A facile and regioselective multicomponent synthesis of chiral aryl-1,2-mercaptoamines in water followed by monoamine oxidase (MAO-N) enzymatic resolution

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Supporting Information

General Methods

^{1}H NMR and ^{13}C NMR spectra were recorded with an Ascend 400 spectrometer Bruker at room temperature (rt) operating at the frequencies indicated. Chemical shifts (δ) are in ppm, referenced to tetramethylsilane. Coupling constants (J) are reported in Hertz. Splitting patterns are abbreviated as follows: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), sextet (sxt), broad (br) or some combination of them. Accurate masses (HRMS) were confirmed by flow injection analysis (H_2O/CH_3CN 1:1 + 0.1% HCOOH). The purity of the compounds was assessed by reverse-phase liquid chromatography coupled with a mass spectrometer (Agilent series 1100 LC/MSD) with a UV detector at λ = 254 nm and an electrospray ionization source (ESI). LC-MS analysis was carried out using an HP 1100 HPLC system coupled with an HP 1050 DAD and an Agilent 1100 Series LC/MSD. The chiral column used to separate the enantiomers was a Chiralpak IG® (5µm, 4.6 mm X 250 mm), supplied by Daicel. Heptane and ethanol were used as an isocratic mobile phase system.

Mass spectra were acquired in positive mode scanning over the mass range of 50–1500. The following ion source parameters were used: drying gas flow, 9 mL/min; nebulize pressure, 40 psig; and drying gas temperature, 350 ºC. All target compounds possessed a purity of ≥ 95% as verified by HPLC analyses. TLC was performed using commercially available pre-coated plates and visualized with UV light at 254 nm; KMnO4 was used to reveal the products.

Flash column chromatography was carried out using Sigma Aldrich silica gel particle size, 40-63 µm particle size 60 Å. All reactions were conducted under a nitrogen atmosphere in oven-dried glassware unless stated otherwise. All solvents and commercially available reagents were used as received.

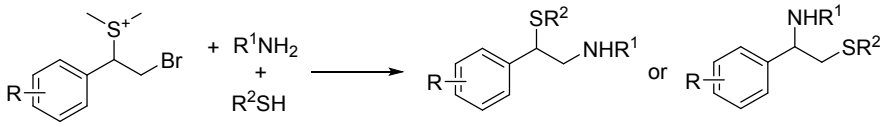
A spectrofuge 16M microcentrifuge was used to remove the cell from MAO-N samples prior to HPLC injection. α_D measurements were taken using a Bellingham and Stanley ADP440+ Polarimeter with a cell length of 5dm.

Microwave irradiations were conducted using a CEM Discover Synthesis Unit. The machine consists of a continuous focused microwave power delivery system with operator, selectable power output from 0 to 300 W. The temperature of the contents of the vessels was monitored using a calibrated infrared temperature control mounted under the reaction vessel. All the experiments were performed using a stirring option whereby the contents of the vessel are stirred by means of rotating magnetic plate located below the floor of the microwave cavity and a Teflon® coated magnetic stirring bar in the vessel.

Synthesis of styrene sulfonium bromides (SBB) 6a-c

Dimethylsulfide (8.9mmols, 400uL) was added at 0 °C to a round bottom flask containing DCM (1.5mL). Bromine (2.3mmol, 100uL) was then added to this solution, followed by the appropriate styrene (3.4 mmol). The resulting mixture was stirred for 30 minutes at 0 °C. After this time, the reaction was brought to room temperature, and Et_2O was added to form a white precipitate. The precipitate was filtered and washed several times with Et_2O to afford the pure products **6a-c**.

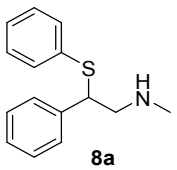
General procedure for the multicomponent synthesis of aryl-1,2-mercaptoamines 8 or 9



Method A. The sulfonium salt SBB **6** (0.576 mmol) was placed into a microwave vial and added with water (5 mL). The appropriate amine (5.76 mmol) and thiol/mercaptane (0.634 mmol) were then added and the reaction mixture was heated at 80 °C for 30 minutes under microwave irradiation (maximum power = 200W; maximum pressure = 150 psi). The reaction was then extracted twice with AcOEt (4 mL) and the organic phase was washed with brine (1 x 10 mL). The organic layers were collected, dried with $MgSO_4$, concentrated in vacuo, and purified on silica gel (3:7 Hexane:Ethyl acetate) to yield the products **8a** and **8c** as oils.

Method B. The sulfonium salt SBB **6** (0.576 mmol) was placed into a microwave vial and added with water (5 mL). The appropriate amine (5.76 mmol) and thiol/mercaptane (0.634 mmol) were then added and the reaction mixture was heated at 150 °C for 20 minutes under microwave irradiation (maximum power = 200W; maximum pressure = 150 psi). The reaction was then extracted twice with AcOEt (4 mL) and the organic phase was washed with brine (1 x 10 mL). The organic layers were collected, dried with $MgSO_4$, concentrated in vacuo, and purified on silica gel (3:7 Hexane:Ethyl acetate) to yield the products **8b**, **8d-p**, **9e**, **9h**, **9n-o** as oils.

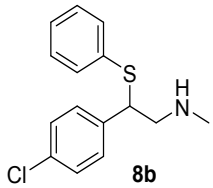
***N*-Methyl-2-phenyl-2-(phenylthio)ethan-1-amine (8a)**



8a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.29 (m, 6H), 7.27-7.18 (m, 4H), 4.45 (t, *J*=7.34 Hz, 1H), 3.11 (dd, *J*=1.28, 7.34 Hz, 2H), 2.47 (s, 3H) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.6, 136.2, 133.9, 133.0, 130.2, 129.2, 128.5, 128.3, 128.2, 127.4, 56.3, 51.3, 35.8 ppm. **HRMS** (ESI) m/z calcd. For $C_{15}H_{18}NS^+$ $[M + H]^+$ 244.1154, found 244.1151. **(*S*)-8a**: $[\alpha]^{20}_D$ = +29.2

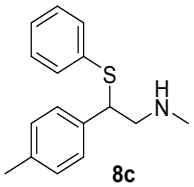
2-(4-Chlorophenyl)-*N*-methyl-2-(phenylthio)ethan-1-amine (8b)



8b

^{1}H NMR (400MHz, $CDCl_3$) δ 7.21-7.11 (m, 10H), 4.25 (t, *J*=7.2 Hz, 1H), 2.97-2.90 (m, 2H), 2.36 (s, 3H) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 132.5, 129.2, 128.9, 128.8, 127.5, 56.4, 52.7, 36.1 ppm. **HRMS** (ESI) m/z calcd. For $C_{15}H_{17}ClNS^+$ $[M + H]^+$ 278.0765, found 278.0757.

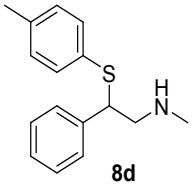
***N*-Methyl-2-(phenylthio)-2-(p-tolyl)ethan-1-amine (8c)**



8c

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.28 (m, 2H), 7.23-7.18 (m, 5H), 7.11 (d, *J*=7.89 Hz, 2H), 4.36 (s, 1H), 3.10-2.92 (m, 2H), 2.41 (s, 3H), 2.32 (s, 3H) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.3, 137.2, 134.8, 132.0, 129.4, 128.8, 127.8, 127.1, 56.7, 52.7, 36.1, 21.2 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{20}NS^+$ $[M + H]^+$ 258.1311, found 258.1305.

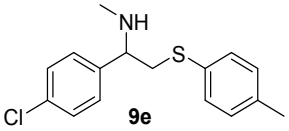
***N*-Methyl-2-phenyl-2-(p-tolylthio)ethan-1-amine (8d)**



8d

^{1}H NMR (400 MHz, CD_3OD) δ 7.31-7.15 (m, 7H), 7.04 (d, *J*=7.79 Hz, 2H), 4.29 (t, *J*=7.47 Hz, 1H), 3.03-2.90 (m, 2H), 2.33 (s, 3H), 2.27 (s, 3H) ppm. **^{13}C NMR** (101 MHz, CD_3OD) δ 141.8, 139.1, 134.5, 131.7, 130.7, 129.7, 129.2, 128.7, 56.9, 54.3, 36.0, 21.3 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{20}NS^+$ $[M + H]^+$ 258.1311, found 258.1304.

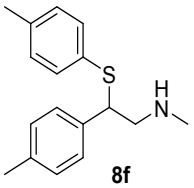
1-(4-Chlorophenyl)-*N*-methyl-2-(*p*-tolylthio)ethan-1-amine (9e)



9e

^{1}H NMR (400MHz, CD_3OD) δ = 7.32-7.29 (m, 2H), 7.26-7.23 (m, 4H), 7.11 (d, *J*=8.0 Hz, 2H), 3.54 (s, 1H), 3.13 (d, J=6.9 Hz, 2H), 2.48 (s, 1H), 2.31 (s, 3H), 2.23-2.13 (m, 3H) ppm. **^{13}C NMR** (101MHz, CD_3OD) δ = 141.8, 138.4, 134.7, 133.3, 132.3, 131.2, 130.7, 130.0, 64.8, 42.8, 34.6, 21.4 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{19}ClNS^+$ $[M + H]^+$ 292.0921, found 292.0911.

***N*-Methyl-2-(p-tolyl)-2-(p-tolylthio)ethan-1-amine (8f)**

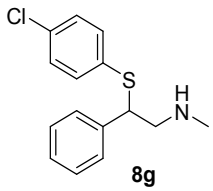


8f

^{1}H NMR (400MHz, CD_3OD) δ = 7.21-7.17 (m, 2H), 7.16-7.08 (m, 4H), 7.08-7.04 (m, J=7.9 Hz, 2H), 4.27 (t, *J*=7.5 Hz, 1H), 2.95 (dd, *J*=5.5, 7.5 Hz, 2H), 2.33 (s, 3H), 2.30 (s, 3H), 2.28 (s, 3H) ppm. **^{13}C**

NMR (101MHz, CD_3OD) δ = 139.1, 138.6, 138.6, 134.5, 131.8, 130.7, 130.4, 129.1, 57.0, 53.9, 35.9, 21.3, 21.2 ppm. **HRMS** (ESI) m/z calcd. For $C_{17}H_{22}NS^+$ $[M + H]^+$ 272.1467, found 272.1459.

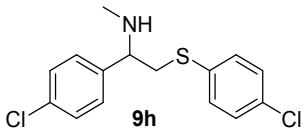
2-((4-Chlorophenyl)thio)-*N*-methyl-2-phenylethan-1-amine (8g)



8g

^{1}H NMR (400MHz, CD_3OD) δ = 7.33-7.20 (m, 9H), 4.40 (s, 1H), 3.01 (dd, *J*=5.5, 7.4 Hz, 2H), 2.37 (s, 3H) ppm. **^{13}C NMR** (101MHz, CD_3OD) δ = 141.4, 135.3, 134.7, 134.4, 130.1, 129.9, 129.2, 128.9, 56.9, 54.0, 36.0 ppm. **HRMS** (ESI) m/z calcd. For $C_{15}H_{17}ClNS^+$ $[M + H]^+$ 278.0765, found 278.0759.

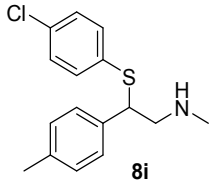
1-(4-Chlorophenyl)-2-((4-chlorophenyl)thio)-*N*-methylethan-1-amine (9h)



9h

^{1}H NMR (400MHz, CD_3OD) δ 7.29-7.25 (m, 4H), 7.24-7.22 (m, 4H), 4.37 (t, *J*=7.4 Hz, 1H), 2.98 (d, *J*= 7.4 Hz, 2H), 2.36 (s, 3H) ppm. **^{13}C NMR** (101MHz, $CDCl_3$) d 140.6, 134.1, 133.3, 131.3, 129.2, 128.8, 128.6, 63.1, 42.4, 34.5 ppm. **HRMS** (ESI) m/z calcd. For $C_{15}H_{16}Cl_2NS^+$ $[M + H]^+$ 312.0375, found 312.0368.

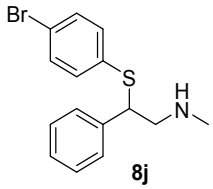
2-((4-Chlorophenyl)thio)-*N*-methyl-2-(p-tolyl)ethan-1-amine (8i)



8i

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.08 (m, 8H), 4.32 (t, *J*=7.29 Hz, 1H), 3.02 (dd, *J*=2.71, 7.29 Hz, 2H), 2.42 (s, 3H), 2.33 (s, 3H) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.4, 136.8, 133.5, 133.3, 129.4, 128.9, 127.7, 56.4, 52.9, 36.0, 21.1 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{19}ClNS^+$ $[M + H]^+$ 292.0921, found 292.0905.

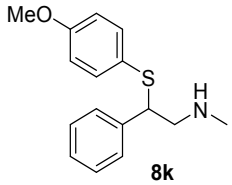
2-((4-Bromophenyl)thio)-*N*-methyl-2-phenylethan-1-amine (8j)



8j

^{1}H NMR (400MHz, CD_3OD) δ = 7.38-7.34 (m, 2H), 7.30-7.28 (m, 4H), 7.25 (dd, *J*=3.6, 5.0 Hz, 1H), 7.21-7.17 (m, 2H), 4.39 (t, *J*=7.4 Hz, 1H), 3.00 (dd, *J*=5.4, 7.4 Hz, 2H), 2.35 (s, 3H) ppm. **^{13}C NMR** (101MHz, CD_3OD) δ = 141.6, 135.6, 135.3, 133.3, 130.1, 129.4, 129.2, 122.8, 57.2, 54.1, 36.2 ppm. **HRMS** (ESI) m/z calcd. For $C_{15}H_{17}BrNS^+$ $[M + H]^+$ 322.0260, found 322.0253.

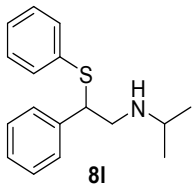
2-((4-Methoxyphenyl)thio)-*N*-methyl-2-phenylethan-1-amine (8k)



8k

^{1}H NMR (400MHz, CD_3OD) δ = 7.30-7.24 (m, 2H), 7.24-7.17 (m, 5H), 6.81-6.76 (m, 2H), 4.18 (t, *J*=7.5 Hz, 1H), 3.74 (s, 3H), 3.04-2.91 (m, 2H), 2.35 (s, 3H) ppm. **^{13}C NMR** (101 MHz, CD_3OD) δ 161.6, 141.8, 137.3, 129.7, 129.2, 128.7, 125.2, 115.6, 56.5, 55.9, 54.9, 35.9 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{20}NOS^+$ $[M + H]^+$ 274.1260, found 274.1254.

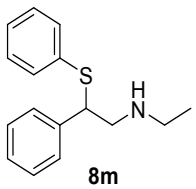
***N*-(2-phenyl-2-(phenylthio)ethyl)propan-2-amine (8l)**



8l

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.09 (m, 10H), 4.28 (t, *J*=7.29 Hz, 1H), 3.00 (dd, *J*=3.81, 7.29 Hz, 2H), 2.83-2.69 (m, 1H), 0.95 (dd, *J*=6.33, 8.99 Hz, 6H) ppm. **^{13}C NMR** (101MHz, $CDCl_3$) δ = 140.5, 134.3, 132.4, 128.8, 128.6, 127.9, 127.5, 127.2, 53.3, 51.9, 48.3, 22.8, 22.7 ppm. **HRMS** (ESI) m/z calcd. For $C_{17}H_{22}NS^+$ $[M + H]^+$ 272.1467, found 272.1462.

