

Supporting Information

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1. General Analytical Information

Nuclear Magnetic Resonance spectra were recorded on Bruker Avance 400 MHz or 600 MHz instruments at ambient temperature. All ^1H NMR spectra were measured in part per million (ppm) relative to the signals of tetramethylsilane (TMS, 0.00 ppm) added into the deuterated chloroform (CDCl_3 , 7.30 ppm) unless otherwise stated. Data for ^1H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets), coupling constants, and integration. All ^{13}C NMR spectra were reported in ppm relative to tetramethylsilane (0.00 ppm) unless otherwise stated, and were obtained with complete ^1H decoupling. All GC analyses were performed on a Perkin-Elmer Clarus 400 GC system with an FID detector. High-resolution mass spectra were obtained with an AB Triple 5600 mass spectrometer by ESI on a TOF mass analyzer. X-ray diffraction spectroscopy was tested using a Shimadzu XRD-6100 model. Scanning electron microscope was tested with TESCAN model MAIA3 LMH, transmission electron microscope, low magnification observation was done with Thermo Fisher model Talos L120C G2, and high magnification observation was done with JEM-ARM200F (NEOARM), a 200KV spherical aberration-corrected transmission electron microscope, which is equipped with the Cold-FEG electron gun (Cold-FEG) and the new high-order spherical aberration corrector (ASCOR) developed by Nihon Electronics. electron gun (Cold-FEG) developed by Nippon Electronics and a new high-order spherical aberration corrector (ASCOR). Fourier transform infrared (FTIR) spectra were measured by a Nicolet iS50 model instrument. Solid-state UV diffuse reflectance spectra were measured by a PE Lambda950 instrument. Fluorescence quantum yield, fluorescence quantum lifetime and fluorescence spectral data were tested using an Edinburgh FLS980. The specific surface area of the materials was tested using a fully automated physisorption instrument ASAP 2020 Plus HD88. XPS spectra were measured using an ESCALAB Xi+. X-ray photoelectron spectrometer from ThermoFisher Scientific, USA. The target was a double-anode Al/Mg target with a power of 400 W. The transient photocurrent response and Mott Schottky Test were measured using a Shanghai Chenhua CHI760E instrument. The photocatalytic materials were fired by Shenzhen Xinbao Instrument and Equipment Company Limited (XEXS5) and Henan Sante Furnace Technology Co.

2. General Reagent Information

Unless otherwise noted, all chemicals used in the preparations of starting materials and in the nickel catalyzed electroreductive carbonylative cross-coupling reactions were commercially available and were used as received without further purifications or prepared according to previous work. Solvents transferred to the glove box without exposure to air.

3. General Procedure for Photocatalytic Carbon-Carbon Coupling Reaction

An oven dried vial (19 x 80 mm) equipped with a stir bar was charged with the LCFOCN-2 (15 mg), p-Methoxyiodobenzene (0.45mmol, 1.5equiv.), the ligand (30 μ mol, 10 mol%). Subsequently, the Ni^{II} catalyst (15 μ mol, 5 mol%), the solvent (anhydrous, 3.8 mL), benzyl chloride (0.3mmol, 1equiv.) and the base (1-5 equiv.) were added in the glove box. Close and tighten the lid with the silicone gasket and remove the reaction flask from the glove box. The reaction mixture was sonicated for 5-10 min followed by stirring for 5 min until a fine dispersion of the solids was achieved. The mixtures were subjected to 20 W blue light irradiation in a photoreactor at 25 °C with rapid stirring (500 rpm). After the respective reaction time, one equivalent of naphthalene C₁₀H₈ (0.3 mmol) was added as an internal standard. Gas chromatography analysis was used to determine yield.

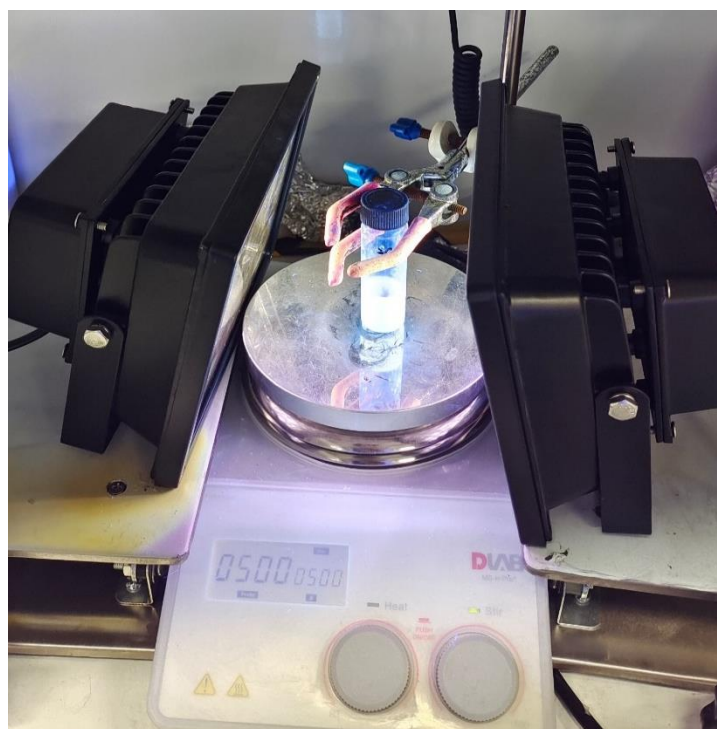


Figure S1 Photocatalytic reactor

4. Preparation of Photocatalysts

Cobalt nitrate hexahydrate (AR), lanthanum nitrate hexahydrate (AR), ferric nitrate hexahydrate (AR), citric acid (AR), urea (AR), anhydrous methanol (AR), and ethanol (AR) were obtained from Beijing Haoke Technology Co., Ltd. All other reagents used in the synthesis were of analytical grade and used without further purification. Deionized water, with a resistivity of 18 M Ω cm, was used throughout the experiments.

In the typical synthesis, for the preparation of pristine g-C₃N₄, a certain amount of urea is placed in a crucible and dried at 80 °C in a blast-drying oven for 10 h. Then it was heated in muffle furnace at the temperature of 550°C for 1.7 h with the heating rate of 5°C/min. For the synthesis of LaCoO₃, calculate the required mass of the reactants in strict accordance with a ratio of cobalt nitrate, lanthanum nitrate and citric acid at 1 : 1 : 2. The above three reactants were weighed and dissolved in 10 mL of secondary water to obtain a purple-red solution. Transfer the solution from the previous step to a round-bottom flask, heat it in a water bath at 80 °C, and stir it magnetically until the solution becomes a purple sol. Pour the sol obtained in the previous step into a clean ceramic crucible and dry it at 120°C in a blast drying box to obtain a swollen purple dry gel, which is ground for later use. Then, the obtained solid was calcined in a muffle furnace at 400°C for 4 hours and then at 700°C for 4 hours. After the machine was naturally cooled, black LaCoO₃ powder was collected. For the synthesis of LaFeO₃, calculate the mass of the required reactants strictly in the ratio 1 : 1 : 2 of ferric nitrate, lanthanum nitrate and citric acid. The above three reactants were weighed and dissolved in 10 ml of secondary water to obtain a pale yellow solution. The solution obtained in the previous step was transferred to a round bottom flask and heated in a water bath at 80 °C with magnetic stirring until the solution turned into an orange sol. The sol obtained in the previous step was poured into a clean ceramic crucible and dried in a blast oven at 120 °C to obtain red-orange color dry gel, which was ground and set aside. The solid obtained was then calcined in a muffle furnace at 400°C for 4 hours and then at 700°C for 4 hours. The brownish LaFeO₃ powder was collected after the machine cooled naturally.

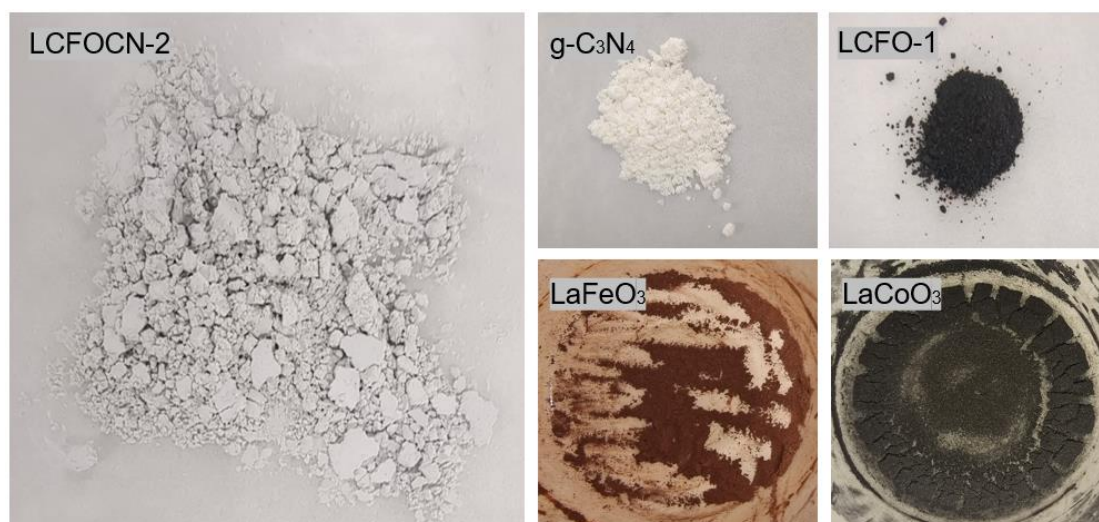


Figure S2 Freshly synthesized photocatalyst.

The synthesis of a series of LCFO photocatalysts was carried out in a similar way to the two substances mentioned above, by adjusting the amounts of cobalt nitrate and iron nitrate, to modulate the elemental cobalt and elemental iron in the product. The obtained catalysts $\text{LaCo}_x\text{Fe}_{1-x}\text{O}_3$ ($x = 0.9, 0.8, 0.6, 0.4, \text{ and } 0.2$) were denoted as LCFO-1, LCFO-2, LCFO-3, LCFO-4, LCFO-5. A series of LCFOCN were prepared by the solvothermal method. A certain weight of LCFO and of $\text{g-C}_3\text{N}_4$ were dispersed in 10 mL anhydrous ethanol separately and sonicated for half an hour. The suspension of LCFO was slowly added to the suspension of $\text{g-C}_3\text{N}_4$. At room temperature, the mixed solution was sonicated for half an hour. During the process of ultrasonication, the system temperature was kept normal. The mixture was stirred at room temperature for 10 h and transferred to a clean hydrothermal kettle. The hydrothermal kettle was heated at 120 °C for 8 h. Then, the mixture was centrifuged for 15 min and dried at 80 °C for 10 h. LCFO@ $\text{g-C}_3\text{N}_4$ nanoflakes with 2, 5, 10, 15, and 20 wt % LCFO contents were prepared and denoted as LCFOCN-1, LCFOCN-2, LCFOCN-3, LCFOCN-4, LCFOCN-5.



Figure S3 Synthesis processes of a series of LCFOCNs photocatalysts.

5. Characterisation of photocatalysts

To develop a cost-effective, operationally simple and efficient photocatalyst for organic coupling reactions, we rationally designed and synthesized a series of composite materials. LaCoO_3 was prepared via a sol-gel method, yielding uniform spherical particles, as observed in the SEM images (Figure S4a and S4b). Likewise, LaFeO_3 was synthesized using the same route and exhibited a plate-like morphology with a relatively flat surface decorated with small protuberances (Figure S4c and S4d), making it a promising substrate candidate. A series of $\text{LaCoO}_3@ \text{LaFeO}_3$ (LCFO) composites were subsequently fabricated through a combination of sol-gel processing and muffle furnace calcination, with representative morphologies shown in Figures S4e and S4f.

Graphitic carbon nitride exhibited a lamellar two-dimensional structure with smooth, rounded edges (Figure S4g and S4h). To integrate LCFO components onto $\text{g-C}_3\text{N}_4$ in tunable proportions, a multi-step process involving ultrasonication, mechanical mixing, and solvothermal treatment was employed. This approach yielded LaCoO_3 -loaded LCCN and LaFeO_3 -loaded LFCN hybrids, as well as a series of LCFOCN- x composites ($x = 1-5$), each differing in loading content of LCFO (see Figure S3).

Notably, SEM analysis of LCFOCN-2 revealed a distinct two-dimensional sheet-like morphology

with thinner and sharper edges relative to pristine g-C₃N₄, and no visible aggregation of LCFO particles was detected (Figure S4i and S4j). These observations suggest that in LCFOCN-2, LaCoO₃ and LaFeO₃ nanoparticles are likely dispersed as individual grains anchored on the g-C₃N₄ substrate.

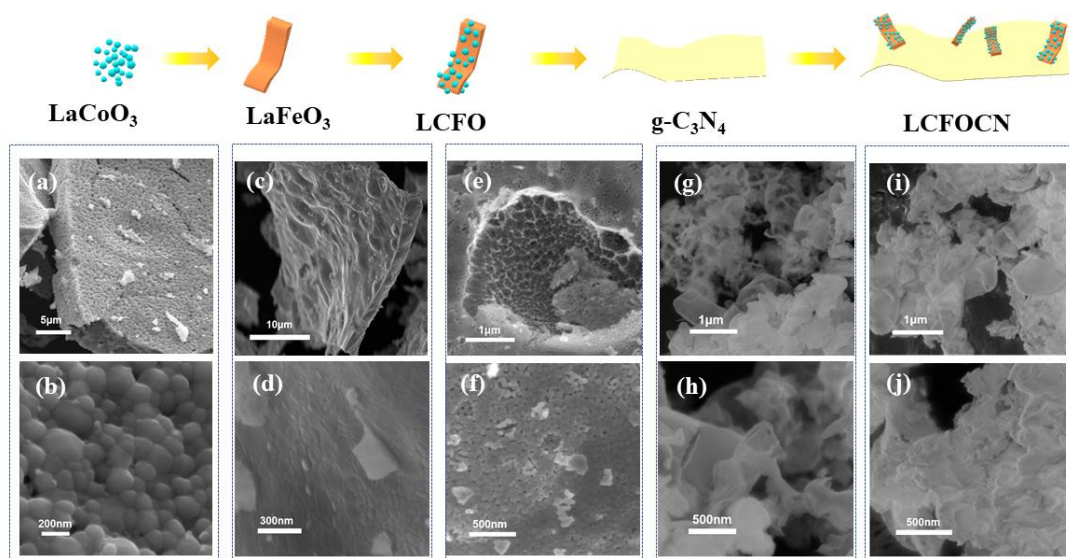


Figure S4. SEM characterization of the catalyst (a, b) LaCoO₃, (c, d) LaFeO₃, (e, f) LCFO, (g, h) g-C₃N₄, (i, j) LCFOCN-2, (k, l) Representative TEM image and (m) EDS elemental mapping of LCFOCN-2, (n) C1s and (o) N1s XPS spectra of g-C₃N₄ and LCFOCN-2.

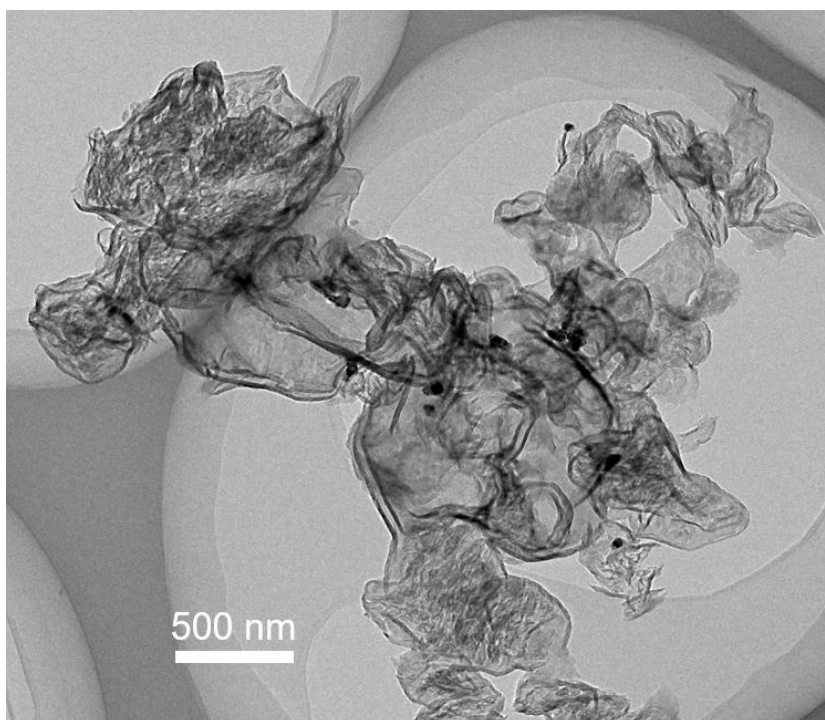


Figure S5 Representative TEM image of LCFOCN-2.

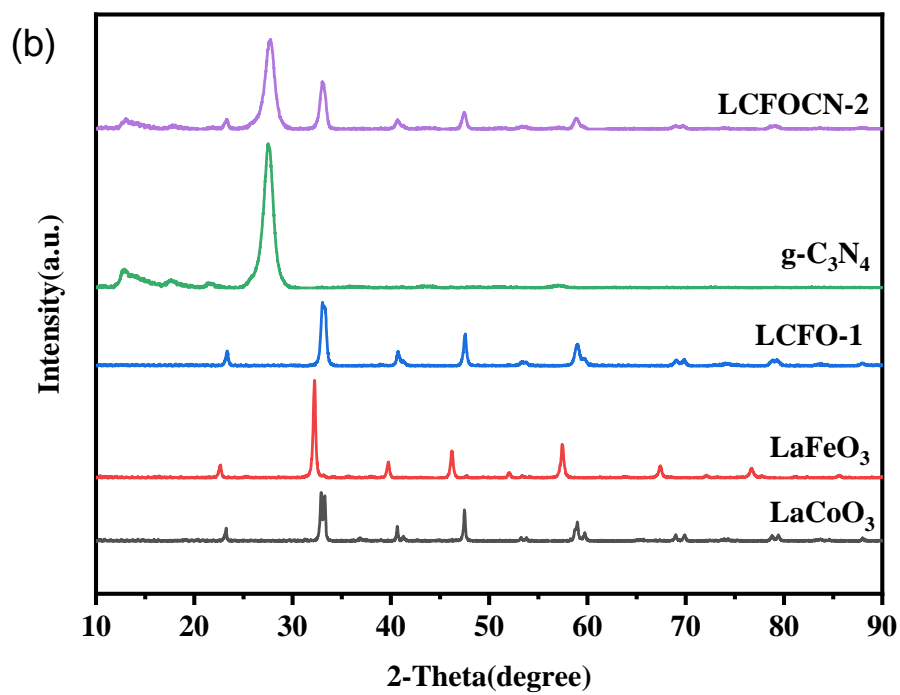
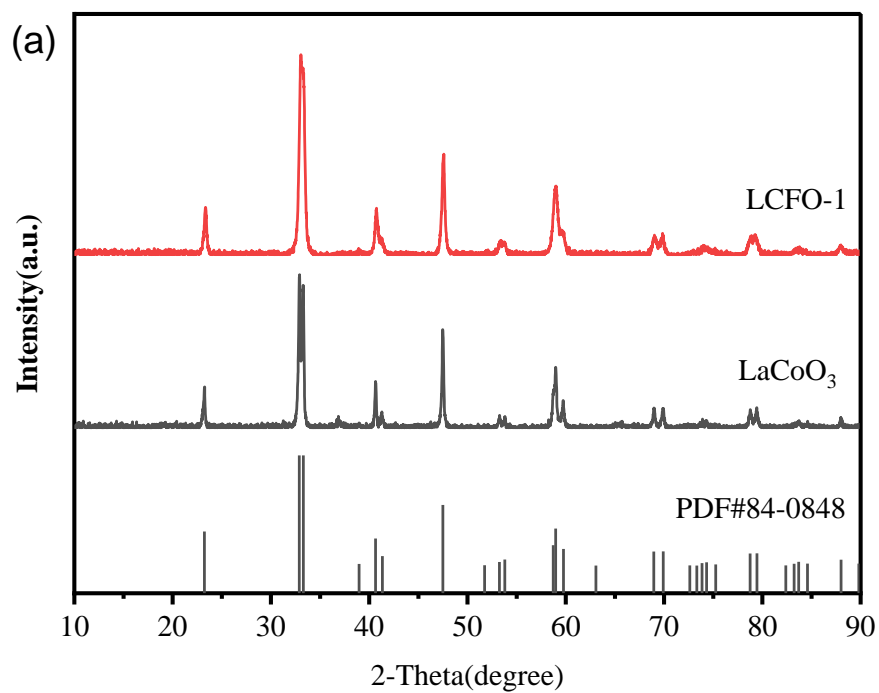


Figure S6 (a) XRD of LaCoO₃ and LCFO-1. (b) XRD of g-C₃N₄, LaCoO₃, LaFeO₃, LCFO-1 and

LCFOCN-2.

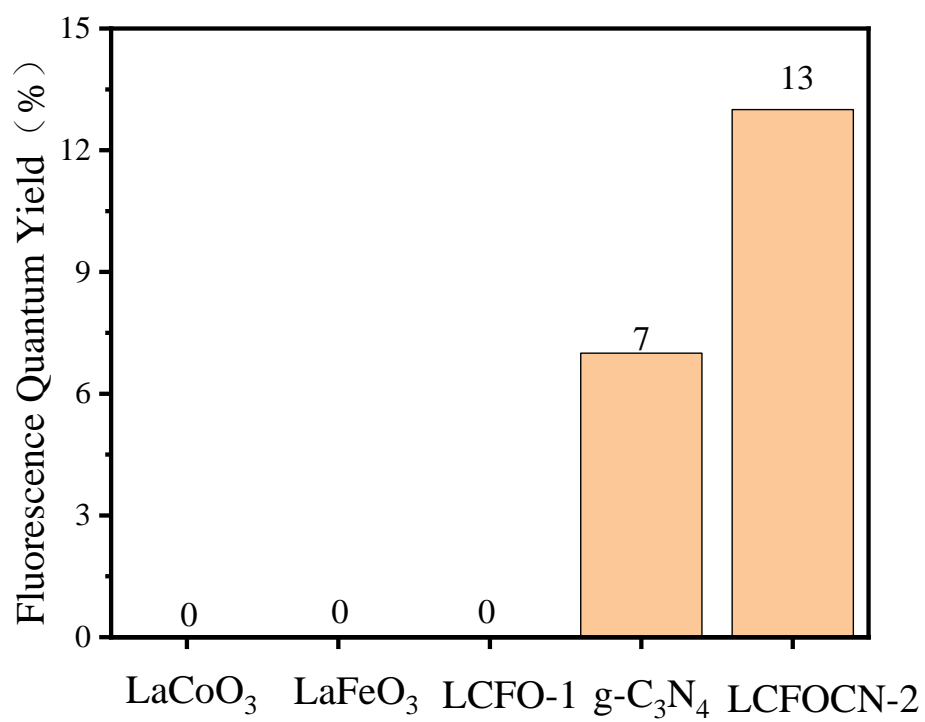


Figure S7 Fluorescence quantum yield of the g-C₃N₄, LaCoO₃, LaFeO₃, LCFO-1 and LCFOCN-2.

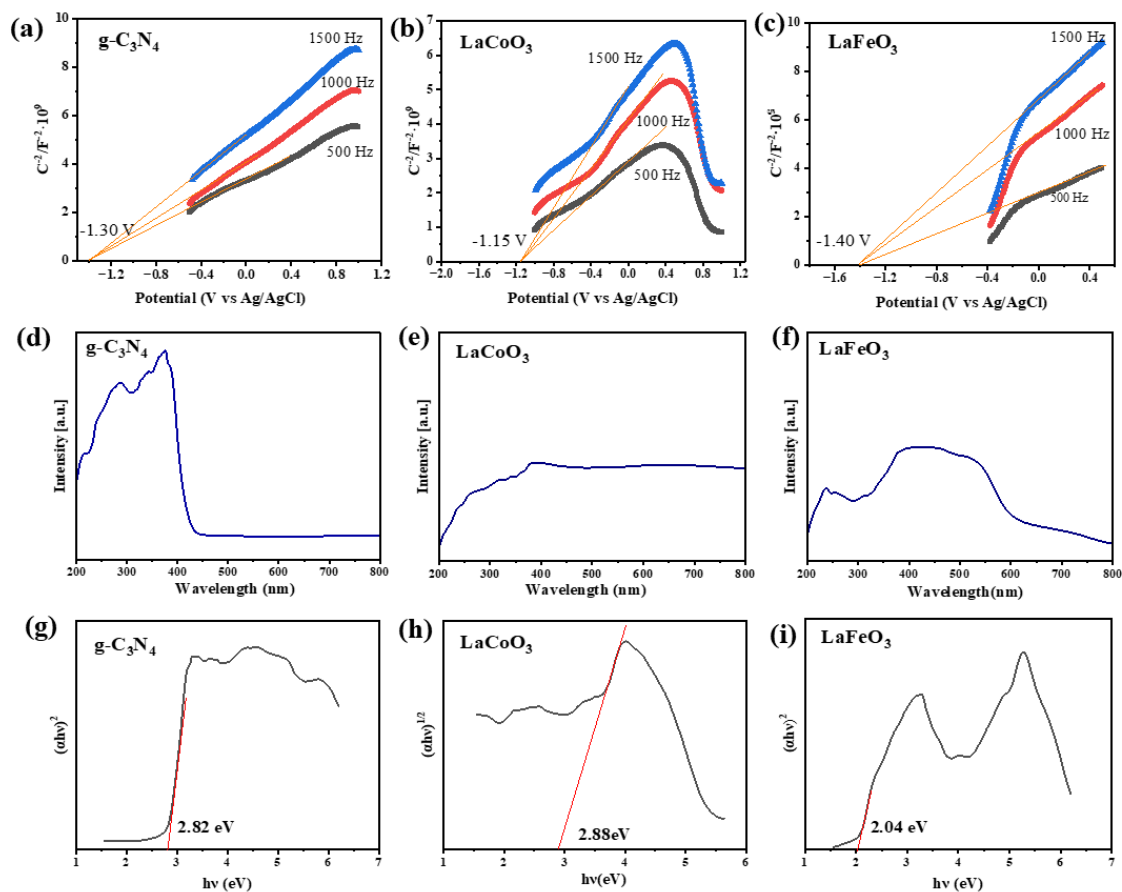


Figure S8. Mott-Schottky test of the catalyst (a) g-C₃N₄, (b) LaCoO₃, (c) LaFeO₃, UV-vis DRS of the catalyst (d) g-C₃N₄ (e) LaCoO₃, (f) LaFeO₃; plots of band gap energies (E_g) for (g) g-C₃N₄, (h) LaCoO₃, (i) LaFeO₃

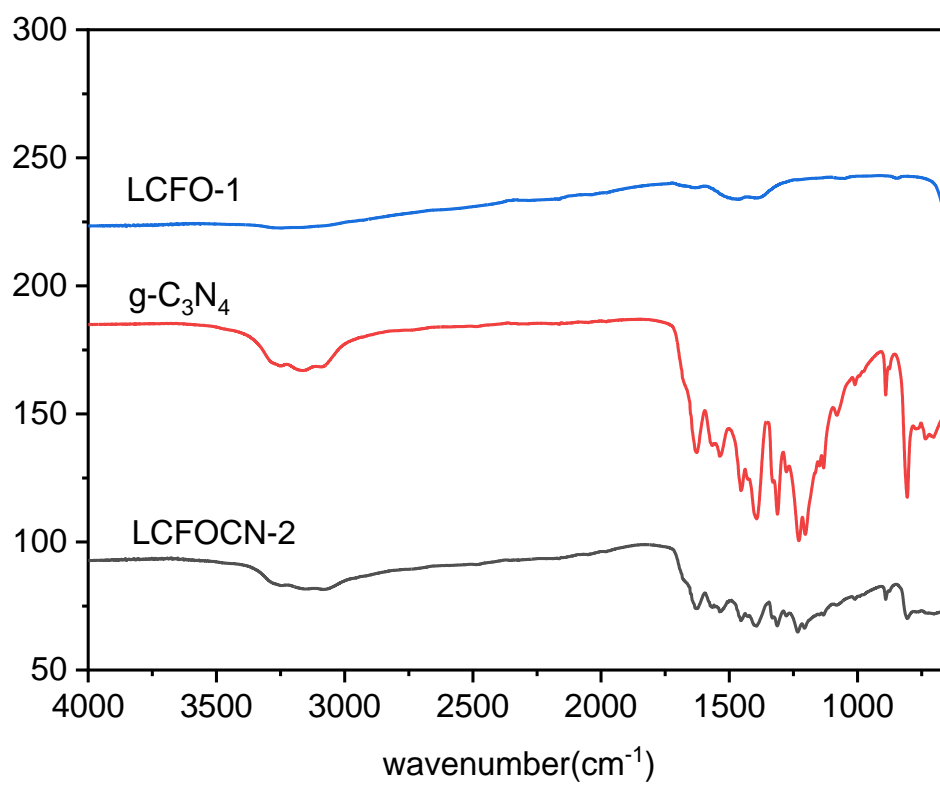
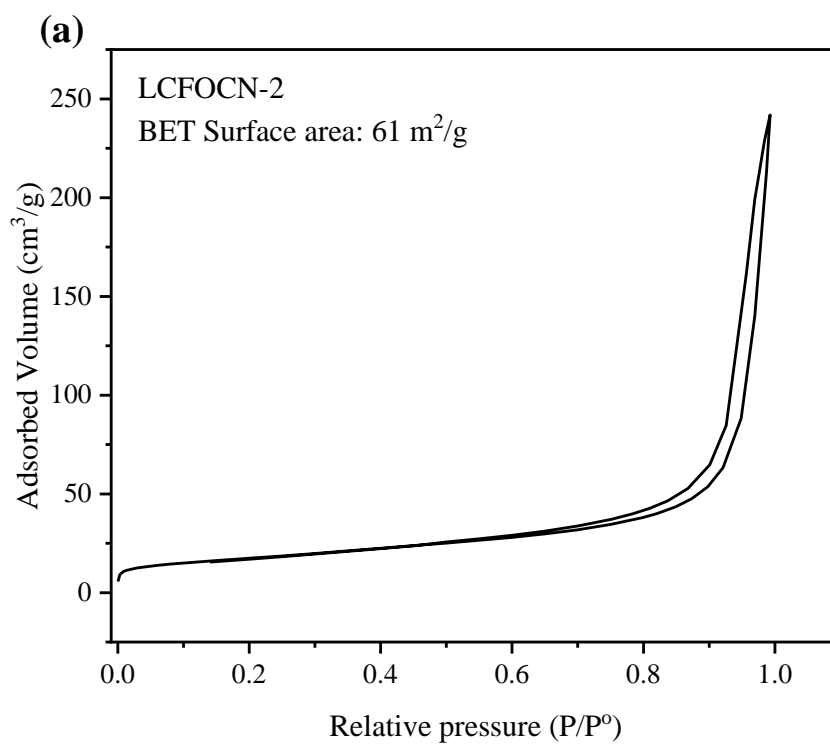
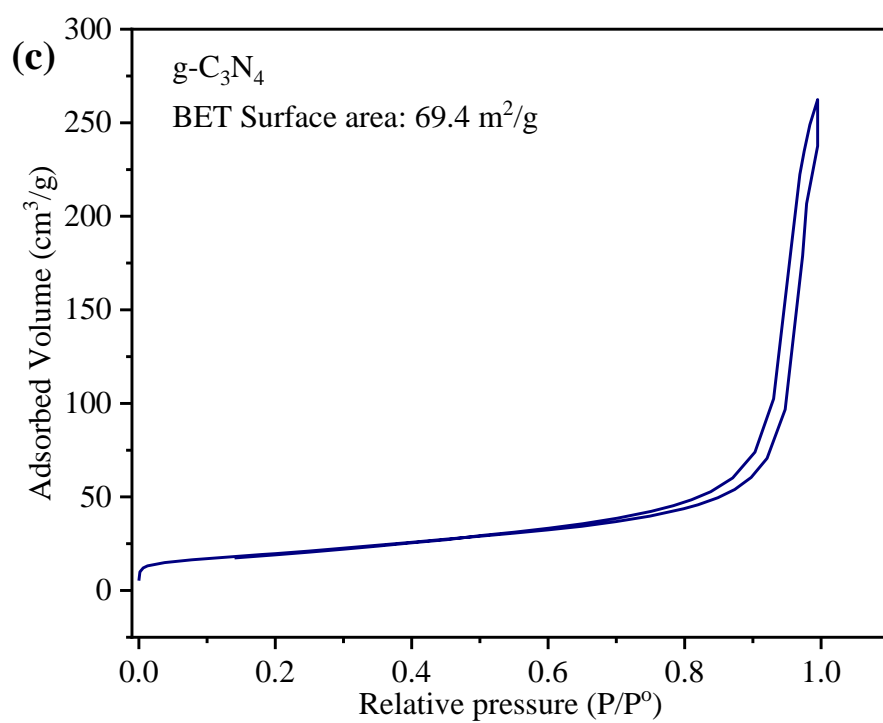
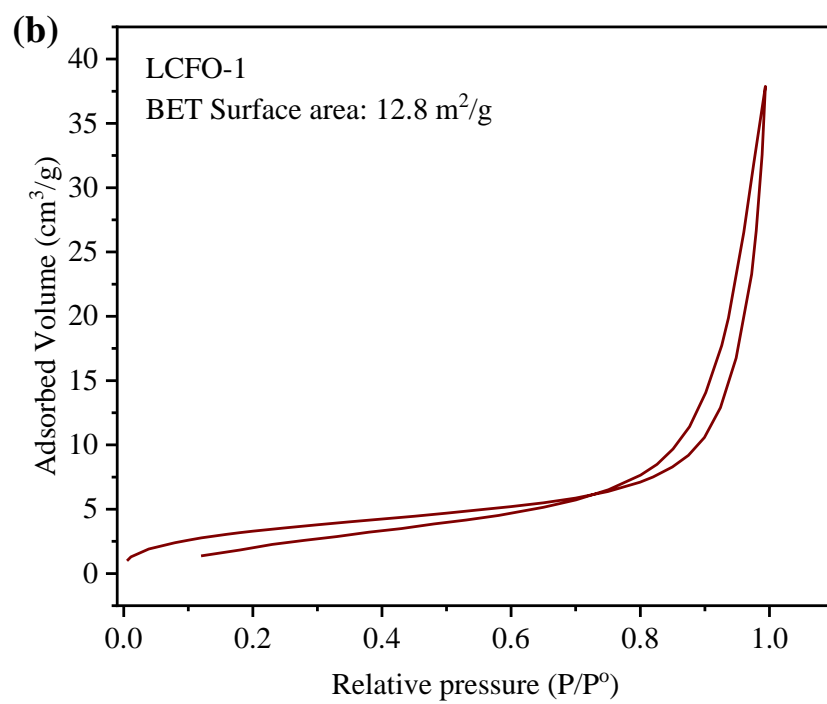
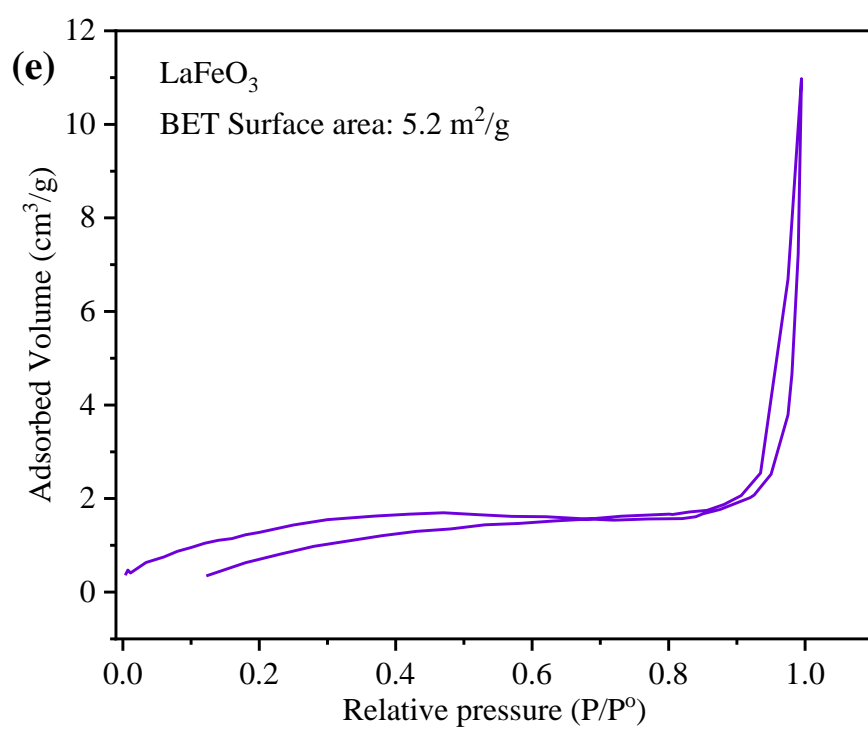
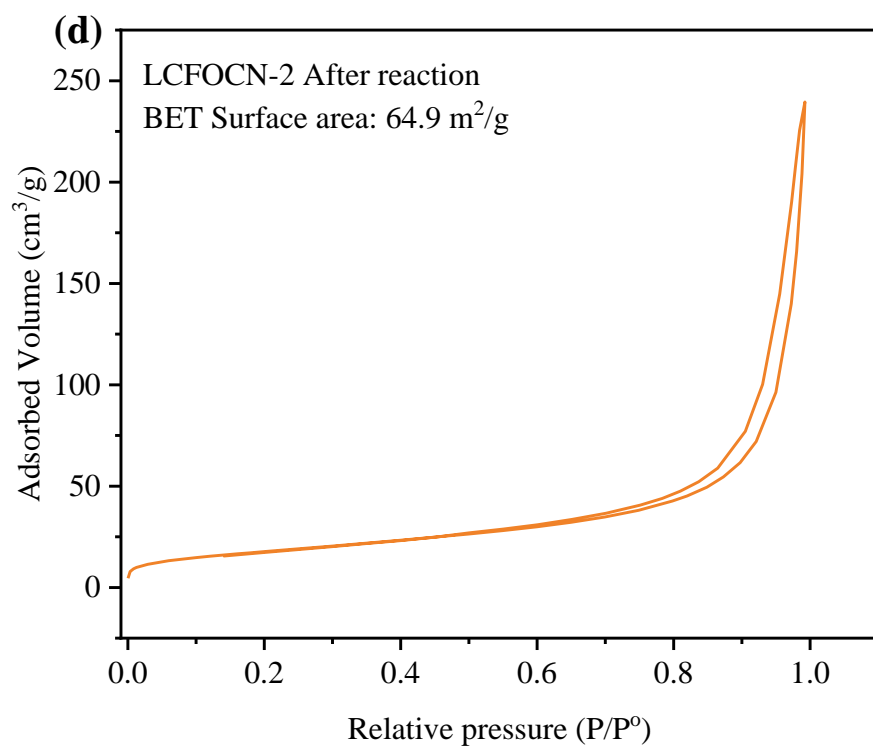


Figure S9 FTIR spectra of LCFO-1, g-C₃N₄, LCFOCN-2.







