Supporting Information

Zwitterionic Salt Catalyzed Site-Selective mono-Bromination of Arenes

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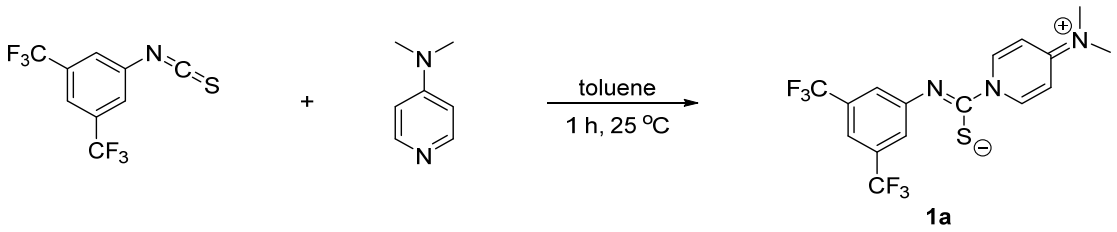
(A) General information

All reactions that required anhydrous conditions were carried by standard procedures under argon atmosphere unless otherwise stated. Commercially available reagents were used as received. The solvents were dried by distillation over the appropriate drying reagents. NMR spectra were recorded on a Brucker ADVANCE III 400MHz spectrometer (^{1}H NMR: 400 MHz, ^{13}C NMR: 100 MHz). Chemical shifts (δ) were reported in ppm relative to $CDCl_3$ (δ 7.26) for the ^{1}H NMR and to $CDCl_3$ (δ 77.16) for the ^{13}C NMR measurements. Mass spectra were recorded on Thermo Finnigan MAT 95 XL spectrometer and Bruker solariX 9.4 Tesla FTICR spectrometer. Thin-layer chromatography (TLC) was carried out on Merck silica gel 60F-254 (0.25 mm) Plates. Flash column chromatography was performed on Merck silica gel (60, particle size 0.040–0.063 mm).

(B) Catalyst preparation



1a

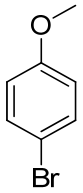
(*Z*)-*N*-[3,5-bis(trifluoromethyl)phenyl]-4-(dimethyliminio)pyridine-1(4H)-carbimidothioate (1a)

The catalyst **1a** was prepared according to the method in the literature.[1]

4-dimethylaminopyridine (367 mg, 3 mmol) was added to a solution of 3,5-bis(trifluoromethyl)phenyl isothiocyanate (814 mg, 3 mmol) in anhydrous toluene (6 mL) at 25 ^{o}C. The resultant mixture was stirred for 1 h. The precipitated product was further recrystallized from toluene to provide a yellow solid of **1a** (2.5 mmol). A stock solution of **1a** was prepared by dissolving 1.0 mmol of **1a** in 100 mL anhydrous dichloromethane.

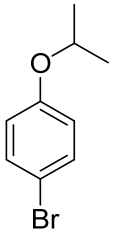
(C) General procedure for the bromination of arene 2 catalyzed by 1a

To a solution of zwitterionic catalyst **1a** in dichloromethane (*c* = 0.00025 M, 1.0 mL) was added substrate **2** (0.5 mmol) and NBS (0.525 mmol) in the absence of light. The resulting mixture was stirred at 25 ^{o}C and monitored by TLC. Upon completion, the reaction was quenched with saturated aqueous Na_2SO_3 (3 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/EtOAc = 100:1 to 5:1) to yield the corresponding brominated product **3**.



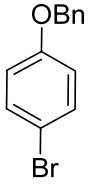
1-Bromo-4-methoxybenzene (3a)[2]

89.9 mg product **3a** was isolated in 96% yield. Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 3H), 6.79 (d, *J* = 8.9 Hz, 2H), 7.38 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 132.3, 115.8, 112.9, 55.5; HRMS (EI) calcd for C_7H_7BrO *m/z* $[M]^+$: 185.9675, found: 185.9679.



1-Bromo-4-isopropoxybenzene (3b)[3]

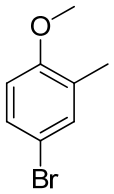
100 mg product **3b** was isolated in 93% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.32 (d, *J* = 6.1 Hz, 6H), 4.45-4.54 (m, 1H), 6.75-6.79 (m, 2H), 7.34-7.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.1, 132.4, 117.8, 112.6, 70.3, 22.3; HRMS (EI) calcd for $C_9H_{11}BrO$ *m/z* $[M]^+$: 213.9988, found: 213.9983.



1-(Benzyloxy)-4-bromobenzene(3c)[2]

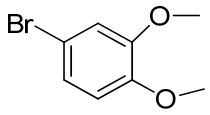
130.2 mg product **3c** was isolated in 99% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.06 (s, 2H), 6.88-6.91 (m, 2H), 7.36-7.47 (m, 7H); ^{13}C NMR (100 MHz,

$CDCl_3$): δ 157.9, 136.6, 132.4, 128.7, 128.2, 127.5, 116.8, 113.2, 70.3. HRMS (EI) calcd for $C_{13}H_{11}BrO$ *m/z* $[M]^+$: 261.9988, found: 261.9985.



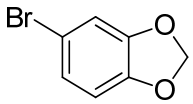
4-Bromo-1-methoxy-2-methylbenzene (3d)[2]

91.5 mg product **3d** was isolated in 91% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.19 (s, 3H), 3.80(s, 3H), 6.68 (d, *J* = 9.0 Hz, 1H), 7.24-7.27 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.0, 133.3, 129.5, 129.1, 112.4, 111.6, 55.6, 16.2. HRMS (EI) calcd for C_8H_9BrO *m/z* $[M]^+$: 199.9831, found: 199.9836.



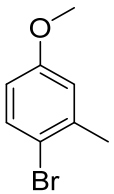
4-Bromo-1,2-dimethoxybenzene (3e)[4]

107.4 mg product **3e** was isolated in 99% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.83 (d, *J* = 4.5 Hz, 6H), 6.70 (d, *J* = 8.5 Hz, 1H), 6.95 (d, *J* = 2.2 Hz, 1H), 7.00 (d, *J* = 2.2 Hz 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 148.3, 123.4, 114.8, 112.7, 112.5, 56.1, 56.0. HRMS (EI) calcd for $C_8H_9BrO_2$ *m/z* $[M]^+$: 215.9780, found: 215.9784.



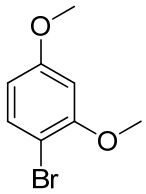
5-Bromobenzo[d][1,3]dioxole (3f)[5]

98.5 mg product **3f** was isolated in 98% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.96 (s, 2H), 6.68 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 7.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.6, 147.0, 124.3, 113.1, 112.3, 109.6, 101.7. HRMS (EI) calcd for $C_7H_5BrO_2$ *m/z* $[M]^+$: 199.9467, found: 199.9466.



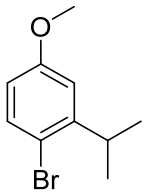
1-Bromo-4-methoxy-2-methylbenzene (3g)[2]

95.5 mg product **3g** was isolated in 95% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.37 (s, 3H), 3.78 (s, 3H), 6.62 (dd, *J* = 2.9 Hz, 8.7 Hz, 1H), 6.79 (d, *J* = 2.8 Hz, 1H), 7.40 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 138.9, 132.9, 116.6, 115.5, 113.0, 55.5, 23.3. HRMS (EI) calcd for C_8H_9BrO *m/z* $[M]^+$: 199.9831, found: 199.9835.



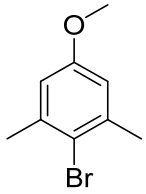
1-Bromo-2,4-dimethoxybenzene (3h)[2]

105.2 mg product **3h** was isolated in 97% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 3H), 3.86 (s, 3H), 6.39 (dd, *J* = 2.7 Hz, 8.7 Hz, 1H), 6.48 (d, *J* = 2.7 Hz, 1H), 7.40 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 156.6, 133.2, 105.9, 102.5, 100.0, 56.2, 55.6. HRMS (EI) calcd for $C_8H_9BrO_2$ *m/z* $[M]^+$: 215.9780, found: 215.9781.



1-Bromo-2-isopropyl-4-methoxybenzene (3i)[6]

103.1 mg product **3i** was isolated in 90% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.24 (d, *J* = 6.8 Hz, 6H), 3.27-3.35 (m, 1H), 3.79 (s, 3H), 6.62 (dd, *J* = 2.8 Hz, 8.7 Hz, 1H), 6.84 (d, *J* = 2.6 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (100 MHz, CDCl3): δ 159.3, 148.6, 133.3, 114.9, 113.1, 112.5, 55.5, 33.1, 22.9. HRMS (ESI) calcd for $C_{10}H_{13}BrO$ *m/z* $[M+Na]^+$: 251.0042, found: 251.0047.



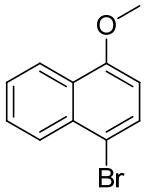
2-Bromo-5-methoxy-1,3-dimethylbenzene (3j)[7]

106.4 mg product **3j** was isolated in 99% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.39 (s, 6H), 3.77 (s, 3H), 6.65 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2, 139.2, 118.3, 113.9, 55.4, 24.2. HRMS (EI) calcd for $C_9H_{11}BrO$ *m/z* $[M]^+$: 213.9988, found: 213.9984.

2-Bromo-4-ethyl-1-methoxybenzene (3k)[8]

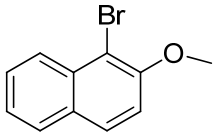
105.4 mg product **3k** was isolated in 98% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.21 (t, *J* = 7.6 Hz, 3H), 2.57 (q, *J* = 7.5 Hz, 2H), 3.86 (s, 3H), 6.82 (d, *J* =

8.3 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 1H), 7.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.9, 138.0, 132.7, 127.8, 112.0, 111.4, 56.3, 27.8, 15.8. HRMS (EI) calcd for $C_9H_{11}BrO$ *m/z* $[M]^+$: 213.9988, found: 213.9986.



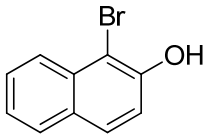
1-Bromo-4-methoxynaphthalene (3l)[2]

114.9 mg product **3l** was isolated in 97% yield. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.98 (s, 3H), 6.67 (d, *J* = 8.2 Hz, 1H), 7.55 (m, 1H), 7.64 (m, 2H), 8.20 (d, *J* = 8.4 Hz, 1H), 8.30 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 132.5, 129.6, 127.9, 127.0, 126.9, 126.0, 122.5, 113.3, 104.6, 55.8. HRMS (EI) calcd for $C_{11}H_9BrO$ *m/z* $[M]^+$: 235.9831, found: 235.9830.



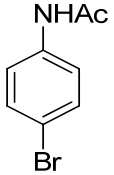
1-Bromo-2-methoxynaphthalene (3m)[6]

117.3 mg product **3m** was isolated in 99% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.02 (s, 3H), 7.25 (d, *J* = 9.0 Hz, 1H), 7.25 (d, *J* = 9.0 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.79 (t, *J* = 7.9 Hz, 2H), 8.24 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.8, 133.2, 129.9, 129.1, 128.1, 127.8, 126.2, 124.4, 113.6, 108.7, 57.1. HRMS (EI) calcd for $C_{11}H_9BrO$ *m/z* $[M]^+$: 235.9831, found: 235.9833.



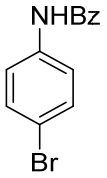
1-Bromonaphthalen-2-ol (3n)[9]

105.9 mg product **3n** was isolated in 95% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.99 (s, 1H), 7.29 (d, *J* = 8.9 Hz), 7.41 (m, 1H), 7.59 (m, 1H), 7.74 (d, *J* = 8.8 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 8.06 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6, 132.4, 129.8, 129.4, 128.3, 127.9, 125.4, 124.2, 117.2, 106.2. HRMS (EI) calcd for $C_{10}H_7BrO$ *m/z* $[M]^+$: 221.9675, found: 221.9675.



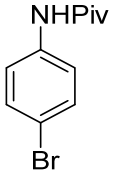
N-(4-Bromophenyl)acetamide (3o)[2]

99.5 mg product **3o** was isolated in 93% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.17 (s, 3H), 7.30 (s, 1H), 7.38-7.43 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.5, 137.1, 132.1, 121.5, 117.0, 24.7. HRMS(ESI) calcd for C_8H_8BrNO *m/z* $[M+H]^+$: 213.9862, found: 213.9865.



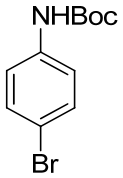
N-(4-Bromophenyl)benzamide (3p)[10]

124.2 mg product **3p** was isolated in 90% yield. White solid; ^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 7.52-7.55 (m, 4H), 7.58-7.62 (m, 1H), 7.76-7.80 (m, 2H), 7.94-7.96 (m, 2H), 10.38 (s, 1H); ^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 165.7, 138.6, 134.7, 131.7, 131.4, 128.4, 127.7, 122.2, 115.3. HRMS (ESI) calcd for $C_{13}H_{10}BrNO$ *m/z* $[M+H]^+$: 276.0019, found: 276.0021.



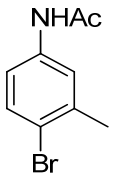
N-(4-Bromophenyl)-2,2-dimethylpropanamide (3q)[10]

126.7 mg product **3q** was isolated in 99% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.29 (s, 9H), 7.38-7.43 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.8, 137.2, 131.9, 121.7, 116.8, 39.8, 27.7. HRMS (ESI) calcd for $C_{11}H_{14}BrNO$ *m/z* $[M+H]^+$: 256.0332, found: 256.0337.



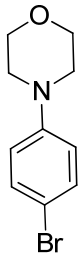
tert-butyl-(4-Bromophenyl)carbamate (3r)[11]

133.3 mg product **3r** was isolated in 98% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.50 (s, 9H), 6.61 (s, 1H), 7.25 (d, J = 8.6 Hz, 2H), 7.37 (dt, J = 2.8 Hz, 8.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 137.6, 131.9, 120.2, 115.5, 81.0, 28.4. HRMS (EI) calcd for $C_{11}H_{14}BrONO_2$ *m/z* $[M]^+$: 271.0202, found: 271.0205.



