Supporting Information

Copper-Catalyzed α-Methylenation of Benzylpyridines Using Dimethylacetamide as One-Carbon Source

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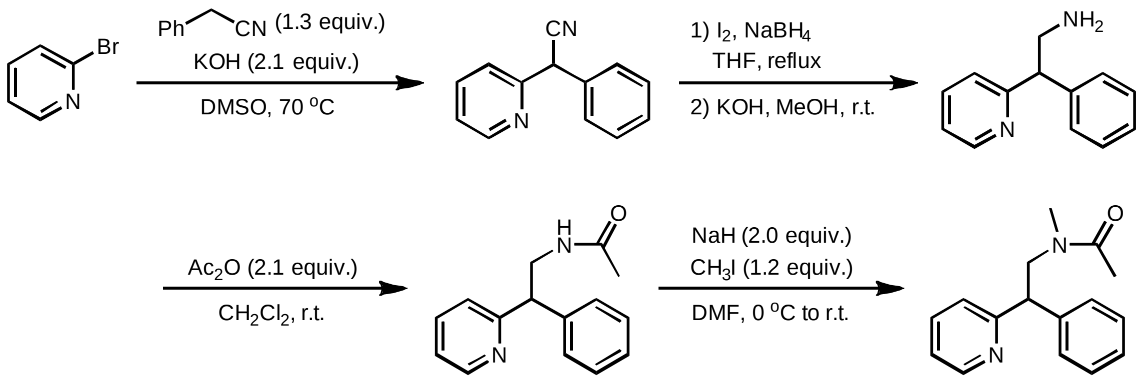
Experimental Section

General. ^{1}H and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively, for $CDCl_3$ solutions. HRMS data were obtained by EI using a double focusing mass spectrometer or APCI using a TOF mass spectrometer. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m). GC-MS analysis was carried out using a CBP-1 capillary column (i. d. 0.25 mm x 25 m). IR spectra were recorded as thin film. The structures of all products listed below were unambiguously determined by ^{1}H and ^{13}C NMR with the aid of NOE, COSY, HSQC, and HMBC experiments.

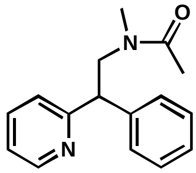
Benzylpyridines **1b-c**, **1e-i**, and **1k**,[S1] **1j**, and **1l-m**,[S2] 2-benzyl-4-methylpyridine (**1n**),[S3] 2-benzylpyrimidine (**1q**),[S4] and di(2-pyridyl)methane (**1r**)[S5] were prepared according to published procedures. Compound **E** was prepared as noted below. Other reagents were commercially available.

Preparation of *N*-methyl-*N*-[2-phenyl-2-(pyridin-2-yl)ethyl]acetamide (E).

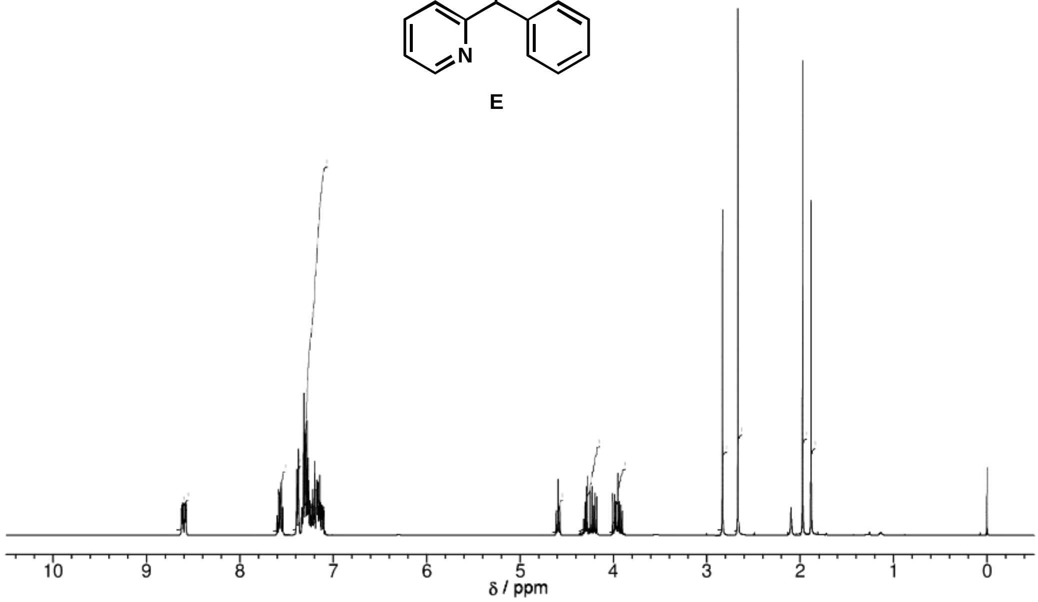


First, 2-phenyl-2-(pyridin-2-yl)ethan-1-amine was prepared according to a reported method.[S6, S7] Thus obtained 2-phenyl-2-(pyridin-2-yl)ethan-1-amine (1 mmol, 198 mg), acetic anhydride (2.1 mmol, 214 mg), and CH_2Cl_2 (1 mL) were added to a 20 mL two-necked flask with a calcium chloride tube and a rubber cup. Then, the resulting mixture was stirred under air at ambient temperature for 12 h. After the consumption of amine, which was confirmed by GC, the reaction mixture was extracted with CH_2Cl_2 (100 mL). The organic layer was washed by water (100 mL, three times), and dried over Na_2SO_4. After evaporation of the solvents under vacuum, the residue was dissolved in DMF (1.5 mL). Then, NaH (2 mmol, 48 mg) and methyl iodide (1.2 mmol, 170 mg) were added to the solution. The resulting mixture was stirred at 0 ^{o}C for 2 h, and allowed to warm to rt. After 8 h, the reaction mixture was extracted with ethyl acetate (100 mL). The organic layer was washed by water (100 mL, three times) and dried over Na_2SO_4. The mixture was then concentrated in vacuo and purified by column chromatography on silica gel using ethyl acetate-methanol (10:1, v/v) to afford *N*-methyl-*N*-[2-phenyl-2-(pyridin-2-yl)ethyl]acetamide (**E**) (167 mg, 66%). This product was isolated as a mixture of two rotamers (25 ^{o}C, 5:4).

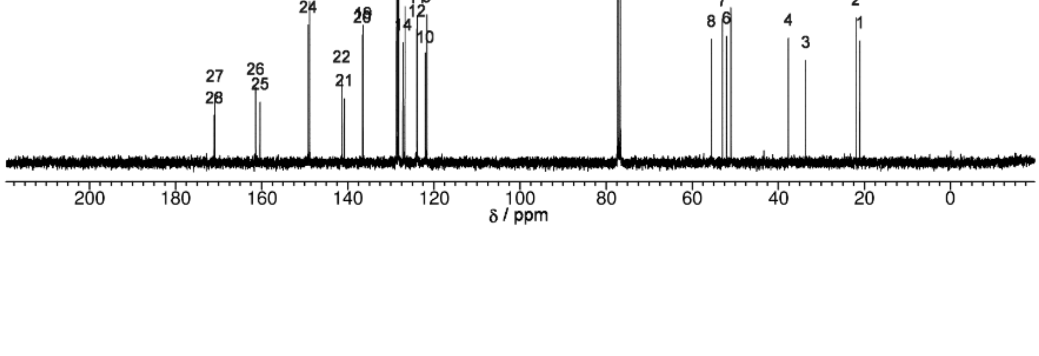
***N*-Methyl-*N*-[2-phenyl-2-(pyridin-2-yl)ethyl]acetamide (E).** Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88 (s, 1.34H), 1.97 (s, 1.66H), 2.67 (s, 1.66H), 2.83 (s, 1.34H), 3.90-4.01 (m, 1.11H), 4.18-4.32 (m, 1.34H), 4.59 (t, *J* = 8.0 Hz, 0.55H), 7.10-7.34 (m, 6H), 7.37-7.39 (m, 1H), 7.54-7.60 (m, 1H), 8.58 (dq, *J* = 0.9, 4.8 Hz, 0.55H), 8.62 (dq, *J* = 0.9, 4.8 Hz, 0.45H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.0, 22.0, 33.6, 37.7, 51.0, 52.0, 53.0, 55.5, 121.6, 122.0, 123.85, 123.93, 126.8, 127.2, 128.2, 128.3, 128.4, 128.7, 136.4, 136.6, 140.8, 141.4, 148.9, 149.2, 160.4, 161.4, 170.9, 171.0; HRMS *m/z* Calcd for $C_{16}H_{19}N_2O$ $(M+H^+)$ 255.1497, found 255.1498.



E



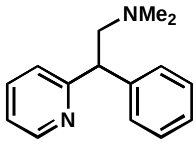
No.	ppm	Hz	Height	No.	ppm	Hz
1	21.04	2116.8	23.12	21	140.84	14169.8
2	21.99	2212.2	27.50	22	141.40	14227.0
3	33.59	3379.4	19.43	23	148.91	14981.9
4	37.66	3788.7	23.63	24	149.19	15010.5
5	51.01	5132.6	29.41	25	160.38	16136.6
6	52.03	5235.4	23.98	26	161.38	16237.1
7	53.00	5332.9	27.31	27	170.85	17189.3
8	55.53	5587.5	23.41	28	171.02	17206.9
9	121.62	12236.1	28.05			
10	121.98	12272.7	20.40			
11	123.85	12461.3	27.84			
12	123.93	12469.3	25.36			
13	126.75	12752.5	29.57			
14	127.20	12798.0	22.74			
15	128.16	12894.1	42.65			
16	128.31	12909.5	65.72			
17	128.43	12921.2	64.16			
18	128.70	12948.4	47.81			
19	136.43	13726.7	24.94			
20	136.61	13745.1	24.19			



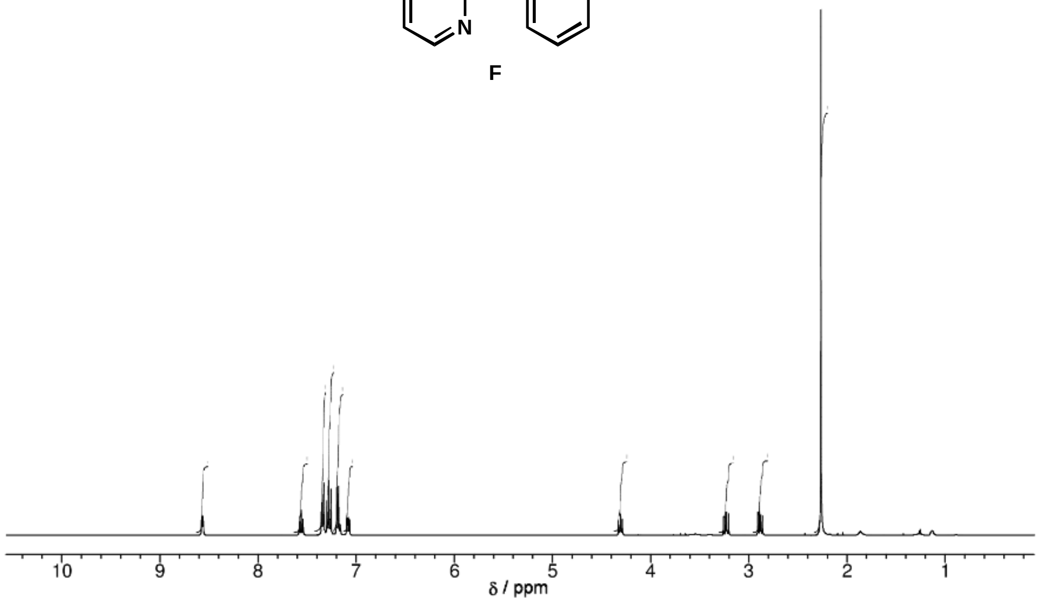
General Procedure for Methylenation of Benzylpyridines. To a 20 mL two-necked flask with a reflux condenser, a balloon, and a rubber cup were added benzylpyridine **1** (0.5 mmol), $Cu(OAc)_2 \cdot H_2O$ (0.05 mmol, 10 mg), $Na_2S_2O_8$ (1 mmol, 238 mg), 1-methylnaphthalene (ca. 40 mg) as internal standard, and DMA (2.5 mL). Then, the resulting mixture was stirred under nitrogen at 120 ^{o}C (bath temperature) for 4 h. After cooling, the reaction mixture was extracted with ethyl acetate (100 mL) and ethylenediamine (1 mL). The organic layer was washed by water (100 mL, three times), and dried over Na_2SO_4. After evaporation of the solvents under vacuum, product **2** was isolated by column chromatography on silica gel using hexane-ethyl acetate (5:1, v/v) as eluent.

Isolation of Intermediate F. To a 20 mL two-necked flask with a reflux condenser, a balloon, and a rubber cup were added 2-benzylpyridine (**1a**) (0.5 mmol, 85 mg), $Cu(OAc)_2 \cdot H_2O$ (0.05 mmol, 10 mg), $Na_2S_2O_8$ (1 mmol, 238 mg), 1-methylnaphthalene (ca. 40 mg) as internal standard, and DMA (2.5 mL). Then, the resulting mixture was stirred under nitrogen at 120 ^{o}C (bath temperature) for 30 min. After cooling, the reaction mixture was extracted with ethyl acetate (100 mL) and ethylenediamine (1 mL). The organic layer was washed by water (100 mL, three times), and dried over Na_2SO_4. The mixture was then concentrated in vacuo and purified by column chromatography on silica gel using methanol to afford intermediate **F** (10 mg, 9%), along with **2a** (22 mg, 24%) and recovered **1a** (50 mg, 59%).

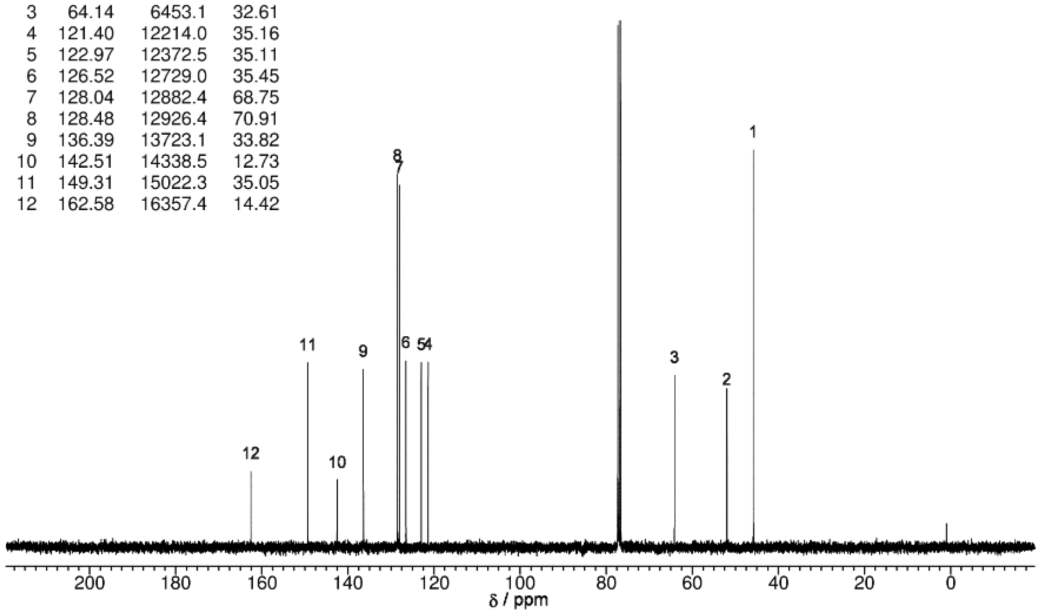
***N,N*-Dimethyl-2-phenyl-2-(pyridin-2-yl)ethan-1-amine (F).** Mp 58-59 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.26 (s, 6H), 2.88 (dd, J = 7.0, 12.4 Hz, 1H), 3.23 (dd, J = 8.3, 12.4 Hz, 1H), 4.31 (t, J = 7.7 Hz, 1H), 7.08 (ddd, J = 1.1, 4.9, 7.5 Hz, 1H), 7.16-7.20 (m, 2H), 7.26-7.30 (m, 2H), 7.33-7.35 (m, 2H), 7.56 (td, J = 1.8, 7.7 Hz, 1H), 8.57 (dq, J = 0.9, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 45.8, 52.0, 64.1, 121.4, 123.0, 126.5, 128.0, 128.5, 136.4, 142.5, 149.3, 162.6; HRMS *m/z* Calcd for $C_{15}H_{19}N_2$ (M+H^{+}) 227.1548, found 227.1549.



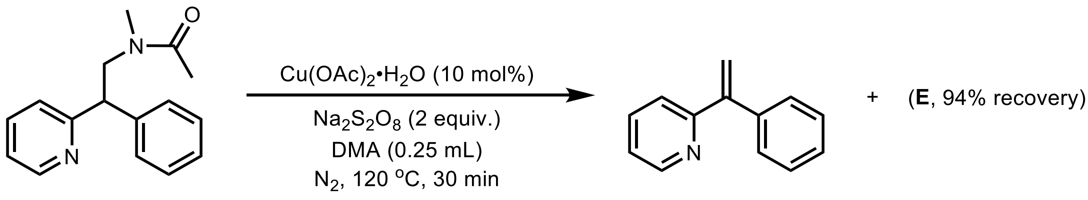
F



No.	ppm	Hz	Height
1	45.84	4611.8	75.44
2	51.95	5227.3	28.45
3	64.14	6453.1	32.61
4	121.40	12214.0	35.16
5	122.97	12372.5	35.11
6	126.52	12729.0	35.45
7	128.04	12882.4	68.75
8	128.48	12926.4	70.91
9	136.39	13723.1	33.82
10	142.51	14338.5	12.73
11	149.31	15022.3	35.05
12	162.58	16357.4	14.42



Reaction of E under Standard Conditions.



