Supplementary Information

Benzylic C-H Heteroarylation of *N*-(Benzyloxy)phthalimides with Cyanopyridines Enabled by Photoredox 1,2-Hydrogen Atom Transfer

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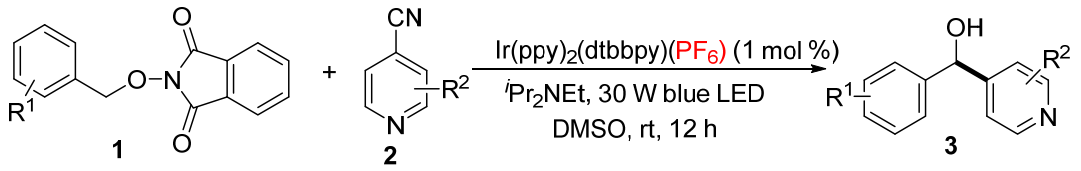
(A) General Experimental Procedures

(a) General information

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker 500 (500, 125, and 471 MHz) advance spectrometer at room temperature in $CDCl_3$ (solvent signals, δ 7.26 and 77.0 ppm) using TMS as internal standard. Low-resolution mass spectra (LRMS) data were measured on GCMS-QP2010 Ultra. High-resolution mass spectra (HRMS) was recorded on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry. Melting Points were recorded on Hanon MP100 Apparatus. Unless otherwise noted, all reactions were carried out using standard Schlenk techniques, and all starting materials and solvents were commercially available and were used without further purification. Column chromatography was performed on silica gel (300-400 mesh) using petroleum ether (PE)/ethyl acetate (EA). The light source were used 30W Blue LEDs (manufacturer: liang yuan ligbting, model: LY-PD001**,** wavelength range: 450-460 nm, λ_{max} = 455 nm), with wrap in foil, less than 5cm from the light source to the irradiation vessel.

Figure S1 The light reaction apparatus

(b) Typical Experimental Procedure for Synthesis of Diarylmethanols 3:



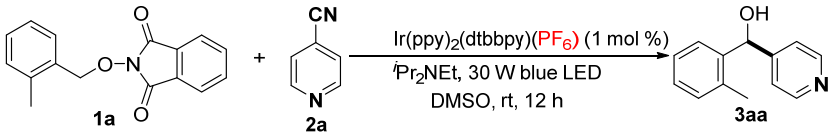
To a Schlenk tube were added *N*-alkoxyphthalimide **1** (0.2 mmol), 4-cyanopyridines **2** (0.4 mmol), *N*,*N*-diisopropylethylamine (0.44 mmol), $Ir(ppy)_2(dtbbpy)PF_6$ (1 mol %, 0.002 mmol), and DMSO (2 mL). Then the tube was charged with argon three times, and was stirred at room temperature under 30W blue LED light for 12 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was concentrated in vacuum, diluted in diethyl ether, and washed with brine. The aqueous phase was re-extracted with 15 mL diethyl ether three times. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.

(c) Experimental Procedure for the 1 mmol Scale.

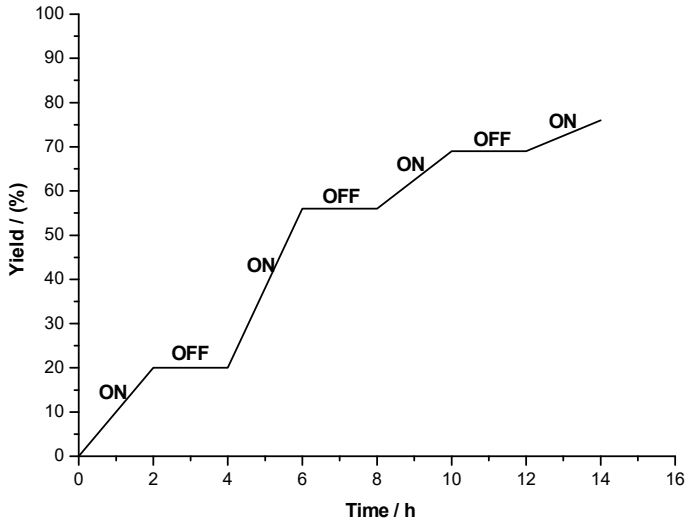
To a Schlenk tube were added *N*-alkoxyphthalimide **1** (1 mmol; 267.1 mg), 4-cyanopyridines **2** (2 mmol; 208.0 mg), *N*,*N*-diisopropylethylamine (2.2 mmol; 283.8 mg), $Ir(ppy)_2(dtbbpy)PF_6$ (1 mol %, 0.01 mmol; 9.14 mg), and DMSO (10 mL). Then the tube was charged with argon three times, and was stirred at room temperature under 30W blue LED light for 12 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was concentrated in vacuum, diluted in diethyl ether, and washed with brine. The aqueous phase was re-extracted with 20 mL diethyl ether three times. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate = 2:1) to afford the desired products **3aa** in 85% yield (169.2 mg).

(d) The Light on/off Experiments

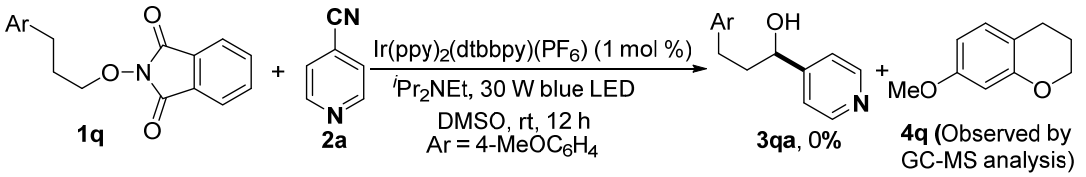
Figure S2 The Light on/off Experiments



Time/h	0	2(blue)	4(dark)	6(blue)	8(dark)	10(blue)	12(dark)	14(blue)
Yield/%	0	20	20	56	56	69	69	76



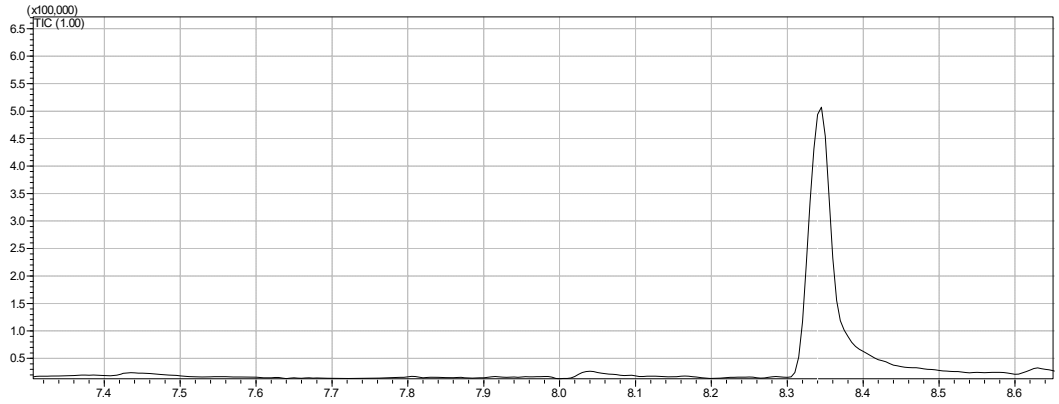
(d) Control Experiments

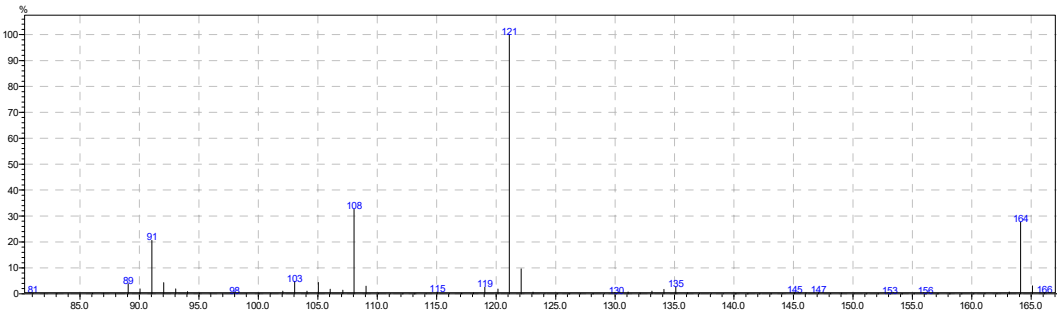


Chemical Formula: $C_{10}H_{12}O_2$

Exact Mass: 164.0837

GC-MS dates of 4q:





[MS Spectrum]

# of Peaks 414

Raw Spectrum 8.335 (scan : 868)

Background No Background Spectrum

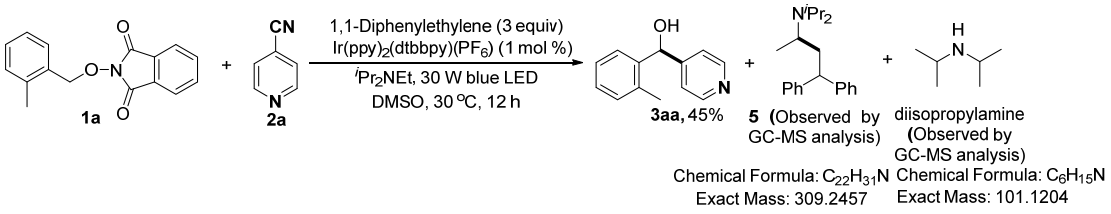
Base Peak m/z 121.10 (Inten : 173,542)

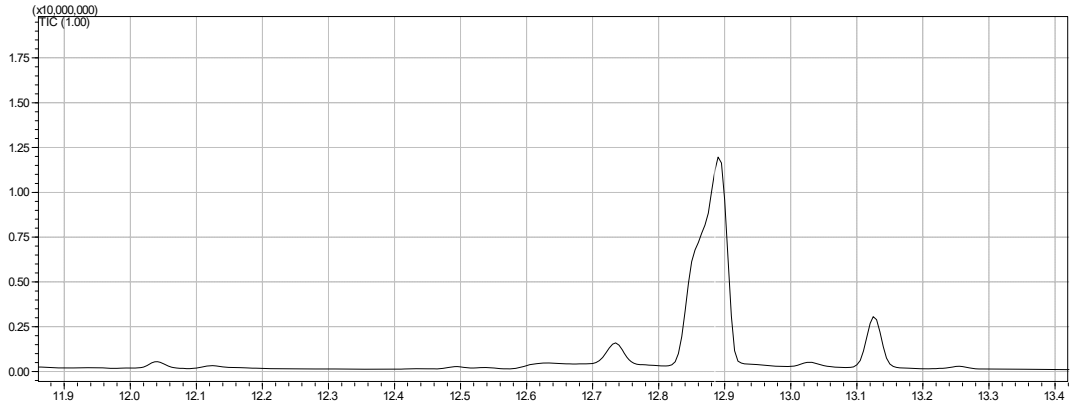
Event# 1

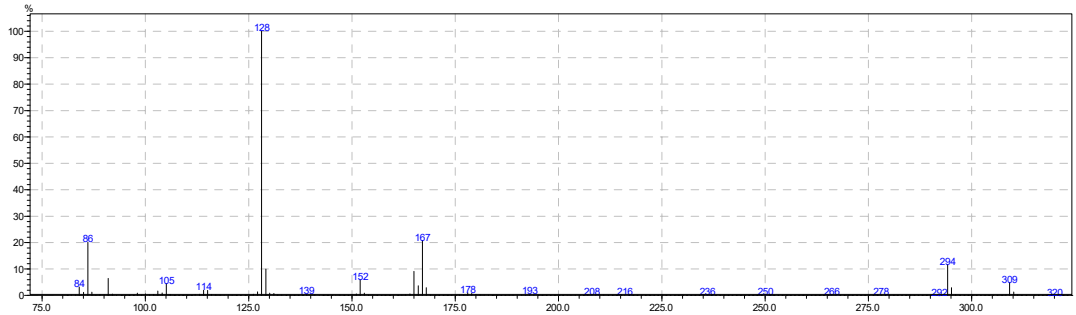
m/z	Absolute Intensity	Relative Intensity	m/z	Absolute Intensity	Relative Intensity	m/z	Absolute Intensity	Relative Intensity
89.05	6627	3.82	93.05	3389	1.95	106.05	3119	1.80
90.05	3269	1.88	103.05	8176	4.71	107.15	2347	1.35
91.05	35766	20.61	104.10	1753	1.01	108.05	54977	31.68
92.05	7524	4.34	105.10	7559	4.36	109.05	5030	2.90

119.05	4369	2.52	123.10	1202	0.69	135.10	4660	2.69
120.15	3195	1.84	124.10	119	0.07	163.15	1159	0.67
121.10	**173542**	**100.00**	133.10	1724	0.99	**164.10**	**47964**	**27.64**
122.10	17106	9.86	134.10	3012	1.74	165.10	5361	3.09

GC-MS dates of 5 and diisopropylamine:







[MS Spectrum]

# of Peaks 260

Raw Spectrum 12.870 (scan : 1775)

Background 12.790 (scan : 1759)

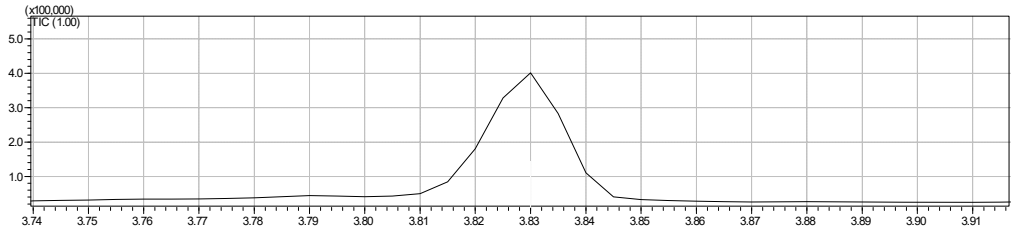
Base Peak m/z 128.15 (Inten : 3,481,902)

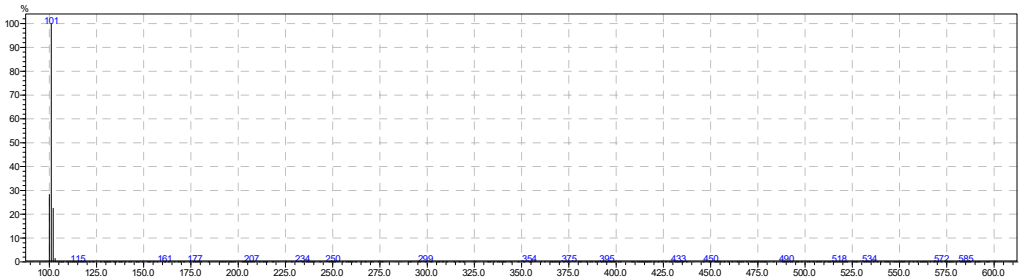
Event# 1

m/z Absolute Intensity Relative Intensity

81.05	555	0.02	82.05	5732	0.16	83.05	6426	0.18

84.00	114947	3.30	114.05	65286	1.88	167.05	689923	19.81
85.05	40549	1.16	115.00	66849	1.92	168.05	97063	2.79
86.05	705472	20.26	**128.15**	**3481902**	**100.00**	178.00	35333	1.01
87.05	41025	1.18	129.10	345753	9.93	294.15	345169	9.91
91.00	213742	6.14	151.05	20176	0.58	295.15	84878	2.44
92.00	16529	0.47	152.00	204229	5.87	308.20	5327	0.15
103.00	60431	1.74	153.00	30482	0.88	**309.15**	**137340**	**3.94**
104.05	24465	0.70	164.05	20454	0.59	310.15	36578	1.05
105.05	94371	2.71	165.00	323641	9.29			
113.05	13808	0.40	166.05	127220	3.65			





[MS Spectrum]

# of Peaks 275

Raw Spectrum 3.830 (scan : 567)

Background 3.810 (scan : 563)

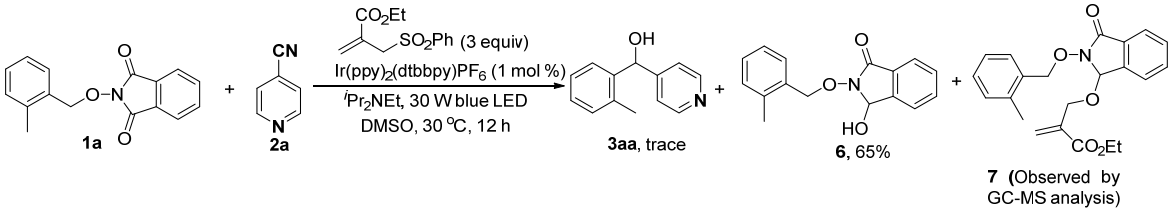
Base Peak m/z 101.10 (Inten : 230,123)

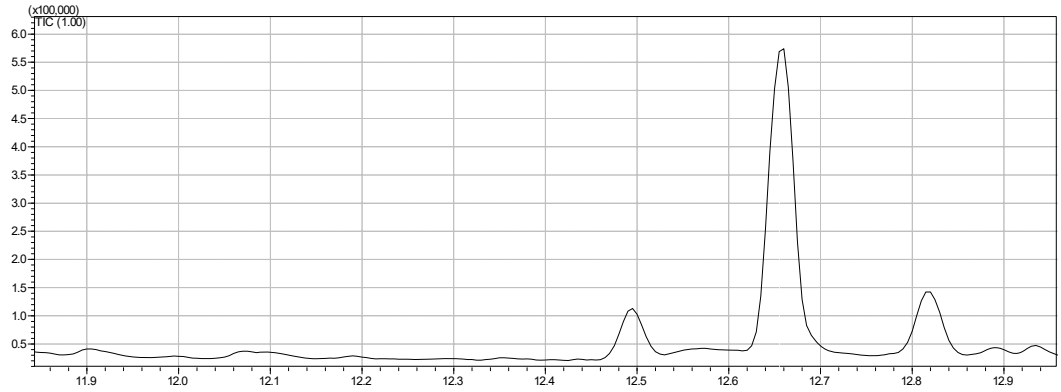
Event# 1

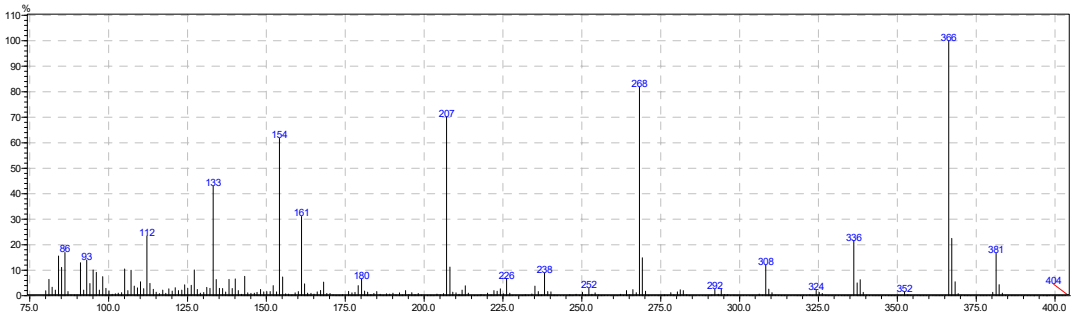
m/z Absolute Intensity Relative Intensity

100.10	64952	28.22	102.10	51729	22.48	104.10	116	0.05
101.10	**230123**	**100.00**	103.10	3377	1.47	105.10	51	0.02

GC-MS dates of 7:







[MS Spectrum]

# of Peaks 421

Raw Spectrum 12.655 (scan : 1732)

Background No Background Spectrum

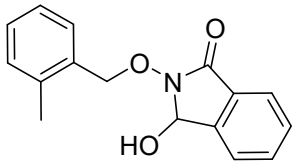
Base Peak m/z 366.30 (Inten : 57,239)

Event# 1

m/z	Absolute Intensity	Relative Intensity						
80.05	1119	1.95	85.10	6374	11.14	94.05	2757	4.82
81.05	3683	6.43	86.10	9716	16.97	95.05	5803	10.14
82.05	1919	3.35	91.05	7437	12.99	96.05	5257	9.18
83.10	1276	2.23	92.05	1296	2.26	97.05	1273	2.22
84.10	8937	15.61	93.05	7989	13.96	98.10	4300	7.51

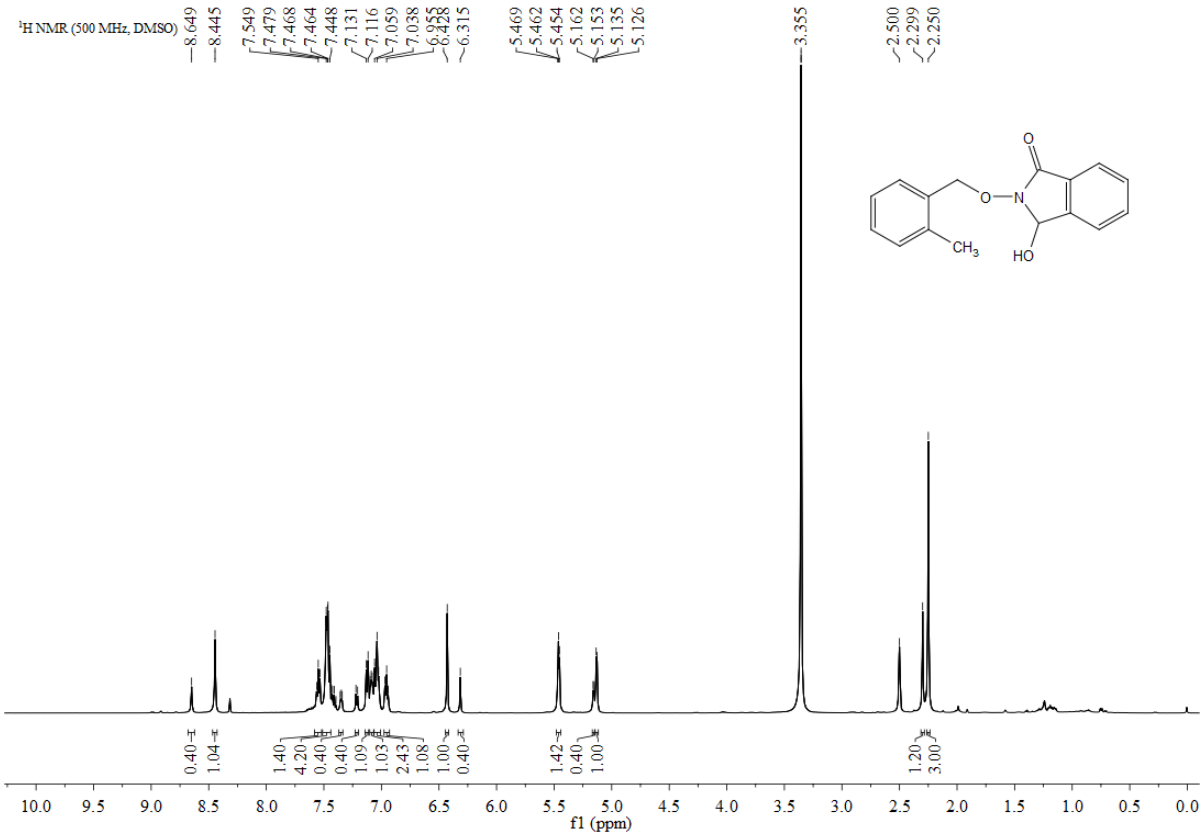
99.10	1657	2.89	138.15	3674	6.42	240.20	873	1.53
100.10	1086	1.90	139.15	1621	2.83	250.20	761	1.33
105.05	6008	10.50	140.15	3771	6.59	252.20	1652	2.89
106.10	1164	2.03	141.10	1174	2.05	264.20	1182	2.07
107.10	5716	9.99	143.10	4374	7.64	266.20	1383	2.42
108.10	2154	3.76	152.15	2285	3.99	267.25	701	1.22
109.10	1766	3.09	154.15	35338	61.74	268.25	46871	81.89
110.10	3113	5.44	155.15	4188	7.32	269.20	8548	14.93
111.10	1604	2.80	161.10	17806	31.11	281.20	1343	2.35
112.10	13386	23.39	162.10	2668	4.66	282.20	1196	2.09
113.10	2778	4.85	167.15	1202	2.10	292.20	1492	2.61
114.15	1463	2.56	168.15	3056	5.34	294.20	1479	2.58
117.10	1268	2.22	179.10	2292	4.00	308.30	6870	12.00
119.10	1562	2.73	180.15	3742	6.54	309.25	1493	2.61
120.10	1052	1.84	181.15	1048	1.83	324.25	1240	2.17
121.10	1820	3.18	182.10	796	1.39	336.25	12372	21.61
122.10	1185	2.07	194.15	1161	2.03	337.25	2893	5.05
123.15	1300	2.27	207.15	40145	70.14	338.25	3611	6.31
124.10	2450	4.28	208.15	6468	11.30	339.25	763	1.33
125.05	1696	2.96	212.05	1280	2.24	**366.30**	**57239**	**100.00**
126.15	2358	4.12	213.05	2273	3.97	367.30	12884	22.51
127.15	5781	10.10	222.15	1145	2.00	368.30	3121	5.45
128.10	1439	2.51	223.15	994	1.74	380.30	774	1.35
131.10	1881	3.29	224.15	1571	2.74	**381.30**	**9545**	**16.68**
132.15	1657	2.89	226.15	3839	6.71	382.30	2505	4.38
133.10	24593	42.97	235.15	2176	3.80	383.30	586	1.02
134.10	3630	6.34	236.15	943	1.65	384.30	126	0.22
135.10	1658	2.90	238.20	5100	8.91			
136.10	1646	2.88	239.15	946	1.65			

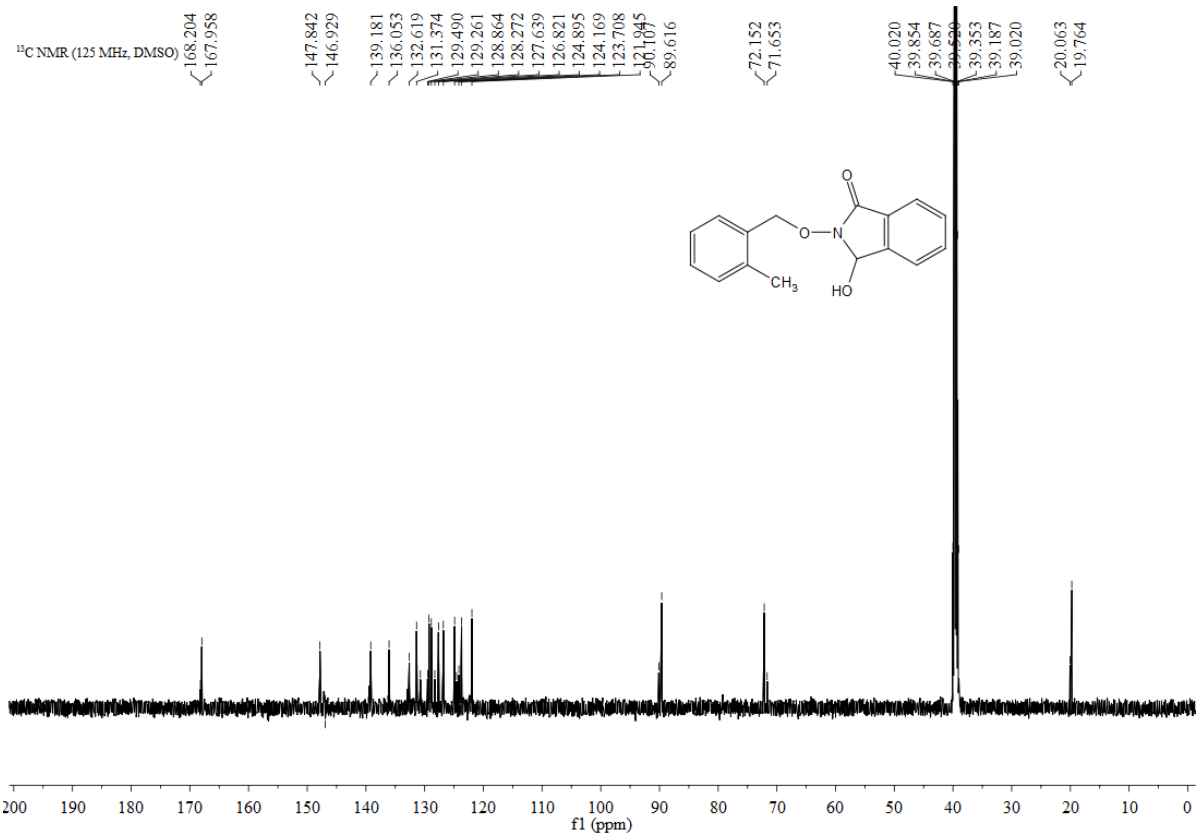
3-hydroxy-2-((2-methylbenzyl)oxy)isoindolin-1-one (6):



35.0 mg, 65% yield; white solid; dr = 1 : 0.4; ^{1}H NMR (500 MHz, DMSO) δ (ppm) 8.65 (s, 0.4H), 8.44 (s, 1H), 7.55 (t, *J* = 6.9 Hz, 1.4H), 7.51 - 7.44 (m, 4.2H), 7.37 - 7.33 (m, 0.4H), 7.21 (d, *J* = 7.4 Hz, 0.4H), 7.12 (d, *J* = 7.7 Hz, 1H), 7.10 - 7.07 (m, 1H), 7.06 - 7.01 (m, 2.4H), 6.95 (t, *J* = 7.0 Hz, 1H), 6.43 (s, 1H), 6.31 (s, 0.4H), 5.48 - 5.44 (m, 1.4 H), 5.16 (d, *J* = 4.4 Hz, 0.4H), 5.13 (d, *J* = 4.3 Hz, 1H), 2.30 (s, 1.2H), 2.25 (s, 3H); ^{13}C NMR (125 MHz, DMSO) δ (ppm) 168.2, 168.0, 147.8, 146.9, 139.4, 139.2, 136.1, 136.0, 132.6, 131.4, 130.7, 129.5, 129.3, 128.9, 128.3, 127.6, 126.9, 126.8, 124.9, 124.6, 124.2, 123.7, 121.9, 90.1, 89.6, 72.2, 71.7, 20.1, 19.8; HRMS *m/z* (ESI) calcd for $C_{16}H_{16}NO_3$ ([M+H]$^+$) 270.1125, found 270.1129.