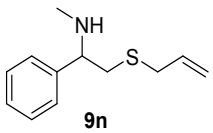
***N*-ethyl-2-phenyl-2-(phenylthio)ethan-1-amine (8m)**



8m

^{1}H NMR (400 MHz, $CDCl_3$) d 7.25-7.10 (m, 14H), 4.34 (t, J = 7.34 Hz, 1H), 3.03 (dd, *J*=3.07, 7.29 Hz, 2H), 2.66-2.55 (m, 2H), 1.23-1.15 (m, 6H) ppm. **^{13}C NMR** (101MHz, $CDCl_3$) δ = 140.5, 134.5, 132.3, 128.7, 128.6, 127.9, 127.4, 127.1, 54.3, 53.3, 43.6, 15.2 ppm **HRMS** (ESI) m/z calcd. For $C_{16}H_{20}NS^+$ $[M + H]^+$ 258.1311, found 258.1305.

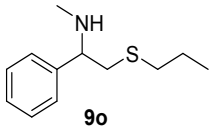
2-(Allylthio)-*N*-methyl-1-phenylethan-1-amine (9n)



9n

^{1}H NMR (400 MHz, CD_3OD) δ 7.26 (d, *J*=1.10 Hz, 2H), 7.23-7.17 (m, 3H), 5.70-5.58 (m, 1H), 5.02-4.92 (m, 2H), 3.61 (s, 1H), 2.93 (d, *J*=7.24 Hz, 2H), 2.68 (dd, *J*=3.21, 6.88 Hz, 2H), 2.16 (s, 3H) ppm. **^{13}C NMR** (101 MHz, CD_3OD) δ 135.8, 133.4, 129.8, 129.1, 128.9, 117.8, 65.3, 38.2, 35.7, 34.1 ppm. **HRMS** (ESI) m/z calcd. For $C_{12}H_{18}NS^+$ $[M + H]^+$ 208.1154, found 208.1149.

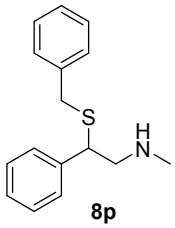
***N*-Methyl-1-phenyl-2-(propylthio)ethan-1-amine (9o)**



9o

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.25 (m, 4H), 7.24-7.14 (m, 3H), 3.55 (dd, *J*=4.59, 9.26 Hz, 1H), 2.83-2.72 (m, 1H), 2.72-2.60 (m, 1H), 2.45-2.35 (m, 2H), 2.26-2.22 (m, 3H), 1.59-1.45 (m, 2H), 0.90 (t, *J*=7.34 Hz, 3H) ppm. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.7, 128.5, 127.1, 64.2, 40.5, 34.6, 34.5, 23.0, 13.4 ppm. **HRMS** (ESI) m/z calcd. For $C_{12}H_{18}NS^+$ $[M + H]^+$ 210.1311, found 210.1305.

2-(benzylthio)-*N*-methyl-2-phenylethan-1-amine (8p)



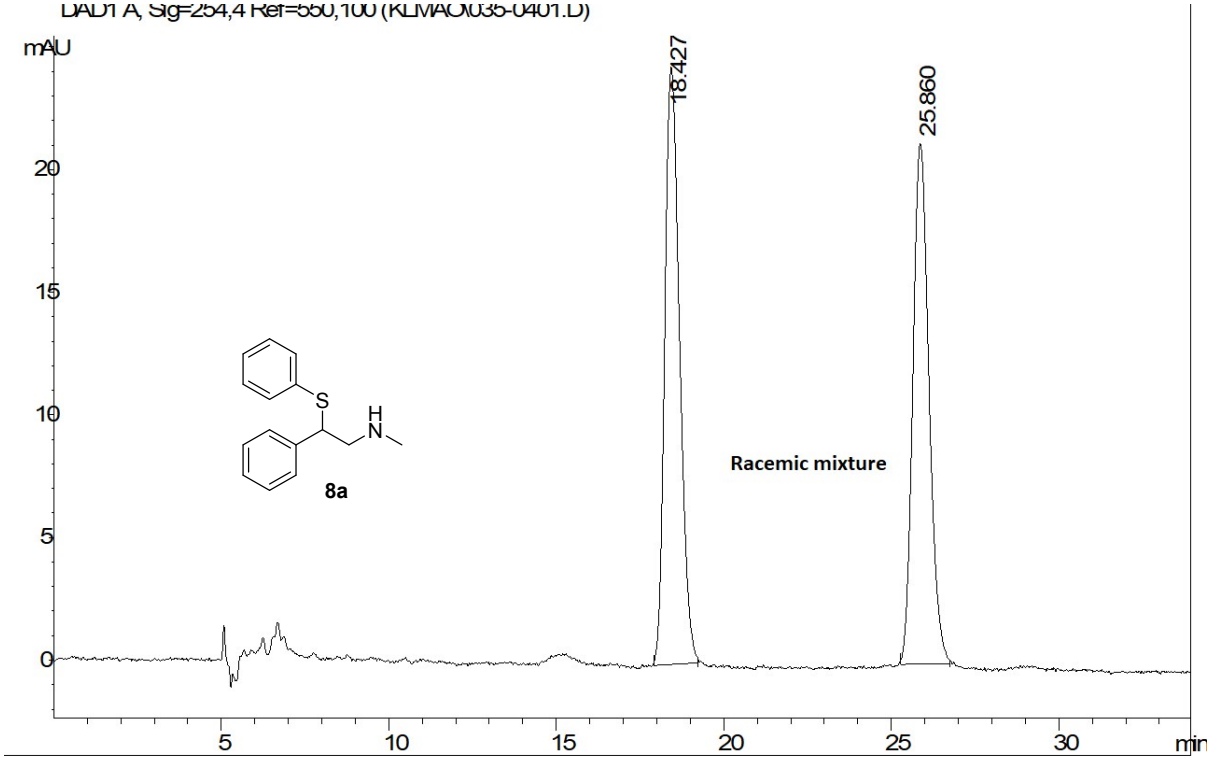
8p

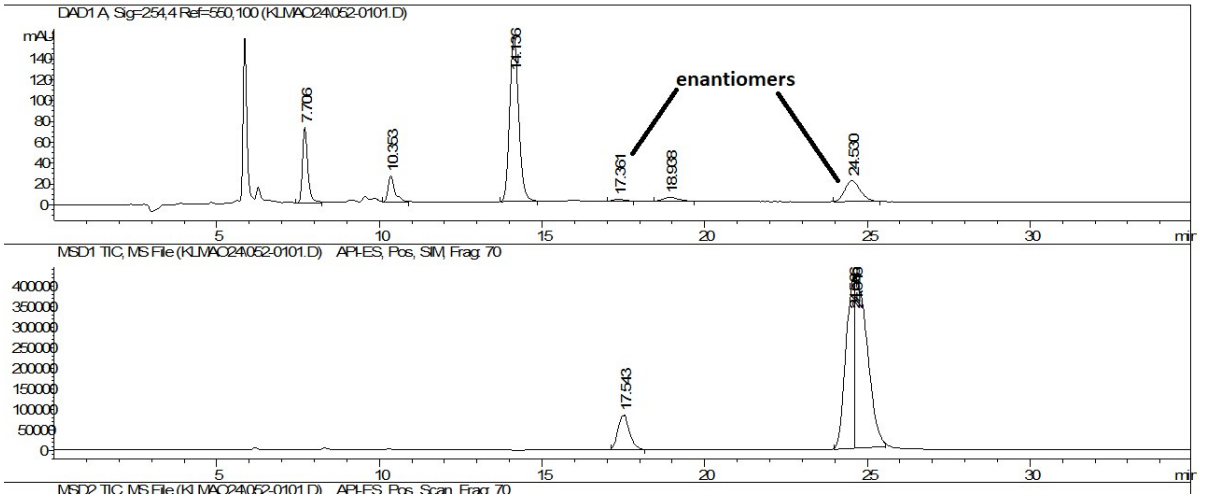
^{1}H NMR (400MHz, CD_3OD) δ = 7.38-7.30 (m, 4H), 7.30-7.18 (m, 6H), 3.87 (t, *J*=7.5 Hz, 1H), 3.64-3.43 (m, 2H), 2.88 (dd, *J*=0.8, 7.5 Hz, 2H), 2.26 (s, 3H) ppm. **^{13}C NMR** (101 MHz, CD_3OD) δ 142.1, 139.8, 130.2, 129.9, 129.6, 129.4, 128.8, 128.2, 57.3, 49.7, 36.2, 35.8 ppm. **HRMS** (ESI) m/z calcd. For $C_{16}H_{20}NS^+$ $[M + H]^+$ 258.1311, found 258.1307.

General procedure for the enzymatic kinetic resolution of 1,2-mercaptoamines 8

In a 15 mL Falcon tube, the required 1,2-mercaptoamine **8** (0.02 mmol) in DMF (10 μL) was added to a potassium phosphate buffer solution (750 μL, 1 M, pH = 7.8). The pH of the solution was adjusted to 7.8 by addition of NaOH. Cell pellet from *E. coli* cultures (100 mg) containing MAO-N D9 was added to the solution. The tube was placed in a shaking incubator and shaken at 37 °C and 250 rpm. Reactions were monitored by chiral phase HPLC. Upon completion of the reaction (typically after 24 h), AcOEt (30 mL) was added to the reaction mixture. The 1,2-mercaptoamine **8** were extracted by centrifugation (3184 x g, 5 min.) and the aqueous phase was extracted again with AcOEt (20 mL). The combined organic phases were dried over $MgSO_4$ and concentrated under vacuum. The crude was analysed by LC-MS to determine the er (and ee %) of the biotransformation. The crude was finally purified on silica gel (3:7 Hexane:Ethyl acetate) to yield the enantioenriched products **8**.

Examples of HPLC spectra





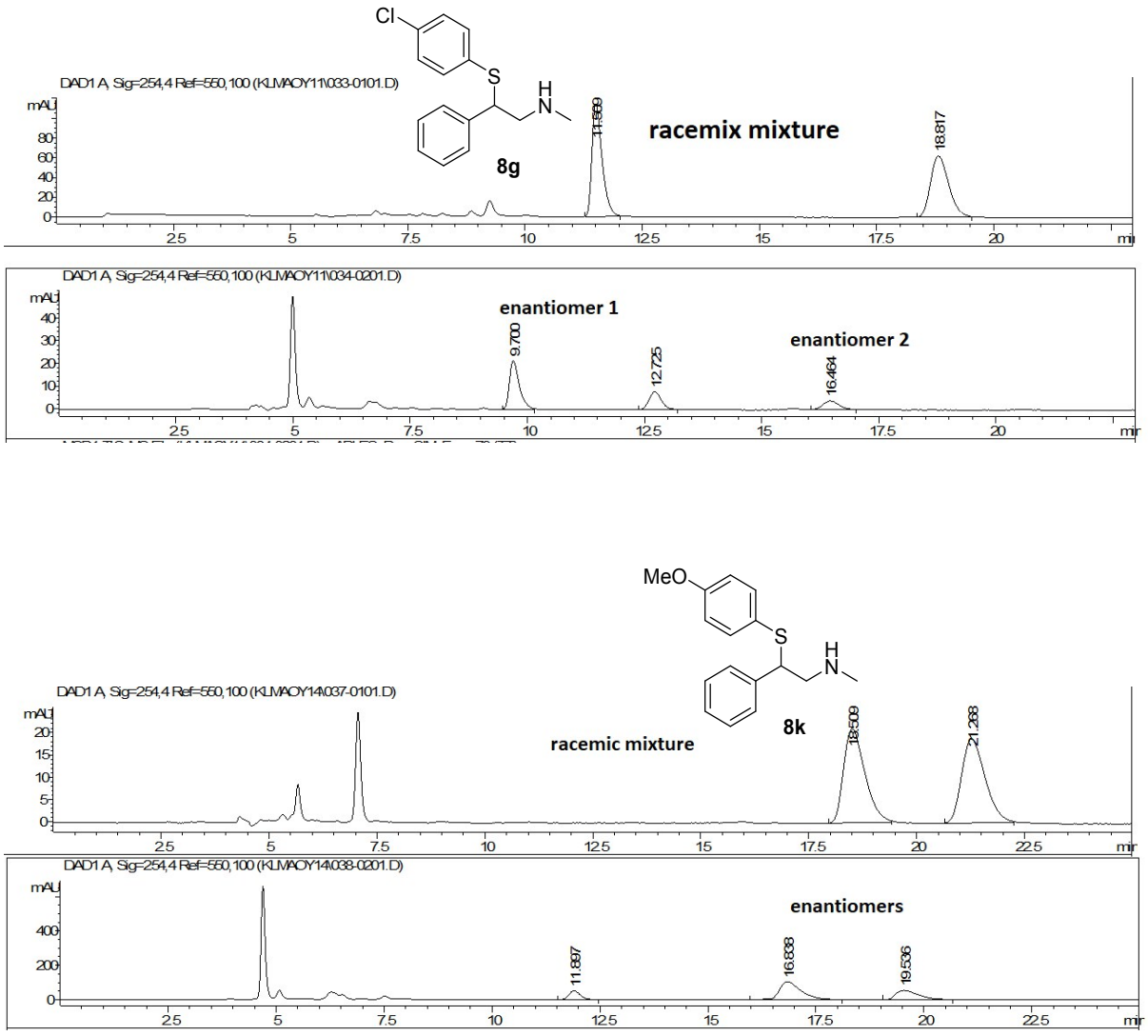
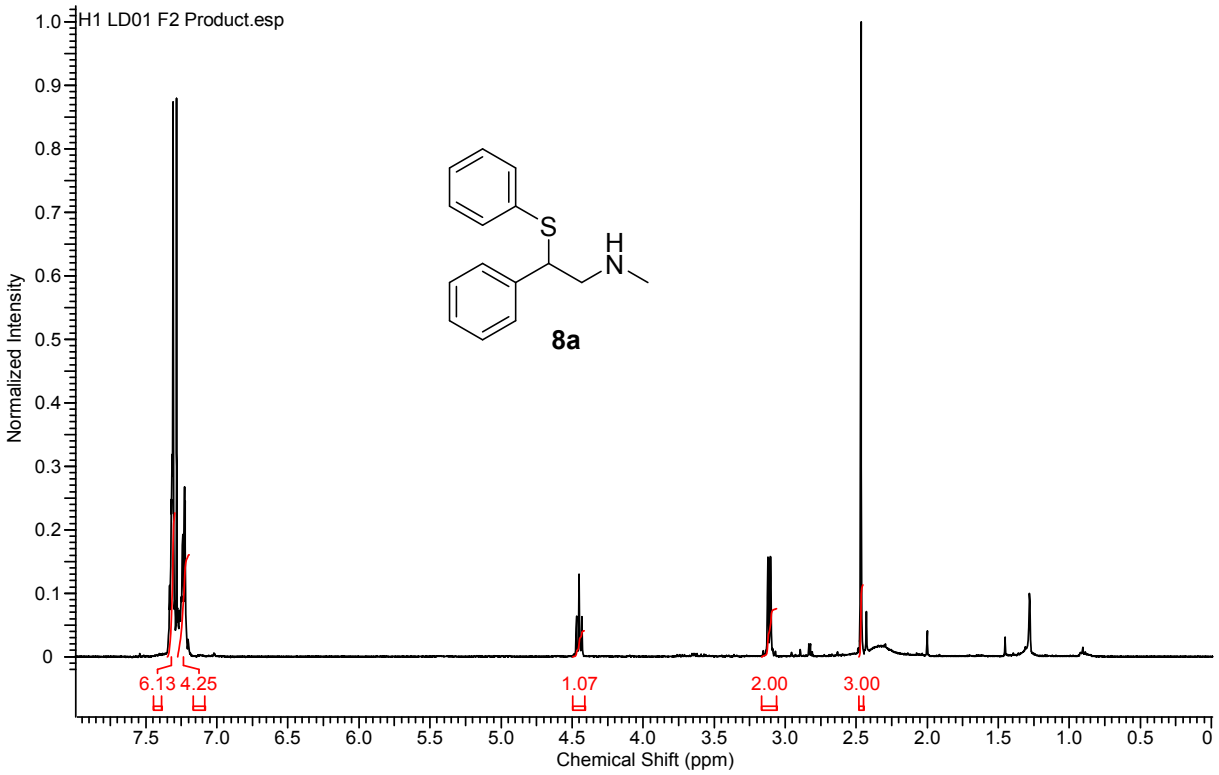
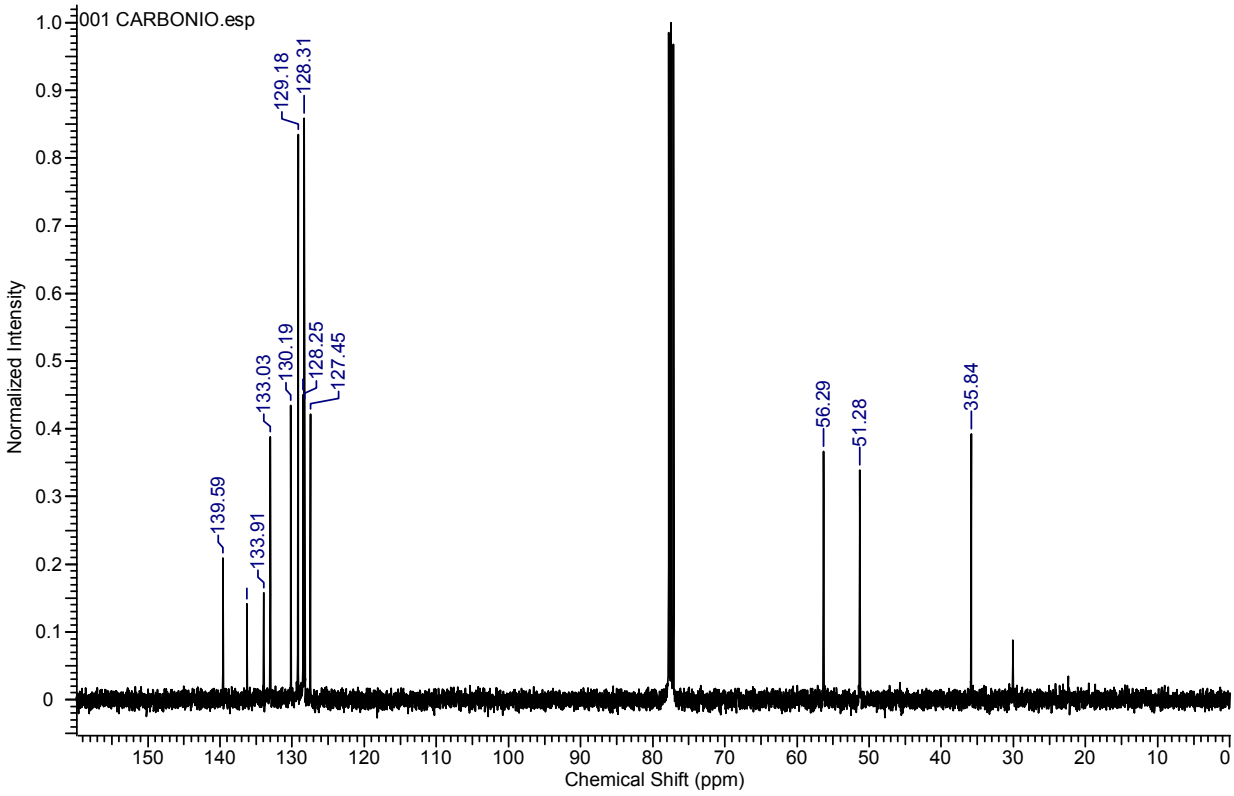


