

Supporting Information-II

HFIP-mediated C-3-Alkylation of Indoles and Synthesis of Indolo[2-3-*b*]quinolines & Related Natural Products

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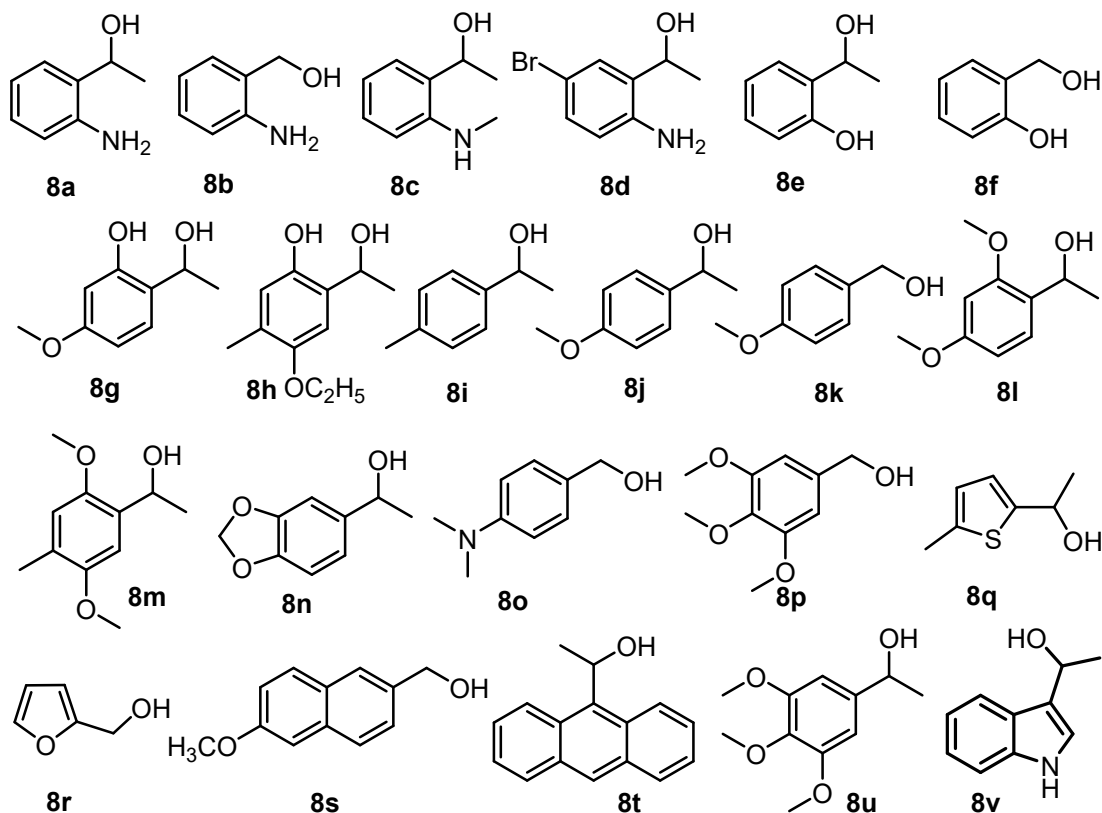
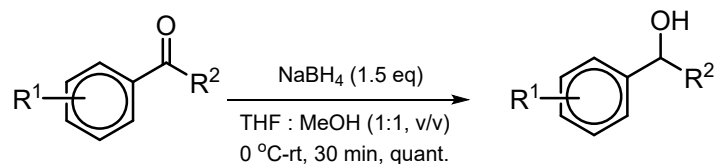
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Table of Contents

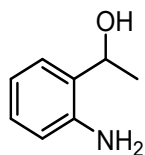
<u>S. No.</u>	<u>Contents</u>	<u>Page No.</u>
<u>1</u>	<u>Preparation of Starting Materials</u>	<u>S2</u>
<u>2</u>	<u>Characterisation Data</u>	<u>S3-S6</u>
<u>3</u>	<u>NMR Spectra</u>	<u>S7-S27</u>
<u>4</u>	<u>Reaction Standardization through HPLC</u>	<u>S29-S41</u>
<u>5</u>	<u>HRMS data</u>	<u>S42-S44</u>

1) Preparation of starting Material: All required alcohols were synthesized using modified version of known protocol.¹



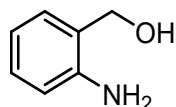
To a solution of respective carbonyl compound (1 equiv.) in THF: MeOH (1:1, v/v), NaBH_4 (1.5 equiv.) was added slowly at $0\text{ }^\circ\text{C}$. After the addition, the cold bath was removed and the reaction mixture was allowed to come to room temperature. After the complete consumption of starting material, as confirmed with TLC, the reaction mixture was quenched with brine and extraction was done with Ethyl acetate (3 times). The combined organic layer was dried over anhydrous Na_2SO_4 , concentrated under reduced pressure and the resulting residue was washed through a short silica gel pad (except **compound 8v** which was used as such for further reaction; reason being decomposition on column purification) to afford the desired product (**8a-8u**)

2) Characterization data for starting material:



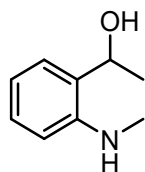
1-(2-aminophenyl)ethan-1-ol (8a):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.10 (m, 2H), 6.73 (t, $J = 7.4$ Hz, 1H), 6.67 (d, $J = 7.8$ Hz, 1H), 4.92 (q, $J = 6.6$ Hz, 1H), 1.59 (d, $J = 6.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 145.3, 128.7, 128.4, 126.7, 118.2, 116.8, 69.8, 21.6



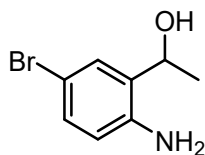
(2-aminophenyl)methanol (8b):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.18 – 7.10 (m, 1H), 7.08-7.04 (m, 1H), 6.76-6.67 (m, 2H), 4.63 (d, $J = 12.8$ Hz, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 145.7, 129.4, 129.3, 125.2, 118.5, 116.3, 64.2



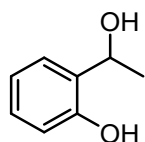
1-(2-(methylamino)phenyl)ethan-1-ol (8c)

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.11 (m, 1H), 6.96 (d, $J = 7.4$ Hz, 1H), 6.57 (dd, $J = 14.1, 7.6$ Hz, 2H), 4.74 (q, $J = 6.6$ Hz, 1H), 2.73 (s, 3H), 1.46 (d, $J = 6.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 147.9, 128.9, 127.8, 126.2, 116.4, 110.6, 69.8, 30.5, 21.4



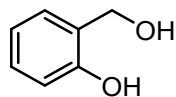
1-(2-amino-5-bromophenyl)ethan-1-ol (8d):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.20 – 7.12 (m, 2H), 6.53 (d, $J = 8.0$ Hz, 1H), 4.83 (q, $J = 6.5$ Hz, 1H), 4.25 (s, 2H), 2.13 (s, 1H), 1.55 (d, $J = 6.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 144.2, 131.2, 130.4, 129.4, 118.3, 109.9, 69.1, 21.5



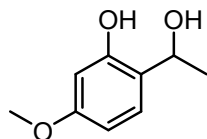
2-(1-hydroxyethyl)phenol (8e):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.15 (d, $J = 22.6$ Hz, 1H), 7.16 (t, $J = 7.1$ Hz, 1H), 6.98 (d, $J = 6.2$ Hz, 1H), 6.85 (d, $J = 7.3$ Hz, 2H), 5.03 (q, $J = 6.4$ Hz, 1H), 1.56 (d, $J = 6.6$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 155.2, 128.9, 128.7, 126.6, 120.1, 117.0, 71.3, 23.4



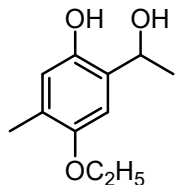
2-(hydroxymethyl)phenol (8f):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.23 – 7.12 (m, 2H), 6.99 (d, $J = 7.4$ Hz, 1H), 6.86-6.76 (m, 2H), 4.80 (s, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 156.1, 129.7, 128.0, 124.8, 120.3, 116.6, 64.7



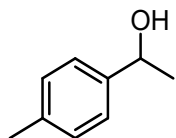
2-(1-hydroxyethyl)-5-methoxyphenol (8g):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.87 (d, $J = 8.4$ Hz, 1H), 6.44 (d, $J = 2.1$ Hz, 1H), 6.38 (dd, $J = 8.3, 2.1$ Hz, 1H), 5.01 (q, $J = 6.5$ Hz, 1H), 3.75 (s, 3H), 1.55 (d, $J = 6.7$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 160.4, 156.8, 127.2, 121.1, 105.8, 102.6, 71.2, 55.4, 23.6



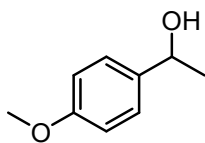
4-ethoxy-2-(1-hydroxyethyl)-5-methylphenol (8h):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48 (s, 1H), 6.65 (s, 1H), 6.42 (s, 1H), 3.92 (q, $J = 7.0$ Hz, 2H), 2.16 (s, 3H), 1.54 (d, $J = 6.6$ Hz, 3H), 1.37 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 150.5, 148.7, 127.9, 126.1, 119.4, 110.7, 71.5, 64.9, 23.6, 16.0, 15.2



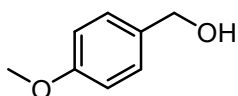
1-(p-tolyl)ethan-1-ol (8i):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.29 (d, $J = 7.8$ Hz, 2H), 7.20 (d, $J = 7.8$ Hz, 2H), 4.87 (q, $J = 6.4$ Hz, 1H), 2.39 (s, 3H), 1.50 (d, $J = 6.5$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.0, 137.1, 129.2, 129.2, 125.4, 125.4, 70.2, 25.1, 21.1



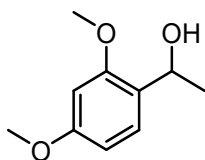
1-(4-methoxyphenyl)ethan-1-ol (8j):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.30 (d, $J = 8.7$ Hz, 2H), 6.89 (d, $J = 8.7$ Hz, 2H), 4.84 (m, 1H), 3.81 (s, 2H), 1.47 (d, $J = 6.4$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 158.9, 138.1, 126.7, 126.7, 113.8, 113.8, 69.86, 55.31, 25.07



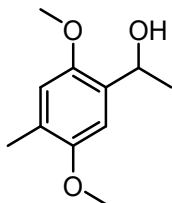
(4-methoxyphenyl)methanol (8k):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.29 (d, $J = 7.3$ Hz, 2H), 6.90 (d, $J = 8.6$ Hz, 2H), 4.60 (s, 2H), 3.82 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 159.2, 133.2, 128.7, 128.7, 114.0, 114.0, 64.9, 55.4



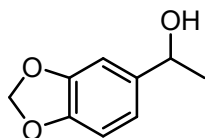
1-(2,4-dimethoxyphenyl)ethan-1-ol(8l):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.27–7.22 (m, 1H), 6.50–6.44 (m, 1H), 5.04 (s, 1H), 3.84 (s, 3H), 3.80 (s, 3H), 2.58 (s, 1H), 1.49 (d, $J = 6.5$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 160.2, 157.8, 126.8, 126.1, 104.1, 98.8, 66.2, 55.5, 55.4, 22.9



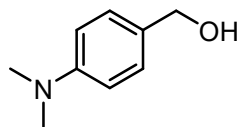
1-(2,5-dimethoxy-4-methylphenyl)ethan-1-ol(8m):

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.86 (s, 1H), 6.70 (s, 1H), 5.07 (q, $J = 6.4$ Hz, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 2.22 (s, 3H), 1.49 (d, $J = 6.5$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 151.9, 150.1, 131.5, 126.1, 114.0, 109.0, 66.7, 56.2, 56.0, 23.4, 16.3



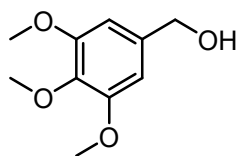
1-(benzo[d][1,3]dioxol-5-yl)ethan-1-ol(8n):

^1H NMR (400 MHz, CDCl_3) δ 6.82 (s, 1H), 6.78-6.68 (m, 2H), 5.88 (s, 2H), 4.72 (q, $J = 5.2$ Hz, 1H), 3.35 (s, 1H), 1.38 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.6, 146.6, 140.1, 118.6, 108.0, 106.1, 100.9, 69.9, 25.1



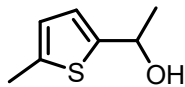
(4-(dimethylamino)phenyl)methanol(8o):

^1H NMR (400 MHz, CDCl_3) δ 7.27 (d, $J = 8.6$ Hz, 2H), 6.76 (d, $J = 8.7$ Hz, 2H), 4.58 (s, 2H), 2.98 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 150.4, 129.1, 128.7, 128.7, 112.8, 112.8, 65.4, 40.8, 40.8



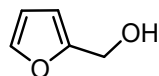
(3,4,5-trimethoxyphenyl)methanol(8p):

^1H NMR (400 MHz, CDCl_3) δ 6.50 (s, 2H), 4.52 (s, 2H), 3.75 (d, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 153.0, 136.9, 136.7, 103.5, 103.5, 64.9, 60.7, 55.8, 55.8.



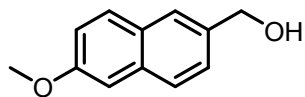
1-(5-methylthiophen-2-yl)ethan-1-ol(8q):

^1H NMR (400 MHz, CDCl_3) δ 6.75 (d, $J = 3.3$ Hz, 1H), 6.60-6.57 (m, 1H), 5.02 (q, $J = 6.3$ Hz, 1H), 2.46 (s, 3H), 1.56 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.5, 139.2, 124.7, 123.2, 66.4, 25.1, 15.5



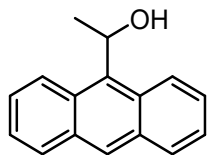
furan-2-ylmethanol(8r)

^1H NMR (400 MHz, CDCl_3) δ 7.37 – 7.35 (m, 1H), 6.31 – 6.24 (m, 2H), 4.53 (s, 2H), 2.95 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.1, 142.5, 110.4, 107.8, 57.2



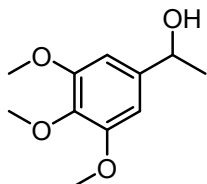
(6-methoxynaphthalen-2-yl)methanol(8s):

^1H NMR (400 MHz, CDCl_3) δ 7.73 (t, $J = 7.0$ Hz, 3H), 7.45 (dd, $J = 8.5, 1.4$ Hz, 1H), 7.17 – 7.14 (m, 2H), 4.82 (s, 2H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 135.8, 131.8, 129.5, 129.5, 128.9, 128.1, 125.7, 125.7, 124.9, 124.9, 67.4, 23.6



1-(anthracen-9-yl)ethan-1-ol (8t):

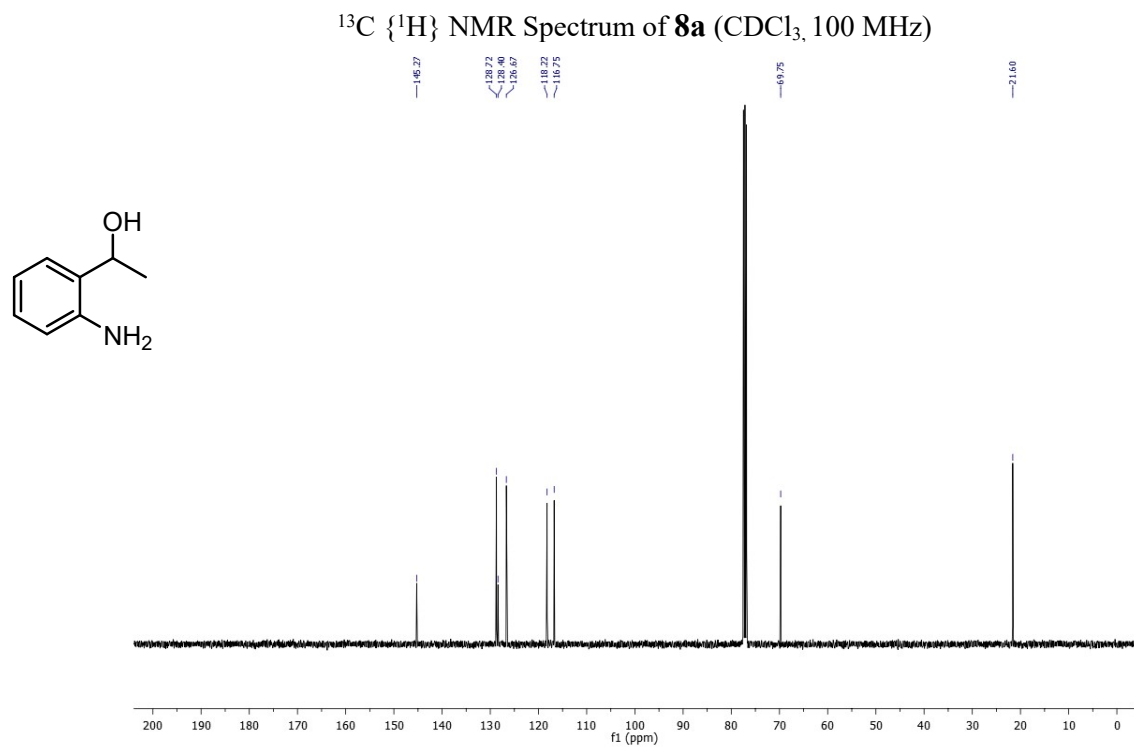
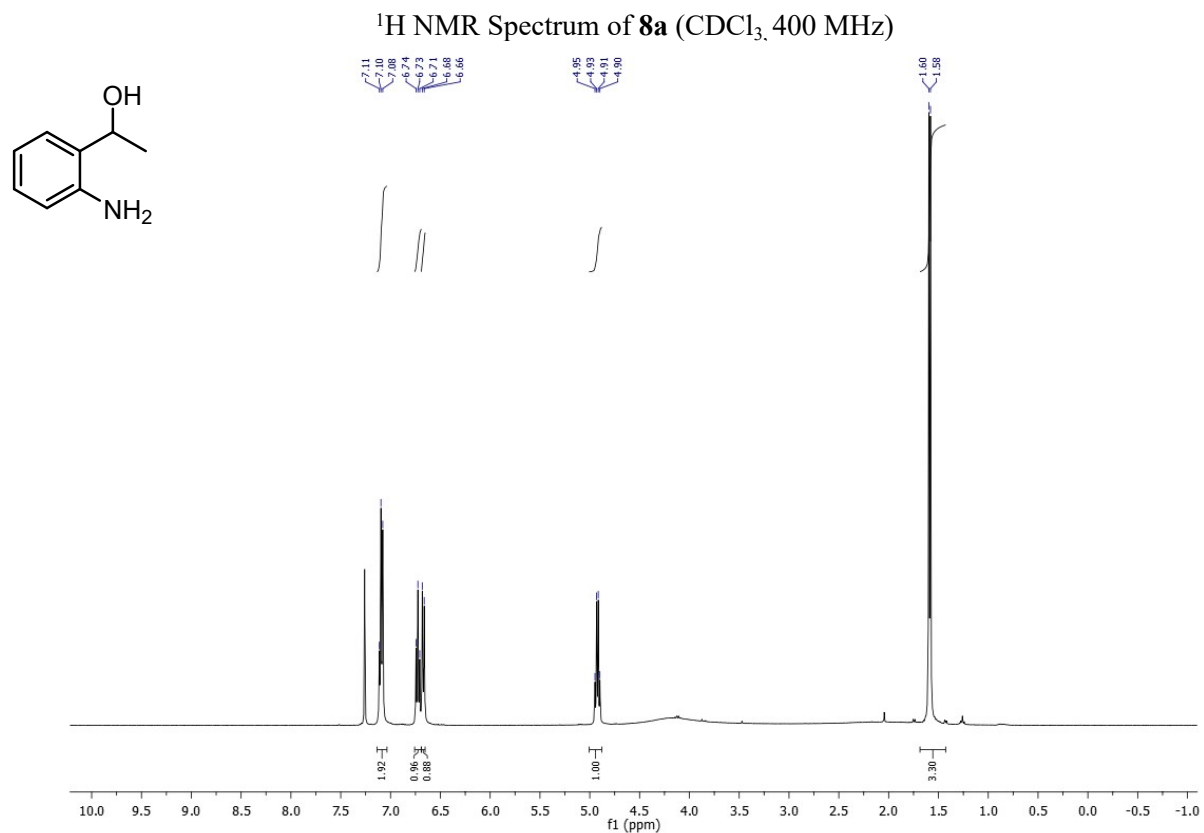
^1H NMR (400 MHz, CDCl_3) δ 8.68 (d, $J = 8.3$ Hz, 2H), 8.40 (s, 1H), 8.01 (d, $J = 7.4$ Hz, 2H), 7.52 – 7.43 (m, 4H), 6.49 (d, $J = 6.8$ Hz, 1H), 1.94 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 157.9, 136.2, 134.2, 129.5, 129.5, 128.9, 127.3, 126.0, 126.0, 125.7, 125.7, 119.1, 105.8, 105.8, 65.7, 55.4



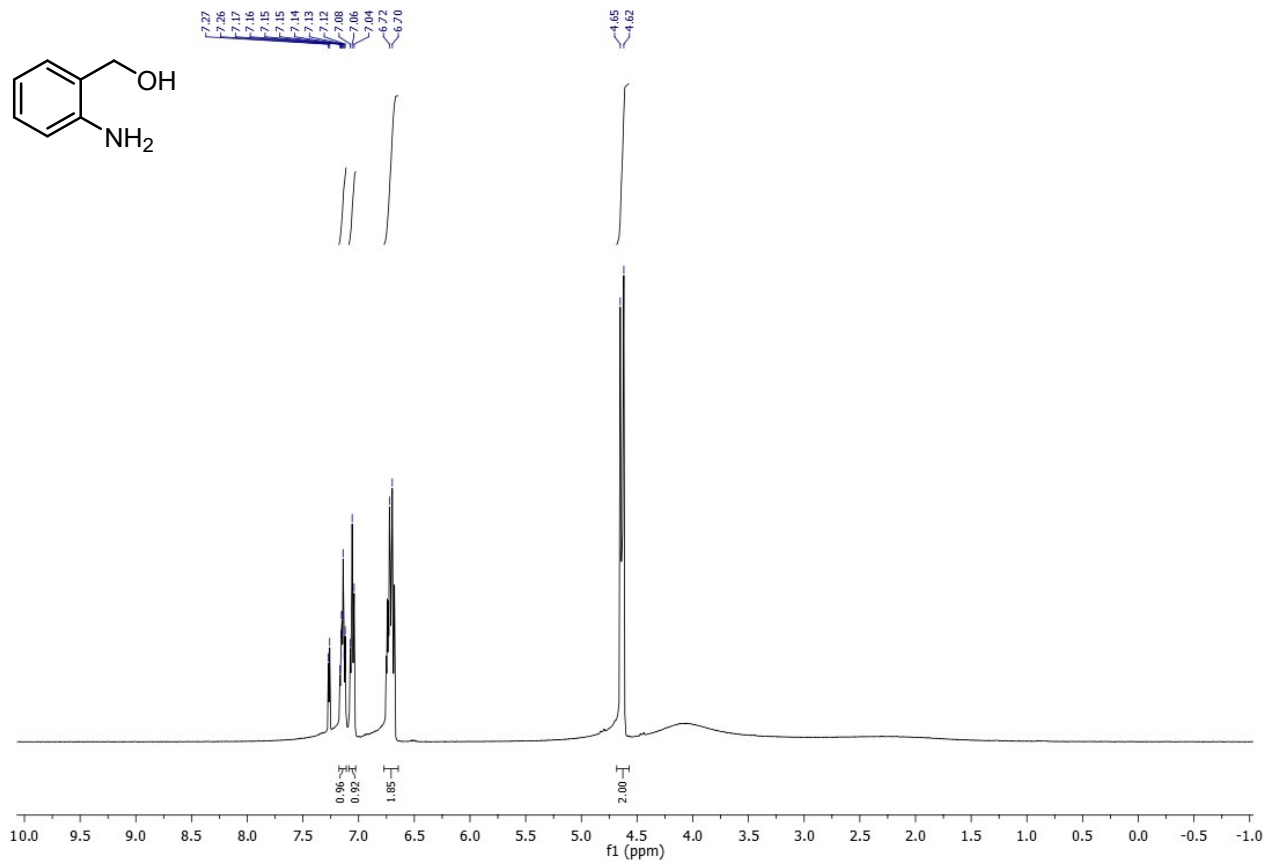
1-(3,4,5-trimethoxyphenyl)ethan-1-ol (8u):

^1H NMR (400 MHz, CDCl_3) δ 6.56 (s, 2H), 4.79 (q, $J = 6.4$ Hz, 1H), 3.83 (s, 6H), 3.79 (s, 3H), 1.45 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.2, 153.2, 141.9, 136.9, 102.2, 102.2, 70.5, 60.8, 56.1, 56.1, 25.3.