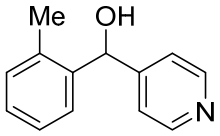
3-hydroxy-2-((2-methylbenzyl)oxy)isoindolin-1-one (6)





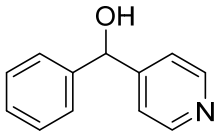
(B) Analytical data

Pyridin-4-yl(*o*-tolyl)methanol (3aa):



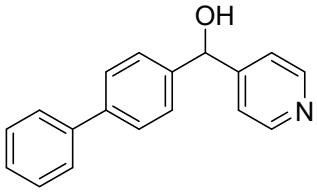
35.8 mg, 90% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 123.2-125.9 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.41 (d, *J* = 5.0 Hz, 2H), 7.31 - 7.15 (m, 6H), 5.97 (s, 1H), 3.63 (s, 1H), 2.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.4, 149.4, 140.5, 135.7, 130.9, 128.1, 127.2, 126.4, 121.7, 72.0, 19.4; LRMS (EI, 70 eV) *m/z* (%): 199 (M^+, 25), 181 (100), 121 (32), 106 (33), 93 (59), 80 (25). The analytical data are in accordance with those reported in the literature.[1]

Phenyl(pyridin-4-yl)methanol (3ba):



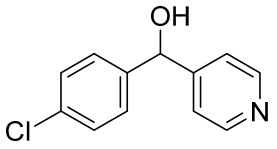
31.5 mg, 85% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 113.2-114.9 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.34 (d, *J* = 6.0 Hz, 2H), 7.33 - 7.29 (m, 6H), 7.28 - 7.25 (m, 1H), 5.75 (s, 1H), 4.28 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 153.3 149.1, 142.9, 128.7, 128.0, 126.8, 121.3, 74.6; LRMS (EI, 70 eV) *m/z* (%): 185 (M^+, 100), 167 (10), 139 (8), 107 (40), 80 (49). The analytical data are in accordance with those reported in the literature.[2]

[1,1'-Biphenyl]-4-yl(pyridin-4-yl)methanol (3ca):



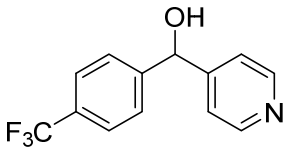
35.5 mg, 68% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 100.2-101.7 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.47 (s, 2H), 7.60 - 7.54 (m, 4H), 7.45 - 7.39 (m, 4H), 7.37 - 7.32 (m, 3H), 5.83 (s, 1H), 3.75 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.7, 149.6, 141.6, 141.2, 140.4, 128.8, 127.54, 127.49, 127.2, 127.1, 121.3, 74.67; LRMS (EI, 70 eV) *m/z* (%): 261 (M^+, 5), 259 (57), 195 (33), 181 (100), 152 (56), 106 (12); HRMS *m/z* (ESI) calcd for $C_{18}H_{16}NO$ ($[M+H]^+$) 262.1226, found 262.1235.

(4-Chlorophenyl)(pyridin-4-yl)methanol (3da):



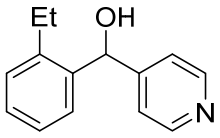
38.5 mg, 88% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 128.7-129.5 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.38 (d, *J* = 5.5 Hz, 2H), 7.31 - 7.26 (m, 6H), 5.75 (s, 1H), 4.46 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.8, 149.3, 141.3, 133.9, 128.9, 128.1, 121.3, 74.02; LRMS (EI, 70 eV) *m/z* (%): 221 (M^++2, 4), 219 (M^+, 10), 217 (31), 141 (33), 139 (100), 125 (23), 113 (12), 111 (36). The analytical data are in accordance with those reported in the literature.[3]

Pyridin-4-yl(4-(trifluoromethyl)phenyl)methanol (3ea):



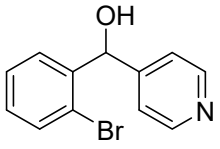
32.9 mg, 65% yield; R_f = 0.2 (PE/EA = 10 : 1); white solid; m.p. 106.4-107.6 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.41 (d, *J* = 5.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 5.5 Hz, 2H), 5.83 (s, 1H), 4.35 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.4, 149.5, 146.4, 130.3 (q, J_{C-F} = 3.5 Hz), 126.9, 125.7 (q, *J* = 3.5 Hz), 123.9 (q, *J* = 270.5 Hz), 121.3, 74.2; ^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm) -62.6; LRMS (EI, 70 eV) *m/z* (%): 253 (M^+, 50), 173 (100), 145 (67), 106 (15), 92 (12); HRMS *m/z* (ESI) calcd for $C_{13}H_{11}F_3NO$ ($[M+H]^+$) 254.0787, found 254.0795.

(2-Ethylphenyl)(pyridin-4-yl)methanol (3fa):



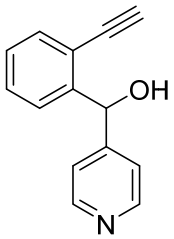
36.6 mg, 86% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 117.2-118.7 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.29 (d, *J* = 6.0 Hz, 2H), 7.26 - 7.22 (m, 4H), 7.21 - 7.15 (m, 2H), 6.01 (s, 1H), 4.90 (s, 1H), 2.66 (q, *J* = 7.7 Hz, 2H), 1.14 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 153.5, 149.0, 141.8, 140.1, 128.9, 128.2, 127.7, 126.3, 121.7, 71.0, 25.3, 15.5; LRMS (EI, 70 eV) *m/z* (%): 213 (M^+, 5), 211 (17), 195 (100), 180 (55), 133 (36), 105(89); HRMS *m/z* (ESI) calcd for $C_{14}H_{16}NO$ ($[M+H]^+$) 214.1226, found 214.1232.

(2-Bromophenyl)(pyridin-4-yl)methanol (3ga):



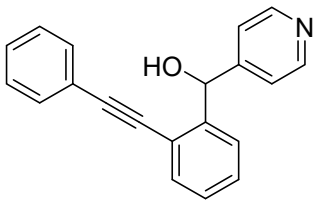
31.6 mg, 60% yield; R_f = 0.2 (PE/EA = 10 : 1); white solid; m.p. 116.7-117.6 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.37 (d, *J* = 5.5 Hz, 2H), 7.34 - 7.27 (m, 6H), 5.76 (s, 1H), 4.37 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 153.1, 149.3, 142.9, 128.7, 128.1, 126.8, 121.3, 74.7. The analytical data are in accordance with those reported in the literature.[4]

(2-Ethynylphenyl)(pyridin-4-yl)methanol (3ha):



33.4 mg, 80% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 144.0-144.9 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.37 (d, *J* = 5.5 Hz, 2H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.38 - 7.35 (m, 2H), 7.35 - 7.32 (m, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 6.32 (s, 1H), 4.52 (s, 1H), 3.31 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.6, 149.2, 145.3, 133.0, 129.6, 127.8, 126.6, 121.3, 120.2, 82.5, 81.5, 71.7; LRMS (EI, 70 eV) *m/z* (%): 209(M^+, 100), 180(67), 152(26), 131(52), 103(31); HRMS *m/z* (ESI) calcd for $C_{14}H_{12}NO$ ($[M+H]^+$) 210.0913, found 210.0916.

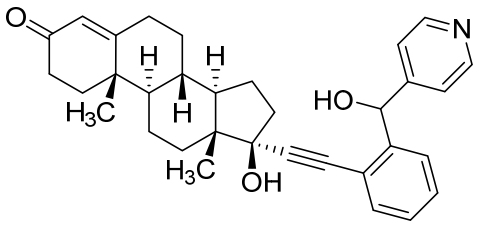
(2-(phenylethynyl)phenyl)(pyridin-4-yl)methanol (3ia):



46.7 mg, 82% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 132.3-134.3 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.37 (d, *J* =6.0 Hz, 2H), 7.52 (d, *J* = 7.5

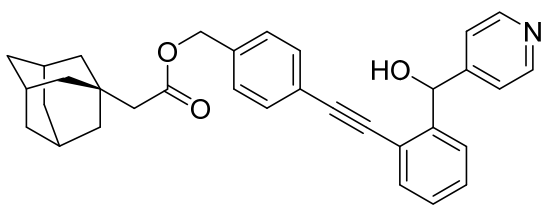
Hz, 1H), 7.47 (d, *J* = 7.7 Hz, 1H), 7.44 - 7.41 (m, 2H), 7.39 (d, *J* = 6.0 Hz, 2H), 7.35 – 7.30 (m, 4H), 7.26 (t, *J* = 7.5 Hz, 1H), 6.37 (s, 1H), 4.80 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.8, 149.2, 144.6, 132.4, 131.4, 129.0, 128.6, 128.4, 127.8, 126.6, 122.6, 121.4, 121.3, 94.5, 87.1, 72.1; LRMS (EI, 70 eV) *m/z* (%): 285 (M^+, 5), 283 (88), 282 (100), 254 (46), 176 (53), 151 (27), 127 (29); HRMS *m/z* (ESI) calcd for $C_{20}H_{16}NO$ ($[M+H]^+$) 286.1226, found 286.1233.

(*8R,9S,10R,13S,14S,17S*)-17-hydroxy-17-((2-(hydroxy(pyridin-4-yl)methyl)phenyl)ethynyl)-10,13-dimethyl-1,2,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-3H-cyclopenta[a]phenanthren-3-one (3ja):



57.4mg, 58% yield; R_f = 0.1 (PE/EA = 1 : 2); white solid; m.p. 89.5-90.6 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.30 (t, *J* = 6.0 Hz, 2H), 7.41 - 7.33 (m, 2H), 7.27 (s, 3H), 7.21 (t, *J* = 6.5 Hz, 1H), 6.20 (d, *J* = 4.0 Hz, 1H), 5.68 (d, *J* = 4.0 Hz, 1H), 4.74 (s, 1H), 2.38 - 2.19 (m, 5H), 2.08 - 2.02 (m, 1H), 1.97 - 1.91 (m, 1H), 1.79 (s, 1H), 1.69 - 1.51 (m, 6H), 1.47 - 1.33 (m, 3H), 1.14 (s, 3H), 0.98 - 0.76 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 200.0, 171.6, 152.9, 148.9, 146.4, 144.2, 132.4, 132.3, 128.9, 127.7, 126.7, 123.7, 121.5, 121.3, 98.4, 83.6, 83.5, 79.83, 79.79, 71.9, 53.4, 50.3, 47.2, 47.1, 39.01, 38.97, 38.5, 36.09, 36.06, 35.5, 33.8, 32.8, 32.66, 32.64, 31.5, 31.4, 23.1, 20.6, 17.3, 12.89, 12.87; HRMS *m/z* (ESI) calcd for $C_{33}H_{38}NO_3$ ($[M+H]^+$) 496.2846, found 496.2859.

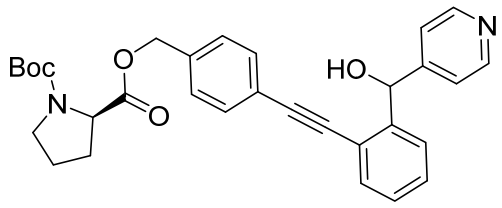
4-((2-(hydroxy(pyridin-4-yl)methyl)phenyl)ethynyl)benzyl-2-((*3r,5r,7r*)-adamantan -1-yl)acetate (3ka):



63.8 mg, 65% yield; R_f = 0.1 (PE/EA = 2 : 1); white solid; m.p. 152.9-153.7 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.43 (d, *J* = 5.5 Hz, 2H), 7.53 (d, *J* = 7.5 Hz, 1H), 7.47 (d, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 5.5 Hz, 2H), 7.34 (t, *J* = 8.5 Hz, 3H), 7.28 (d, *J* = 7.5 Hz, 1H), 6.36 (s, 1H), 5.09 (s, 2H), 2.12 (s, 2H), 1.95 (s, 3H), 1.69 (d, *J* = 12.0 Hz, 3H), 1.62 - 1.59 (m, 9H); ^{13}C

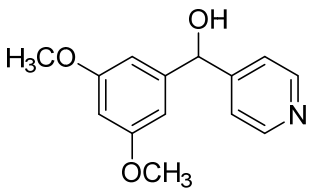
NMR (125 MHz, $CDCl_3$) δ (ppm) 171.5, 152.5, 149.4, 144.5, 136.8, 132.4, 131.5, 129.1, 128.2, 127.9, 126.7, 122.4, 121.3, 121.2, 94.3, 87.5, 72.3, 65.2, 48.8, 42.3, 36.6, 32.9, 28.5; HRMS *m/z* (ESI) calcd for $C_{33}H_{34}NO_3$ ($[M+H]^+$) 492.2533, found 492.2543.

1-(tert-butyl) 2-(4-((2-(hydroxyl (pyridin-4-yl)methyl)phenyl)ethynyl)benzyl) (*2R*)-pyrrolidine-1,2- dicarboxylate (3la):



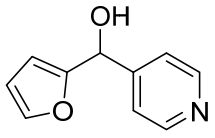
87.0 mg, 85% yield; R_f = 0.2 (PE/EA = 1 : 1); white solid; m.p. 82.7-83.6 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.39 (d, *J* = 4.5 Hz, 2H), 7.49 (dd, *J* = 15.5, 7.5 Hz, 2H), 7.43 - 7.36 (m, 4H), 7.34 - 7.29 (m, 3H), 7.26 - 7.23 (m, 1H), 6.35 (s, 1H), 5.20 - 5.07 (m, 2H), 4.38 - 4.34 (m, 0.46H), 4.28 - 4.23 (m, 0.65H), 3.53 - 3.36 (m, 2H), 2.23 - 2.13 (m, 1H), 1.96 - 1.82 (m, 3H), 1.43 (s, 4H), 1.32 (s, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 172.9, 172.7, 154.4, 153.7, 152.8, 152.7, 149.2, 144.7, 136.4, 136.1, 132.35, 132.30, 131.5, 131.4, 129.2, 129.1, 128.2, 127.9, 127.7, 126.6, 122.7, 122.4, 121.3, 121.1, 121.0, 94.1, 93.9, 87.7, 87.6, 80.0, 79.9, 72.1, 72.0, 66.0, 59.1, 58.8, 46.5, 46.2, 30.8, 29.8, 28.3, 28.2, 24.2, 23.5; HRMS *m/z* (ESI) calcd for $C_{31}H_{33}N_2O_5$ ($[M+H]^+$) 513.2384, found 513.2393.

(3,5-dimethoxyphenyl)(pyridin-4-yl)methanol (3ma):



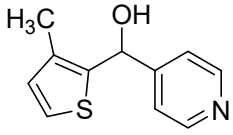
35.3 mg, 72% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 139.6-140.7 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.49 (d, *J* = 6.0 Hz, 2H), 7.32 (d, *J* = 6.0 Hz, 2H), 6.50 (d, *J* = 2.0 Hz, 2H), 6.38 (t, *J* = 2.5 Hz, 1H), 5.70 (s, 1H), 3.76 (s, 6H), 3.21 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 161.1, 152.3, 149.6, 145.0, 121.2, 104.7, 99.8, 74.9, 55.4; LRMS (EI, 70 eV) *m/z* (%): 245 (M^+, 83), 195 (100), 180 (56), 152 (20), 115 (21), 91 (28); HRMS *m/z* (ESI) calcd for $C_{14}H_{16}NO_3$ ($[M+H]^+$) 246.1125, found 246.1130.

Furan-2-yl(pyridin-4-yl)methanol (3na):



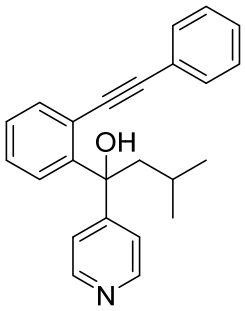
24.5 mg, 70% yield; R_f = 0.3 (PE/EA = 2 : 1); yellow liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.56 (s, 2H), 7.42 - 7.35 (m, 3H), 6.34 (s, 1H), 6.17 (s, 1H), 5.84 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 154.6, 149.9, 149.6, 143.0, 121.4, 110.4, 108.0, 68.4; LRMS (EI, 70 eV) *m/z* (%): 175 (M+, 3), 173 (42), 145 (10), 95 (100). The analytical data are in accordance with those reported in the literature.[5]

(3-methylthiophen-2-yl)(pyridin-4-yl)methanol (3oa):



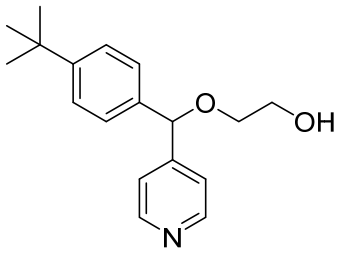
27.1 mg, 66% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 96.1-97.6 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.40 (s, 2H), 7.33 (s, 2H), 7.16 (s, 1H), 6.79 (s, 1H), 6.05 (s, 1H), 4.51 (s, 1H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.7, 149.1, 139.7, 134.5, 130.2, 124.3, 121.3, 68.6, 14.0; LRMS (EI, 70 eV) *m/z* (%): 205 (M^+, 5), 203 (34), 125 (100), 97 (10); HRMS *m/z* (ESI) calcd for $C_{11}H_{12}NOS$ ($[M+H]^+$) 206.0634, found 206.0640.

3-methyl-1-(2-(phenylethynyl)phenyl)-1-(pyridin-4-yl)butan-1-ol (3pa):



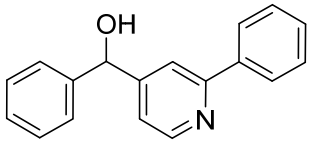
42.3 mg, 62% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 148.3 - 149.5 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.47 (d, *J* = 5.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 6.5 Hz, 1H), 7.41 (t, *J* = 7.0 Hz, 1H), 7.34 - 7.30 (m, 4H), 7.26 - 7.22 (m, 4H), 3.99 (s, 1H), 2.55 - 2.49 (m, 1H), 2.12 - 2.06 (m, 1H), 1.79 - 1.69 (m, 1H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.86 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.2, 149.3, 146.9, 134.7, 131.1, 128.8, 128.4, 128.3, 127.5, 126.3, 122.2, 121.6, 120.8, 96.4, 88.0, 78.8, 48.6, 24.71, 24.69, 24.2; LRMS (EI, 70 eV) *m/z* (%): 341(M+, 44), 298(21), 284(100), 254(9); HRMS *m/z* (ESI) calcd for $C_{24}H_{24}NO$ ($[M+H]^+$) 342.1852, found 342.1857.

2-((4-(tert-Butyl)phenyl)(pyridin-4-yl)methoxy)ethan-1-ol (3ra):



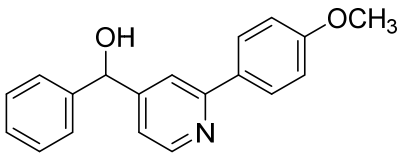
8.6 mg, 15% yield; R_f = 0.3 (PE/EA = 1 : 1); Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.53 (d, *J* = 4.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 4.5 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 5.35 (s, 1H), 3.80 (t, *J* = 4.0 Hz, 2H), 3.59 (t, *J* = 4.0 Hz, 2H), 1.30 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 151.3, 151.2, 149.6, 137.1, 126.9, 125.7, 121.6, 82.6, 70.5, 61.9, 34.5, 31.3; LRMS (EI, 70 eV) *m/z* (%): 285 (M^+, 47), 240 (100), 224 (82), 163 (47), 106 (42), 91 (27); HRMS *m/z* (ESI) calcd for $C_{18}H_{24}NO_2$ ($[M+H]^+$) 286.1802, found 286.1809.

Phenyl(2-phenylpyridin-4-yl)methanol (3bb):



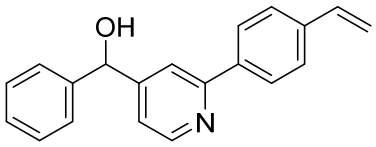
40.2 mg, 77% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; white solid; m.p. 121.3-122.0 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.47 (d, *J* = 5.0 Hz, 1H), 7.86 (d, *J* = 7.0 Hz, 2H), 7.69 (s, 1H), 7.42 - 7.36 (m, 3H), 7.31 - 7.24 (m, 5H), 7.15 (d, *J* = 5.0 Hz, 1H), 5.72 (s, 1H), 4.00 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 157.5, 153.6, 149.3, 142.7, 139.1, 134.1, 128.9, 128.7, 128.6, 128.1, 127.0, 126.8, 123.4, 119.8, 118.3, 74.9; LRMS (EI, 70 eV) *m/z* (%): 261 (M^+, 63), 230 (26), 127 (21), 105 (100); HRMS *m/z* (ESI) calcd for $C_{18}H_{16}NO$ ($[M+H]^+$) 262.1226, found 262.1231.

(2-(4-Methoxyphenyl)pyridin-4-yl)(phenyl)methanol (3bc):



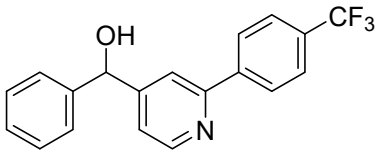
34.9 mg, 60% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 132.3-133.7 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.49 (s, 1H), 7.88 (s, 2H), 7.68 (s, 1H), 7.40 - 7.27 (m, 5H), 7.13 (s, 1H), 6.95 (s, 2H), 5.77 (s, 1H), 3.83 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 160.4, 157.3, 153.0, 149.5, 142.7, 131.9, 128.8, 128.3, 128.2, 126.8, 119.1, 117.3, 114.0, 75.2, 55.3; HRMS *m/z* (ESI) calcd for $C_{19}H_{18}NO_2$ ($[M+H]^+$) 292.1332, found 292.1339.

Phenyl(2-(4-vinylphenyl)pyridin-4-yl)methanol (3bd):



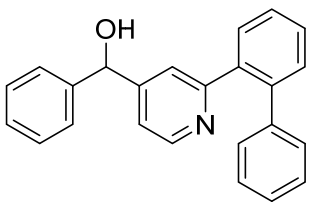
23.0 mg, 40% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; white solid; m.p. 134.1-135.7 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.51 (s, 1H), 7.88 (s, 2H), 7.73 (s, 1H), 7.46 (s, 2H), 7.38 - 7.30 (m, 5H), 7.16 (s, 1H), 6.74 (s, 1H), 5.86 - 5.72 (m, 2H), 5.29 (s, 1H), 3.57 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 157.2, 153.1, 149.6, 142.6, 138.5, 138.2, 136.3, 128.9, 128.3, 127.1, 126.8, 126.5, 119.8, 117.8, 114.5, 75.2; LRMS (EI, 70 eV) *m/z* (%): 287(M^+, 21), 286 (94), 244 (57), 148 (63), 124 (100), 91 (59); HRMS *m/z* (ESI) calcd for $C_{20}H_{18}NO$ ($[M+H]^+$) 288.1383, found 288.1388.

Phenyl(2-(4-(trifluoromethyl)phenyl)pyridin-4-yl)methanol (3be):



41.4 mg, 63% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 134.1-135.7 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.55 (d, *J* = 5.0 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.75 (s, 1H), 7.69 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 5.0 Hz, 3H), 7.22 (d, *J* = 5.0 Hz, 1H), 5.79 (s, 1H), 2.99 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 156.1, 153.6, 149.8, 142.5, 134.3, 131.7 (q, *J* = 211.7 Hz), 128.9, 128.4, 127.3, 126.8, 125.6 (q, *J* = 3.6 Hz), 123.6, 120.6, 118.3, 75.1; ^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm) -62.5; LRMS (EI, 70 eV) *m/z* (%): 329 (7), 289 (100), 260 (20), 105 (93); HRMS *m/z* (ESI) calcd for $C_{19}H_{15}F_3NO$ ($[M+H]^+$) 330.1100, found 330.1109.

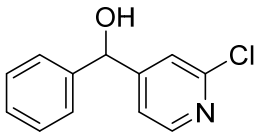
(2-([1,1'-biphenyl]-2-yl)pyridin-4-yl)(phenyl)methanol (3bf):



39.1 mg, 58% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 153.7-155.1 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.49 (d, *J* = 5.0 Hz, 1H), 7.63 (d, *J* = 7.0 Hz, 1H), 7.47 - 7.37 (m, 3H), 7.25 - 7.13 (m, 6H), 7.07 (t, *J* = 8.5 Hz, 3H), 6.95 (s, 2H), 6.84 (s, 1H), 5.43 (s, 1H), 2.91 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 159.2, 151.5, 149.5, 142.1, 141.3, 140.6, 139.3, 130.4, 130.3, 129.7, 128.7, 128.5, 128.1, 127.9, 127.6, 126.6, 126.5, 123.1, 119.0, 74.9; LRMS (EI, 70 eV) *m/z* (%): 337 (M^+, 100), 228 (14), 105 (10); HRMS *m/z* (ESI) calcd for

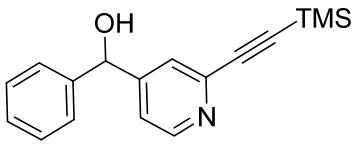
$C_{24}H_{20}NO$ ([M+H]$^+$) 338.1539, found 338.1550.

(2-chloropyridin-4-yl)(phenyl)methanol (3bg):



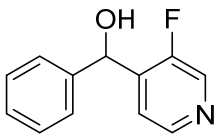
28.5 mg, 65% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 115.9-116.7 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.41 (d, *J* = 6.0 Hz, 2H), 7.35 - 7.33 (m, 4H), 7.31 (d, *J* = 6.0 Hz, 2H), 5.77 (s, 1H), 3.90 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.9, 149.4, 142.8, 128.8, 128.2, 126.8, 121.3, 74.8. The analytical data are in accordance with those reported in the literature.[6]

Phenyl(2-((trimethylsilyl)ethynyl)pyridin-4-yl)methanol (3bh):



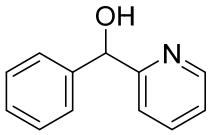
29.8 mg, 53% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 121.3-122.9 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.46 (d, *J* = 5.0 Hz, 1H), 7.50 (s, 1H), 7.38 – 7.30 (m, 5H), 7.25 (s, 1H), 5.77 (s, 1H), 2.75 (s, 1H), 0.25 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 152.7, 149.9, 142.9, 142.3, 128.9, 128.4, 126.9, 124.6, 120.6, 103.7, 94.8, 74.7, -0.3; LRMS (EI, 70 eV) *m/z* (%): 281(M$^+$, 51), 275(70), 274(57), 266(100), 105(18); HRMS *m/z* (ESI) calcd for $C_{17}H_{20}NOSi$ ([M+H]$^+$) 282.1309, found 282.1315.