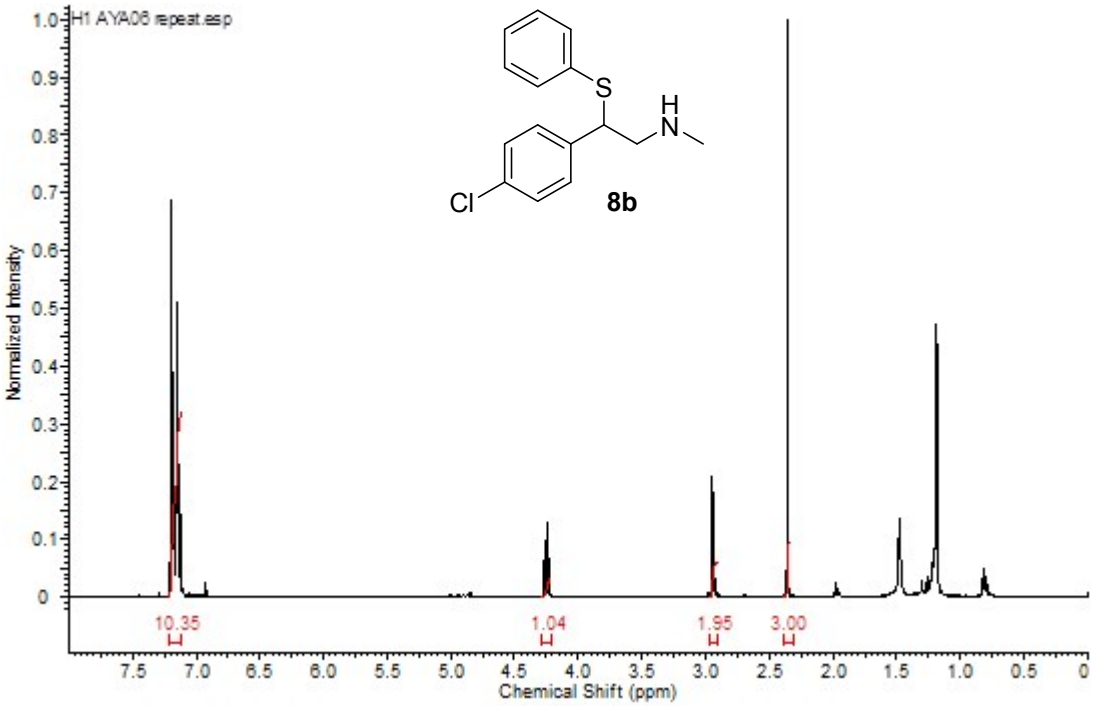
Table S1. Retention times, separation conditions, areas of the peaks and ee for the biocatalytic deracemization

Cmpd	Biocatalyst	Heptane: EtOH	Retention Time (min.)	ee (%)
8a	MAO-D9	60:40	17.4 and 24.5	90
8b	MAO-D9	60:40	14.7 and 25.9	21
8c	MAO-D9	60:40	7.3 and 22.9	34
8d	MAO-D9	60:40	13.4 and 14.1	17
8g	MAO-D9	60:40	9.7 and 16.5	55
8k	MAO-D9	60:40	16.8 and 19.5	30
8l	MAO-D9	90:10	5.5 and 6.1	4
9n	MAO-D9	60:40	3.7 and 3.9	1
9o	MAO-D9	60:40	4.1 and 4.7	7
8p	MAO-D9	60:40	6.5 and 7.8	43

Copies of spectra

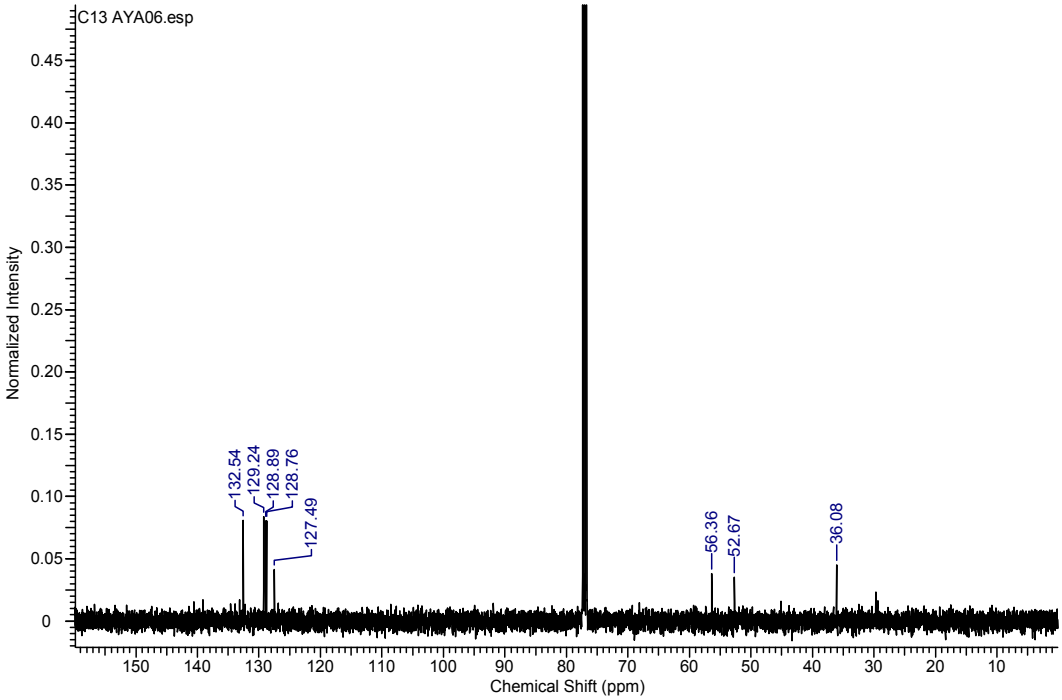


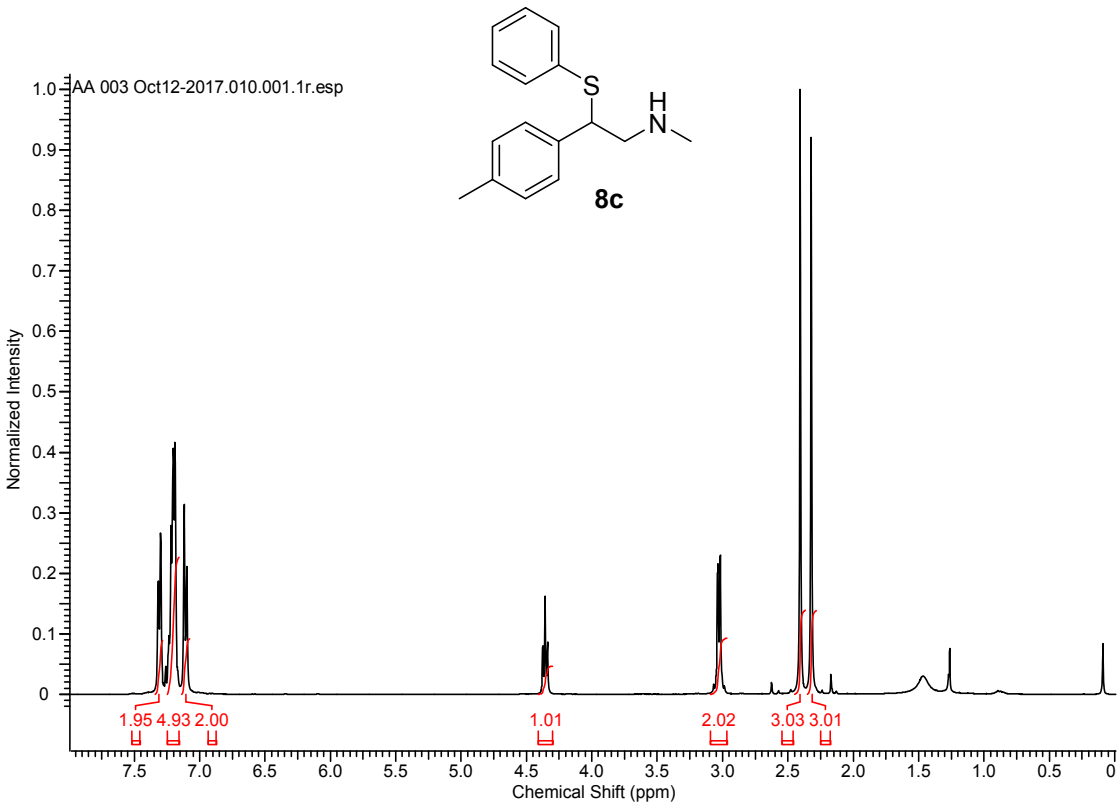

001 CARBONIO.esp
Normalized Intensity
Chemical Shift (ppm)
139.59
133.91
133.03
130.19
129.18
128.31
128.25
127.45
56.29
51.28
35.84

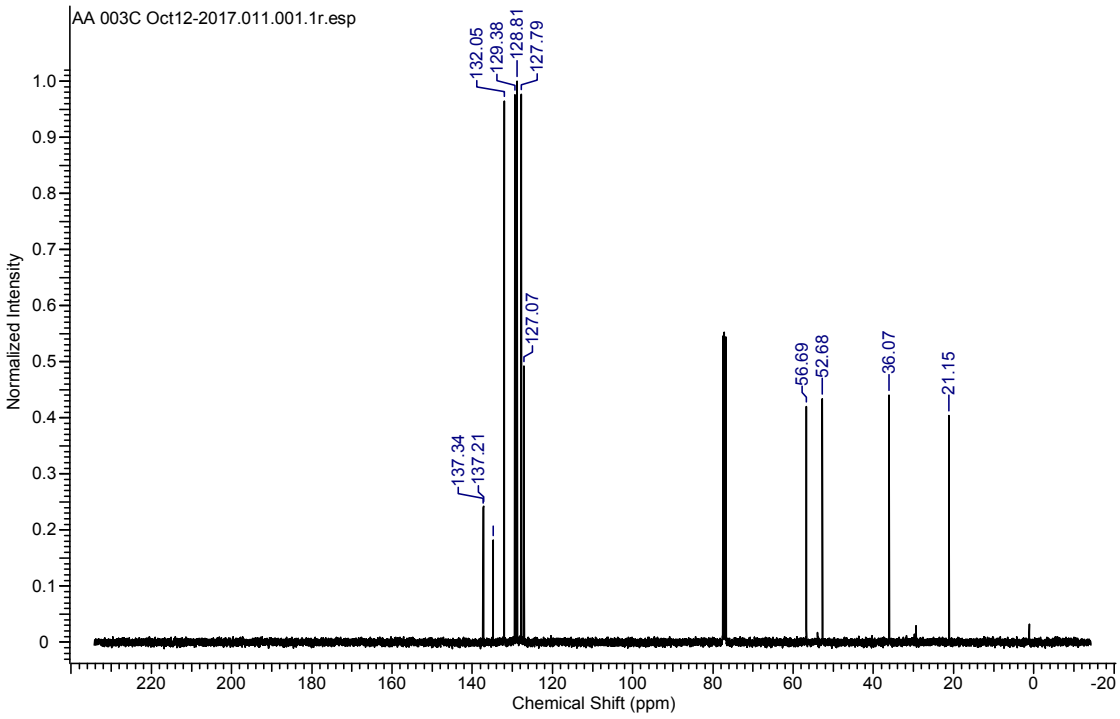

H1 AYA08 repeat.esp
Normalized Intensity
Chemical Shift (ppm)
10.35
1.04
1.95
3.00