To a 20 mL two-necked flask with a reflux condenser, a balloon, and a rubber cup were added *N*-methyl-*N*-[2-phenyl-2-(pyridin-2-yl)ethyl]acetamide (**E**) (0.04 mmol, 10 mg), $Cu(OAc)_2{\cdot}H_2O$ (0.004 mmol, 0.8 mg), $Na_2S_2O_8$ (0.08 mmol, 19 mg), 1-methylnaphthalene (ca. 10 mg) as internal standard, and DMA (0.25 mL). Then, the resulting mixture was stirred under nitrogen at 120 ^oC (bath temperature) for 30 min. It was confirmed by GC analysis that only a trace amount of **2a** was formed and 94% of **E** was recovered. In addition, treatment of a mixture of **E** (0.04 mmol, 10 mg) and 1-(4-chlorophenyl)-1-(2-pyridyl)methane (**1d**) (0.04 mmol, 8 mg) with $Cu(OAc)_2{\cdot}H_2O$ (0.008 mmol, 1.6 mg), $Na_2S_2O_8$ (0.16 mmol, 38 mg), and 1-methylnaphthalene (ca. 20 mg) as internal standard in DMA (0.5 mL) under nitrogen at 120 ^oC (bath temperature) for 4 h selectively gave **2d** in 91% yield, along with a negligible amount of **2a** (3%). In this case, 70% of **E** was recovered.

Procedure for Oxygenation of 2-Benzylpyridine (1a). To a 20 mL two-necked flask with a reflux condenser, a calcium chloride tube, and a rubber cup were added 2-benzylpyridine (**1a**) (0.5 mmol, 85 mg), $Cu(OAc)_2{\cdot}H_2O$ (0.05 mmol, 10 mg), 1-methylnaphthalene (ca. 40 mg) as internal standard, and DMA (2.5 mL). Then, the resulting mixture was stirred at 120 ^oC (bath temperature) for 48 h under air. After cooling, the reaction mixture was extracted with ethyl acetate (100 mL) and ethylenediamine (1 mL). The organic layer was washed by water (100 mL, three times), and dried over Na_2SO_4. The mixture was then concentrated in vacuo and purified by column chromatography on silica gel using hexane-ethyl acetate (3:1, v/v) to afford 2-benzoylpyridine (**3**) (81 mg, 89%).

General Procedure for Dimerization of Benzylpyridines. To a 20 mL two-necked flask with a reflux condenser, a balloon, and a rubber cup were added benzylpyridine **1** (0.5 mmol), $Cu(OAc)_2{\cdot}H_2O$ (1 mmol, 200 mg), 1-methylnaphthalene (ca. 40 mg) as internal standard, and DMA (2.5 mL). Then, the resulting mixture was stirred under nitrogen at 120 ^oC (bath temperature) for 8 h. After cooling, the reaction mixture was extracted with ethyl acetate (100 mL) and ethylenediamine (1 mL). The organic layer was washed by water (100 mL, three times), and dried over Na_2SO_4. After evaporation of the solvents under vacuum, product **4** was isolated by column chromatography on silica gel using hexane-ethyl acetate (2:1, v/v) as eluent.

X-ray Crystal-Structure Analysis. The configuration of *meso*-**4o** was determined for a white microcrystal obtained from acetonitrile/dioxane (Figure S1).

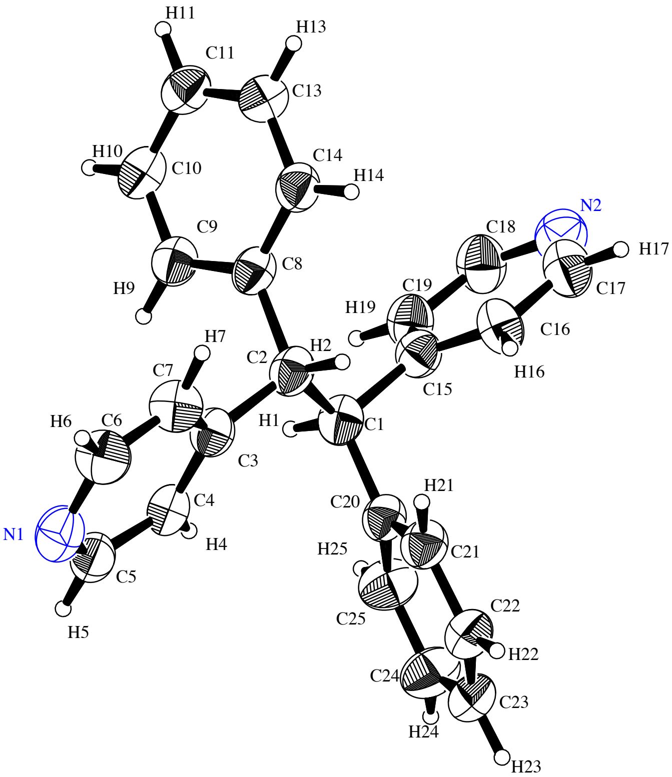


Figure S1. ORTEP drawing of compound *meso*-**4o**•$C_4H_8O_2$.

Crystal data: $C_{28}H_{28}N_2O_2$, Mw = 424.54, monoclinic, space group P 1 2_1/c 1, T = 296 K, a = 8.4619(3), b = 16.3894(6), c = 16.8746(7), β = 102.4883(17), V = 2284.88(15), Z = 4, 4146 reflections measured, R = 0.1041, Rw = 0.3693.

Characterization Data of Products

2-(1-Phenylvinyl)pyridine (2a).[3a] Oil, 63 mg (70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 (d, J = 1.5 Hz, 1H), 5.99 (d, J = 1.5 Hz, 1H), 7.20 (ddd, J = 1.1, 4.8, 7.5 Hz, 1H), 7.26 (dt, J = 1.0, 7.9 Hz, 1H), 7.32-7.36 (m, 5H), 7.62 (td, J = 1.8, 7.8 Hz, 1H), 8.64 (dq, J = 1.0, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.6, 122.4, 122.8, 127.8, 128.2, 128.4, 136.2, 140.3, 149.2, 149.4, 158.5; HRMS *m/z* Calcd for $C_{13}H_{12}N$ (M+H$^+$) 182.0970, found 182.0969.

2-(1-Phenylvinyl-2,2-d_2)pyridine (2a-d_2). Oil, 51 mg (55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (ddd, J = 1.1, 4.8, 7.5 Hz, 1H), 7.26 (dt, J = 1.0, 7.9 Hz, 1H), 7.32-7.36 (m, 5H), 7.62 (td, J = 1.8, 7.8 Hz, 1H), 8.64 (dq, J = 1.0, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.5(5) (J = 27.9 Hz), 122.4, 122.8, 127.8, 128.3, 128.4, 136.2, 140.3, 149.0, 149.4, 158.5; HRMS *m/z* Calcd for $C_{13}H_{10}D_2N$ (M+H$^+$) 184.1095, found 184.1094.

2-[1-(*p*-Tolyl)vinyl]pyridine (2b). Oil, 53 mg (55%); IR: (neat, cm^{-1}) 3024, 1581, 1512, 1466, 1427, 1334, 1041, 910, 802; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.37 (s, 3H), 5.58 (d, J = 1.5 Hz, 1H), 5.93 (d, J = 1.5 Hz, 1H), 7.15-7.21 (m, 3H), 7.24-7.29 (m, 3H), 7.61 (td, J = 1.8, 7.6 Hz, 1H), 8.63 (dq, J = 1.0, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 117.0, 122.3, 122.8, 128.3, 128.9, 136.2, 137.5, 137.6, 149.0, 149.3, 158.7; HRMS *m/z* Calcd for $C_{14}H_{14}N$ (M+H$^+$) 196.1126, found 196.1125.

2-[1-(4-Methoxyphenyl)vinyl]pyridine (2c).[2a] Oil, 25 mg (24%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (s, 3H), 5.55 (d, J = 1.5 Hz, 1H), 5.86 (d, J = 1.5 Hz, 1H), 6.87-6.91 (m, 2H), 7.20 (ddd, J = 1.1, 4.9, 7.6 Hz, 1H), 7.27-7.30 (m, 3H), 7.63 (td, J = 1.8, 7.6 Hz, 1H), 8.64 (dq, J = 1.0, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 113.7, 116.4, 122.3, 122.8, 129.5, 132.8, 136.2, 148.7, 149.3, 158.9, 159.4; HRMS *m/z* Calcd for $C_{14}H_{14}NO$ (M+H$^+$) 212.1075, found 212.1077.

2-[1-(4-Chlorophenyl)vinyl]pyridine (2d).[2a] Oil, 87 mg (81%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 (d, J = 1.2 Hz, 1H), 5.96 (d, J = 1.2 Hz, 1H), 7.21 (ddd, J = 1.2, 4.8, 7.6 Hz, 1H), 7.26-7.34 (m, 5H), 7.64 (td, J = 1.8, 7.7 Hz, 1H), 8.63 (dq, J = 1.0, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 118.0, 122.5, 122.6, 128.4, 129.7, 133.7, 136.4, 138.7, 148.1, 149.4, 158.1; HRMS *m/z* Calcd for $C_{13}H_{11}ClN$ (M+H$^+$) 216.0580, found 216.0577.

2-[1-[4-(Trifluoromethyl)phenyl]vinyl]pyridine (2e). Oil, 104 mg (83%); IR: (neat, cm^{-1}) 3008, 1581, 1327, 1165, 1126, 1072, 849; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.67 (d, J = 1.0 Hz, 1H), 6.05 (d, J = 1.0 Hz, 1H), 7.23 (ddd, J = 1.1, 4.9, 7.6 Hz, 1H), 7.30 (dt, J = 1.0, 8.0 Hz, 1H), 7.47 (d, J = 8.1 Hz, 2H), 7.62 (d, J = 8.1 Hz, 2H), 7.66 (td, J = 1.8, 7.7 Hz, 1H), 8.64 (dq, J = 1.0, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.1, 122.6, 122.7, 124.2 (q, J = 272.2 Hz), 125.2 (q, J = 3.7 Hz), 128.7, 129.8 (q, J = 32.3 Hz), 136.5, 143.9, 148.2, 149.5, 157.8; HRMS *m/z* Calcd for $C_{14}H_{11}F_3N$ (M+H$^+$) 250.0844, found 250.0845.

4-(1-(Pyridin-2-yl)vinyl)benzonitrile (2f). Oil, 77 mg (75%); IR: (neat, cm^{-1}) 3055, 2229, 1581, 1466, 1403, 925, 848, 802; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.70 (d, J = 0.9 Hz, 1H), 6.04 (d, J = 0.9 Hz, 1H), 7.26 (ddd, J = 1.1, 4.9, 7.6 Hz, 1H), 7.33 (dt, J = 1.0, 7.9 Hz, 1H), 7.47 (d, J = 8.7 Hz, 2H), 7.65 (d, J = 8.7 Hz, 2H), 7.70 (td, J = 1.8, 7.7 Hz, 1H), 8.63 (dq, J = 1.0, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 111.4, 118.8, 119.7, 122.6, 122.8, 129.0, 132.0, 136.6, 144.9, 147.9, 149.4, 157.4; HRMS m/z Calcd for $C_{14}H_{10}N_2$ (M^+) 206.0844, found 206.0837.

2-[1-[3-(Trifluoromethyl)phenyl]vinyl]pyridine (2g). Oil, 83 mg (67%); IR: (neat, cm^{-1}) 3008, 1581, 1466, 1435, 1311, 1122, 1072, 802; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.66 (d, J = 1.1 Hz, 1H), 6.04 (d, J = 1.1 Hz, 1H), 7.23 (ddd, J = 1.1, 4.8, 7.6 Hz, 1H), 7.30 (dt, J = 1.1, 7.9 Hz, 1H), 7.45-7.60 (m, 3H), 7.64 (s, 1H), 7.67 (td, J = 1.7, 7.6 Hz, 1H), 8.64 (dq, J = 1.0, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 118.9, 122.6, 122.7, 124.1 (q, J = 272.2 Hz), 124.5 (q. J = 3.7 Hz), 125.1 (q. J = 3.7 Hz), 128.7, 130.7 (q, J = 32.3 Hz), 131.7 (d, J = 1.5 Hz), 136.5, 141.1, 148.1, 149.5, 157.8; HRMS m/z Calcd for $C_{14}H_{10}F_3N$ (M^+) 249.0765, found 249.0767.

2-[1-(*o*-Tolyl)vinyl]pyridine (2h).[S8] Oil, 67 mg (69%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.07 (s, 3H), 5.39 (d, J = 2.0 Hz, 1H), 6.42 (d, J = 2.0 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 7.15 (ddd, J = 1.1, 4.9, 7.5 Hz, 1H), 7.20-7.30 (m, 4H), 7.54 (td, J = 1.8, 7.6 Hz, 1H), 8.63 (dq, J = 0.9, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.0, 118.6, 121.5, 122.2, 125.9, 127.7, 129.98, 130.04, 136.2, 136.4, 140.4, 148.5, 149.5, 157.3; HRMS m/z Calcd for $C_{14}H_{13}N$ (M^+) 195.1048, found 195.1047.

2-[1-(2-Fluorophenyl)vinyl]pyridine (2i).[S8] Oil, 76 mg (76%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 (d, J = 1.5 Hz, 1H), 6.28 (d, J = 1.5 Hz, 1H), 7.05-7.10 (m, 1H), 7.15-7.22 (m, 3H), 7.31-7.38 (m, 2H), 7.61 (td, J = 1.8, 7.8 Hz, 1H), 8.61 (dq, J = 0.9, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 115.6 (d, J = 22.0 Hz), 120.2, 121.4, 122.4, 124.2 (d, J = 3.7 Hz), 128.2 (d, J = 14.7 Hz), 129.6 (d, J = 8.1 Hz), 131.4 (d, J = 3.6 Hz), 136.4, 143.8, 149.2, 157.3, 160.0 (d, J = 248.7 Hz); HRMS m/z Calcd for $C_{13}H_{10}FN$ (M^+) 199.0797, found 199.0794.

2-[1-(2-Bromophenyl)vinyl]pyridine (2j).[S8] Oil, 79 mg (61%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.46 (d, J = 1.5 Hz, 1H), 6.44 (d, J = 1.5 Hz, 1H), 7.03 (dt, J = 1.0, 8.0 Hz, 1H), 7.16 (ddd, J = 1.1, 4.8, 7.6 Hz, 1H), 7.23 (dt, J = 4.6, 8.0 Hz, 1H), 7.36-7.38 (m, 2H), 7.55-7.62 (m, 2H), 8.62 (dq, J = 0.9, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.6, 121.5, 122.3, 123.3, 127.5, 129.2, 131.6, 132.8, 136.4, 141.6, 148.4, 149.4, 156.3; HRMS m/z Calcd for $C_{13}H_{11}BrN$ ($M+H^+$) 260.0075, found 260.0072.

2-[1-(Naphthalen-1-yl)vinyl]pyridine (2k). Oil, 75 mg (65%); IR: (neat, cm^{-1}) 3008, 1581, 1466, 1427, 1157, 1049, 926, 779; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.57 (d, J = 2.1 Hz, 1H), 6.67 (d, J = 2.1 Hz, 1H), 6.83 (d, J = 7.9 Hz, 1H), 7.12 (ddd, J = 1.1, 4.8, 7.5 Hz, 1H), 7.32 (ddd, J = 1.2, 6.9, 8.2 Hz, 1H), 7.40-7.53 (m, 4H), 7.71 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 8.1 Hz, 2H), 8.65 (dq, J = 0.9, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 120.0, 122.1, 122.3, 125.5, 125.8, 125.9, 126.2, 127.3, 128.1, 128.2, 131.9, 133.6, 136.4, 138.6, 147.3, 149.4, 157.5; HRMS m/z Calcd for $C_{17}H_{13}N$ (M^+) 231.1048, found 231.1049.

2-[1-(Pyridin-3-yl)vinyl]pyridine (2l). Oil, 56 mg (61%); IR: (neat, cm^{-1}) 3046, 1581, 1473, 1411, 1335, 1026, 918, 802; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.68 (d, J = 1.1 Hz, 1H), 6.07 (d, J = 1.1 Hz, 1H), 7.24 (ddd, J = 1.1, 4.8, 7.6 Hz, 1H), 7.28-7.34 (m, 2H), 7.65-7.70 (m, 2H), 8.58 (dd, J = 1.6, 4.9 Hz, 1H), 8.63-8.65 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.0, 122.4, 122.7, 123.0, 135.7, 135.9, 136.5, 146.1, 148.9, 149.35, 149.40, 157.5; HRMS m/z Calcd for $C_{12}H_{11}N_2$ (M+H$^+$) 183.0922, found 183.0925.

2-[1-(Thiophen-3-yl)vinyl]pyridine (2m). Oil, 15 mg (16%); IR: (neat, cm^{-1}) 3008, 1581, 1466, 1434, 1288, 1149, 995, 795; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.69 (d, J = 1.4 Hz, 1H), 5.81 (d, J = 1.4 Hz, 1H), 7.18 (dd, J = 1.4, 4.9 Hz, 1H), 7.23 (ddd, J = 1.1, 4.9, 7.6 Hz, 1H), 7.28 (dd, J = 1.4, 2.9 Hz, 1H), 7.31 (dd, J = 2.9, 4.9 Hz, 1H), 7.38 (dt, J = 1.1, 7.8 Hz, 1H), 7.67 (td, J = 1.8, 7.7 Hz, 1H), 8.65 (dq, J = 0.9, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 116.6, 122.5, 122.6, 123.4, 125.4, 127.4, 136.4, 140.9, 143.7, 149.3, 158.6; HRMS m/z Calcd for $C_{11}H_{10}NS$ (M+H$^+$) 188.0534, found 188.0536.

4-Methyl-2-(1-phenylvinyl)pyridine (2n) + 2-(1-Phenylvinyl)-4-vinylpyridine (2n'). 51 mg (52%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 3H, **2n**), 5.44 (d, J = 10.8 Hz, 1H, **2n'**), 5.58 (d, J = 1.5 Hz, 1H, **2n**), 5.62 (d, J = 1.5 Hz, 1H, **2n'**), 5.91 (d, J = 17.6 Hz, 1H, **2n'**), 5.96 (d, J = 1.5 Hz, 1H, **2n**), 5.98 (d, J = 1.5 Hz, 1H, **2n'**), 6.62 (dd, J = 10.8, 17.6 Hz, 1H, **2n'**), 7.02-7.04 (m, 1H, **2n**), 7.08-7.09 (m, 1H, **2n**), 7.21-7.24 (m, 2H, **2n'**), 7.32-7.37 (m, 5H+5H, **2n+2n'**), 8.50 (d, J = 4.9 Hz, 1H, **2n**), 8.59 (d, J = 5.0 Hz, 1H, **2n'**); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.0, 117.5, 117.7, 118.6, 119.3, 120.2, 123.4, 123.7, 127.7, 127.8, 128.2, 128.27, 128.33, 128.4, 134.8, 140.3, 140.5, 145.1, 147.3, 149.1 (overlapped), 149.2, 149.7, 158.4, 159.1; HRMS m/z Calcd for $C_{14}H_{14}N$ (M+H$^+$) 196.1126, found 196.1127, and $C_{15}H_{14}N$ (M+H$^+$) 208.1126, found 208.1128.