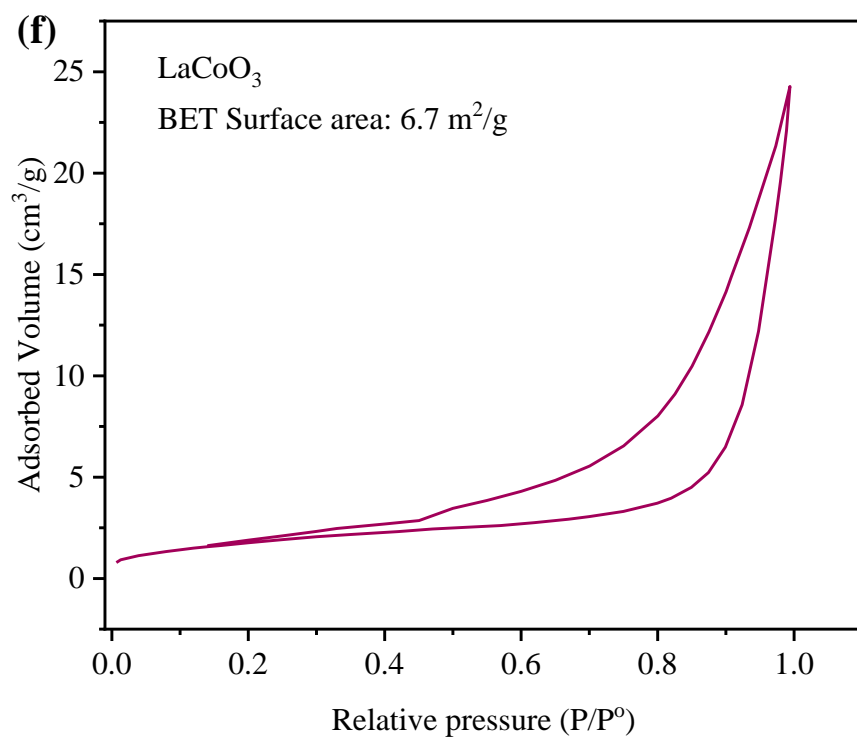
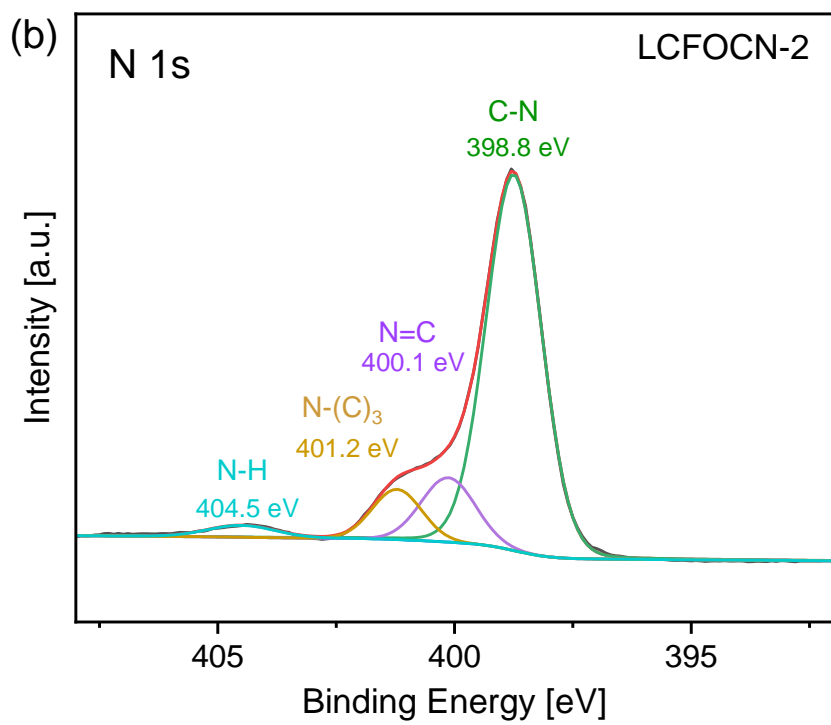
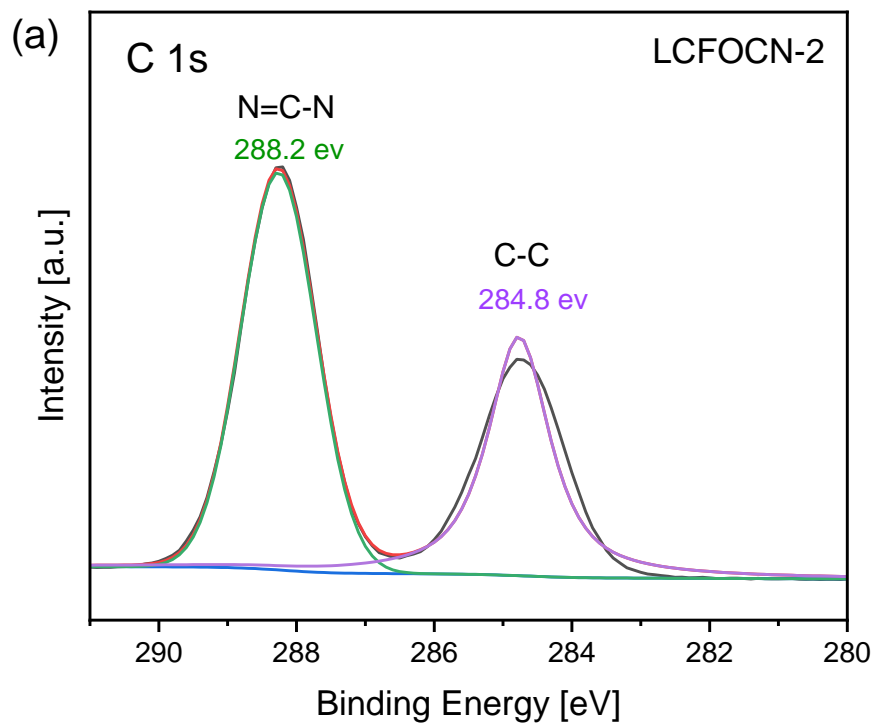


Figure S10 Nitrogen adsorption-desorption isotherms and corresponding BET surface area of (a)LCFOCN-2 before reaction, (b)LCFO-1, (c)g-C₃N₄, (d)LCFOCN-2 after reaction (e)LaFeO₃ and (f)LaCoO₃.



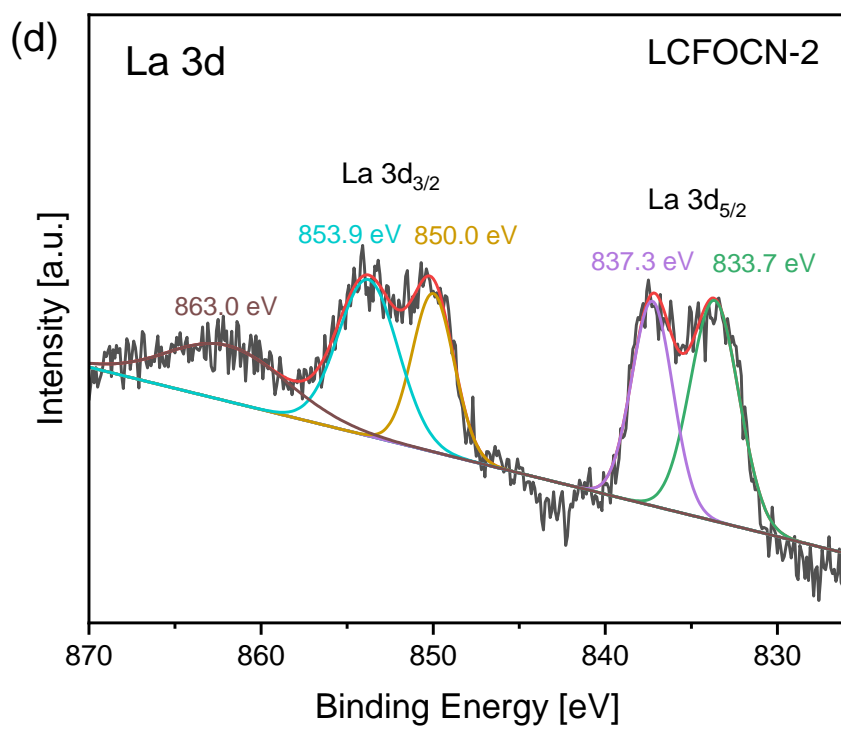
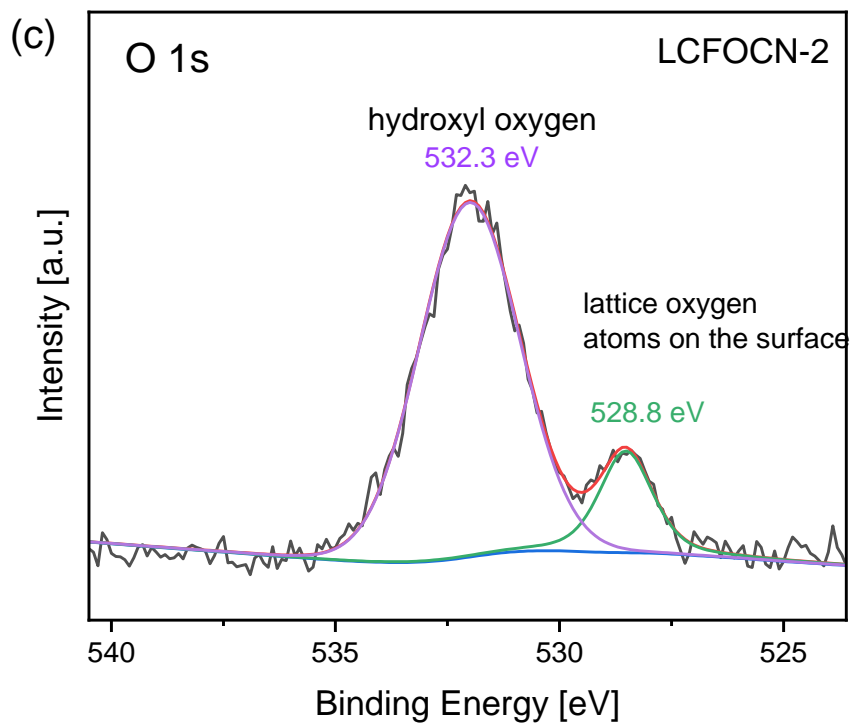
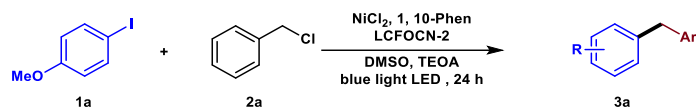


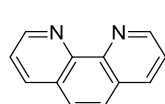
Figure S11 XPS spectra of the LCFOCN-2 of (a)C 1s, (b)N 1s, (c)O 1s, (d)La 3d.

6. Reaction Optimization

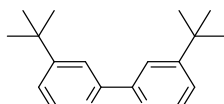


Entry	Deviation from standard conditions	Yield 3a (%)
1	None	98 (92)
2	LCFOCN-1 instead of LCFOCN-2	46
3	LCFOCN-3 instead of LCFOCN-2	53
4	LCFOCN-4 instead of LCFOCN-2	42
5	LCFOCN-5 instead of LCFOCN-2	30
6	g-C ₃ N ₄ instead of LCFOCN-2	22
7	4-CzIPN instead of LCFOCN-2	35
8	LCFO-1,2,3,4,5 instead of LCFOCN-2	Trace
9	no light	0
10	no photocatalyst	0
11	no NiCl_2 and no ligand	0
12	no ligand	0
13	no TEOA	0
14	DMF instead of DMSO	47
15	NMP instead of DMSO	42
16	$\text{Ni}(\text{acac})_2$ instead of NiCl_2	33
17	NiBr_2 instead of NiCl_2	48
18	NiF_2 instead of NiCl_2	32
19	NiI_2 instead of NiCl_2	10
20	bpy instead of 1,10-phen	68

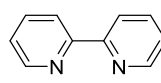
21	dtbpy instead of 1,10-phen	59
22	dmbpy instead of 1,10-phen	26
23	1.8ml DMSO	56
24	2.8ml DMSO	71
25	4.8ml DMSO	79



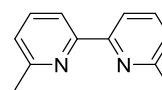
1,10-phen



dtbpy



bpy

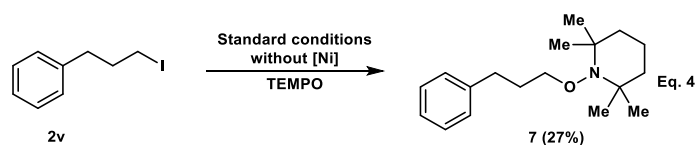
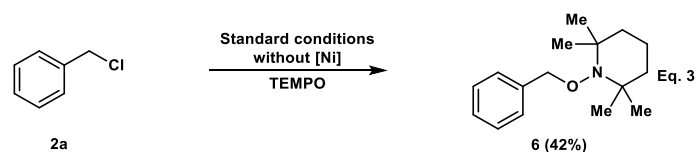
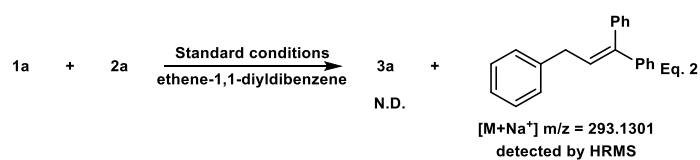


dmbpy

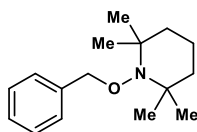
^a Reaction condition: 1a(0.3 mmol), 2a(0.3 mmol), NiCl₂(5 mol%), 1-10 phen(5 mol%), DMSO (3.8 mL), triethanolamine(TEOA 5eq) 395 nm LED light irradiation, 24 h. Yield was measured by GC using naphthalene as an internal standard.

7. Mechanistic Experiments

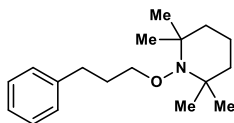
7.1 Radical trapping experiments



General procedure A was followed, but additionally, TEMPO (3 equivalent) was added along with the solvent. 5, 6, 7 was detected by GC-MS. However, careful analysis of the crude reaction mixture revealed the formation of a small amount (approximately 46%) of 1-(benzyloxy)-2,2,6,6-tetramethyl piperidine, (approximately 27%) of 3-phenyl-1-(2',2',6',6'-tetramethyl-1'-piperidinyloxy)-propane. The addition of ethene-1,1-diyl dibenzene to the standard reaction of 1a with 2a was also able to capture the benzyl radical. The obtained products were able to be detected by high-resolution mass spectrometry

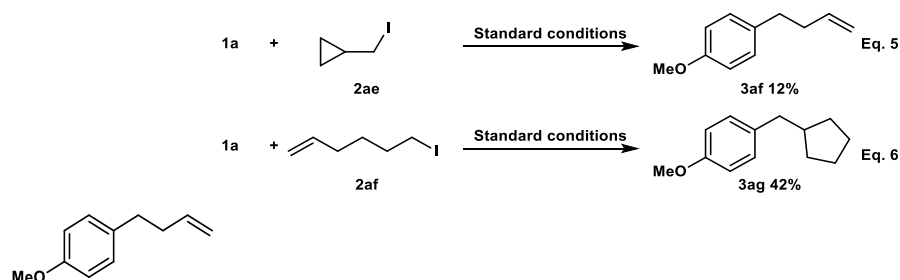


Yellow liquid. ^1H NMR (400 MHz, Chloroform- d) δ 7.42 – 7.27 (m, 5H), 4.83 (s, 2H), 1.61 – 1.47 (m, 6H), 1.27 (s, 6H), 1.16 (s, 6H). The spectroscopic characterization of the Bn-TEMPO adduct matched with data reported in the literature¹.

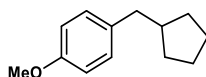


Yellow liquid. ^1H NMR (400 MHz, Chloroform- d): δ 7.20-7.05 (m, 5H), 3.70 (t, J = 6 Hz, 2H), 2.63 (t, J = 8 Hz, 2H), 1.82-1.72 (m, 2H), 1.53-1.20 (m, 6H), 1.06 (s, 6H), 1.04 (s, 6H). The spectroscopic characterization of the Bn-TEMPO adduct matched with data reported in the literature².

7.2 Radical clock experiment



^1H NMR (400 MHz, Chloroform- d) δ 7.15 – 7.05 (d, J = 8.4 Hz, 2H), 6.83 (d, J = 8.4 Hz, 2H), 5.87 (ddt, J = 17.2, 10.4, 6.7 Hz, 1H), 5.17 – 4.88 (m, 2H), 3.79 (s, 3H), 2.83 – 2.55 (m, 2H), 2.44 – 2.18 (m, 2H). The spectroscopic characterization matched with data reported in the literature³.



^1H NMR (400 MHz, Chloroform- d) δ 7.01 (dd, J = 9.0, 2.6 Hz, 2H), 6.81 – 6.69 (m, 2H), 3.71 (s, 3H), 2.47 (d, J = 7.4 Hz, 2H), 2.03 – 1.91 (m, 1H), 1.70 – 1.50 (m, 4H), 1.55 – 1.39 (m, 2H), 1.20-1.01 (m, 2H). The spectroscopic characterization matched with data reported in the literature⁴.

7.3. Light On-off experiment

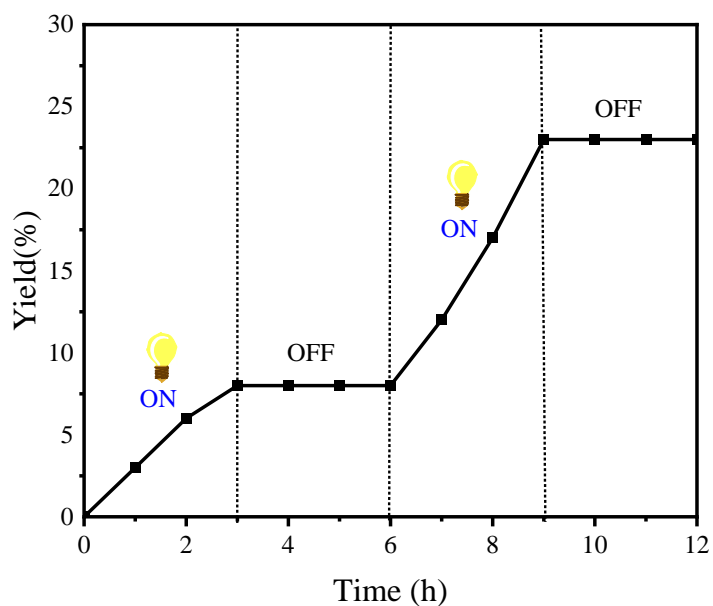


Figure S12 Time-dependent yield of the model reaction under alternating light ON/OFF conditions.

To demonstrate the pivotal role of light irradiation in modulating the reaction outcome, a time-resolved yield profile was recorded under alternating light ON/OFF conditions. As illustrated in Figure X, the yield increased steadily during the initial illumination period (0–3 h), reaching approximately 8%. Upon switching off the light (3–6 h), the reaction plateaued, indicating negligible progress in the absence of light. When the light was reintroduced at 6 h, the yield resumed a sharp increase, reaching ~23% by 9 h. A subsequent light-off period (9–12 h) again resulted in stagnation of the yield.

This clearly indicates that the reaction is highly dependent on light activation, exhibiting a "photogated" behavior where the reaction is effectively "switched on" only in the presence of light. Such temporal control underscores the potential for precise external regulation of reaction kinetics in photocatalytic systems, enabling on-demand modulation of product formation.

8. Catalyst Recycling

An oven dried glass vial (18 x 66mm) equipped with a stir bar was charged with the LCFOCN-2 (15 mg) as photocatalysts, p-Methoxyiodobenzene (0.45mmol, 1.5equiv.), the ligand (30 μ mol, 10 mol%). Subsequently, the Ni^{II} catalyst (15 μ mol, 5 mol%), the solvent (anhydrous, 3.8 mL), benzyl chloride (0.3mmol, 1equiv.) and the base (5 equiv.) were added in the glove box. Close and tighten the lid with the silicone gasket and remove the reaction flask from the glove box. The reaction mixture was sonicated for 5-10 min followed by stirring for 5 min until a fine dispersion of the solids was achieved. The mixture was irradiated in the photoreactor at room temperature with rapid stirring (500 rpm). After the respective 24 hours, one equivalent of naphthalene C₁₀H₈ (0.3 mmol) was added as an internal standard. Gas chromatography analysis was used to determine yield. The reaction solution was centrifuged and the non-homogeneous photocatalyst precipitated at the bottom of the centrifuge tube, the solid from the centrifugation process was washed three times with ethyl acetate, then centrifuged and dried. The solid obtained was put into the next cycle of the reaction as photocatalysts. The products obtained in each case were quantified using gas chromatography plus naphthalene as an internal standard. Repeat the process five times to obtain the cyclic test results.

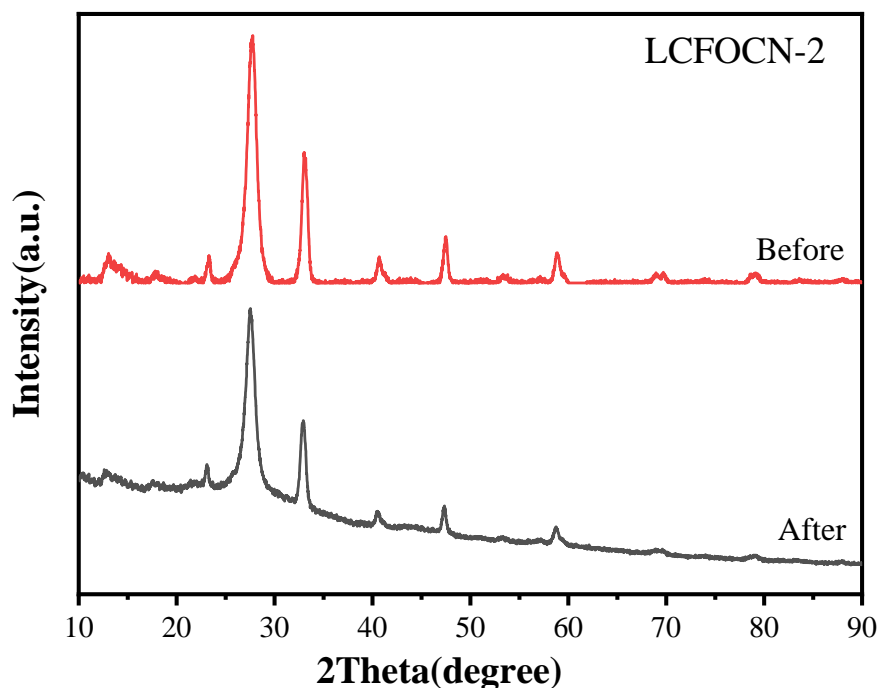


Figure S13 XRD patterns of LCFOCN-2 before and after the reaction cycles.

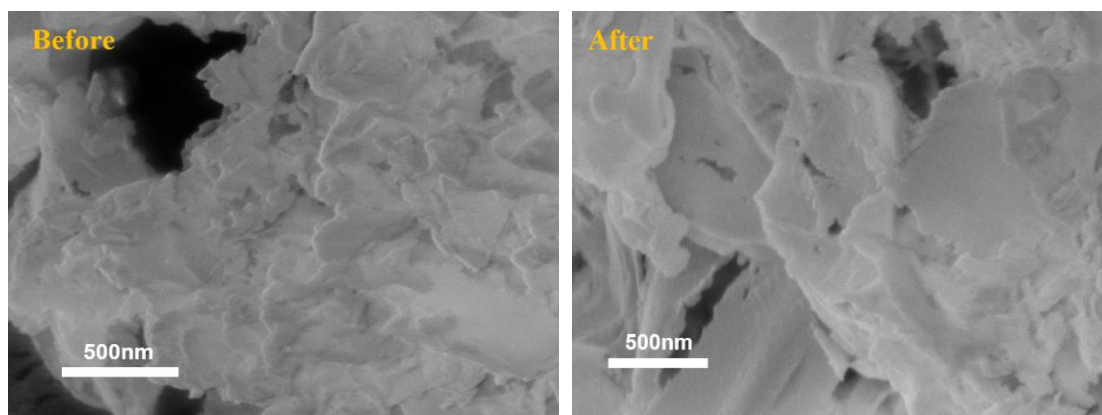


Figure S14 SEM images of LCFOCN-2 before and after catalytic cycling.

9. Large Scale Reaction Procedure

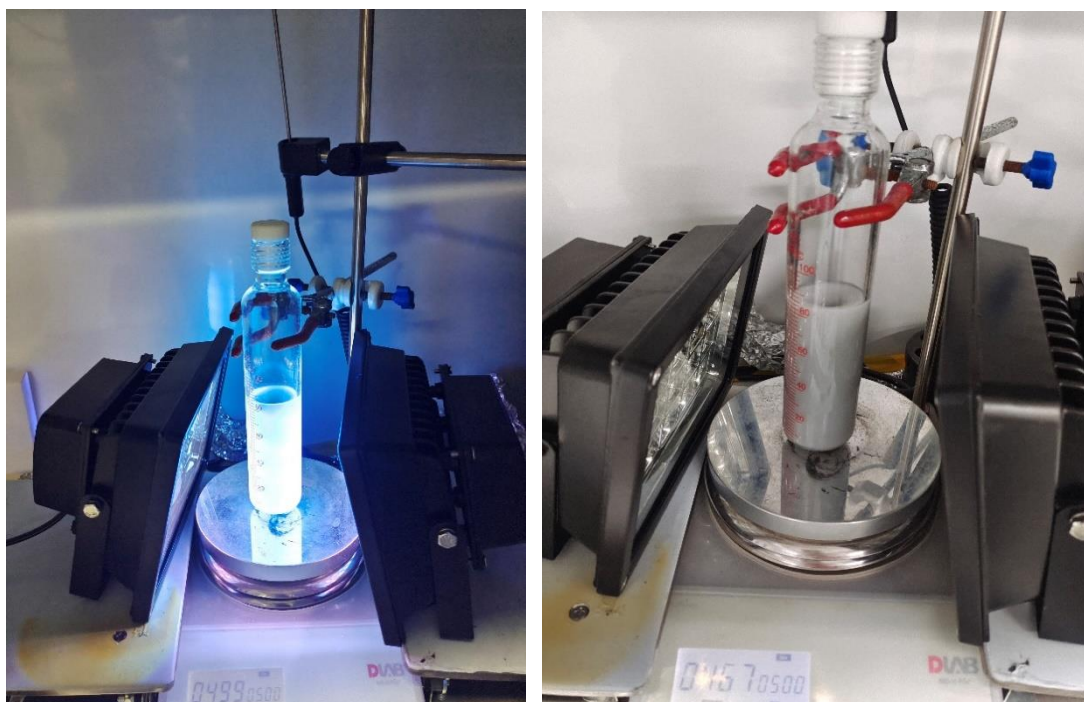
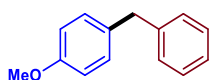


Figure S15 Large Scale Reactor.

LCFOCN-2 (300 mg), 1,10-phen (5 mol%), p-methoxyiodobenzene (6 mmol), NiCl_2 (10 mol%) were sequentially added to a 100 mL custom-made long round-bottomed screw-necked flask

equipped with a Teflon-coated magnetic stirring bar and a Teflon cap, followed by 80 mL of DMSO in a three-necked flask, TEOA (5eq) benzyl chloride (6 mmol). All steps were performed in a glove box. It was then removed from the glove box. The reaction mixture was stirred and the photocatalytic reaction was irradiated with 20w 395nm LED lamps from both sides of the reaction flask for 24 h at room temperature. After the reaction, DMSO was washed with water (4 X80 mL). The product was extracted from the crude reaction mixture with ethyl acetate (4 X80 mL). The organic layers were combined and washed with brine (160 mL). Dry over Na₂SO₄, filter and concentrate under reduced pressure. The product was purified by silica gel column chromatography using petroleum ether /EA = 80/1 (v/v) as eluent. The final product was obtained in 70% yield.

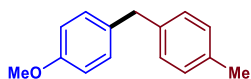
10. Analytical Data of Substrates and Products



1-benzyl-4-methoxybenzene 3a⁵ Colorless liquid (54.6 mg, 92% yield). The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.33 (ddt, *J* = 9.3, 7.0, 3.6 Hz, 2H), 7.23 (dt, *J* = 7.9, 2.6 Hz, 3H), 7.16 (dd, *J* = 8.7, 3.4 Hz, 2H), 7.03 – 6.74 (m, 2H), 3.98 (s, 2H), 3.81 (s, 3H).

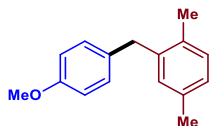
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.34, 140.97, 132.62, 129.25, 128.20, 127.82, 125.36, 113.24, 54.59, 40.40.



1-methoxy-4-(4-methylbenzyl)benzene 3b⁶ Yellow liquid (56.0 mg, 88% yield). The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (600 MHz, Chloroform-*d*) δ 7.18 – 6.97 (m, 6H), 6.87 – 6.78 (m, 2H), 3.90 (s, 2H), 3.79 (s, 3H), 2.32 (s, 3H).

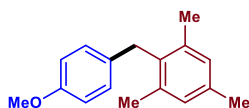
¹³C NMR (151 MHz, Chloroform-*d*) δ 157.94, 138.56, 135.46, 133.59, 129.81, 129.14, 128.71, 113.88, 55.27, 40.62, 21.01.



2-(4-methoxybenzyl)-1,4-dimethylbenzene 3c⁷ Yellow liquid (39.3 mg, 58% yield). The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.05 (dd, J = 8.6, 2.7 Hz, 3H), 6.94 (d, J = 21.3 Hz, 2H), 6.82 (d, J = 8.6 Hz, 2H), 3.89 (s, 2H), 3.79 (s, 3H), 2.29 (s, 3H), 2.20 (s, 3H).

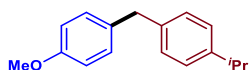
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.71, 139.08, 135.32, 133.34, 132.51, 130.55, 130.12, 129.60, 126.94, 113.71, 55.21, 38.47, 20.98, 19.16.



2-(4-methoxybenzyl)-1,3,5-trimethylbenzene 3d⁸ Pale yellow needle-like crystals (29.5 mg, 41% yield). The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 6.95 – 6.86 (m, 4H), 6.80 – 6.74 (m, 2H), 3.95 (s, 2H), 3.76 (s, 3H), 2.29 (s, 3H), 2.20 (s, 6H).

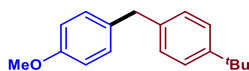
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.64, 136.98, 135.60, 134.17, 132.06, 128.89, 128.73, 113.76, 55.26, 33.79, 20.94, 20.14.



1-isopropyl-4-(4-methoxybenzyl)benzene 3e⁹ Yellow liquid (61.2 mg, 85% yield). The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.19 – 7.03 (m, 6H), 6.88 – 6.81 (m, 2H), 3.90 (s, 2H), 3.78 (s, 3H), 2.87 (p, J = 6.9 Hz, 1H), 1.23 (d, J = 7.0 Hz, 6H).

¹³C NMR (151 MHz, Chloroform-*d*) δ 157.92, 146.50, 138.92, 133.50, 129.87, 128.66, 126.47, 113.85, 55.26, 40.63, 33.69, 24.05.



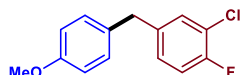
1-(tert-butyl)-4-(4-methoxybenzyl)benzene 3f¹⁰ White liquid (31.3 mg, 41%) The product was

purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*)

δ 7.32 – 7.29 (m, 2H), 7.17 – 7.07 (m, 4H), 6.87 – 6.80 (m, 2H), 3.90 (s, 2H), 3.79 (s, 3H), 1.31 (s, 9H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 157.94, 148.76, 138.56, 133.45, 129.90, 128.39, 125.34, 113.87, 55.27, 40.53, 34.37, 31.42.



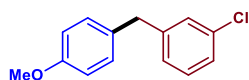
2-chloro-1-fluoro-4-(4-methoxybenzyl)benzene 3g Colorless liquid (47.3 mg, 63%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.13 (m, 1H), 7.12 – 7.05 (m, 2H), 7.05 – 6.92 (m, 2H), 6.88 – 6.81 (m, 2H), 3.86 (s, 2H), 3.79 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.27, 156.64 (d, *J* = 246.9 Hz), 138.65 (d, *J* = 4.0 Hz), 132.17, 130.70, 129.83, 128.33 (d, *J* = 7.0 Hz), 120.71 (d, *J* = 17.6 Hz), 116.39 (d, *J* = 20.9 Hz), 114.10, 55.29, 40.00.

¹⁹F NMR (377 MHz, Chloroform-*d*) δ -119.77.

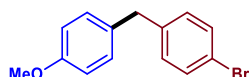
HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₁₄H₁₃ClFO⁺ 251.0634; Found 251.0474.



1-chloro-3-(4-methoxybenzyl)benzene 3h¹¹ Yellow liquid (40.4 mg, 58%). The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.16 – 7.05 (m, 3H), 7.00 (dd, *J* = 14.8, 7.7 Hz, 3H), 6.84 – 6.70 (m, 2H), 3.82 (s, 2H), 3.72 (s, 3H).

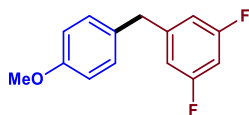
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.13, 142.61, 133.18, 131.26, 128.85, 128.62, 127.85, 125.94, 125.17, 112.97, 54.23, 39.64.



1-bromo-4-(4-methoxybenzyl)benzene 3i¹² Yellow liquid (40.6 mg, 49%). The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.32 (m, 2H), 7.13 – 6.98 (m, 4H), 6.88 – 6.79 (m, 2H), 3.87 (s, 2H), 3.79 (s, 3H).

¹³C NMR (151 MHz, Chloroform-*d*) δ 158.14, 140.60, 132.57, 131.49, 130.56, 129.83, 119.83, 114.00, 55.27, 40.42.



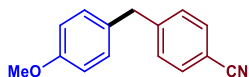
1,3-difluoro-5-(4-methoxybenzyl)benzene 3j¹³ Yellow liquid (54.8 mg, 78%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.13 – 7.04 (m, 2H), 6.89 – 6.81 (m, 2H), 6.73 – 6.55 (m, 3H), 3.89 (s, 2H), 3.80 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 164.32 (dd, *J* = 249.0, 13.1 Hz), 158.32, 145.58 (t, *J* = 9.1 Hz), 131.48, 129.91, 114.08, 111.70 – 111.28 (dd, *J* = 25.2, 6.1 Hz), 101.45 (t, *J* = 25.4 Hz), 55.26, 40.69 (t, *J* = 2.0 Hz).

¹⁹F NMR (377 MHz, Chloroform-*d*) δ -110.44.

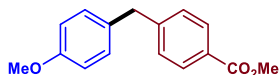
HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₁₄H₁₃OF₂⁺ 235.0929; Found 235.0921.



4-(4-methoxybenzyl)benzonitrile 3k¹² Yellow solid (44.2 mg, 66%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 40/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.3 Hz, 2H), 7.28 (s, 1H), 7.26 (d, *J* = 0.8 Hz, 1H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.97 (s, 2H), 3.79 (s, 3H).

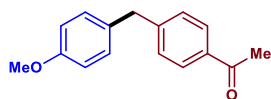
¹³C NMR (101 MHz, Chloroform-*d*) δ 158.34, 147.28, 132.32, 131.41, 129.98, 129.54, 119.11, 114.15, 109.90, 55.30, 41.11.



methyl 4-(4-methoxybenzyl)benzoate 3l¹⁴ Yellow liquid (40.0 mg, 52%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 50/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 3.96 (s, 2H), 3.89 (s, 3H), 3.78 (s, 3H).

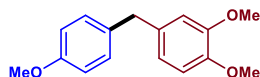
¹³C NMR (101 MHz, Chloroform-*d*) δ 167.14, 158.16, 147.07, 132.24, 129.95, 129.84, 128.86, 127.98, 114.01, 55.28, 52.07, 41.05.



1-(4-(4-methoxybenzyl)phenyl)ethan-1-one 3m¹⁵ Yellow oil The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.80 – 6.71 (m, 2H), 3.89 (s, 2H), 3.70 (s, 3H), 2.49 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 197.83, 158.21, 147.34, 135.20, 132.15, 129.92, 129.00, 128.65, 114.06, 55.28, 41.03, 26.59.

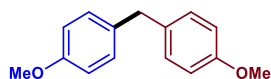


1,2-dimethoxy-4-(4-methoxybenzyl)benzene 3n¹⁶ Yellow liquid (55.0 mg, 71%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.10 (d, *J* = 8.5 Hz, 2H), 6.85 – 6.81 (m, 2H), 6.79 (d, *J* = 8.1 Hz, 1H), 6.73 – 6.68 (m, 2H), 3.87 (s, 2H), 3.85 (s, 3H), 3.83 (s, 3H), 3.79 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 157.92, 148.88, 147.30, 134.15, 133.51, 129.73, 120.73, 113.85, 112.06, 111.12, 55.92, 55.80, 55.29, 40.59.

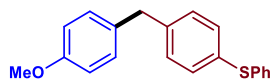
HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₁₆H₁₉O₃⁺ 259.1329; Found 259.1341.



bis(4-methoxyphenyl)methane 3o¹⁷ Yellow solid (56.1 mg, 82%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.18 – 7.05 (m, 4H), 6.92 – 6.76 (m, 4H), 3.89 (s, 2H), 3.80 (s, 6H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 157.94, 133.76, 129.77, 113.88, 55.28, 40.16.

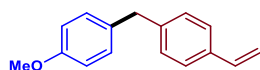


(4-(4-methoxybenzyl)phenyl)(phenyl)sulfane 3p¹⁸ Yellow liquid (27.5 mg, 30%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.21 (m, 7H), 7.11 (t, *J* = 8.6 Hz, 4H), 6.83 (d, *J* = 8.7 Hz, 2H), 3.90 (s, 2H), 3.78 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.06, 141.07, 136.48, 132.76, 132.47, 131.85, 130.35, 129.92, 129.74, 129.14, 126.72, 113.95, 55.30, 40.63.

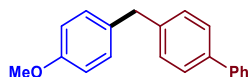
HRMS (ESI) *m/z*: [M] Calcd for C₂₀H₁₉SO 306.1078; Found 306.1057.



1-methoxy-4-(4-vinylbenzyl)benzene 3q¹⁹ Yellow liquid (23.5 mg, 35%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.2 Hz, 2H), 7.12 (dd, *J* = 14.1, 8.4 Hz, 4H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.69 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.71 (dd, *J* = 17.6, 0.9 Hz, 1H), 5.20 (dd, *J* = 10.9, 1.0 Hz, 1H), 3.92 (s, 2H), 3.79 (s, 3H).

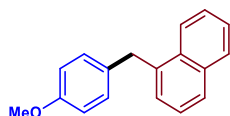
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.93, 141.26, 136.56, 135.38, 133.09, 129.80, 128.95, 126.27, 113.84, 113.13, 55.23, 40.71.



4-(4-methoxybenzyl)-1,1'-biphenyl 3r²⁰ Colorless liquid (55.1 mg, 67%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.56 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.51 (dd, *J* = 8.4, 2.4 Hz, 2H), 7.42 (td, *J* = 7.7, 4.4 Hz, 2H), 7.35 – 7.29 (m, 1H), 7.24 (dt, *J* = 5.4, 2.5 Hz, 2H), 7.14 (dd, *J* = 8.9, 2.8 Hz, 2H), 6.93 – 6.78 (m, 2H), 3.96 (s, 2H), 3.78 (s, 3H).

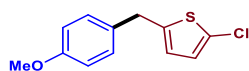
¹³C NMR (101 MHz, Chloroform-*d*) δ 158.06, 141.06, 140.75, 138.98, 133.16, 129.92, 129.22, 128.74, 127.21, 127.07, 127.03, 113.96, 55.29, 40.71.



1-(4-methoxybenzyl)naphthalene 3s¹² Pale yellow solid (61.0 mg, 82%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 80/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.99 (m, 1H), 7.90 – 7.85 (m, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.49 – 7.41 (m, 3H), 7.29 (dd, *J* = 7.0, 1.1 Hz, 1H), 7.15 – 7.11 (m, 2H), 6.86 – 6.81 (m, 2H), 4.41 (s, 2H), 3.78 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 157.93, 137.09, 133.95, 132.70, 132.12, 129.71, 128.70, 127.18, 127.10, 125.98, 125.60, 125.58, 124.31, 113.89, 55.27, 38.19.

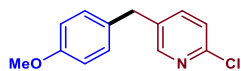


2-chloro-5-(4-methoxybenzyl)thiophene 3t Yellow liquid (46.4 mg, 65%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.26 (d, *J* = 1.7 Hz, 2H), 7.15 – 7.11 (m, 2H), 6.88 – 6.84 (m, 2H), 4.02 (s, 2H), 3.80 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.71, 150.47, 141.76, 138.26, 130.58, 129.47, 114.25, 55.31, 32.65.

HRMS (ESI) m/z : $[M + K]^+$ Calcd for $C_{12}H_{11}KClSO^+$ 276.9851; Found 276.9831.

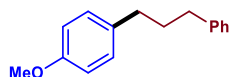


2-chloro-5-(4-methoxybenzyl)pyridine 3u²¹ Yellow solid (40.6 mg, 58%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.26 (d, J = 2.5 Hz, 1H), 7.41 (dd, J = 8.2, 2.5 Hz, 1H), 7.23 (d, J = 8.2 Hz, 1H), 7.10 – 7.04 (m, 2H), 6.88 – 6.82 (m, 2H), 3.90 (s, 2H), 3.79 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.37, 149.70, 149.27, 139.21, 136.06, 131.29, 129.81, 124.08, 114.20, 55.31, 37.35.

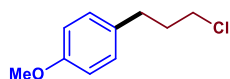
HRMS (ESI) m/z : $[M + H]^+$ Calcd for $C_{13}H_{13}NClO^+$ 234.0680; Found 234.0690.



1-methoxy-4-(3-phenylpropyl)benzene 3v²² Colorless liquid (36.0 mg, 53%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H), 7.18 (dt, J = 7.9, 1.5 Hz, 3H), 7.14 – 7.06 (m, 2H), 6.89 – 6.77 (m, 2H), 3.79 (s, 3H), 2.61 (m, 4H), 1.96 – 1.88 (m, 2H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 157.72, 142.40, 134.40, 129.34, 128.48, 128.32, 125.74, 113.73, 55.29, 35.41, 34.53, 33.25.

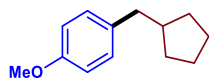


1-(3-chloropropyl)-4-methoxybenzene 3w²³ Colorless liquid (35.3 mg, 64%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.16 – 7.07 (m, 2H), 6.88 – 6.78 (m, 2H), 3.79 (s, 3H), 3.51 (t, J = 6.5 Hz, 2H), 2.72 (t, J = 7.4 Hz, 2H), 2.09 – 2.01 (m, 2H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.02, 132.72, 129.47, 113.92, 55.28, 44.23, 34.25, 31.84.

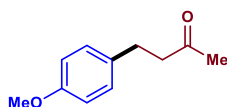
HRMS (ESI) m/z : $[M + K]^+$ Calcd for $C_{10}H_{13}KOCl^+$ 223.0287; Found 223.0299.



