Electronic Supplementary Material (ESI) for Inorganic Chemistry Frontiers.

Supporting Information

In-situ ball-milling gram-scale preparation of polyoxoniobates-intercalated MgAl-layered double hydroxides for selective Aldol and Michael addition cascade reactions in water

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Table of contents

Experiment section

Chemical materials. The sources of basic materials and testing kits were purchased from certified companies as following: aromatic aldehydes, aromatic ketones, niobium pentyloxide (Aladdin Chemicals Co., Ltd); sodium hydroxide, potassium hydroxide, ethyl acetate, petroleum ether, magnesium nitrate and aluminum nitrate (Sinopharm Chemical Reagent Co., Ltd.); 3-(4,5)-dimethylthiahiazo(-2)-3,5-diphenytetrazoliumromide (MTT) (Energy Chemical Co., Ltd.). All chemicals and solvents were used in this study without any further purification.

Synthesis of $K_7HNb_6O_{19}\cdot 13H_2O$. $K_7HNb_6O_{19}\cdot 13H_2O$ was synthesized according to the previous report.[1] Nb_2O_5 (0.05 mol, 13.3 g) was added to the melt of KOH (0.46 mol, 26 g) in a nickel crucible. After heating 30 min at 380 °C, the mixture was slowly cooled down to room temperature and quickly poured into 100 mL deionized water. The above mixture was filtrated and the filtrate was put in refrigerator at 0 °C for 12 h and needle-shaped solid was obtained. Finally, the solid was collected and cleaned five times with ethanol-water (V/V=1:1). They were dried in vacuo overnight to yield 9.6 g $K_7HNb_6O_{19}\cdot 13H_2O$ solid (denoted as Nb_6).

Synthesis of Mg_3Al-LDH-Nb_6-X%. $Mg(NO_3)_2\cdot 6H_2O$ (18 mmol, 4.66 g), $Al(NO_3)_3\cdot 9H_2O$ (8 mmol, 3 g), NaOH (0.05 mol, 2 g), with various amounts of Nb_6 (0.5 g, 1 g, 2 g and 4 g, respectively) were put into the 25 mL stainless steel milling pot. The powder of the starting materials was ball-milled with 2 mL amounts of H_2O as the initiator using 5 steel balls with 10 mm diameter for 30 min at a rotation speed

of 250 r. In order to eliminate the excess Nb_6 and inorganic salts, which were in between the particles and attached to the surface of the composites, thus the obtained solids were extensively washed with large amounts of water consecutively, and they were further stirred and ultra-sonicated until no Nb_6 was present in the solution as evidenced by UV-vis spectroscopy. Then the products were dried under vacuum overnight to yield Mg_3Al-LDH-Nb_6-X% (X = 8, 15, 18 and 22) products. IR (KBr, cm^{-1}): 3365 (m), 2220 (m), 1655 (m), 1370 (w), 705 (w), 528 (m).

Catalyst characterization. ^1H NMR and ^{13}C NMR spectra of the organic compounds were acquired on a AVANCE NEO 500 spectrometer by using $CDCl_3$ as the solvent and TMS (tetramethylsilane) as the internal reference. Fourier transform infrared spectra (FT-IR) were recorded on a NICOLET 5700 instrument ranging from 4000 to 400 cm^{-1}. Powder X-ray diffraction analysis (PXRD) patterns were collected on a smart lab diffractometer from Rigaku equipped with a 9 kW rotating anode Cu source (45 kV, 200 mA, 5-50°). Nitrogen sorption experiments were carried out at 77 K on ASAP-2460 analyser. The samples were degassed at 150 °C for 10 h before analysis. X-ray photoelectron spectroscopy (XPS) measurements were undertaken with a K-Alpha spectrometer (Thermo Scientific Ltd., USA). Scanning electron microscopic (SEM) images were viewed on a Thermo Fisher Scientific scanning electron microscope. Transmission electron microscopy (TEM) analysis was carried out on a Thermo Scientific TM Talos™ F200X electron microscope.

Selective synthesis of aromatic 1,5-dione derivatives. In a typical experiment, aromatic aldehydes (0.5 mmol), aromatic ketones (1 mmol), catalyst (0.05 mmol) and

2 mL H_2O were successively added into a 10 mL glass tube. After being stirred at 60 °C for 1 h, the reaction mixture was cooled to room, filtered through a sand core funnel and extracted with ethyl acetate, washed with brine (5 mL) for three times, dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography on silica gel. The resulting products were identified by ^1H NMR and ^{13}C NMR.

Selective synthesis of polysubstituted cyclohexanol derivatives. The synthetic procedure of polysubstituted cyclohexanols was similar to those of syntheses of aromatic 1,5-diones except that TBAB (0.25 mmol) was used, the amount of catalyst, the reaction time and temperature were changed in the reactions.

Recycling process for the selective synthesis of polysubstituted cyclohexanol derivatives. The reuse experiment was carried out for synthesis of polysubstituted cyclohexanols. In a typical experiment, benzaldehyde (0.5 mmol), acetophenone (1 mmol), catalyst (0.1 mmol) and TBAB (0.25 mmol) were successively added in a 10 mL double-neck round bottom flask with 2 mL H_2O. The reaction mixture was stirred for 24 h at 90 °C. After the reaction was completed, the catalyst was retrieved by filtration, washed and soaked with H_2O (10 mL) and EtOH (10 mL) three times, and air-dried prior to being used for the reuse experiment.

Cytotoxicity activity of cyclohexanols and aromatic 1,5-diones. For determination of cytotoxic or growth inhibition effect of polysubstituted cyclohexanols and aromatic 1,5-diones on cancerous cells, MTT assay was performed according to a reported method.[2] HeLa and A549 cells were employed for the cell viability study. Both of the cells were seeded at a concentration of 4000 cells per well

in a 96 well cell culture plate in DMEM/High glucose supplemented with 10% (v/v) fetal bovine serum (Gibco) and 1 x antibiotics (Hyclone) at 37 °C and humidified 5% CO_2. The media was replaced with same volume of DMEM with respective concentrations of polysubstituted cyclohexanols and aromatic 1,5-diones (0-20 mg/mL). Then the cells in the 96 well plate were allowed to grow for another 48 h at 37 °C and humidified 5% CO_2. After 48 h of treatment, the cells in the wells were incubated with 100 µL (1 mg/ml in incomplete media) of MTT solution for 4 h at 37 °C and humidified 5% CO_2 atmosphere. The wells were replaced with 100 µL of DMSO solution. The absorbance of the DMSO dissolved formazan crystals were measured at 490 nm with a spectrophotometer (I MarkTM Microplate Absorbance Reader). The cell viability was calculated as $(A_{sample}/A_{control}) \times 100$, where A_{sample} is the absorbance of the sample and $A_{control}$ is the absorbance of the control.

Table S1. The elemental analysis of Mg_3Al-LDH-Nb_6-X% composites

Entry	Samples	Mg (wt%)	Al (wt%)	Nb (wt%)	X value
1	Mg_3Al-LDH-Nb_6-X%	12.41	5.06	6.10	17
2		10.98	4.47	8.69	25
3		10.54	4.34	10.10	29
4		9.82	4.02	10.45	32

Table S2. The results of synthesis of polysubstituted cyclohexanols under various conditions

The starting materials	Conditions	Solvent	Yield (%)	Ref.
Chalcone	(20 mol%) NaO*t*Bu, (1.5 eq) NHC	Et_2O	80	3
Benzaldehyde and acetophenone	Solid NaOH-K_2CO_3 (2:1, ground)	None	91	4
Chalcone and acetophenone	(16 eq) NaOH	None	86	5

Gram scale for synthesis of Mg_3Al-LDH-Nb_6-29%

Synthesis of Mg_3Al-LDH-Nb_6-29%. $Mg(NO_3)_2{\cdot}6H_2O$ (18 mmol, 4.66 g), $Al(NO_3)_3{\cdot}9H_2O$ (8 mmol, 3 g), NaOH (0.05 mol, 2 g), with 2 g Nb_6 were put into the 25 mL stainless steel milling pot. The powder of the starting materials was ball-milled with 2 mL amounts of H_2O as the initiator using 5 steel balls with 10 mm diameter for 30 min at a rotation speed of 250 r. In order to eliminate the excess Nb_6 and inorganic salts, which were in between the particles and attached to the surface of the composites, thus the obtained solids were extensively washed with large amounts of water consecutively, and they were further stirred and ultra-sonicated until no Nb_6 was present in the solution as evidenced by UV-vis spectroscopy.[6] Then the products were dried under vacuum overnight to yield 2.16 g Mg_3Al-LDH-Nb_6-29% product.

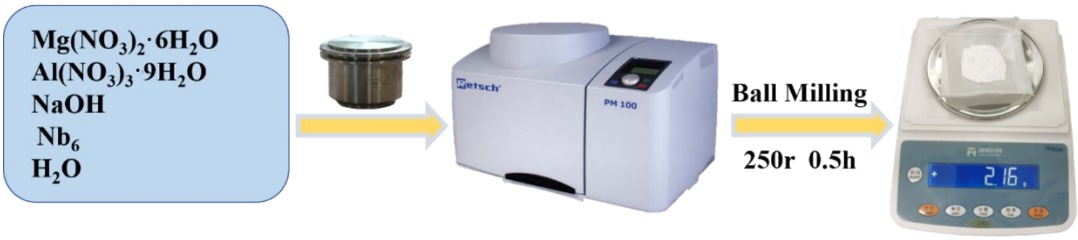


Figure S1. One-pot synthetic protocol for producing Mg_3Al-LDH-Nb_6-29% (weight 2.16 g).

EDS spectra of Mg_3Al-LDH-Nb_6-29% catalyst

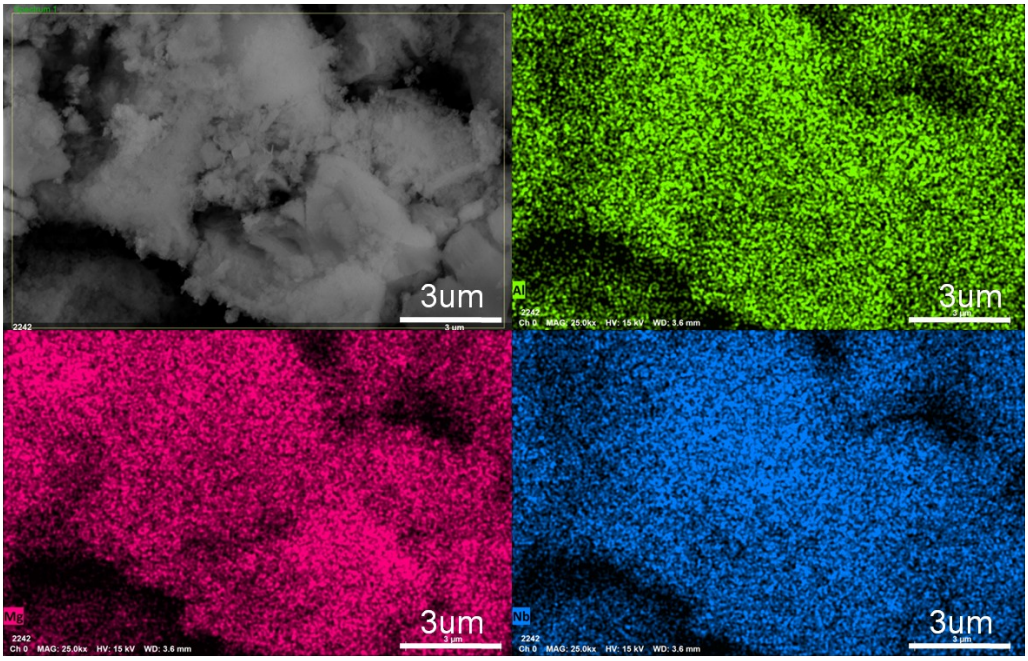


Figure S2. The EDS elemental mapping of Mg_3Al-LDH-Nb_6.

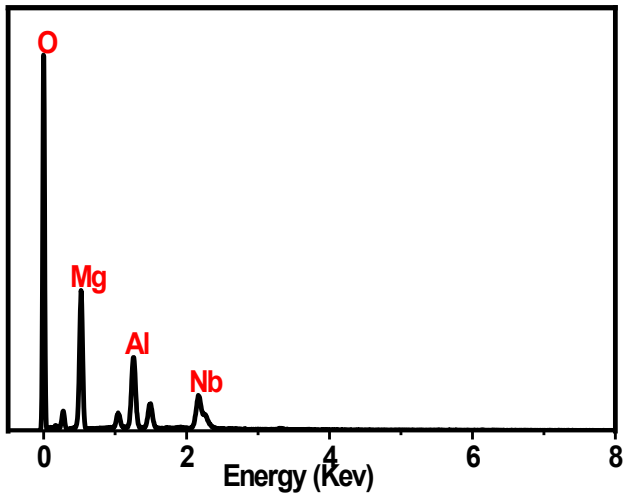


Figure S3. The EDS spectrum of Mg_3Al-LDH-Nb_6 composite, which gives the compositional information.

TG and UV spectra of Mg_3Al-LDH-Nb_6-29% catalyst

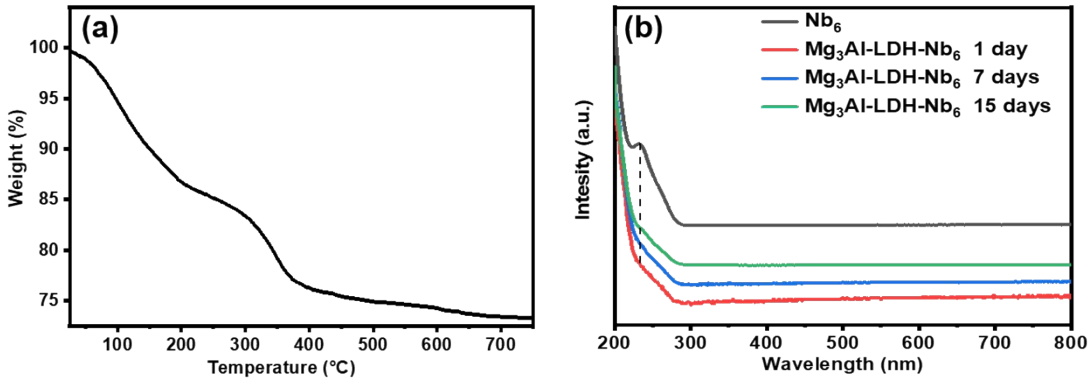


Figure S4. (a) TG patterns of Mg_3Al-LDH-Nb_6; (b) UV spectra of Mg_3Al-LDH-Nb_6 (H_2O), Nb_6 (H_2O) and 1 day, 7 days, 15 days.

Gram scale for synthesis of polysubstituted cyclohexanol product (4l)

In a typical experiment, benzaldehyde (0.02 mol), *p*-bromoacetophenone (0.04 mol), catalyst (0.004 mol), TBAB (0.01 mol) and 15 mL H_2O were successively added into a 50 mL glass flask. After being stirred at 90 °C for 24 h, the reaction mixture was cooled to room, filtered through a sand core funnel and extracted with ethyl acetate, washed with brine (50 mL) for three times, dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography on silica gel. The resulting products were identified by ^1H NMR and ^{13}C NMR.

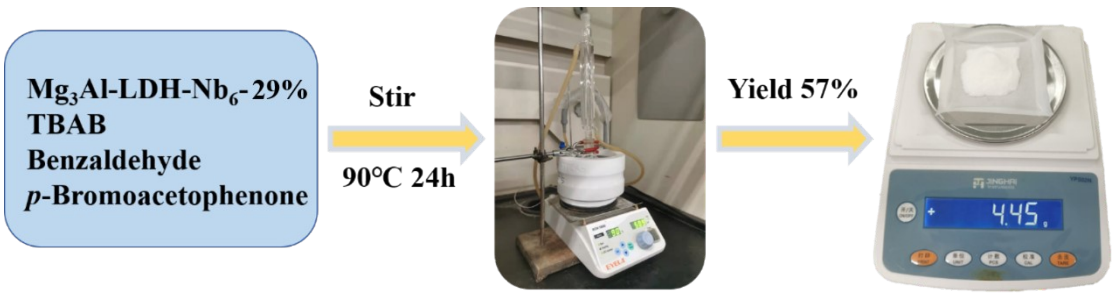


Figure S5. One-pot synthetic protocol for achieving **4l** (weight 4.45g).