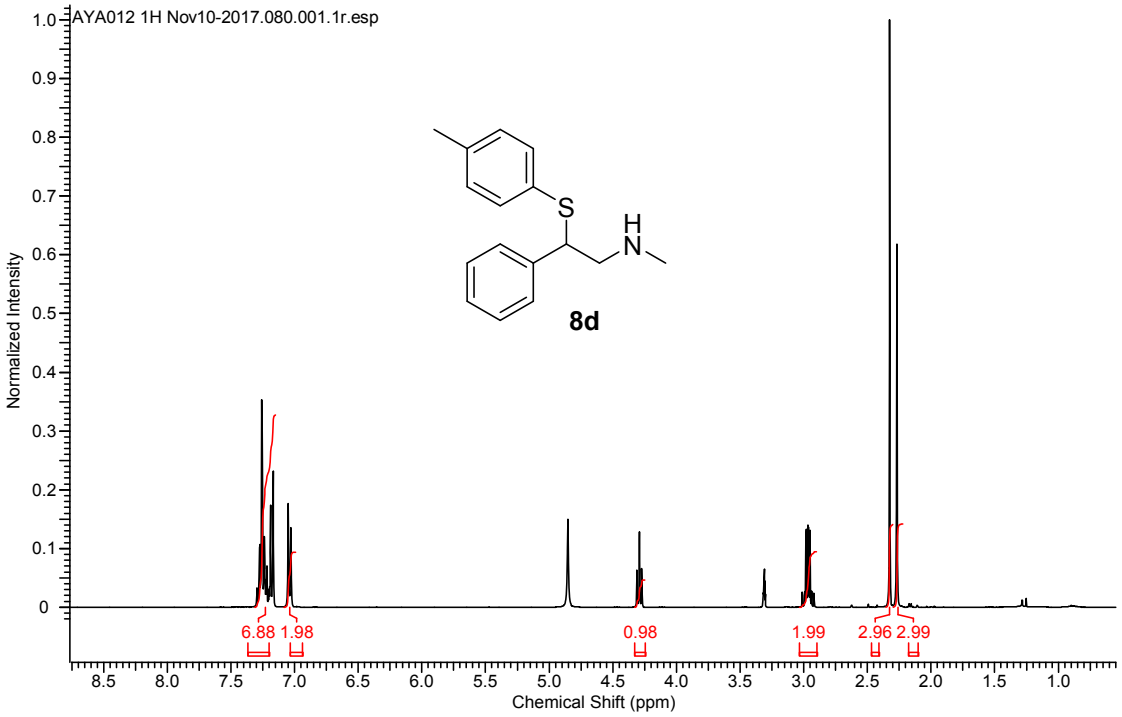
S
H
N
Cl

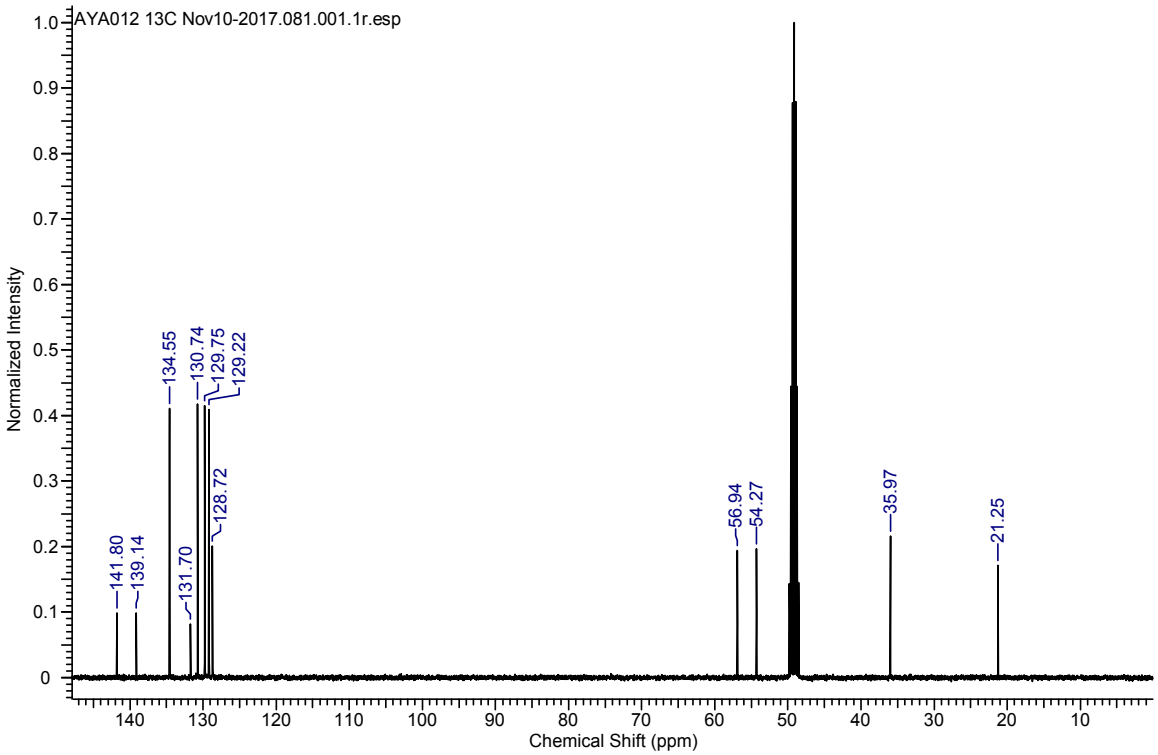
8b

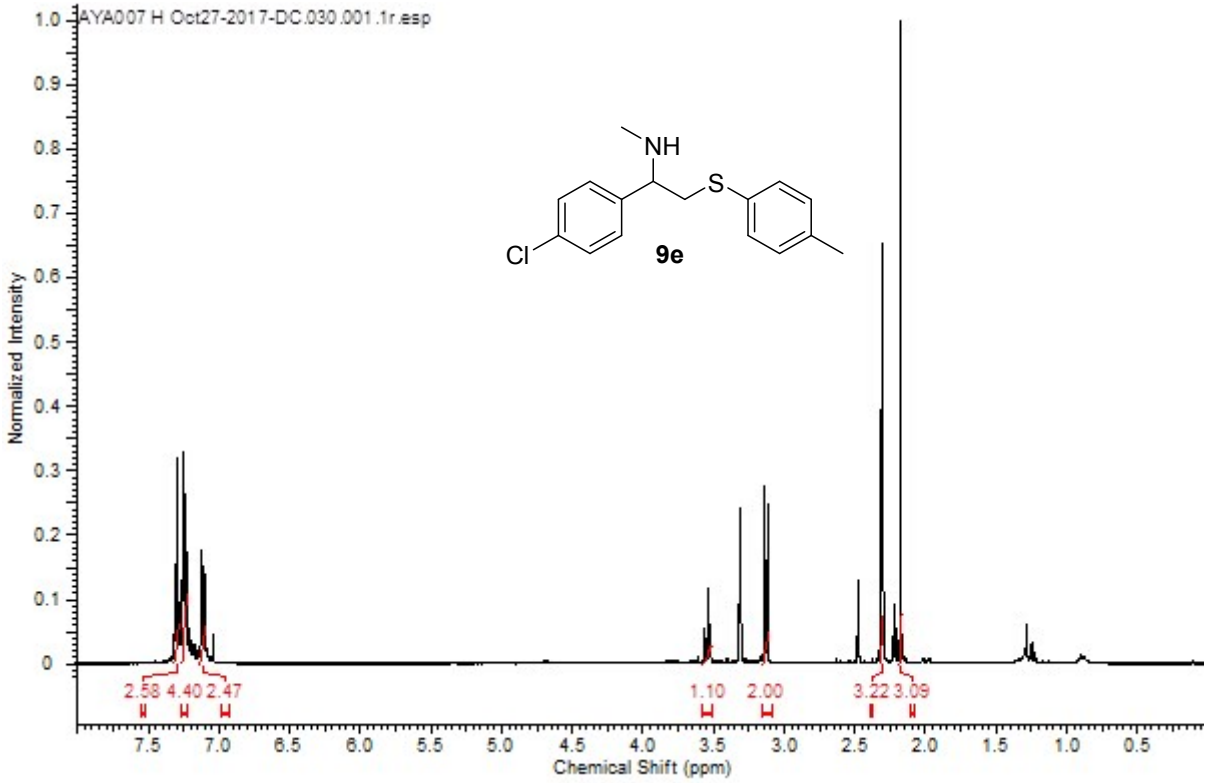

C13 AYA06.esp
Normalized Intensity
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
132.54
129.24
128.89
128.76
127.49
56.36
52.67
36.08
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

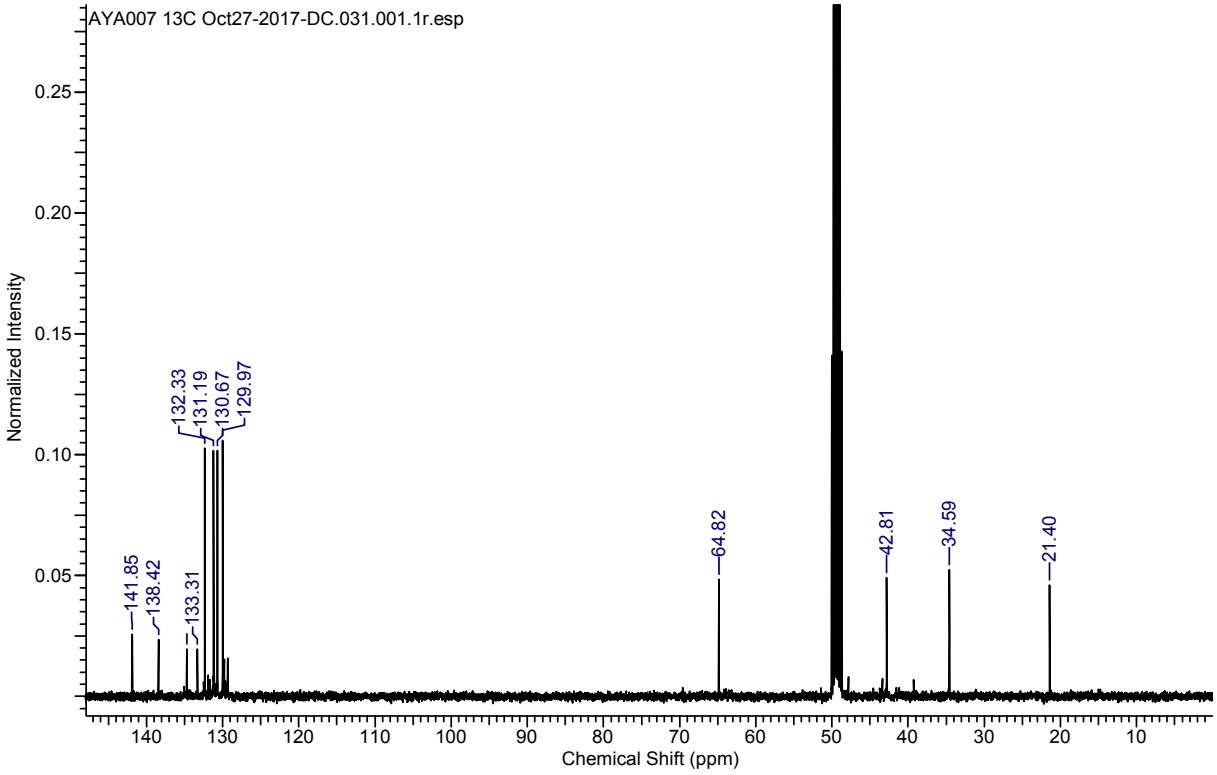

AA 003 Oct12-2017.010.001.1r.esp
S
H
N
8c
Normalized Intensity
1.95 4.93 2.00
1.01
2.02
3.03 3.01
Chemical Shift (ppm)

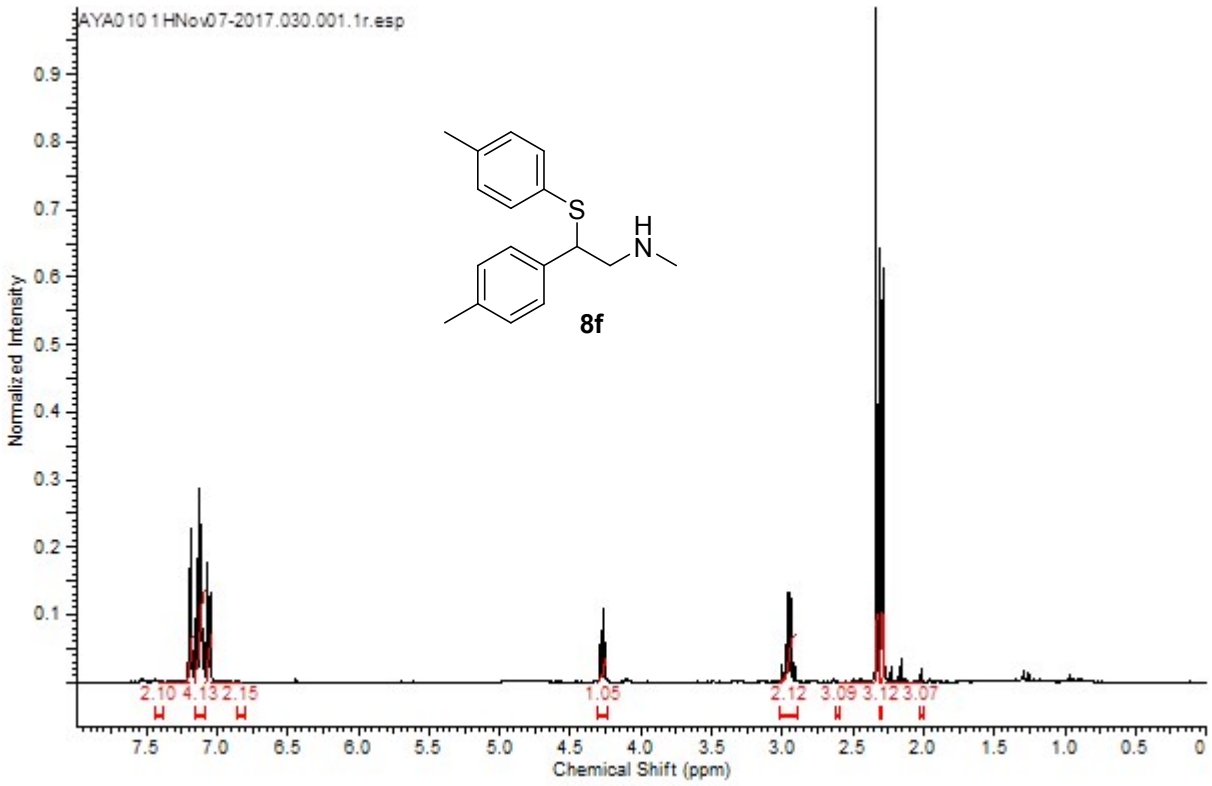

AA 003C Oct12-2017.011.001.1r.esp
132.05
129.38
128.81
127.79
127.07
137.34
137.21
56.69
52.68
36.07
21.15
Normalized Intensity
Chemical Shift (ppm)