1-(cyclopentylmethyl)-4-methoxybenzene 3x²⁴ Yellow liquid (30.2 mg, 53%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.08 (dd, *J* = 9.0, 2.6 Hz, 2H), 6.88 – 6.76 (m, 2H), 3.78 (s, 3H), 2.54 (d, *J* = 7.4 Hz, 2H), 2.11 – 1.99 (m, 1H), 1.75 – 1.57 (m, 4H), 1.55 – 1.47 (m, 2H), 1.27 – 1.10 (m, 2H).

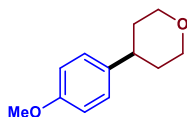
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.60, 134.56, 129.64, 113.55, 55.24, 42.19, 41.18, 32.42, 24.95.



4-(4-methoxyphenyl)butan-2-one 3y²⁵ Yellow liquid (24.6 mg, 46%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 40/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.19 – 6.96 (m, 2H), 6.88 – 6.66 (m, 2H), 3.75 (s, 3H), 2.90 – 2.60 (m, 4H), 2.10 (s, 3H).

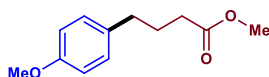
¹³C NMR (101 MHz, Chloroform-*d*) δ 208.05, 157.98, 133.03, 129.24, 113.90, 55.19, 45.37, 30.04, 28.89.



4-(4-methoxyphenyl)tetrahydro-2H-pyran 3z²⁶ White solid (31.7 mg, 55%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (600 MHz, CDCl₃) δ 7.19 – 7.11 (m, 2H), 6.90 – 6.83 (m, 2H), 4.12 – 4.02 (m, 2H), 3.80 (s, 3H), 3.52 (td, *J* = 11.5, 2.9 Hz, 2H), 2.71 (tt, *J* = 11.5, 4.4 Hz, 1H), 1.83 – 1.74 (m, 4H).

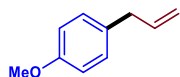
¹³C NMR (151 MHz,) δ 158.03, 138.12, 127.59, 113.88, 68.45, 55.26, 40.70, 34.20.



methyl 4-(4-methoxyphenyl)butanoate 3ab²⁵ Yellow liquid (40.6 mg, 65%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.13 – 7.05 (m, 2H), 6.87 – 6.79 (m, 2H), 3.78 (s, 3H), 3.66 (s, 3H), 2.59 (t, *J* = 7.6 Hz, 2H), 2.32 (t, *J* = 7.5 Hz, 2H), 1.93 (m, 2H).

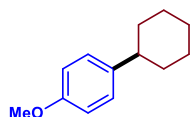
¹³C NMR (101 MHz, Chloroform-*d*) δ 173.96, 157.84, 133.37, 129.32, 113.74, 55.18, 51.43, 34.15, 33.28, 26.67.



1-allyl-4-methoxybenzene 3ac^{19,27} Yellow liquid (18.7 mg, 42%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.11 (m, 2H), 6.98 – 6.84 (m, 2H), 6.03 (m, 1H), 5.19 – 5.08 (m, 2H), 3.84 (s, 3H), 3.40 (dd, *J* = 6.7, 1.6 Hz, 2H).

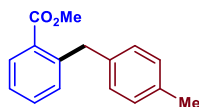
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.94, 137.85, 132.05, 129.46, 115.37, 113.79, 55.20, 39.30.



1-cyclohexyl-4-methoxybenzene 3ad²⁸ Yellow solid (26.2 mg, 46%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (600 MHz, Chloroform-*d*) δ 7.17 – 7.12 (m, 2H), 6.88 – 6.82 (m, 2H), 3.80 (s, 3H), 2.46 (m, 1H), 1.92 – 1.81 (m, 4H), 1.75 (m, 1H), 1.45 – 1.35 (m, 4H), 1.30 – 1.22 (m, 1H). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.58 – 7.53 (m, 1H), 7.32 (d, *J* = 4.3 Hz, 1H), 7.16 – 7.10 (m, 1H), 6.86 – 6.81 (m, 1H), 3.78 (s, 3H), 2.44 (d, *J* = 3.6 Hz, 1H), 1.85 (d, *J* = 9.7 Hz, 2H), 1.56 (s, 2H), 1.41 – 1.34 (m, 2H), 1.25 (s, 4H).

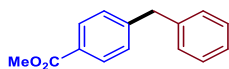
¹³C NMR (151 MHz,) δ 157.62, 140.36, 127.60, 113.62, 55.20, 43.66, 34.70, 26.93, 26.15.



methyl 2-(4-methylbenzyl)benzoate 4a²⁹ Colorless liquid (54.7 mg, 76%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.41 (td, *J* = 7.5, 1.5 Hz, 1H), 7.27 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.20 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.05 (q, *J* = 8.2 Hz, 4H), 4.33 (s, 2H), 3.83 (s, 3H), 2.30 (s, 3H).

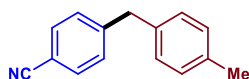
¹³C NMR (101 MHz, Chloroform-*d*) δ 168.16, 142.49, 137.84, 135.40, 131.95, 131.48, 130.64, 129.95, 129.05, 128.80, 126.16, 51.94, 39.17, 21.02.



methyl 4-benzylbenzoate 4b³⁰ Colorless oil (44.8 mg, 66%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, J = 8.3 Hz, 2H), 7.33 – 7.24 (m, 4H), 7.24 – 7.10 (m, 3H), 4.03 (s, 2H), 3.89 (s, 3H).

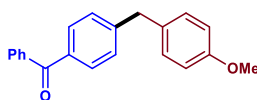
¹³C NMR (101 MHz, Chloroform-*d*) δ 167.08, 146.54, 140.14, 129.84, 128.97, 128.88, 128.63, 128.13, 126.40, 52.02, 41.94.



4-(4-methylbenzyl)benzonitrile 4c¹⁹ Yellow needle solid (29.8 mg, 48%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.52 (m, 2H), 7.27 (d, J = 8.2 Hz, 2H), 7.14 – 7.02 (m, 4H), 3.98 (s, 2H), 2.32 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 147.03, 136.24, 136.22, 132.23, 129.52, 129.39, 128.79, 119.07, 109.84, 41.51, 20.98.

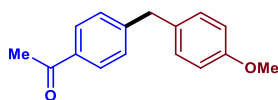


(4-(4-methoxybenzyl)phenyl)(phenyl)methanone 4d White solid (61.6 mg, 68%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.76 (dd, J = 18.5, 8.0 Hz, 4H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.5 Hz, 2H), 4.00 (s, 2H), 3.79 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 196.42, 158.20, 146.66, 137.82, 135.42, 133.72, 132.22, 130.45, 129.96, 129.94, 128.69, 128.21, 114.04, 55.26, 41.05.

HRMS (ESI) m/z : $[M + H]^+$ Calcd for $C_{21}H_{19}O_2^+$ 303.1380; Found 303.1377.

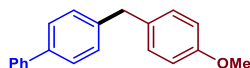


1-(4-(4-methoxybenzyl)phenyl)ethan-1-one 4e¹⁵ Yellow oil (49.0 mg, 68%) The product was

purified by silica gel column chromatography, using petroleum ether /EA = 100/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.80 – 6.71 (m, 2H), 3.89 (s, 2H), 3.70 (s, 3H), 2.49 (s, 3H).

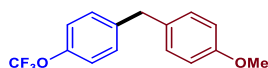
¹³C NMR (101 MHz, Chloroform-*d*) δ 197.83, 158.21, 147.34, 135.20, 132.15, 129.92, 129.00, 128.65, 114.06, 55.28, 41.03, 26.59.



4-(4-methoxybenzyl)-1,1'-biphenyl 4f³¹ White solid (51.0 mg, 62%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 120/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.47 (m, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.26 – 7.21 (m, 1H), 7.16 (d, *J* = 7.9 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 6.76 (d, *J* = 8.7 Hz, 2H), 3.88 (s, 2H), 3.70 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.08, 141.08, 140.77, 139.00, 133.17, 129.94, 129.24, 128.75, 127.23, 127.09, 127.05, 113.98, 55.30, 40.72.

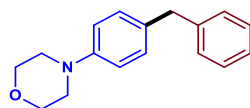


1-methoxy-4-(4-(trifluoromethoxy)benzyl)benzene 4g¹² White liquid (37.2 mg, 44%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.20 – 7.16 (m, 2H), 7.14 – 7.06 (m, 4H), 6.84 (dd, *J* = 8.9, 2.4 Hz, 2H), 3.92 (s, 2H), 3.79 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 158.14, 147.51, 140.34, 132.51, 129.96, 129.84, 120.98, 120.23 (q, *J* = 258 Hz), 113.99, 55.25, 40.29.

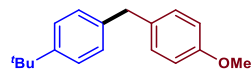
¹⁹F NMR (377 MHz, Chloroform-*d*) δ -57.91.



4-(4-benzylphenyl)morpholine 4h³² Yellow solid (20.5 mg, 27%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.21 (m, 3H), 7.21 – 7.17 (m, 2H), 7.16 – 7.07 (m, 2H), 6.84 (dd, *J* = 9.0, 7.1 Hz, 2H), 3.91 (s, 2H), 3.85 (dt, *J* = 6.0, 2.9 Hz, 4H), 3.15 – 3.09 (m, 4H).

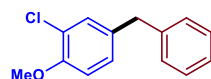
¹³C NMR (101 MHz, Chloroform-*d*) δ 149.60, 141.59, 132.73, 129.63, 128.81, 128.38, 125.91, 115.89, 66.95, 49.60, 40.99.



1-(tert-butyl)-4-(4-methoxybenzyl)benzene 4i¹⁰ Colorless oil (31.3 mg, 41%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 120/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.29 (m, 2H), 7.17 – 7.07 (m, 4H), 6.87 – 6.80 (m, 2H), 3.90 (s, 2H), 3.79 (s, 3H), 1.31 (s, 9H).

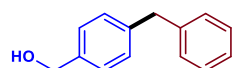
¹³C NMR (101 MHz, Chloroform-*d*) δ 157.94, 148.76, 138.56, 133.45, 129.90, 128.39, 125.34, 113.87, 55.27, 40.53, 34.37, 31.42.



4-benzyl-2-chloro-1-methoxybenzene 4k¹² Colorless liquid (43.9 mg, 63%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 2H), 7.19 (ddt, J = 12.9, 8.3, 1.9 Hz, 4H), 7.03 (dd, J = 8.4, 2.2 Hz, 1H), 6.84 (d, J = 8.4 Hz, 1H), 3.89 (s, 2H), 3.87 (s, 3H).

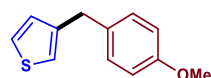
¹³C NMR (101 MHz, Chloroform-*d*) δ 153.39, 140.76, 134.39, 130.63, 128.82, 128.57, 128.04, 126.26, 122.32, 112.12, 56.20, 40.77.



(4-(4-methylbenzyl)phenyl)methanol 4l³³ Pale yellow solid (33.1 mg, 52%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.35 – 7.09 (m, 9H), 5.10 (t, J = 5.6 Hz, 1H), 4.43 (d, J = 5.6 Hz, 2H), 3.91 (s, 2H).

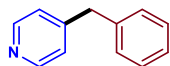
¹³C NMR (101 MHz, DMSO-*d*₆) δ 141.95, 140.64, 140.08, 129.09, 128.87, 128.85, 127.12, 126.36, 63.18, 41.28.



3-(4-methoxybenzyl)thiophene 4n³⁴ Colorless liquid (24.5 mg, 40%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.25 – 7.23 (m, 1H), 7.16 – 7.07 (m, 2H), 6.97 – 6.88 (m, 2H), 6.87 – 6.76 (m, 2H), 3.92 (s, 2H), 3.79 (s, 3H).

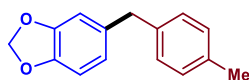
¹³C NMR (101 MHz, Chloroform-*d*) δ 158.04, 142.06, 132.75, 129.69, 128.41, 125.58, 121.02, 113.89, 55.28, 35.66.



4-benzylpyridine 4b³⁵ White solid (13.7 mg, 27%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.63 – 8.39 (m, 2H), 7.46 (dt, *J* = 7.8, 2.0 Hz, 1H), 7.41 – 7.00 (m, 6H), 3.98 (s, 2H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 150.13, 147.61, 139.75, 128.81, 128.65, 126.45, 123.40, 39.01.

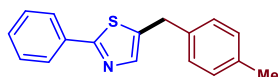


5-(4-methylbenzyl)benzo[d][1,3]dioxole 4p Yellow solid (44.7 mg, 56%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

¹H NMR (400 MHz, Chloroform-*d*) δ 7.11 – 7.03 (m, 4H), 6.75 – 6.69 (m, 1H), 6.64 (h, *J* = 1.7 Hz, 2H), 5.89 (s, 2H), 3.84 (s, 2H), 2.31 (s, 3H).

¹³C NMR (101 MHz, Chloroform-*d*) δ 147.69, 145.80, 138.25, 135.61, 135.34, 129.18, 128.68, 121.64, 109.38, 108.14, 100.81, 41.22, 21.02.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₁₅H₁₄NaO₂⁺ 249.0886; Found 249.0707.



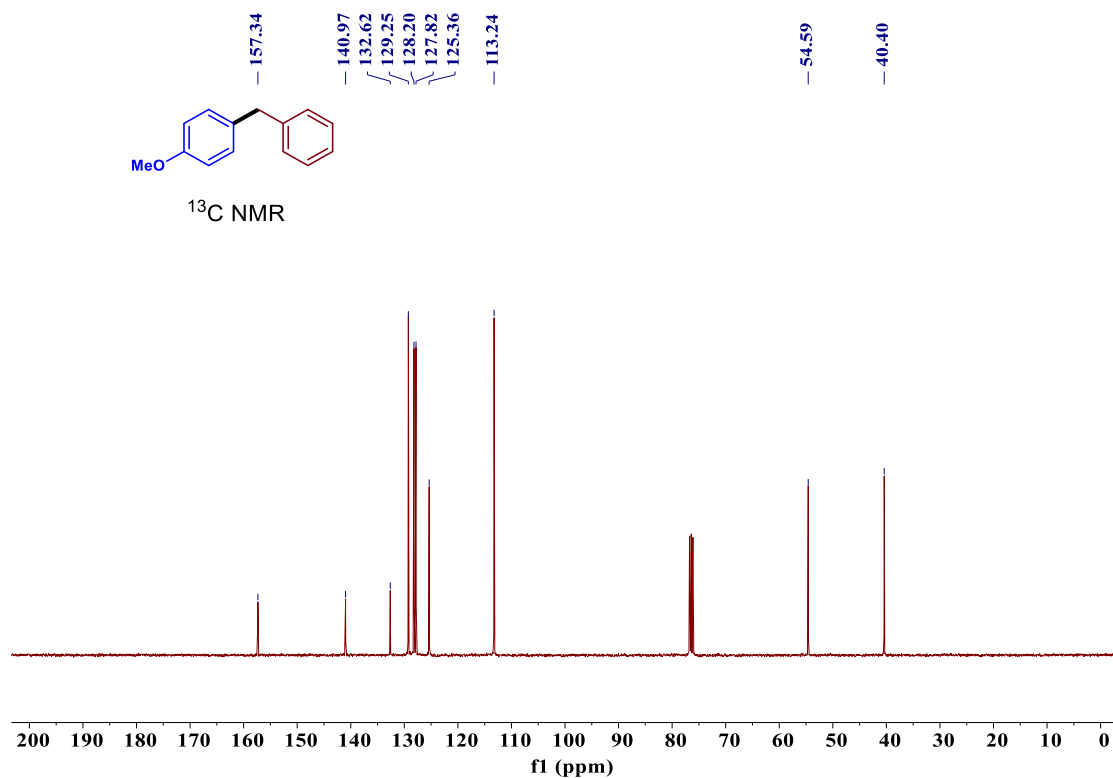
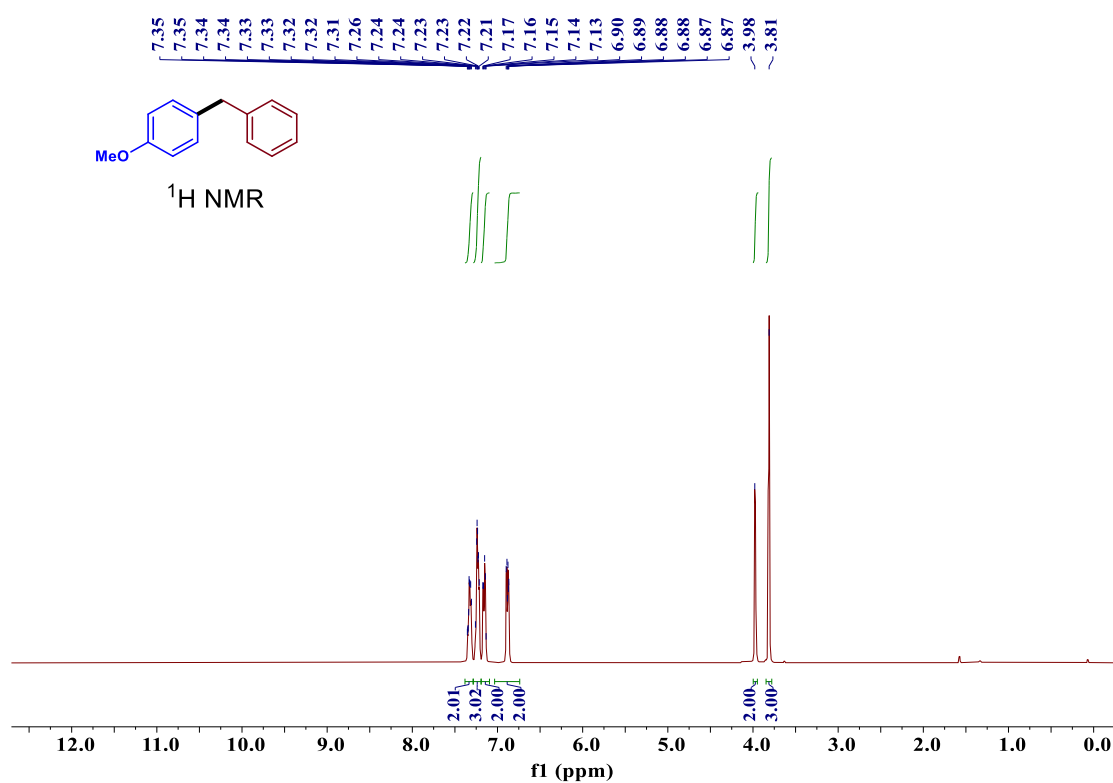
5-(4-methylbenzyl)-2-phenylthiazole 4q Yellow liquid (33.4 mg, 42%) The product was purified by silica gel column chromatography, using petroleum ether /EA = 60/1 (v/v) as an eluent.

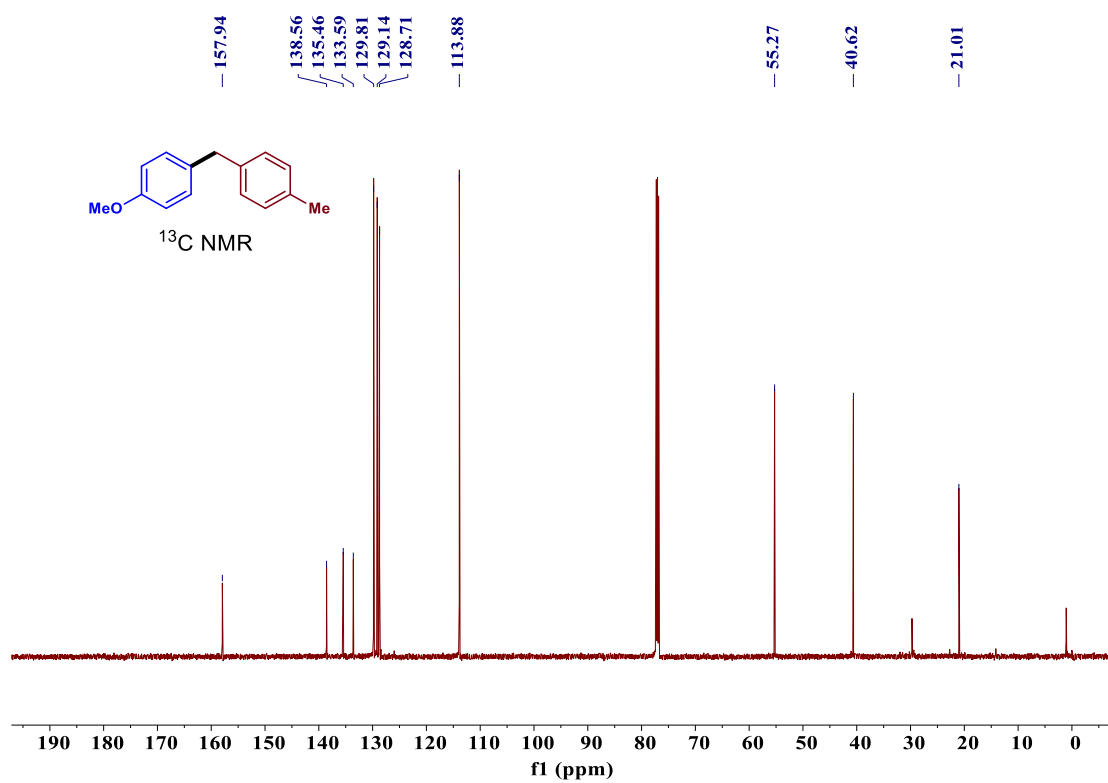
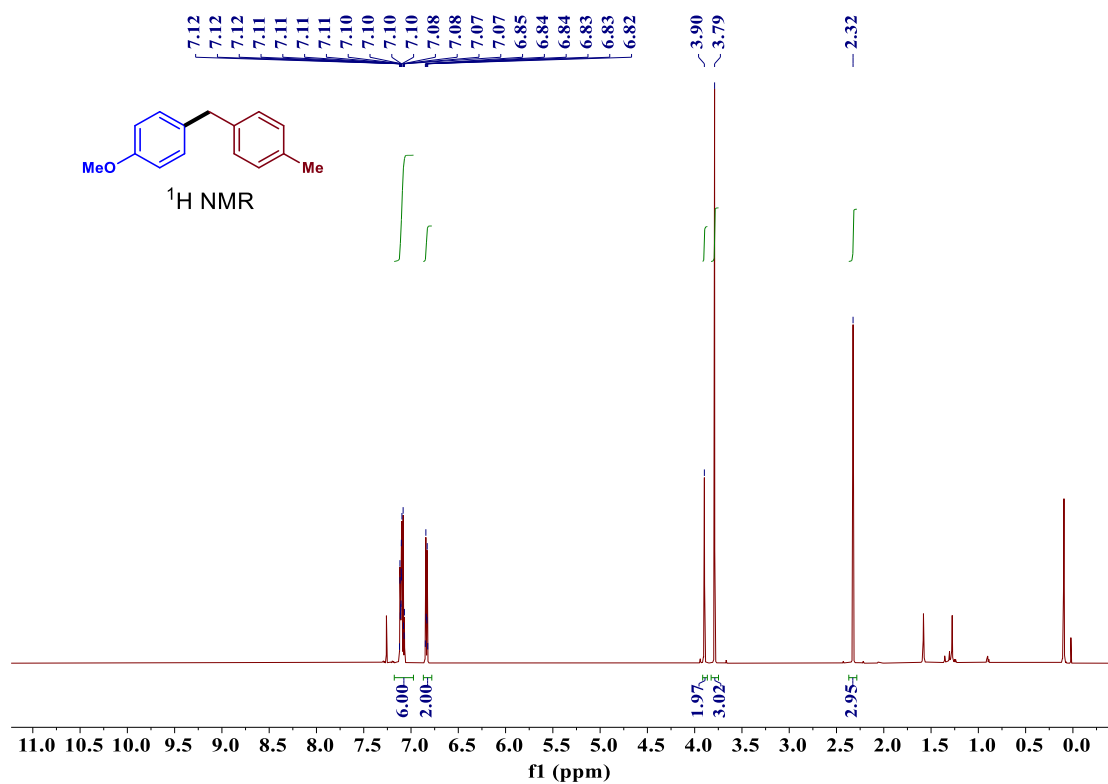
¹H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 7.3, 2.2 Hz, 2H), 7.39 – 7.27 (m, 3H), 7.14 (d, *J* = 7.7 Hz, 2H), 7.06 (d, *J* = 7.7 Hz, 2H), 6.64 (s, 1H), 4.07 (s, 2H), 2.25 (s, 3H).

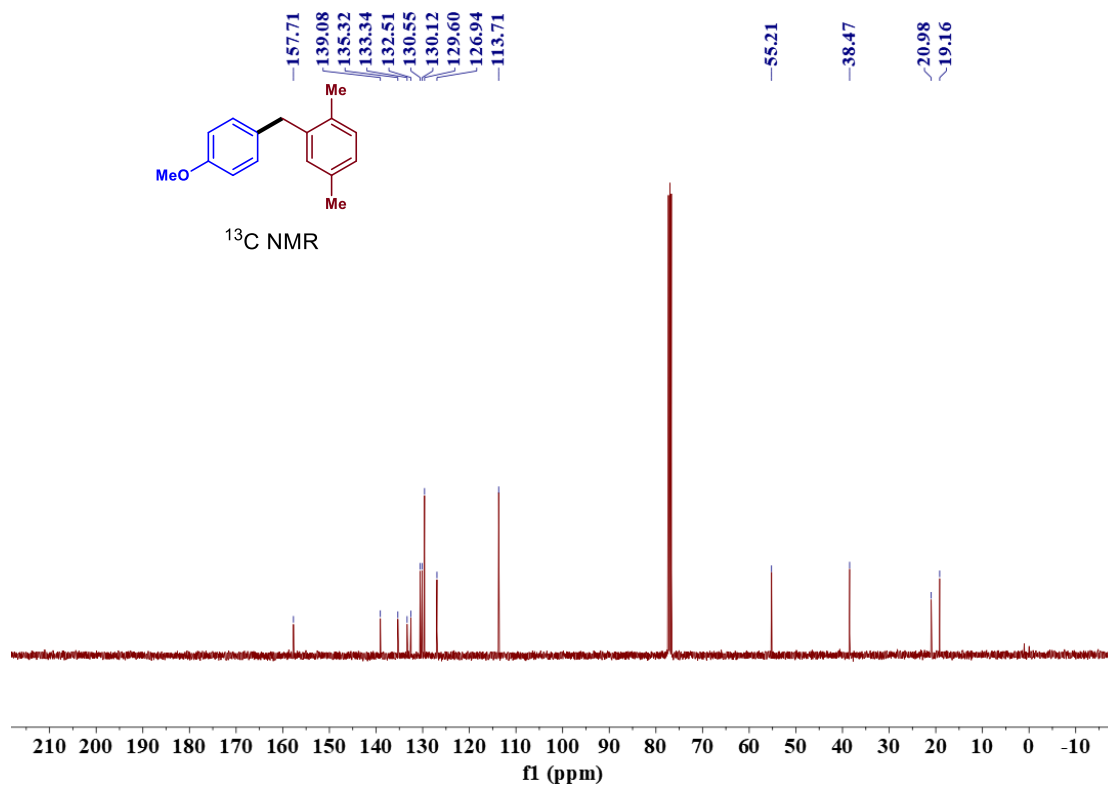
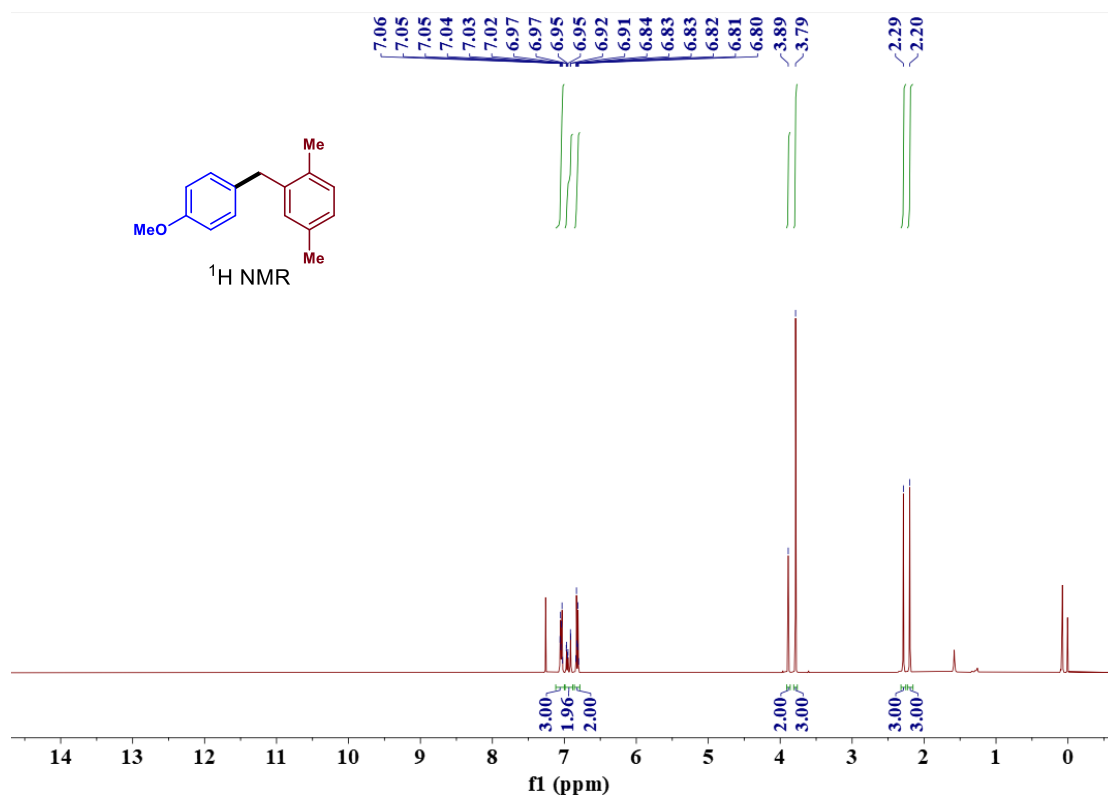
¹³C NMR (101 MHz, Chloroform-*d*) δ 167.91, 157.88, 136.00, 135.97, 133.78, 129.90, 129.28, 129.06, 128.91, 126.56, 114.27, 37.66, 21.13.

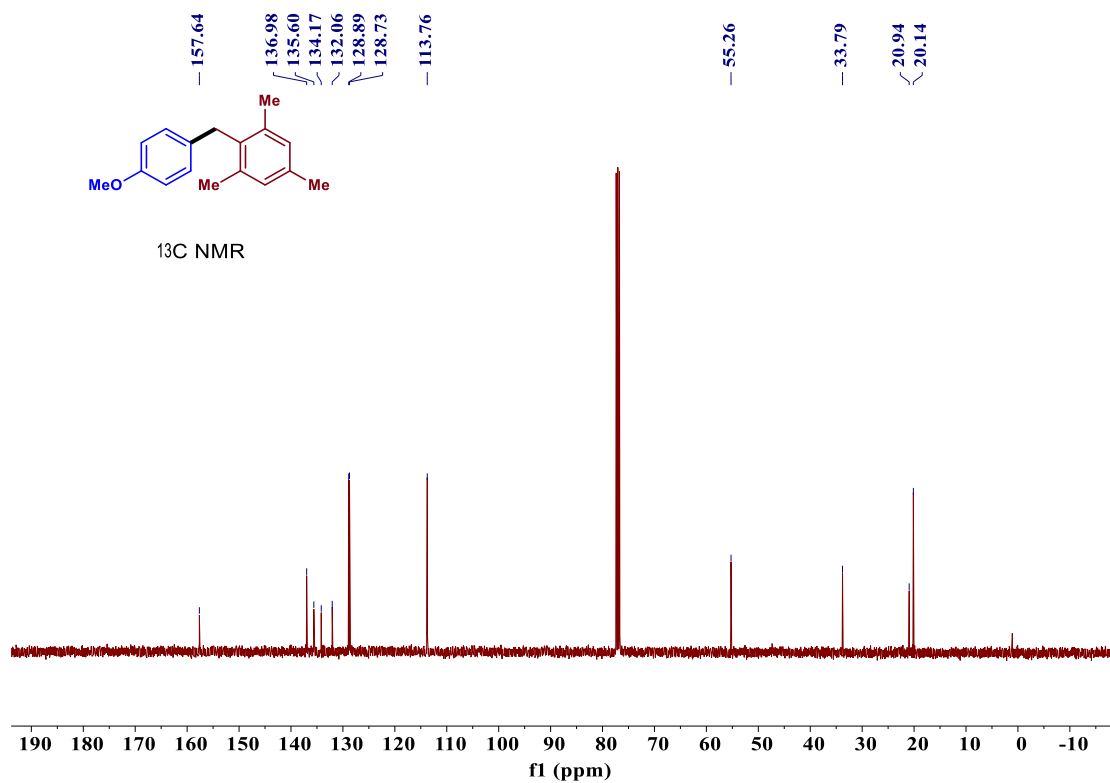
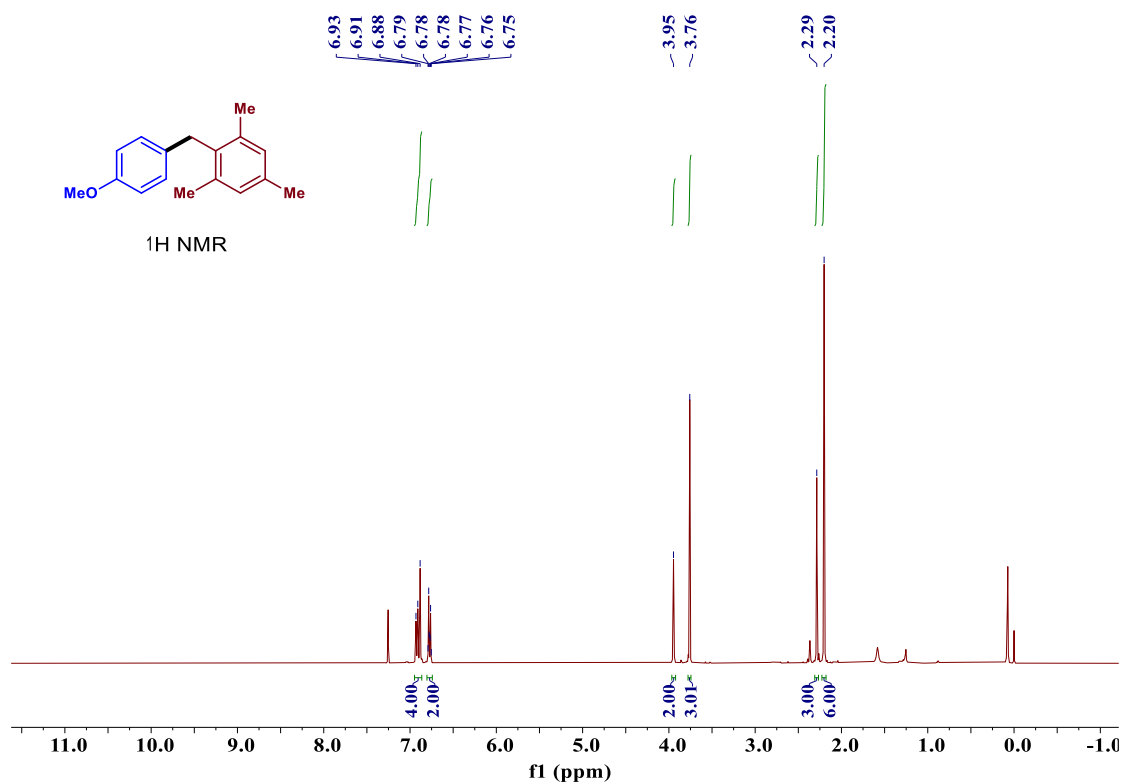
HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₁₇H₁₆NS⁺ 266.0998; Found 266.0998.