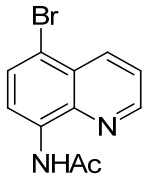
N-(4-Bromo-3-methylphenyl)acetamide (3s)[2]

104.9 mg product **3s** was isolated in 92% yield. Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.12 (s, 3H), 2.26 (s, 3H), 7.19 (dd, *J* = 1.9 Hz, 8.5 Hz, 1H), 7.34-7.38 (m, 2H), 8.77 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.5, 138.3, 137.2, 132.4, 122.6, 119.5, 119.3, 24.3, 22.9. HRMS (ESI) calcd for $C_9H_{10}BrNO$ *m/z* $[M+H]^+$: 228.0019, found: 228.0021.



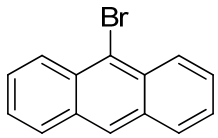
4-(4-Bromophenyl)morpholine (3t)[12]

118.1 mg product **3t** was isolated in 98% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.10 (t, *J* = 4.9 Hz, 4H), 3.84 (t, *J* = 4.8 Hz, 4H), 6.76 (m, 2H), 7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3, 131.9, 117.3, 112.1, 66.8, 49.1. HRMS (EI) calcd for $C_{10}H_{13}NOBr$ *m/z* $[M]^+$: 241.0097, found: 241.0095.



N-(5-Bromoquinolin-8-yl)acetamide (3u)[13]

125.9 mg product **3u** was isolated in 95% yield. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.31 (s, 3H), 7.45 (q, *J* = 4.2 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 8.38 (d, *J* = 8.5 Hz, 1H), 8.55 (d, *J* = 8.4 Hz, 1H), 8.70 (d, *J* = 4.0 Hz, 1H), 9.65 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.7, 148.5, 138.7, 135.8, 134.3, 130.8, 127.0, 122.6, 116.7, 114.1, 25.2. HRMS (ESI) calcd for $C_{11}H_9BrN_2O$ *m/z* $[M+H]^+$: 264.9971, found: 264.9978.



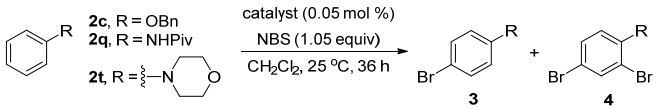
9-Bromoanthracene (3v)[2]

110.5 mg product **3v** was isolated in 86% yield. Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 2H), 7.60 (t, *J* = 7.7 Hz, 2H), 7.97 (d, *J* = 8.4 Hz, 2H), 8.4 (s, 1H), 8.53 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.3, 132.3, 130.7, 128.7, 127.8, 127.4, 127.3, 125.8. HRMS (APCI) calcd for $C_{14}H_9Br$ *m/z* $[M+H]^+$: 256.9954, found: 256.9960.

(D) Representative procedure for the large scale preparation of 3

To a solution of zwitterionic catalyst **1a** in dichloromethane (c = 0.0001 M, 25.0 mL) was added substrate **2a** (2.7 g, 25.0 mmol) and NBS (4.6 g, 26.2 mmol) in the absence of light. The resultant mixture was stirred at 25 ^{o}C for 36 h. Upon completion, the reaction was quenched with saturated aqueous Na_2SO_3 (30 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/EtOAc = 100:1) to yield **3a** as an orange oil (98%, 4.58 g).

Table S1. Bromination of 2t with Different Catalysts[a]



entry	substrate	catalyst	Br source	ratio of **2**:**3**:**4**[b]
1	**2t**	concn HCl	NBS	31:52:18
2	**2t**	$FeCl_3$	NBS	37:51:12
3	**2t**	Ph_3PS	NBS	0:79:21
4	**2t**	none	Br_2	33:61:5
5	**2t**	**1a**	NBS	2:98:0
6	**2c**	**1a**	NBS	1:99:0
7	**2q**	**1a**	NBS	1:99:0

[a] Reactions were carried out with substrate **2t** (0.5 mmol), catalyst (0.05 mol %), Br source (0.525 mmol) in CH_2Cl_2 (1 mL) at 25 ^{o}C for 36 h in the absence of light. [b] The ratios were determined by GC/MS analysis.

(E) References

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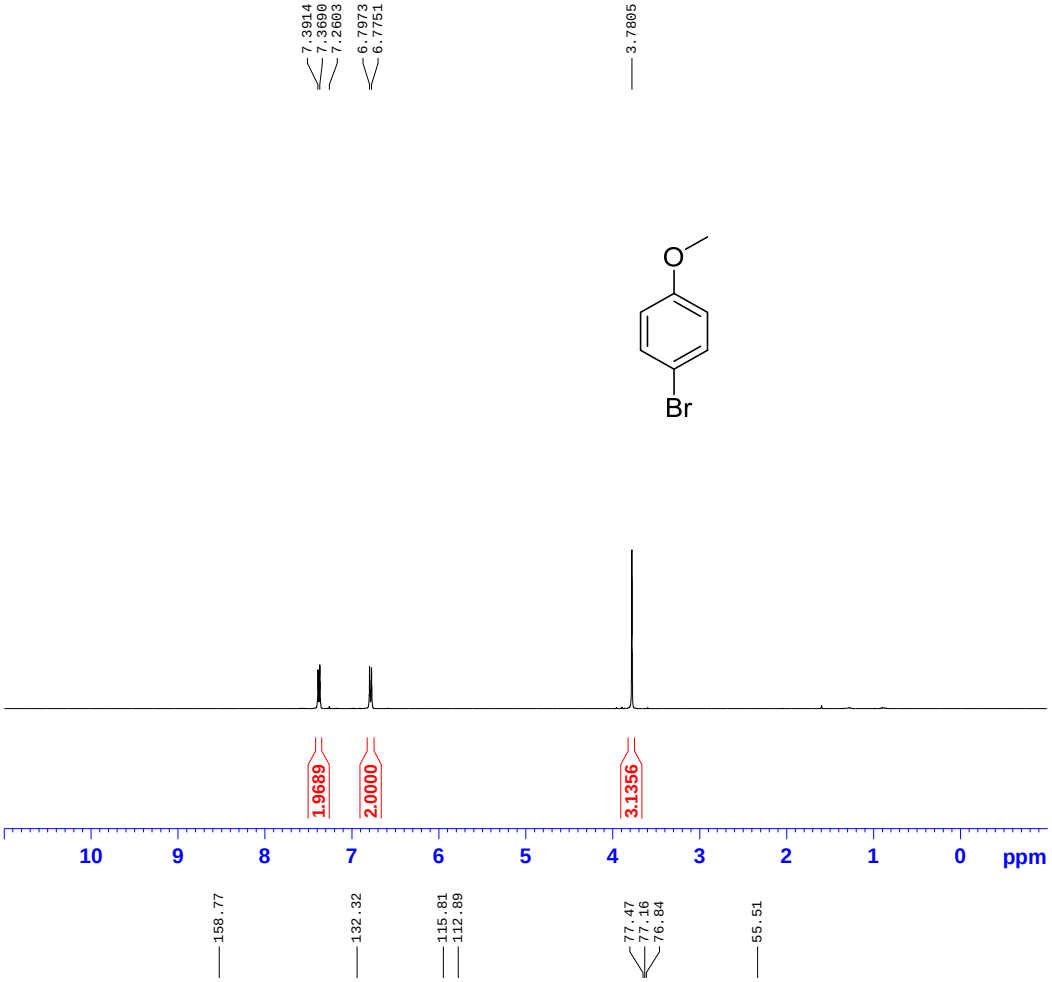
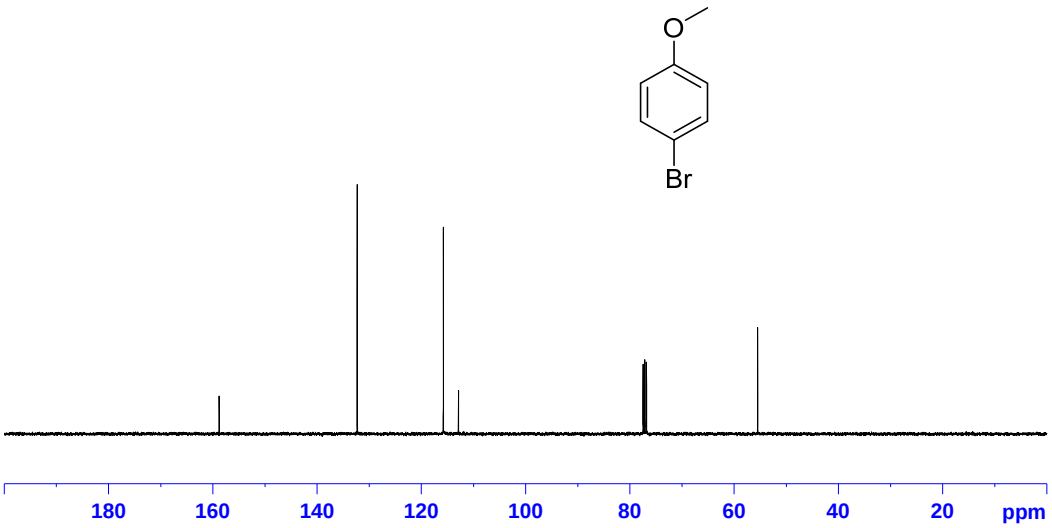
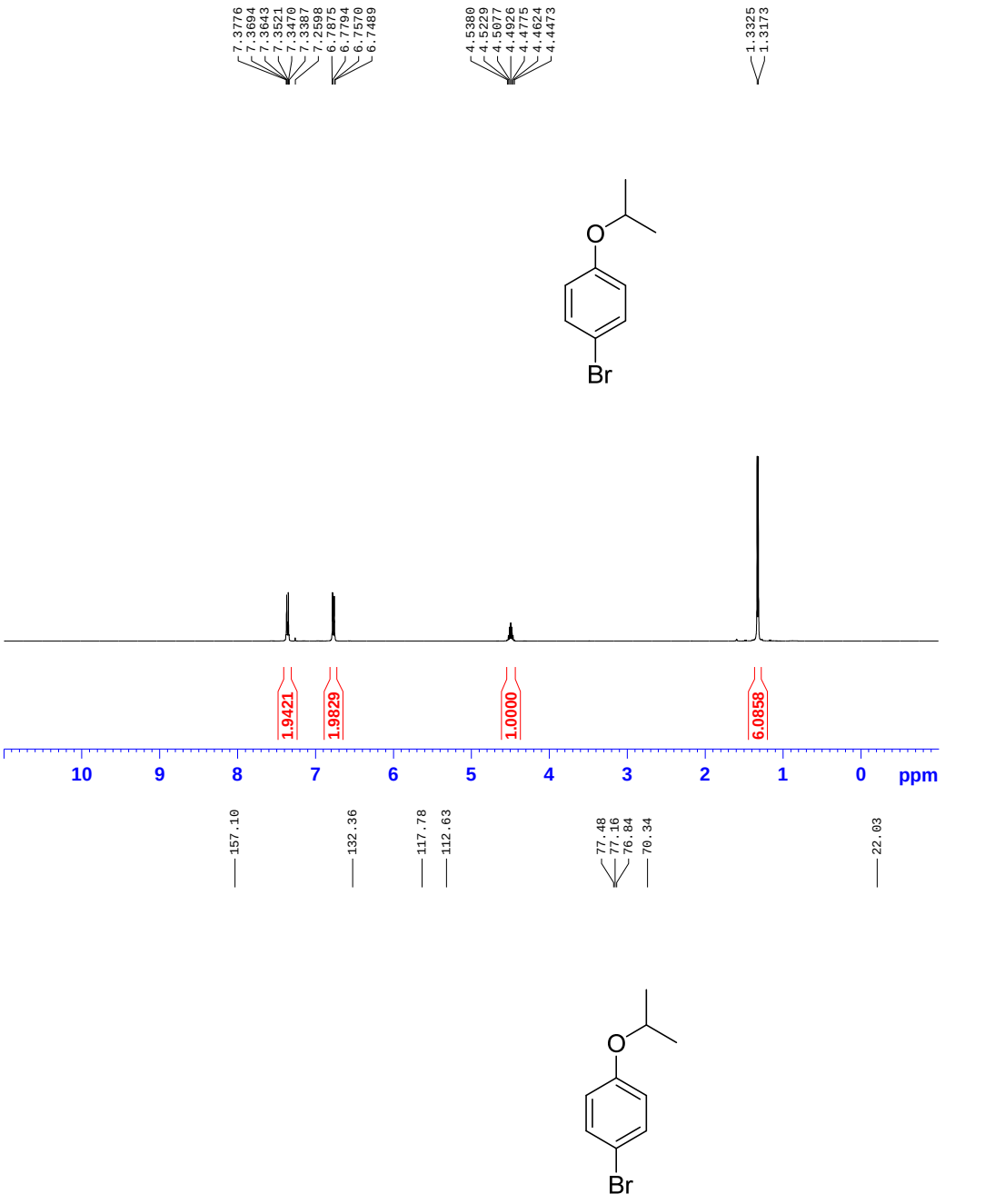
(F) ^{1}H and ^{13}C Spectra

1-bromo-4-methoxybenzene (3a)