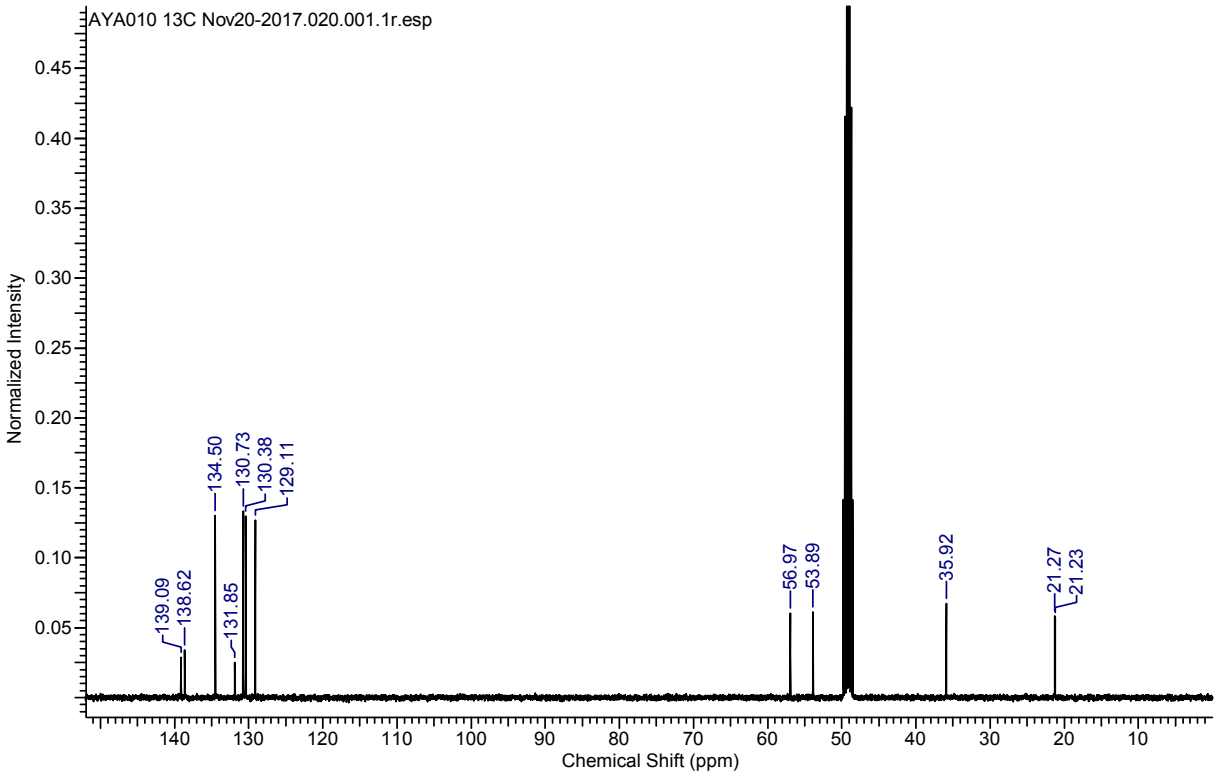

AYA012 1H Nov10-2017.080.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
6.88
1.98
0.98
1.99
2.96
2.99
S
H
N
8d

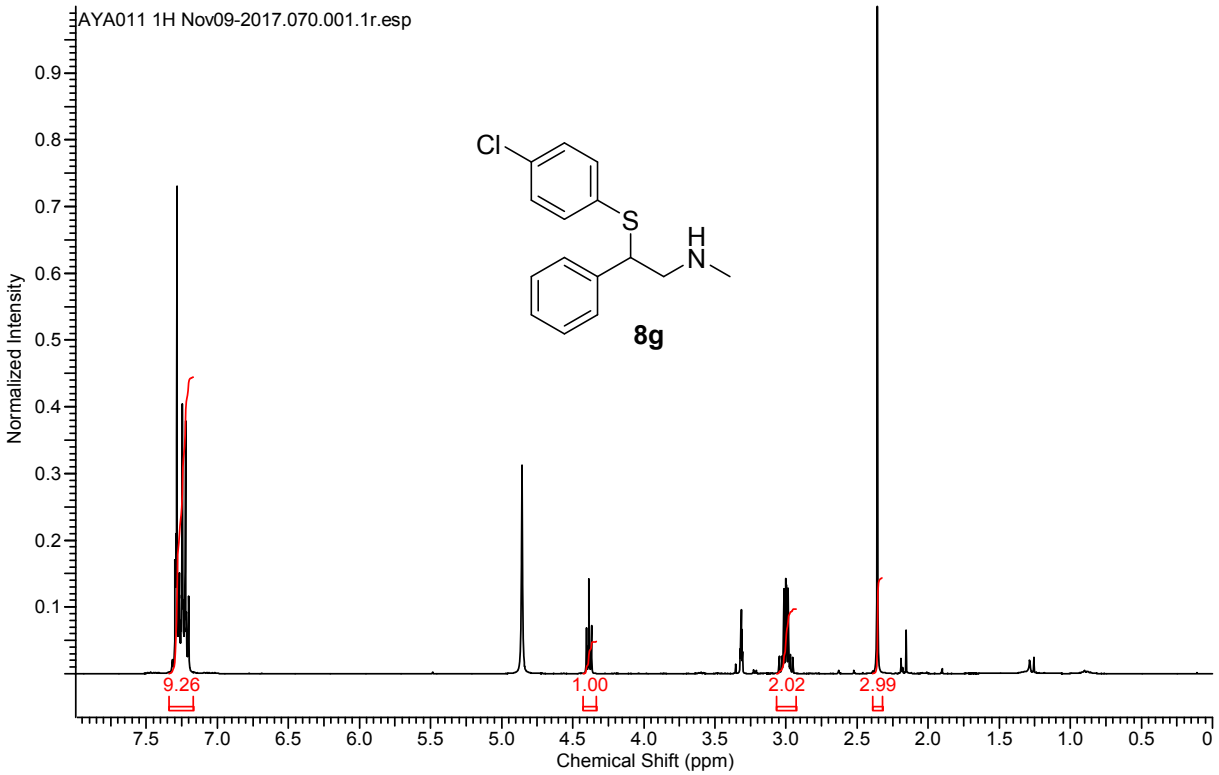

AYA012 13C Nov10-2017.081.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
141.80
139.14
134.55
131.70
130.74
129.75
129.22
128.72
56.94
54.27
35.97
21.25

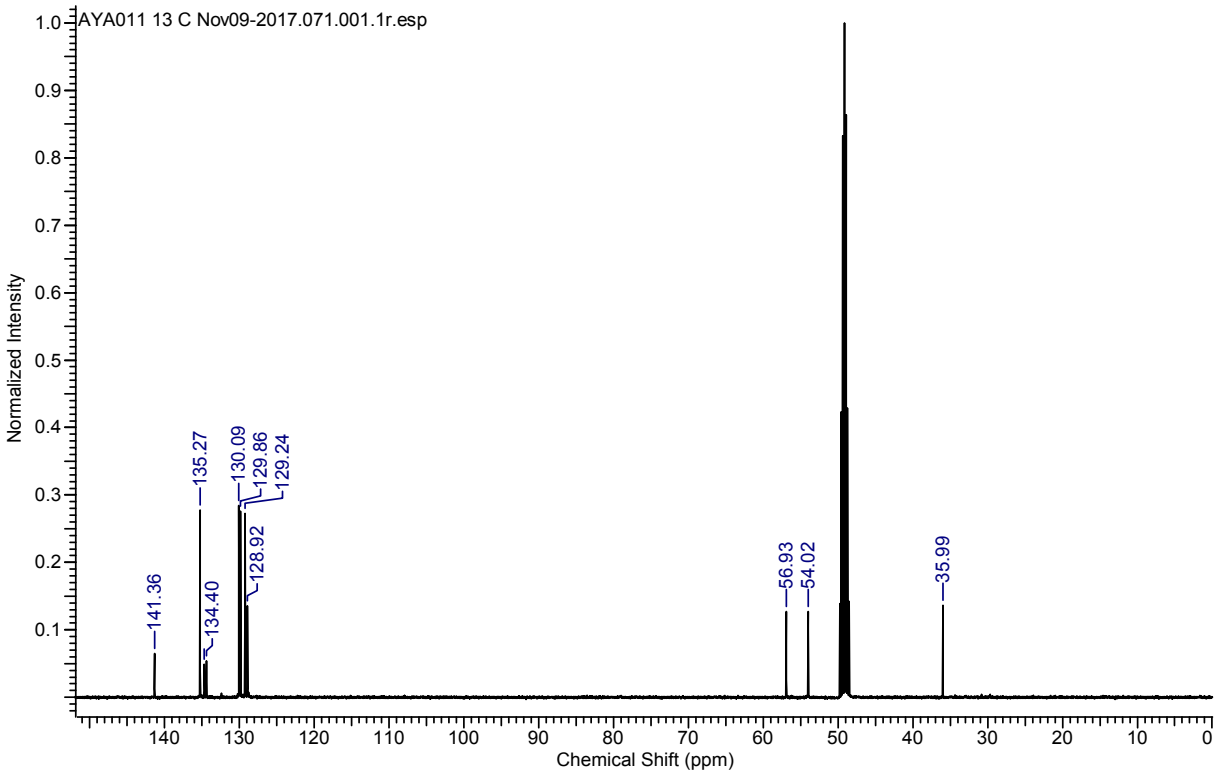

AYA007 H Oct27-2017-DC.030.001.1r.esp
NH
S
Cl
9e
Normalized Intensity
2.58 4.40 2.47
1.10 2.00
3.22 3.09
Chemical Shift (ppm)

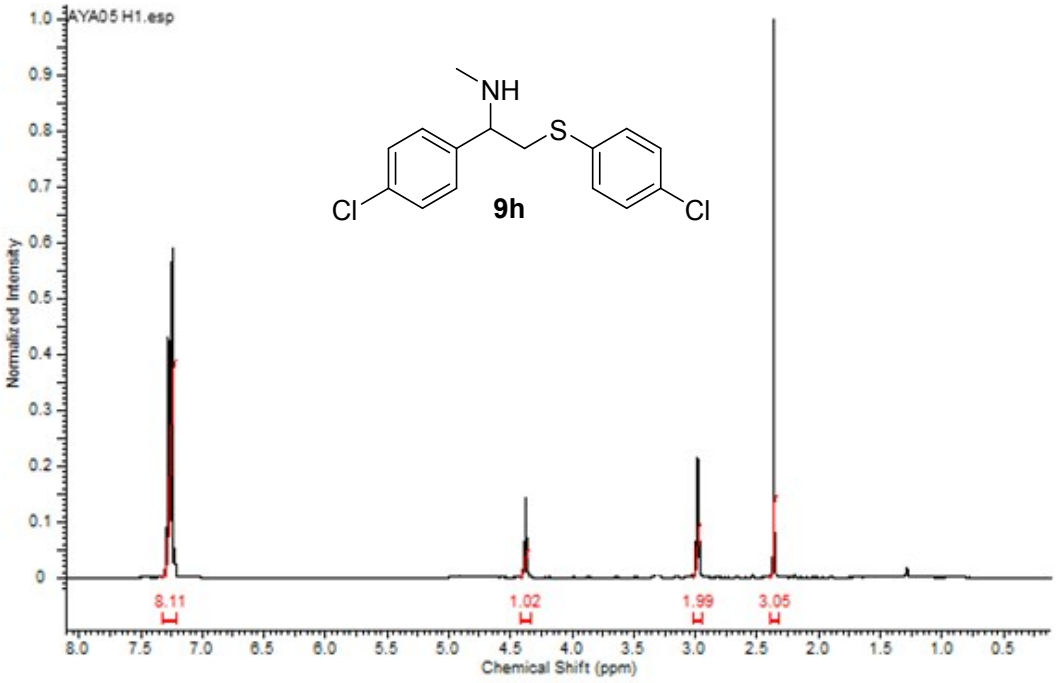

AYA007 13C Oct27-2017-DC.031.001.1r.esp
Normalized Intensity
132.33
131.19
130.67
129.97
141.85
138.42
133.31
64.82
42.81
34.59
21.40
Chemical Shift (ppm)

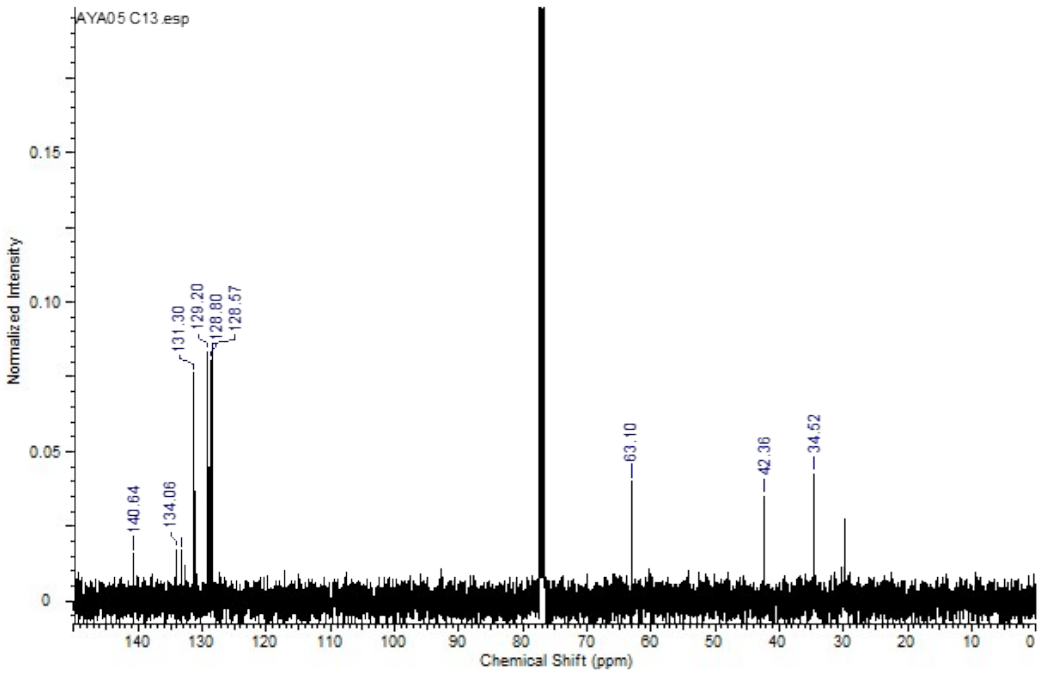

AYA010 1HNov07-2017.030.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
2.10 4.13 2.15
1.05
2.12 3.09 3.12 3.07
S
H
N
8f

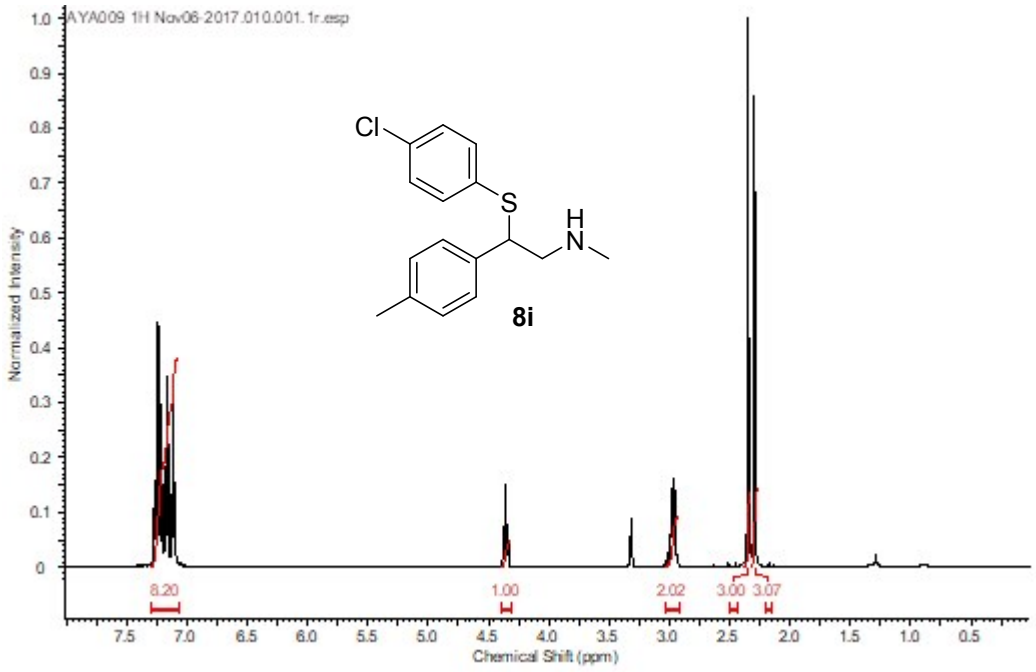

AYA010 13C Nov20-2017.020.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
139.09
138.62
131.85
134.50
130.73
130.38
129.11
56.97
53.89
35.92
21.27
21.23