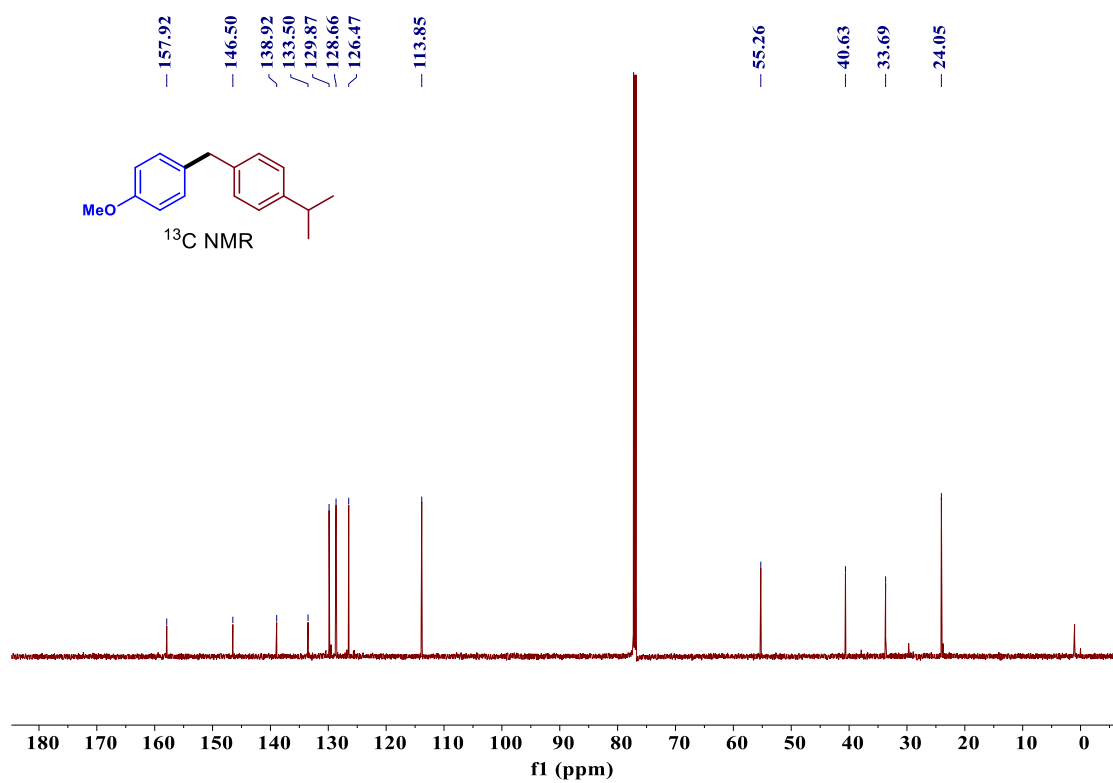
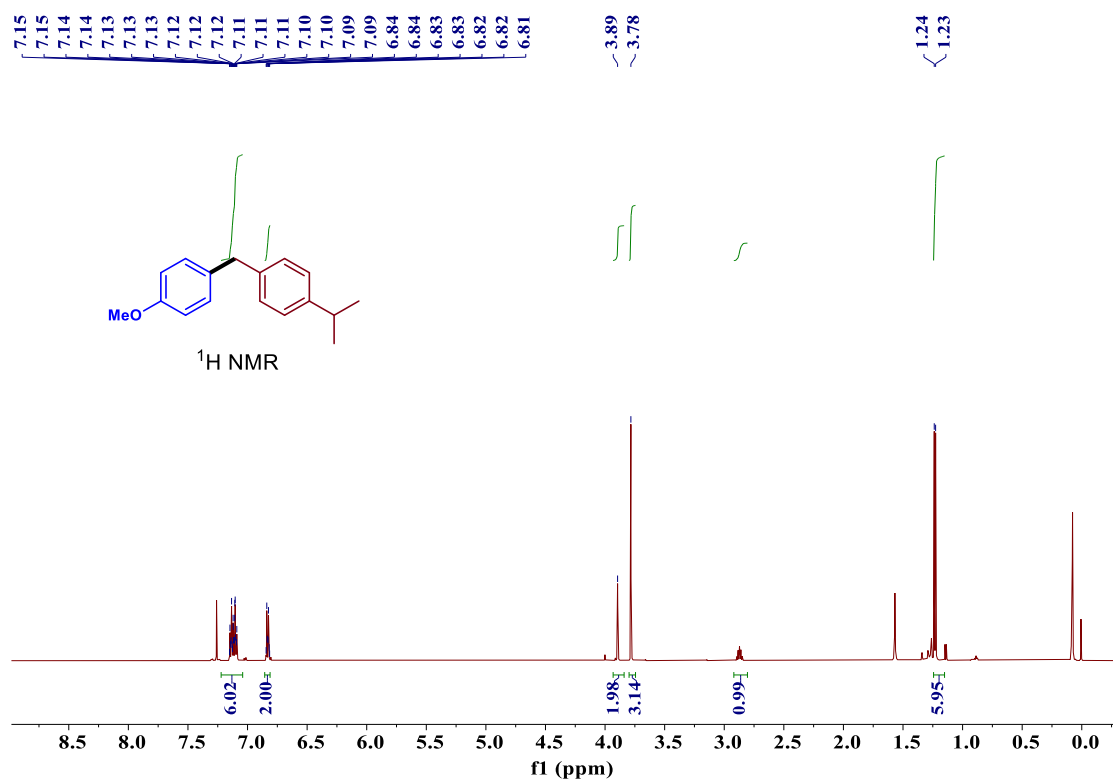
11. NMR Spectra of Substrates and Products

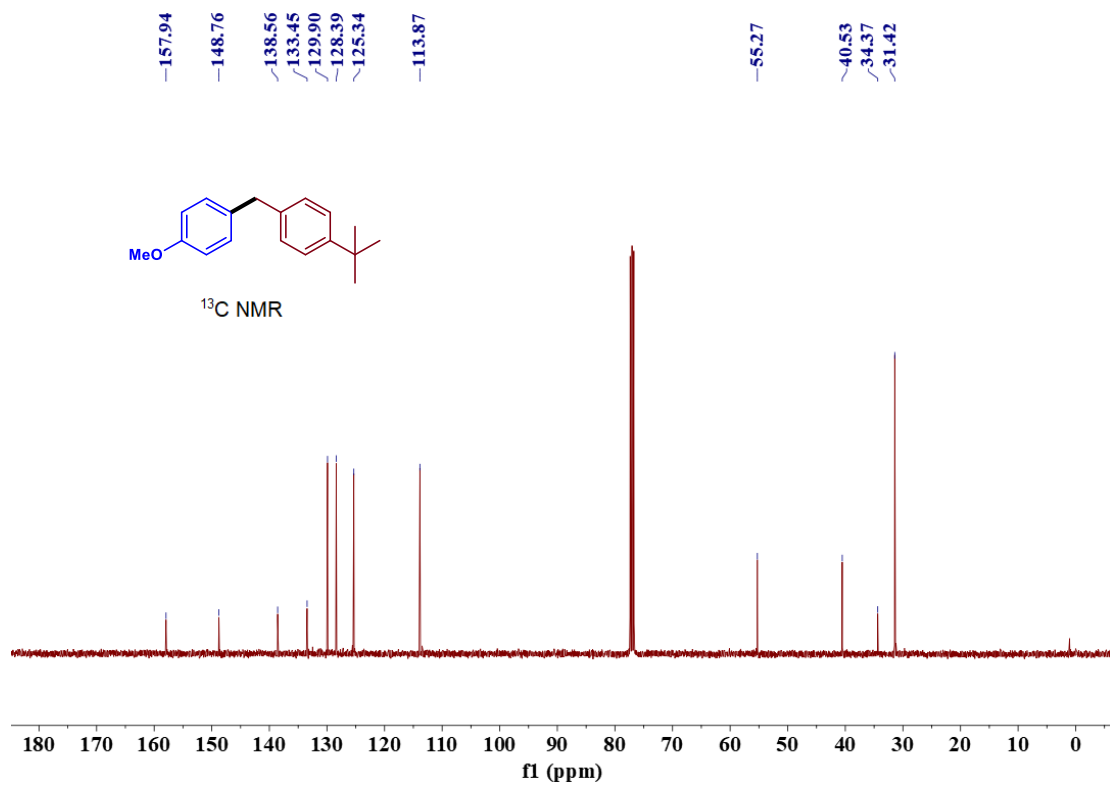
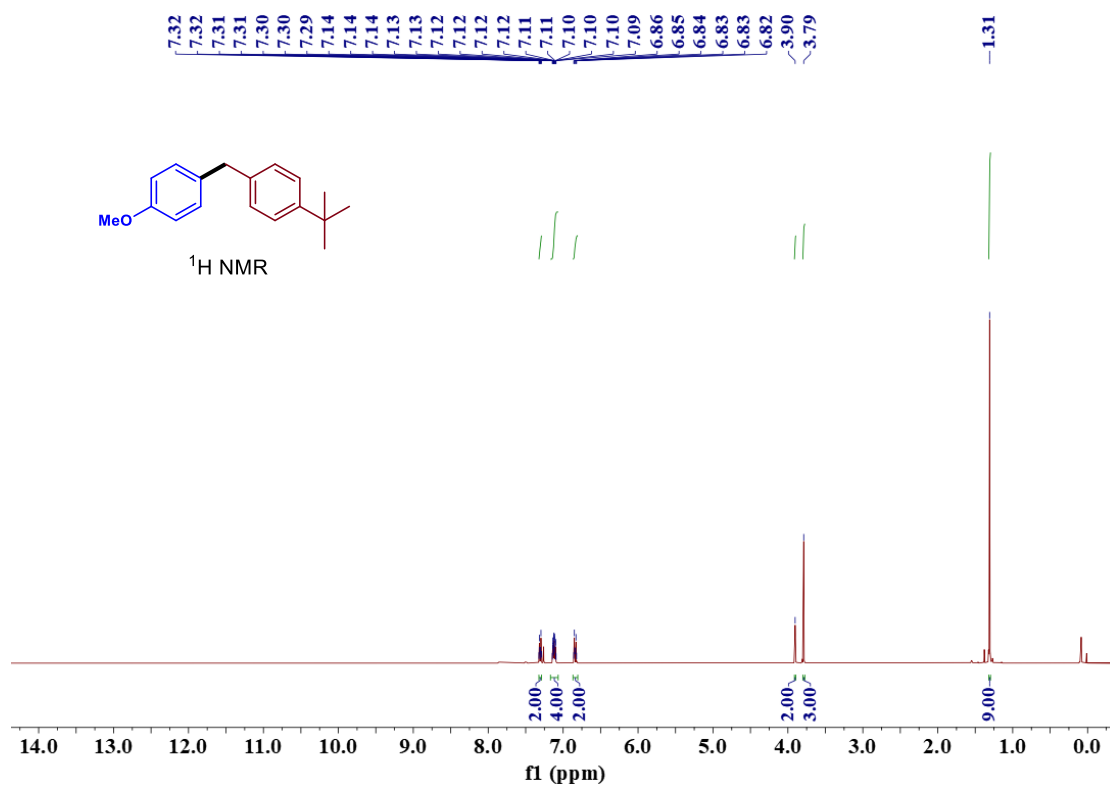


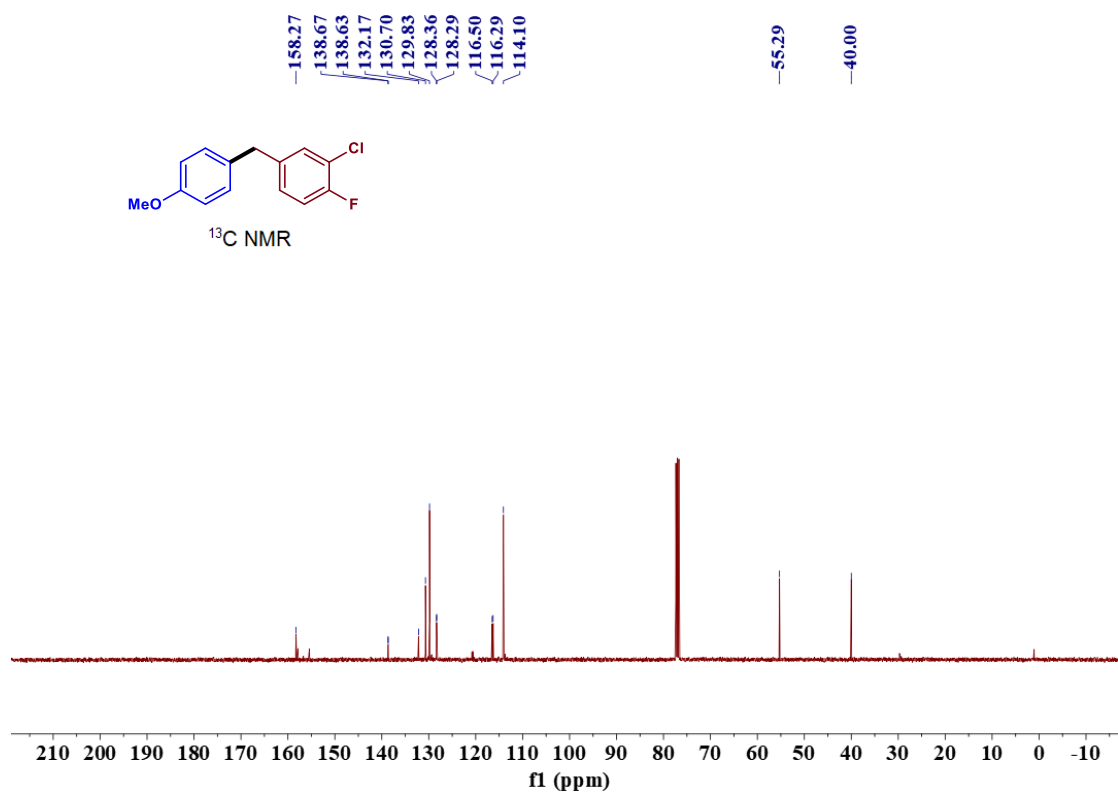
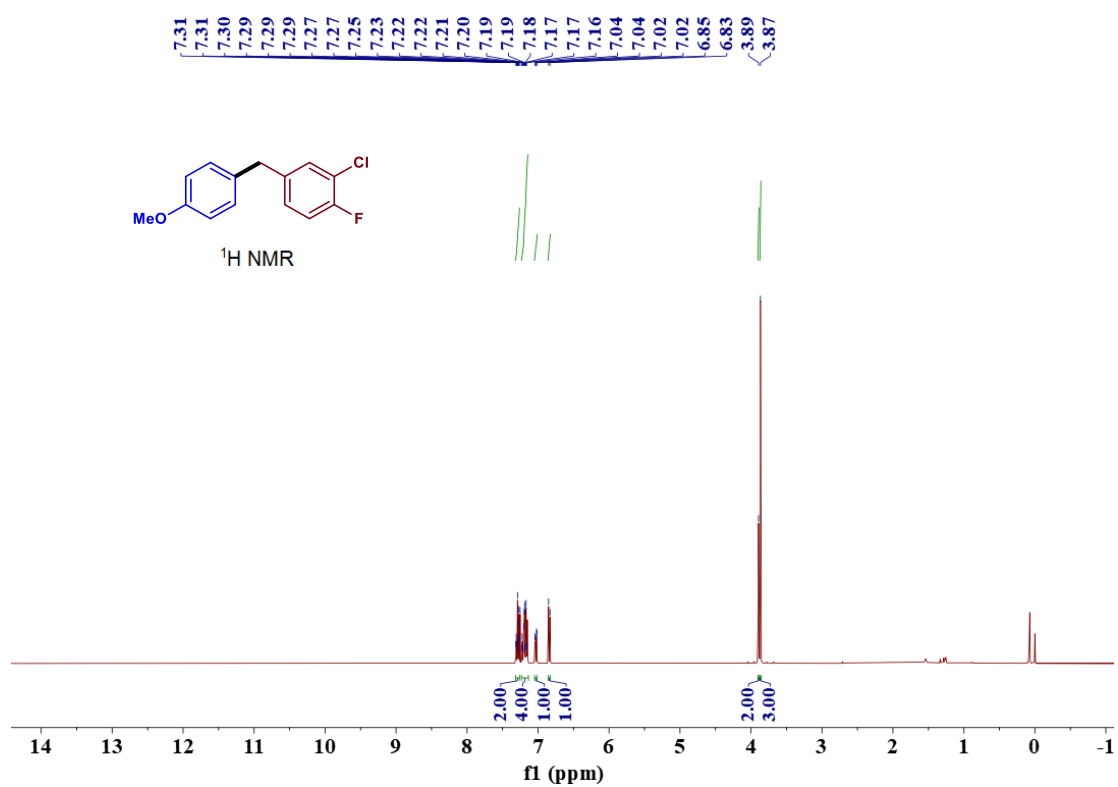


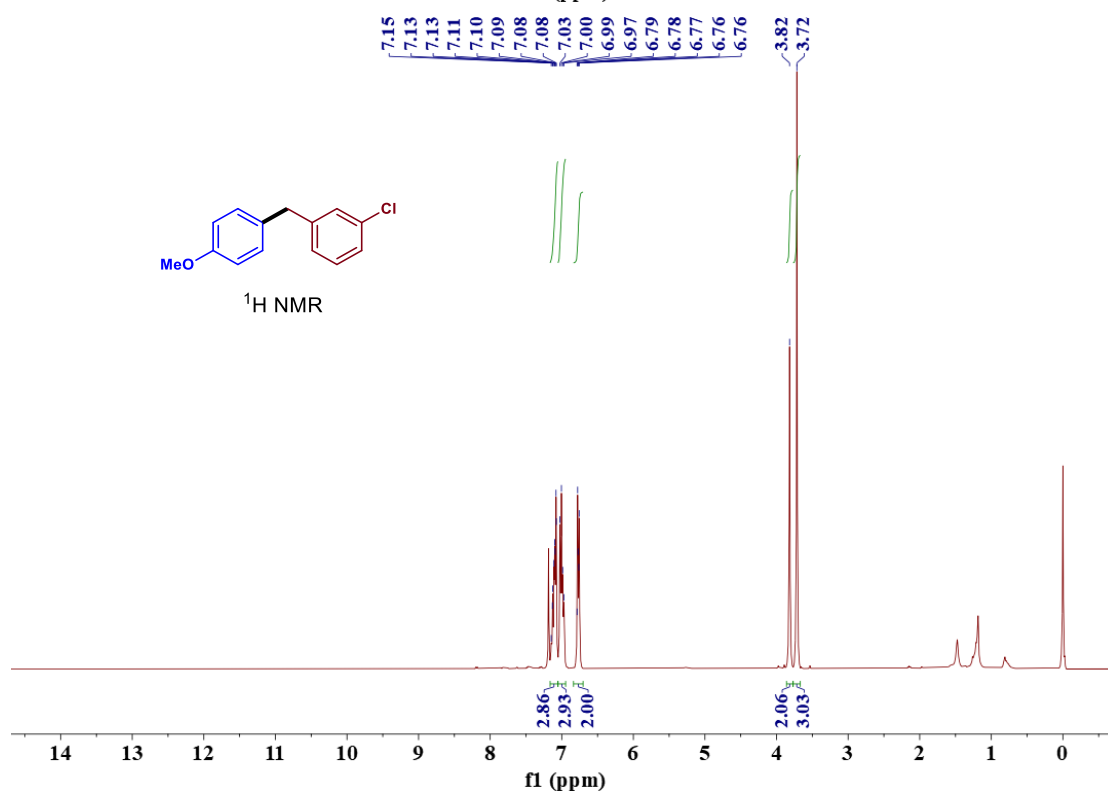
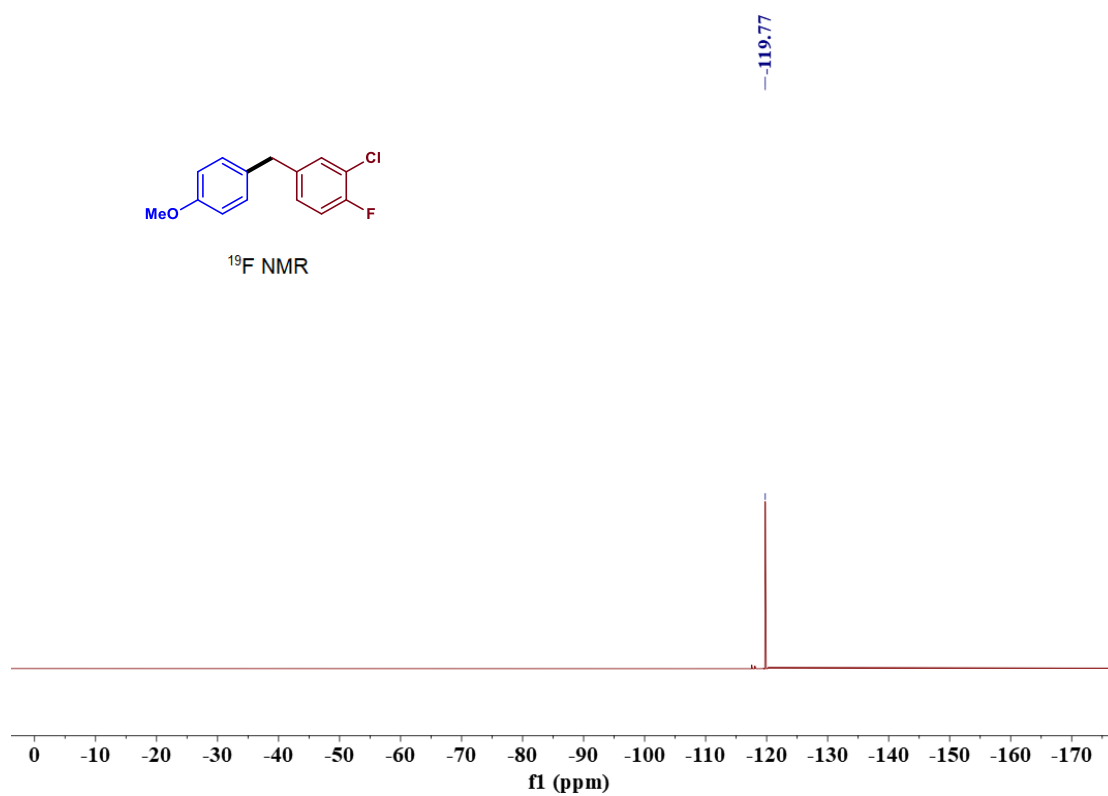


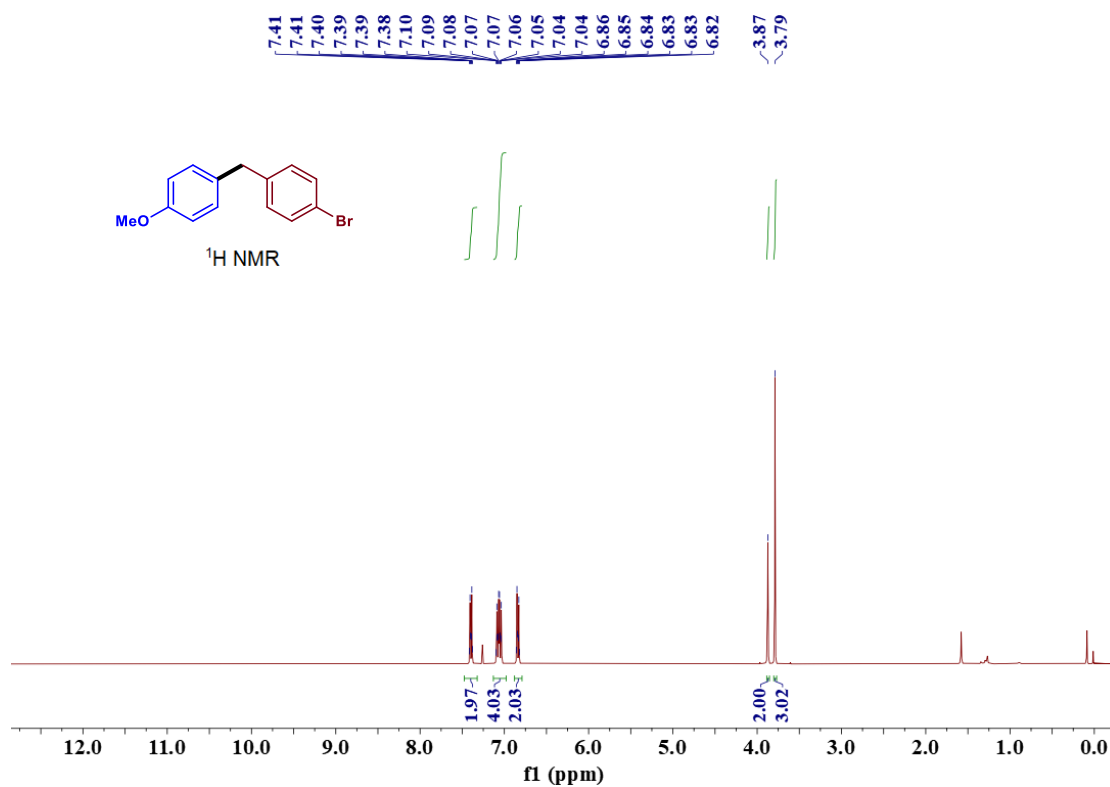
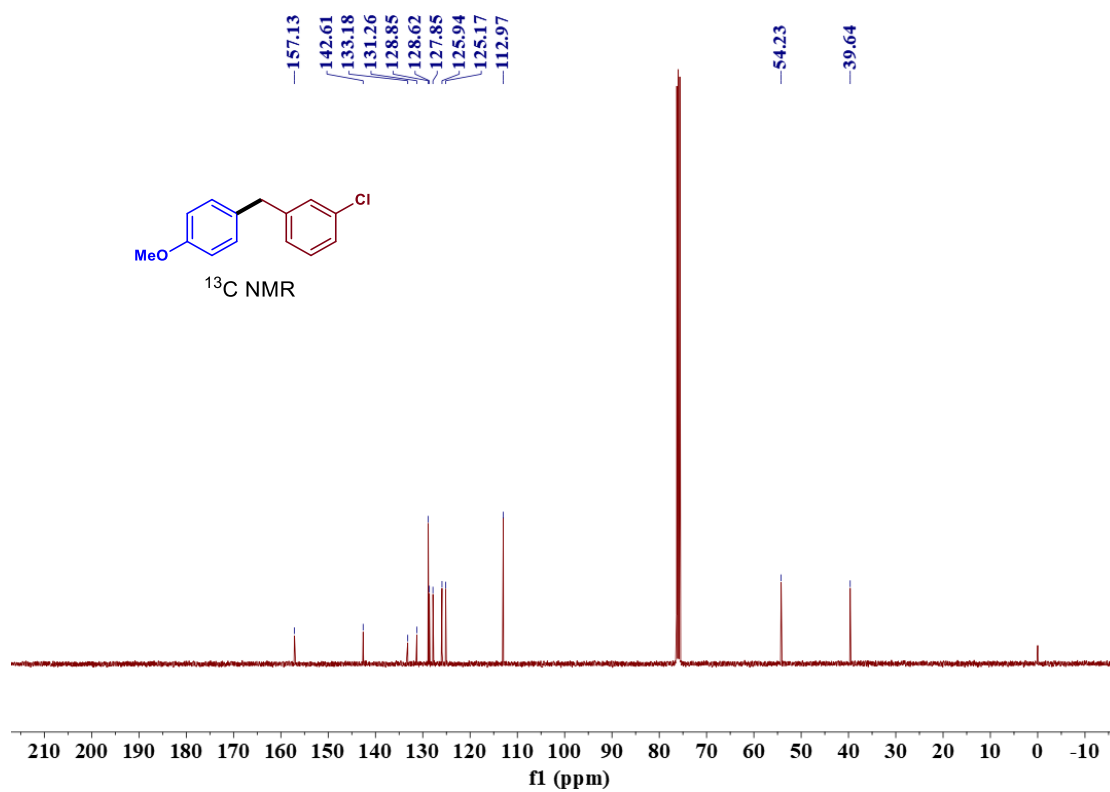


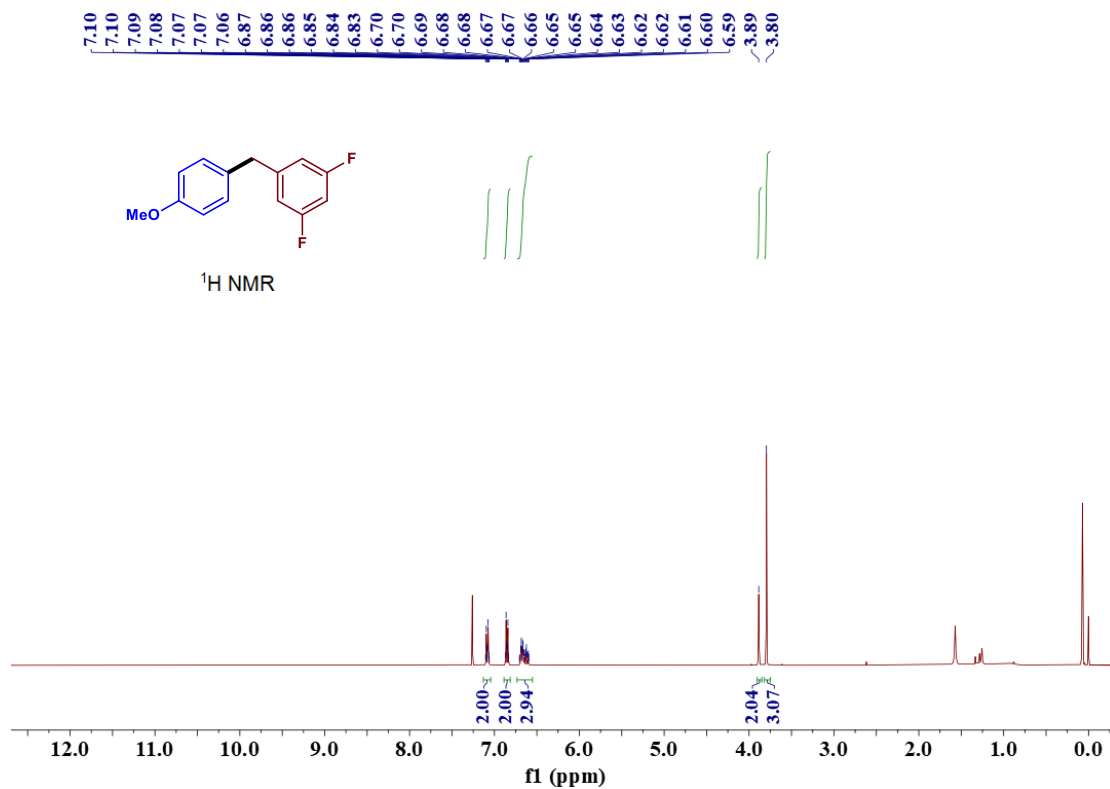
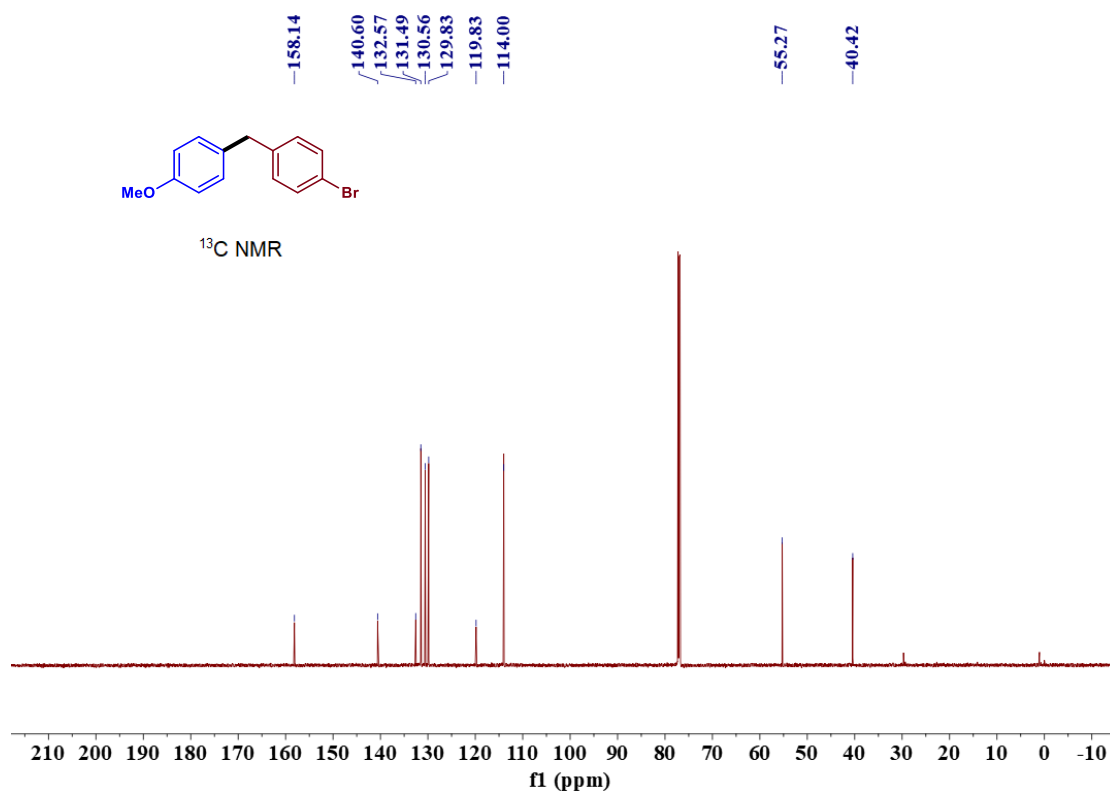


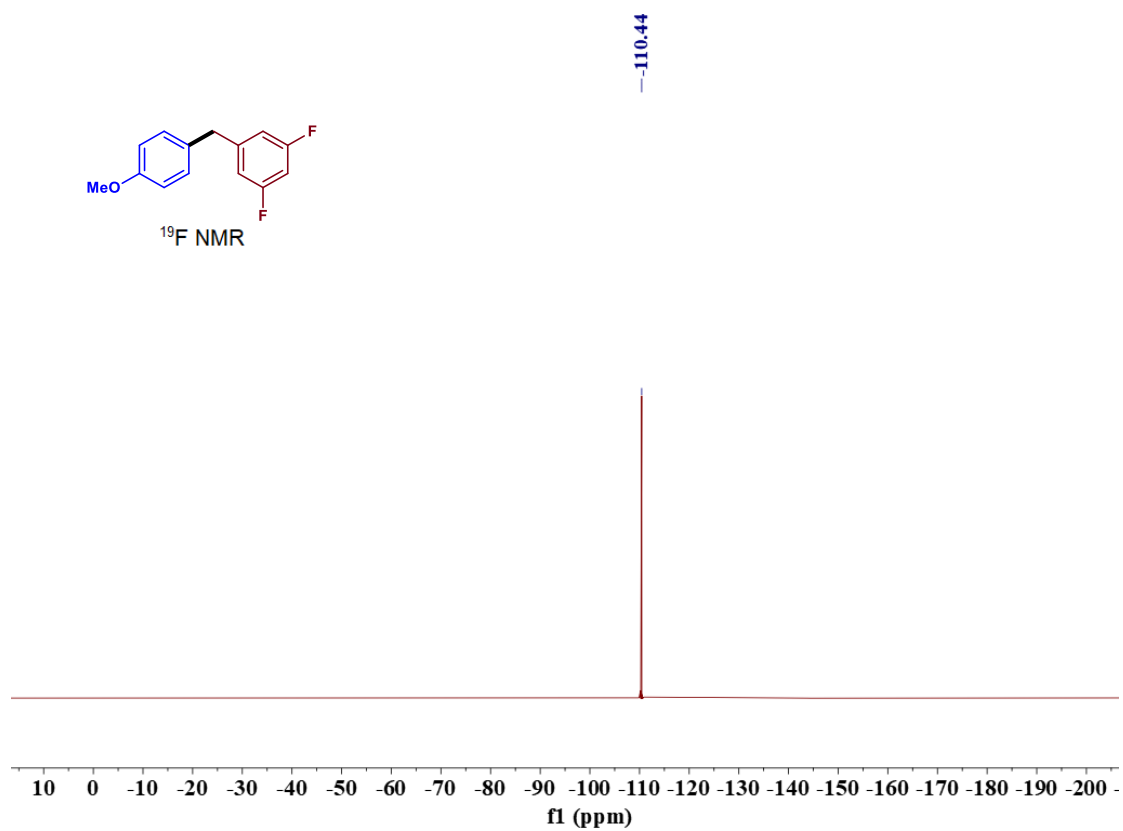
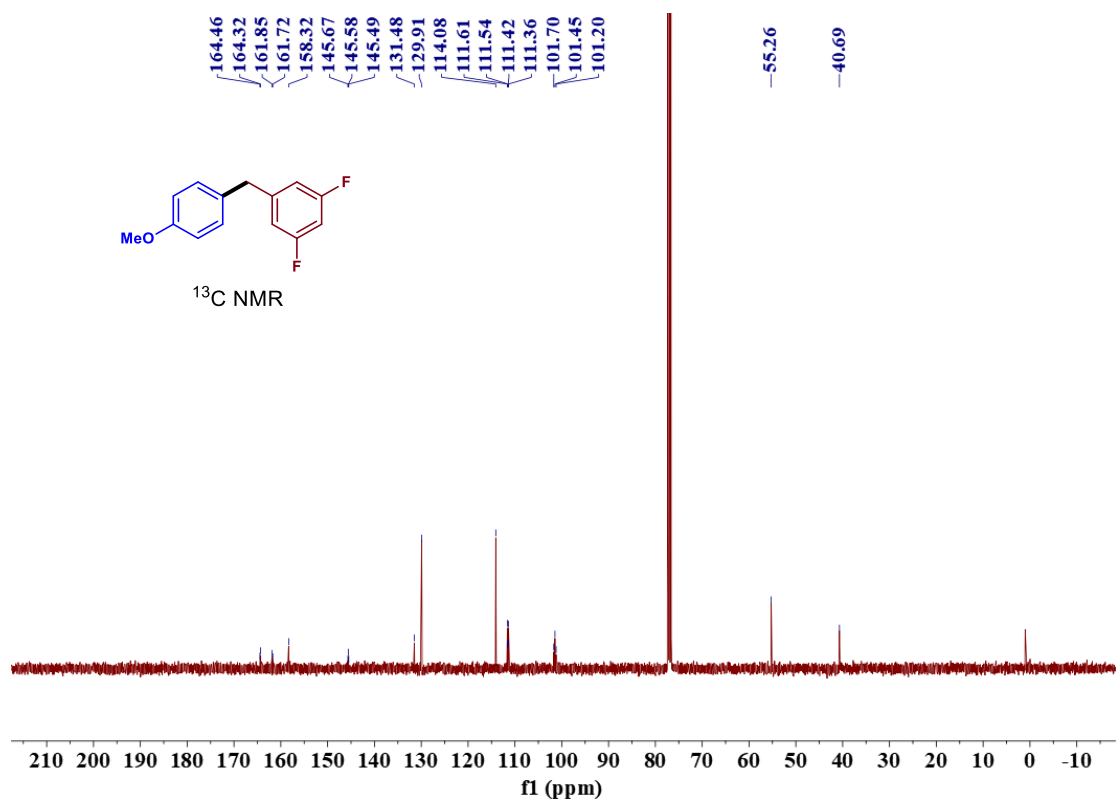


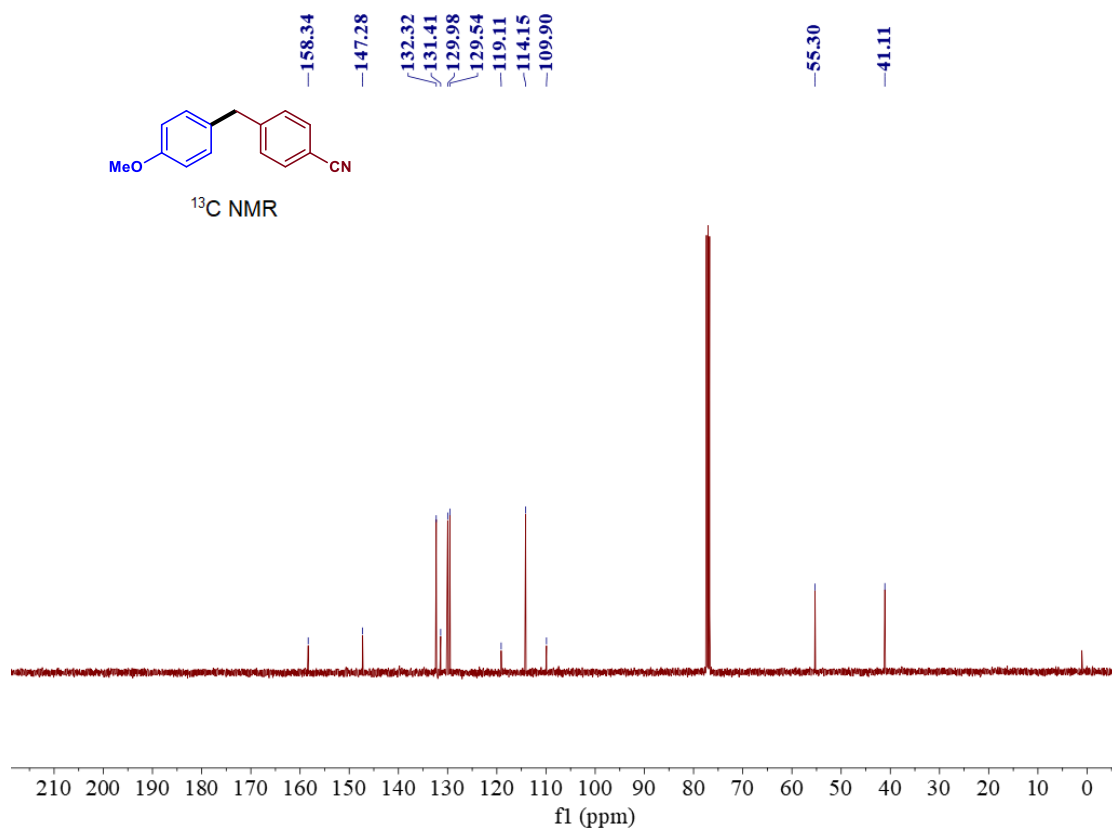
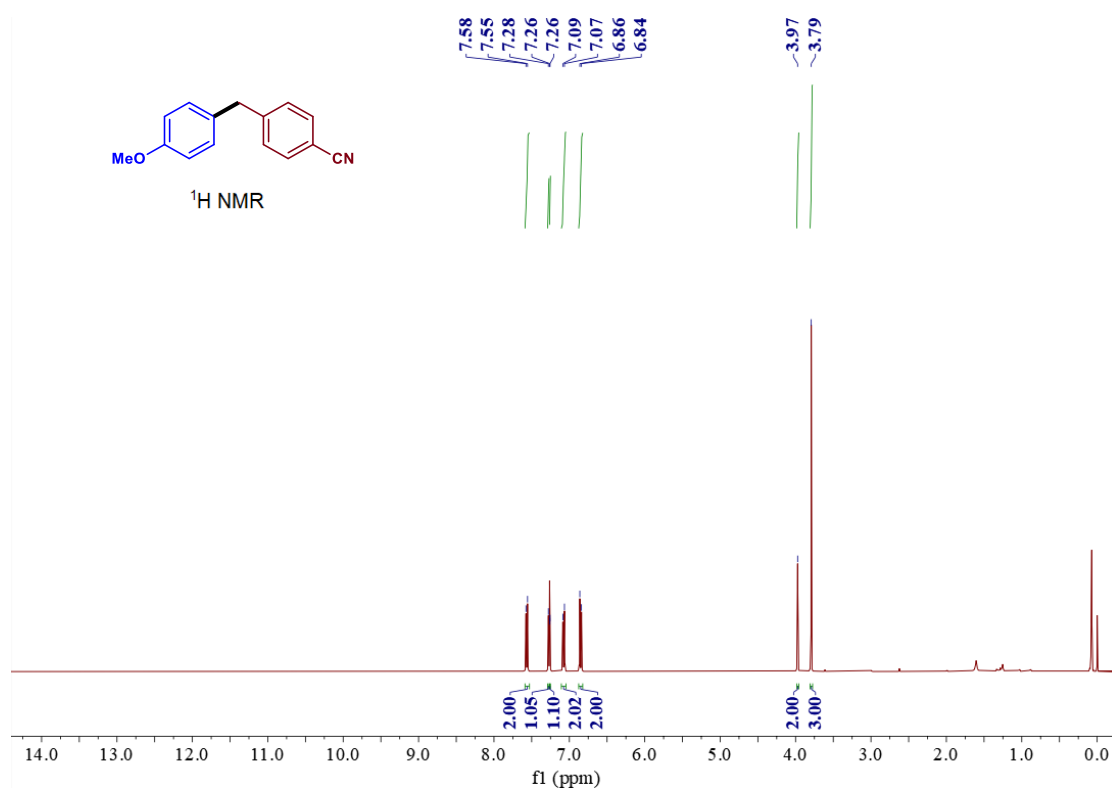


