Supporting Information

Transition Metal Free chemoselective C-H Hydroxylation of Bisarylmethanes Enabled by a Phosphite as Sacrifical Reductant

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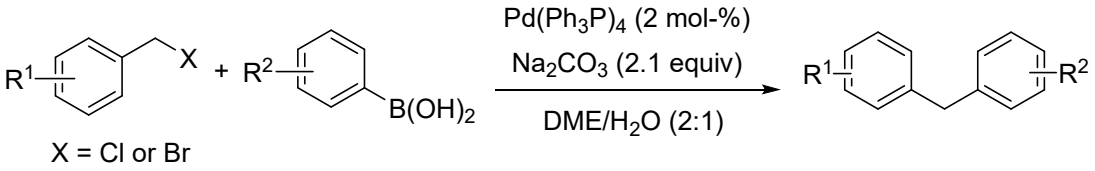
1. General Information

Unless otherwise noted, all reagents were obtained commercially and used without further purification. Unless otherwise specified, all other reagents were purchased from Acros, Aldrich, Fisher, Adamas-beta Co. Ltd. or TCI and used without further purification. ^{1}H NMR spectra was recorded at 400 MHz, ^{13}C NMR spectra was recorded at 100 MHz. ^{1}H NMR spectra was recorded with tetramethylsilane (δ 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ 77.00 ppm) as internal reference. Chemical shifts were reported in parts per million (ppm, δ) downfield from tetramethylsilane. Proton coupling patterns are described as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), and broad (br). Chromatography was carried out with silica gel (200-300 mesh) using mixtures of petroleum ether and ethyl acetate (or dichlormethan and methanol) as eluents. Mass Spectra were obtained from the mass spectrometry facility of East China University of Science and Technology. Mass spectrometry analysis was carried out using an electrospray spectrometer Waters Micromass Q-TOF premier Mass Spectrometer (Waters, Milford, MA, USA). Gas Chromatography analysis was carried out using 6890 plus GC system (FID/6890 PLUS, Agilent, USA). Gas Chromatography analysis was carried out using U3000/Q-Exactive plus (Thermo Scientific, USA).

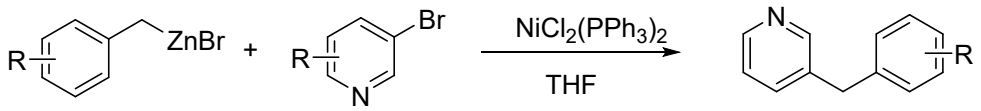
2. General procedure for the preparation of substrates

2.1 General procedure A:

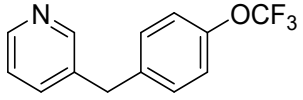


Na_2CO_3 (4.2 mmol), boronic acid (2.4 mmol), $Pd(PPh_3)_4$ (0.04 mmol), 1,2-dimethoxyethane (4 mL) and water (2 mL) were added into a dried, sealed tube with (ar)alkyl halide (2.0 mmol) under N_2. The tube was sealed and heated for 4 h-8 h at 100 °C. The resulting mixture was cooled and diluted with water when the reaction was monitored by TLC. The aqueous layer was extracted with CH_2Cl_2. The organic layer was dried with $MgSO_4$, filtered, and the solvents were evaporated. The residue was purified by column chromatography.[1]

2.2 General procedure B:

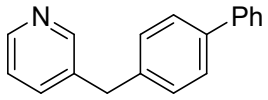


Benzyl bromide (6.0 mmol) and Zn powder (585 mg, 9.0 mmol) in THF (10 mL) were stirred for 2 h at room temperature. The supernatant solution was transferred to a solution (6 mL) containing 3-bromopyridine (5.0 mmol) and $NiCl_2(PPh_3)_2$ (654 mg, 1.0 mmol) in THF. The reaction mixture was stirred for 20 h at room temperature before quenching with 10% ammonia. The product was extracted with CH_2Cl_2 and purified by column chromatography (silica gel, Dichlormethan/Methanol = 50/1).[2]

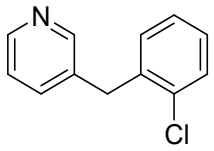


3-(4-(Trifluoromethoxy)benzyl)pyridine (**3c**, pale yellow oil), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 50/1), 734 mg (58% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.58 (s, 1H), 8.54 (d, J = 4.4 Hz, 1H), 7.56 (d, J = 8.0 Hz, 1H), 7.32 (dd, J_1 = 7.6 Hz, J_2 = 5.2 Hz, 1H), 7.19 - 7.13 (m, 4H), 4.00 (s,

2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.5, 148.0, 147.5, 138.0, 137.8, 137.0, 130.2, 124.4, 121.4, 120.4, 38.2 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{11}F_3NO$ 254.0793; Found 254.0798.



3-([1,1'-Biphenyl]-4-ylmethyl)pyridine (**3d**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 50/1), 797 mg (65% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.68 (s, 1H), 8.61 (d, *J* = 4.4 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.56 - 7.52 (m, 4H), 7.45 - 7.32 (m, 4H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.05 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.0, 146.8, 140.5, 139.8, 139.3, 138.5, 137.6, 129.2, 128.8, 127.6, 127.4, 127.0, 124.8, 38.4 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{16}N$ 246.1283; Found 246.1287.



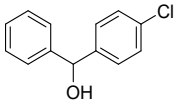
3-(2-Chlorobenzyl)pyridine (**3f**, yellow oil), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 50/1), 530 mg (52% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.51 (s, 1H), 8.47 (s, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.39 - 7.37 (m, 1H), 7.21 - 7.16 (m, 4H), 4.10 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3, 147.8, 137.4, 136.2, 135.0, 134.2, 131.0, 129.8, 128.2, 127.1, 123.4, 36.6 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{11}NCl$ 204.0580; Found 204.0585.

3. General procedure for the preparation of substrates

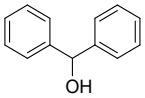
3.1 Typical procedure for preparing hydroxylation products (2a as an example):

Under air atmosphere, 257 μL $P(OEt)_3$ (1.5 mmol) was added into a solution of KO*t*Bu (168 mg, 1.5 mmol) in 1 mL anhydrous DMSO at room temperature. Substrate **1a** (61 mg, 0.3 mmol) was then added into the solution. The reaction was

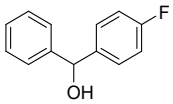
completed in about 30 min. The mixture was quenched with 10 mL H_2O when the reaction finished monitored by TLC. Then the mixture was extracted with CH_2Cl_2 (30 mL), washed sequentially with saturated sodium chloride solution (30 mL). The combined organic phase was dried over $MgSO_4$, filtered and evaporated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Petroleum ether/ EtOAc = 4:1) to give **2a** as a white solid (47.2 mg, 71% yield).



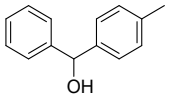
(4-Chlorophenyl)(phenyl)methanol[3] (**2a**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 47.2 mg (71% yield). Mp: 57-59 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.35 - 7.34 (m, 4H), 7.31 - 7.27 (m, 5H), 5.80 (s, 1H), 2.35 (br s, 1H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.4, 142.2, 133.3, 128.7, 128.6, 127.9, 126.5, 75.6 ppm.



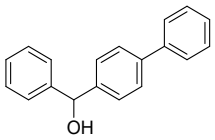
Diphenylmethanol[4] (**2b**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 38.0 mg (68% yield). (68% yield). Mp: 65-67 ^oC. ^1H NMR (400 MHz, $CDCl_3$): δ 7.40 - 7.33 (m, 8H), 7.29 - 7.26 (m, 2H), 5.84 (s, 1H), 2.34 (d, J = 2.4 Hz, 1H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.8, 128.5, 127.6, 126.6, 76.2 ppm.



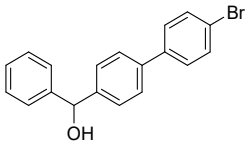
(4-Fluorophenyl)(phenyl)methanol[4] (**2c**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 42.3 mg (70% yield)., 42.0 mg (70% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 - 7.28 (m, 7H), 7.04 - 7.00 (m, 2H), 5.81 (s, 1H), 2.41 (s, 1H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 162.2 (d, $^{1}J_{C\text{-}F}$ = 244.4 Hz), 143.7, 139.6 (d, $^{4}J_{C\text{-}F}$ = 3.0 Hz), 128.6, 128.30 (q, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 127.8, 126.5, 115.3 (d, $^{2}J_{C\text{-}F}$ = 21.4 Hz), 75.6 ppm.



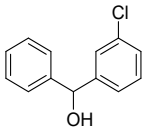
Phenyl(p-tolyl)methanol[3] (**2d**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 23.9 mg (2 h, 40% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.30 (m, 4H), 7.26 - 7.24 (m, 3H), 7.14 - 7.12 (m, 2H), 5.79 (s, 1H), 2.32 (s, 3H), 2.25 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 140.9, 137.2, 129.1, 128.4, 127.4, 126.5, 126.4, 76.0, 21.1 ppm.



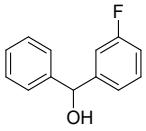
[1,1'-biphenyl]-4-yl(phenyl)methanol[5] (**2e**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 62 mg (79% yield). Mp: 93-96 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 - 7.57 (dd, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 4H), 7.47 - 7.43 (m, 6H), 7.39 - 7.28 (m, 4H), 5.90 (s, 1H), 2.35 (s, 1H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.8, 142.9, 140.8, 140.5, 128.8, 128.6, 127.7, 127.3, 127.3, 127.1, 127.0, 126.6, 76.1 ppm.



(4'-Bromo-[1,1'-biphenyl]-4-yl)(phenyl)methanol[6] (**2f**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 63 mg (62% yield). Mp: 135-137 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 - 7.48 (m, 4H), 7.44 - 7.38 (m, 6H), 7.36 - 7.32 (m, 2H), 7.29 - 7.25 (m, 1H), 5.85 (s, 1H), 2.36 (s, 1H) ppm. ^{13}C NMR (101 MHz, CDCl3): δ 143.7, 143.3, 139.7, 139.2, 131.9, 128.7, 128.6, 127.8, 127.1, 127.1, 126.6, 121.6, 76.0 ppm.

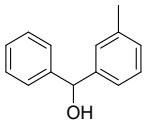


(3-Chlorophenyl)(phenyl)methanol[7] (**2g**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 47.3 mg (71% yield).. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (s, 1H), 7.33 - 7.26 (m, 5H), 7.23 - 7.20 (m, 3H), 5.74 (s, 1H), 2.46 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8, 143.2, 134.4, 129.8, 128.7, 128.0, 127.7, 126.6, 124.7, 75.7 ppm.

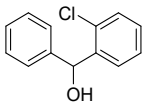


(3-Fluorophenyl)(phenyl)methanol[8] (**2h**, pale yellow oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 46.0 mg (75% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 - 7.24 (m, 6H), 7.12 - 7.08 (m, 2H), 6.93 (td, J_1 = 8.8 Hz, J_2 = 2.0 Hz, 1H), 5.76 (s, 1H), 2.46 (d, J = 2.4 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0 (d, $^{1}J_{C\text{-}F}$ = 144.3 Hz), 146.4 (d, $^{5}J_{C\text{-}F}$ = 6.9 Hz),

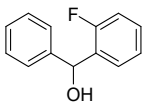
143.3, 130.0 (d, $^{4}J_{C\text{-}F}$ = 8.2 Hz), 128.7, 127.9, 126.6, 122.1 (d, $^{6}J_{C\text{-}F}$ = 2.8 Hz), 114.4 (d, $^{3}J_{C\text{-}F}$ = 21.0 Hz), 113.4 (d, $^{2}J_{C\text{-}F}$ = 22.2 Hz), 75.7 (d, J = 1.6 Hz) ppm.



Phenyl(m-tolyl)methanol[4] **(2i,** white solid**)** purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 33.0 mg (55% yield). Mp: 52-54°C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 - 7.35 (m, 4H), 7.31 - 7.28 (m, 1H), 7.24 - 7.19 (m, 2H), 7.11 (d, J = 7.2 Hz, 1H), 5.82 (s, 1H), 2.37 (s, 3H), 2.34 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 143.7, 138.1, 128.4, 128.4, 128.3, 127.5, 127.2, 126.5, 123.6, 76.2, 21.4 ppm.

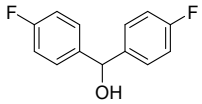


(2-Chlorophenyl)(phenyl)methanol[3] (**2j**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 48.8 mg (74% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (dd, J_1 = 8.0 Hz, J_2 = 1.6 Hz, 1H), 7.38 - 7.23 (m, 7H), 7.20 (td, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1H), 6.20 (s, 1H), 2.46 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.2, 141.0, 132.5, 129.5, 128.7, 128.5, 128.0, 127.8, 127.1, 126.9, 72.7 ppm.

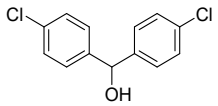


(2-Fluorophenyl)(phenyl)methanol[9] (**2k**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 42.1 mg (70% yield). Mp: 39-41°C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (td, J_1 = 7.6 Hz, J_2 = 1.6 Hz,

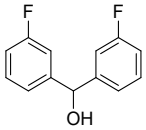
1H), 7.39 (d, J = 7.2 Hz, 2H), 7.34 - 7.31 (m, 2H), 7.28 - 7.21 (m, 2H), 7.15 - 7.12 (m, 1H), 7.03 - 6.98 (m, 1H), 6.12 (s, 1H), 2.40 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.9 (d, $^1J_{C\text{-}F}$ = 244.7 Hz), 142.8, 131.0 (d, $^3J_{C\text{-}F}$ = 12.9 Hz), 129.2 (d, $^4J_{C\text{-}F}$ = 8.1 Hz), 128.6, 127.8, 127.7 (d, $^5J_{C\text{-}F}$ = 3.8 Hz), 126.4, 124.4 (d, $^6J_{C\text{-}F}$ = 3.6 Hz), 115.4 (d, $^2J_{C\text{-}F}$ = 21.5 Hz), 70.1 (d, J = 3.4 Hz) ppm.



Bis(4-Fluorophenyl)methanol[10] (**2l**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 41.0 mg (62% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 - 7.30 (m, 4H), 7.05 - 7.00 (m, 4H), 5.81 (s, 1H), 2.27 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.2 (d, $^1J_{C\text{-}F}$ = 244.7 Hz), 139.4 (d, $^4J_{C\text{-}F}$ = 3.1 Hz), 128.2 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 115.4 (d, $^2J_{C\text{-}F}$ = 21.4 Hz), 75.0 (d, J = 5.7 Hz) ppm.

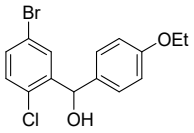


Bis(4-Chlorophenyl)methanol[11] (**2m,** white solid), purification by flash column chromatography(eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 58.3 mg (76% yield). Mp: 93-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 - 7.25 (m, 8H), 5.75 (s, 1H), 2.38 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 133.5, 128.7, 127.8, 74.9 ppm.

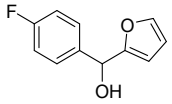


Bis(3-Fluorophenyl)methanol[12] **(2n)** white solid**,** purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 52.7 mg (80%

yield). Mp: 44-47 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 - 7.28 (m, 2H), 7.14 - 7.07 (m, 4H), 6.97 (td, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 2H), 5.78 (s, 1H), 2.50 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0 (d, $^{1}J_{C\text{-}F}$ = 245.0 Hz), 145.8 (d, $^{5}J_{C\text{-}F}$ = 6.6 Hz), 130.2 (d, $^{4}J_{C\text{-}F}$ = 8.1 Hz), 122.1 (d, $^{6}J_{C\text{-}F}$ = 2.9 Hz), 114.7 (d, $^{3}J_{C\text{-}F}$ = 21.0 Hz), 113.4 (d, $^{2}J_{C\text{-}F}$ = 22.1 Hz), 75.1 (t, J = 1.7 Hz) ppm.

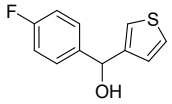


(5-Bromo-2-chlorophenyl)(4-ethoxyphenyl)methanol[13] (**2o**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1，Rf = 0.5), 64.9 mg (63% yield). Mp: 101-103 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 2.4 Hz, 1H), 7.32 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 7.25 (s, 1H), 7.23 (s, 1H), 7.16 (d, J = 8.4 Hz, 1H), 6.85 (s, 1H), 6.83 (s, 1H), 6.04 (s, 1H), 4.00 (q, J = 7.2 Hz, 2H), 2.39 (s, 1H), 1.39 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 143.2, 133.5, 131.5, 131.2, 130.9, 130.6, 128.4, 120.9, 114.5, 72.2, 63.4, 14.8 ppm.

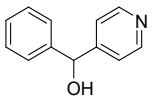


(4-Fluorophenyl)(furan-2-yl)methanol[14] (**2p**, pale yellow oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 26.1 mg (45% yield).^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 - 7.39 (m, 3H), 7.06 (t, J = 8.4 Hz, 2H), 6.32 (dd, J_1 = 2.8 Hz, J_2 = 2.0 Hz, 1H), 6.11 (d, J = 3.2 Hz, 1H), 5.81 (s, 1H), 2.50 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.5 (d, $^{1}J_{C\text{-}F}$ = 244.7 Hz), 155.7, 142.7, 136.5 (d, $^{4}J_{C\text{-}F}$ = 3.1 Hz), 128.4 (d, $^{3}J_{C\text{-}F}$ = 8.2 Hz), 115.3 (d, $^{2}J_{C\text{-}F}$ = 21.5

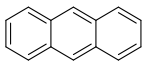
Hz), 110.3, 107.5, 69.5 ppm.



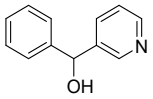
(4-Fluorophenyl)(thiophen-3-yl)methanol[15] (**2q**, yellow oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 35.8 mg (57% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.33 (m, 2H), 7.30 - 7.28 (m, 1H), 7.17 - 7.16 (m, 1H), 7.06 - 7.01 (m, 2H), 6.97 (dd, J_1 = 4.8 Hz, J_2 = 0.8 Hz, 1H), 5.86 (s, 1H), 2.41 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.2 (d, $^{1}J_{C\text{-}F}$ = 244.5 Hz), 145.1, 139.1 (d, $^{4}J_{C\text{-}F}$ = 3.1 Hz), 128.1 (d, $^{3}J_{C\text{-}F}$ = 8.1 Hz), 126.4, 126.2, 121.7, 115.3 (d, $^{2}J_{C\text{-}F}$ = 21.3 Hz), 72.1 ppm.



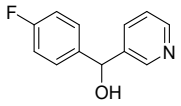
Phenyl(pyridin-4-yl)methanol[16] (**2r**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 40/1, Rf = 0.2), 35.2 mg (63% yield). Mp: 114-116 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (d, J = 6.0 Hz, 2H), 7.33 - 7.26 (m, 7H), 5.76 (s, 1H), 4.44 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.8, 149.2, 142.8, 128.8, 128.2, 126.8, 121.6, 74.7 ppm.



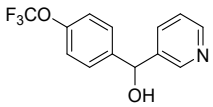
Anthracene[17] (**2s**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.8), 44.2 mg (82% yield). Mp: 215-217 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (s, 2H), 8.04 - 8.01 (m, 4H), 7.50 - 7.47 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 131.7, 128.2, 126.2, 125.4 ppm.



Phenyl(pyridin-3-yl)methanol[18] (**4a**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 40/1, Rf = 0.2), 36.0 mg (64% yield). Mp: 65-67 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (s, 1H), 8.36 (s, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.34 - 7.26 (m, 5H), 7.22 (dd, J_1 = 7.2 Hz, J_2 = 4.8 Hz, 1H), 5.82 (s, 1H), 3.98 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 148.0, 143.2, 139.7, 134.4, 128.7, 127.9, 126.6, 123.5, 73.9 ppm.

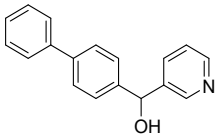


(4-Fluorophenyl)(pyridin-3-yl)methanol[19] (**4b**, yellow oil), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 40/1, Rf = 0.3), 40.6 mg (68% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (s, 1H), 8.36 (s, 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.32 - 7.29 (m, 2H), 7.23 (dd, J_1 = 7.6 Hz, J_2 = 5.2 Hz, 1H), 7.03 - 6.99 (m, 2H), 5.81 (s, 1H), 4.32 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.3 (d, $^{1}J_{C\text{-}F}$ = 244.9 Hz), 148.4, 147.9, 139.6, 139.1 (d, $^{4}J_{C\text{-}F}$ = 3.0 Hz), 134.4, 128.3 (d, $^{3}J_{C\text{-}F}$ = 8.1 Hz), 123.6, 115.6 (d, $^{2}J_{C\text{-}F}$ = 21.3 Hz), 73.2 ppm.

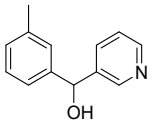


Pyridin-3-yl(4-(trifluoromethoxy)phenyl)methanol (4c) white solid**,** purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 53.1 mg (66% yield). Mp: 87-91 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 8.30 (s, 1H), 7.67 (d, J = 7.6 Hz, 1H), 7.38 (s, 1H), 7.35 (s, 1H), 7.23 (dd, J_1 = 7.2 Hz, J_2 =

4.8 Hz, 1H), 7.18 (s, 1H), 7.16 (s, 1H), 5.82 (s, 1H), 5.18 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.6 (d, J = 1.5 Hz), 148.3, 147.7, 142.0, 139.7, 134.7, 128.0, 123.7, 121.1, 120.4 (q, J_{C-F} = 255.6 Hz), 73.0 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{11}F_3NO_2$ 270.0742; Found 270.0741.

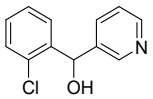


[1,1'-biphenyl]-4-yl(pyridin-3-yl)methanol (4d) white solid**,** purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 47.4 mg (60% yield). Mp: 94-97 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (s, 1H), 8.42 (s, 1H), 7.78 (d, J = 8.0 Hz, 1H), 7.60 - 7.58 (m, 4H), 7.48 - 7.44 (m, 4H), 7.38 (t, J = 7.6 Hz, 1H), 7.29 (dd, J_1 = 7.6 Hz, J_2 = 2.8 Hz, 1H), 5.91 (s, 1H), 4.03 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 148.0, 142.3, 140.8, 140.6, 139.8, 134.5, 128.8, 127.4, 127.4, 127.1, 127.0, 123.6, 73.7 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{16}NO$ 262.1232; Found 262.1237.

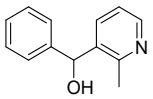


Pyridin-3-yl(m-tolyl)methanol (4e) white solid**,** purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 39.0 mg (65% yield). Mp: 62-64 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (s, 1H), 8.35 (s, 1H), 7.70 (d, J = 7.6 Hz, 1H), 7.24 - 7.20 (m, 2H), 7.14 (t, J = 8.0 Hz, 2H), 7.08 (d, J = 7.6 Hz, 1H), 5.78 (s, 1H), 3.74 (brs, 1H), 2.32 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 148.0, 143.3, 139.9, 138.4, 134.4, 128.6, 128.6, 127.2, 123.6, 123.5, 73.9, 21.5

ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{14}NO$ 200.1075; Found 200.1072.



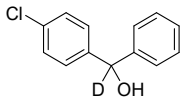
(2-Chlorophenyl)(pyridin-3-yl)methanol[18] (**4f**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 40.1 mg (61% yield). Mp: 81-83 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (s, 1H), 8.31 (s, 1H), 7.66 (t, *J* = 8.0 Hz, 2H), 7.32 - 7.27 (m, 2H), 7.23 - 7.19 (m, 2H), 6.19 (s, 1H), 4.79 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 148.1, 140.6, 138.6, 134.9, 132.2, 129.5, 128.9, 127.8, 127.2, 123.5, 70.1 ppm.



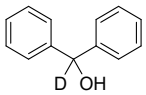
(2-Methylpyridin-3-yl)(phenyl)methanol[18] (**4g**, colorless oil), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 36.2 mg (60% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (d, *J* = 4.0 Hz, 1H), 7.88 (dd, J_1 = 8.0 Hz, J_2 = 1.2 Hz, 1H), 7.34 - 7.26 (m, 5H), 7.15 (dd, J_1 = 7.6 Hz, J_2 = 4.8 Hz, 1H), 5.94 (s, 1H), 3.73 (brs, 1H), 2.39 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6, 147.6, 142.2, 137.3, 134.26, 128.7, 128.0, 127.2, 121.4, 72.5, 22.2 ppm.

3.1 Typical procedure for preparing hydroxylation-deuteration products (3s as an example): Under air atmosphere, 155 μL $P(OEt)_3$ (0.9 mmol) was added to a solution of KO*t*Bu (168 mg, 1.5 mmol) in 1 mL DMSO-d_6 at room temperature. Substrate **1s** (51 mg, 0.3 mmol) was added into the solution. The mixture was quenched with 10 mL H_2O after the reaction finished monitored by TLC. The mixture was extracted with CH_2Cl_2 (15 mL × 3), washed sequentially with saturated sodium

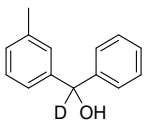
chloride solution (30 mL). The combined organic phase was dried over $MgSO_4$, filtered and evaporated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Dichlormethan/Methanol = 50:1 - 30:1) to give **3s** as a white solid (36 mg, 65% yield).



(4-Chlorophenyl)(phenyl)methan-*d*-ol[19] (**2a'**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 43.8 mg (67% yield). Mp: 58-60 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 - 7.33 (m, 4H), 7.32 - 7.27 (m, 5H), 5.80 (s, 0.03H), 2.33 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.4, 142.1, 133.3, 128.6, 128.6, 127.8, 126.5, 75.2 (t, $J_{C\text{-}D}$ = 21.9 Hz) ppm.

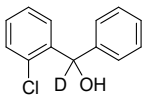


Diphenylmethan-*d*-ol[19] (**2b'**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 22.0 mg (40% yield). Mp: 63-65 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 - 7.31 (m, 8H), 7.28 - 7.25 (m, 2H), 5.84 (s, 0.03H), 2.26 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.7, 128.5, 127.6, 126.5, 75.8 (t, $J_{C\text{-}D}$ = 21.9 Hz) ppm.

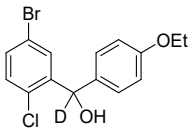


Phenyl(m-tolyl)methan-*d*-ol[19] (**21'**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.5), 36.1 mg (61% yield). Mp: 49-51 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 - 7.30 (m, 4H), 7.27 - 7.18

(m, 3H), 7.14 (d, J = 7.6 Hz, 1H), 7.06 (d, J = 7.2 Hz, 1H), 5.77 (s, 0.02H), 2.32 (s, 3H), 2.28 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 143.7, 138.1, 128.4, 128.4, 128.3, 127.4, 127.1, 126.5, 123.6, 75.8 (t, $J_{C\text{-}D}$ = 22.0 Hz), 21.4 ppm.

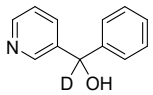


(2-Chlorophenyl)(phenyl)methan-*d*-ol[20] (**2g'**, colorless oil), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 51.9 mg (79% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (dd, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 2H), 7.42 - 7.39 (m, 2H), 7.36 - 7.27 (m, 5H), 7.23 (td, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1H) 6.22 (d, J = 2.8 Hz, 0.03H), 2.47 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.1, 140.9, 132.5, 129.5, 128.7, 128.4, 128.0, 127.7, 127.1, 126.9, 72.3 (t, $J_{C\text{-}D}$ = 22.3 Hz) ppm.

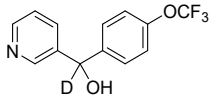


(5-Bromo-2-chlorophenyl)(4-ethoxyphenyl)methan-*d*-ol (**2o'**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 4/1, Rf = 0.4), 63.2 mg (61% yield). Mp: 101-103 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J = 2.0 Hz, 1H), 7.33 (dd, J_1 = 8.0 Hz, J_2 = 2.4 Hz, 1H), 7.26 (s, 1H), 7.24 (s, 1H), 7.18 - 7.16 (m, 1H), 6.84 (d, J = 8.8 Hz, 2H), 6.05 (s, 0.02H), 4.01 (q, J = 7.2 Hz, 2H), 2.41 (s, 1H), 1.40 (t, $J_{C\text{-}D}$ = 7.2 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 143.1, 133.5, 131.6, 131.2, 131.0, 130.7, 128.4, 121.0, 114.6, 71.8 (t, $J_{C\text{-}D}$ = 22.0 Hz), 63.5, 14.8 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{14}DO_2ClBr$ 342.0007;

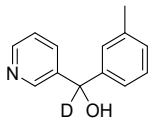
Found 342.0002.



Phenyl(pyridin-3-yl)methan-*d*-ol (**4a'**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 40.8 mg (73% yield). Mp: 65-67 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (s, 1H), 8.31 (d, J = 4.0 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.33 - 7.26 (m, 5H), 7.19 (dd, J_1 = 8.0 Hz, J_2 = 4.8 Hz, 1H), 5.80 (s, 0.02H), 4.66 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 147.8, 143.2, 139.8, 134.4, 128.6, 127.8, 126.5, 123.4, 73.3 (t, $J_{C\text{-}D}$ = 21.8 Hz) ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{11}DNO$ 187.0982; Found 187.0981.



Pyridin-3-yl(4-(trifluoromethoxy)phenyl)methan-*d*-ol (**4c'**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 56.8 mg (70% yield). Mp: 87-89 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (d, J = 1.2 Hz, 1H), 8.30 (dd, J_1 = 4.8 Hz, J_2 = 1.6 Hz, 1H), 7.68 - 7.66 (m, 1H), 7.36 (d, J = 8.8 Hz, 2H), 7.22 (dd, J_1 = 7.6 Hz, J_2 = 4.8 Hz, 1H), 7.17 (d, J = 8.0 Hz, 2H), 5.81 (s, 0.01H), 5.05 (brs, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.7 (d, J = 1.4 Hz), 148.4, 147.8, 141.9, 139.5, 134.6, 128.0, 123.7, 121.1, 120.4 (q, $J_{C\text{-}F}$ = 255.7 Hz), 72.6 (t, $J_{C\text{-}D}$ = 21.8 Hz) ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{10}DF_3NO_2$ 271.0805; Found 271.0805.



Pyridin-3-yl(m-tolyl)methan-*d*-ol (**4e'**, white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 30/1, Rf = 0.3), 37.0 mg (61% yield). Mp: 62-64 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (s, 1H), 8.34 (d, *J* = 3.6 Hz, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.24 - 7.19 (m, 2H), 7.14 (t, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 7.6 Hz, 1H), 5.78 (s, 0.01H), 3.96 (brs, 1H), 2.32 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 147.9, 143.2, 139.8, 138.4, 134.4, 128.6, 128.6, 127.2, 123.6, 123.5, 73.4 (t, $J_{C\text{-}D}$ = 22.0 Hz), 21.4 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{13}DNO$ 201.1138; Found 201.1138.

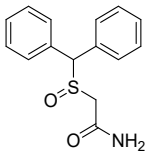
Synthesis of Modafinil and Adrafinil.

Synthesis of 5a: A mixture of **2b** (184 mg, 1.0 mmol) and thioglycolic acid (92 mg, 1.0 mmol) in trifluoroacetic acid (1.5 mL) was stirred for 2.5 h at room temperature. The solvent was removed and H_2O (10 mL) was added, the crude solid was obtained by filtration. The solid was washed with hexanes (10 mL) and dried to afford **5b** (237.7 mg, 0.92 mmol, 92% yield) as a white solid without further purification.

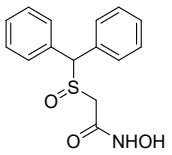
Synthesis of 6b: Oxalyl chloride (0.5 mL) and 20 μL DMF were added into a solution of **5b** in anhydrous DCM (3 mL). The reaction was stirred for 3 h at room temperature. Then 2 mL MeOH was added dropwise at 0 ℃. The solvent was removed under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Petroleum ether/ EtOAc = 30:1) to give **6b** as a colorless oil (213.0 mg, 0.78 mmol, 85% yield).

Synthesis of 7b: H_2O_2 (30% w/w aqueous solution, 1.56 mmol) was slowly added into a solution of **6b** in AcOH (2 mL). The reaction was stirred for 15 min at 50 ℃ and quenched by the addition of saturated $Na_2S_2O_3$ aqueous solution (1 mL). Then the mixture was extracted with CH_2Cl_2 (15 mL × 3). The combined organic phase was dried over $MgSO_4$, filtered and evaporated under reduced pressure to afford **7b** as a colorless oil (220.4 mg, 0.76 mmol, 98% yield) without further purification.

Synthesis of Modafinil (Modafinil as the example): A mixture of **7b** and NH_3 (2.0 mol/L in MeOH, 2 mL) was stirred for 4.5 h at 50 ℃. The solvent was evaporated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Dichlormethan/Methanol = 15/1) to give Modafinil as a white solid (161.3 mg, 0.59 mmol, 78% yield).



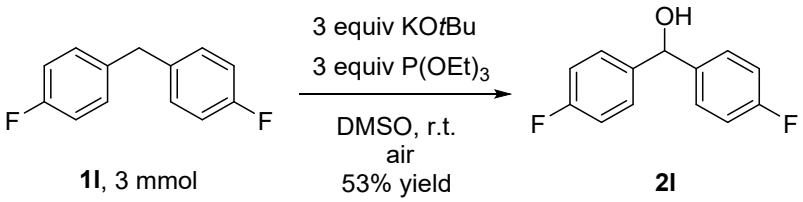
Modafinil [21]: (2-(Benzhydrylsulfinyl)acetamide) (white solid), purification by flash column chromatography (eluent: Dichlormethan/Methanol = 15/1), 161.3 mg (total 41% yield from **1b**). ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.68 (brs, 1H), 7.53 - 7.51 (m, 4H), 7.42 (td, J_1 = 7.2 Hz, J_2 = 1.2 Hz, 4H), 7.38 - 7.32 (m, 3H), 5.35 (s, 1H), 3.38 (d, J = 13.6 Hz, 1H), 3.24 (d, J = 13.6 Hz, 1H) ppm. ^{13}C NMR (100 MHz, DMSO-d_6): δ 167.2, 137.9, 135.7, 130.5, 129.8, 129.2, 128.7, 128.7, 69.6, 56.9 ppm.



Adrafinil [21]: (2-(Benzhydrylsulfinyl)-*N*-hydroxyacetamide) (white solid),

purification by flash column chromatography (eluent: Dichlormethan/Methanol = 15/1), 161.3 mg (total 38% yield from **1b**). ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.79 (brs, 1H), 9.15 (brs, 1H), 7.51 - 7.36 (m, 10H), 5.39 (s, 1H), 3.32 (s, 1H), 3.08 (d, J = 13.2 Hz, 1H) ppm. ^{13}C NMR (100 MHz, DMSO-d_6): δ 161.2, 137.0, 134.9, 129.7, 129.1, 128.6, 128.0, 128.0, 69.0, 53.8 ppm.

3.3 10-Equivalent-Scale Experiments (2l as the example).

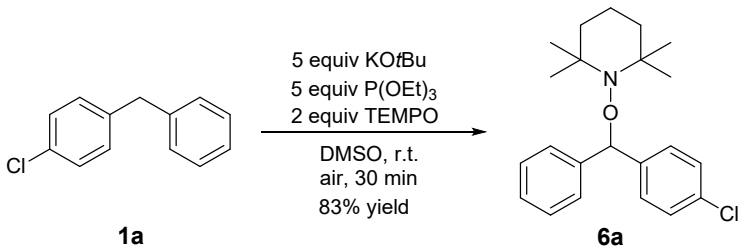


1l, 3 mmol **2l**

Under air atmosphere, 1.55 mL $P(OEt)_3$ (9 mmol) was added to a solution of KO*t*Bu (1.01 g, 9 mmol) in 6 mL DMSO-d_6 at room temperature. Substrate **1l** (0.61 g, 3 mmol) was added into the solution. The mixture was quenched with 20 mL H_2O after 2 h. Then the mixture was extracted with CH_2Cl_2 (25 mL × 3), washed sequentially with saturated sodium chloride solution (30 mL). The combined organic phase was dried over $MgSO_4$, filtered and evaporated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Petroleum ether/EtOAc = 4/1) to give **2l** as a colorless oil (350 mg, 53% yield).

4. Mechanism study

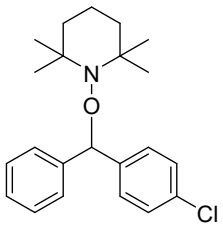
4.1 Radical trapping experiments.



1a **6a**

4-Chlorodiphenylmethane (**1a**, 61 mg, 0.3 mmol) was added into a mixture of TEMPO (70 mg, 0.45 mmol), KO*t*Bu (168 mg, 1.5 mmol) and DMSO (1.0 mL) at 25 °C under air. The reaction mixture was then stirred for 30 min. The mixture was

quenched with 20 mL H_2O, diluted by CH_2Cl_2 (30 mL), washed with saturated NaCl (aq), dried over Na_2SO_4, and the organic phase was evaporated under reduced pressure. The resulting residue was purified by flash chromatography over silica gel (Petroleum ether/EtOAc = 50/1) to give **6a** as a colorless oil (89 mg, 83% yield).



1-((4-Chlorophenyl)(phenyl)methoxy)-2,2,6,6-tetramethylpiperidine (**6a**, white solid), purification by flash column chromatography (eluent: Petroleum ether/EtOAc = 50/1), 89 mg (83% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 - 7.22 (m, 8H), 7.18 - 7.14 (m, 3H), 5.62 (s, 1H), 1.55 (s, 1H), 1.42 (d, *J* = 4.4 Hz, 4H), 1.29 - 1.26 (m, 1H), 1.14 (s, 6H), 0.73 (d, *J* = 6.0 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 143.1, 132.0, 128.1, 128.0, 127.7, 126.5, 126.3, 89.7, 59.6, 40.1, 33.8, 33.6, 20.1, 16.8 ppm. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}ClNO$ 358.1938; Found 358.1937.

4.2 EPR experiments.

The first and the second oven-dried schlenk tube equipped with a stir bar were loaded with DMSO (1.0 mL), KO*t*Bu (168 mg, 1.5 mmol), and $P(OEt)_3$ (155 μL, 0.9 mmol). The third and the fourth oven-dried schlenk tube equipped with a stir bar were loaded with DMSO (1.0 mL), KO*t*Bu (168 mg, 1.5 mmol), $P(OEt)_3$ (155 μL, 0.9 mmol) and Diphenylmethane **1b** (50 mg, 0.3 mmol). These mixtures were stirred at 25 ℃ for ten min, followed by the addition of 5,5-dimethyl 1-pyridine *N*-oxide (DMPO) (68 mg, 0.6 mmol) to the second and the fourth solution respectively. These solutions were stirred for another five min with sampling and analyzing by EPR respectively.