(3-fluoropyridin-4-yl)(phenyl)methanol (3bi):



27.2 mg, 67% yield; R_f = 0.3 (PE/EA = 2 : 1); white solid; m.p. 120.2-121.1 ºC (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.31 (d, *J* = 5.0 Hz, 1H), 8.23 (s, 1H), 7.61 (t, *J* = 5.5 Hz, 1H), 7.43 - 7.26 (m, 5H), 6.09 (s, 1H), 3.77 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 156.6 (d, J_{C-F} = 254.2 Hz), 145.8 (d, J_{C-F} = 5.0 Hz), 141.5, 139.9 (d, *J* = 11.0 Hz), 137.5 (d, *J* = 24.0 Hz), 128.8, 128.3, 126.5, 121.5, 69.1 (d, J_{C-F} = 2.1 Hz); ^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm) -131.7; LRMS (EI, 70 eV) *m/z* (%): 203 (M$^+$, 100), 185 (12), 124 (43), 107 (40), 105 (19), 97 (50). The analytical data are in accordance with those reported in the literature.[7]

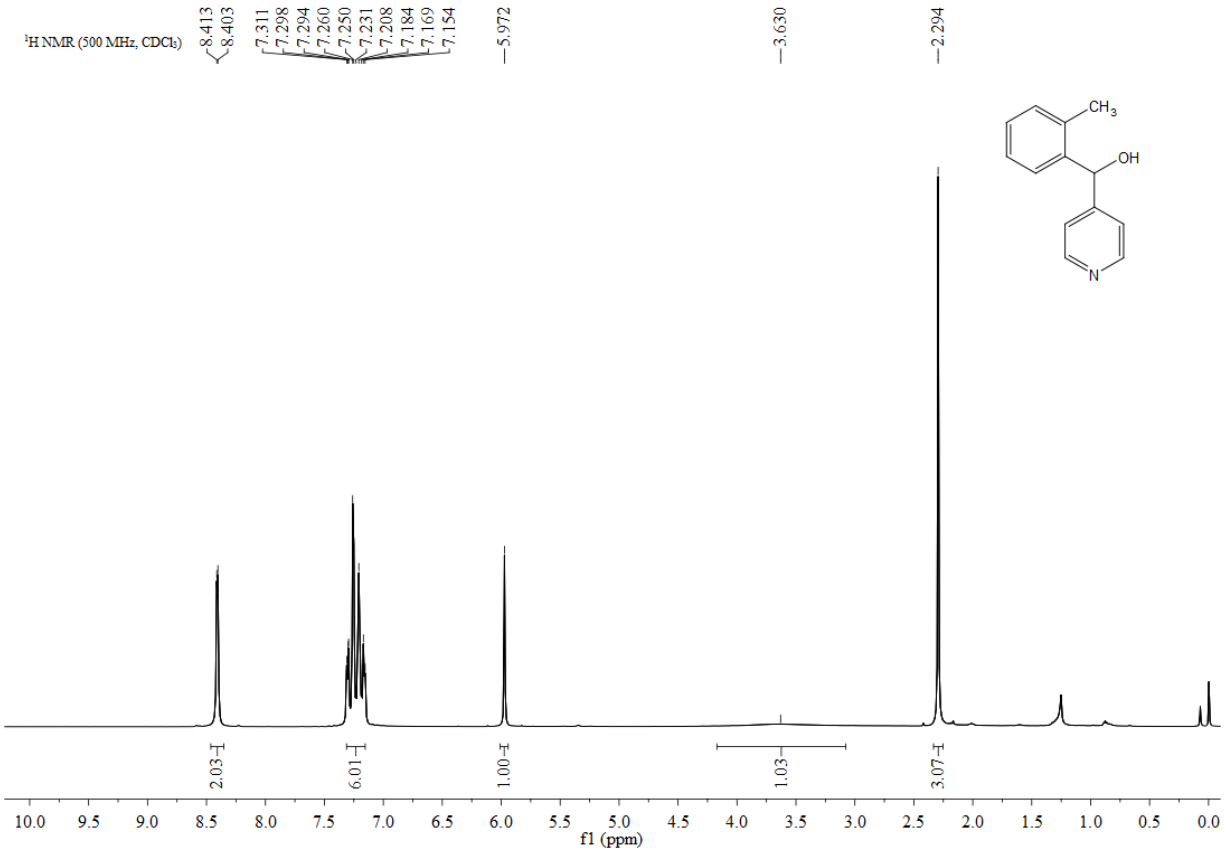
Phenyl(pyridin-2-yl)methanol (3bj):

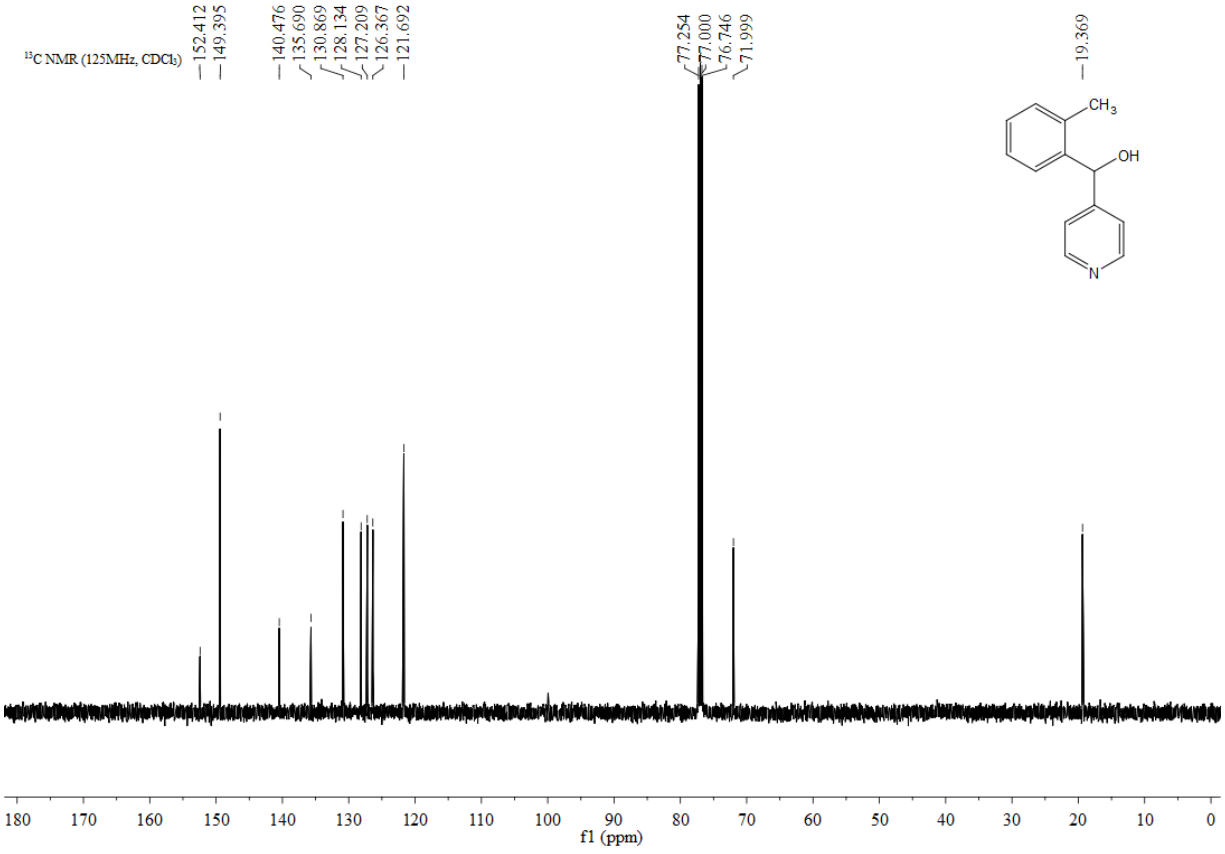


29.5 mg, 70% yield; R_f = 0.2 (PE/EA = 2 : 1); white solid; m.p. 109.3-110.9 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm) 8.57 (d, *J* = 5.0 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 7.0 Hz, 2H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 7.0 Hz, 1H), 7.20 (dd, *J* = 7.0, 5.5 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 5.75 (s, 1H), 5.28 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) 160.8, 147.8, 143.2, 136.8, 128.6, 127.8, 127.1, 122.4, 121.4, 74.9; LRMS (EI, 70 eV) *m/z* (%):185 (M^+, 100), 167 (18), 107 (38), 80 (52). The analytical data are in accordance with those reported in the literature.[2]

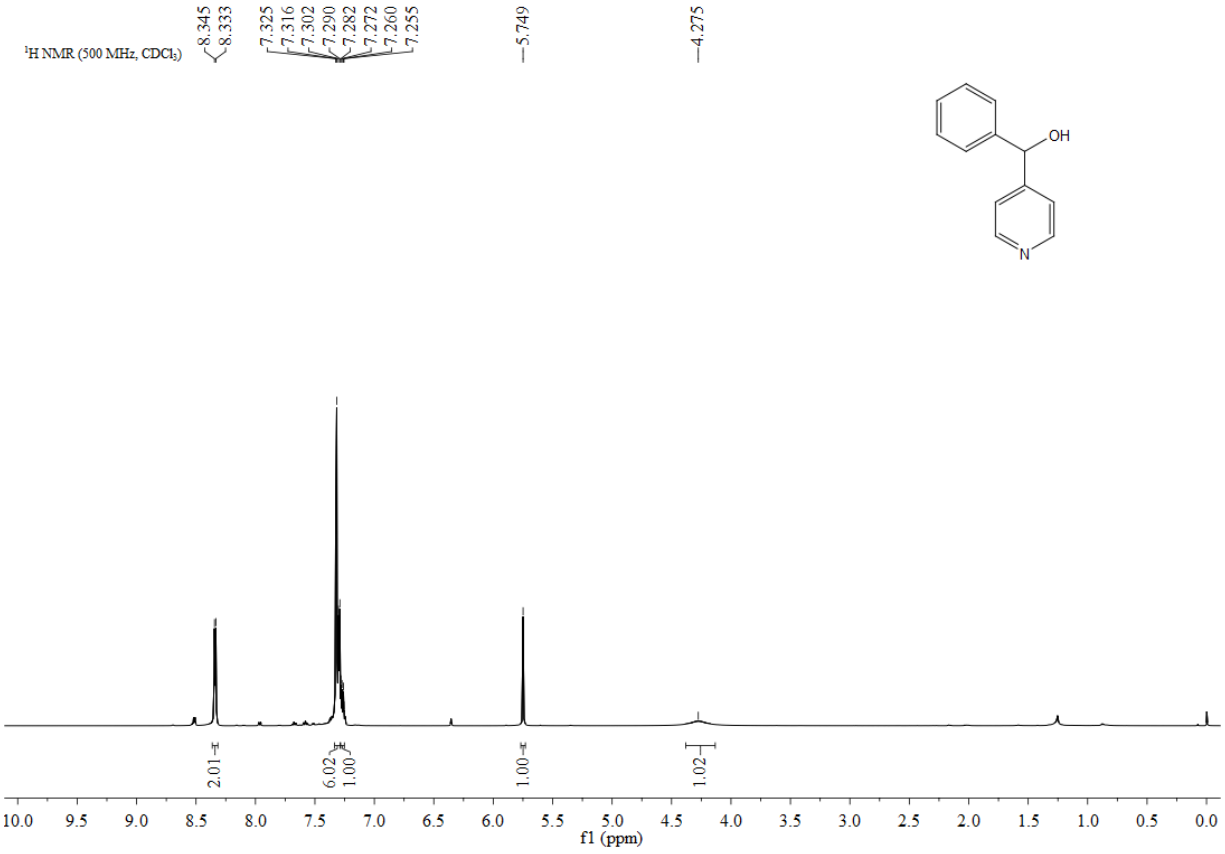
(C) Spectra

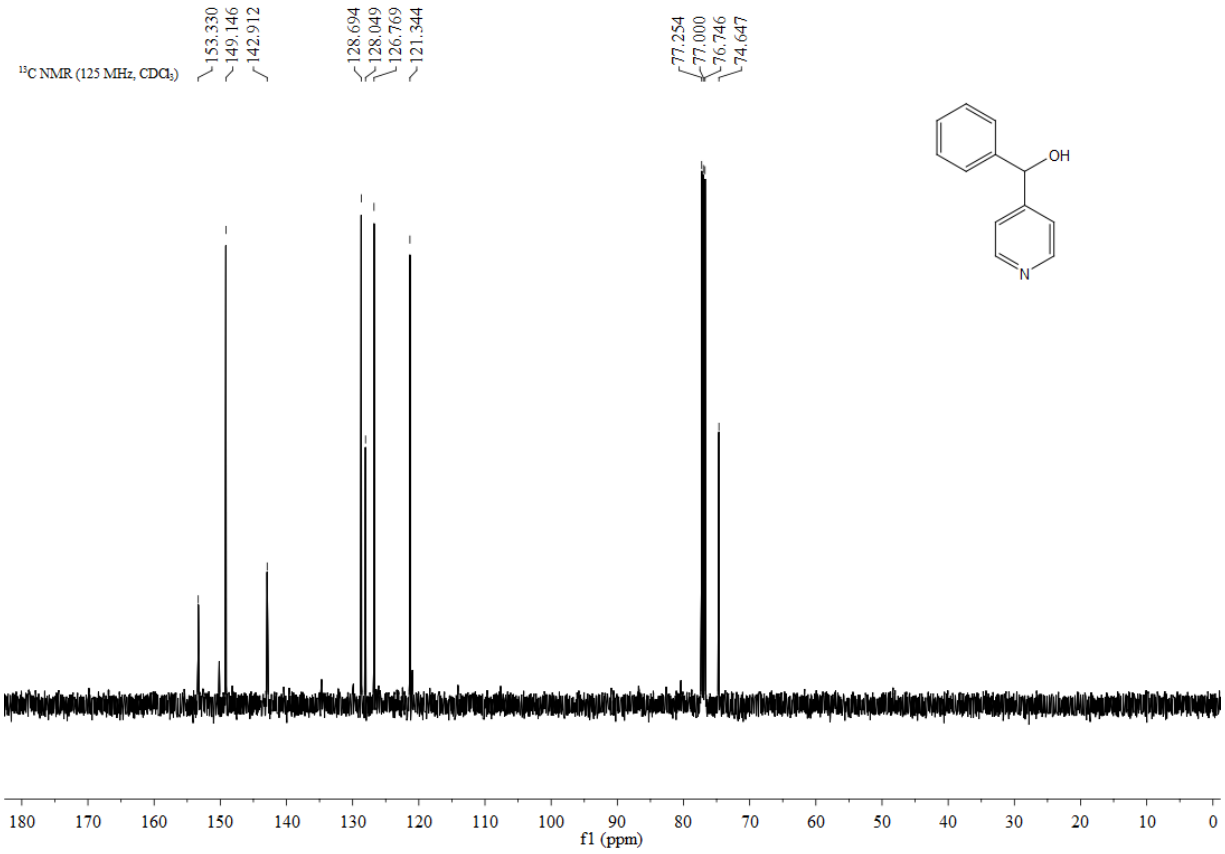
Pyridin-4-yl(*o*-tolyl)methanol (3aa)



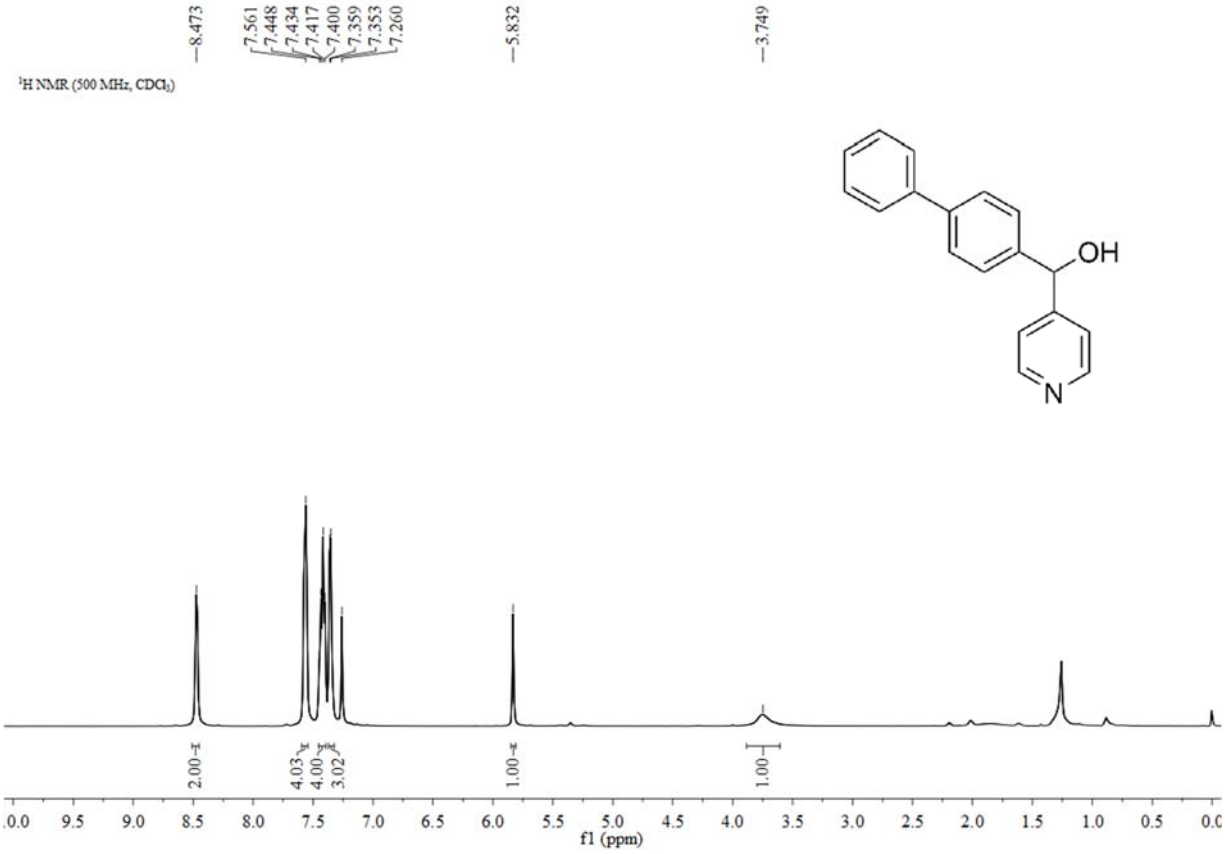


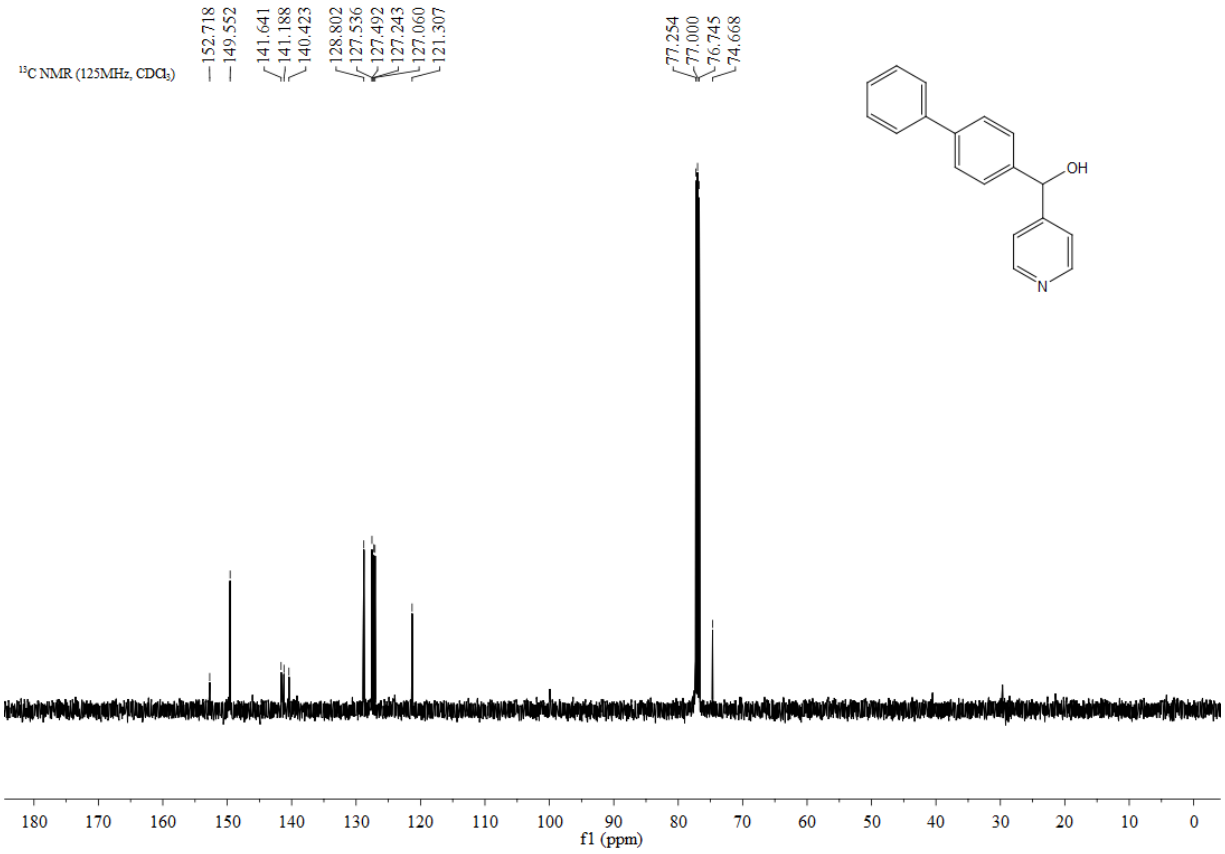
Phenyl(pyridin-4-yl)methanol (3ba)



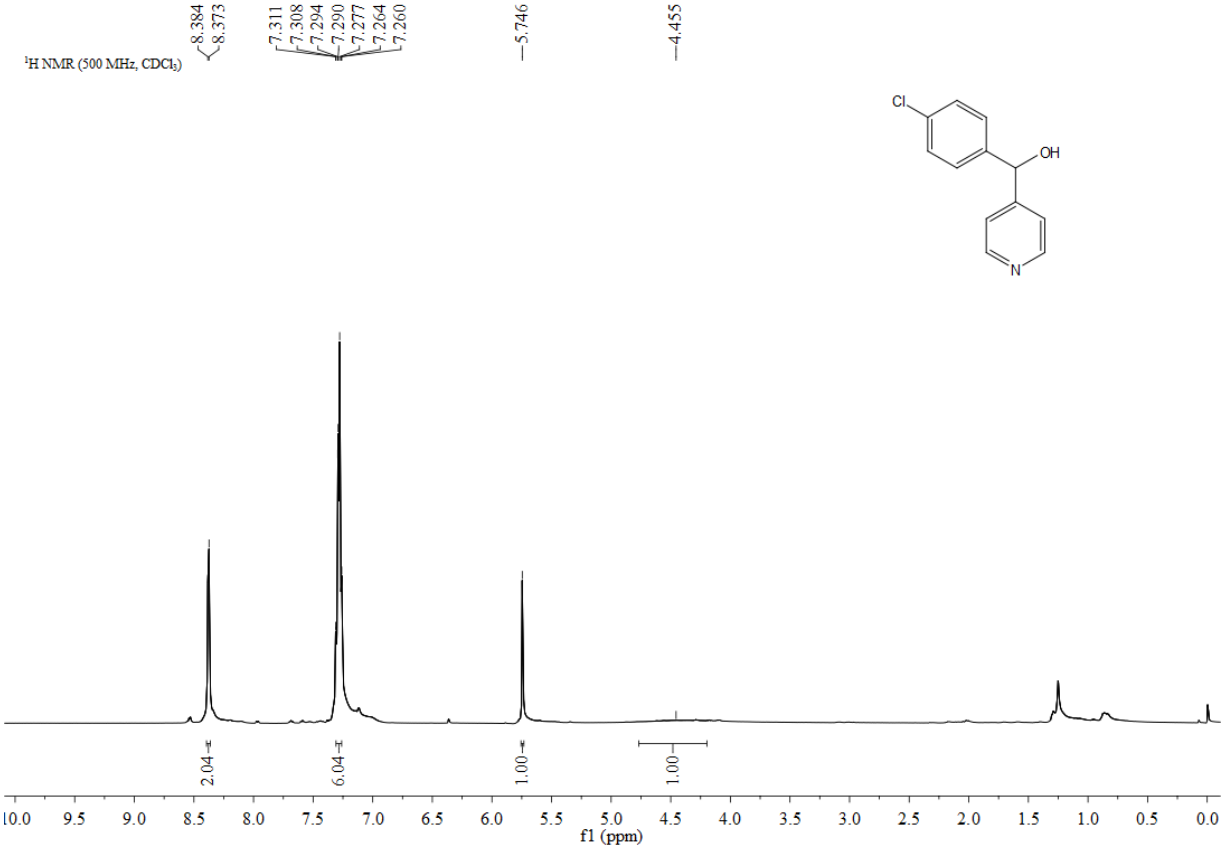


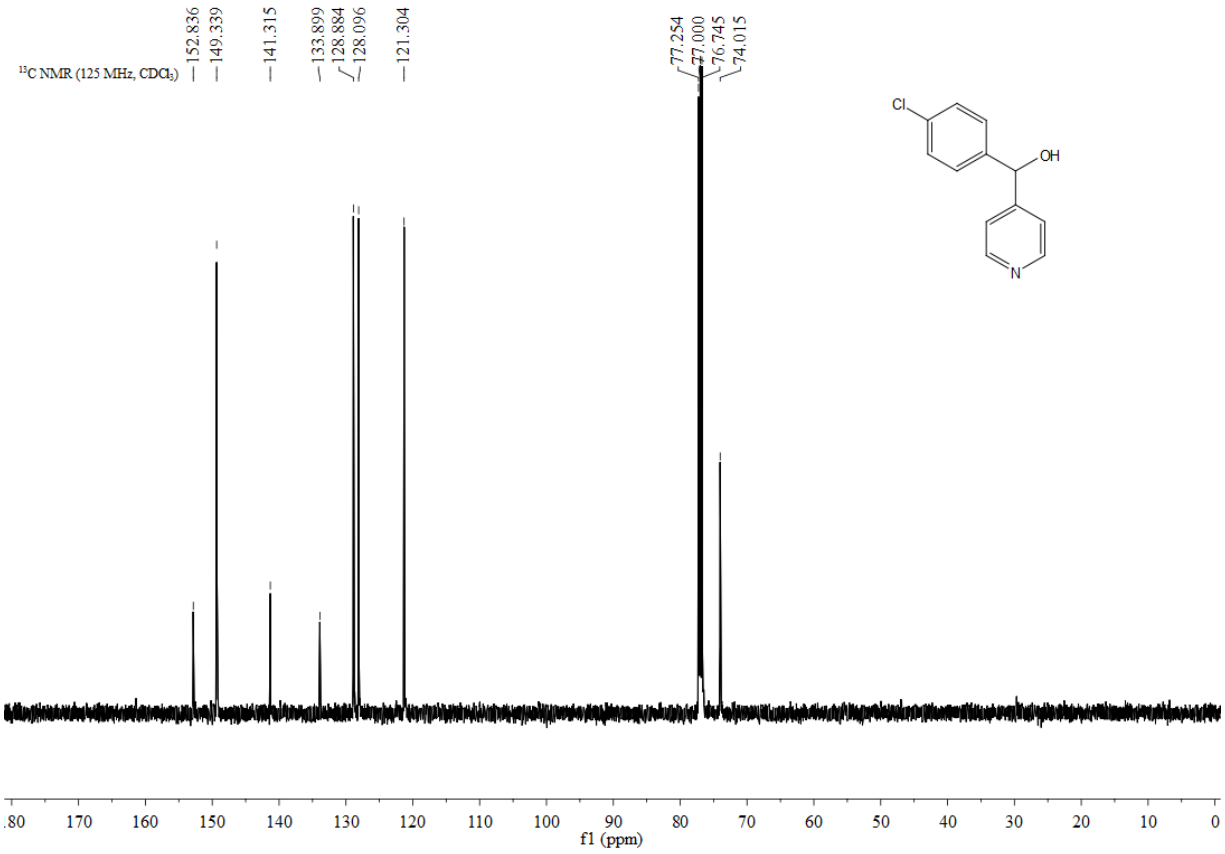
[1,1'-biphenyl]-4-yl(pyridin-4-yl)methanol (3ca)



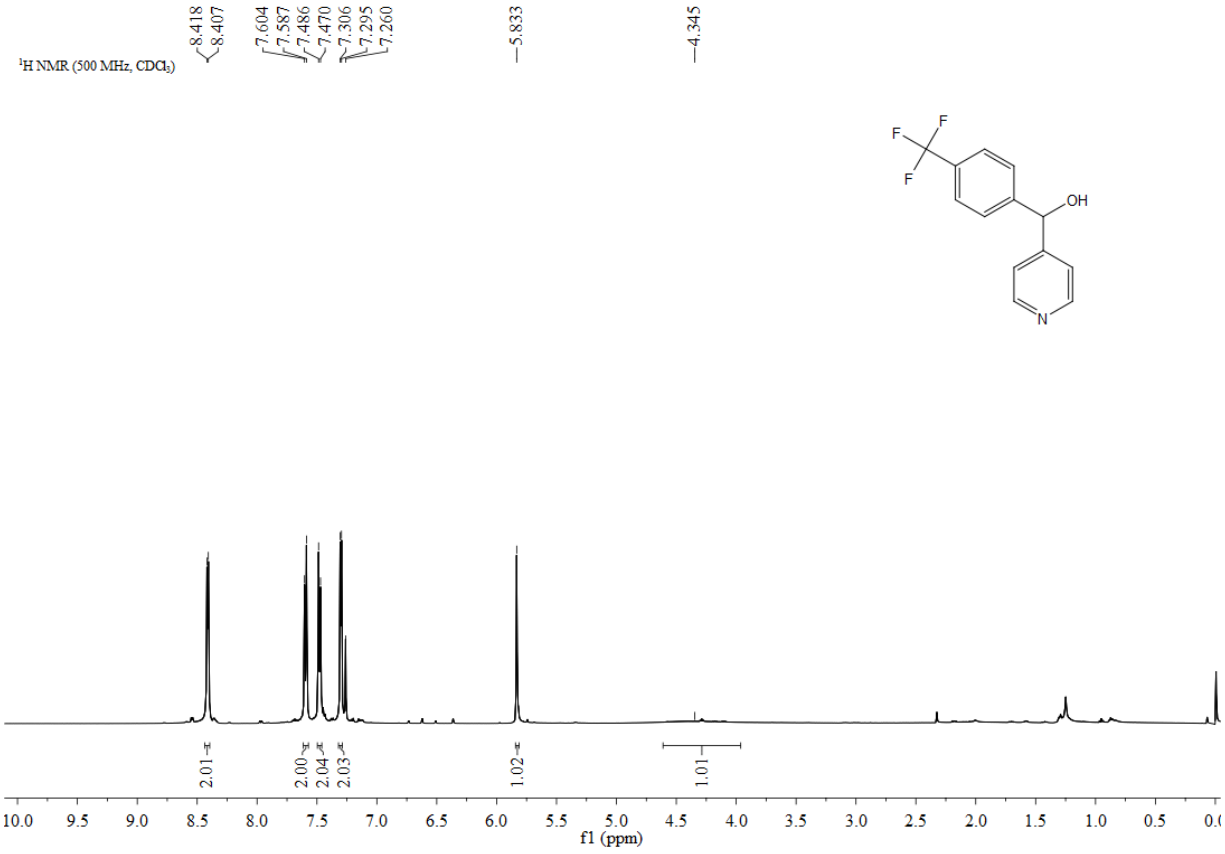


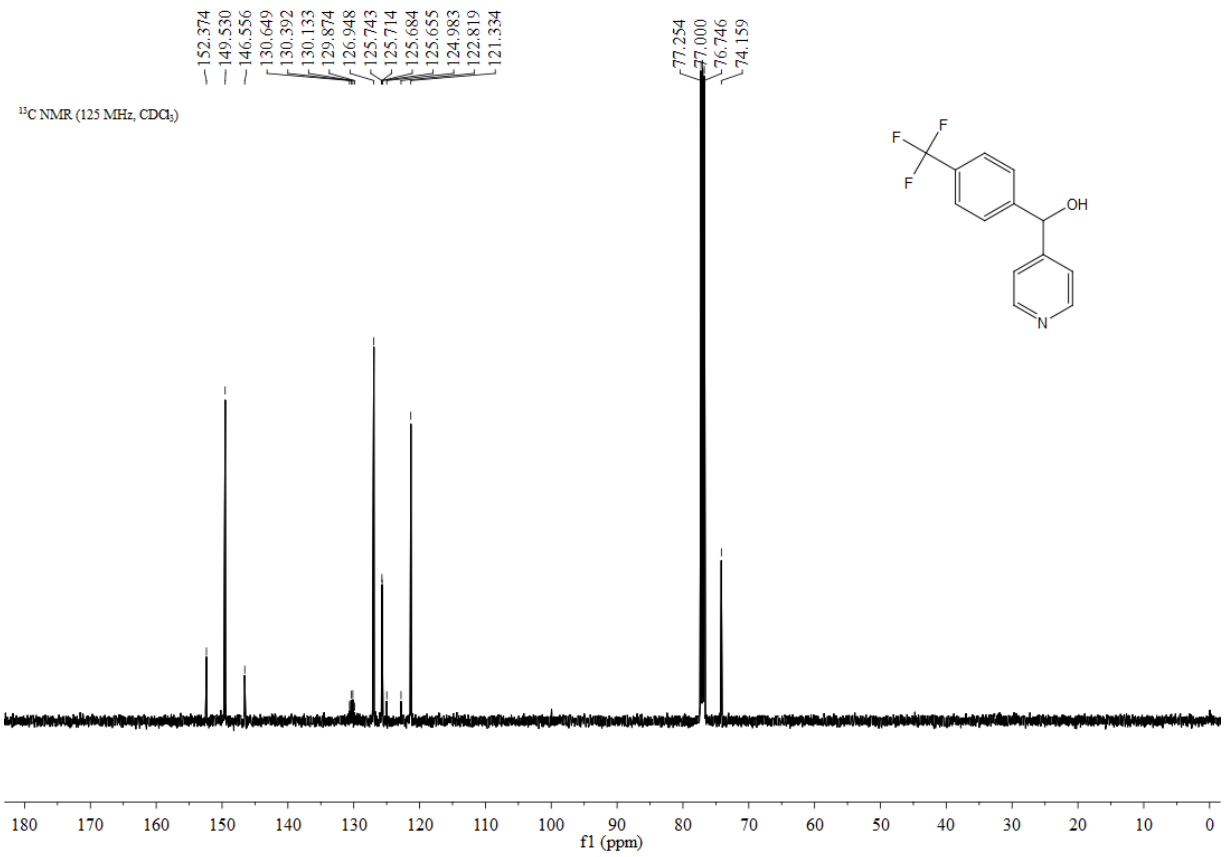
(4-chlorophenyl)(pyridin-4-yl)methanol (3da)





