Totals : 8954.36816 563.61041

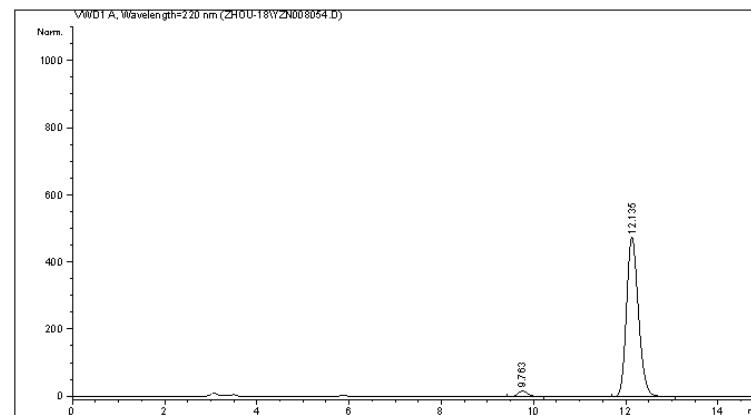


(±)-3af

*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008054.D
Sample Name: zz-2-55B

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 3/29/2018 8:30:32 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/29/2018 8:26:52 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:04:04 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 220nm
```



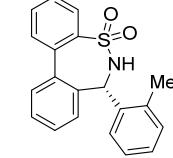
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

#	RetTime	Type	Width	Area	Height	Area
	[min]		[min]	[mAU]	*s	[mAU]
1	9.763	BB	0.2175	235.49289	16.64380	2.6824
2	12.135	BB	0.2774	8543.53125	473.72354	97.3176

Totals : 8779.02414 490.36734

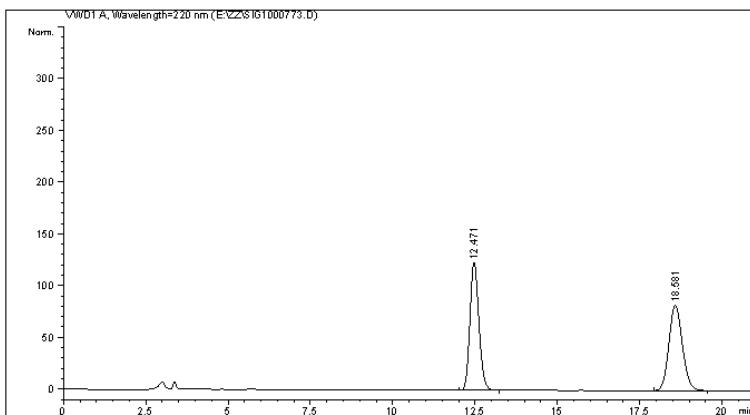


(+)-3af

*** End of Report ***

Data File E:\ZZ\SIG1000773.D
Sample Name: zz-2-62A(+-)

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 肖 1
Injection Date : 4/10/2018 9:14:07 AM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/10/2018 8:47:37 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:28:52 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



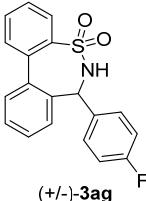
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

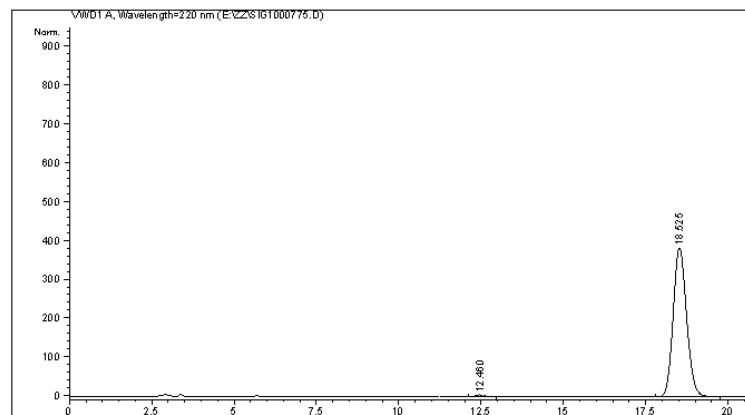
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 12.471	BB	0.2937	2350.76953	123.68821	49.9554
2 18.581	BB	0.4414	2354.96387	82.65630	50.0446

Totals : 4705.73340 206.34451



Data File E:\ZZ\SIG1000775.D
Sample Name: zz-2-62A

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 肖 1
Injection Date : 4/10/2018 10:04:53 AM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/10/2018 10:04:11 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:29:56 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



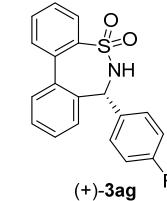
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

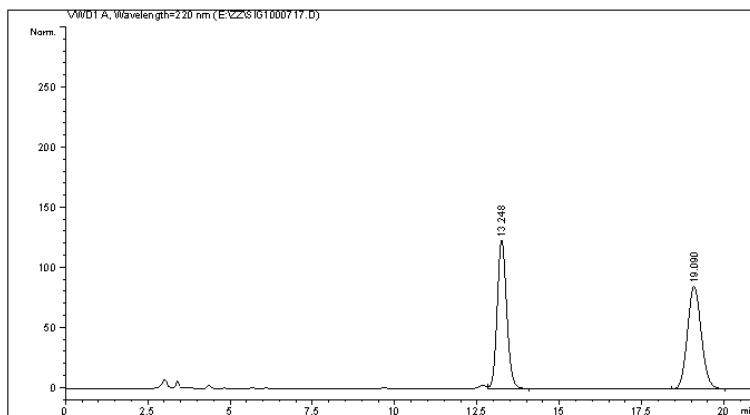
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 12.460	BB	0.2926	85.59999	4.50734	0.7801
2 18.525	BB	0.4404	1.08877e4	383.33890	99.2199

Totals : 1.09733e4 387.84624



Data File E:\ZZ\SIG1000717.D
Sample Name: zz-2-58A(+-)

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 肖 1
Injection Date : 4/2/2018 10:28:18 AM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/2/2018 10:24:36 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:09:23 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



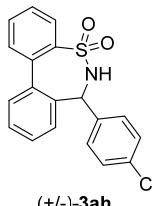
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

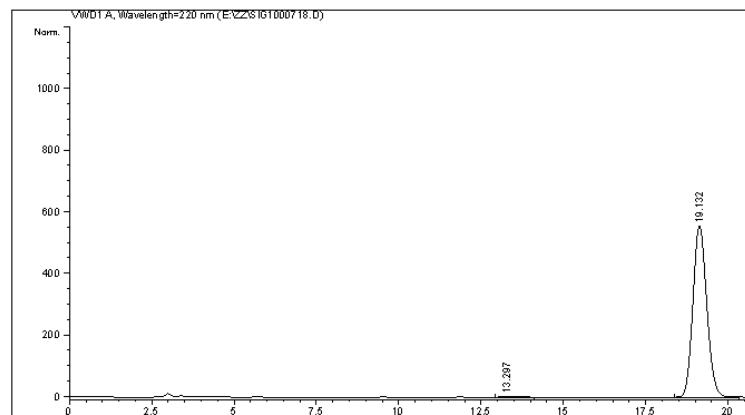
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.248	VB	0.3105	2484.96216	123.62598	50.0388
2 19.090	BB	0.4513	2481.11328	85.31990	49.9612

Totals : 4966.07544 208.94588



Data File E:\ZZ\SIG1000718.D
Sample Name: zz-2-58A

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 肖 1
Injection Date : 4/2/2018 11:09:59 AM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/2/2018 10:51:20 AM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:10:34 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



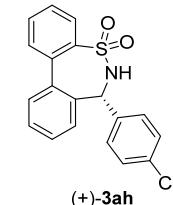
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

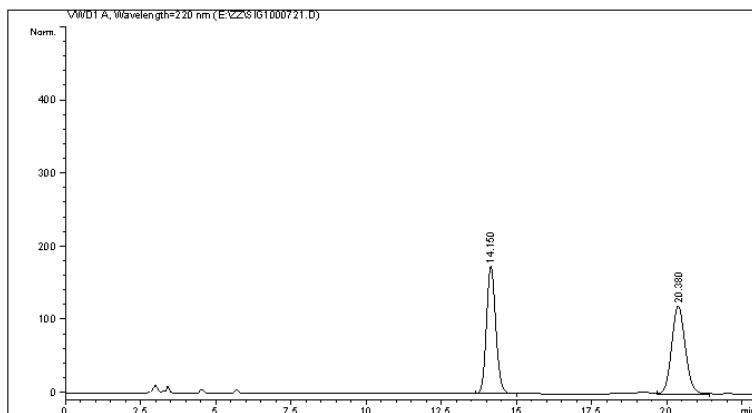
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.297	BB	0.3724	82.18174	3.22421	0.5029
2 19.132	BB	0.4540	1.6260e4	556.24695	99.4971

Totals : 1.63422e4 559.47116



Data File E:\ZZ\SIG1000721.D
Sample Name: zz-2-58B(+-)