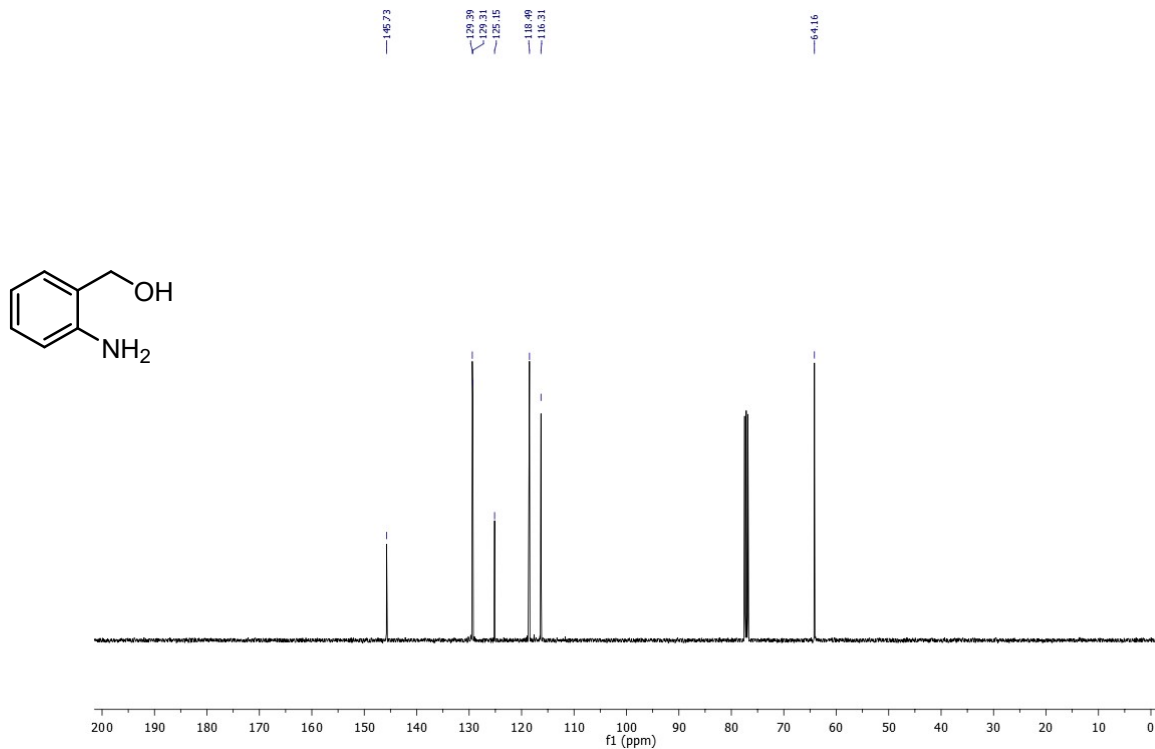
3) NMR spectra of starting materials:



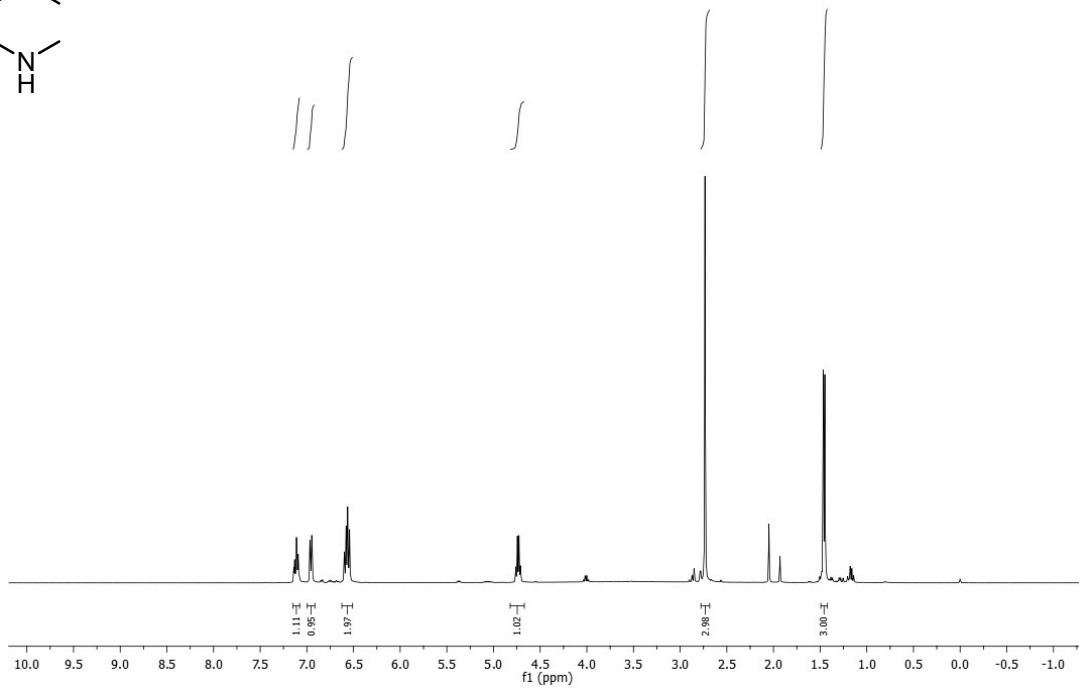
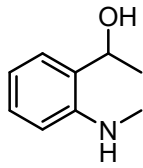
¹H NMR Spectrum of **8b** (CDCl₃, 400 MHz)



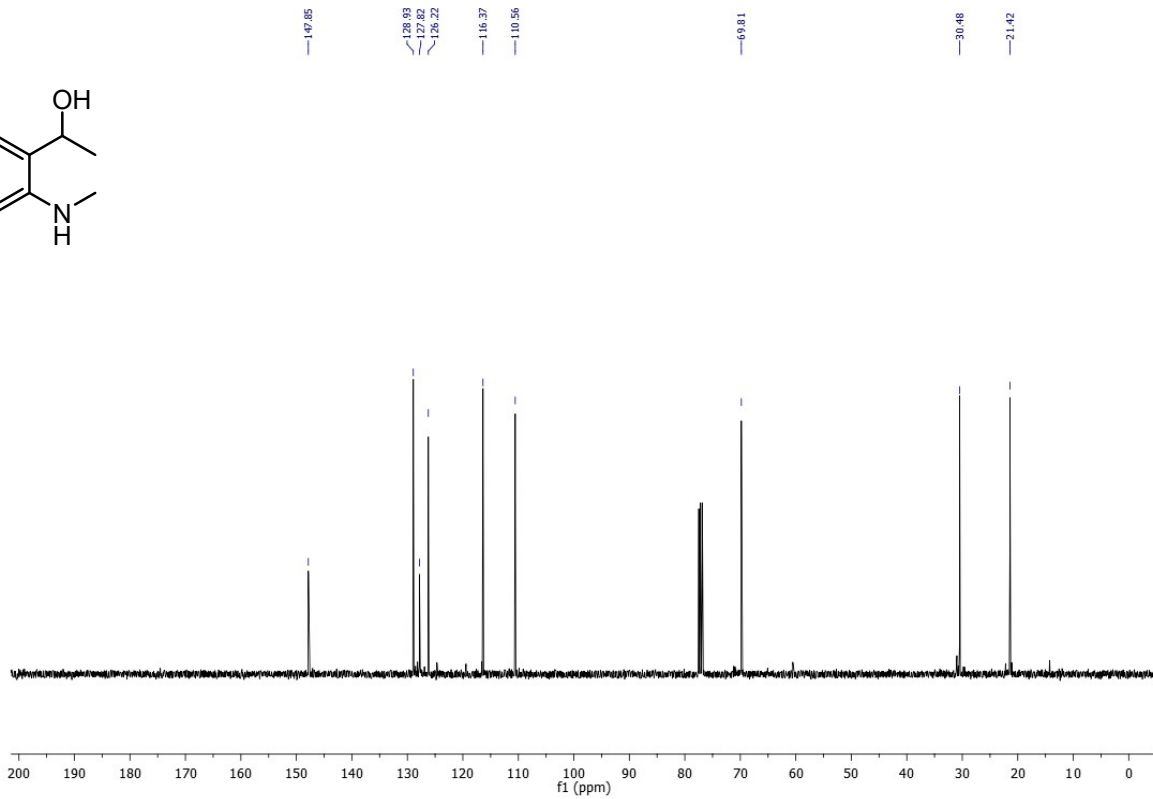
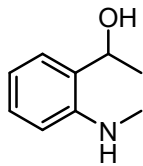
¹³C {¹H} NMR Spectrum of **8b** (CDCl₃, 100 MHz)



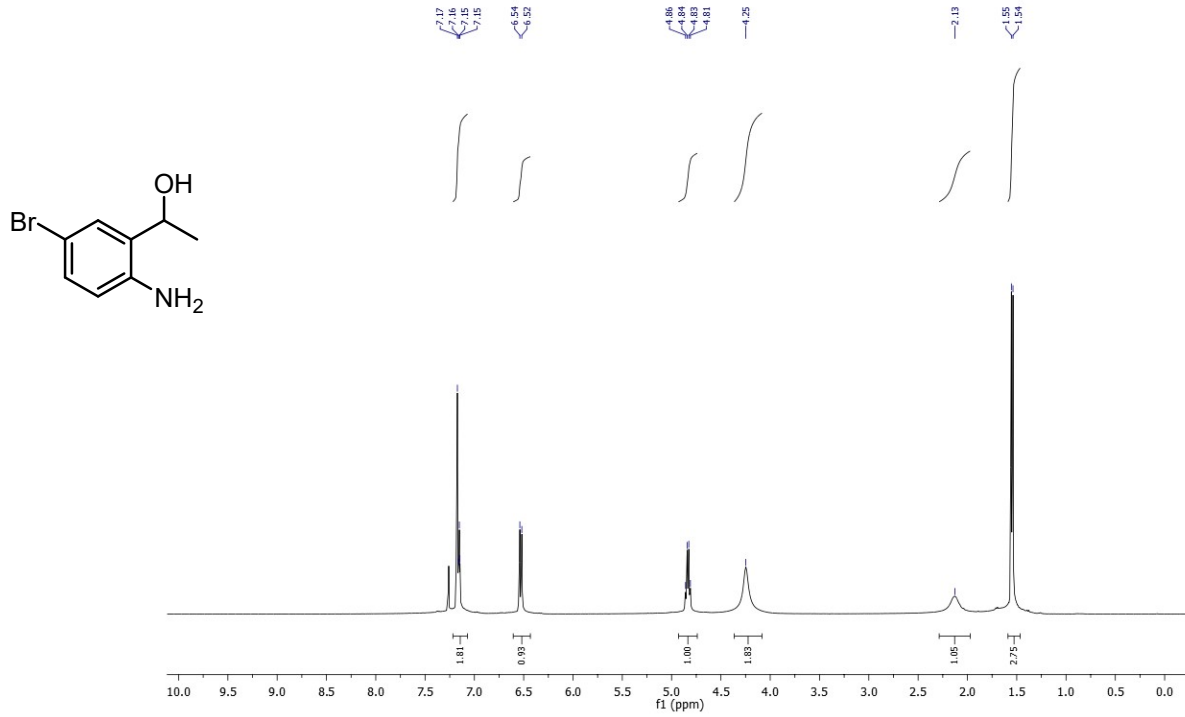
¹H NMR Spectrum of **8c** (CDCl₃, 400 MHz)



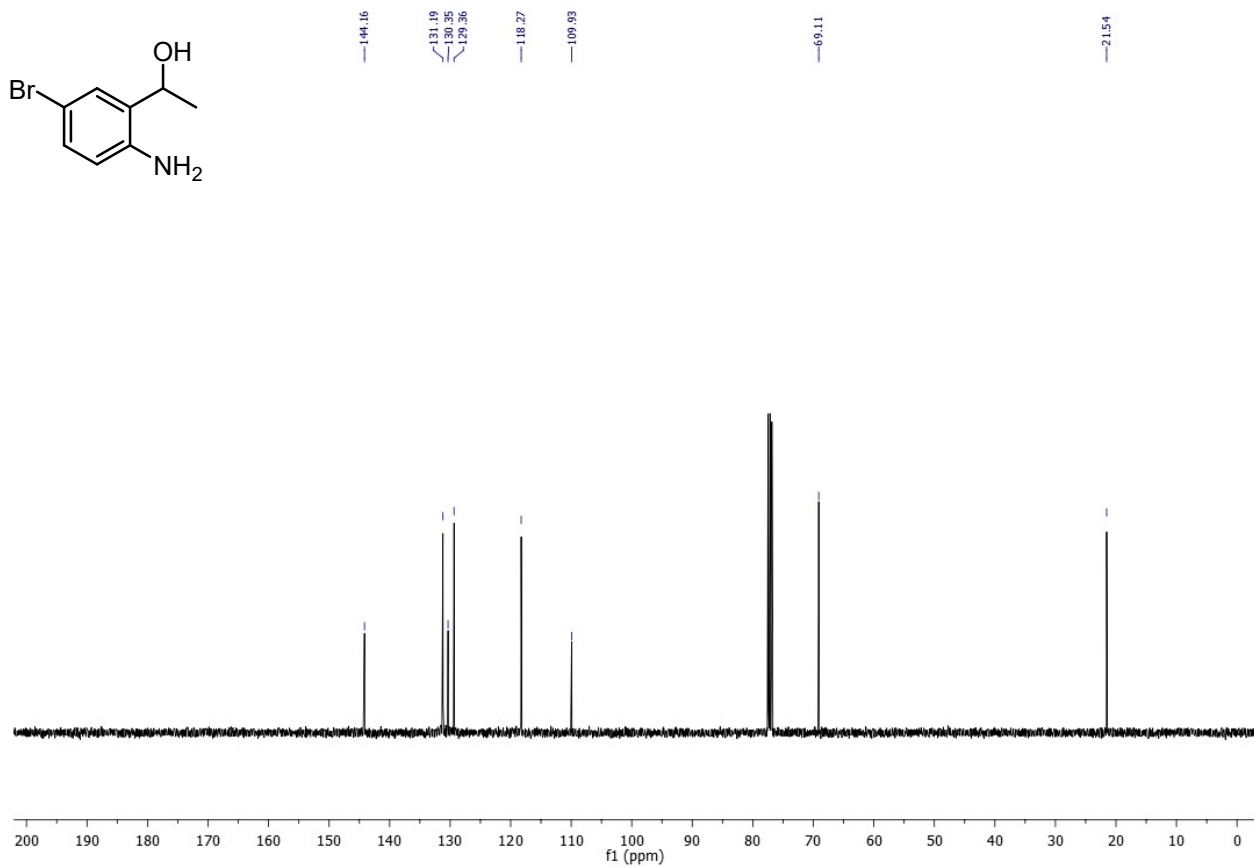
¹³C {¹H} NMR Spectrum of **8c** (CDCl₃, 100 MHz)



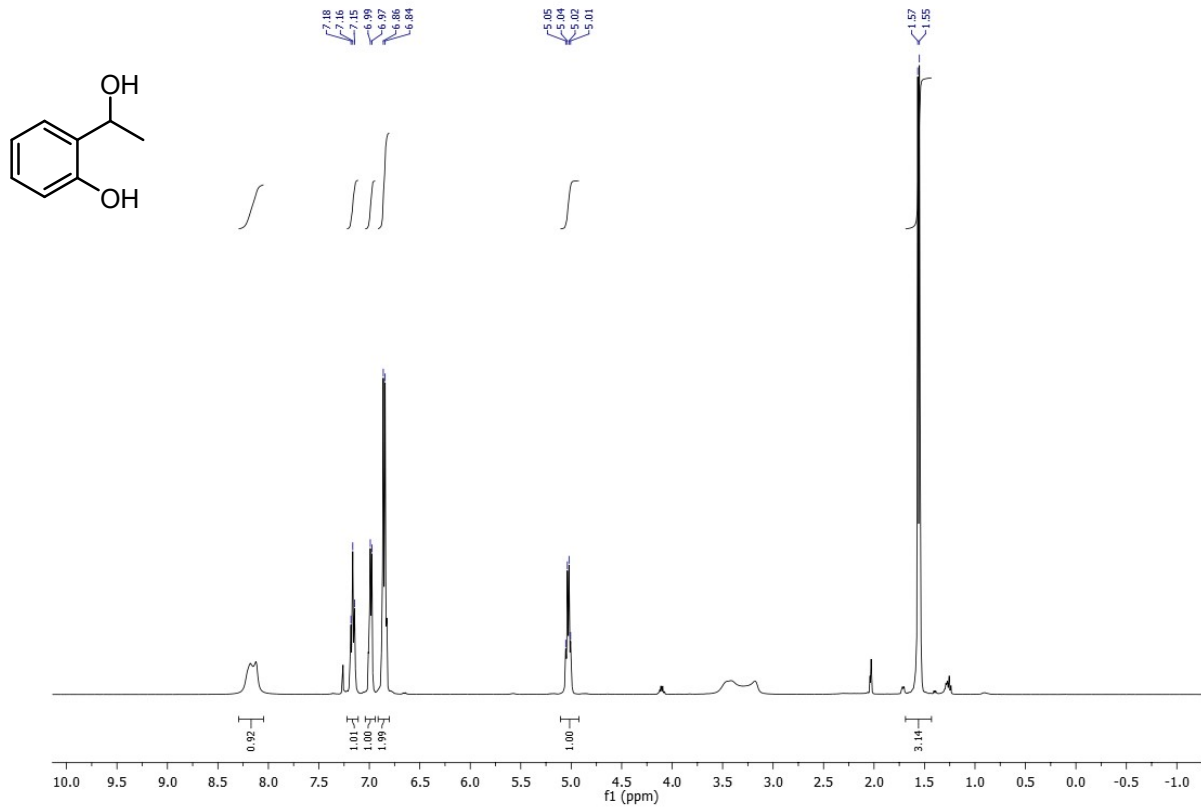
¹H NMR Spectrum of **8d** (CDCl₃, 400 MHz)



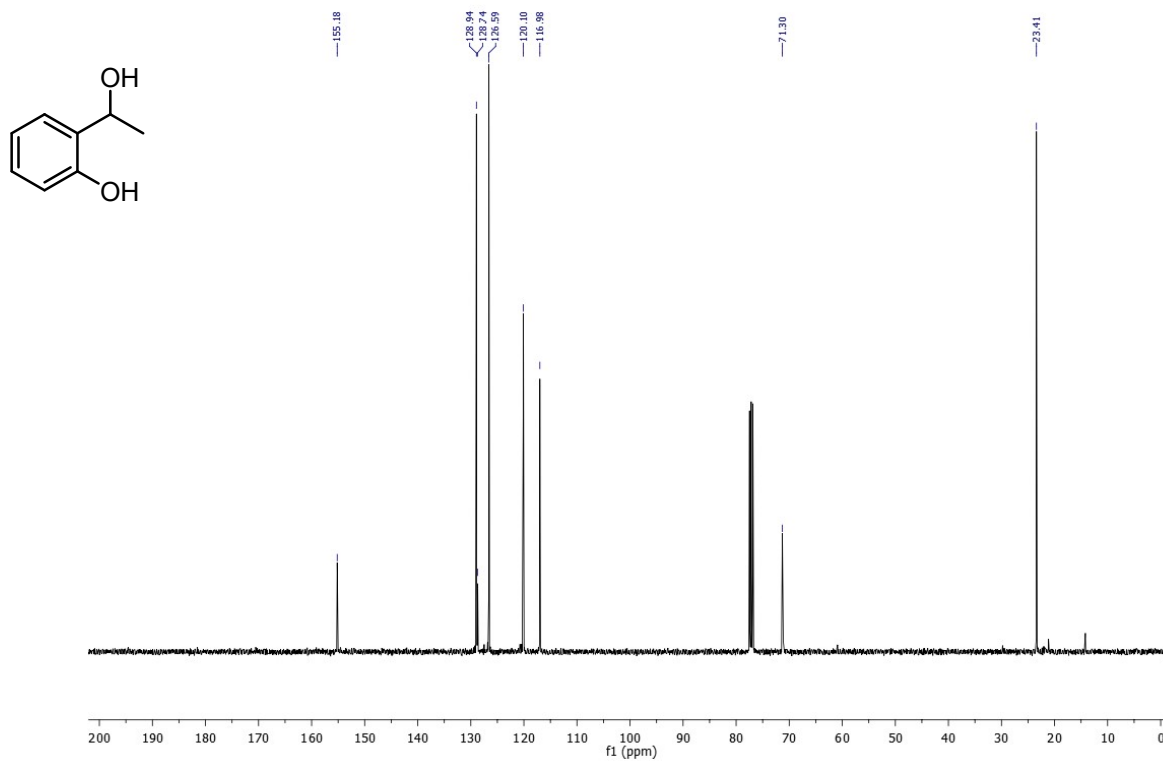
¹³C {¹H} NMR Spectrum of **8d** (CDCl₃, 100 MHz)