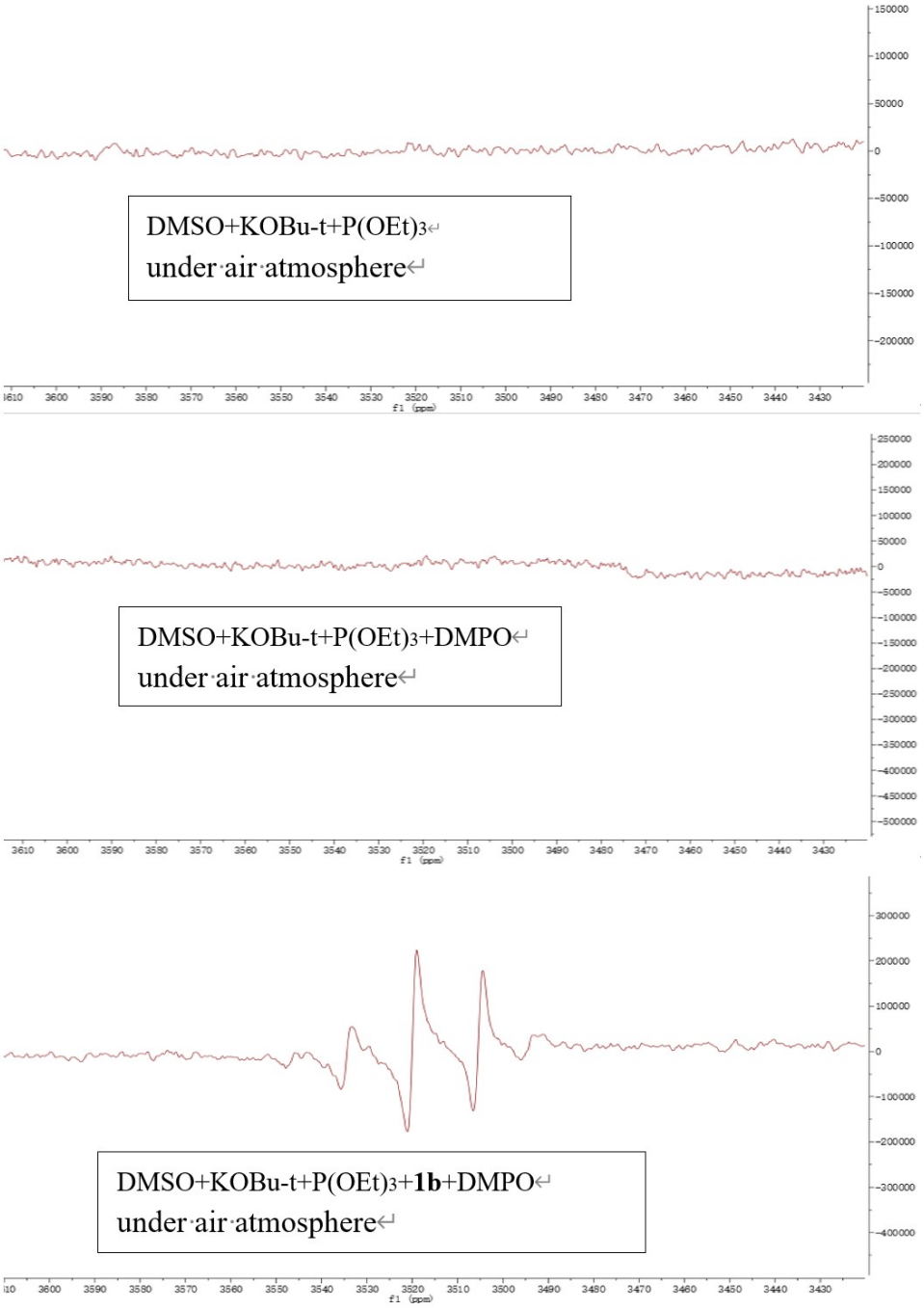
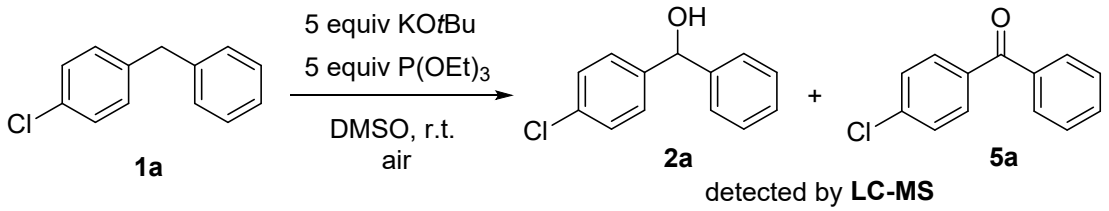


Figure S1. The EPR spectrum

4.3 LC-MS and GC-MS analysis.



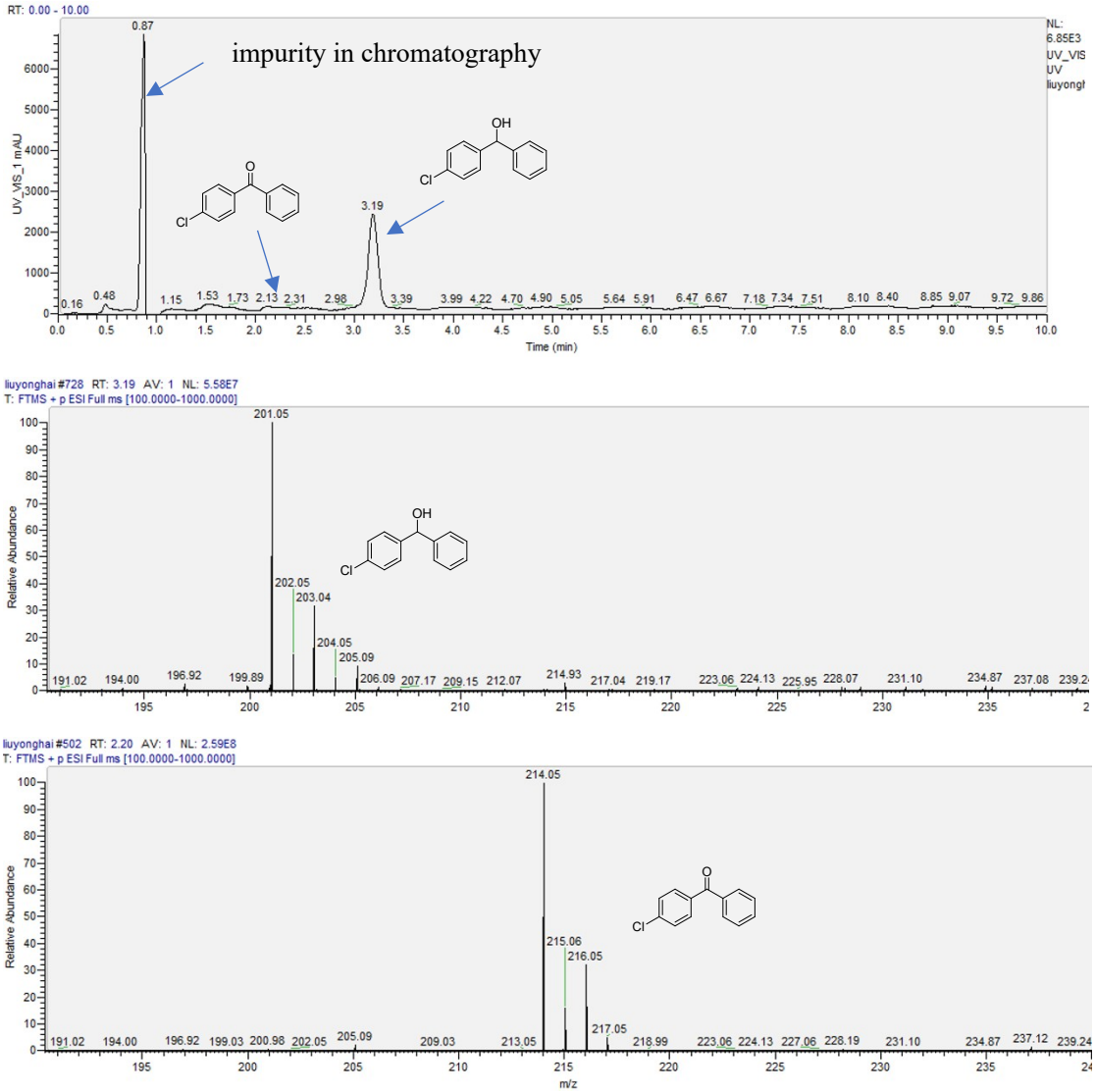
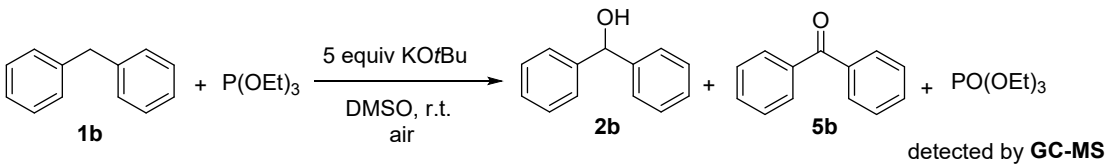


Figure S2. LC-MS analysis for characterization of corresponding byproducts by employing **1c** as the substrate.



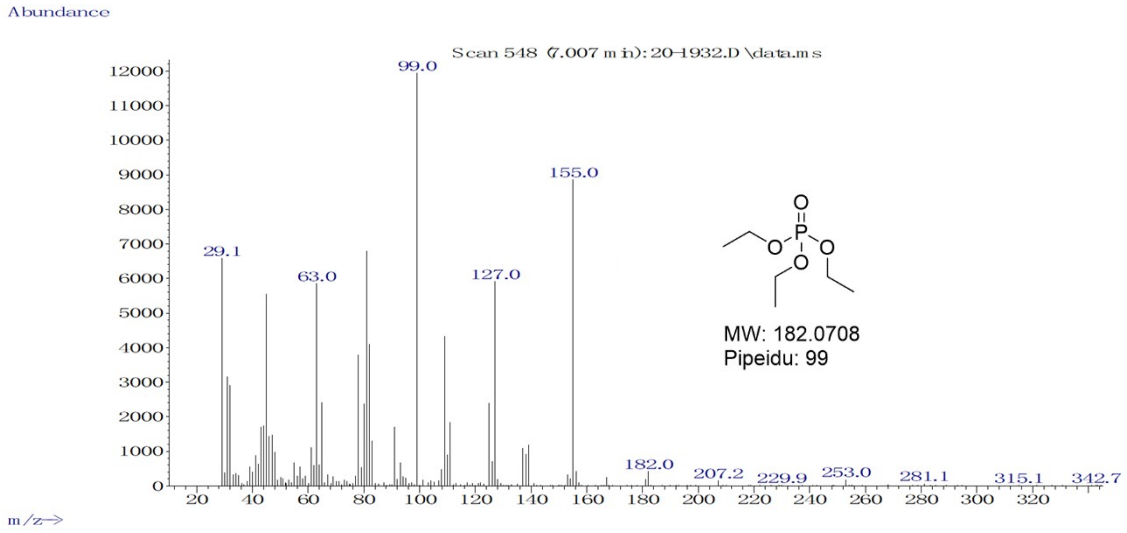


Figure S3. GC-MS analyses for characterization of Triethyl phosphate by employing **1a** as the substrate.

4.4 Peroxide reduction by $P(OEt)_3$.

4.4.1 ^{1}H NMR Experiments. Hydroperoxydiphenylmethane was divided into two samples equally and the second one was treated with 3 equivalent $P(OEt)_3$. The samples were analysed by ^{1}H NMR and the spectrums were displayed below. Hydroperoxydiphenylmethane and $P(OEt)_3$ reacts nearly quantitatively at a 1:1 relationship to give diphenylmethanol and $PO(OEt)_3$ respectively. Hydroperoxydiphenylmethane was prepared according to the method reported.[22]

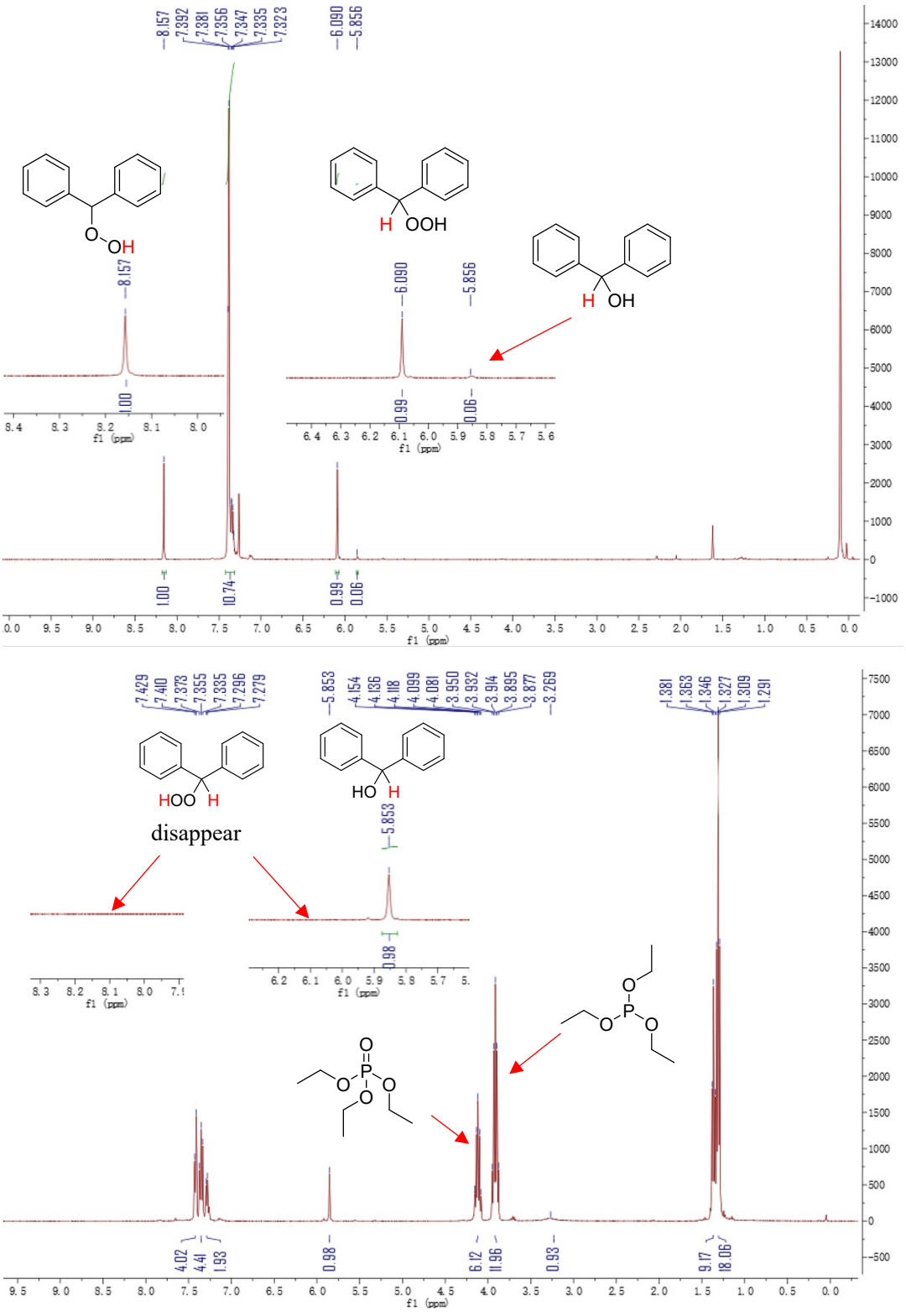


Figure S4. Peroxide reduction analysed by ^{1}H NMR spectrum

4.4.2. The formation of $PO(OEt)_3$ and product (2b as example) analysed by GC.

Mesitylene was used as internal standard in GC, and the standard curve was showed as below:

m ($PO(OEt)_3$)	s ($PO(OEt)_3$)	m (Mesitylene)	s (Mesitylene)	s($PO(OEt)_3$) /s(Mesitylene)	m($PO(OEt)_3$) /m(Mesitylene)
42.8 mg	818.41	37.2	1775.86	0.46085	1.15054
51.1	1031.33	19.1	981.9	1.05034	2.67539
36.8	1216.34	55.9	4647.16	0.26174	0.65832

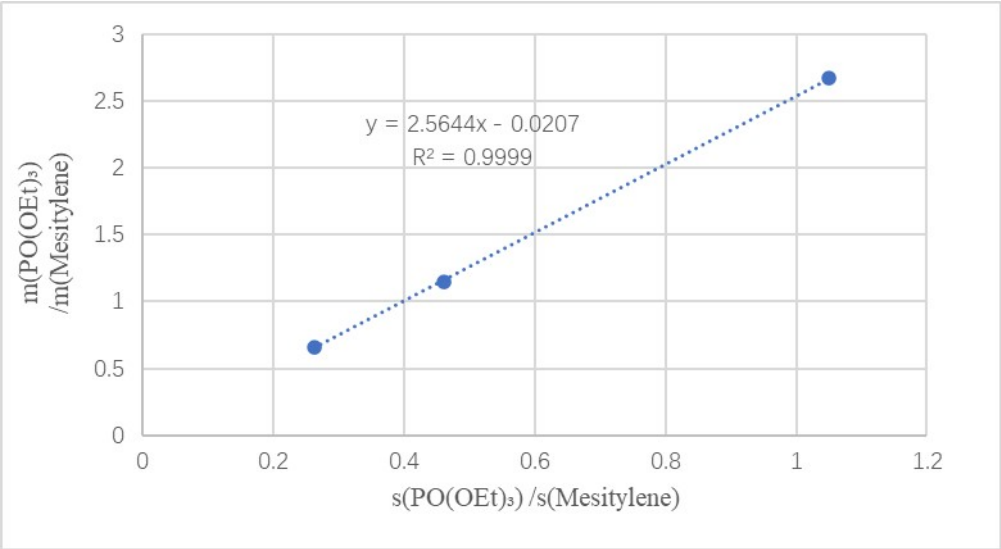


Figure S5. Standard curve of $PO(OEt)_3$ and Mesitylene

m (**2b**)	s (**2b**)	m (Mesitylene)	s (Mesitylene)	s(**2b**) /s(Mesitylene)	m(**2b**) /m(Mesitylene)
48.7	2533.28	50.5	3130.14	0.80932	0.96436
57.6	2477.63	42	2152.46	1.15107	1.37143
19.1	1328.61	31.6	2420.47	0.54891	0.60443

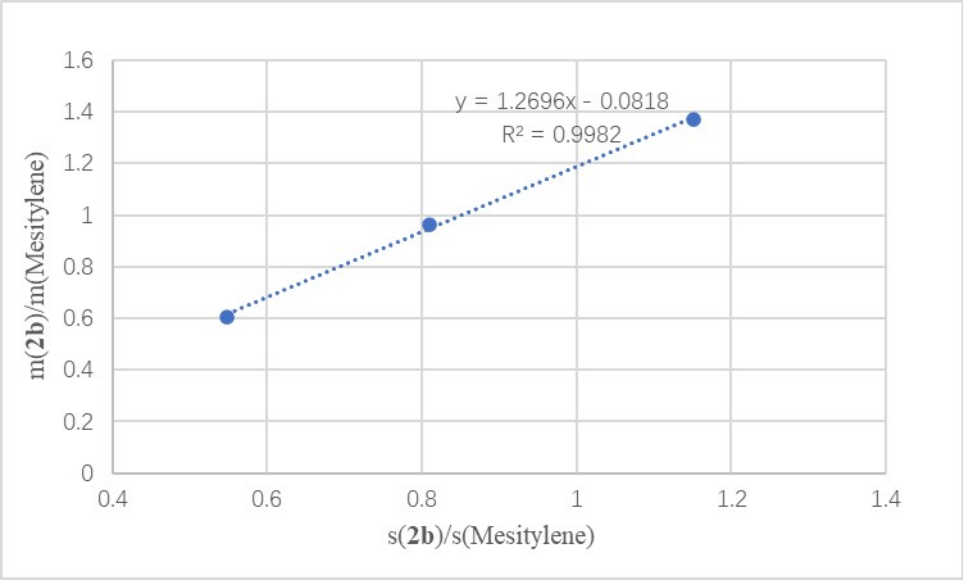


Figure S6. Standard curve of **2b** and Mesitylene

According to the correction equations above, we can quantitatively analyze the amount of **2b** and $PO(OEt)_3$ produced at each time point. And the results were showed as below:

([P] mmol)

Time (min)	[**2b**]	[$PO(OEt)_3$]
3	0.04670	0.08646
6	0.06687	0.13015
10	0.10581	0.15316
15	0.15094	0.19794
20	0.19084	0.24146

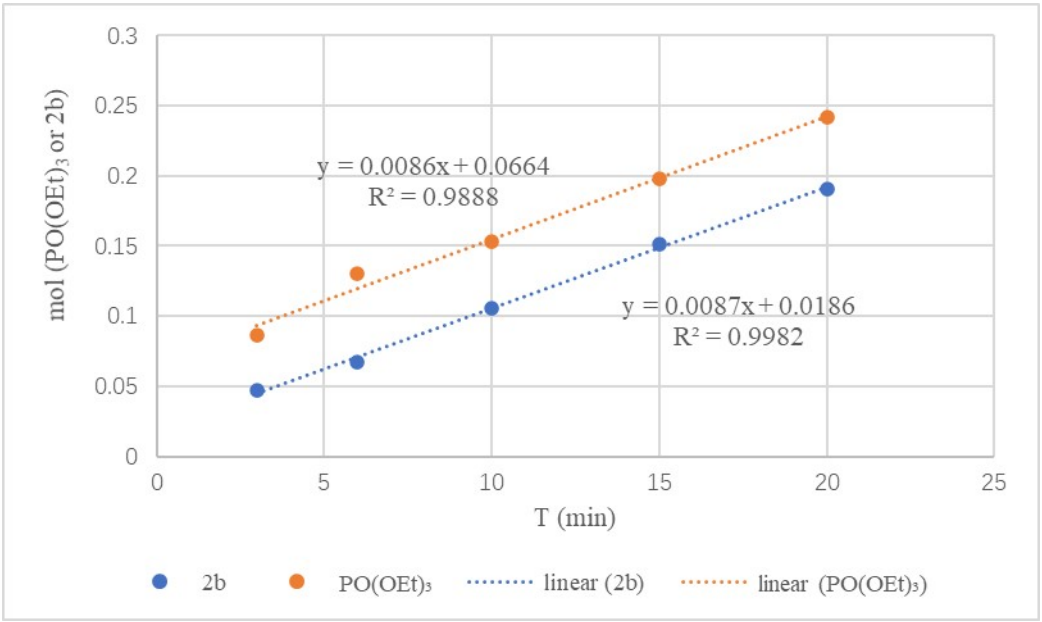
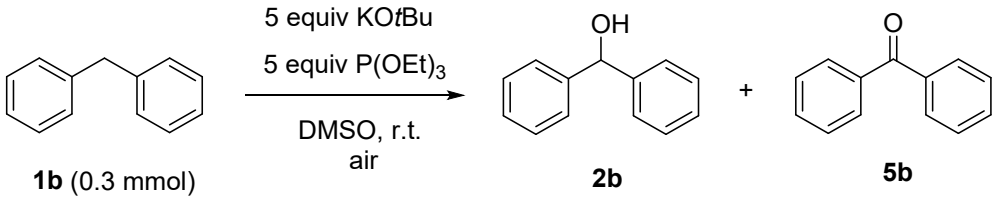


Figure S7. The formation of $PO(OEt)_3$ and product

4.5 Reaction studies by GC analysis.

4.5.1 Reaction progress analysis. The rate of reaction using Diphenylmethane (**1b**) as the substrate at 298 K was analysed by GC using Mesitylene as internal standard.



m (**1b**)	s (**1b**)	m (Mesitylene)	s (Mesitylene)	s(**1b**) /s(Mesitylene)	m(**1b**) /m(Mesitylene)
49	3121.35	50.5	3130.14	0.99719	0.97030
25.8	1377.13	42	2152.46	0.63979	0.61429
26.5	2162.49	31.6	2420.47	0.89342	0.83861

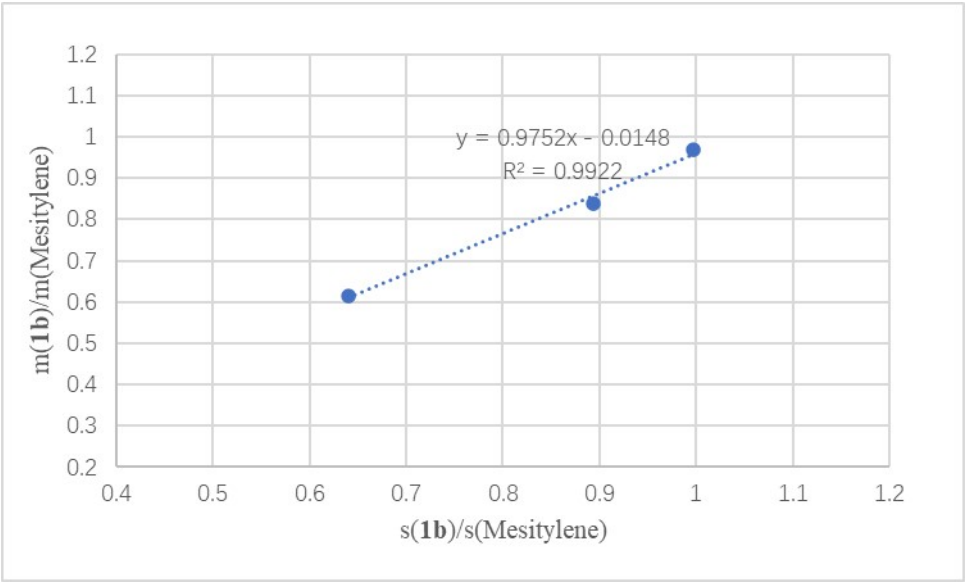


Figure S8. Standard curve of **1b** and Mesitylene

m (**5b**)	s (**5b**)	m (Mesitylene)	s (Mesitylene)	s(**5b**) /s(Mesitylene)	m(**5b**) /m(Mesitylene)
33.3	1361.12	33.7	1445	0.94195	0.98813
44.3	2017.52	22.6	1201.15	1.67966	1.96018
63	2728.34	35.4	1744.36	1.56409	1.77966

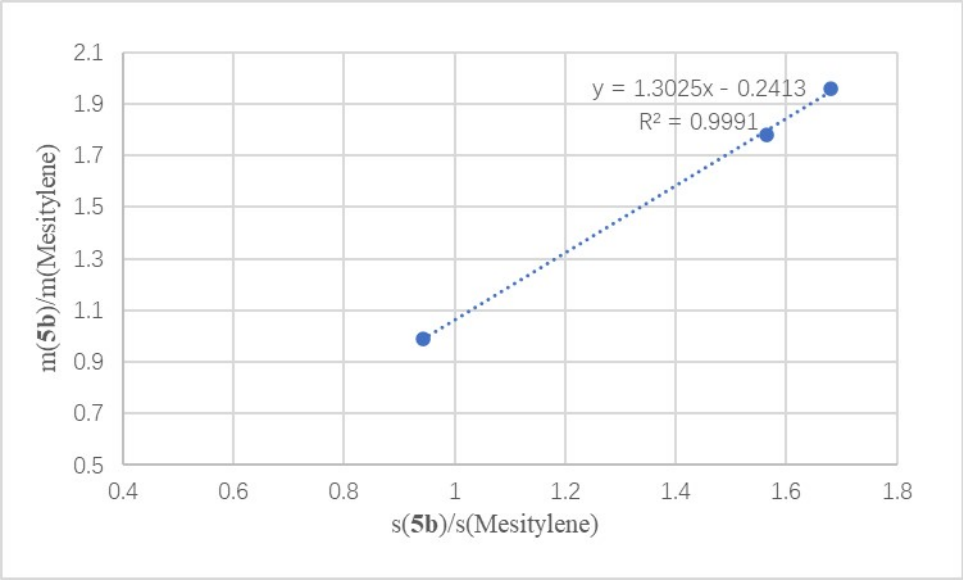


Figure S9. Standard curve of **5b** and Mesitylene

The amounts of **1b**, **2b** and **5b** at each time point were calculated by GC data and standard curves, and the results were showed as below:

([S] mmol)

Time (min)	[**1b**]	[**2b**]	[**5b**]
0	0.3	0	0
3	0.18586	0.04670	0.00088
6	0.16728	0.06687	0.00171
7.5	0.15082	0.07903	0.00222
10	0.11057	0.10581	0.00349
15	0.07978	0.15094	0.00766
20	0.04522	0.19084	0.01344
30	0.04542	0.21032	0.02129
60	0.02830	0.19900	0.02391

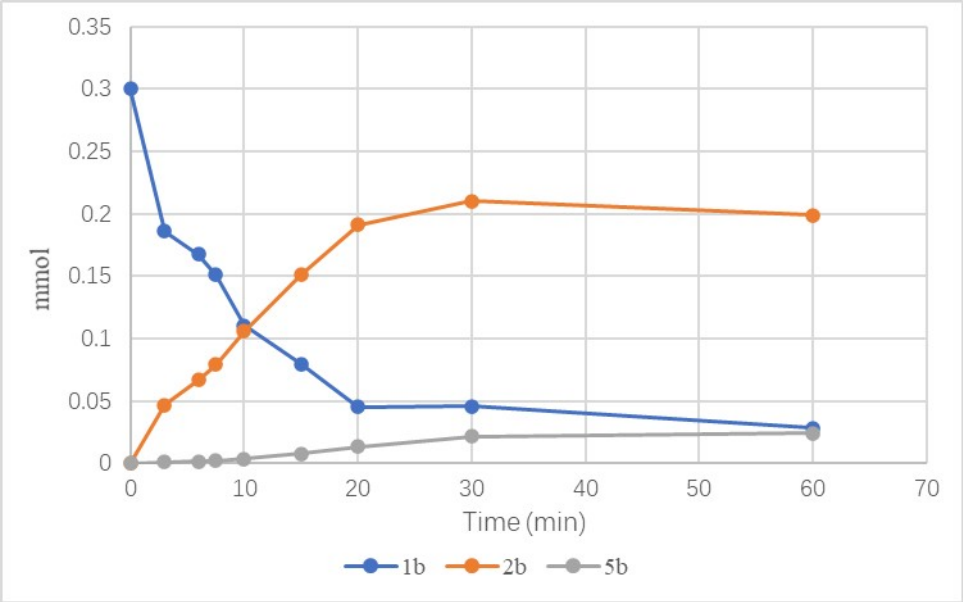


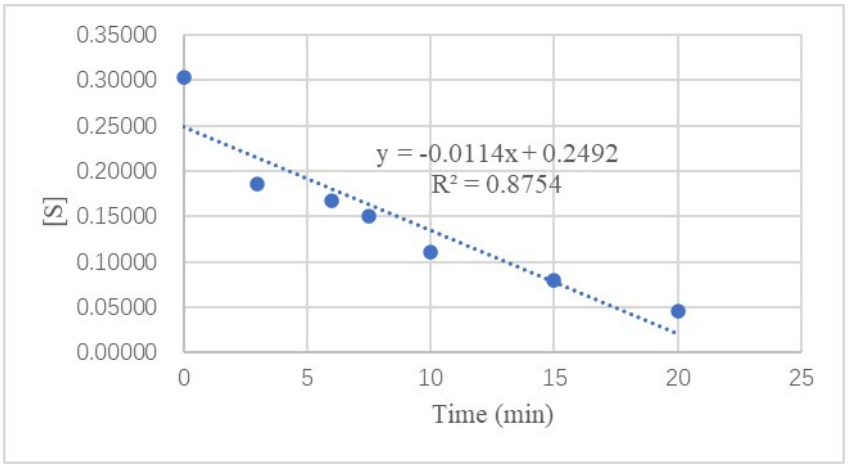
Figure S10. The reaction progress

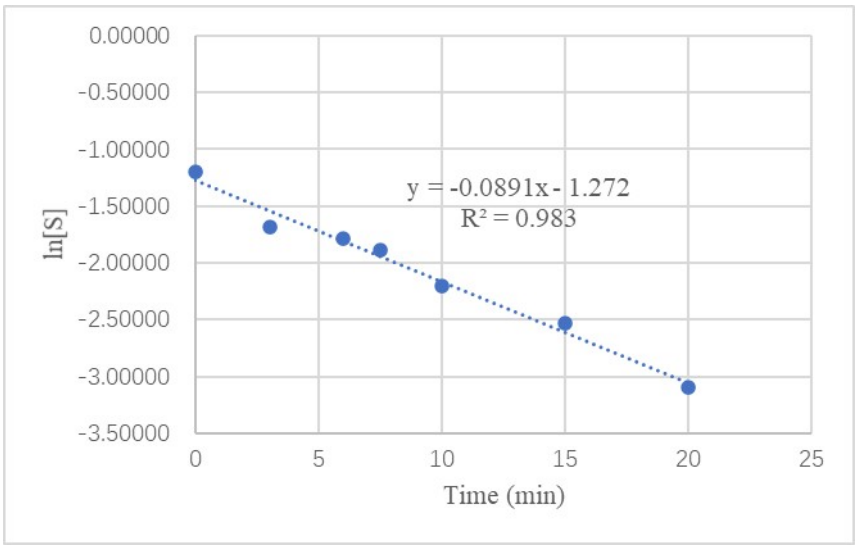
The product (**2b**) increased rapidly in the first 20 minutes with the rapid decreasing of the starting material (**1b**). Then the reaction tended to be flat. By-product (**5b**) was slowly formed in small amounts throughout the reaction process.

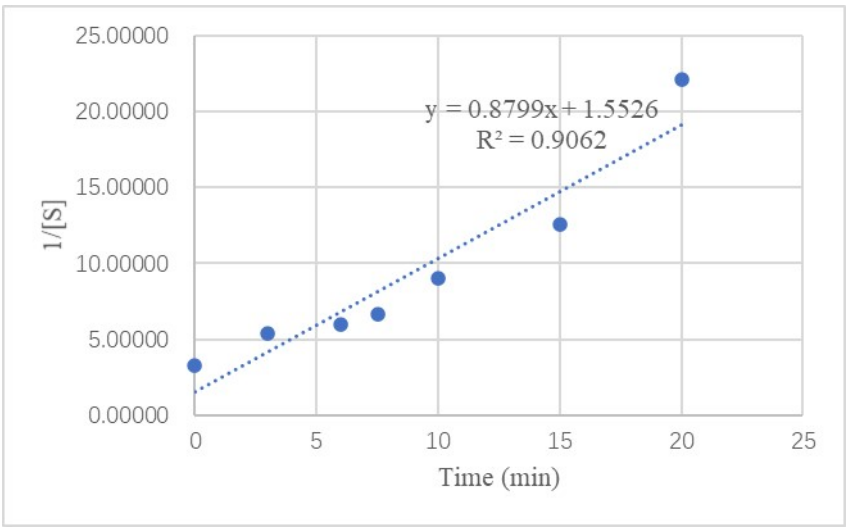
4.5.2 Reaction order analysis.

([S] = **[1b]**, 5 equiv $P(OEt)_3$)

Time (min)	[S]	ln[S]	1/[S]
0	0.30316	-1.19349	3.29859
3	0.18586	-1.68275	5.38032
6	0.16728	-1.78811	5.97813
7.5	0.15082	-1.89169	6.63059
10	0.11057	-2.20210	9.04400
15	0.07978	-2.52843	12.53383
20	0.04522	-3.09622	22.11426



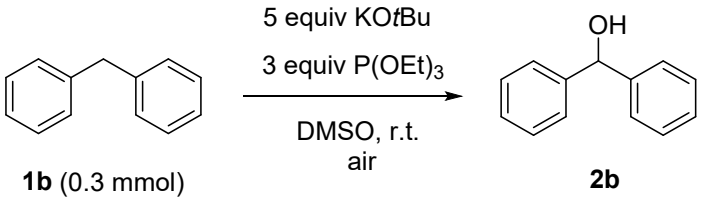




According to the curve fitted above, the first 20 minutes of the reaction showed a 1st-order dependence on the concentration of the substrate, and $t_{1/2}$ = 7.78 min.

4.6 Hammett analysis.

Several substrates **1b**, **1g**, **1h**, **1i** using 3 equivalent $P(OEt)_3$ at 298 K were analyzed by GC using Mesitylene as internal standard. All reactions displayed a 1st-order dependence on the concentration of the substrates. From the 1st-order rate constants, k(min^{-1}) for **1b**, **1g**, **1h**, and **1i**, a strong linear correlation between $\log(k_R/k_H)$ and σ_p-constant (Hammett plot below) was observed.