LC-MS experimental data of the intermediates 3a, 4a, 5a

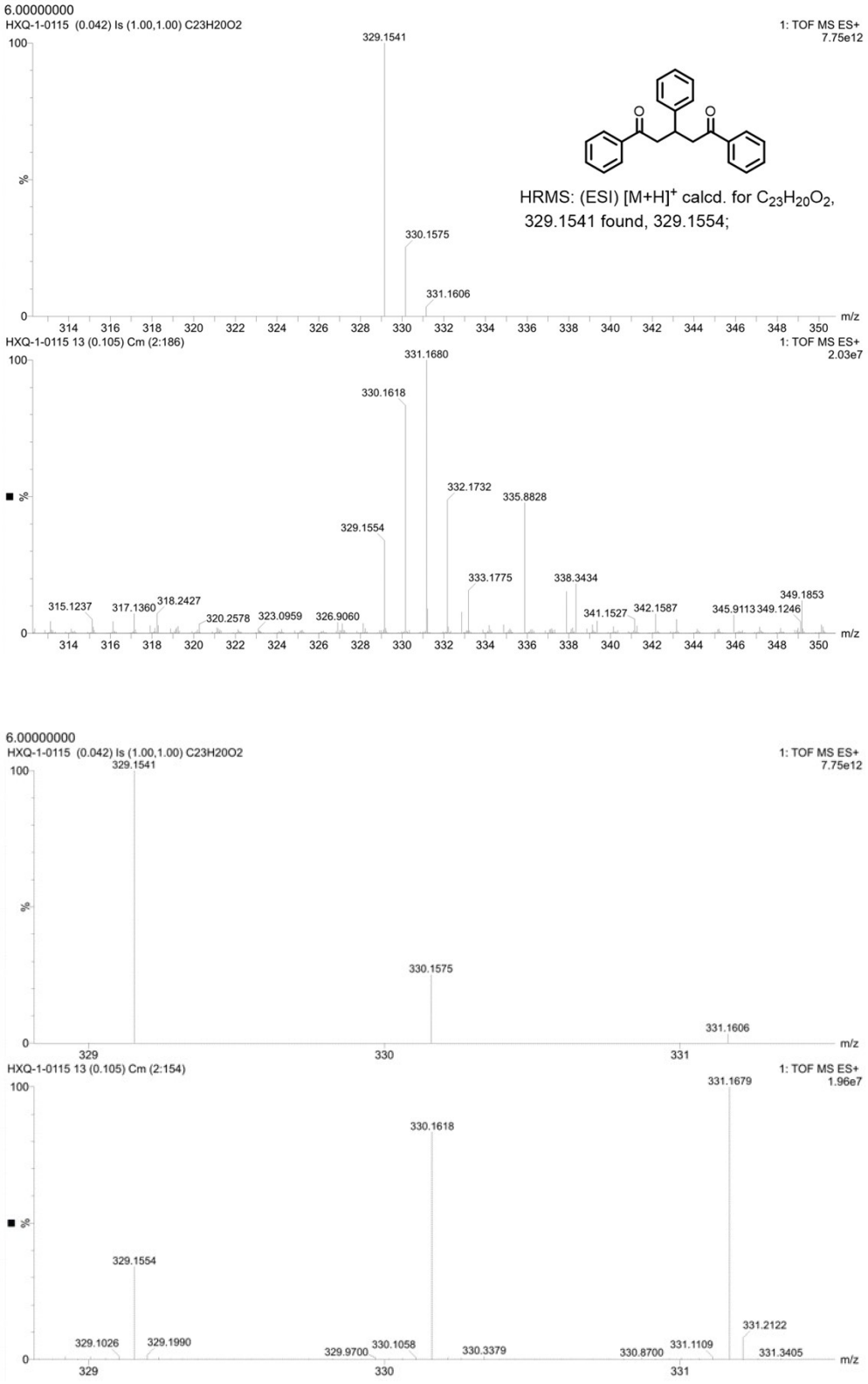


Figure S6. LC-MS spectra of **3a**

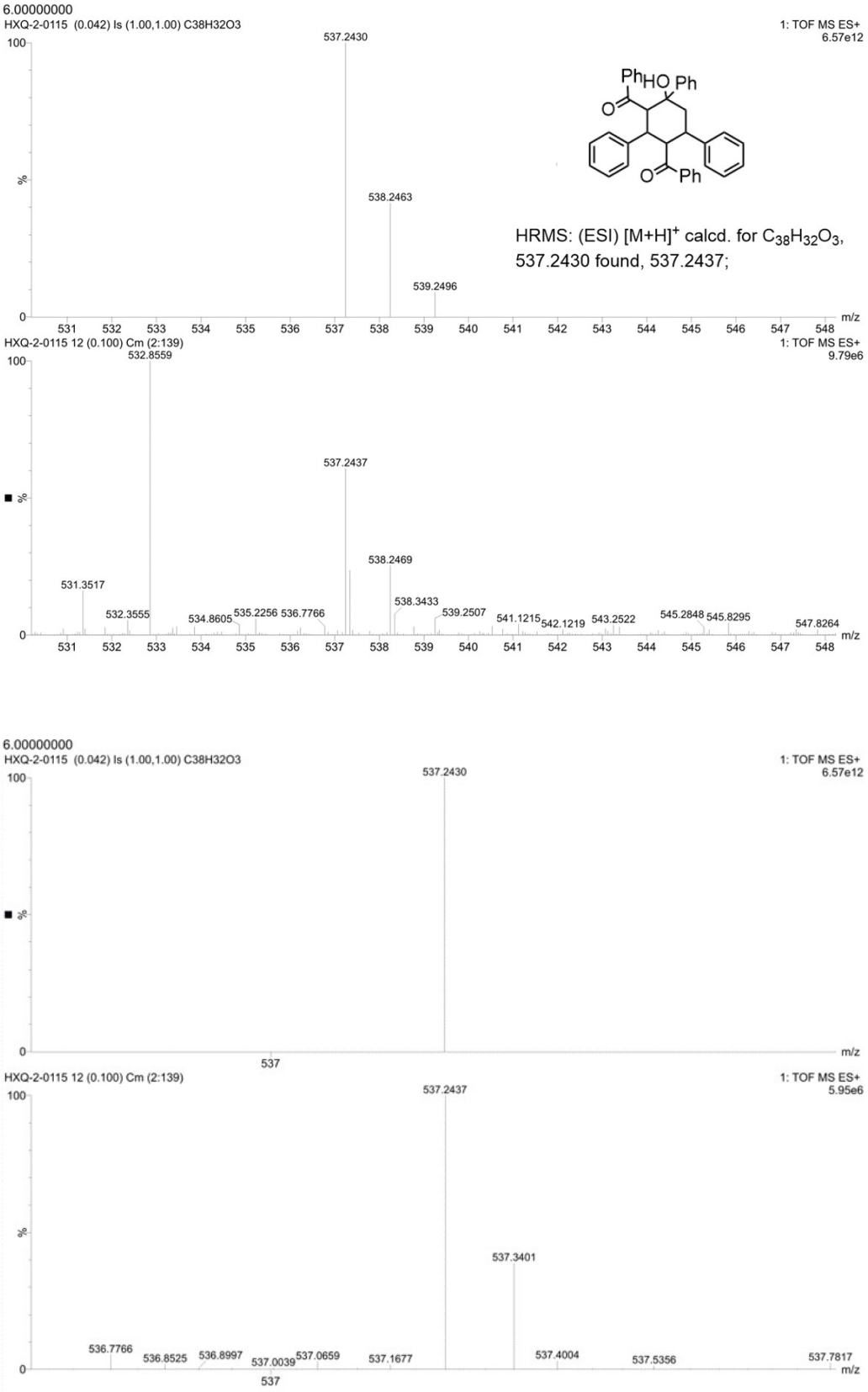


Figure S7. LC-MS spectra of **4a**

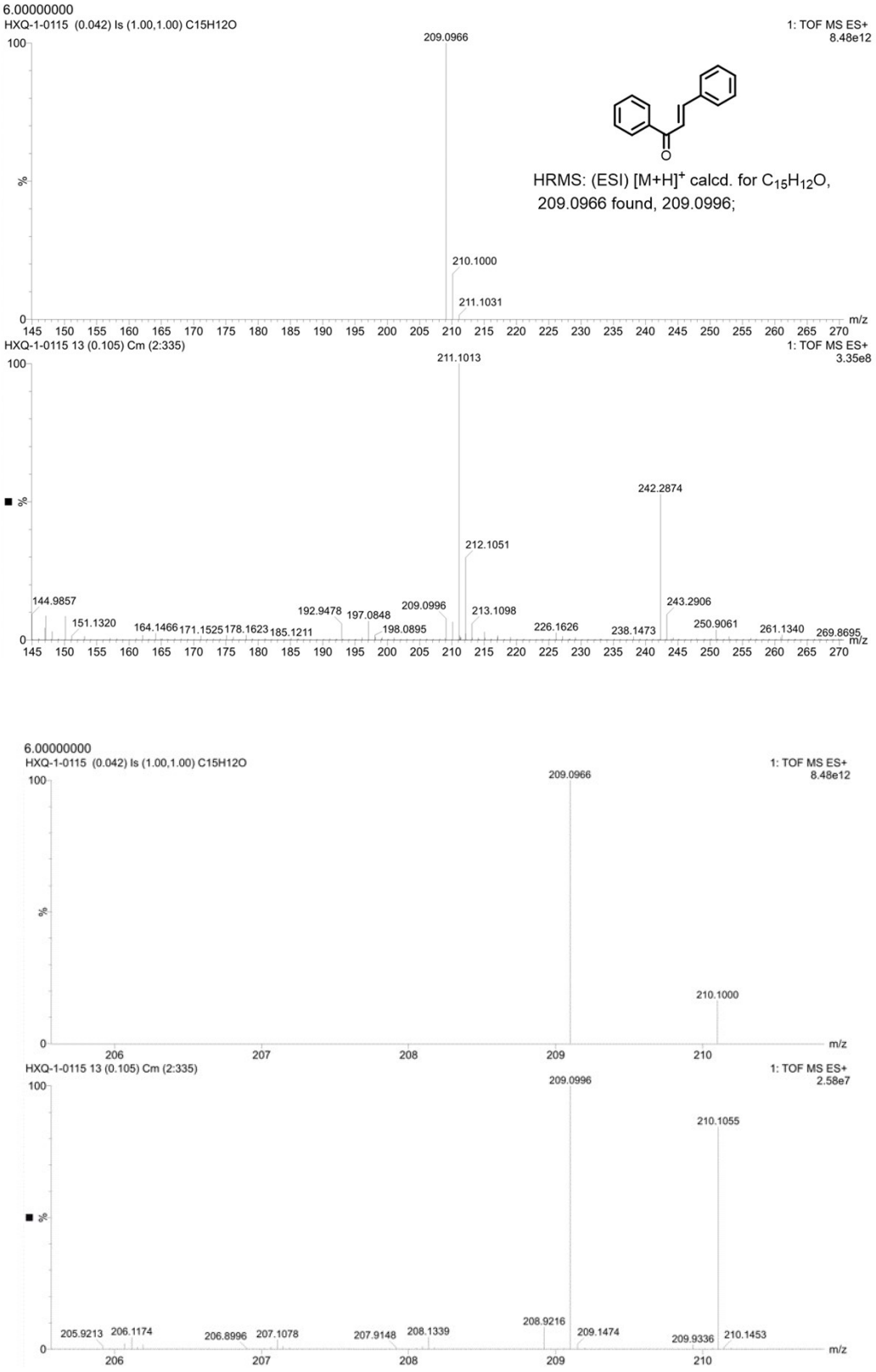


Figure S8. LC-MS spectra of **5a**

TPD spectra of Mg_3Al-LDH-Nb_6-29% catalyst

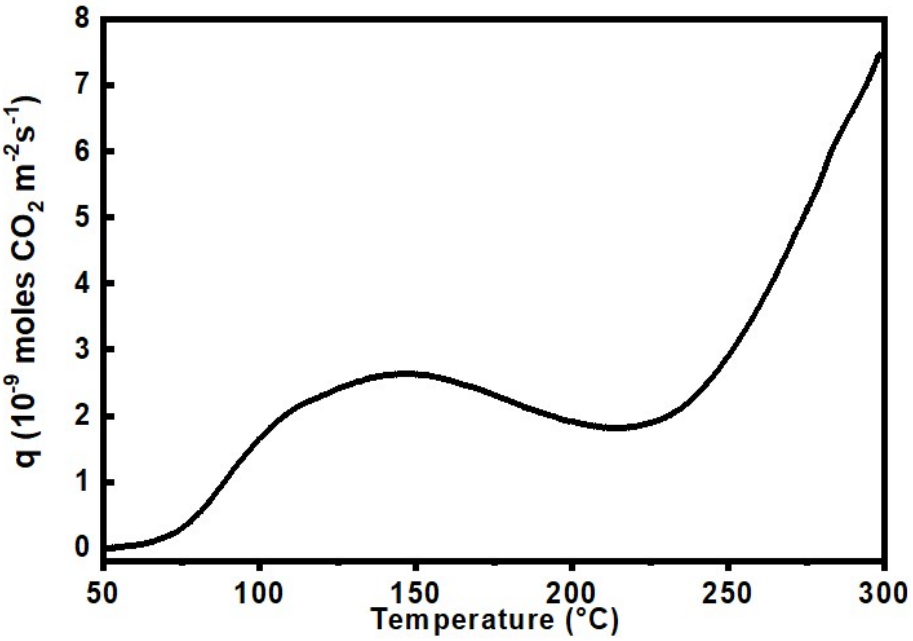
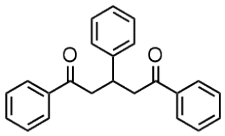


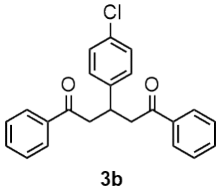
Figure S9. TPD spectra of CO_2 obtained after CO_2 adsorption

NMR spectra of products



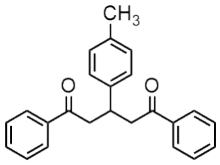
3a

1,3,5-triphenylpentane-1,5-dione (3a). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.5 Hz, 4H), 7.54 (t, *J* = 7.4 Hz, 2H), 7.44 (t, *J* = 7.4 Hz, 4H), 7.30 – 7.25 (m, 4H), 7.19 – 7.16 (m, 1H), 4.09 – 4.06 (m, 1H), 3.49 (d, *J* = 16.6 Hz, 2H), 3.36 (d, *J* = 16.6 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.69, 143.96, 137.06, 133.20, 128.75, 128.72, 128.27, 127.60, 126.82, 45.04, 37.31. HRMS: (ESI) $[M+H]^+$ calcd. for $C_{23}H_{20}O_2$, 329.1541 found, 329.1554.



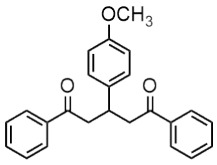
3b

3-(4-chlorophenyl)-1,5-diphenylpentane-1,5-dione (3b). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 7.7 Hz, 4H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 4H), 7.25 – 7.23 (m, 4H), 4.08 – 4.04 (m, 1H), 3.47 (d, *J* = 16.7 Hz, 2H), 3.35 (d, *J* = 16.7 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.35, 142.46, 136.94, 133.35, 132.49, 129.05, 128.87, 128.79, 128.24, 44.87, 36.66.



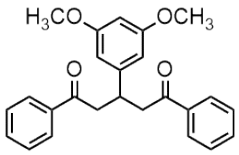
3c

1,5-diphenyl-3-(p-tolyl)pentane-1,5-dione (3c). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.3 Hz, 4H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 4H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 7.9 Hz, 2H), 4.06 – 4.0 (m, 1H), 3.47 (d, *J* = 16.5 Hz, 2H), 3.34 (d, *J* = 16.5 Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.81, 140.92, 137.12, 136.32, 133.16, 129.44, 128.71, 128.29, 127.43, 45.19, 37.02, 29.84, 21.14.



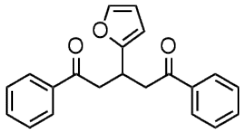
3d

3-(4-methoxyphenyl)-1,5-diphenylpentane-1,5-dione (3d). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.2 Hz, 4H), 7.53 (t, *J* =7.3 Hz, 2H), 7.44 (t, *J* = 7.3 Hz, 4H), 7.19 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 4.03 – 4.00 (m, 1H), 3.76 (s, 3H), 3.46 (d, *J* = 16.5 Hz, 2H), 3.31 (d, *J* = 16.5 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.89, 137.16, 133.19, 128.74, 128.57, 128.31, 114.18, 55.36, 45.32, 36.72.



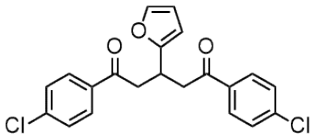
3e

3-(3,5-dimethoxyphenyl)-1,5-diphenylpentane-1,5-dione (3e). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.4 Hz, 4H), 7.52 (t, *J* = 7.4 Hz, 2H), 7.42 (t, *J* = 7.7 Hz, 4H), 6.43 (s, 2H), 6.28 (s, 1H), 4.04 – 4.00 (m, 1H), 3.71 (s, 6H), 3.44 (d, *J* = 16.7 Hz, 2H), 3.33 (d, *J* = 16.7 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.56, 160.90, 146.47, 136.99, 133.13, 128.64, 128.18, 105.75, 98.39, 76.93, 55.28, 44.82, 37.40.



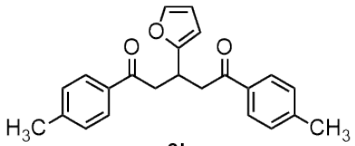
3f

3-(furan-2-yl)-1,5-diphenylpentane-1,5-dione (3f). ^{1}H NMR (500 MHz, Chlorzoform-*d*) δ 7.97 (d, *J* = 8.6 Hz, 4H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 4H), 7.27 (d, *J* = 1.8 Hz, 1H), 6.23 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.06 (d, *J* = 3.2 Hz, 1H), 4.17 – 4.22 (m, 1H), 3.45 – 3.43 (m, 4H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.36, 156.52, 141.29, 136.93, 133.27, 128.73, 128.26, 110.34, 105.59, 42.27, 30.90.



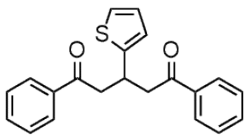
3g

1,5-bis(4-chlorophenyl)-3-(furan-2-yl)pentane-1,5-dione (3g). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.4 Hz, 4H), 7.42 (d, *J* = 8.4 Hz, 4H), 7.29 – 7.25 (m, 1H), 6.23 (dd, *J* = 2.6, 1.9 Hz, 1H), 6.04 (d, *J* = 2.6 Hz, 1H), 4.18 – 4.14 (m, 1H), 3.40 – 3.39 (m, 4H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.12, 156.08, 141.43, 139.79, 135.19, 129.68, 129.06, 110.40, 105.74, 42.16, 30.92.



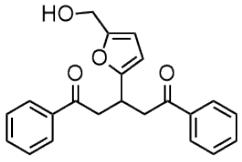
3h

3-(furan-2-yl)-1,5-di-p-tolylpentane-1,5-dione (3h). ^{1}H NMR (500 MHz, Chloroform-d) δ 7.87 (d, *J* = 8.2 Hz, 4H), 7.26 – 7.24 (m, 5H), 6.22 (dd, *J* = 2.9, 1.9 Hz ,1H), 6.04 (d, *J* = 2.9 Hz, 1H), 4.18 – 4.15 (m, 1H), 3.40 – 3.39 (m, 4H), 2.40 (s, 6H).^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.05, 156.72, 144.04, 141.24, 134.52, 129.40, 128.41, 110.33, 105.51, 42.22, 31.07, 21.77.



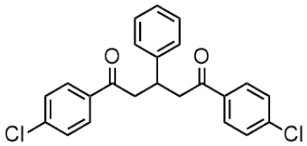
3i

1,5-diphenyl-3-(thiophen-2-yl)pentane-1,5-dione (3i). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 7.2 Hz, 4H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 4H), 6.99 (d, *J* = 4.5 Hz, 1H), 6.84 – 6.72 (m, 2H), 4.36 – 4.31 (m, 1H), 3.42 (d, *J* = 16.9 Hz, 2H), 3.32 (d, *J* = 16.9 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.11, 147.57, 136.86, 133.23, 128.67, 128.17, 126.78, 124.32, 123.38, 45.64, 32.43.



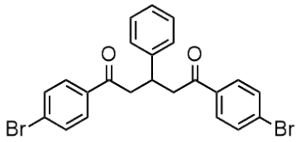
3j

3-(5-(hydroxymethyl)furan-2-yl)-1,5-diphenylpentane-1,5-dione (3j). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 7.3 Hz, 4H), 7.54 (t, *J* = 7.7 Hz, 2H), 7.44 (t, *J* = 7.7 Hz, 4H), 6.11 (d, *J* = 3.2 Hz, 1H), 5.98 (d, *J* = 3.2 Hz, 1H), 4.47 (s, 2H), 4.15 – 4.14 (m, 1H), 3.43 – 3.40 (m, 4H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.47, 156.48, 152.91, 136.79, 133.30, 128.70, 128.24, 108.63, 106.43, 57.47, 42.17, 31.09.



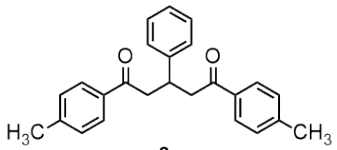
3k

1,5-bis(4-chlorophenyl)-3-phenylpentane-1,5-dione (3k). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 8.6 Hz, 4H), 7.41 (d, *J* = 8.6 Hz, 4H), 7.29 – 7.24 (m, 4H), 7.22 – 7.16 (m, 1H), 4.04 – 4.01 (m, 1H), 3.45 (d, *J* = 16.6 Hz, 2H), 3.30 (d, *J* = 12.5 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.47, 143.55, 139.74, 135.32, 129.70, 129.07, 128.87, 127.53, 127.04, 44.94, 37.35.



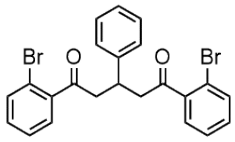
3l

1,5-bis(4-bromophenyl)-3-phenylpentane-1,5-dione (3l). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.6 Hz, 4H), 7.58 (d, *J* = 8.6 Hz, 4H), 7.30 – 7.23 (m, 4H), 7.21 – 7.15 (m, 1H), 4.04 – 4.01 (m, 1H), 3.45 (d, *J* = 16.6 Hz, 2H), 3.29 (d, *J* = 16.6 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.54, 143.39, 135.57, 131.94, 129.68, 128.75, 128.35, 127.40, 126.92, 44.79, 37.18.



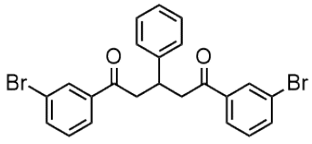
3m

3-phenyl-1,5-di-p-tolylpentane-1,5-dione (3m). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 8.2 Hz, 4H), 7.27 – 7.25 (m, 4H), 7.23 – 7.21 (m, 4H), 7.17 – 7.16 (m, 1H), 4.08 – 4.02 (m, 1H), 3.47 (d, *J* = 16.5 Hz, 2H), 3.31 (d, *J* = 16.5 Hz, 2H), 2.38 (s, 6H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.47, 143.55, 139.74, 135.33, 129.70, 129.07, 128.87, 127.53, 127.04, 44.94, 37.35.



3n

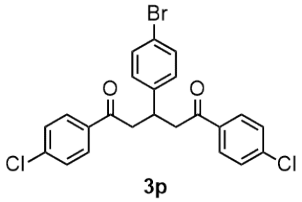
1,5-bis(2-bromophenyl)-3-phenylpentane-1,5-dione (3n). ^{1}H NMR (500 MHz, Chloroform-d) δ 7.54 (d, *J* = 7.8 Hz, 2H), 7.29 – 7.20 (m, 8H), 7.19 – 7.14 (m, 3H), 3.96 (t, *J* = 7.2 Hz, 1H), 3.44 (d, *J* = 17.0 Hz, 2H), 3.31 (d, *J* = 17.0 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-d) δ 202.37, 142.74, 141.55, 133.60, 131.57, 128.64, 128.50, 127.74, 127.41, 126.90, 118.61, 48.63, 37.20.



3o

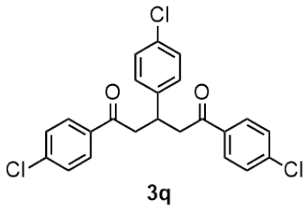
1,5-bis(3-bromophenyl)-3-phenylpentane-1,5-dione (3o). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.05 (t, *J* = 1.6 Hz, 2H), 7.87 (d, *J* = 7.8 Hz, 2H), 7.67 (d, *J* = 7.9 Hz, 2H), 7.33 (t, *J* = 7.9 Hz, 2H), 7.31 – 7.28 (m, 4H), 7.21 – 7.18 (m, 1H), 4.06 – 4.00 (m, 1H), 3.45 (d, *J* = 16.8 Hz, 2H), 3.32 (d, *J* = 16.8 Hz, 2H). ^{13}C NMR (126 MHz,

Chloroform-*d*) δ 197.20, 143.45, 138.67, 136.13, 131.34, 130.35, 128.89, 127.53, 127.07, 126.78, 123.13, 44.93, 37.02.



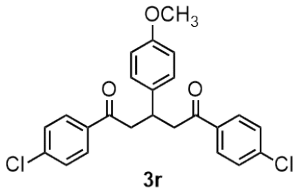
3p

1,5-bis(4-bromophenyl)-3-(4-chlorophenyl)pentane-1,5-dione (3p). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 – 7.77 (m, 4H), 7.59 – 7.56 (m, 4H), 7.26 – 7.18 (m, 4H),4.00 – 3.99 (m, 1H), 3.43 (d, *J* = 16.9 Hz, 2H), 3.26 (d, *J* = 16.9 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.24, 142.03, 135.51, 132.65, 132.09, 129.72, 128.94, 128.61, 44.70, 36.52.



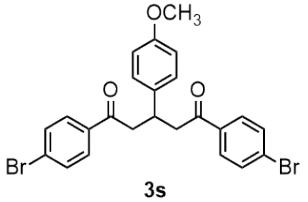
3q

1,3,5-tris(4-chlorophenyl)pentane-1,5-dione (3q). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 8.5 Hz, 4H), 7.42 (d, *J* = 8.5 Hz, 4H), 7.26 – 7.18 (m, 4H), 4.03 – 3.99 (m, 1H), 3.47 – 3.41 (m, 2H), 3.29 – 3.24 (m, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.08, 142.07, 139.91, 135.17, 132.72, 129.66, 129.13, 128.98, 128.97, 44.76, 36.62.



3r

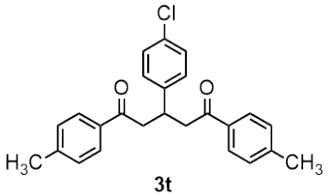
1,5-bis(4-chlorophenyl)-3-(4-methoxyphenyl)pentane-1,5-dione (3r). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.86 (d, *J* =8.4 Hz, 4H), 7.38 (d, *J* = 8.6 Hz, 4H), 7.16 (d, *J* = 8.5 Hz, 2H), 6.79 (d, *J* = 8.5 Hz, 2H), 3.98 – 3.95 (m, 1H), 3.72 (s, 3H), 3.42 (d, *J* = 16.6 Hz, 2H), 3.25 (d, *J* = 16.6 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.43, 158.34, 139.48, 135.42, 135.20, 129.57, 128.88, 128.37, 114.06, 55.16, 45.04, 36.47.



3s

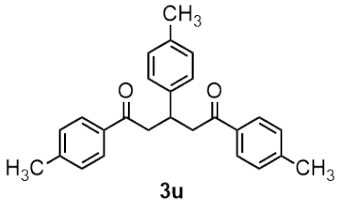
1,5-bis(4-bromophenyl)-3-(4-methoxyphenyl)pentane-1,5-dione (3s). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 8.5 Hz, 4H), 7.38 (d, *J* = 8.5 Hz, 4H), 7.16 (d, *J* = 8.6 Hz, 2H), 6.79 (d, *J* = 8.6 Hz, 2H), 3.98 – 3.95 (m, 1H), 3.72 (s, 3H), 3.44 (d, *J* = 16.6 Hz, 2H), 3.25 (d, *J* = 16.6 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ

197.43, 158.34, 139.48, 135.42, 135.20, 129.57, 128.88, 128.37, 114.06, 55.16, 45.04, 36.47.



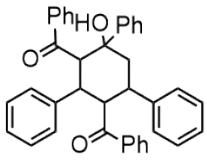
3t

3-(4-chlorophenyl)-1,5-di-p-tolylpentane-1,5-dione (3t). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.2 Hz, 4H), 7.24 – 7.18 (m, 8H), 4.09 – 3.99 (m, 1H), 3.43 (d, *J* = 16.7 Hz, 2H), 3.28 (d, *J* = 16.7 Hz, 2H), 2.37 (s, 6H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.89, 144.01, 142.56, 134.36, 132.21, 129.34, 128.97, 128.68, 128.26, 44.67, 36.66, 21.66.



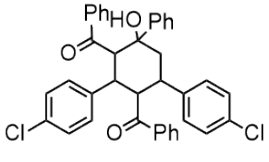
3u

1,3,5-tri-p-tolylpentane-1,5-dione (3u). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.2 Hz, 4H), 7.23 (d, *J* = 8.2 Hz, 4H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 4.03 – 3.99 (m, 1H), 3.43 (d, *J* = 16.4 Hz, 2H), 3.30 (d, *J* = 16.4 Hz, 2H), 2.39 (s, 6H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 198.51, 143.91, 141.06, 136.22, 134.64, 129.41, 129.38, 128.43, 127.43, 45.13, 37.17, 21.76, 21.15.



4a

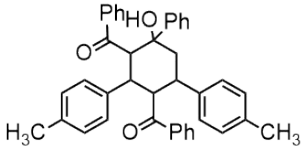
(4-hydroxy-2,4,6-triphenylcyclohexane-1,3-diyl)bis(phenylmethanone) (4a). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 7.5 Hz, 2H), 7.28 – 7.26 (m, 4H), 7.24 – 7.21 (m, 4H), 7.20 – 7.17 (m, 3H), 7.09 (t, *J* = 7.6 Hz, 2H), 7.05 – 7.01 (m, 5H), 6.98 (t, *J* = 7.0 Hz, 1H), 6.82 (t, *J* = 7.5 Hz, 2H), 6.72 (t, *J* = 7.5 Hz, 1H), 5.38 (d, *J* = 2.5 Hz, 1H), 4.49 (d, *J* = 11.0 Hz, 1H), 4.24 – 4.20 (m, 2H), 4.09 – 4.03 (m, 1H), 2.54 – 2.48 (m, 1H), 2.27 (d, *J* = 14.1 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 207.32, 203.72, 146.07, 142.26, 139.16, 138.80, 138.32, 132.80, 131.91, 128.51, 128.33, 128.23, 128.14, 127.86, 127.82, 127.70, 127.54, 127.15, 127.06, 126.86, 124.97, 75.50, 56.94, 56.92, 48.23, 46.03, 43.51. HRMS: (ESI) $[M+H]^+$ calcd. for $C_{38}H_{32}O_3$, 537.2430 found, 537.2437.



4b

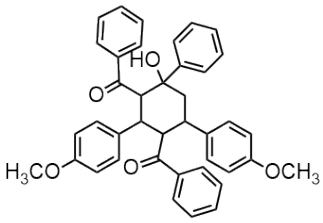
(2,6-bis(4-chlorophenyl)-4-hydroxy-4-phenylcyclohexane-1,3-

diyl)bis(phenylmethanone) (4b). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 7.7 Hz, 2H), 7.28 – 7.23 (m, 6H), 7.20 – 7.17 (m, 4H), 7.10 – 7.05 (m, 9H), 6.80 (d, *J* = 8.1 Hz, 2H), 5.32 (d, *J* = 2.5 Hz, 1H), 4.44 (d, *J* = 10.7 Hz, 1H), 4.18 – 4.11 (m, 2H), 4.07 – 4.01 (m, 1H), 2.48 – 2.42 (m, 1H), 2.22 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.81, 203.0, 145.64, 140.60, 138.68, 138.07, 137.31, 133.14, 132.89, 132.47, 129.43, 128.68, 128.43, 128.41, 128.03, 128.02, 127.83, 127.52, 127.34, 124.88, 75.39, 56.72, 56.67, 47.61, 45.90, 42.94.



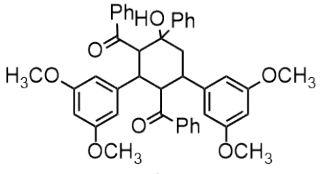
4c

(4-hydroxy-4-phenyl-2,6-di-p-tolylcyclohexane-1,3-diyl)bis(phenylmethanone) (4c). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.49 (m, 2H), 7.26 – 7.21 (m, 6H), 7.20 – 7.13 (m, 5H), 7.06 – 6.99 (m, 6H), 6.88 (d, *J* = 7.8 Hz, 2H), 6.61 (d, *J* =5.7 Hz, 2H), 5.33 (d, *J* = 2.5 Hz, 1H), 4.44 (d, *J* = 11.1 Hz, 1H), 4.15 (d, *J* = 14.2 Hz, 2H), 4.03 – 3.98 (m, 1H), 2.50 – 2.41 (m, 1H), 2.21 (d, *J* = 14.2 Hz, 1H), 2.12 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 207.49, 203.92, 146.15, 139.29, 138.43, 136.47, 136.23, 132.62, 131.69, 129.13, 128.84, 128.27, 127.97, 127.89, 127.75, 127.63, 127.06, 124.99, 75.54, 57.23, 57.19, 47.78, 46.24, 43.10, 29.85, 20.99, 20.81.