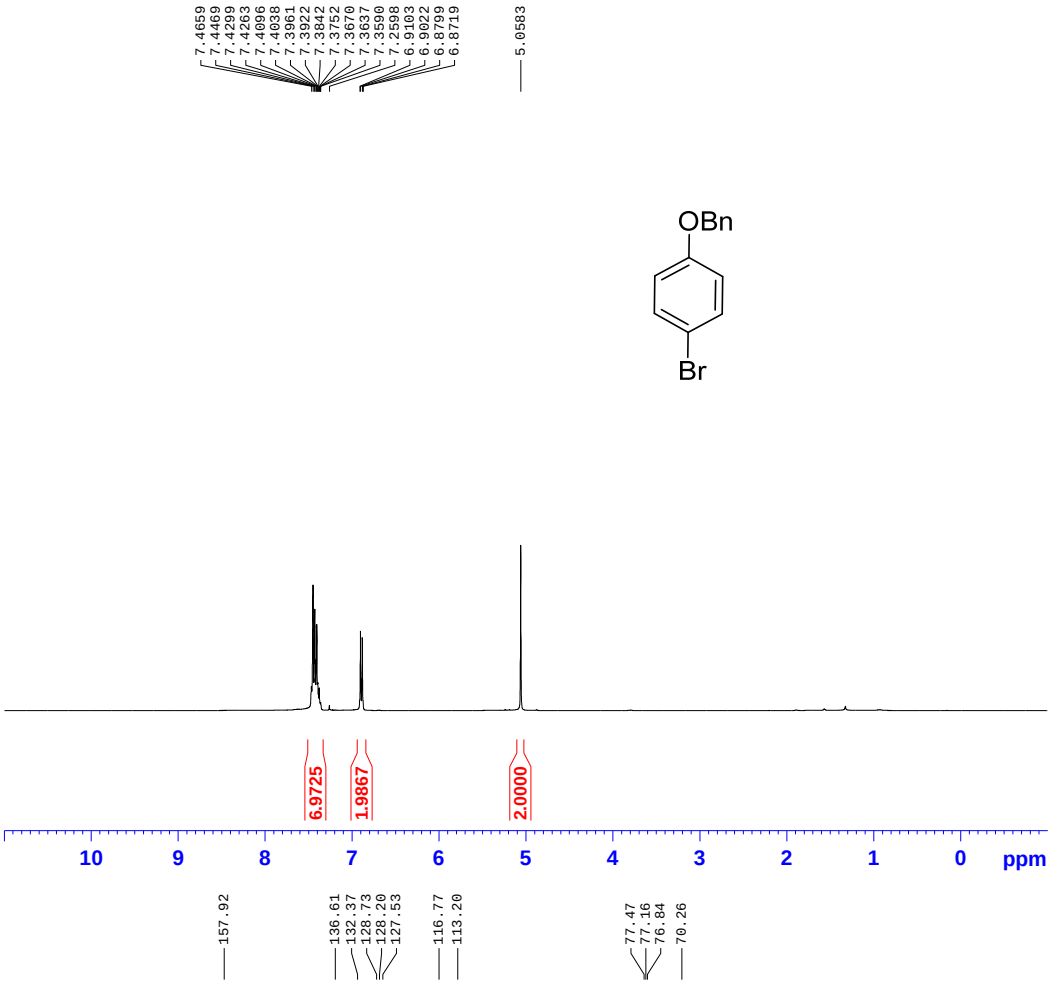


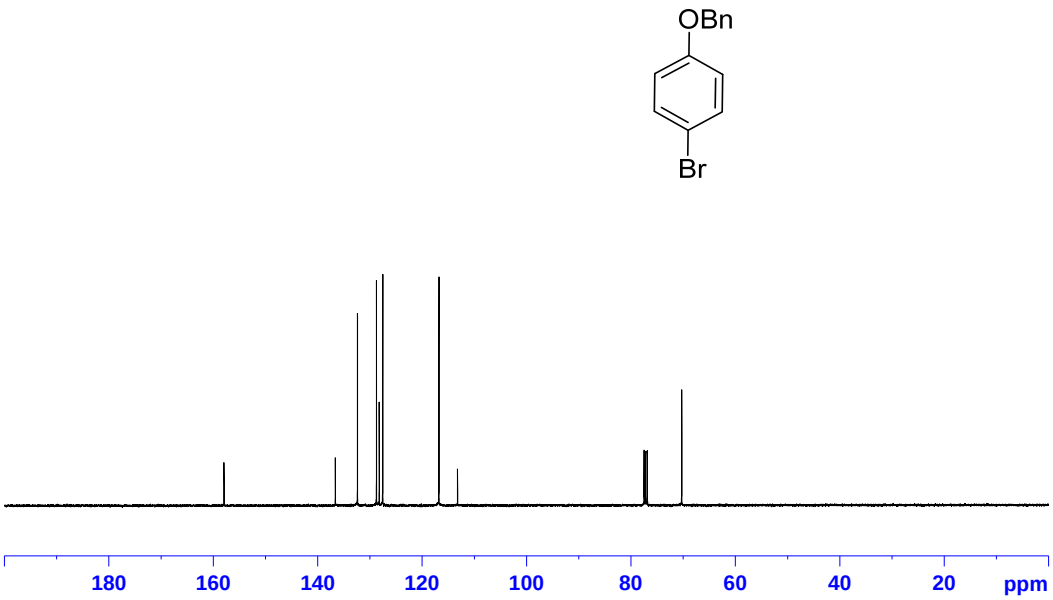


1-bromo-4-isopropoxybenzene (3b)

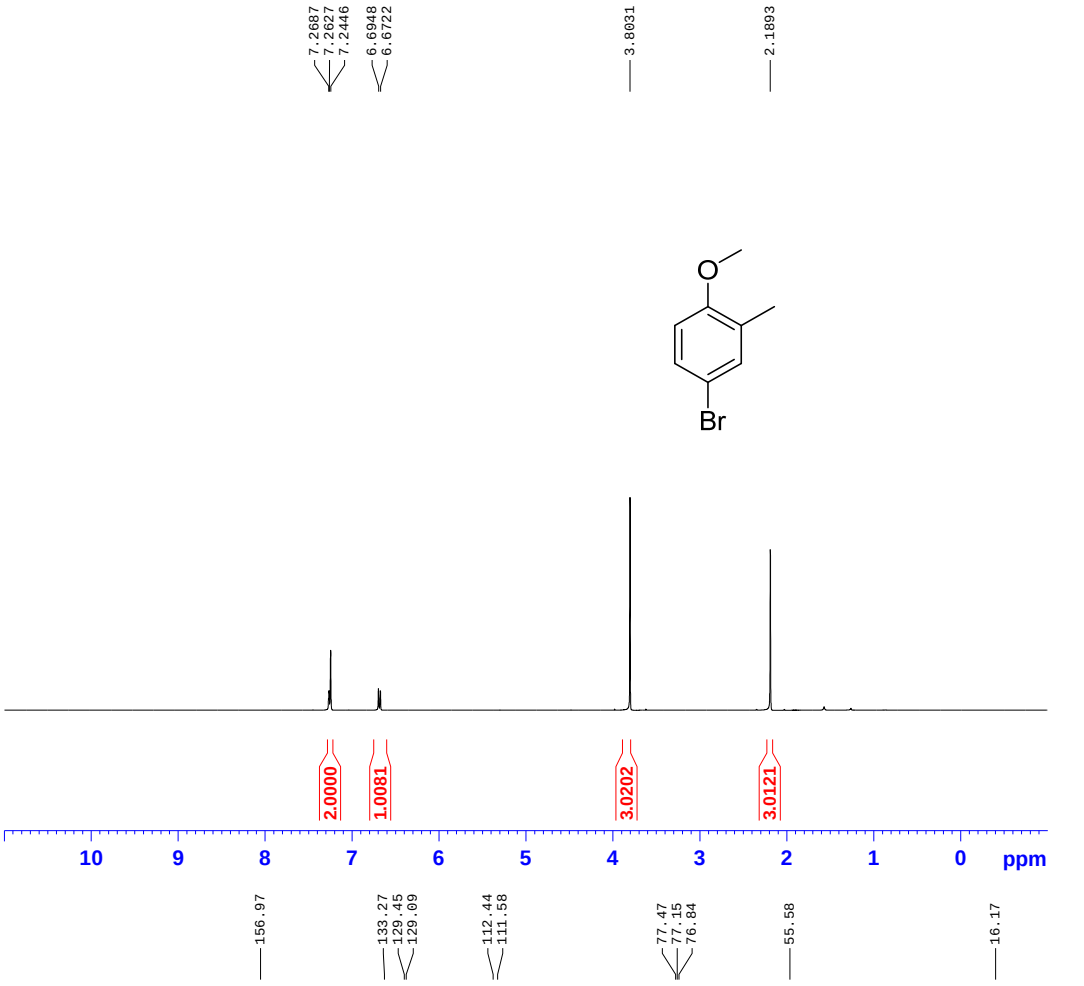


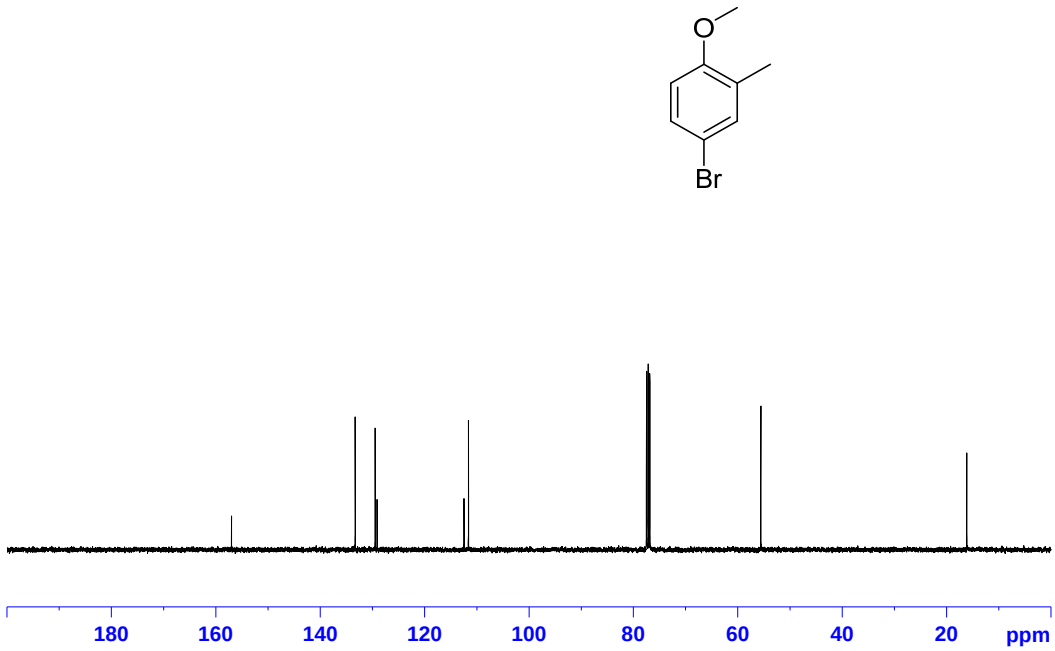
1-(benzyloxy)-4-bromobenzene (3c)



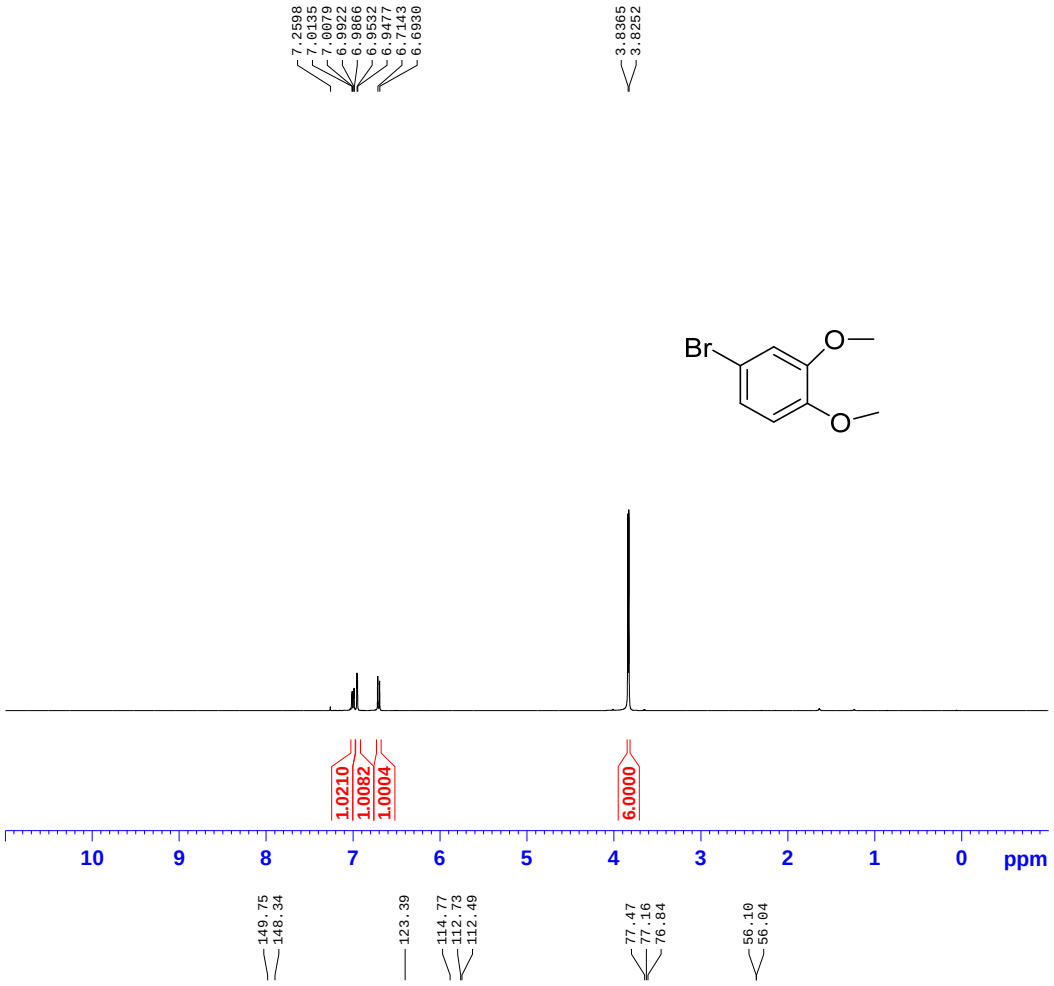


4-bromo-1-methoxy-2-methylbenzene (3d)