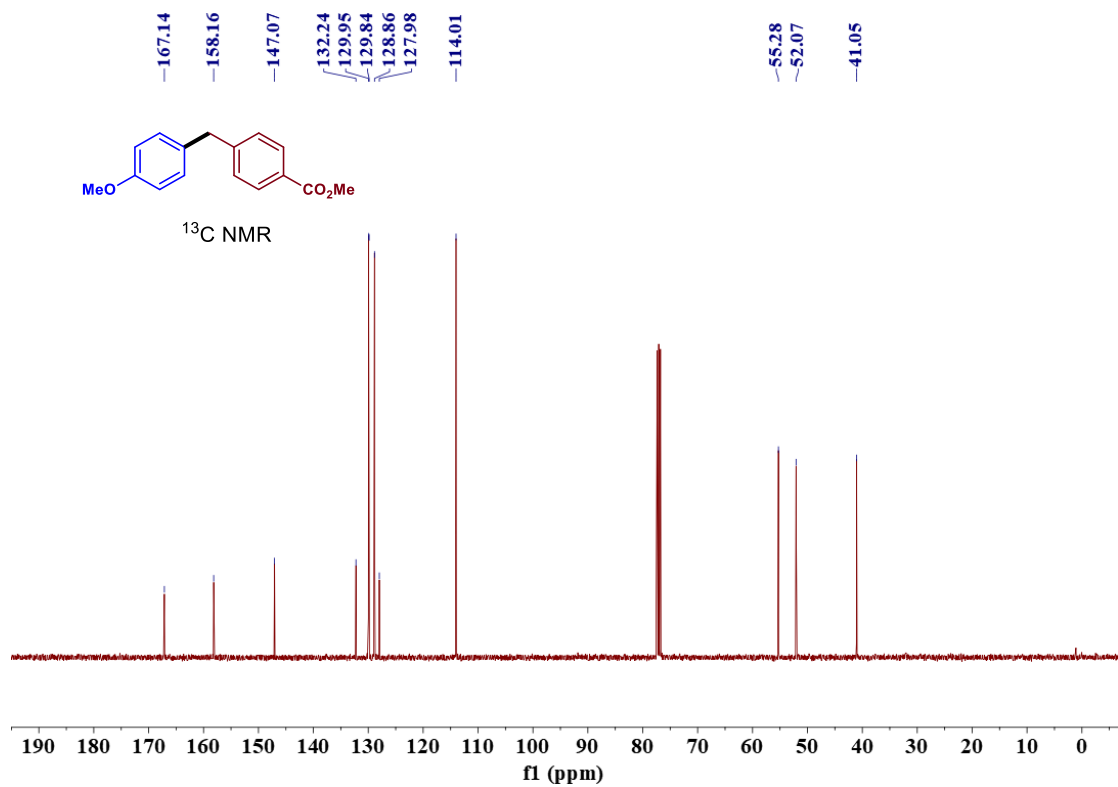
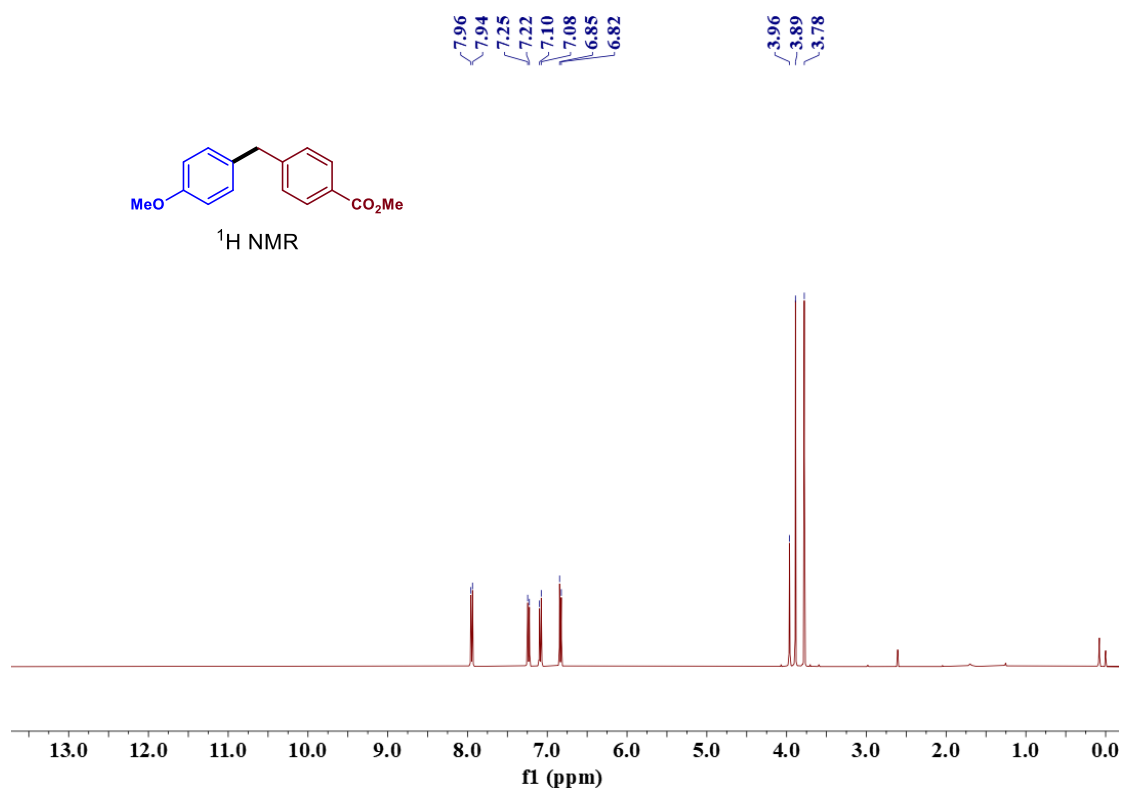


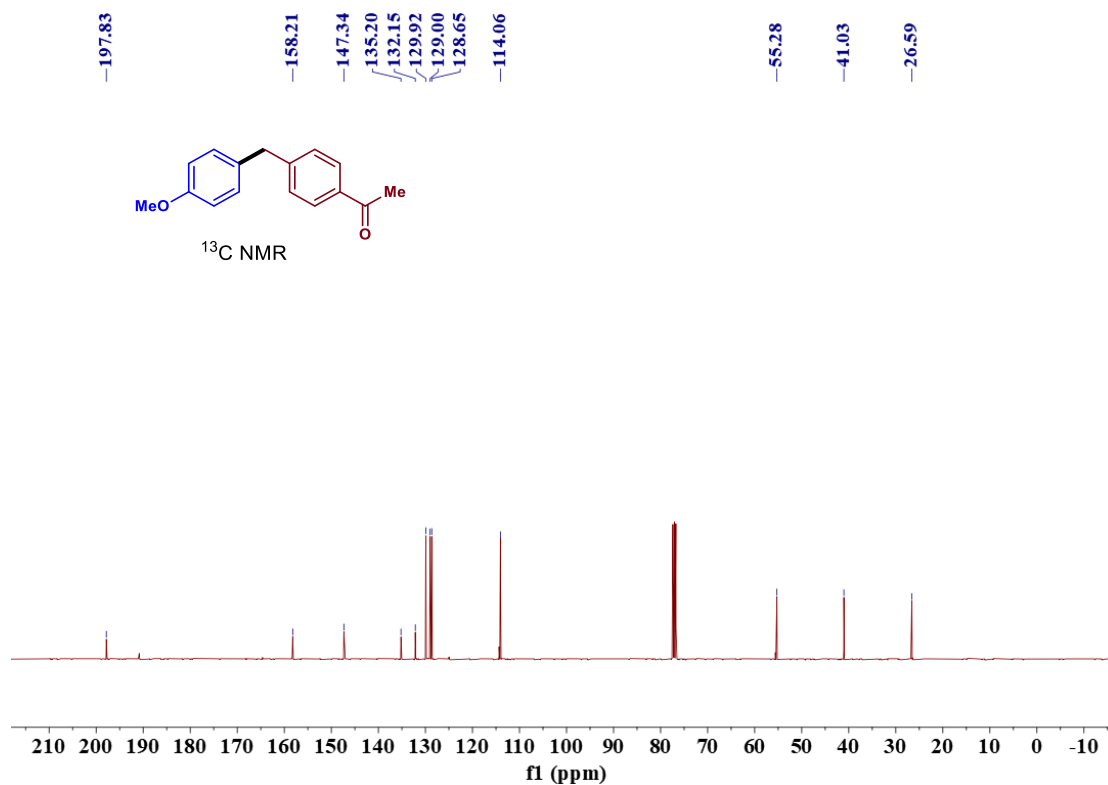
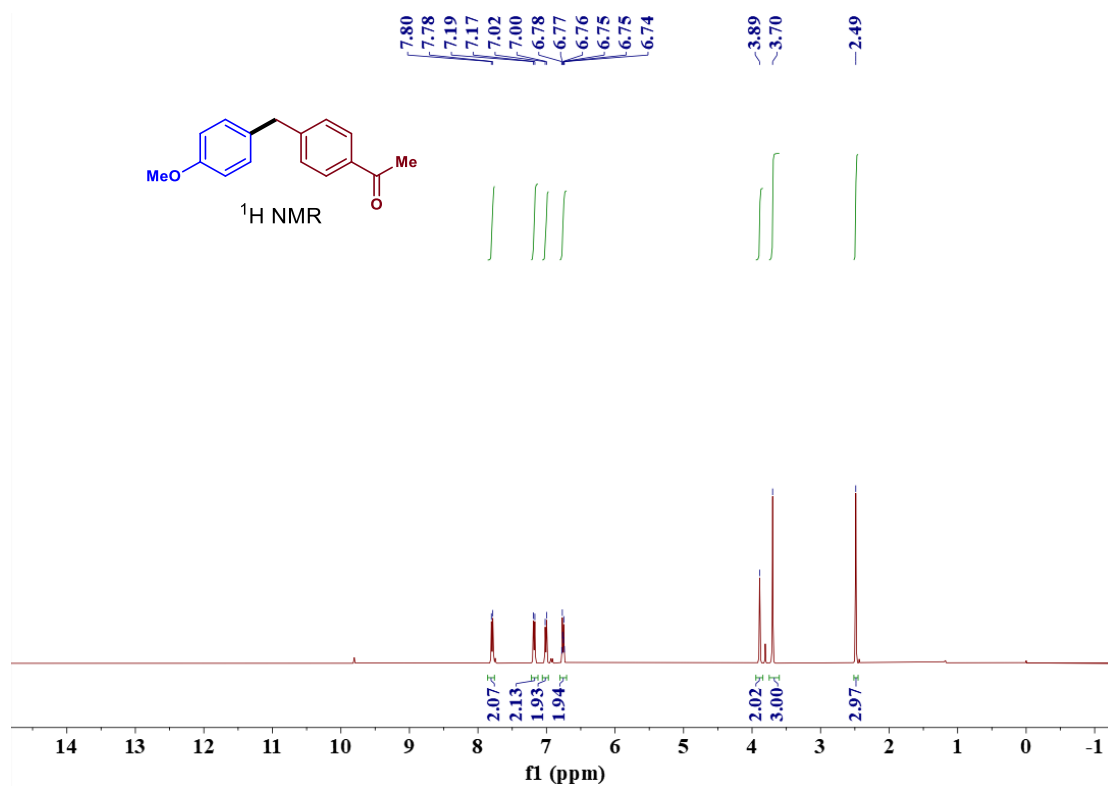


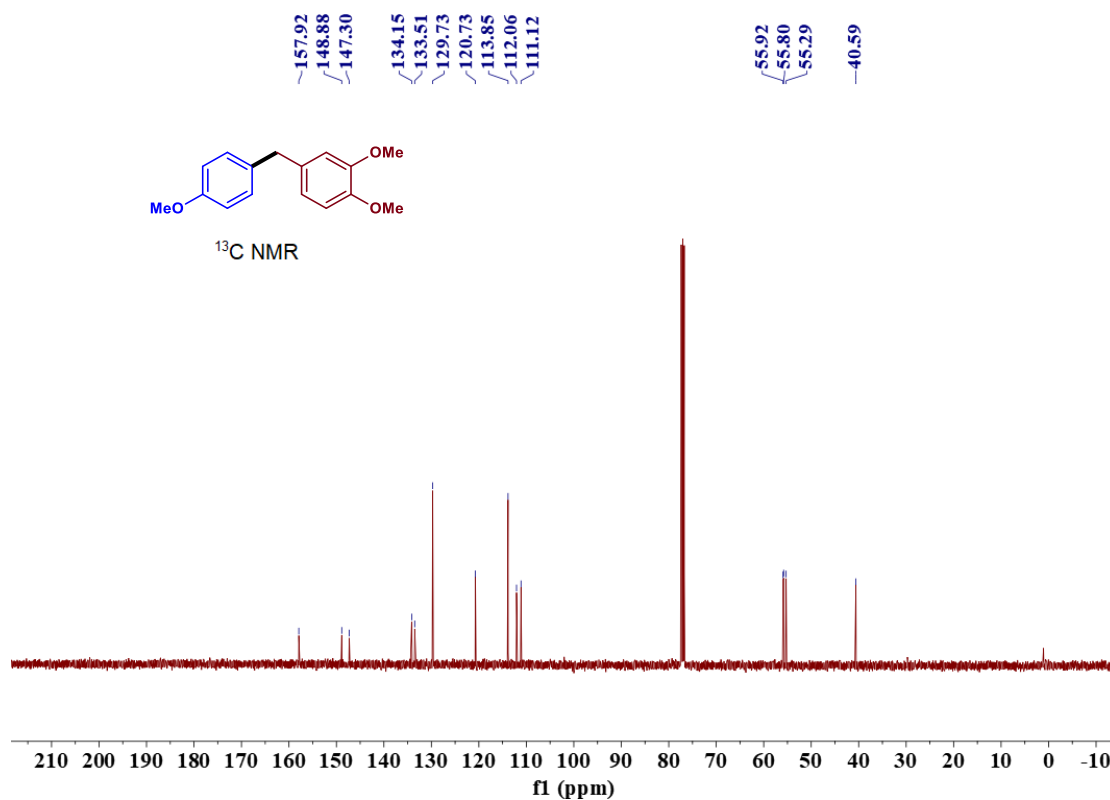
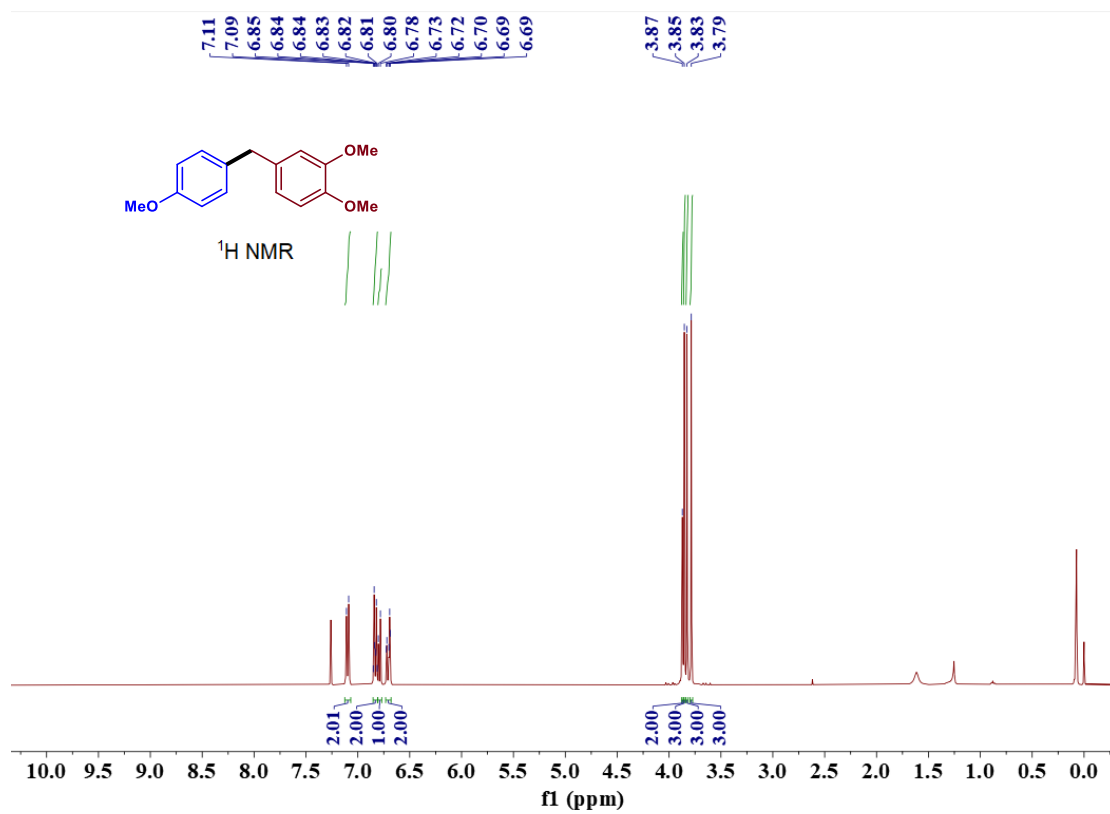


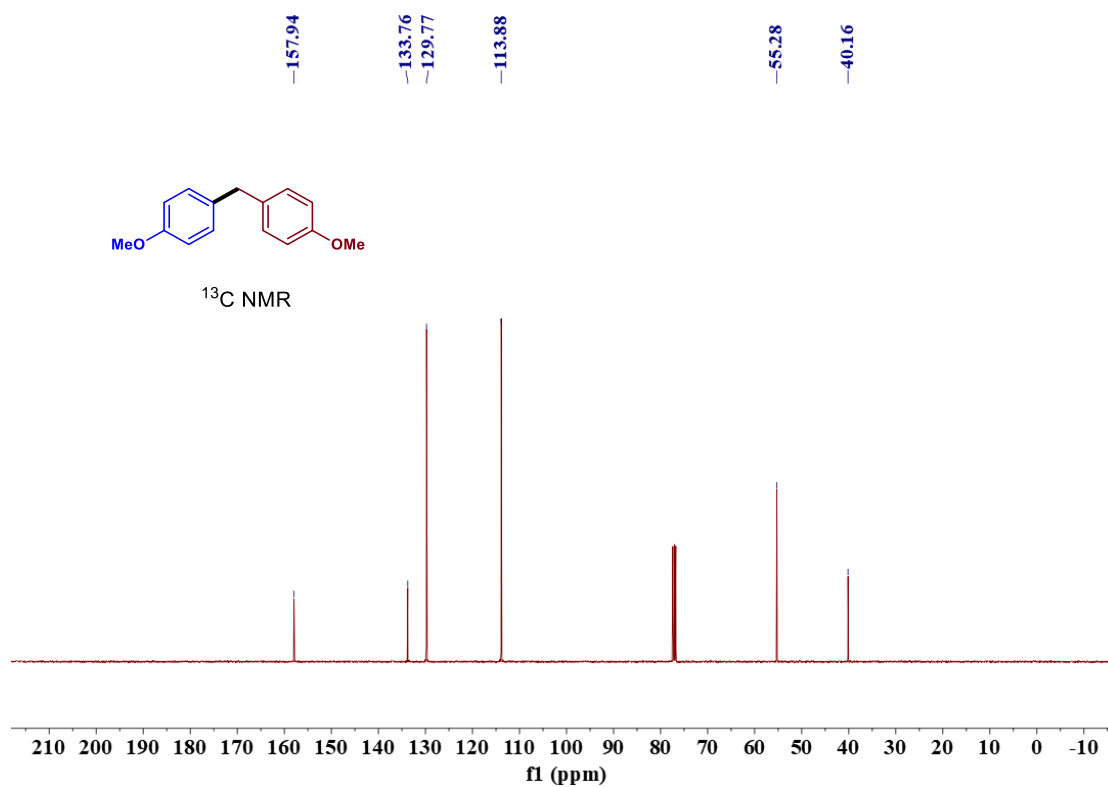
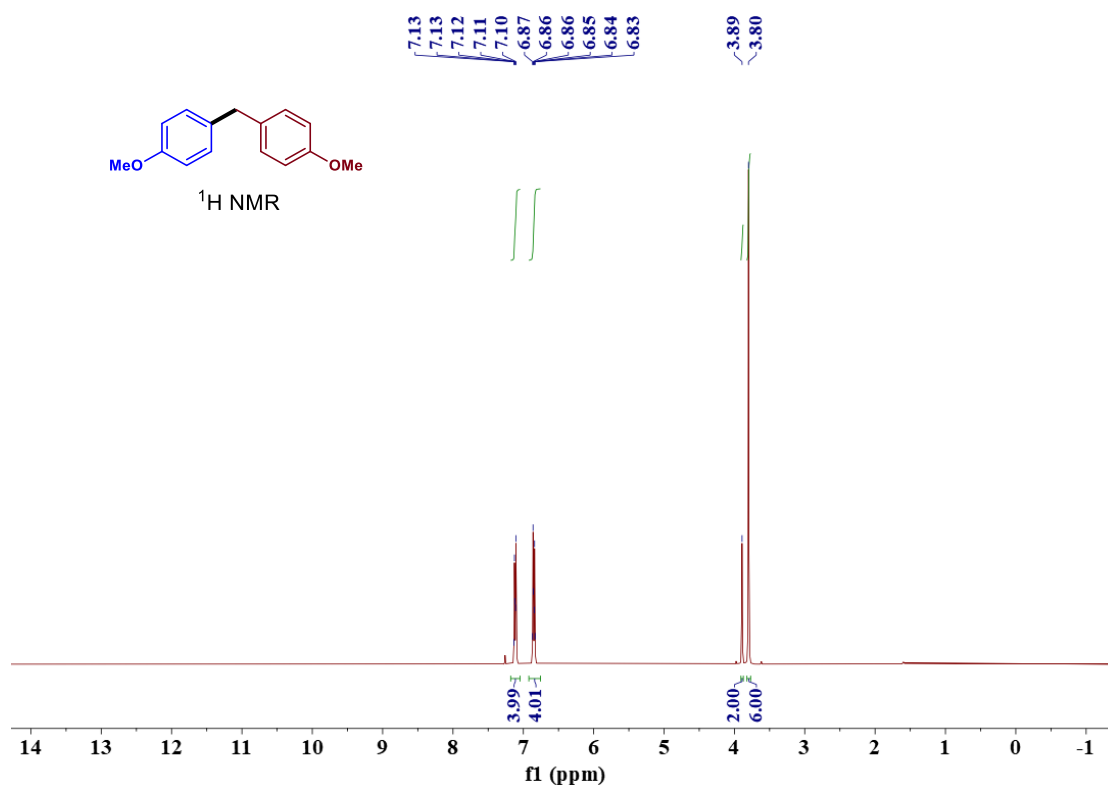


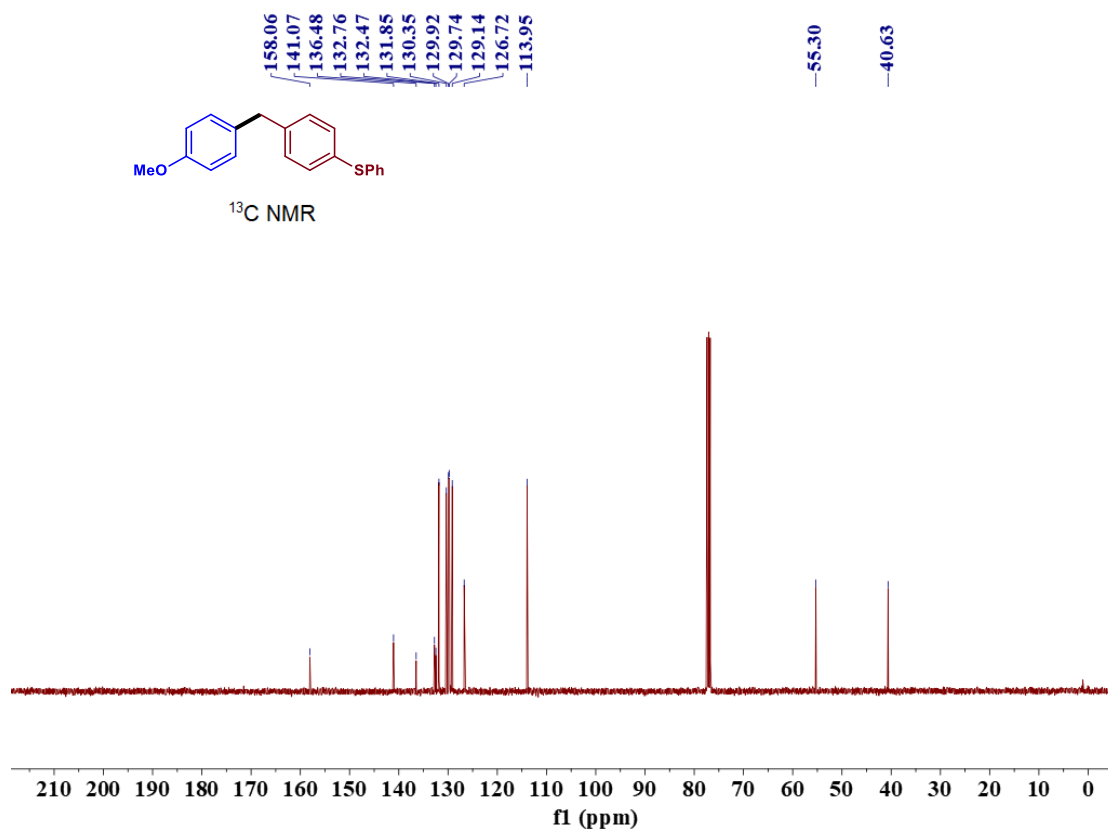
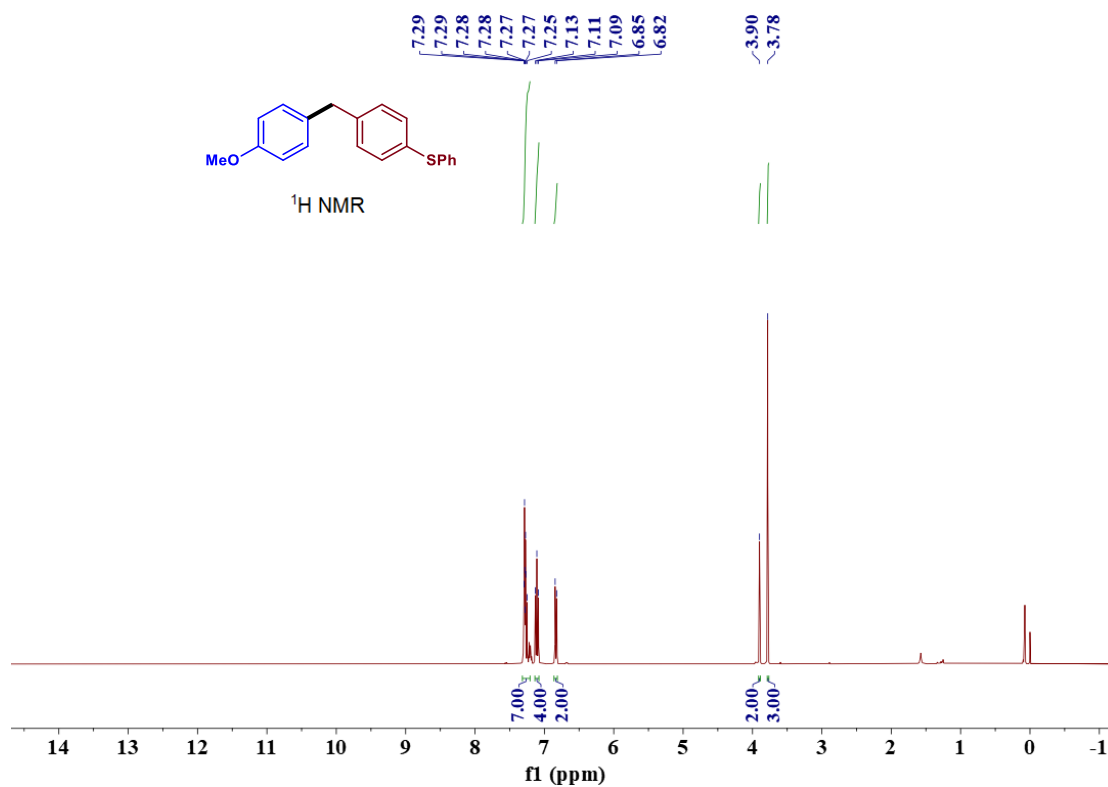


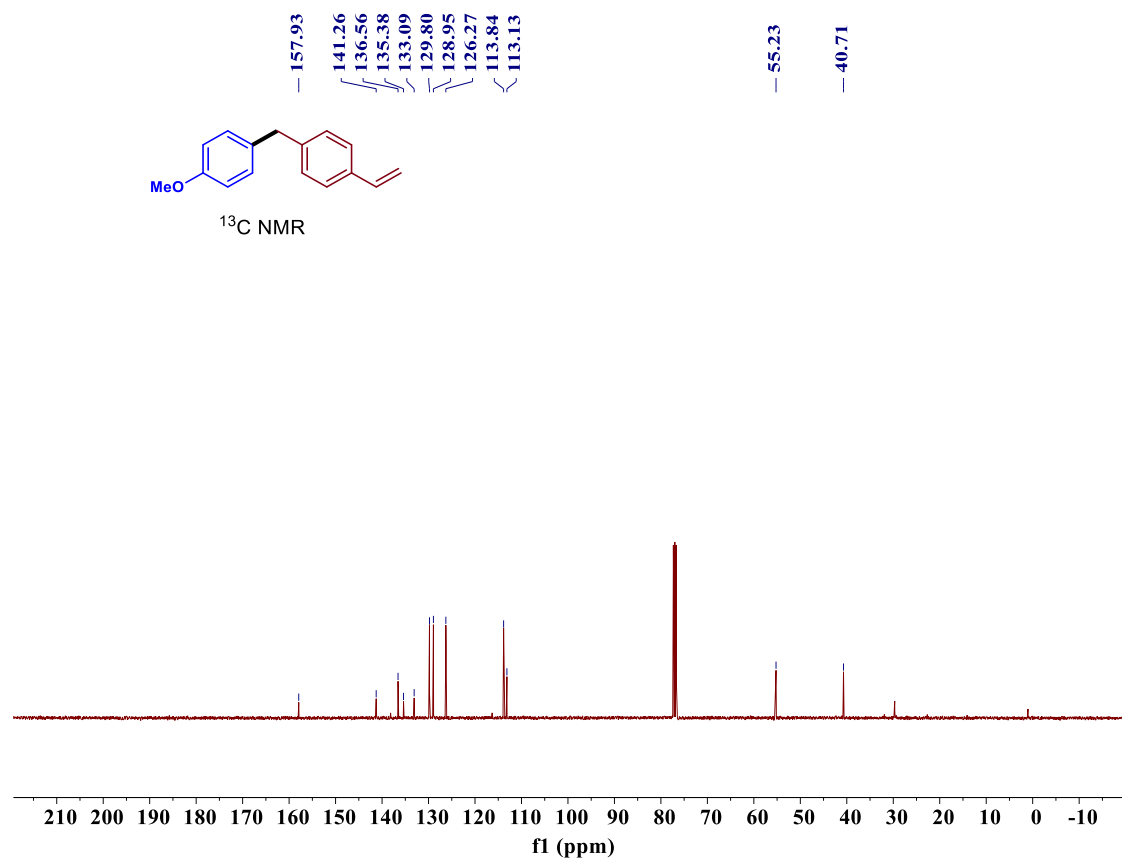
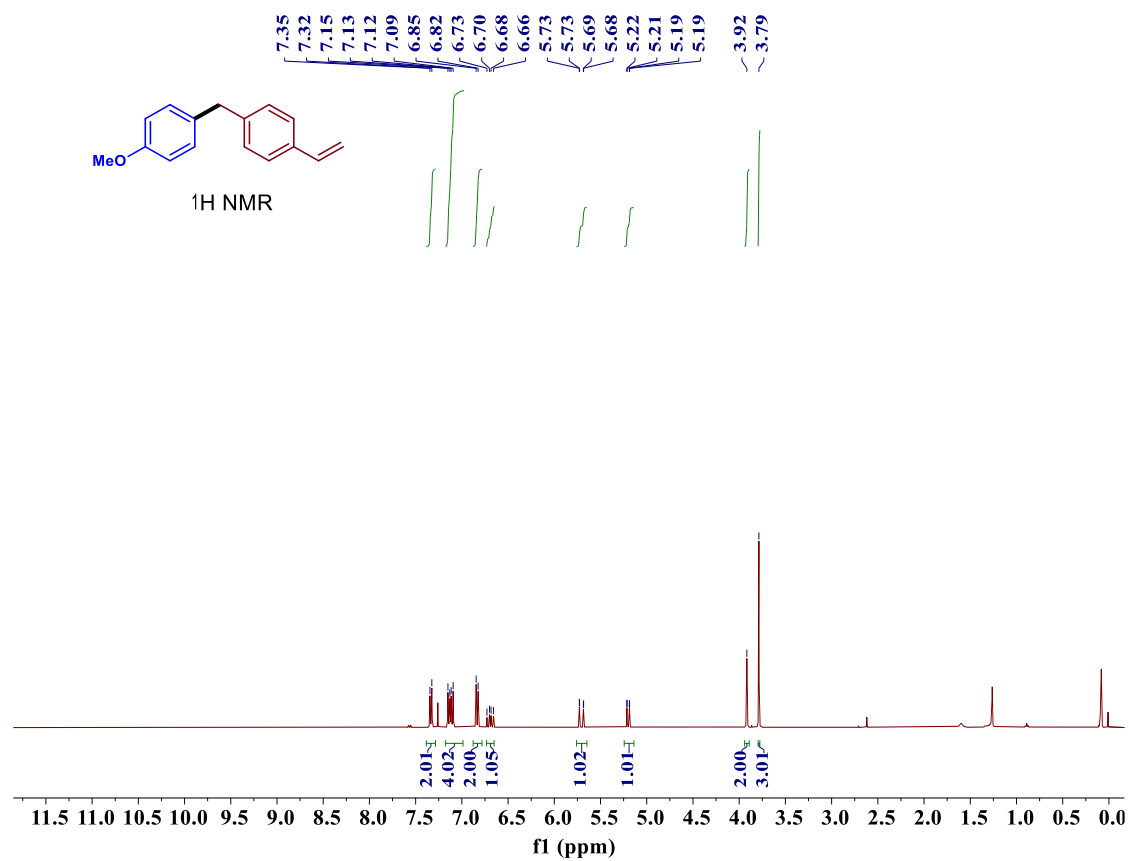


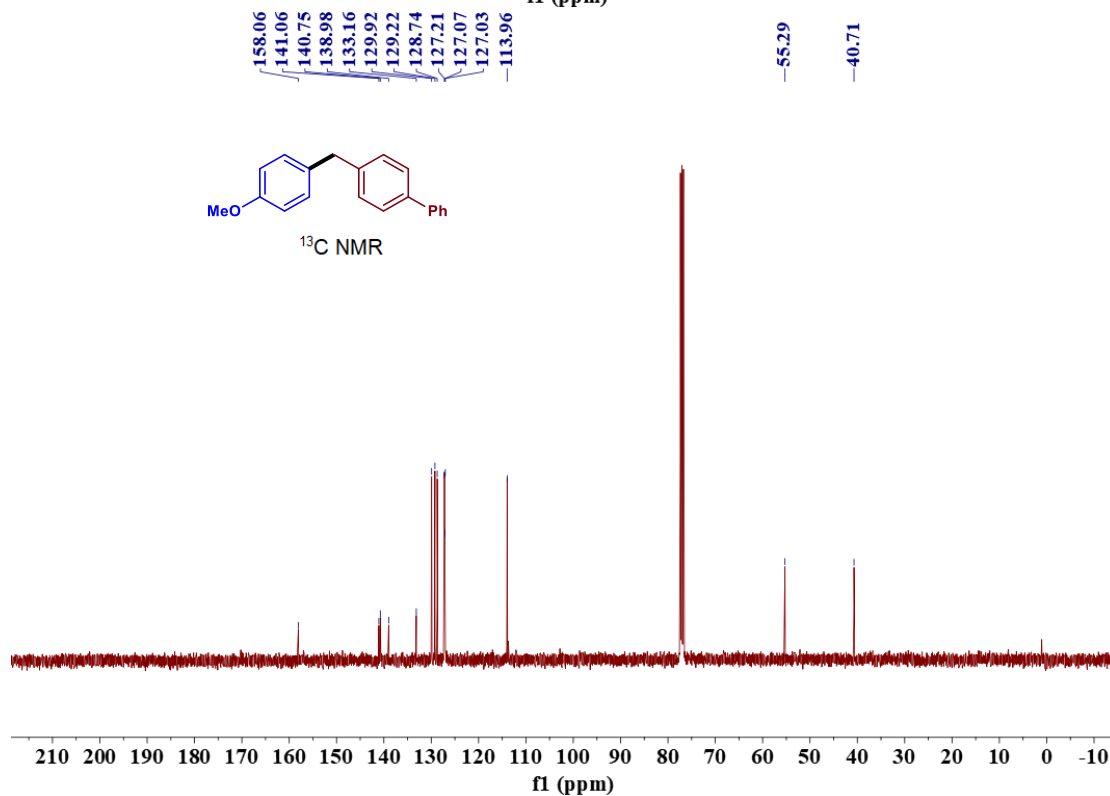
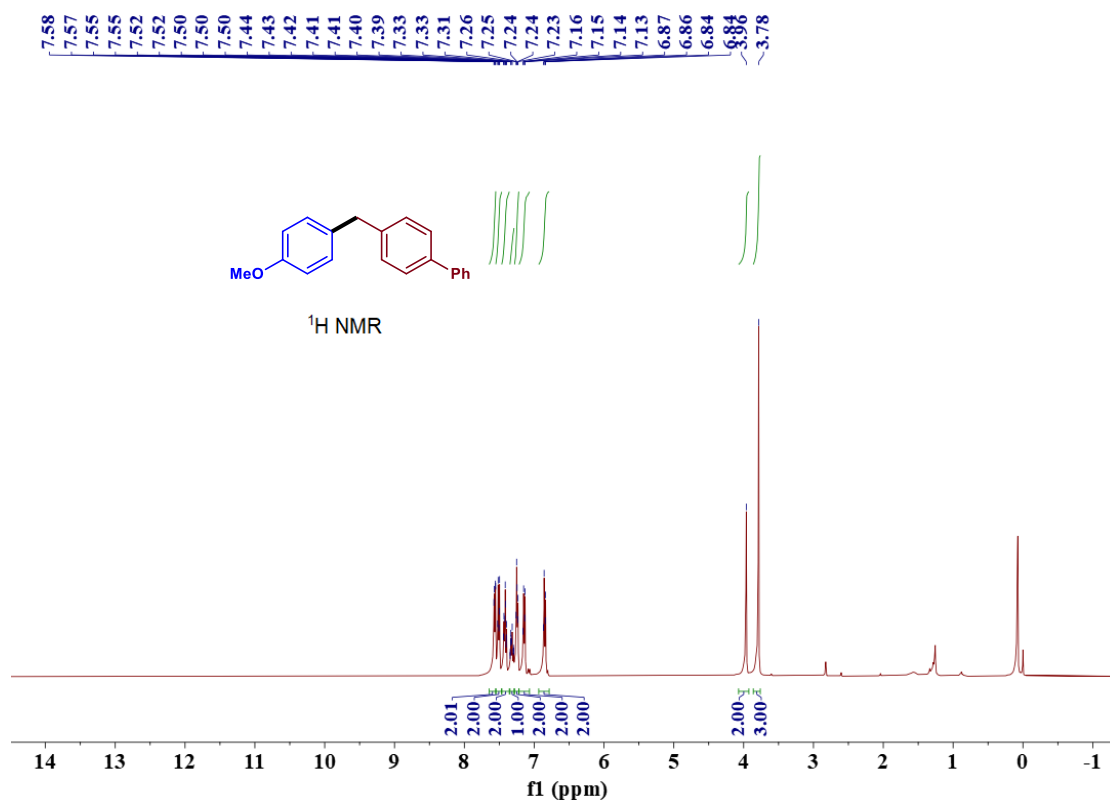