```
=====
Acq. Operator : HFE-258          Location : Vial 91
Acq. Instrument : IVIS 1        Inj Volume : 2.000 µl
Injection Date : 4/2/2018 3:07:58 PM
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed   : 4/2/2018 3:05:01 PM by HFE-258
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 8/19/2018 9:14:35 PM
(modified after loading)
Sample Info    : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



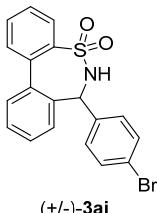
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

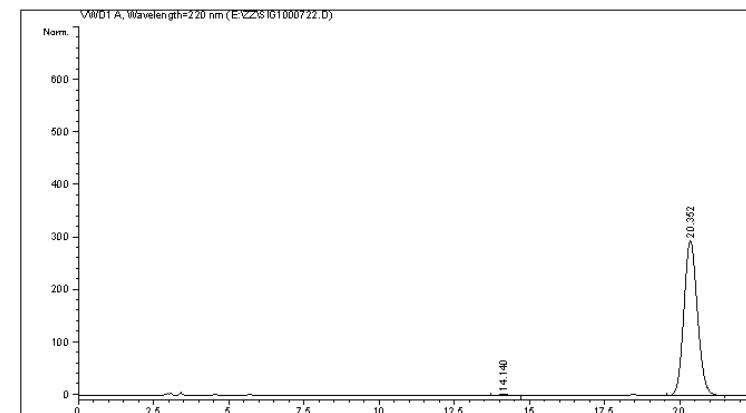
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 14.150	BB	0.3333	3751.73291	174.14243	49.9861
2 20.380	VB	0.4855	3753.81616	120.15101	50.0139

Totals : 7505.54907 294.29343



Data File E:\ZZ\SIG1000722.D
Sample Name: zz-2-58B

```
=====
Acq. Operator : HFE-258          Location : Vial 91
Acq. Instrument : IVIS 1        Inj Volume : 2.000 µl
Injection Date : 4/2/2018 3:41:11 PM
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed   : 4/2/2018 3:37:06 PM by HFE-258
(modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 8/19/2018 9:15:34 PM
(modified after loading)
Sample Info    : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



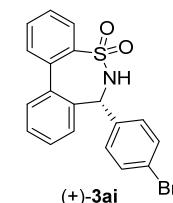
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

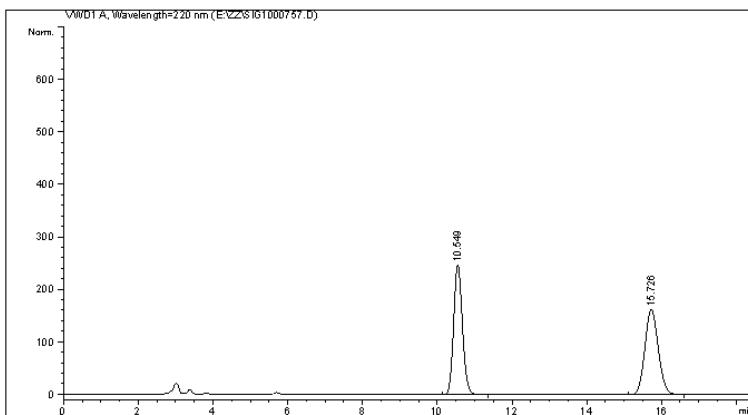
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 14.140	BB	0.3374	47.42809	2.18327	0.5130
2 20.352	BB	0.4850	9197.86328	294.76514	99.4870

Totals : 9245.29137 296.94841



Data File E:\ZZ\SIG1000757.D
Sample Name: zz-2-60A(+-)

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 蜂 1
Location : Vial 91
Injection Date : 4/6/2018 2:27:32 PM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/6/2018 1:47:26 PM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:17:08 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



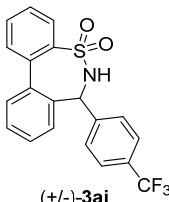
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

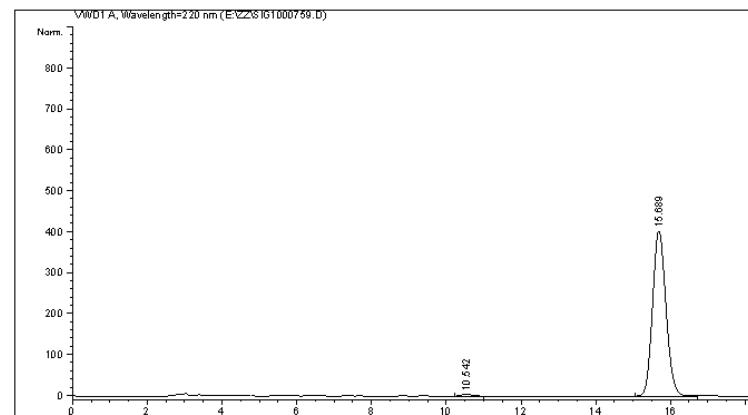
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 10.549	BB	0.2531	4038.20068	246.57562	50.0410
2 15.726	BB	0.3852	4031.58252	162.61316	49.9590

Totals : 8069.78320 409.18878



Data File E:\ZZ\SIG1000759.D
Sample Name: zz-2-60A

```
=====
Acq. Operator : HFE-258
Acq. Instrument : 仪 蜂 1
Location : Vial 91
Injection Date : 4/6/2018 3:12:05 PM
Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed : 4/6/2018 3:11:24 PM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:22:14 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



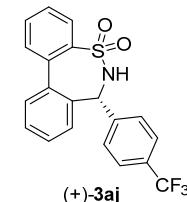
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

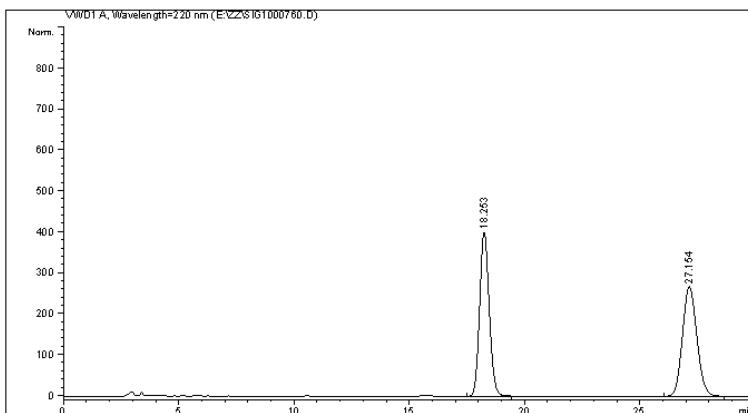
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 10.542	BB	0.2511	92.49078	5.70737	0.9122
2 15.689	BB	0.3860	1.00470e4	404.23911	99.0878