4d

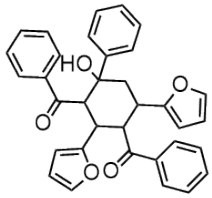
(4-hydroxy-2,6-bis(4-methoxyphenyl)-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone) (4d). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.51 (m, 2H), 7.25 – 7.20 (m, 7H), 7.19 – 7.15 (m, 4H), 7.07 – 7.02 (m, 6H), 6.65 – 6.61 (m, 2H), 6.38 – 6.34 (m, 2H), 5.34 (d, *J* = 2.4 Hz, 1H), 4.45 – 4.40 (m, 1H), 4.14 – 4.09 (m, 2H), 4.00 (d, *J* = 14.0 Hz, 1H), 3.64 (s, 3H), 3.47 (s, 3H), 2.47 – 2.41 (m, 1H), 2.20 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 207.53, 204.05, 158.37, 146.14, 139.25, 138.39, 134.49, 132.75, 131.87, 130.95, 129.04, 128.30, 127.87, 127.84, 127.76, 127.58, 127.09, 124.97, 113.94, 113.70, 75.57, 57.48, 57.23, 55.30, 55.15, 47.40, 46.30, 42.69, 29.85.



4e

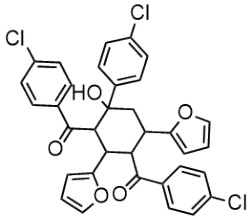
(2,6-bis(3,5-dimethoxyphenyl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone) (4e). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.52 (d, *J* =

7.7 Hz, 2H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.26 – 7.23 (m, 2H), 7.18 (t, *J* = 7.6 Hz, 2H), 7.12 – 7.03 (m, 5H), 6.41 (s, 2H), 6.26 (s, 2H), 6.08 (s, 1H), 5.79 (s, 1H), 5.40 (s, 1H), 4.41 (d, *J* = 11.6 Hz, 1H), 4.17 (t, *J* = 11.0 Hz, 1H), 4.07 (t, *J* = 11.3 Hz, 1H), 3.99 – 3.93 (m, 1H), 3.64 (s, 6H), 3.49 (s, 6H), 2.43 – 2.38 (m, 1H), 2.28 – 2.18 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 207.20, 203.32, 160.77, 160.49, 145.98, 144.67, 141.08, 139.22, 138.34, 132.82, 132.01, 128.34, 127.88, 127.81, 127.76, 127.68, 127.17, 124.93, 106.42, 99.41, 98.98, 75.48, 56.70, 56.13, 55.42, 55.36, 48.41, 46.17, 43.83.



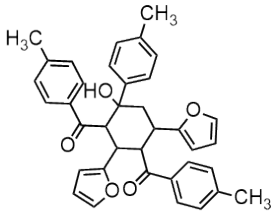
4f

(2,6-di(furan-2-yl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone) (4f). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.58 – 7.56 (m, 2H), 7.52 – 7.50 (m, 2H), 7.47 – 7.42 (m, 2H), 7.37 – 7.30 (m, 2H),7.24 (t, *J* = 11.1 Hz, 2H), 7.20 – 7.14 (m, 4H), 7.09 (d, *J* = 1.9 Hz, 1H), 7.07 – 7.03 (m, 1H), 6.85 (d, *J* = 1.8 Hz, 1H), 6.00 – 5.99 (m, 1H), 5.92 – 5.91 (m, 1H),5.75 – 5.74 (m, 1H),5.68 – 5.67 (m, 1H), 5.35 (d, *J* = 2.5 Hz, 1H), 4.62 (d, *J* = 11.7 Hz, 1H), 4.48 (t, *J* = 11.7 Hz, 1H), 4.19 (t, *J* = 11.7 Hz, 1H), 4.12 – 4.06 (m, 1H), 2.48 – 2.42 (m, 1H), 2.21 (d, *J* = 14.1 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.99, 203.24, 155.34, 151.76, 145.68, 141.48, 141.26, 133.18, 132.44, 128.34, 128.04, 128.02, 127.96, 127.79, 127.16, 124.87, 110.24, 110.17, 108.53, 106.35, 75.16, 53.87, 51.86, 43.98, 41.21, 36.70.



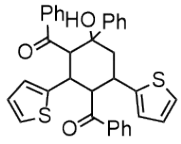
4g

(4-(4-chlorophenyl)-2,6-di(furan-2-yl)-4-hydroxycyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone) (4g). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.53 – 7.49 (m, 2H), 7.45 – 7.40 (m, 4H), 7.22 – 7.16 (m, 6H), 7.10 (d, *J* = 1.9 Hz, 1H), 6.91 (d, *J* = 1.4 Hz, 1H), 6.02 (d, *J* = 3.2 Hz, 1H), 5.91 (d, *J* = 3.2 Hz, 1H), 5.73 (d, *J* = 1.3 Hz, 2H), 5.32 (d, *J* = 2.6 Hz, 1H), 4.50 (d, *J* = 11.7 Hz, 1H), 4.40 (t, *J* = 11.4 Hz, 1H), 4.14 (t, *J* = 11.4 Hz, 1H), 4.06 – 4.01 (m, 1H), 2.39 – 2.33 (m, 1H), 2.17 (d, *J* = 14.1 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.36, 201.77, 154.82, 151.32, 144.22, 141.70, 141.43, 140.23, 139.07, 135.87, 135.28, 133.21, 129.30, 129.17, 128.66, 128.61, 128.45, 126.30, 110.53, 110.33, 108.76, 106.56, 74.95, 53.48, 51.67, 43.92, 41.11, 36.63.



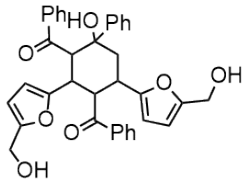
4h

(2,6-di(furan-2-yl)-4-hydroxy-4-(p-tolyl)cyclohexane-1,3-diyl)bis(p-tolylmethanone) (4h). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.37 (m, 4H), 7.08 (d, *J* = 1.9 Hz, 1H), 7.07 – 6.90 (m, 6H), 6.85 (d, *J* = 1.8 Hz, 1H), 5.99 (d, *J* = 3.2 Hz, 1H), 5.89 (d, *J* = 3.1 Hz, 1H), 5.71 (d, *J* = 3.2 Hz, 1H), 5.67 (d, *J* = 3.2 Hz, 1H), 5.44 (d, *J* = 2.7 Hz, 1H), 4.57 (d, *J* = 11.8 Hz, 1H), 4.42 (t, *J* = 11.4 Hz, 1H), 4.16 (t, *J* = 11.4 Hz, 1H), 4.09 – 4.04 (m, 1H), 2.42 – 2.36 (m, 1H), 2.28 (s, 3H), 2.26 (s, 3H), 2.22 – 2.18 (m, 1H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.43, 202.68, 155.61, 152.01, 144.05, 143.03, 141.35, 141.17, 136.56, 134.89, 128.99, 128.75, 128.74, 128.22, 127.95, 124.77, 110.17, 110.12, 108.34, 106.18, 75.06, 53.34, 51.64, 44.30, 41.27, 36.71, 21.71, 21.68, 20.98.



4i

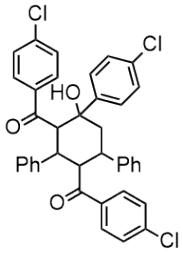
(4-hydroxy-4-phenyl-2,6-di(thiophen-2-yl)cyclohexane-1,3-diyl)bis(phenylmethanone) (4i). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.45 (d, *J* = 7.8 Hz, 2H), 7.38 (d, *J* = 7.8 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.23 – 7.19 (m, 2H), 7.12 – 7.07 (m, 4H), 7.05 – 7.0 (m, 2H), 6.97 (t, *J* = 5.0 Hz, 1H), 6.87 (d, *J* = 5.0 Hz, 1H), 6.73 – 6.50 (m, 4H), 6.32 (d, *J* = 5.0 Hz, 1H), 5.21 (d, *J* = 2.5 Hz, 1H), 4.47 (t, *J* = 11.3 Hz, 1H), 4.40 – 4.27 (m, 2H), 4.05 (t, *J* = 11.3 Hz, 1H), 2.44 – 2.38 (m, 1H), 2.36 – 2.29 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.68, 203.09, 145.49, 145.17, 142.06, 138.63, 137.84, 132.93, 132.16, 128.23, 127.92, 127.75, 127.71, 127.66, 127.14, 127.01, 126.57, 126.27, 125.56, 124.83, 123.91, 123.26, 75.23, 58.93, 57.71, 46.90, 43.33, 39.04.



4j

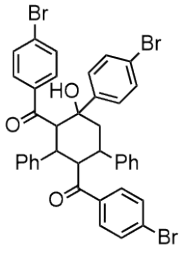
(4-hydroxy-2,6-bis(5-(hydroxymethyl)furan-2-yl)-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone) (4j). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.70 – 7.65 (m, 2H), 7.54 – 7.49 (m, 4H), 7.41 – 7.37 (m, 1H), 7.36 – 7.33 (m, 1H), 7.29 – 7.26 (m, 2H), 7.21 – 7.17 (m, 4H), 7.10 – 7.05 (m, 1H), 5.93 – 5.87 (m, 2H), 5.70 (d, *J* = 3.2 Hz, 1H), 5.56 (d, *J* = 3.2 Hz, 1H), 5.36 (d, *J* = 2.7 Hz, 1H), 4.66 (d, *J* = 11.7 Hz, 1H), 4.58 (t, *J* = 11.7 Hz, 1H), 4.34 – 4.27 (m, 2H), 4.12 – 4.03 (m, 4H), 2.47 – 2.40 (m, 1H), 2.25 – 2.19 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.83, 203.12,

155.58, 153.21, 152.85, 151.99, 138.00, 137.42, 133.33, 132.66, 128.42, 128.15, 128.13, 128.07, 127.96, 127.26, 124.82, 109.33, 108.59, 108.55, 107.12, 75.21, 57.49, 57.20, 53.65, 51.66, 44.07, 41.40, 36.85.



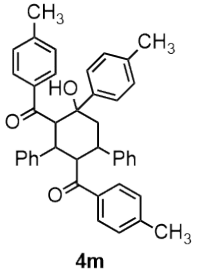
4k

(4-(4-chlorophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone) (4k). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 8.6 Hz, 2H), 7.3 (d, *J* = 8.6 Hz, 2H), 7.25 – 7.20 (m, 4H), 7.17 (d, *J* = 8.5 Hz, 2H), 7.15 – 7.11 (m, 4H), 7.13 – 7.06 (m, 4H), 7.03 – 7. 0 (m, 1H), 6.88 (t, *J* = 7.5 Hz, 2H), 6.82 – 6.77 (m, 1H), 5.34 (d, *J* = 2.5 Hz, 1H), 4.41 – 4.31 (m, 1H), 4.16 – 4.08 (m, 2H), 4.03 – 3.98 (m, 1H),2.44 – 2.38 (m, 1H), 2.21 (d, *J* = 14.1 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.63, 202.28, 144.57, 141.75, 139.84, 138.52, 138.40, 137.16, 136.13, 133.20, 129.23, 128.89, 128.70, 128.58, 128.51, 128.42, 128.12, 127.98, 127.49, 127.18, 126.39, 75.26, 56.70, 56.49, 48.12, 45.83, 43.41.



4l

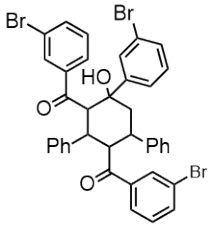
(4-(4-bromophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,3-diyl)bis((4-bromophenyl)methanone) (4l). ^{1}H NMR (500 MHz, Chloroform-d) δ 7.45 – 7.37 (m, 2H), 7.35 – 7.30 (m, 2H), 7.27 – 7.21 (m, 4H), 7.20 – 7.04 (m, 10H), 7.04 – 6.99 (m, 1H), 6.91 – 6.83 (m, 2H), 6.82 – 6.74 (m, 1H), 5.35 (d, *J* = 2.4 Hz, 1H), 4.46 – 4.26 (m, 1H), 4.17 – 4.09 (m, 2H), 4.07 – 3.91 (m, 1H), 2.41 (d, *J* = 14.0, 1H), 2.22 (d, *J* = 14.0, 1H). ^{13}C NMR (126 MHz, Chloroform-d) δ 205.74, 202.37, 144.96, 141.57, 138.20, 137.42, 136.39, 131.41, 131.29, 130.97, 129.16, 128.86, 128.59, 128.56, 128.41, 127.84, 127.40, 127.16, 127.08, 126.61, 121.25, 75.18, 56.56, 56.25, 47.99, 45.65, 43.28.



4m

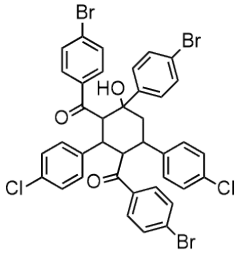
(4-hydroxy-2,6-diphenyl-4-(p-tolyl)cyclohexane-1,3-diyl)bis(p-tolylmethanone)

(4m). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.24 (m, 3H), 7.19 (t, *J* = 7.9 Hz, 4H), 7.14 – 7.03 (m, 4H), 6.97 (d, *J* = 7.8 Hz, 2H), 6.86 – 6.78 (m, 6H), 6.70 (t, *J* = 7.5 Hz, 1H), 5.46 (d, *J* = 2.4 Hz, 1H), 4.44 (d, *J* = 11.0 Hz, 1H), 4.20 – 4.13 (m, 2H), 4.07 – 4.00 (m, 1H), 2.48 – 2.44 (m, 1H), 2.22 (d, *J* = 3.5 Hz, 1H), 2.19 (s, 3H), 2.17 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.72, 203.08, 143.65, 143.36, 142.55, 142.45, 138.98, 136.51, 128.94, 128.51, 128.42, 128.40, 128.17, 128.12, 128.10, 127.76, 126.87, 126.71, 124.85, 75.39, 56.51, 56.34, 48.24, 46.35, 43.50, 21.60, 21.52, 20.95.



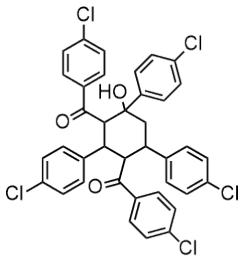
4o

(4-(3-bromophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,3-diyl)bis((3-bromo phenyl)methanone) (4o). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.69 (s, 1H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.39 (d, *J* = 7.0 Hz, 1H), 7.36 – 7.29 (m, 2H), 7.28 – 7.24 (m, 2H), 7.21 (d, *J* = 7.8 Hz, 1H), 7.18 (d, *J* = 7.9 Hz, 1H), 7.16 – 7.11 (m, 4H), 7.08 (t, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.98 (t, *J* = 7.9 Hz, 1H), 6.94 – 6.89 (m, 3H), 6.82 (t, *J* = 7.5 Hz, 1H), 5.16 (d, *J* = 2.5 Hz, 1H), 4.42 – 4.29 (m, 1H), 4.16 – 4.09 (m, 2H), 4.01 – 3.96 (m, 1H), 2.47 – 2.41 (m, 1H), 2.25 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.56, 202.29, 148.20, 141.54, 140.61, 139.66, 138.29, 135.90, 134.86, 130.81, 130.56, 130.50, 130.11, 129.59, 129.38, 128.72, 128.58, 128.37, 128.02, 127.61, 127.26, 126.23, 125.88, 123.59, 122.85, 122.31, 122.04, 75.24, 57.08, 57.01, 47.91, 45.61, 43.45.



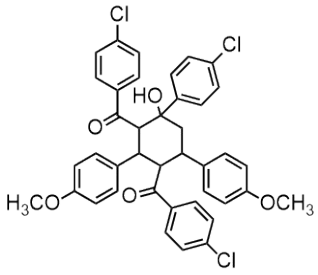
4p

(4-(4-bromophenyl)-2,6-bis(4-chlorophenyl)-4-hydroxycyclohexane-1,3-diyl)bis((4-bromophenyl)methanone) (4p). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.37 (d, *J* =8.5 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.28 – 7.26 (m, 1H), 7.25 – 7.24 (m, 1H), 7.19 – 7.07 (m, 10H), 7.06 – 7.01 (m, 2H), 6.88 – 6.85 (m, 2H), 5.23 (d, *J* = 2.5 Hz, 1H), 4.32 (d, *J* = 11.5 Hz, 1H), 4.15 – 4.11 (m, 1H), 4.05 – 3.97 (m, 2H), 2.38 – 2.32 (m, 1H), 2.18 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.84, 202.62, 158.59, 158.50, 144.61, 139.75, 138.43, 137.19, 136.14, 133.88, 133.09, 130.40, 129.23, 128.93, 128.89, 128.51, 128.47, 128.41, 128.15, 126.36, 114.04, 113.86, 75.31, 57.16, 56.71, 55.27, 55.14, 55.10, 47.30, 46.10, 42.60.



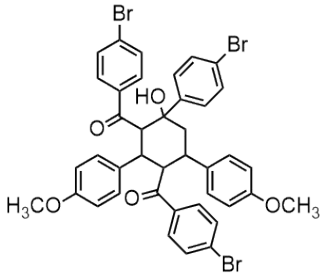
4q

(2,4,6-tris(4-chlorophenyl)-4-hydroxycyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone) (4q). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 8.6 Hz, 2H), 7.23 (t, *J* = 8.6 Hz, 4H), 7.19 – 7.14 (m, 4H), 7.13 – 7.09 (m, 6H), 7.08 (d, *J* = 3.0 Hz, 2H), 7.04 (d, *J* =10.0 Hz, 2H), 6.89 – 6.84 (m, 2H), 5.24 (d, *J* = 2.5 Hz, 1H), 4.32 (d, *J* = 11.5 Hz, 1H), 4.12 (t, *J* = 11.0 Hz, 1H), 4.06 – 3.96 (m, 2H), 2.37 – 2.33 (m, 1H), 2.18 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.04, 201.50, 144.10, 140.33, 140.10, 139.26, 136.91, 135.88, 133.43, 132.99, 129.29, 129.21, 128.89, 128.73, 128.68, 128.66, 128.49, 126.31, 75.14, 56.40, 56.27, 47.46, 45.74, 42.84.



4r

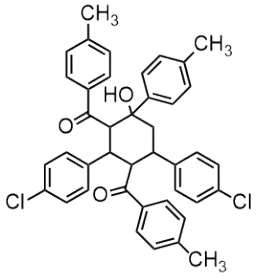
(4-(4-chlorophenyl)-4-hydroxy-2,6-bis(4-methoxyphenyl)cyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone) (4r). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 8.6 Hz, 2H), 7.22 – 7.12 (m, 8H), 7.08 (d, *J* = 8.6 Hz, 2H), 7.04 (d, *J* = 8.6 Hz, 2H), 6.99 (m, 2H), 6.65 – 6.64 (m, 2H), 6.41 – 6.39 (m, 2H), 5.30 (d, *J* = 2.4 Hz, 1H), 4.32 (d, *J* = 10.9 Hz, 1H), 4.08 – 4.02 (m, 2H), 3.96 – 3.91 (m, 1H), 3.65 (s, 3H), 3.51 (s, 3H), 2.37 – 2.32 (m, 2H), 2.16 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 205.24, 201.67, 144.62, 140.06, 137.06, 136.87, 136.29, 133.42, 133.00, 131.63, 131.46, 129.27, 129.24, 128.96, 128.89, 128.73, 126.64, 121.58, 75.18, 56.38, 56.19, 47.45, 45.70, 42.82, 27.06.



4s

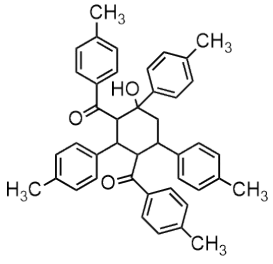
(4-(4-bromophenyl)-4-hydroxy-2,6-bis(4-methoxyphenyl)cyclohexane-1,3-

diyl)bis((4-bromophenyl)methanone) (4s). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.38(d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.26 – 7.20 (m, 4H), 7.15 – 7.13 (m, 2H), 7.12 – 7.08 (m, 4H), 6.99 (d, *J* = 7.7 Hz, 2H), 6.63 (d, *J* = 8.6 Hz, 2H), 6.40 (d, *J* = 8.8 Hz, 2H), 5.29 (d, *J* = 2.4 Hz, 1H), 4.33 – 4.27 (m, 1H), 4.06 – 4.03 (m, 2H), 3.96 – 3.91 (m, 1H), 3.66 (s, 3H), 3.52 (s, 3H), 2.37 – 2.32 (m, 1H), 2.16 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.02, 202.65, 145.18, 138.73, 137.70, 137.10, 136.69, 136.66, 135.31, 131.49, 131.35, 131.02, 129.35, 129.32, 129.14, 128.49, 127.81, 127.06, 126.75, 121.29, 75.34, 56.89, 56.68, 47.64, 46.00, 43.00, 21.03, 20.89.



4t

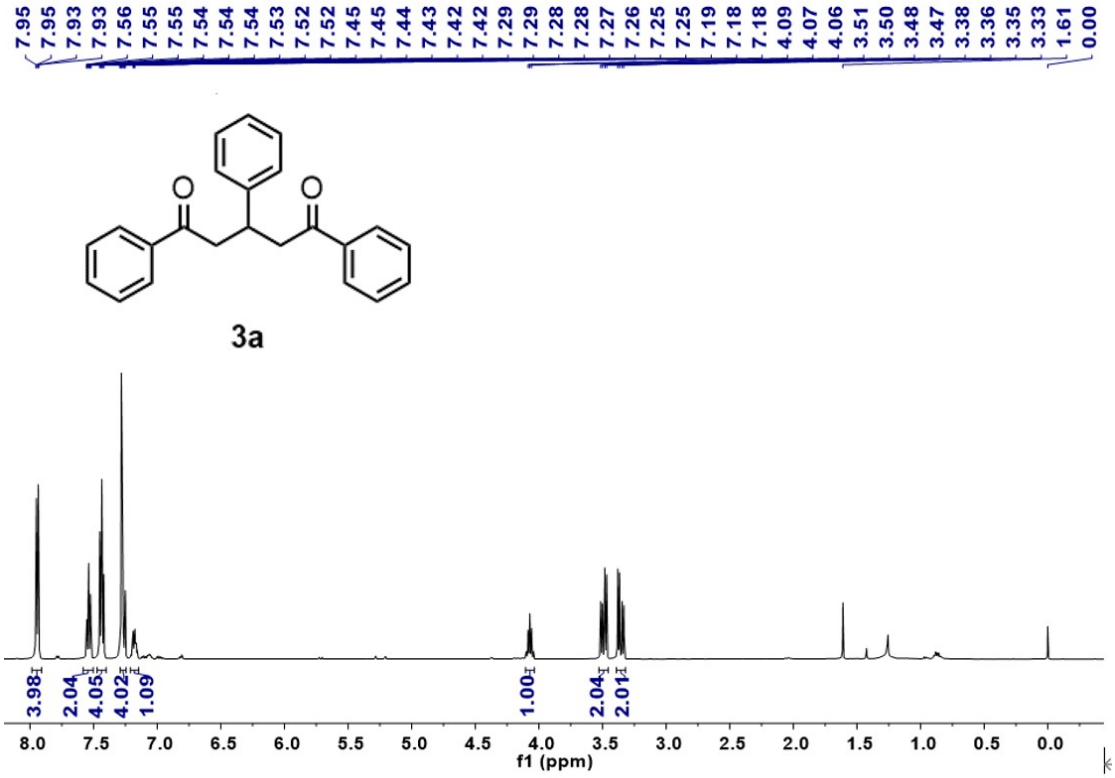
(2,6-bis(4-chlorophenyl)-4-hydroxy-4-(p-tolyl)cyclohexane-1,3-diyl)bis(p-tolylmethanone) (4t). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 7.9 Hz, 2H), 7.22 – 7.16 (m, 6H), 7.05 (d, *J* = 7.9 Hz, 4H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.89 (d, *J* = 8.3 Hz, 4H), 6.79 (d, *J* = 8.2 Hz, 2H), 5.40 (d, *J* = 2.5 Hz, 1H), 4.39 (d, *J* = 11.2 Hz, 1H), 4.17 – 4.05 (m, 2H), 4.01 (d, *J* = 12.2 Hz, 1H), 2.37 (d, *J* = 13.3 Hz, 2H), 2.23 (s, 6H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.22, 202.31, 144.17, 144.17, 142.93, 140.79, 137.52, 136.77, 135.98, 135.49, 132.67, 132.45, 129.41, 129.05, 128.75, 128.72, 128.61, 128.32, 128.16, 127.78, 124.76, 75.27, 56.22, 56.11, 47.57, 46.21, 42.92, 21.66, 21.59, 20.96.