$k_{\mathbf{1b}} = 0.0582\ min^{-1}$

([S] = **[1b]**, 3 equiv $P(OEt)_3$)

Time (min)	[S]	ln[S]
5	0.20570	-1.58136
10	0.15054	-1.89350
15	0.11489	-2.16379

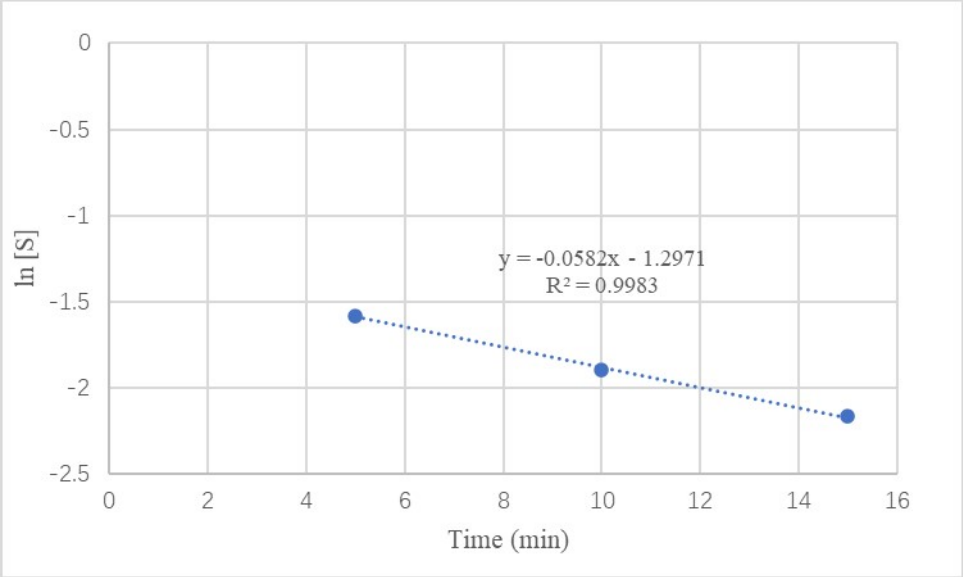
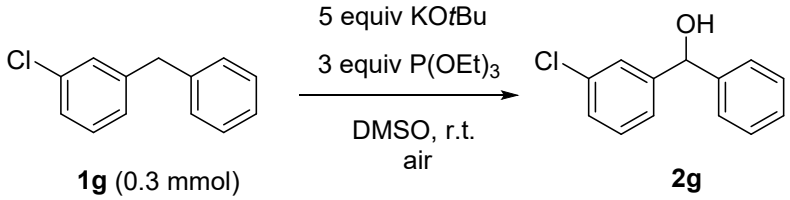


Figure S12. 1st-order rate constants of **1b**



1g (0.3 mmol) **2g**

$k_{1g} = 0.1766\ min^{-1}$

m (**1g**)	s (**1g**)	m (Mesitylene)	s (Mesitylene)	s(**1g**) /s(Mesitylene)	m(**1g**) /m(Mesitylene)
43.2	1446.48	40	1694.99	0.85339	1.08000
30.8	1090.96	30.1	1316.16	0.82890	1.02326
23.2	938.89	23	1143.83	0.82083	1.00870

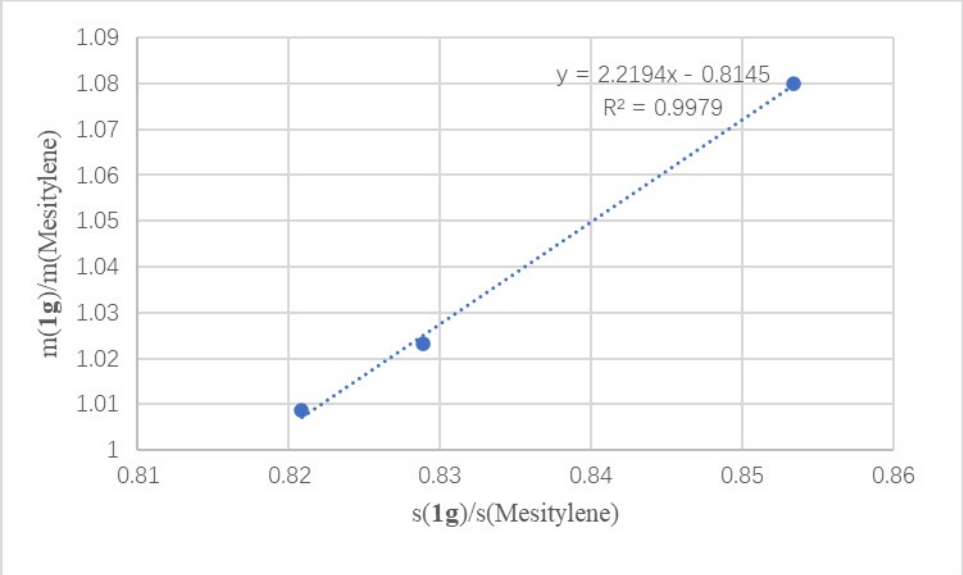


Figure S13. Standard curve of **1g** and Mesitylene

([S] = **[1g]**, 3 equiv $P(OEt)_3$)

Time (min)	[S]	ln[S]
5	0.11181	-2.37765
10	0.03180	-3.23379
15	0.01393	-4.14358

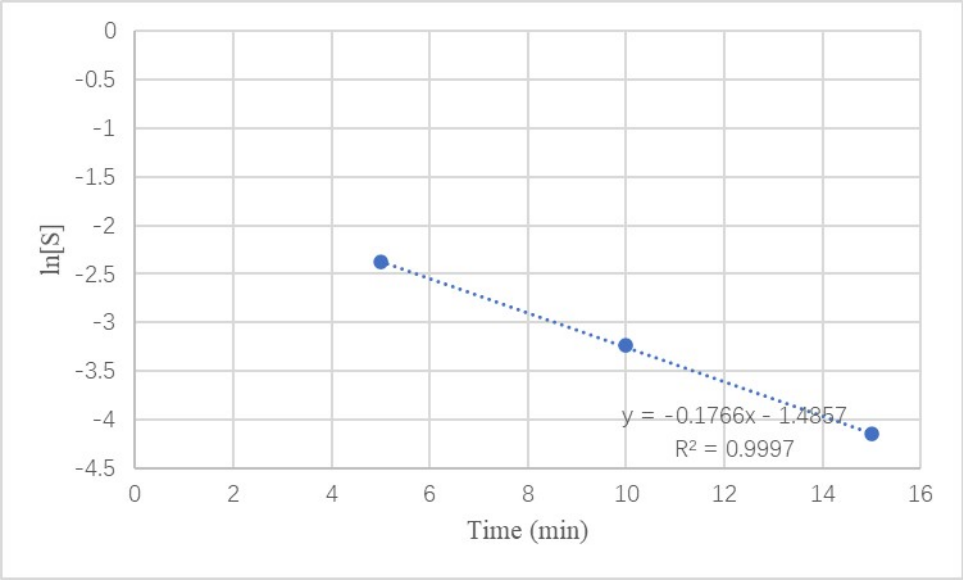
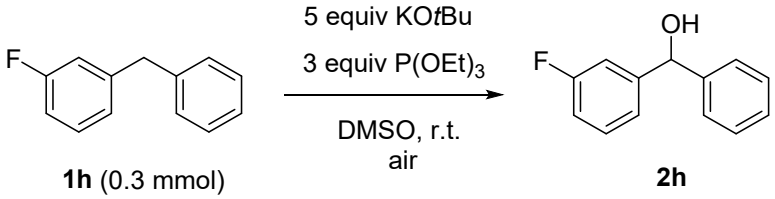


Figure S14. 1st-order rate constants of **1g**



$k_{\mathbf{1h}} = 0.1766\ min^{-1}$

m (**1h**)	s (**1h**)	m (Mesitylene)	s (Mesitylene)	s(**1h**) /s(Mesitylene)	m(**1h**) /m(Mesitylene)
11.1	1003.09	20.6	2356.1	0.42574	0.53883
7.8	427.88	17.8	1232.18	0.34725	0.43820
23.5	1769.68	32.1	3073.5	0.57579	0.73209

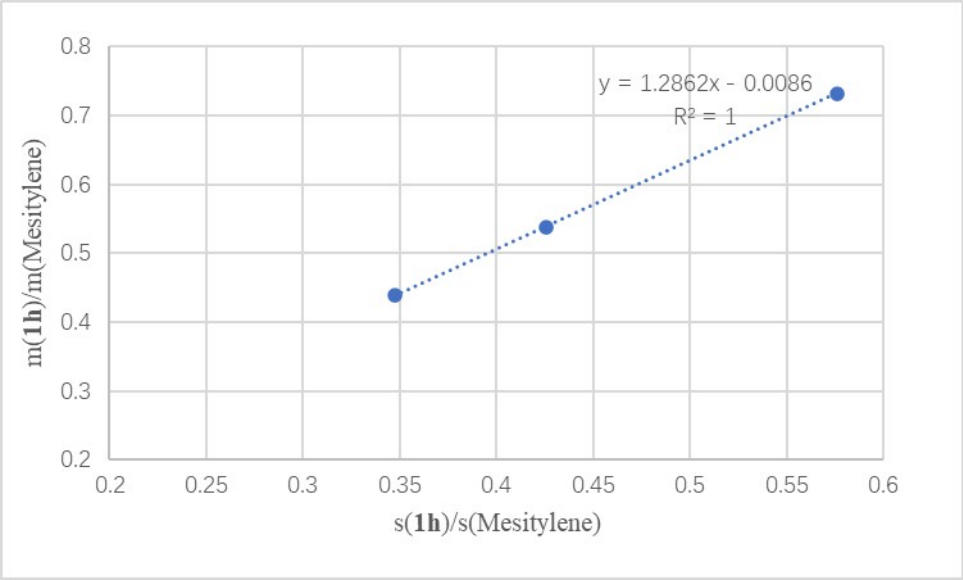


Figure S15. Standard curve of **1h** and Mesitylene

([S] = **[1h]**, 3 equiv $P(OEt)_3$)

Time (min)	[S]	ln[S]
5	0.09277	-2.37765
10	0.03941	-3.23379
15	0.01587	-4.14358

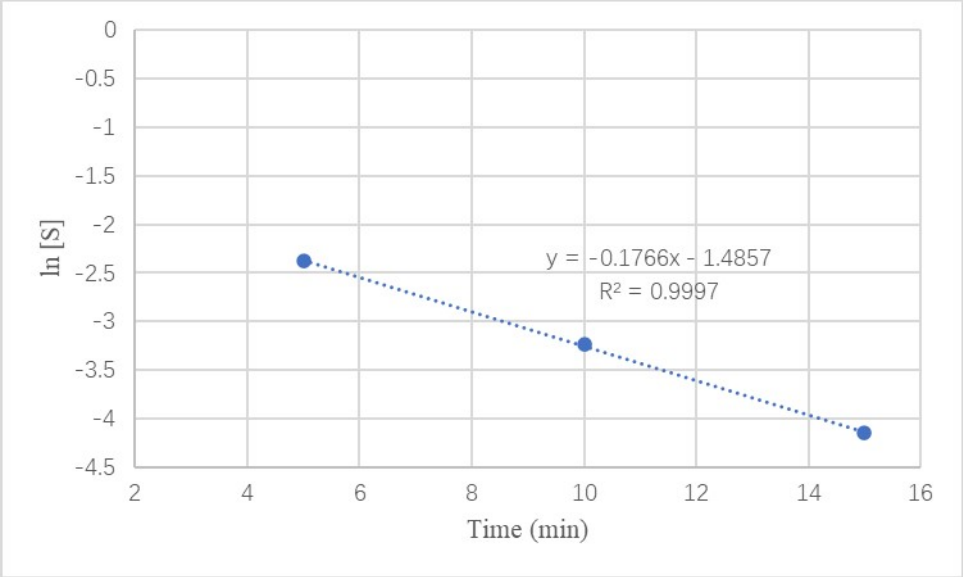
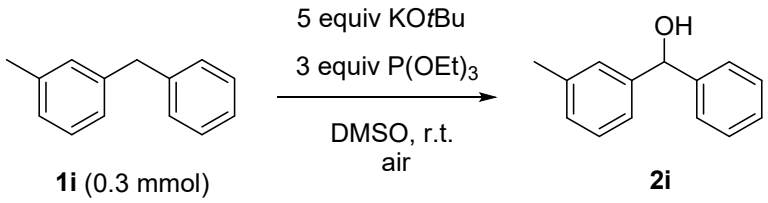


Figure S16. 1st-order rate constants of **1h**



$k_{1i} = 0.0326\ min^{-1}$

m (**1i**)	s (**1i**)	m (Mesitylene)	s (Mesitylene)	s(**1i**) /s(Mesitylene)	m(**1i**) /m(Mesitylene)
34.4	1635.16	29.1	1440.43	1.13519	1.18213
28	1992.83	36.9	2736.34	0.72828	0.75881
21	1480.95	26.5	1839.54	0.80507	0.79245

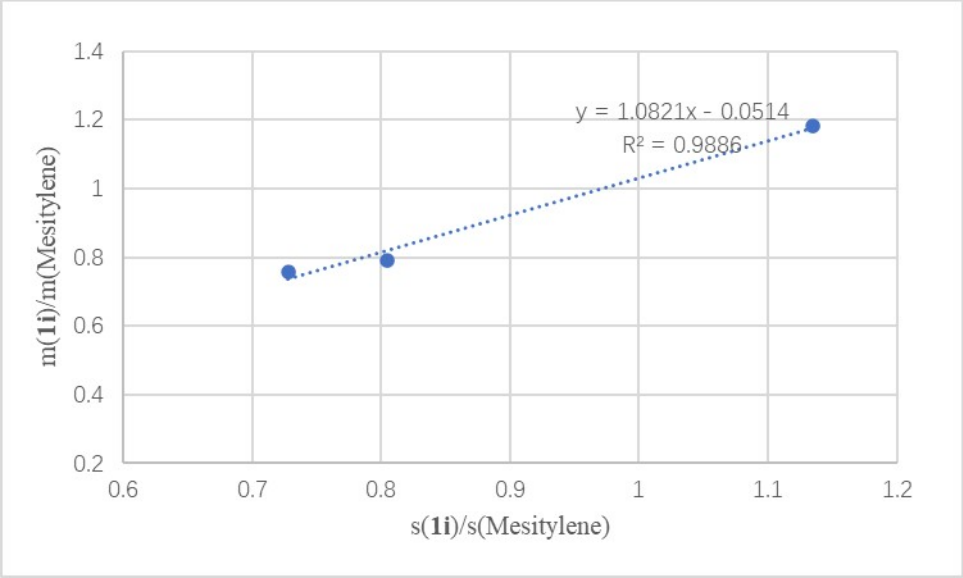


Figure S17. Standard curve of **1i** and Mesitylene

([S] = **[1i]**, 3 equiv $P(OEt)_3$)

Time (min)	[S]	ln[S]
10	0.17832	-1.72420
15	0.14942	-1.90098
20	0.12866	-2.05056

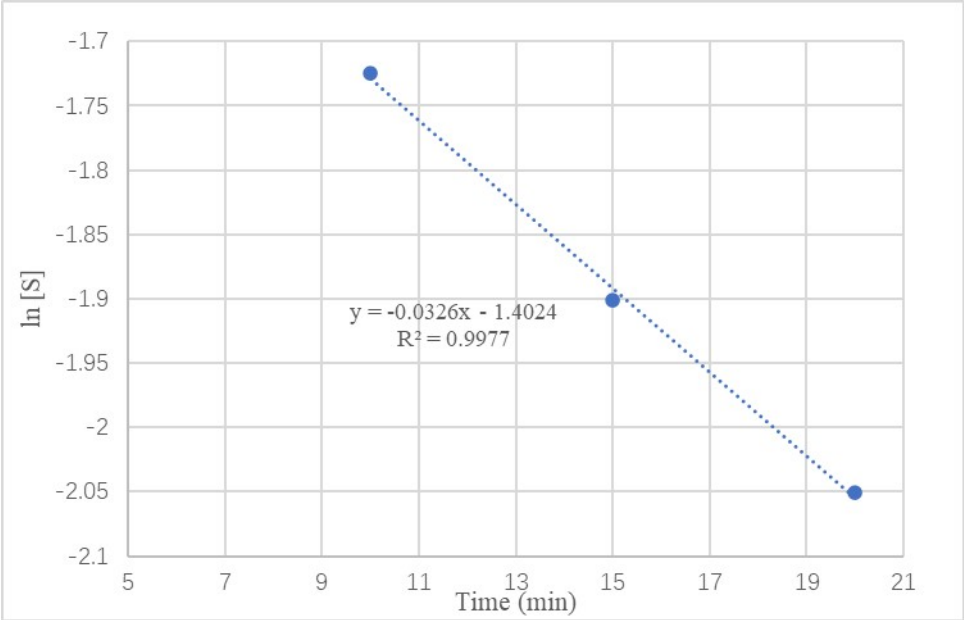
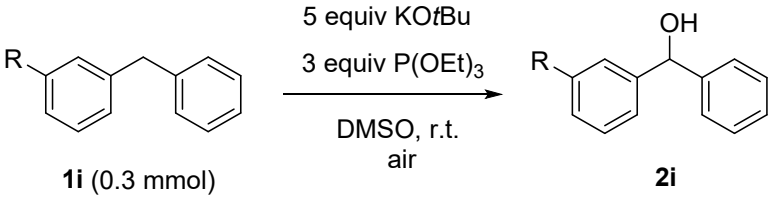


Figure S18. 1st-order rate constants of **1i**



1i (0.3 mmol) **2i**

substrate	R	σ_p	k(min^{-1})	$\log(k_R/k_H)$
1b	m-H	0	0.0582	0
1g	m-F	+0.337	0.1766	0.4821
1h	m-Cl	+0.373	0.1766	0.4821
1i	m-Me	-0.069	0.0326	-0.2517

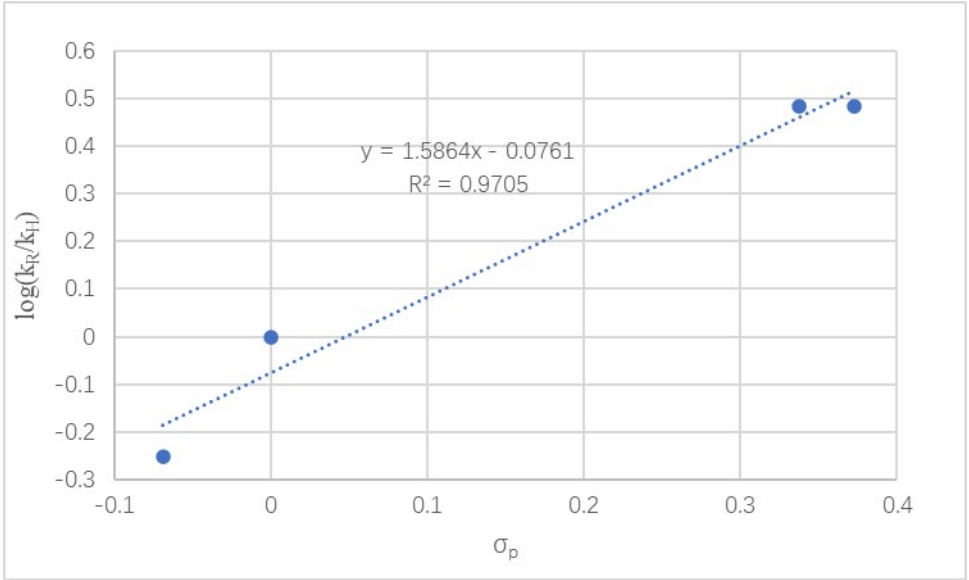
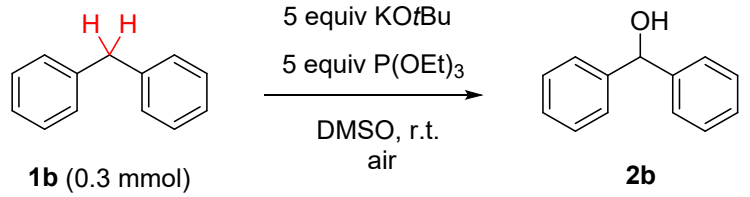


Figure S19. The Hammett analysis

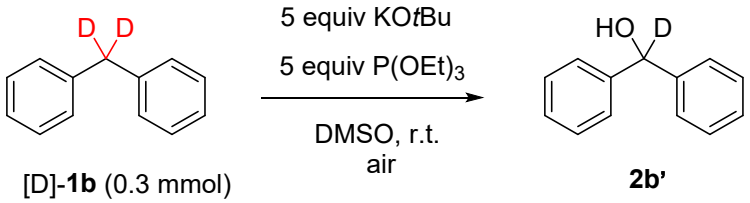
4.7 Kinetic Isotope Effect (KIE) Experiments.

4.7.1 Kinetic Isotope Effect under standard conditions between **1b** and **[D]-1b**.



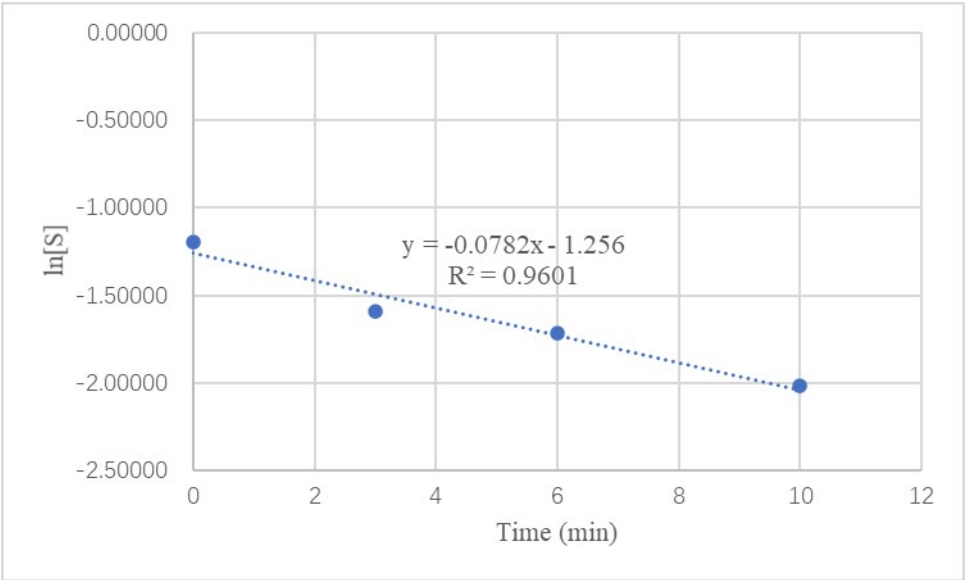
1b (0.3 mmol) **2b**

$K_H = 0.0891$



[D]-**1b** (0.3 mmol) **2b'**

Time (min)	[S]	ln[S]
0	0.30330	-1.19303
3	0.20448	-1.58727
6	0.18018	-1.71378
10	0.13316	-2.01623

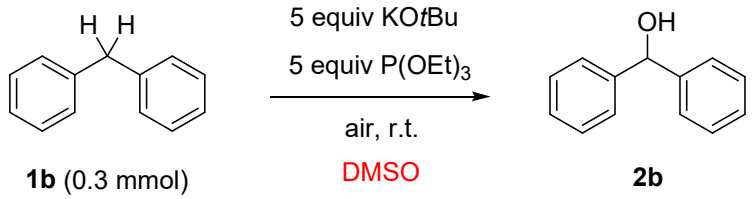


$K_D = 0.0782$

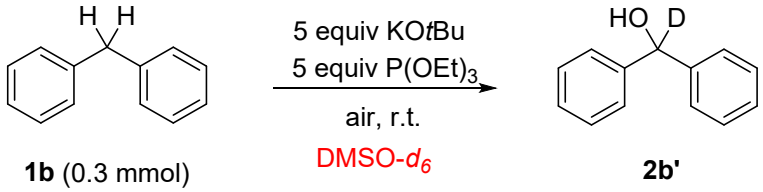
$K_H/K_D = 0.0891/0.0782 = 1.14$

Figure S20. KIE Experiment of **1b**

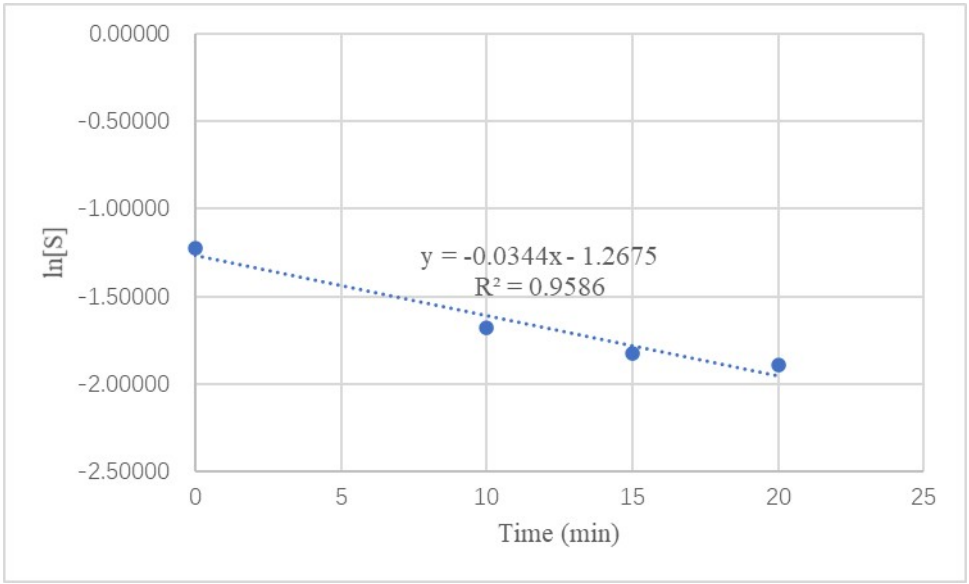
4.7.2 Kinetic Isotope Effect of **1b** under DMSO and DMSO-d_6.



$K_H = 0.0891$



Time (min)	[S]	ln[S]
0	0.29354	-1.22574
10	0.18728	-1.67517
15	0.16158	-1.82277
20	0.15051	-1.89370



$K_{D'} = 0.0344$

$K_H/K_{D'} = 0.0891/0.0344 = 2.59$

Figure S21. KIE Experiment of **DMSO**

5. Reference

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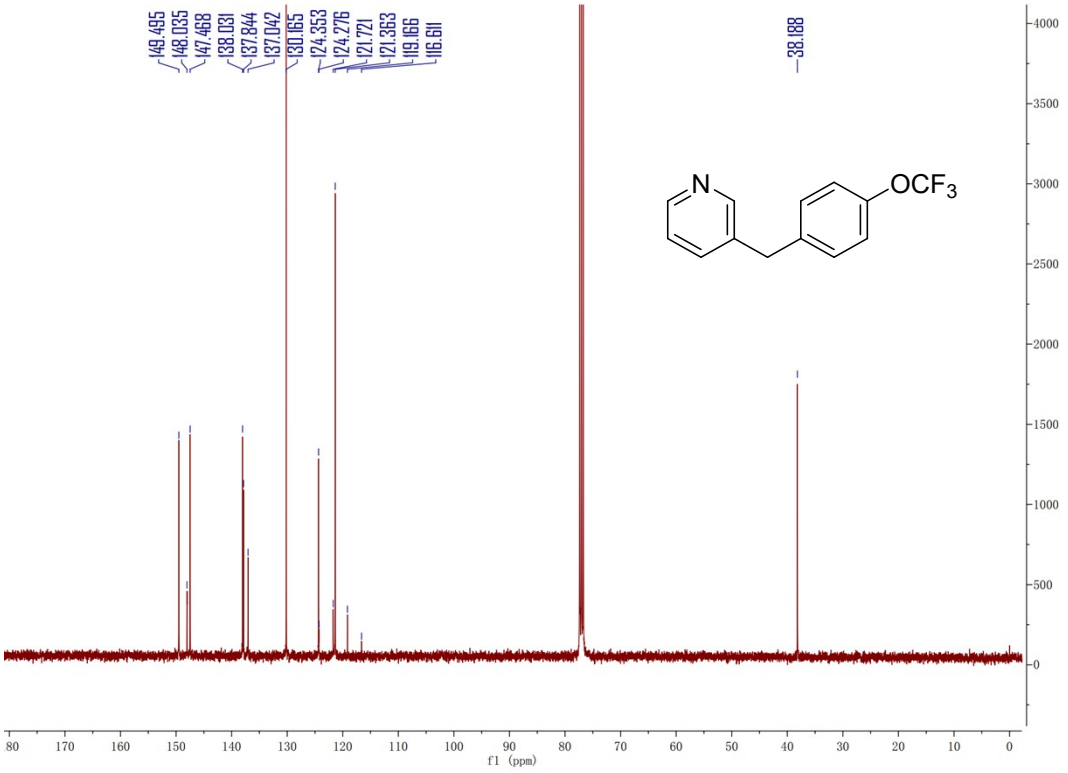
6. ^{1}H and ^{13}C-NMR Spectra Data

3-(4-(Trifluoromethoxy)benzyl)pyridine (3c)

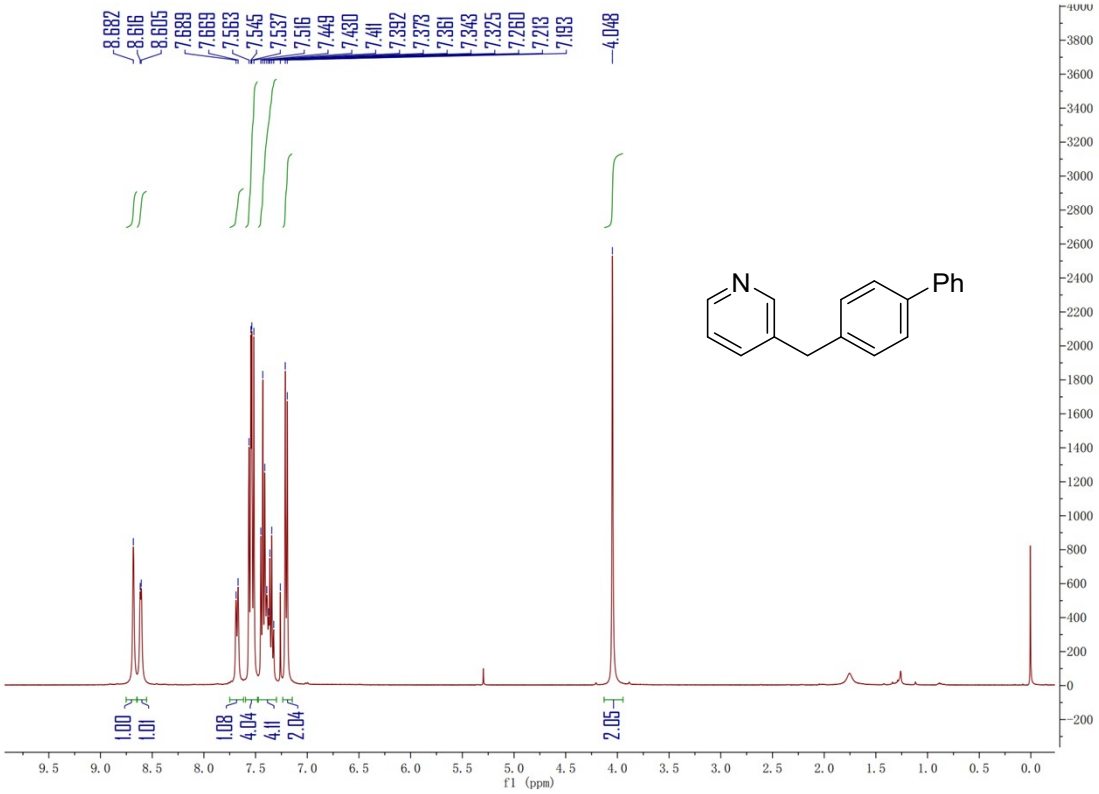
[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]



3-([1,1'-Biphenyl]-4-ylmethyl)pyridine (3d)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



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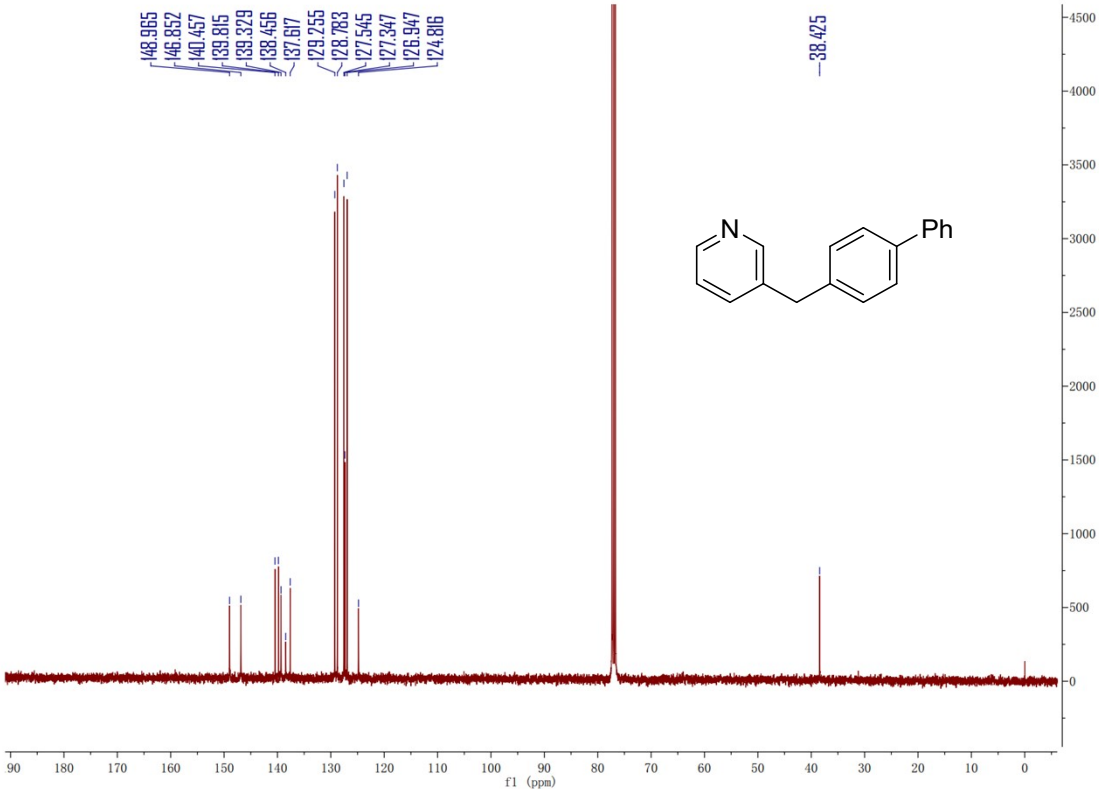

3-(2-Chlorobenzyl)pyridine (3f)

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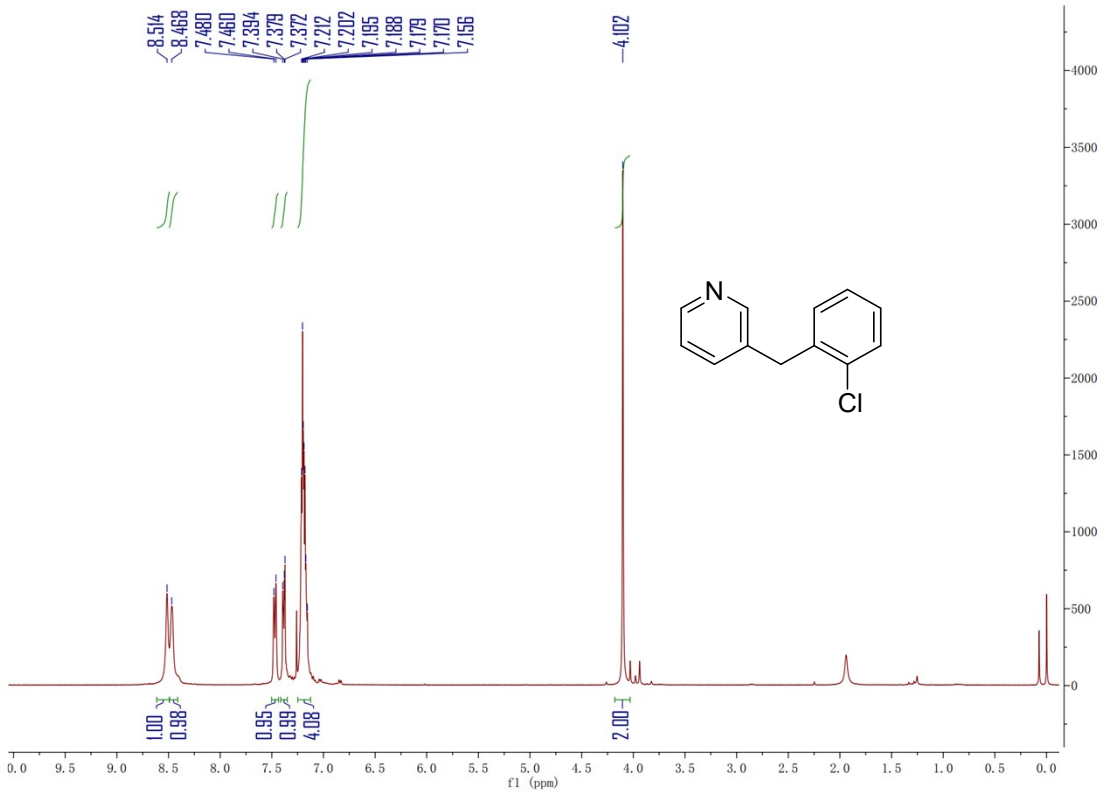

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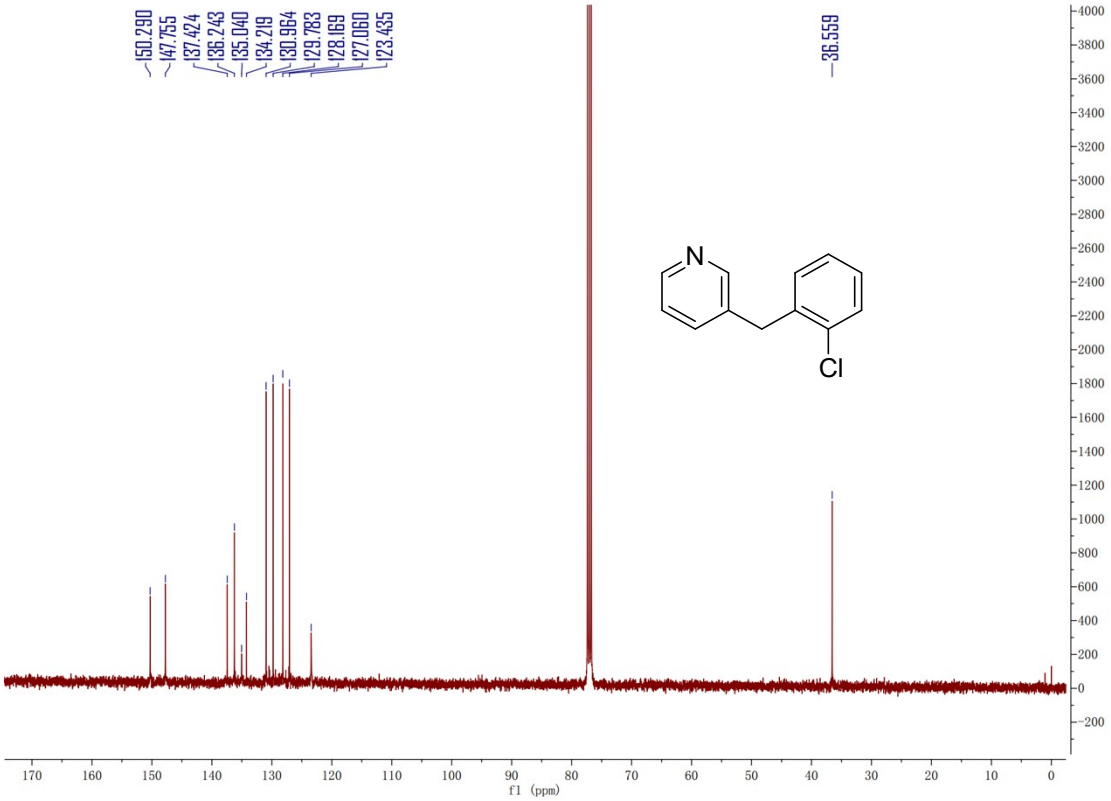
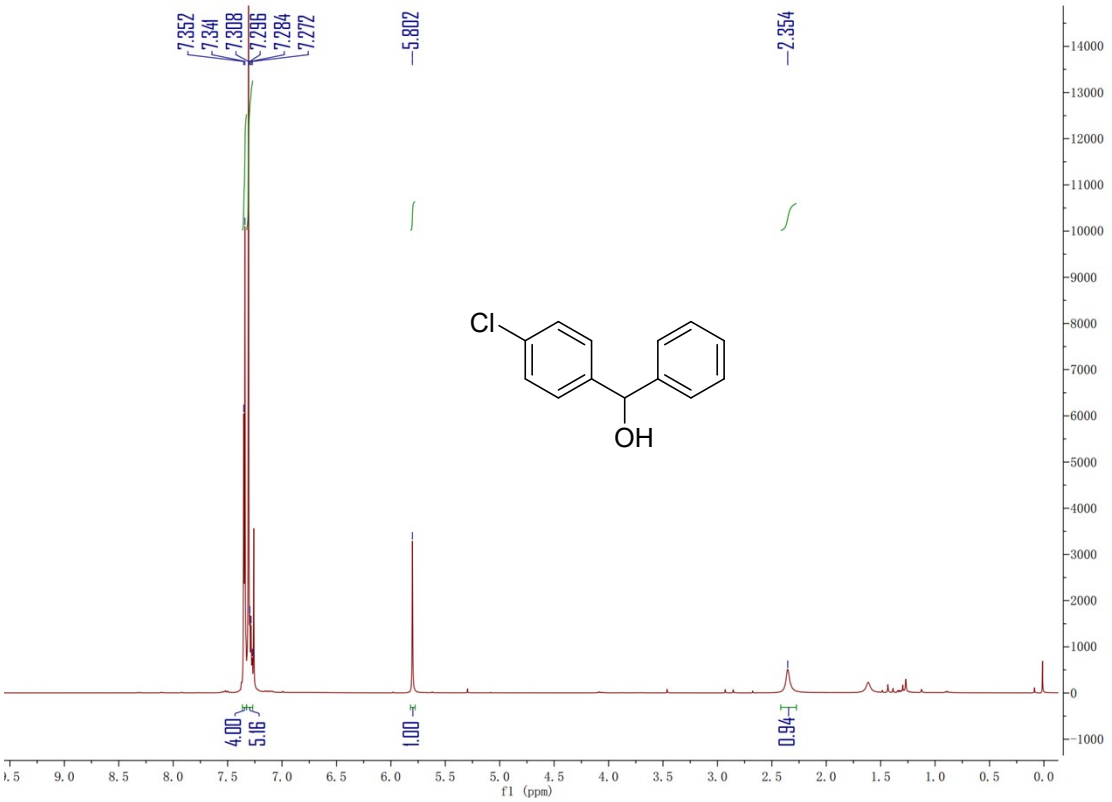


(4-Chlorophenyl)(phenyl)methanol (2a)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