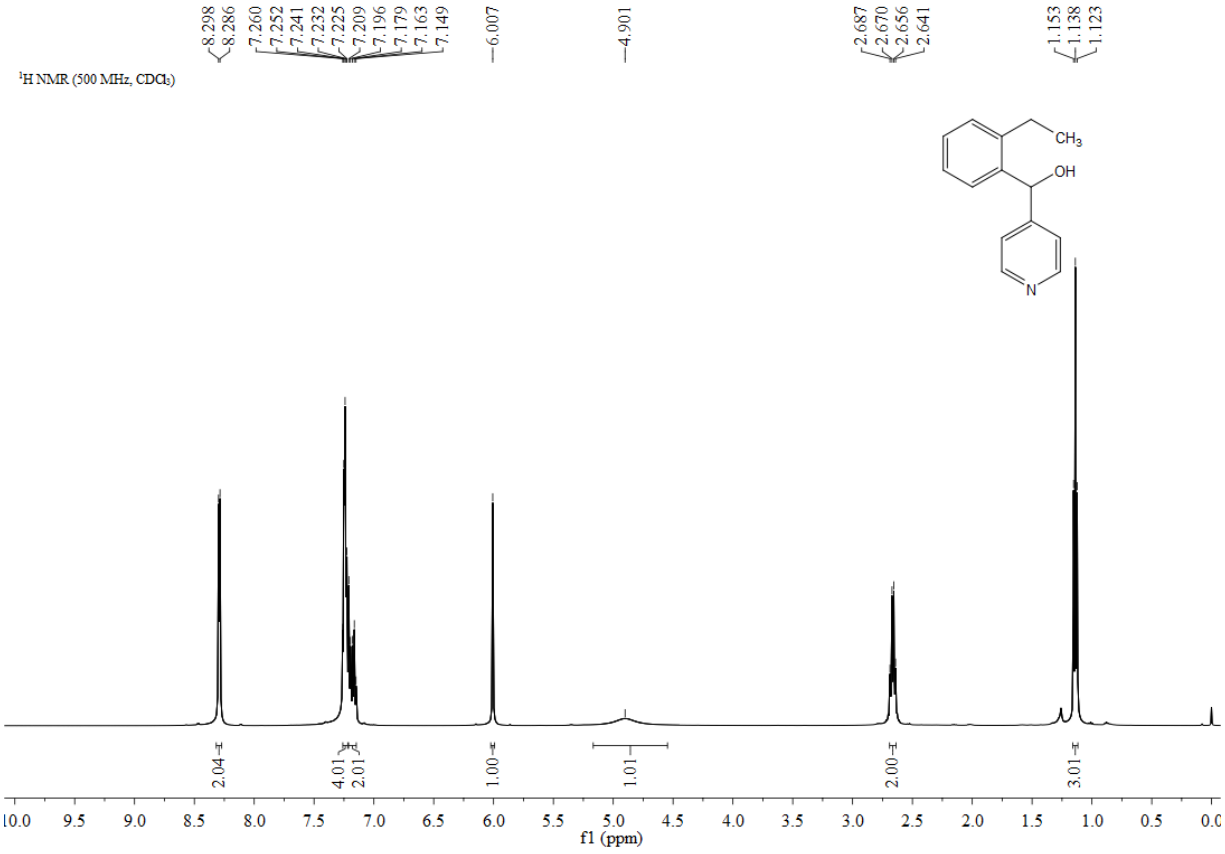
Pyridin-4-yl(4-(trifluoromethyl)phenyl)methanol (3ea)

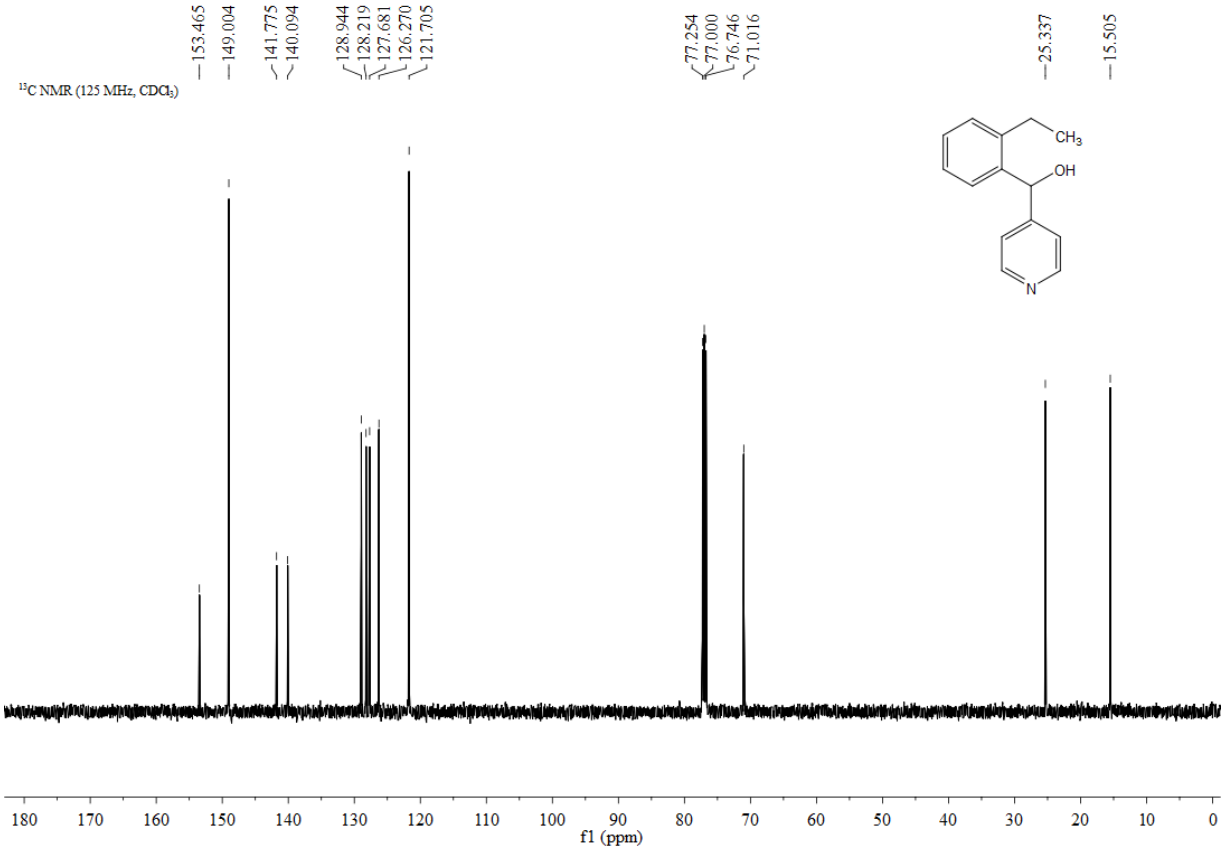




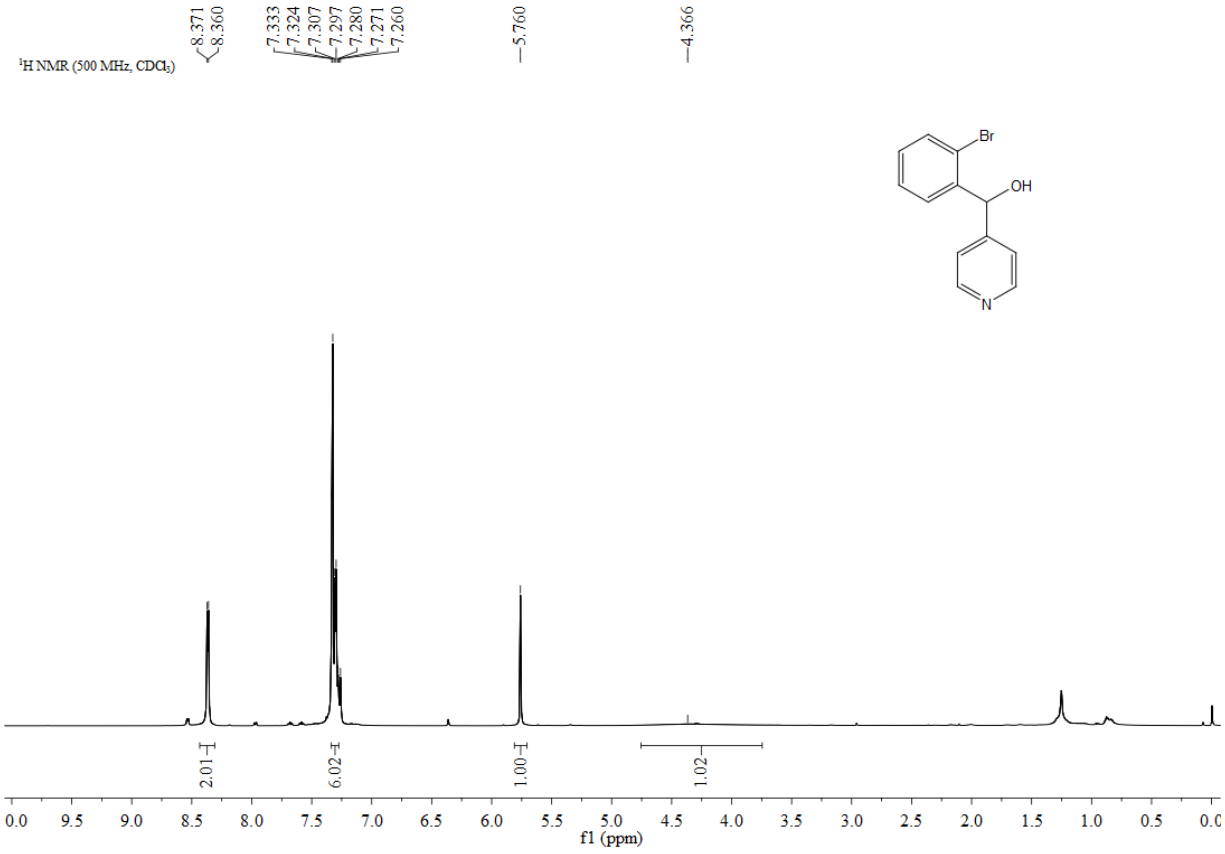
^{19}F NMR (471 MHz, $CDCl_3$)

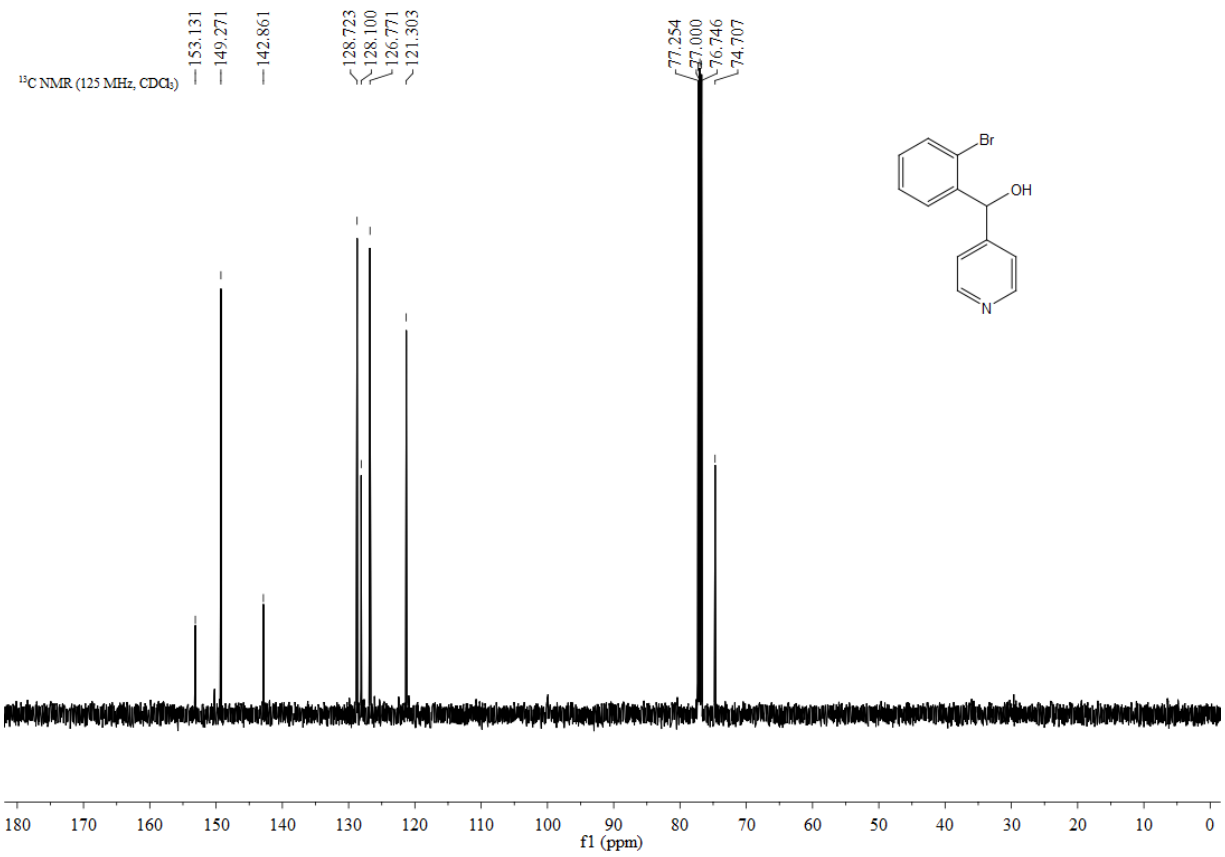
(2-ethylphenyl)(pyridin-4-yl)methanol (3fa)



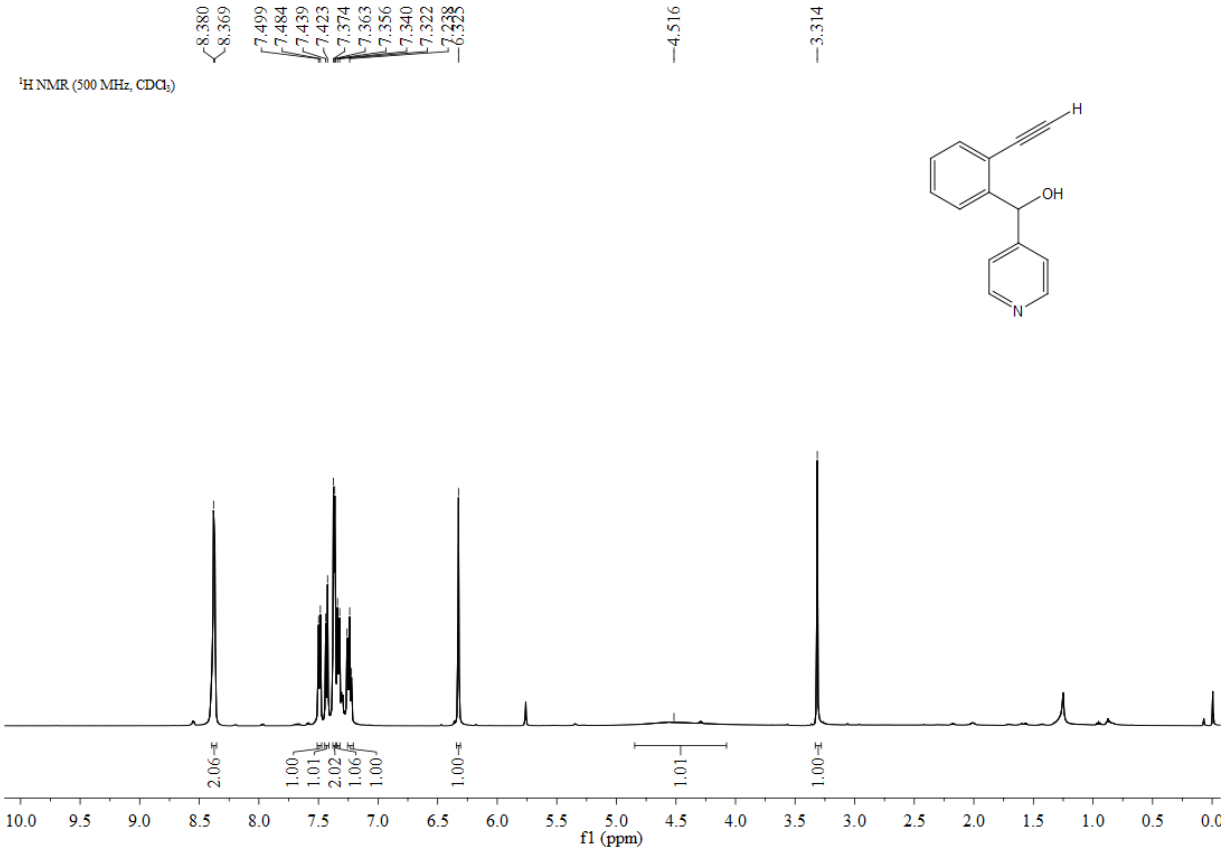


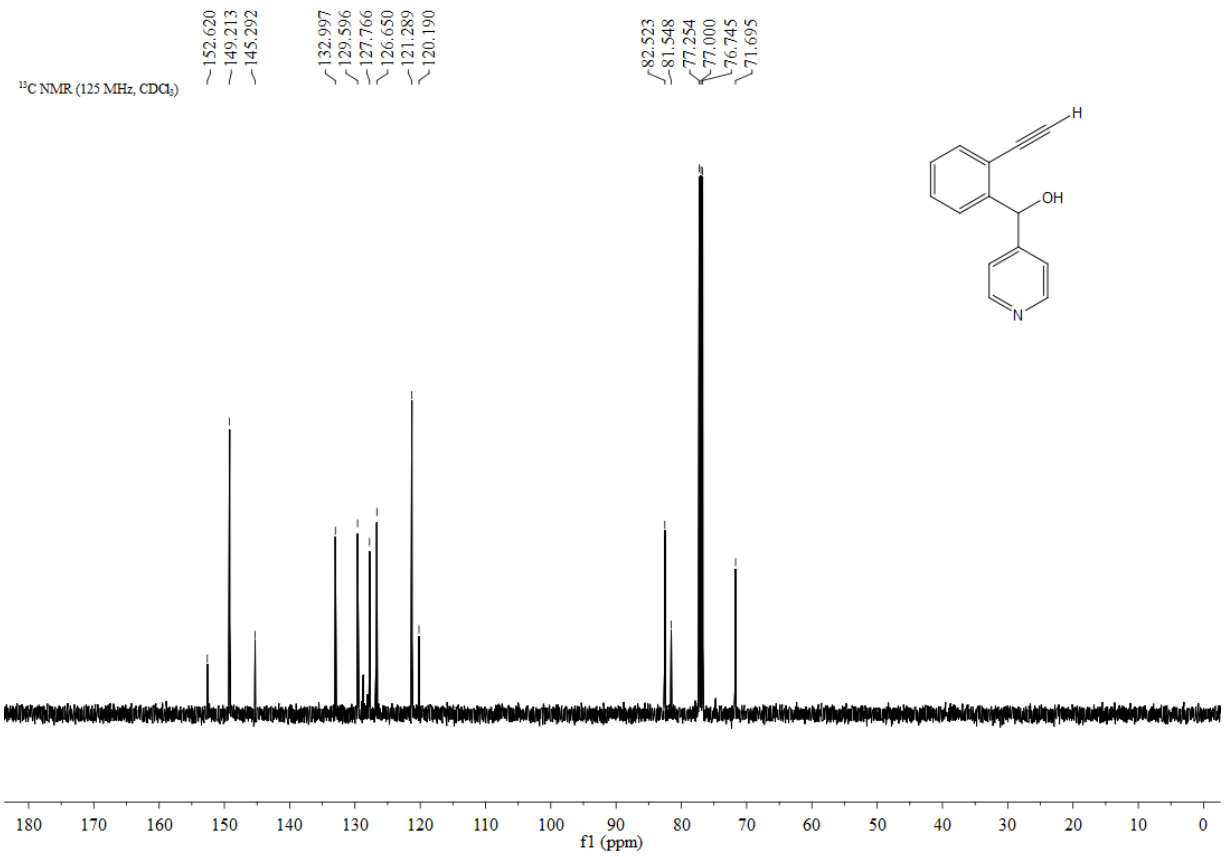
(2-bromophenyl)(pyridin-4-yl)methanol (3ga)



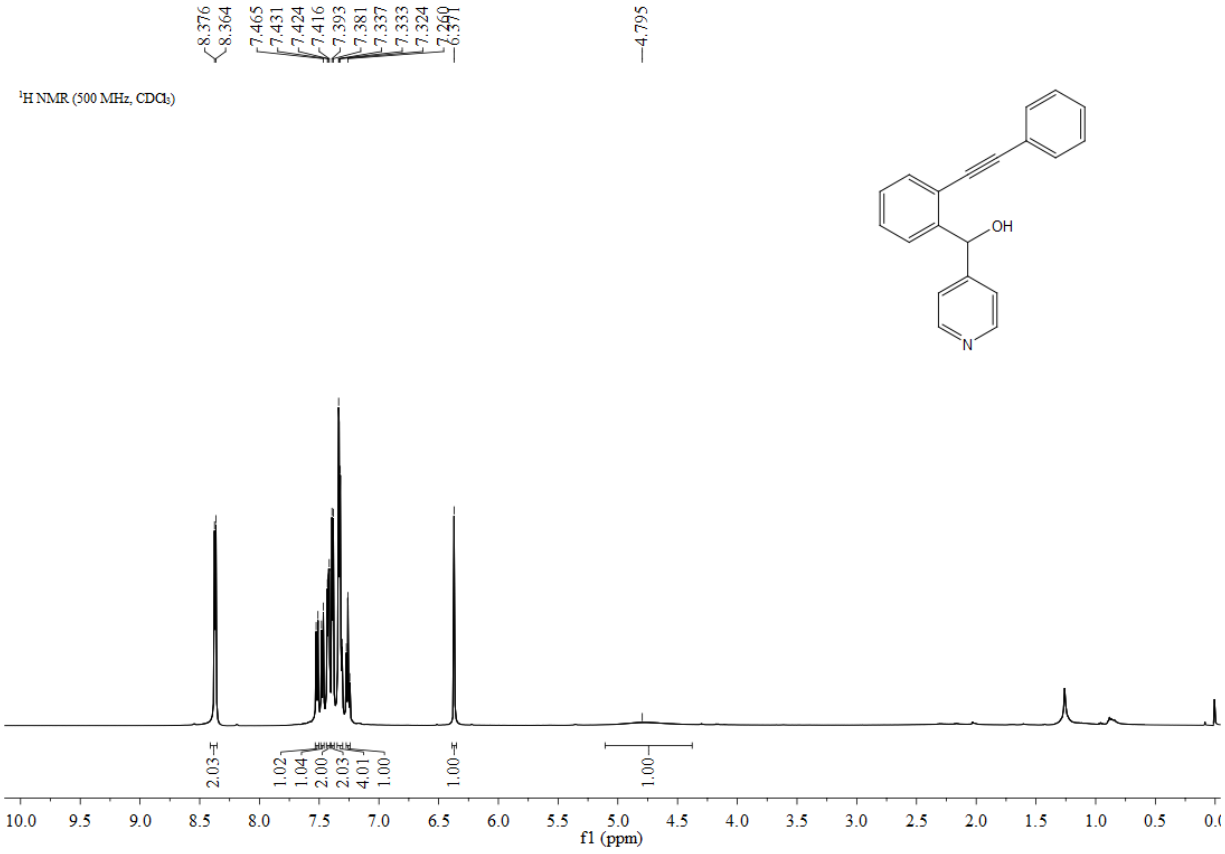


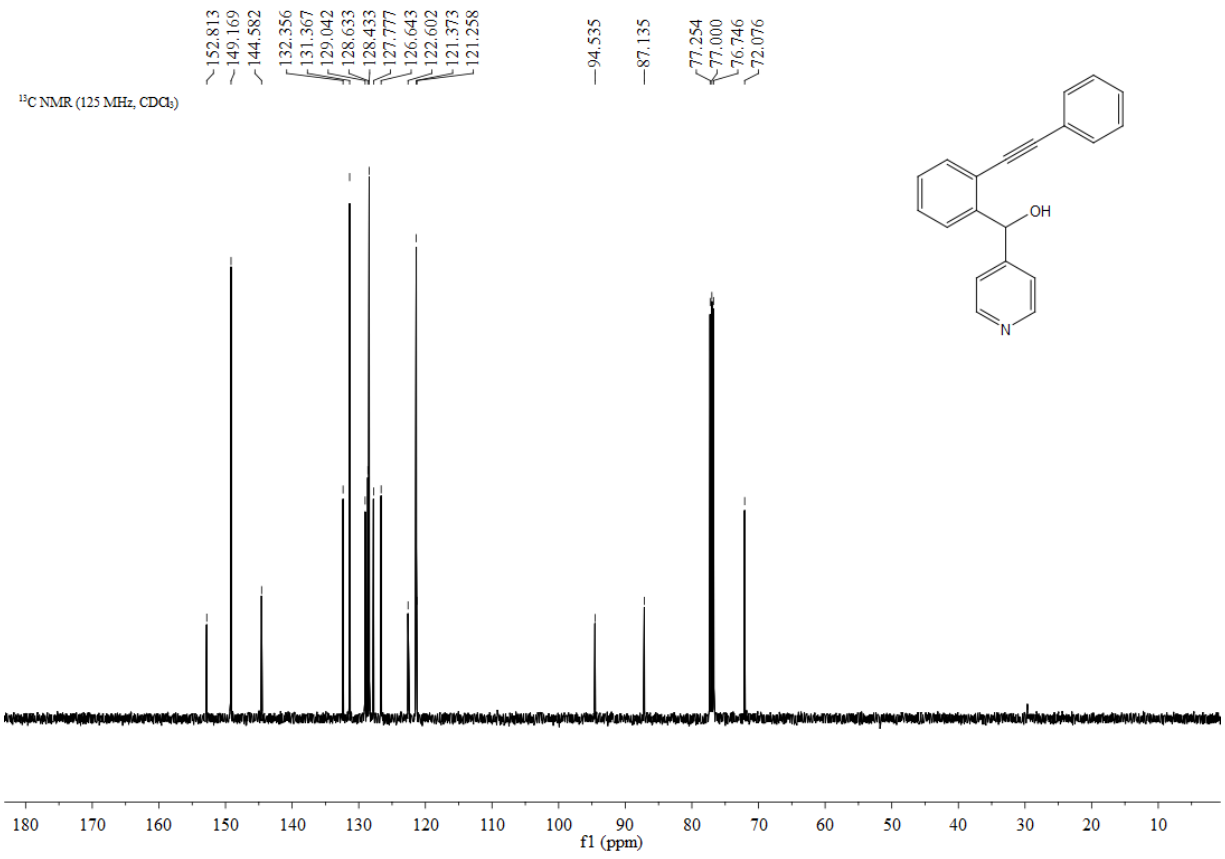
(2-ethynylphenyl)(pyridin-4-yl)methanol (3ha)