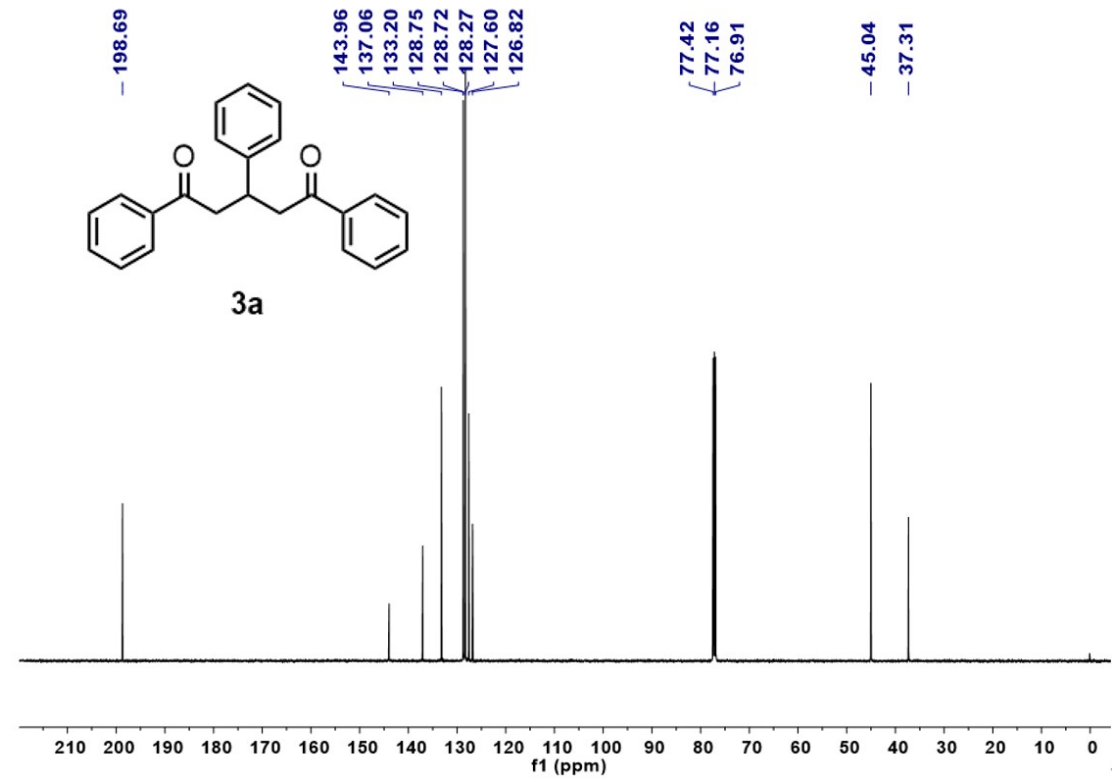
4u

(4-hydroxy-2,4,6-tri-p-tolylcyclohexane-1,3-diyl)bis(p-tolylmethanone) (4u). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 9.1 Hz, 4H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.01 – 6.93 (m, 4H), 7.01 – 6.93 (m, 6H), 6.61 (d, *J* = 7.9 Hz, 2H), 5.40 (d, *J* = 2.5 Hz, 2H), 4.39 (d, *J* = 11.0 Hz, 1H), 4.15 – 4.07 (m, 2H), 4.00 – 3.98 (m, 1H), 2.44 – 2.38 (m, 1H), 2.20 (s, 6H), 2.18 – 2.14 (m, 4H), 2.12 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 206.91, 203.25, 143.46, 143.43, 142.34, 139.48, 136.62, 136.42, 136.23, 136.05, 135.96, 135.87, 129.05, 128.89, 128.75, 128.43, 128.34, 128.21, 127.94, 127.89, 124.87, 75.43, 56.66, 47.75, 46.57, 43.08, 29.85, 21.60, 21.53, 21.01, 20.95, 20.84.