Totals : 1.01395e4 409.94648



Data File E:\ZZ\SIG1000760.D
Sample Name: zz-2-61A(+-)

```
=====
Acq. Operator : HFE-258                               Location : Vial 91
Acq. Instrument : 仪 色 1                               Inj Volume : 2.000 μl
Injection Date : 4/8/2018 3:36:01 PM
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed   : 4/8/2018 3:31:53 PM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 8/19/2018 9:23:46 PM
(modified after loading)
Sample Info    : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



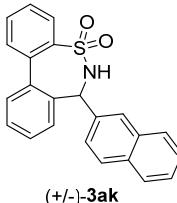
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

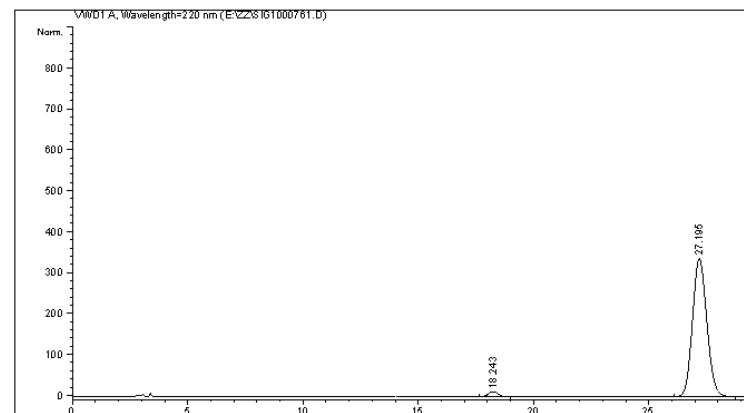
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 18.253	BB	0.4450	1.15551e4	401.24536	49.9594
2 27.154	BB	0.6726	1.15738e4	267.57956	50.0406

Totals : 2.31289e4 668.82492



Data File E:\ZZ\SIG1000761.D
Sample Name: zz-2-61A

```
=====
Acq. Operator : HFE-258                               Location : Vial 91
Acq. Instrument : 仪 色 1                               Inj Volume : 2.000 μl
Injection Date : 4/8/2018 4:10:16 PM
Acq. Method   : C:\CHEM32\1\METHODS\FM-4-4 LC.M
Last changed   : 4/8/2018 4:07:44 PM by HFE-258
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 8/19/2018 9:23:46 PM
(modified after loading)
Sample Info    : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



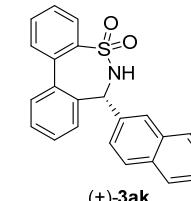
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

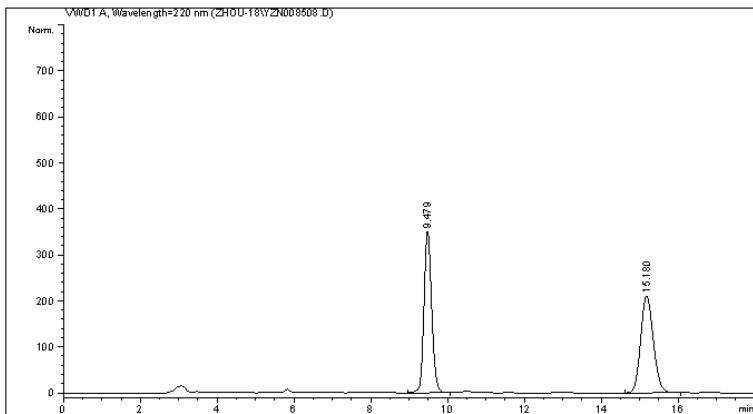
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 18.243	BB	0.4419	360.27094	12.62411	2.4091
2 27.195	BB	0.6722	1.45943e4	337.69812	97.5909

Totals : 1.49546e4 350.32223



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008508.D
Sample Name: zz-2-87(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/15/2018 9:02:40 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/15/2018 9:00:48 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:09:05 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```



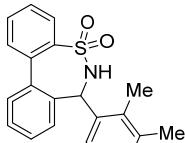
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 9.479	BB	0.2135	4896.93262	351.40527	50.2977		
2 15.180	BB	0.3562	4638.96924	211.11191	49.7023		

Totals : 9735.90186 562.51718

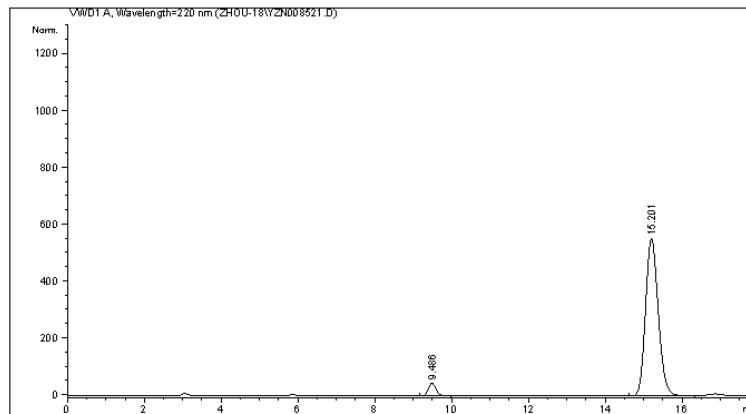


(+/-)-3al

*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008521.D
Sample Name: zz-2-87

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/15/2018 1:58:20 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/15/2018 1:57:48 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:11:22 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```



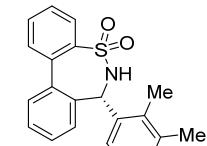
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 9.486	BB	0.2102	607.35504	44.48985	4.5727		
2 15.201	BB	0.3562	1.26747e4	553.13171	95.4273		

Totals : 1.32821e4 597.62156

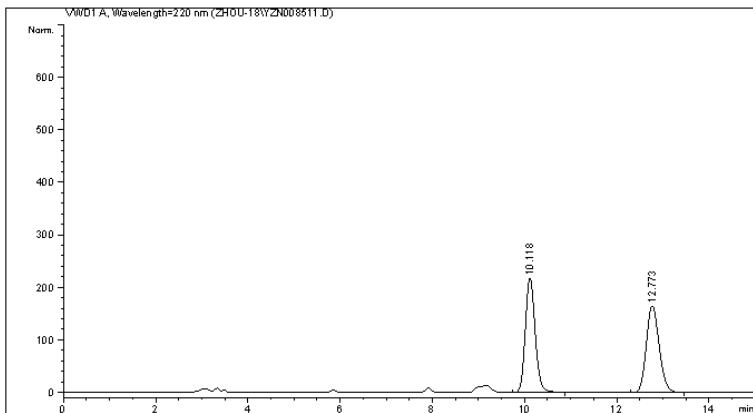


(+)-3al

*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008511.D
Sample Name: zz-2-89(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/15/2018 10:09:31 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/15/2018 10:07:23 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:12:55 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```

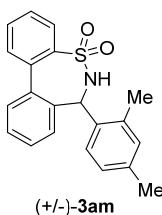


```
=====
Area Percent Report
=====
```