4-(1-Phenylvinyl)pyridine (2o).[4b] Oil, 68 mg (75%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 (d, J = 0.7 Hz, 1H), 5.61 (d, J = 0.7 Hz, 1H), 7.24 (dd, J = 1.7, 4.5 Hz, 2H), 7.28-7.32 (m, 2H), 7.34-7.39 (m, 3H), 8.58 (dd, J = 1.7, 4.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 116.9, 122.7, 128.1, 128.2, 128.4, 139.7, 147.9, 148.7, 149.9; HRMS m/z Calcd for $C_{13}H_{11}N$ (M$^+$) 181.0891, found 181.0889.

4-[1-(4-Chlorophenyl)vinyl)]pyridine (2p). Oil, 60 mg (56%); IR: (neat, cm^{-1}) 3410, 1597, 1489, 1412, 1281, 1095, 918, 833; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.59 (d, J = 0.4 Hz, 1H), 5.62 (d, J = 0.4 Hz, 1H), 7.21-7.25 (m, 4H), 7.32-7.35 (m, 2H), 8.59 (dd, J = 1.7, 4.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.3, 122.6, 128.6, 129.4, 134.2, 138.2, 146.8, 148.2, 150.0; HRMS m/z Calcd for $C_{13}H_{10}ClN$ (M$^+$) 215.0502, found 215.0500.

2-(1-Phenylvinyl)pyrimidine (2q). Oil, 59 mg (65%); IR: (neat, cm^{-1}) 3055, 1558, 1496, 1419, 1350, 1072, 926, 833; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.83 (d, J = 1.7 Hz, 1H), 6.48 (d, J = 1.7 Hz, 1H), 7.17 (t, J = 4.8 Hz, 1H), 7.32-7.43 (m, 5H), 8.75 (d, J = 4.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.1, 122.4, 127.8, 128.0, 128.6, 139.4, 148.3, 156.9, 166.2; HRMS m/z Calcd for $C_{12}H_{11}N_2$ (M+H$^+$) 183.0922, found 183.0921.

2-Benzoylpyridine (3).[13a] Mp 40-41 ºC, 81 mg (89%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.50 (m, 3H), 7.57-7.61 (m, 1H), 7.89 (td, J = 1.7, 7.7 Hz, 1H), 8.02-8.08 (m, 3H), 8.72 (dq, J = 0.9, 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 124.5, 126.1, 128.1, 130.9, 132.8, 136.2, 137.0, 148.5, 155.0, 193.8; HRMS *m/z* Calcd for $C_{12}H_9NO$ (M^+) 183.0684, found 183.0685.

1,2-Diphenyl-1,2-di(pyridin-2-yl)ethane (4a).[S9] 69 mg (82%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.27 (d, J = 8.3 Hz, 4H), 6.90 (dddd, J = 1.1, 3.7, 4.8, 8.6 Hz, 4H), 6.98-7.03 (m, 4H), 7.08-7.14 (m, 10H), 7.23 (dt, J = 1.1, 7.8 Hz, 2H), 7.33-7.41 (m, 8H), 7.44-7.46 (m, 4H), 8.42 (dq, J = 1.0, 4.9 Hz, 2H), 8.47 (dq, J = 1.0, 4.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 57.6, 57.7, 120.8, 121.0, 123.9, 124.2, 126.0, 126.1, 127.9, 128.0, 128.6, 128.8, 135.9, 136.0, 142.0, 142.2, 148.8, 149.2, 162.0, 162.5; HRMS *m/z* Calcd for $C_{24}H_{20}N_2$ (M^+) 336.1626, found 336.1624.

***meso*-1,2-Diphenyl-1,2-di(pyridin-4-yl)ethane (*meso*-4o).**[S9] Mp 234-235 ºC, 37 mg (44%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.73 (s, 2H), 7.05-7.19 (m, 14H), 8.34 (d, J = 5.5 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.2, 123.6, 127.0, 128.3, 128.7, 140.8, 149.7, 151.5; HRMS *m/z* Calcd for $C_{24}H_{21}N_2$ ($M+H^+$) 337.1705, found 337.1708.

***dl*-1,2-Diphenyl-1,2-di(pyridin-4-yl)ethane (*dl*-4o).**[S9] Mp 217-218 ºC, 35 mg (42%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.73 (s, 2H), 7.05-7.16 (m, 14H), 8.37 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.1, 123.6, 126.8, 128.3, 128.5, 140.8, 149.9, 151.5; HRMS *m/z* Calcd for $C_{24}H_{21}N_2$ ($M+H^+$) 337.1705, found 337.1707.

1,1,2,2-Tetra(pyridin-2-yl)ethane (4r).[S10] Mp > 300 ºC, 70 mg (82%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 (s, 2H), 6.90 (ddd, J = 1.7, 4.8, 7.1 Hz, 4H), 7.37-7.43 (m, 8H), 8.44 (ddd, J = 1.0, 1.7, 4.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 58.8, 121.1, 124.1, 135.9, 149.1, 161.0; HRMS *m/z* Calcd for $C_{22}H_{18}N_4$ (M^+) 338.1531, found 338.1533.

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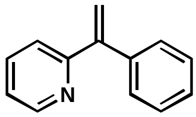

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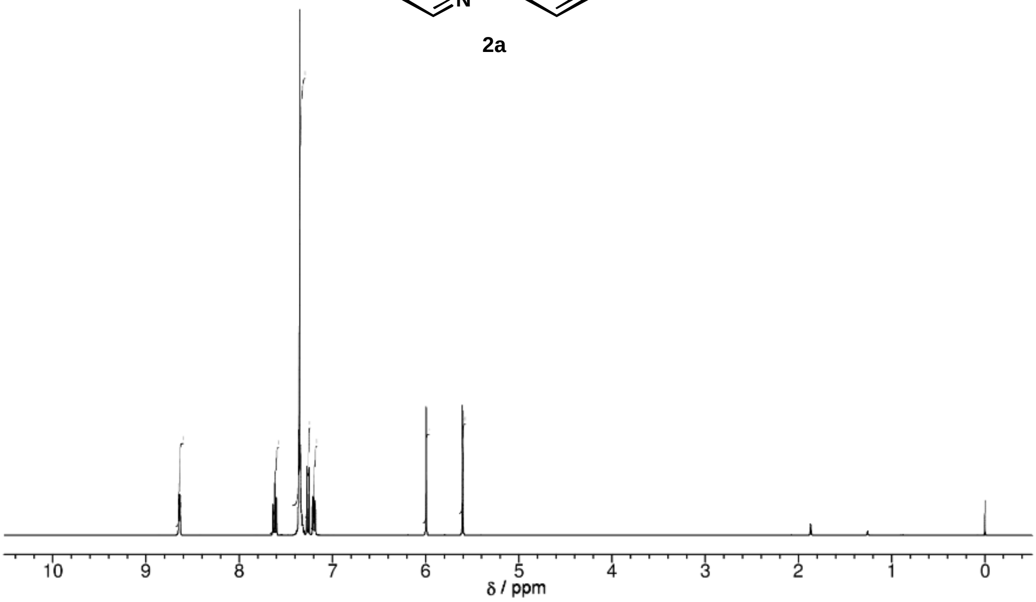

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5	128.24	12902.2	76.68
6	128.37	12916.1	100
7	136.20	13703.2	44.07
8	140.33	14119.2	15.63
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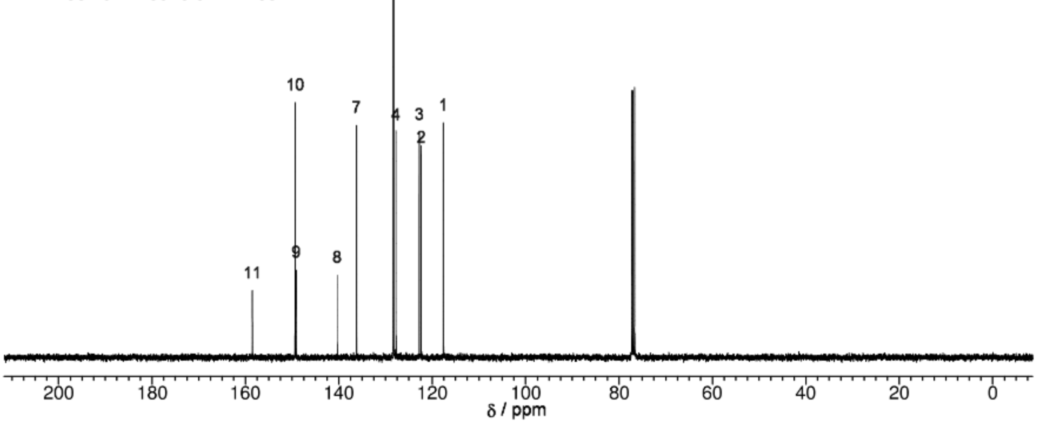

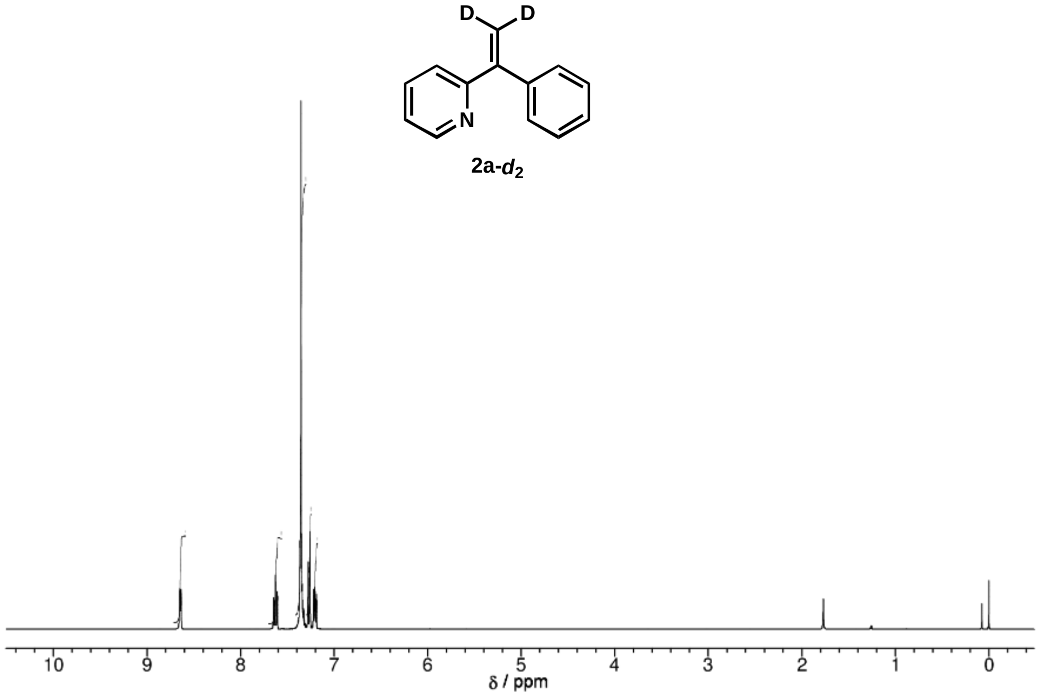

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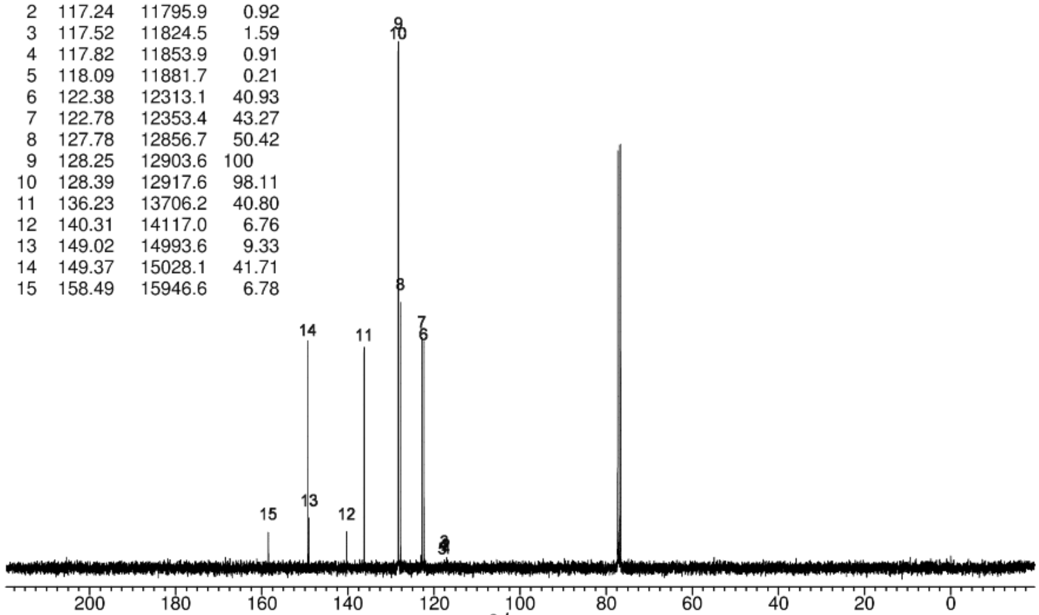
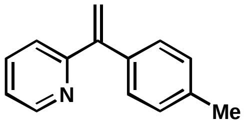
5	118.09	11881.7	0.21
6	122.38	12313.1	40.93
7	122.78	12353.4	43.27
8	127.78	12856.7	50.42
9	128.25	12903.6	100
10	128.39	12917.6	98.11
11	136.23	13706.2	40.80
12	140.31	14117.0	6.76
13	149.02	14993.6	9.33
14	149.37	15028.1	41.71
15	158.49	15946.6	6.78





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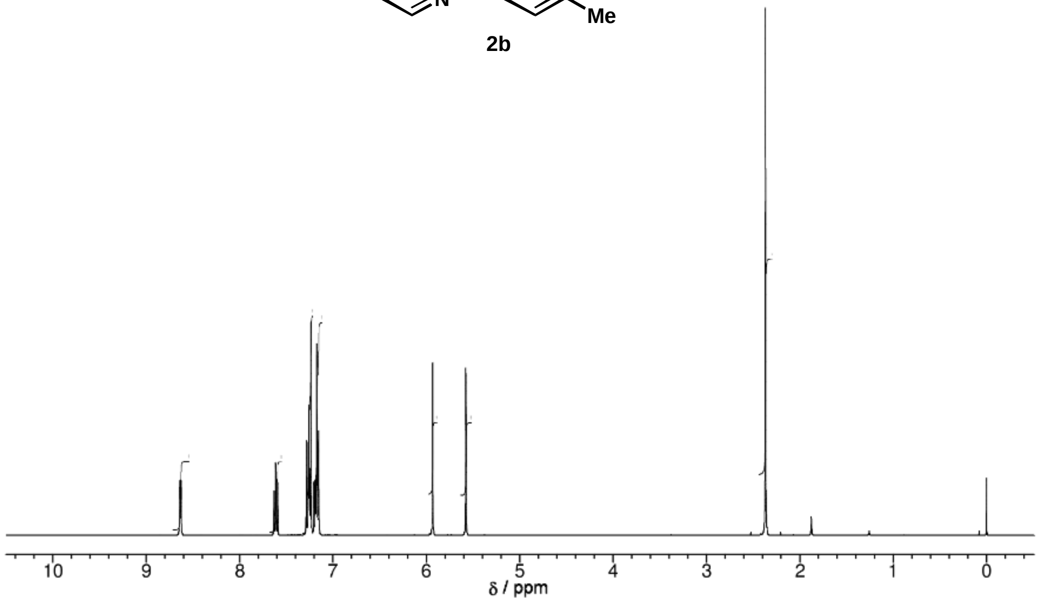

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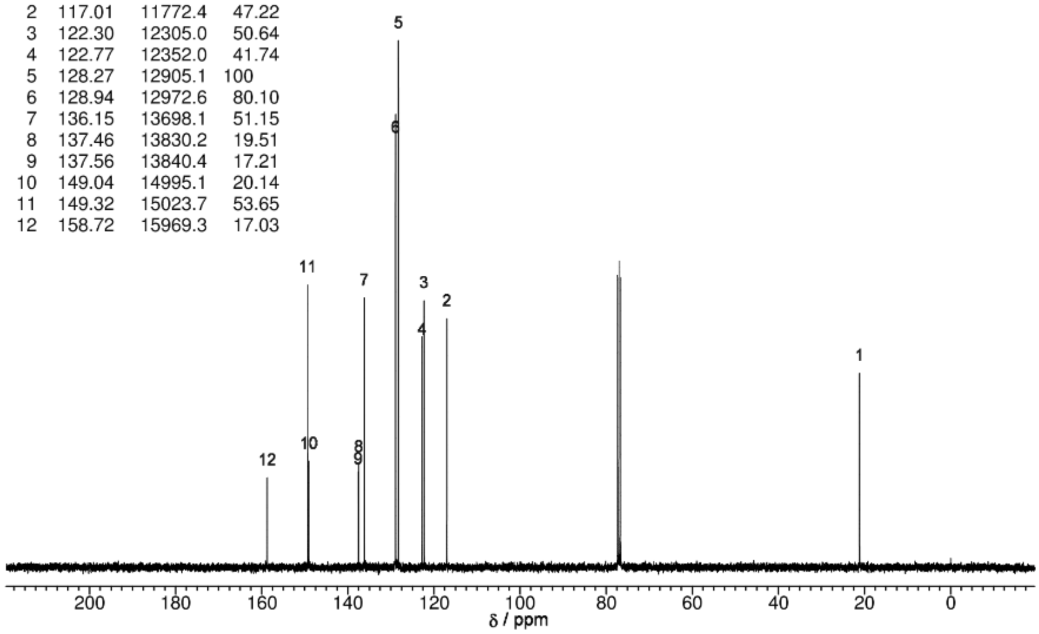
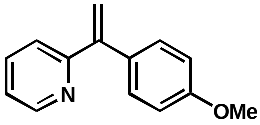
6	128.94	12972.6	80.10
7	136.15	13698.1	51.15
8	137.46	13830.2	19.51
9	137.56	13840.4	17.21
10	149.04	14995.1	20.14
11	149.32	15023.7	53.65
12	158.72	15969.3	17.03