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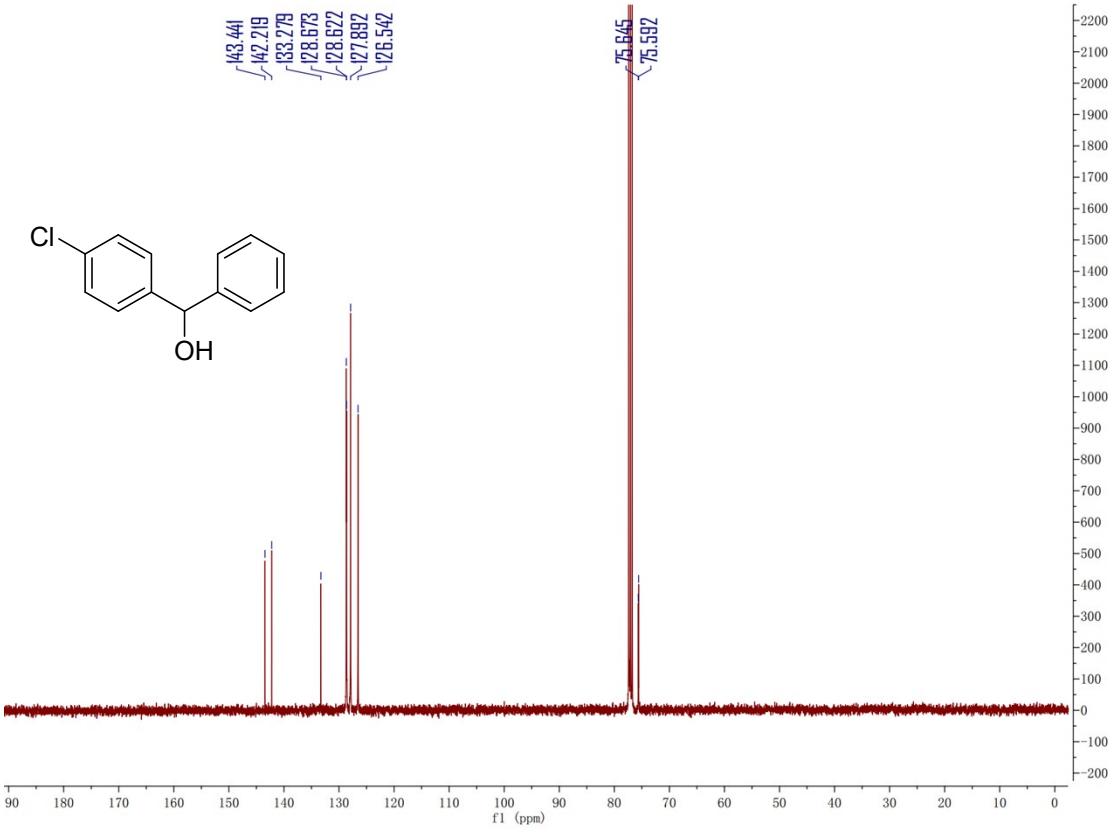
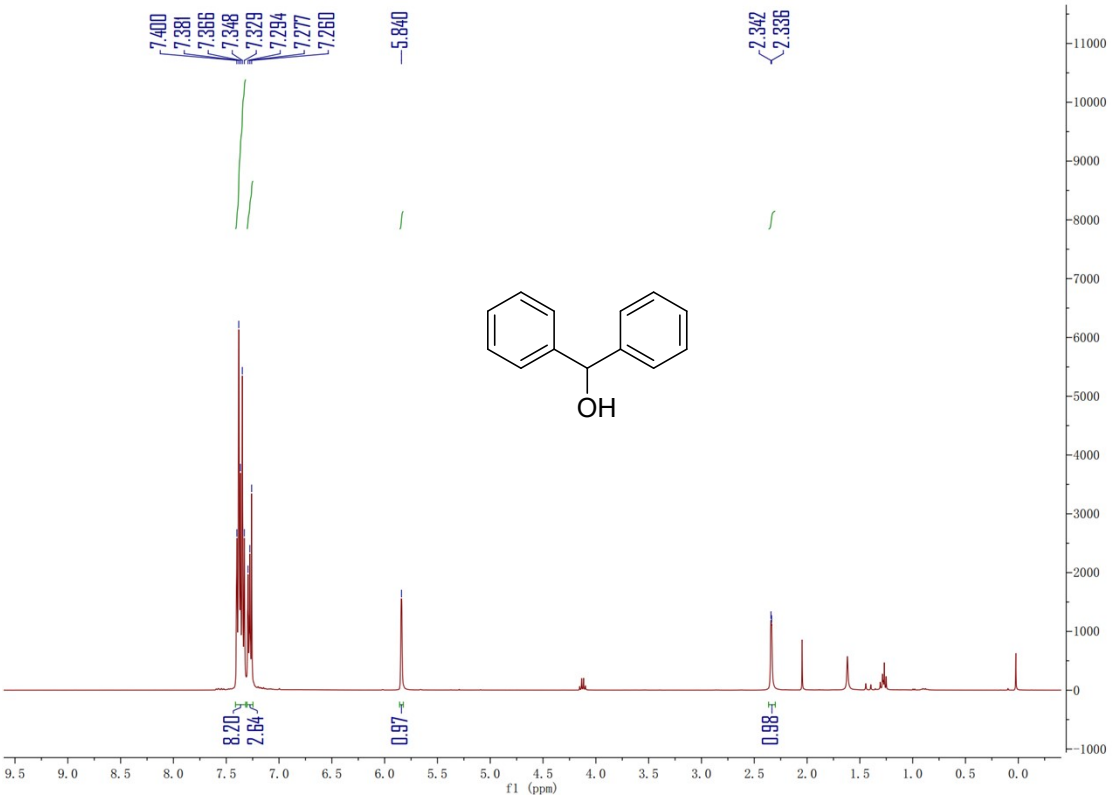


Diphenylmethanol (2b)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



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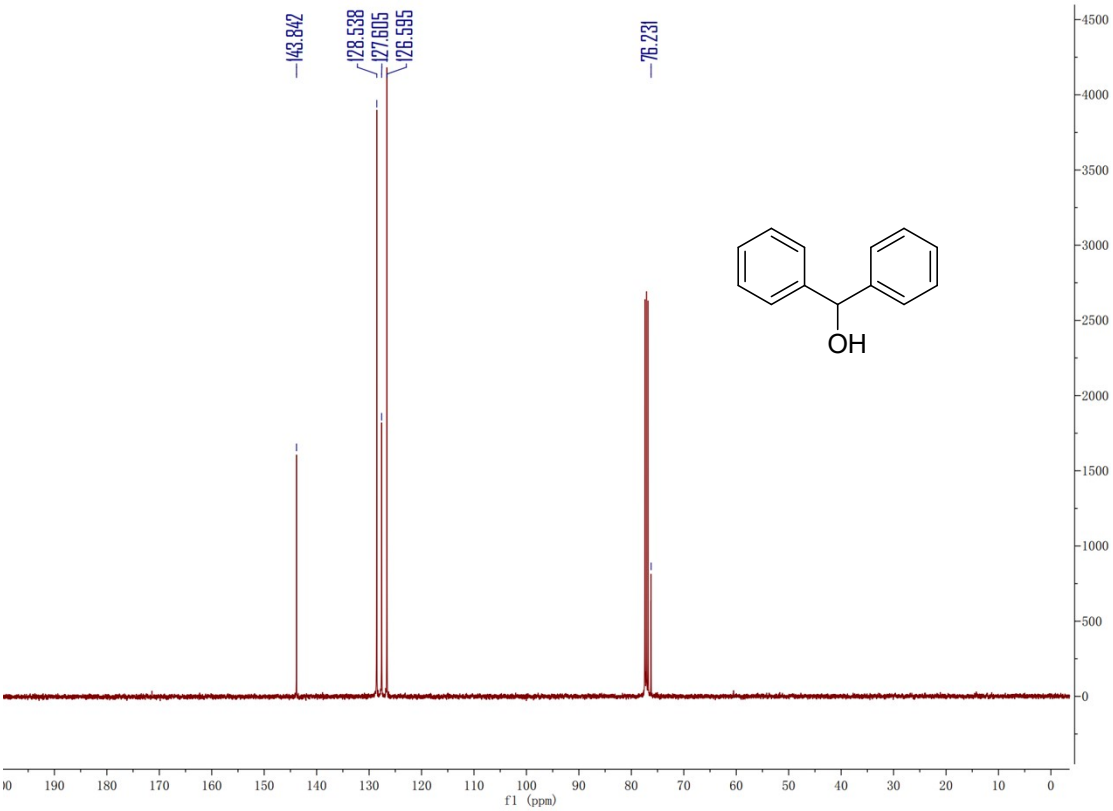
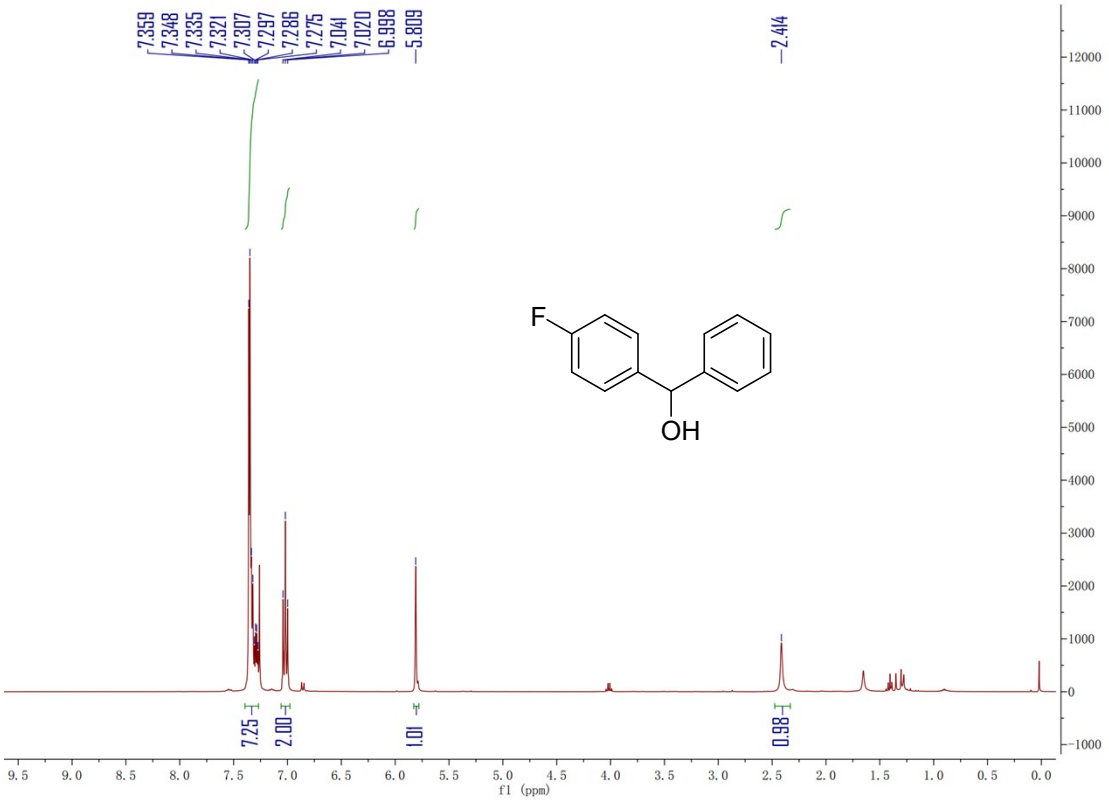


(4-Fluorophenyl)(phenyl)methanol (2c)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



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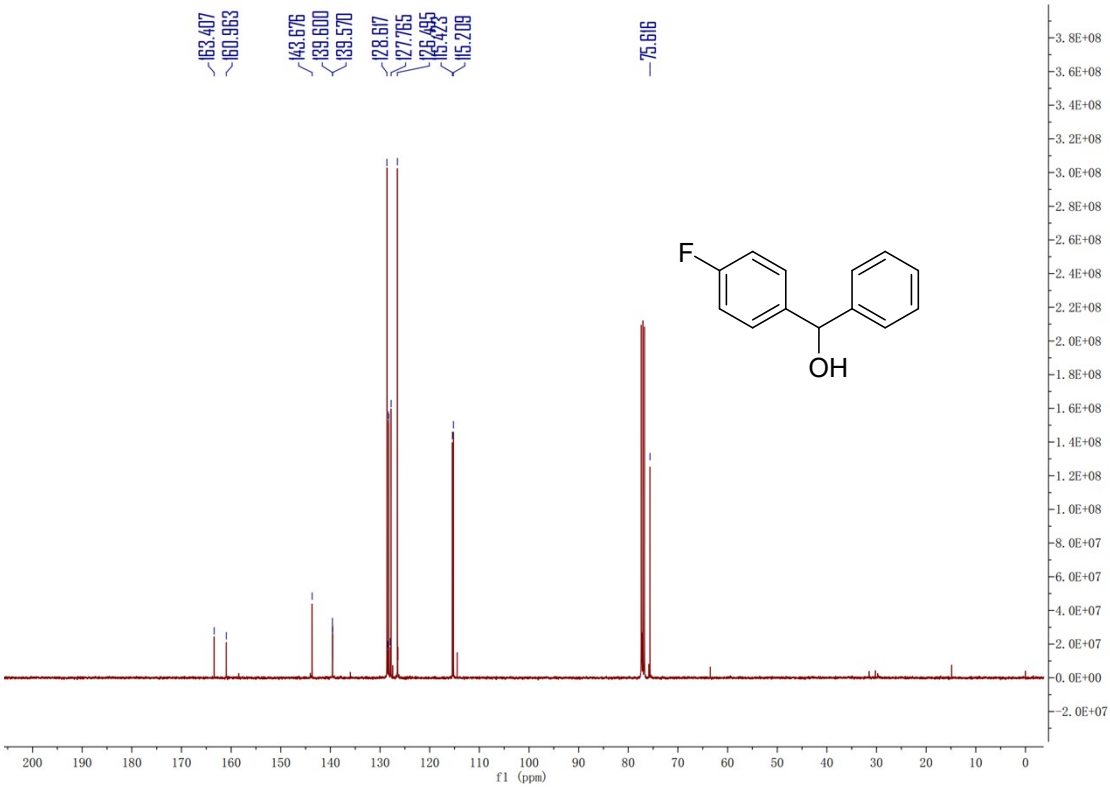
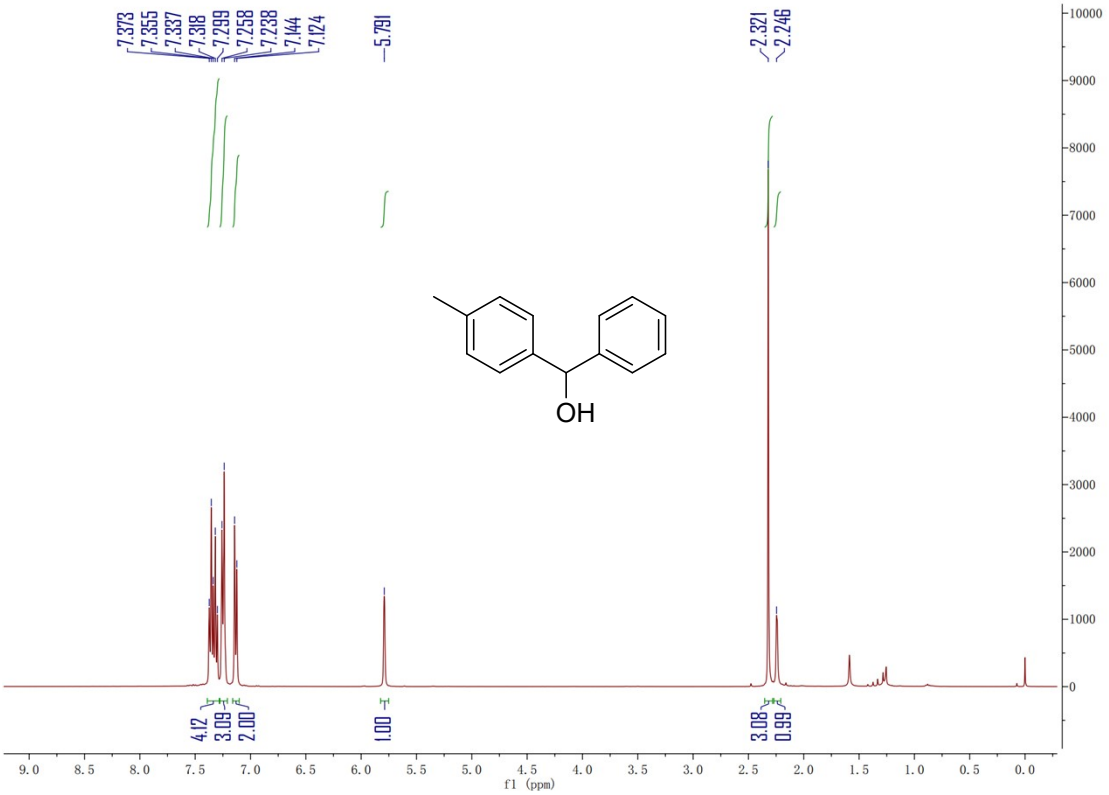


Phenyl(p-tolyl)methanol (2d)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



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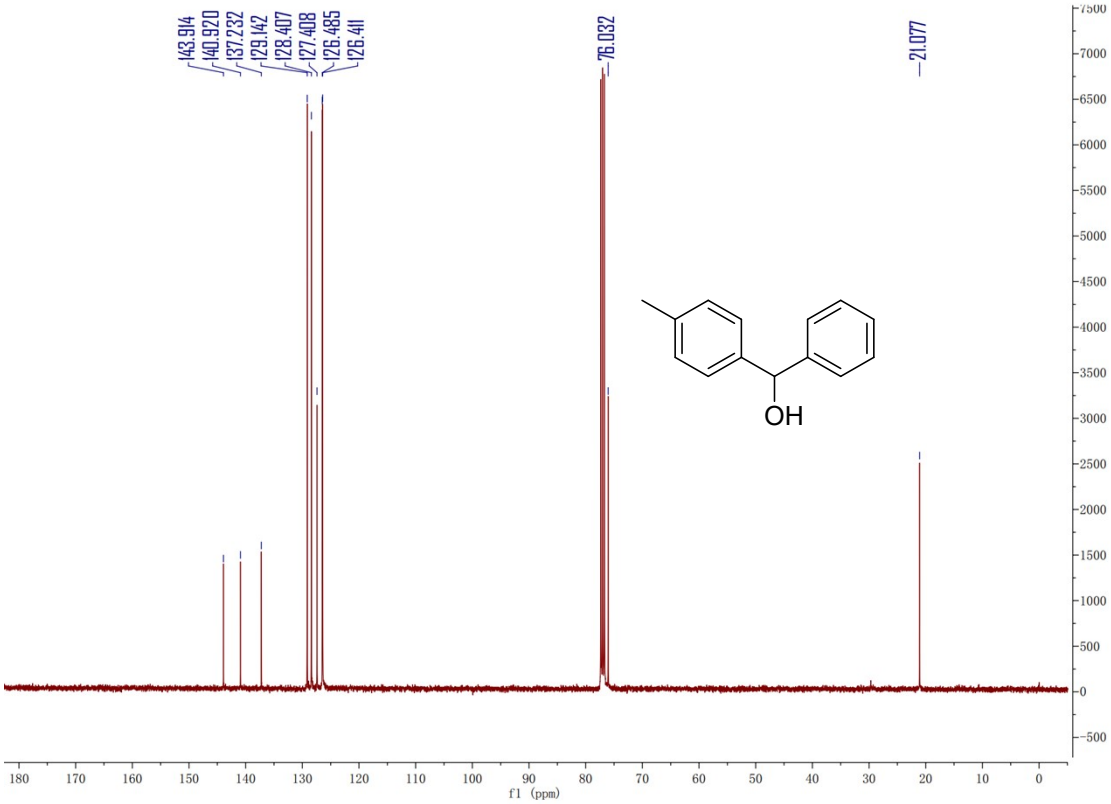
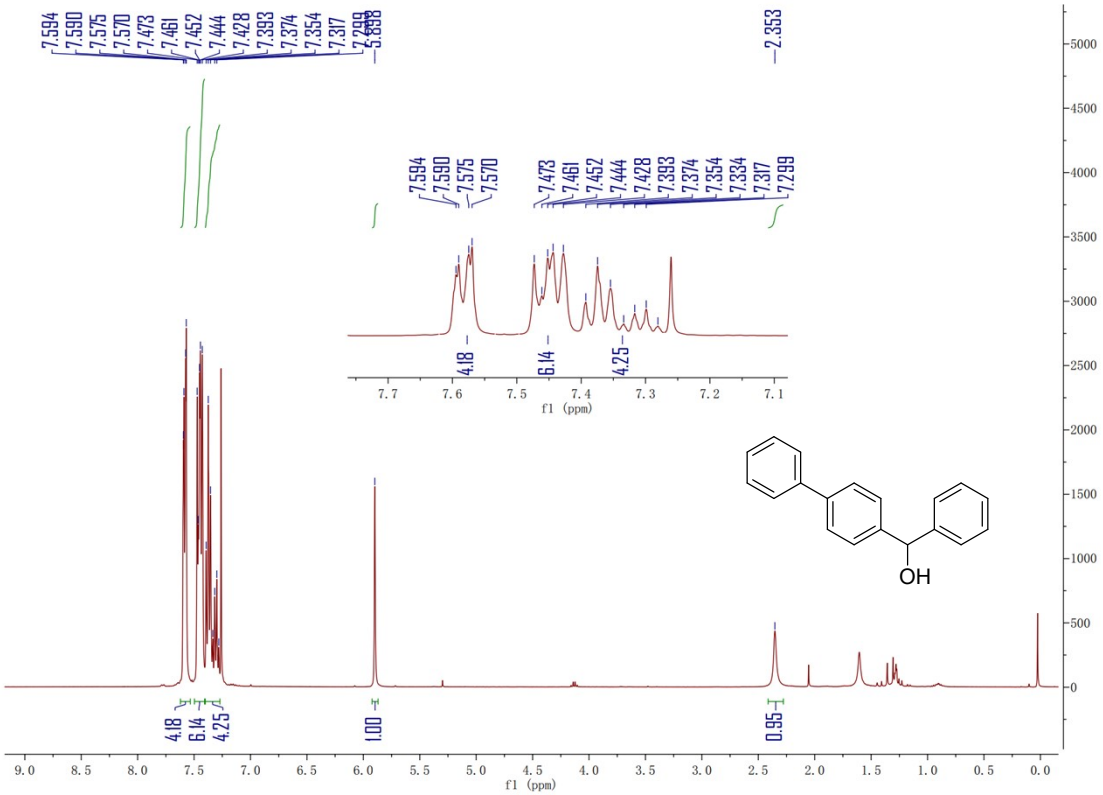
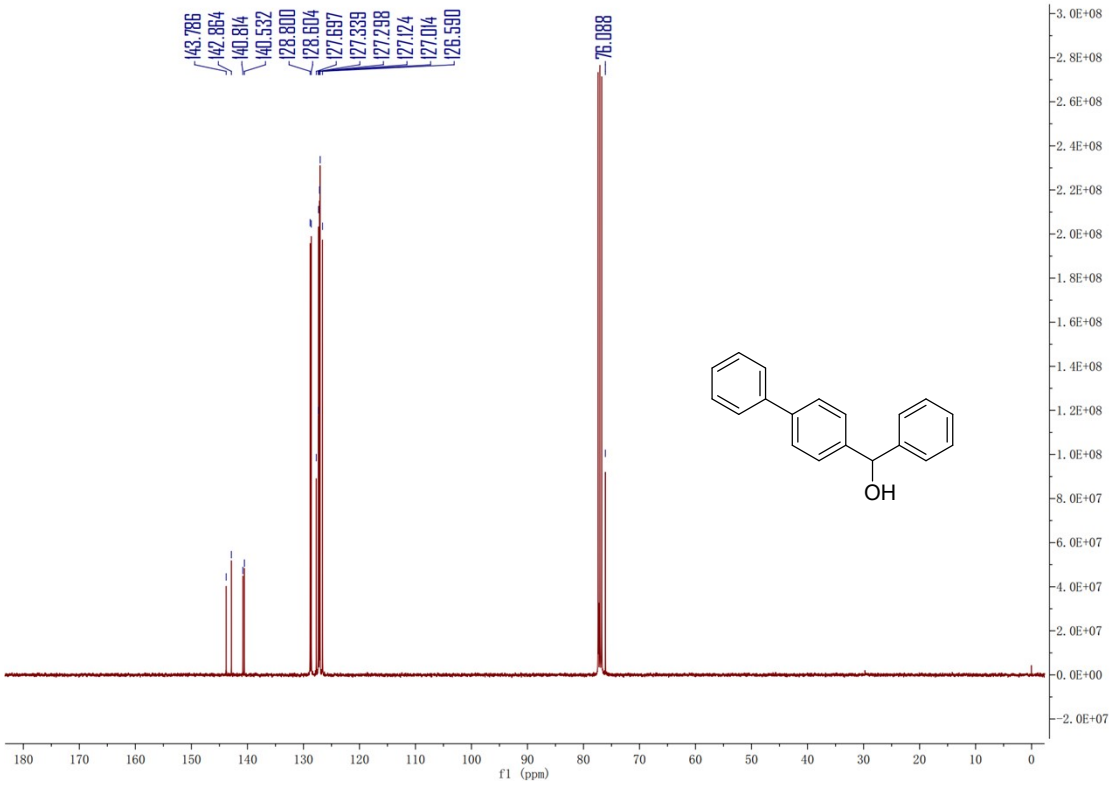


[1,1'-biphenyl]-4-yl(phenyl)methanol (2e)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

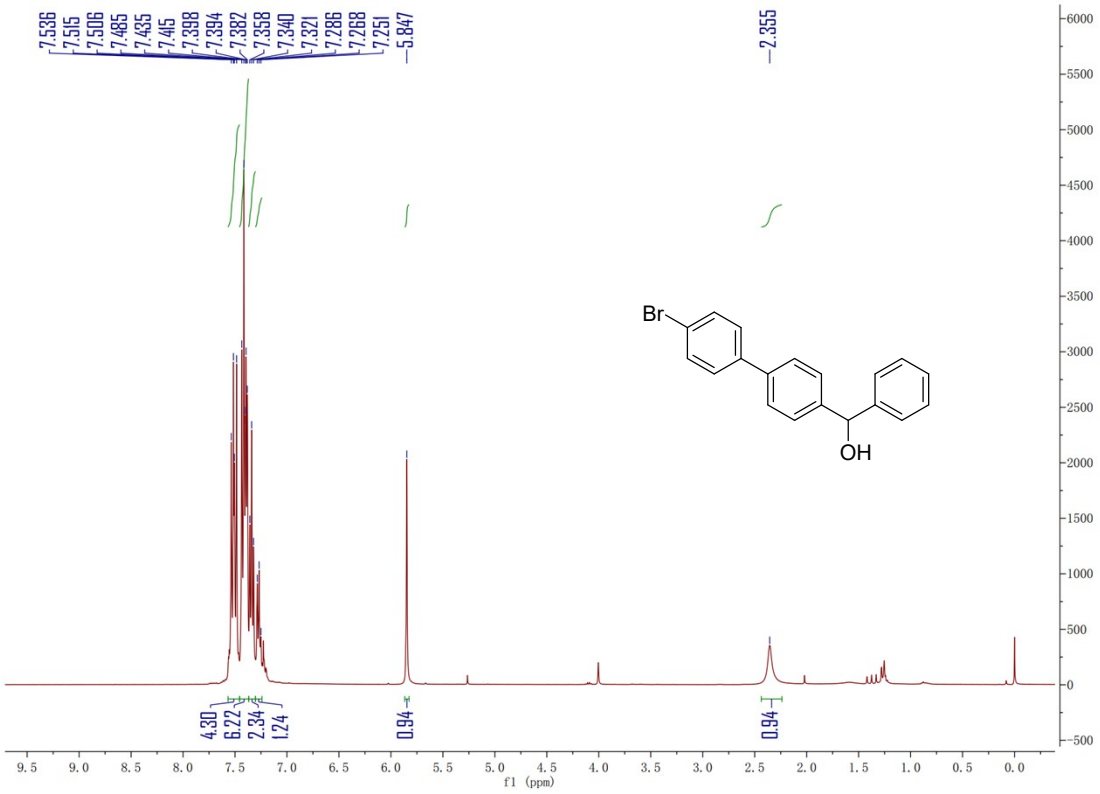


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

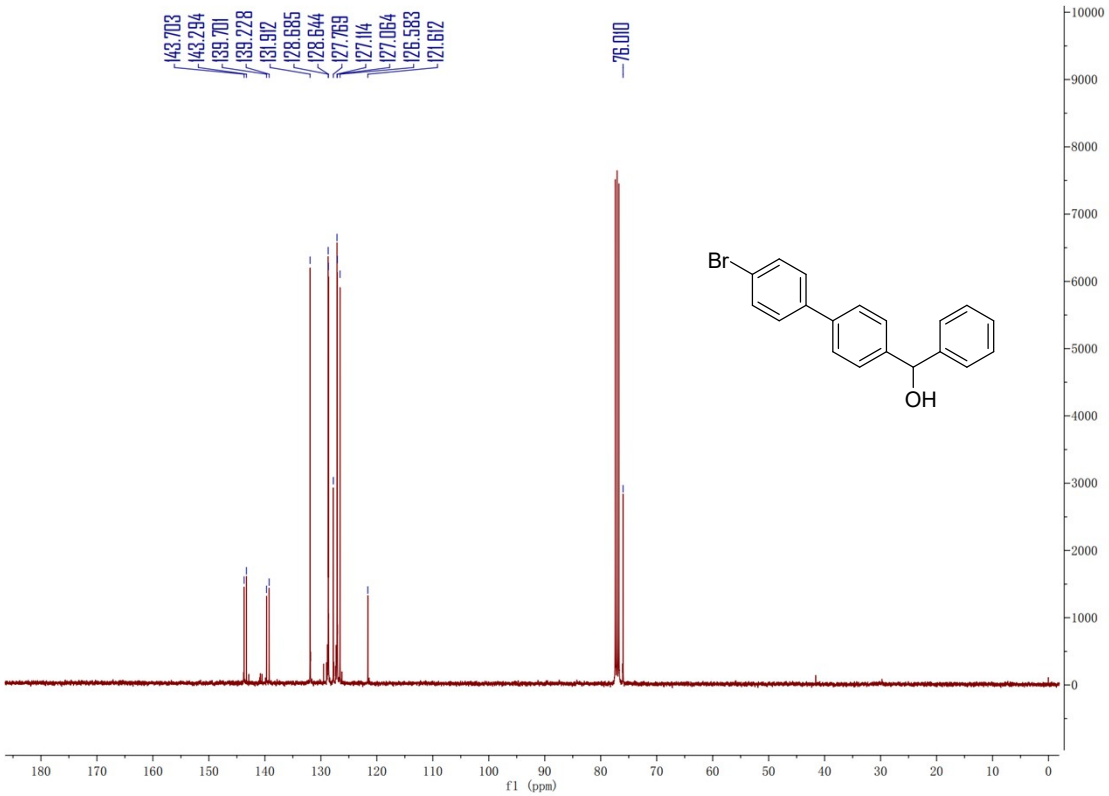


(4'-Bromo-[1,1'-biphenyl]-4-yl)(phenyl)methanol (2f)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

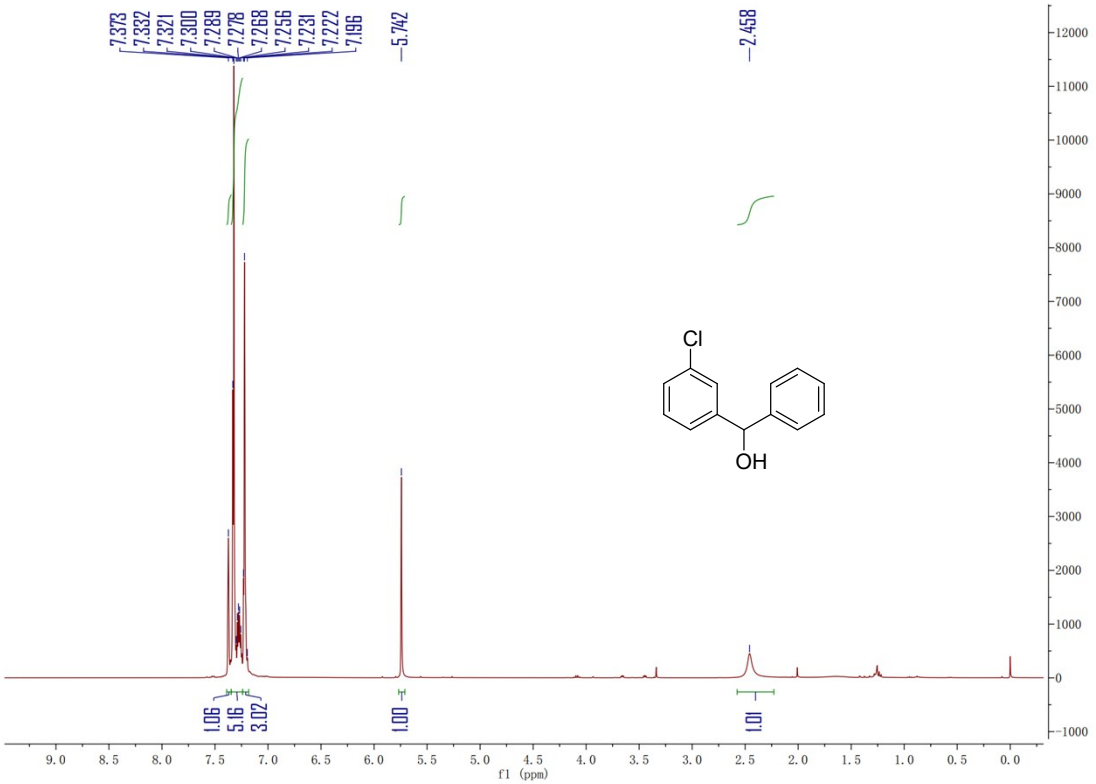


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

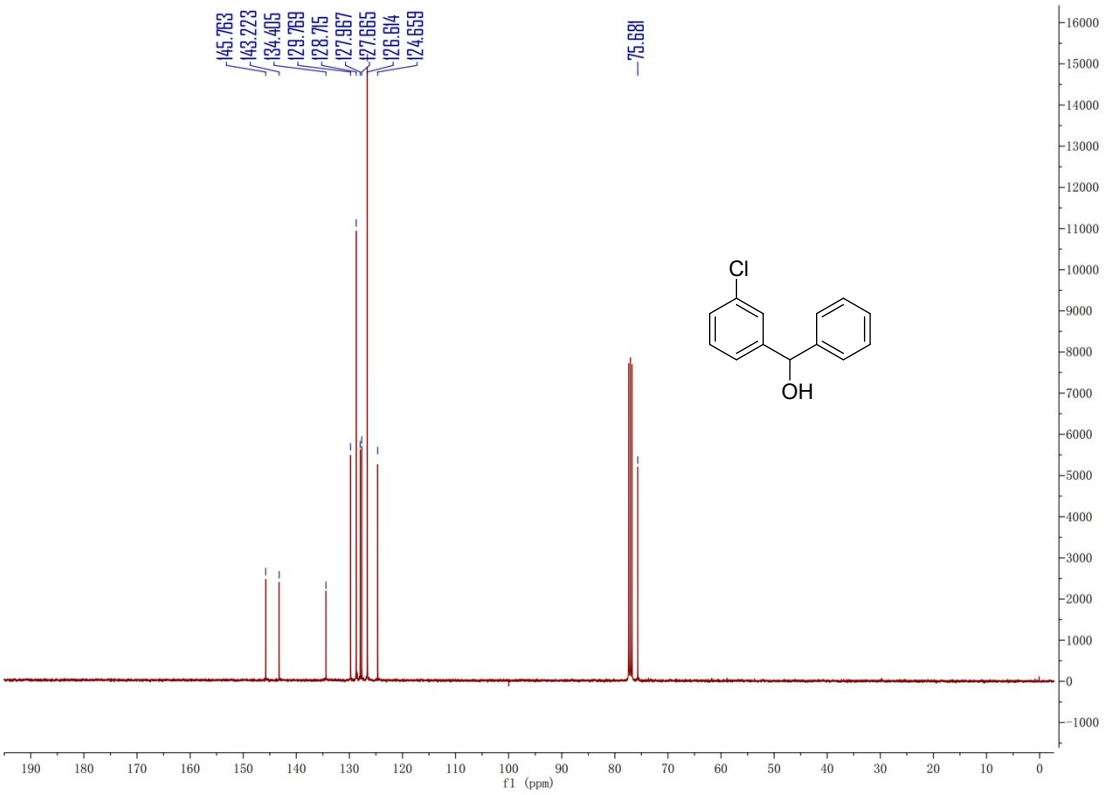


(3-Chlorophenyl)(phenyl)methanol (2g)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

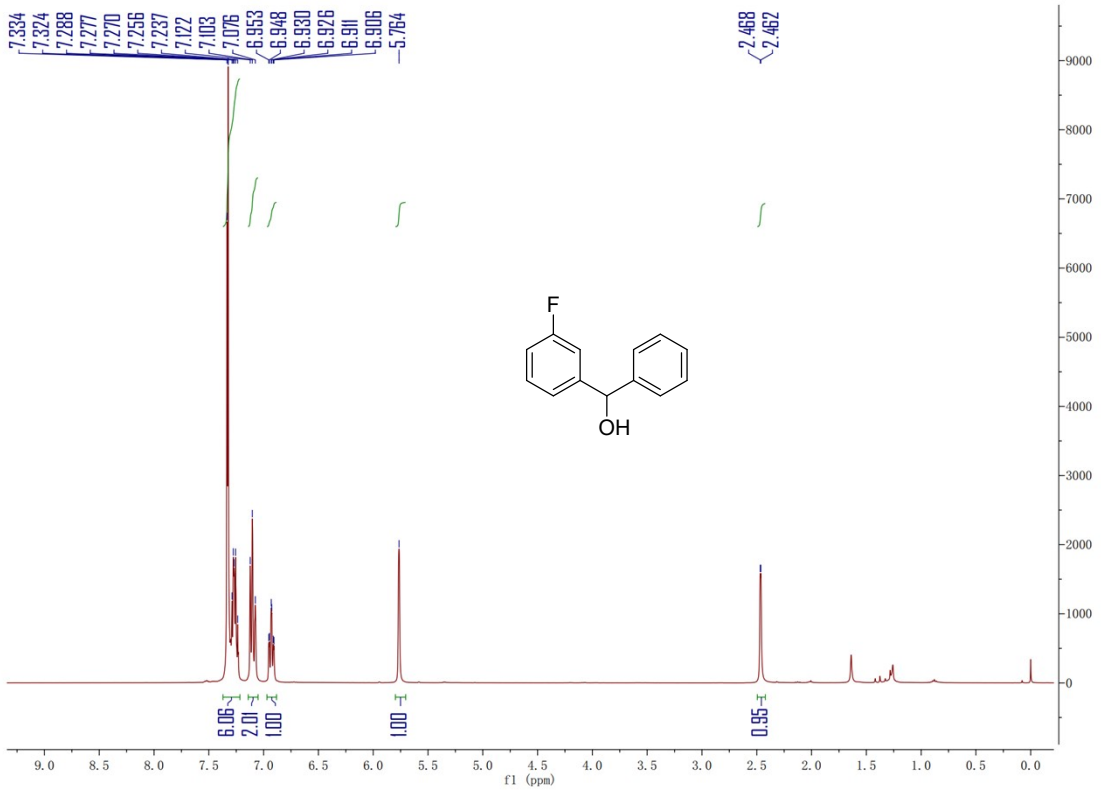


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

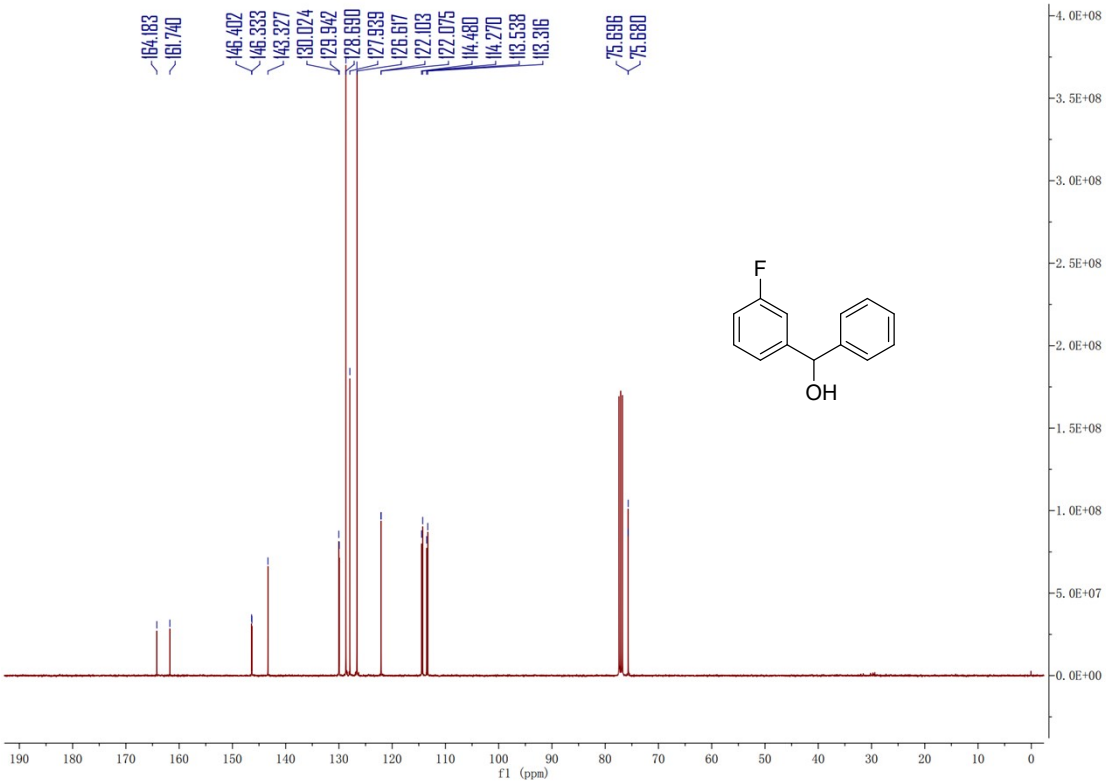


(3-Fluorophenyl)(phenyl)methanol (2h)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