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

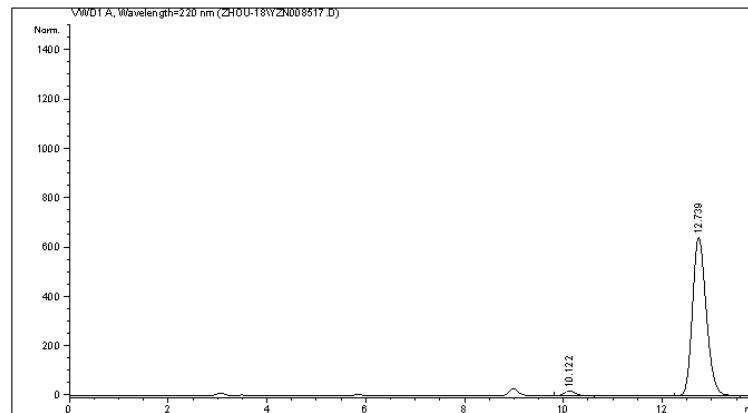
Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 10.118	VB	0.2293	3250.50928	217.97568	50.3392
2 12.773	BB	0.2999	3206.70776	165.76772	49.6608
Totals :			6457.21704	383.74339	



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008517.D
Sample Name: zz-2-89

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/15/2018 12:42:23 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/15/2018 12:29:09 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:15:01 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```

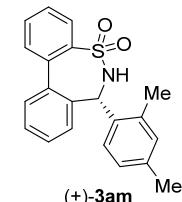


```
=====
Area Percent Report
=====
```

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

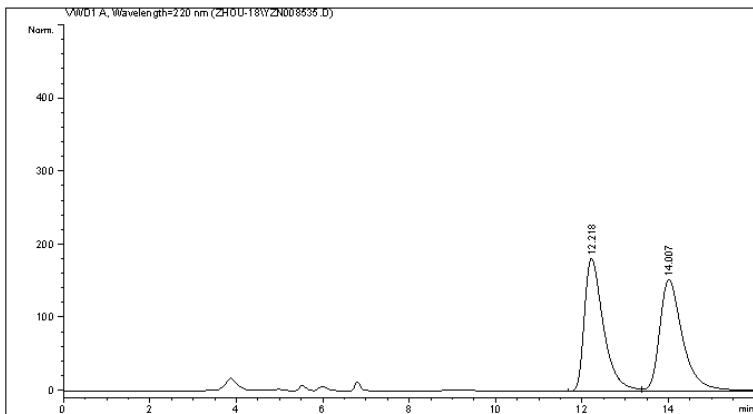
Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 10.122	BB	0.2258	295.97800	20.25657	2.3356
2 12.739	BB	0.2994	1.23767e4	641.25525	97.6644
Totals :			1.26727e4	661.51182	



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008535.D
Sample Name: zz-2-91(+-)

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/16/2018 1:33:10 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/16/2018 1:32:30 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:16:43 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 80/20, 0.8 mL/min, 30 oC, 220 nm
```



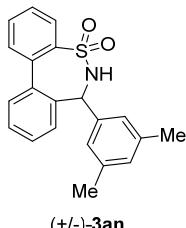
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 12.218	BV	0.4613	5556.32031	181.86340	49.7696
2 14.007	VB	0.5564	5607.76709	152.23785	50.2304

Totals : 1.11641e4 334.10126



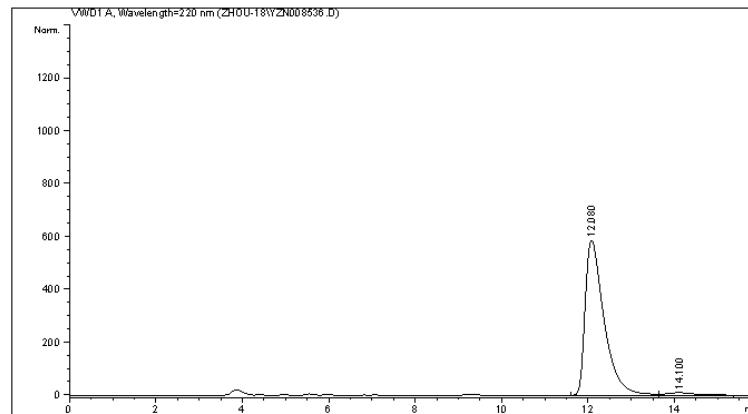
*** End of Report ***

Instrument 1 8/20/2018 11:16:54 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008536.D
Sample Name: zz-2-91

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : Vial 1
Injection Date : 5/16/2018 1:53:46 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/16/2018 1:50:51 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:18:17 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 80/20, 0.8 mL/min, 30 oC, 220 nm
```



```
=====
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	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 12.080	BV	0.4539	1.78074e4	587.76025	97.6556
2 14.100	VB	0.6302	427.48941	9.87708	2.3444

Totals : 1.82349e4 597.63733

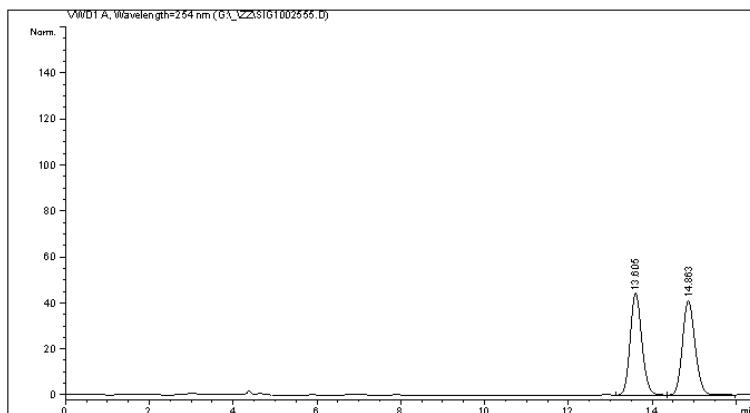
*** End of Report ***

Instrument 1 8/20/2018 11:18:23 AM

Page 1 of 1

Data File G:\V2Z\SIG1002555.D
Sample Name: zz-5-49(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/22/2019 9:17:35 PM Inj Volume : 5.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/22/2019 9:10:02 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:23:22 PM
(modified after loading)
Sample Info : AD-H, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



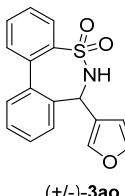
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=254 nm

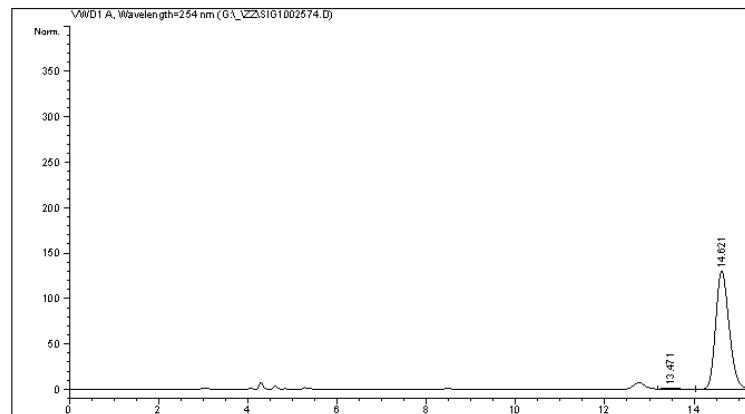
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.605	VV	0.2983	859.40112	44.30006	49.8809
2 14.863	VV	0.3254	863.50519	41.05014	50.1191

Totals : 1722.90631 85.35021



Data File G:\V2Z\SIG1002574.D
Sample Name: zz-5-54

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/26/2019 10:54:30 AM Inj Volume : 5.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/26/2019 10:30:13 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:27:11 PM
(modified after loading)
Sample Info : AD-H, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



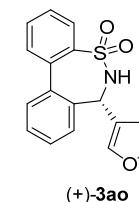
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=254 nm

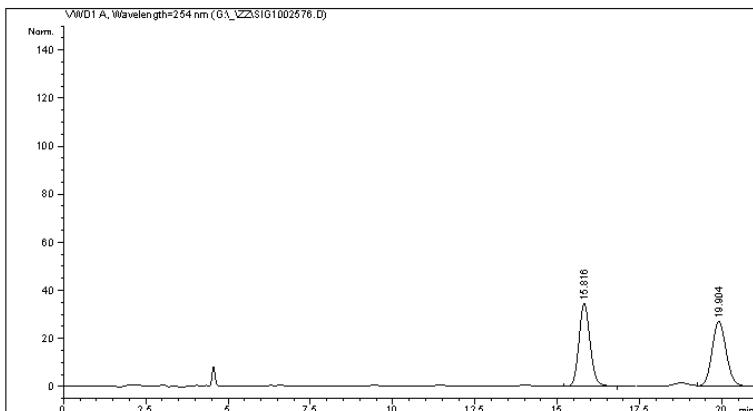
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 13.471	VV	0.3159	33.24145	1.59020	1.2072
2 14.621	VV	0.3245	2720.28979	129.78143	98.7928

Totals : 2753.53124 131.37163



Data File G:\ \ZZ\SIG1002576.D
Sample Name: zz-5-55(+-)

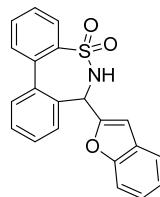
```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Injection Date : 2/26/2019 3:26:09 PM Inj Volume : 5.000 μl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/26/2019 3:24:22 PM (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:29:32 PM (modified after loading)
Sample Info : AD-H, n-hexane/i-PrOH =80/20, 1.0 mL/min, 30 oC, 254 nm
```



Area Percent Report

Signal 1: WWD1 A, Wavelength=254 nm						
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.816	BV	0.3632	810.62933	34.36010	50.2031
2	19.904	VV	0.4587	804.07184	27.50803	49.7969
Totals :				1614.70117	61.41812	

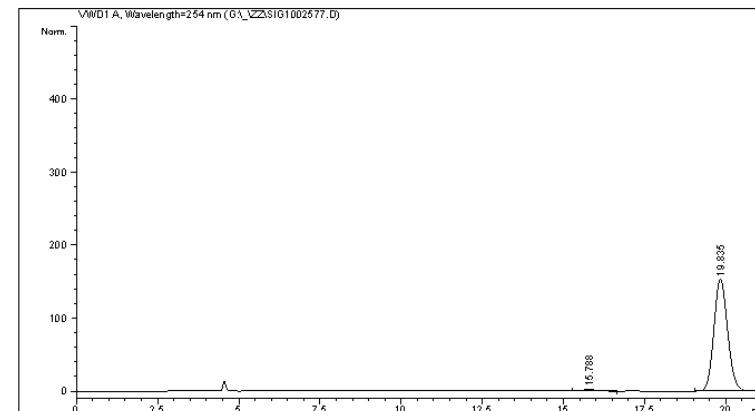
=====
*** End of Report ***



(+/-)-3ap

Data File G:\ZZ\SIG1002577.D
Sample Name: zz-5-55