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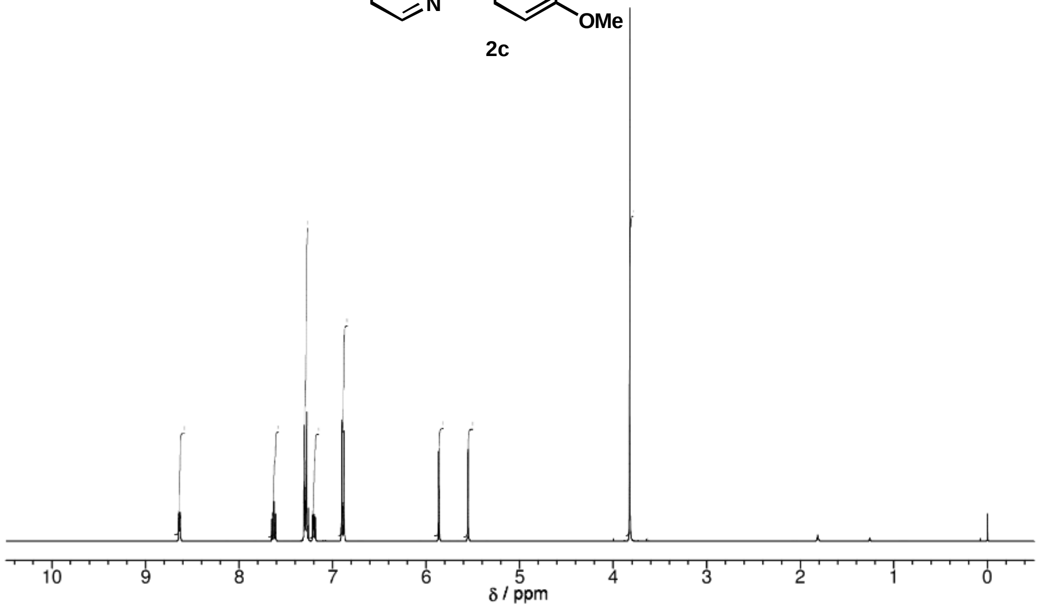

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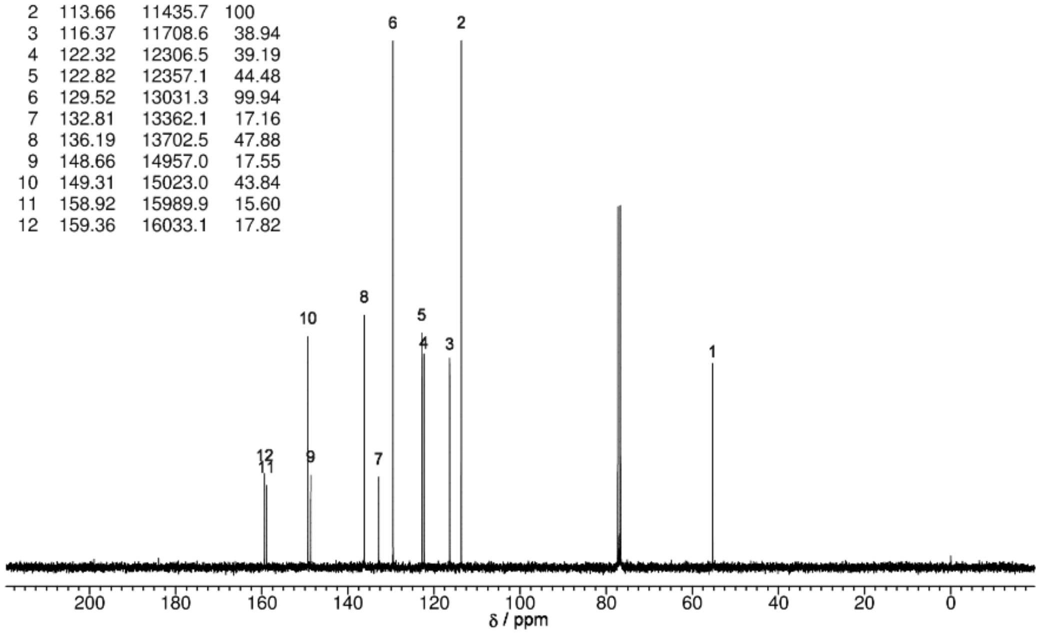
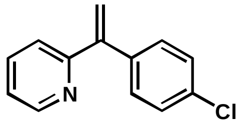
6	129.52	13031.3	99.94
7	132.81	13362.1	17.16
8	136.19	13702.5	47.88
9	148.66	14957.0	17.55
10	149.31	15023.0	43.84
11	158.92	15989.9	15.60
12	159.36	16033.1	17.82





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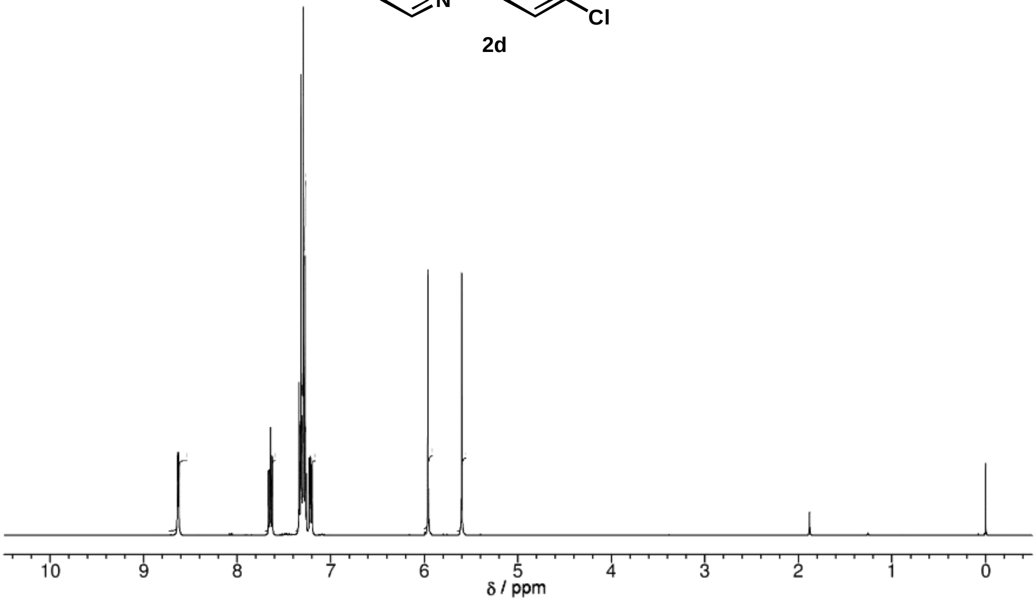

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5	129.69	13048.1	96.05
6	133.70	13451.6	14.41
7	136.35	13718.7	37.86
8	138.74	13958.5	16.20
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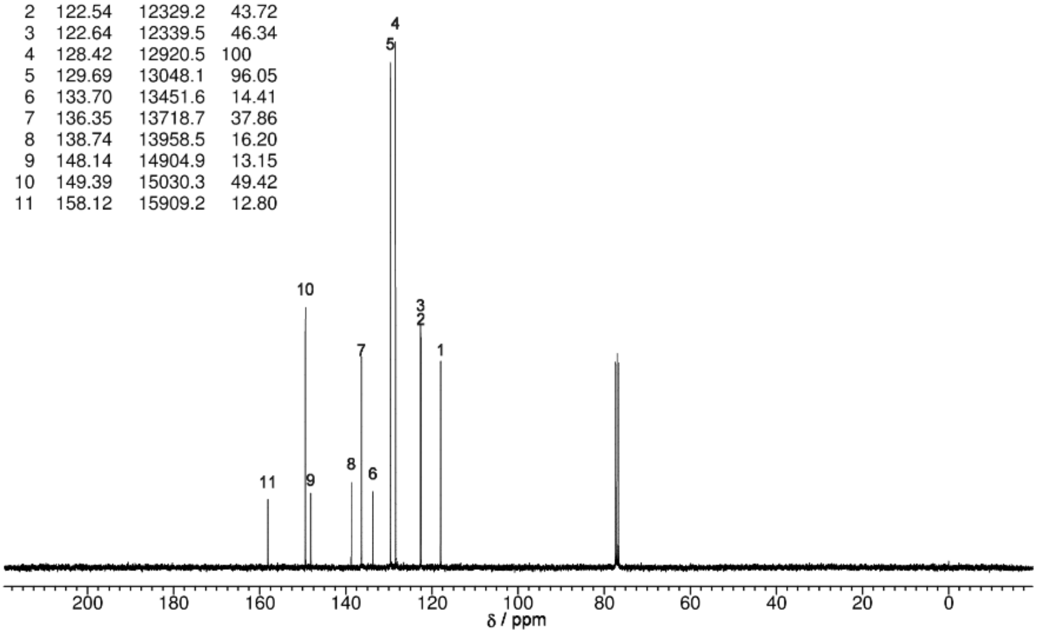

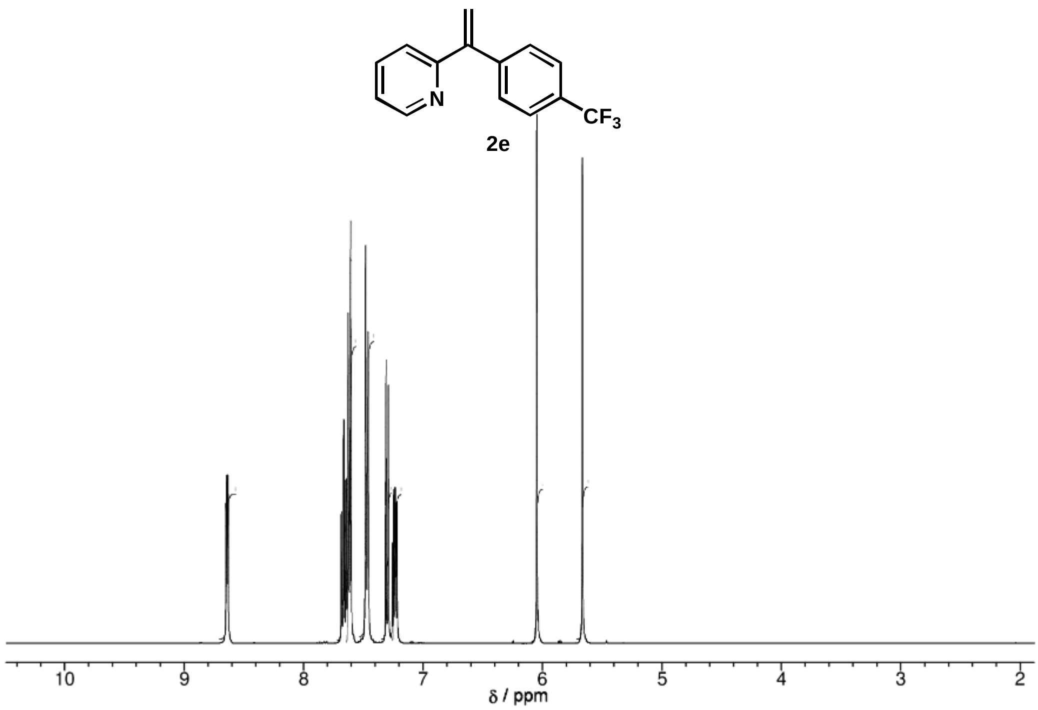

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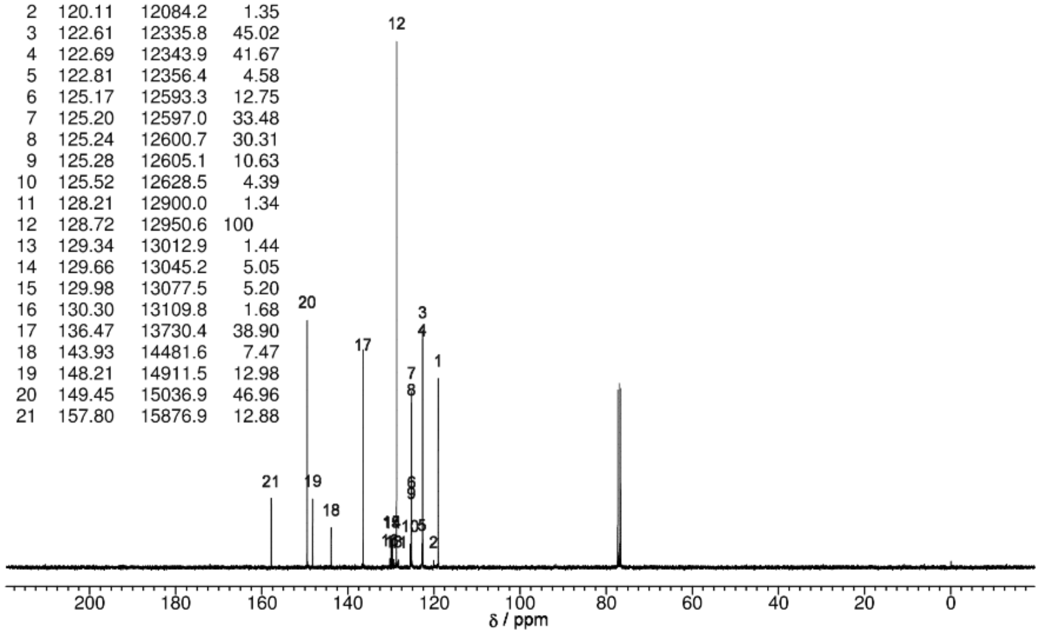
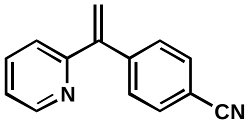
5	122.81	12356.4	4.58
6	125.17	12593.3	12.75
7	125.20	12597.0	33.48
8	125.24	12600.7	30.31
9	125.28	12605.1	10.63
10	125.52	12628.5	4.39
11	128.21	12900.0	1.34
12	128.72	12950.6	100
13	129.34	13012.9	1.44
14	129.66	13045.2	5.05
15	129.98	13077.5	5.20
16	130.30	13109.8	1.68
17	136.47	13730.4	38.90
18	143.93	14481.6	7.47
19	148.21	14911.5	12.98
20	149.45	15036.9	46.96
21	157.80	15876.9	12.88





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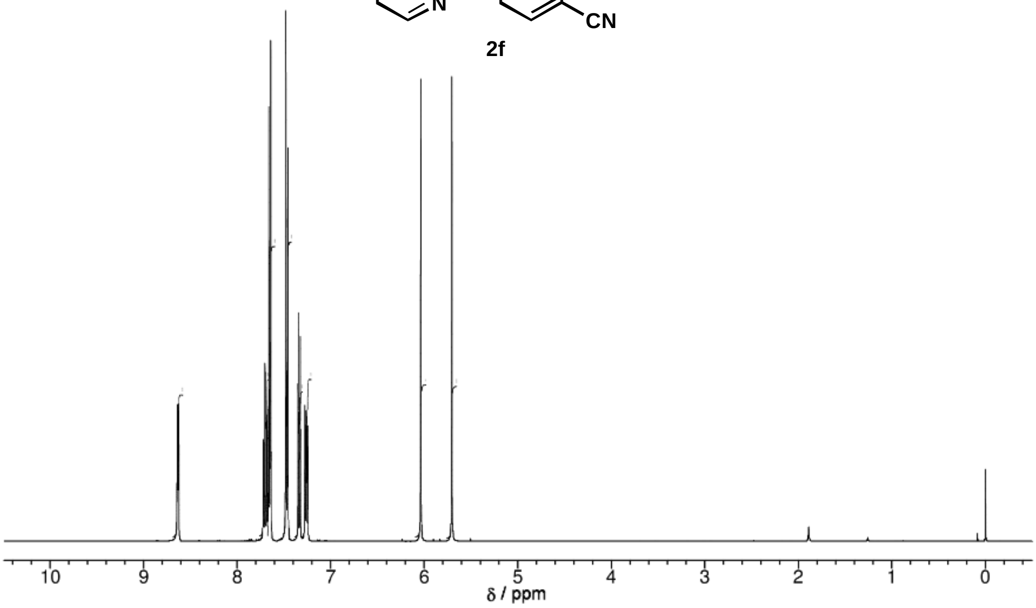