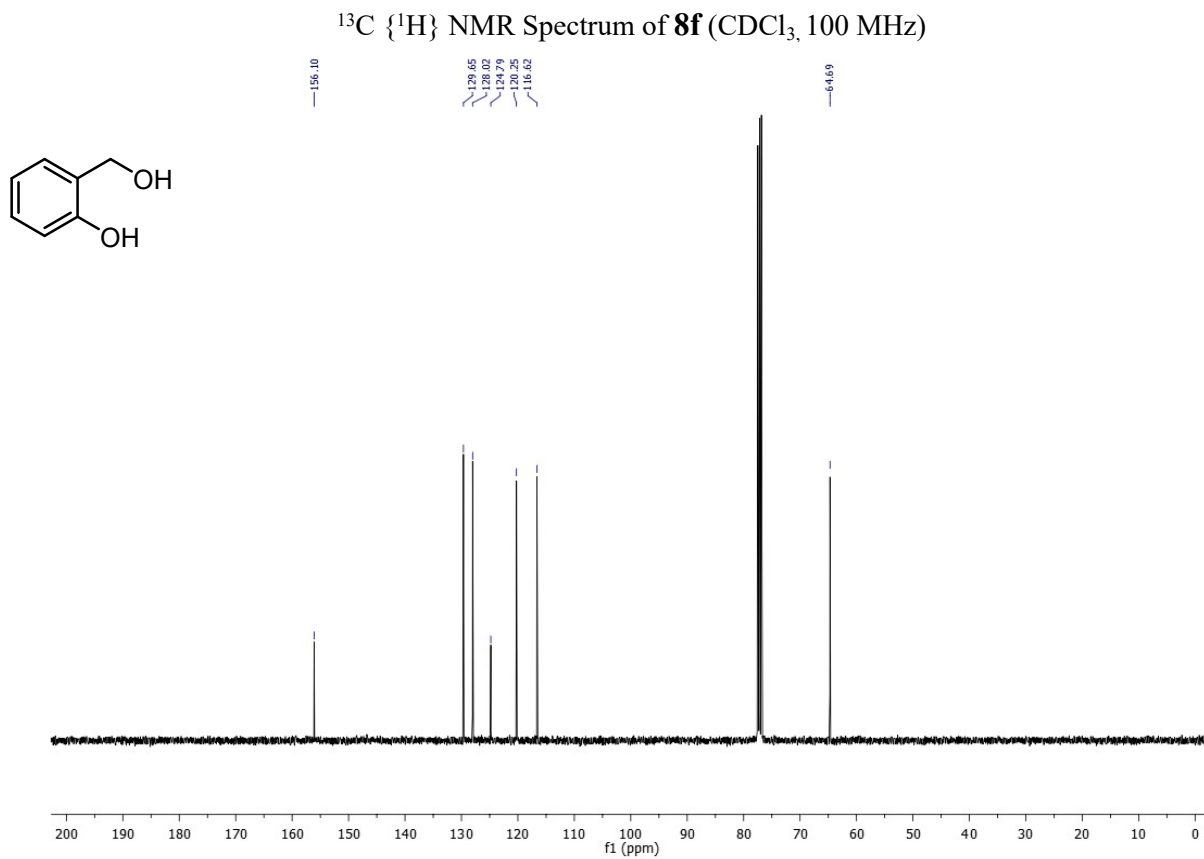
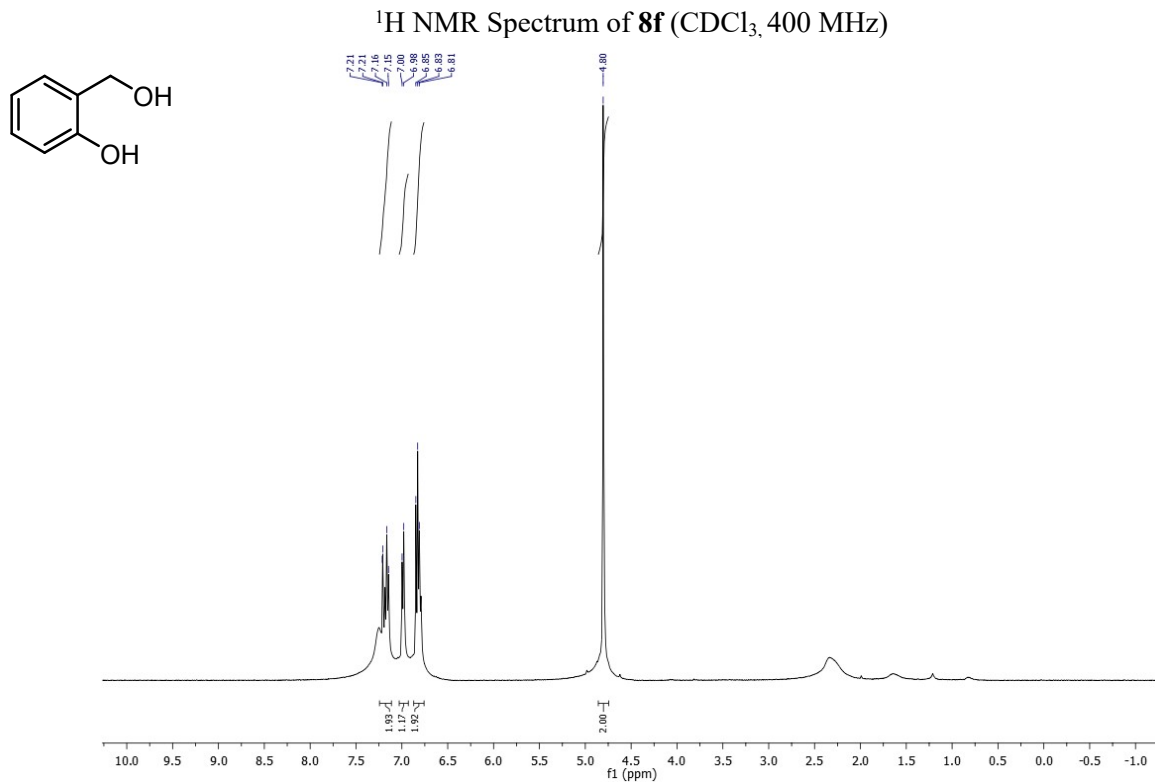


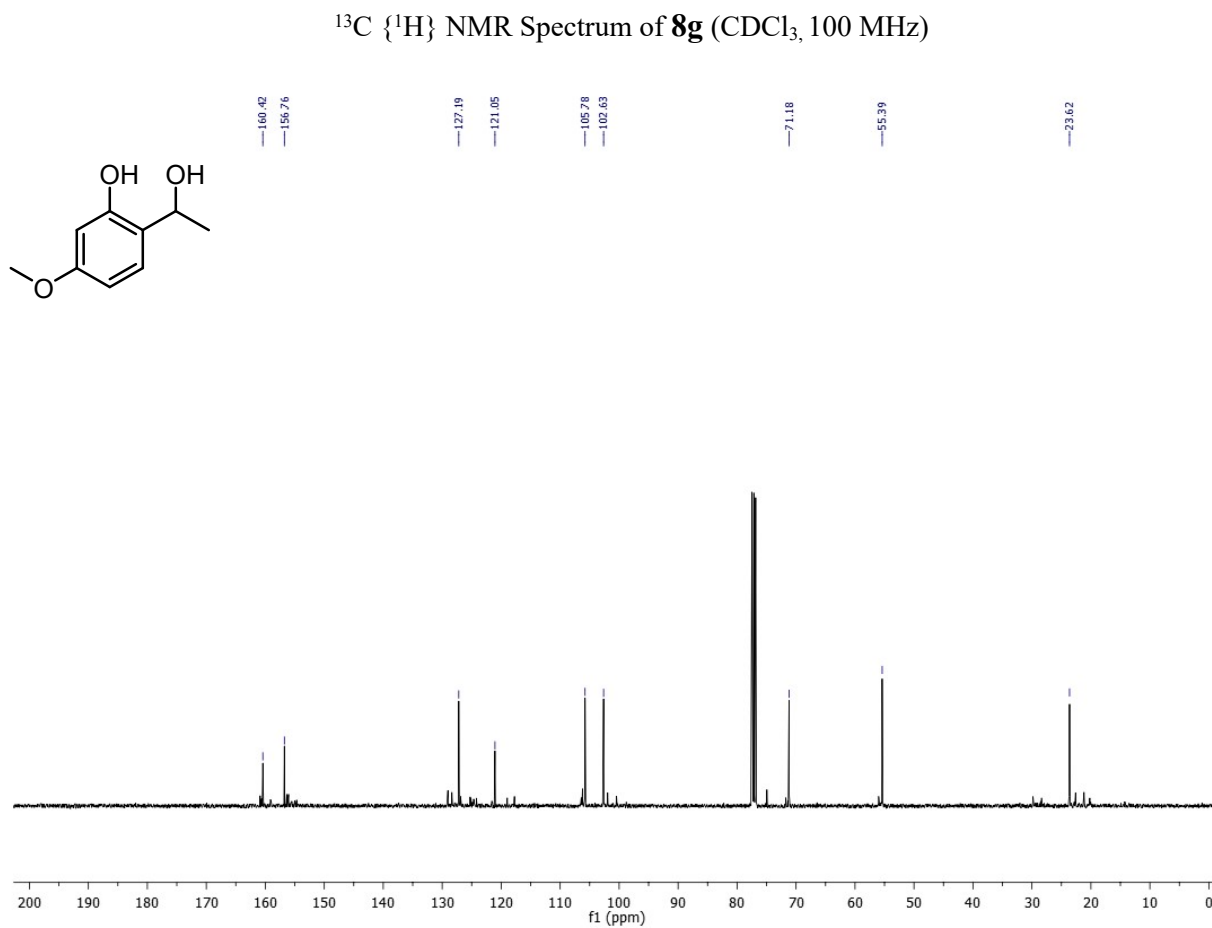
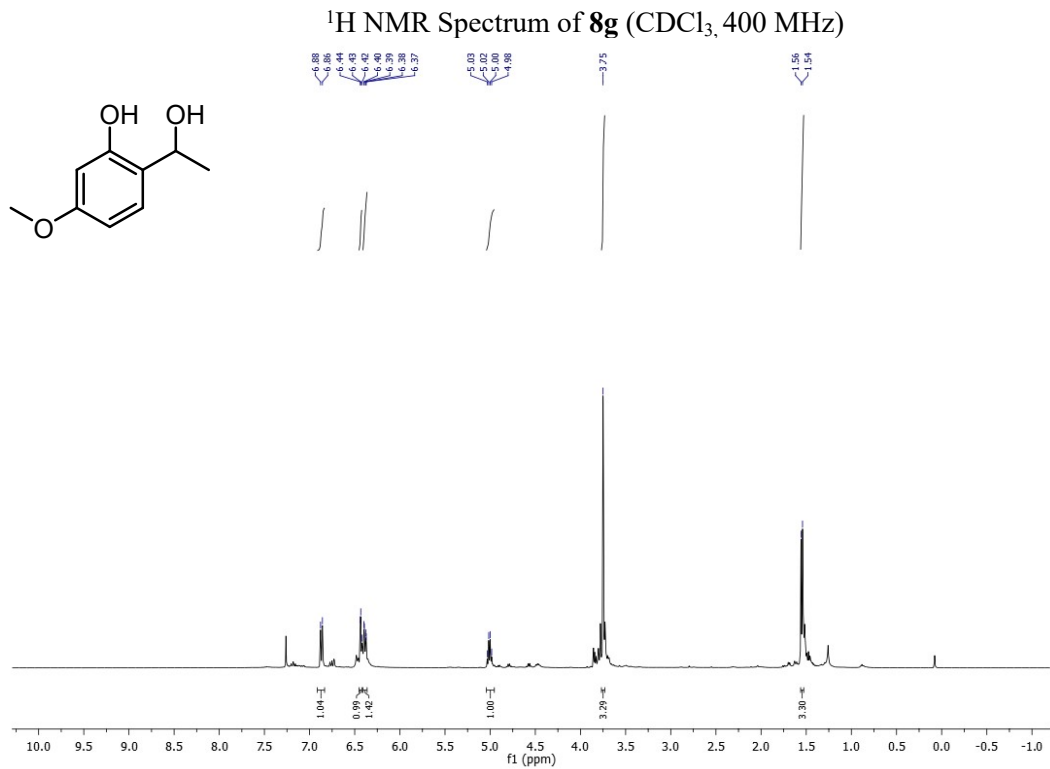
¹H NMR Spectrum of **8e** (CDCl₃, 400 MHz)

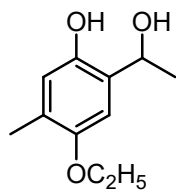


¹³C {¹H} NMR Spectrum of **8e** (CDCl₃, 100 MHz)

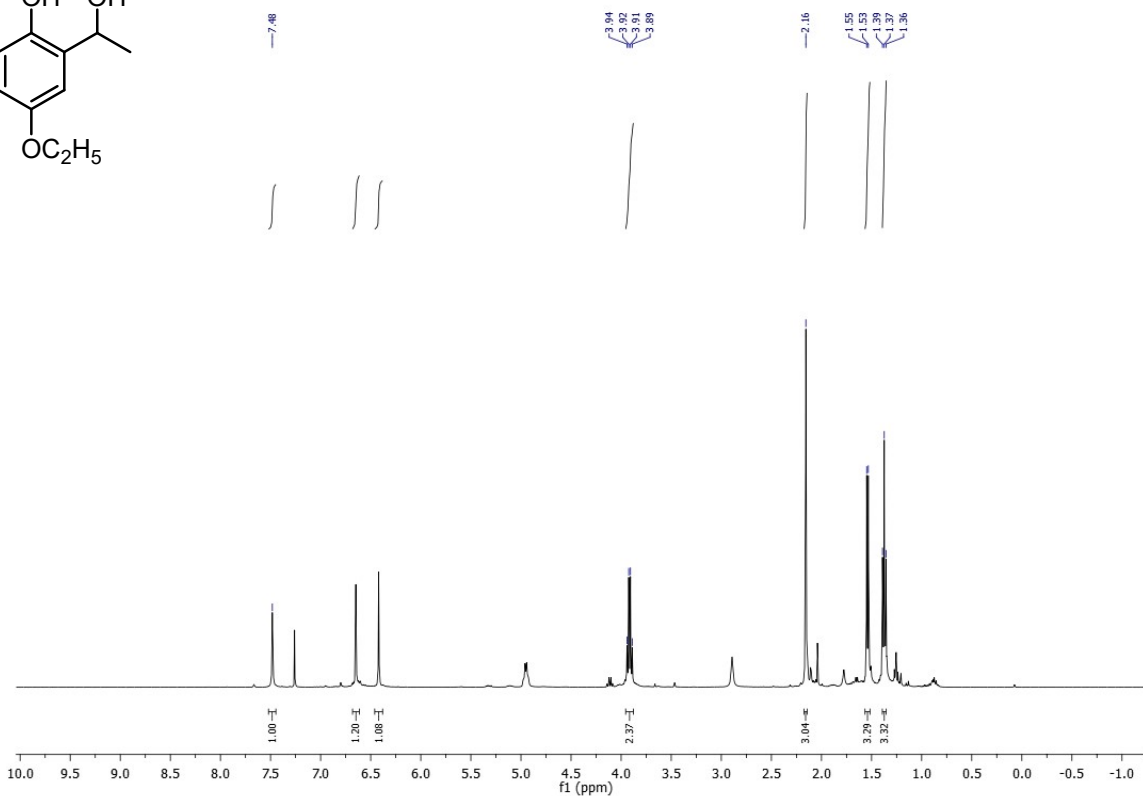




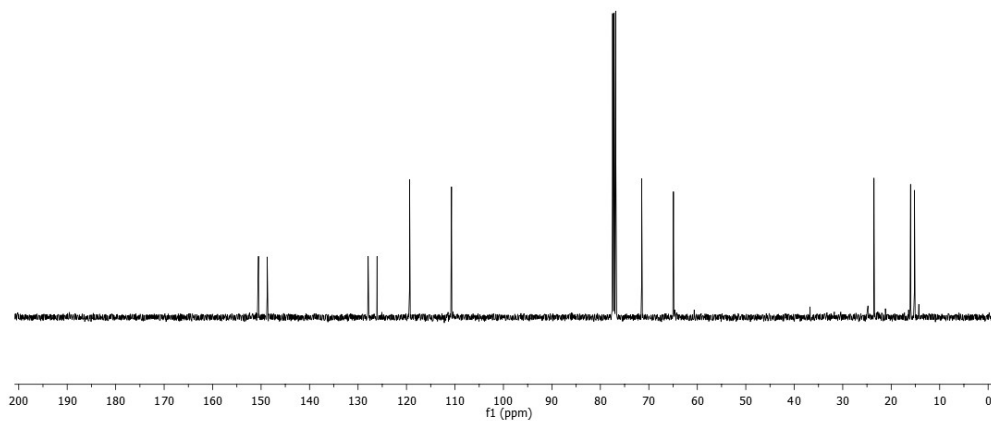
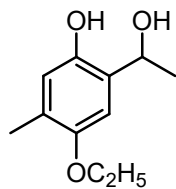


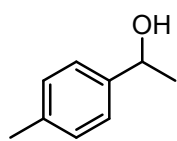


¹H NMR Spectrum of **8h** (CDCl₃, 400 MHz)

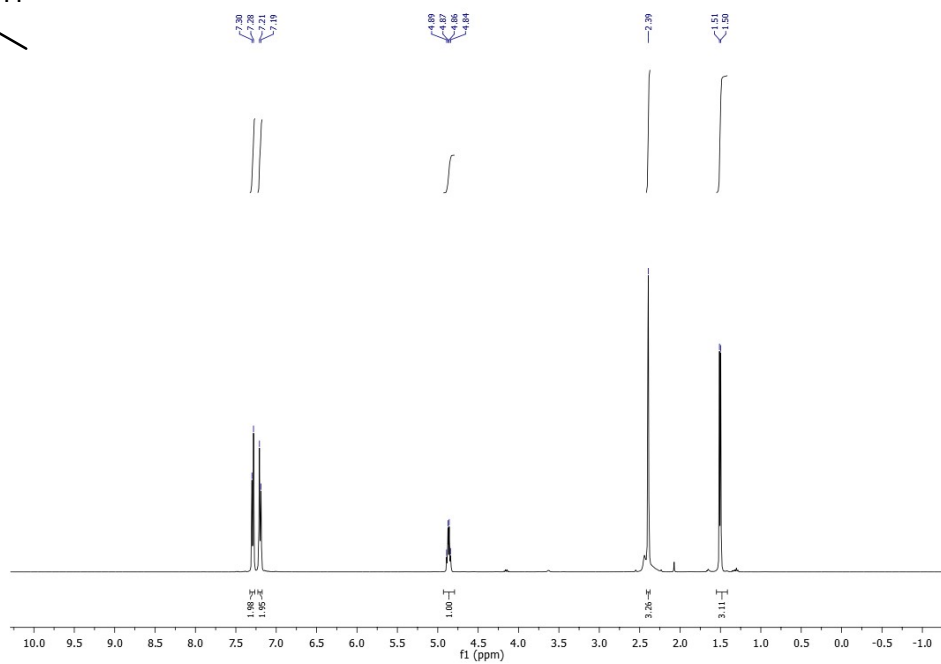


¹³C {¹H} NMR Spectrum of **8h** (CDCl₃, 100 MHz)

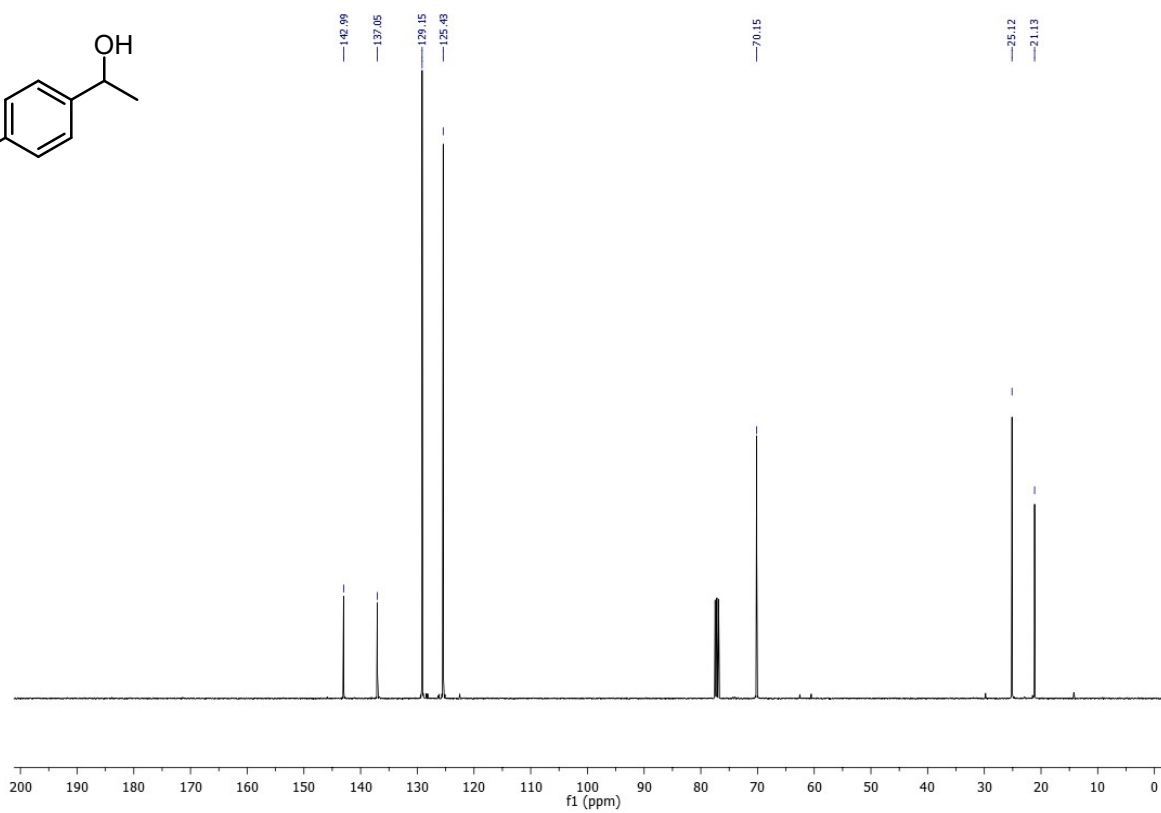
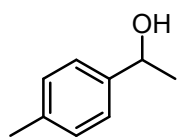