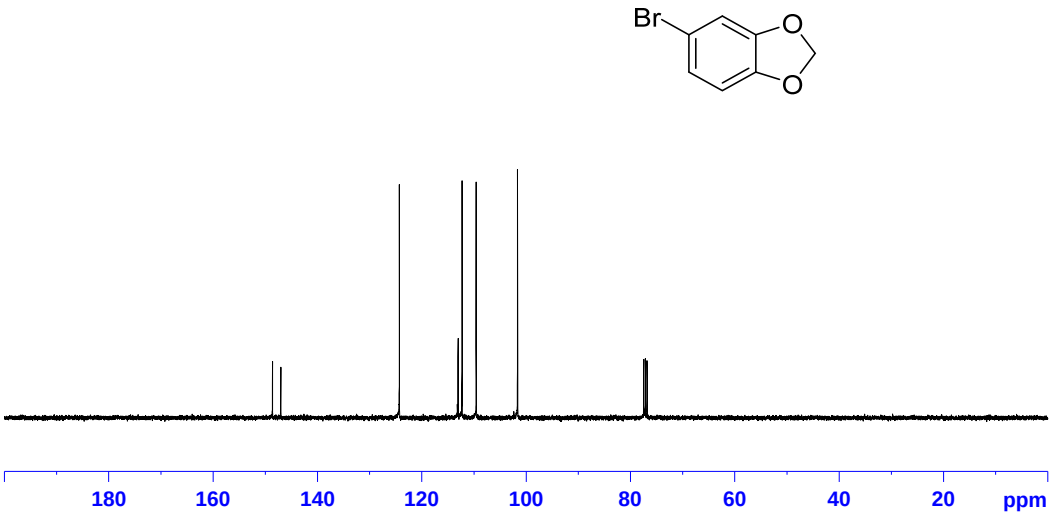




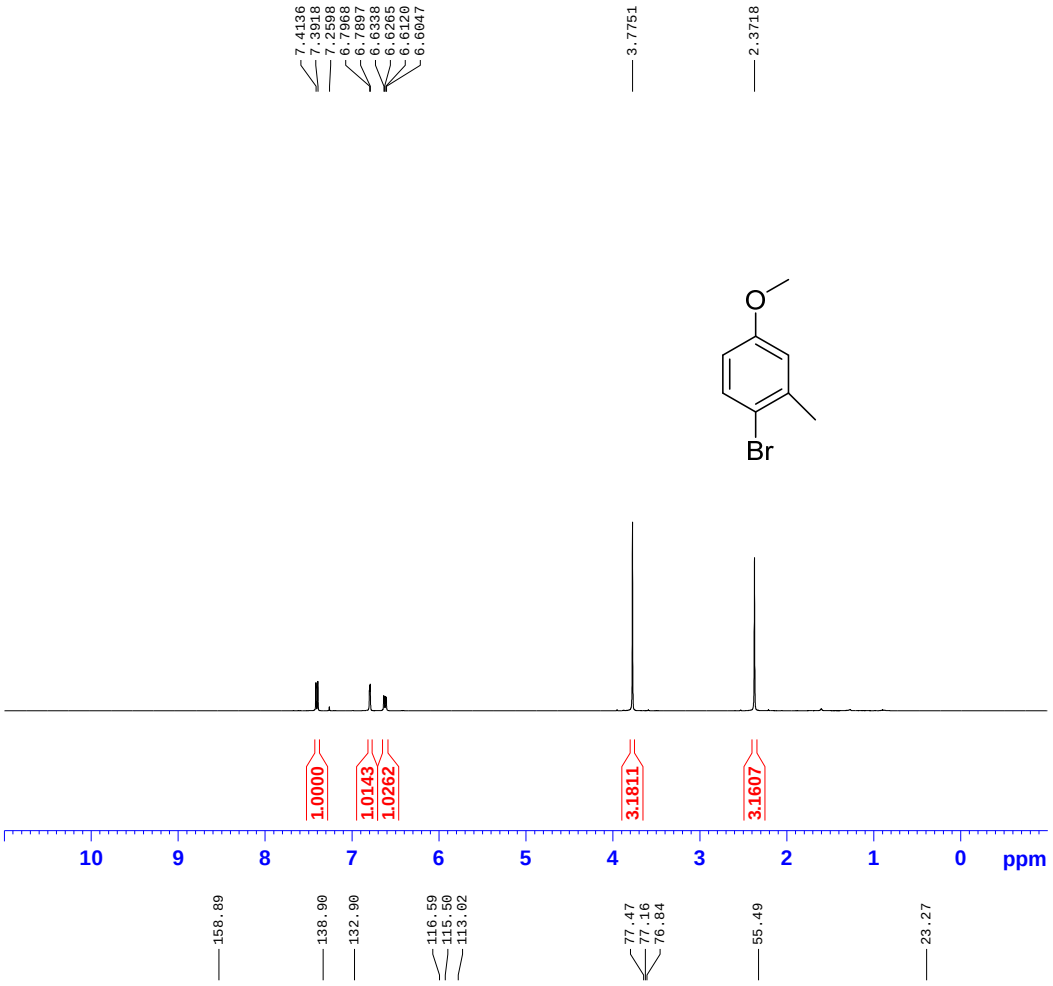
4-bromo-1,2-dimethoxybenzene (3e)

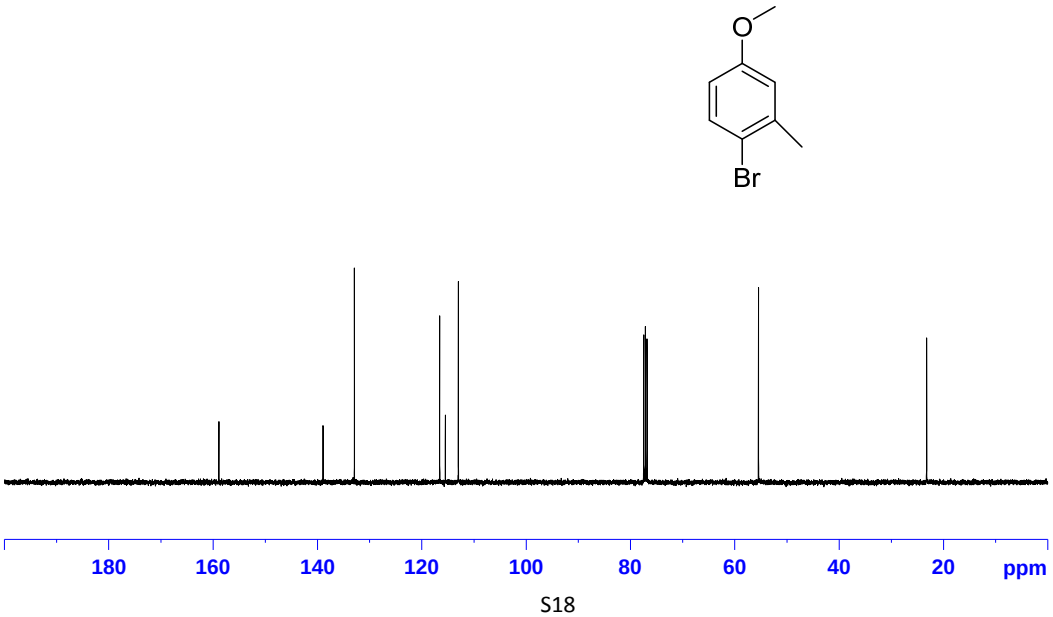


5-bromobenzo[d][1,3]dioxole (3f)

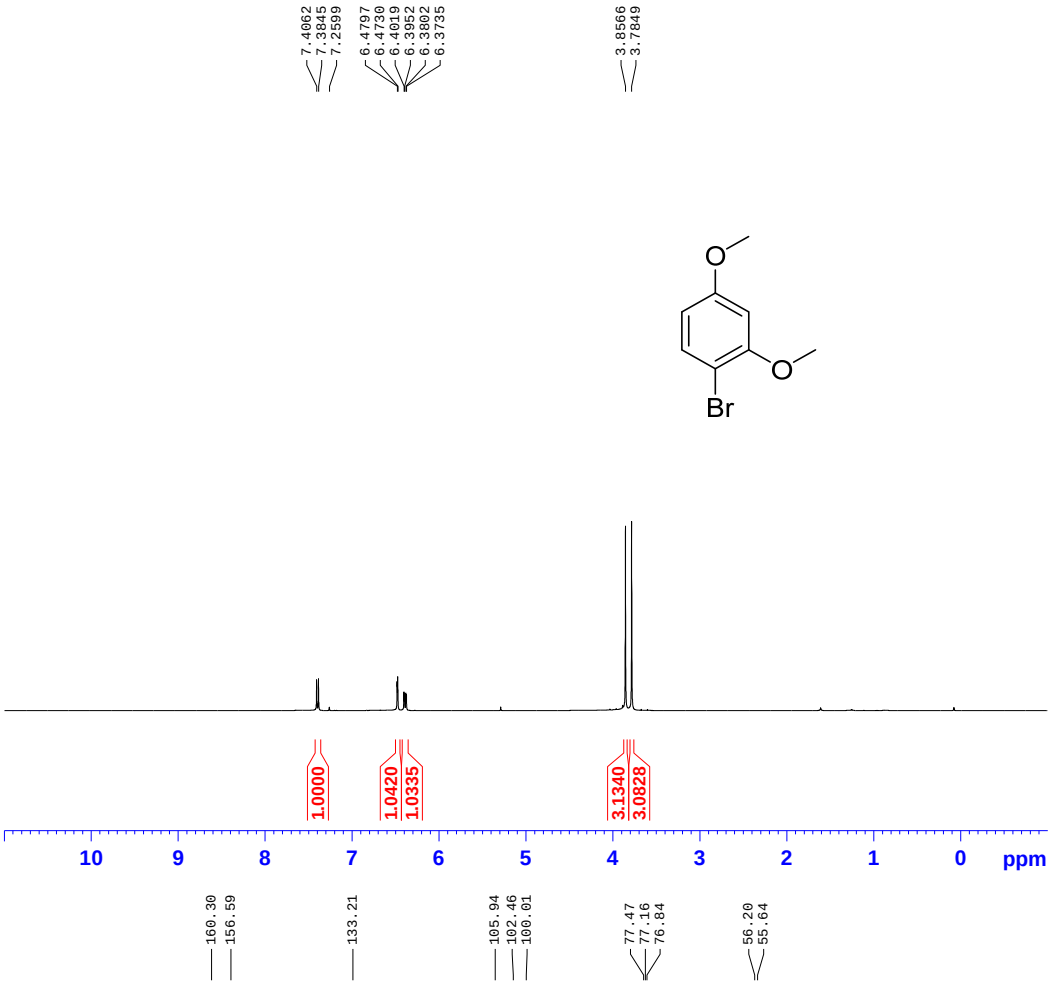


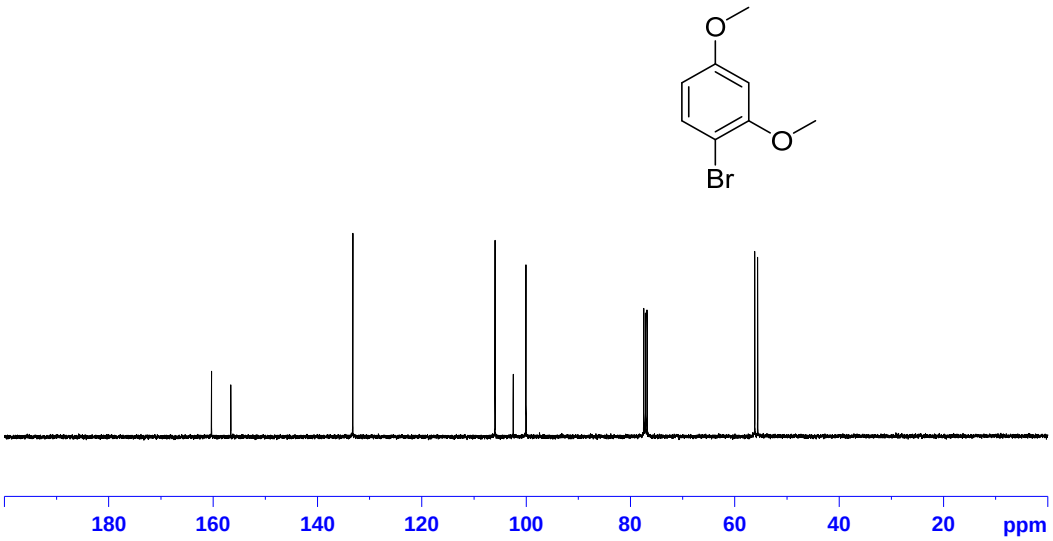
1- bromo-4-methoxy-2-methylbenzene (3g)



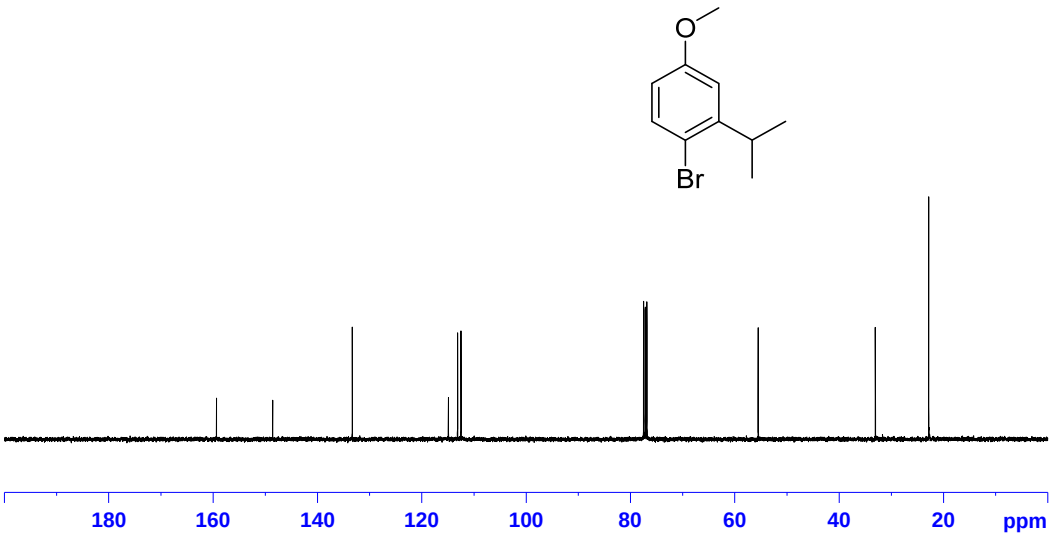


1- bromo-2,4-dimethoxybenzene (3h)