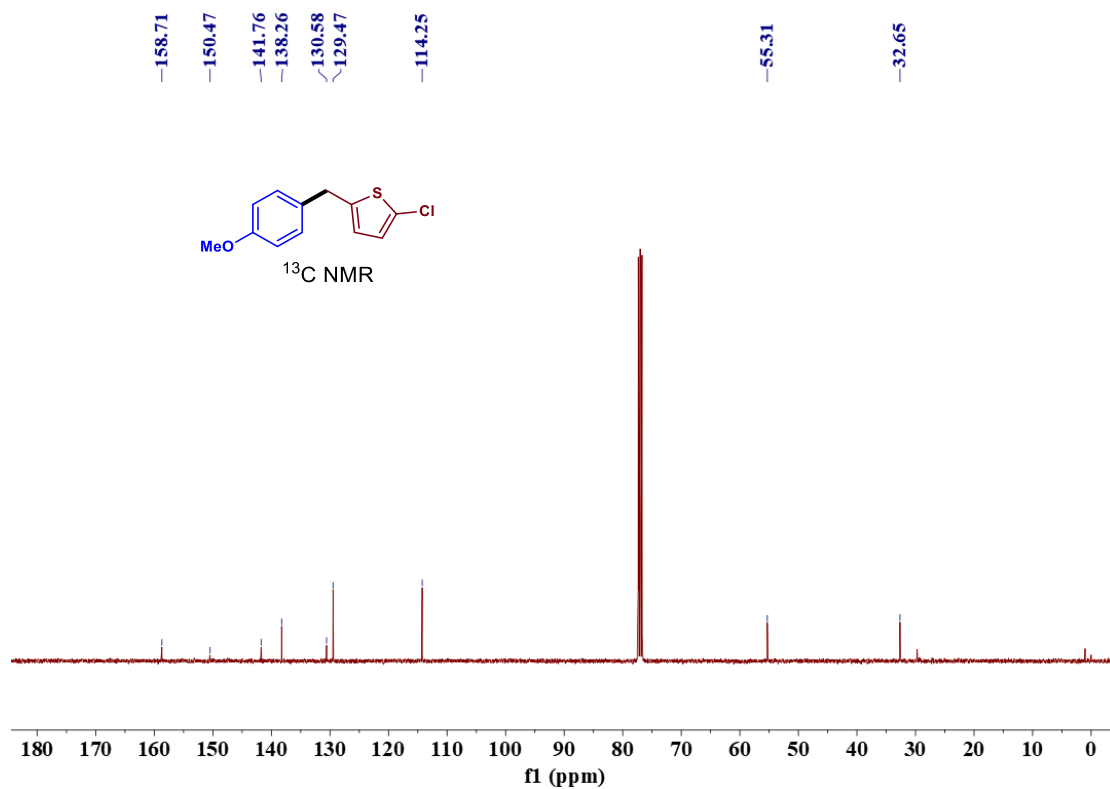
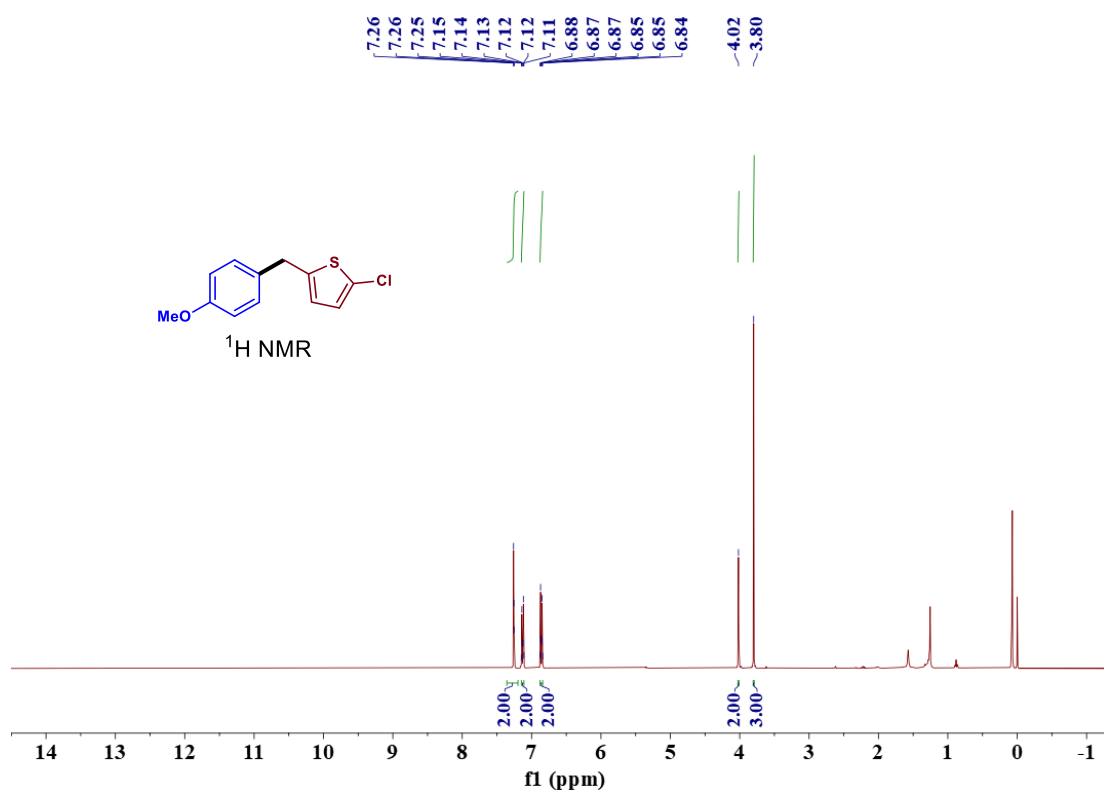


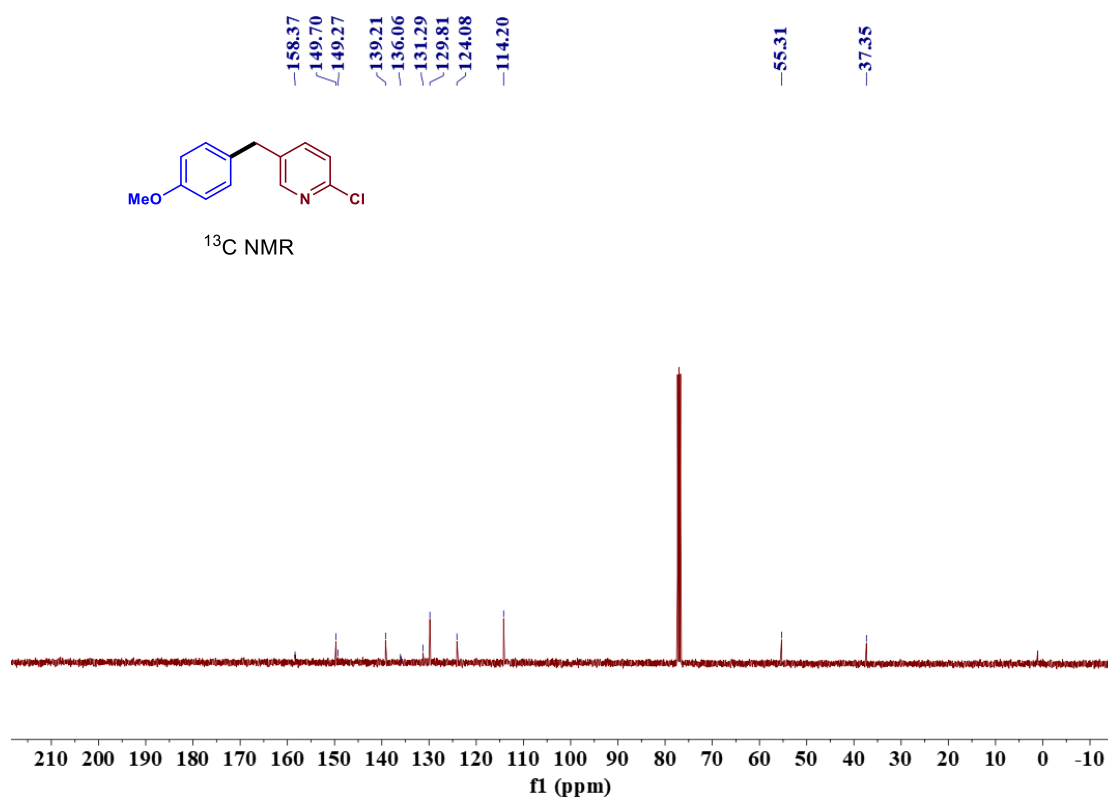
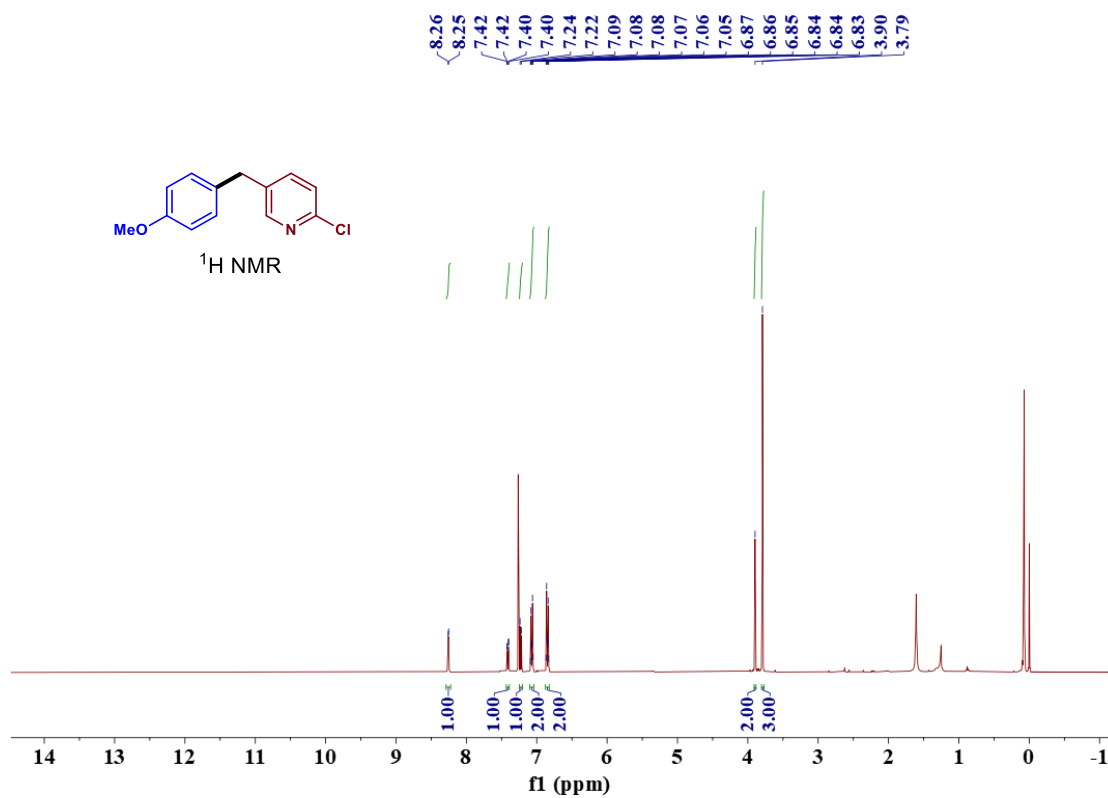


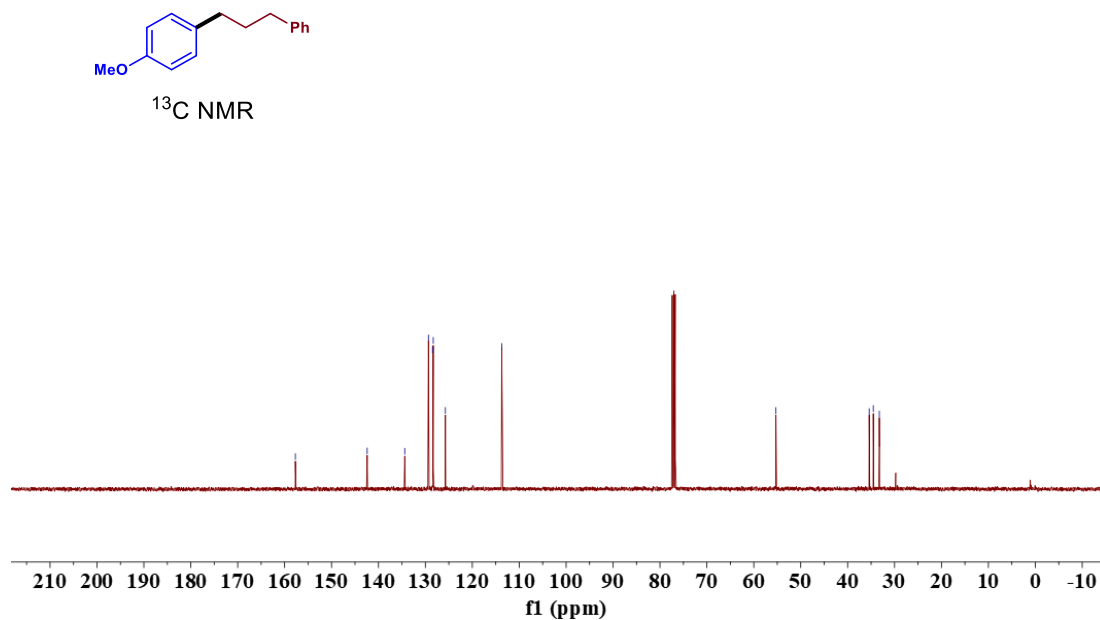
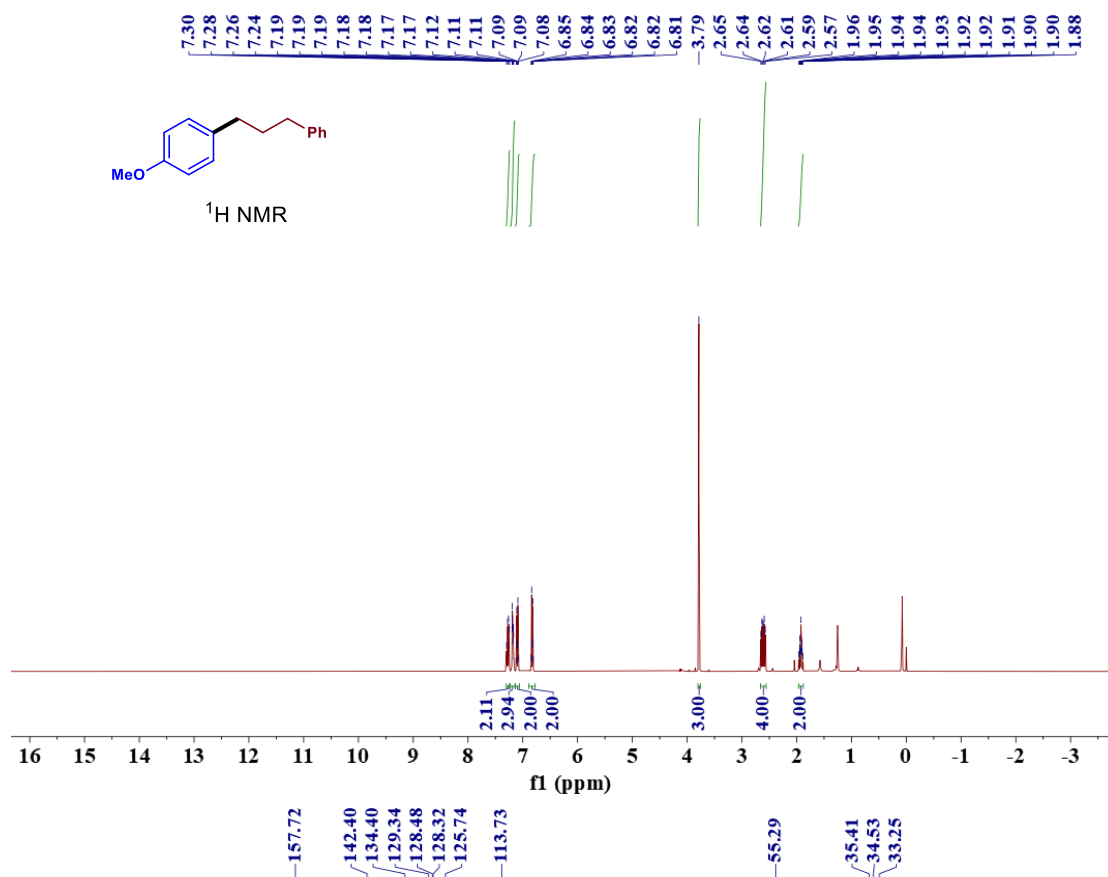


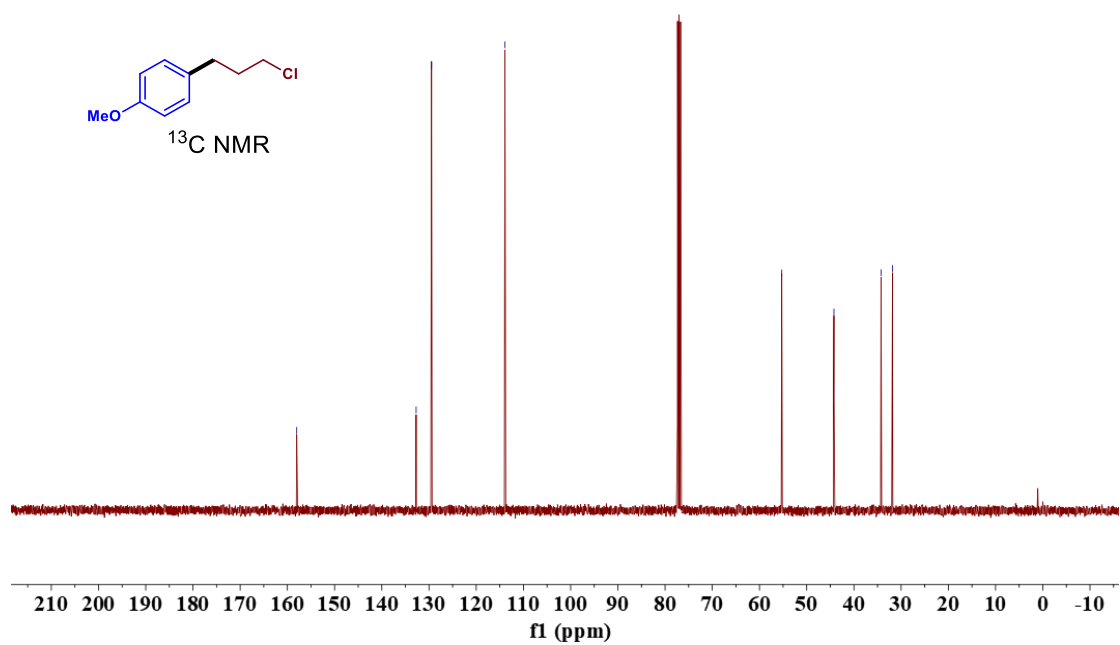
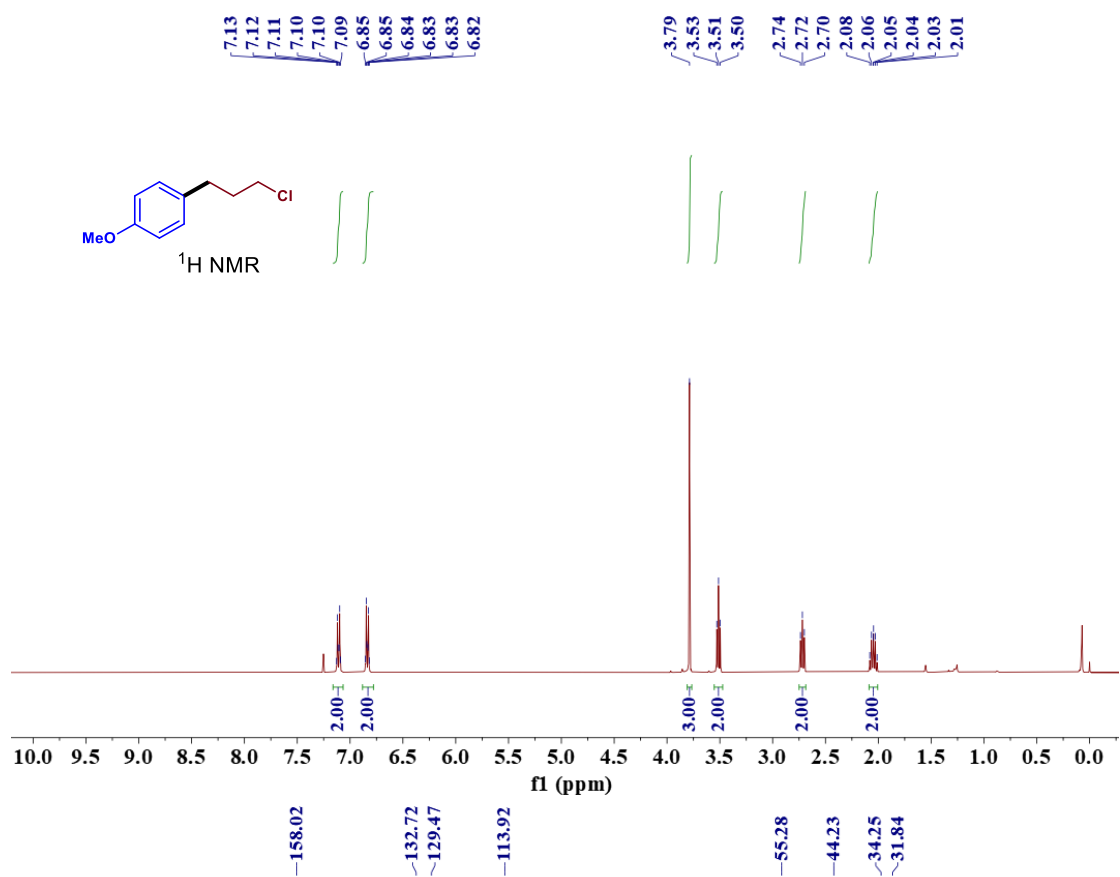


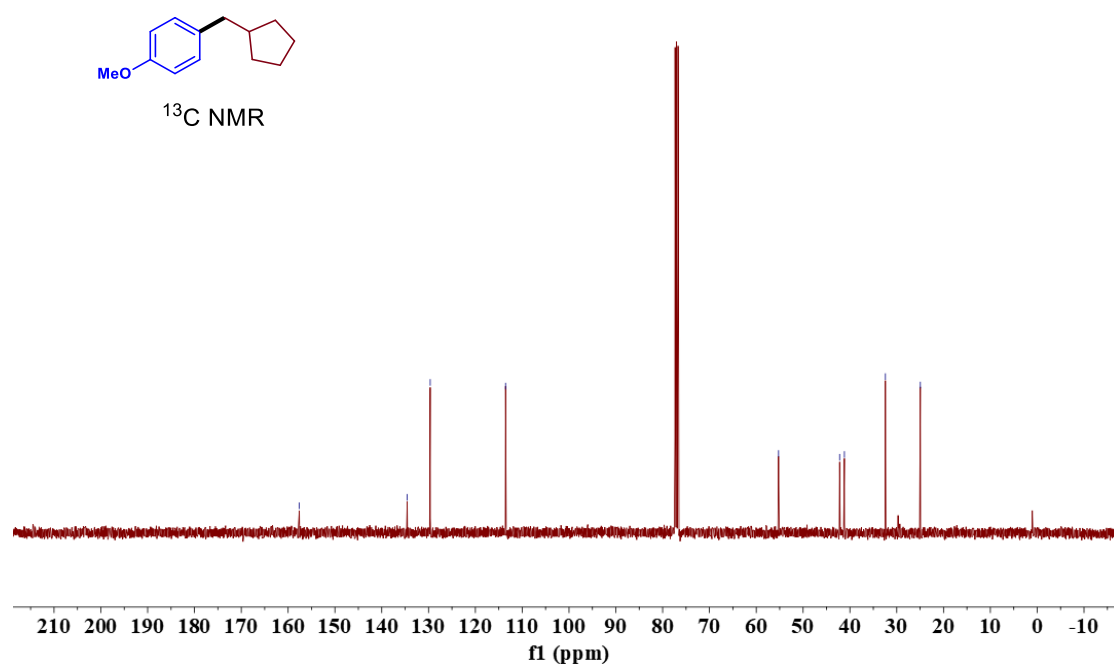
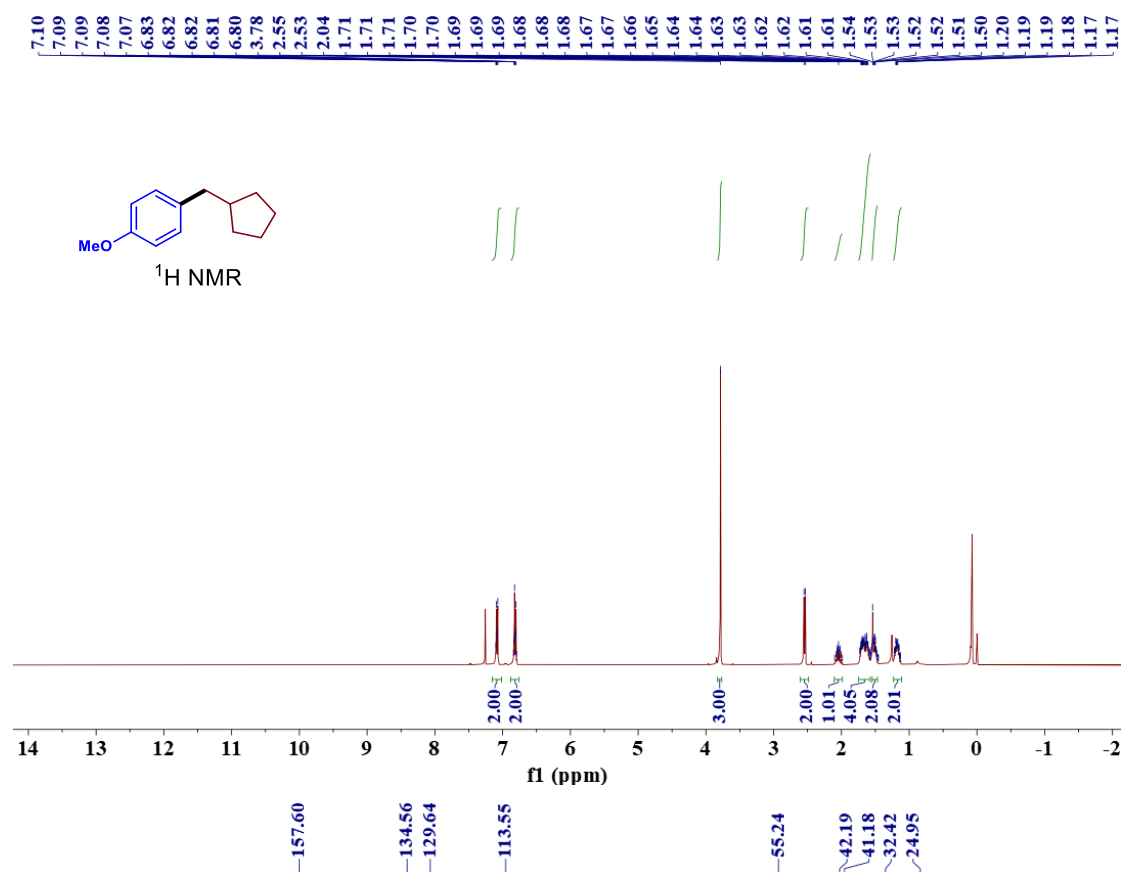


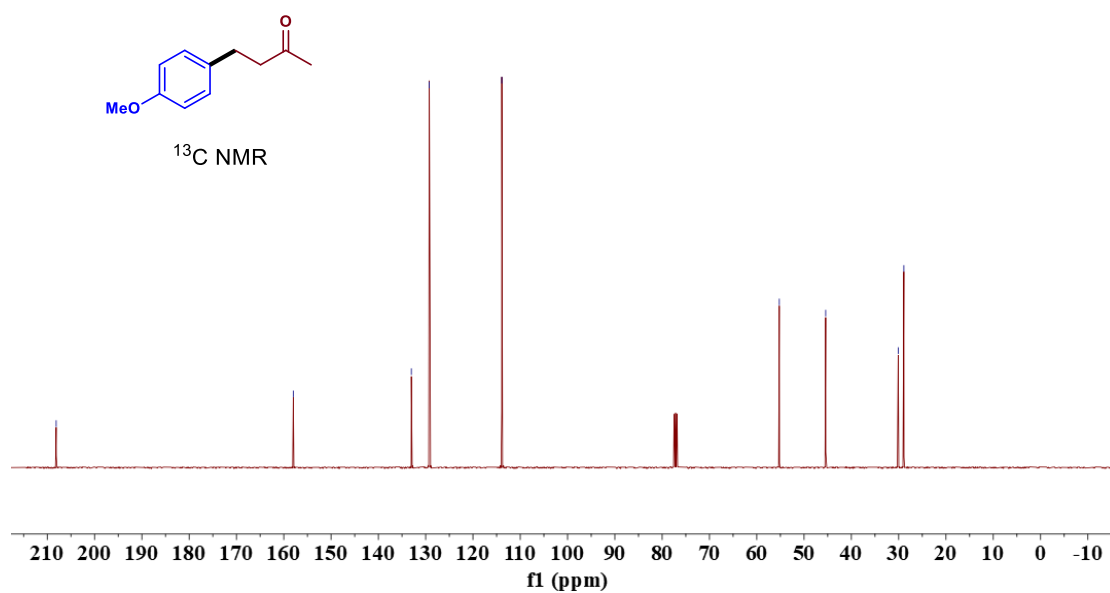
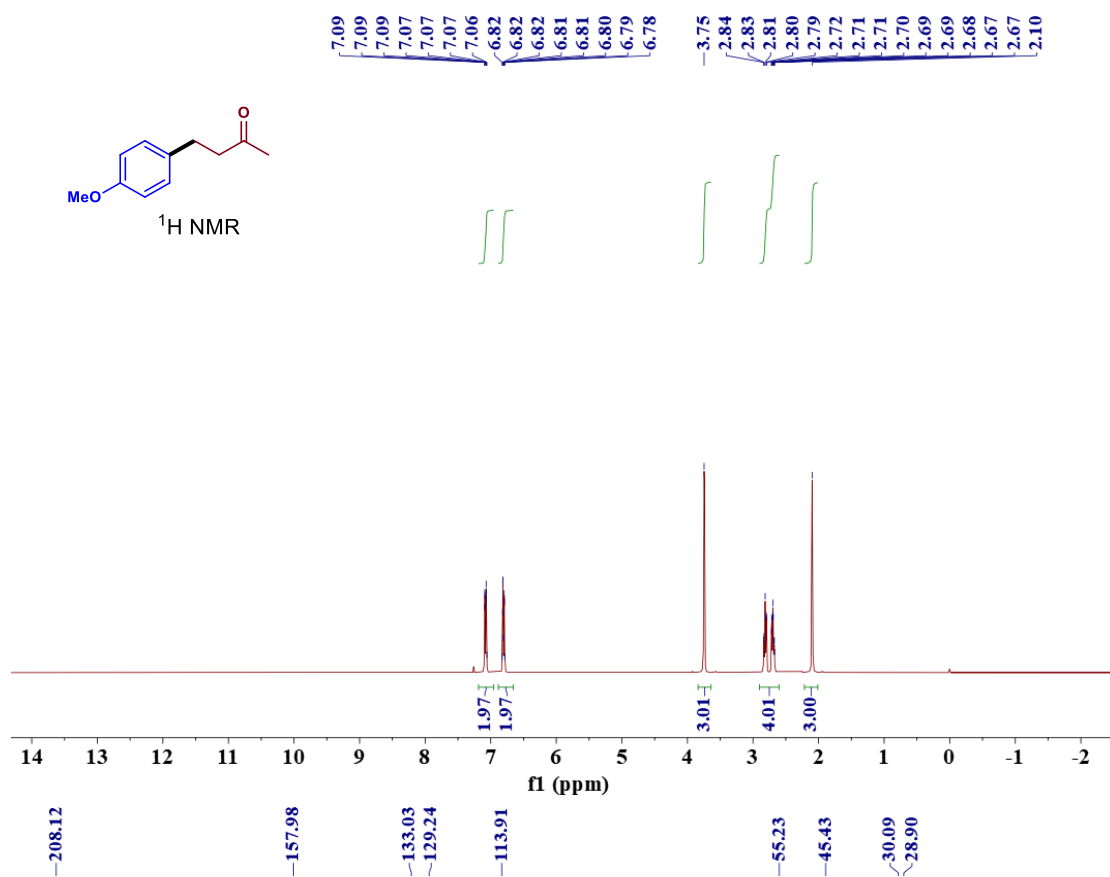


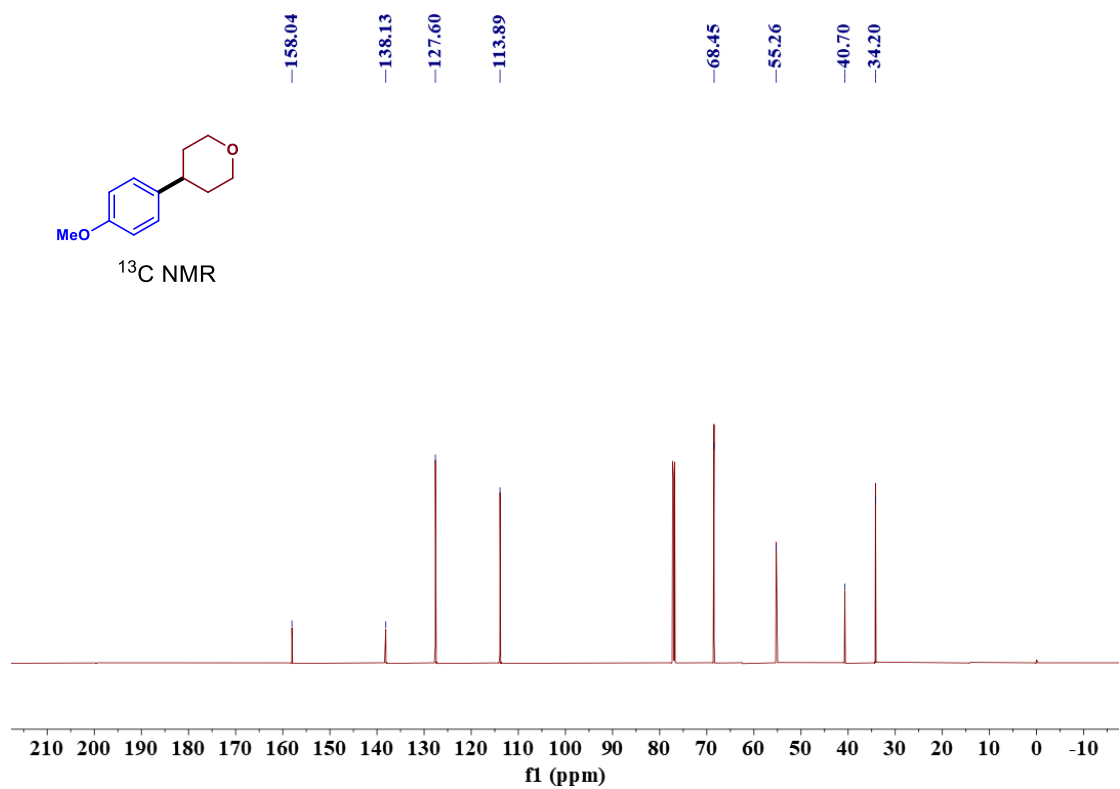
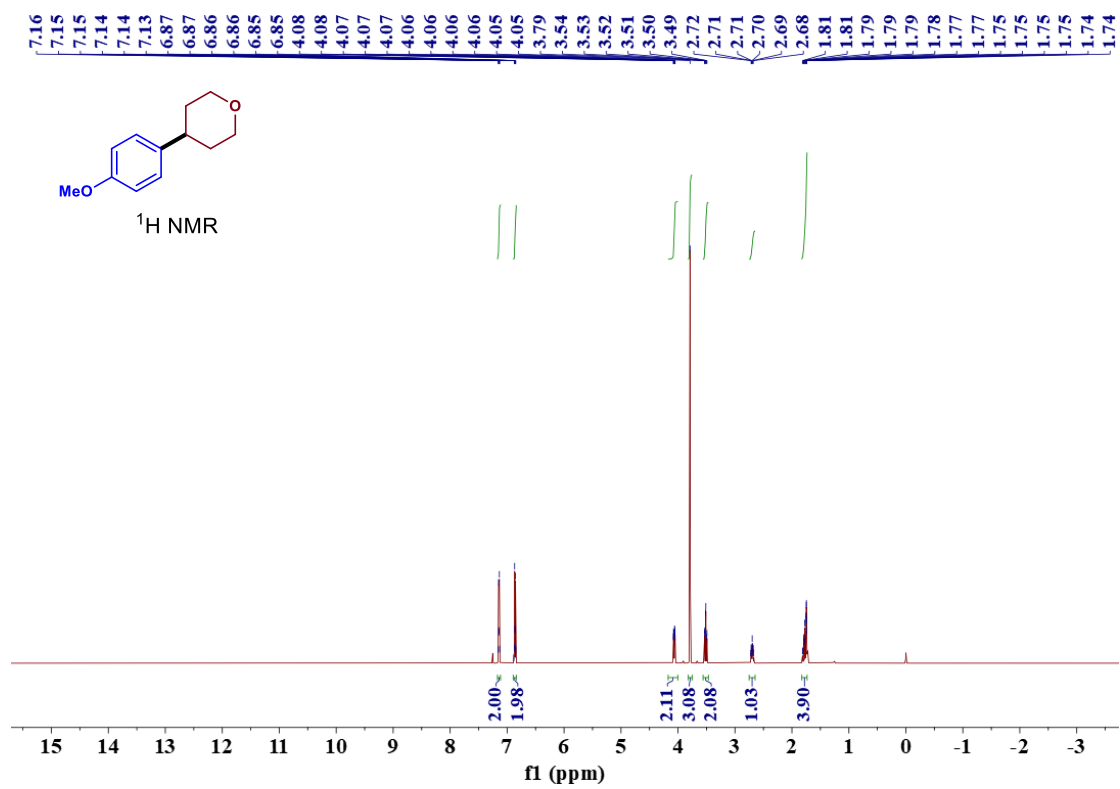


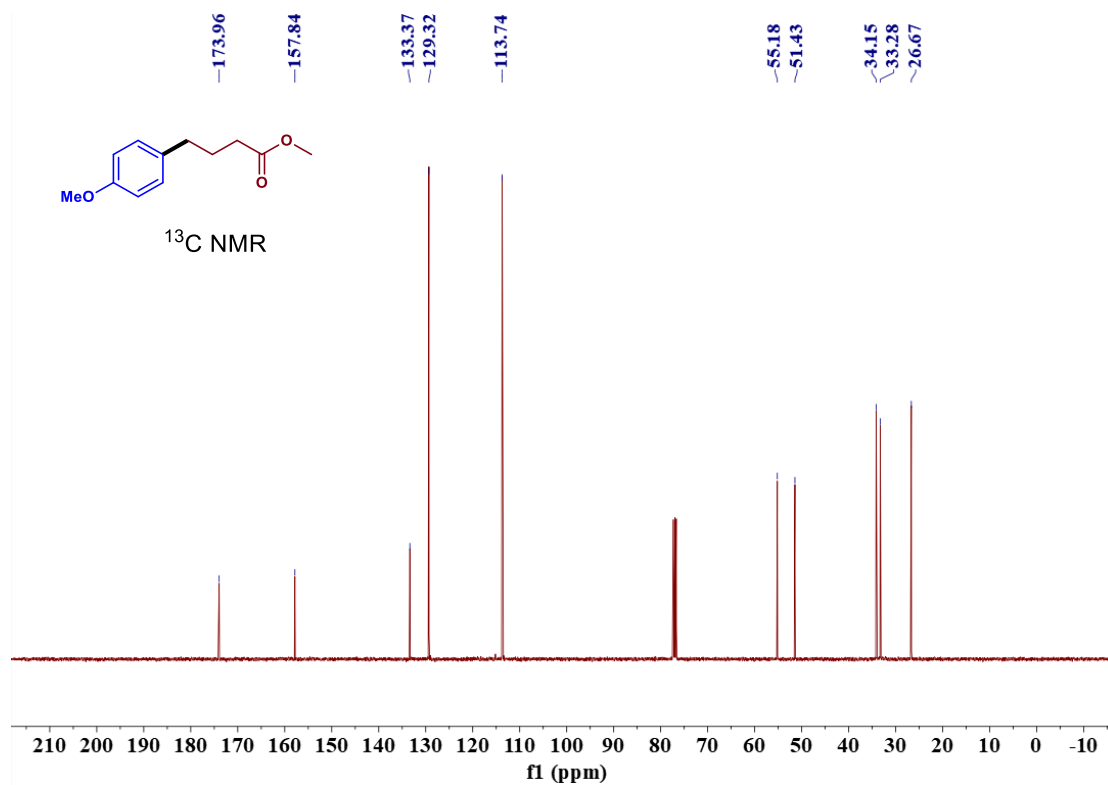
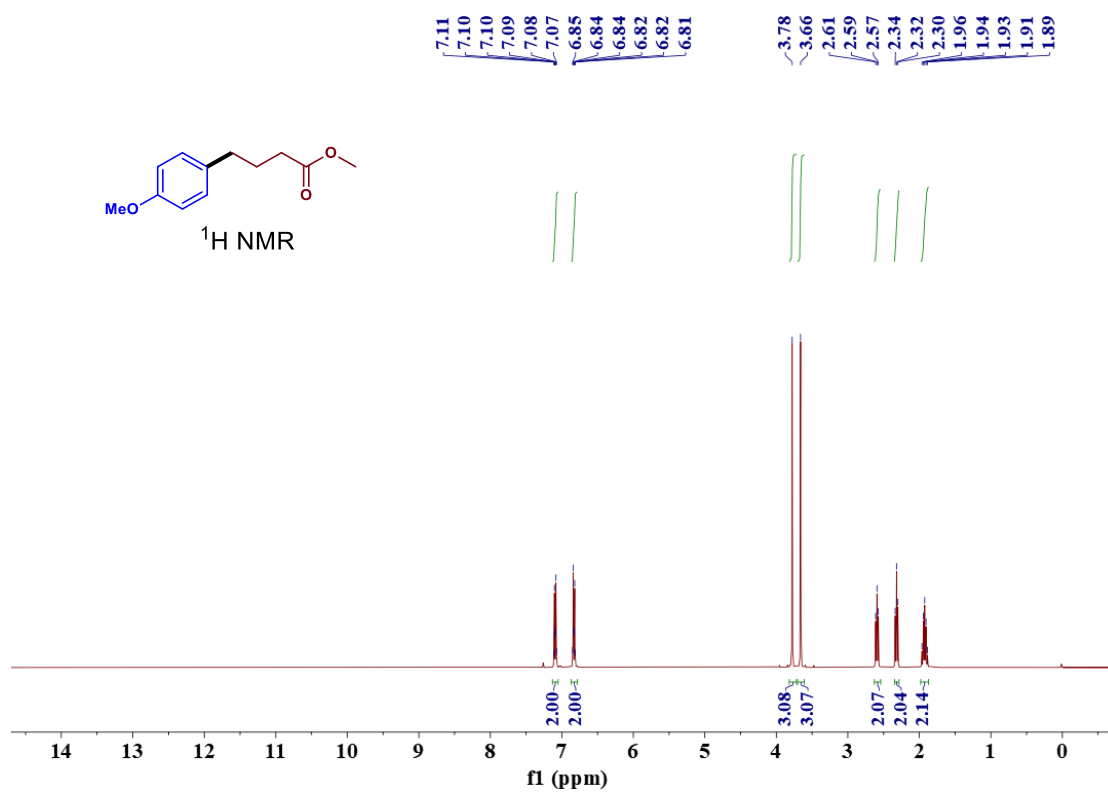