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6	129.00	12979.2	97.11
7	132.02	13282.9	100
8	136.58	13741.4	44.04
9	144.85	14574.0	14.37
10	147.91	14881.4	18.74
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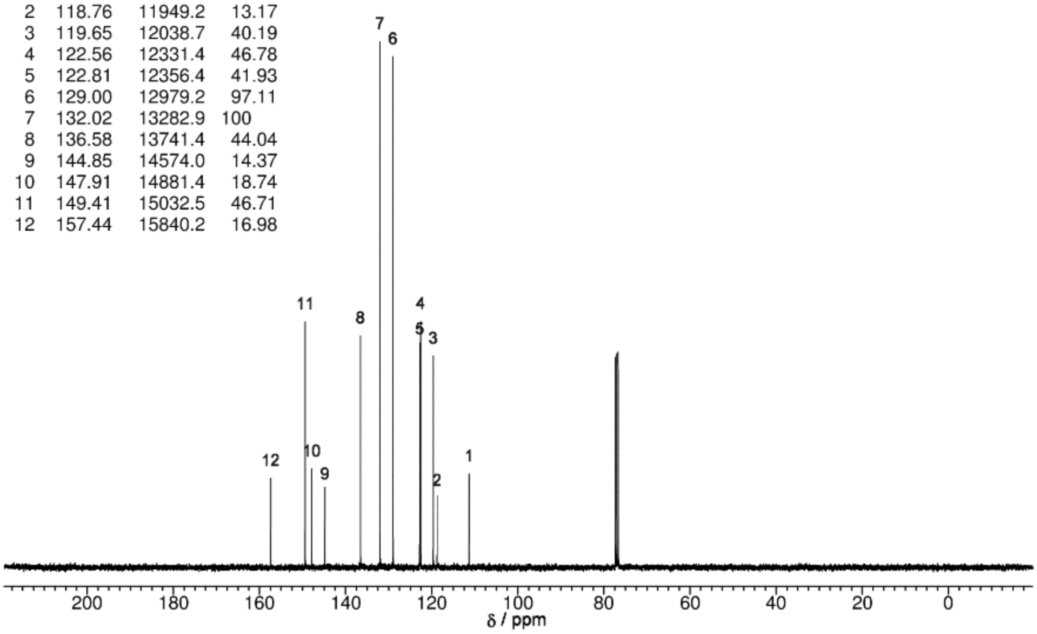
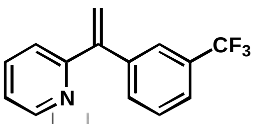
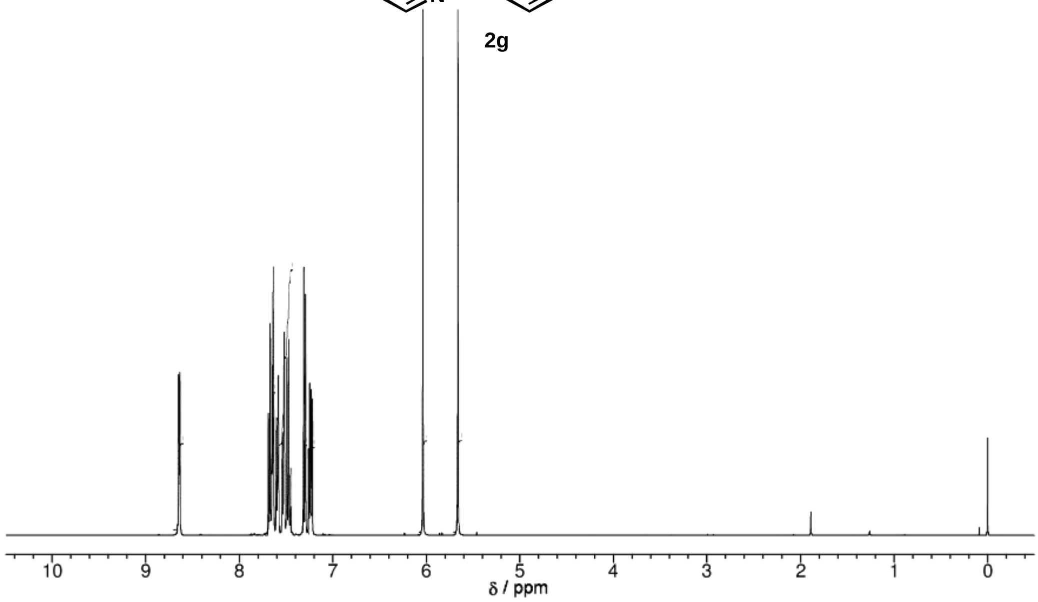
12	157.44	15840.2	16.98

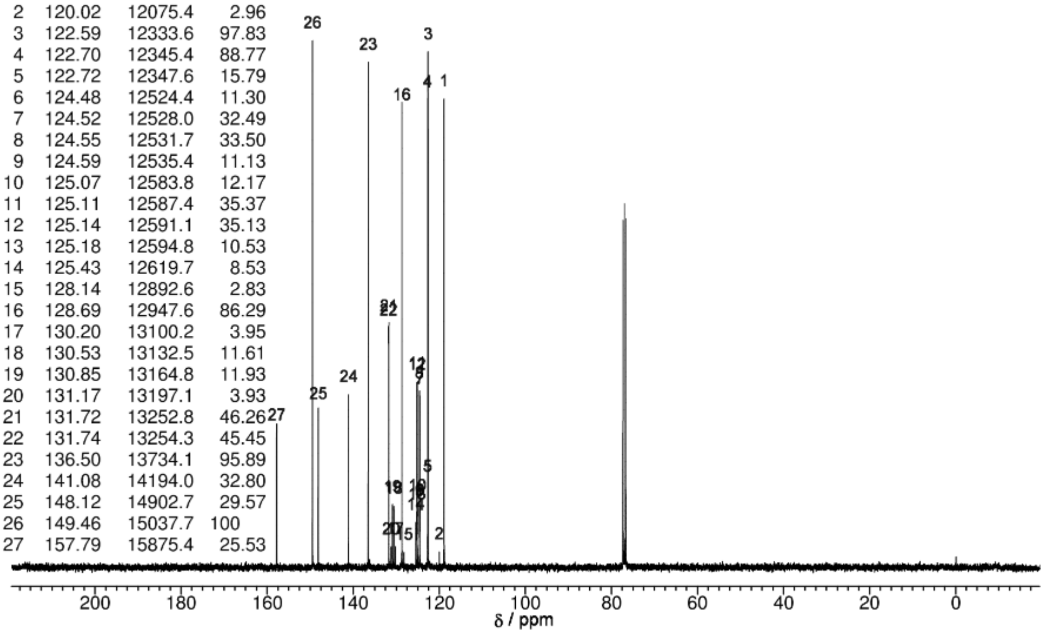


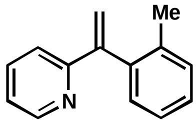


2g

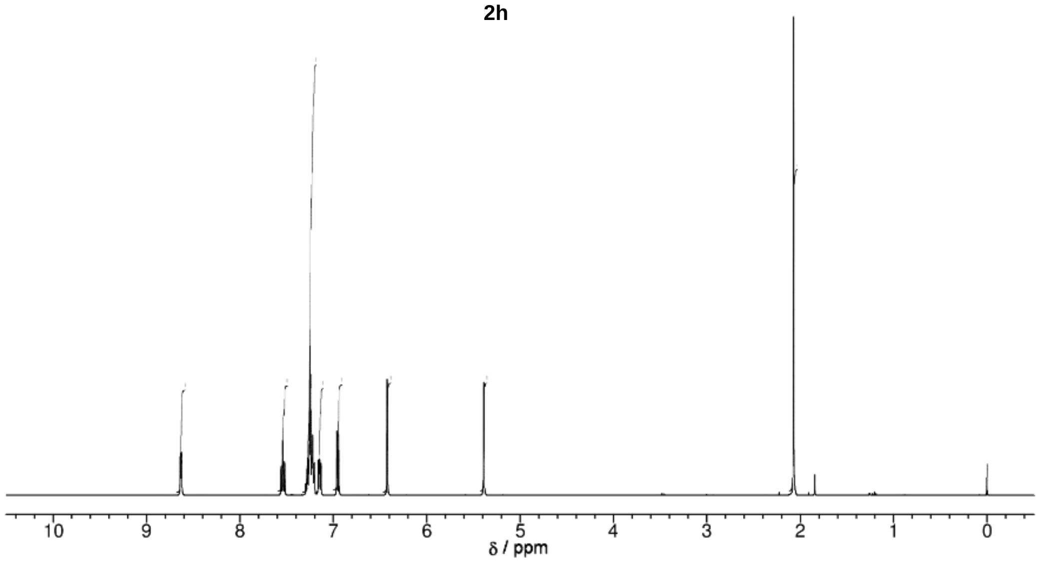


No.	ppm	Hz	Height
1	118.87	11960.2	88.94
2	120.02	12075.4	2.96
3	122.59	12333.6	97.83
4	122.70	12345.4	88.77
5	122.72	12347.6	15.79
6	124.48	12524.4	11.30
7	124.52	12528.0	32.49
8	124.55	12531.7	33.50
9	124.59	12535.4	11.13
10	125.07	12583.8	12.17
11	125.11	12587.4	35.37
12	125.14	12591.1	35.13
13	125.18	12594.8	10.53
14	125.43	12619.7	8.53
15	128.14	12892.6	2.83
16	128.69	12947.6	86.29
17	130.20	13100.2	3.95
18	130.53	13132.5	11.61
19	130.85	13164.8	11.93
20	131.17	13197.1	3.93
21	131.72	13252.8	46.26
22	131.74	13254.3	45.45
23	136.50	13734.1	95.89
24	141.08	14194.0	32.80
25	148.12	14902.7	29.57
26	149.46	15037.7	100
27	157.79	15875.4	25.53

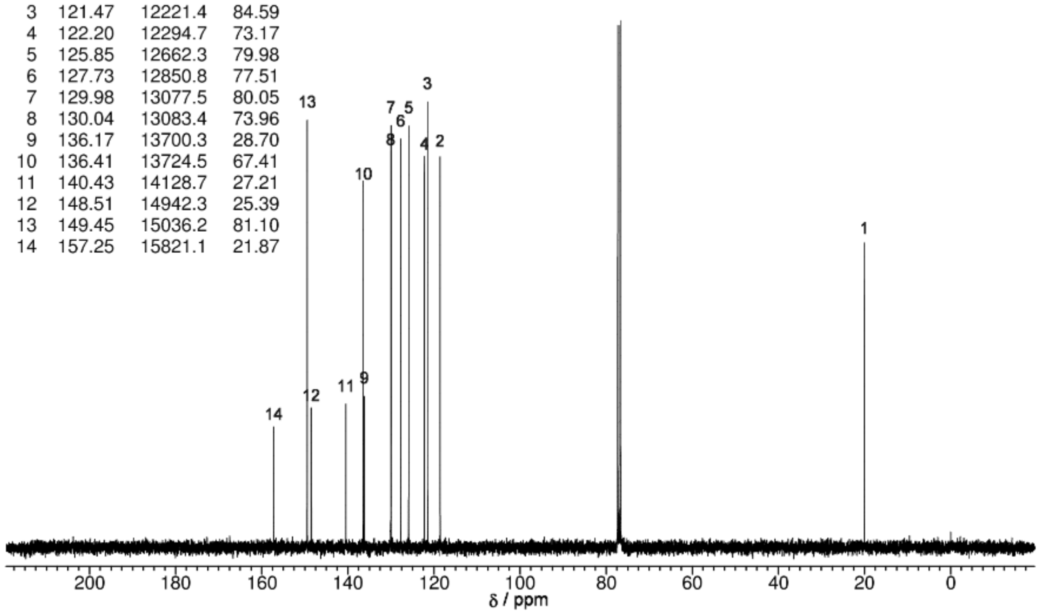


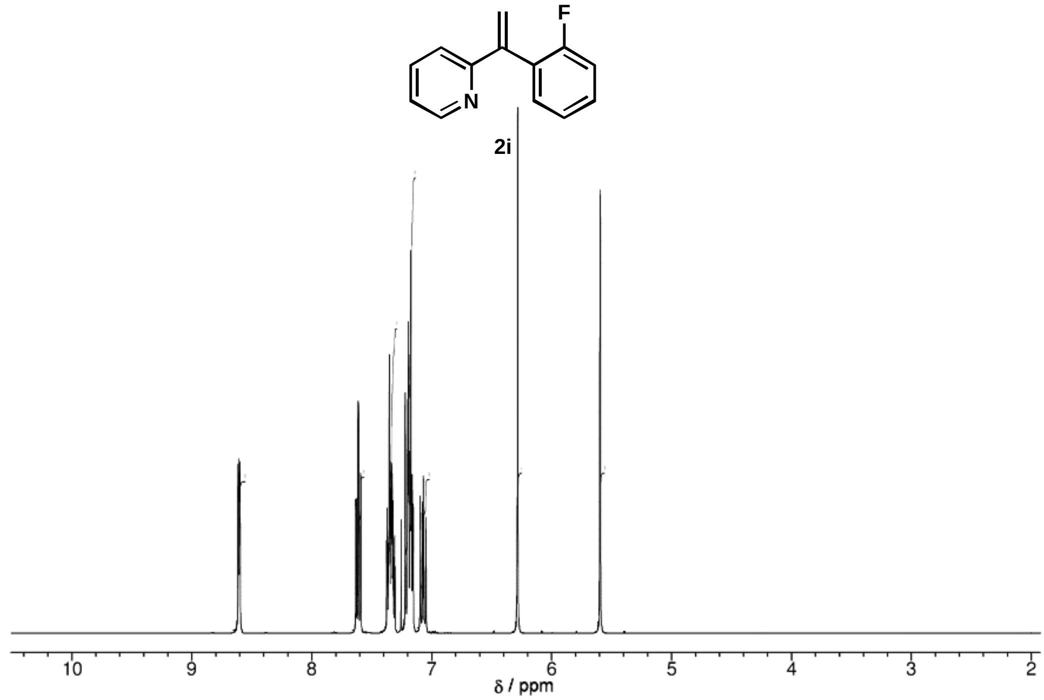


2h



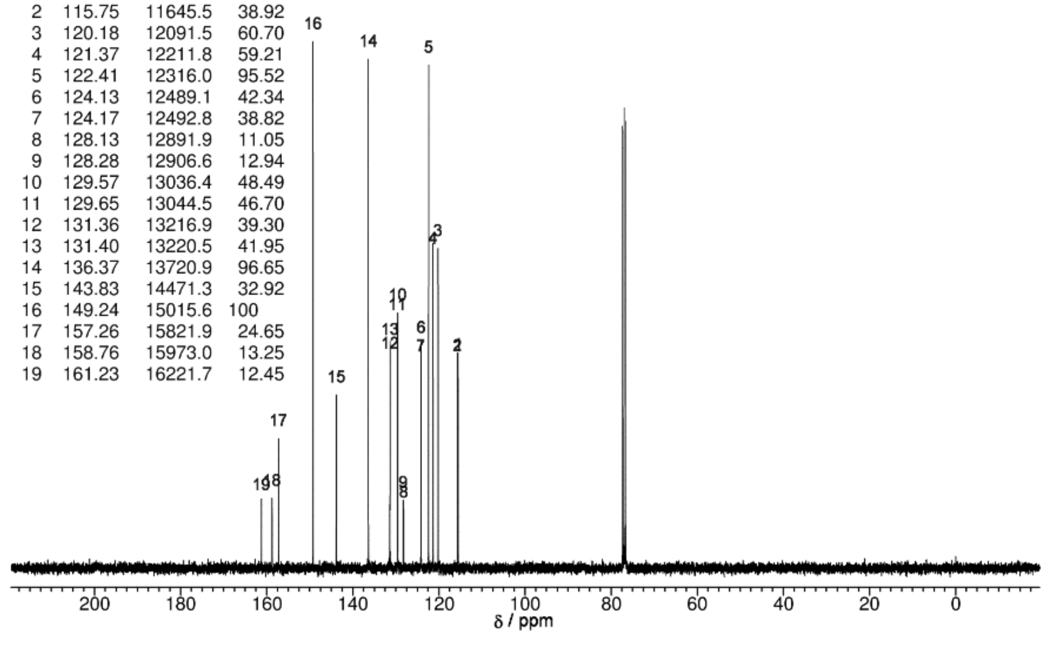
No.	ppm	Hz	Height
1	20.02	2014.1	57.12
2	118.55	11927.9	73.72
3	121.47	12221.4	84.59
4	122.20	12294.7	73.17
5	125.85	12662.3	79.98
6	127.73	12850.8	77.51
7	129.98	13077.5	80.05
8	130.04	13083.4	73.96
9	136.17	13700.3	28.70
10	136.41	13724.5	67.41
11	140.43	14128.7	27.21
12	148.51	14942.3	25.39
13	149.45	15036.2	81.10
14	157.25	15821.1	21.87

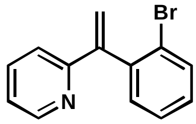




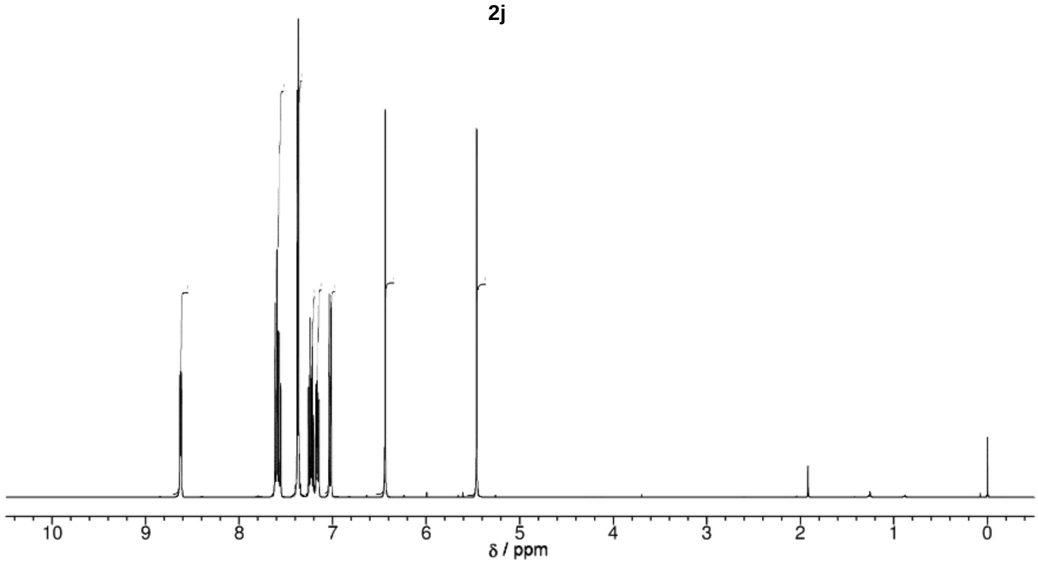
2i

No.	ppm	Hz	Height
1	115.53	11623.5	39.06
2	115.75	11645.5	38.92
3	120.18	12091.5	60.70
4	121.37	12211.8	59.21
5	122.41	12316.0	95.52
6	124.13	12489.1	42.34
7	124.17	12492.8	38.82
8	128.13	12891.9	11.05
9	128.28	12906.6	12.94
10	129.57	13036.4	48.49
11	129.65	13044.5	46.70
12	131.36	13216.9	39.30
13	131.40	13220.5	41.95
14	136.37	13720.9	96.65
15	143.83	14471.3	32.92
16	149.24	15015.6	100
17	157.26	15821.9	24.65
18	158.76	15973.0	13.25
19	161.23	16221.7	12.45