```
=====
Acc. Operator   : 
Acq. Instrument : 仪器 1                               Location : Vial 1
Injection Date : 2/26/2019 3:56:48 PM                 Inj Volume : 5.000 µl
Acc. Method    : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 2/26/2019 3:47:33 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed   : 3/1/2019 7:31:56 PM
                  (modified after loading)
Sample Info    : AD-H, n-hexane/i-ProOH/80/20, 1.0 mL/min, 30 oC, 254 nm
```



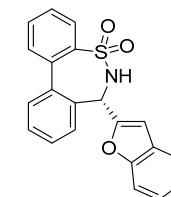
Area Percent Report

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

Signal 1: VWD1 A, Wavelength=254 nm						
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Heiight [mAU]	Area %
1	17.788	BEB	0.3691	61.31554	2.58133	1.3448
2	19.835	BBA	0.4549	4498.02295	153.03374	98.6552

Totals : 4559.33849 155.61507

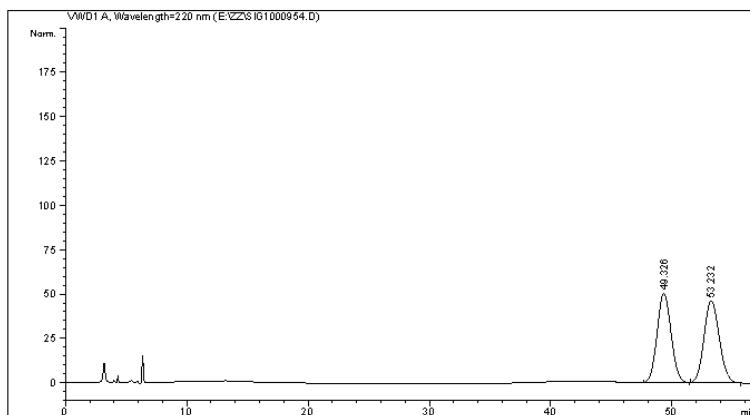
=====
*** End of Report ***



(+)-3a

Data File E:\ZZ\SIG1000954.D
Sample Name: zz-2-97(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/9/2018 12:10:52 PM Inj Volume : 2.000 μl
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/9/2018 11:35:13 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:38:47 PM
(modified after loading)
Sample Info : IC, n-hexane / i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```



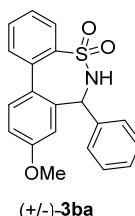
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

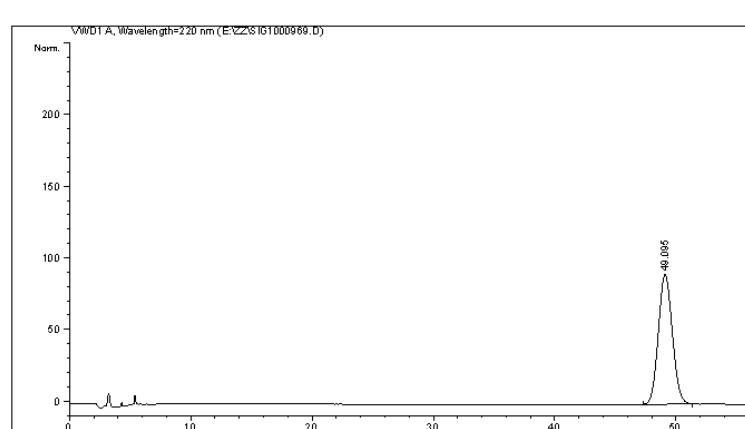
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	49.326	BB	1.2533	4067.06982	50.51767	49.9693
2	53.232	BB	1.3803	4068.80591	46.38844	50.0107

Totals : 8135.87573 96.90611



Data File E:\ZZ\SIG1000969.D
Sample Name: zz-2-97

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/10/2018 7:16:33 PM Inj Volume : 2.000 μl
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/10/2018 7:15:13 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:40:20 PM
(modified after loading)
Sample Info : IC, n-hexane / i-PrOH = 80/20 , 1.0 mL/min, 30 oC, 220 nm
```



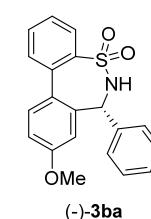
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=220 nm

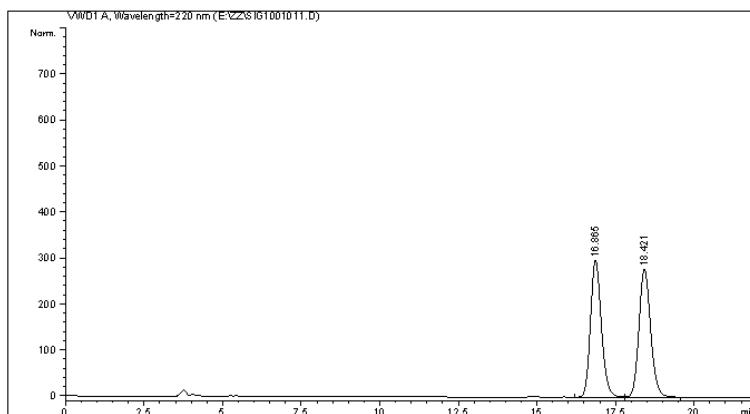
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	49.095	BB	1.2422	7254.51904	90.69852	100.0000