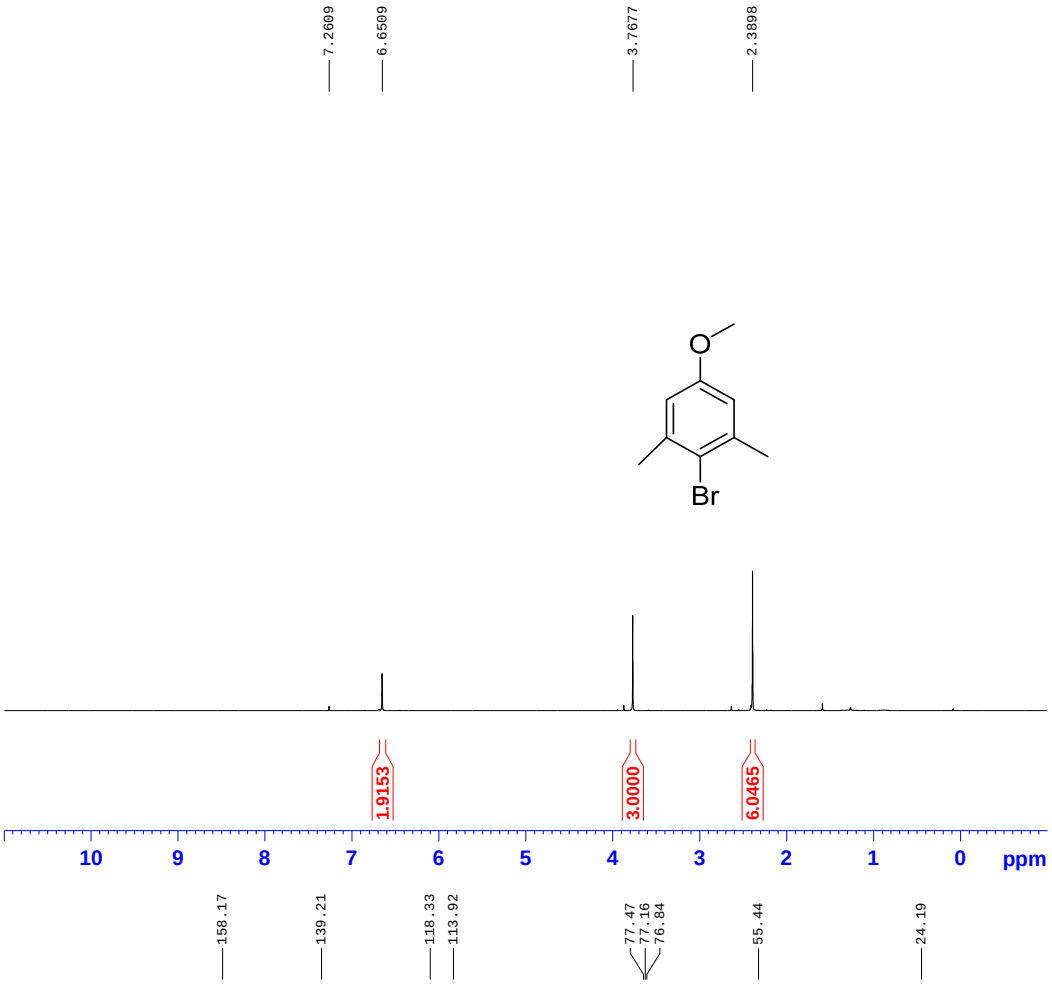


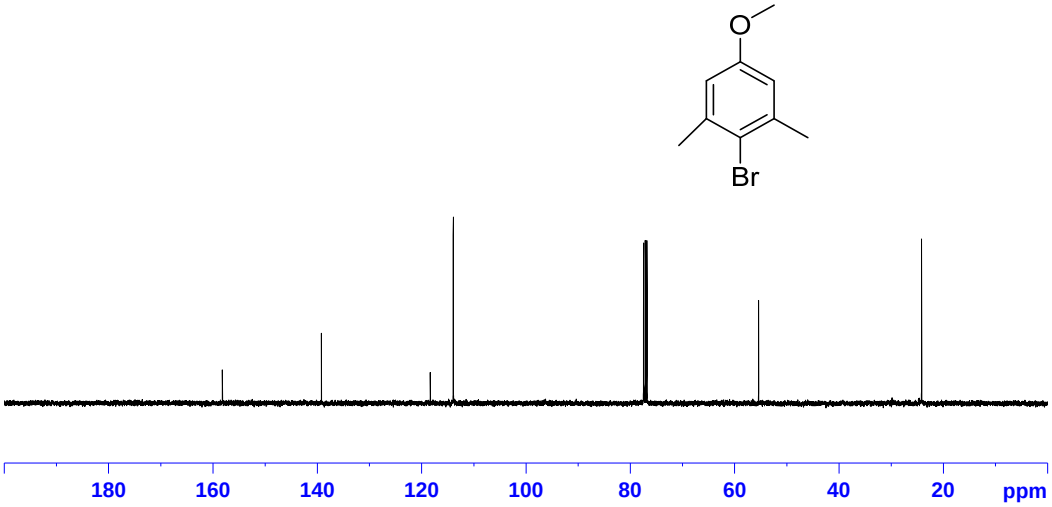


1-bromo-2-isopropyl-4-methoxybenzene (3i)

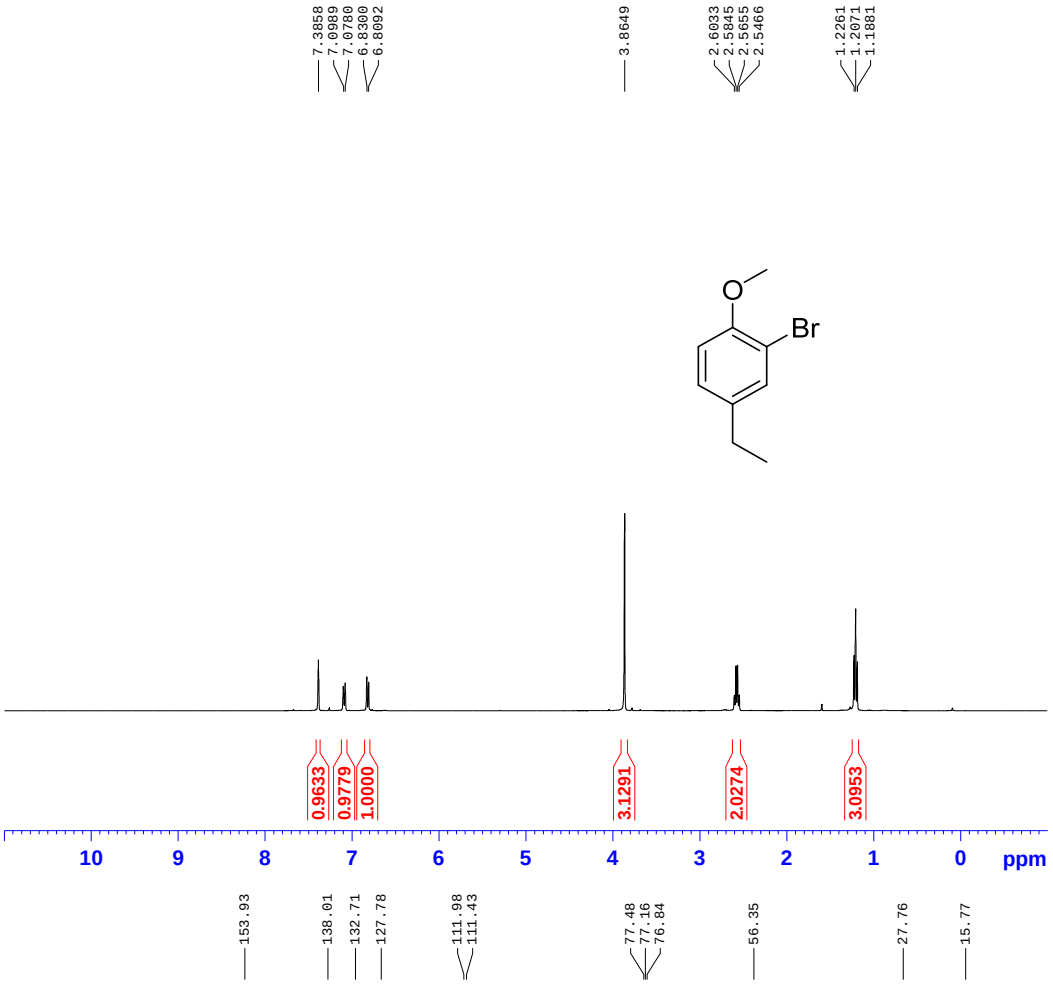


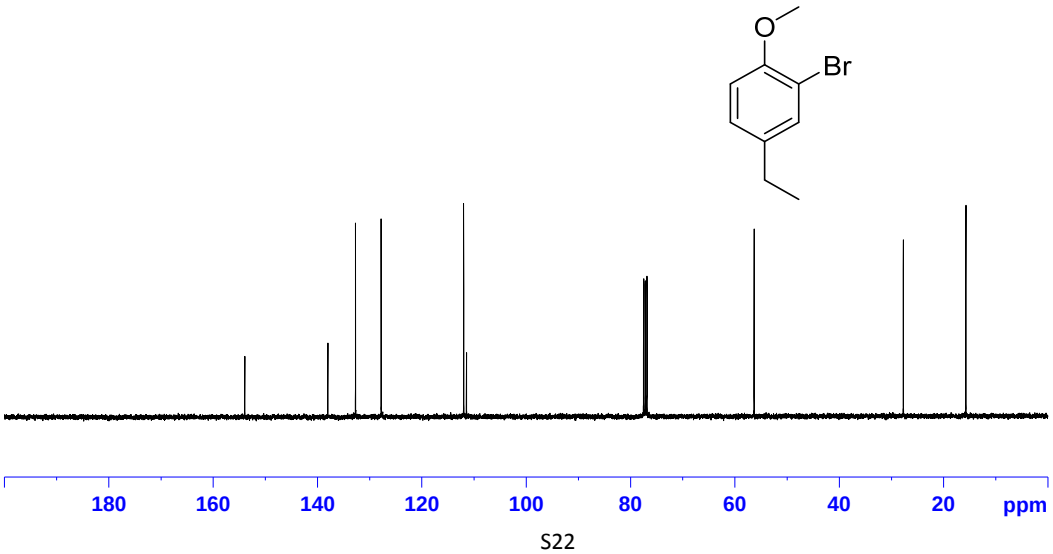
2-bromo-5-methoxy-1,3-dimethylbenzene (3j)



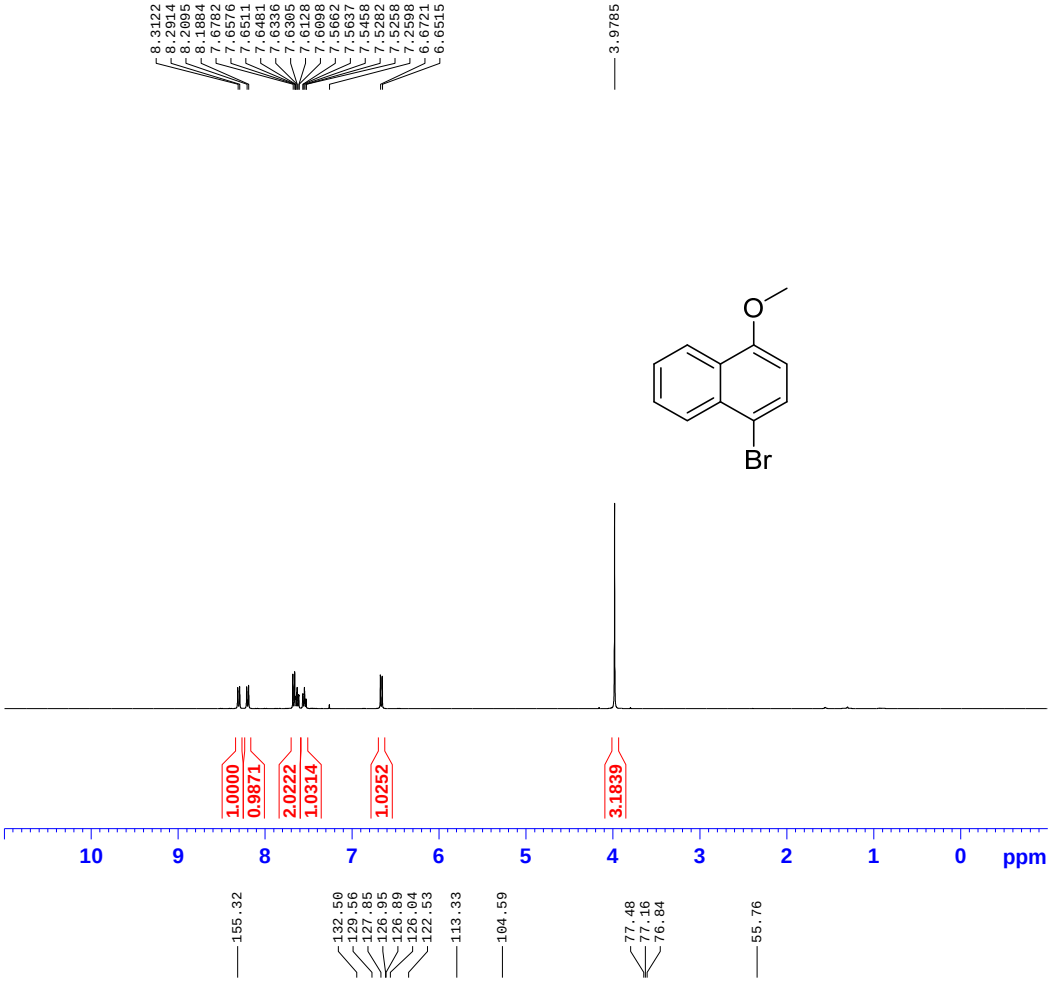


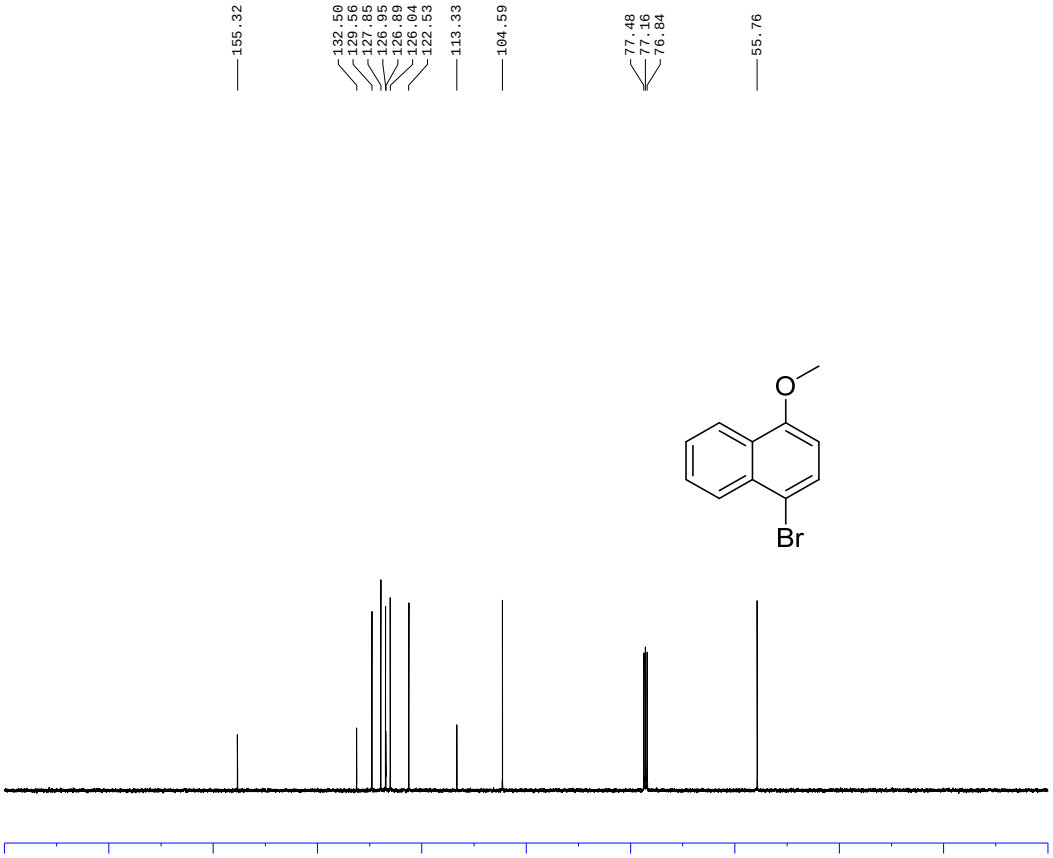
2-bromo-4-ethyl-1-methoxybenzene (3k)