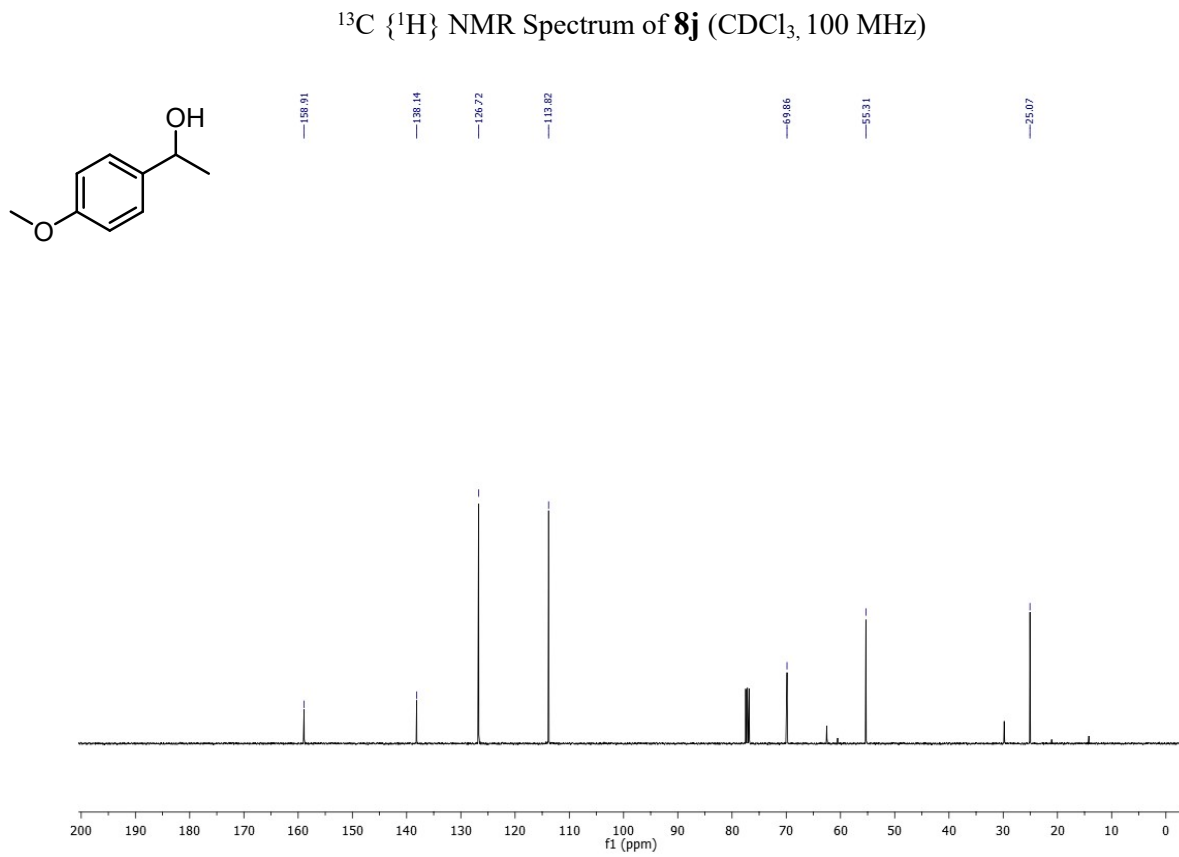
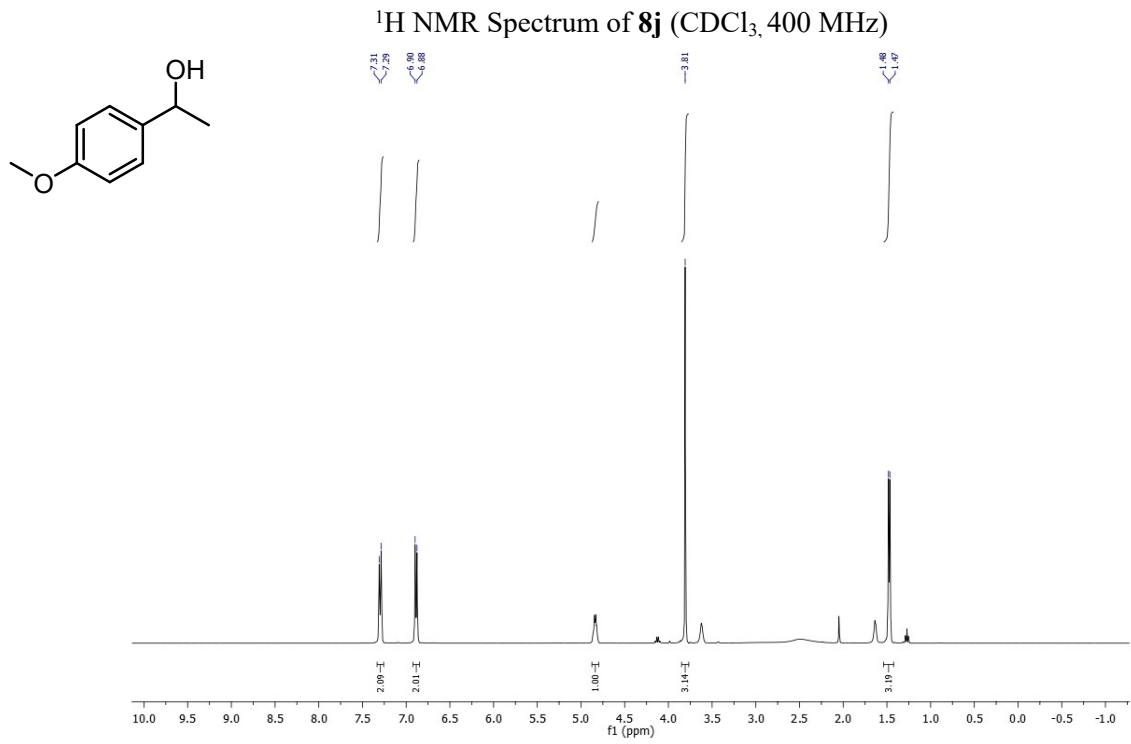


^1H NMR Spectrum of **8i** (CDCl_3 , 400 MHz)

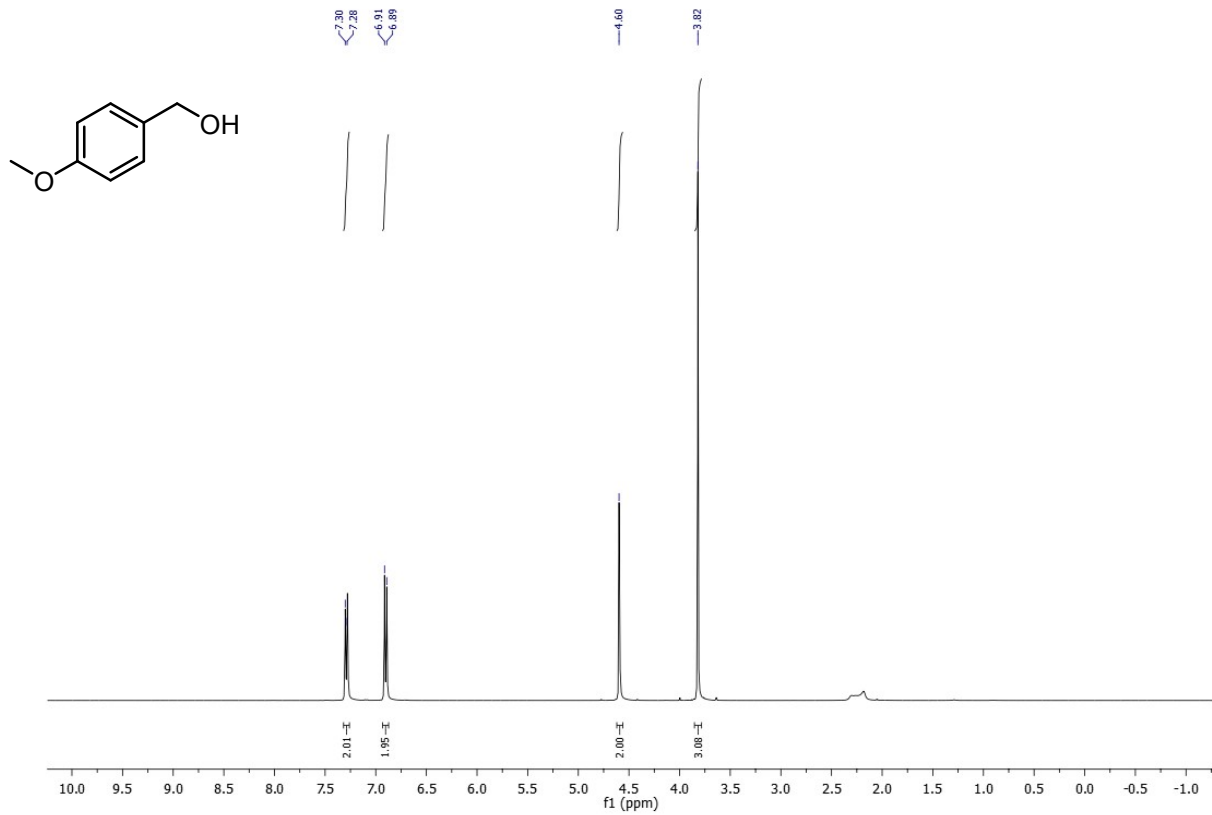


^{13}C $\{^1\text{H}\}$ NMR Spectrum of **8i** (CDCl_3 , 100 MHz)

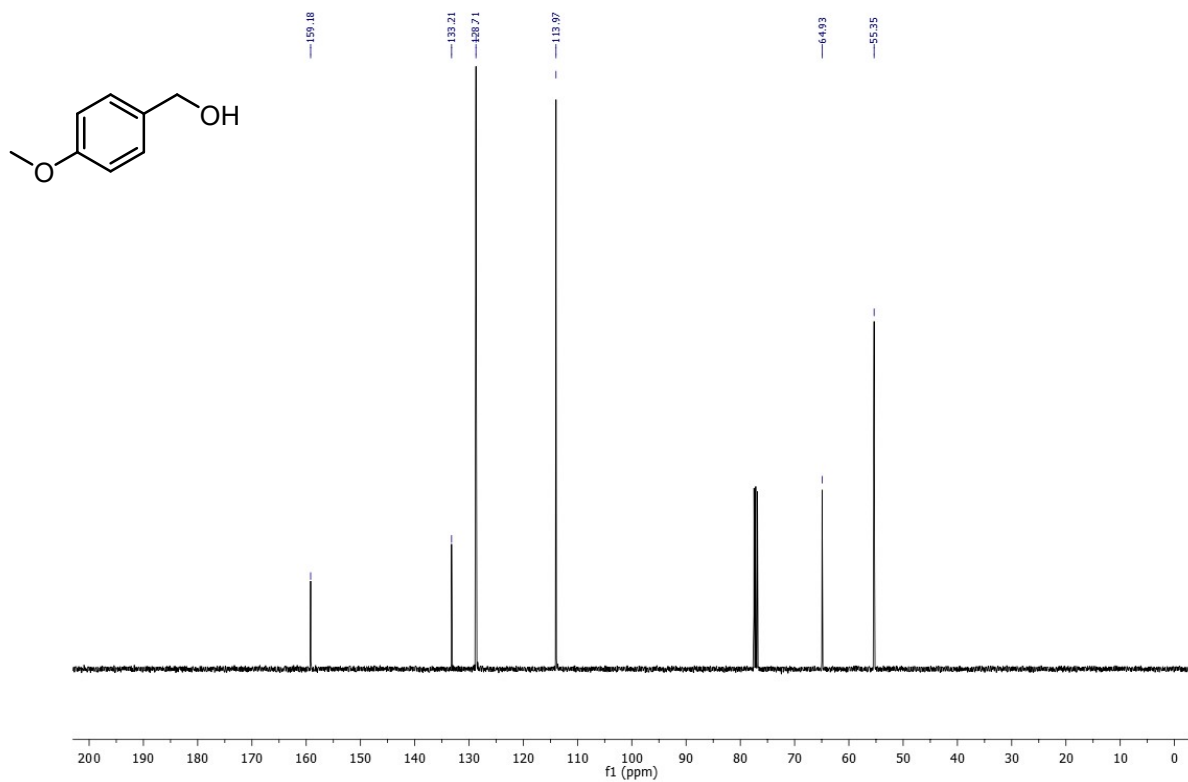




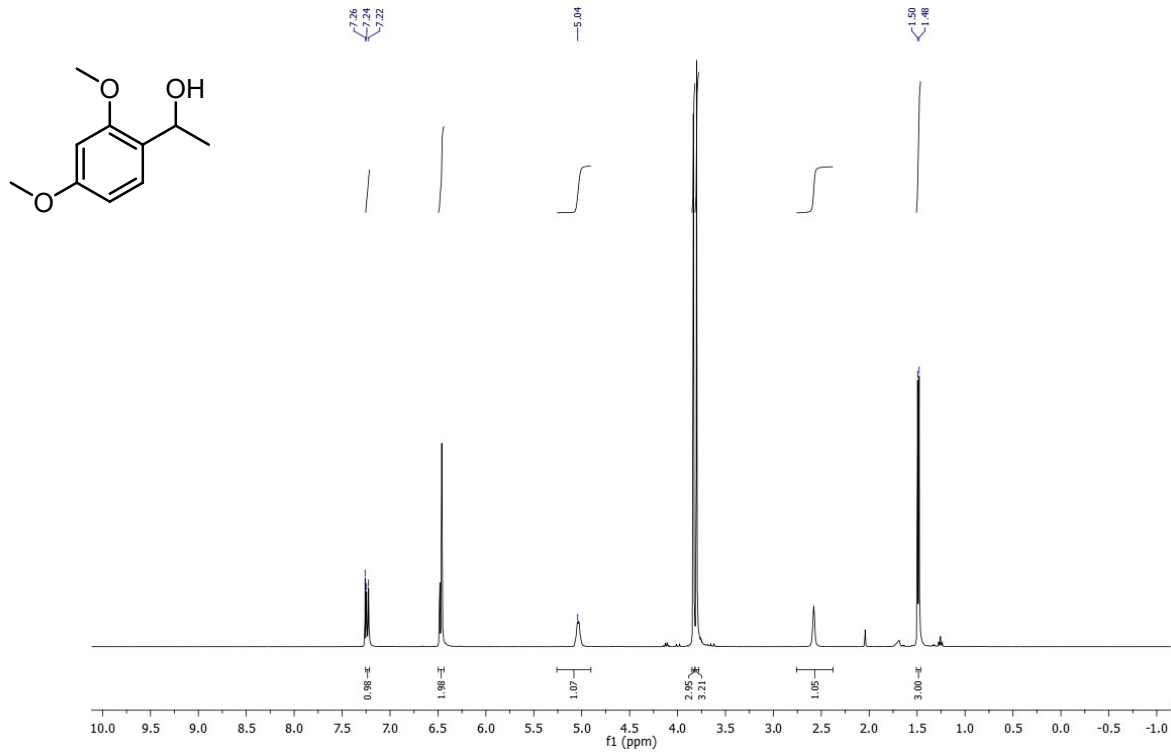
¹H NMR Spectrum of **8k** (CDCl₃, 400 MHz)



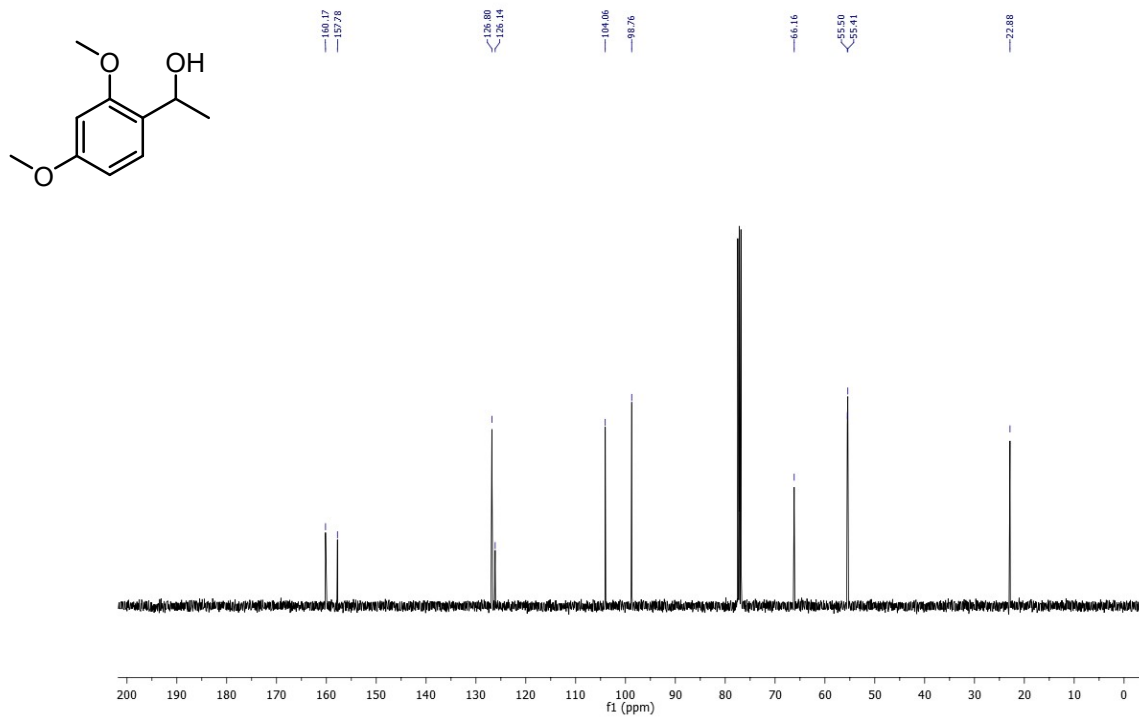
¹³C {¹H} NMR Spectrum of **8k** (CDCl₃, 100 MHz)

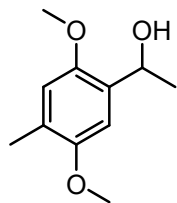


¹H NMR Spectrum of **81** (CDCl₃, 400 MHz)

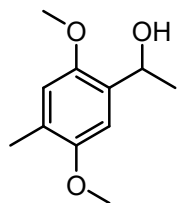
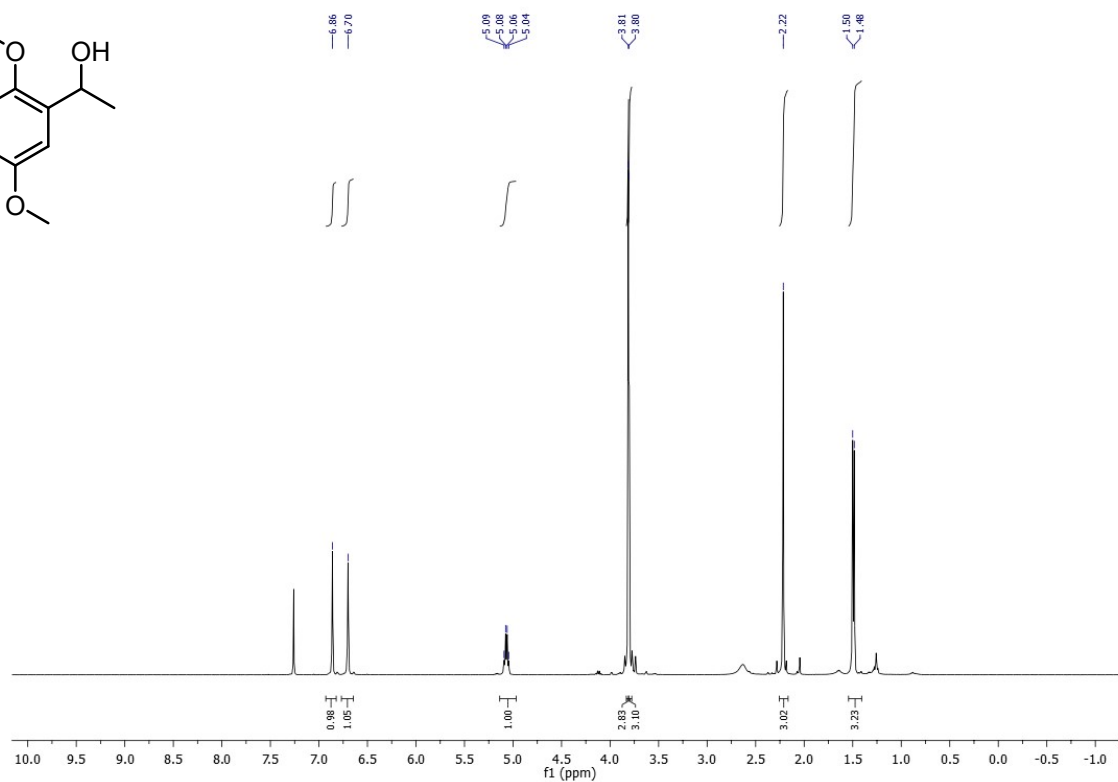


¹³C {¹H} NMR Spectrum of **81** (CDCl₃, 100 MHz)