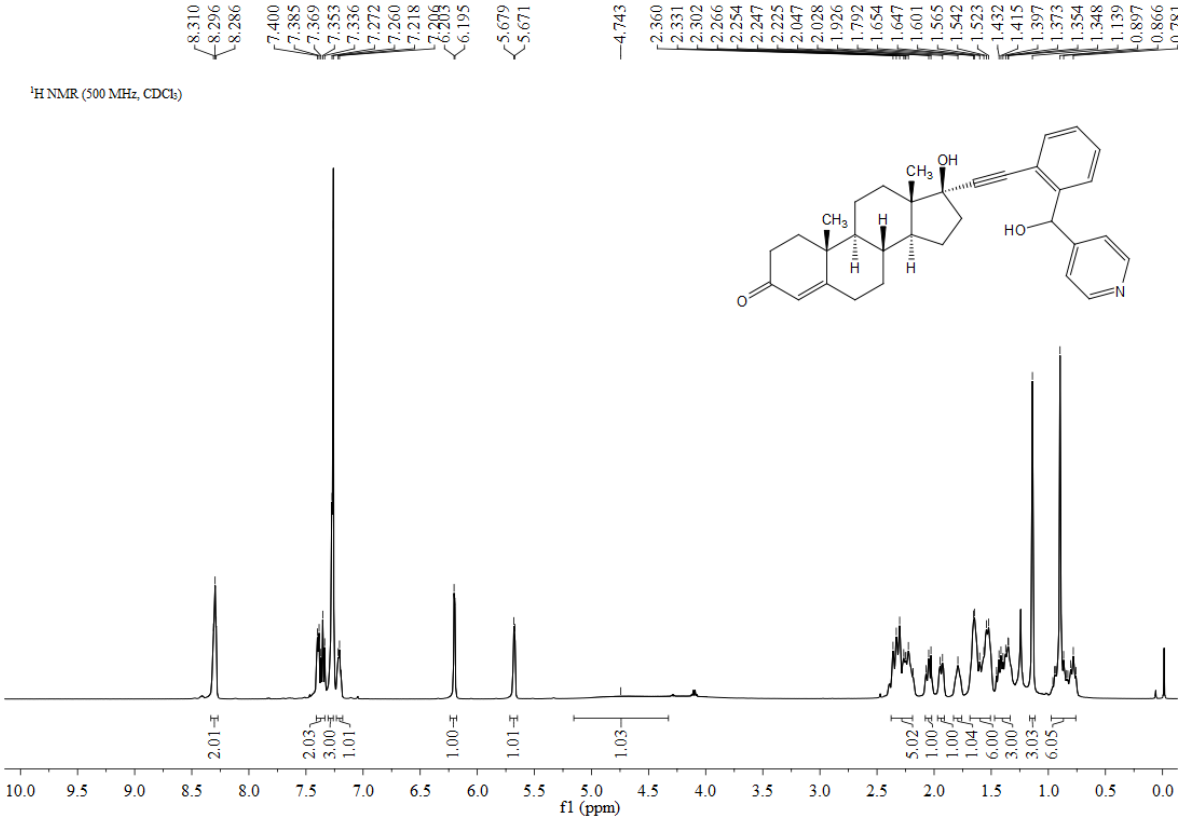


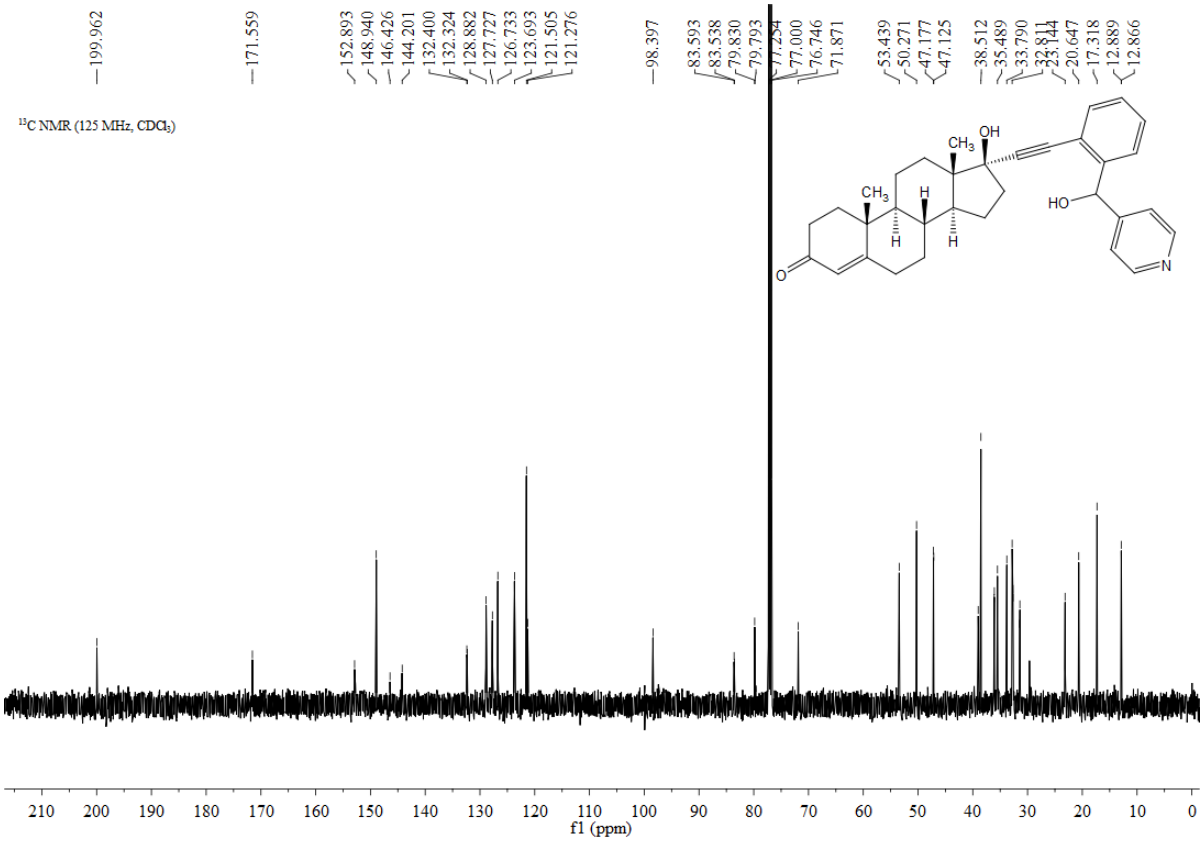
(2-(phenylethynyl)phenyl)(pyridin-4-yl)methanol (3ia)



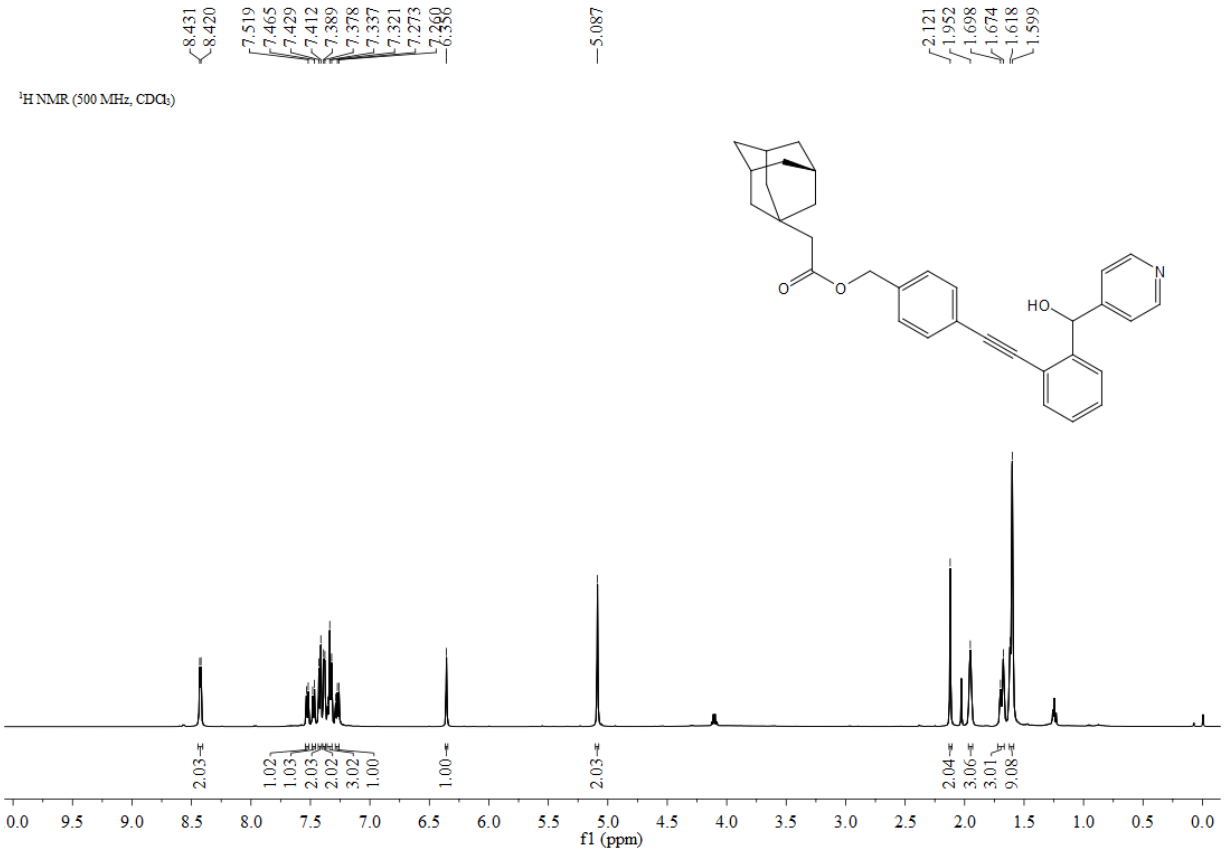


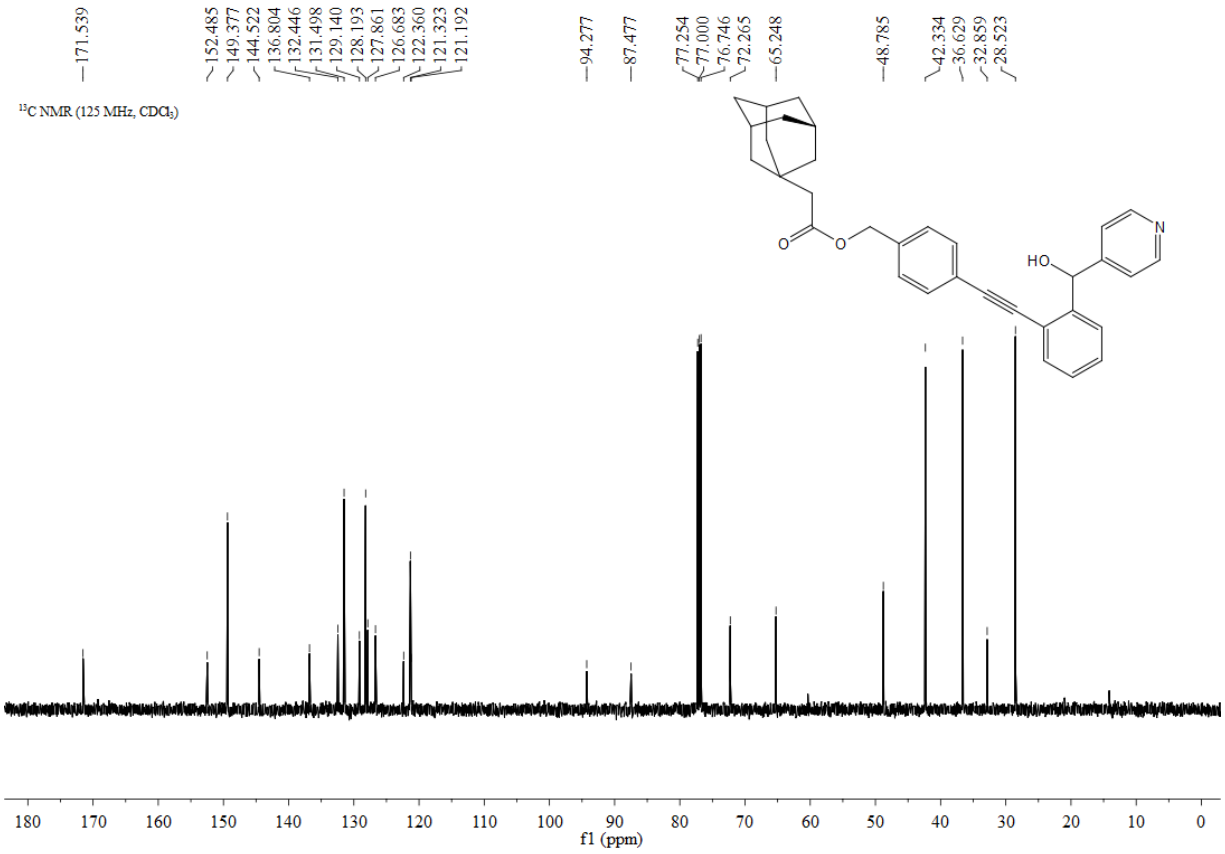
(*8R,9S,10R,13S,14S,17S*)-17-hydroxy-17-((2-(hydroxy(pyridin-4-yl)methyl)phenyl)ethynyl)-10,13-dimethyl-1,2,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-3H-cyclopenta[a]phenanthren-3-one (3ja)



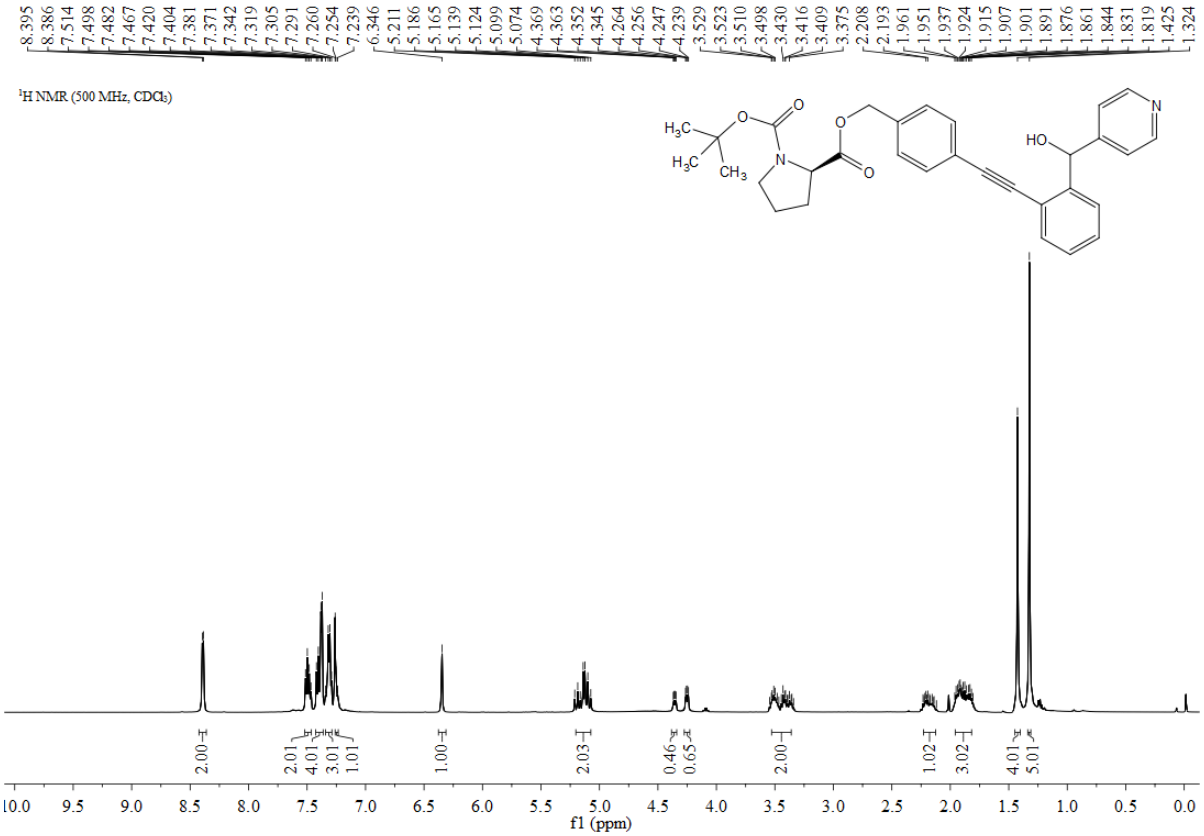


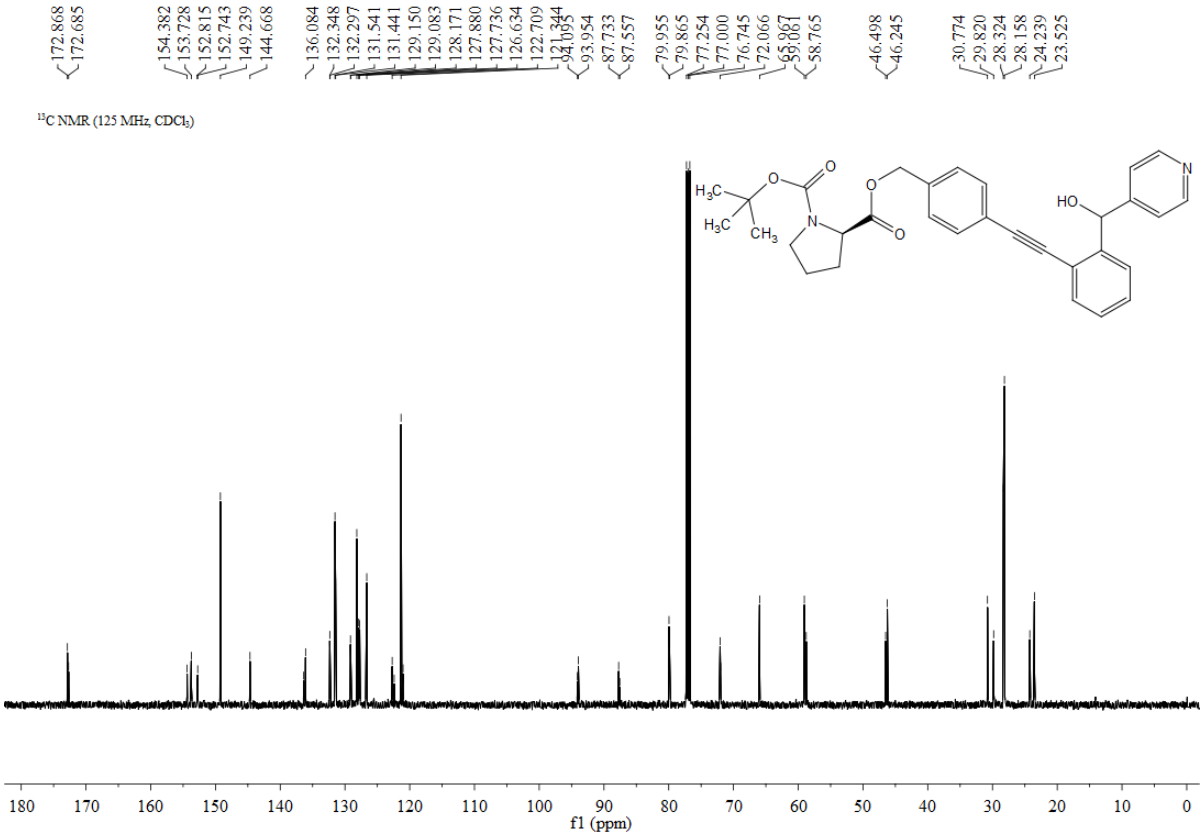
4-((2-(hydroxy(pyridin-4-yl)methyl)phenyl)ethynyl)benzyl-2-((*3r,5r,7r*)-adamanta n -1-yl)acetate (3ka)



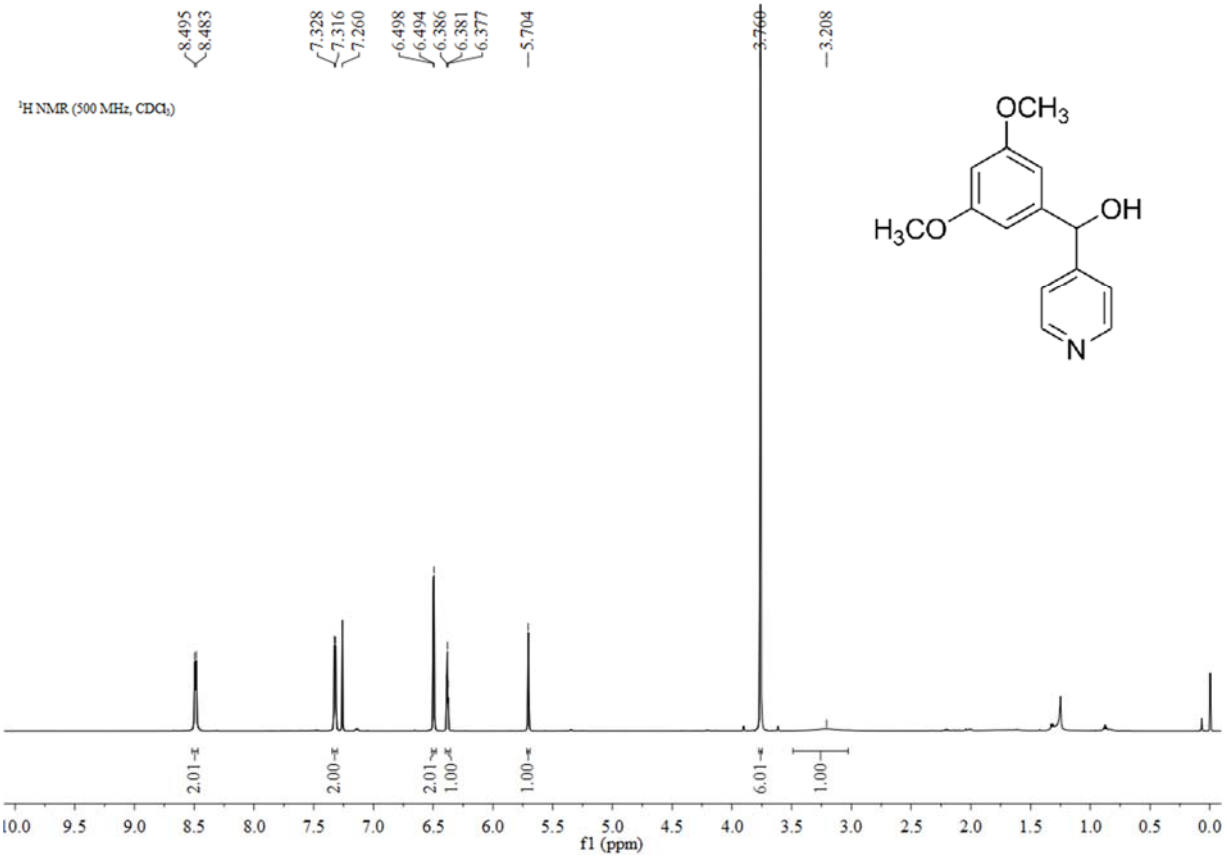


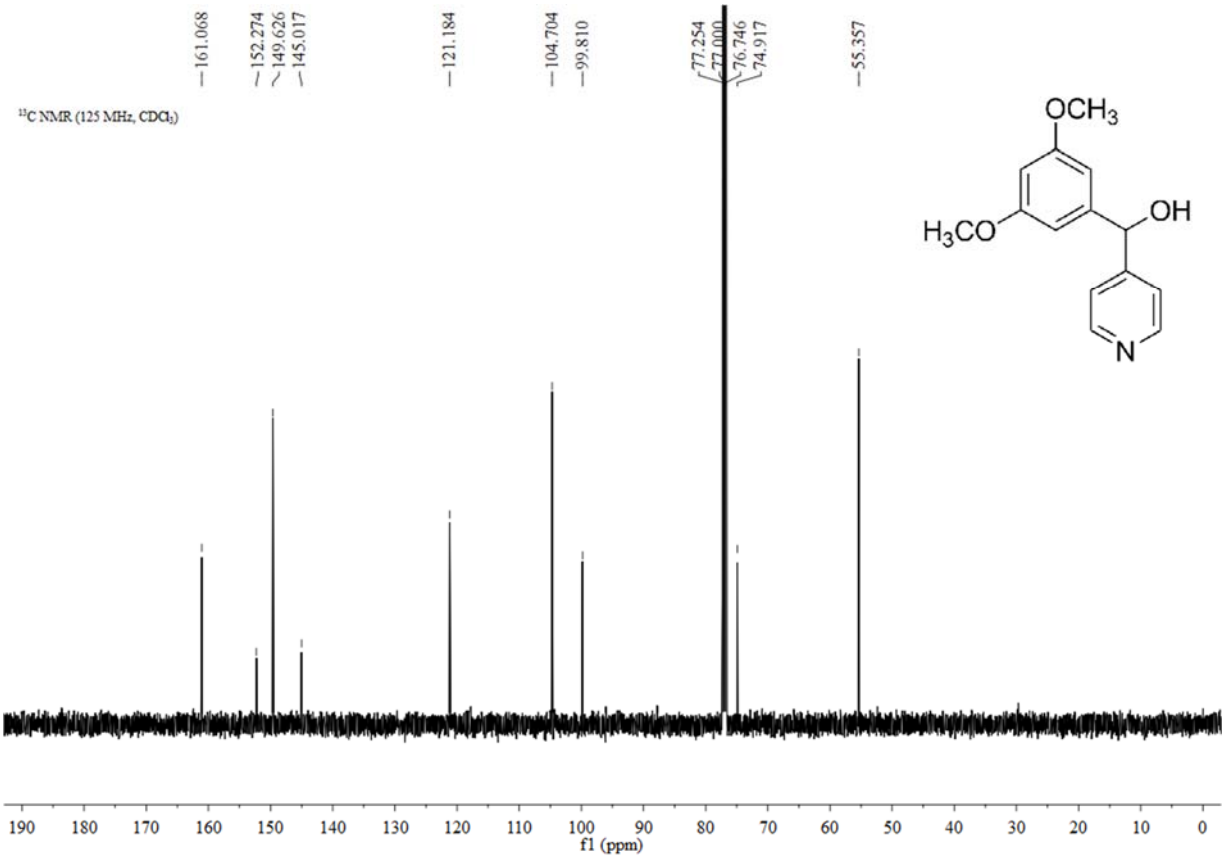
1-(tert-butyl) 2-(4-((2-(hydroxyl (pyridin-4-yl)methyl)phenyl)ethynyl)benzyl) (*2R*)-pyrrolidine-1,2-dicarboxylate (3la)



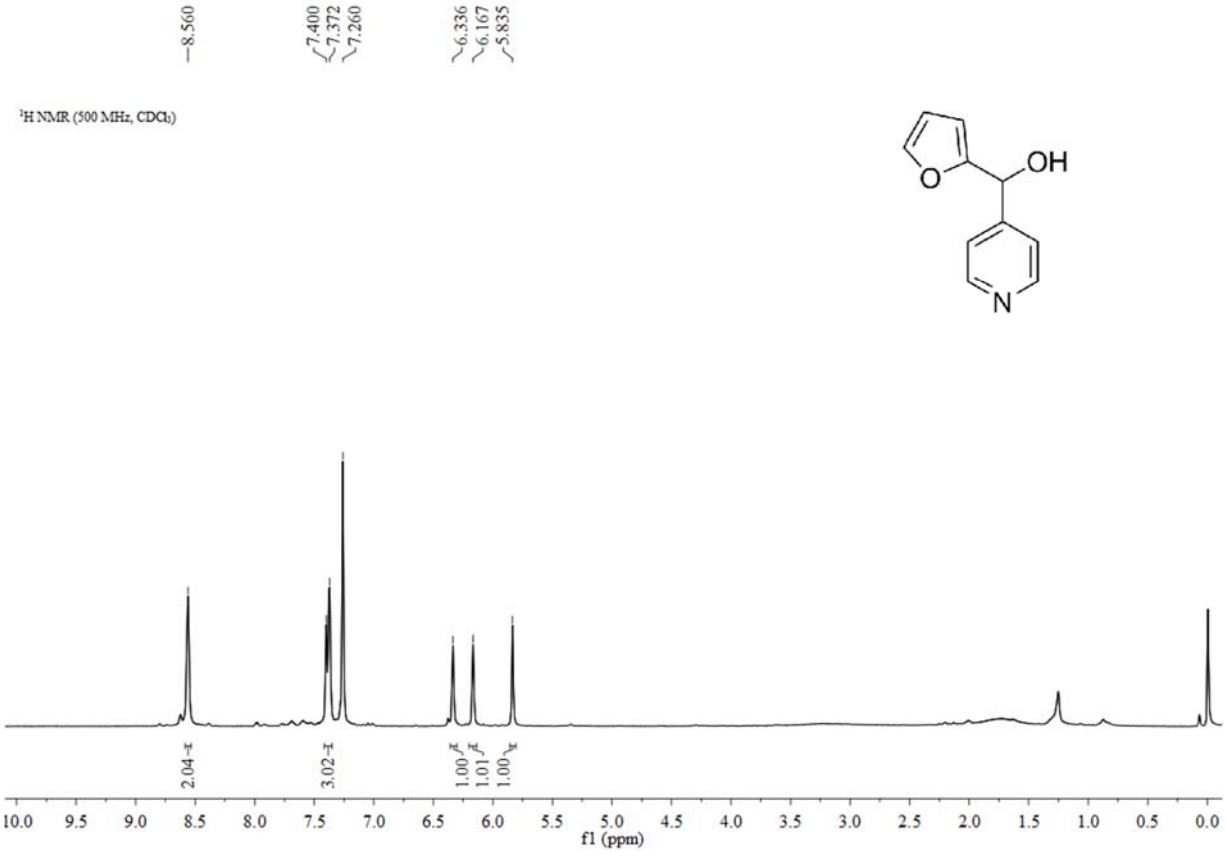


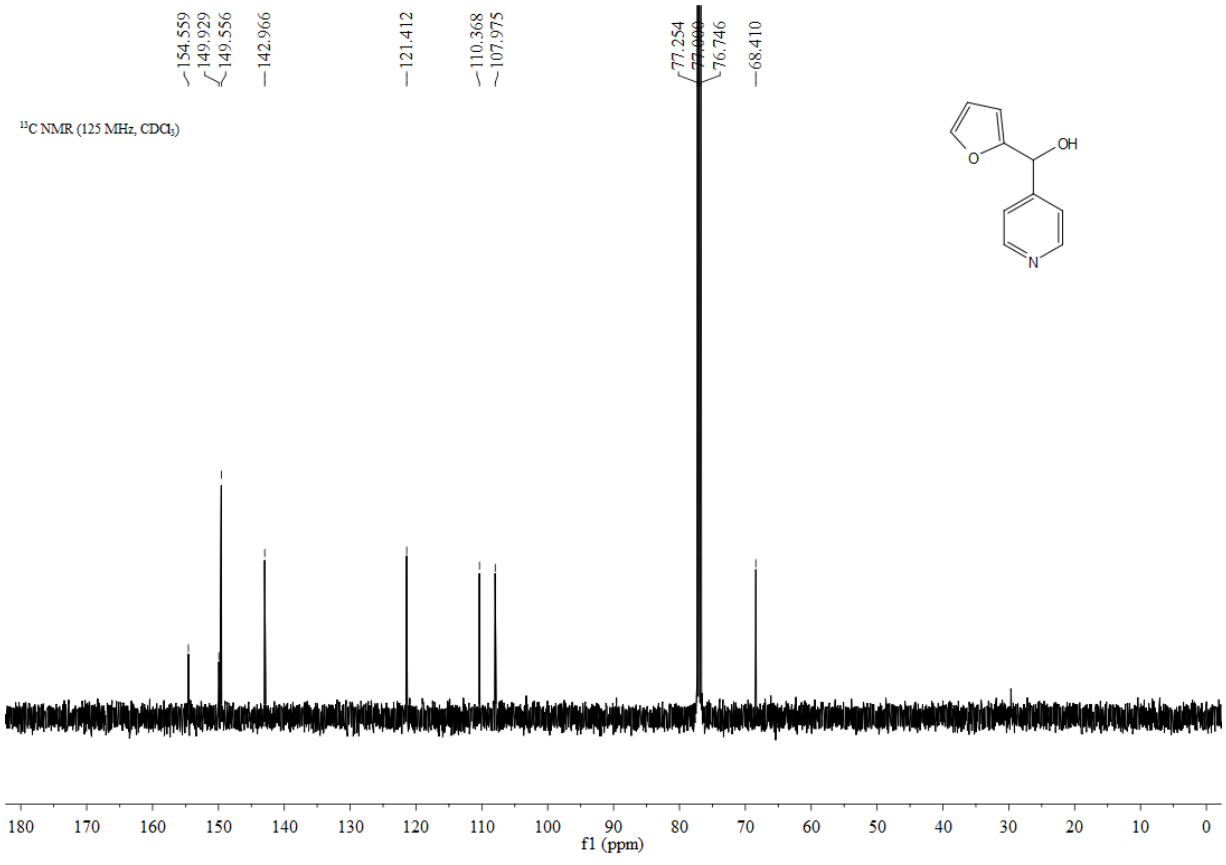
(3,5-dimethoxyphenyl)(pyridin-4-yl)methanol (3ma)