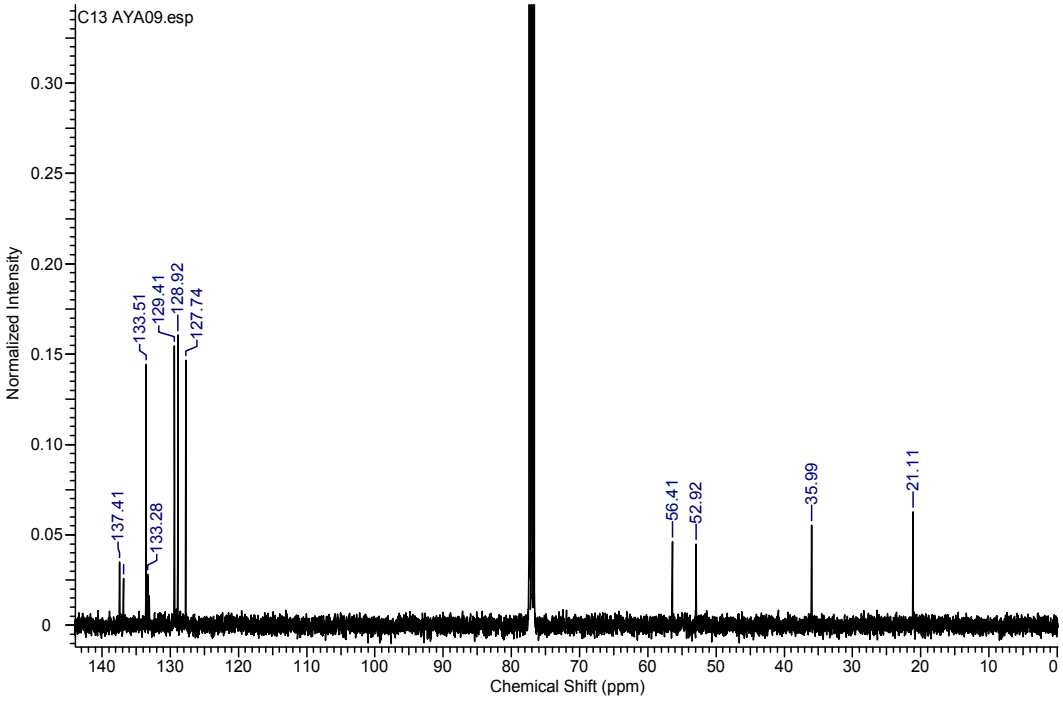

AYA011 1H Nov09-2017.070.001.1r.esp
Cl
S
H
N
8g
Normalized Intensity
9.26
1.00
2.02
2.99
Chemical Shift (ppm)

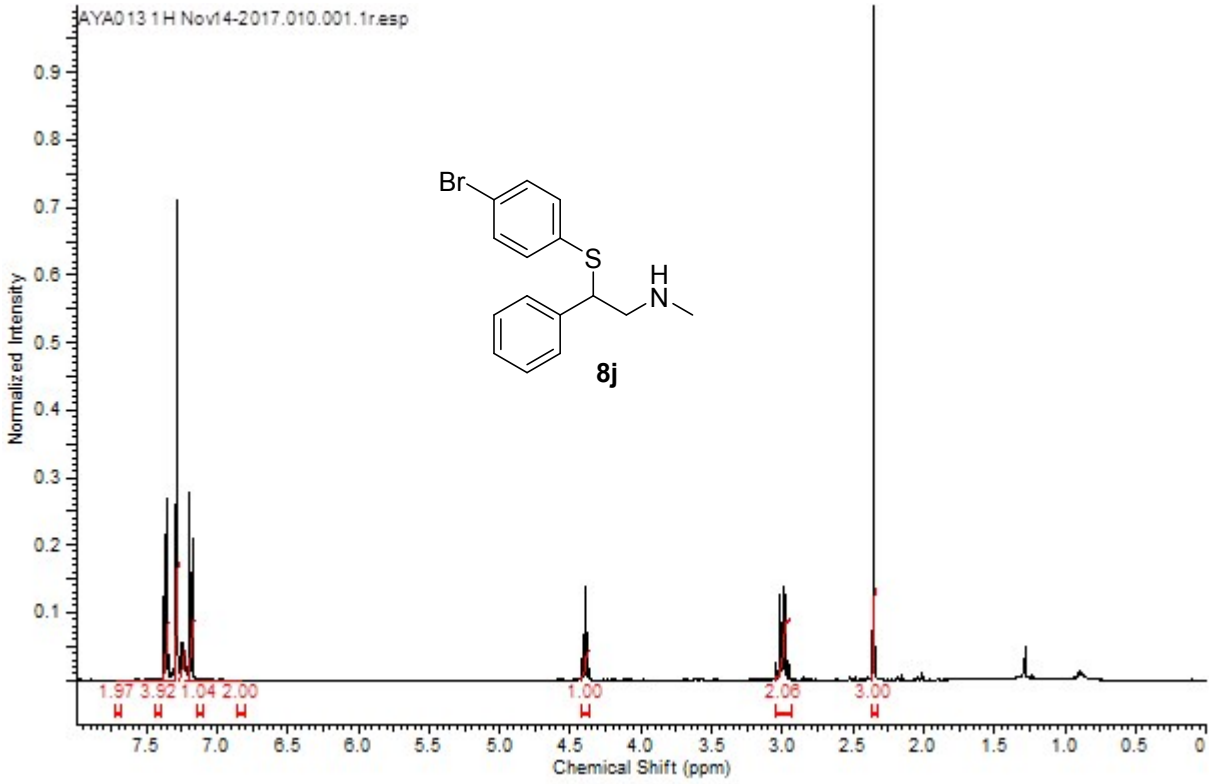

AYA011 13 C Nov09-2017.071.001.1r.esp
Normalized Intensity
141.36
135.27
134.40
130.09
129.86
129.24
128.92
56.93
54.02
35.99
Chemical Shift (ppm)

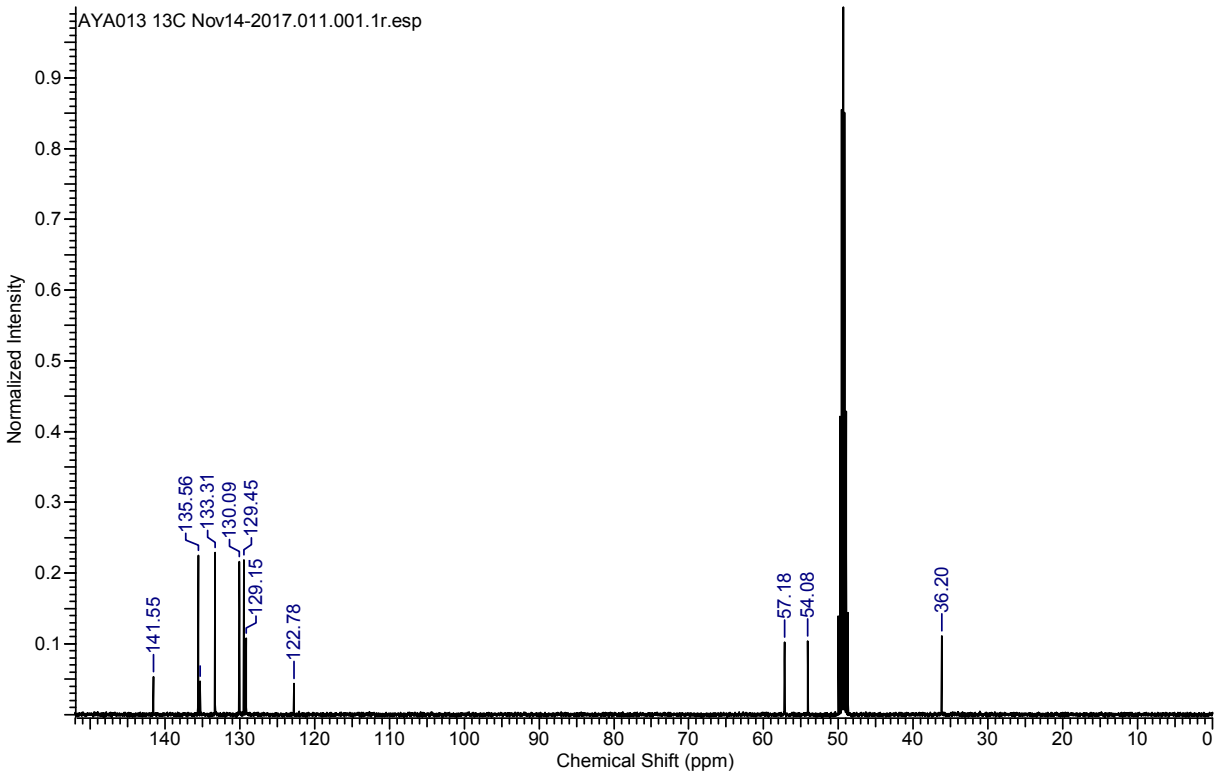

AYA05 H1.esp
NH
S
Cl
Cl
9h
Normalized Intensity
8.11
1.02
1.99
3.05
Chemical Shift (ppm)

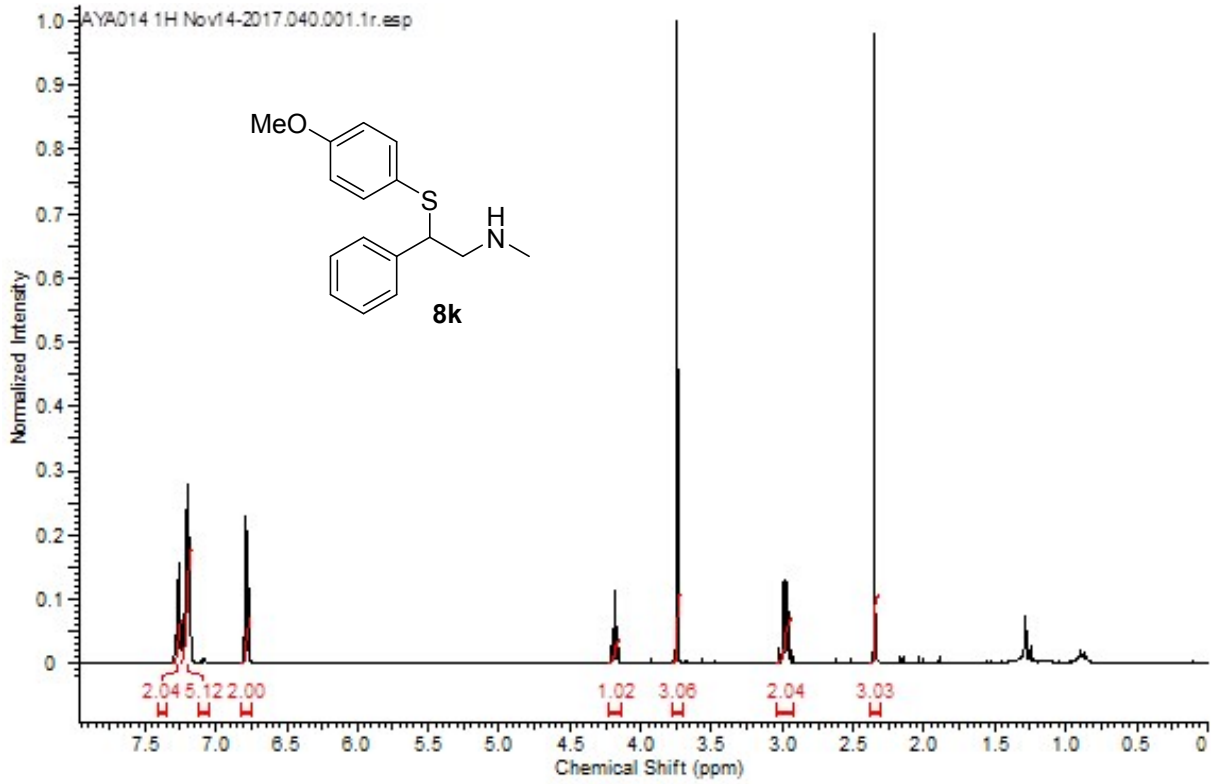

AYA05 C13.esp
Normalized Intensity
140.64
134.06
131.30
129.20
128.80
128.57
63.10
42.36
34.52
Chemical Shift (ppm)

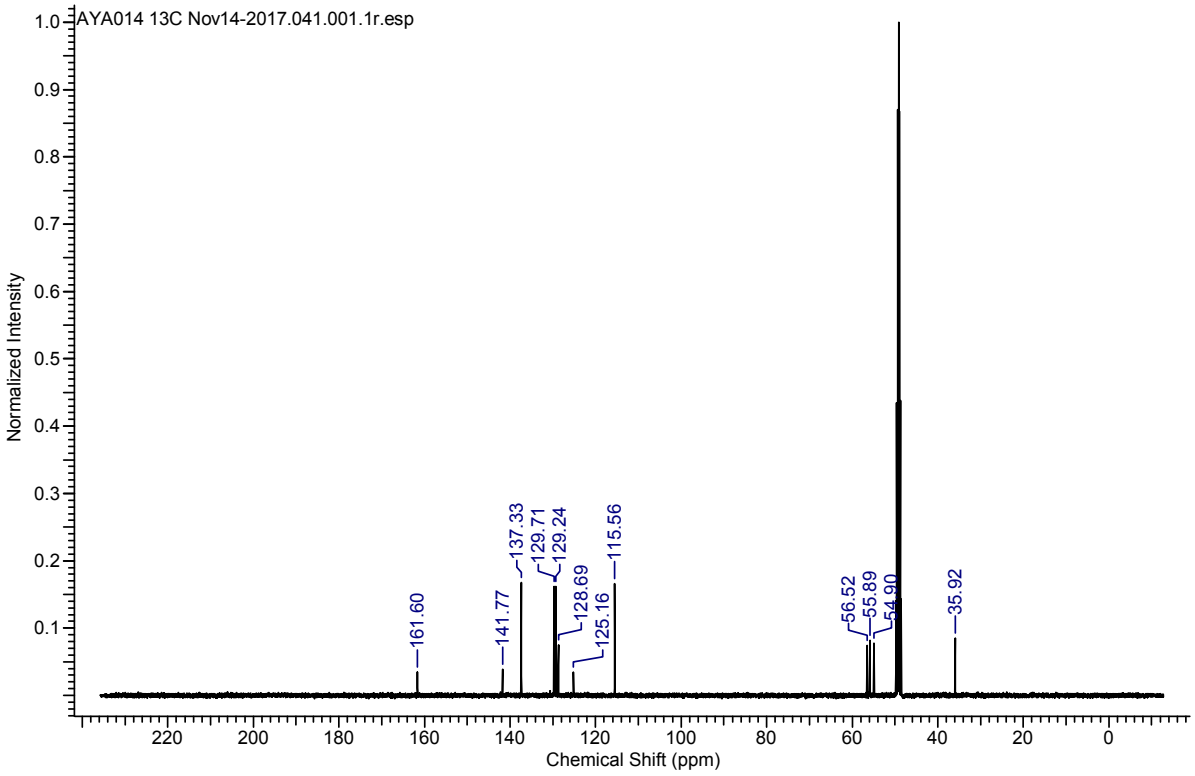

AYA009 1H Nov06-2017.010.001.1r.esp
Cl
S
H
N
8i
Normalized Intensity
8.20
1.00
2.02
3.00
3.07
Chemical Shift (ppm)