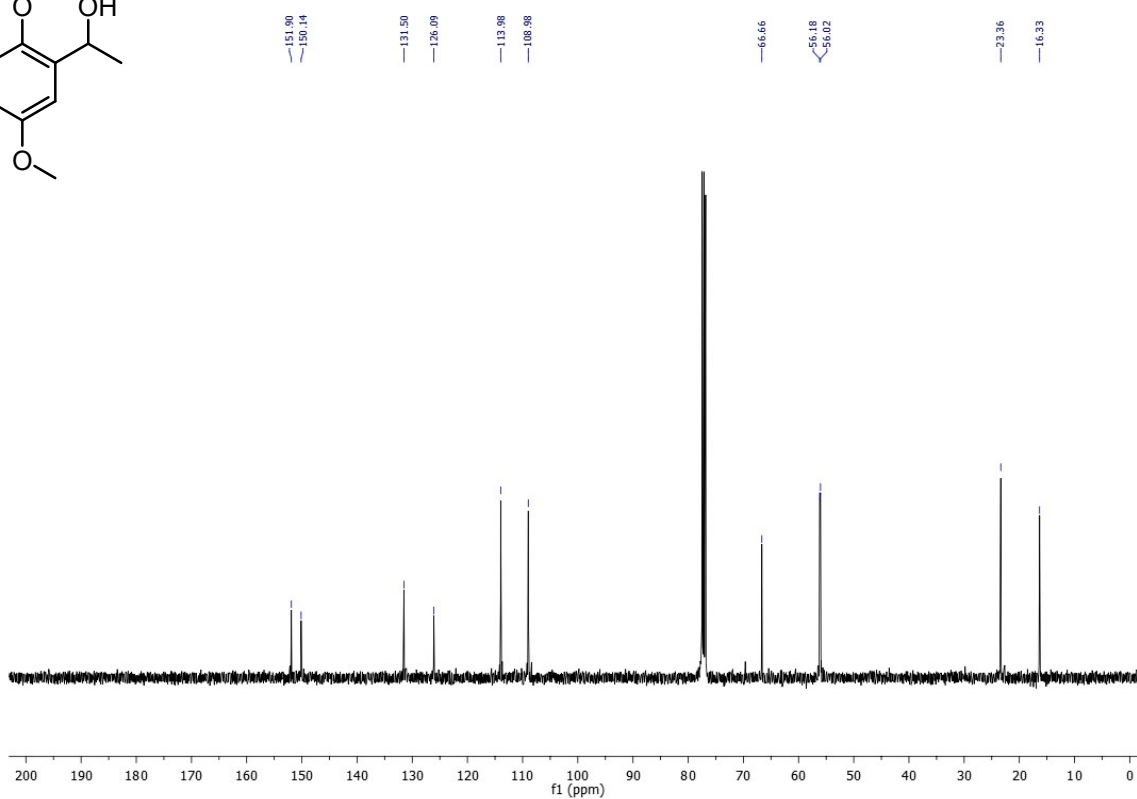




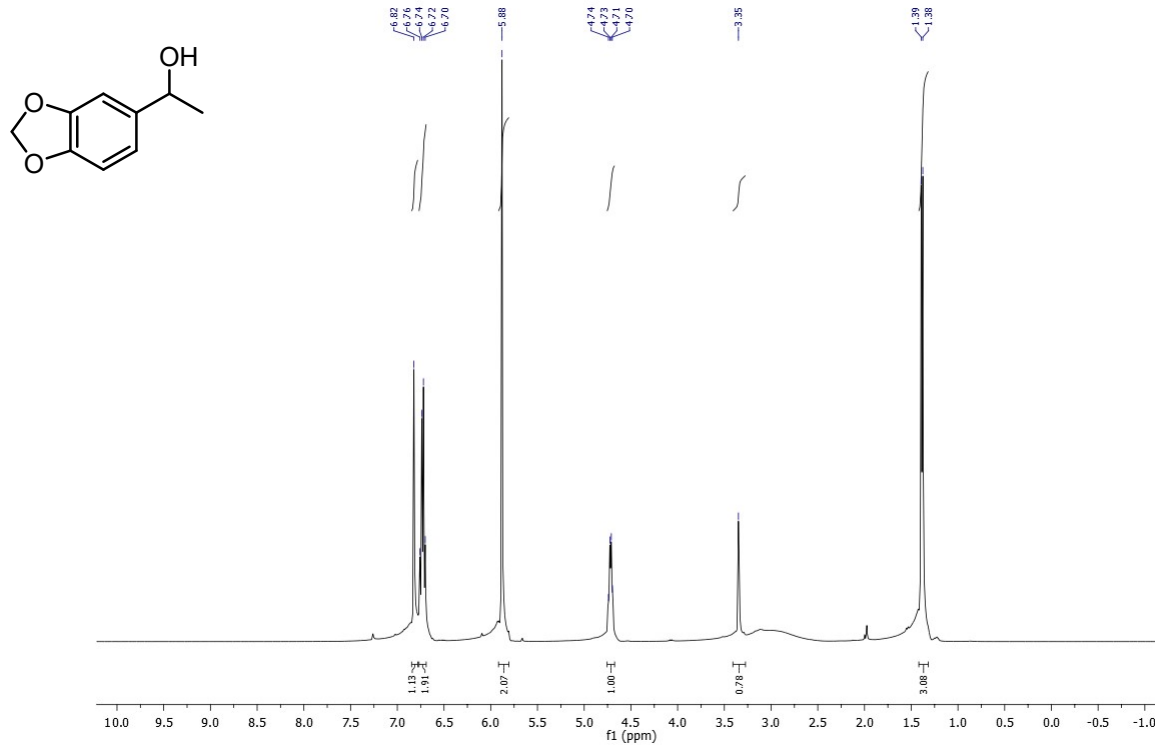
¹H NMR Spectrum of **8m** (CDCl₃, 400 MHz)



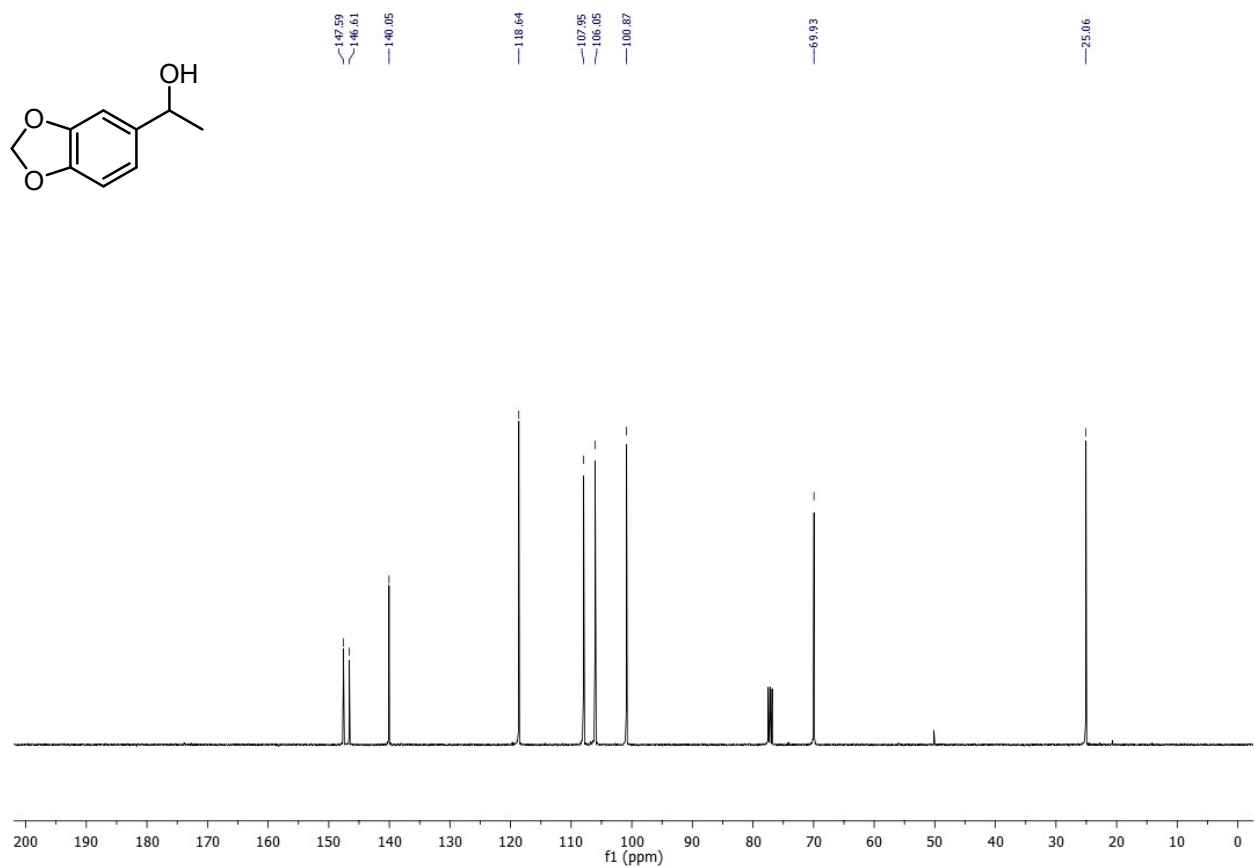
¹³C {¹H} NMR Spectrum of **8m** (CDCl₃, 100 MHz)

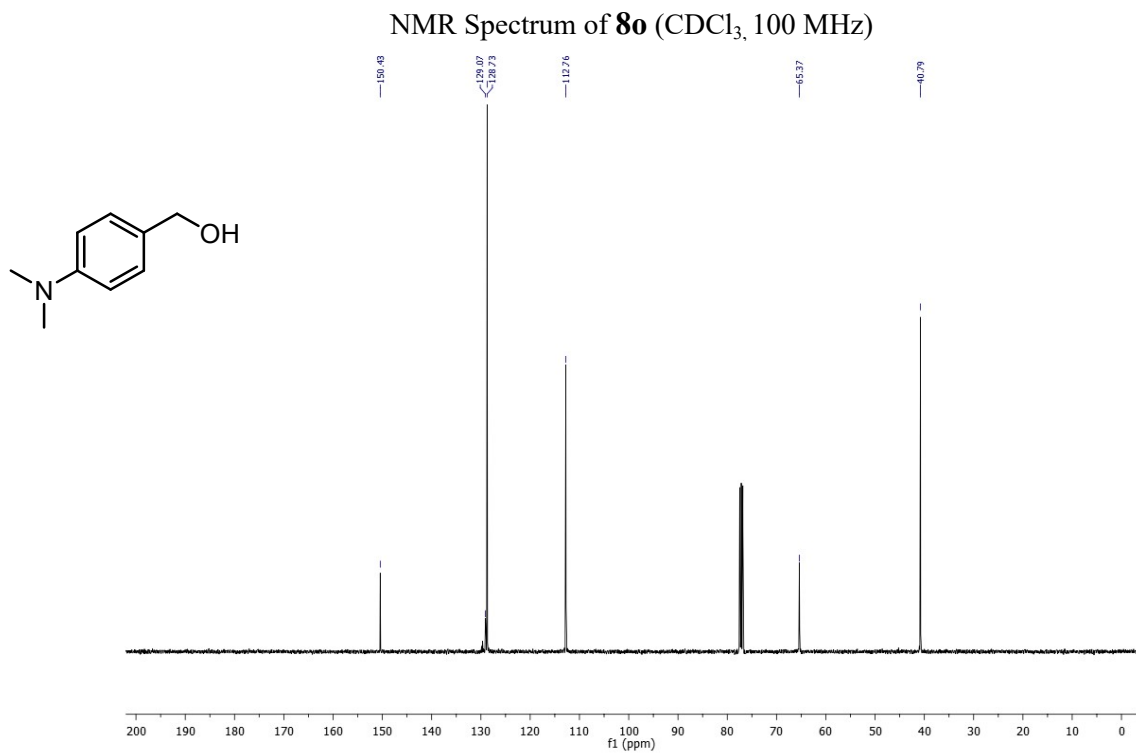
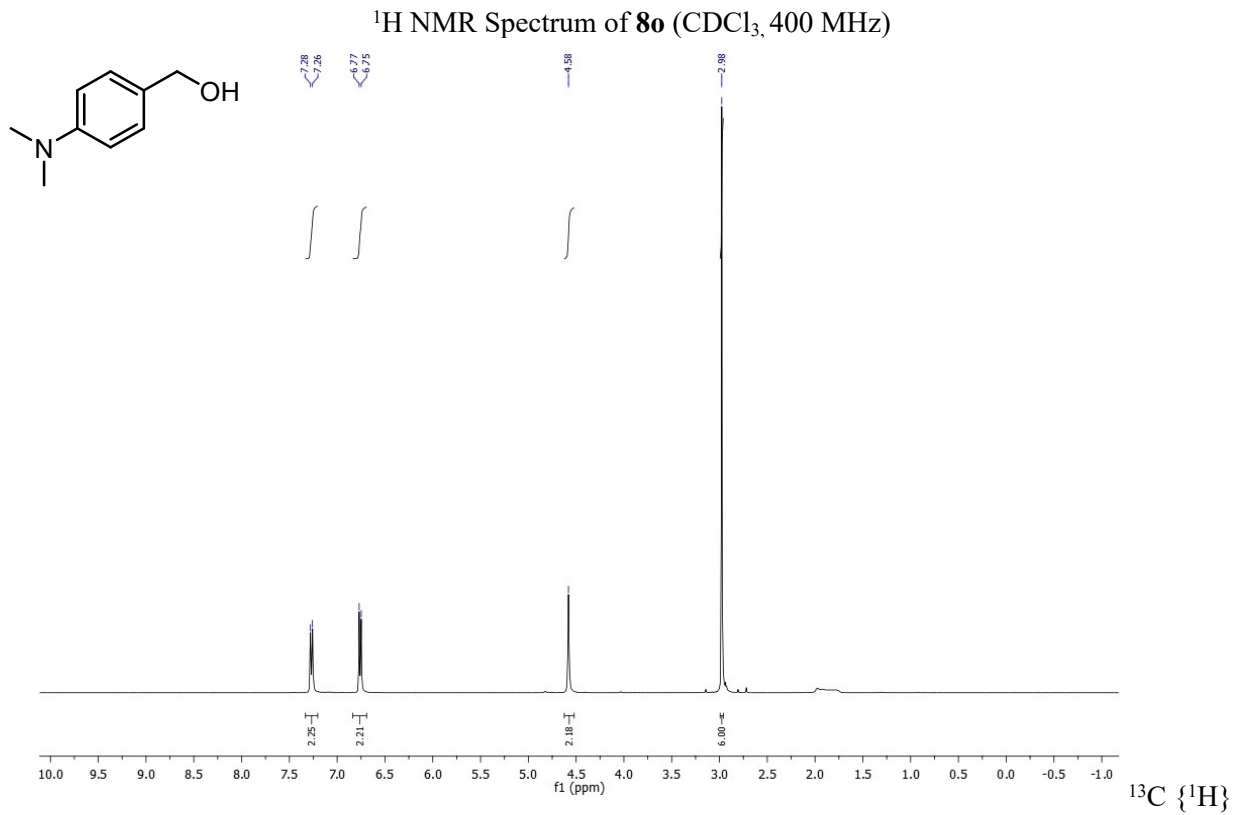


¹H NMR Spectrum of **8n** (CDCl₃, 400 MHz)

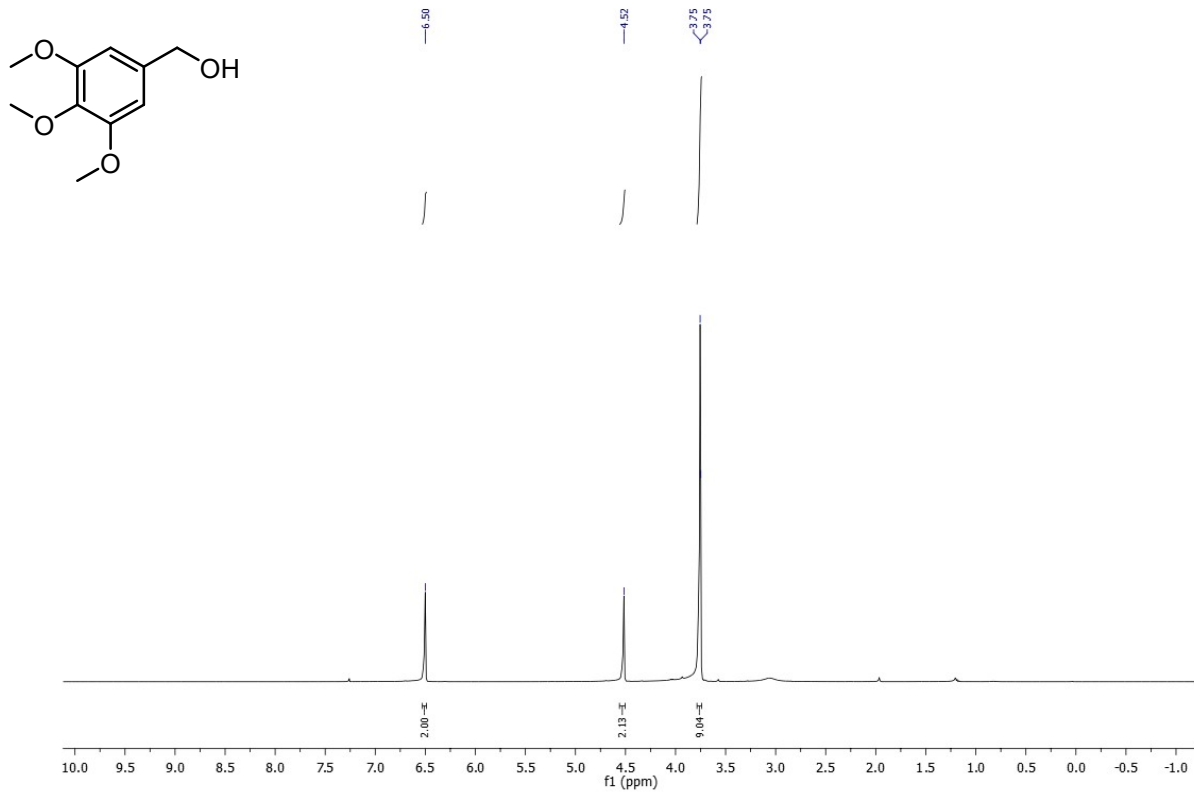


¹³C {¹H} NMR Spectrum of **8n** (CDCl₃, 100 MHz)

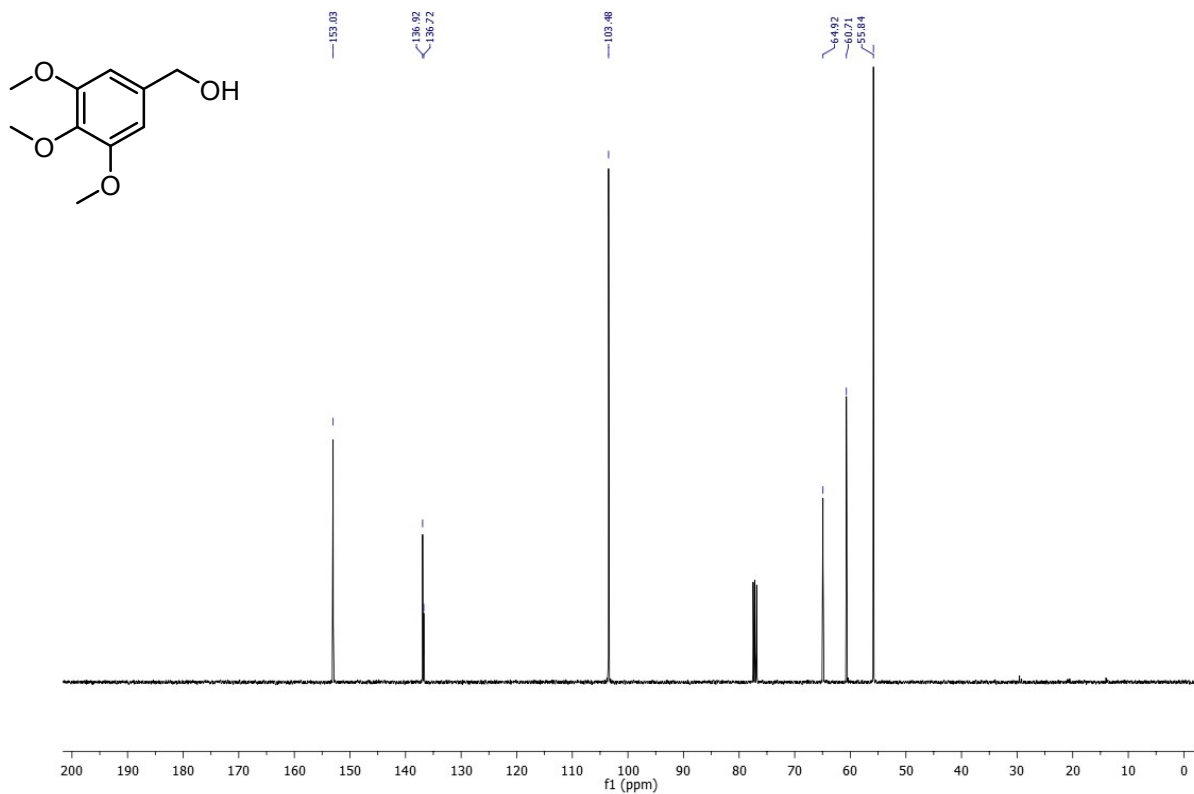


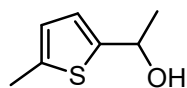


¹H NMR Spectrum of **8p** (CDCl₃, 400 MHz)

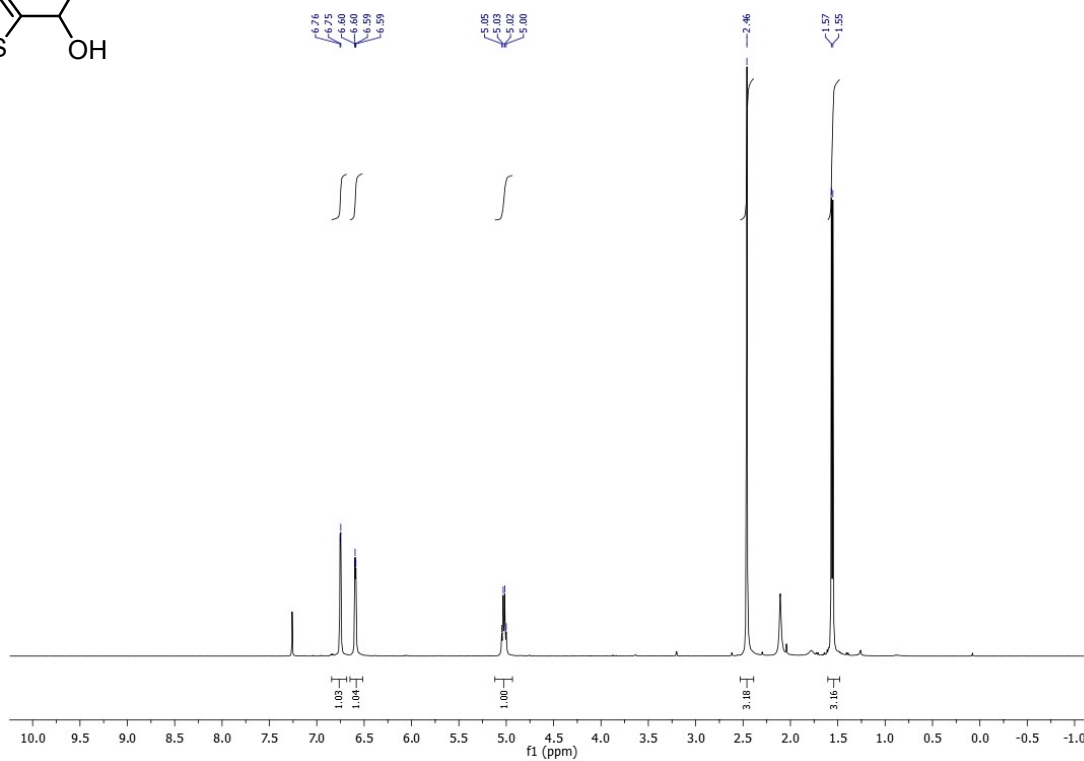


¹³C {¹H} NMR Spectrum of **8p** (CDCl₃, 100 MHz)