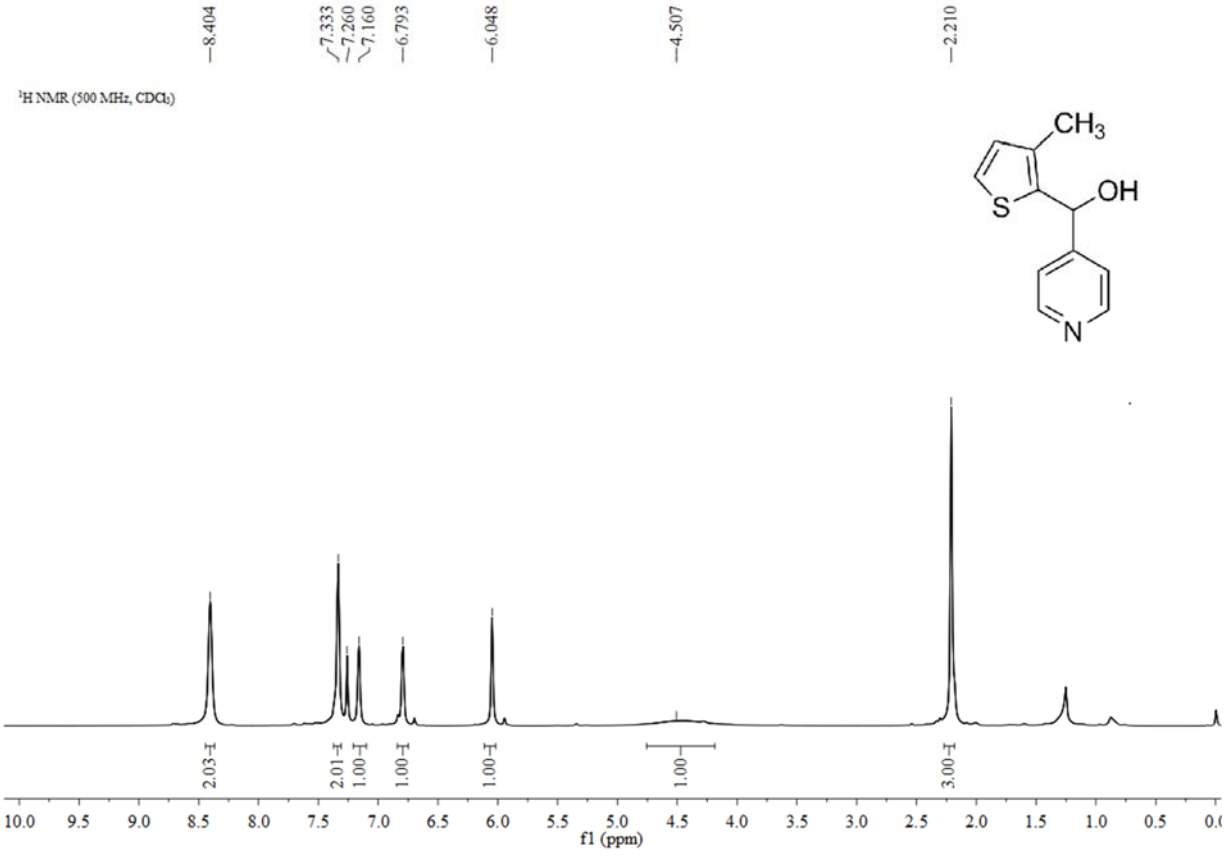


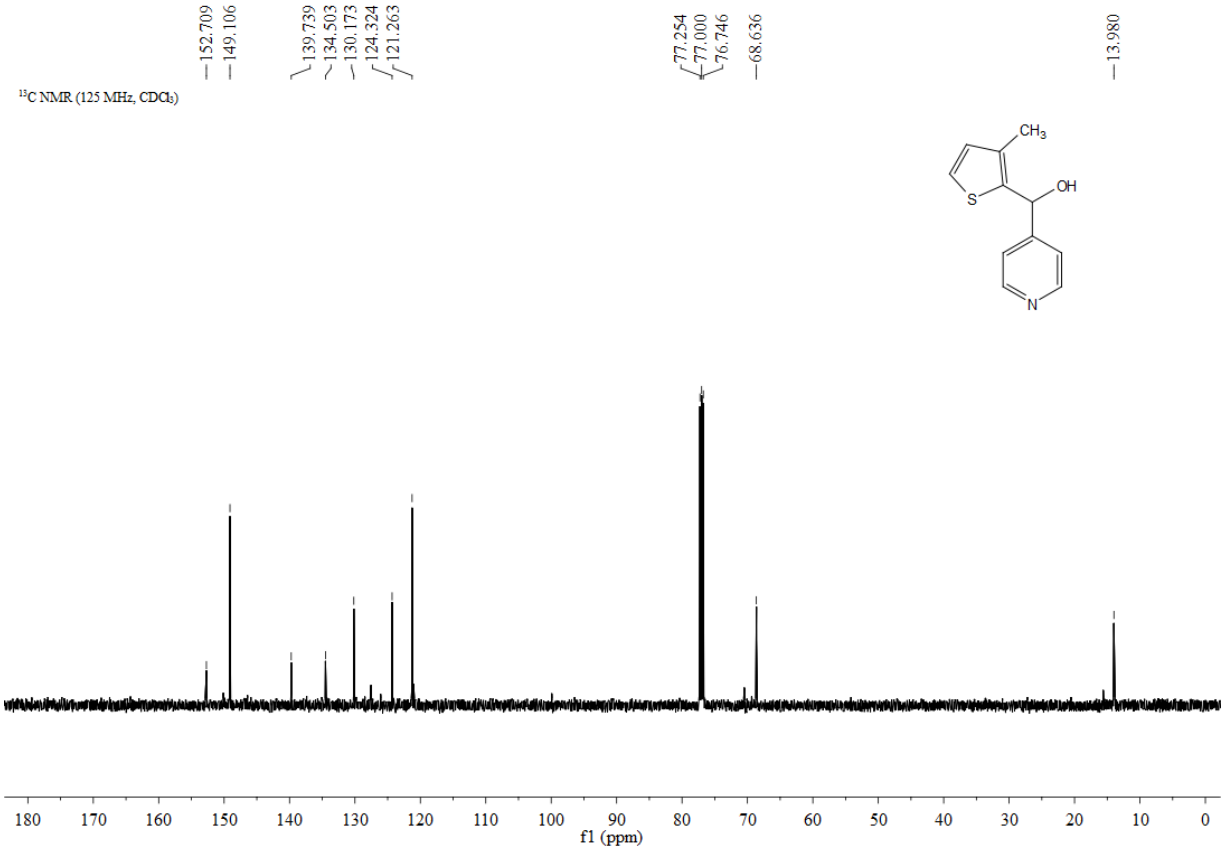
Furan-2-yl(pyridin-4-yl)methanol (3na)



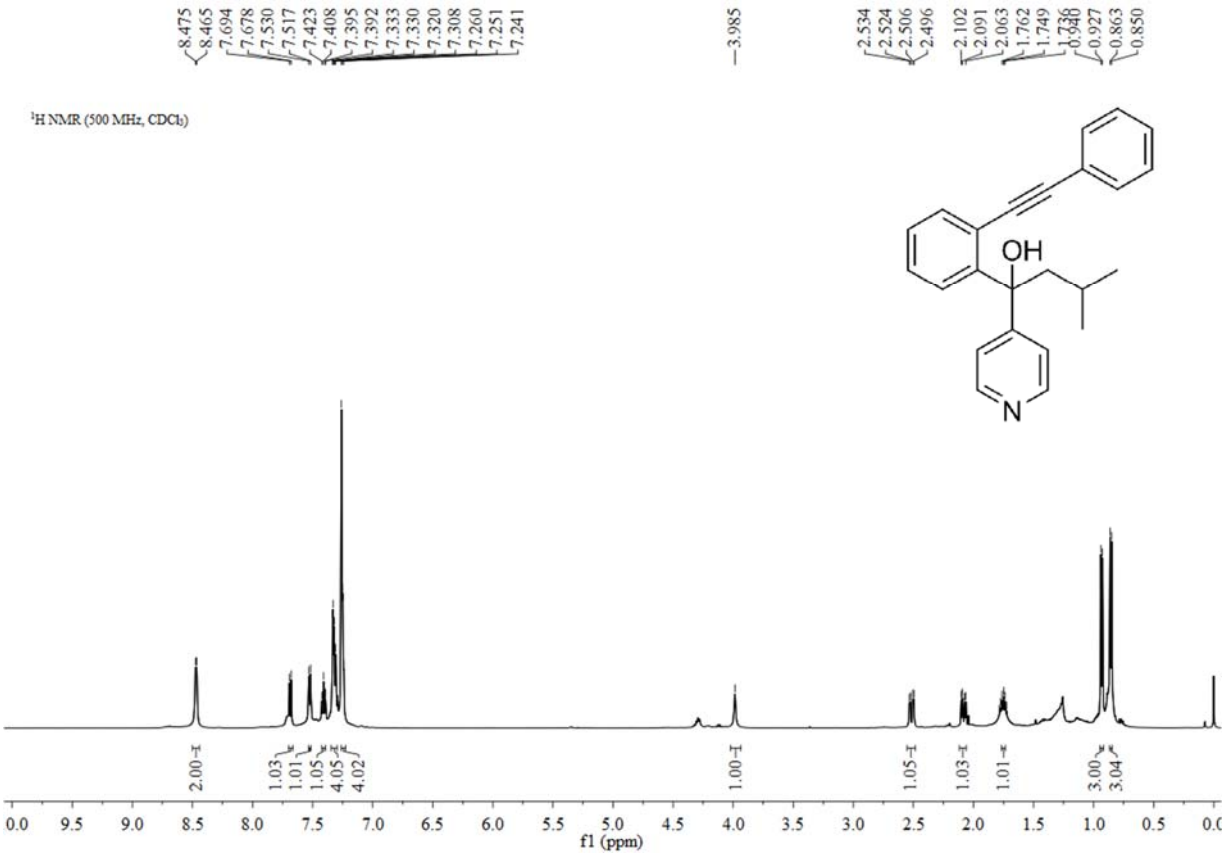


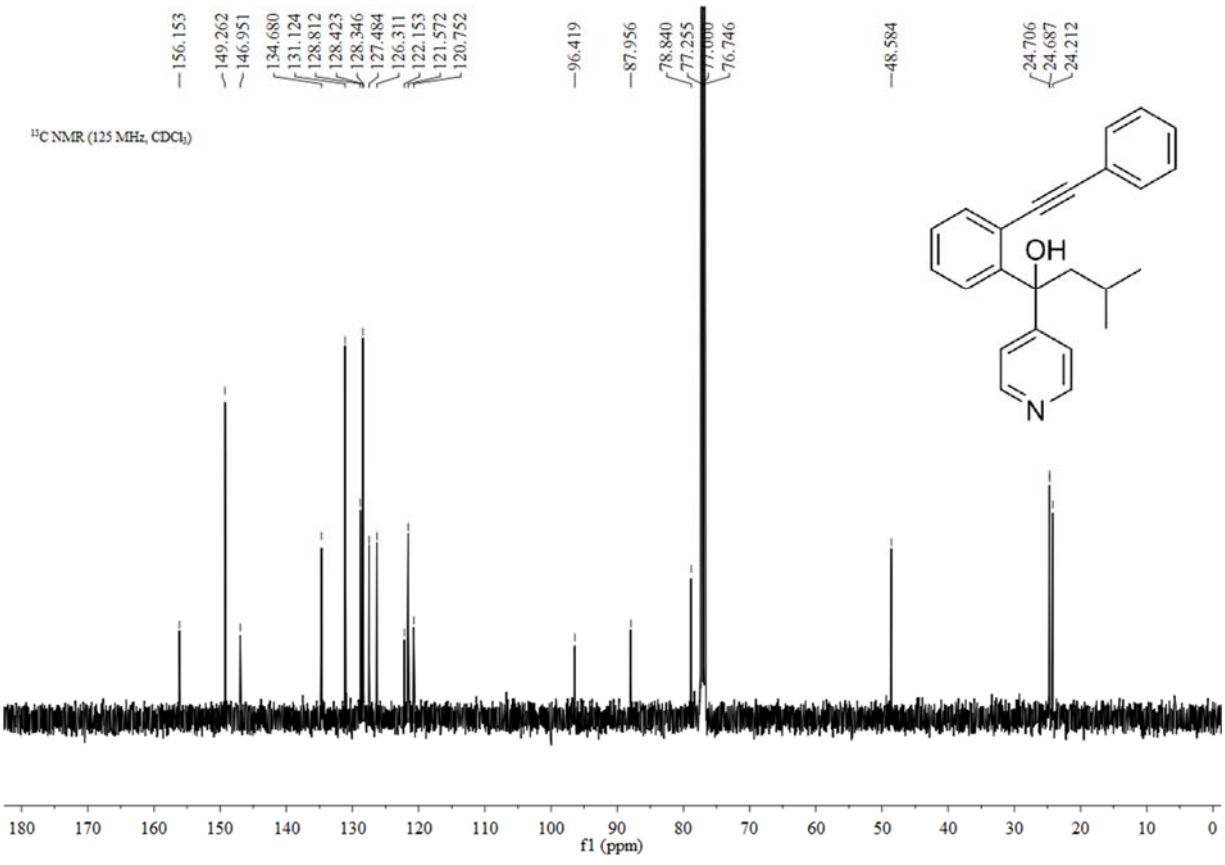
(3-methylthiophen-2-yl)(pyridin-4-yl)methanol (3oa)



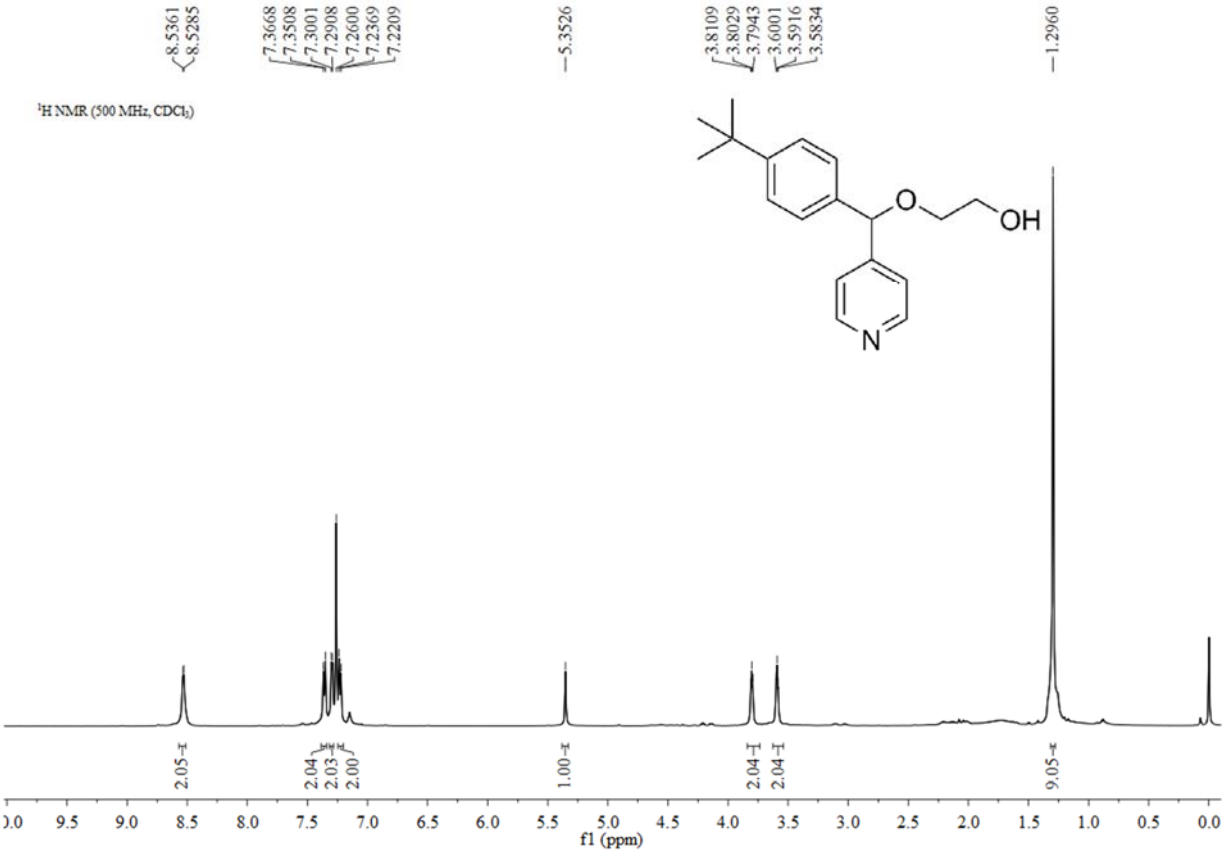


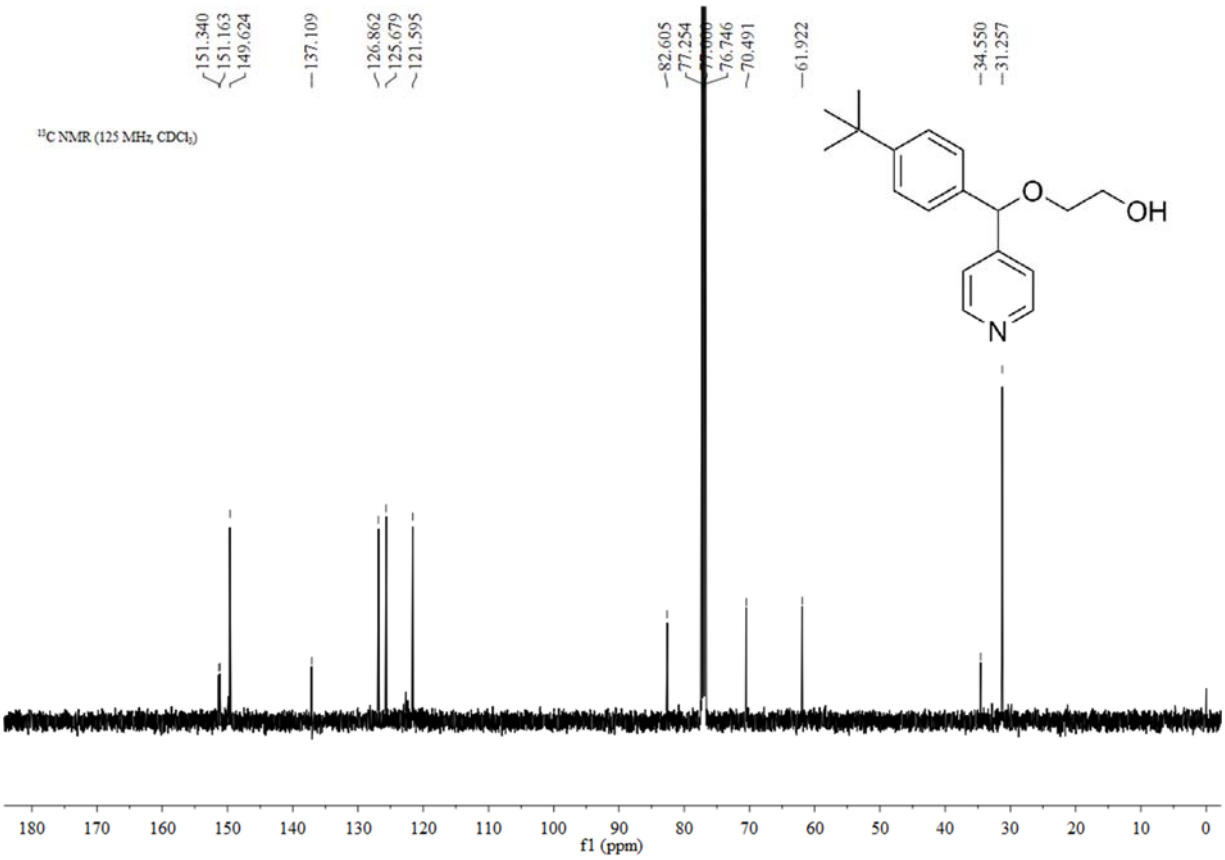
3-methyl-1-(2-(phenylethynyl)phenyl)-1-(pyridin-4-yl)butan-1-ol (3pa):



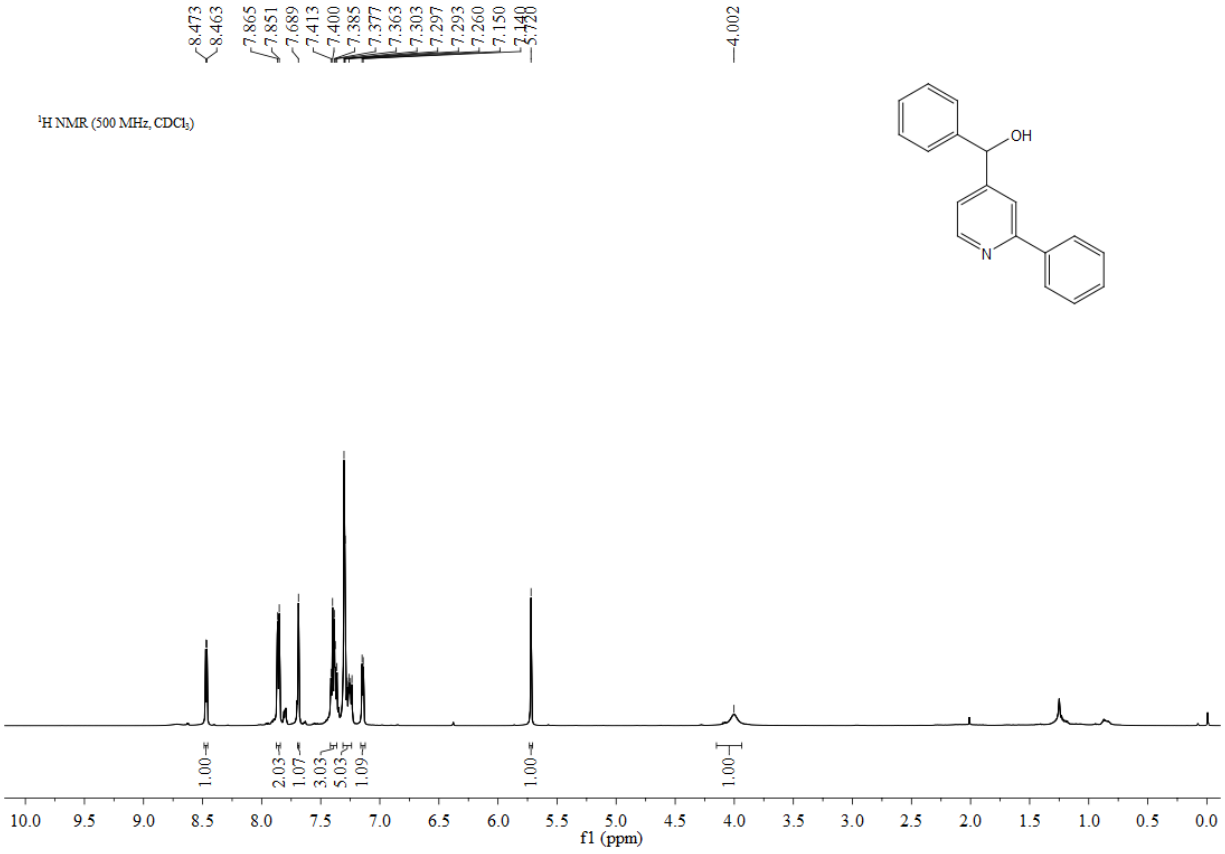


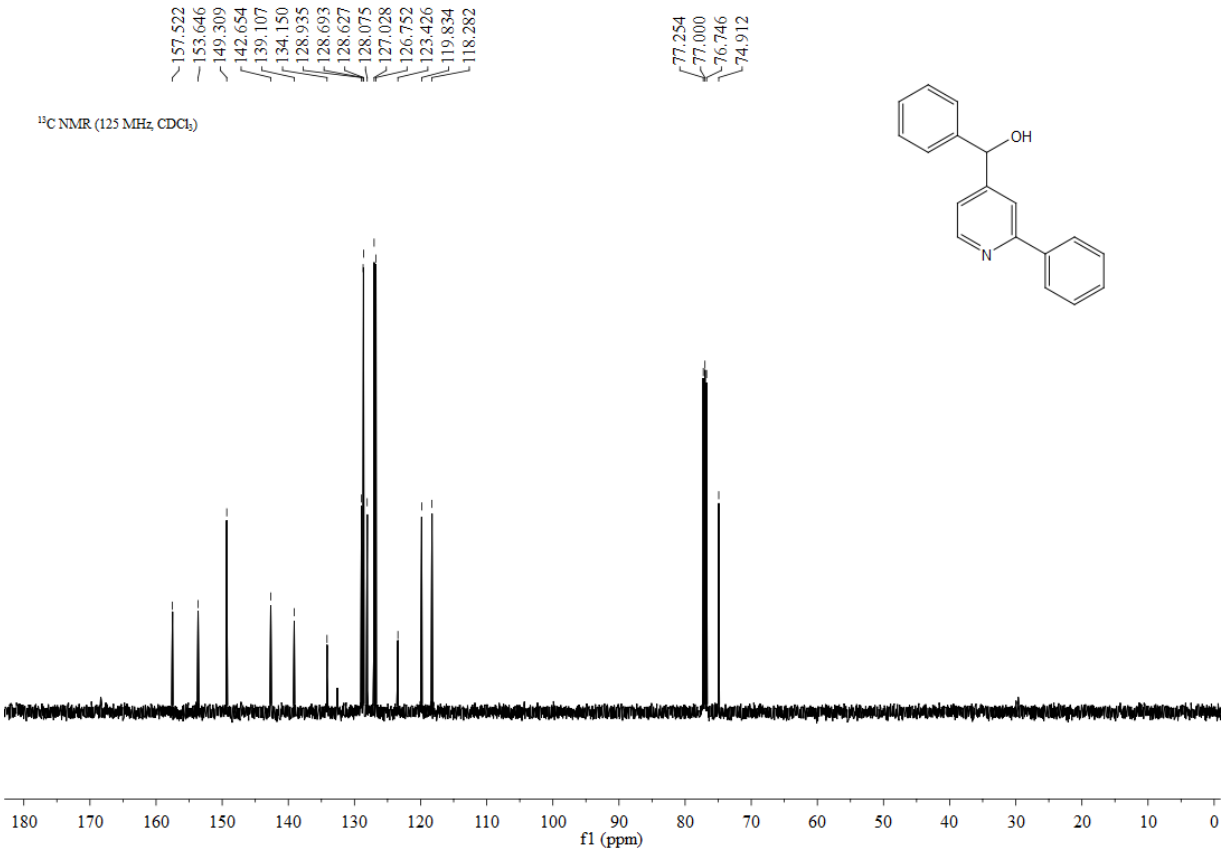
2-((4-(tert-butyl)phenyl)(pyridin-4-yl)methoxy)ethan-1-ol (3ra):