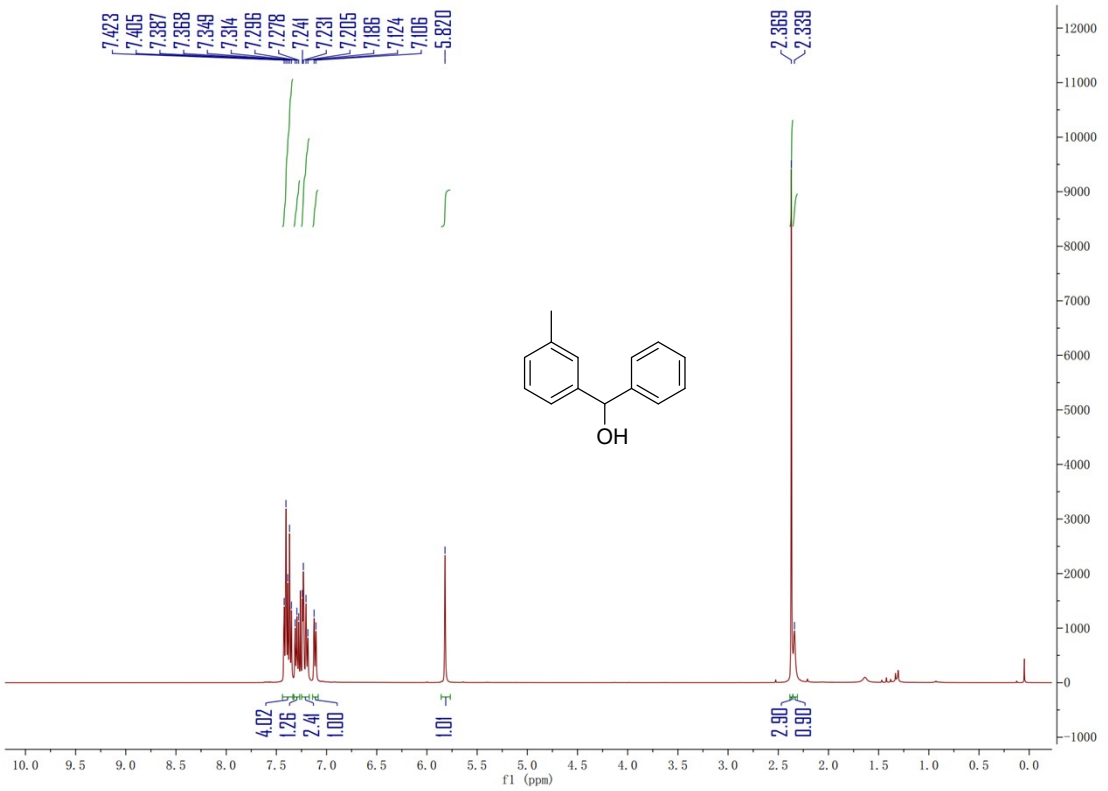


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

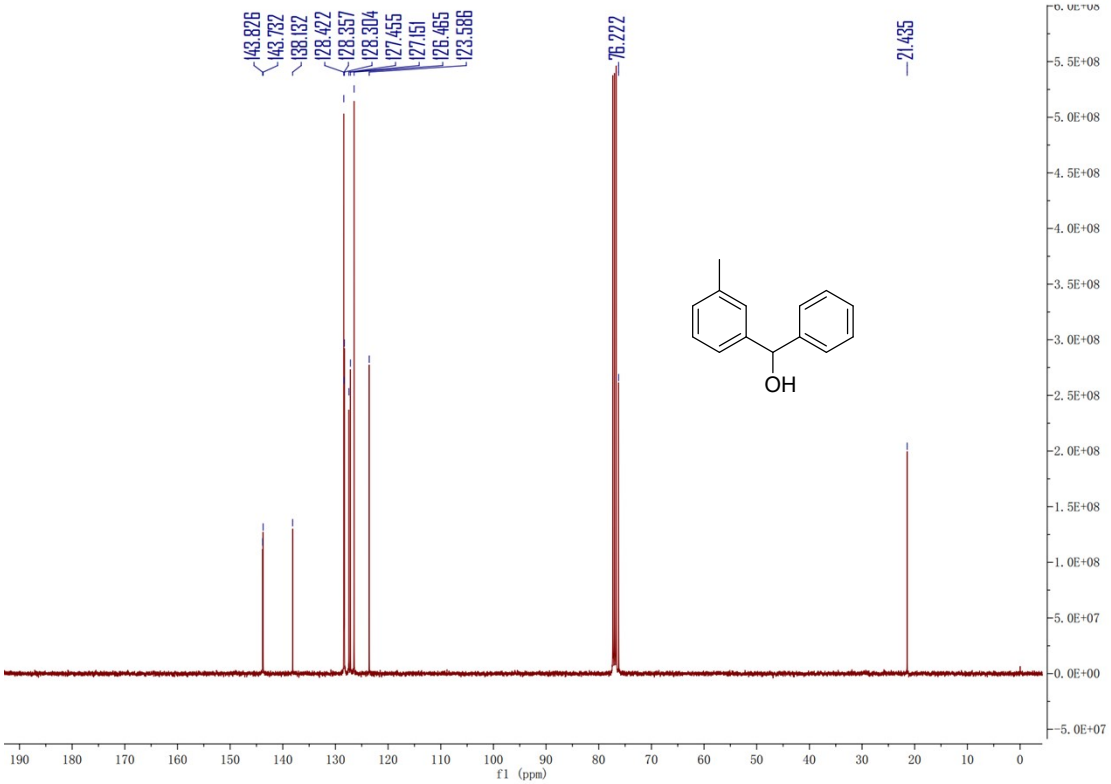


Phenyl(m-tolyl)methanol (2i)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

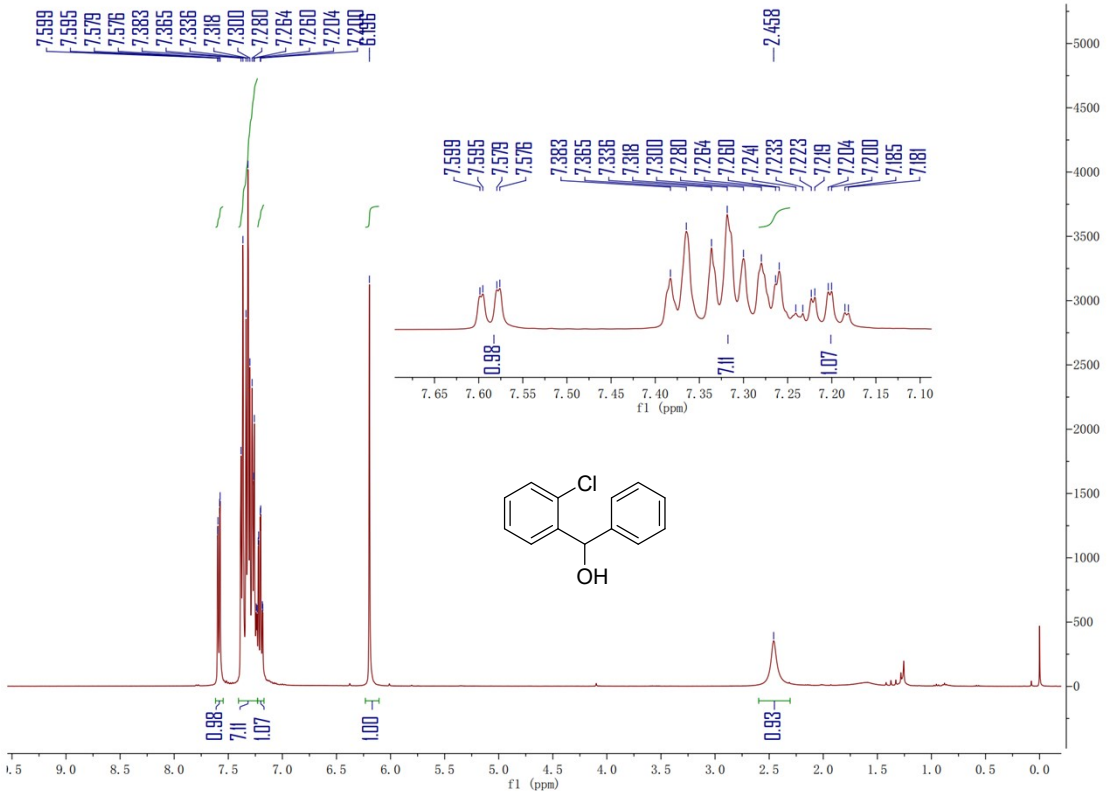


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

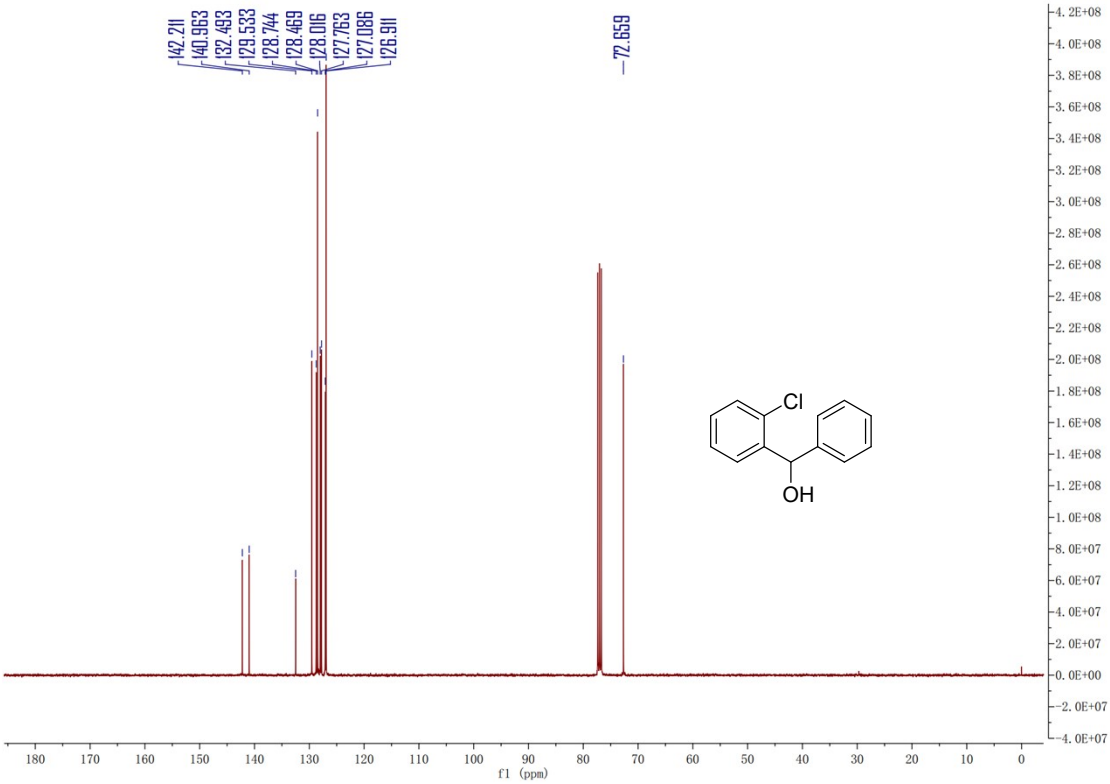


(2-Chlorophenyl)(phenyl)methanol (2j)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

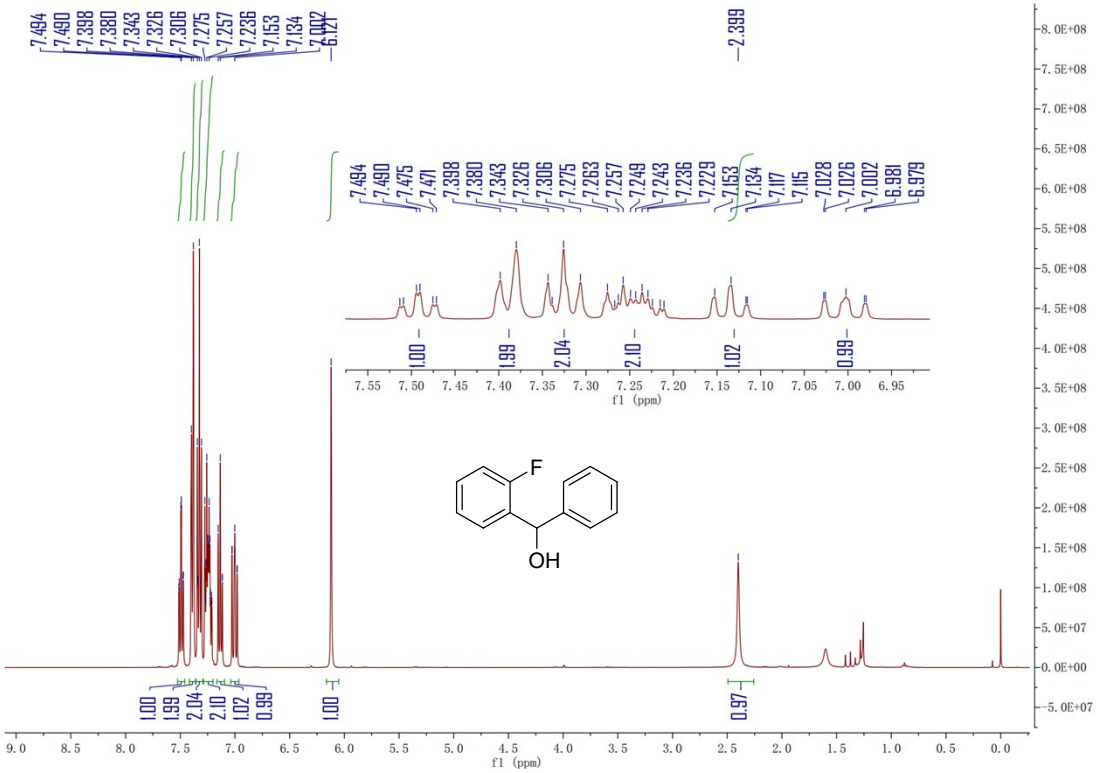


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

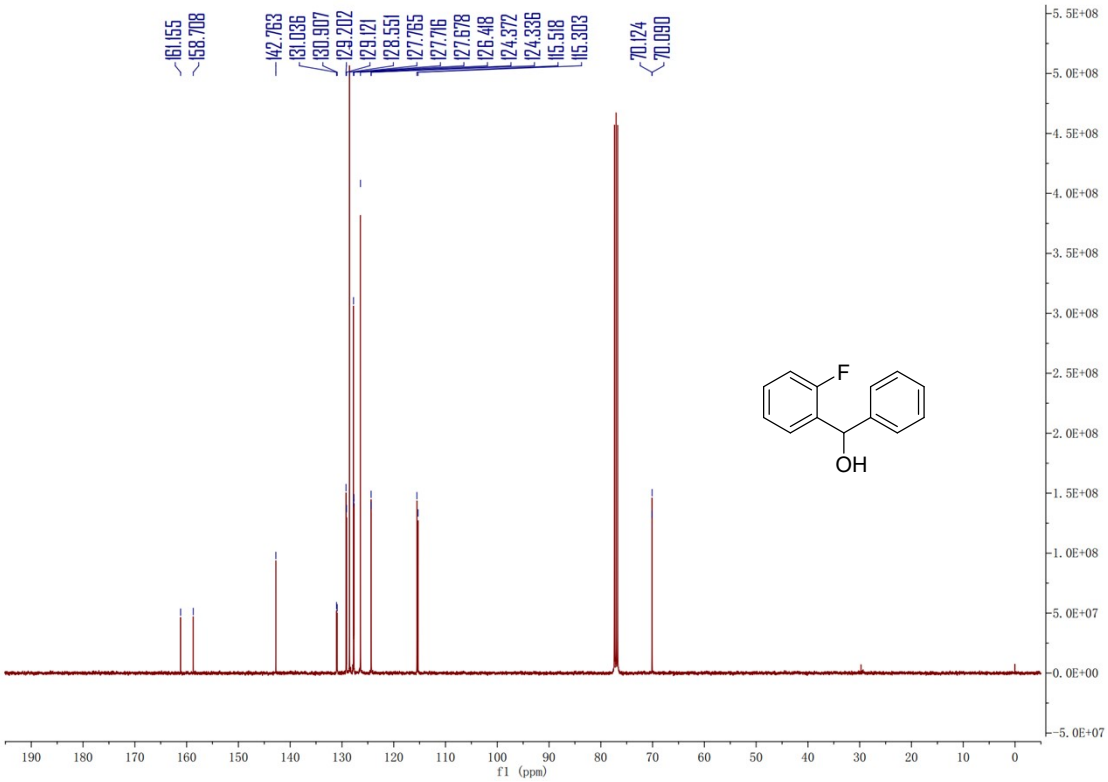


(2-Fluorophenyl)(phenyl)methanol (2k)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

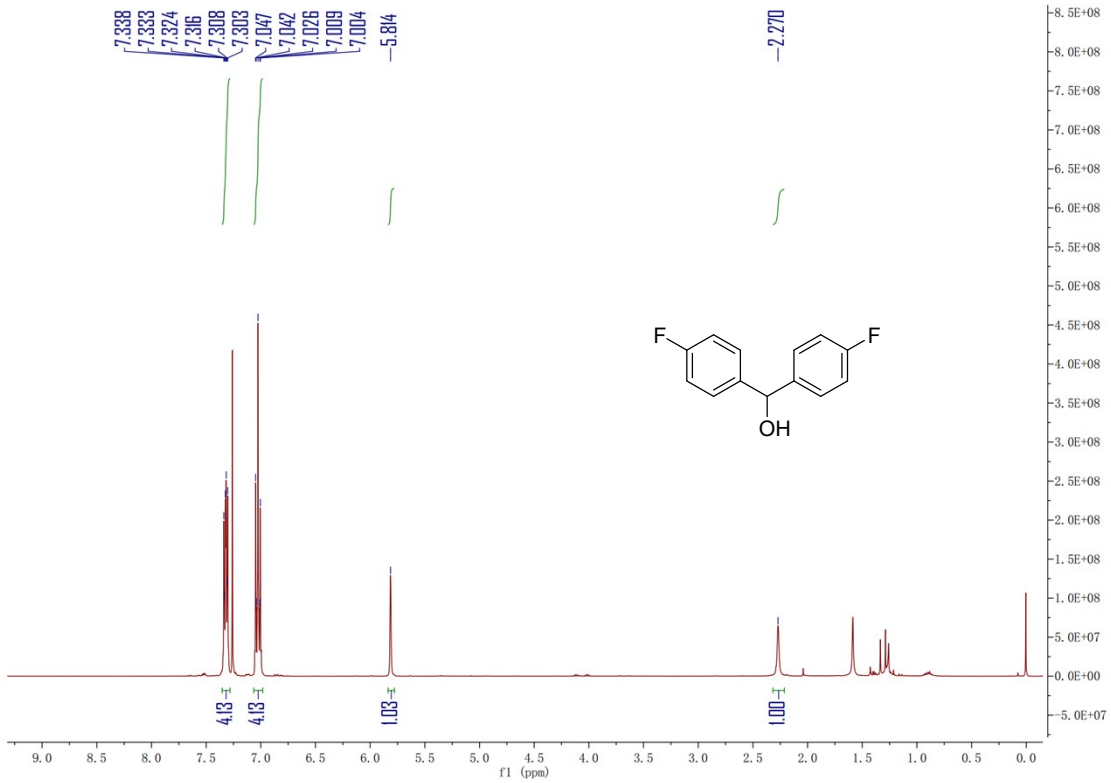


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

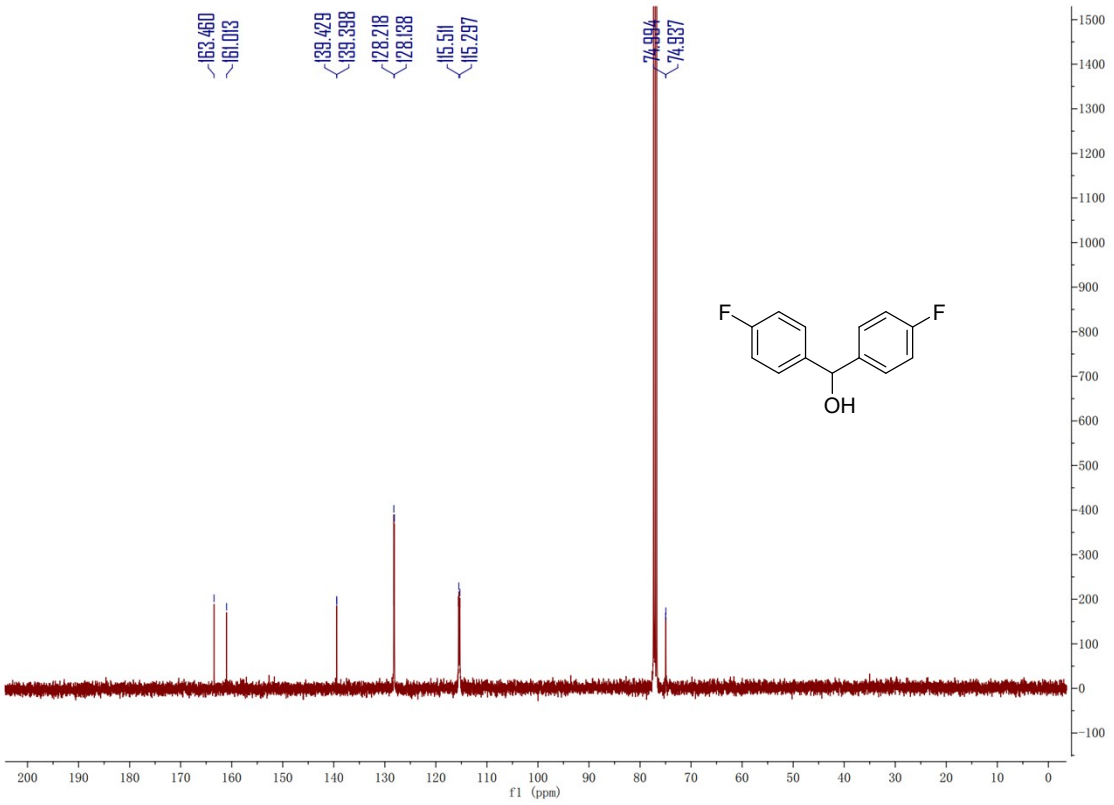


Bis(4-fluorophenyl)methanol (2l)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

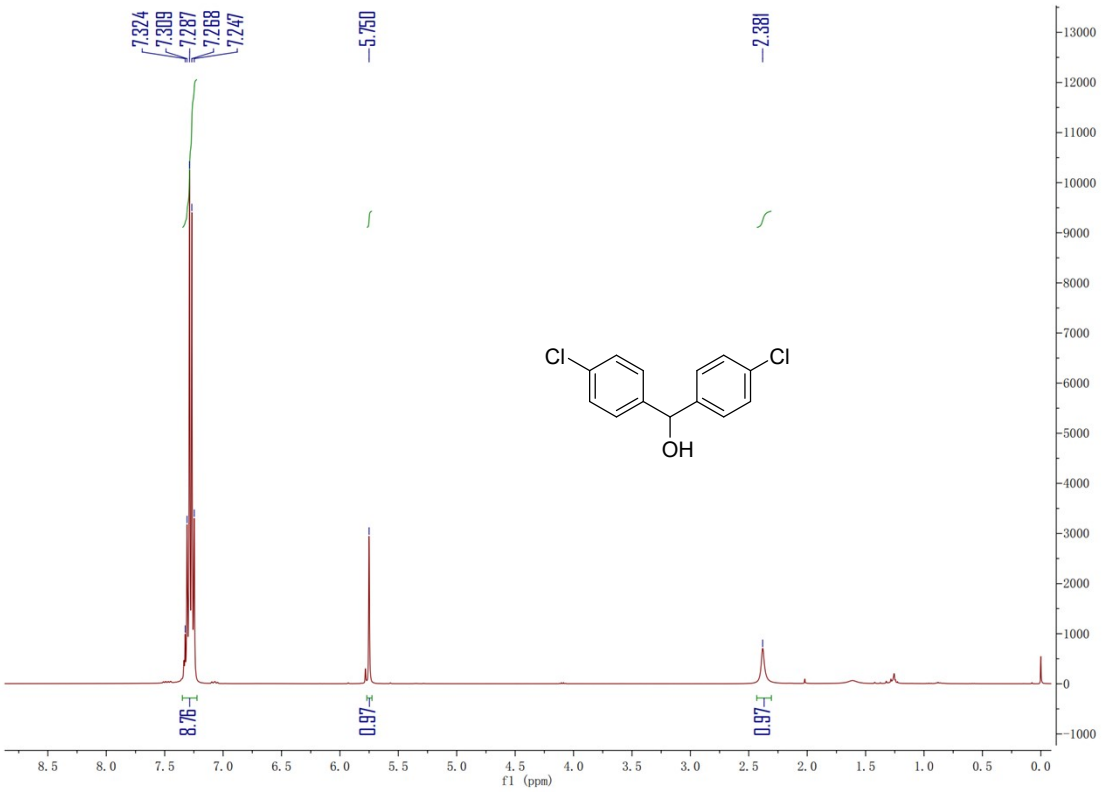


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

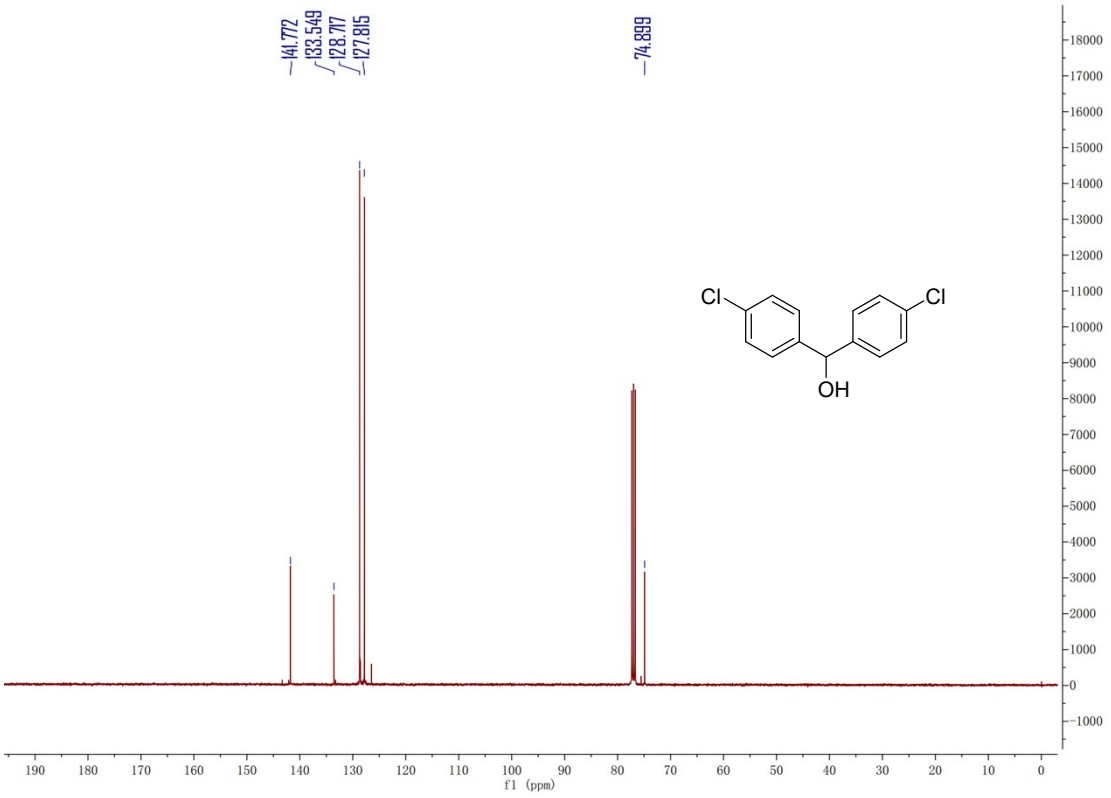


Bis(4-chlorophenyl)methanol (2m)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

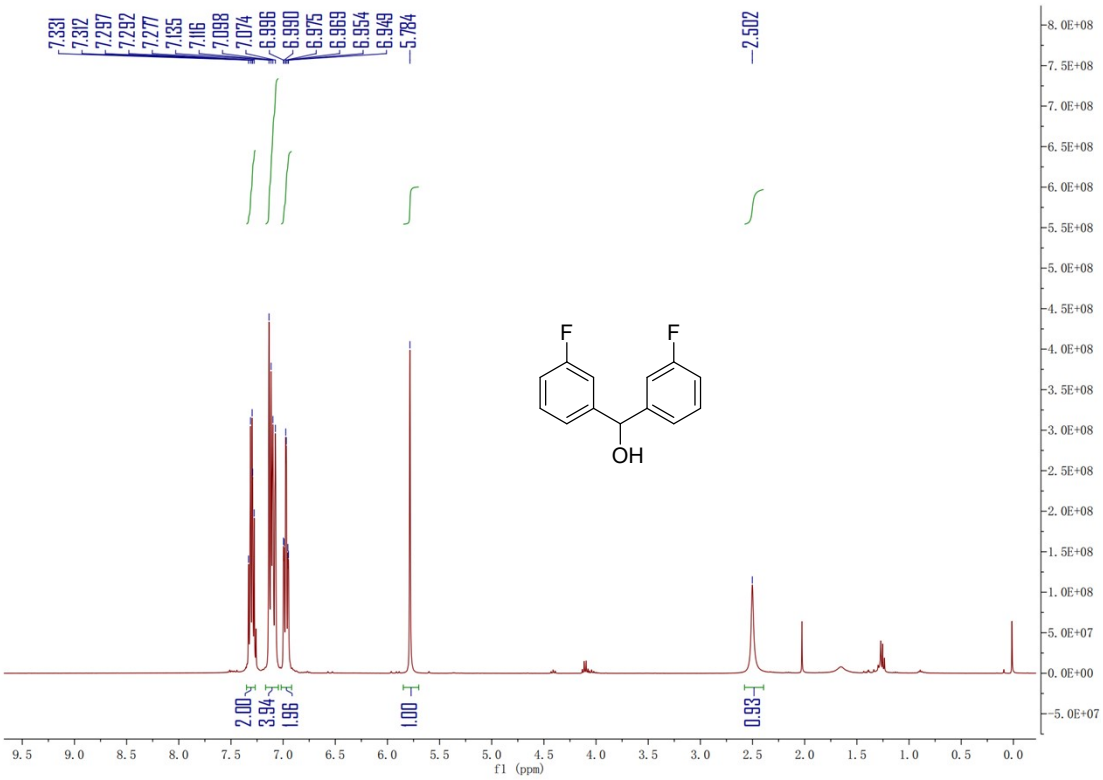


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

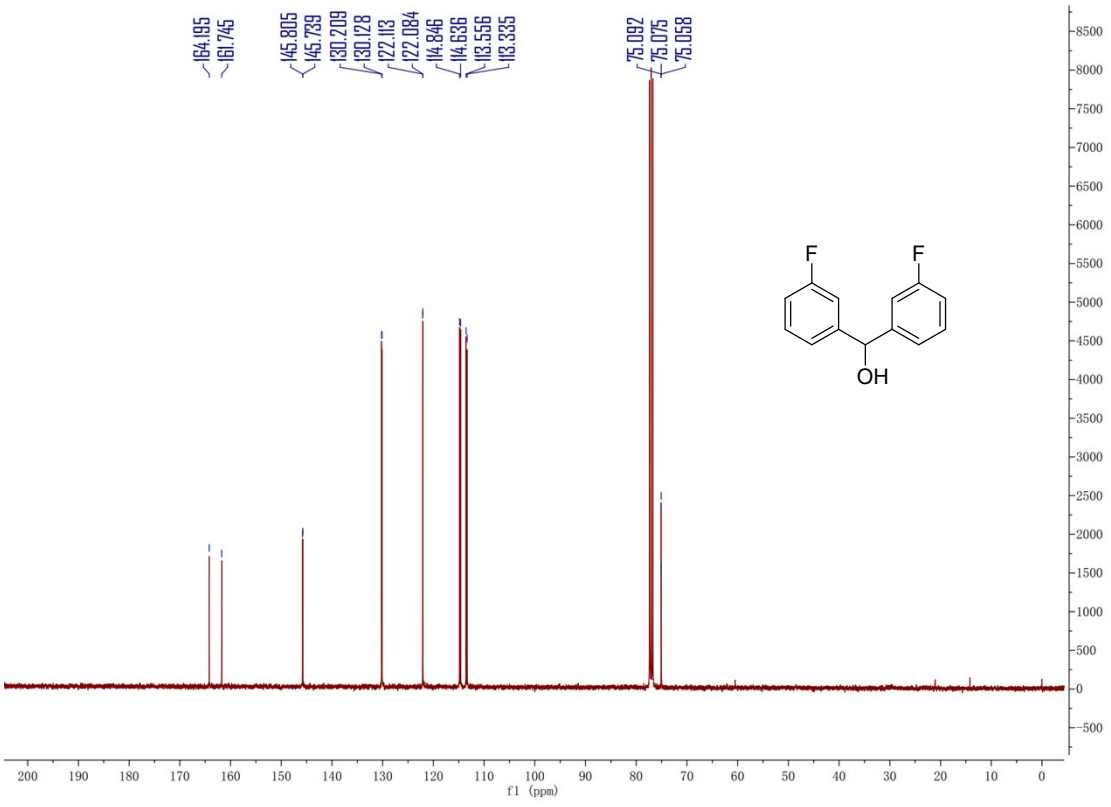


Bis(3-fluorophenyl)methanol (2n)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

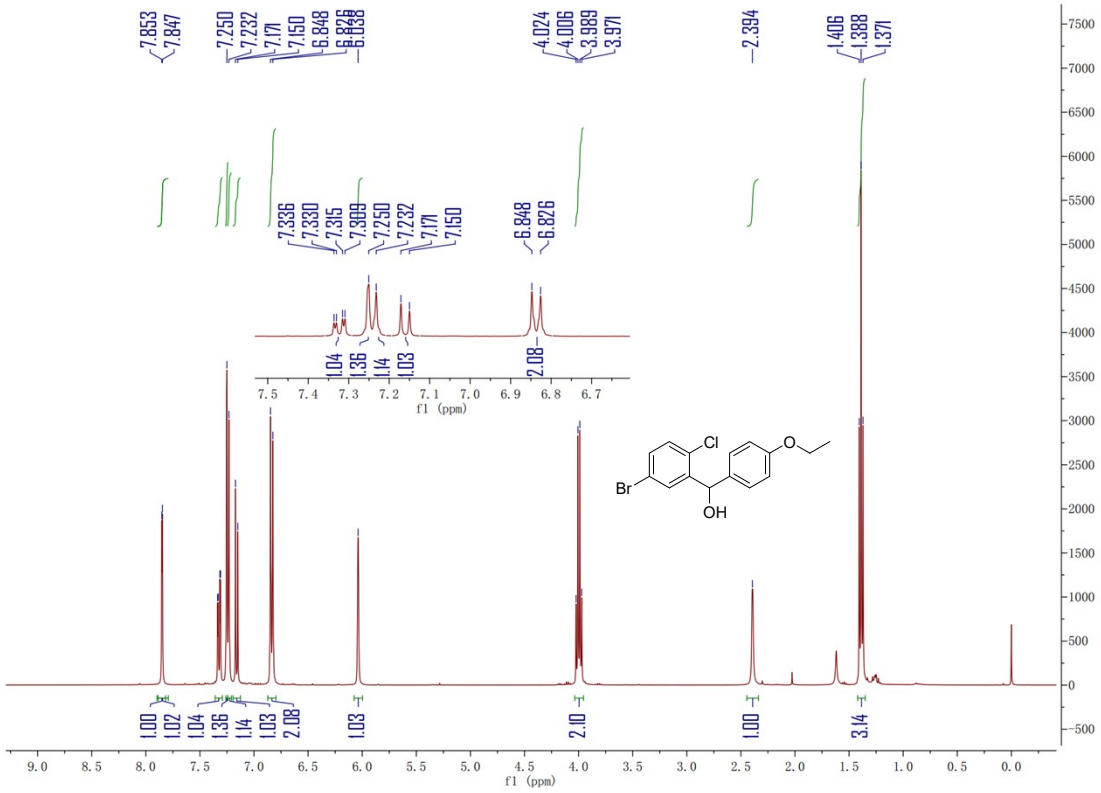


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

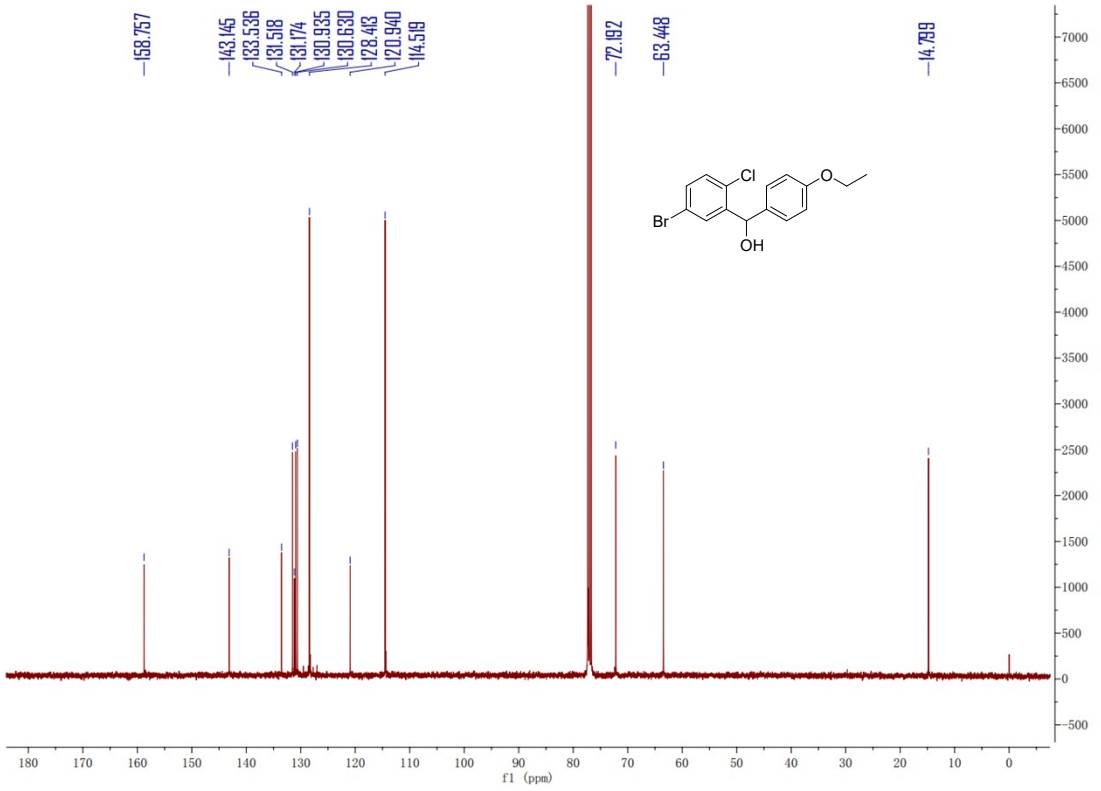


(5-Bromo-2-chlorophenyl)(4-ethoxyphenyl)methanol (2o)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