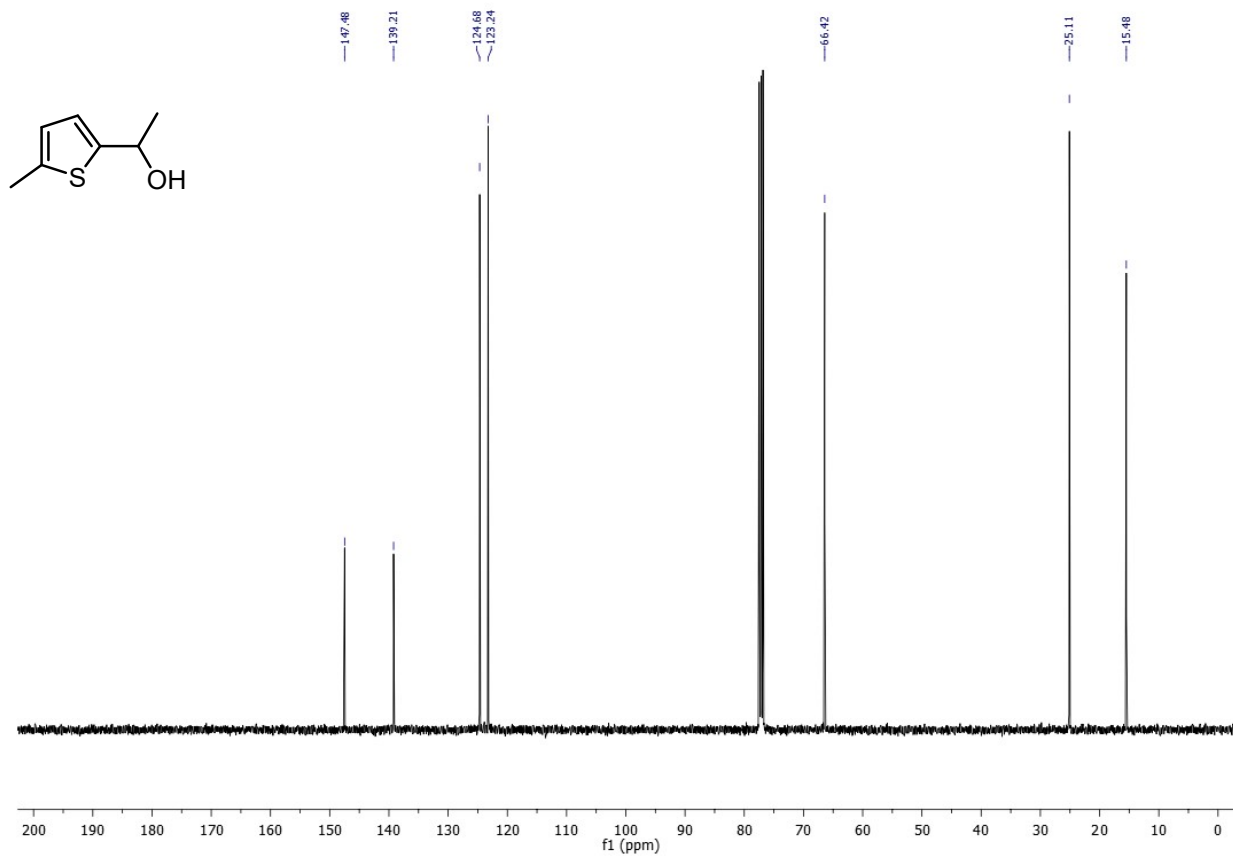




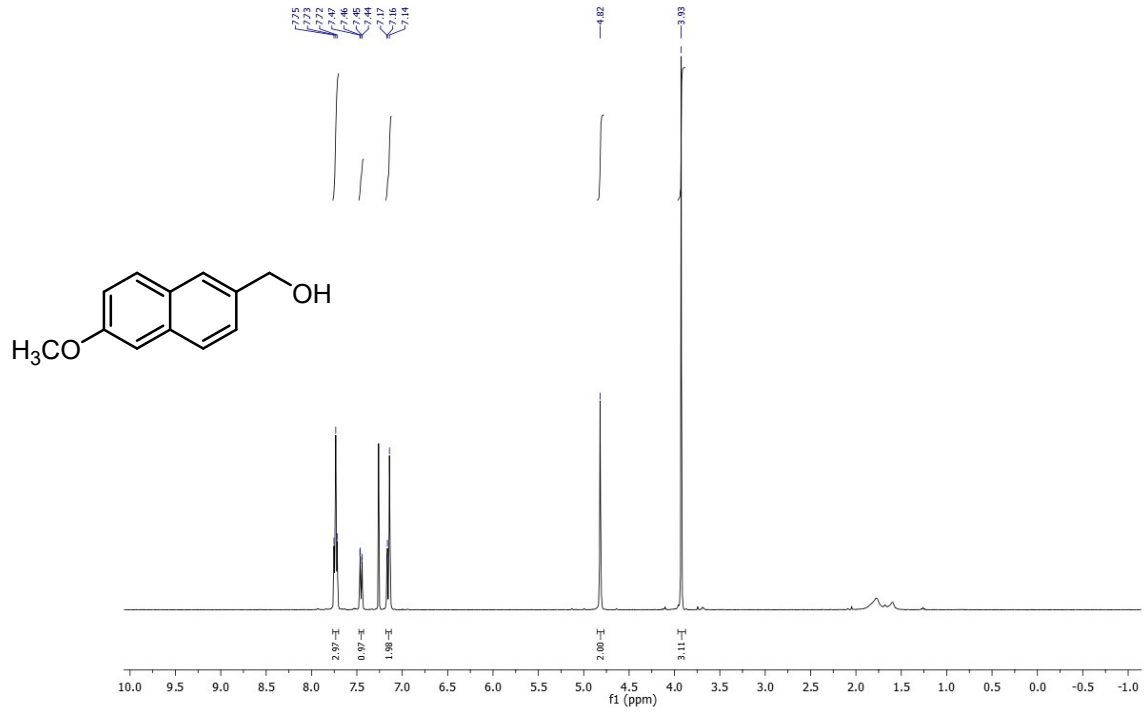
^1H NMR Spectrum of **8q** (CDCl_3 , 400 MHz)



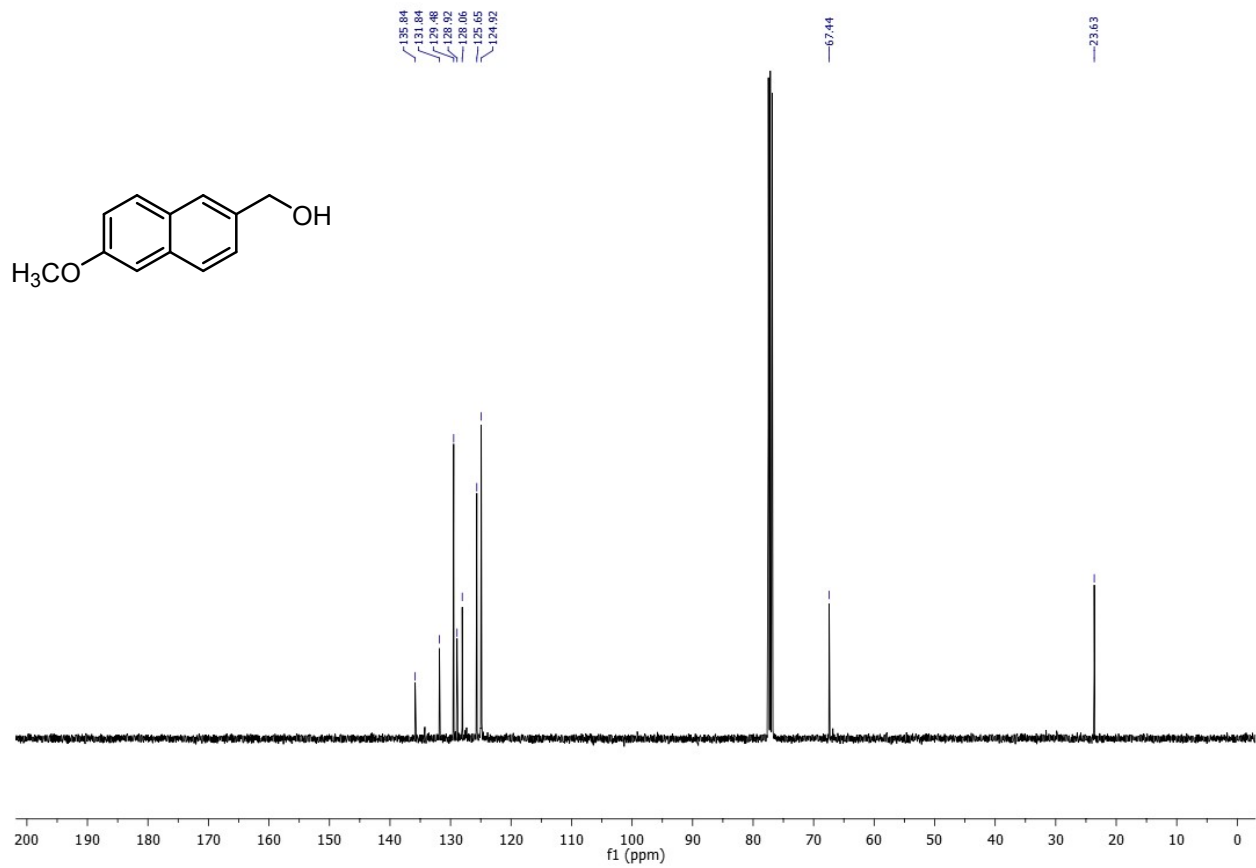
^{13}C $\{^1\text{H}\}$ NMR Spectrum of **8q** (CDCl_3 , 100 MHz)



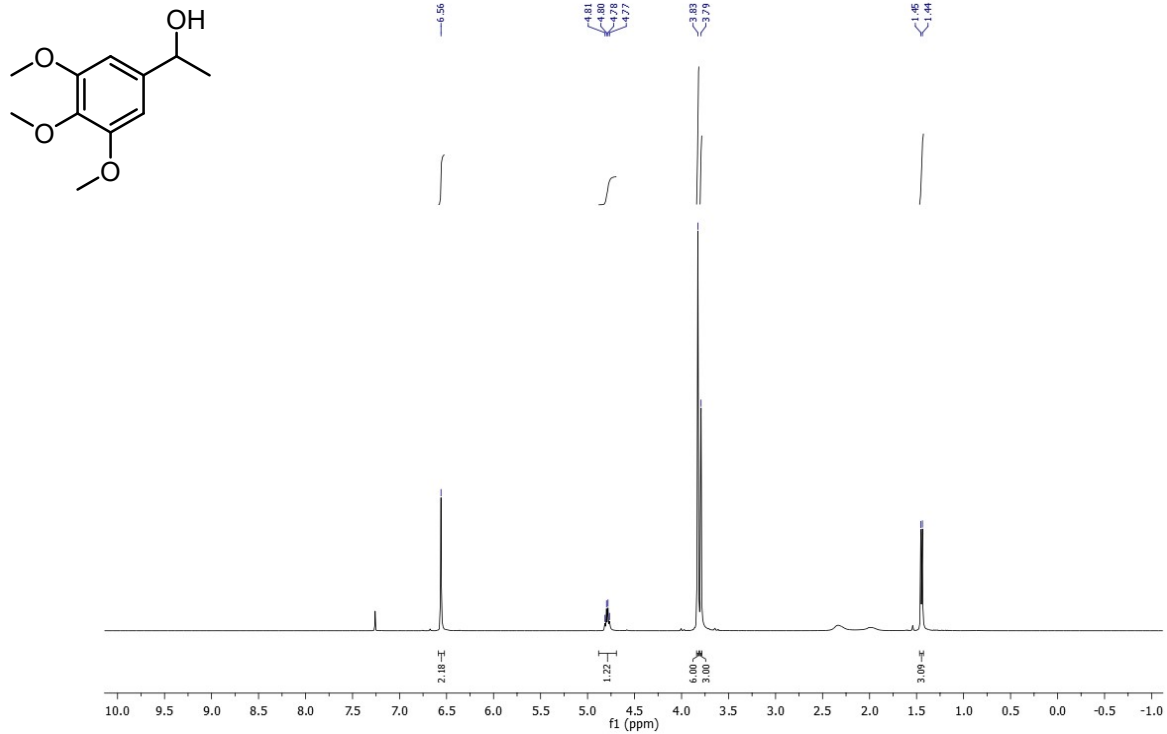
¹H NMR Spectrum of **8s** (CDCl₃, 400 MHz)



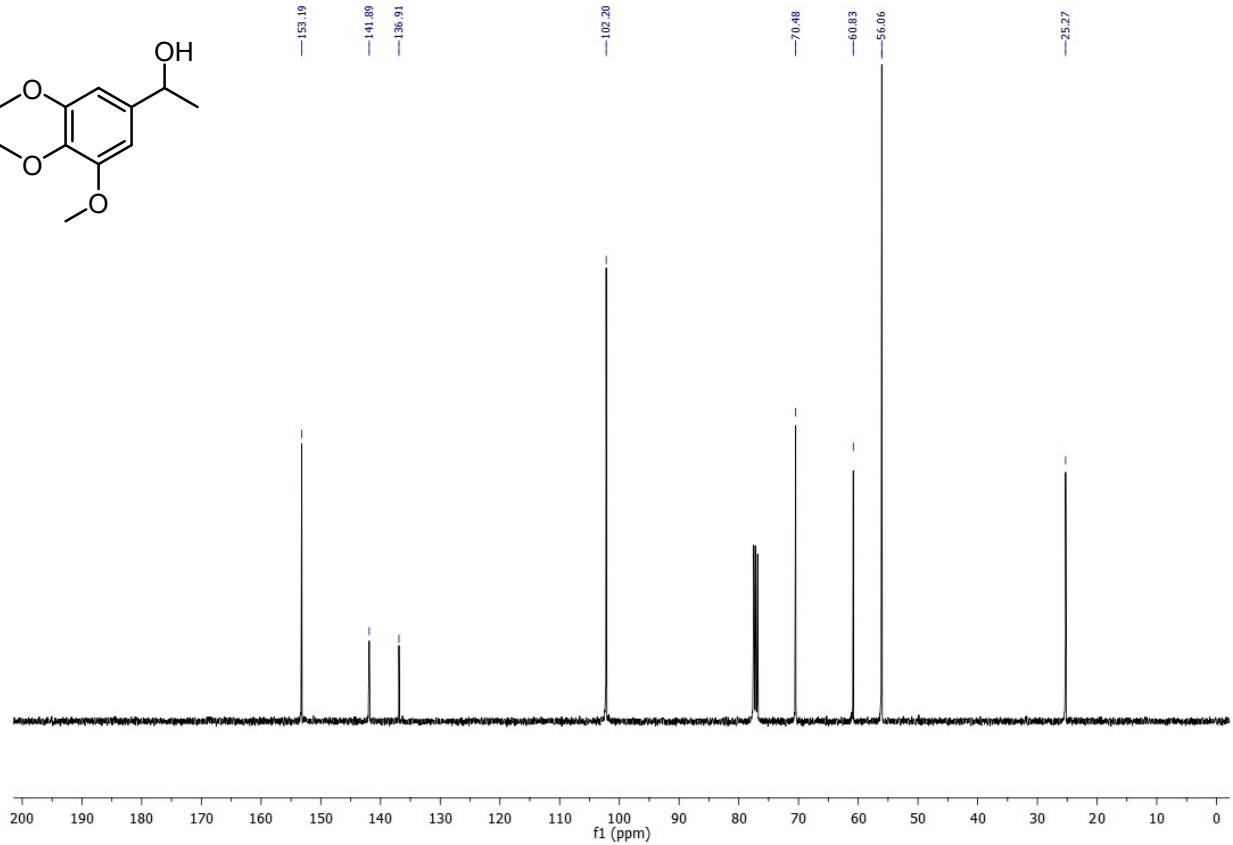
¹³C {¹H} NMR Spectrum of **8s** (CDCl₃, 100 MHz)



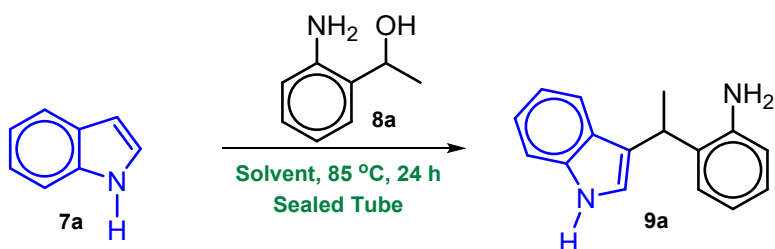
^1H NMR Spectrum of **8u** (CDCl_3 , 400 MHz)



^{13}C $\{^1\text{H}\}$ NMR Spectrum of **8u** (CDCl_3 , 100 MHz)



4) Reaction Standardization through HPLC:



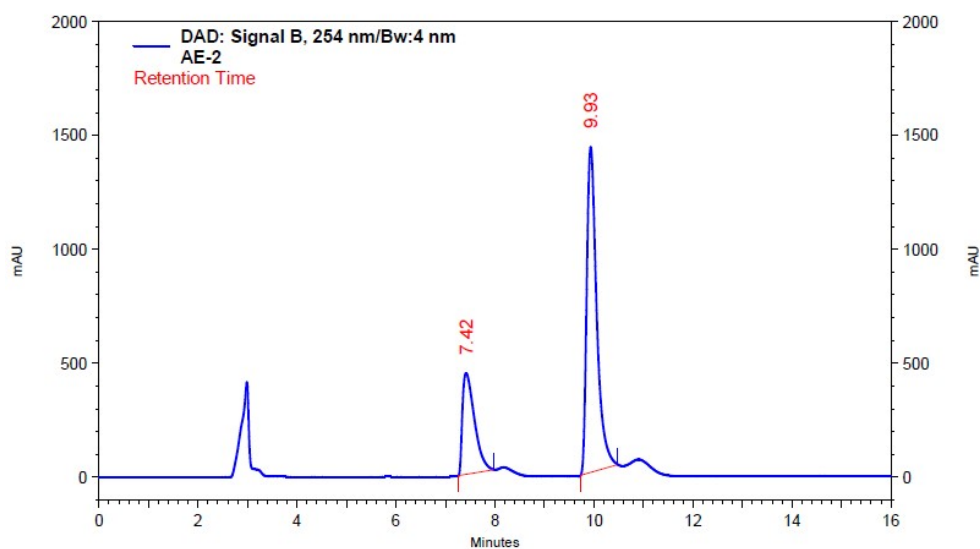
Area % Report When Reaction is Carried Out in Methanol

Data File: C:\Documents and Settings\user\Desktop\hplcMeOH (100%)-2.dat

Method: untitled.met

Acquired: 3/5/2024 12:43:17 AM (GMT +05:30)

Printed: 3/5/2024 1:02:40 AM (GMT +05:30)

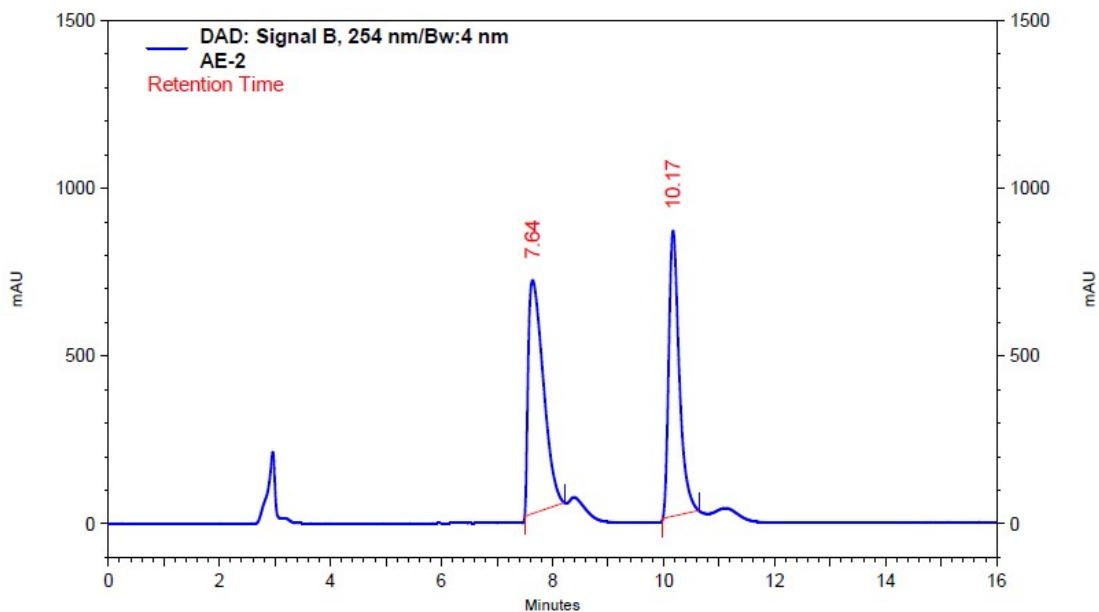


DAD: Signal B, 254 nm/Bw:4 nm Results

Retention Time	Area	Area %	Height	Height %
7.420	15749083	27.76	931122	23.72
9.933	41597266	72.24	2994924	76.28
Totals	57346349	100.00	3926046	100.00

Area % Report When Reaction is Carried Out in Water

Data File: C:\Documents and Settings\user\Desktop\auqib\H2O 100\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 11:08:38 PM (GMT +05:30)
 Printed: 3/5/2024 12:23:36 AM (GMT +05:30)