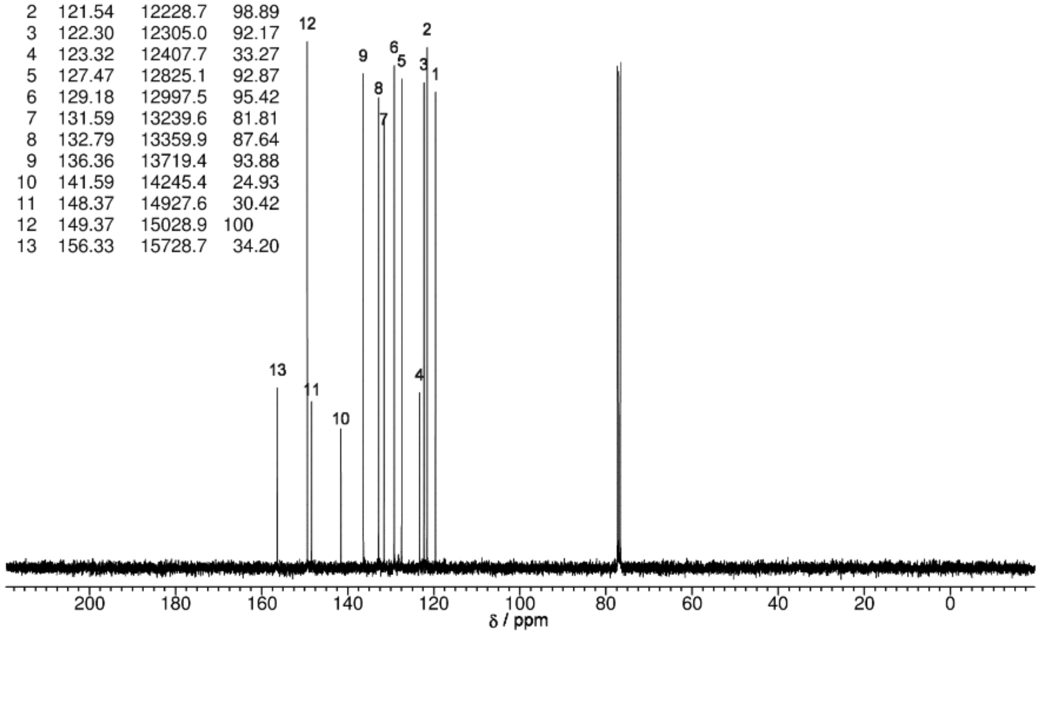


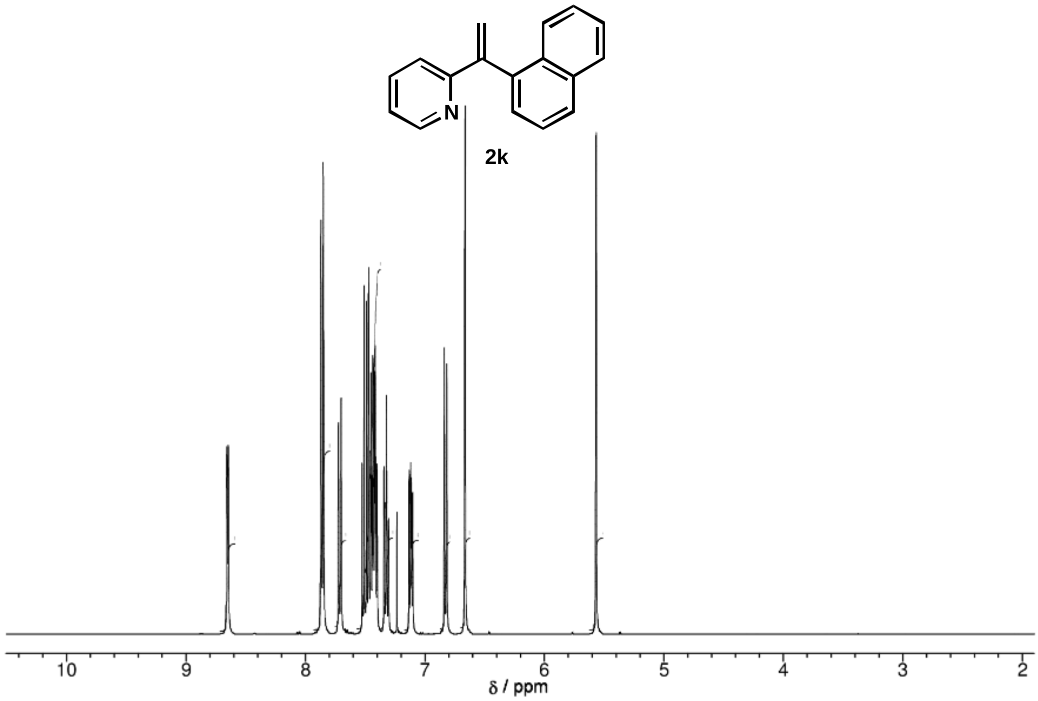


2j

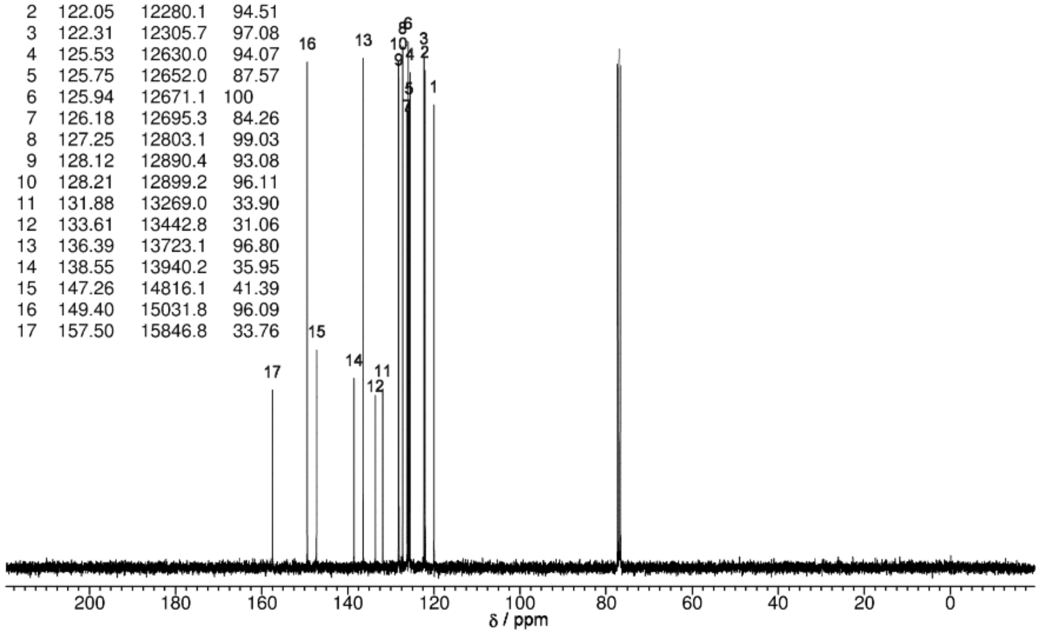


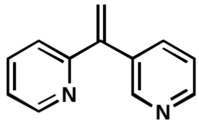
No.	ppm	Hz	Height
1	119.61	12034.3	90.36
2	121.54	12228.7	98.89
3	122.30	12305.0	92.17
4	123.32	12407.7	33.27
5	127.47	12825.1	92.87
6	129.18	12997.5	95.42
7	131.59	13239.6	81.81
8	132.79	13359.9	87.64
9	136.36	13719.4	93.88
10	141.59	14245.4	24.93
11	148.37	14927.6	30.42
12	149.37	15028.9	100
13	156.33	15728.7	34.20



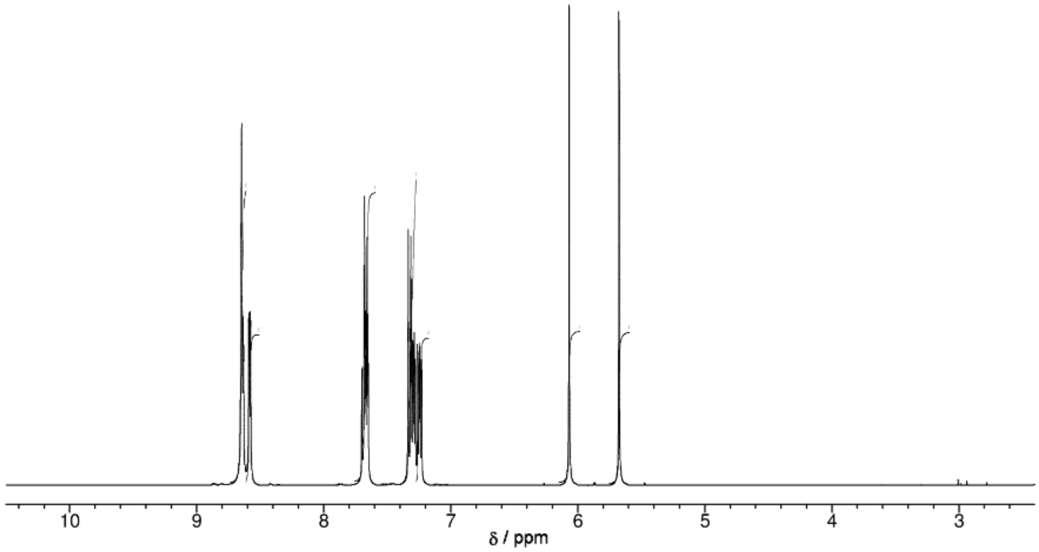


No.	ppm	Hz	Height
1	119.97	12071.0	87.95
2	122.05	12280.1	94.51
3	122.31	12305.7	97.08
4	125.53	12630.0	94.07
5	125.75	12652.0	87.57
6	125.94	12671.1	100
7	126.18	12695.3	84.26
8	127.25	12803.1	99.03
9	128.12	12890.4	93.08
10	128.21	12899.2	96.11
11	131.88	13269.0	33.90
12	133.61	13442.8	31.06
13	136.39	13723.1	96.80
14	138.55	13940.2	35.95
15	147.26	14816.1	41.39
16	149.40	15031.8	96.09
17	157.50	15846.8	33.76

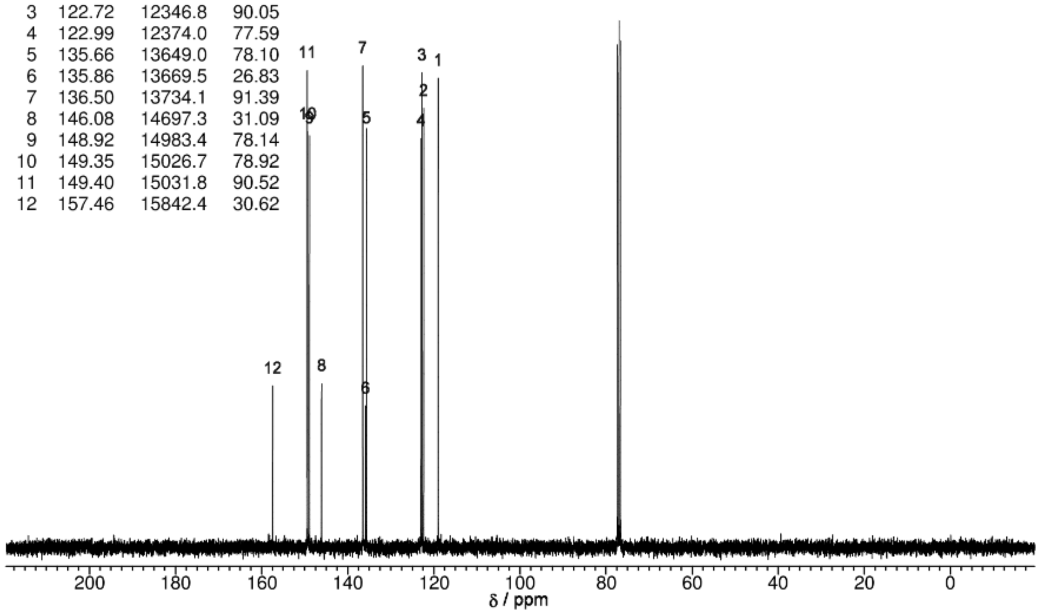


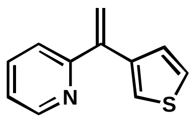


2l

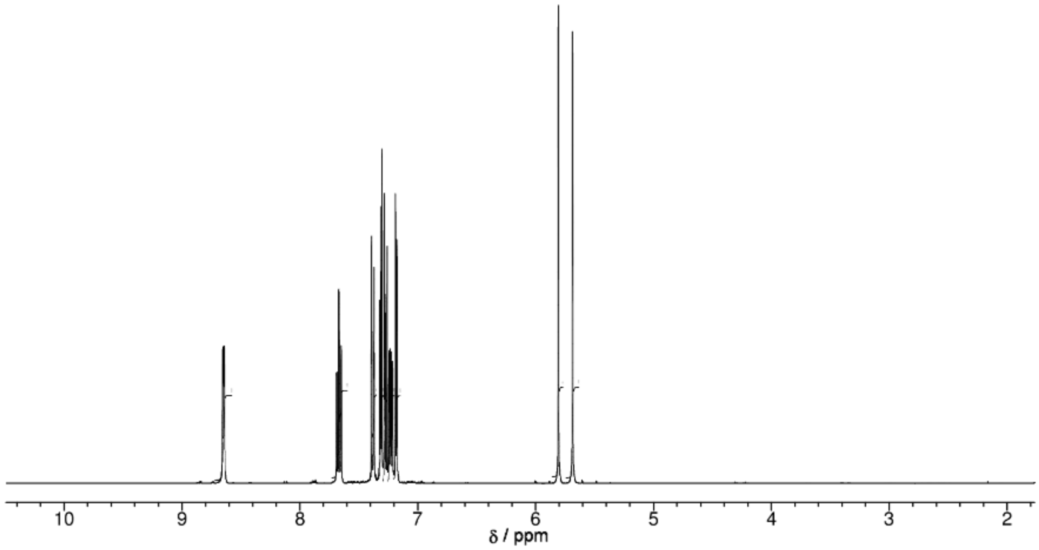


No.	ppm	Hz	Height
1	119.03	11975.6	89.06
2	122.38	12313.1	83.31
3	122.72	12346.8	90.05
4	122.99	12374.0	77.59
5	135.66	13649.0	78.10
6	135.86	13669.5	26.83
7	136.50	13734.1	91.39
8	146.08	14697.3	31.09
9	148.92	14983.4	78.14
10	149.35	15026.7	78.92
11	149.40	15031.8	90.52
12	157.46	15842.4	30.62

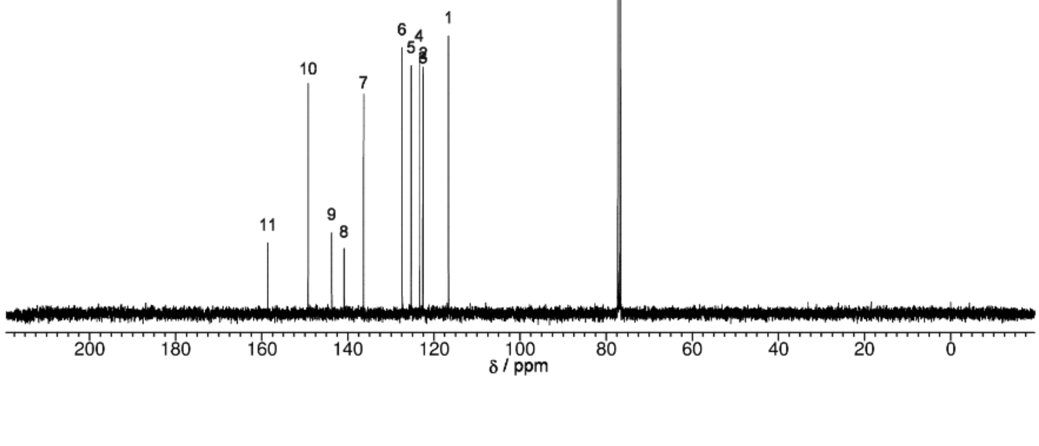


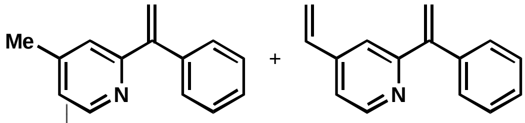


2m

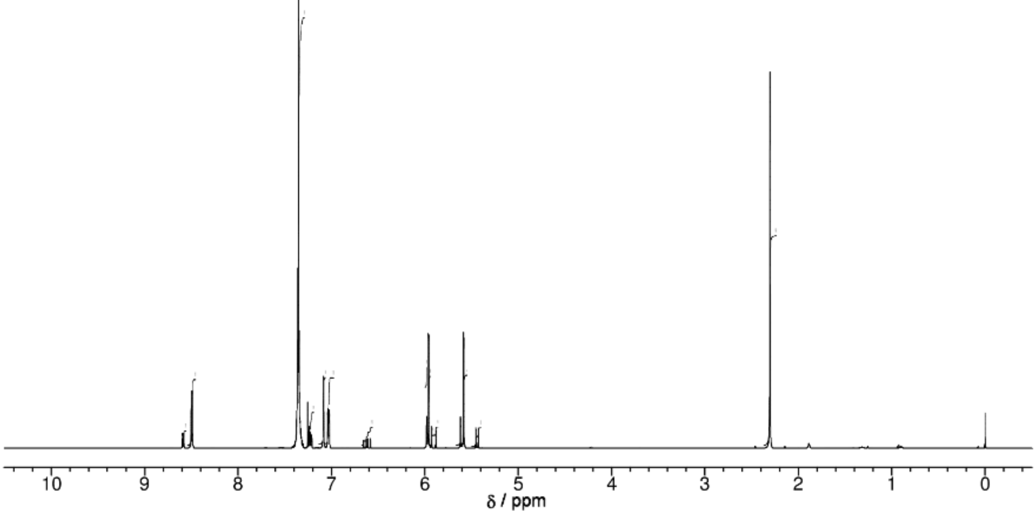


No.	ppm	Hz	Height
1	116.61	11732.8	52.84
2	122.51	12326.3	46.08
3	122.58	12332.9	45.20
4	123.43	12418.7	49.37
5	125.35	12611.7	47.15
6	127.41	12819.3	50.60
7	136.37	13720.9	40.47
8	140.89	14174.9	12.10
9	143.73	14461.1	15.39
10	149.25	15016.4	43.10
11	158.60	15957.6	13.48