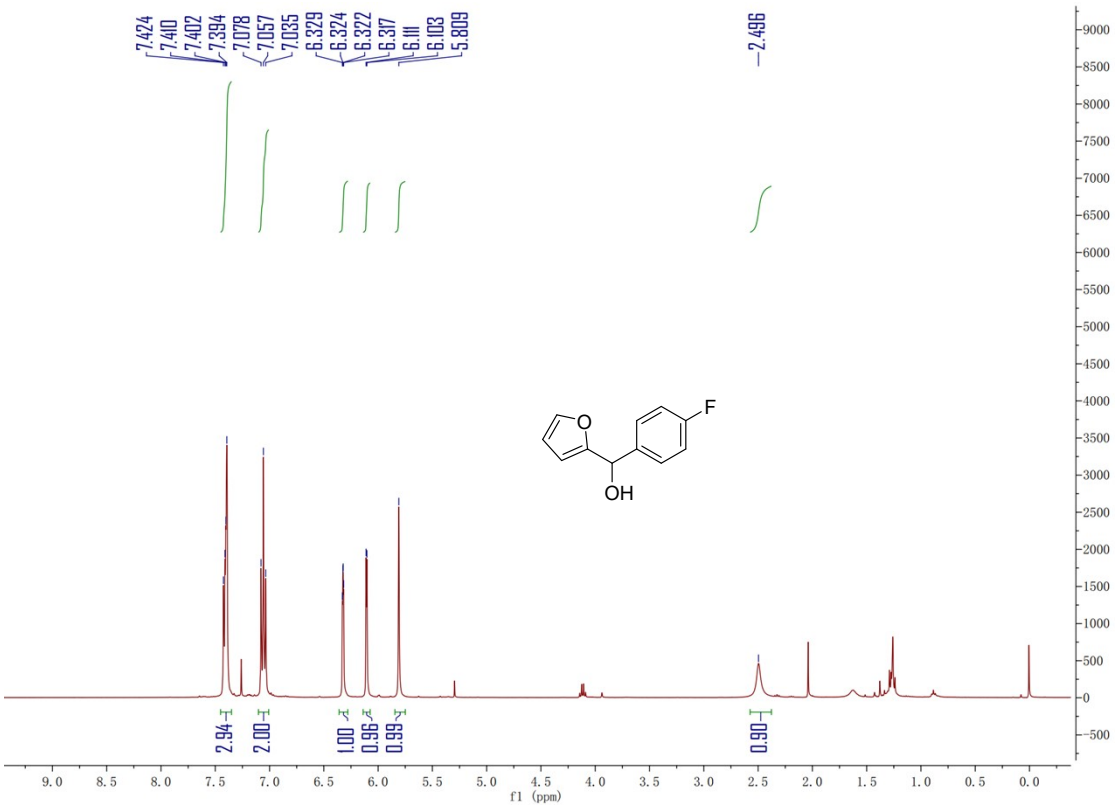


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

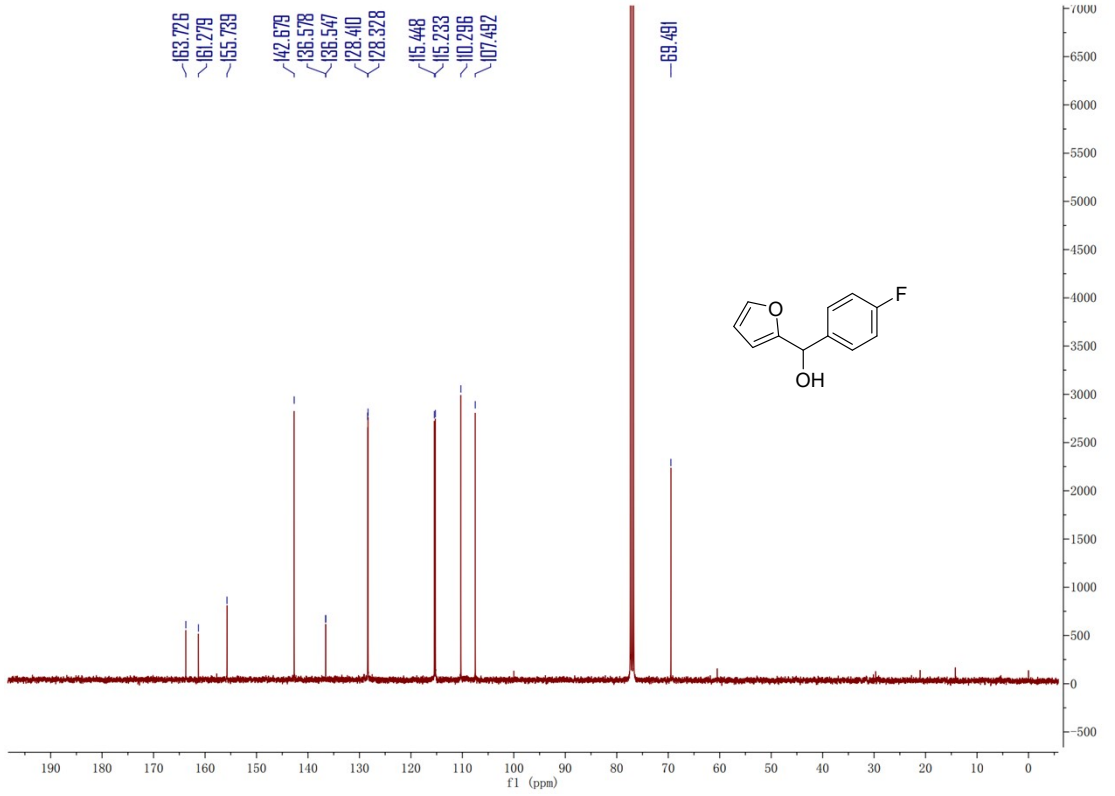


(4-Fluorophenyl)(furan-2-yl)methanol (2p)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

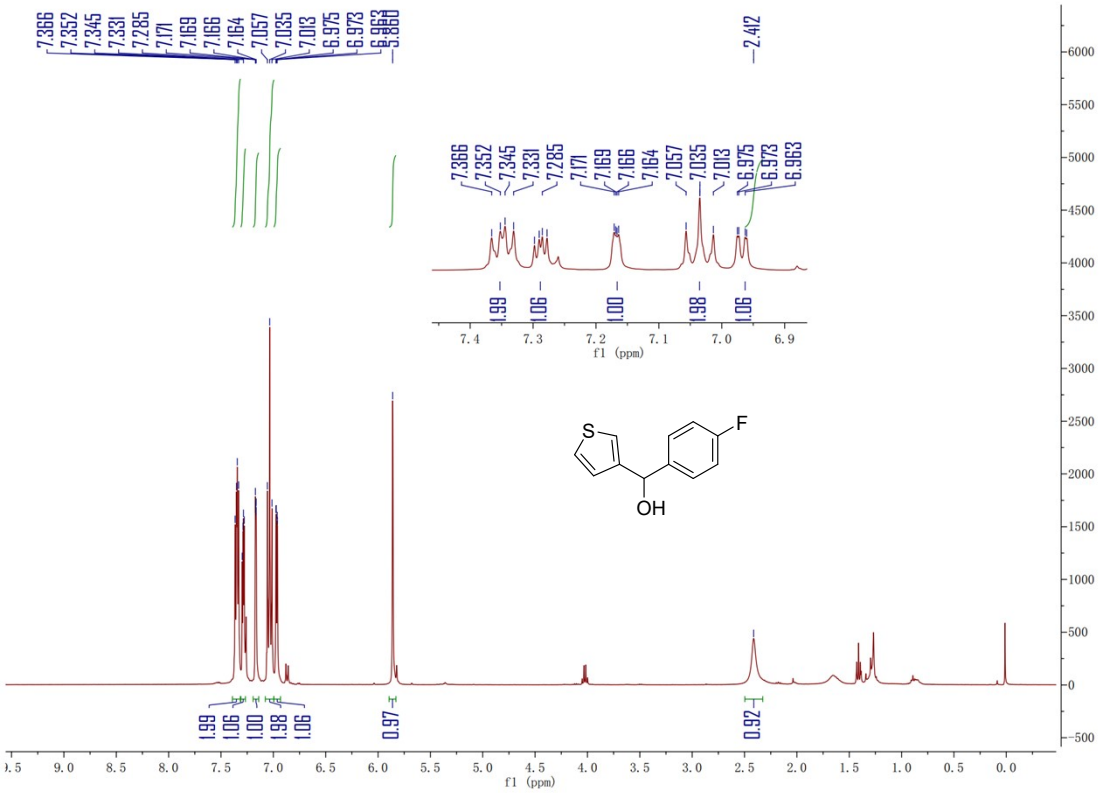


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

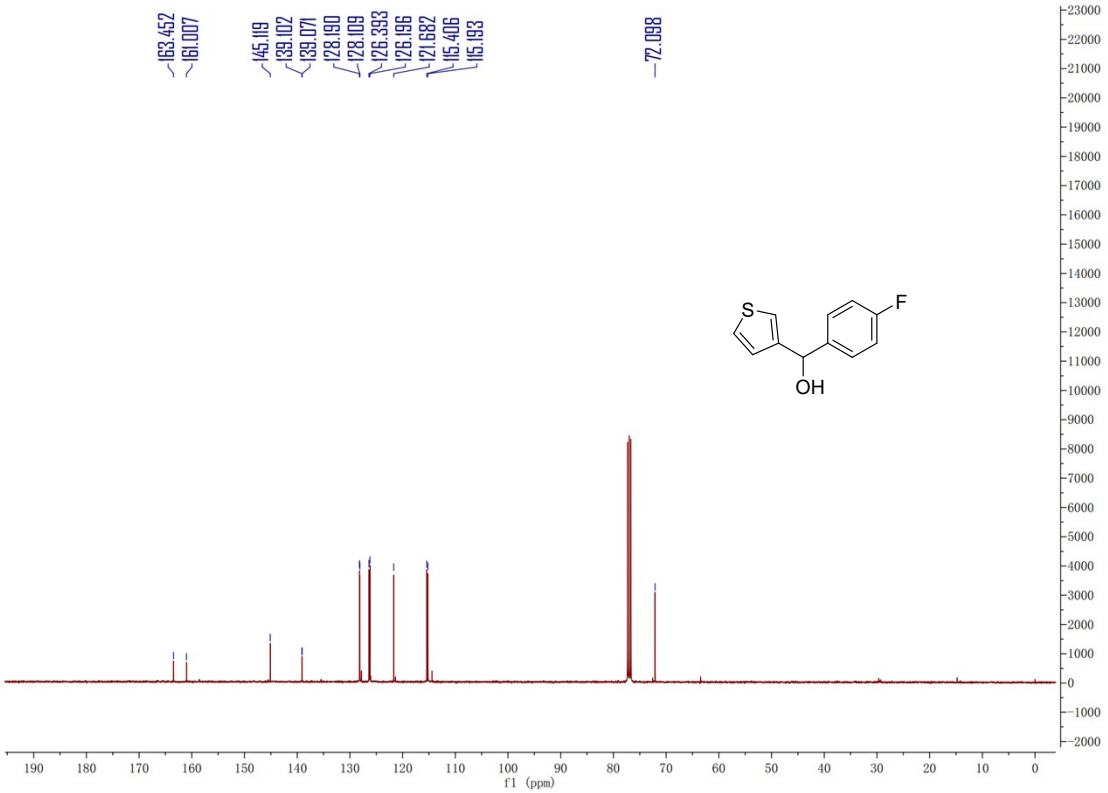


(4-Fluorophenyl)(thiophen-3-yl)methanol (2q)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

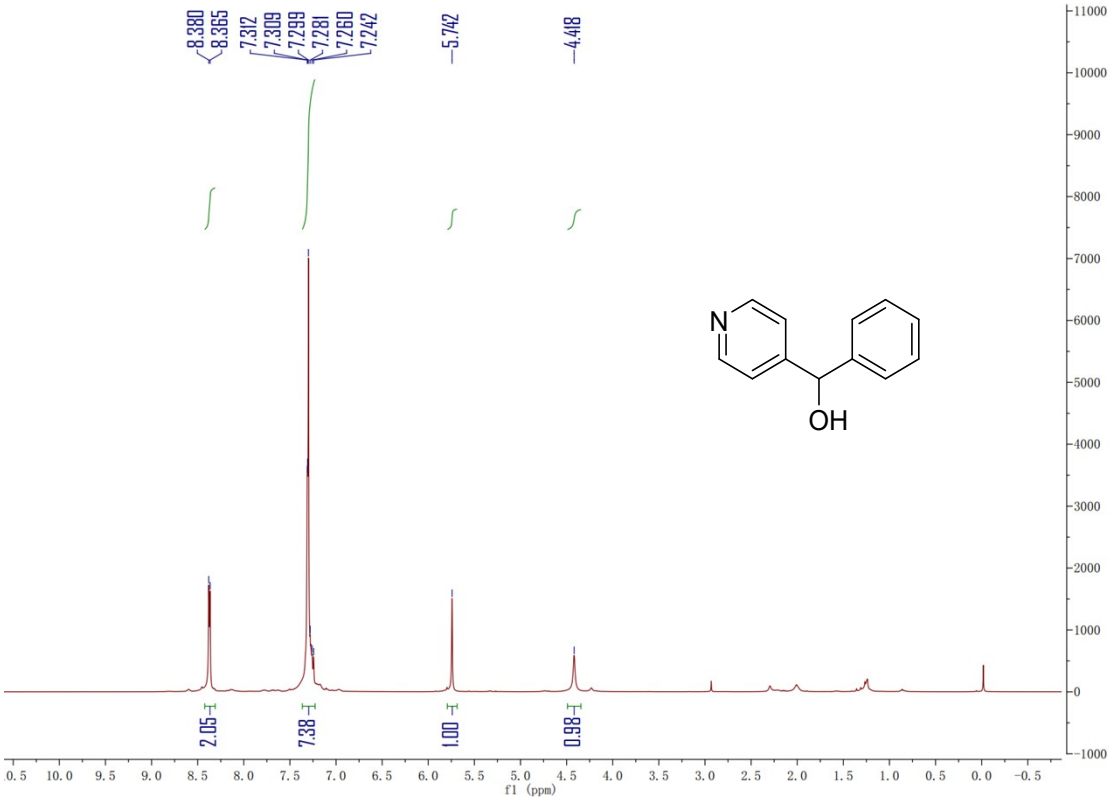


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

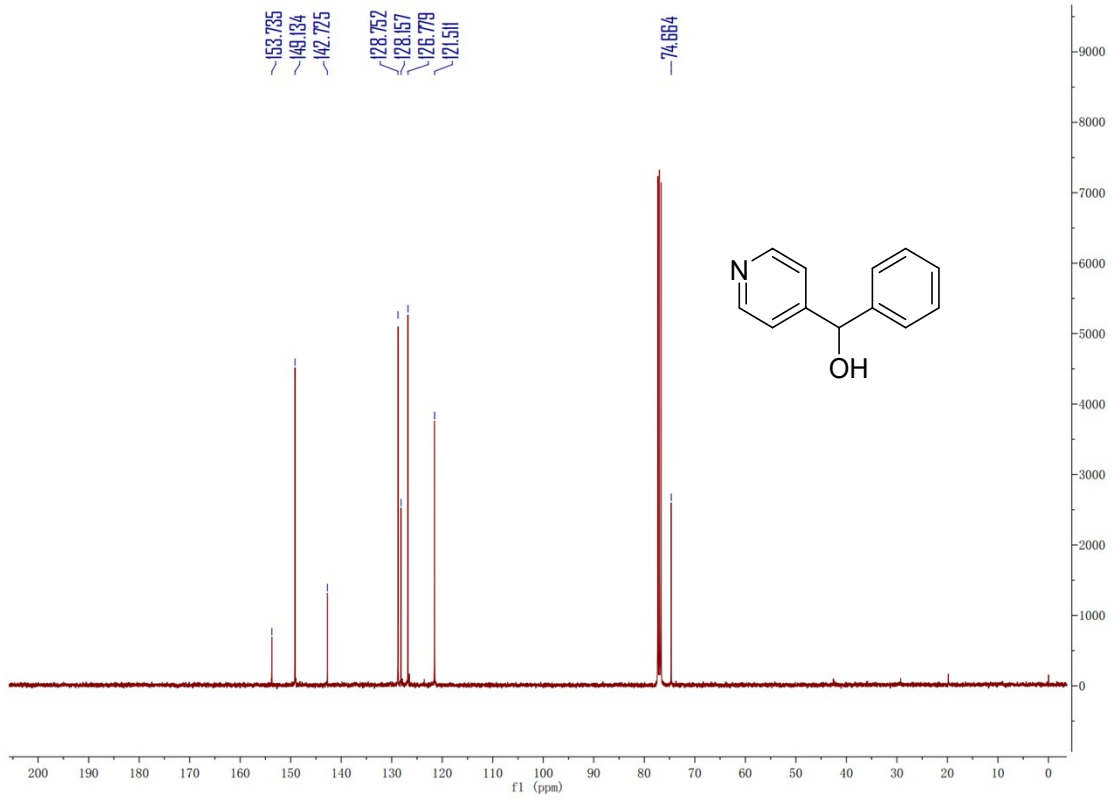


Phenyl(pyridin-4-yl)methanol (2r)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

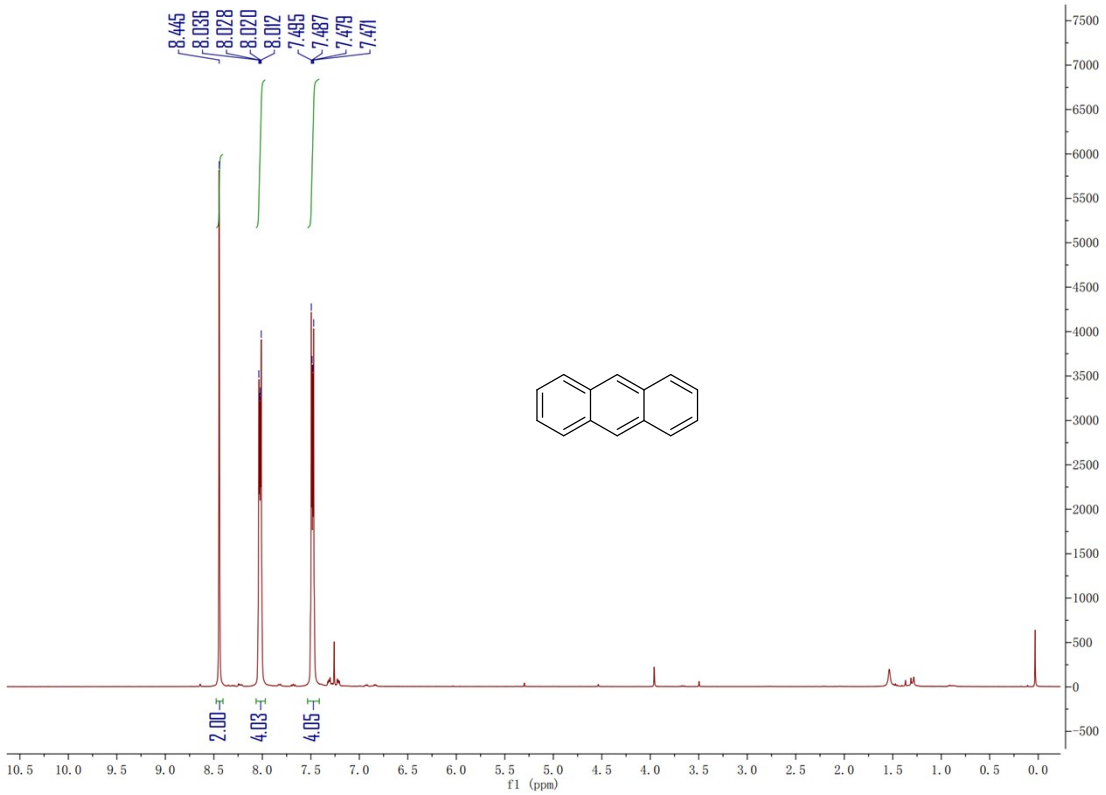


[^{13}C_NMR_100MHz_($CDCl_3$:77.00 ppm)]

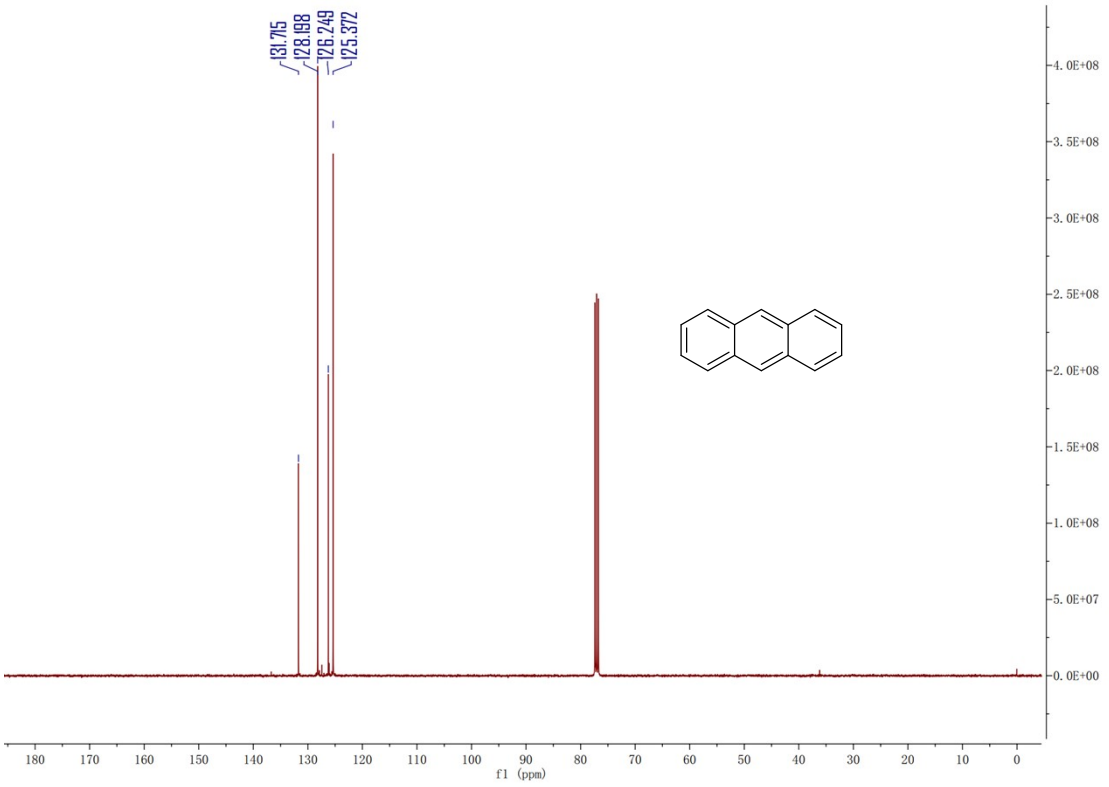


Anthracene (2s)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

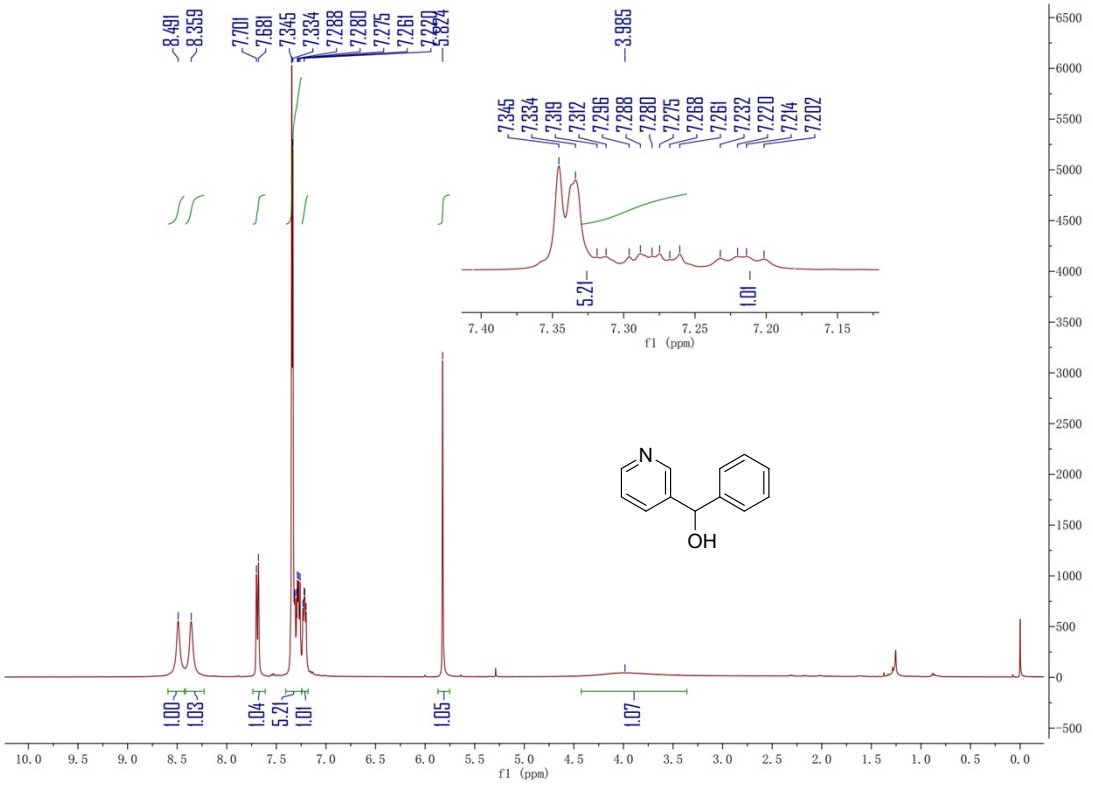


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

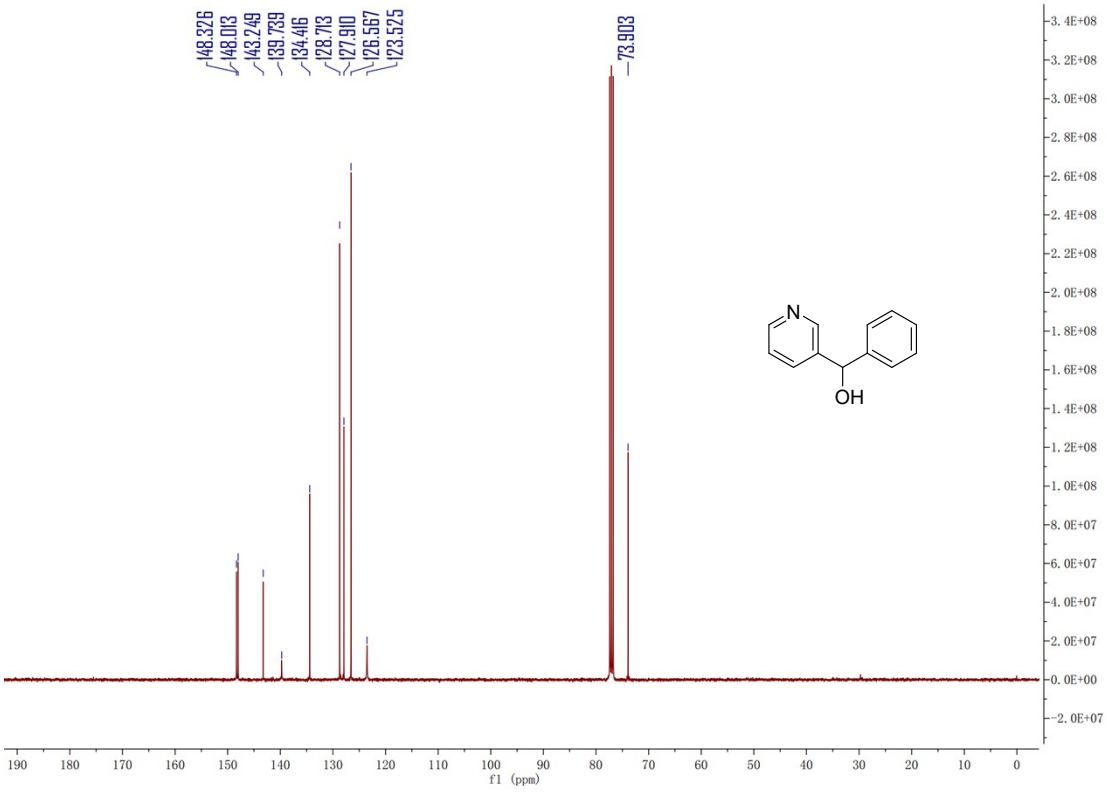


Phenyl(pyridin-3-yl)methanol (4a)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

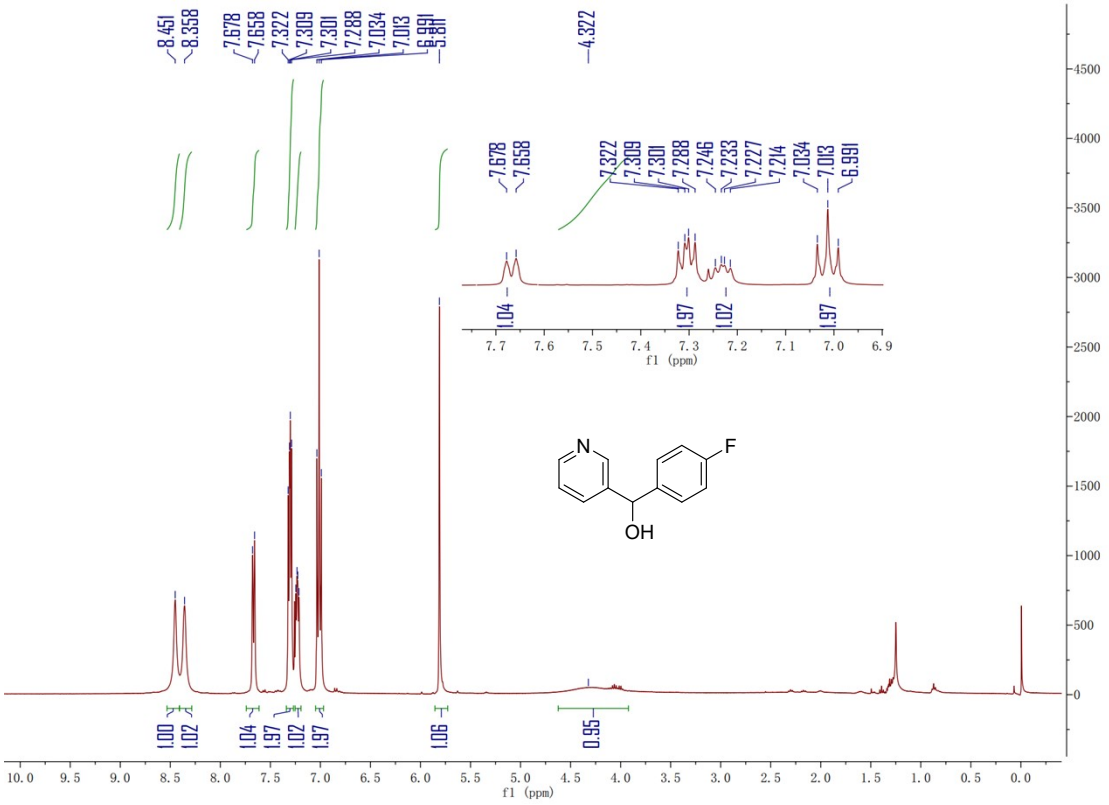


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

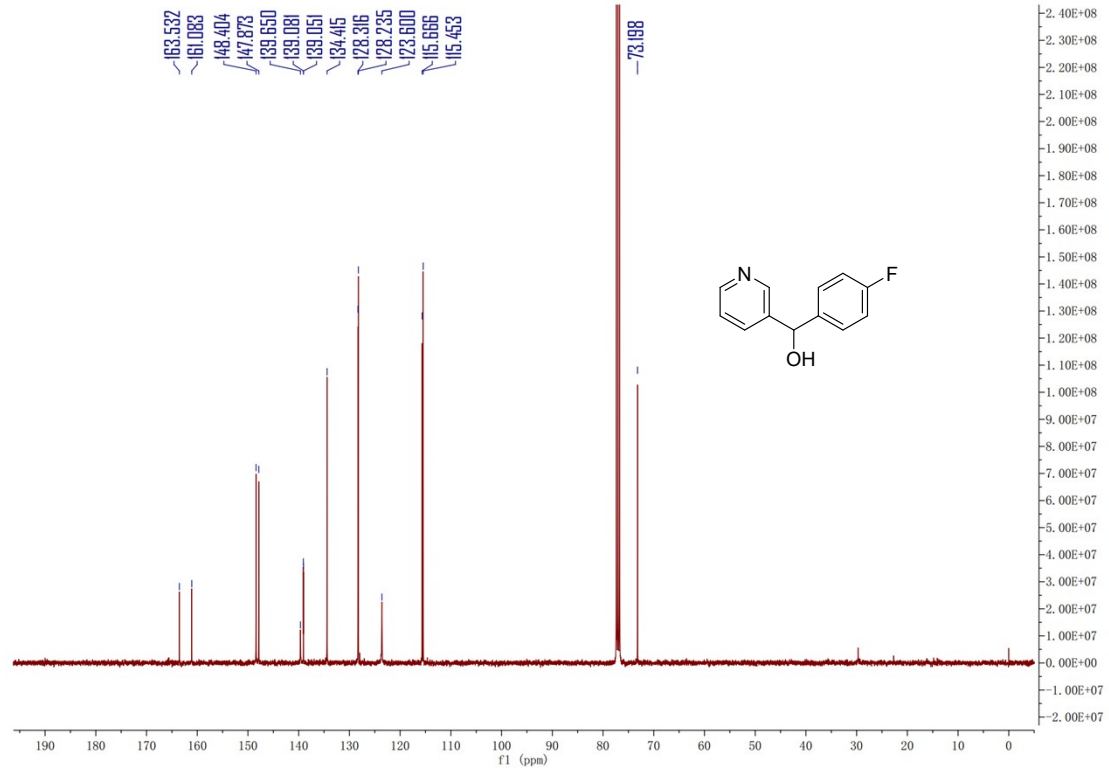


(4-Fluorophenyl)(pyridin-3-yl)methanol (4b)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

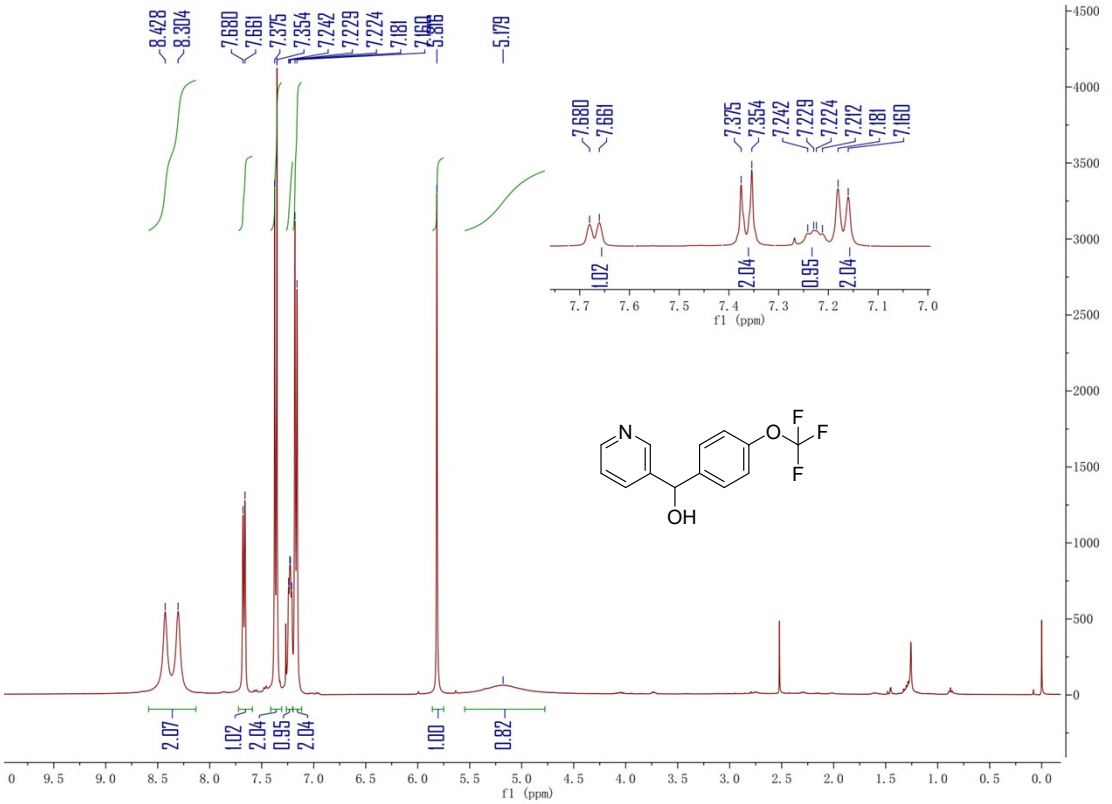


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

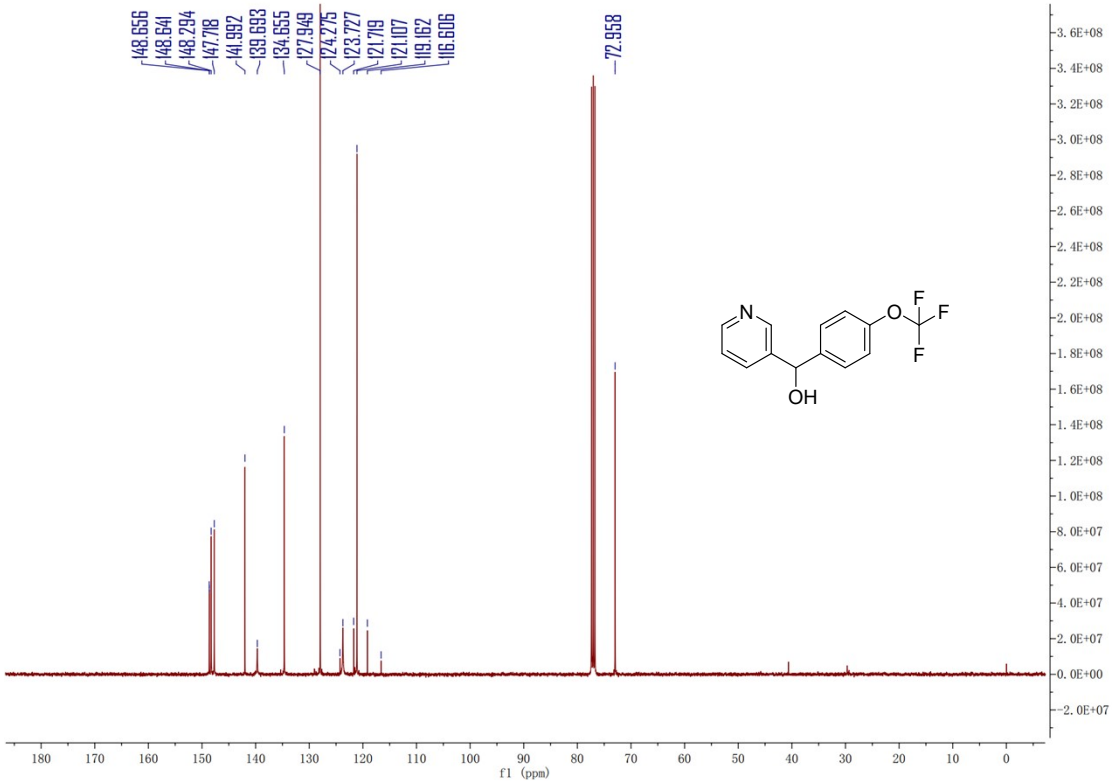


Pyridin-3-yl(4-(trifluoromethoxy)phenyl)methanol (4c)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