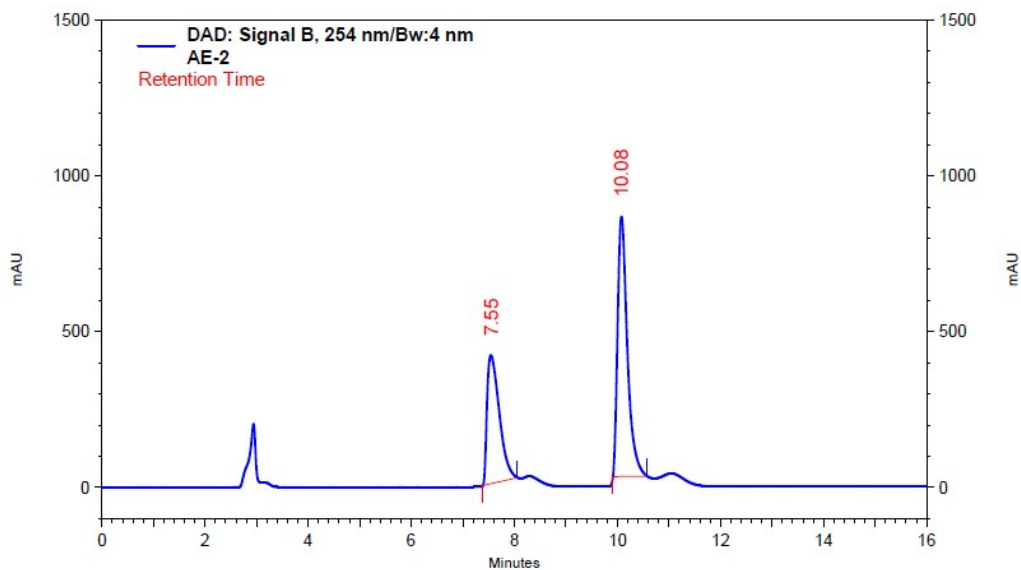


**DAD: Signal B,
 254 nm/Bw:4 nm
 Results**

Retention Time	Area	Area %	Height	Height %
7.640	27466212	54.80	1458730	44.99
10.173	23426722	45.20	1783662	55.01
Totals	50892934	100.00	3242392	100.00

Area % Report When Reaction is Carried Out in Ethanol

Data File: C:\Documents and Settings\user\Desktop\EtOH-100.rslt\AE-2.dat Method: untitled.met
 Acquired: 3/2/2024 2:04:42 PM (GMT +05:30)
 Printed: 3/3/2024 5:52:15 PM (GMT +05:30)



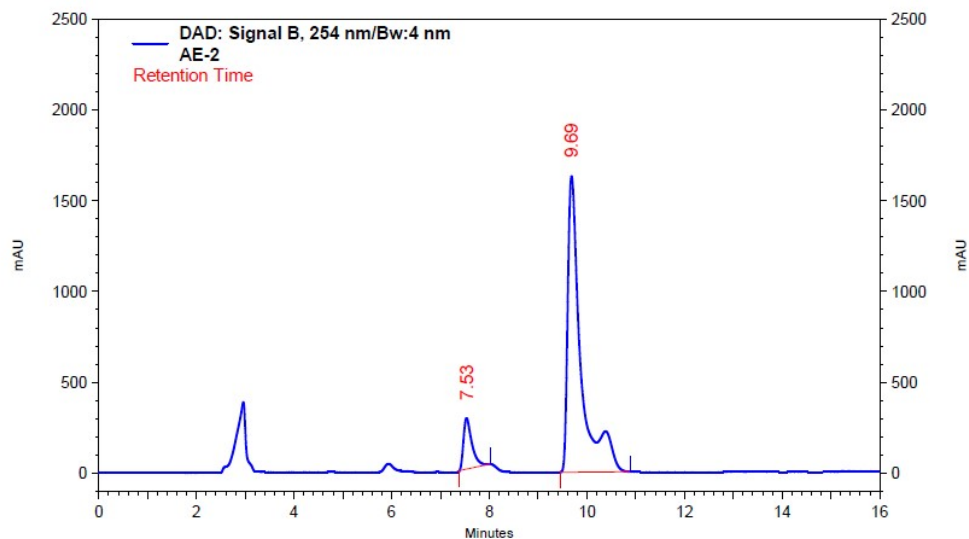
**DAD: Signal B,
 254 nm/Bw:4 nm
 Results**

Retention Time	Area	Area %	Height	Height %
7.547	14249211	40.03	864078	33.07
10.080	22966402	59.97	1748590	66.93

Totals	37215613	100.00	2612668	100.00
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Area % Report When Reaction is Carried Out in Isopropanol

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\ISOPROPENOL FINAL NEW
 NEW.rslt\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 11:51:00 PM (GMT +05:30)
 Printed: 3/5/2024 12:51:32 AM (GMT +05:30)



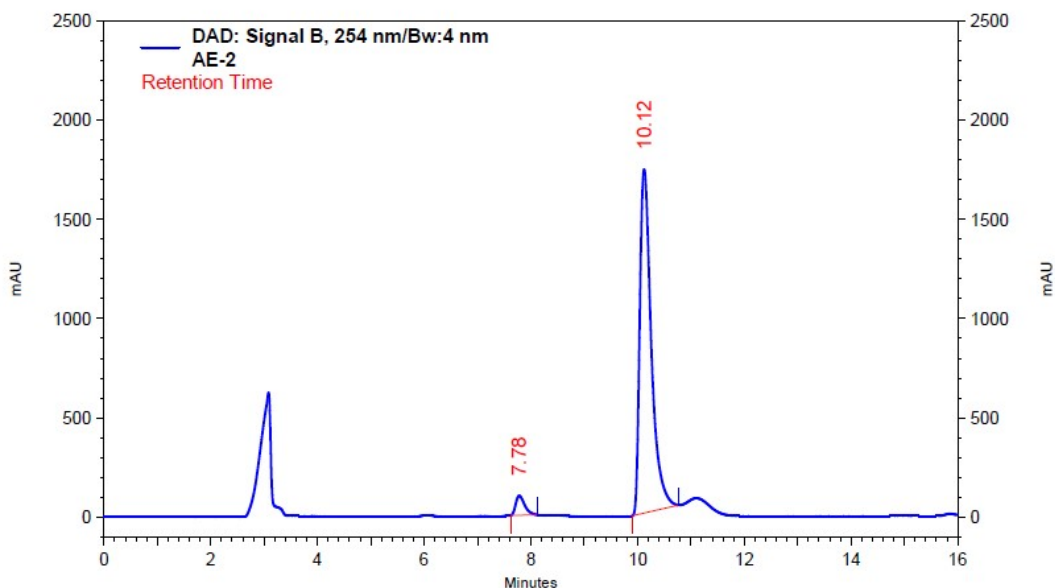
DAD: Signal B, 254 nm/Bw:4 nm Results

Retention Time	Area	Area %	Height	Height %
7.533	6993055	8.45	589571	14.70
9.687	64537025	91.55	3420528	85.30

Totals	71530080	100.00	4010099	100.00
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Area % Report When Reaction is Carried Out in n-Butanol

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\N butanol FINAL NEW.rsl\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 10:03:01 PM (GMT +05:30)
 Printed: 3/5/2024 12:54:26 AM (GMT +05:30)



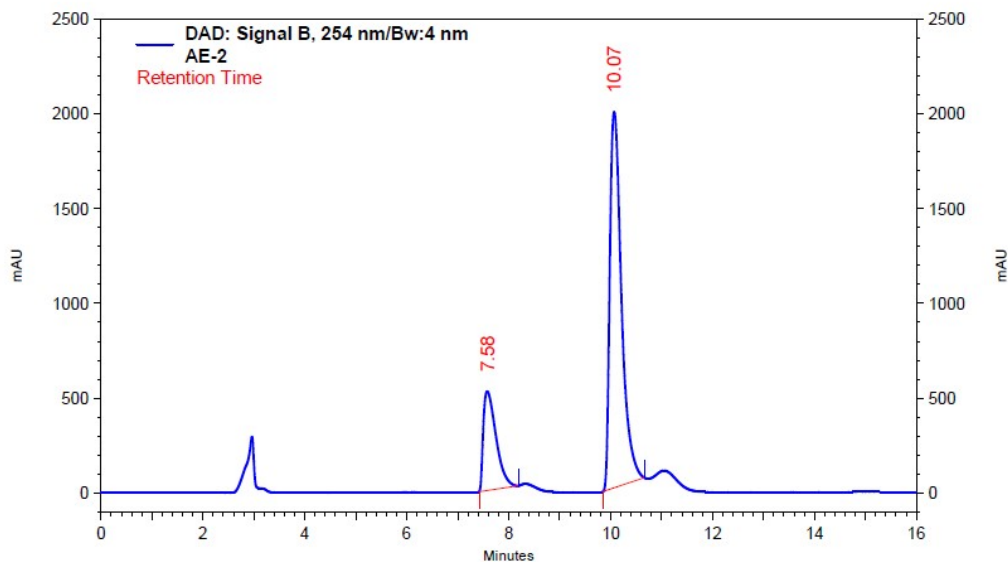
**DAD: Signal B,
 254 nm/Bw:4 nm
 Results**

Retention Time	Area	Area %	Height	Height %
7.780	2405583	4.63	205714	5.36
10.120	54400847	95.27	3631391	94.64

Totals	56806430	100.00	3837105	100.00
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Area % Report When Reaction is Carried Out in tert-Butanol

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\tert butanol FINAL NEW.rslt\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 9:17:49 PM (GMT +05:30)
 Printed: 3/5/2024 12:46:31 AM (GMT +05:30)



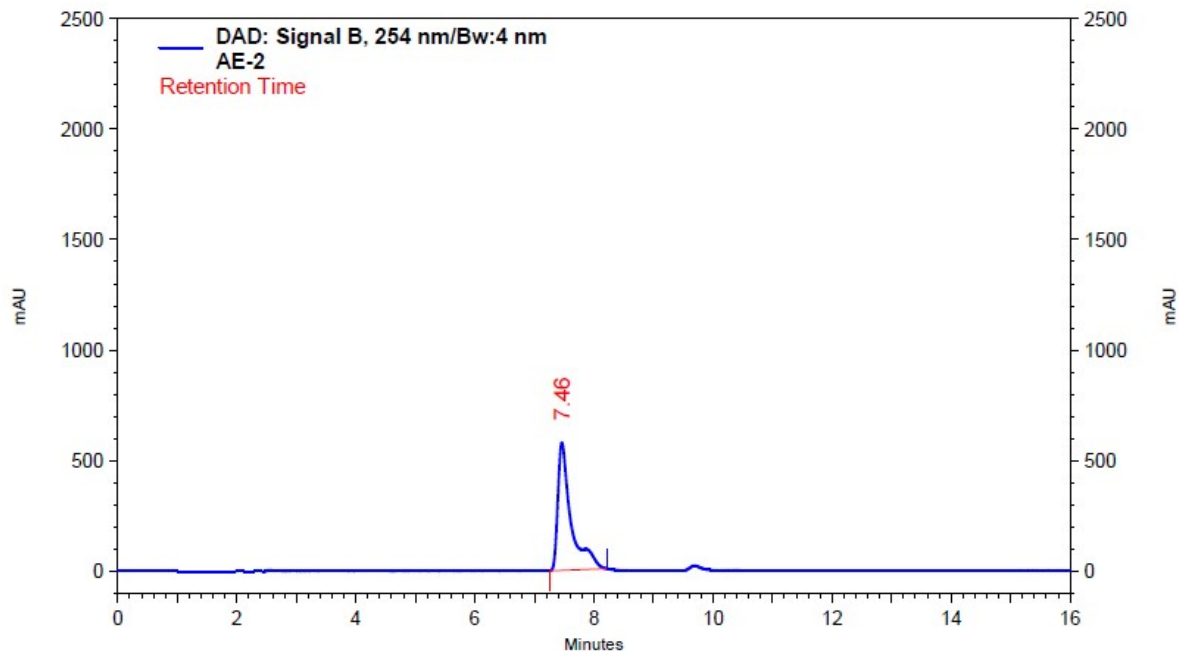
DAD: Signal B, 254 nm/Bw:4 nm Results

Retention Time	Area	Area %	Height	Height %
7.580	19028630	24.70	1097364	20.88
10.067	66234600	75.30	4159156	79.12

Totals	85263230	100.00	5256520	100.00
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Area % Report when reaction is carried out in HFIP

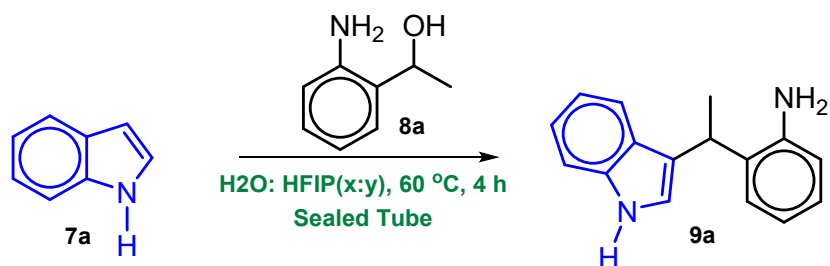
Data File: C:\Documents and Settings\user\Desktop\HFIP .result\AE-2.dat Method: untitled.met
 Acquired: 3/4/2024 8:28:56 PM (GMT +05:30)
 Printed: 3/5/2024 12:27:55 AM (GMT +05:30)



DAD: Signal B, 254 nm/Bw:4 nm Results

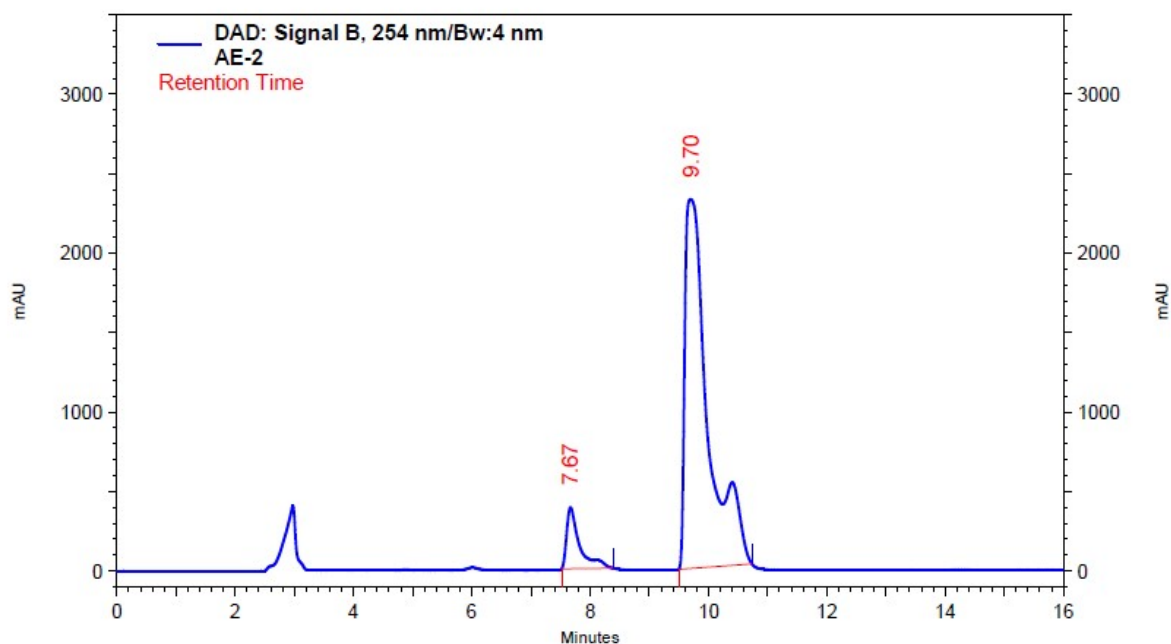
Retention Time	Area	Area %	Height	Height %
7.460	18667654	100.00	1211003	100.00

Totals	18667654	100.00	1211003	100.00
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Area % Report when reaction is carried out in H₂O

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\AQIB H2O.rslt\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 1:09:58 AM (GMT +05:30)
 Printed: 3/4/2024 1:28:18 AM (GMT +05:30)

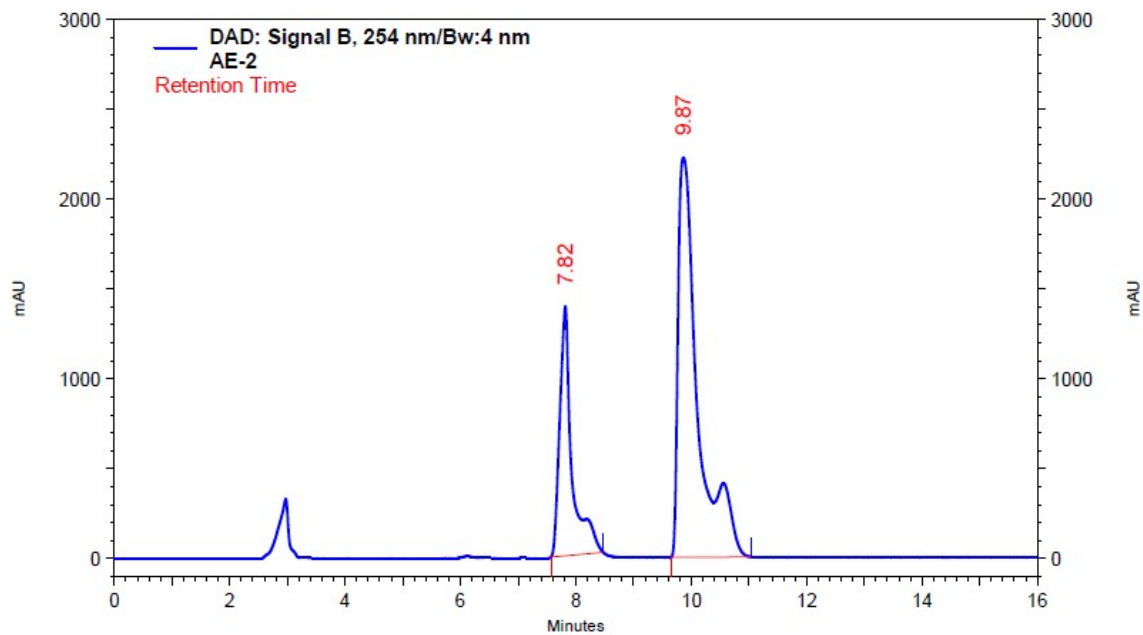


DAD: Signal B, 254 nm/Bw:4 nm Results

Retention Time	Area	Area %	Height	Height %
7.667	12273542	11.62	807646	14.22
9.700	131329868	88.38	4871530	85.78
Totals	143603410	100.00	5679176	100.00

Area % Report when reaction is carried out in H₂O: HFIP (9:1)

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\AQIB 91.rsl\AE-2.dat
 Method: untitled.met
 Acquired: 3/4/2024 12:45:38 AM (GMT +05:30)
 Printed: 3/4/2024 1:03:58 AM (GMT +05:30)



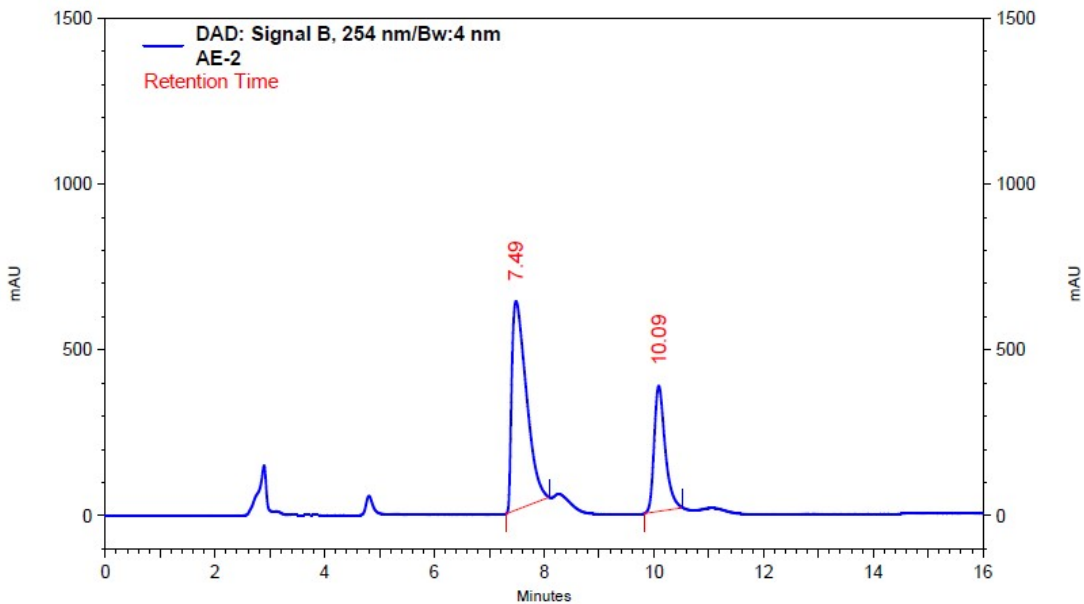
**DAD: Signal B,
254 nm/Bw:4 nm
Results**

Retention Time	Area	Area %	Height	Height %
7.820	42780126	29.89	2910892	38.43
9.867	110581383	70.11	4663803	61.57

Totals	153361509	100.00	7574695	100.00
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Area % Report when reaction is carried out in H₂O: HFIP (8:2)

Data File: C:\Documents and Settings\user\Desktop\AUQIB 8/2-.rslt\AE-2.dat Method: untitled.met
 Acquired: 3/4/2024 12:14:53 AM (GMT +05:30)
 Printed: 3/4/2024 12:38:19 AM (GMT +05:30)



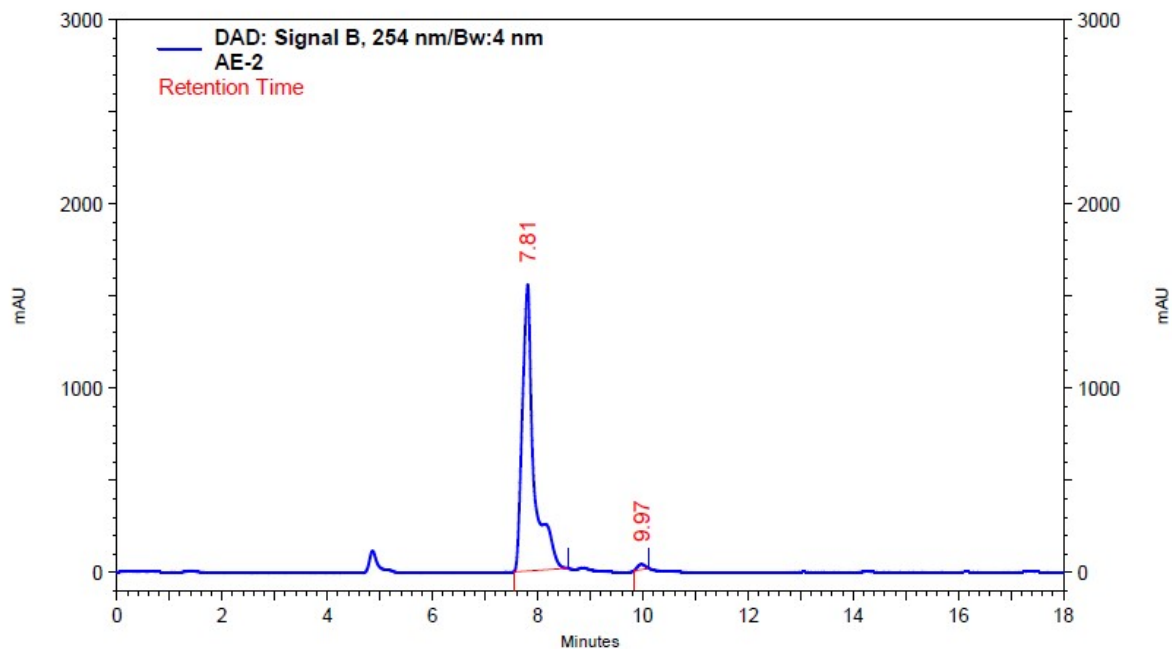
**DAD: Signal B,
254 nm/Bw:4 nm
Results**