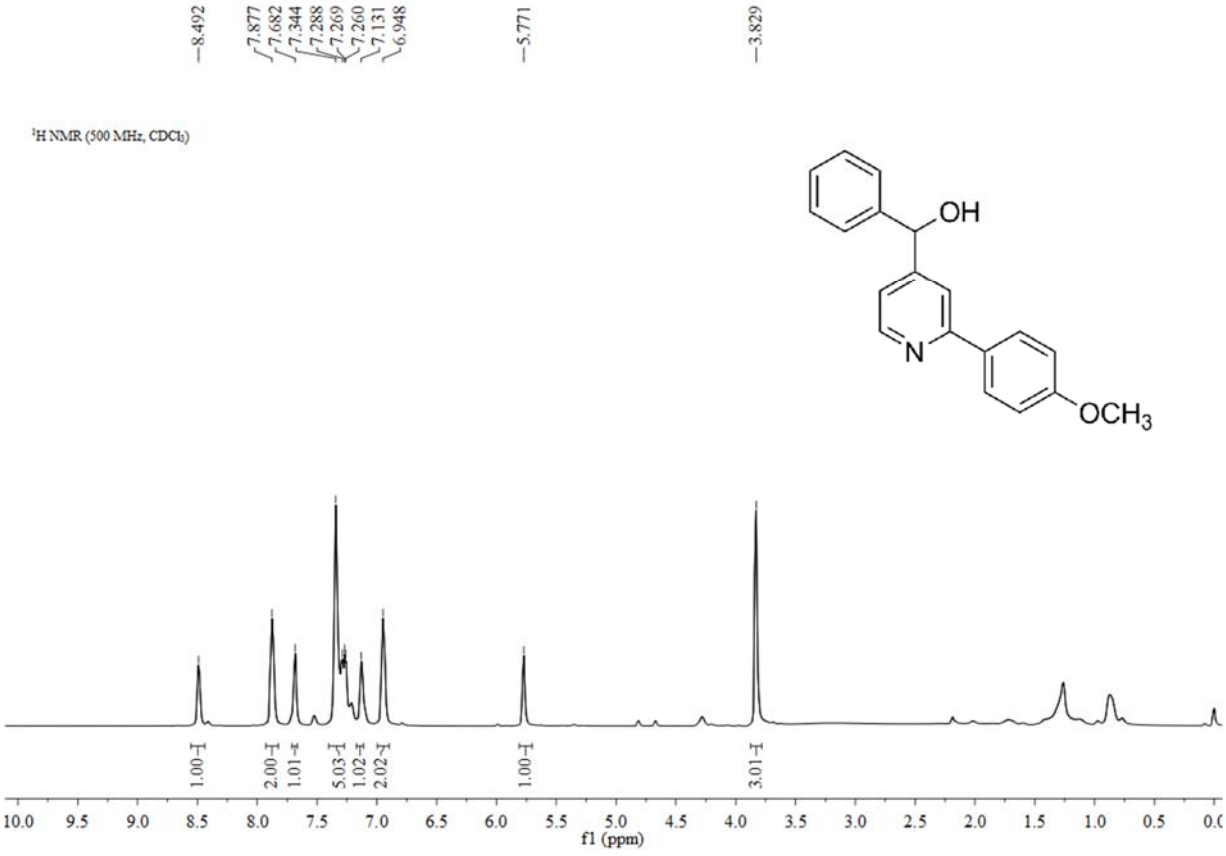


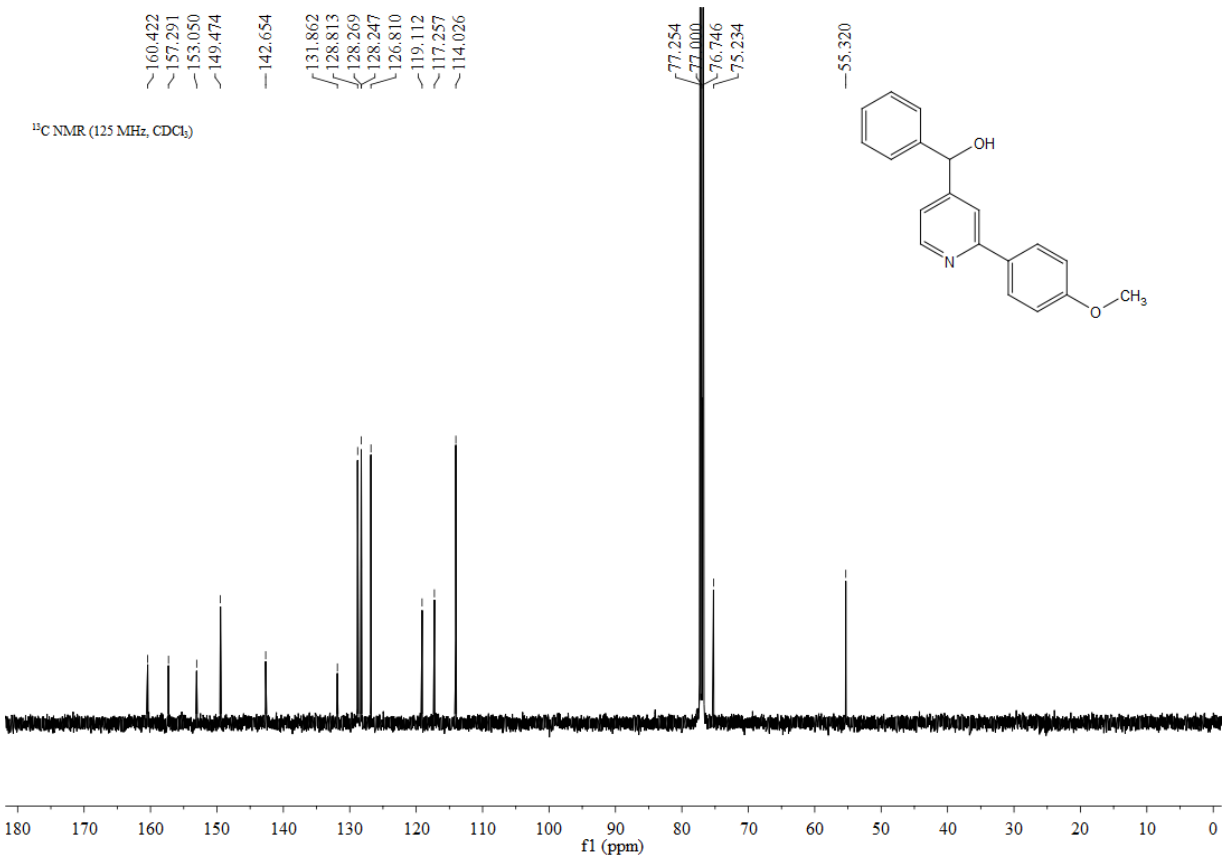
Phenyl(2-phenylpyridin-4-yl)methanol (3bb)



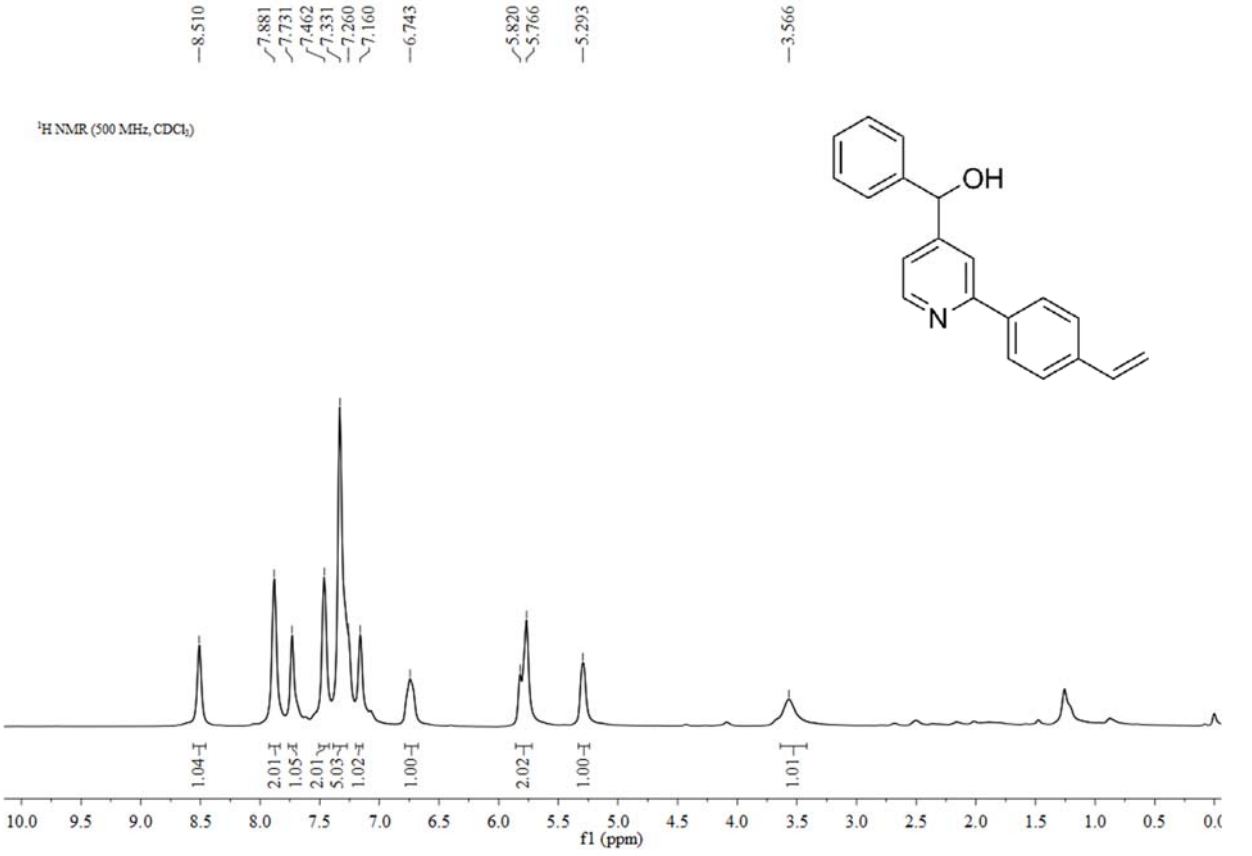


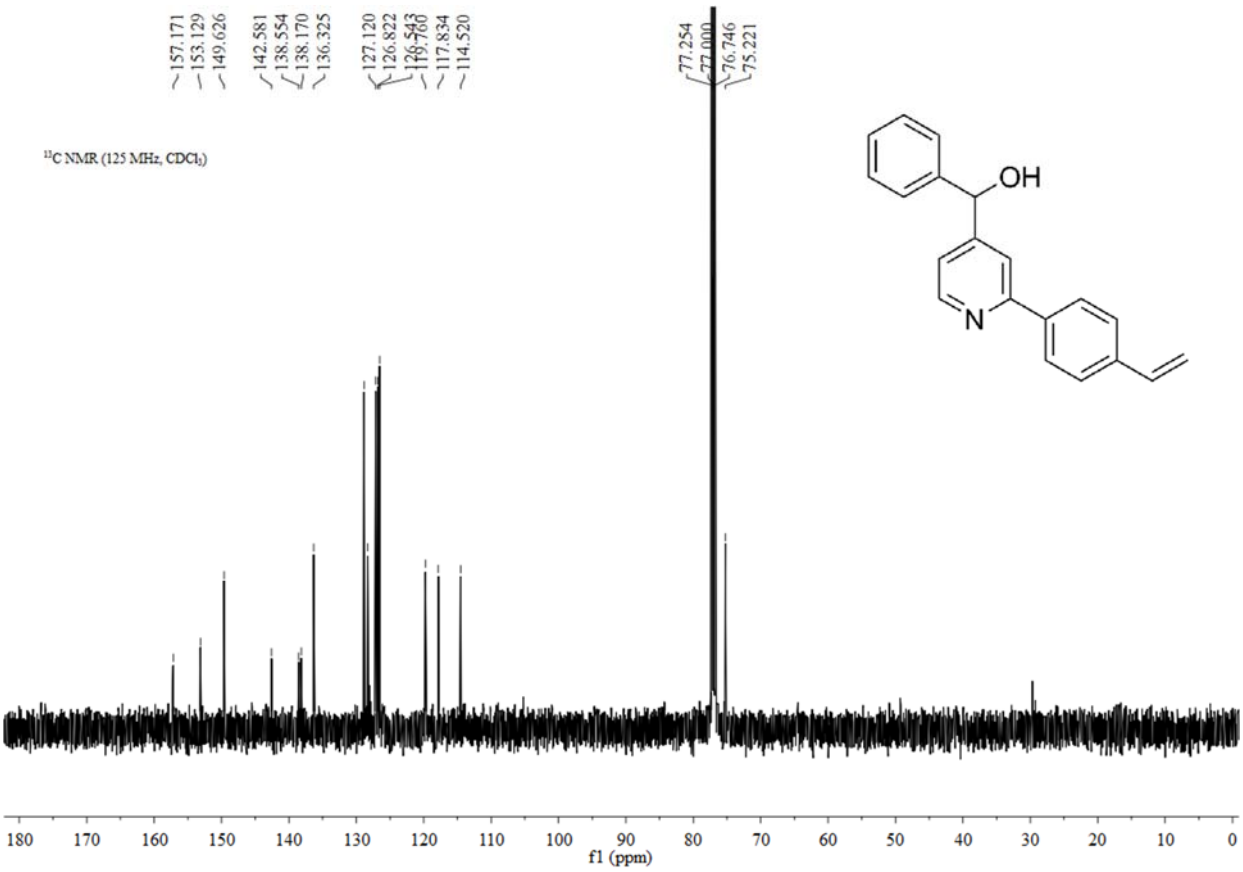
(2-(4-methoxyphenyl)pyridin-4-yl)(phenyl)methanol (3bc)



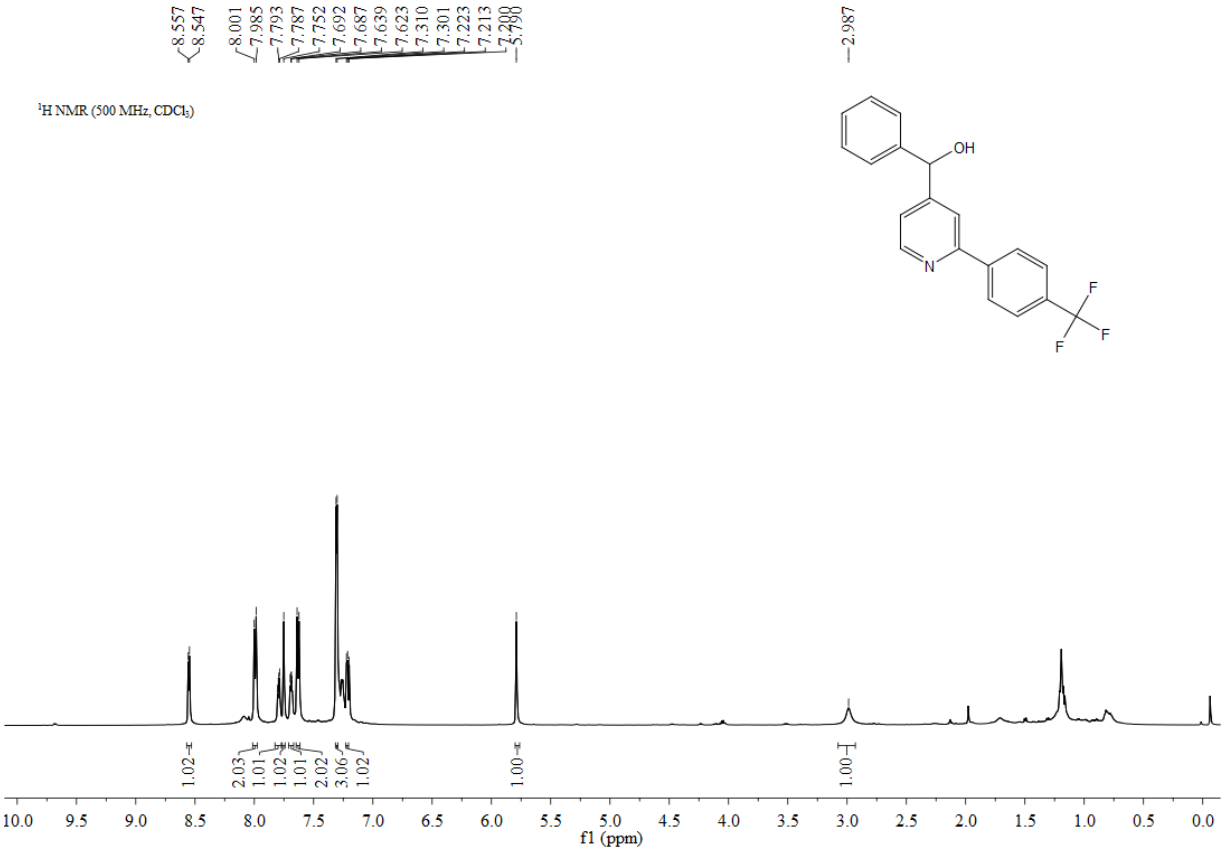


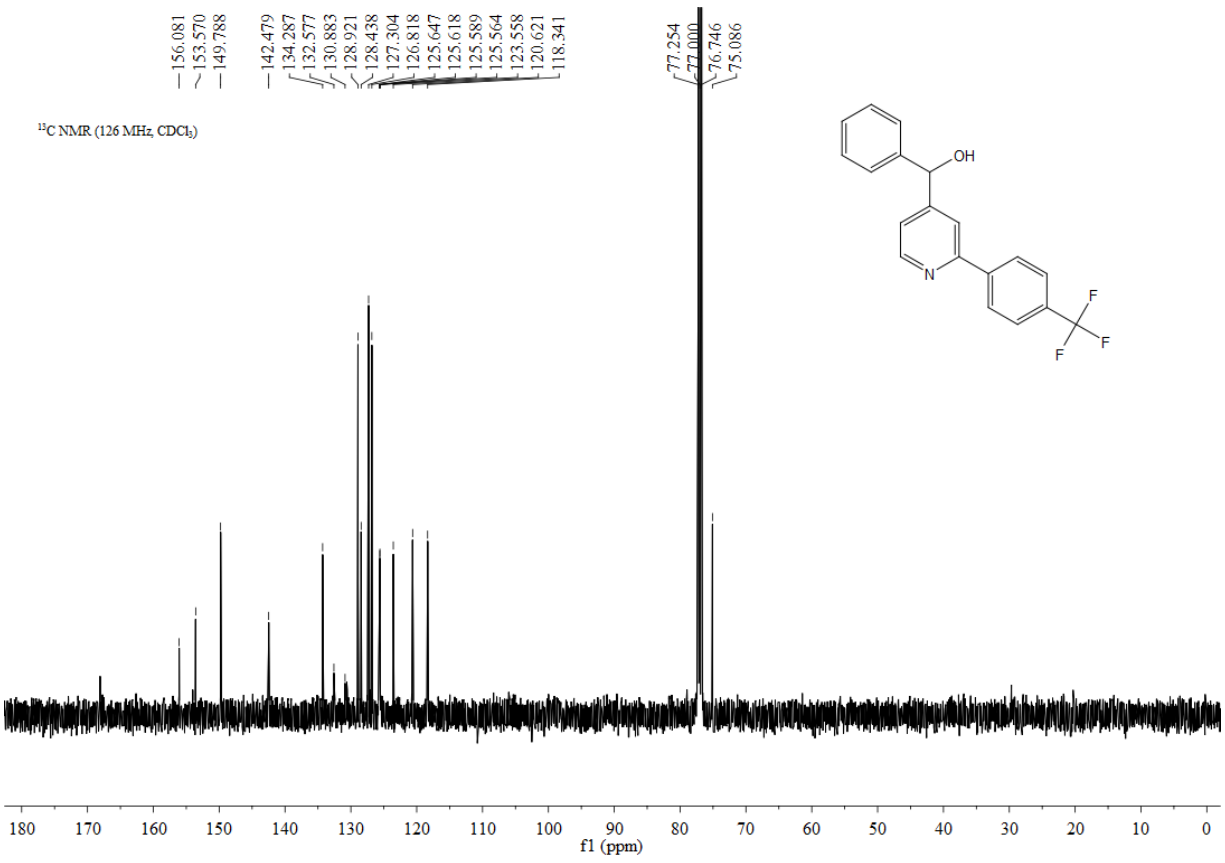
Phenyl(2-(4-vinylphenyl)pyridin-4-yl)methanol (3bd)



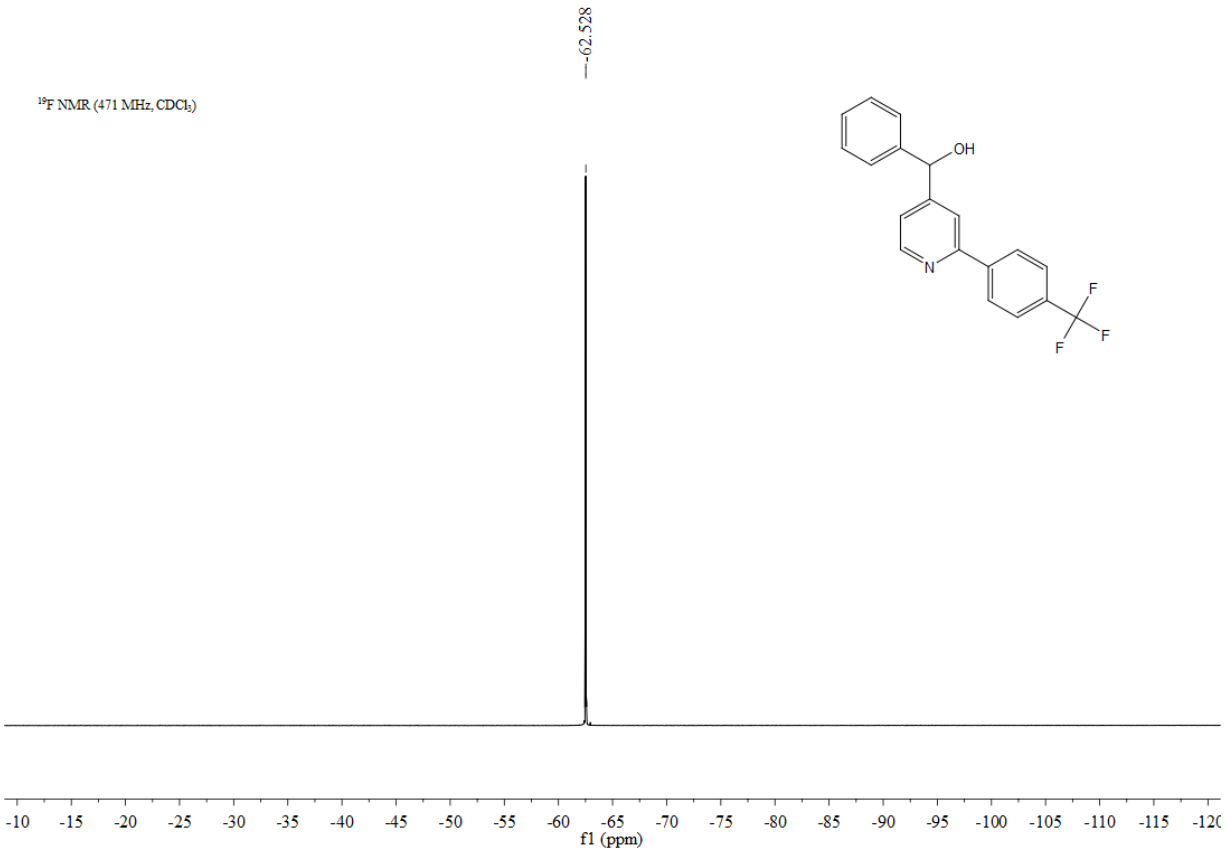


Phenyl(2-(4-(trifluoromethyl)phenyl)pyridin-4-yl)methanol (3be)

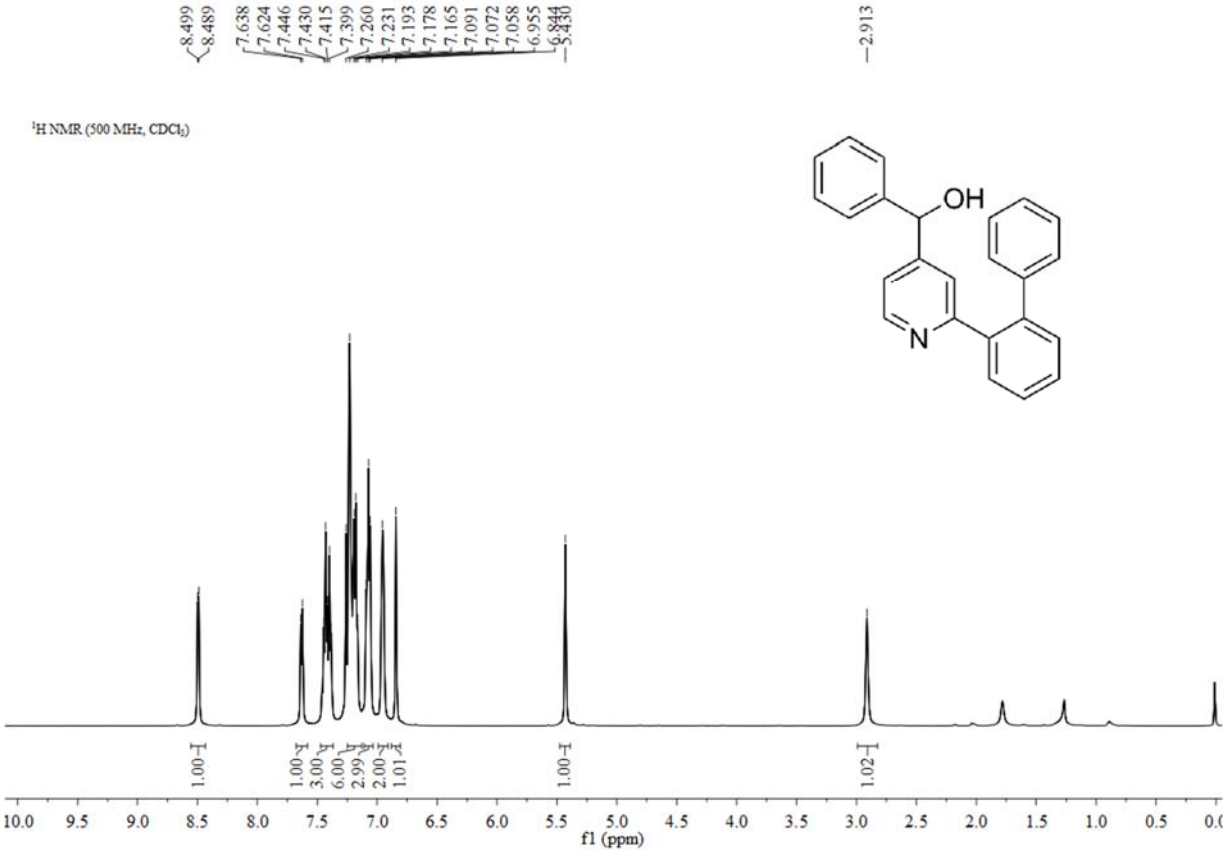


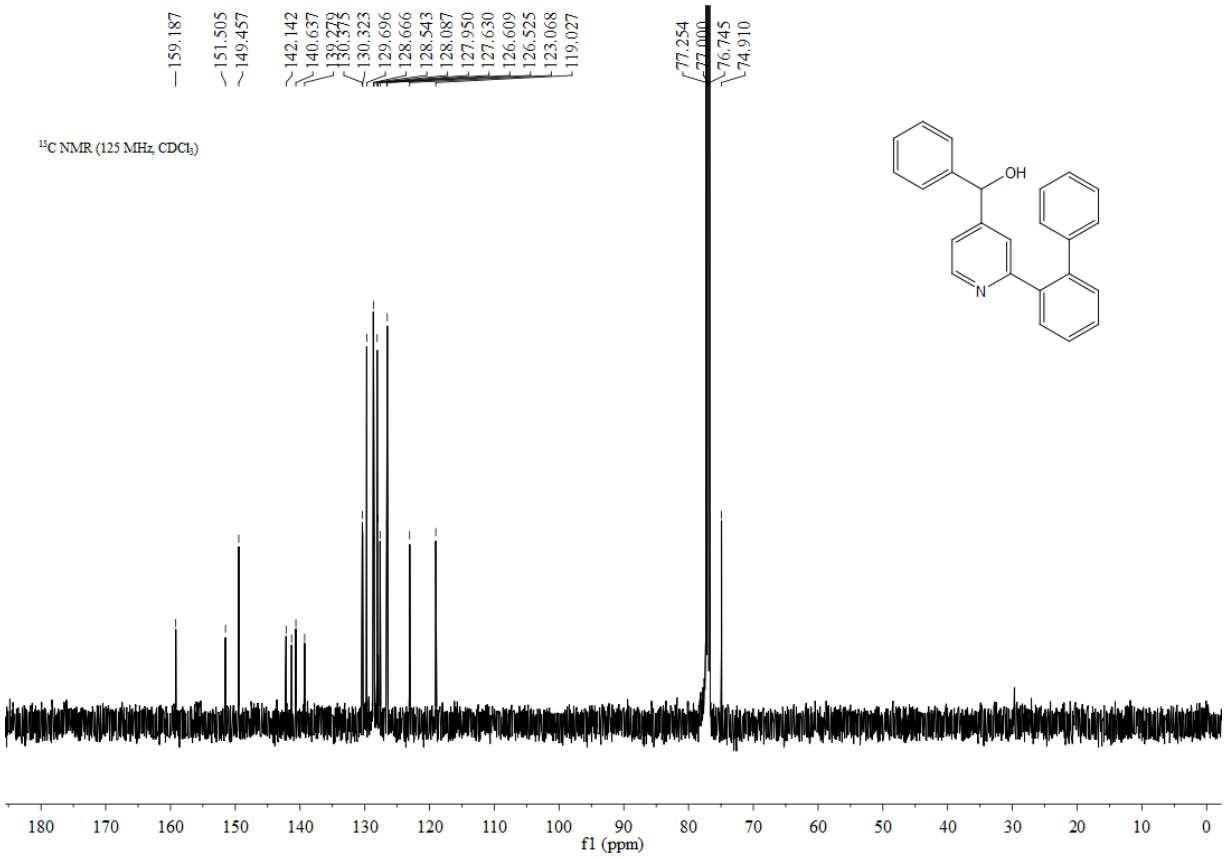


^{19}F NMR (471 MHz, $CDCl_3$)