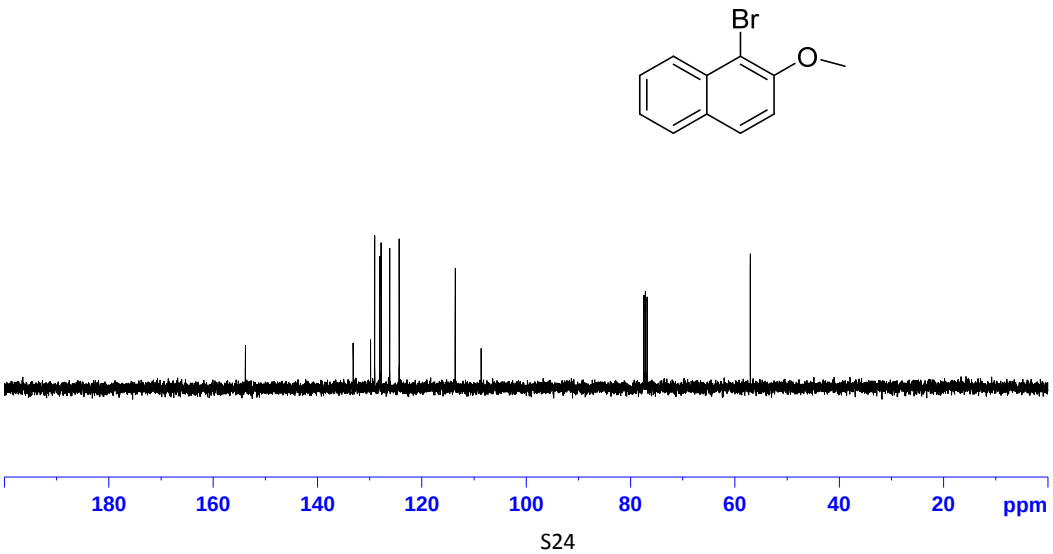


1-bromo-4-methoxynaphthalene (3l)

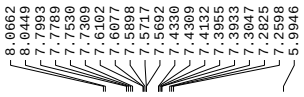


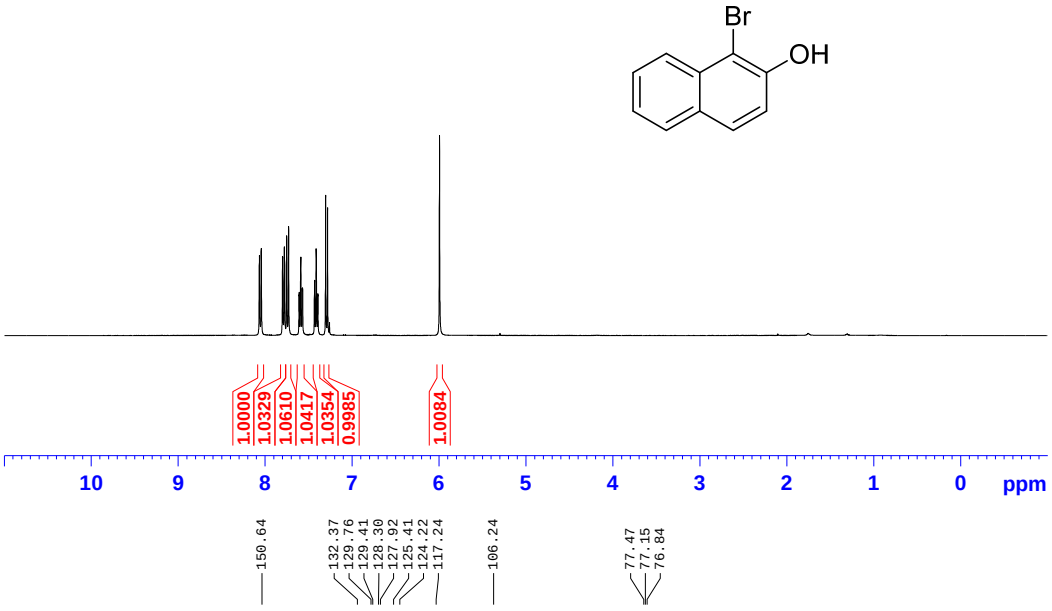


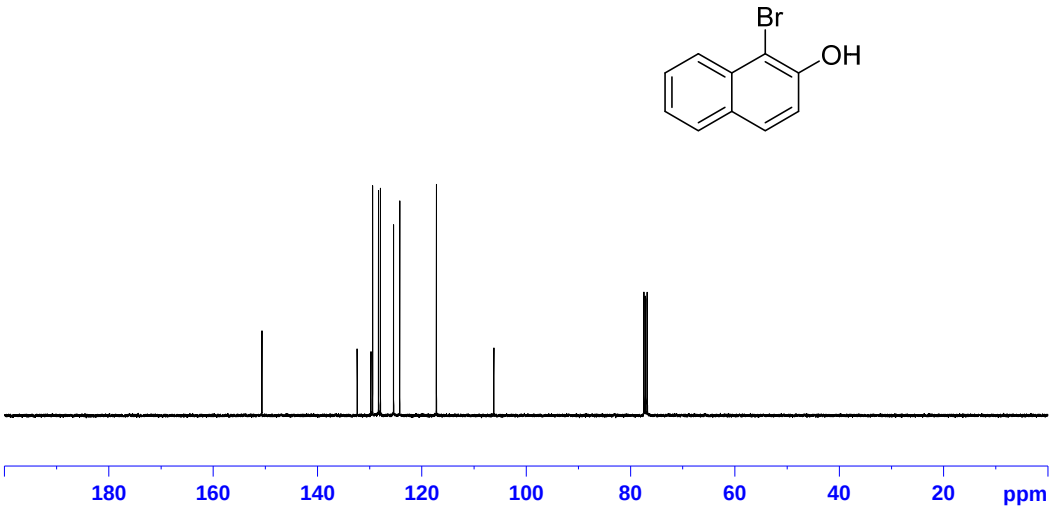
1-bromo-2-methoxynaphthalene (3m)



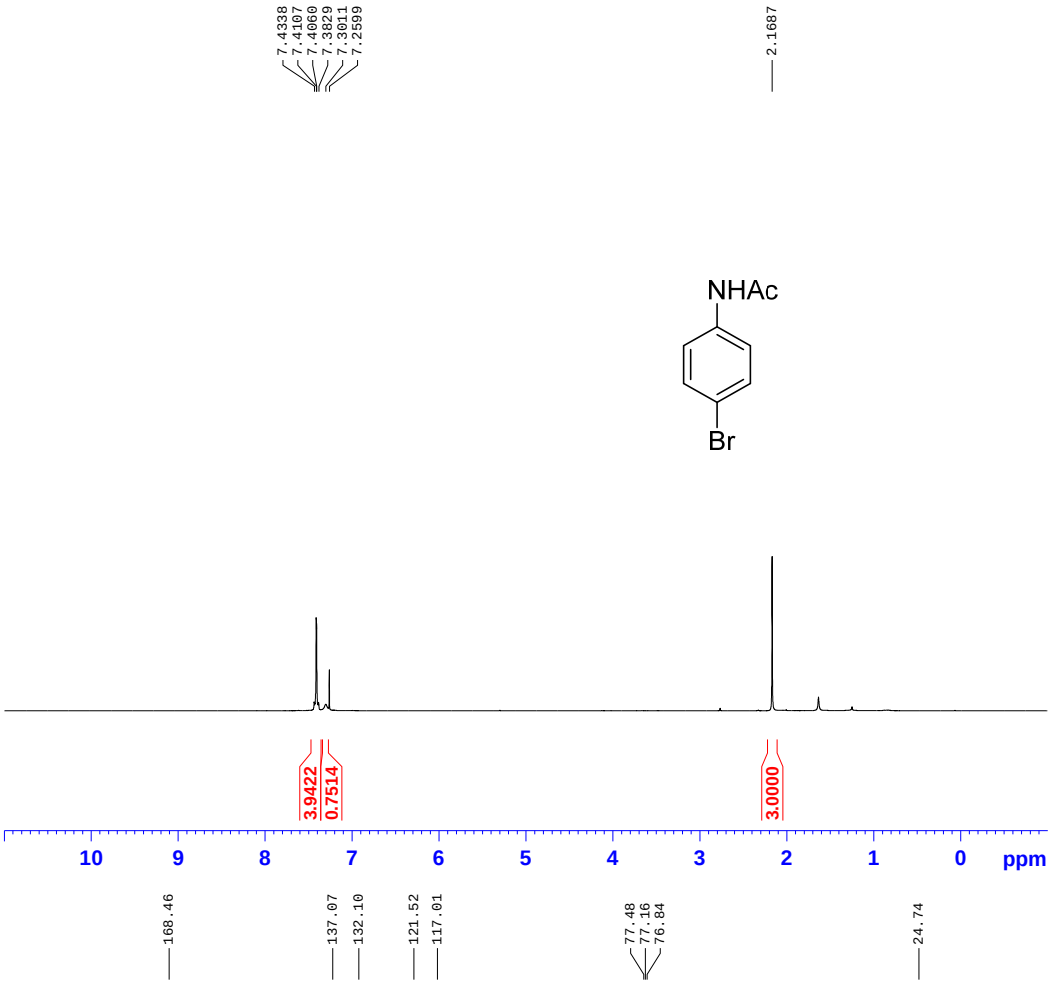
1-bromonaphthalen-2-ol (3n)