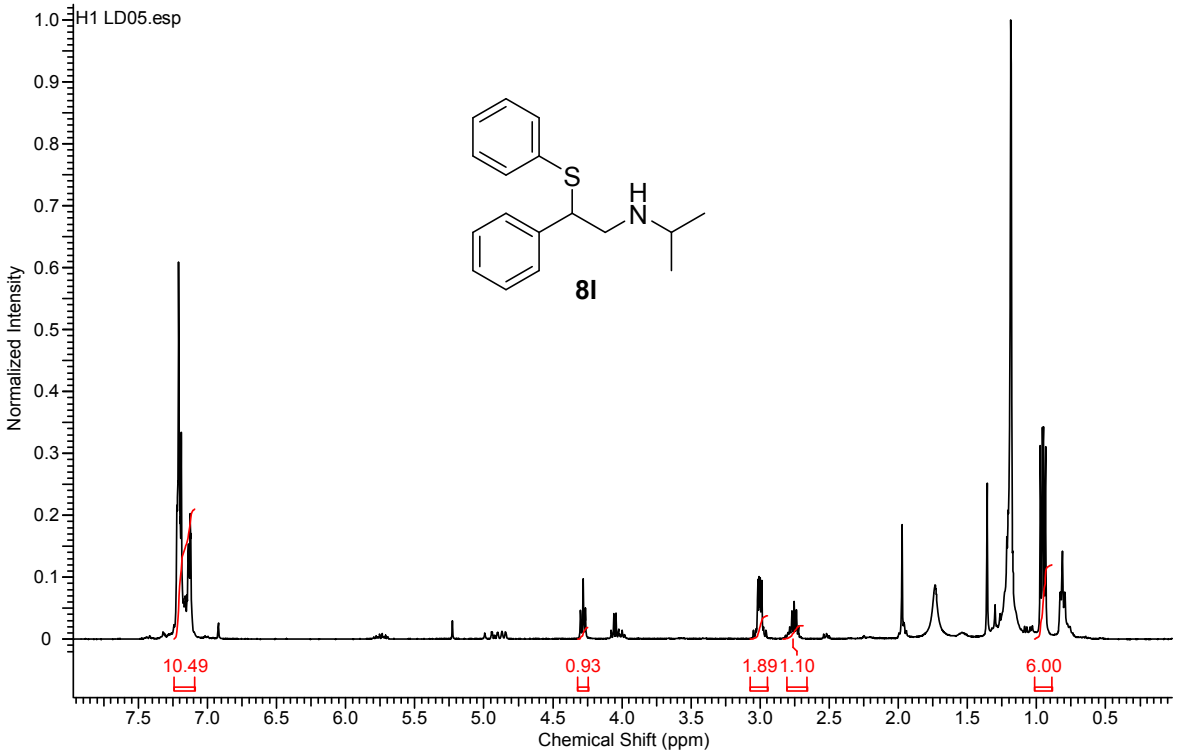

C13 AYA09.esp
Normalized Intensity
137.41
133.51
133.28
129.41
128.92
127.74
56.41
52.92
35.99
21.11
Chemical Shift (ppm)

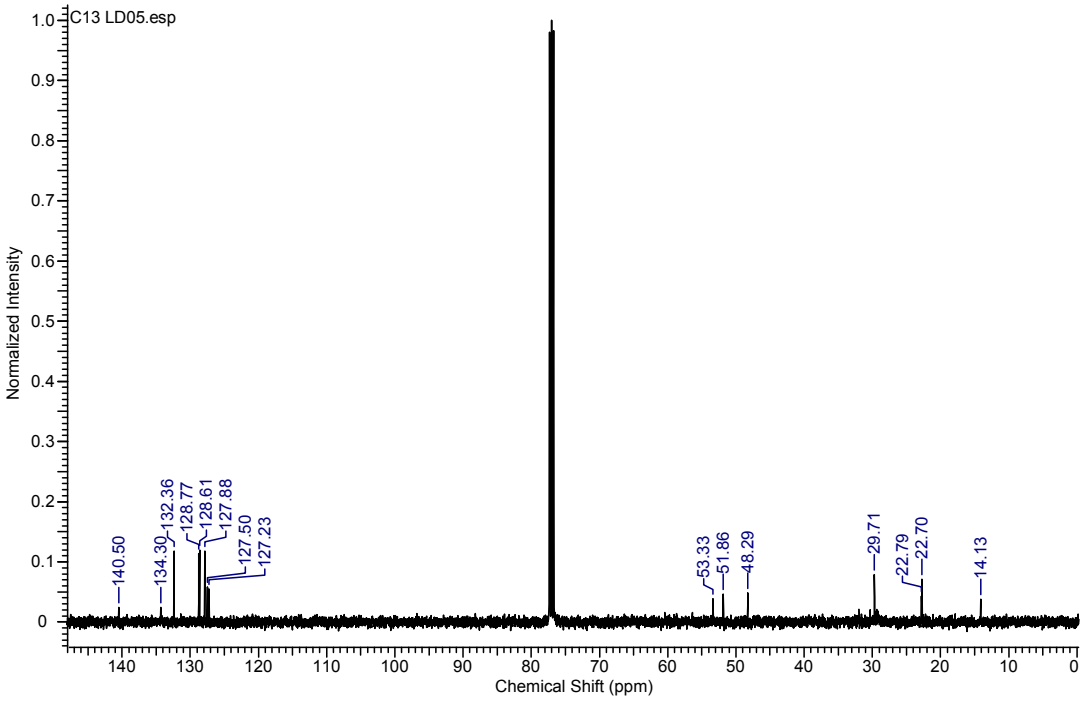

AYA013 1H Nov14-2017.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
1.97
3.92
1.04
2.00
1.00
2.06
3.00
Br
S
H
N
8j

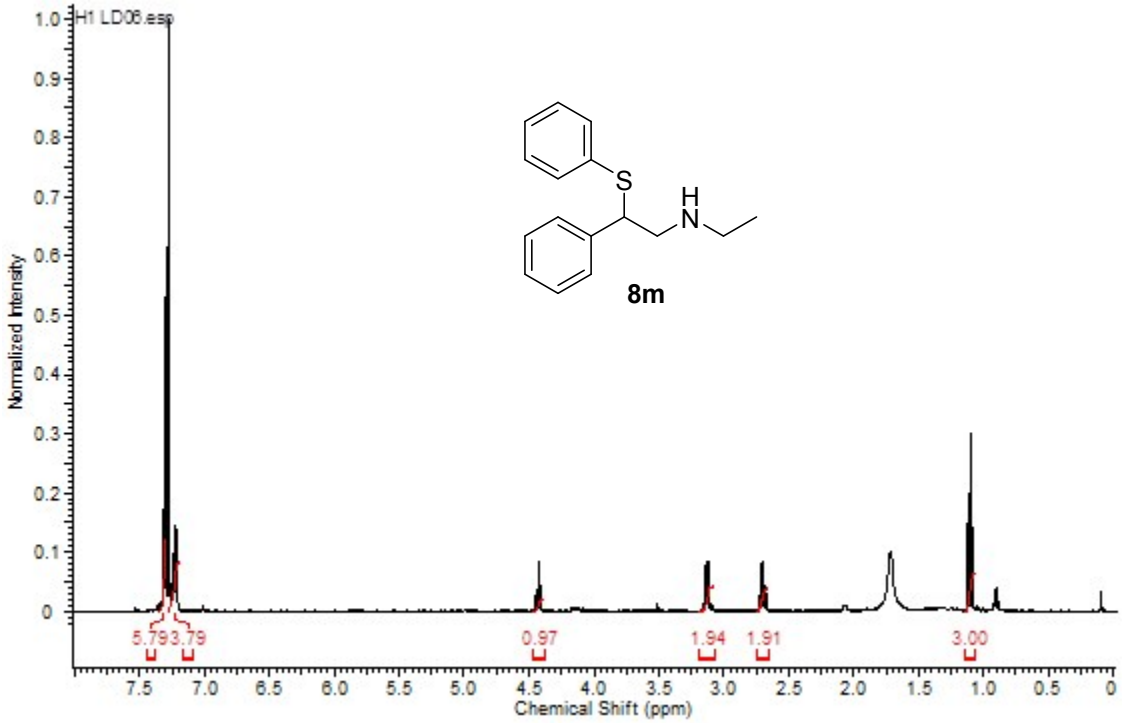

AYA013 13C Nov14-2017.011.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
141.55
135.56
133.31
130.09
129.45
129.15
122.78
57.18
54.08
36.20


AYA014 1H Nov14-2017.040.001.1r.esp
MeO
S
H
N
8k
Normalized Intensity
2.04
5.12
2.00
1.02
3.06
2.04
3.03
Chemical Shift (ppm)

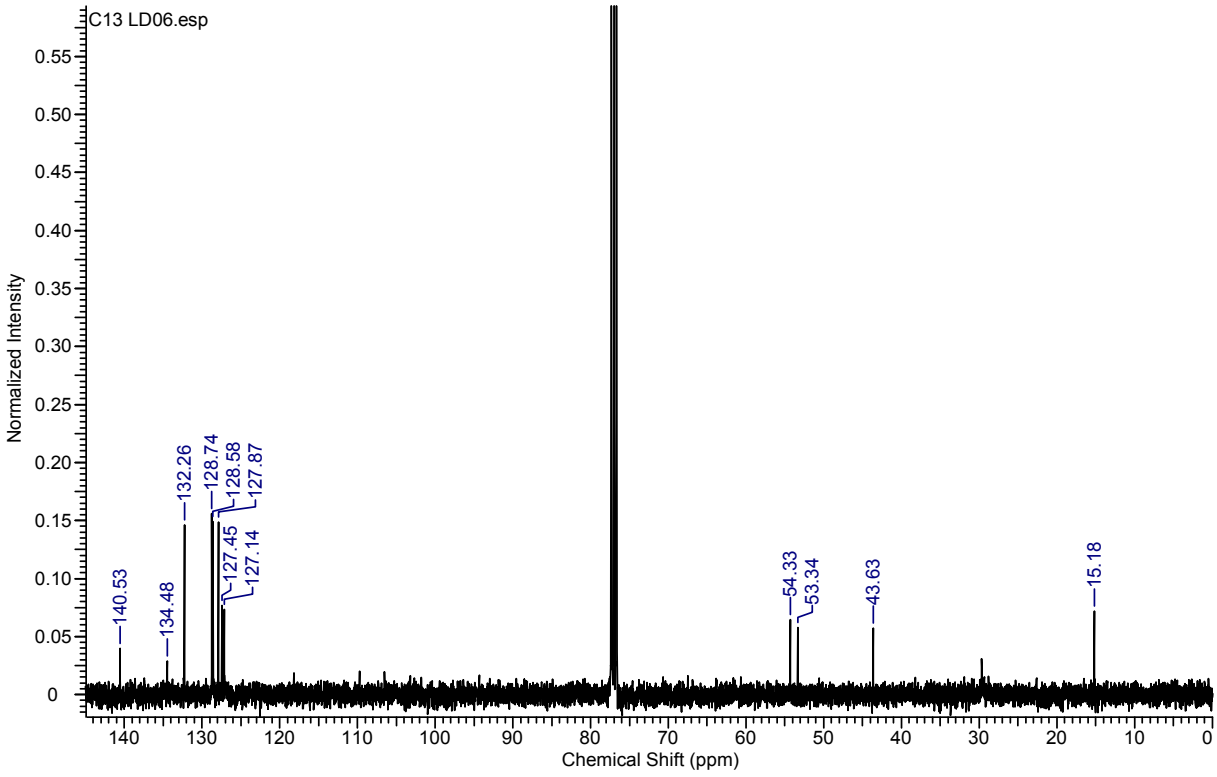

AYA014 13C Nov14-2017.041.001.1r.esp
Normalized Intensity
161.60
141.77
137.33
129.71
129.24
128.69
125.16
115.56
56.52
55.89
54.90
35.92
Chemical Shift (ppm)

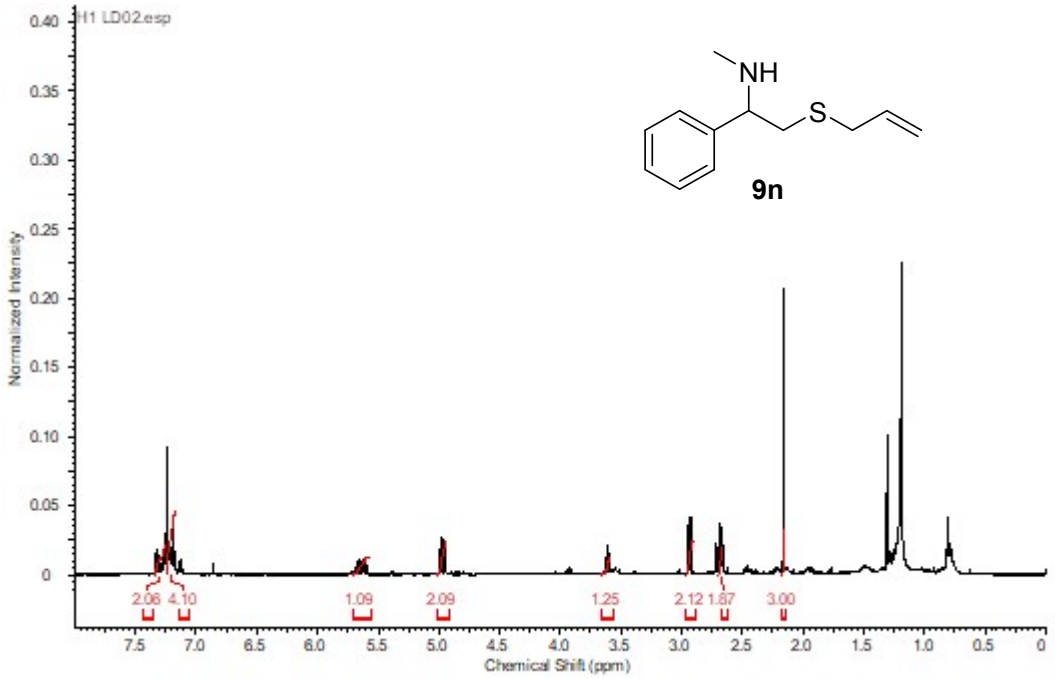

H1 LD05.esp
Normalized Intensity
Chemical Shift (ppm)
10.49
0.93
1.89
1.10
6.00
S
H
N
8l