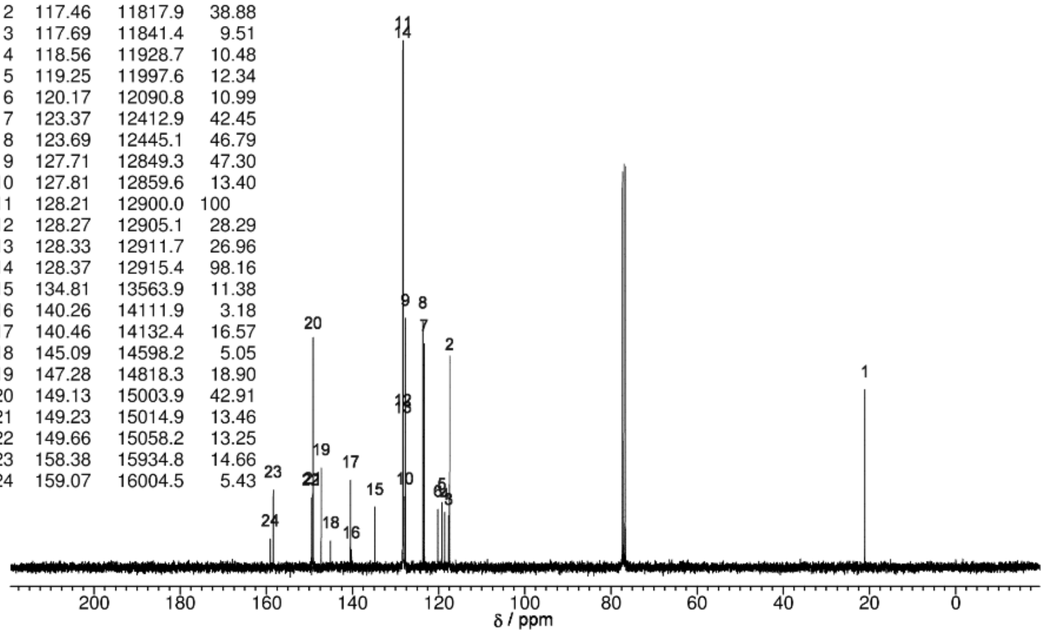


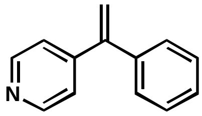


2n+2n'

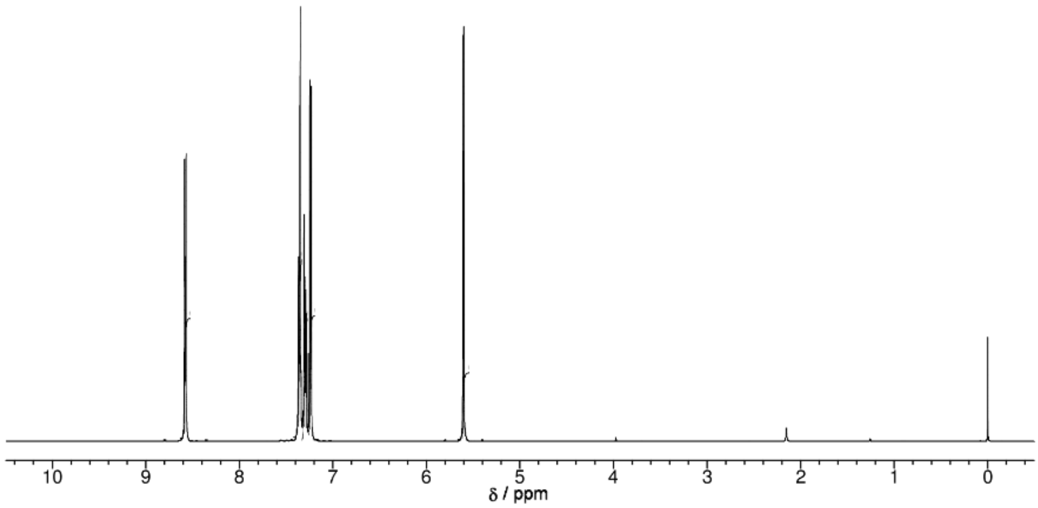


No.	ppm	Hz	Height
1	21.01	2113.9	33.75
2	117.46	11817.9	38.88
3	117.69	11841.4	9.51
4	118.56	11928.7	10.48
5	119.25	11997.6	12.34
6	120.17	12090.8	10.99
7	123.37	12412.9	42.45
8	123.69	12445.1	46.79
9	127.71	12849.3	47.30
10	127.81	12859.6	13.40
11	128.21	12900.0	100
12	128.27	12905.1	28.29
13	128.33	12911.7	26.96
14	128.37	12915.4	98.16
15	134.81	13563.9	11.38
16	140.26	14111.9	3.18
17	140.46	14132.4	16.57
18	145.09	14598.2	5.05
19	147.28	14818.3	18.90
20	149.13	15003.9	42.91
21	149.23	15014.9	13.46
22	149.66	15058.2	13.25
23	158.38	15934.8	14.66
24	159.07	16004.5	5.43

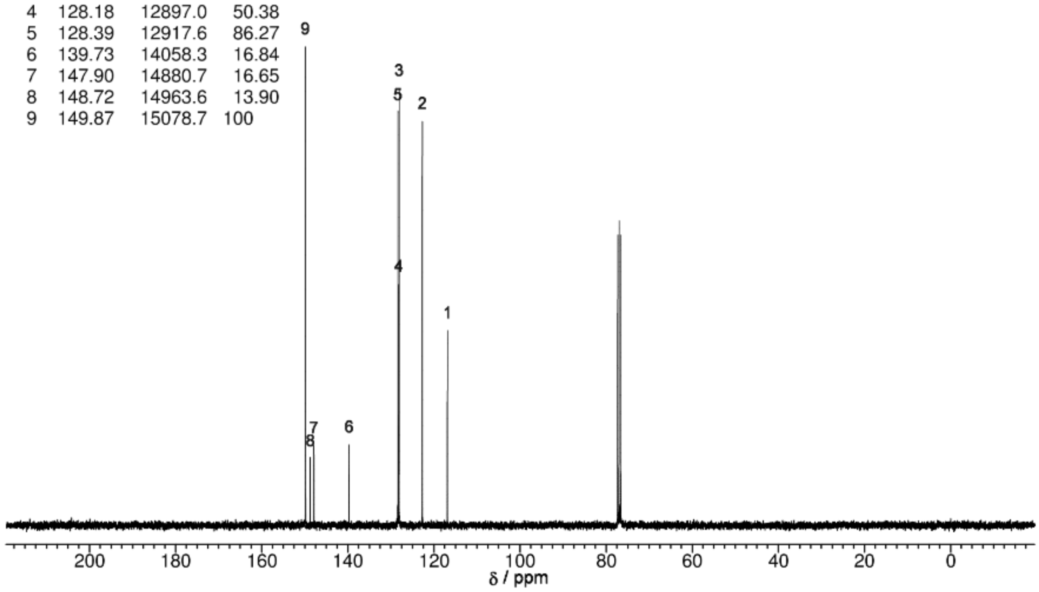


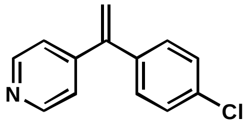


2o

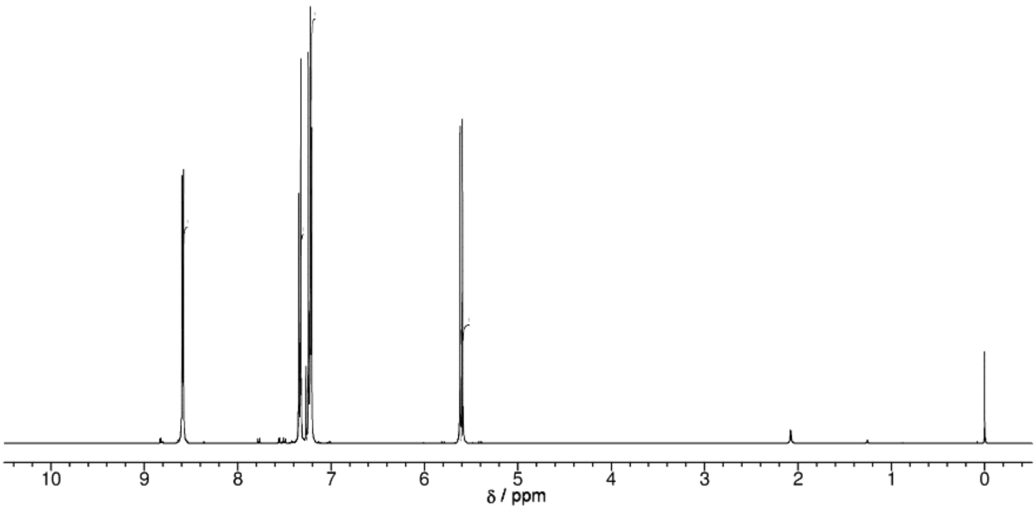


No.	ppm	Hz	Height
1	116.88	11759.2	40.71
2	122.72	12347.6	84.42
3	128.10	12888.2	91.30
4	128.18	12897.0	50.38
5	128.39	12917.6	86.27
6	139.73	14058.3	16.84
7	147.90	14880.7	16.65
8	148.72	14963.6	13.90
9	149.87	15078.7	100

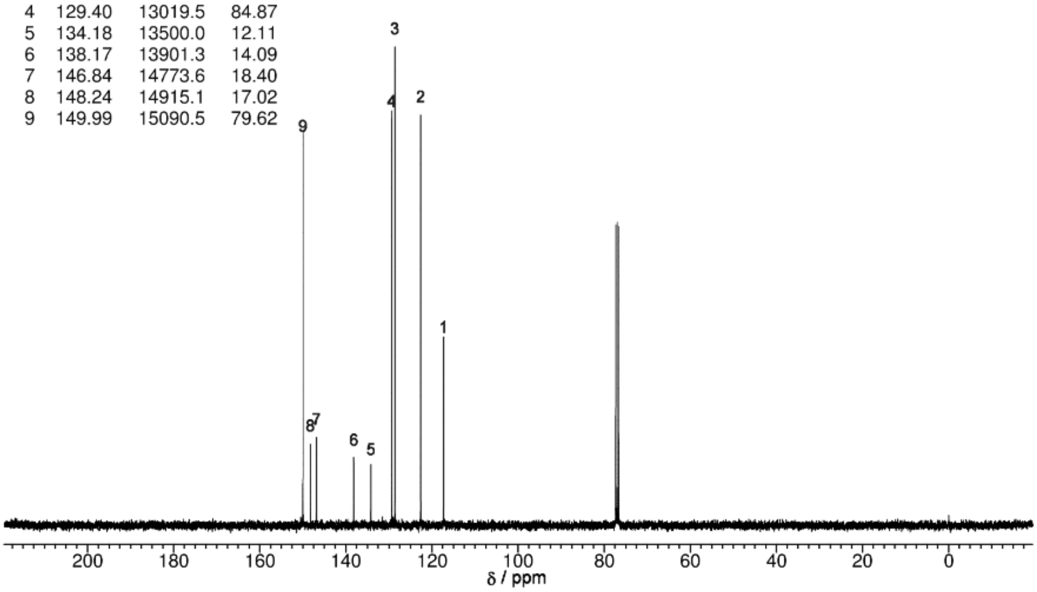


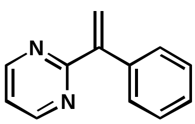


2p

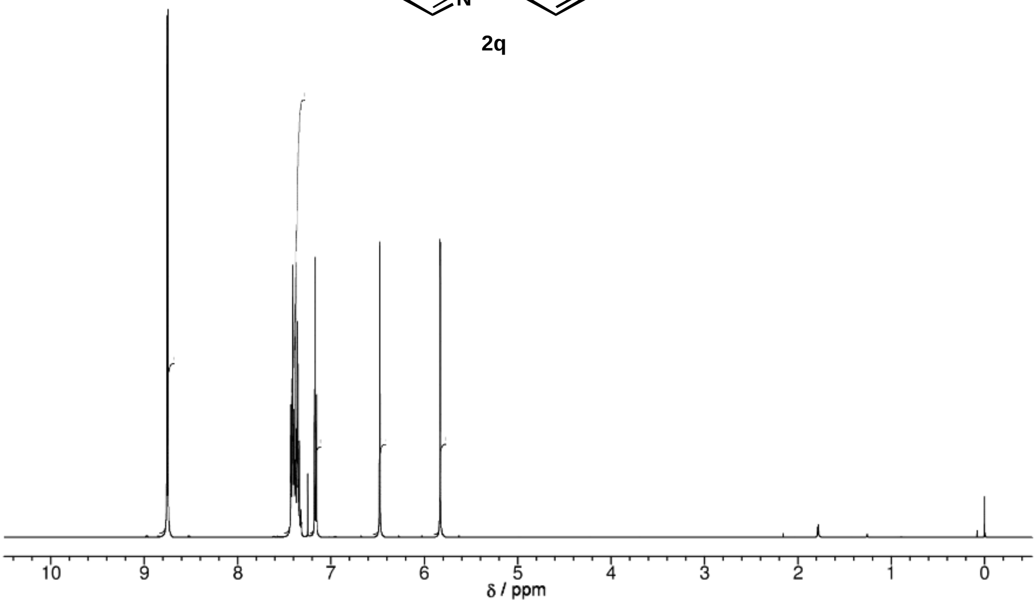


No.	ppm	Hz	Height
1	117.31	11802.5	37.69
2	122.61	12336.6	85.75
3	128.62	12941.0	100
4	129.40	13019.5	84.87
5	134.18	13500.0	12.11
6	138.17	13901.3	14.09
7	146.84	14773.6	18.40
8	148.24	14915.1	17.02
9	149.99	15090.5	79.62

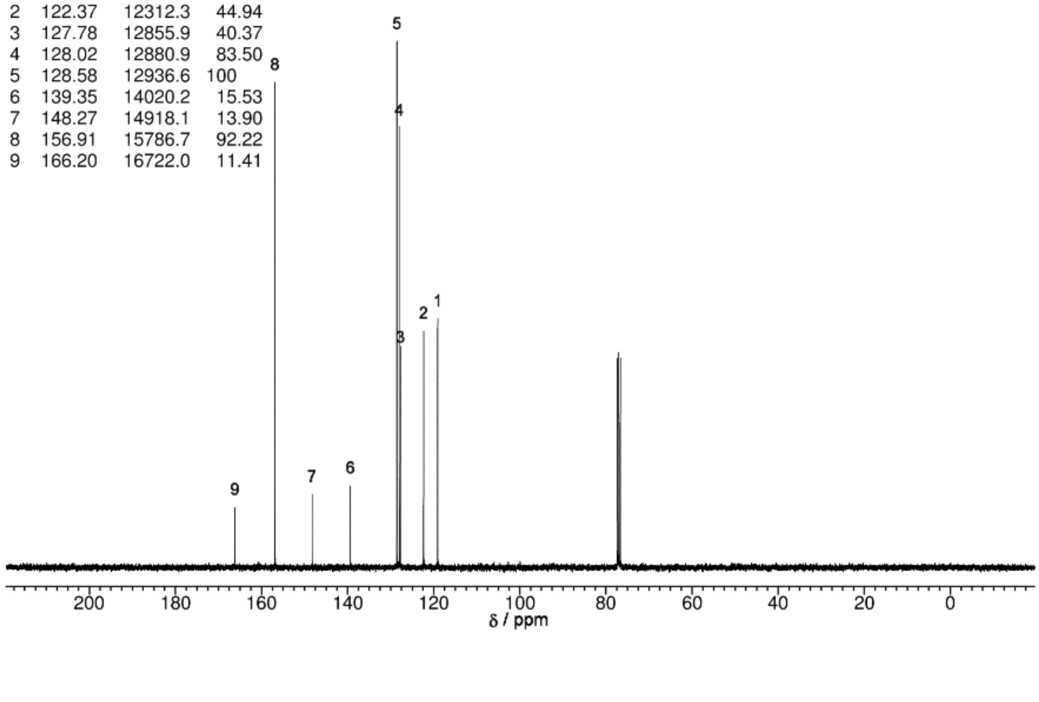


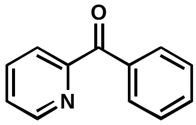


2q

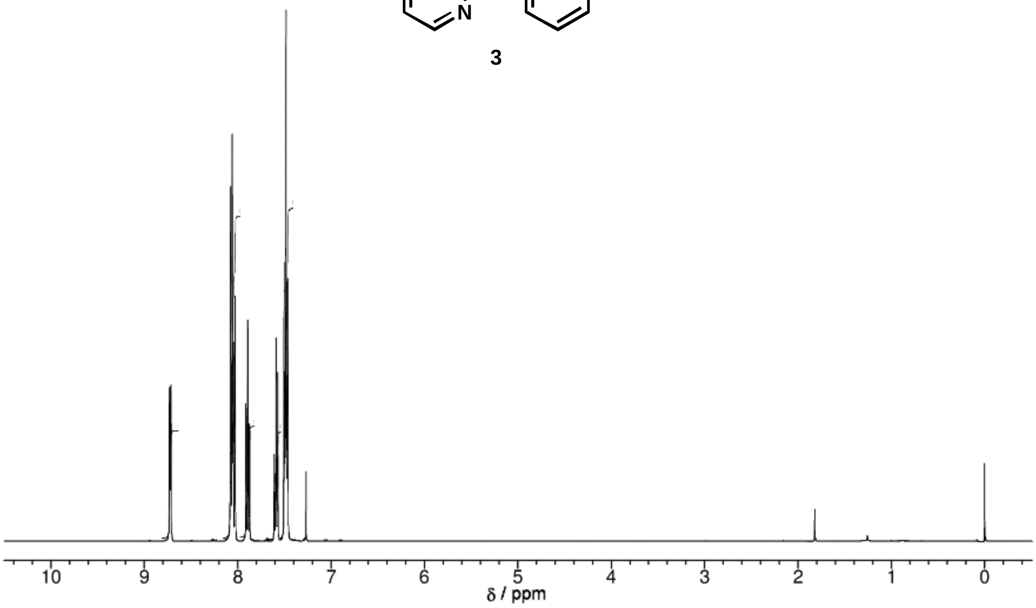


No.	ppm	Hz	Height
1	119.09	11982.2	47.30
2	122.37	12312.3	44.94
3	127.78	12855.9	40.37
4	128.02	12880.9	83.50
5	128.58	12936.6	100
6	139.35	14020.2	15.53
7	148.27	14918.1	13.90
8	156.91	15786.7	92.22
9	166.20	16722.0	11.41