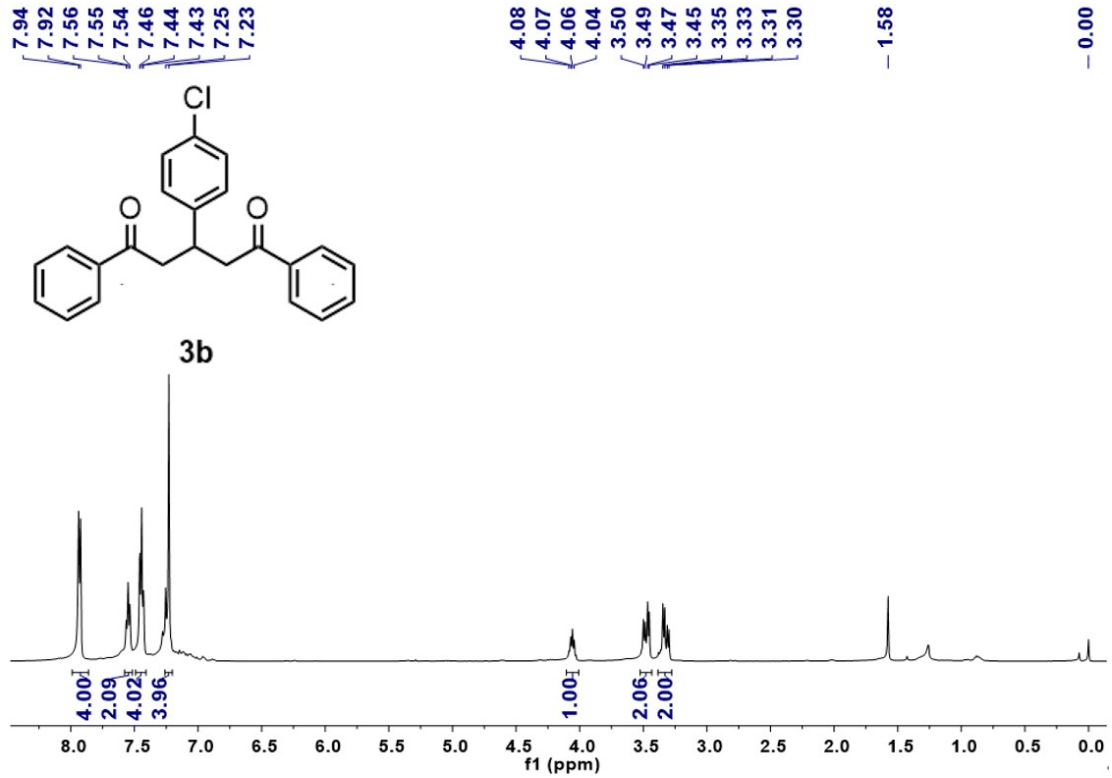
^{1}H NMR of **3a**



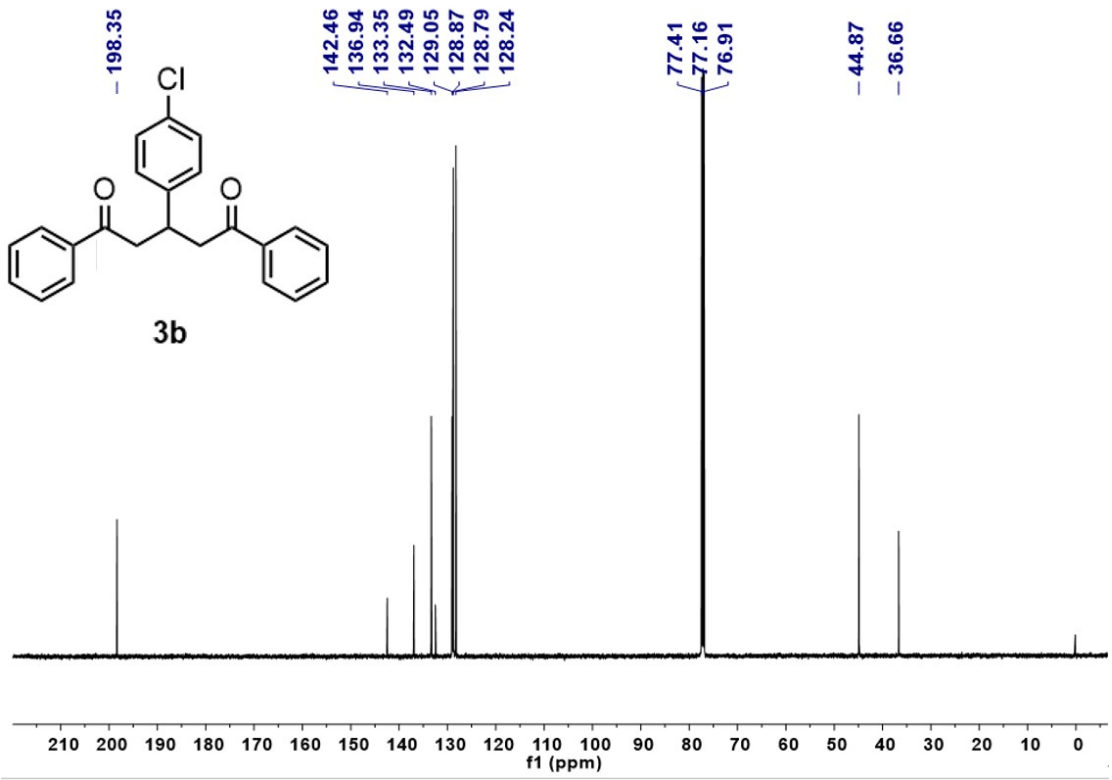
^{13}C NMR of **3a**



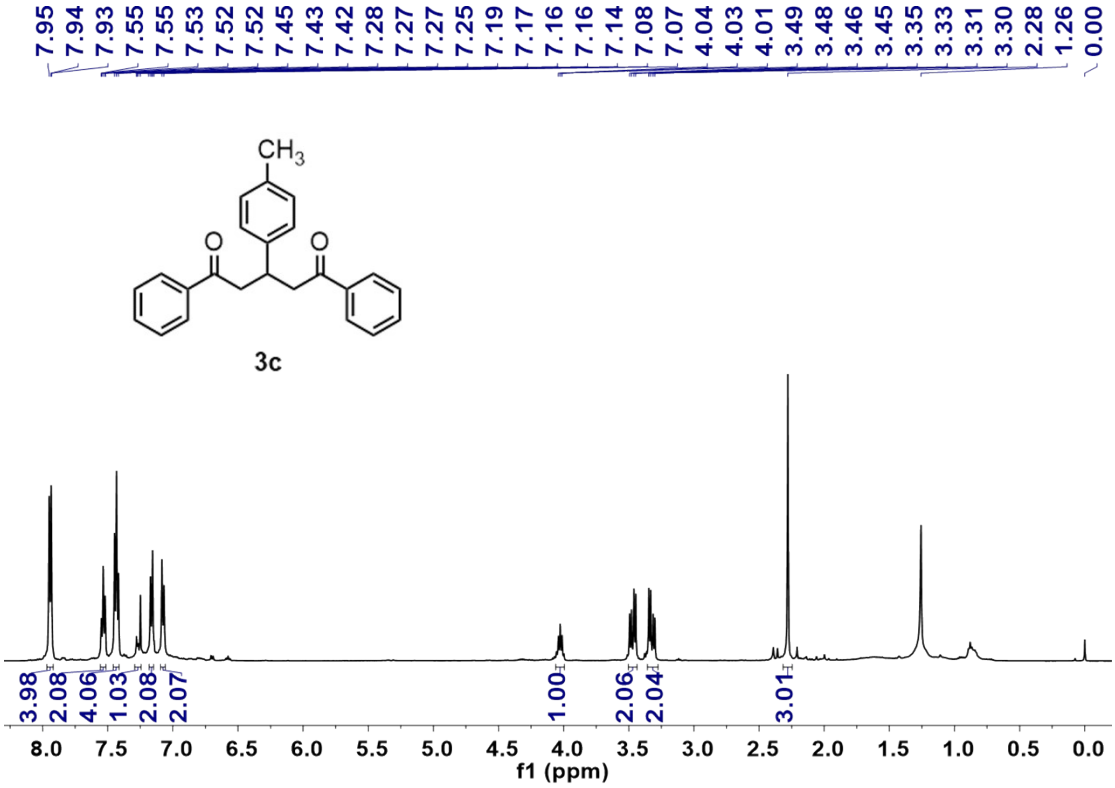
^{1}H NMR of **3b**



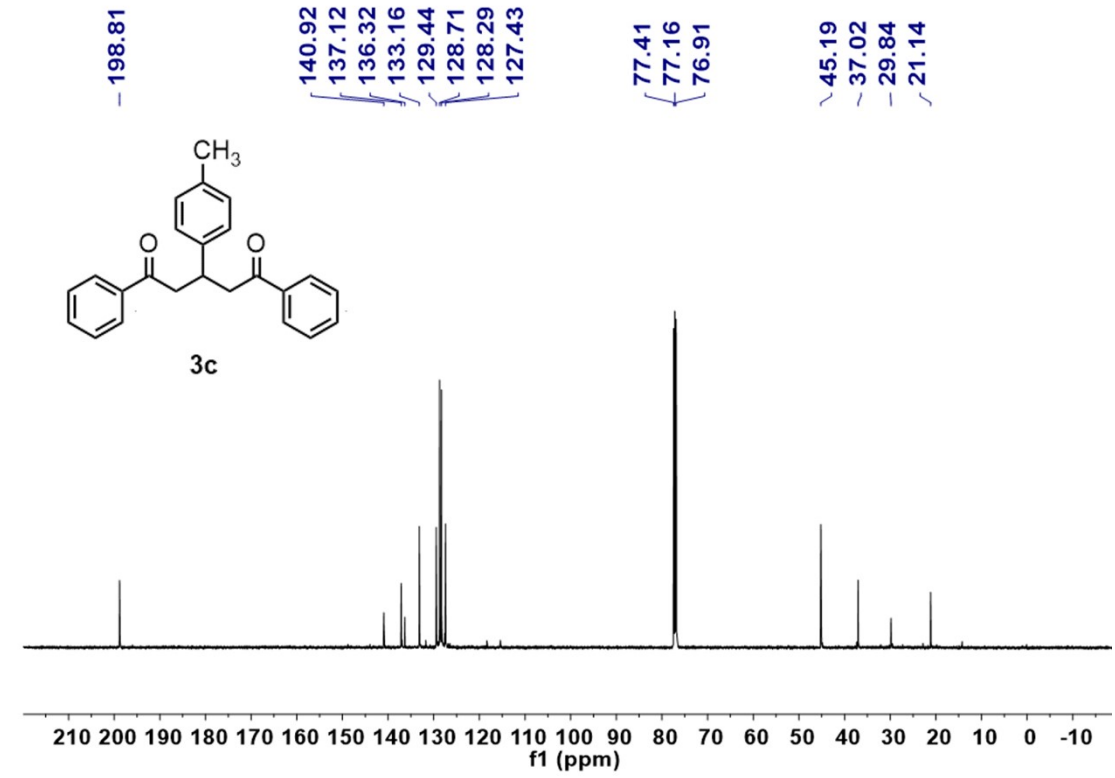
^{13}C NMR of **3b**



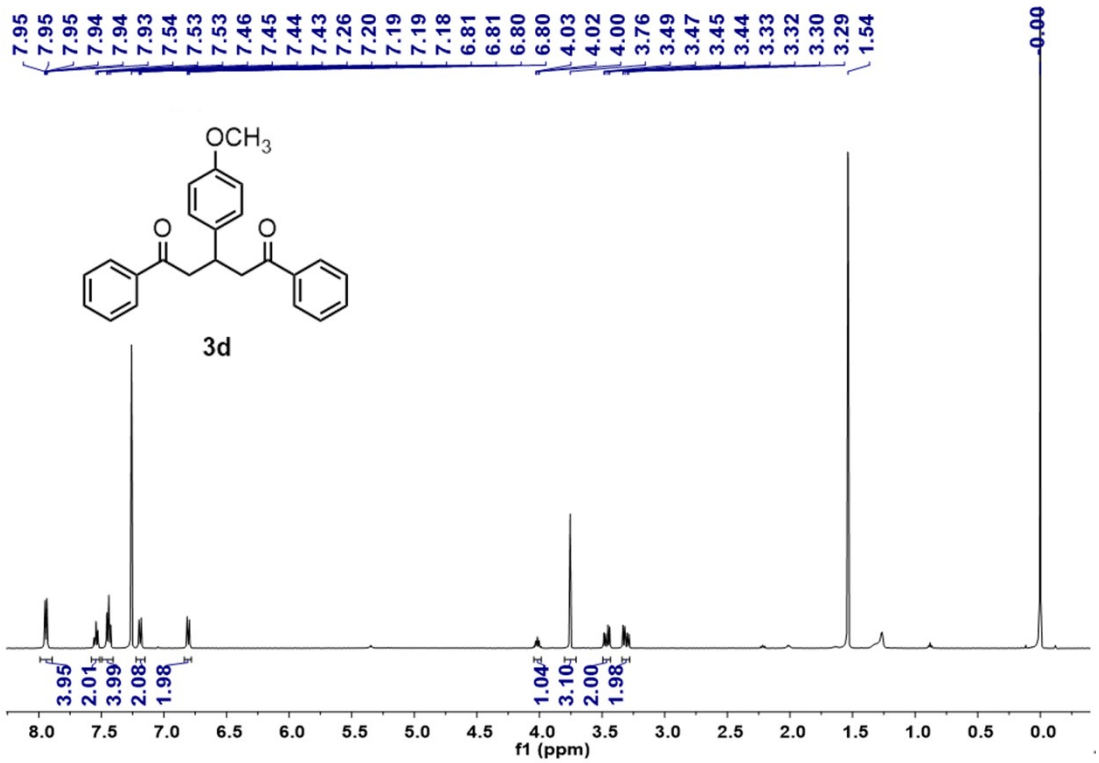
^{1}H NMR of **3c**



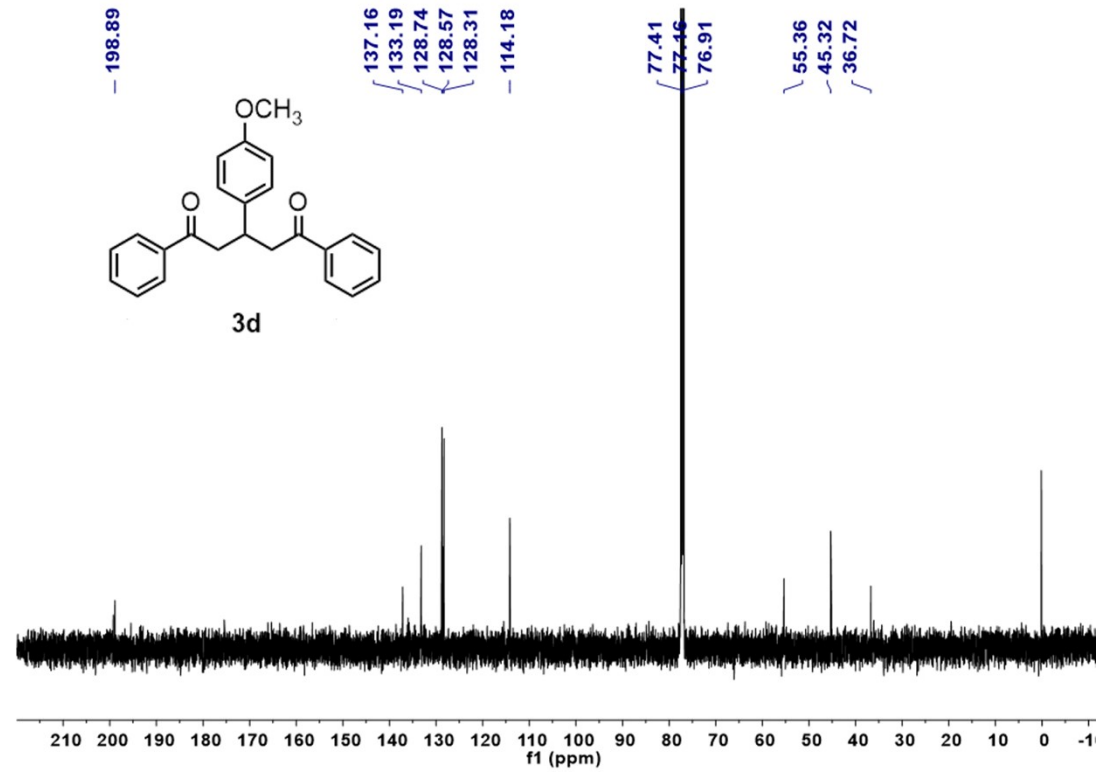
^{13}C NMR of **3c**



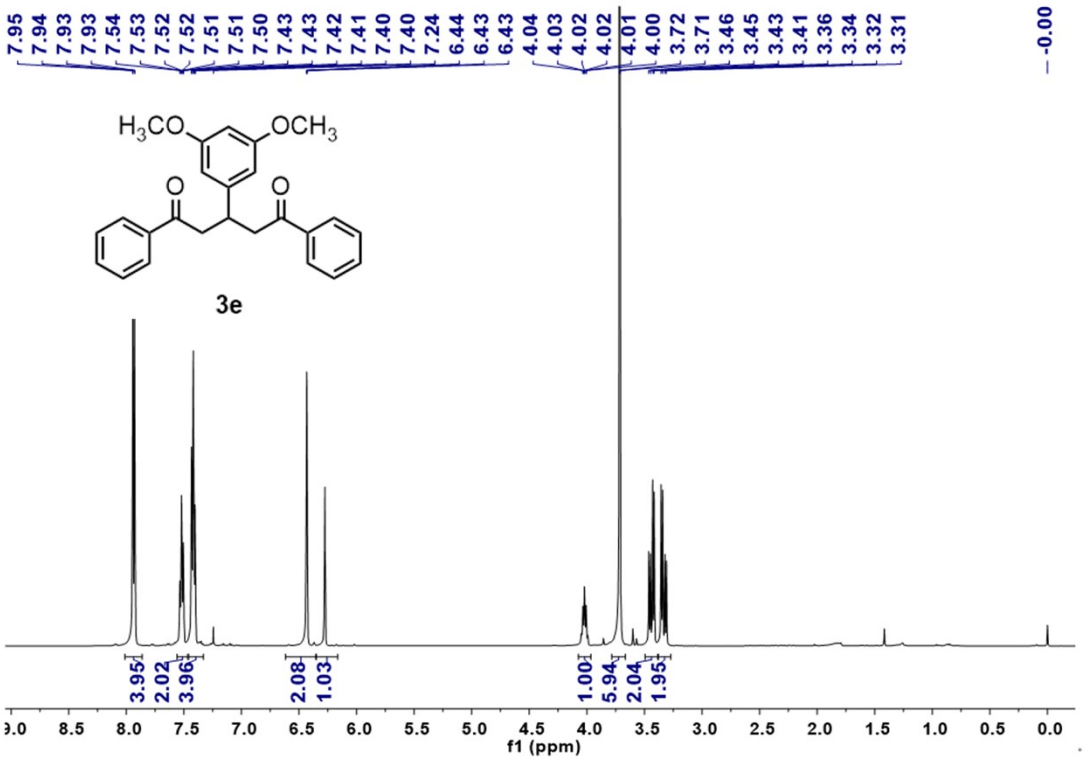
^{1}H NMR of **3d**



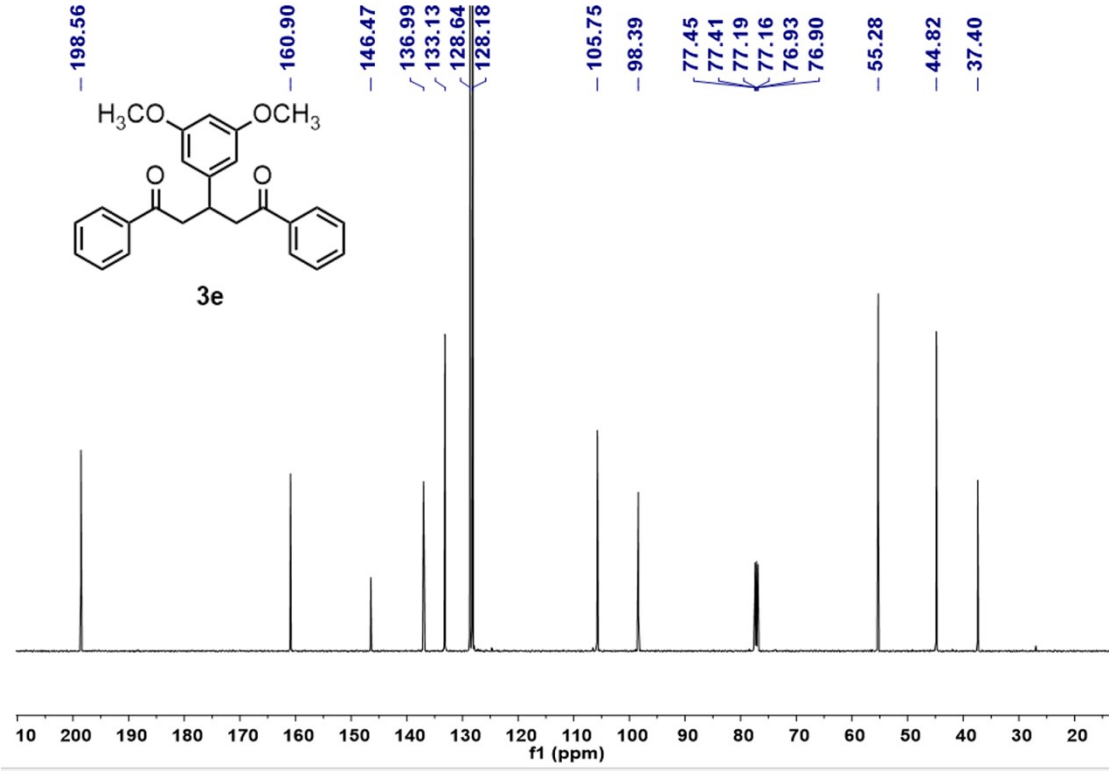
^{13}C NMR of **3d**



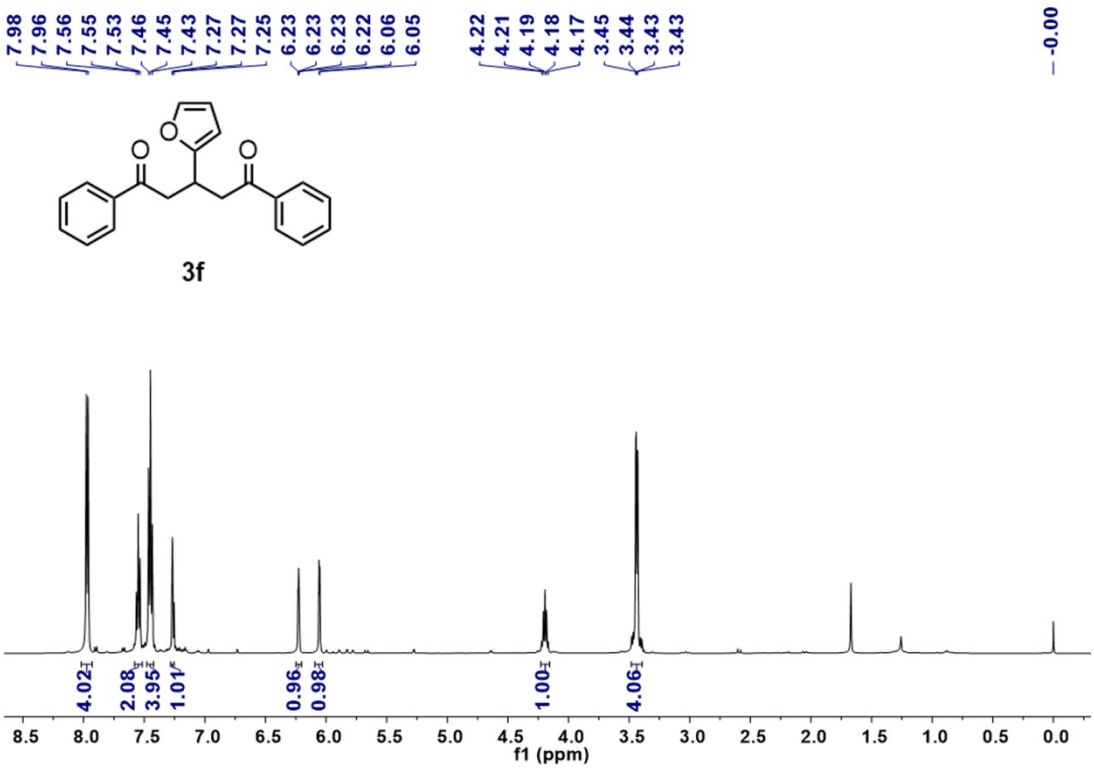
^{1}H NMR of **3e**



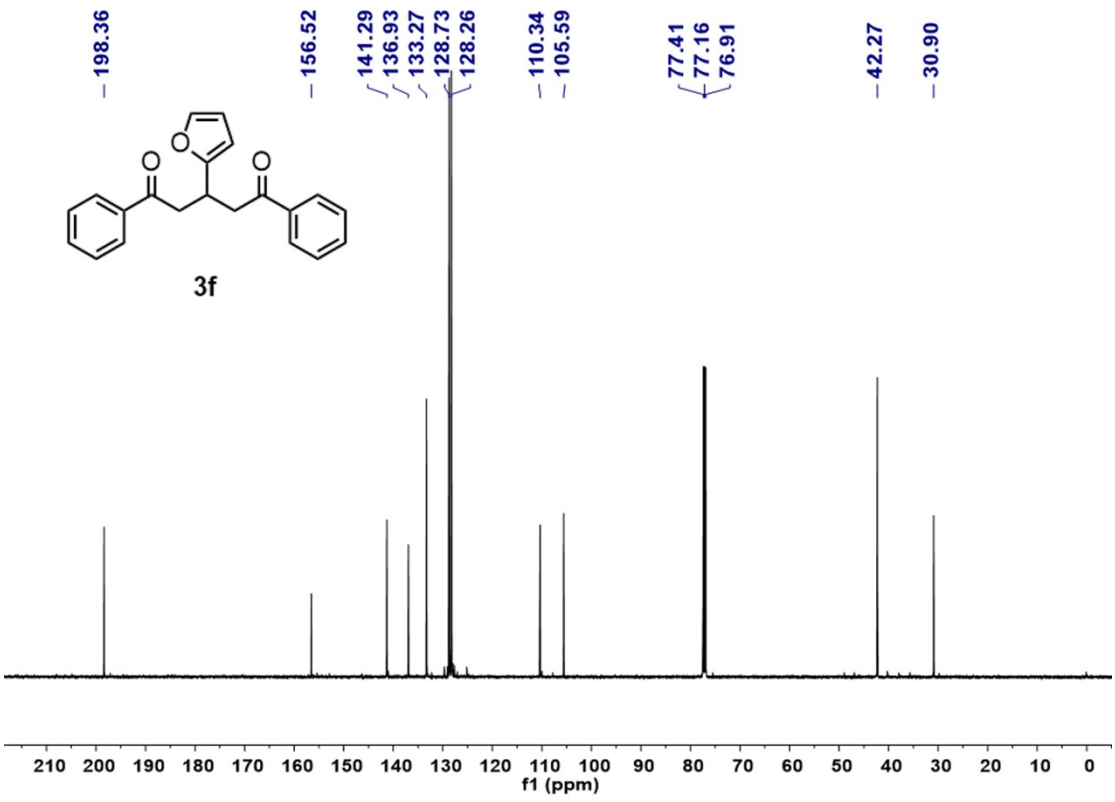
^{13}C NMR of **3e**



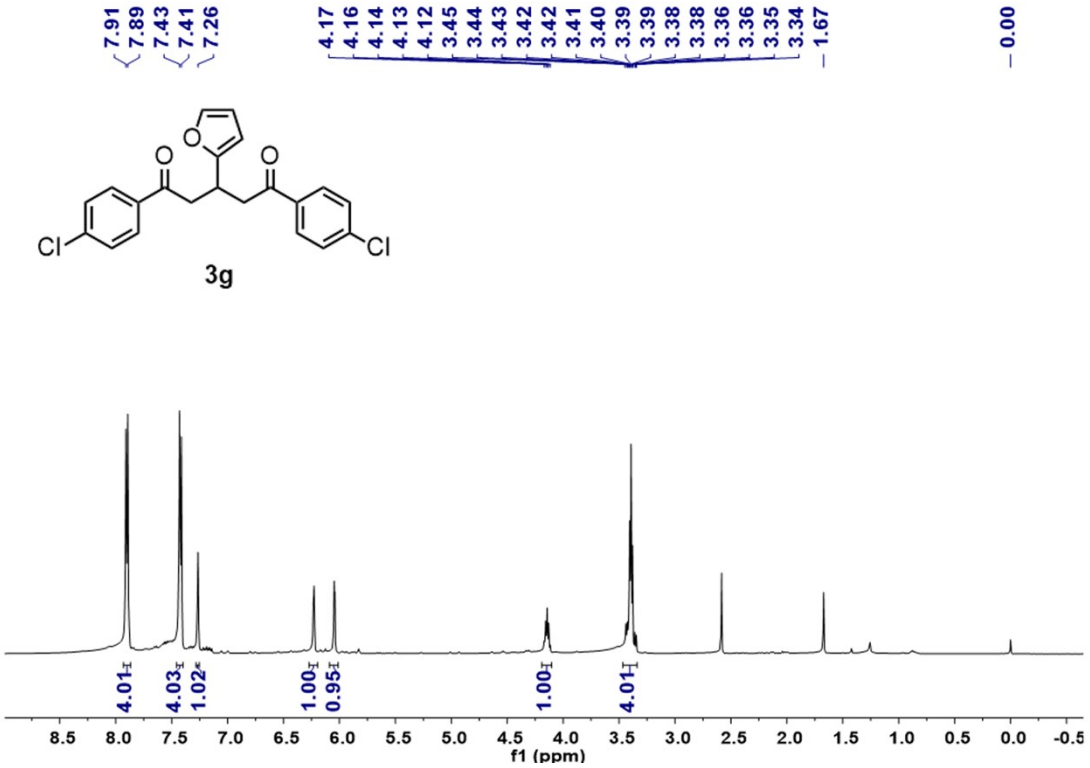
^{1}H NMR of **3f**



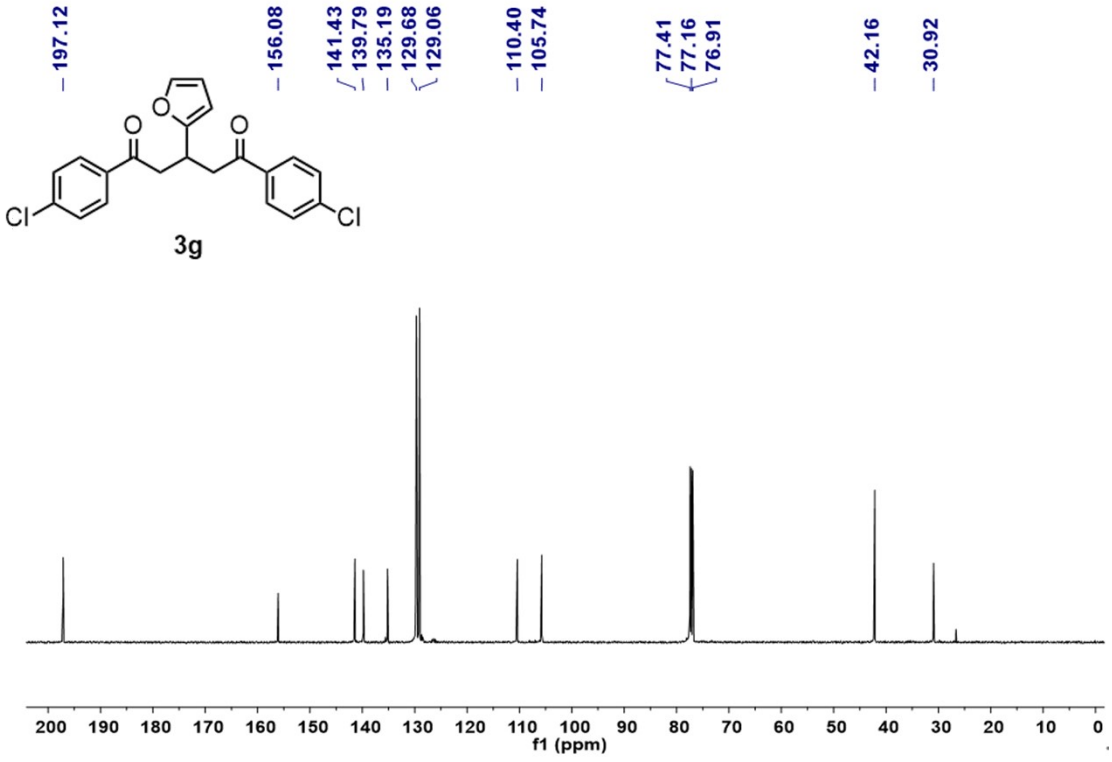
^{13}C NMR of **3f**



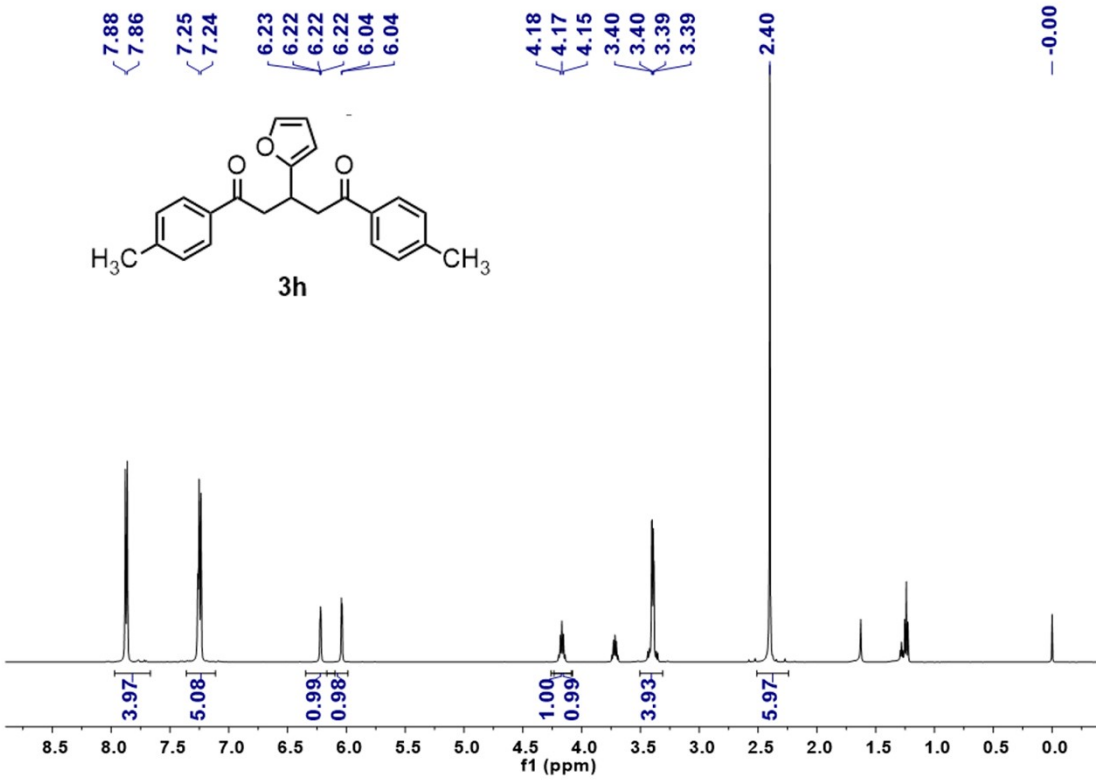
^{1}H NMR of **3g**



^{13}C NMR of **3g**



^{1}H NMR of **3h**



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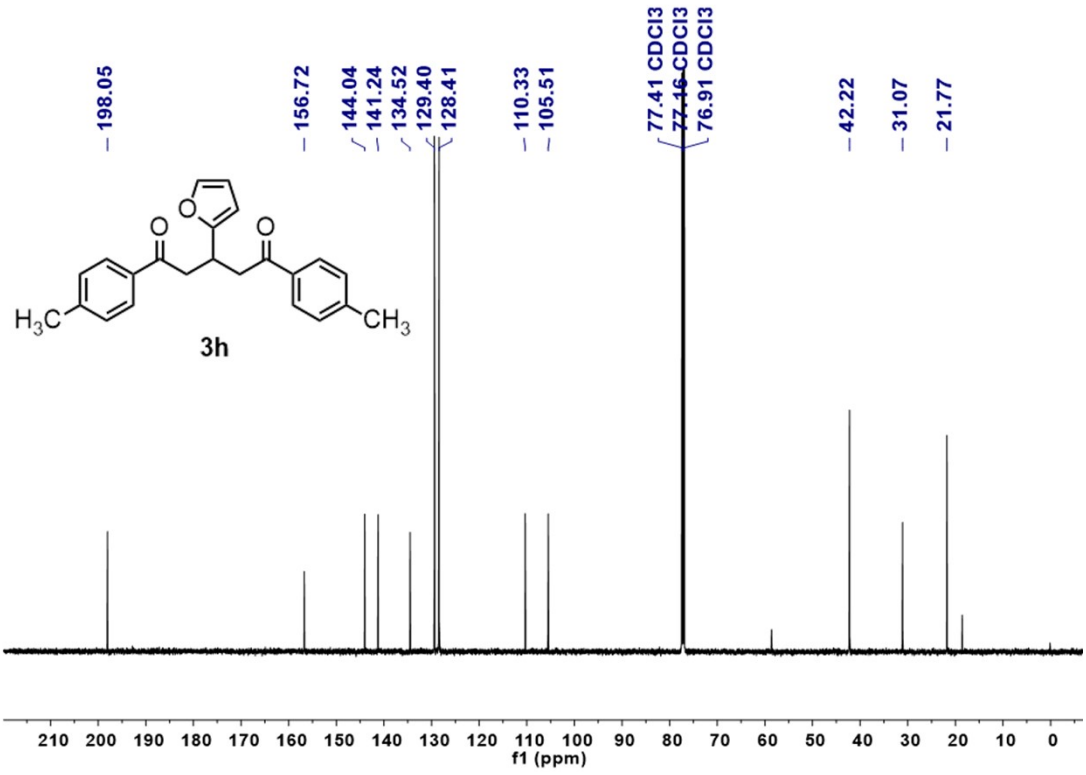
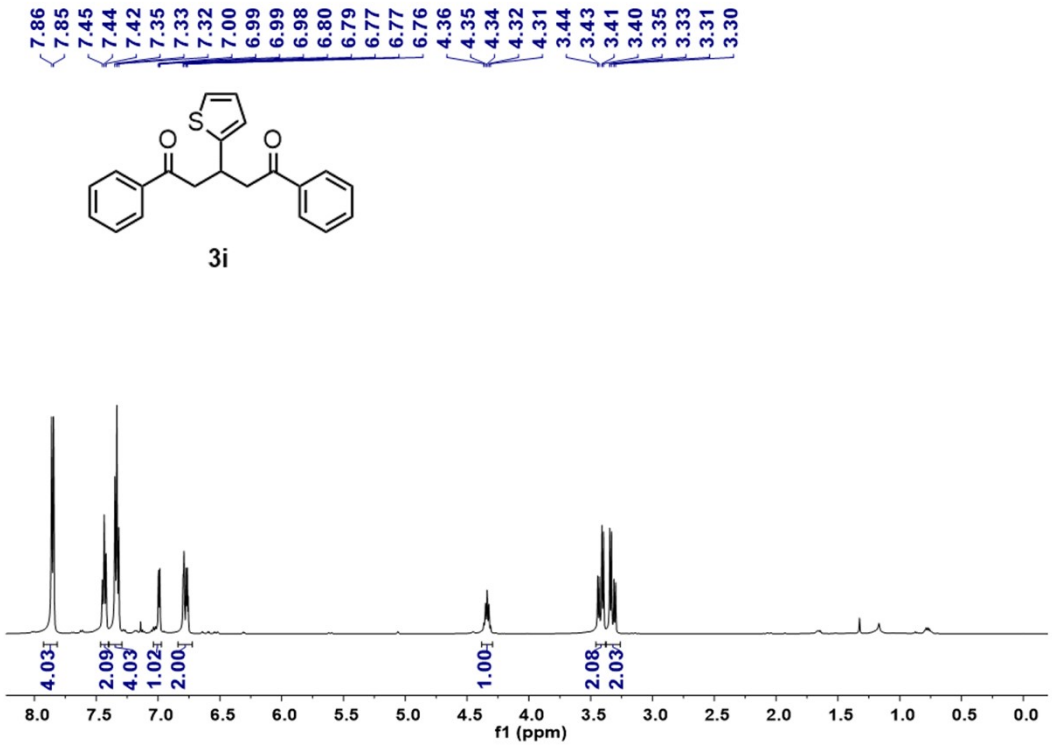
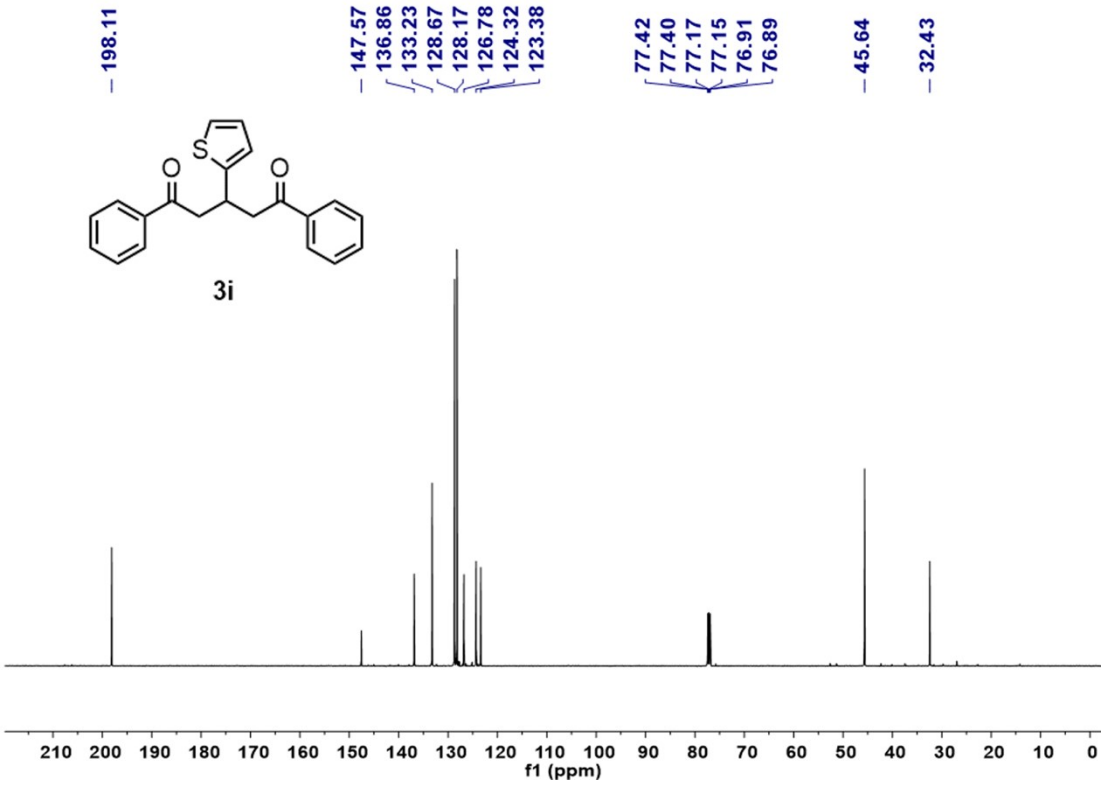