Totals : 7254.51904 90.69852



Data File E:\ZZ\SIG1001011.D
Sample Name: zz-3-9(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/14/2018 10:20:38 PM Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/14/2018 10:15:26 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:41:12 PM
(modified after loading)
Sample Info : AD-H, n-hexane / i-PrOH = 80/20 , 0.8mL/min, 30 oC, 220
mm
```



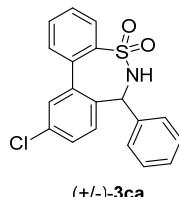
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

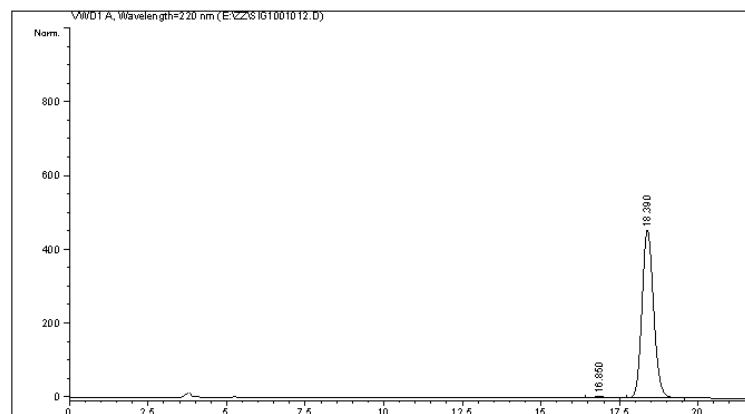
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 16.865	VV	0.3716	7184.28418	297.60495	49.9403
2 18.421	VB	0.3993	7201.46191	277.90320	50.0597

Totals : 1.43657e4 575.50815



Data File E:\ZZ\SIG1001012.D
Sample Name: zz-3-9

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/14/2018 10:54:58 PM Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/14/2018 10:49:50 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:42:22 PM
(modified after loading)
Sample Info : AD-H, n-hexane / i-PrOH = 80/20 , 0.8mL/min, 30 oC, 220
mm
```



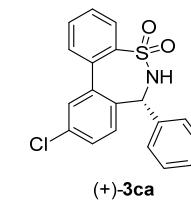
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

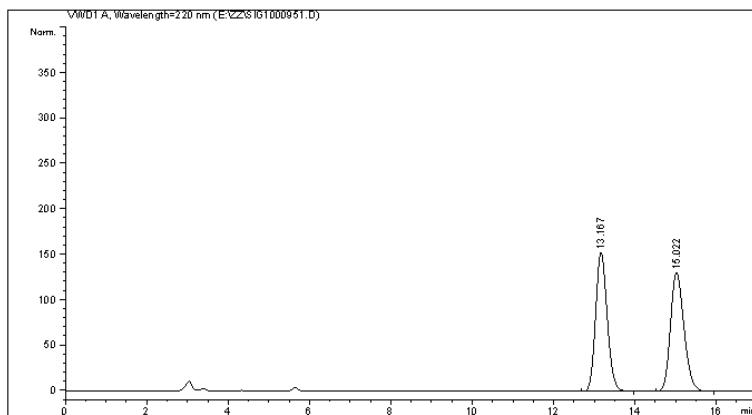
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1 16.850	BB	0.3681	119.02818	4.95856	1.0060
2 18.390	BB	0.3969	1.17133e4	455.73901	98.9940

Totals : 1.18323e4 460.69758



Data File E:\ZZ\SIG1000951.D
Sample Name: zz-2-98(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/9/2018 10:03:25 AM Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/9/2018 10:02:33 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:34:27 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



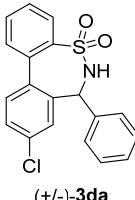
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

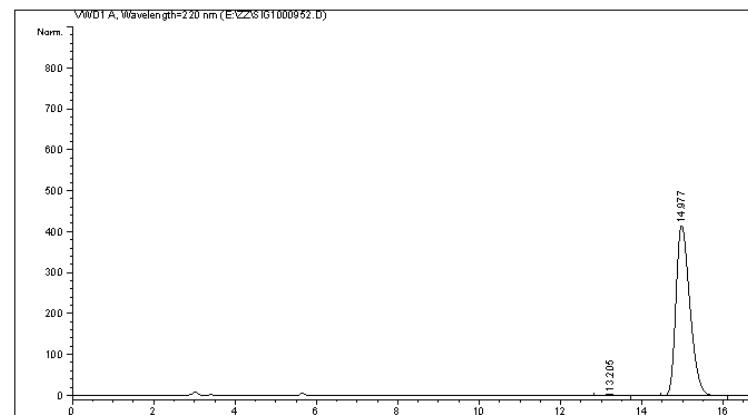
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 13.167	BB	0.3059	3025.71704	152.87488	49.9755
2 15.022	BB	0.3601	3028.68823	130.32420	50.0245

Totals : 6054.40527 283.19908



Data File E:\ZZ\SIG1000952.D
Sample Name: zz-2-98

```
=====
Acq. Operator : 仪器 1 Location : Vial 91
Acq. Instrument : 仪器 1 Location : Vial 91
Injection Date : 5/9/2018 10:31:46 AM Inj Volume : 2.000 μl
=====
Acq. Method : C:\CHEM32\1\METHODS\FM-4-4_LC.M
Last changed : 5/9/2018 10:29:10 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/19/2018 9:36:04 PM
(modified after loading)
Sample Info : AD-H, n-hexane/ i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220
nm
```



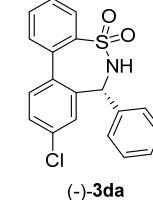
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

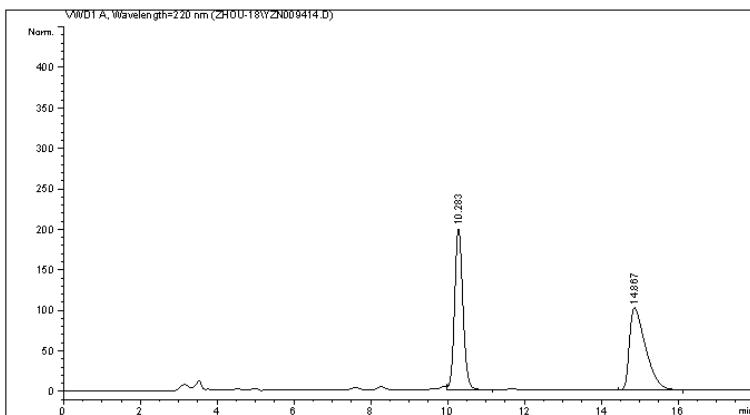
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	
1 13.205	BB	0.3089	70.28666	3.50581	0.7021
2 14.977	BB	0.3686	9941.12891	416.31915	99.2979

Totals : 1.00114e4 419.82496



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN009414.D
Sample Name: zz-3-S2(+-)