Retention Time	Area	Area %	Height	Height %
7.487	24529316	68.89	1320983	62.47
10.087	11078158	31.11	793628	37.53

Totals	35607474	100.00	2114611	100.00
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Area % Report % when reaction is carried out in H₂O: HFIP (7:3)

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\AQIB 73 NEW.rsl\AE-2.dat
 Method: untitled.met
 Acquired: 3/3/2024 9:54:38 PM (GMT +05:30)
 Printed: 3/4/2024 12:58:55 AM (GMT +05:30)



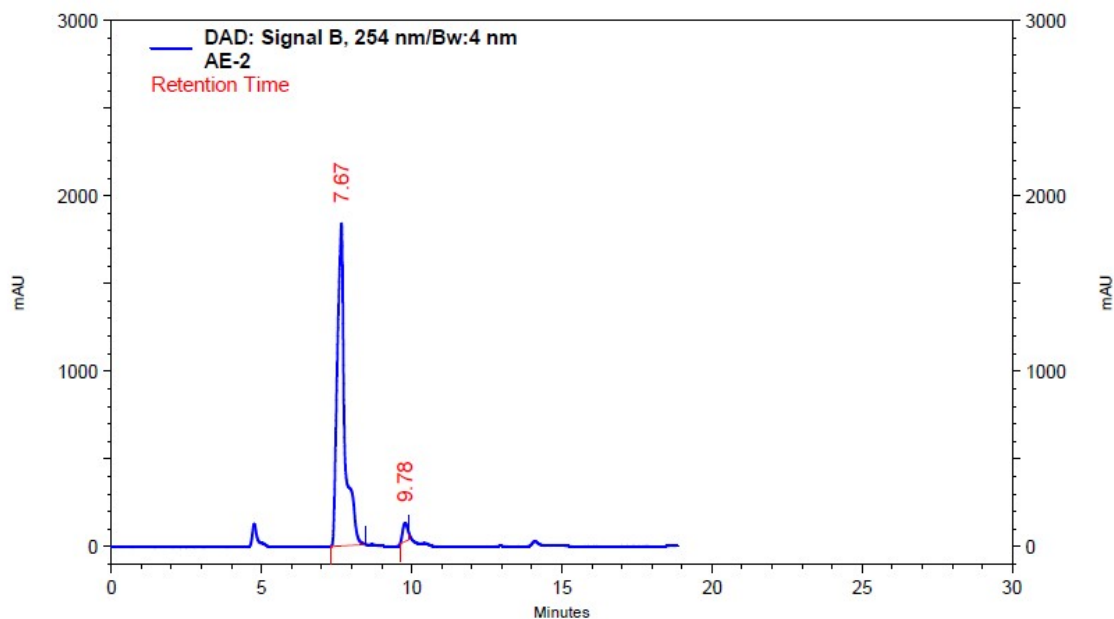
**DAD: Signal B,
 254 nm/Bw:4 nm
 Results**

Retention Time	Area	Area %	Height	Height %
7.813	49557538	99.51	3259982	98.17
9.973	591464	0.49	60793	1.83

Totals	50149002	100.00	3320775	100.00
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Area % Report when reaction is carried out in H₂O: HFIP (6:4)

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\AQIB 32.rslt\AE-2.dat
 Method: untitled.met
 Acquired: 3/3/2024 9:20:45 PM (GMT +05:30)
 Printed: 3/4/2024 12:55:30 AM (GMT +05:30)



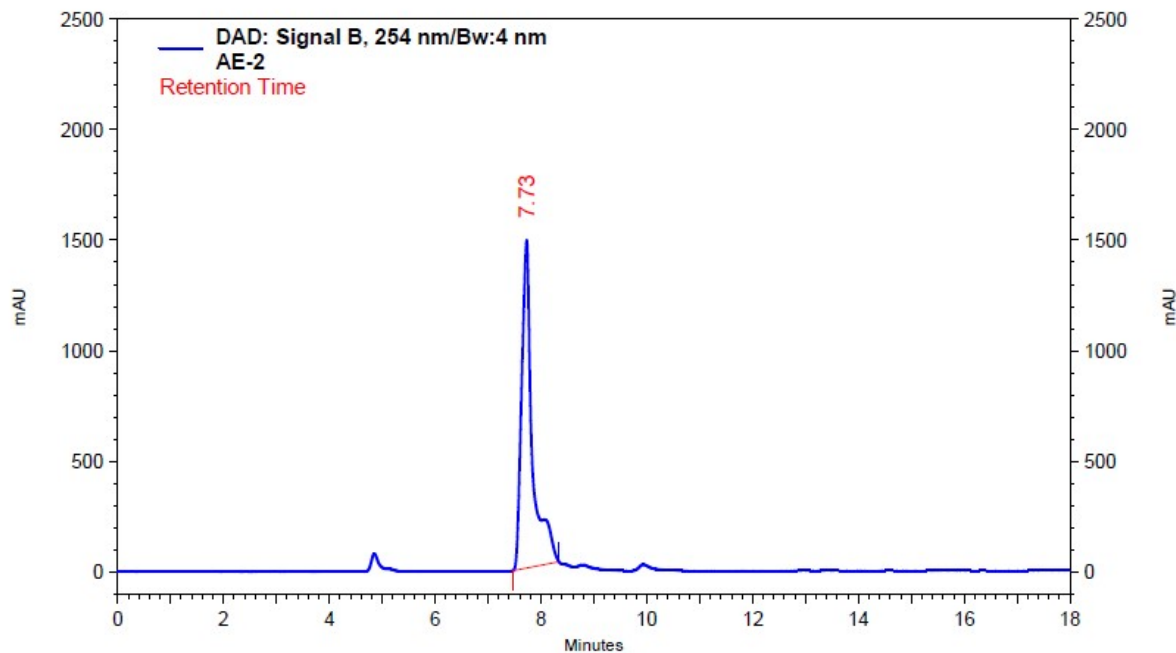
DAD: Signal B, 254 nm/Bw:4 nm Results

Retention Time	Area	Area %	Height	Height %
7.667	67496173	96.43	3852936	94.58
9.780	2498622	3.57	220967	5.42

Totals	69994795	100.00	4073903	100.00
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Area % Report when reaction is carried out in H₂O: HFIP (1:1)

Data File: C:\Documents and Settings\user\Desktop\MOMO SIR\AQIB 11.rslt\AE-2.dat
 Method: untitled.met
 Acquired: 3/3/2024 9:00:40 PM (GMT +05:30)
 Printed: 3/4/2024 12:50:57 AM (GMT +05:30)

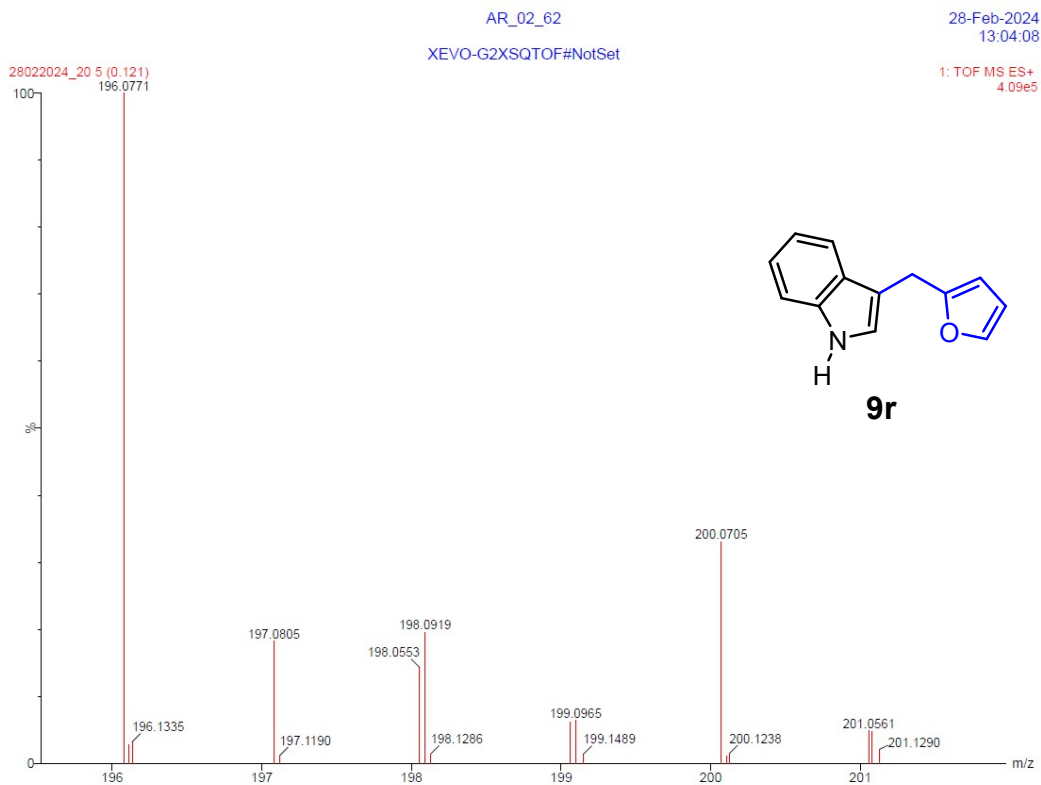
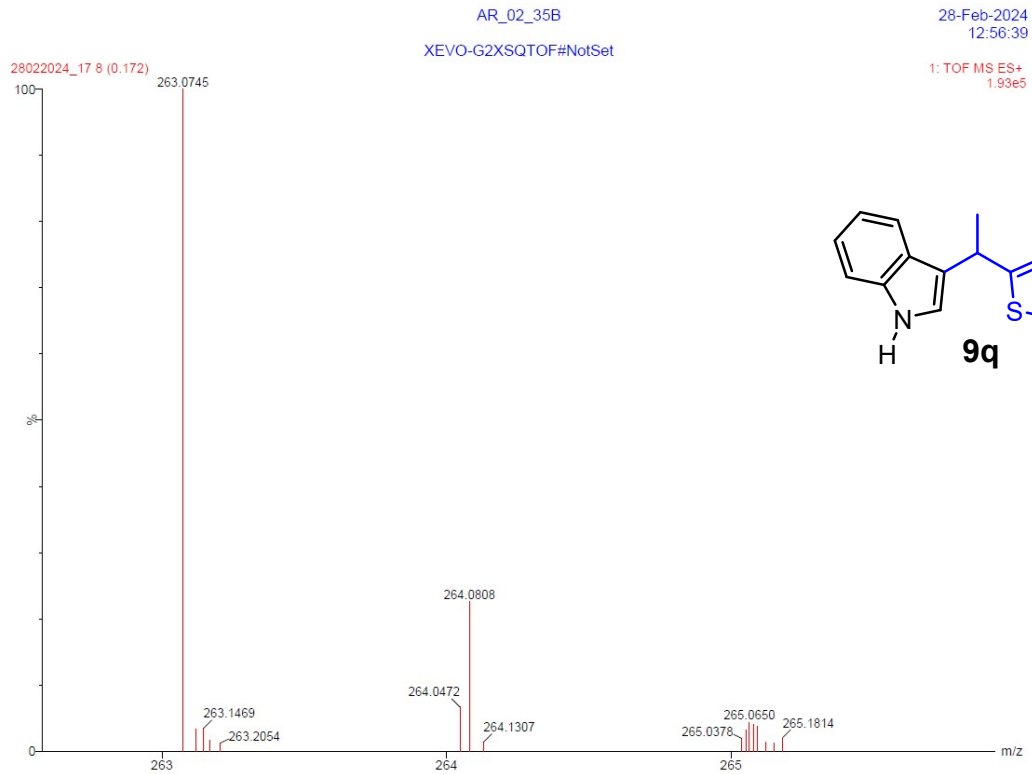


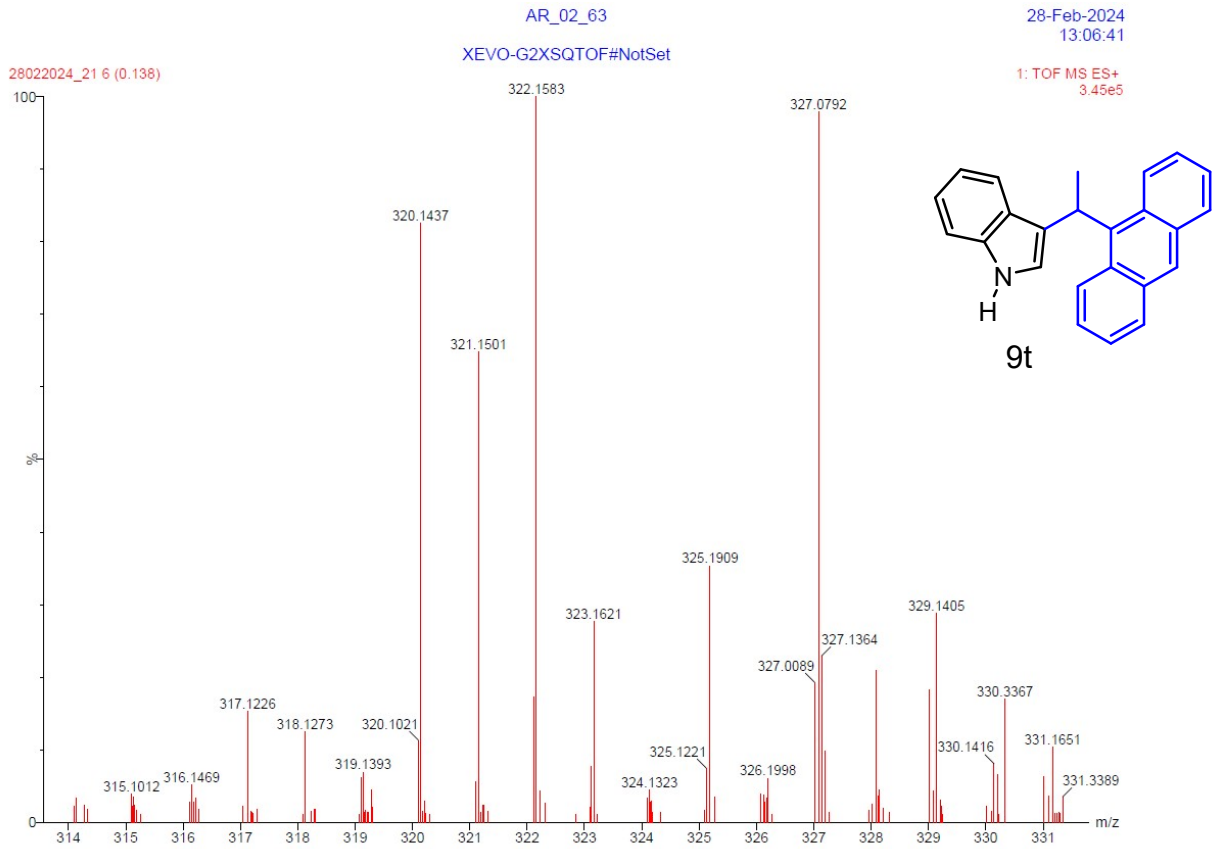
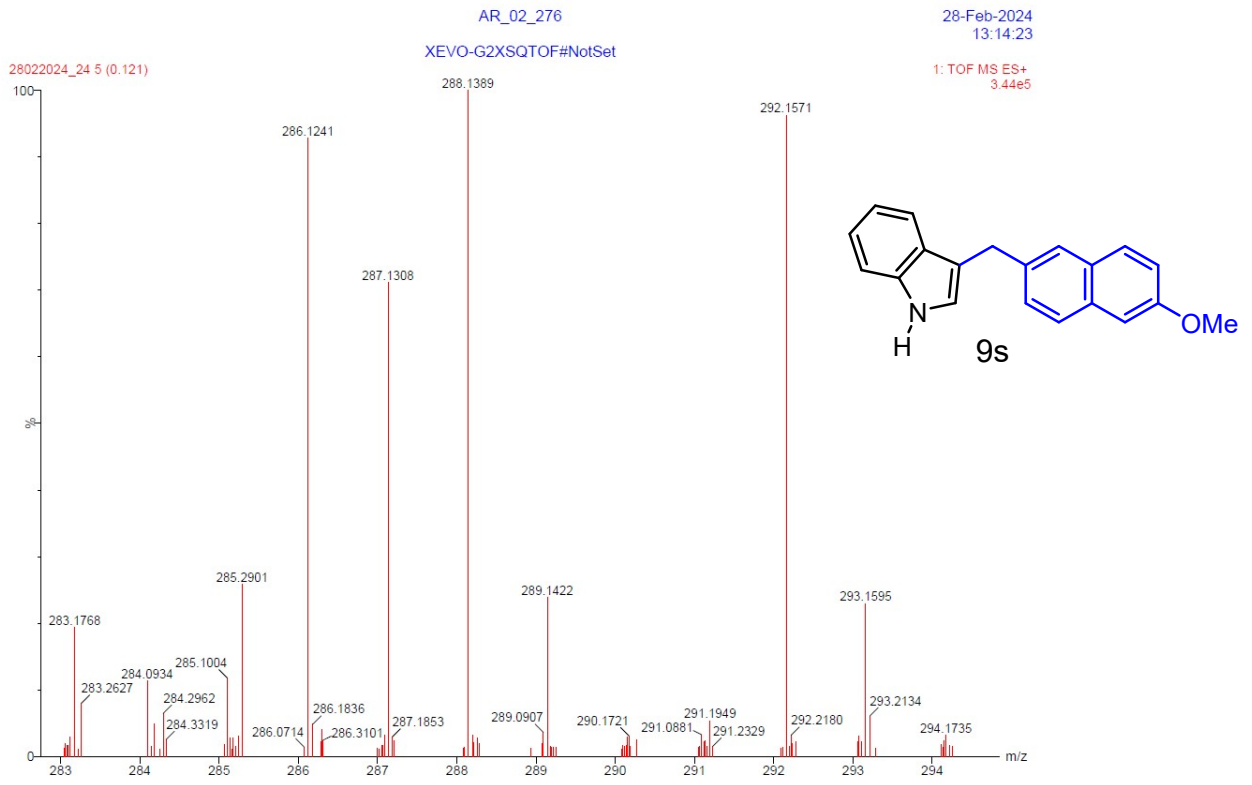
**DAD: Signal B,
 254 nm/Bw:4 nm
 Results**

Retention Time	Area	Area %	Height	Height %
7.727	43693272	100.00	3105474	100.00
Totals				
	43693272	100.00	3105474	100.00

5) HRMS data

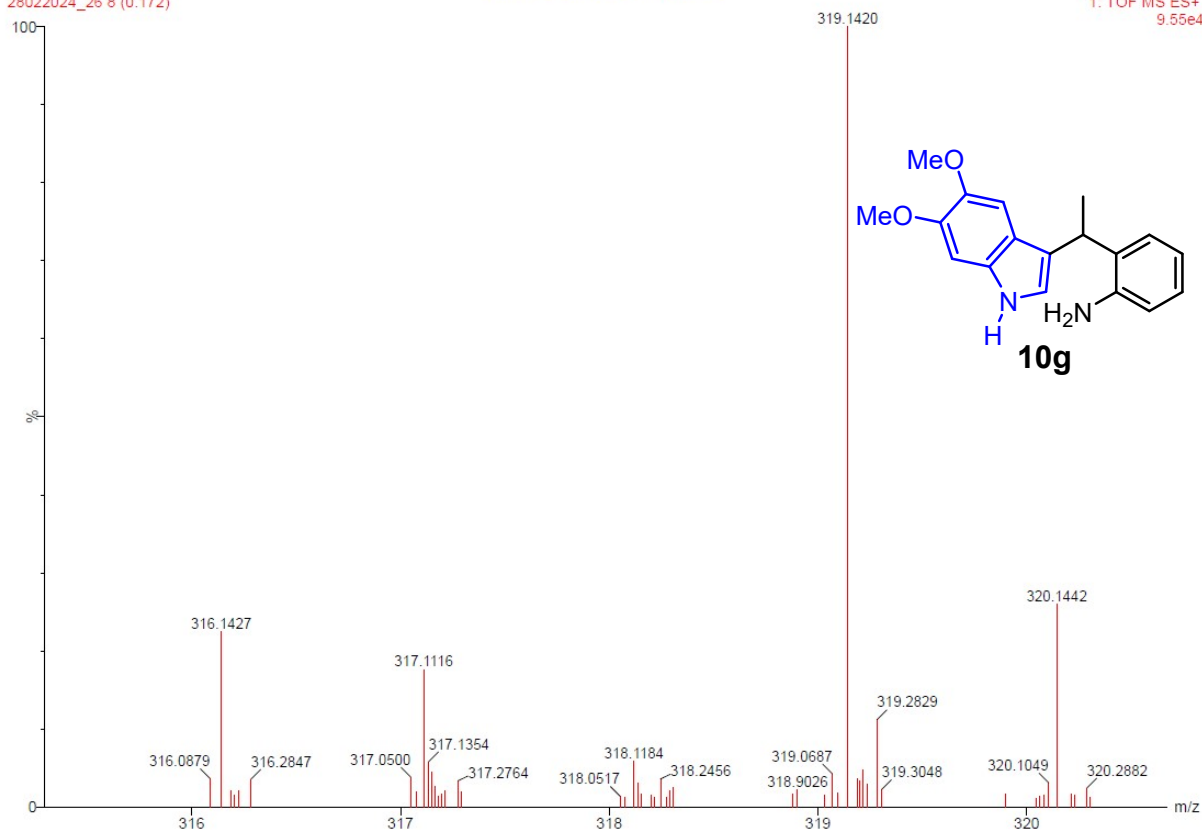
HRMS of Compounds **9q**, **9r**, **9s** **9t** and **10g** are attached below: High-resolution mass spectrometry (HRMS) was measured on an electrospray ionization (ESI) apparatus using the





28022024_26 8 (0.172)

1: TOF MS ES+
9.55e4



References:

1. K, Tanemura, Results Chem., 2022, 4, 100486.