^{1}H NMR of **3i**



^{13}C NMR of **3i**



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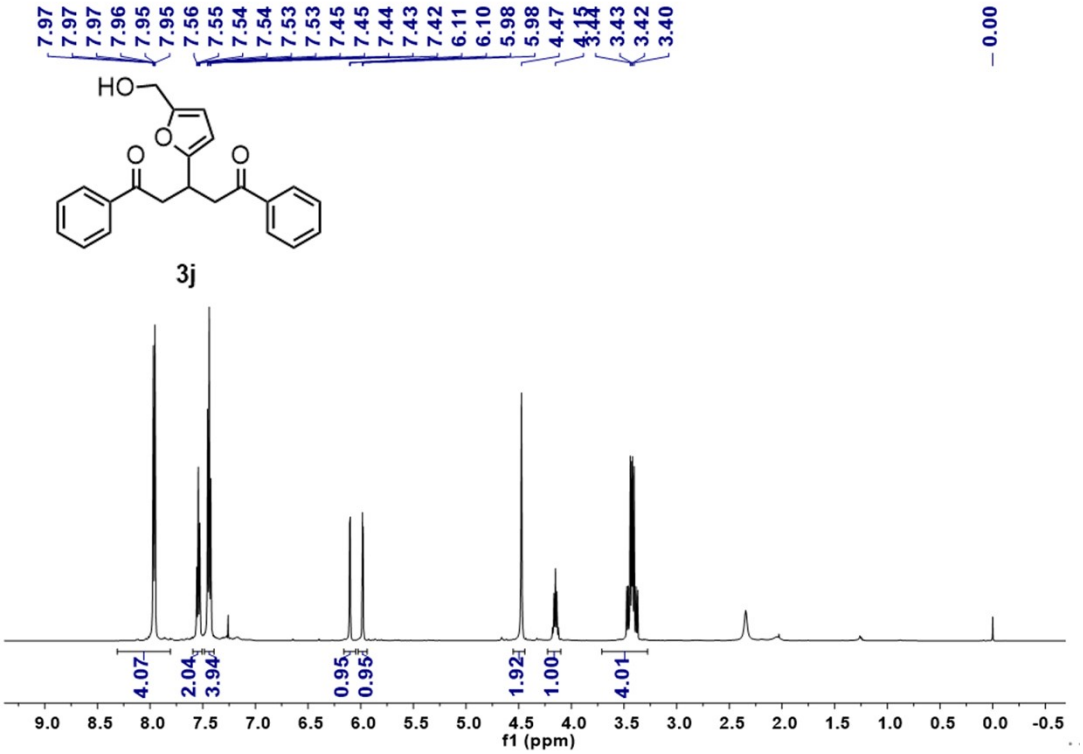
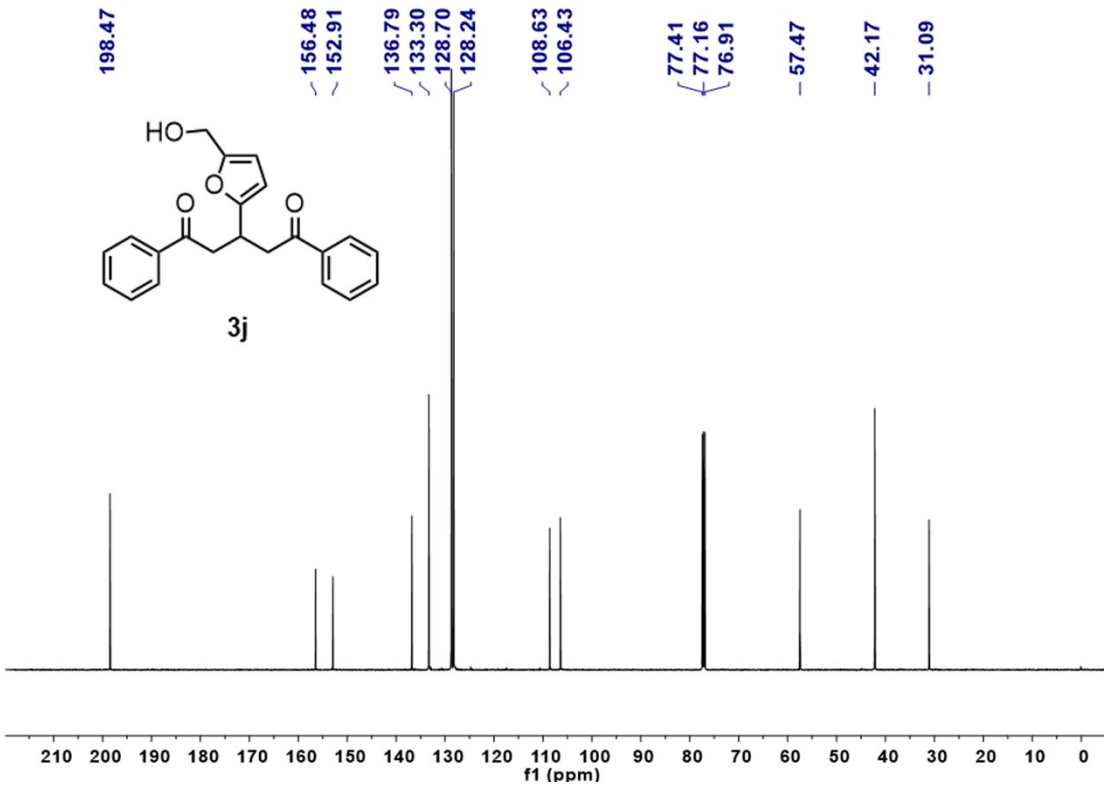
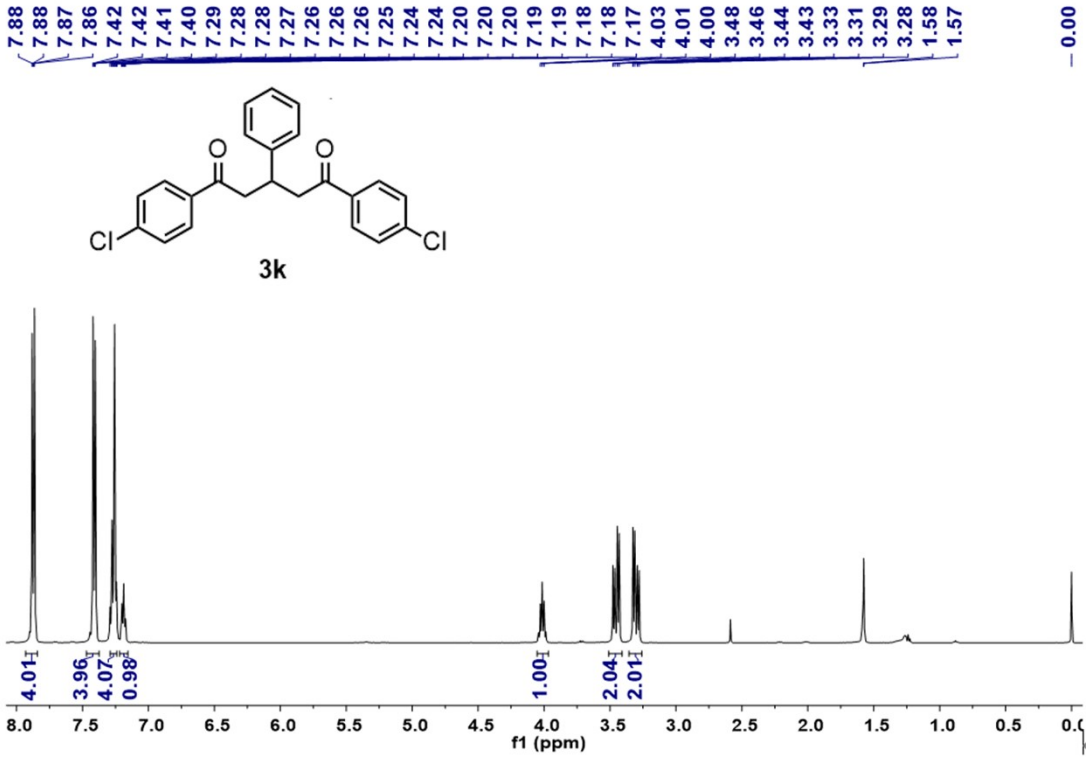


^{13}C NMR of **3j**



^{1}H NMR of **3k**



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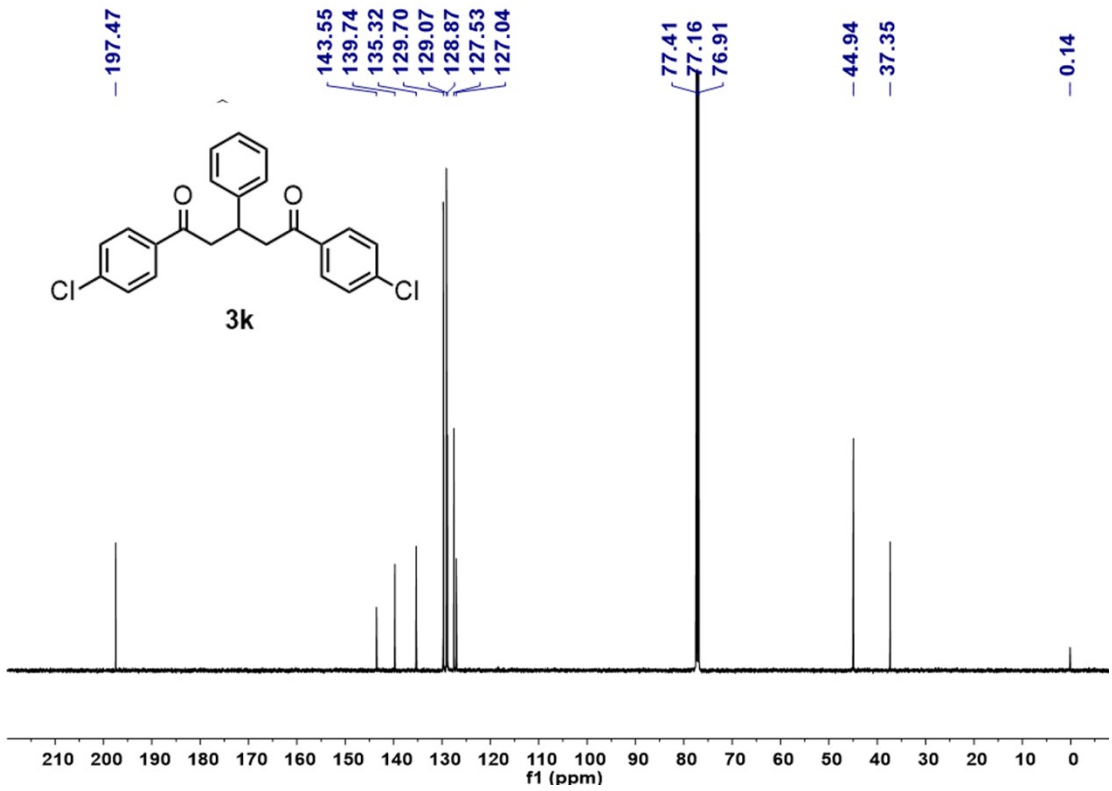
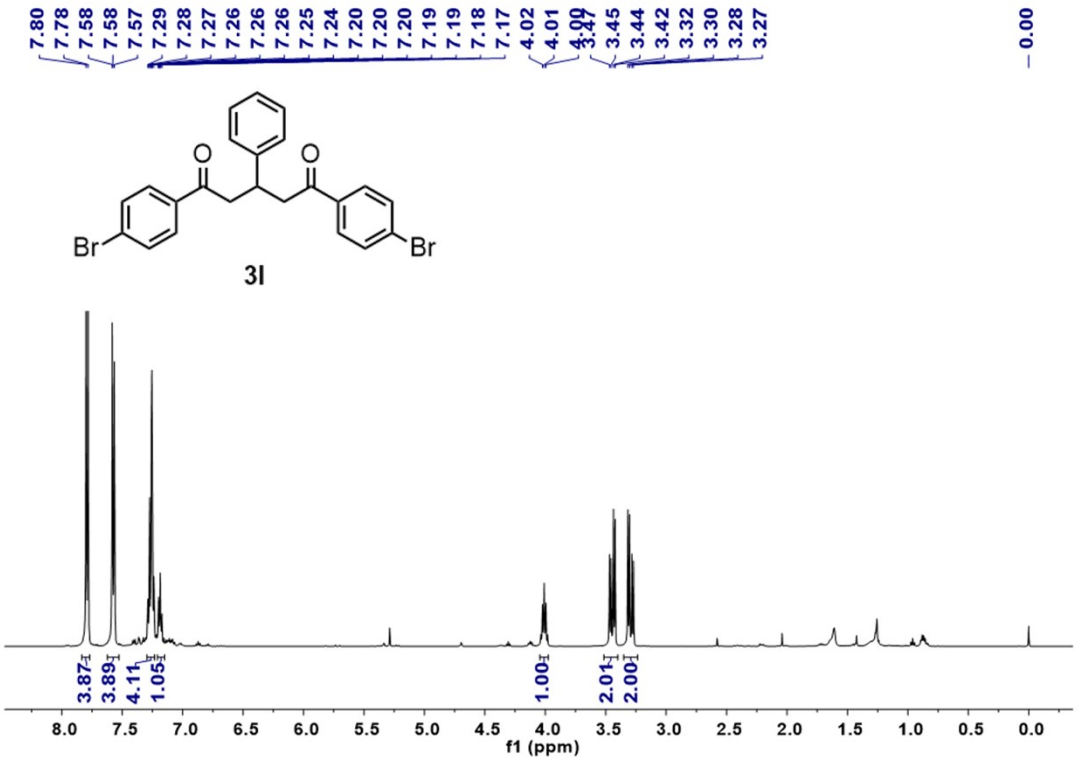
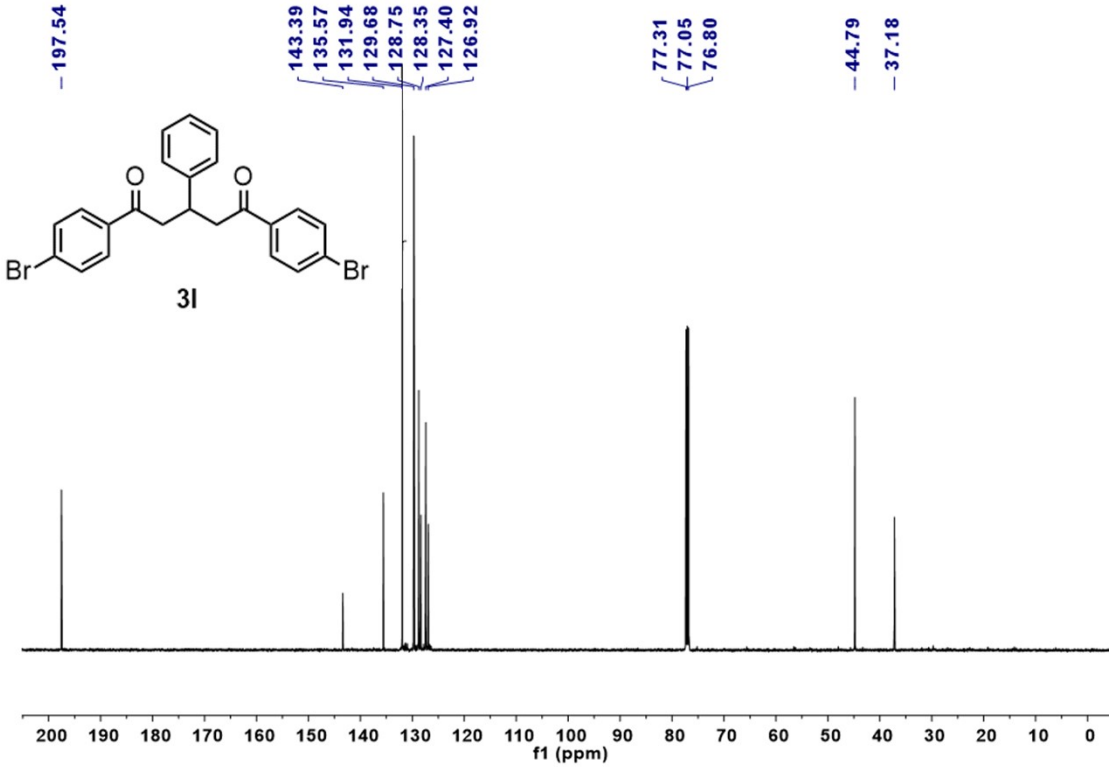