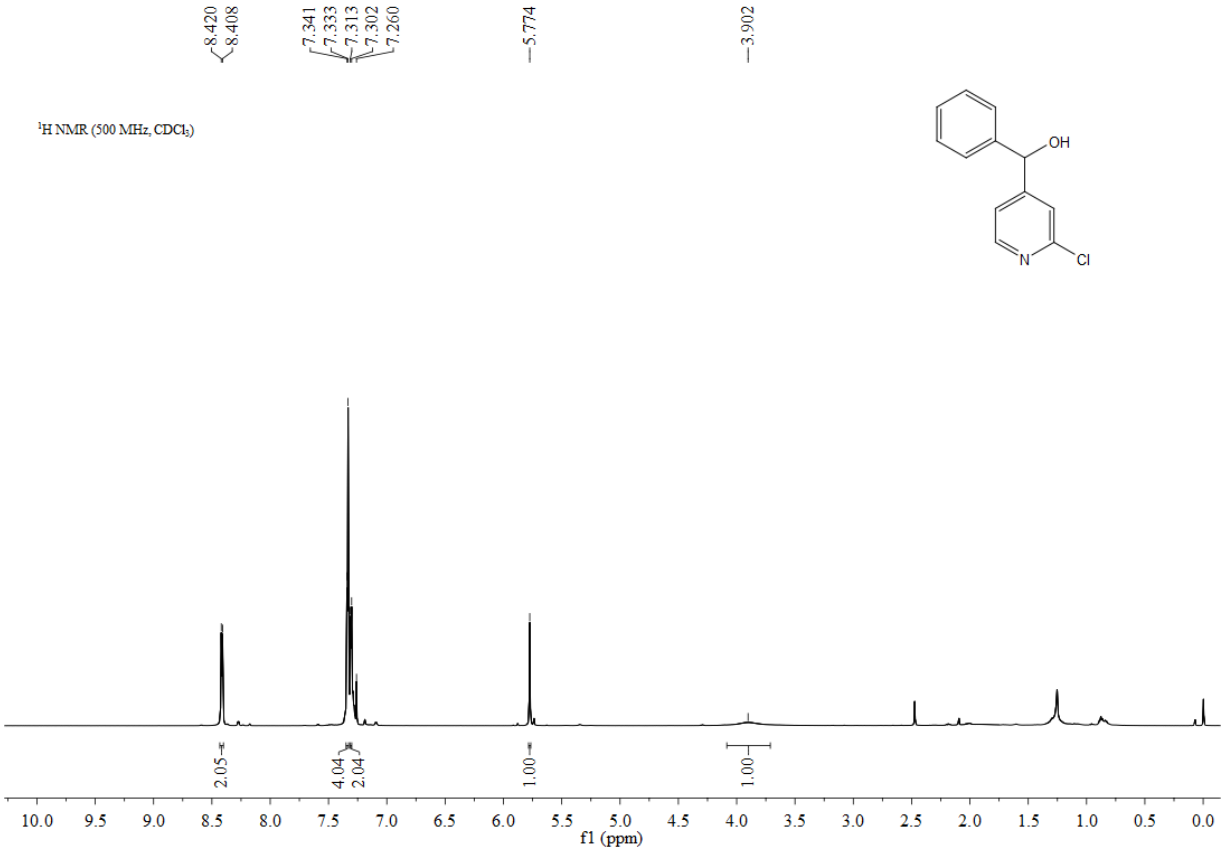


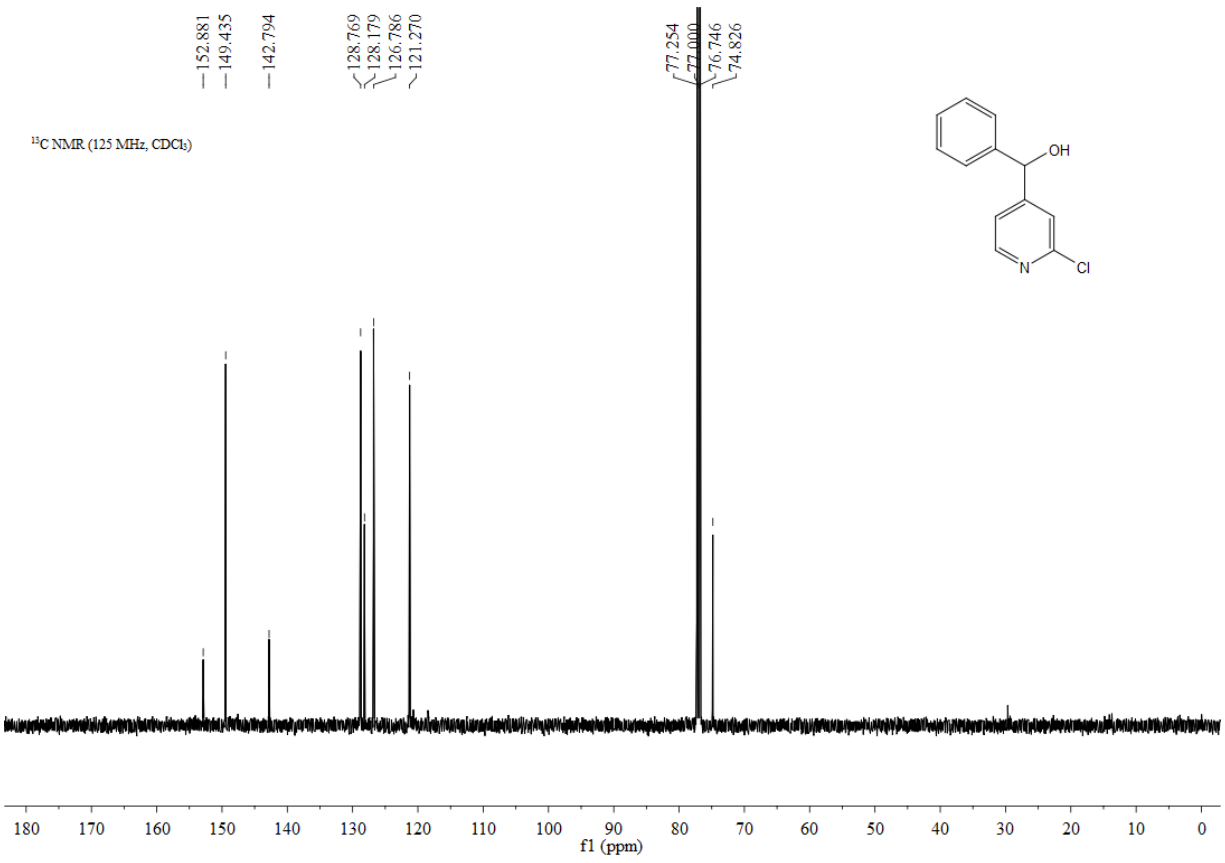
(2-([1,1'-biphenyl]-2-yl)pyridin-4-yl)(phenyl)methanol (3bf)



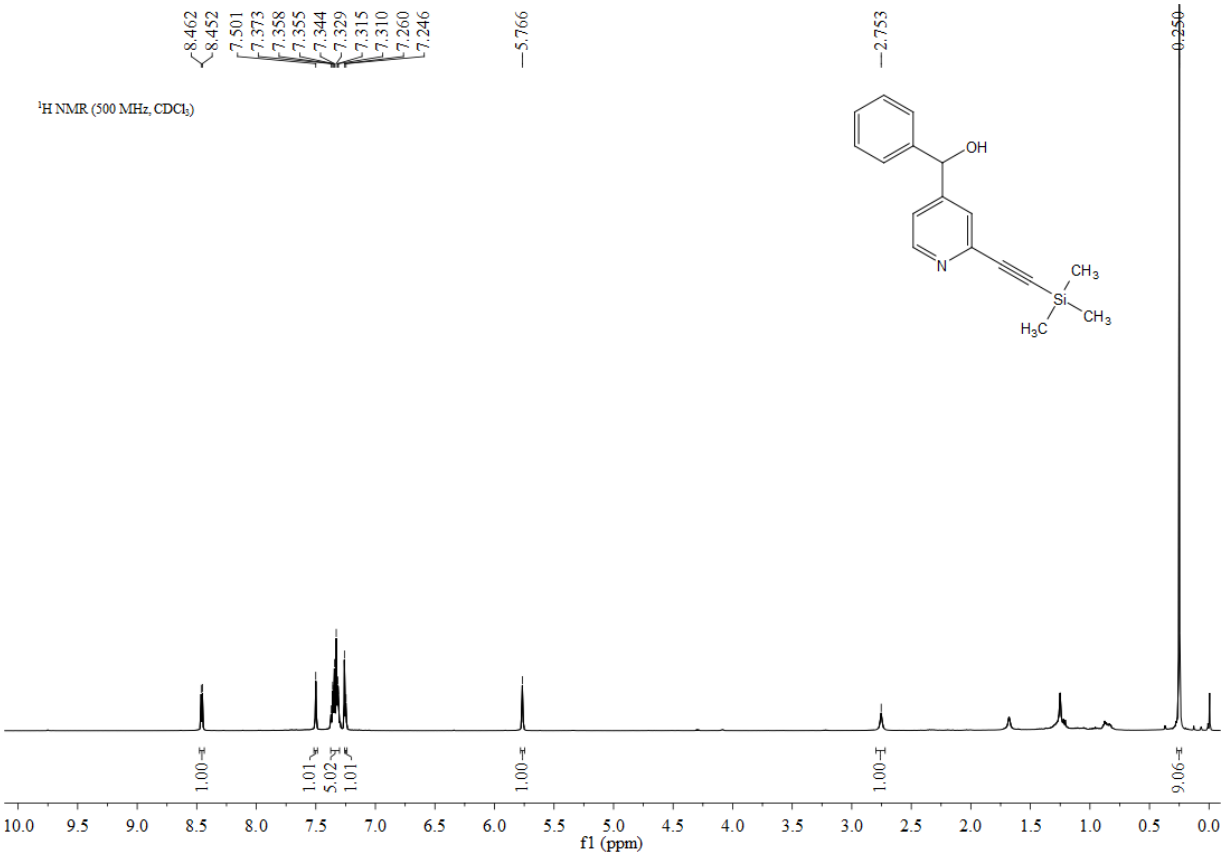


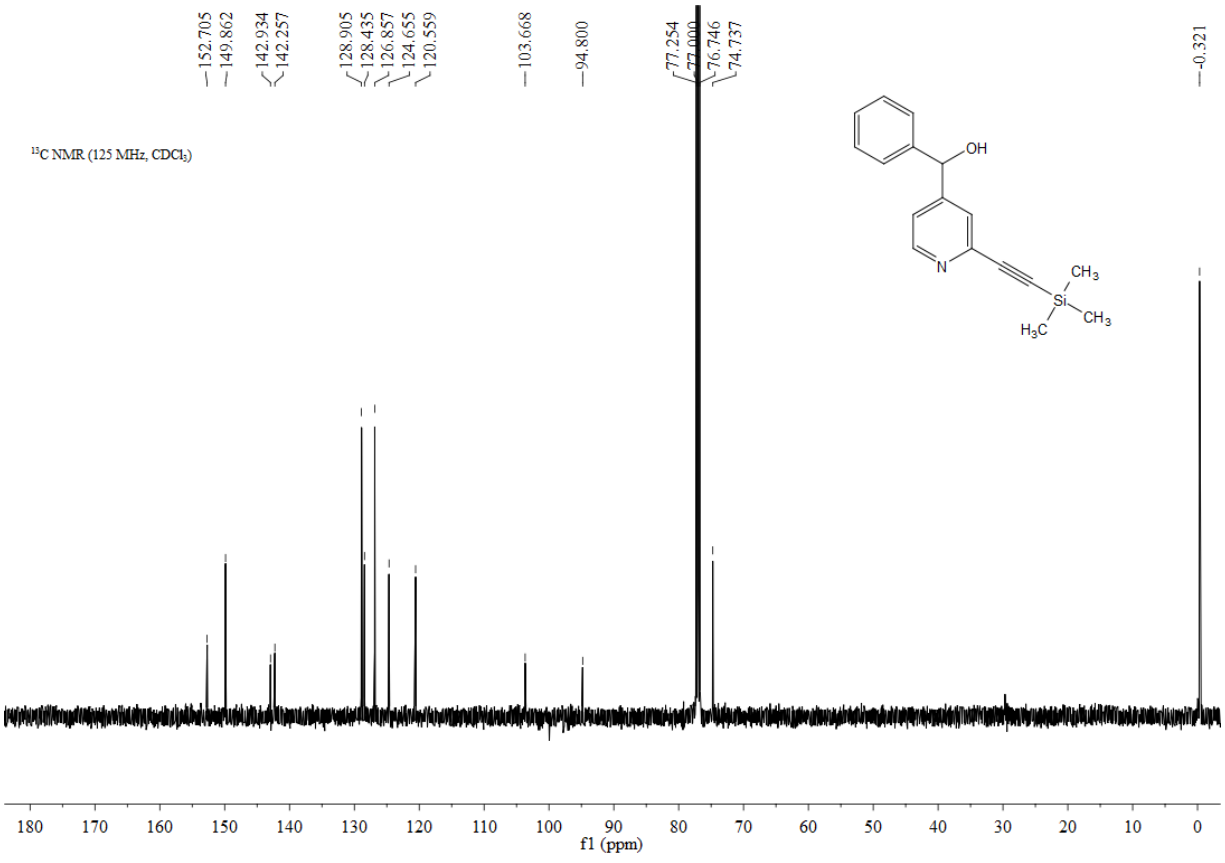
(2-chloropyridin-4-yl)(phenyl)methanol (3bg)



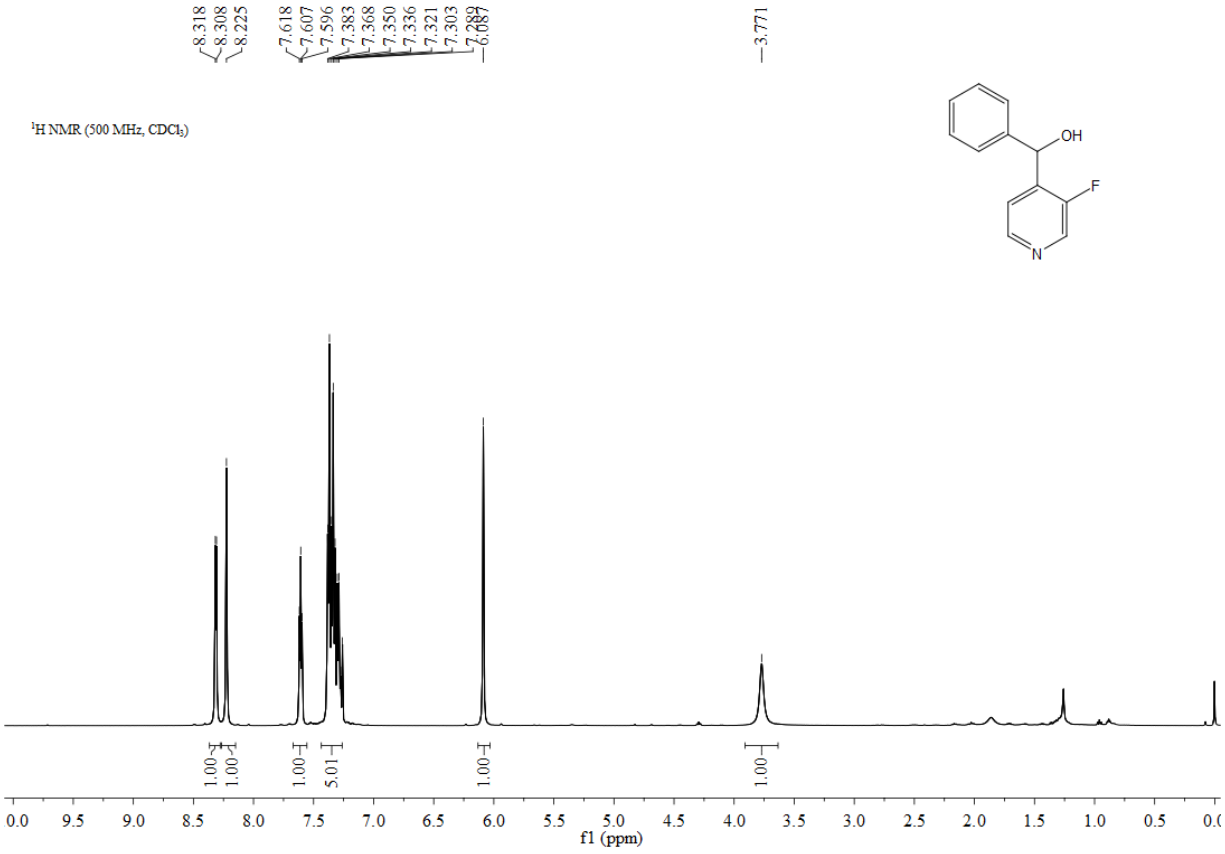


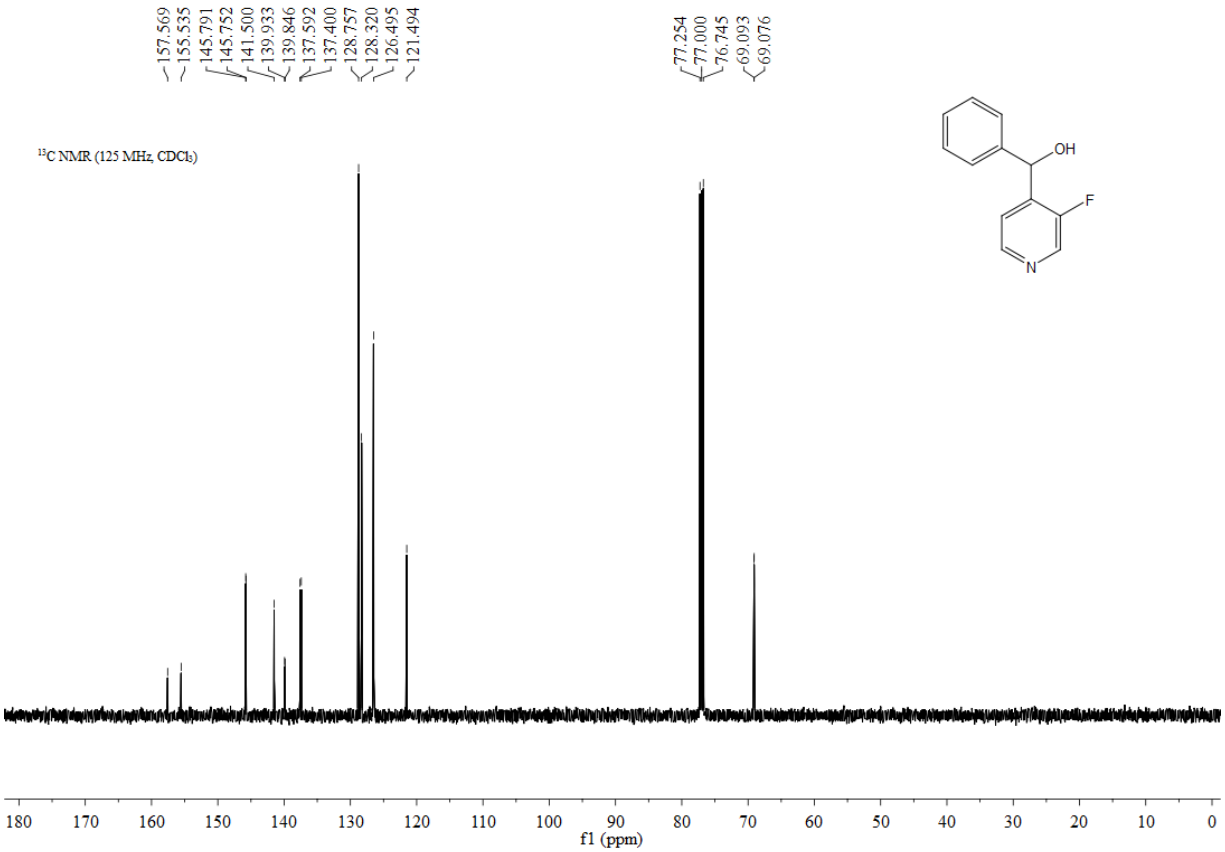
Phenyl(2-((trimethylsilyl)ethynyl)pyridin-4-yl)methanol (3bh)



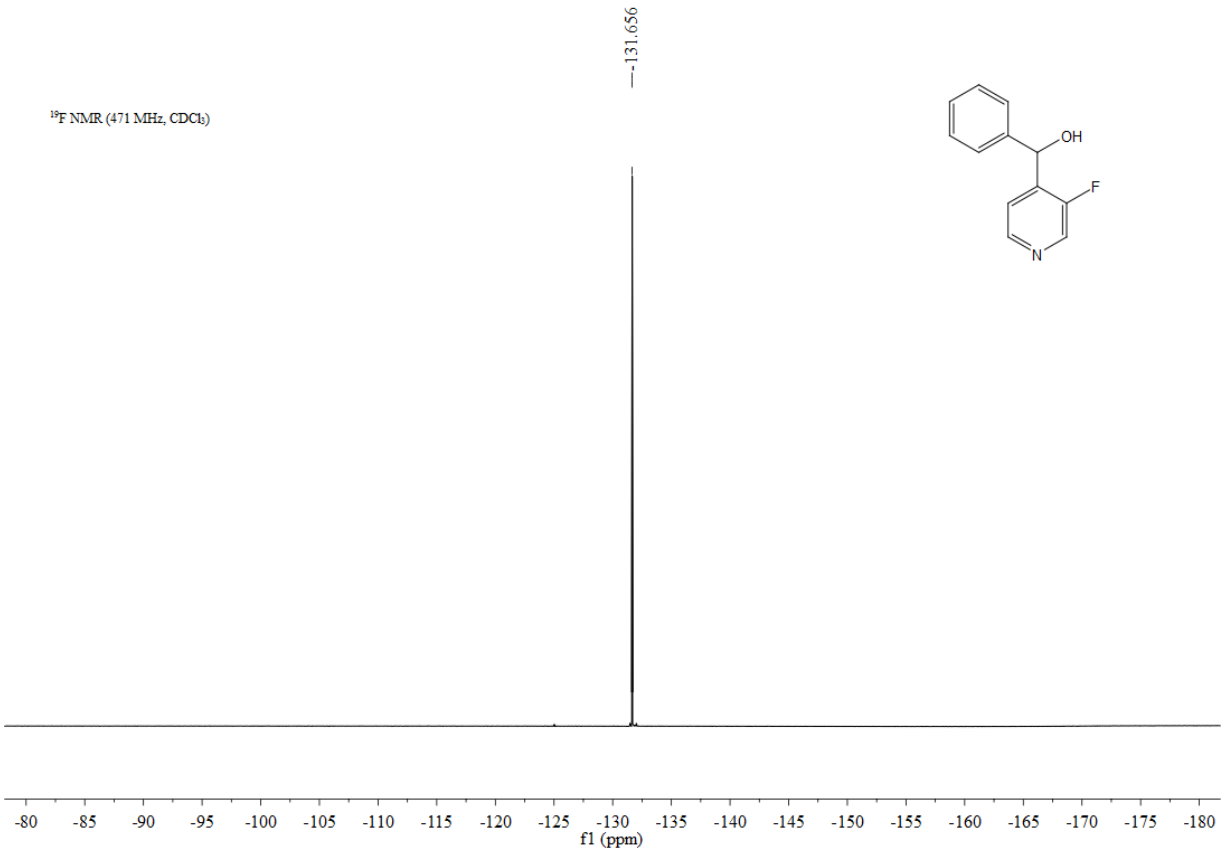


(3-fluoropyridin-4-yl)(phenyl)methanol (3bi)

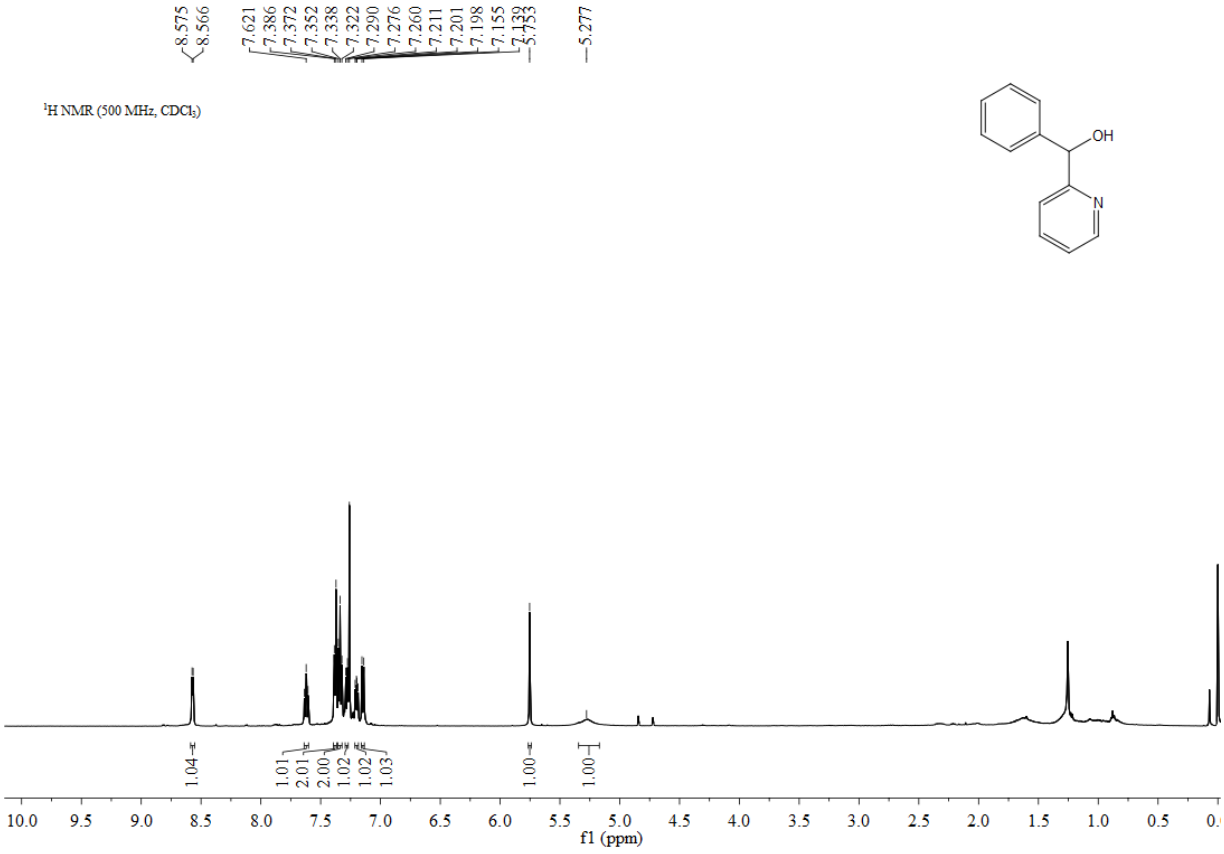


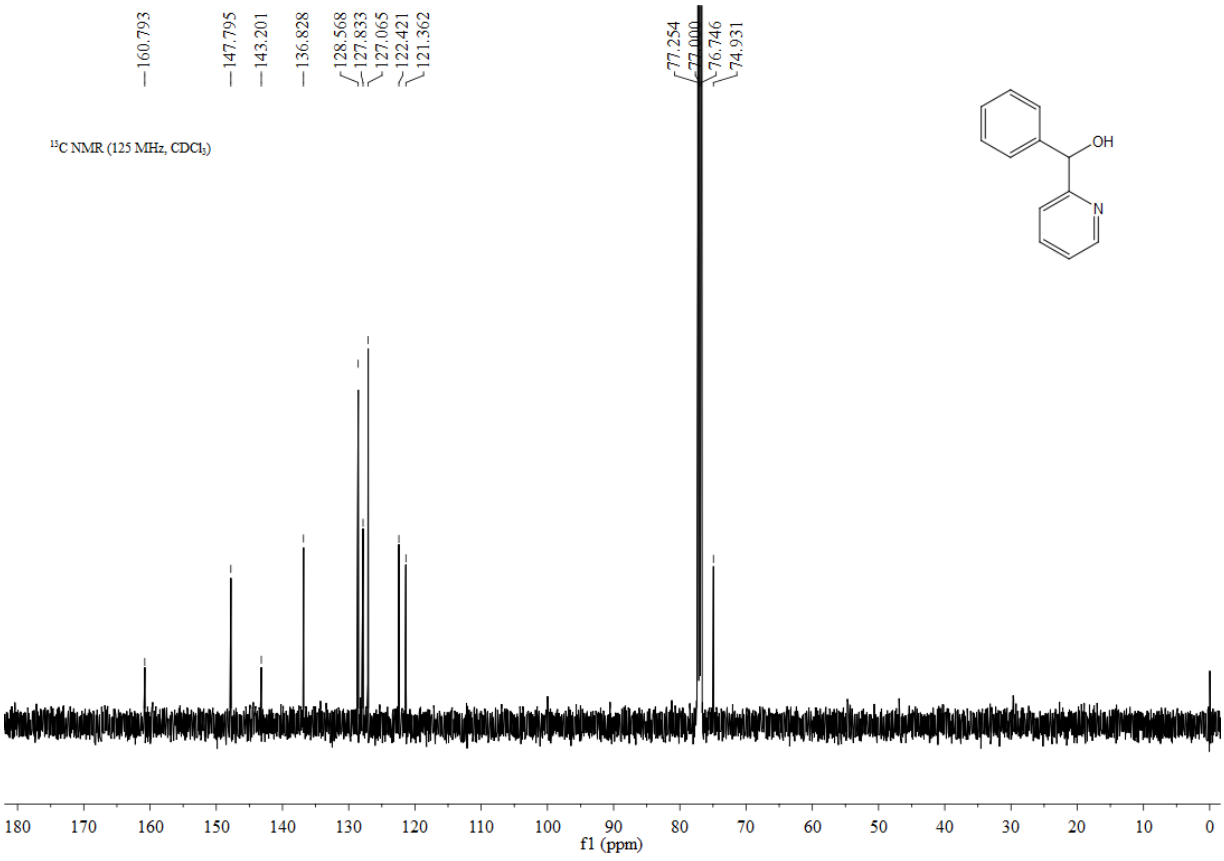


^{19}F NMR (471 MHz, $CDCl_3$)



Phenyl(pyridin-2-yl)methanol (3bj)





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