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1             Location : -
Injection Date : 7/20/2018 10:09:33 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 7/20/2018 9:54:07 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 8/20/2018 11:32:49 AM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH =80/20, 1.0 mL/min, 30 oC, 220 nm
```



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

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

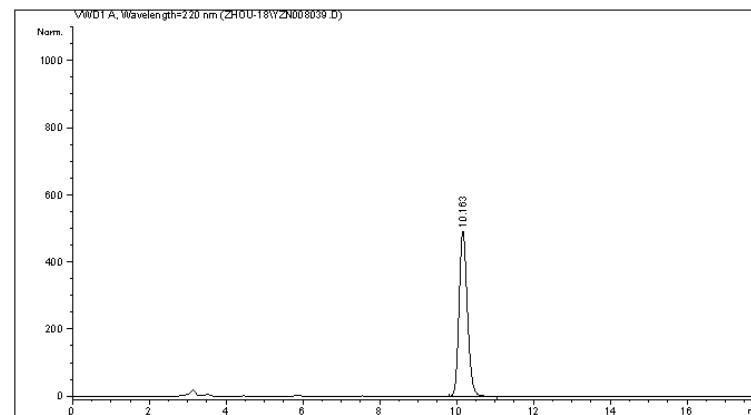
Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 10.283	VB	0.2310	3006.07080	199.58835	50.4767
2 14.867	BB	0.4363	2949.28784	101.57075	49.5233
Totals :			5955.35864	301.15910	



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008039.D
Sample Name: zz-2-48

```
=====
Acq. Operator :                               Location : Vial 1
Acq. Instrument : Instrument 1             Location : -
Injection Date : 3/28/2018 9:34:26 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/28/2018 9:22:11 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 11/7/2018 10:17:18 PM
(modified after loading)
Sample Info : AD-H, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 220 nm
```



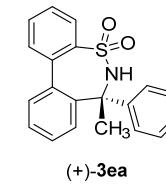
```
=====
Area Percent Report
=====
```

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU *s]	[mAU]	%
1 10.163	BB	0.2314	7374.13086	492.79382	100.0000

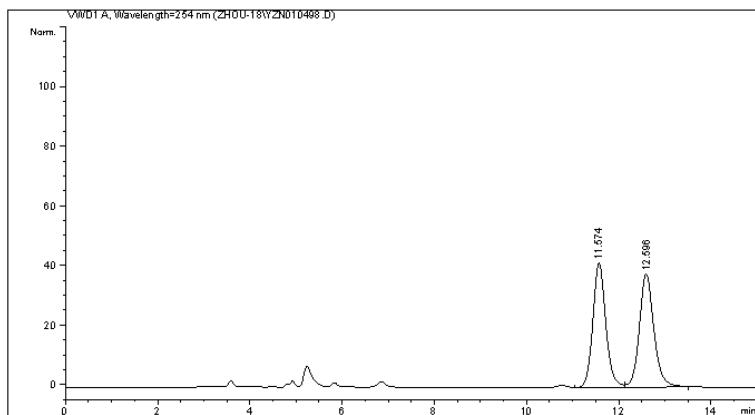
Totals : 7374.13086 492.79382



*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN010498.D
Sample Name: zz-4-49A(+-)

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1             Location : -
Injection Date : 10/19/2018 1:35:12 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 10/19/2018 1:33:49 PM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 11/6/2018 2:26:03 PM
                                         (modified after loading)
Sample Info : IA, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 254 nm
```

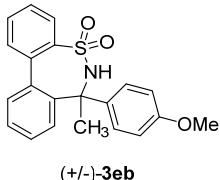


```
=====
Area Percent Report
=====
```

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

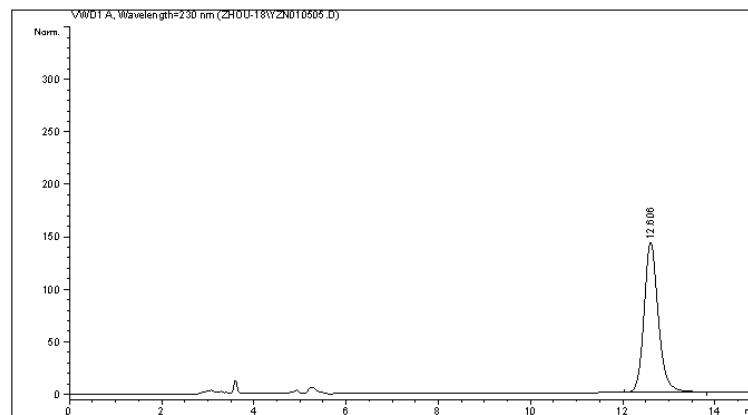
Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 11.574	VV	0.2973	814.47351	41.77811	49.3550		
2 12.596	VB	0.3324	835.76007	37.99205	50.6450		
Totals :			1650.23358		79.77017		



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN010505.D
Sample Name: zz-4-49A

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1             Location : -
Injection Date : 10/19/2018 7:51:53 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 10/19/2018 7:38:33 PM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 11/6/2018 2:28:25 PM
                                         (modified after loading)
Sample Info : IA, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```

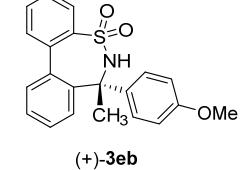


```
=====
Area Percent Report
=====
```

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

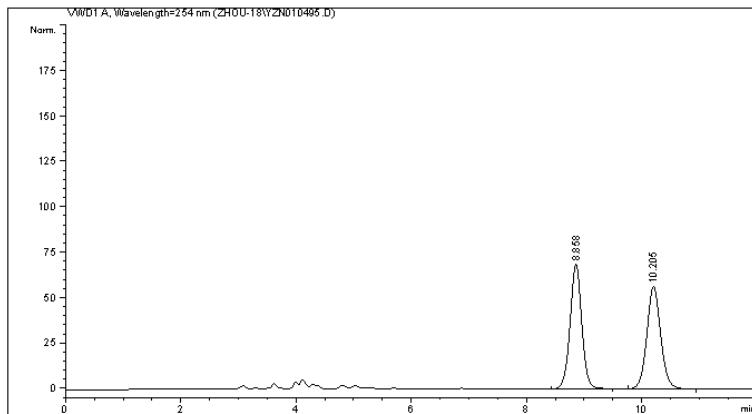
Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 12.606	BB	0.3254	3083.32446	143.26038	100.0000		
Totals :			3083.32446		143.26038		



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN010495.D
Sample Name: zz-4-49B(+-)

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1             Location : -
Injection Date : 10/19/2018 12:47:00 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 10/19/2018 12:44:58 PM
                                                (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 11/6/2018 2:30:01 PM
                                                (modified after loading)
Sample Info : IA, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 254 nm
```

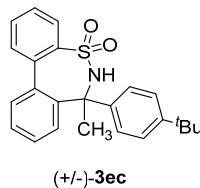


```
=====
Area Percent Report
```

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

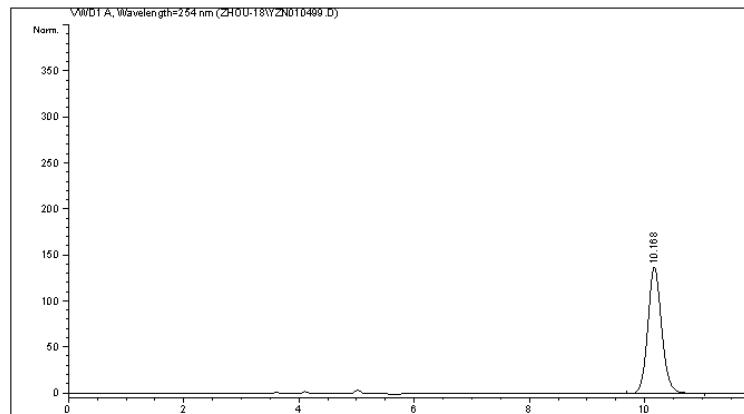
Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 8.858	BB	0.2169	977.57111	68.73567	50.8132		
2 10.205	BB	0.2553	946.28217	56.43370	49.1866		
Totals :			1923.85327		125.16938		



Data File C:\CHEM32\1\DATA\ZHOU-18\YZN010499.D
Sample Name: zz-4-49B

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1             Location : -
Injection Date : 10/19/2018 1:51:33 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 10/19/2018 1:50:25 PM
                                                (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 11/6/2018 2:31:20 PM
                                                (modified after loading)
Sample Info : IA, Hexane/i-PrOH = 80/20, 1.0mL/min, 30 oC, 254 nm
```