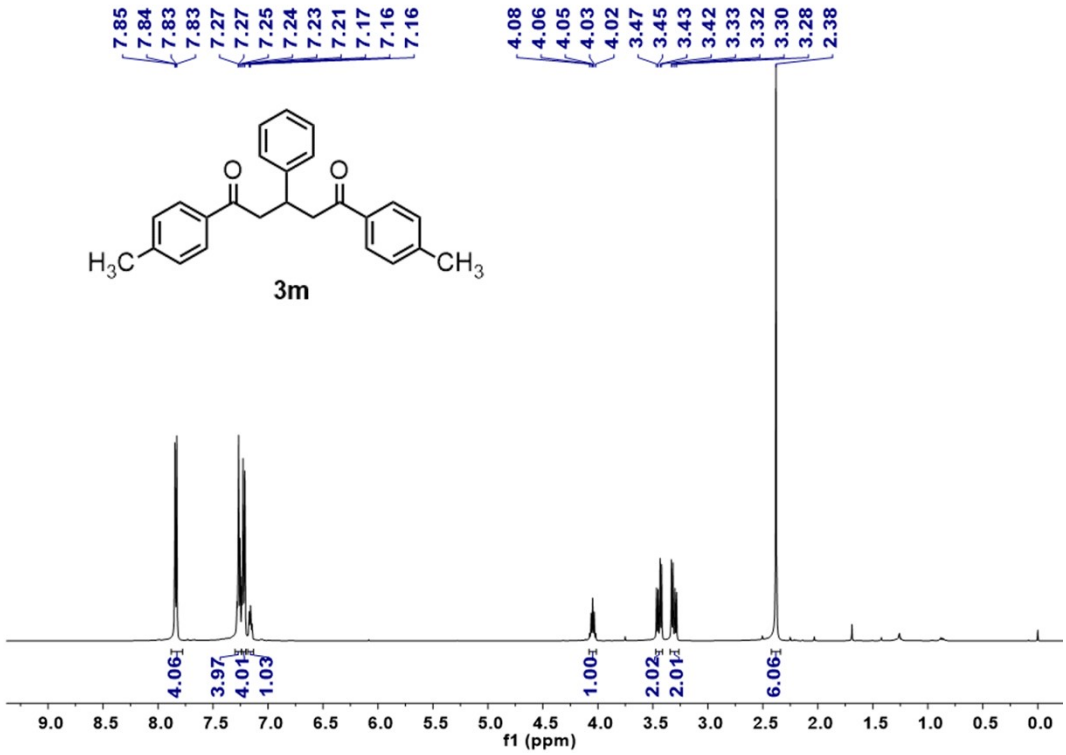
^{1}H NMR of **3l**



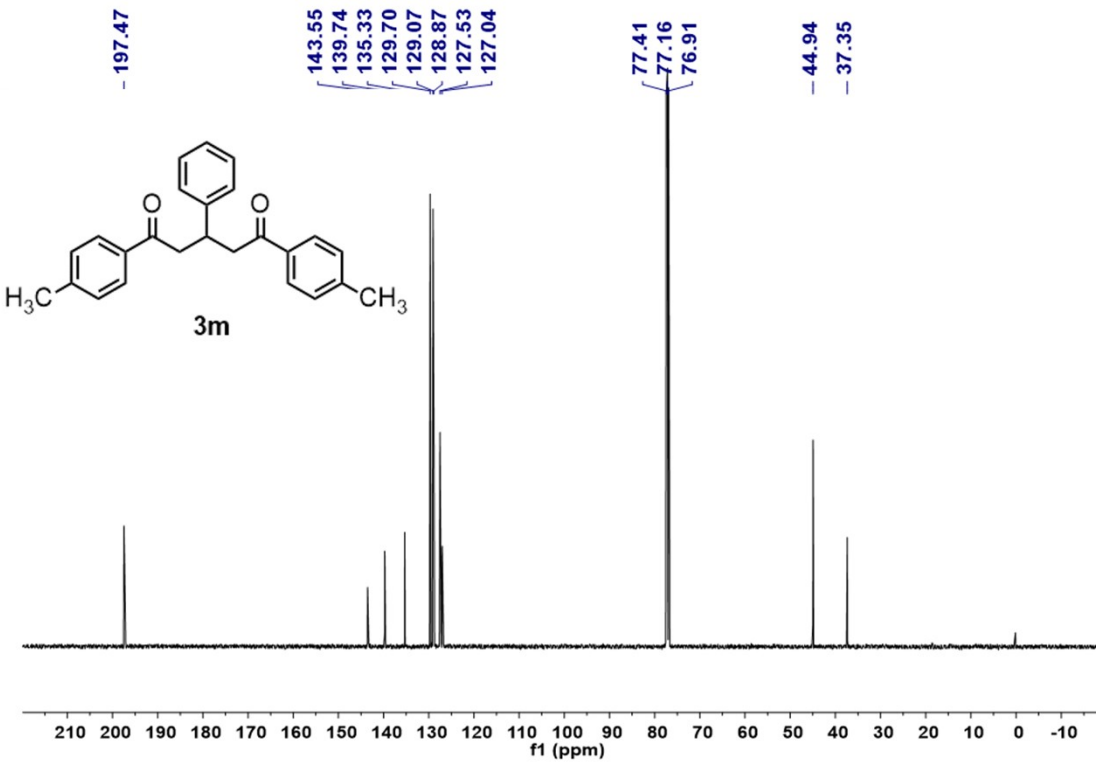
^{13}C NMR of **3l**



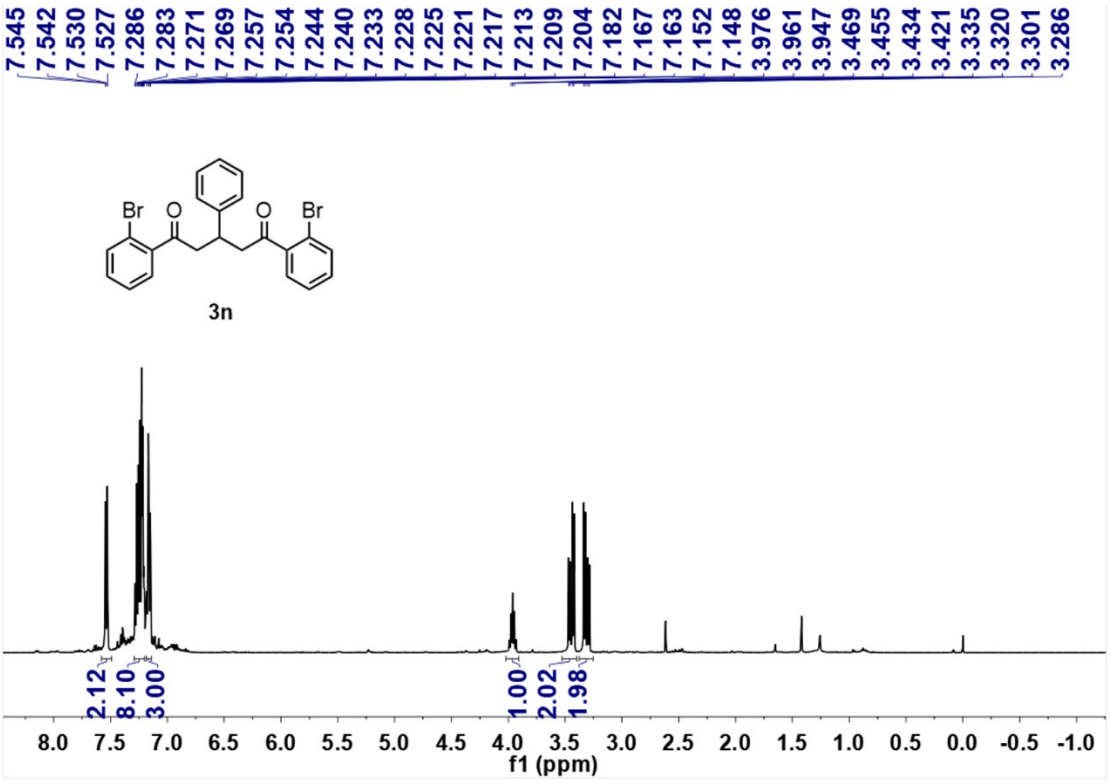
^{1}H NMR of **3m**



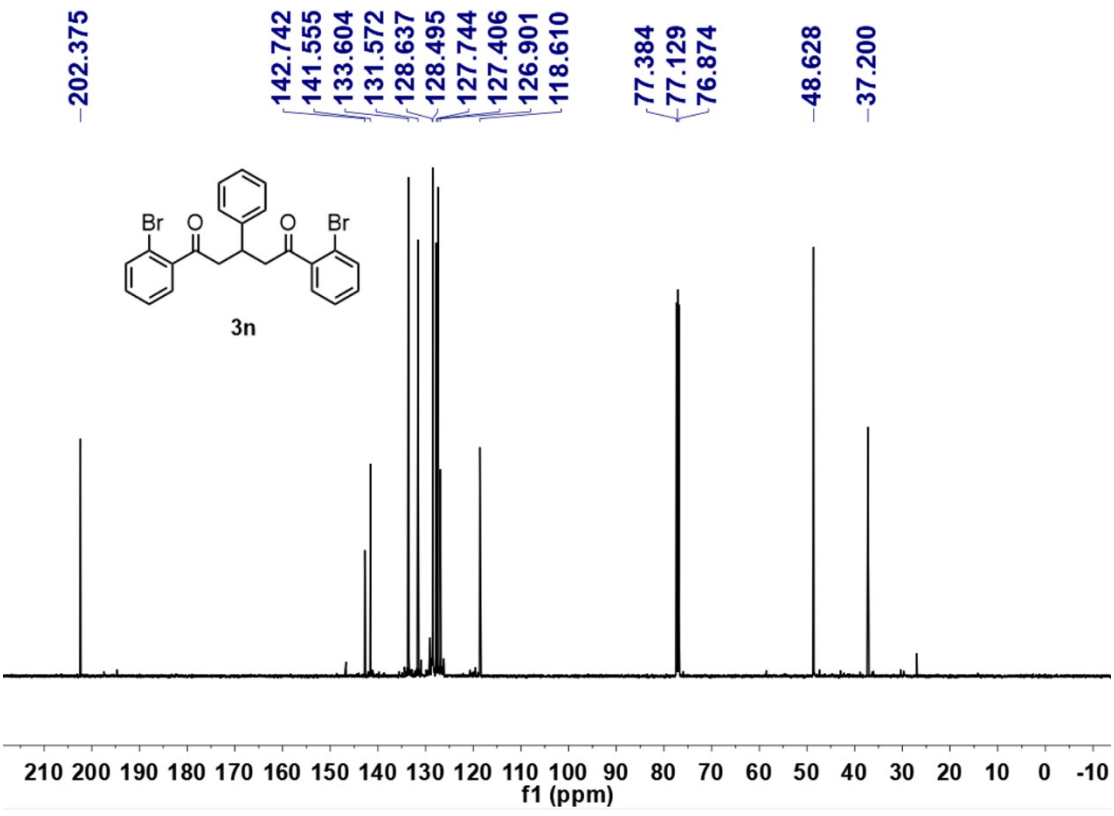
^{13}C NMR of **3m**



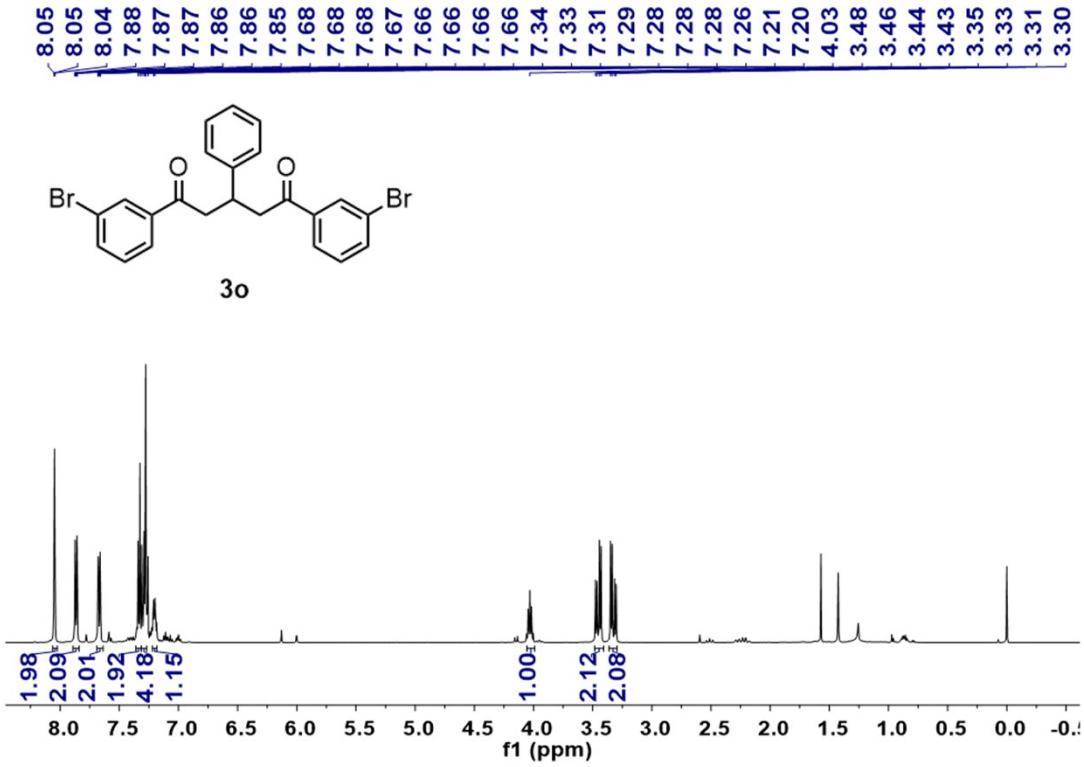
^{1}H NMR of **3n**



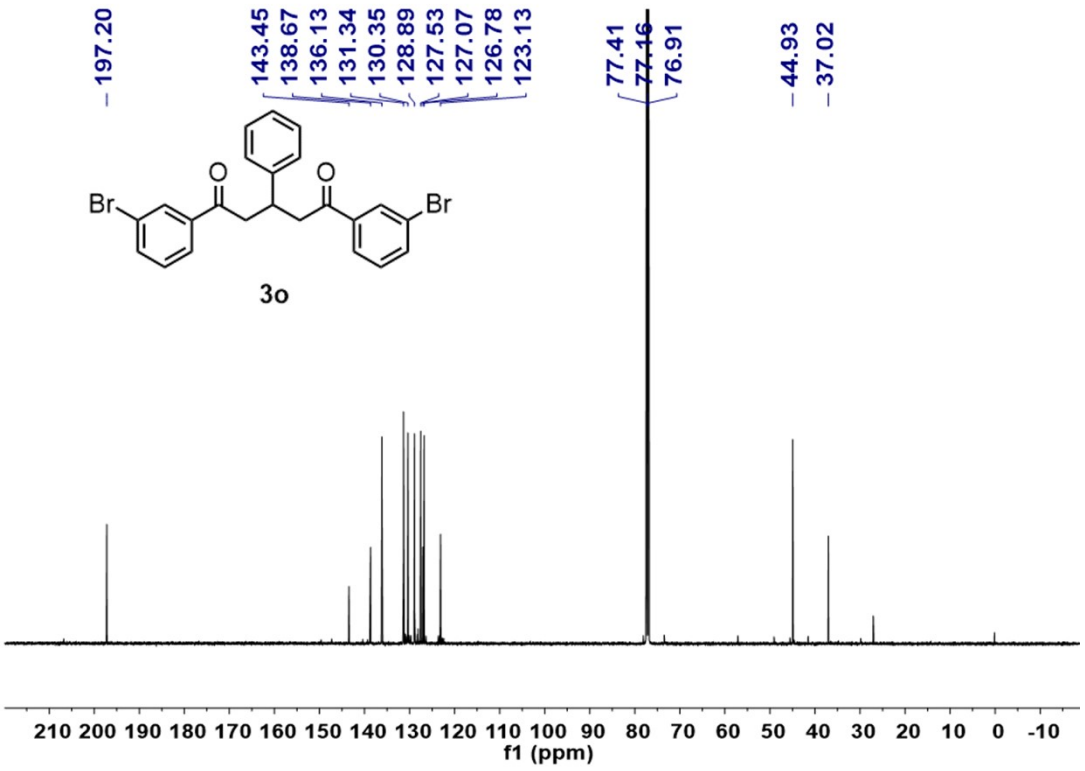
^{13}C NMR of **3n**



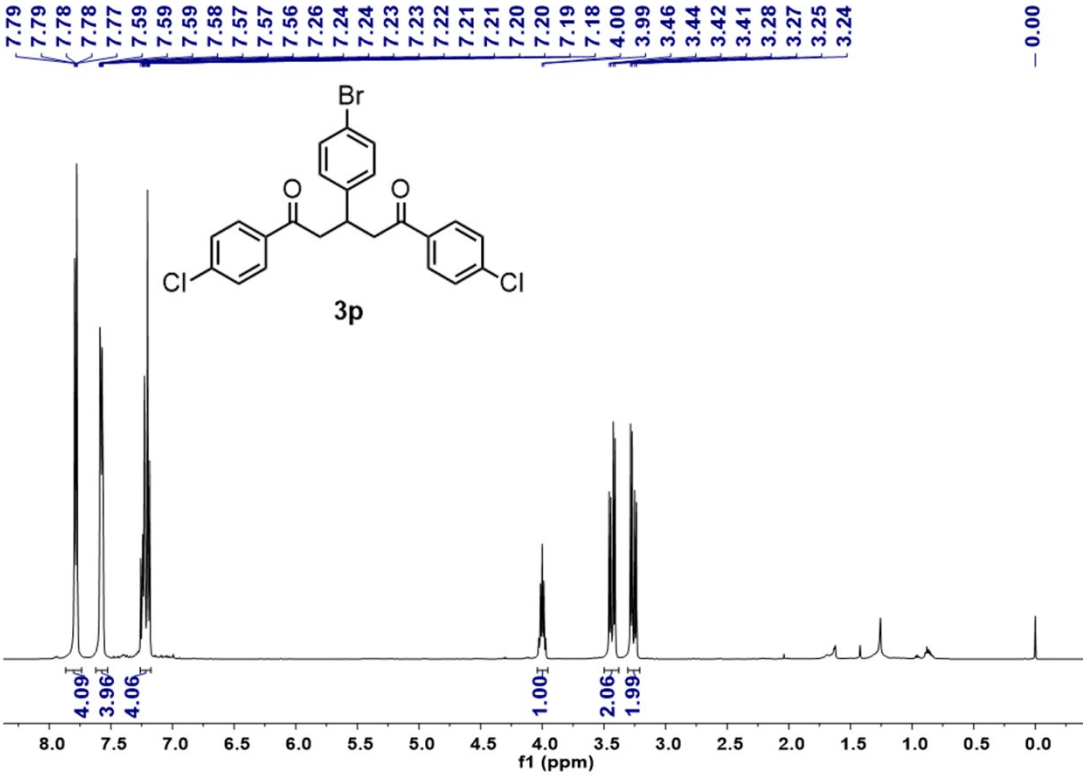
^{1}H NMR of **3o**



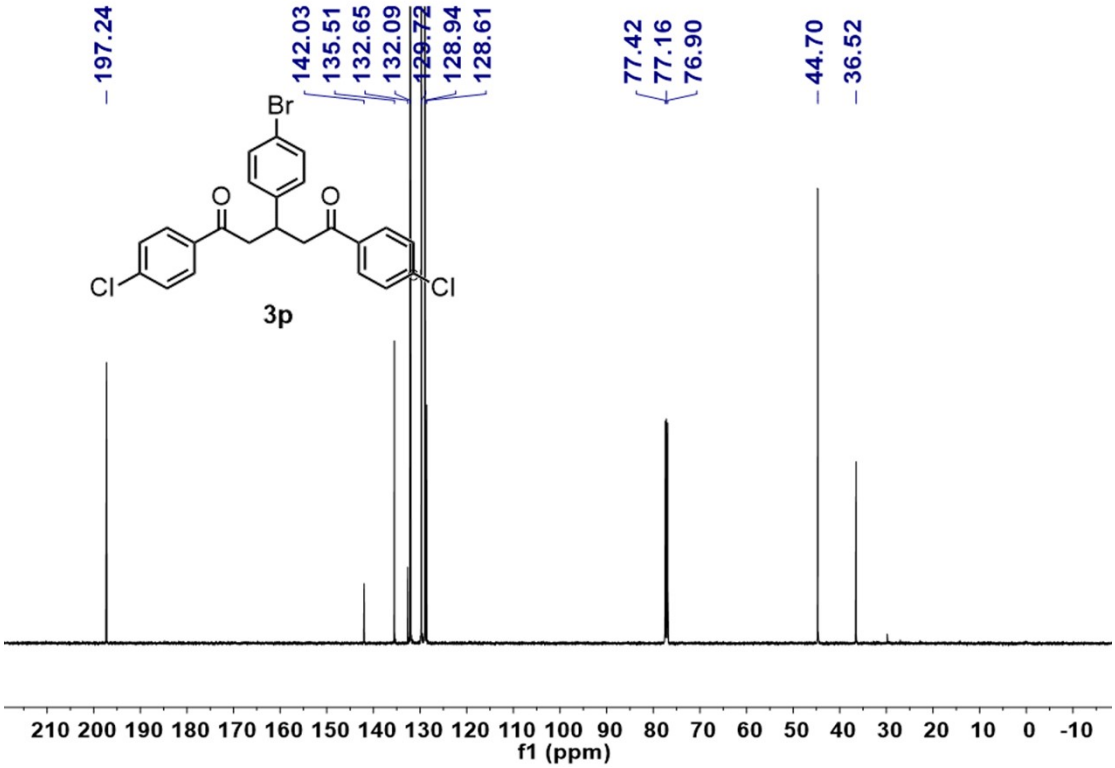
^{13}C NMR of **3o**



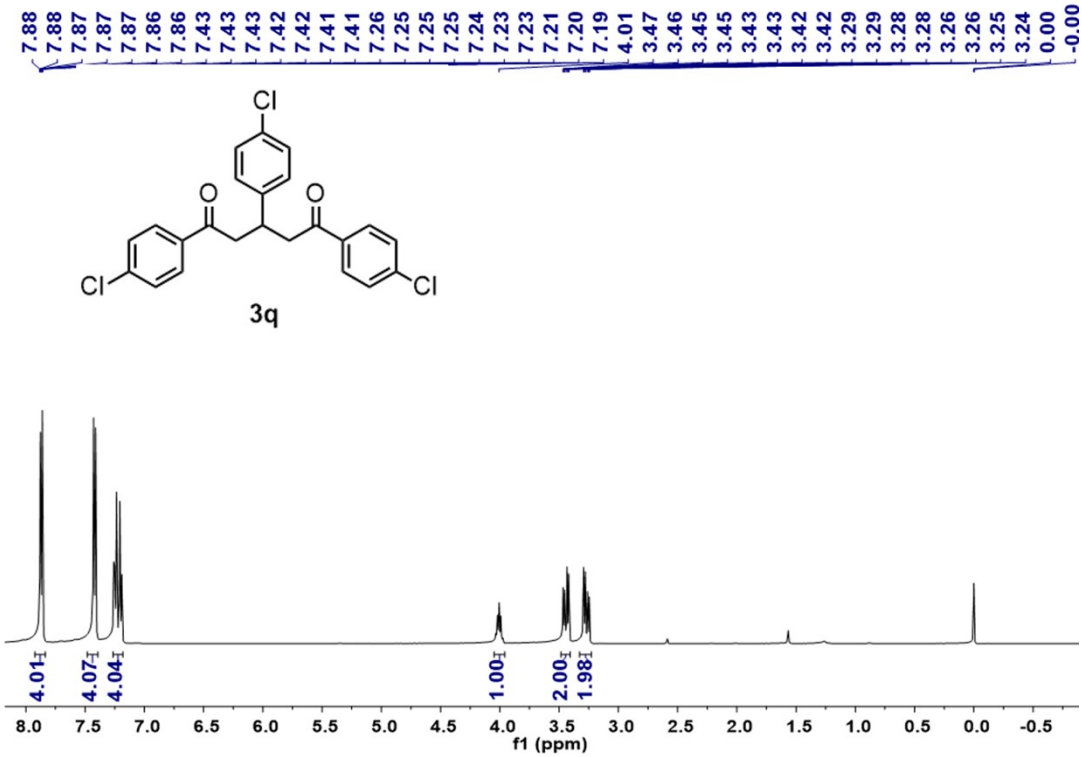
^{1}H NMR of **3p**



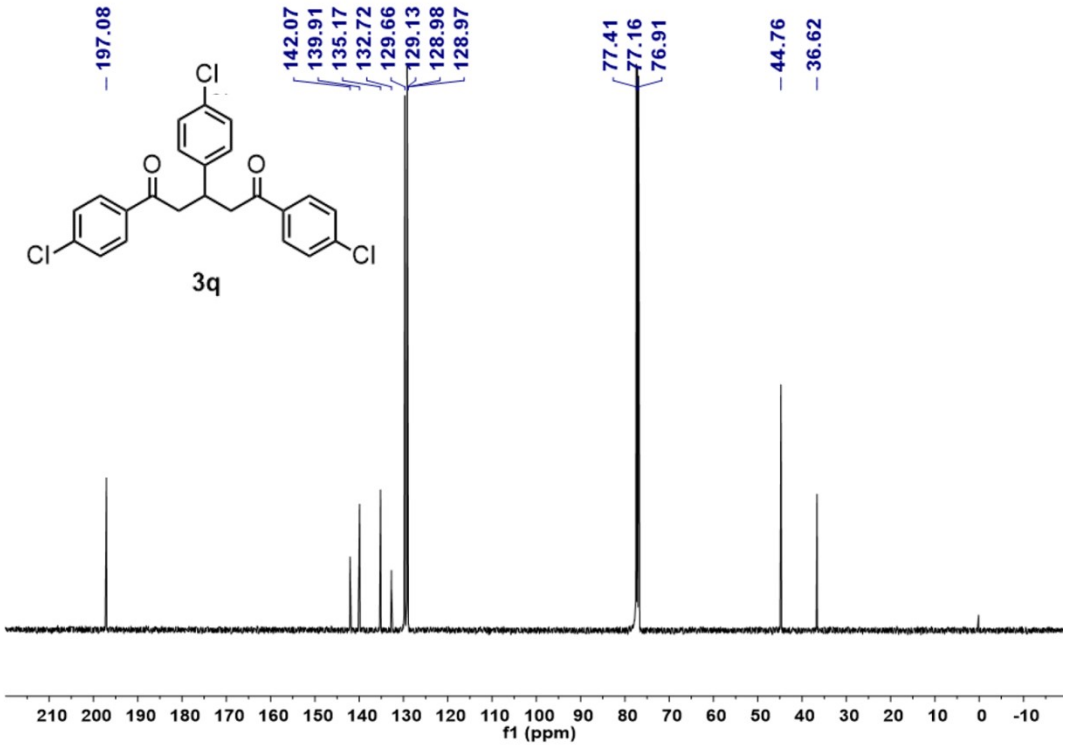
^{13}C NMR of **3p**



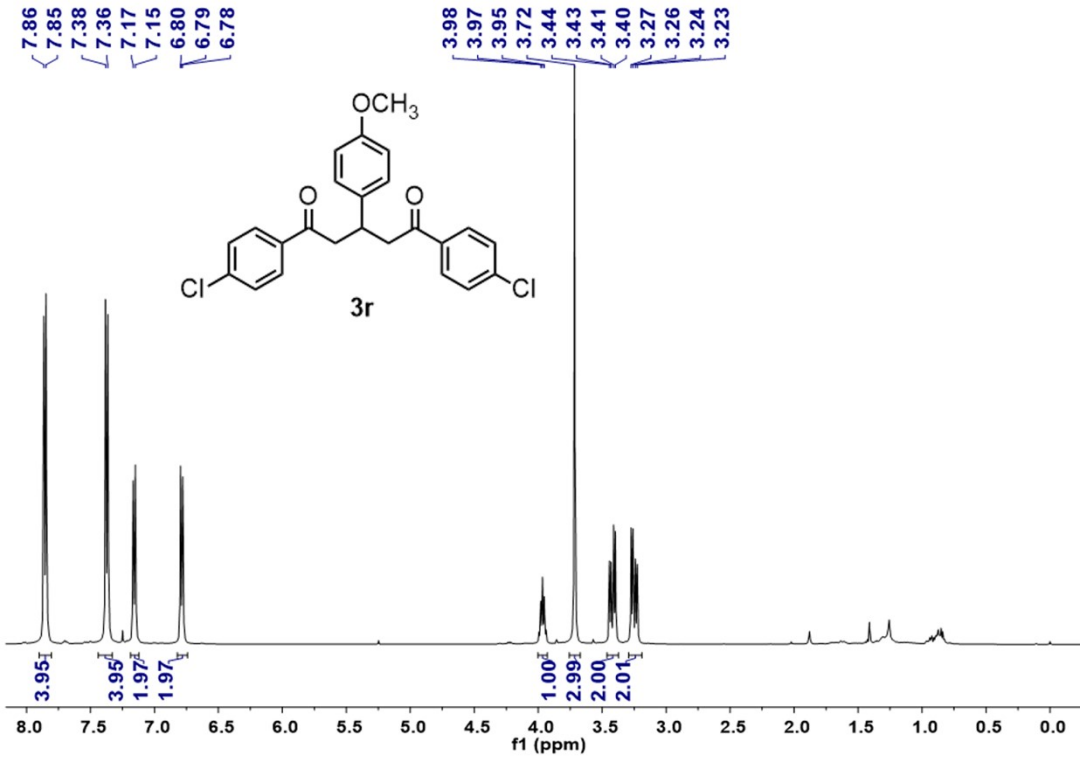
^{1}H NMR of **3q**



^{13}C NMR of **3q**



^{1}H NMR of **3r**



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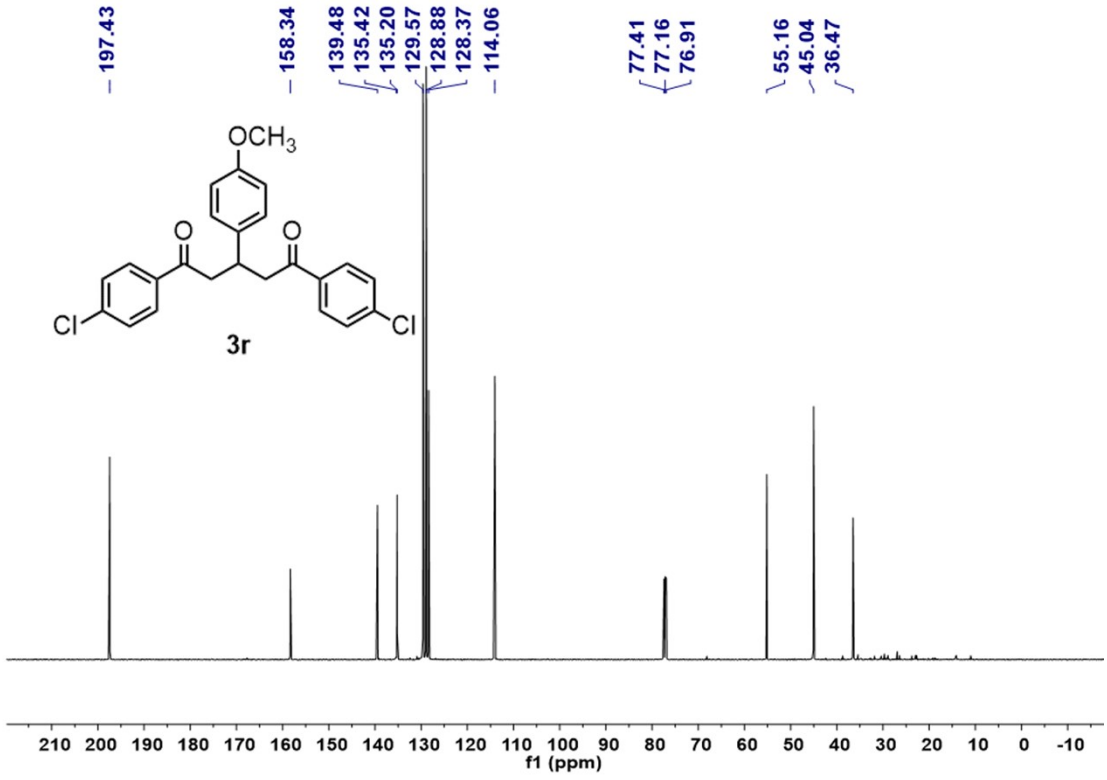

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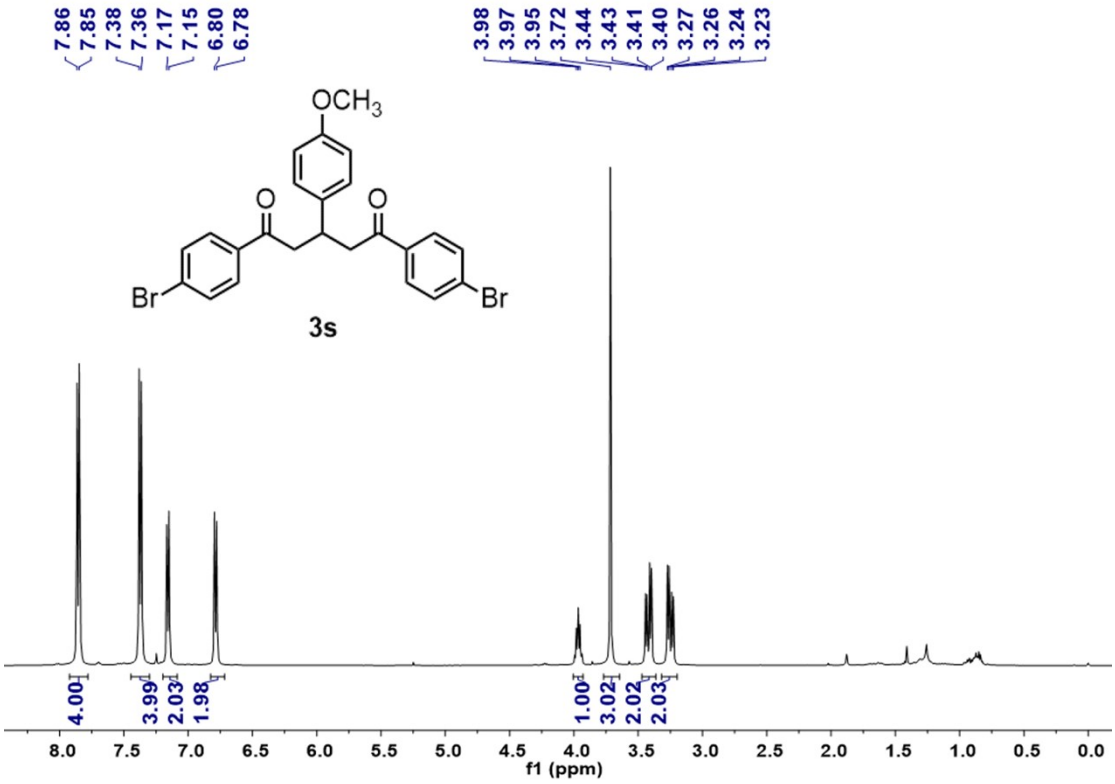
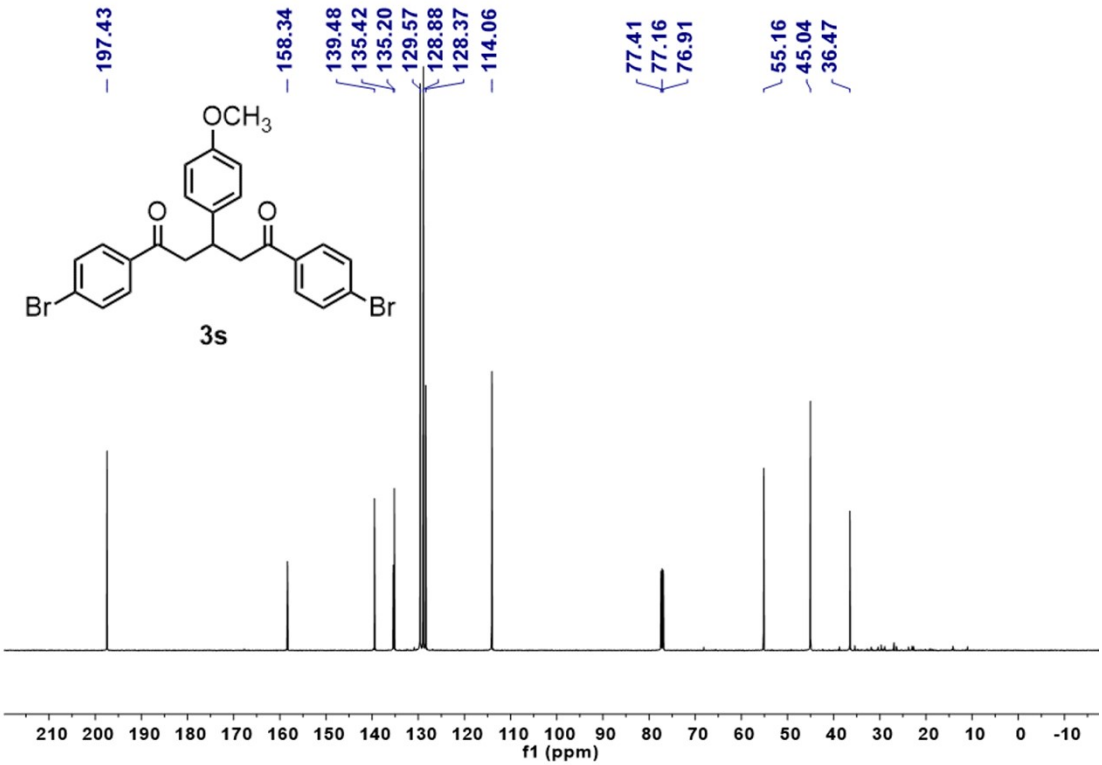
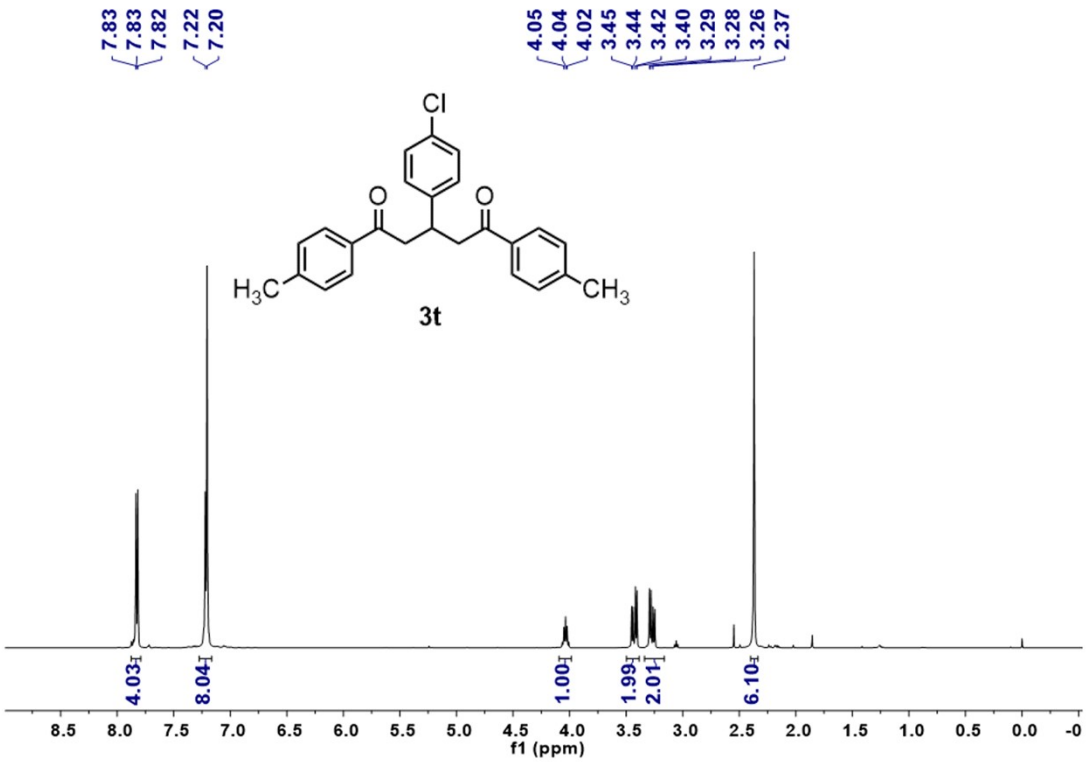