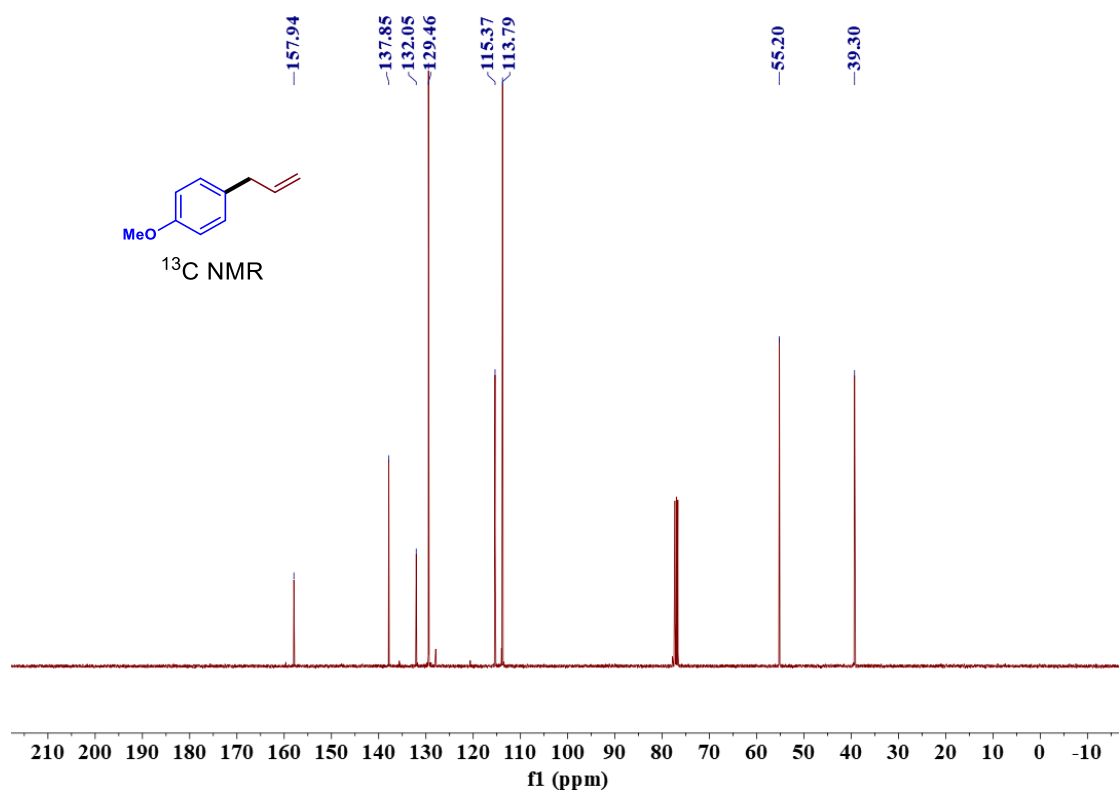
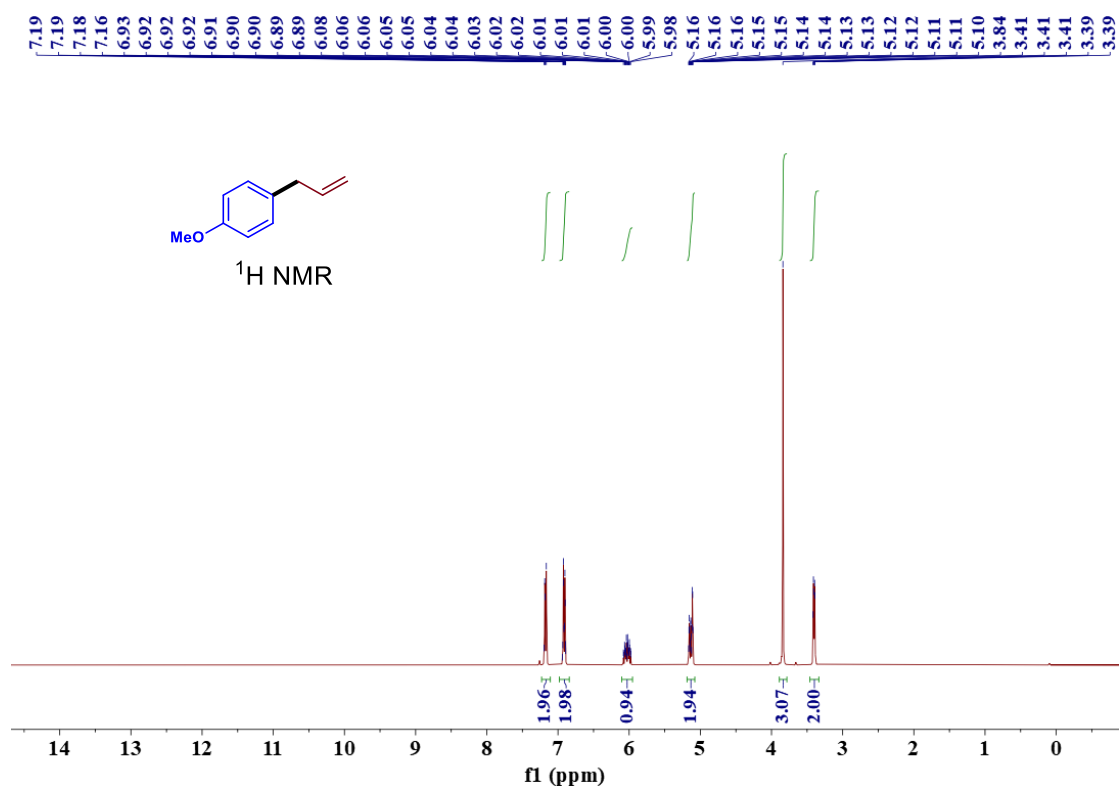


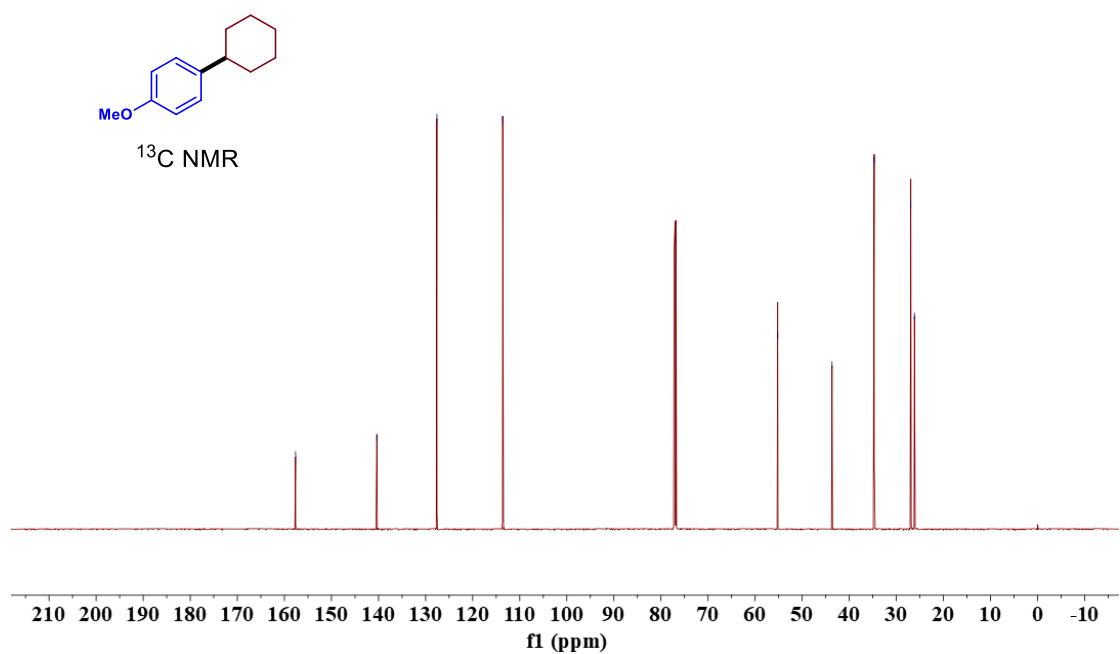
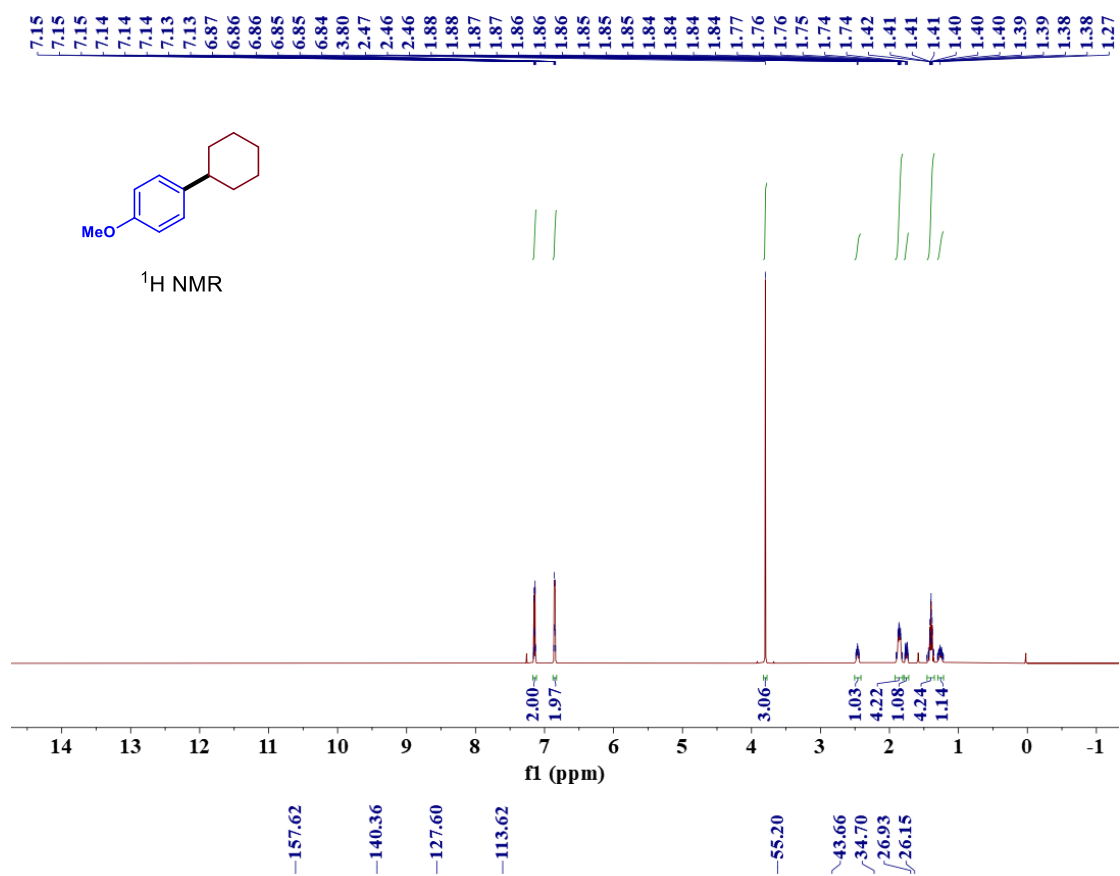


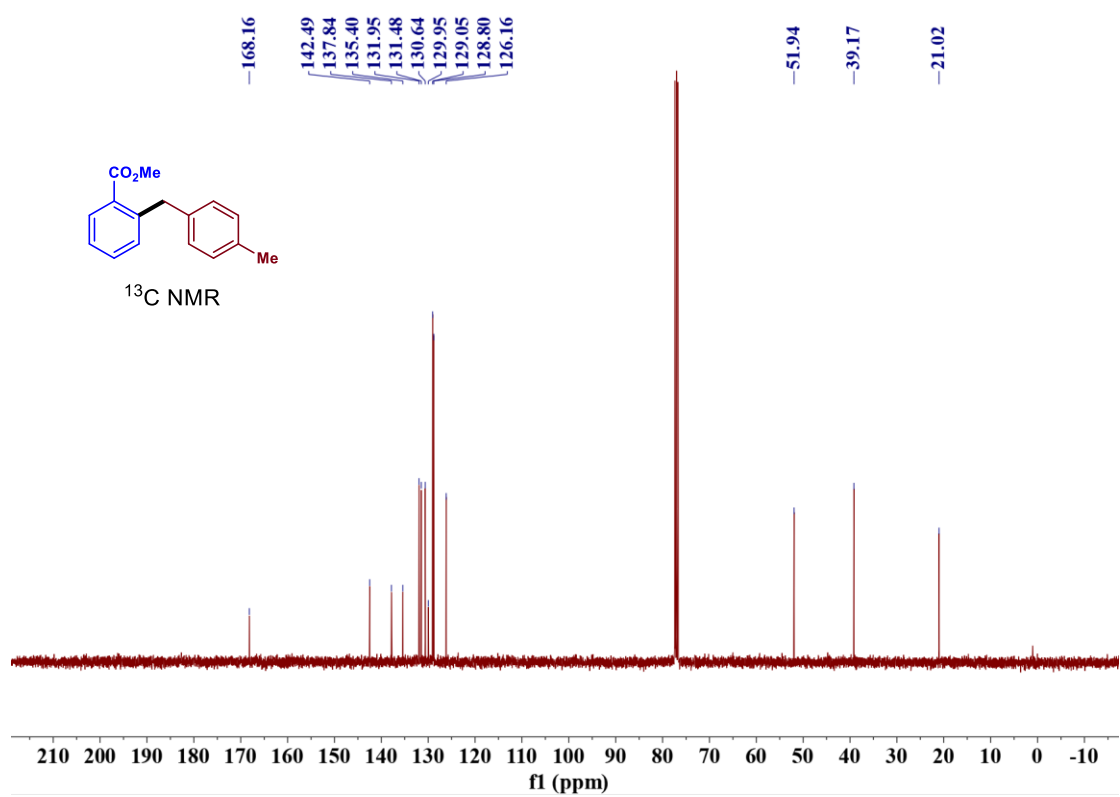
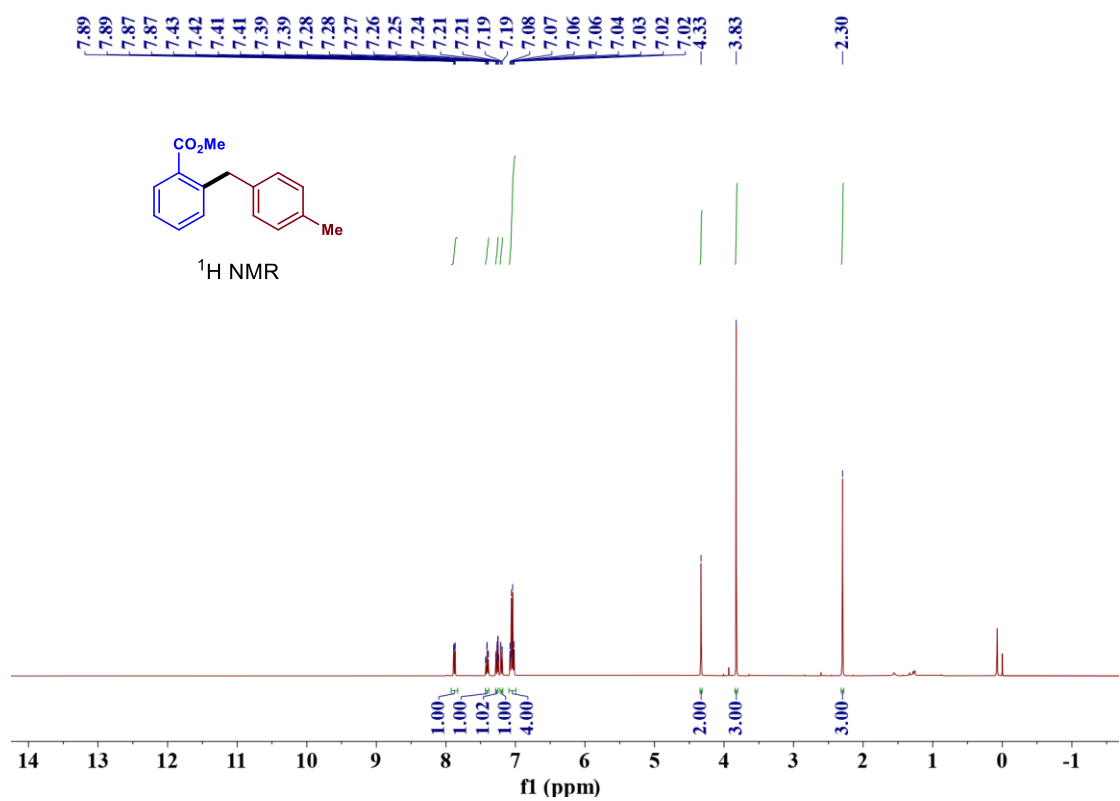


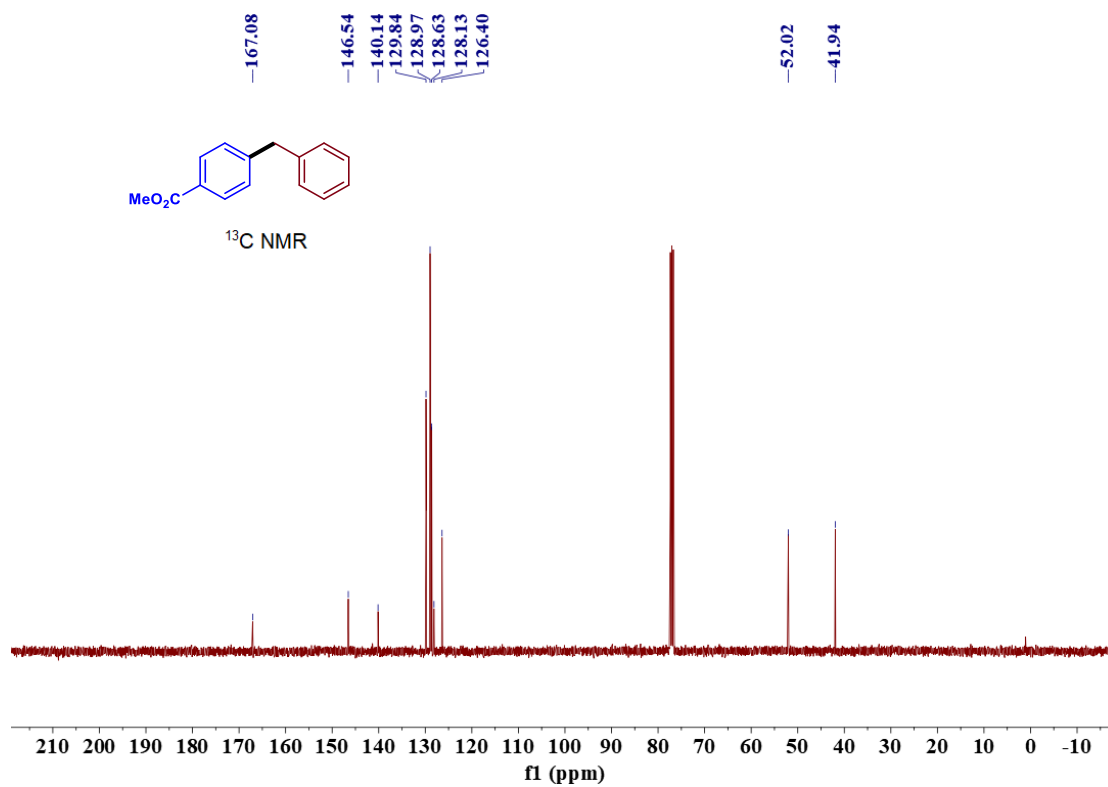
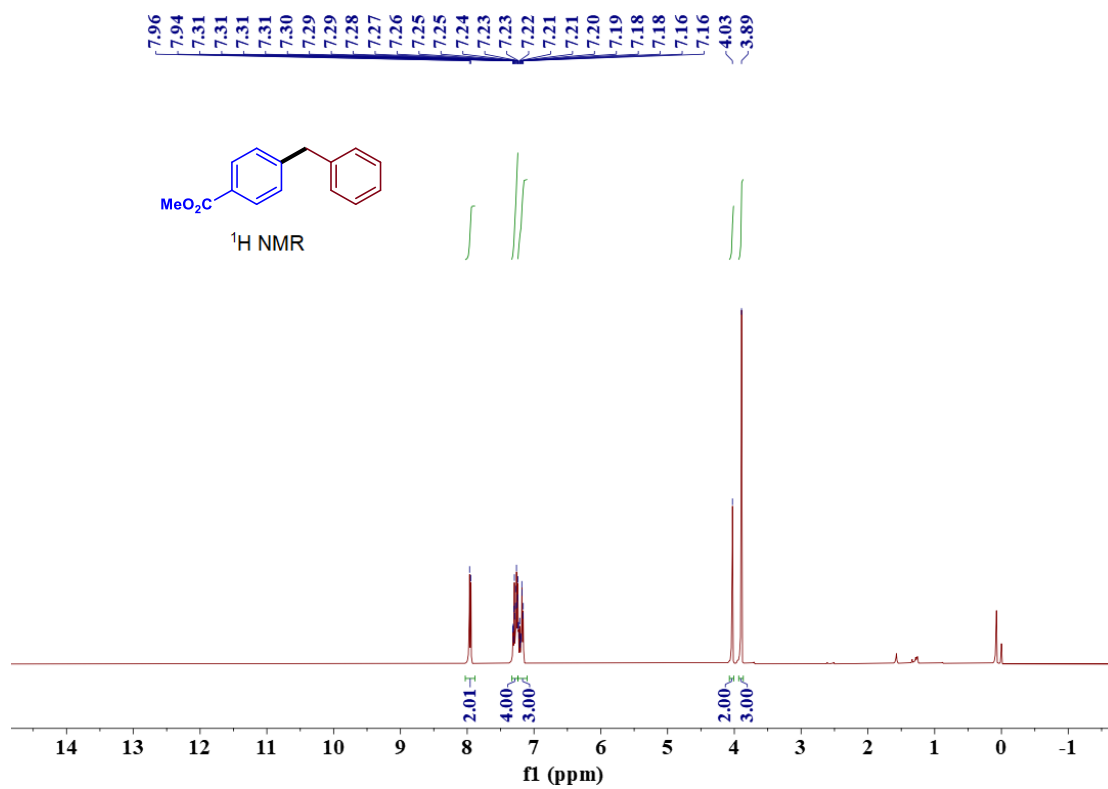


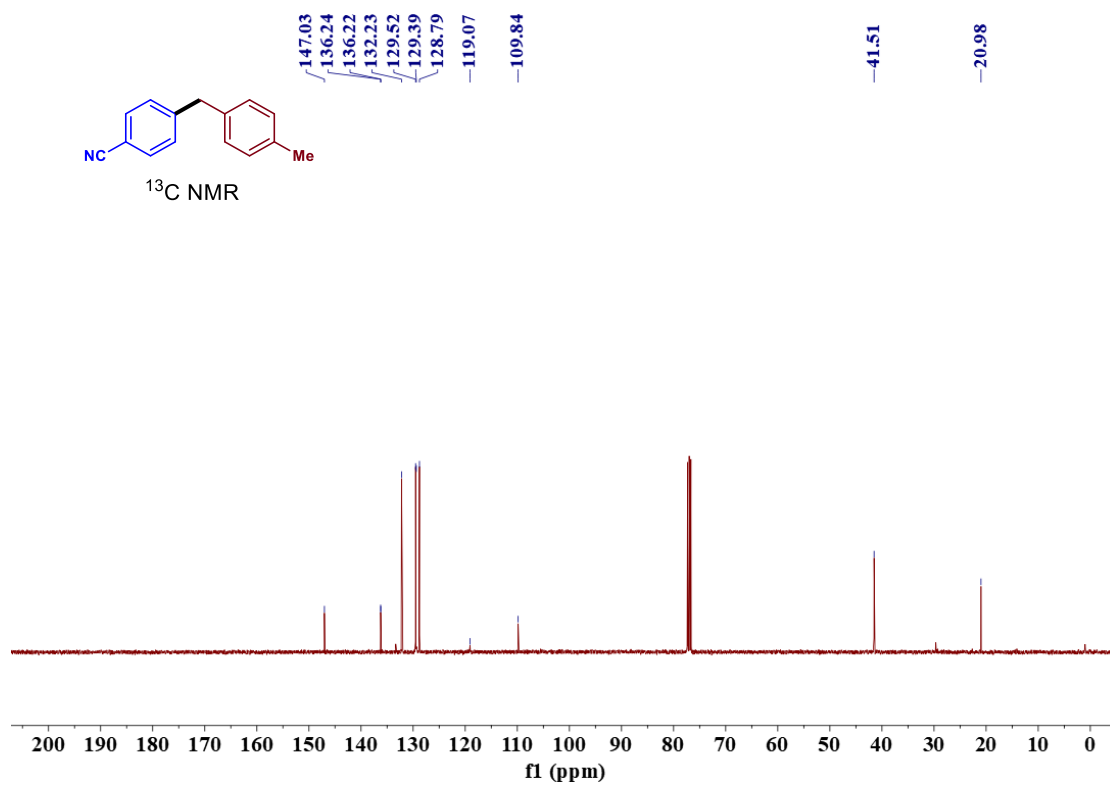
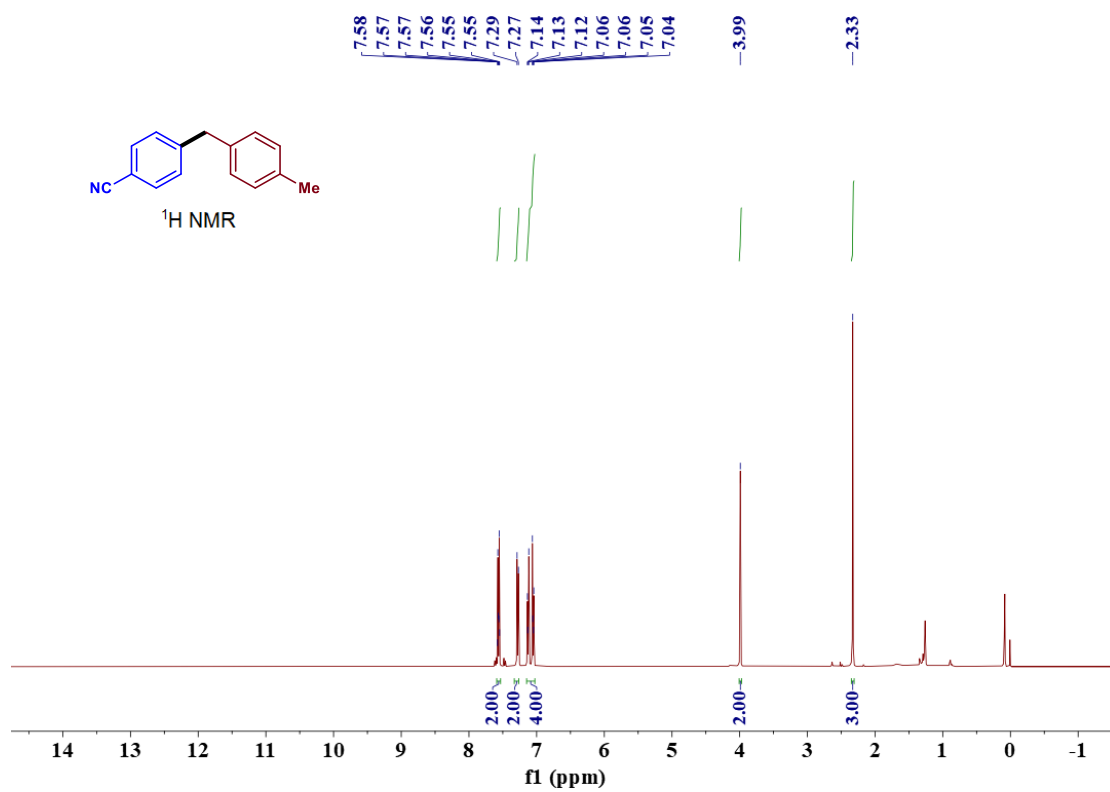


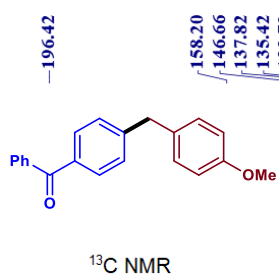


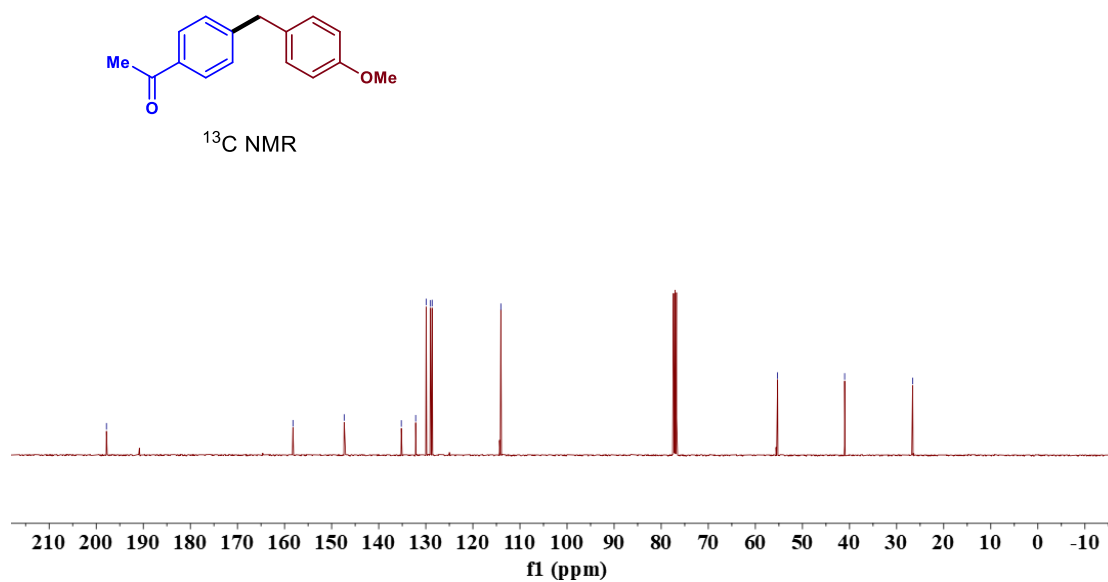
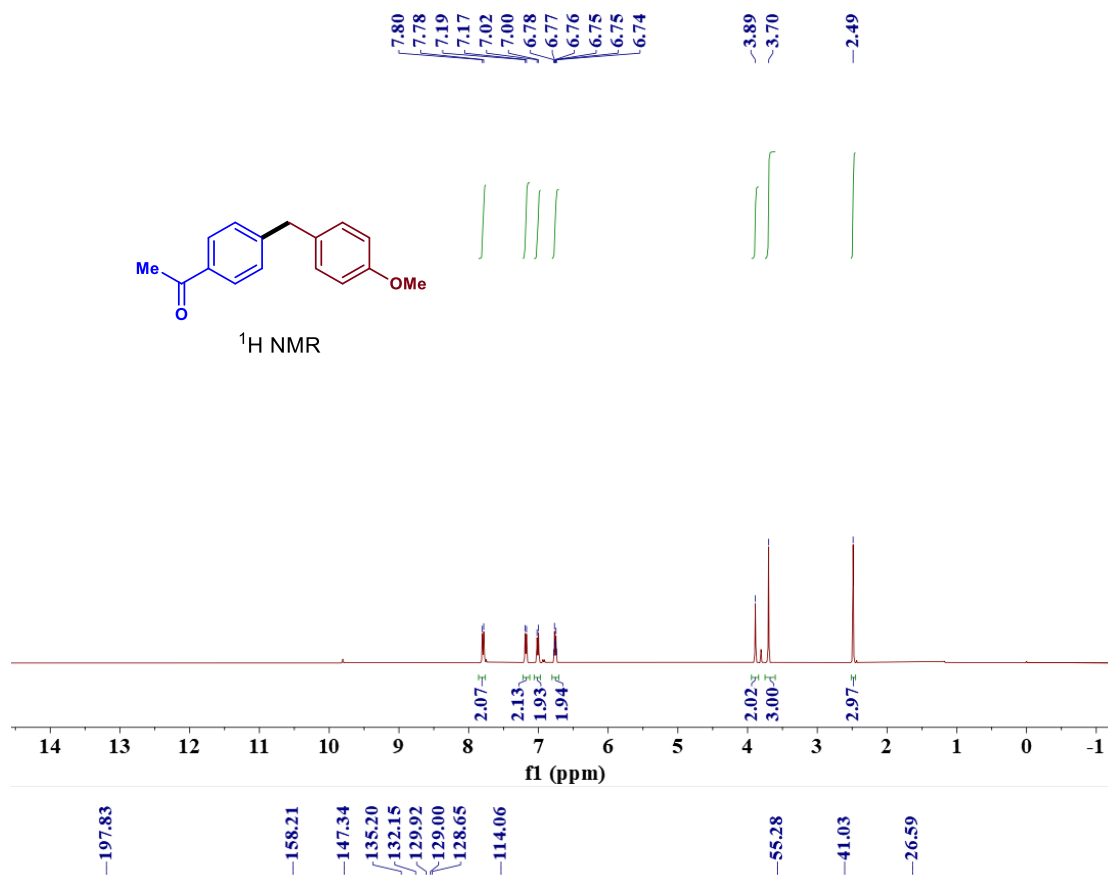


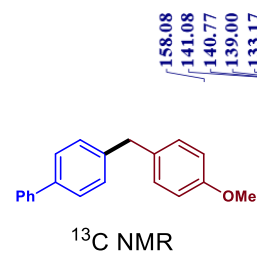
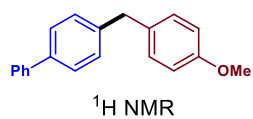


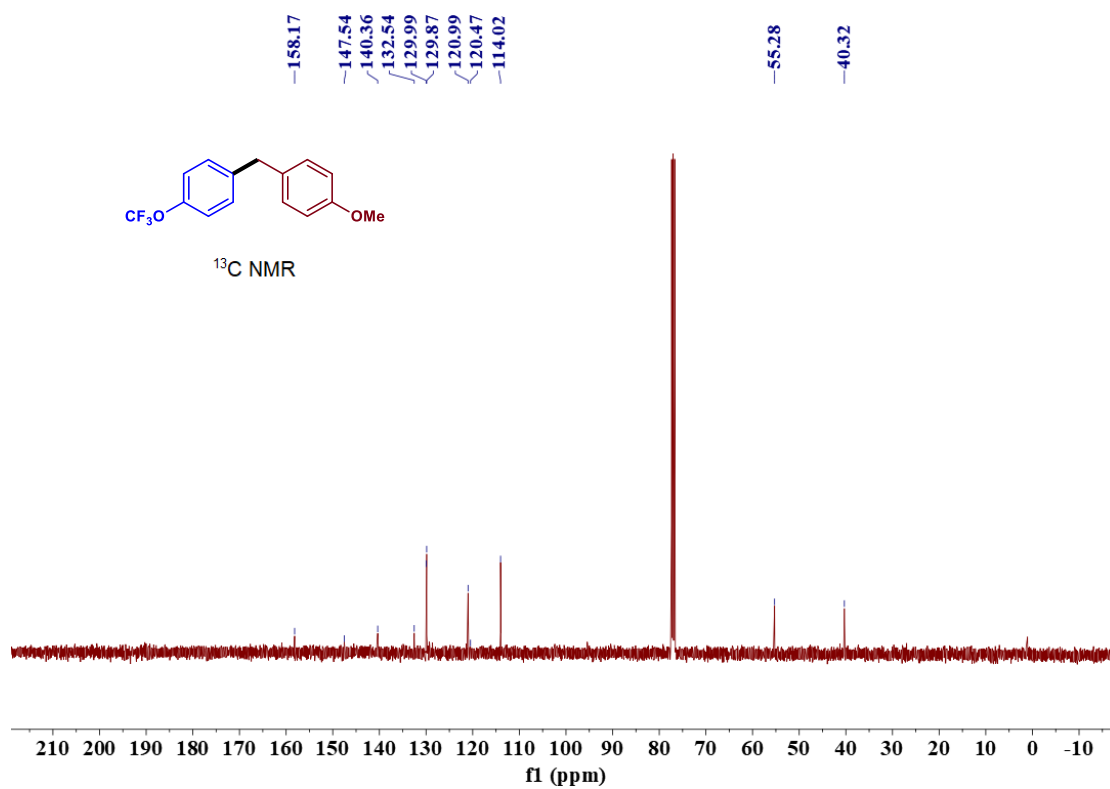
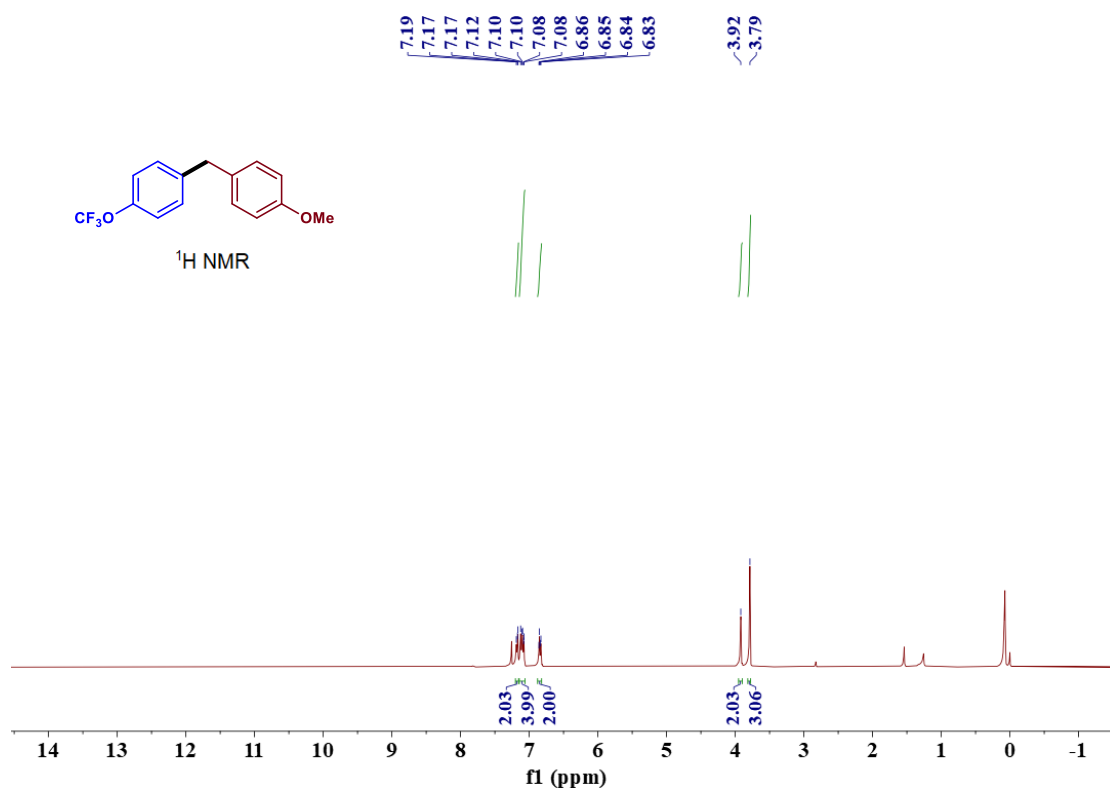