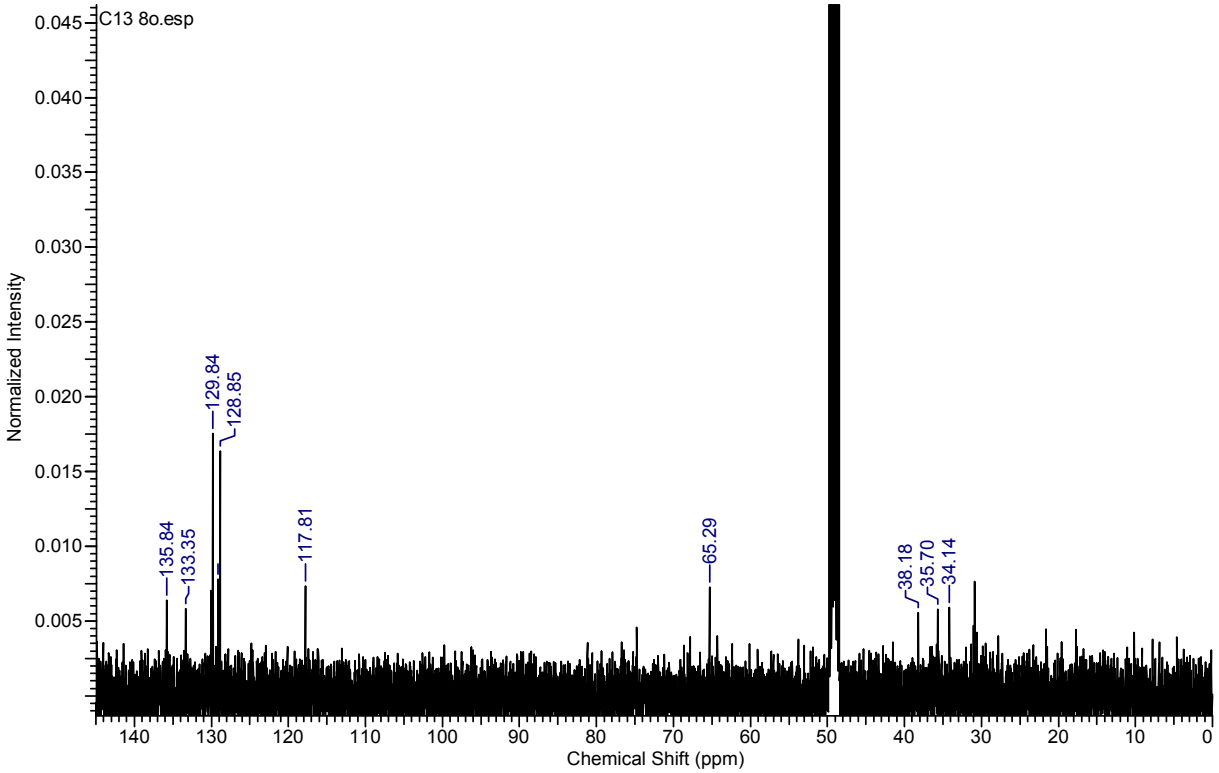

C13 LD05.esp
Normalized Intensity
Chemical Shift (ppm)
140.50
134.30
132.36
128.77
128.61
127.88
127.50
127.23
53.33
51.86
48.29
29.71
22.79
22.70
14.13

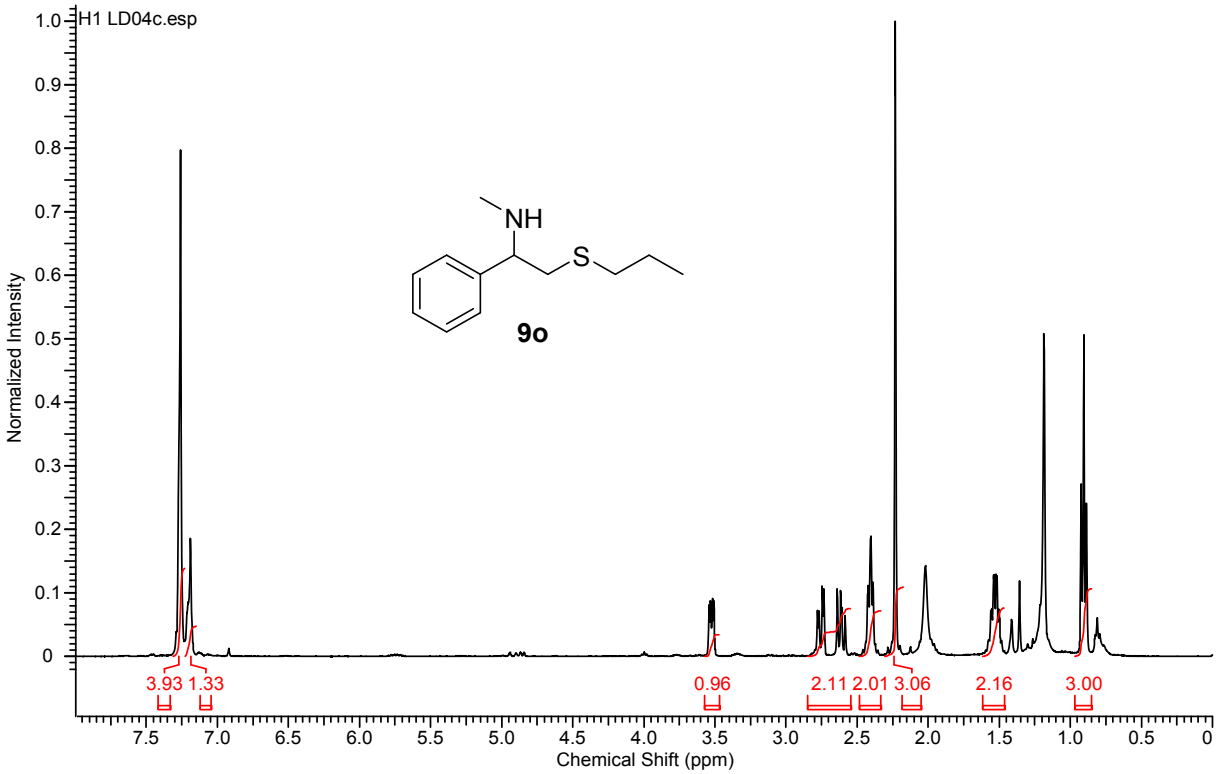

H1 LD06.esp
Normalized Intensity
Chemical Shift (ppm)
5.79
3.79
0.97
1.94
1.91
3.00
S
H
N

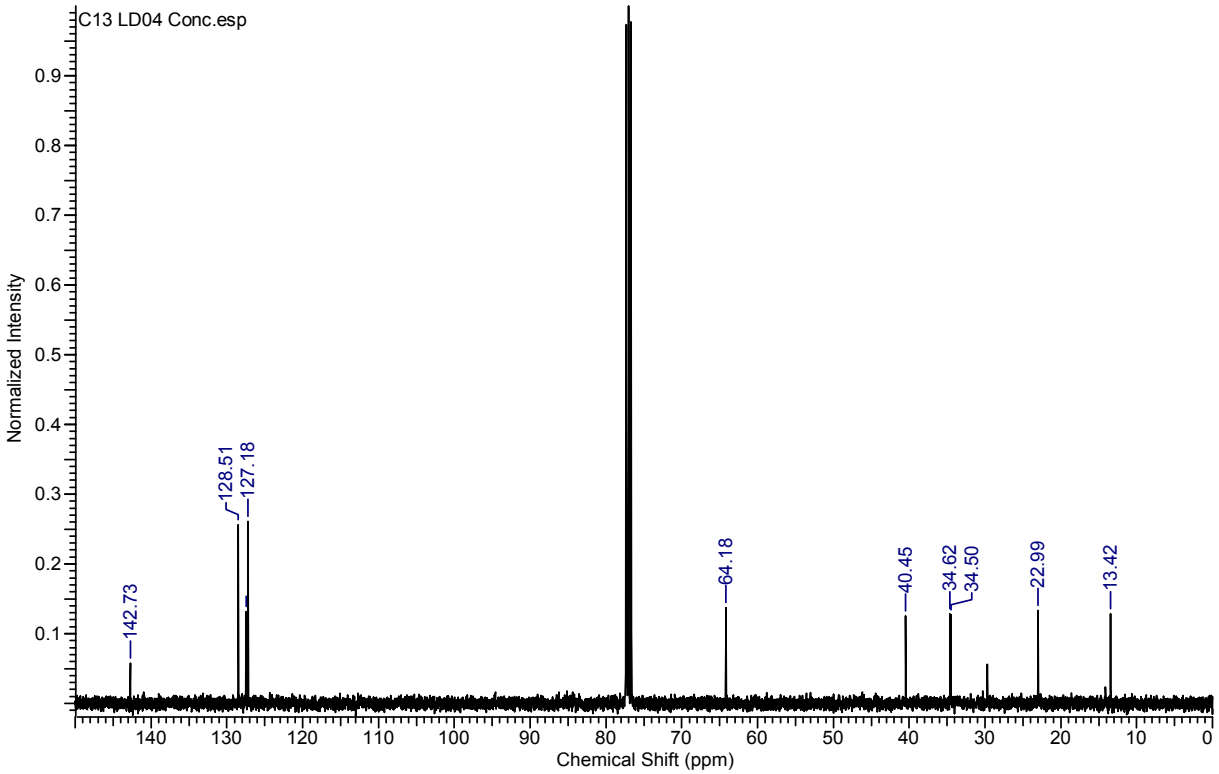
8m

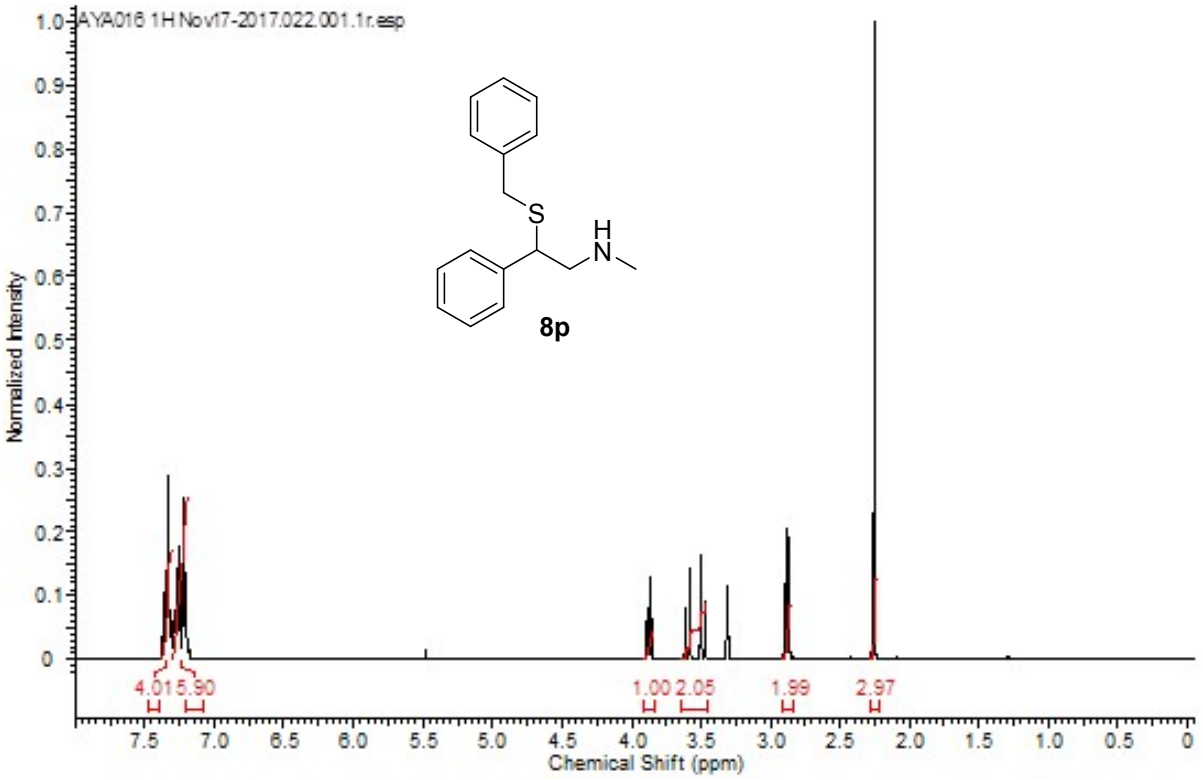

C13 LD06.esp
Normalized Intensity
Chemical Shift (ppm)
140.53
134.48
132.26
128.74
128.58
127.87
127.45
127.14
54.33
53.34
43.63
15.18


H1 LD02.esp
NH
S
9n
Normalized Intensity
2.08
4.10
1.09
2.09
1.25
2.12
1.87
3.00
Chemical Shift (ppm)

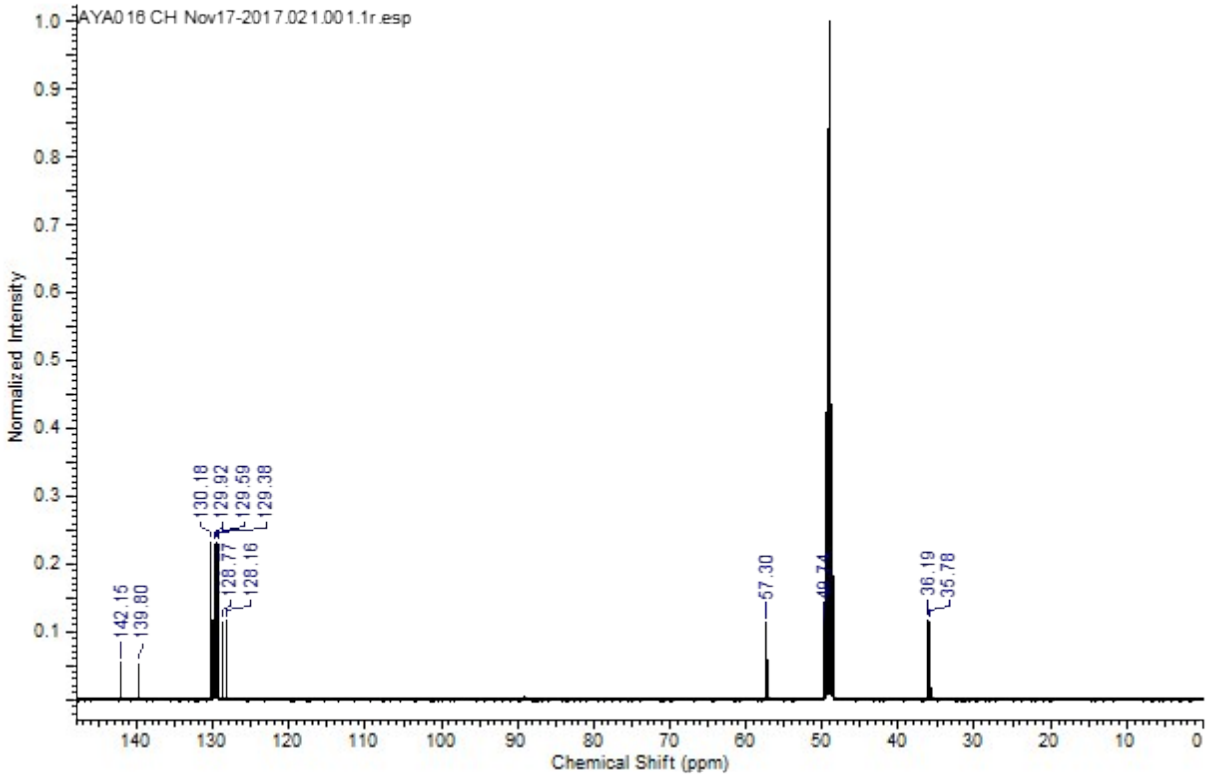

C13 8o.esp
Normalized Intensity
135.84
133.35
129.84
128.85
117.81
65.29
38.18
35.70
34.14
Chemical Shift (ppm)


H1 LD04c.esp
Normalized Intensity
Chemical Shift (ppm)
NH
S
9o
3.93
1.33
0.96
2.11
2.01
3.06
2.16
3.00


C13 LD04 Conc.esp
Normalized Intensity
Chemical Shift (ppm)
142.73
128.51
127.18
64.18
40.45
34.62
34.50
22.99
13.42


AYA016 1H Nov17-2017.022.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
4.01
5.90
1.00
2.05
1.99
2.97
S
H
N

8p


AYA016 CH Nov17-2017.021.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
142.15
139.80
130.18
129.92
129.59
129.38
128.77
128.16
57.30
49.74
36.19
35.78