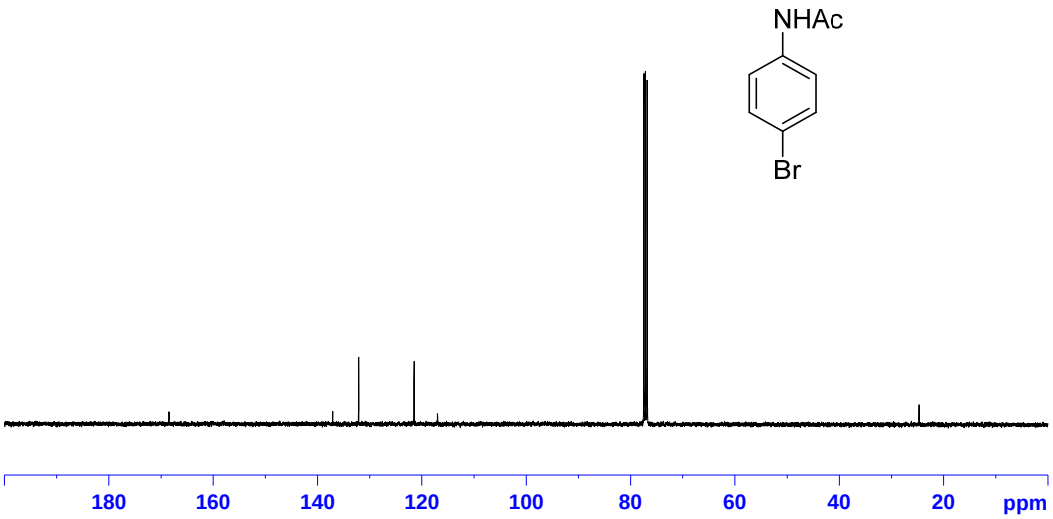




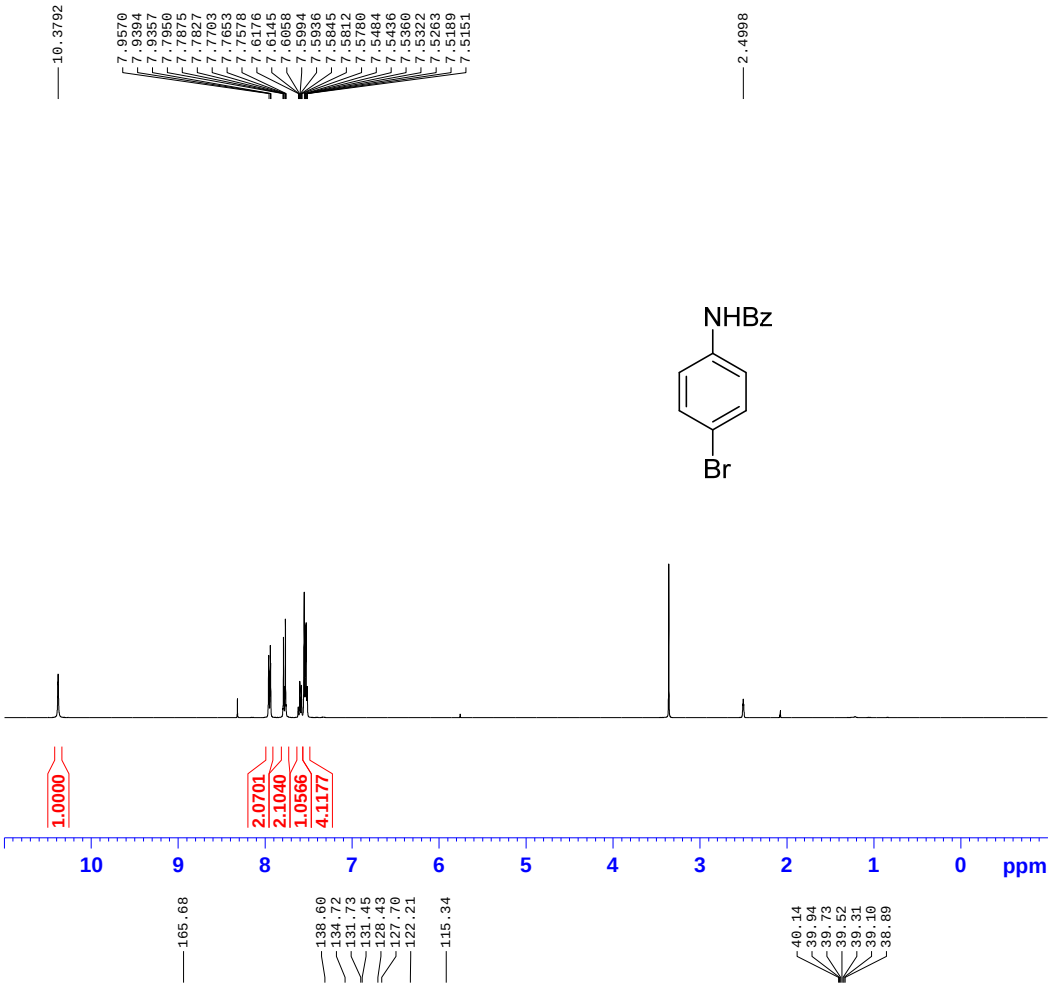


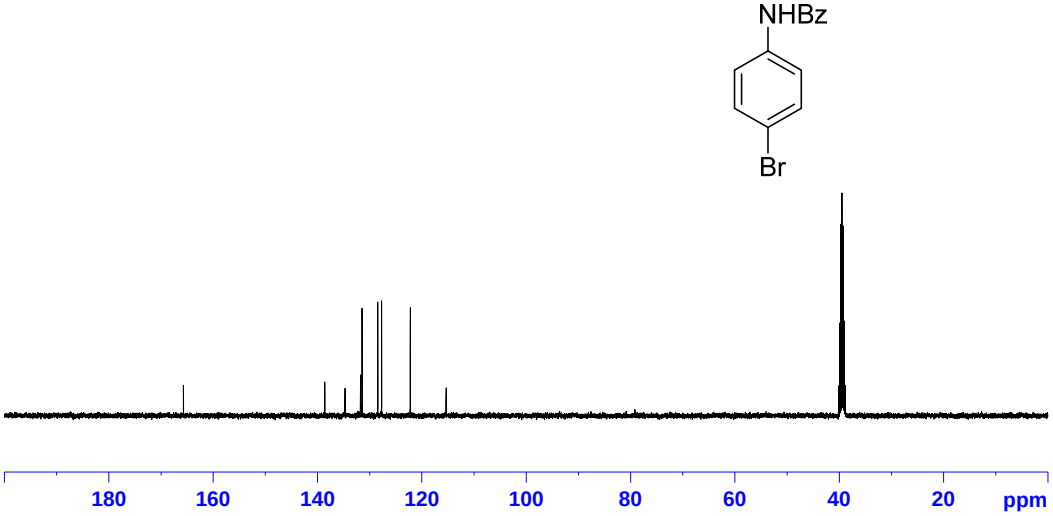
N-(4-bromophenyl)acetamide (3o)



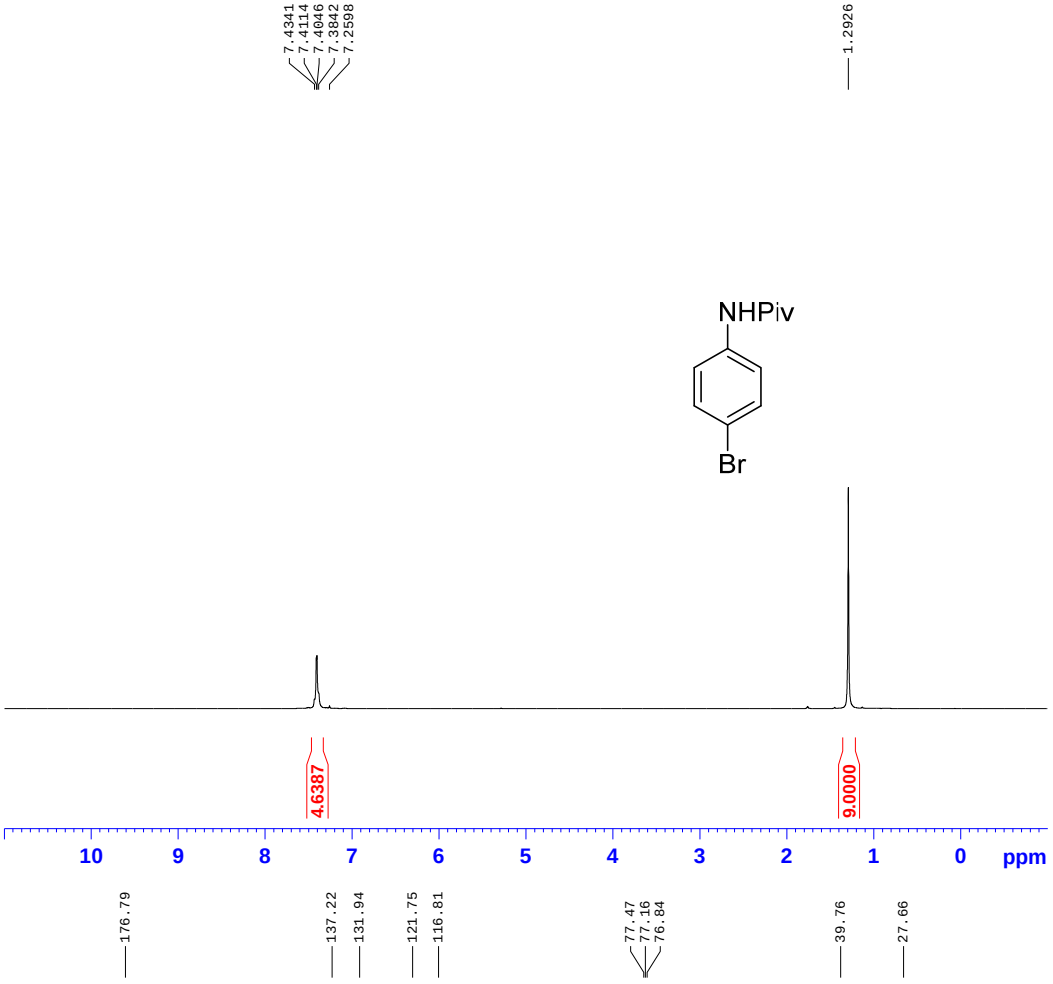


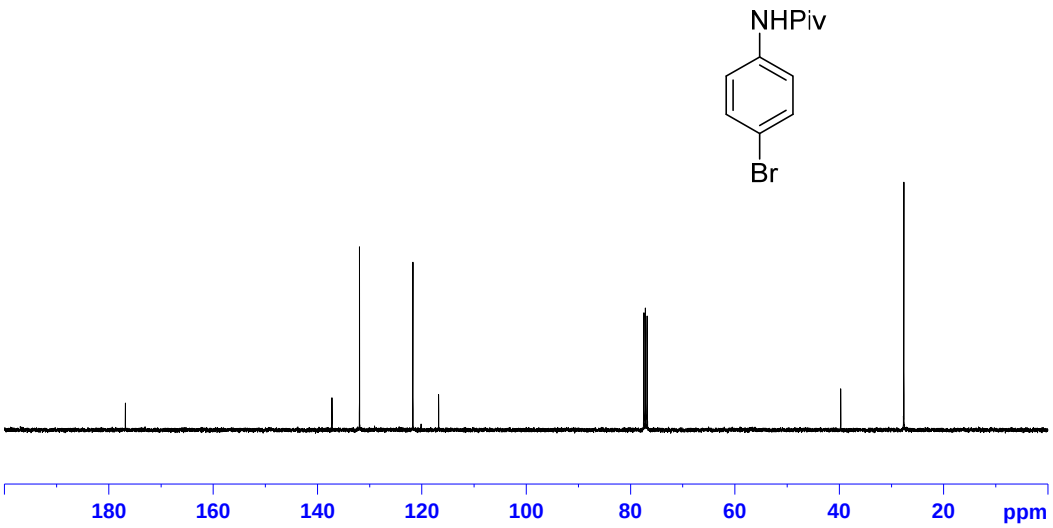
N-(4-bromophenyl)benzamide (3p)



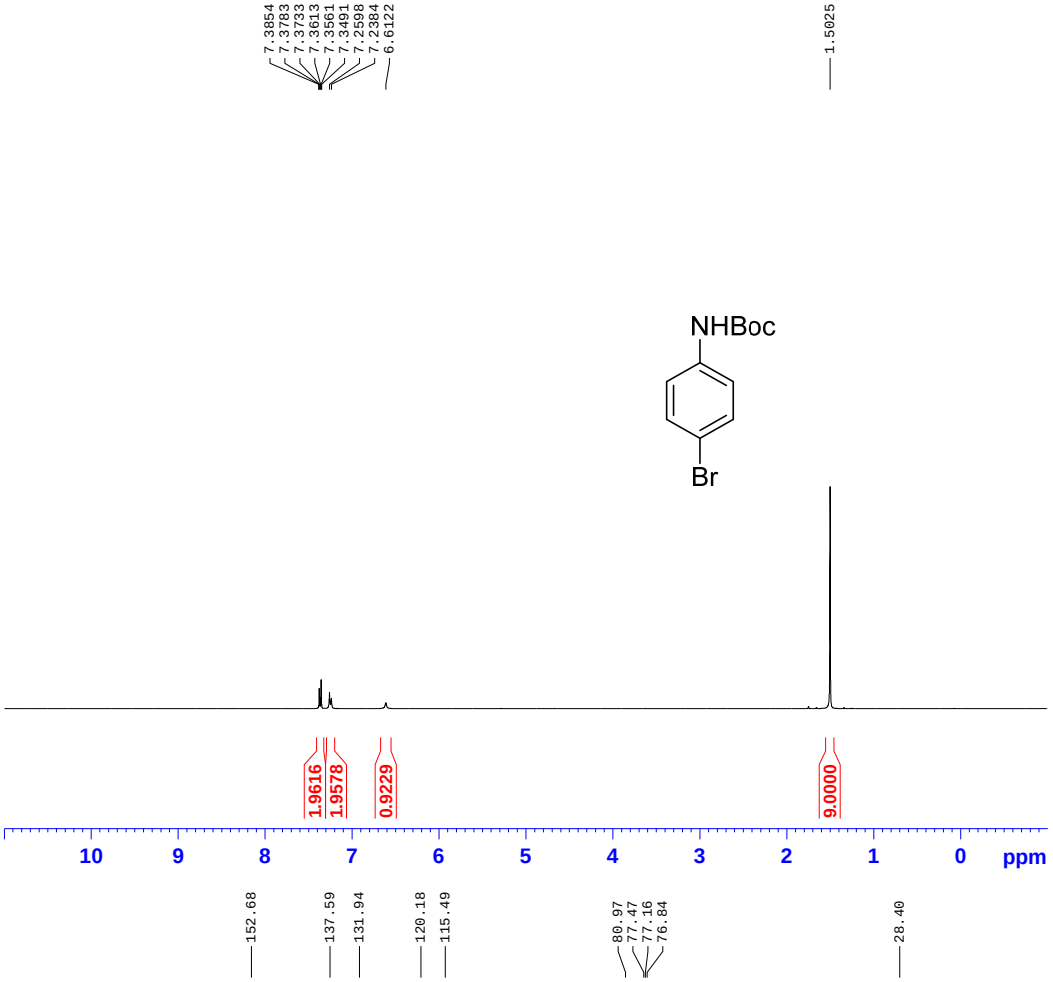


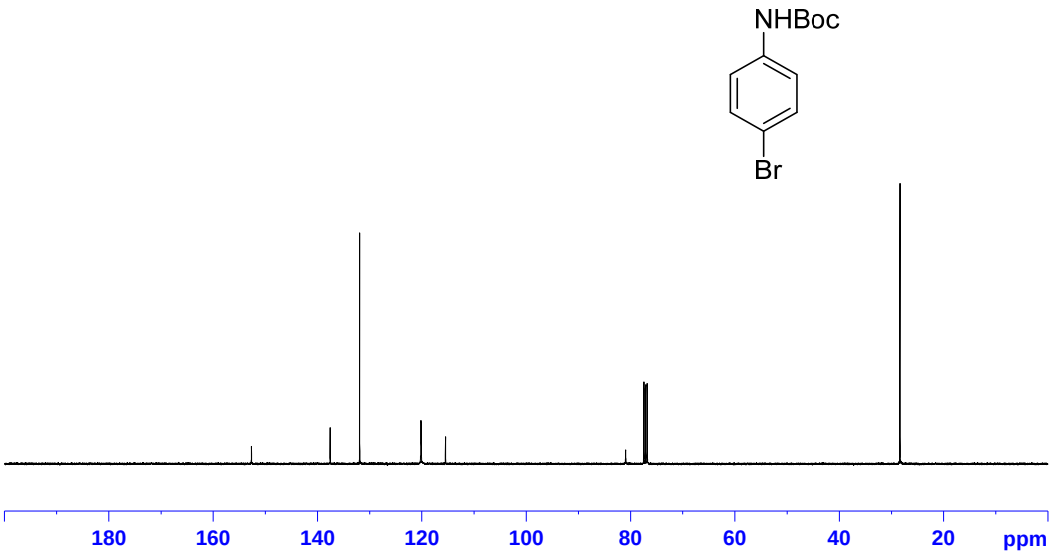
N-(4-Bromophenyl)-2,2-dimethylpropanamide (3q)