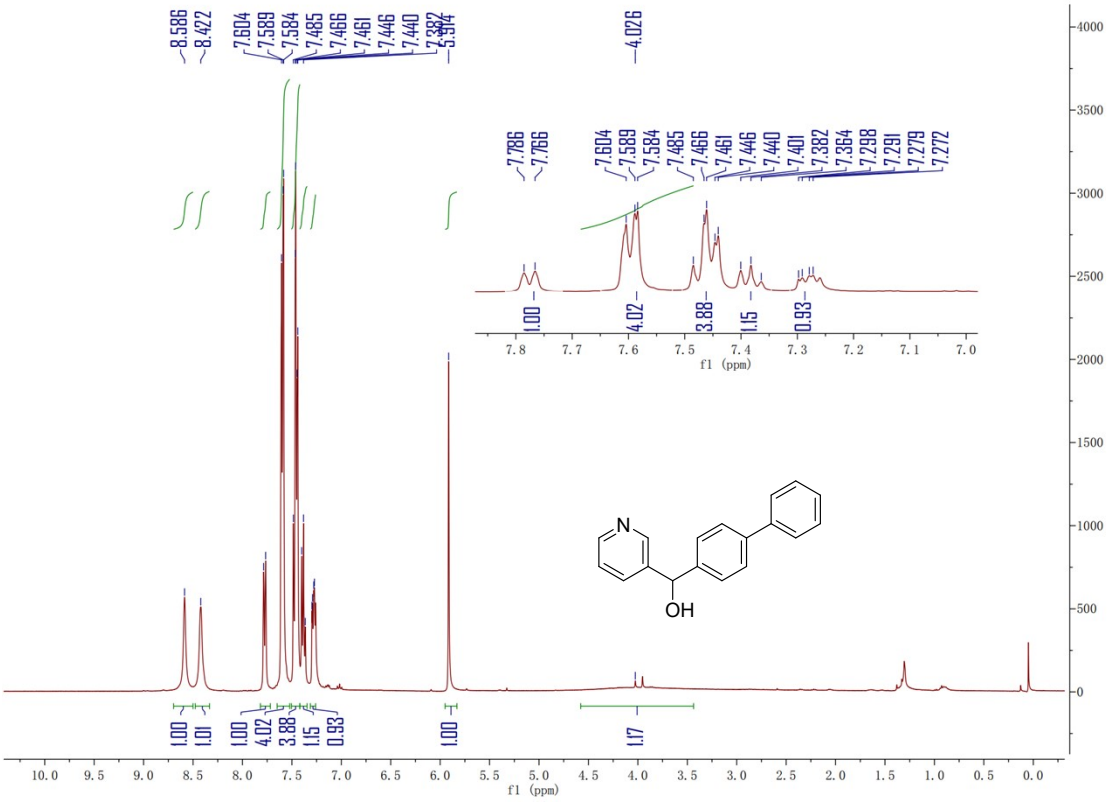


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

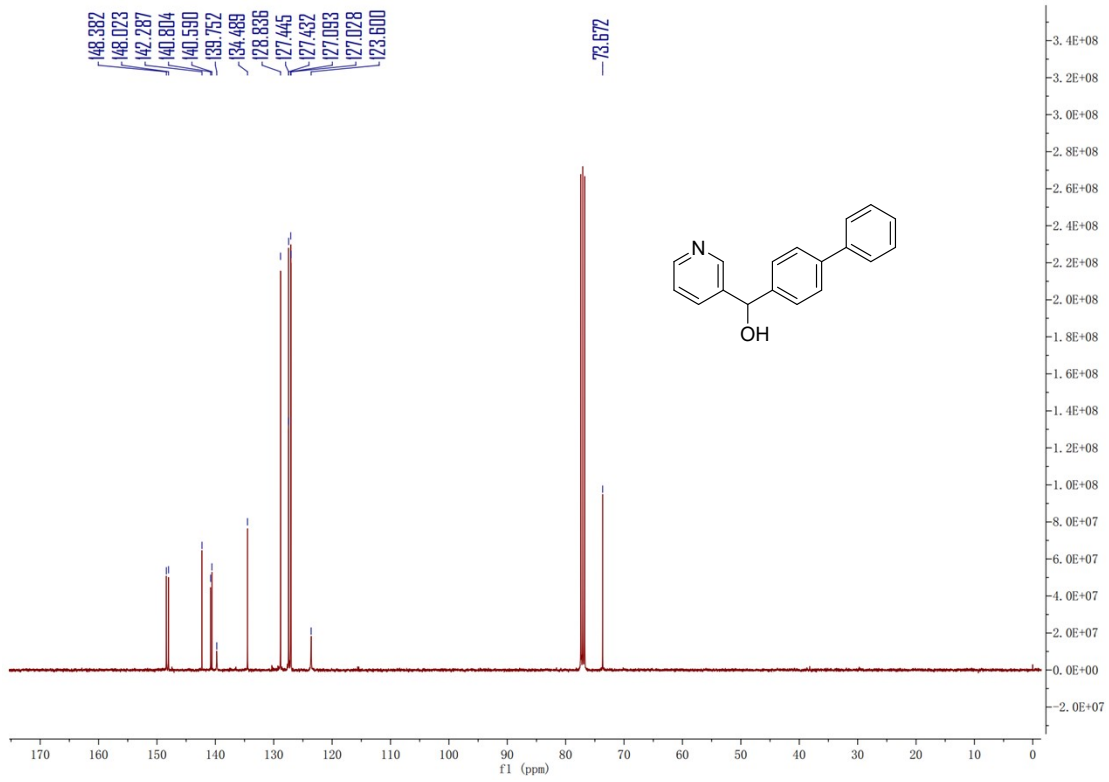


[1,1'-biphenyl]-4-yl(pyridin-3-yl)methanol (4d)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

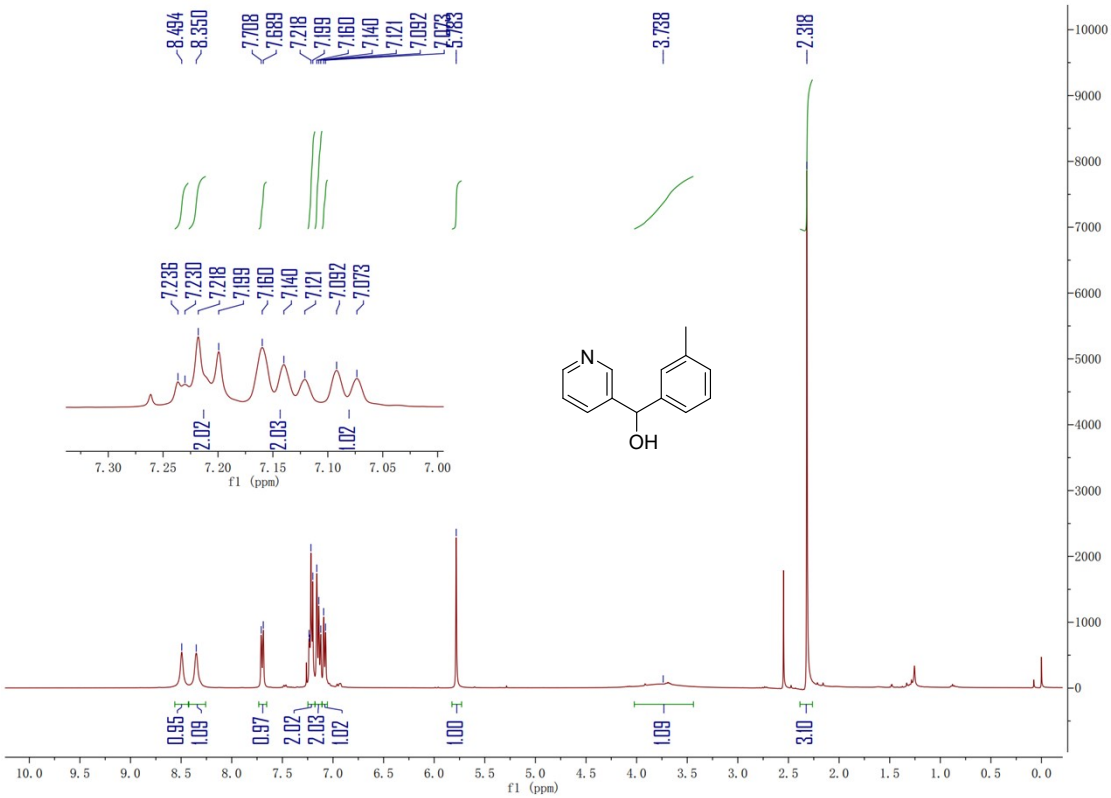


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

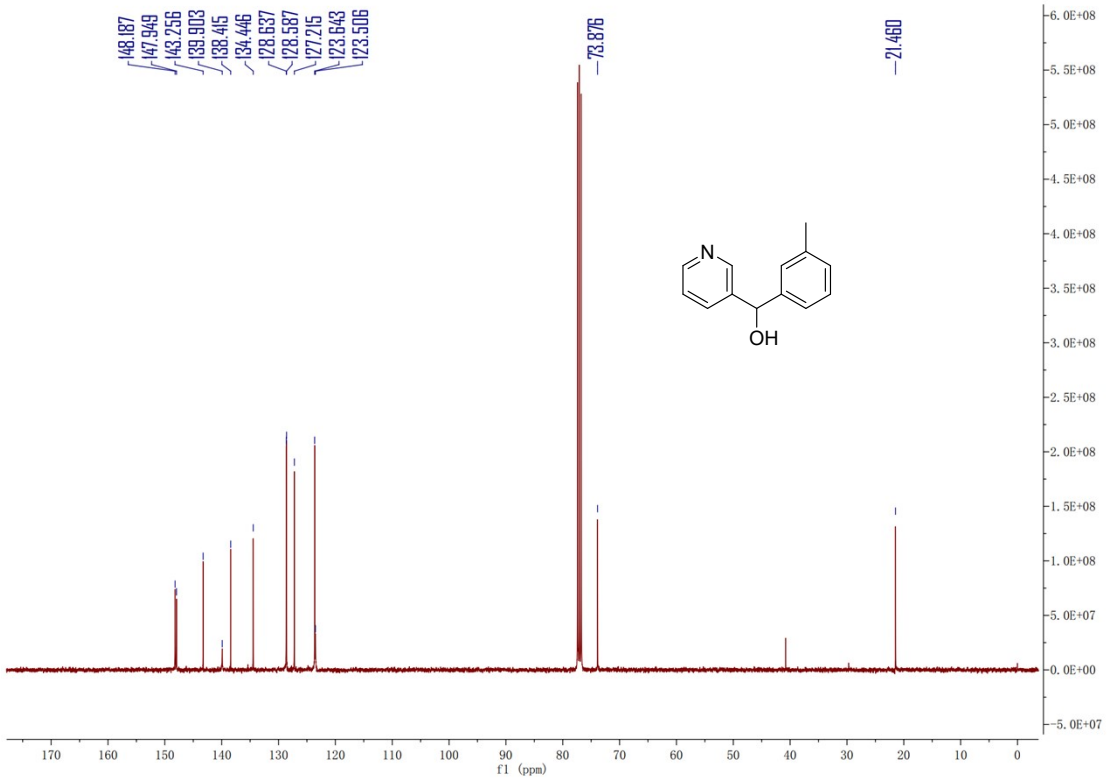


Pyridin-3-yl(m-tolyl)methanol (4e)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

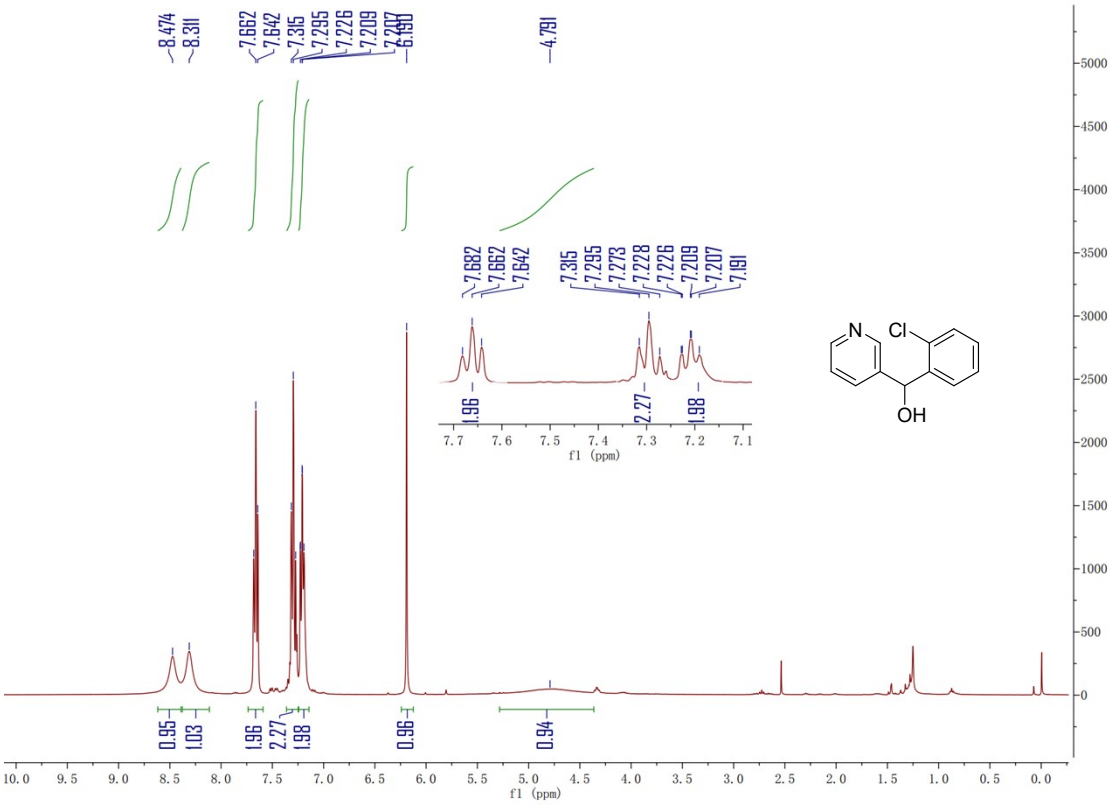


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

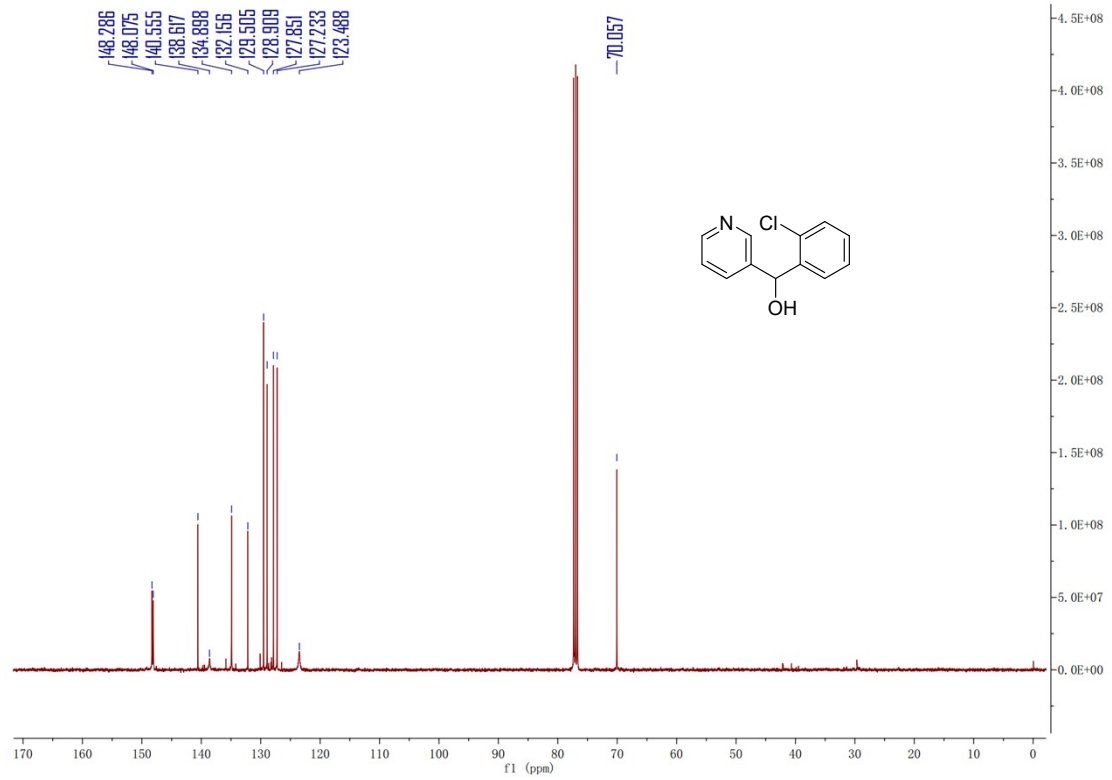


(2-Chlorophenyl)(pyridin-3-yl)methanol (4f)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

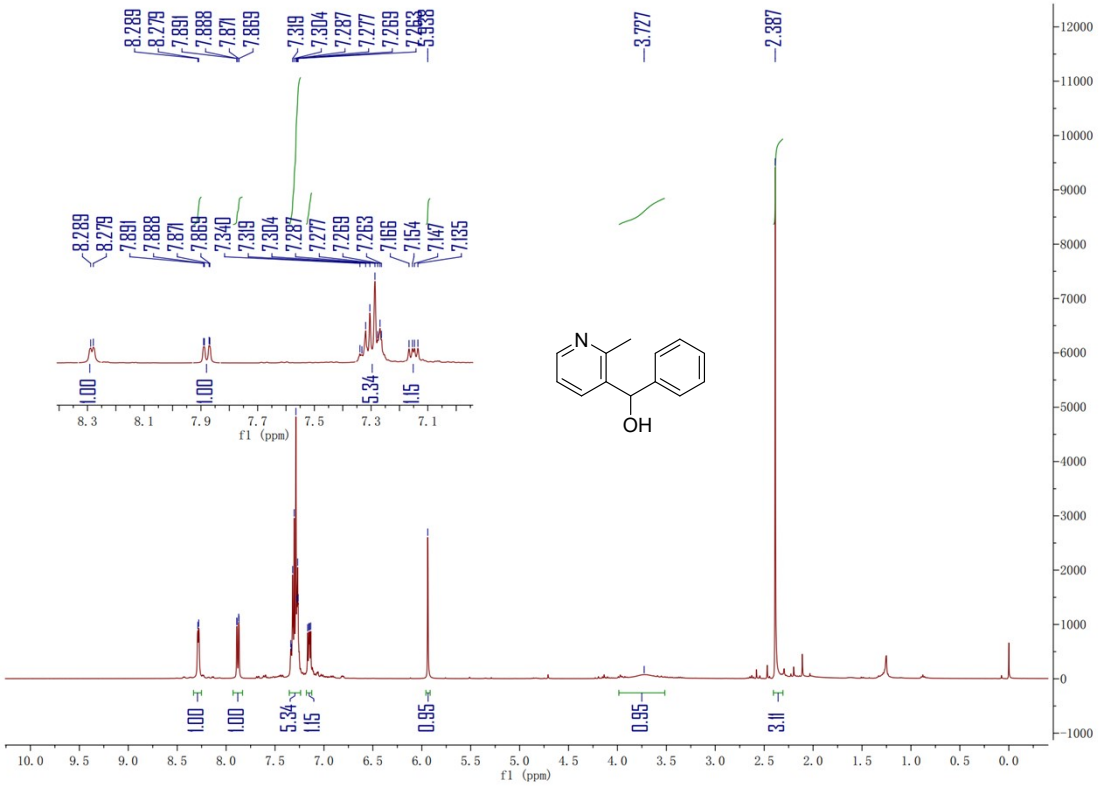


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

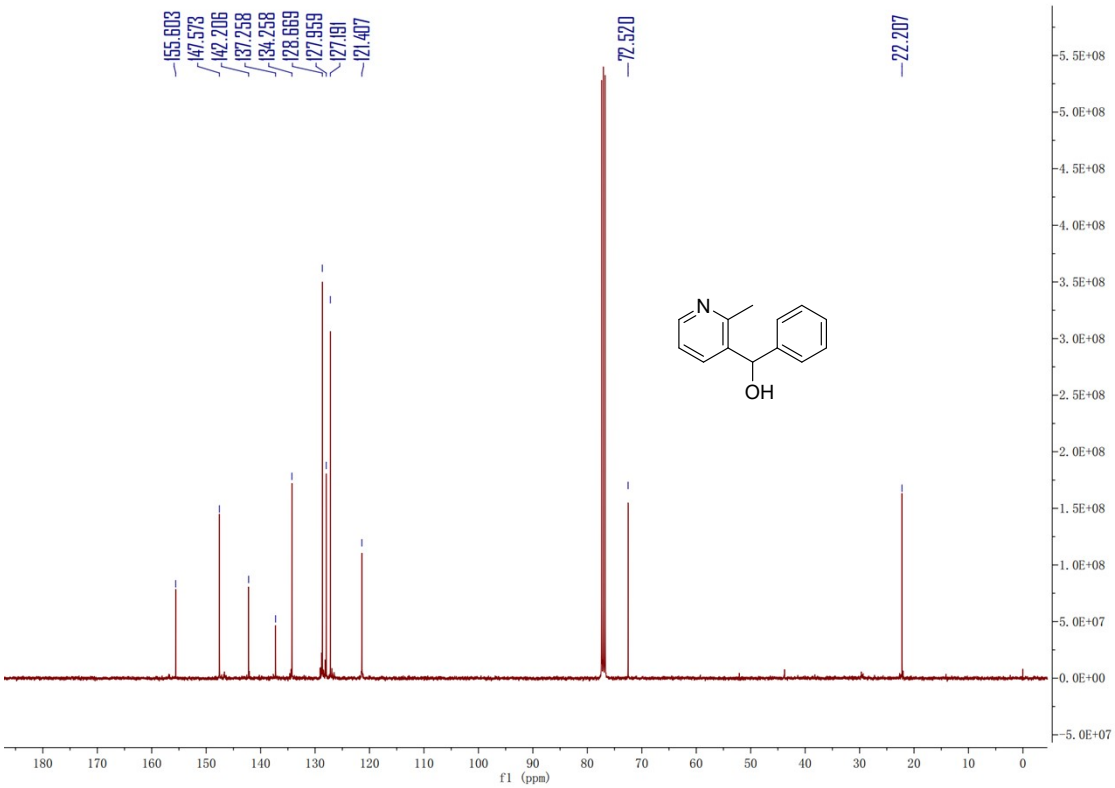


(2-Methylpyridin-3-yl)(phenyl)methanol (4g)

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

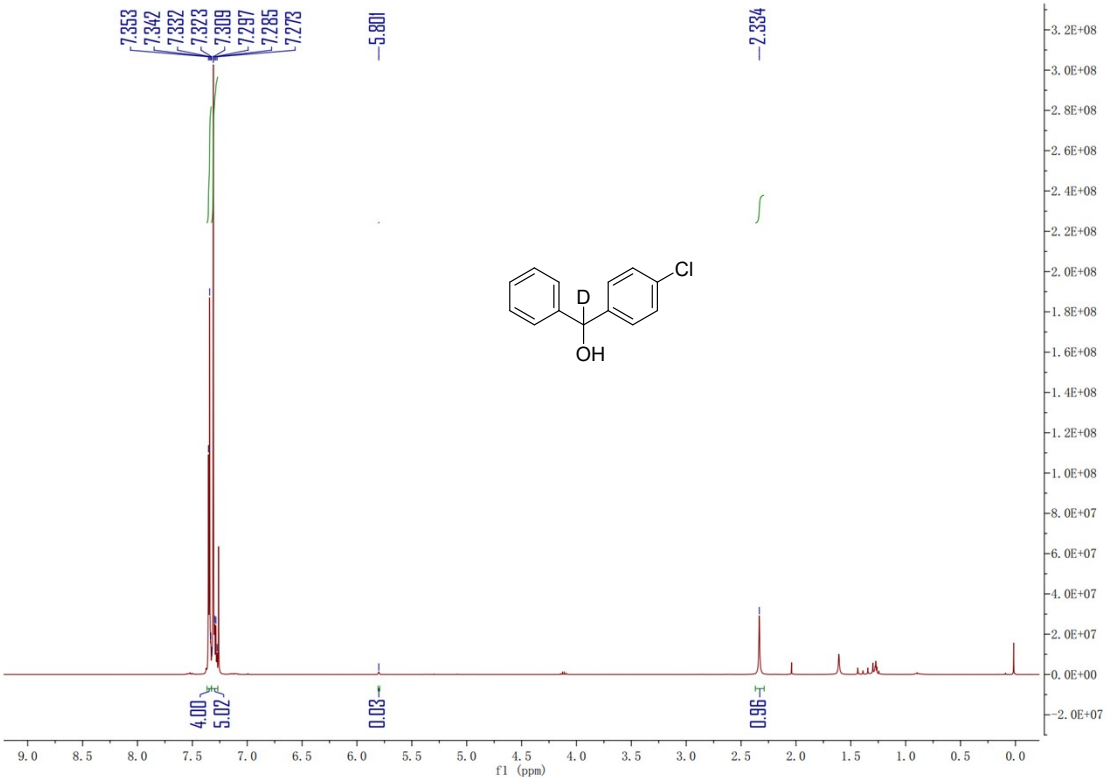


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

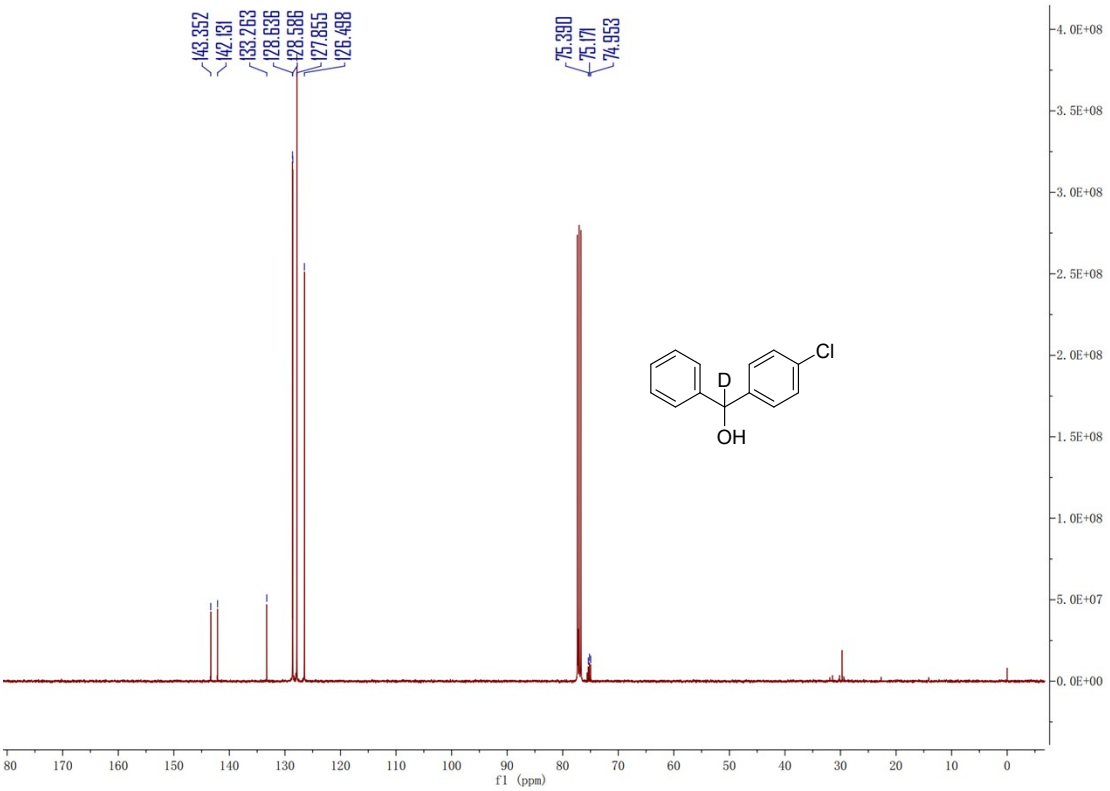


(4-Chlorophenyl)(phenyl)methan-*d*-ol (2a')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

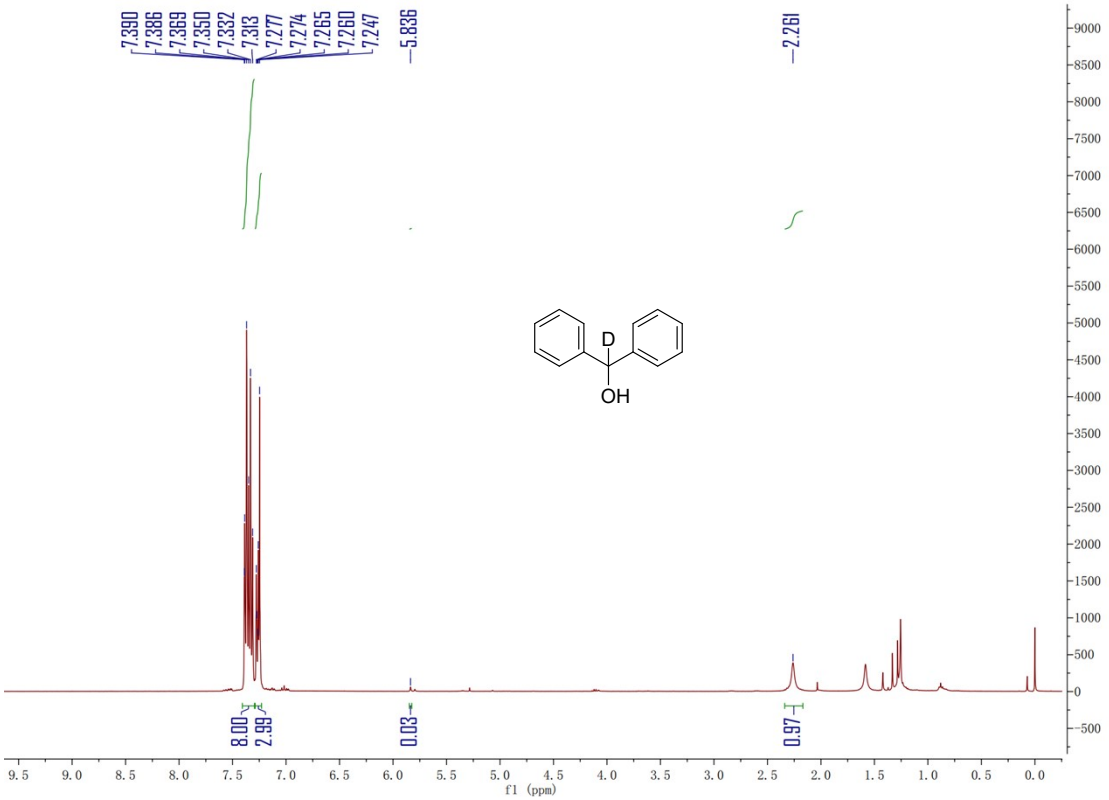


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

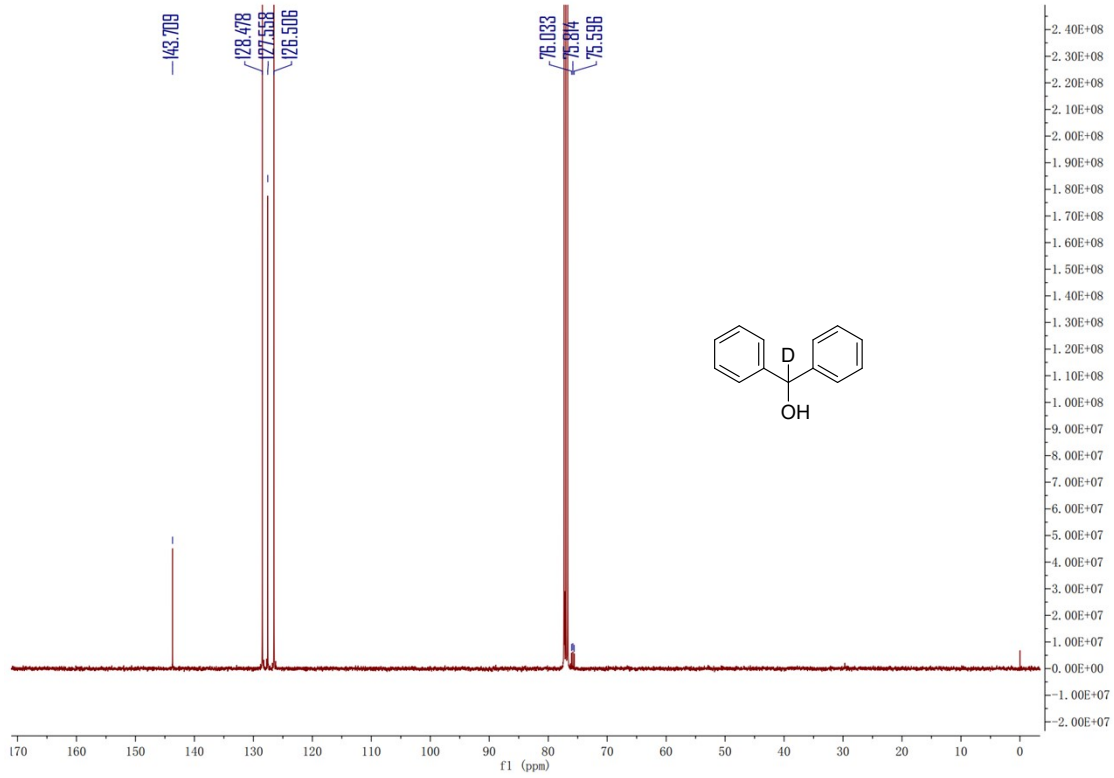


Diphenylmethan-*d*-ol (2b')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

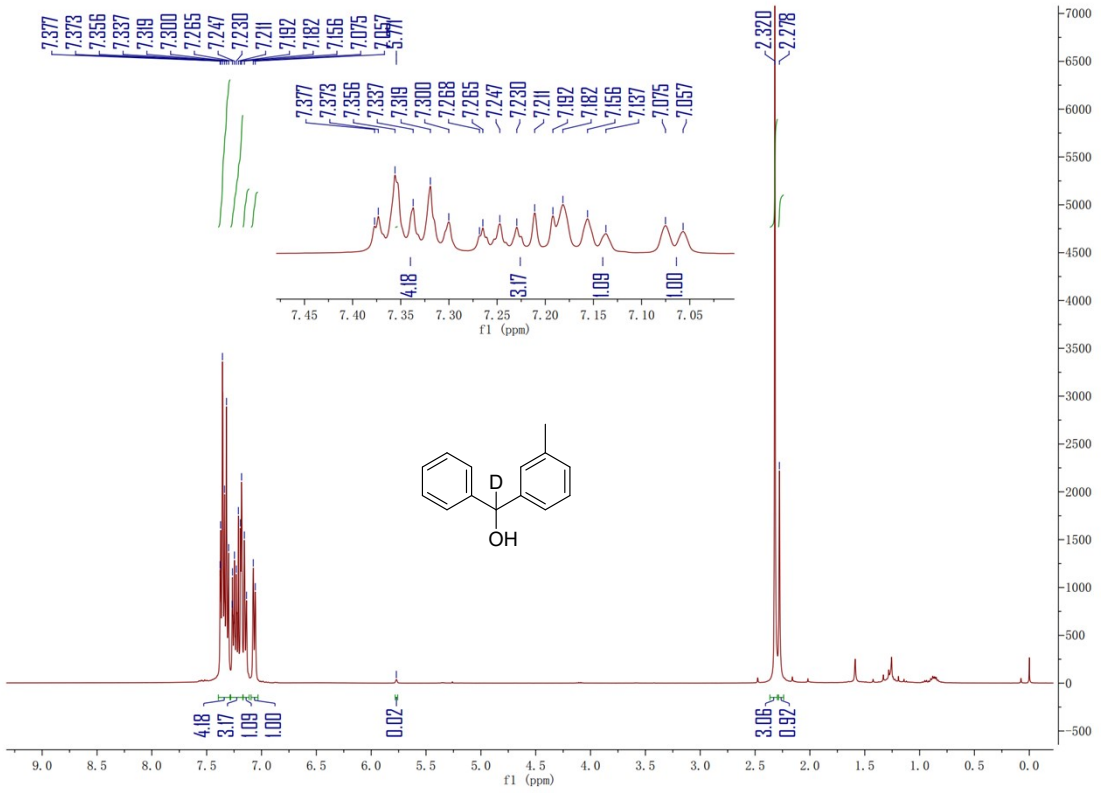


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

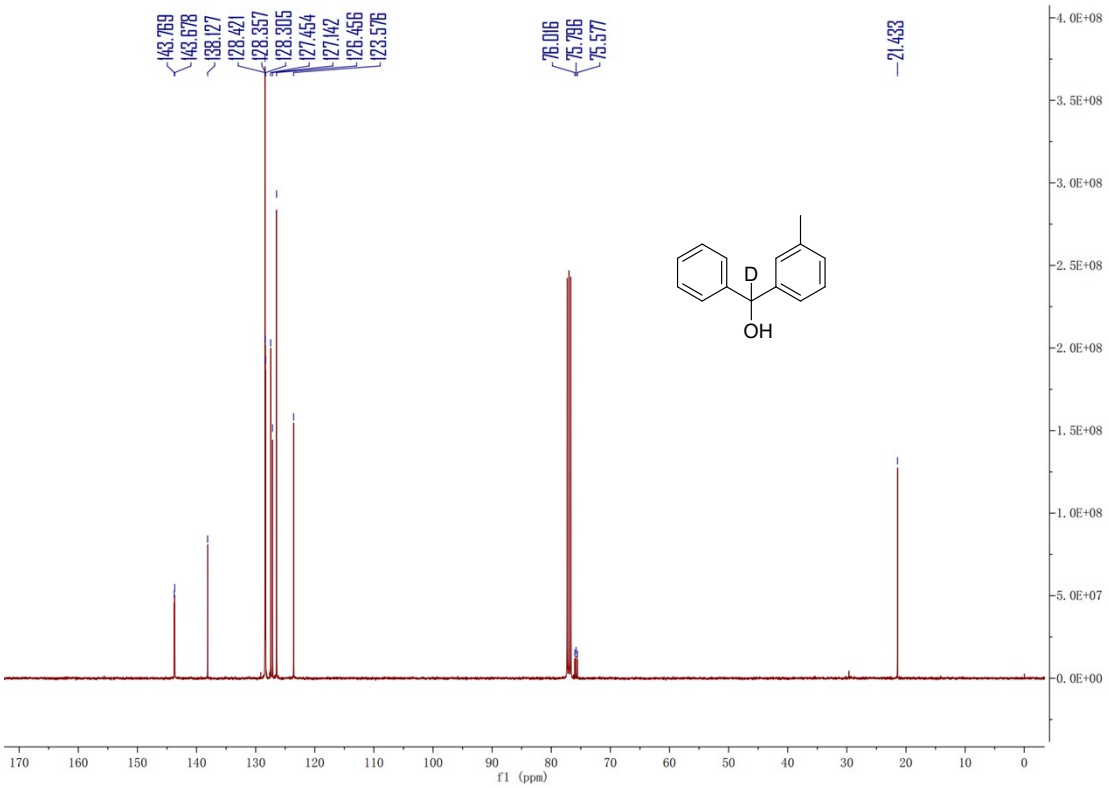


Phenyl(m-tolyl)methan-*d*-ol (2i')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