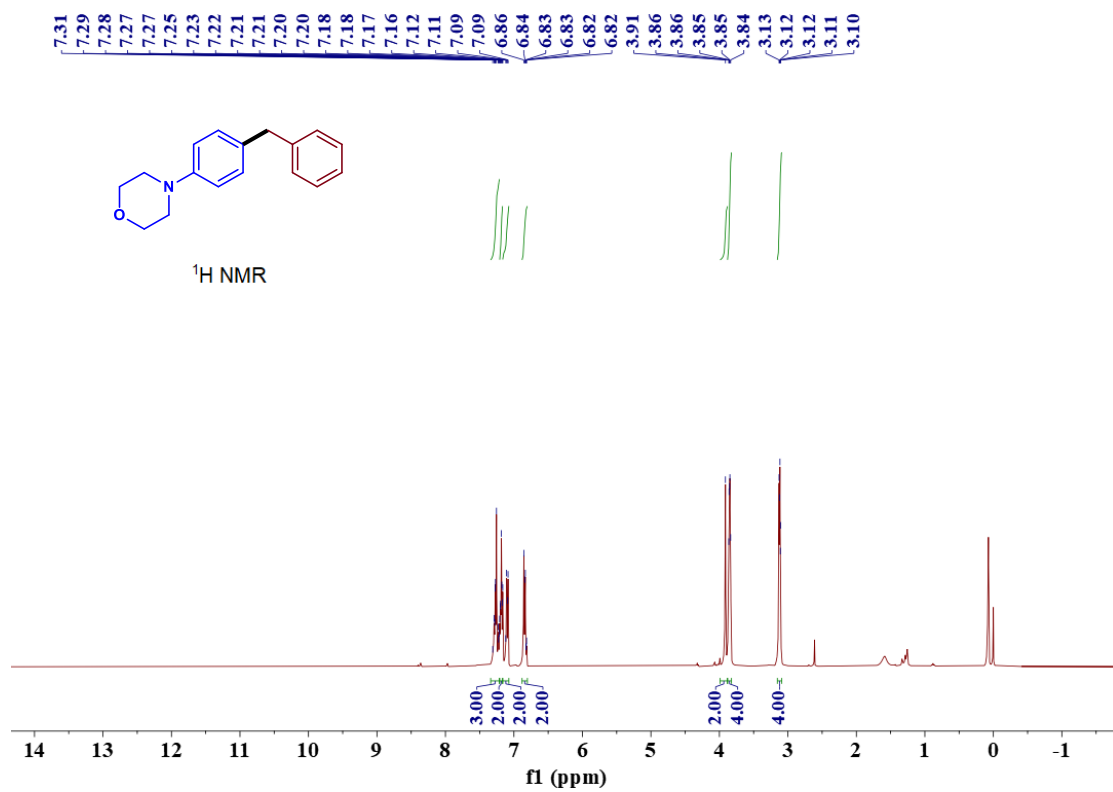
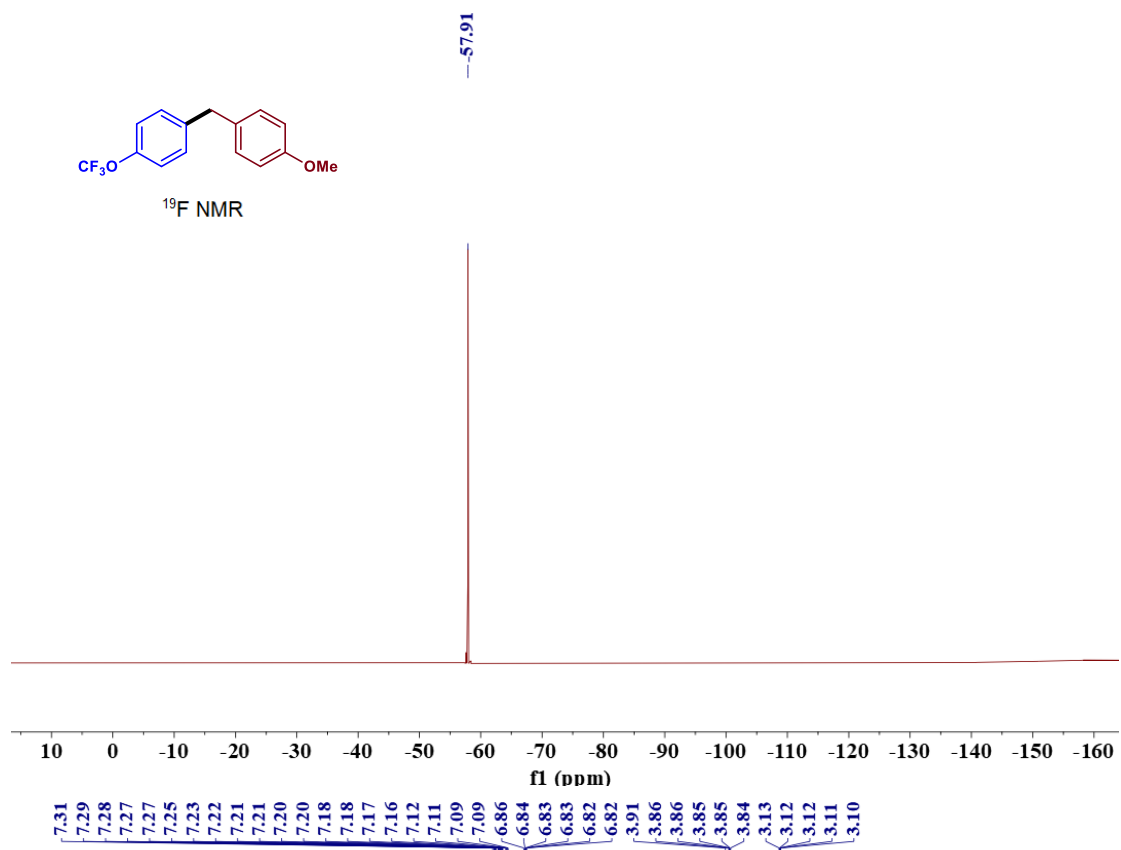


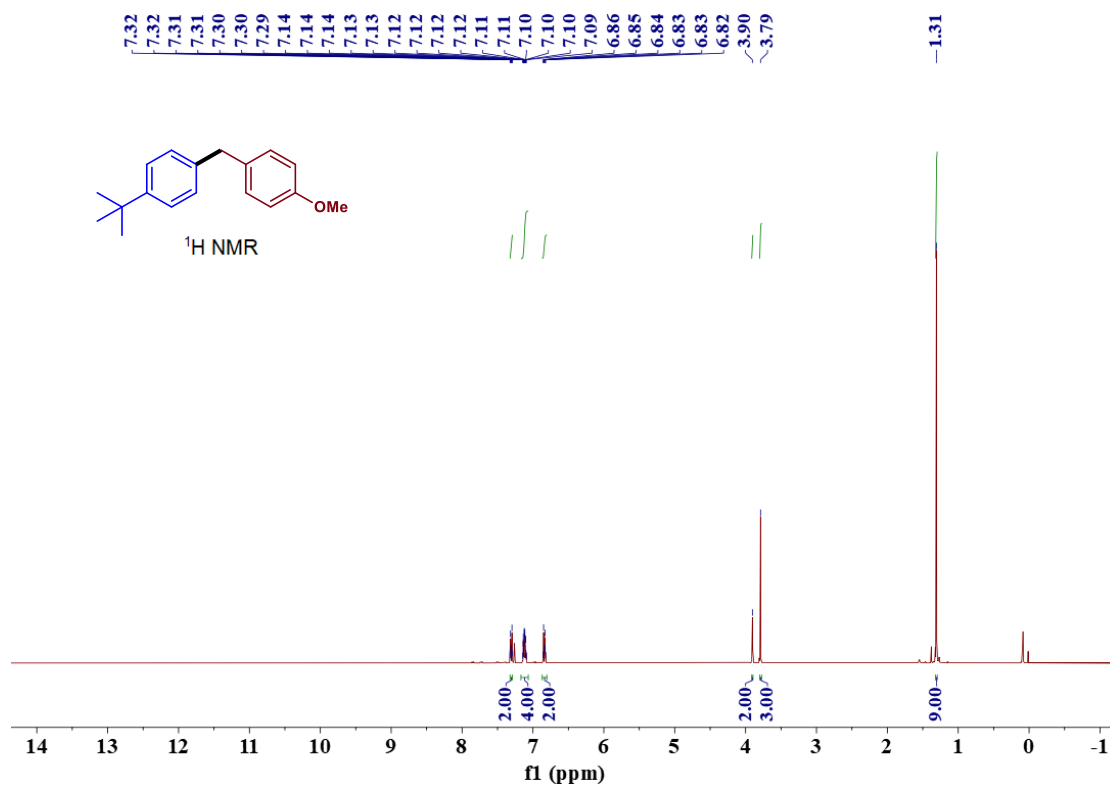
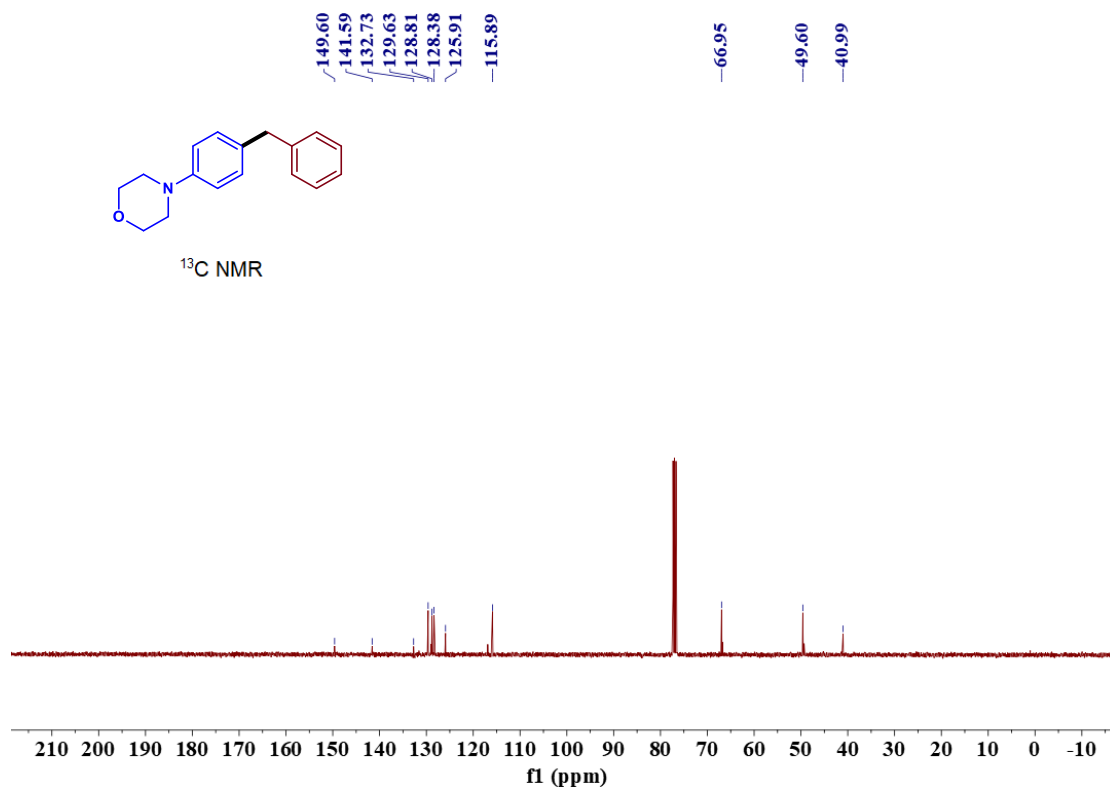


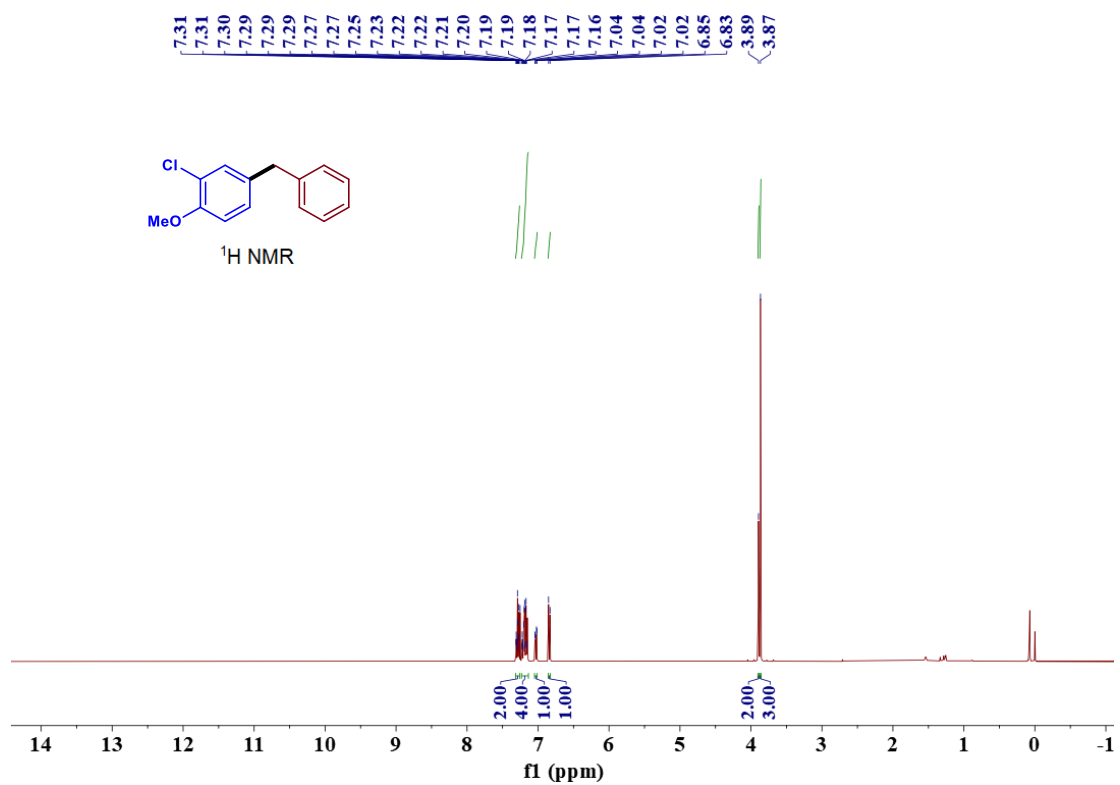
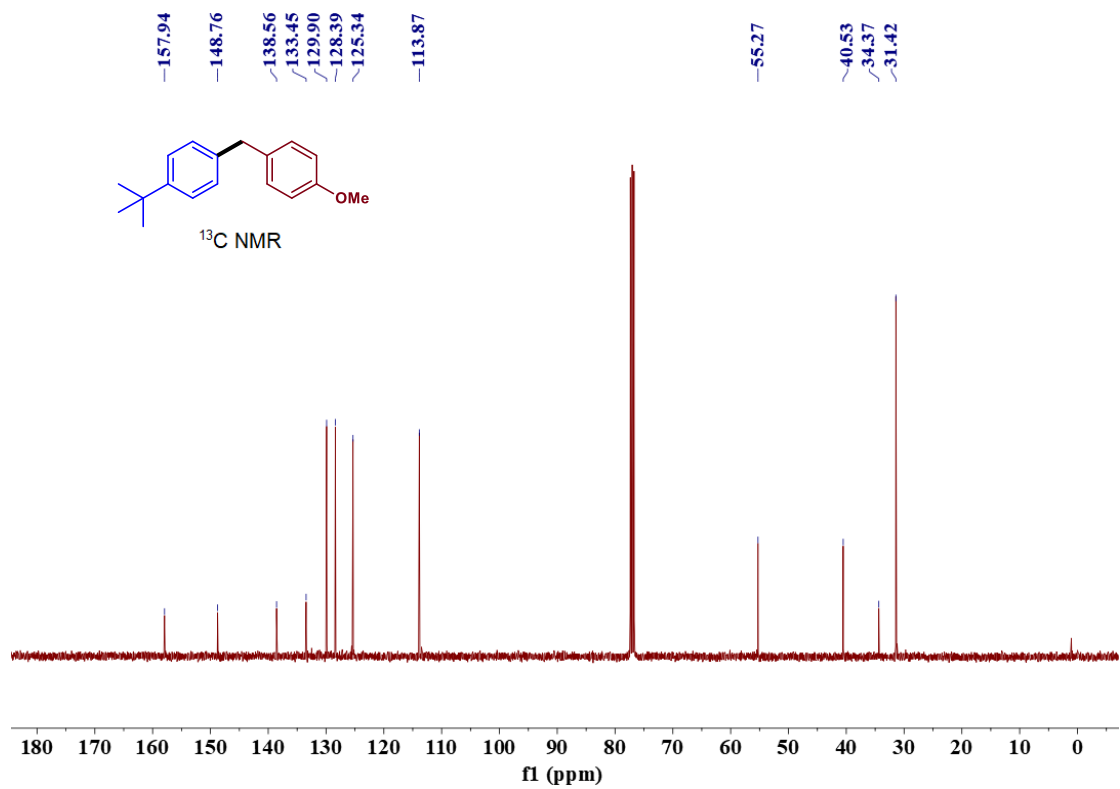


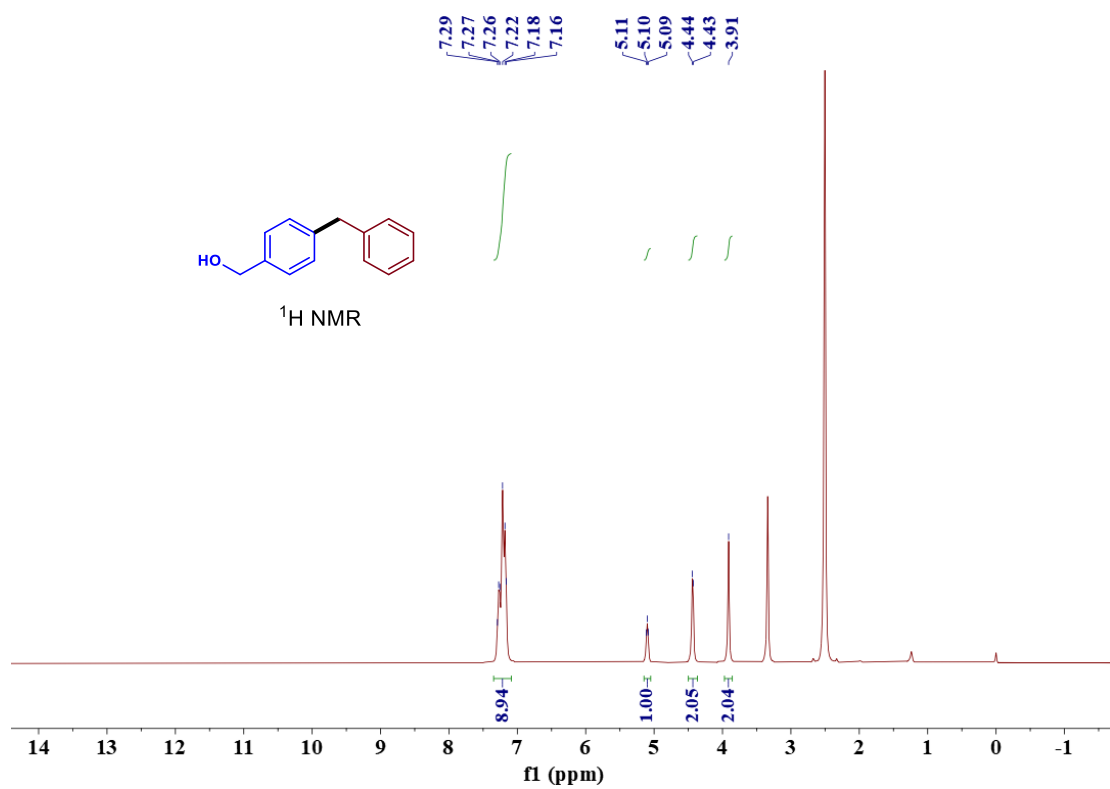
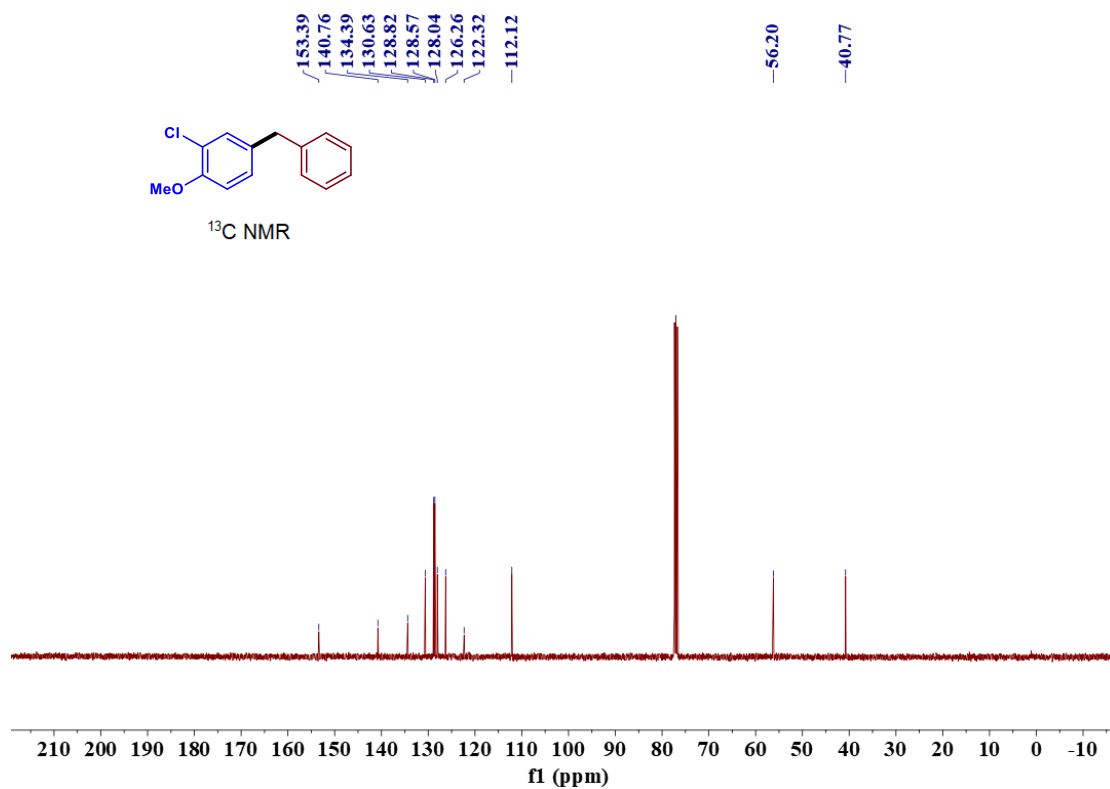


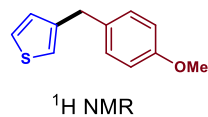
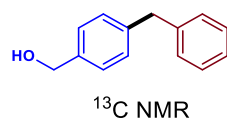


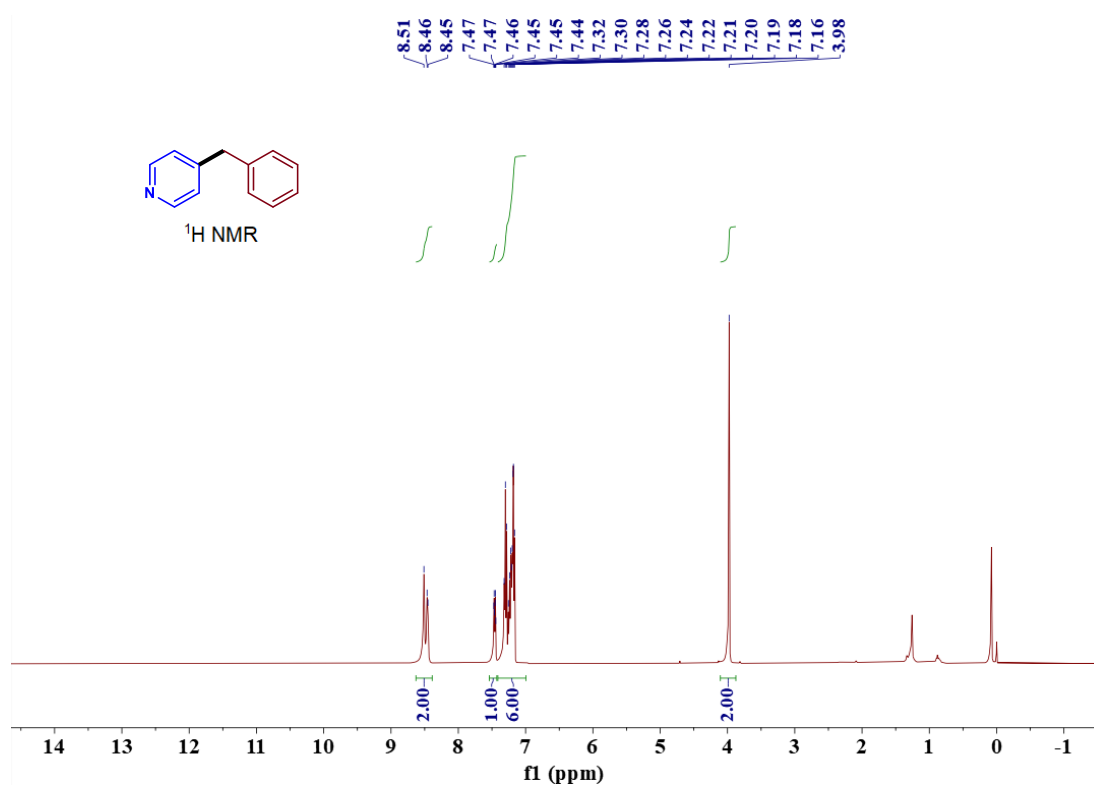
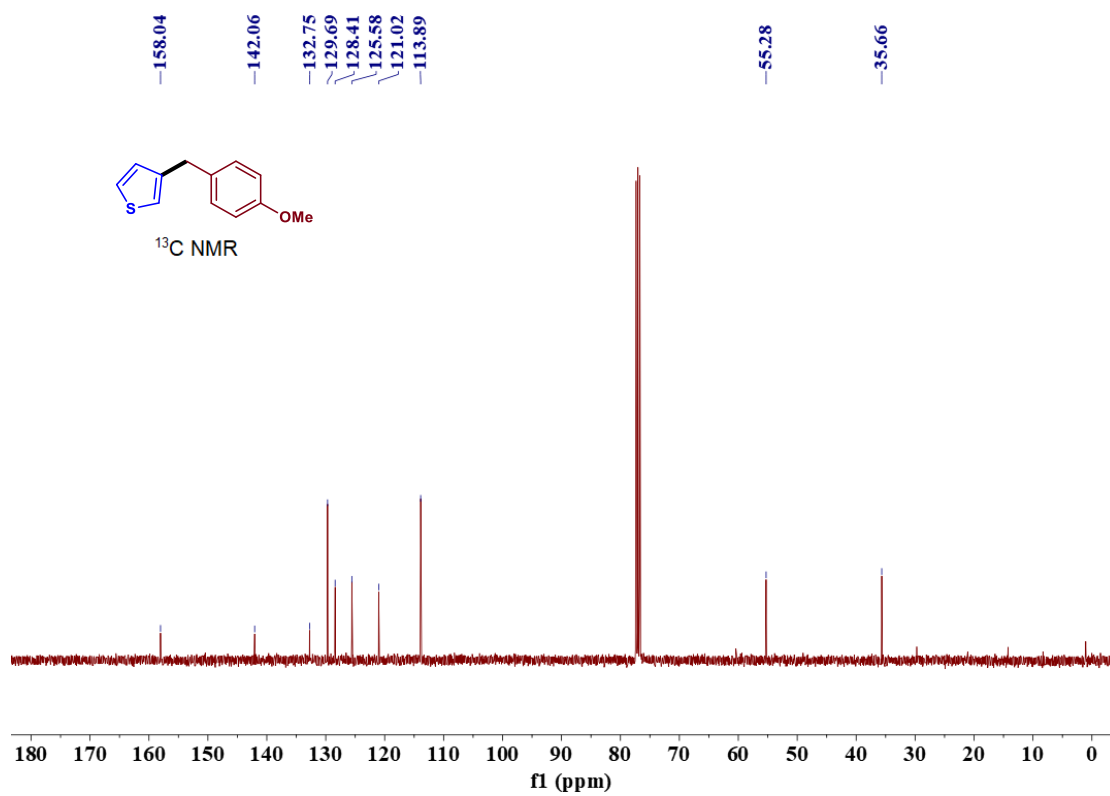


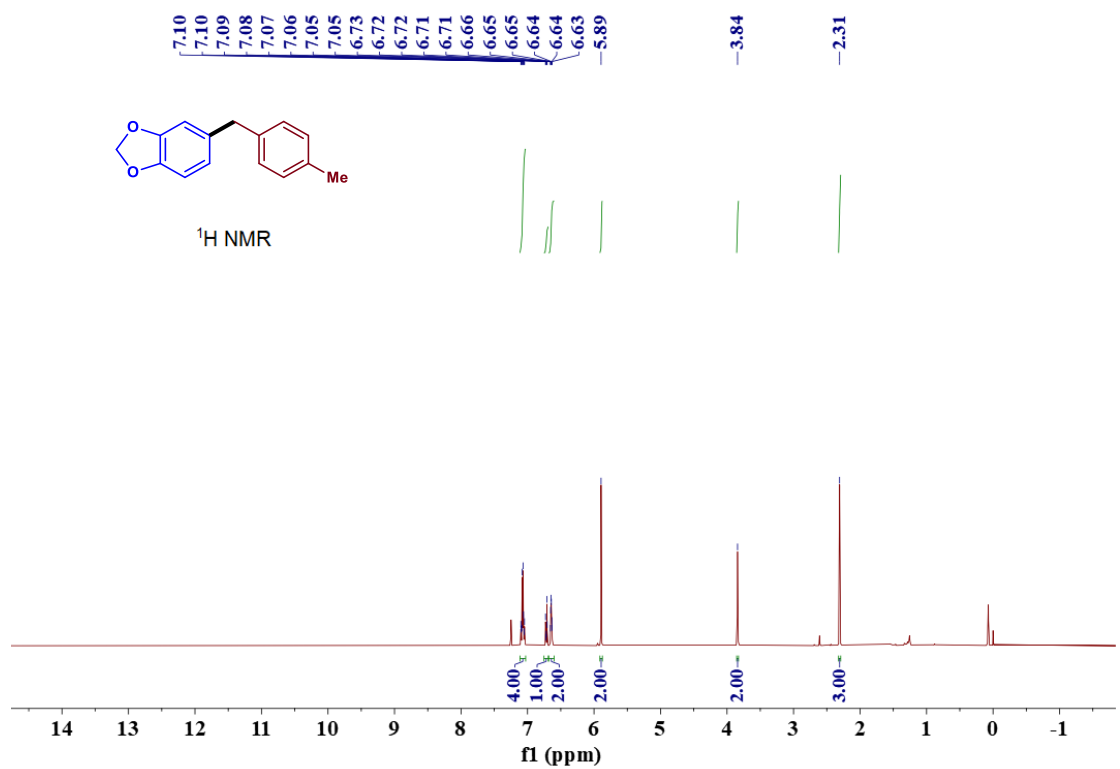
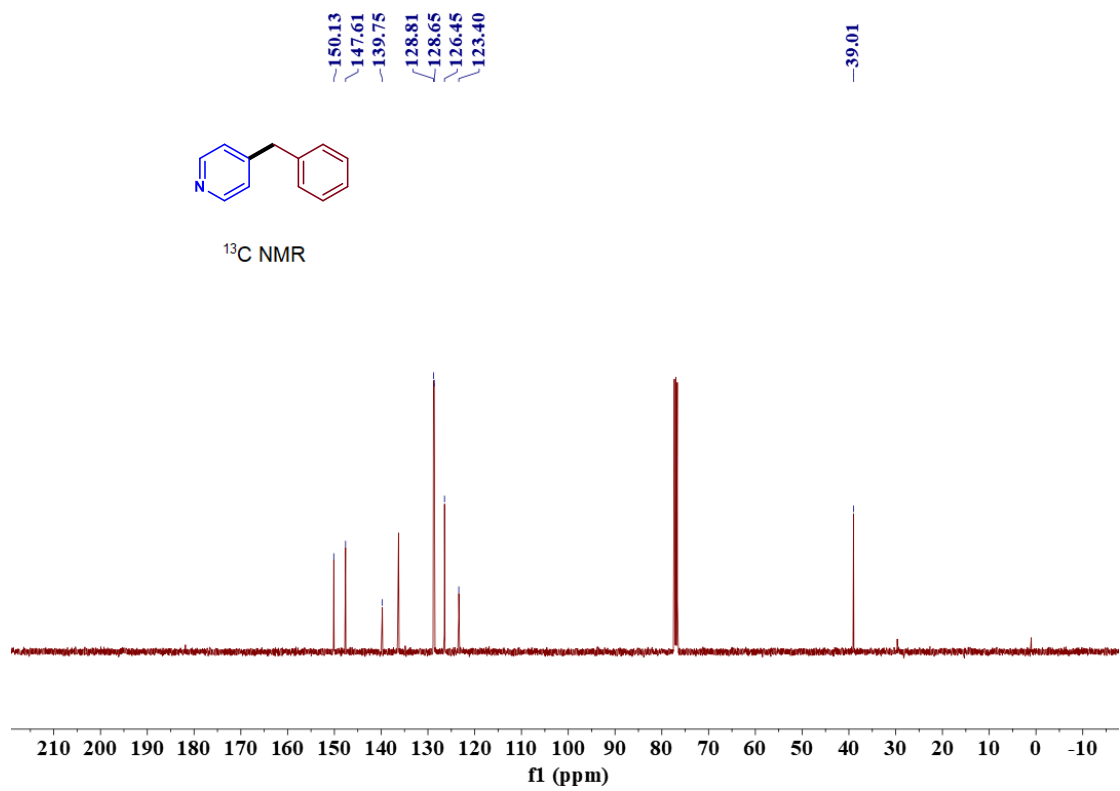


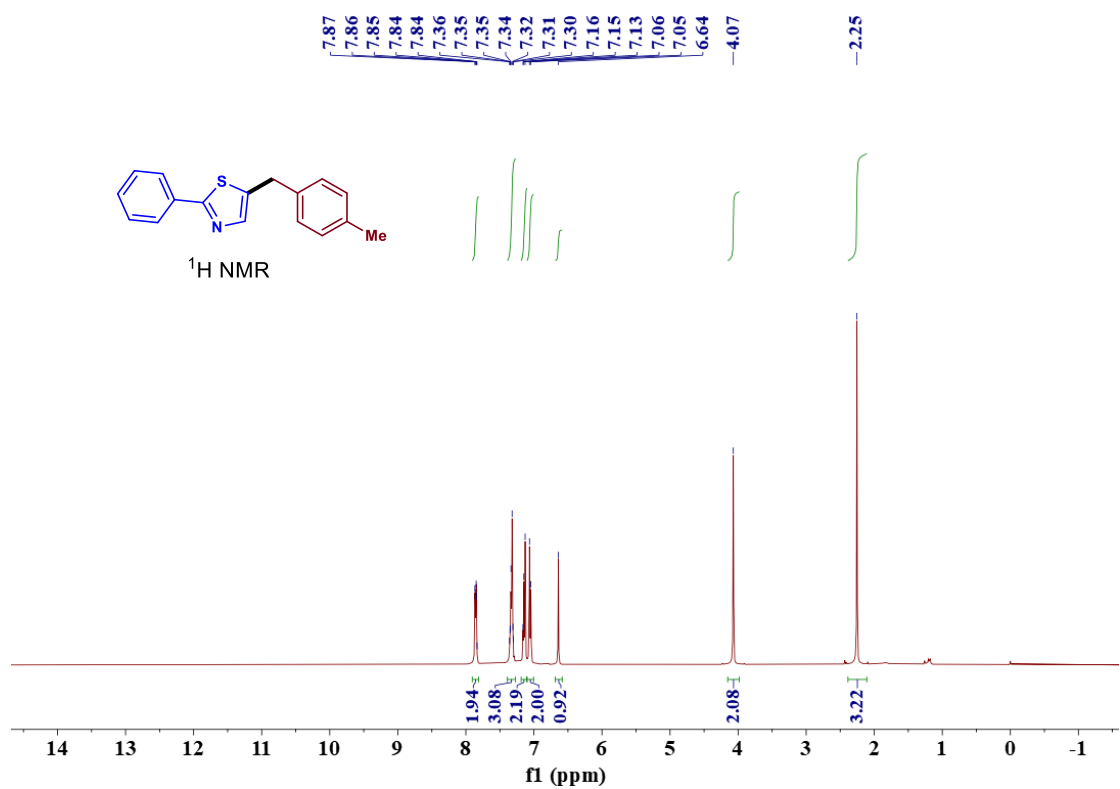
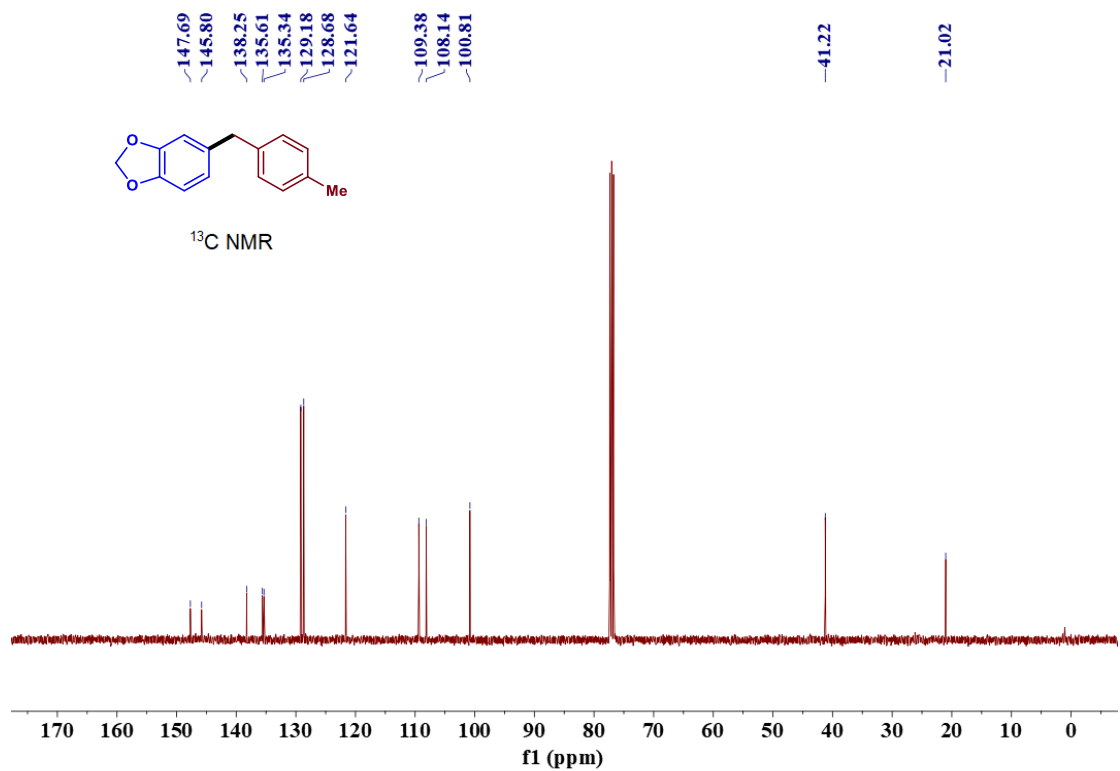


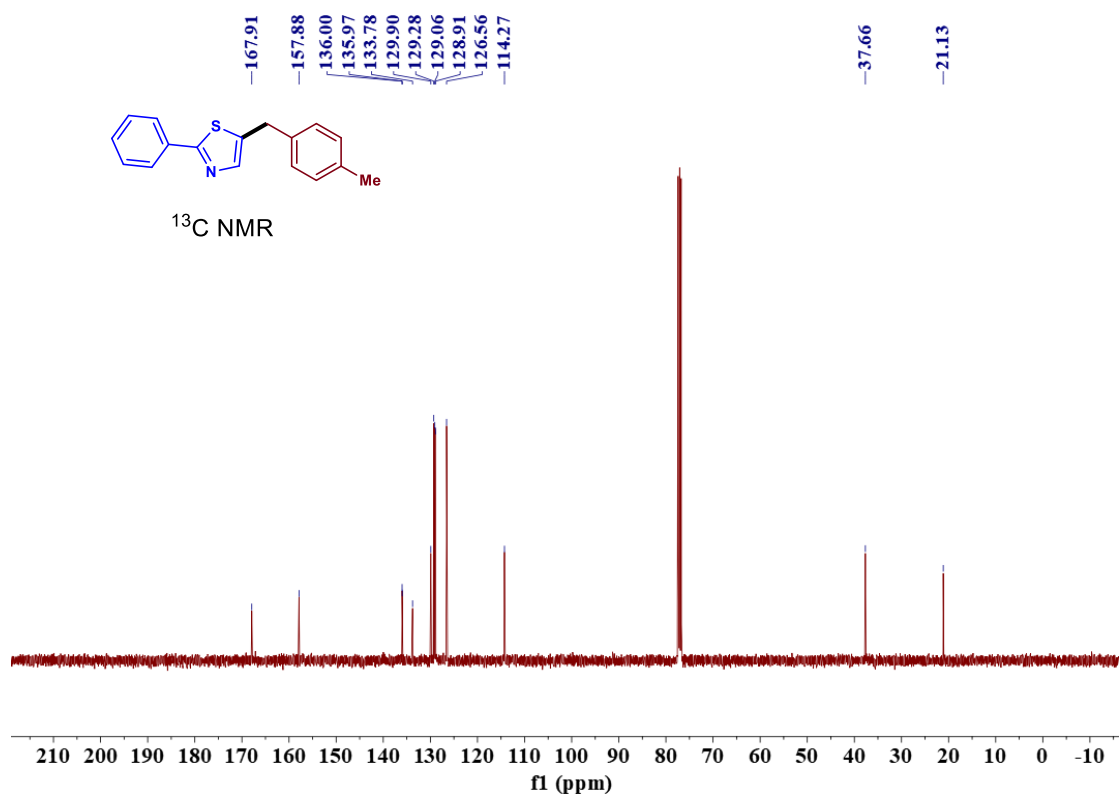












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