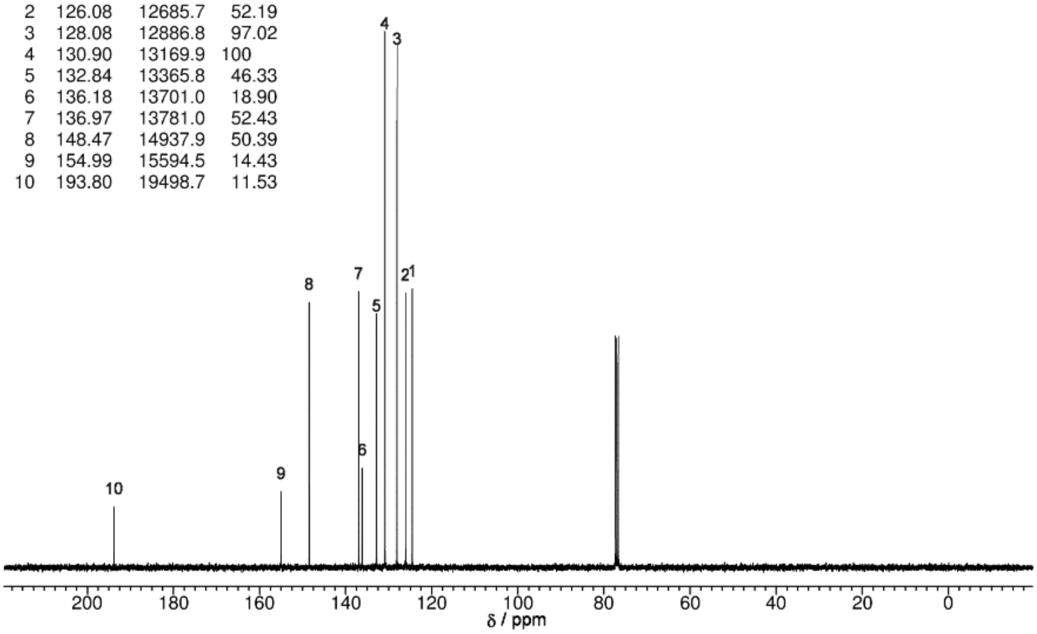


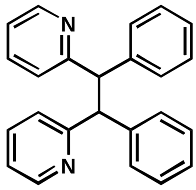


3

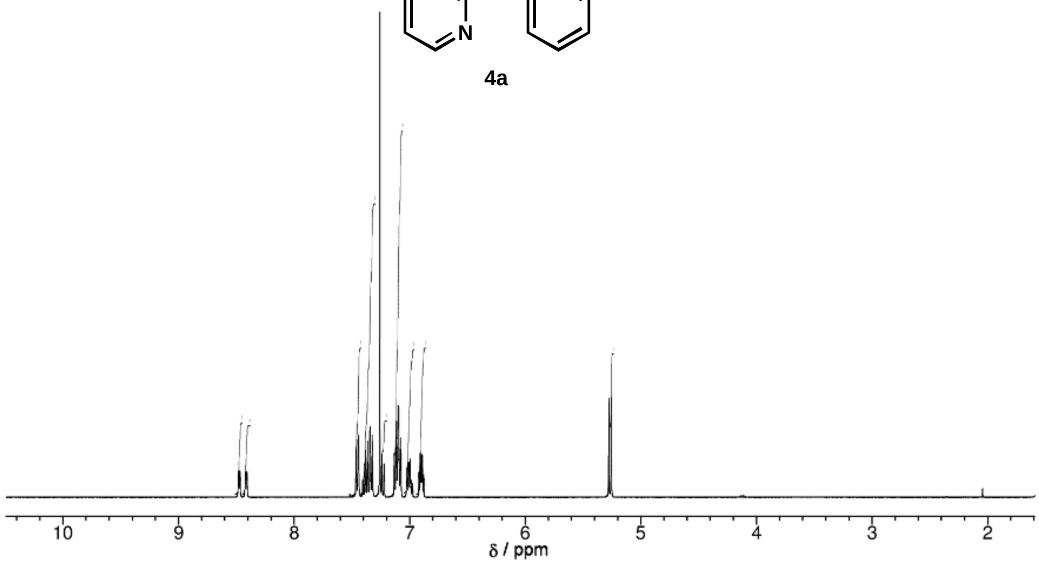


No.	ppm	Hz	Height
1	124.53	12529.5	52.93
2	126.08	12685.7	52.19
3	128.08	12886.8	97.02
4	130.90	13169.9	100
5	132.84	13365.8	46.33
6	136.18	13701.0	18.90
7	136.97	13781.0	52.43
8	148.47	14937.9	50.39
9	154.99	15594.5	14.43
10	193.80	19498.7	11.53

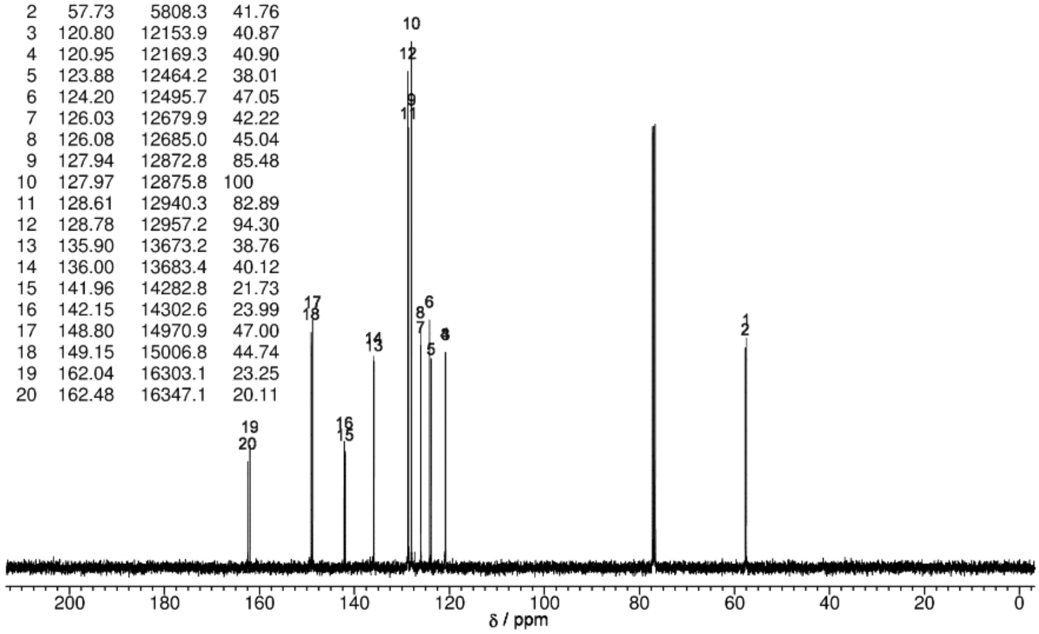


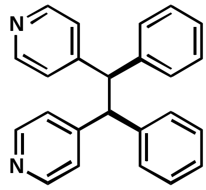


4a

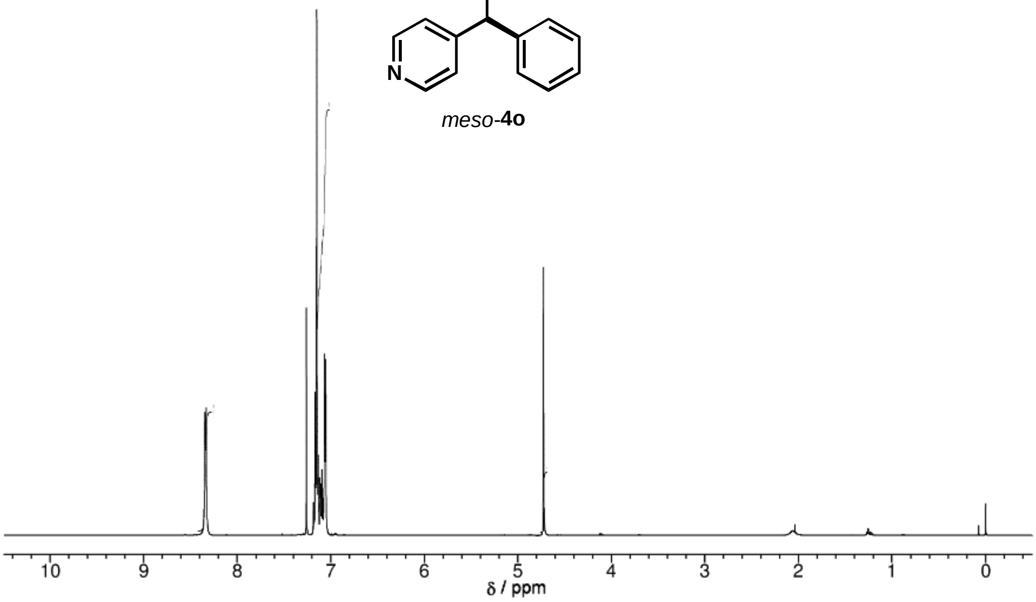


No.	ppm	Hz	Height
1	57.55	5790.0	43.61
2	57.73	5808.3	41.76
3	120.80	12153.9	40.87
4	120.95	12169.3	40.90
5	123.88	12464.2	38.01
6	124.20	12495.7	47.05
7	126.03	12679.9	42.22
8	126.08	12685.0	45.04
9	127.94	12872.8	85.48
10	127.97	12875.8	100
11	128.61	12940.3	82.89
12	128.78	12957.2	94.30
13	135.90	13673.2	38.76
14	136.00	13683.4	40.12
15	141.96	14282.8	21.73
16	142.15	14302.6	23.99
17	148.80	14970.9	47.00
18	149.15	15006.8	44.74
19	162.04	16303.1	23.25
20	162.48	16347.1	20.11

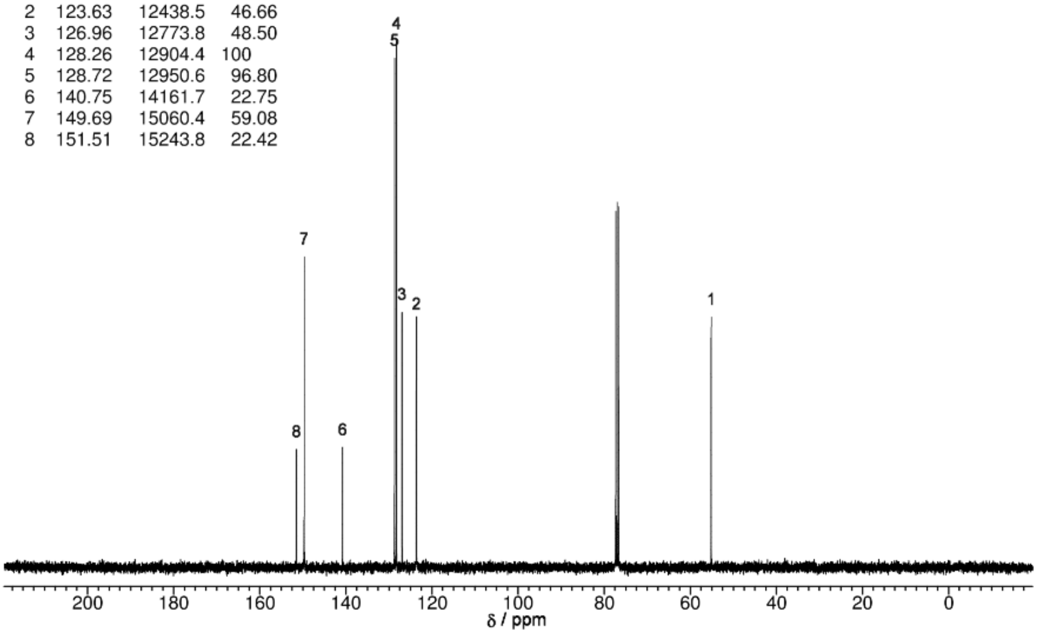


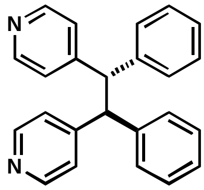


meso-**4o**

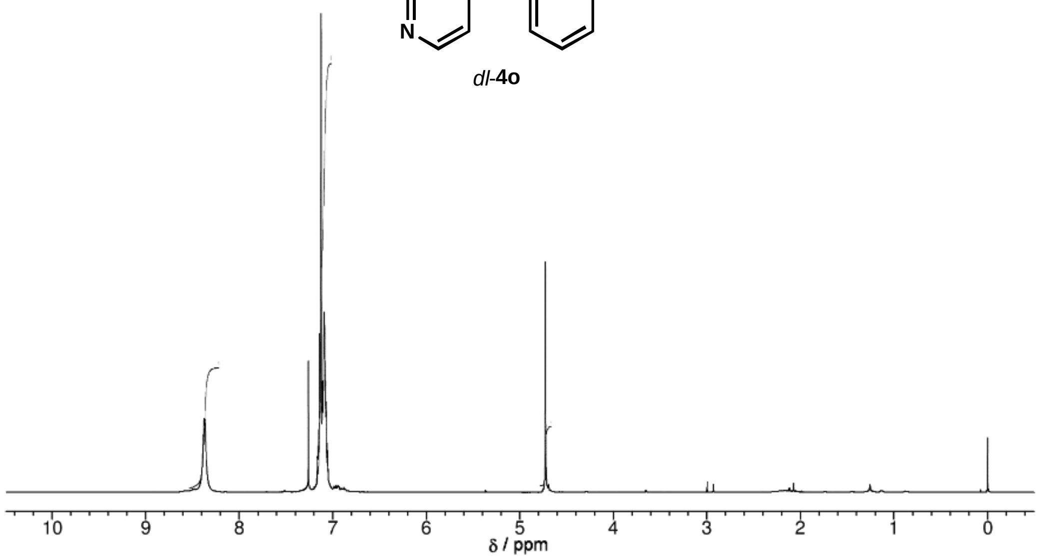


No.	ppm	Hz	Height
1	55.16	5550.1	47.59
2	123.63	12438.5	46.66
3	126.96	12773.8	48.50
4	128.26	12904.4	100
5	128.72	12950.6	96.80
6	140.75	14161.7	22.75
7	149.69	15060.4	59.08
8	151.51	15243.8	22.42

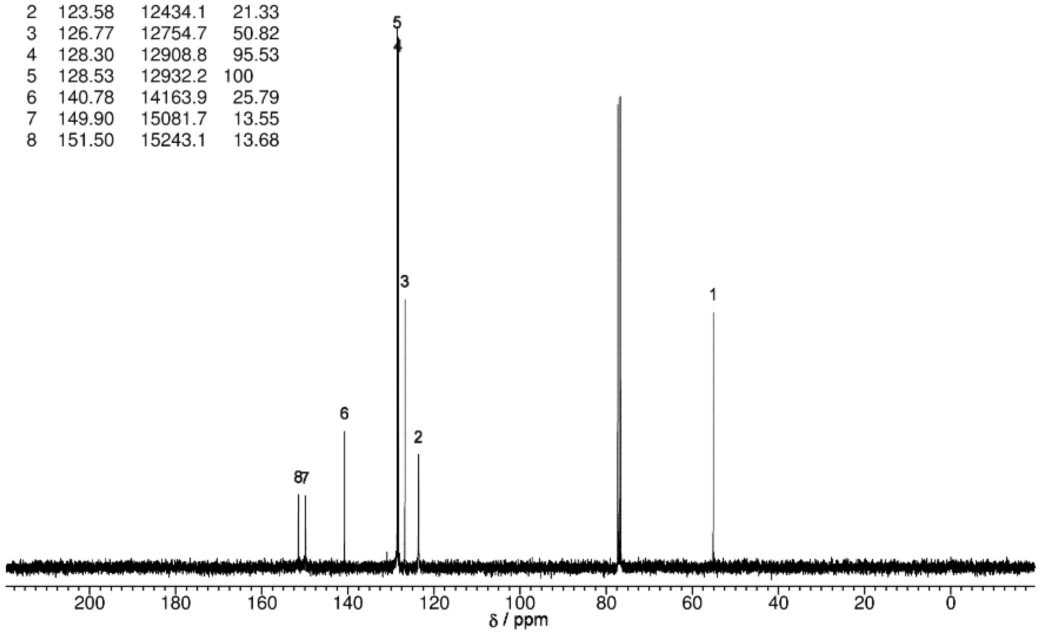


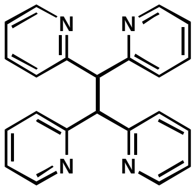


dl-**4o**

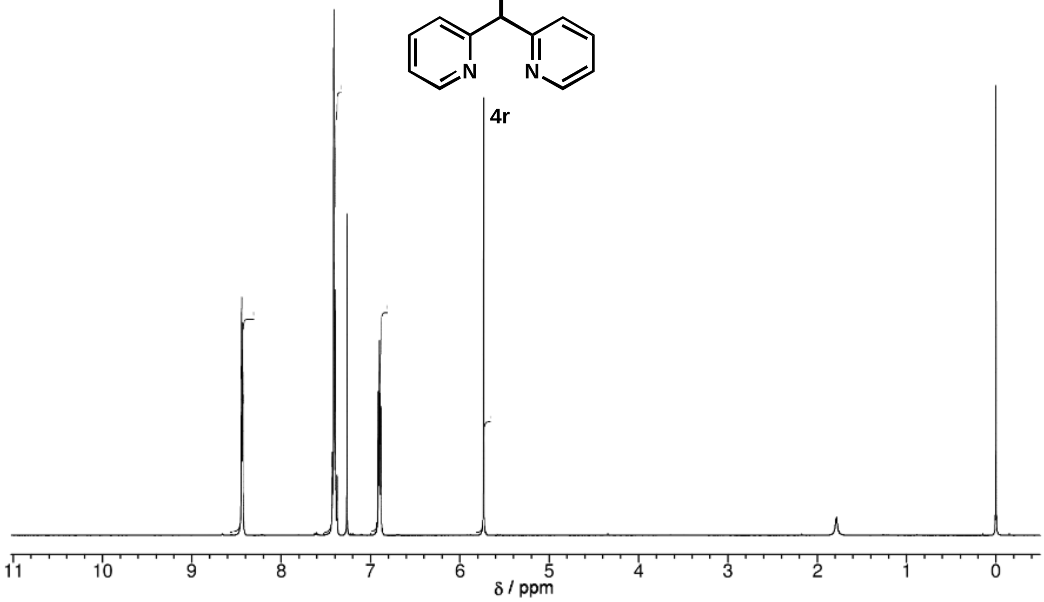


No.	ppm	Hz	Height
1	55.14	5547.9	48.37
2	123.58	12434.1	21.33
3	126.77	12754.7	50.82
4	128.30	12908.8	95.53
5	128.53	12932.2	100
6	140.78	14163.9	25.79
7	149.90	15081.7	13.55
8	151.50	15243.1	13.68





4r



No.	ppm	Hz	Height
1	58.82	5918.3	22.76
2	121.07	12181.0	45.94
3	124.14	12489.9	43.74
4	135.89	13672.4	44.26
5	149.09	15000.2	41.04
6	161.00	16198.2	21.74