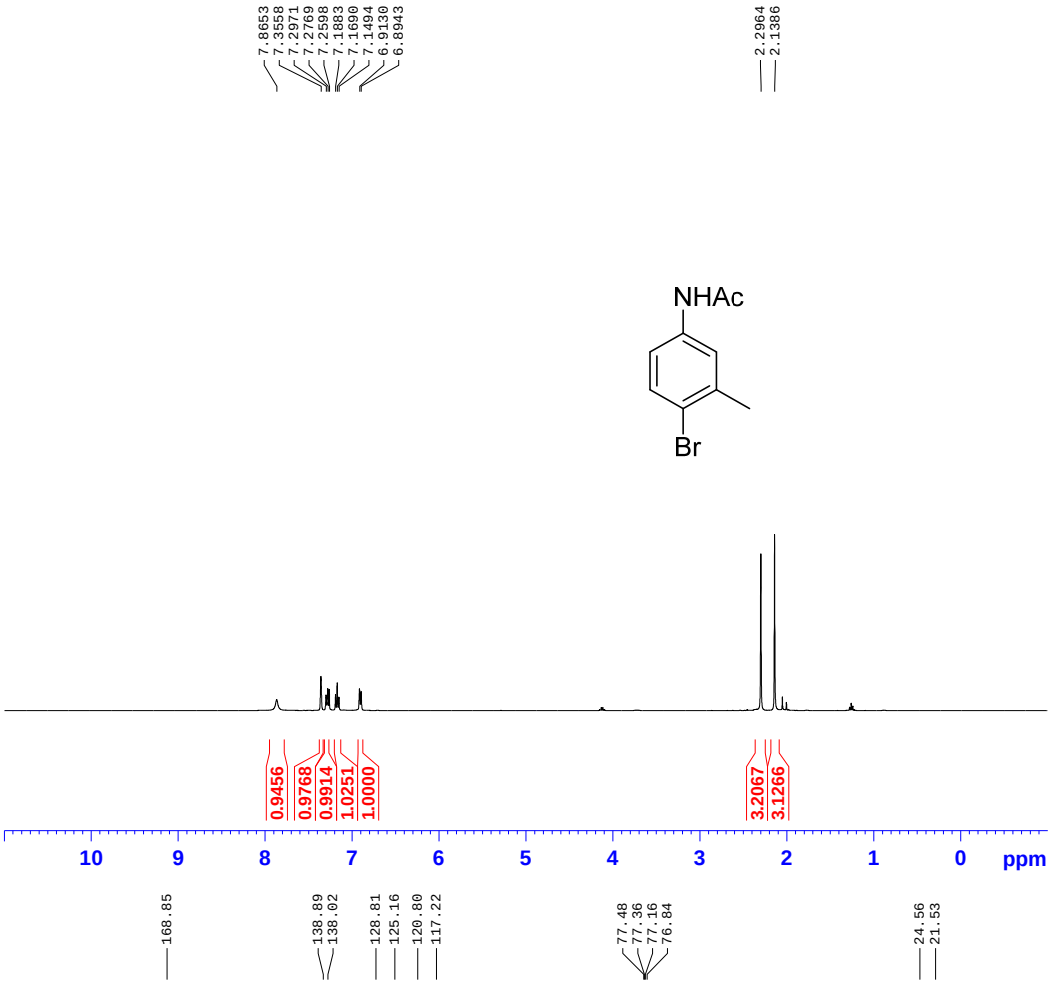


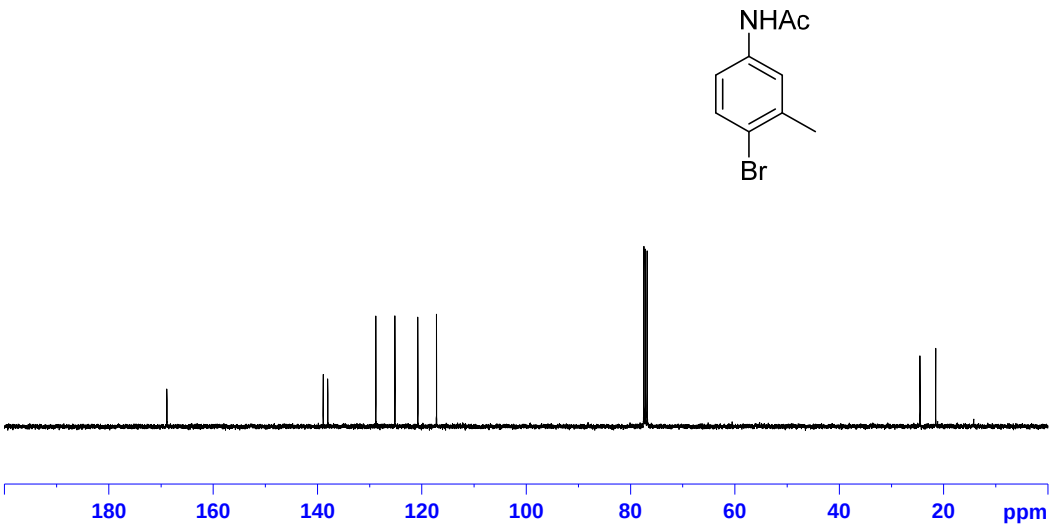
tert-butyl (4-bromophenyl)carbamate (3r)



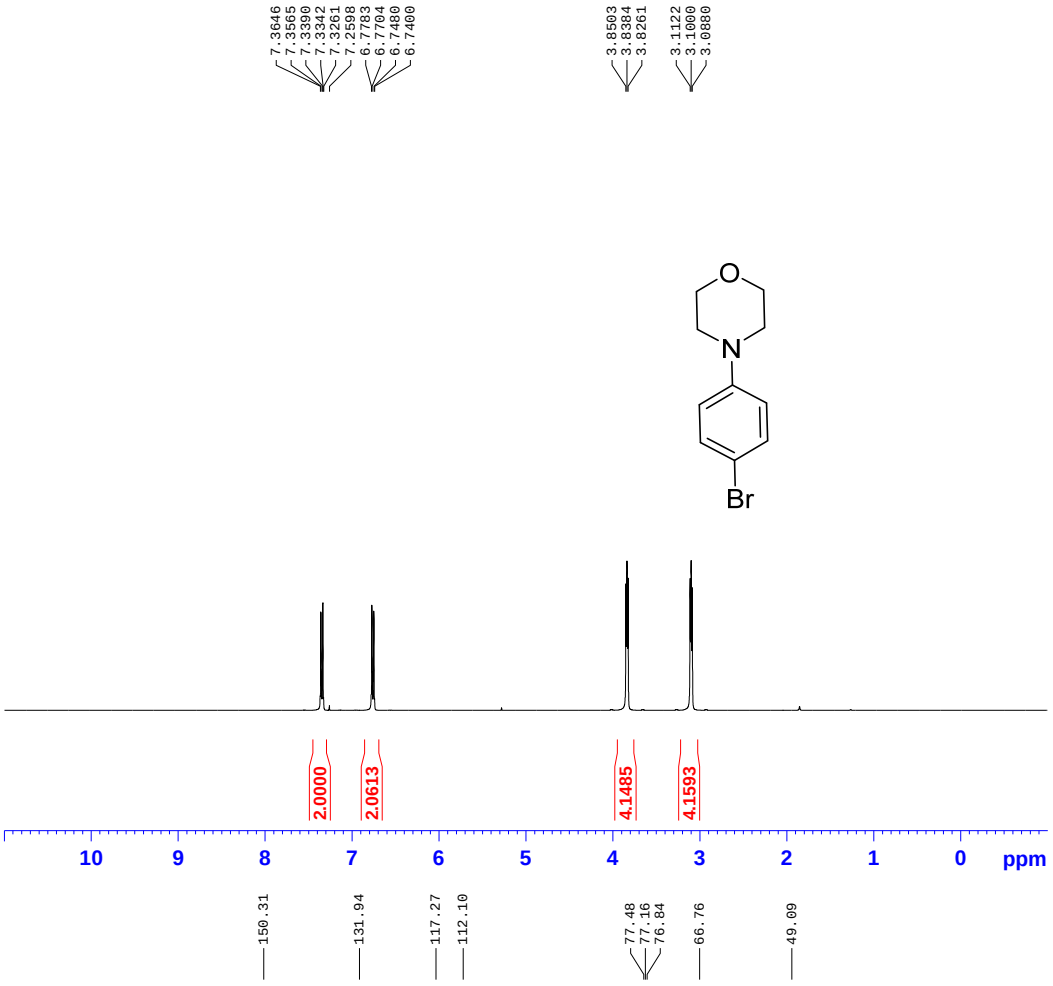


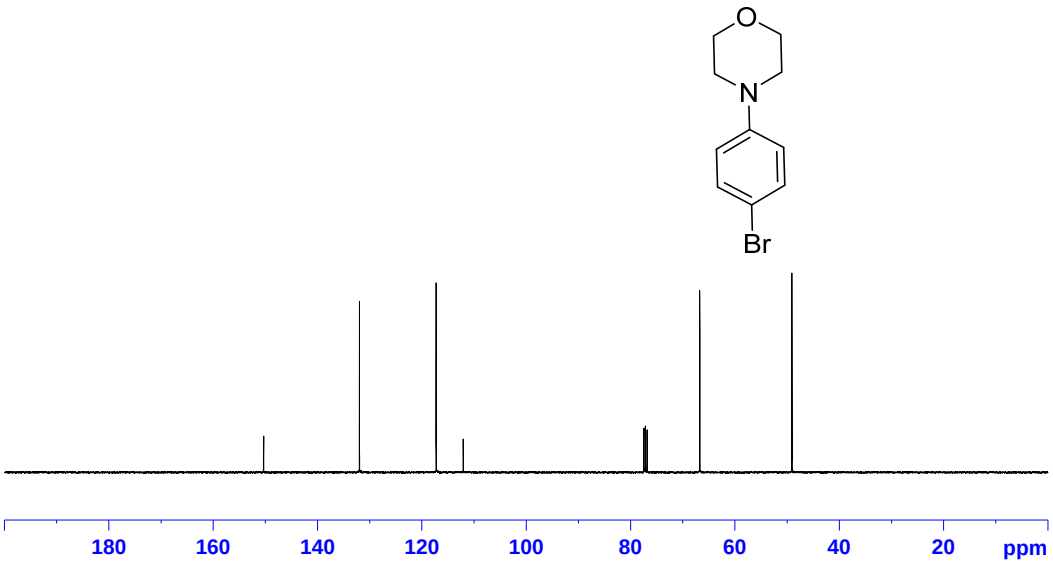
N-(4-bromo-3-methylphenyl)acetamide (3s)



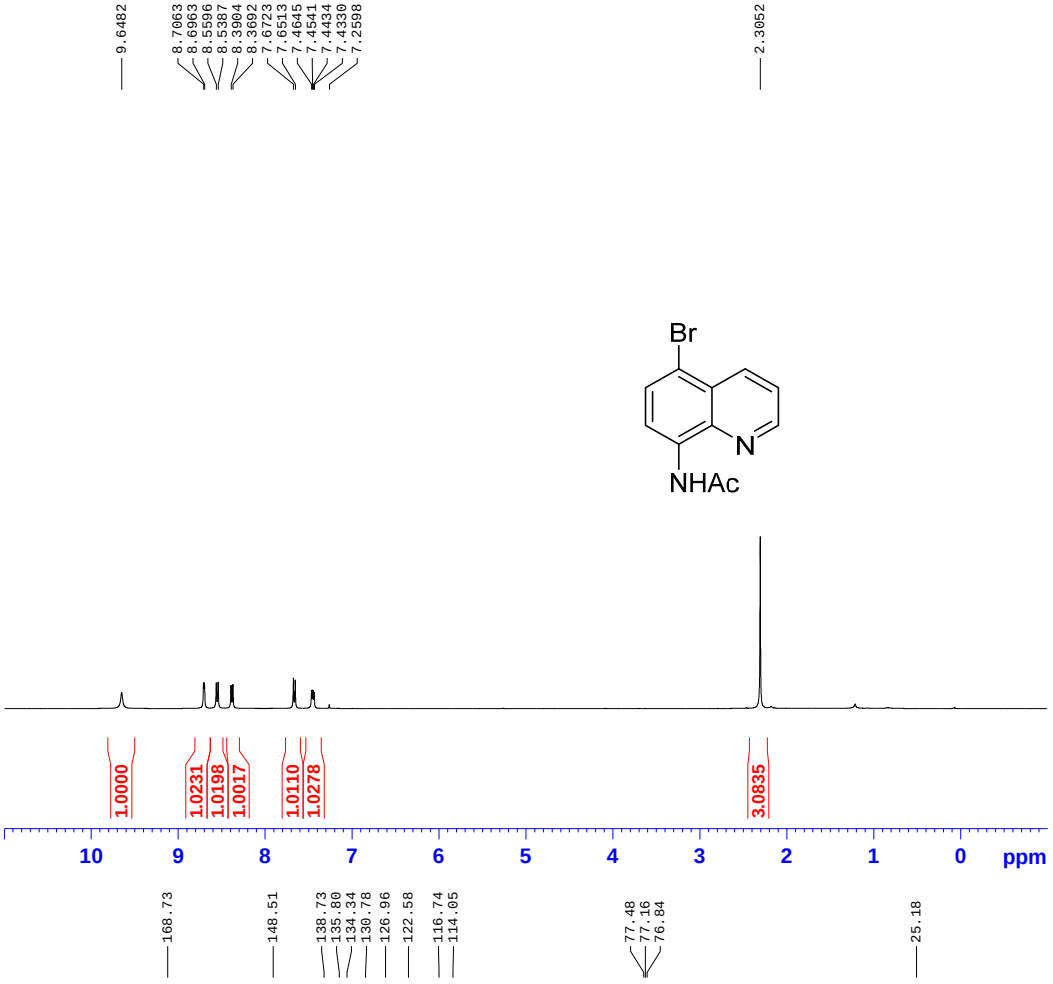


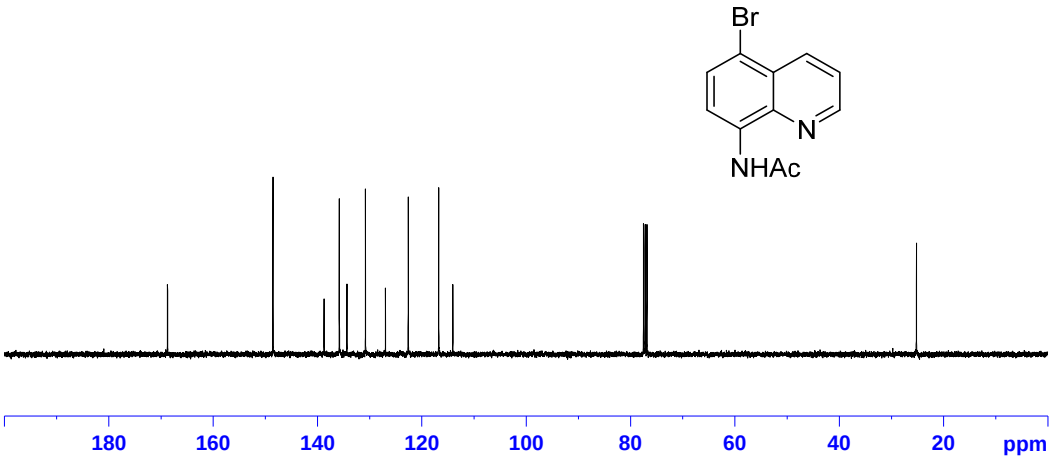
4-(4-bromophenyl)morpholine (3t)





N-(5-bromoquinolin-8-yl)acetamide (3u)





9-bromoanthracene (3v)