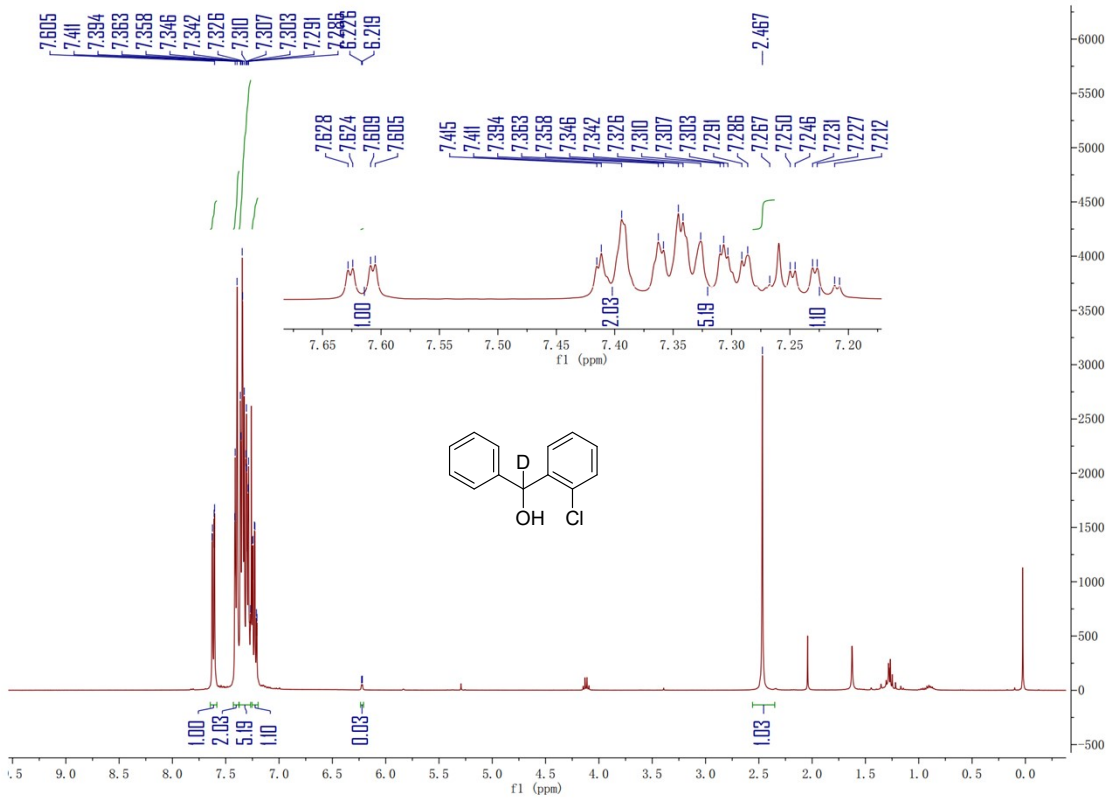


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

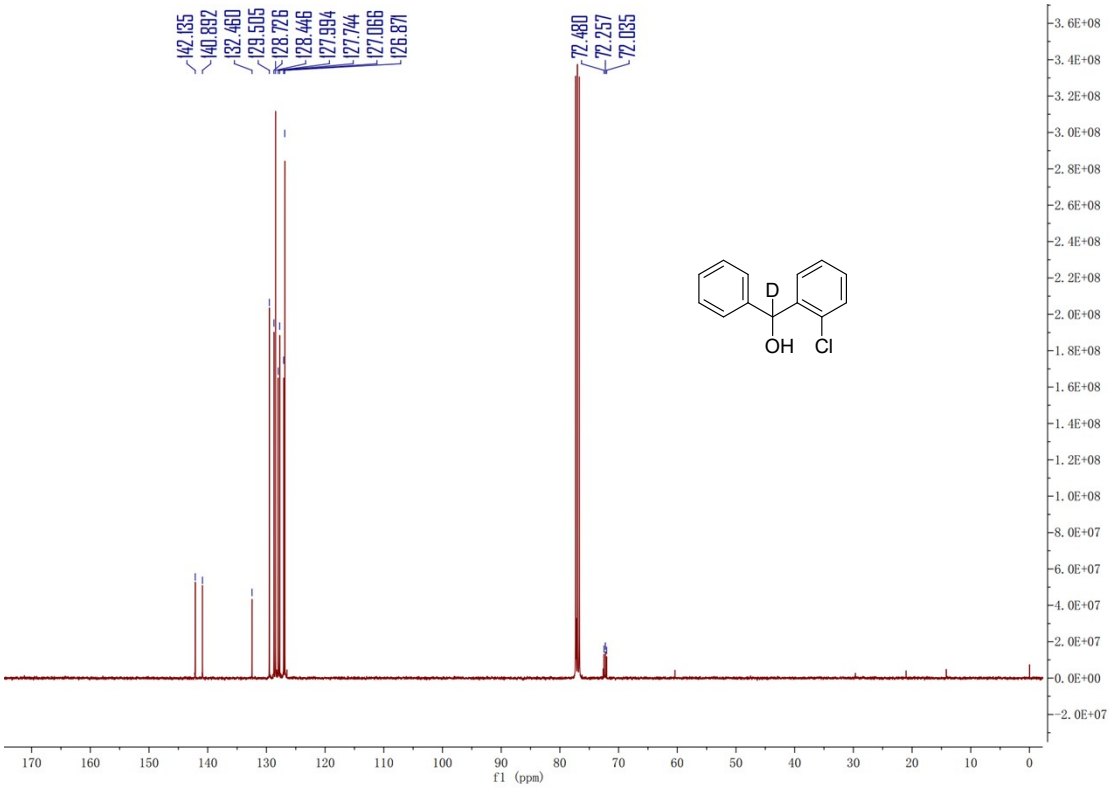


(2-Chlorophenyl)(phenyl)methan-*d*-ol (2g')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

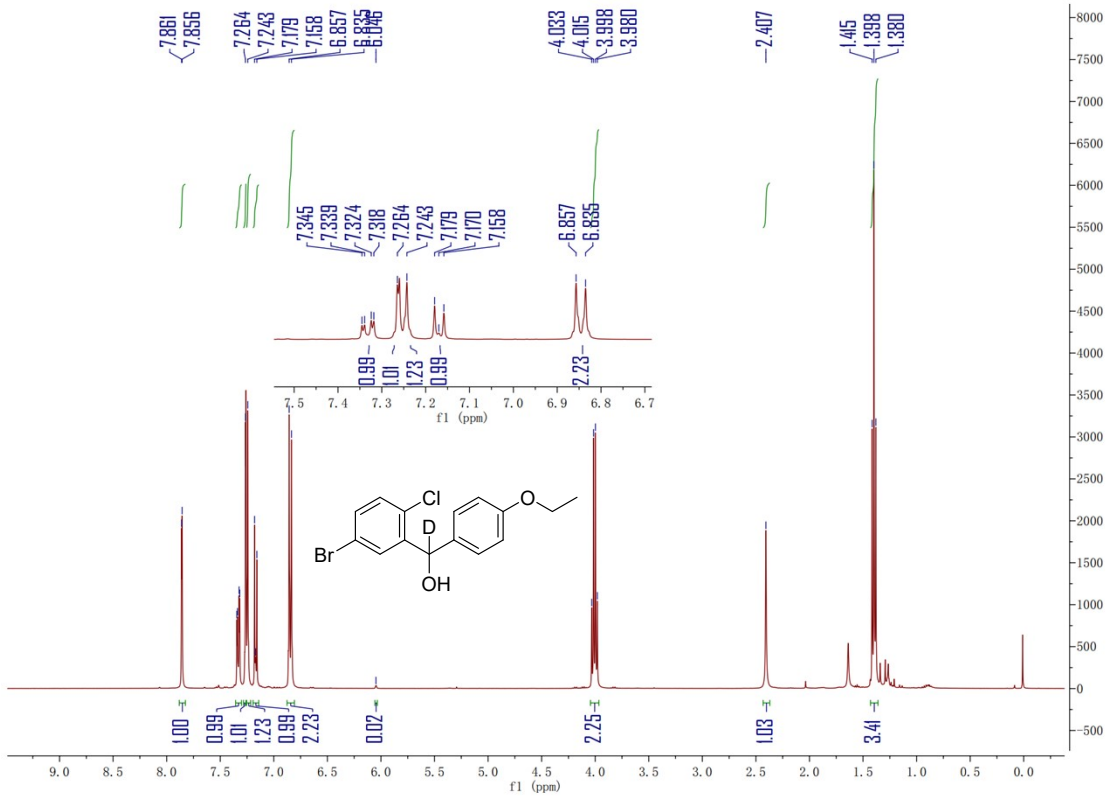


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

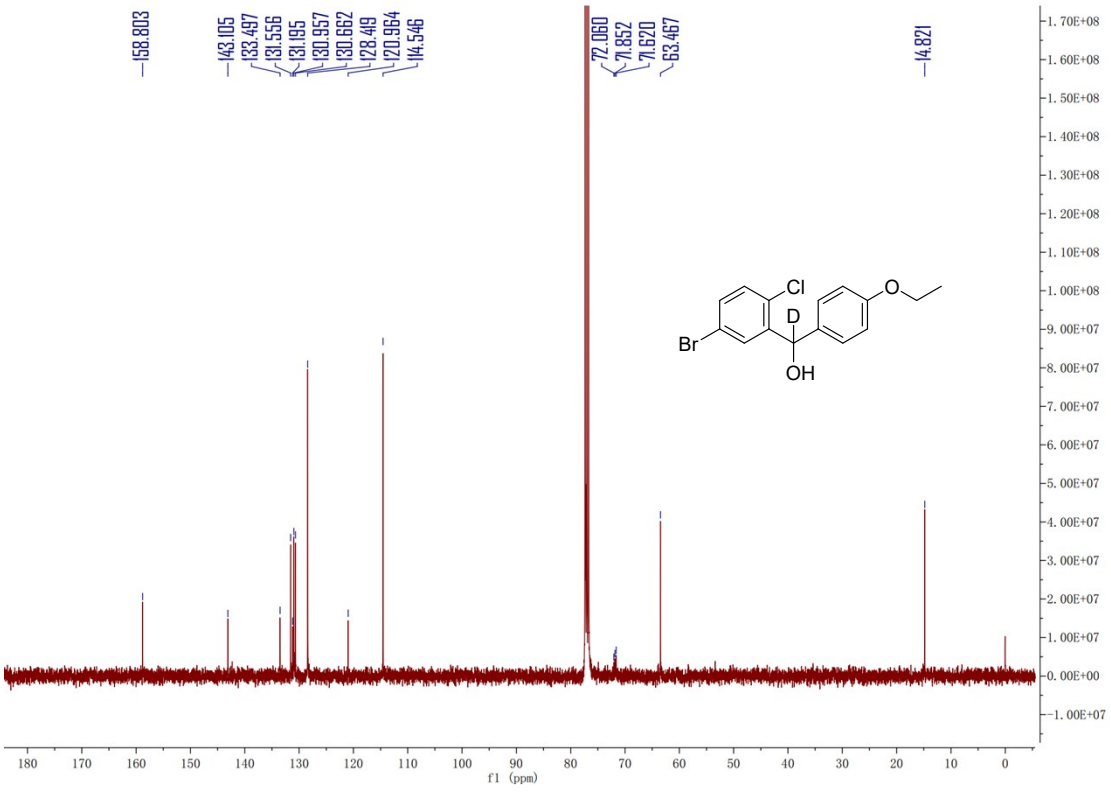


(5-Bromo-2-chlorophenyl)(4-ethoxyphenyl)methan-*d*-ol (2o')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

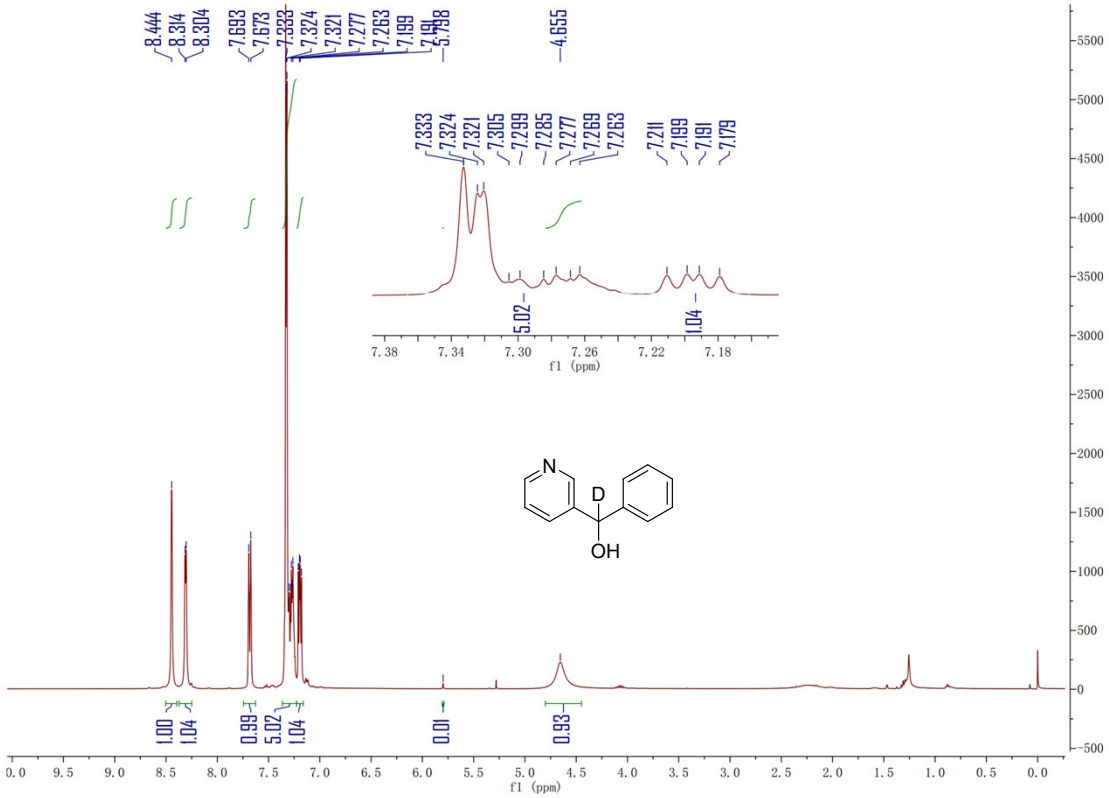


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

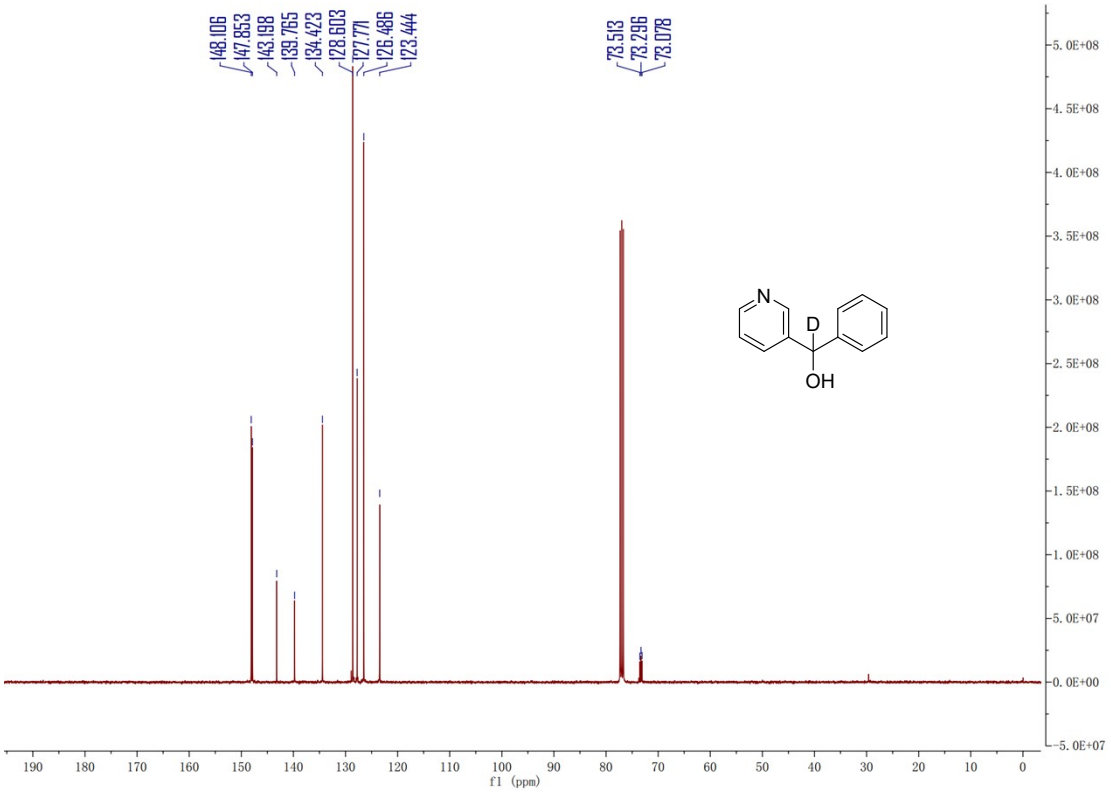


Phenyl(pyridin-3-yl)methan-*d*-ol (4a')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

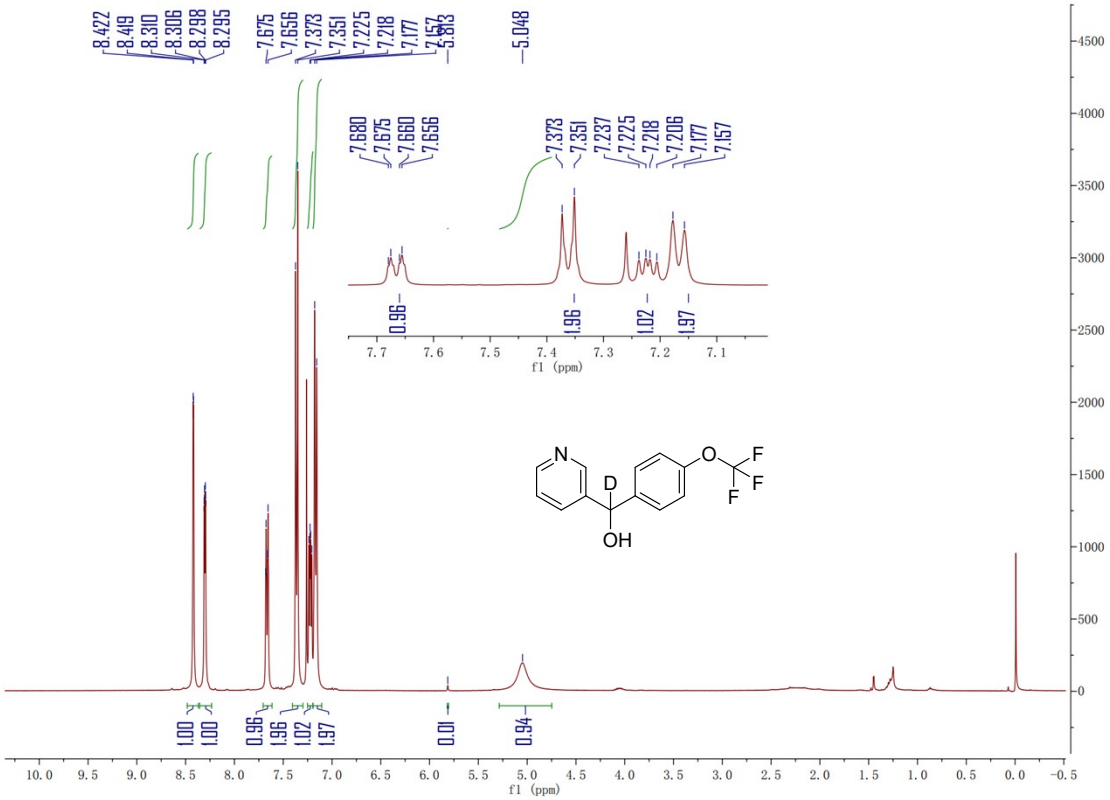


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

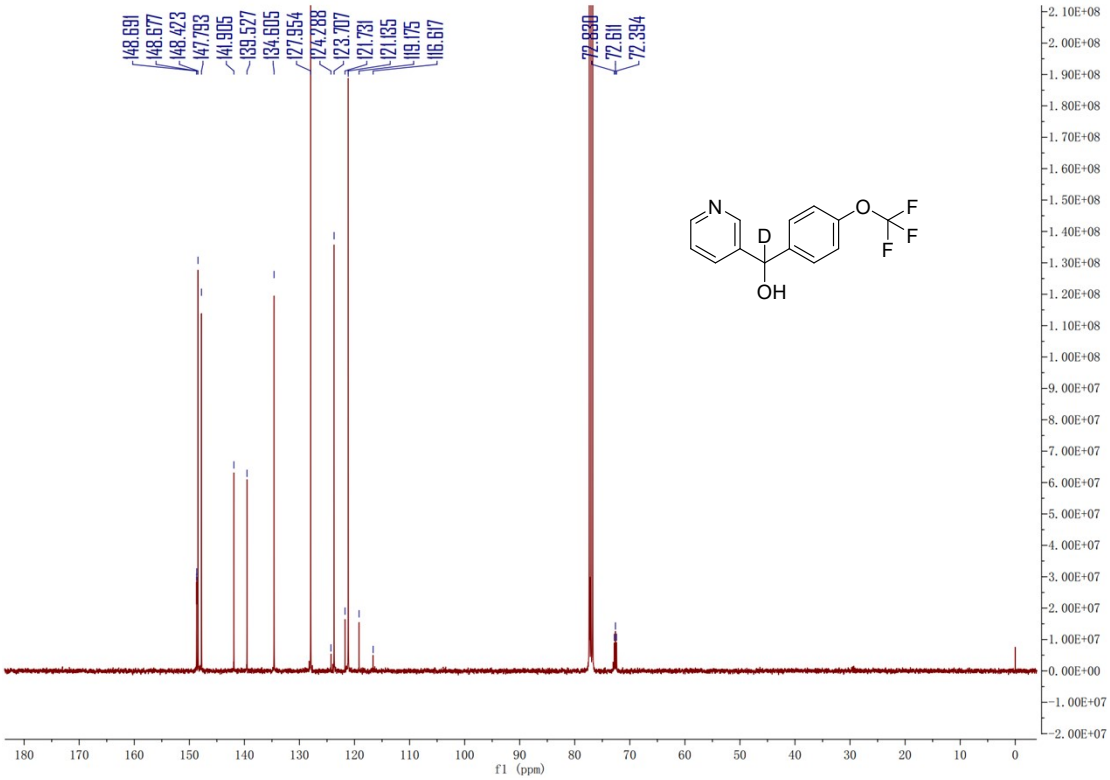


Pyridin-3-yl(4-(trifluoromethoxy)phenyl)methan-*d*-ol (4c')

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]

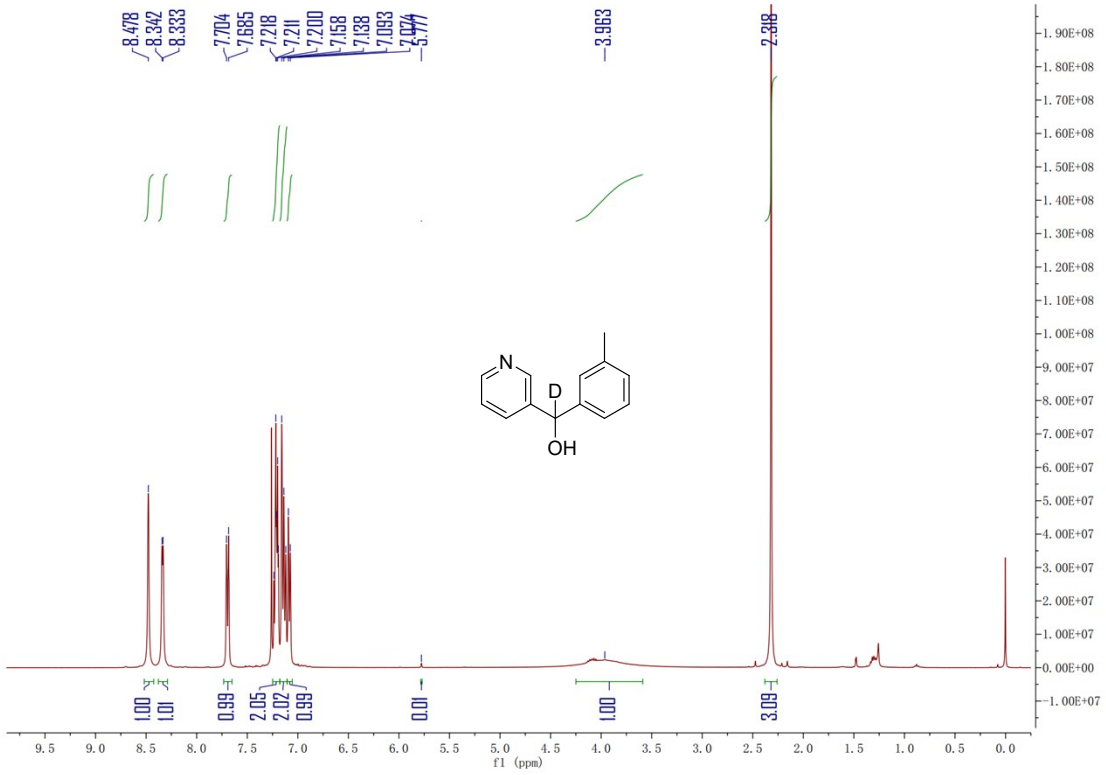


[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]

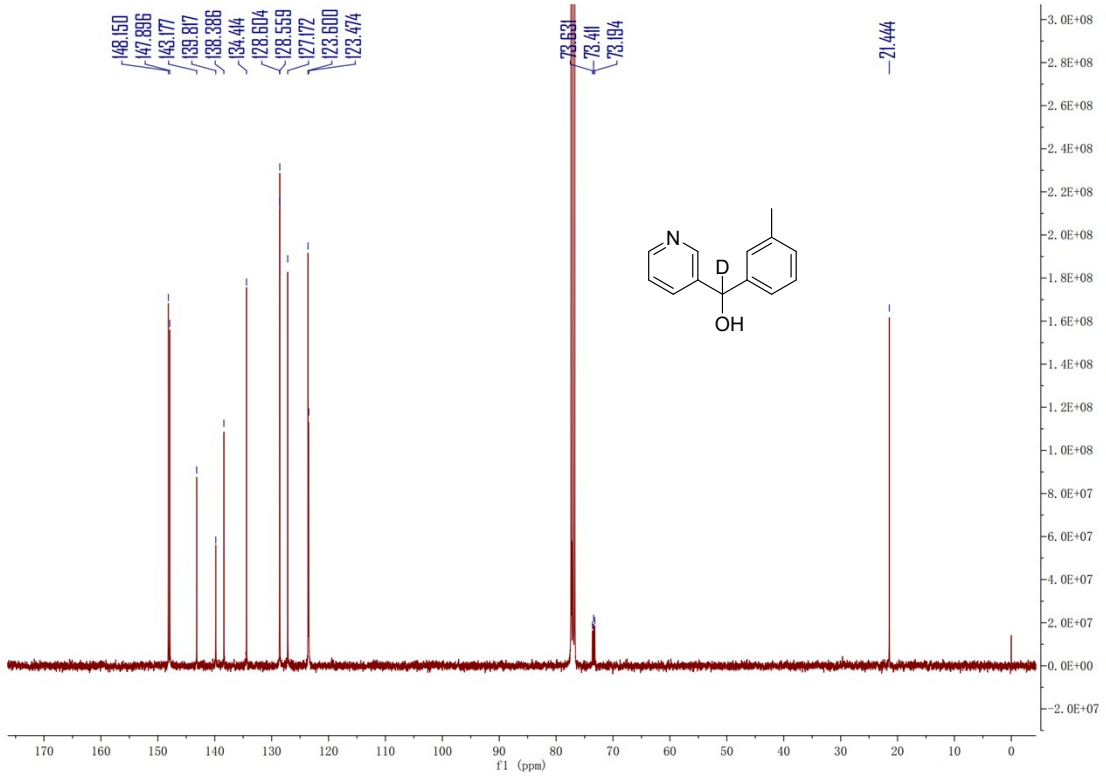


Pyridin-3-yl(m-tolyl)methan-*d*-ol (4e')

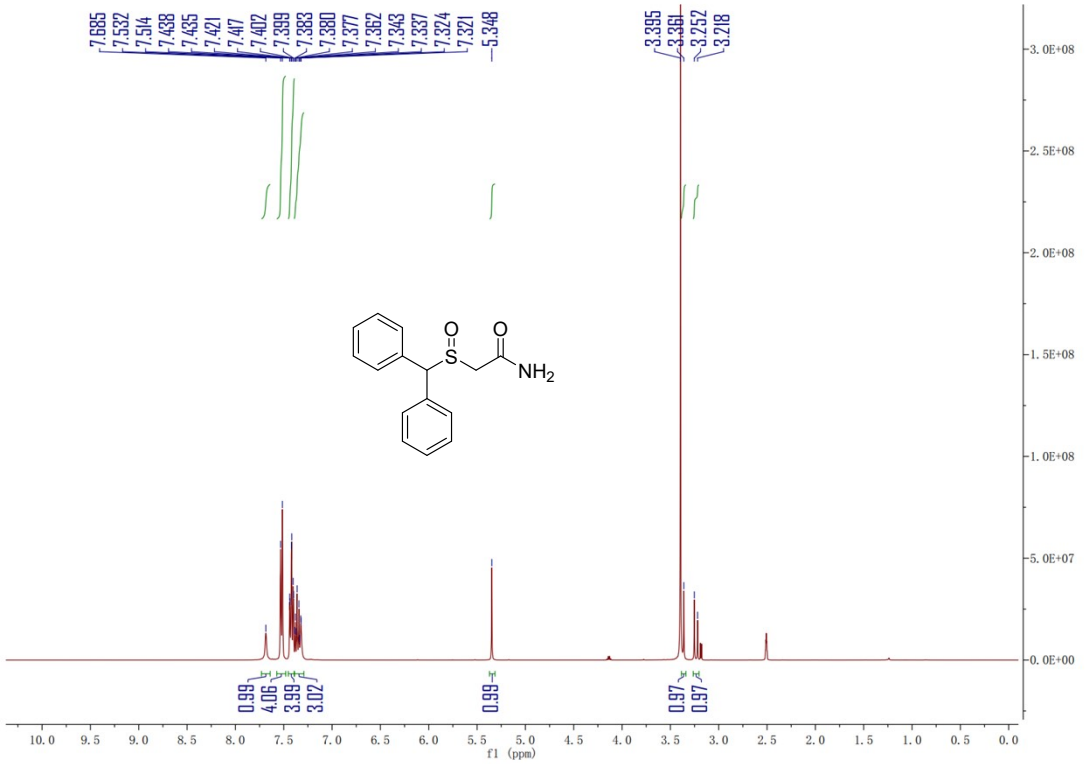
[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



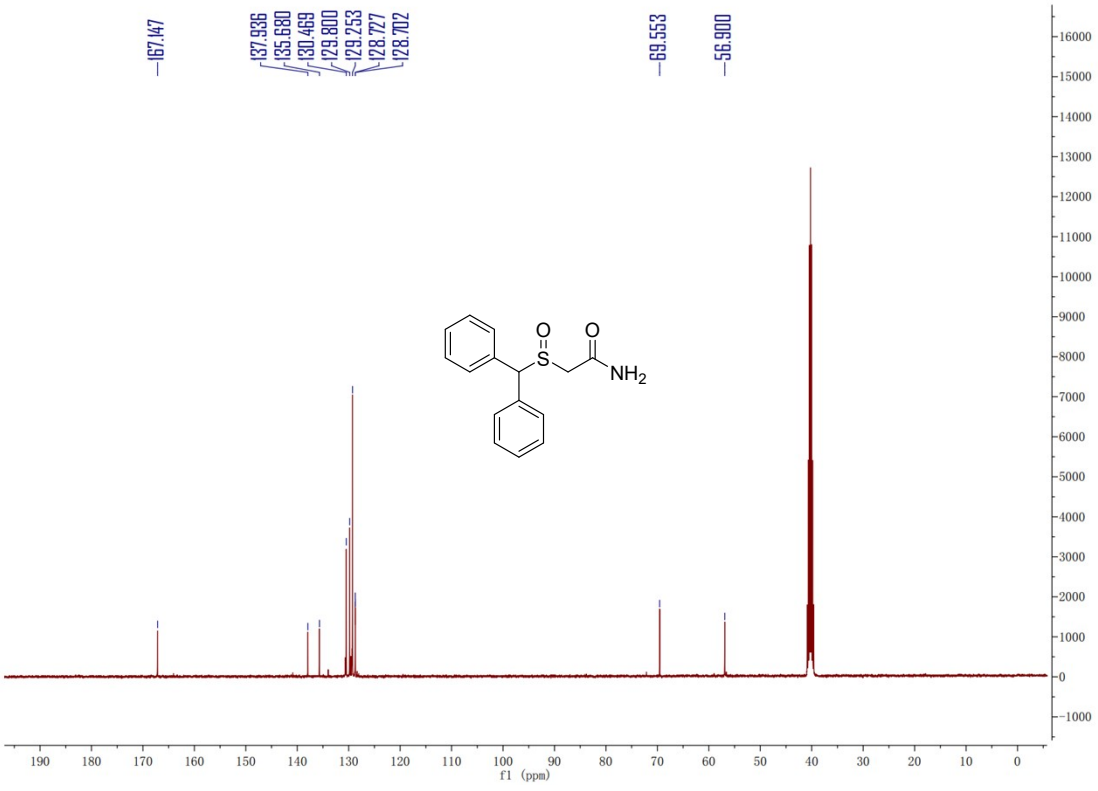
[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]



Modafinil [^{1}H_NMR_400 MHz_(DMSO-d_6: 2.50 ppm)]

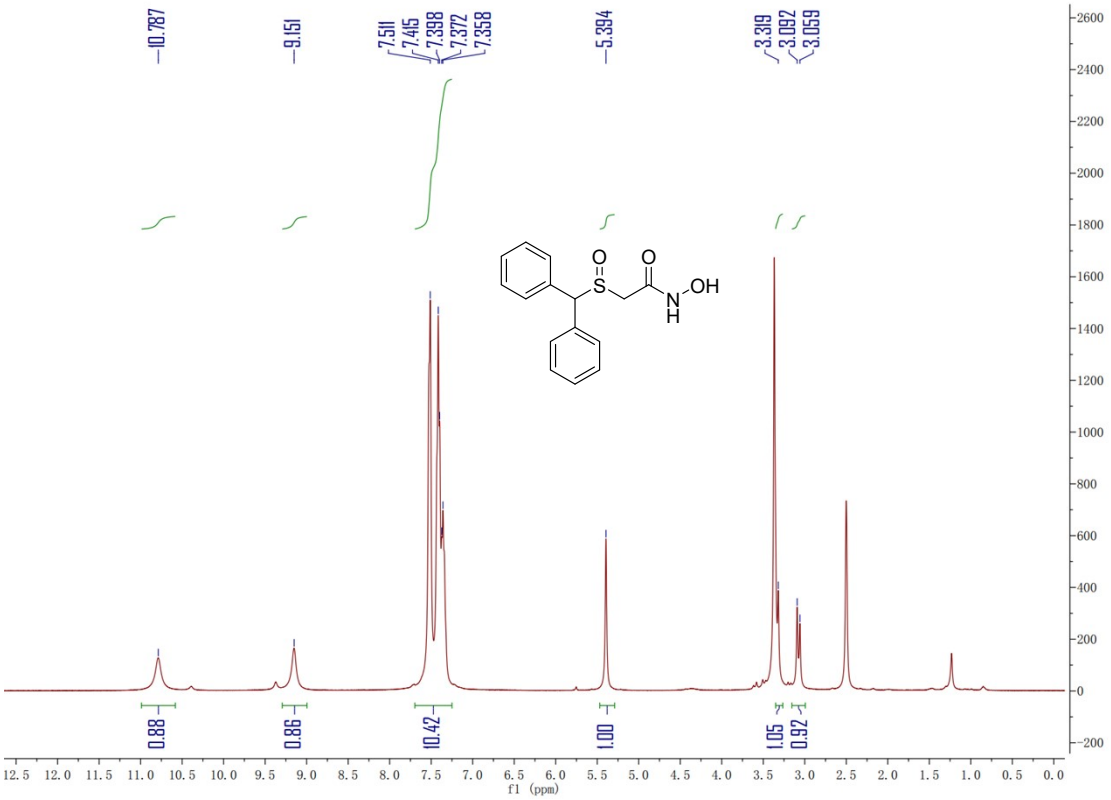


[^{13}C_NMR_100 MHz_(DMSO-d_6: 39.52 ppm)]

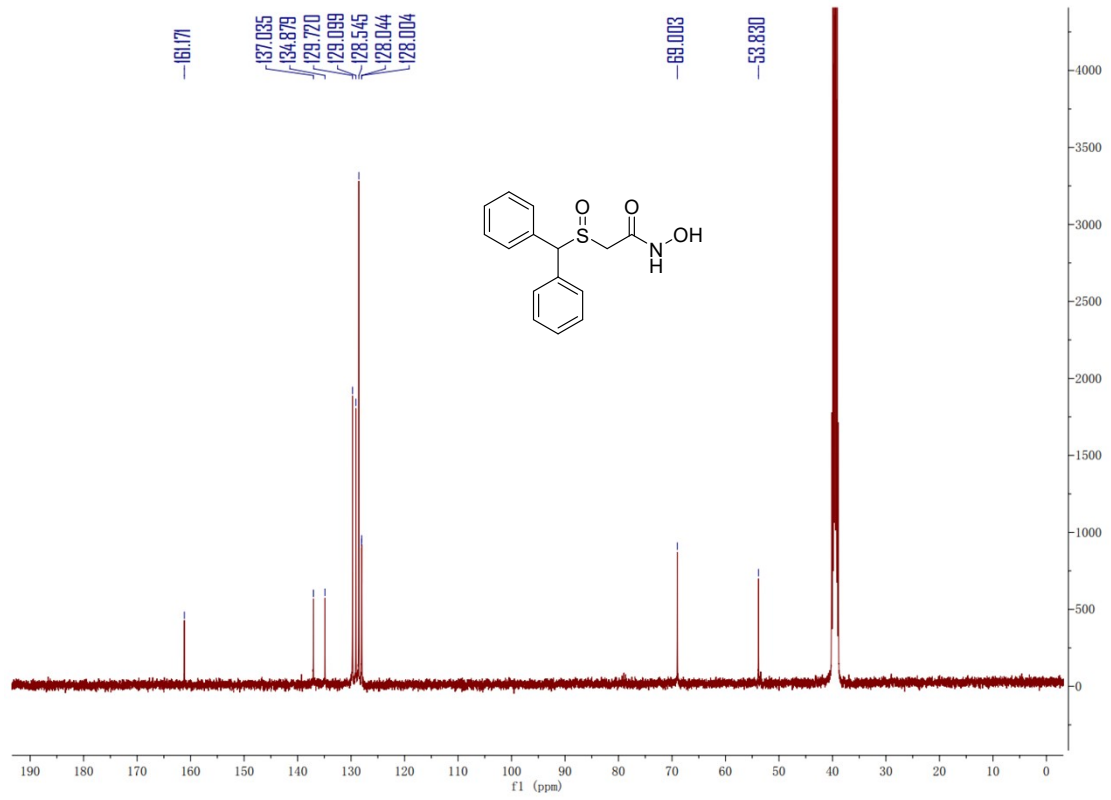


Adrafinil

[^{1}H_NMR_400 MHz_(DMSO-d_6: 2.50 ppm)]

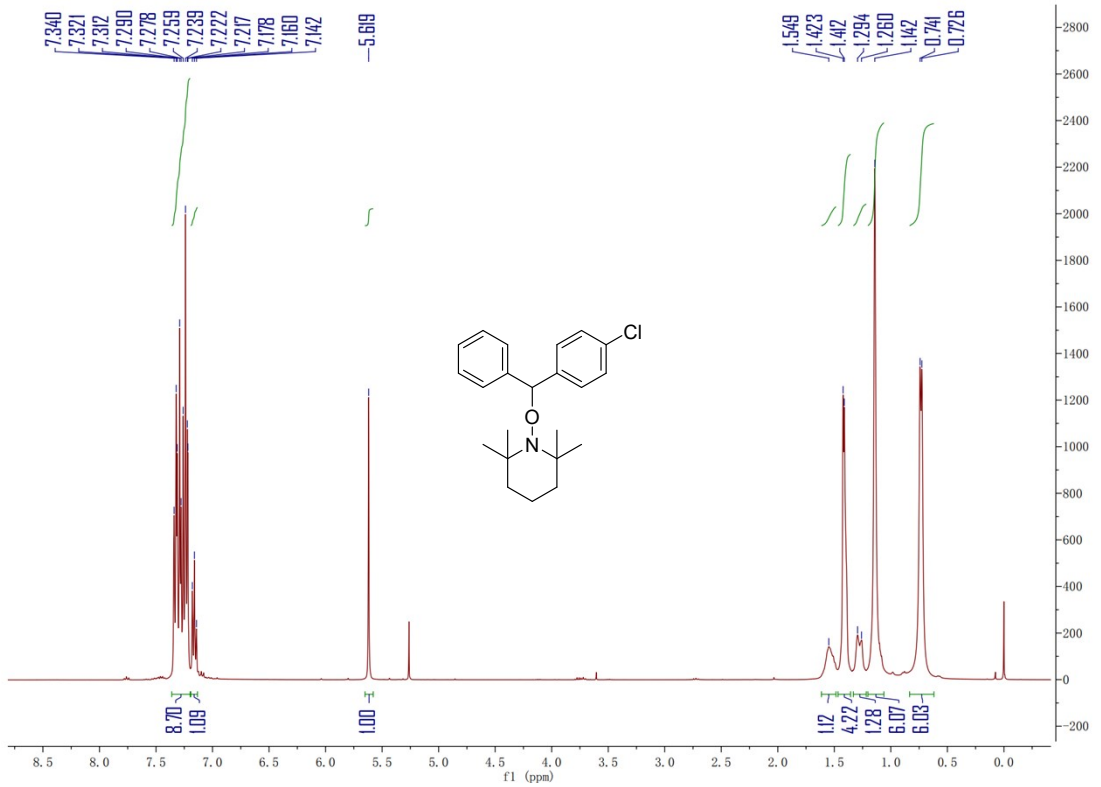


[^{13}C_NMR_100 MHz_(DMSO-d_6: 39.52 ppm)]



1-((4-Chlorophenyl)(phenyl)methoxy)-2,2,6,6-tetramethylpiperidine

[^{1}H_NMR_400 MHz_($CDCl_3$: 7.26 ppm)]



[^{13}C_NMR_100 MHz_($CDCl_3$: 77.00 ppm)]