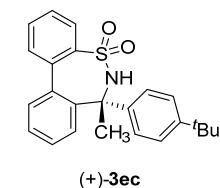
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=254 nm

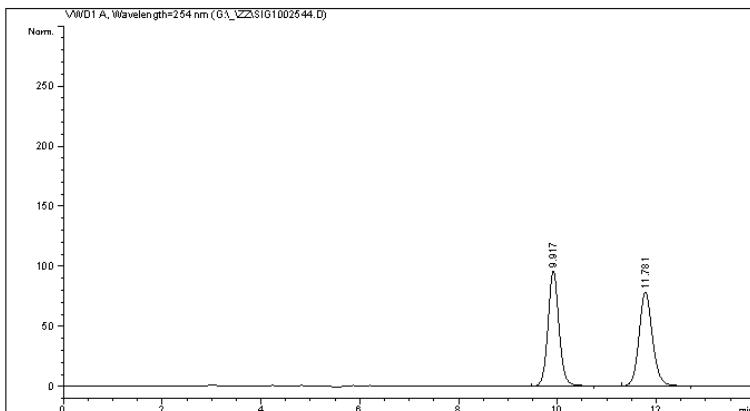
Peak RetTime	Type	Width	Area	Height	Area		
# [min]		[min]	[mAU]	*s	[mAU]	1	%
1 10.168	BB	0.2554	2310.54150	137.76035	100.0000		

Totals : 2310.54150 137.76035



Data File G:\V2Z\SIG1002544.D
Sample Name: zz-5-48A(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/21/2019 8:14:21 PM Inj Volume : 5.000 μl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/21/2019 8:01:04 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:11:01 PM
(modified after loading)
Sample Info : IA, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



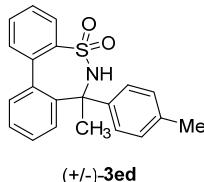
=====
Area Percent Report

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

Signal 1: VWD1 A, Wavelength=254 nm

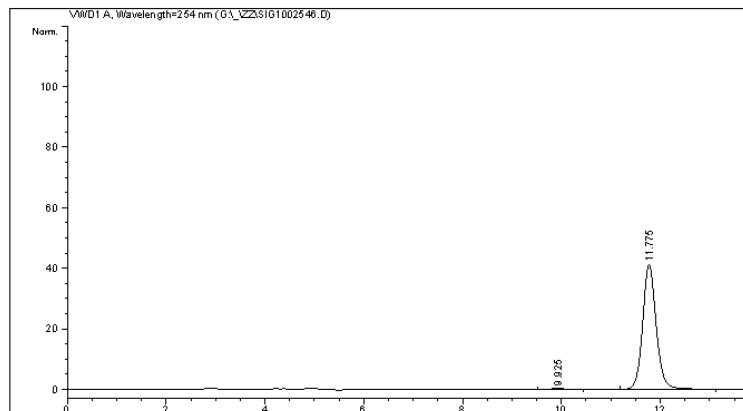
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.917	BB	0.2380	1497.90271	96.01641	50.2076
2	11.781	BB	0.2893	1485.51697	78.32873	49.7924

Totals : 2983.41968 174.34514



Data File G:\V2Z\SIG1002546.D
Sample Name: zz-5-48A

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/21/2019 8:47:53 PM Inj Volume : 5.000 μl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/21/2019 8:45:51 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:13:17 PM
(modified after loading)
Sample Info : IA, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



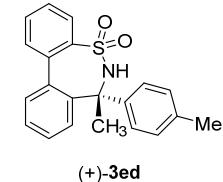
=====
Area Percent Report

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

Signal 1: VWD1 A, Wavelength=254 nm

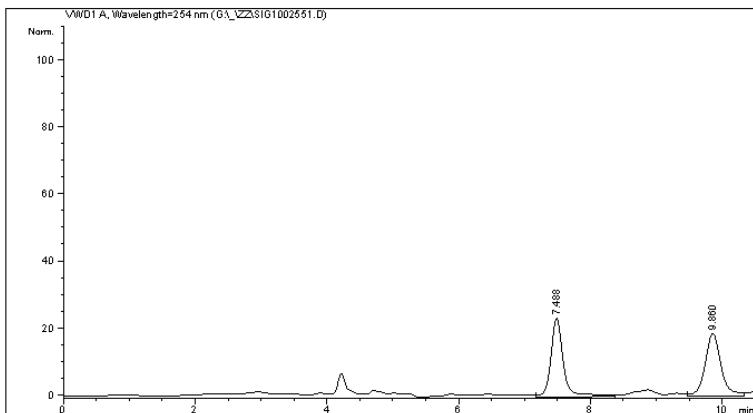
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.925	WB	0.2584	5.34911	3.08403e-1	0.6752
2	11.775	BB	0.2906	786.92102	41.24401	99.3248

Totals : 792.27013 41.55241



Data File G:\V2Z\SIG1002551.D
Sample Name: zz-5-48B(+-)

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/22/2019 3:53:50 PM Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/22/2019 3:51:10 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:16:47 PM
(modified after loading)
Sample Info : IA, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



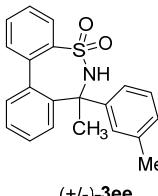
```
=====
Area Percent Report
```

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

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.468	VB	0.1973	312.04053	23.43923	49.9383
2	9.860	VB	0.2518	312.81171	18.63601	50.0617

Totals : 624.85223 42.07524



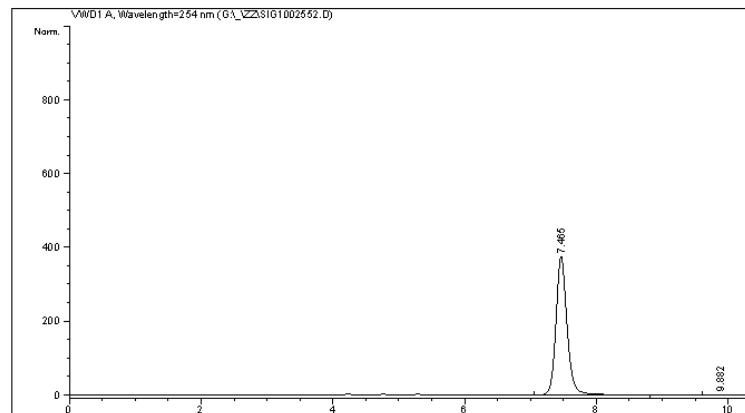
*** End of Report ***

Instrument 1 3/1/2019 7:17:10 PM

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Data File G:\V2Z\SIG1002552.D
Sample Name: zz-5-48B

```
=====
Acq. Operator : 仪器 1 Location : Vial 1
Acq. Instrument : 仪器 1 Location : Vial 1
Injection Date : 2/22/2019 4:19:11 PM Inj Volume : 5.000 µl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 2/22/2019 4:04:57 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed : 3/1/2019 7:18:22 PM
(modified after loading)
Sample Info : IA, n-hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm
```



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

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

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.465	VB	0.1764	4388.61963	375.36118	99.8846
2	9.882	BB	0.2327	5.07140	3.38640e-1	0.1154

Totals : 4393.69103 375.69982

*** End of Report ***

Instrument 1 3/1/2019 7:19:05 PM

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