^{13}C NMR of **3s**



^{1}H NMR of **3t**



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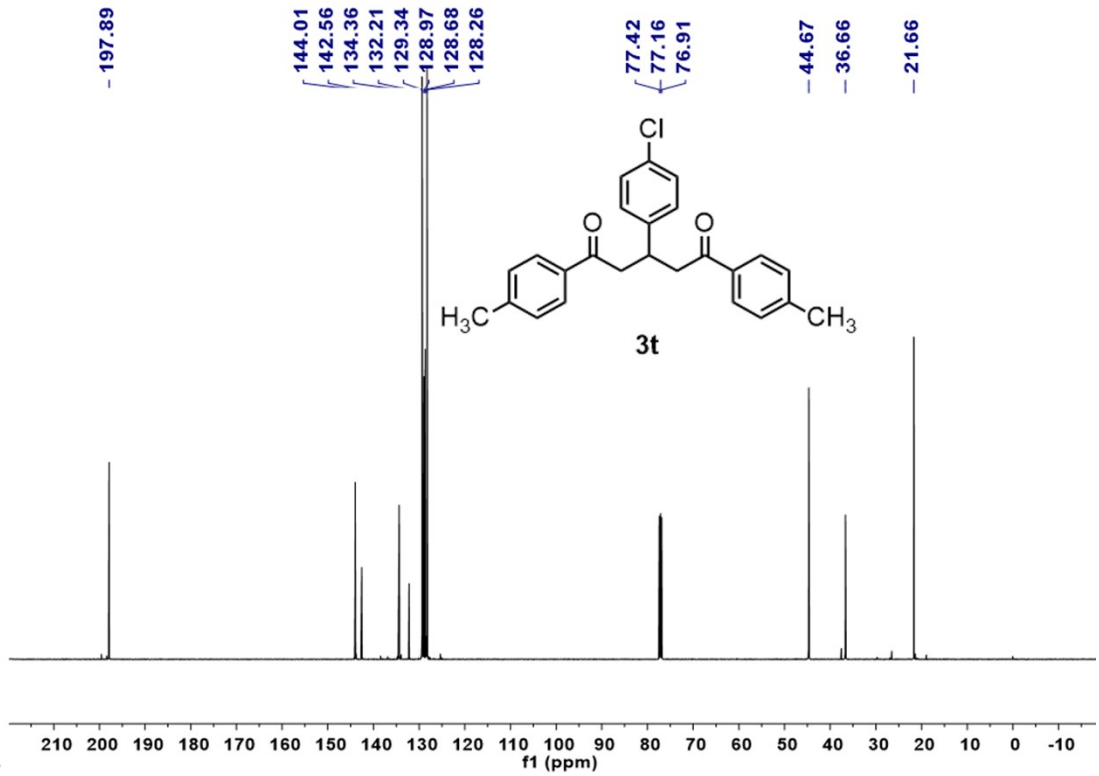
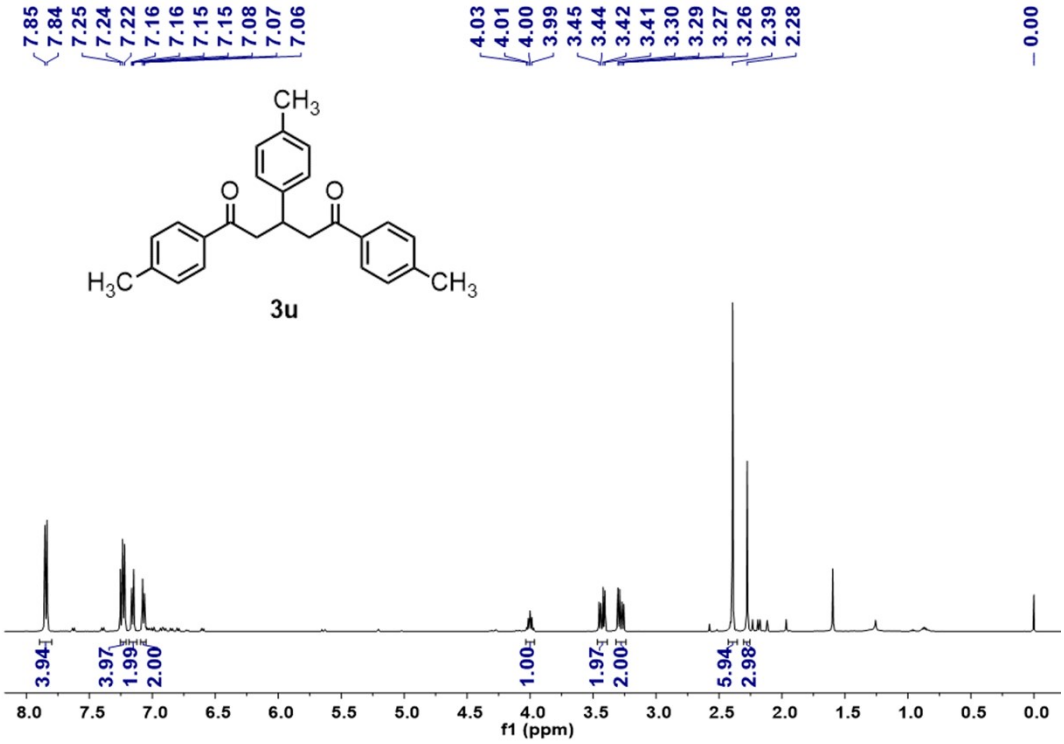
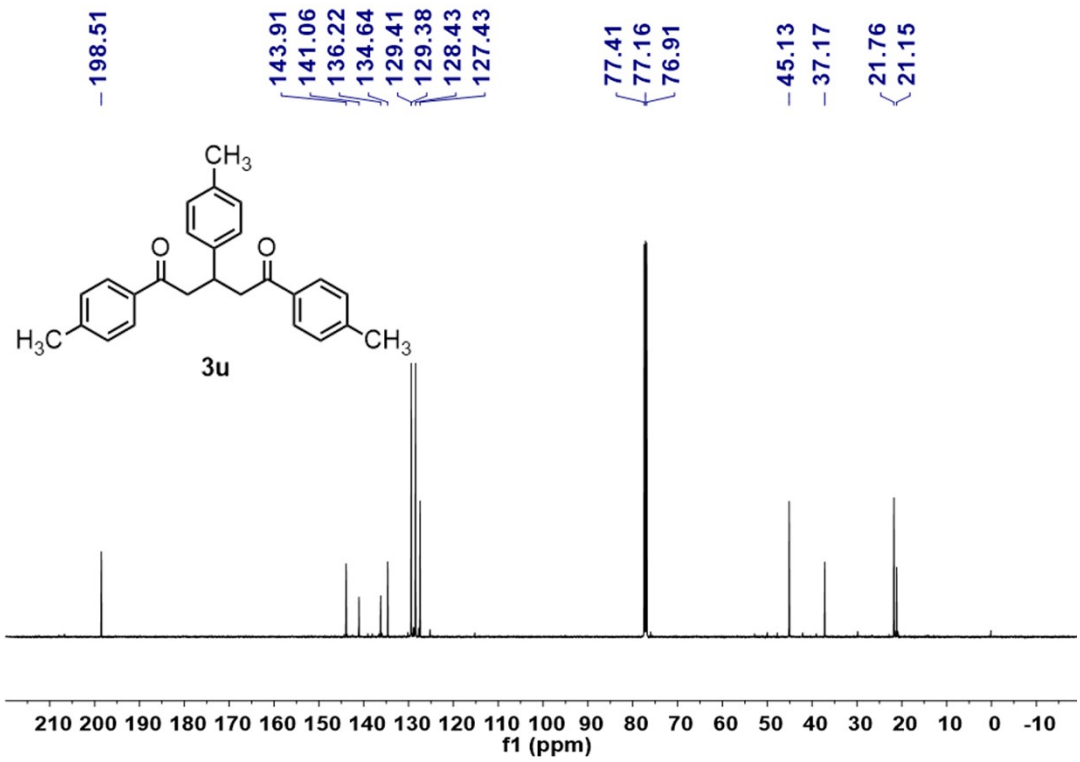


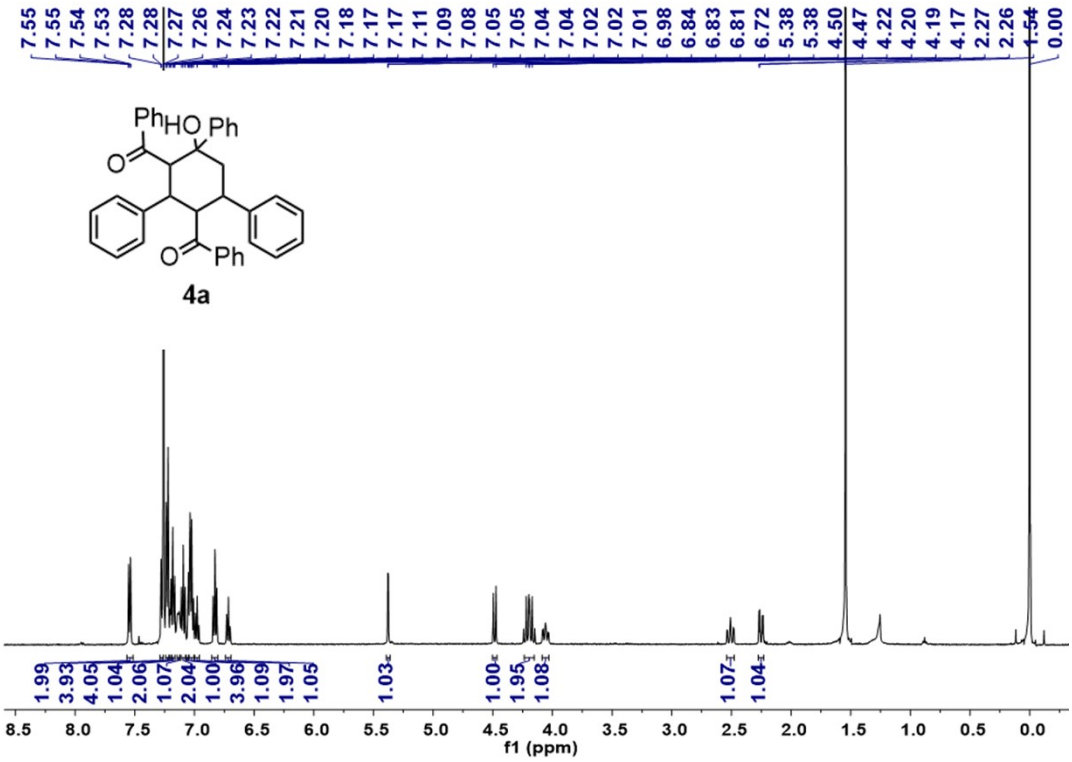
^{1}H NMR of **3u**



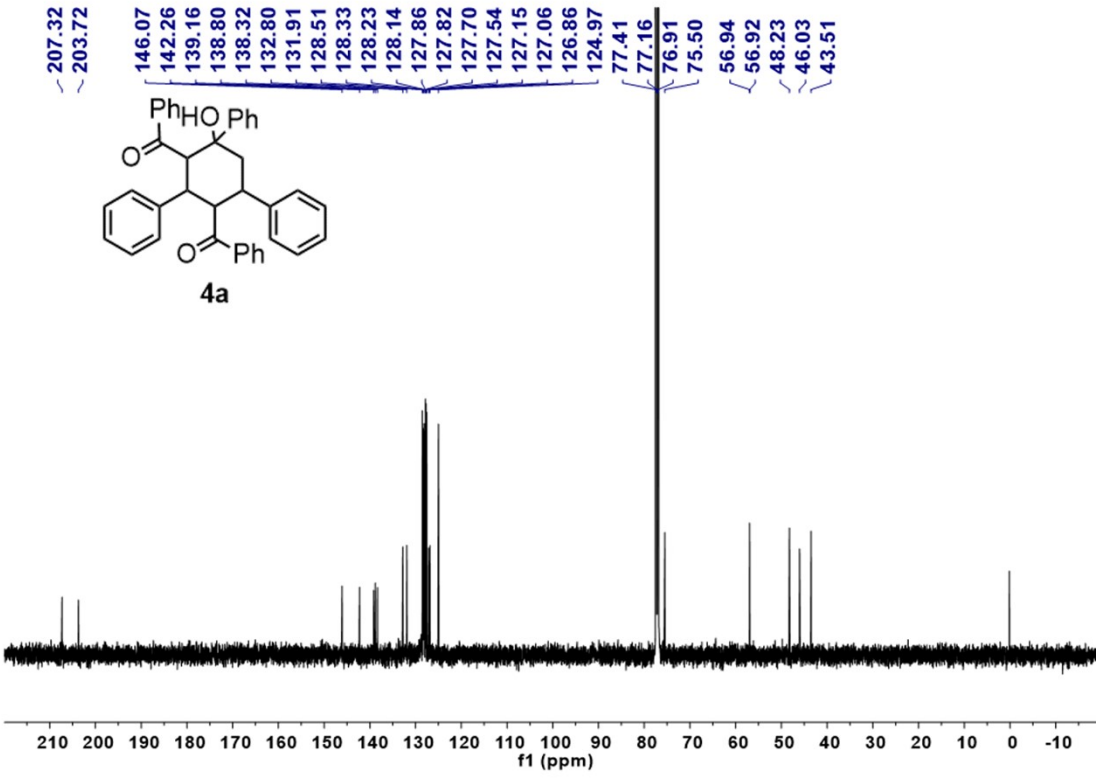
^{13}C NMR of **3u**



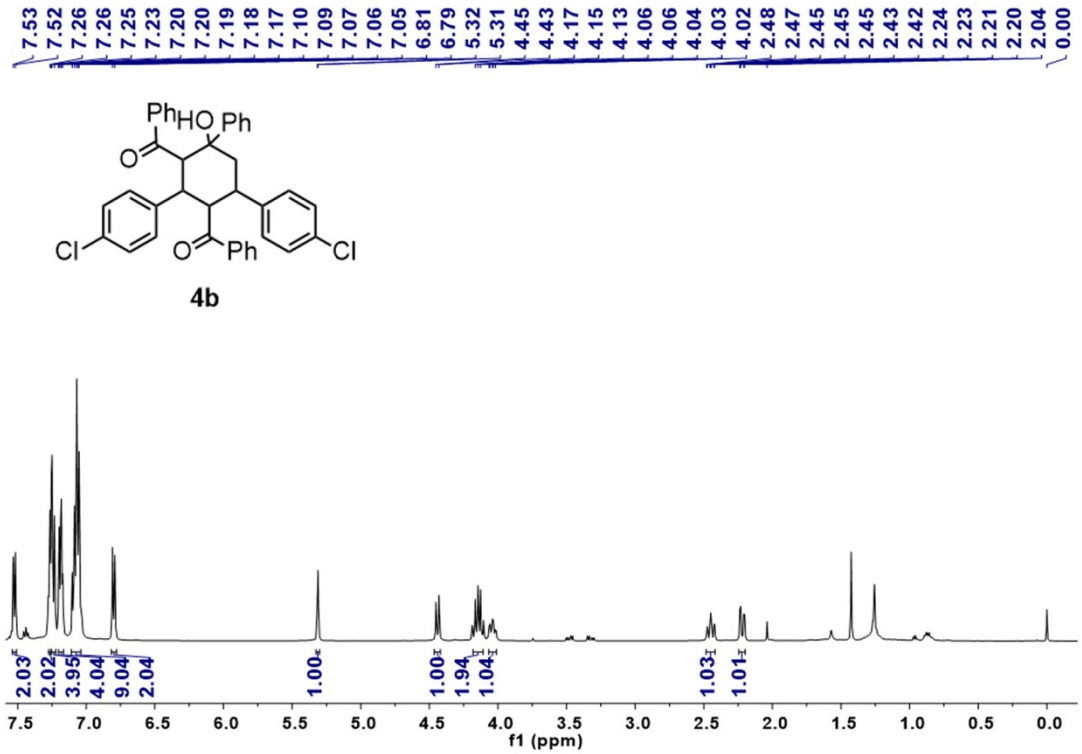
^{1}H NMR of **4a**



^{13}C NMR of **4a**



^{1}H NMR of **4b**



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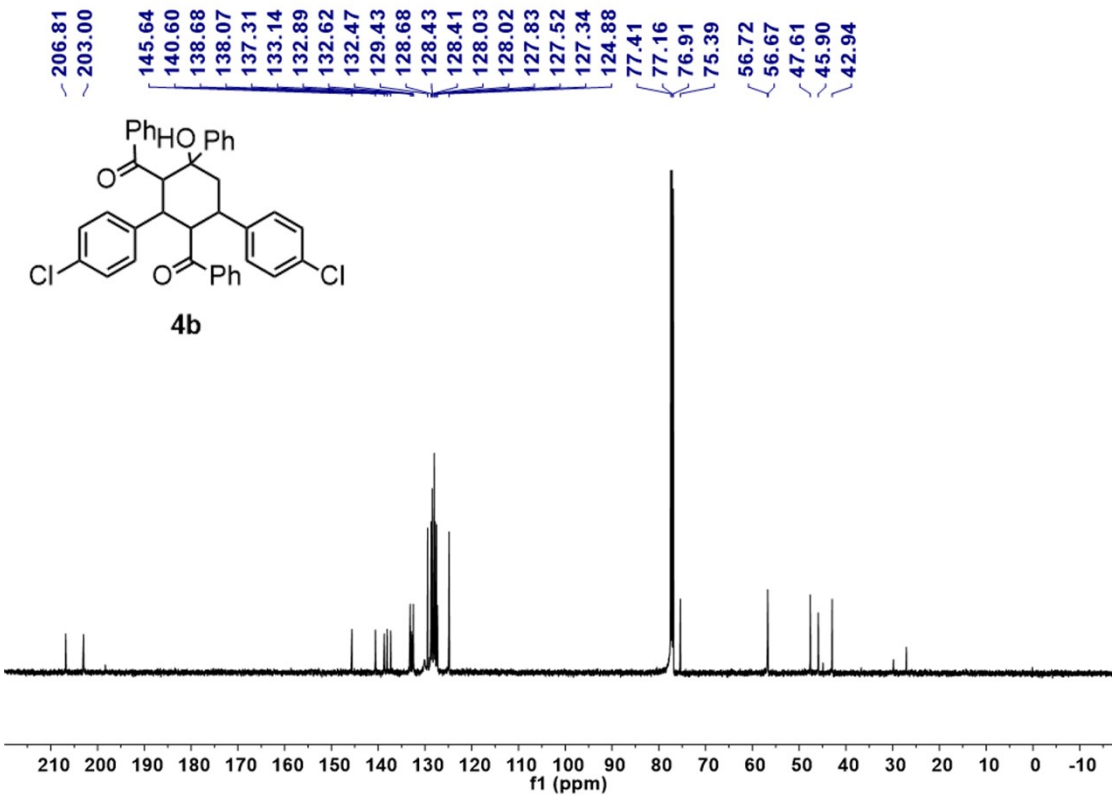
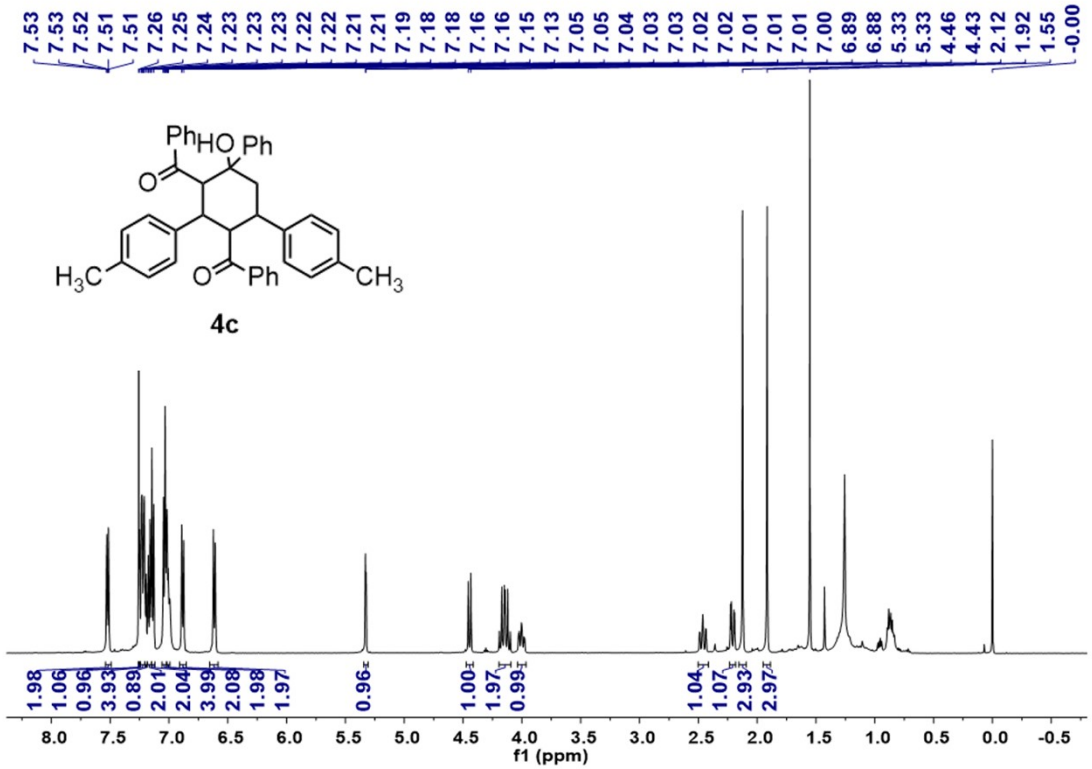
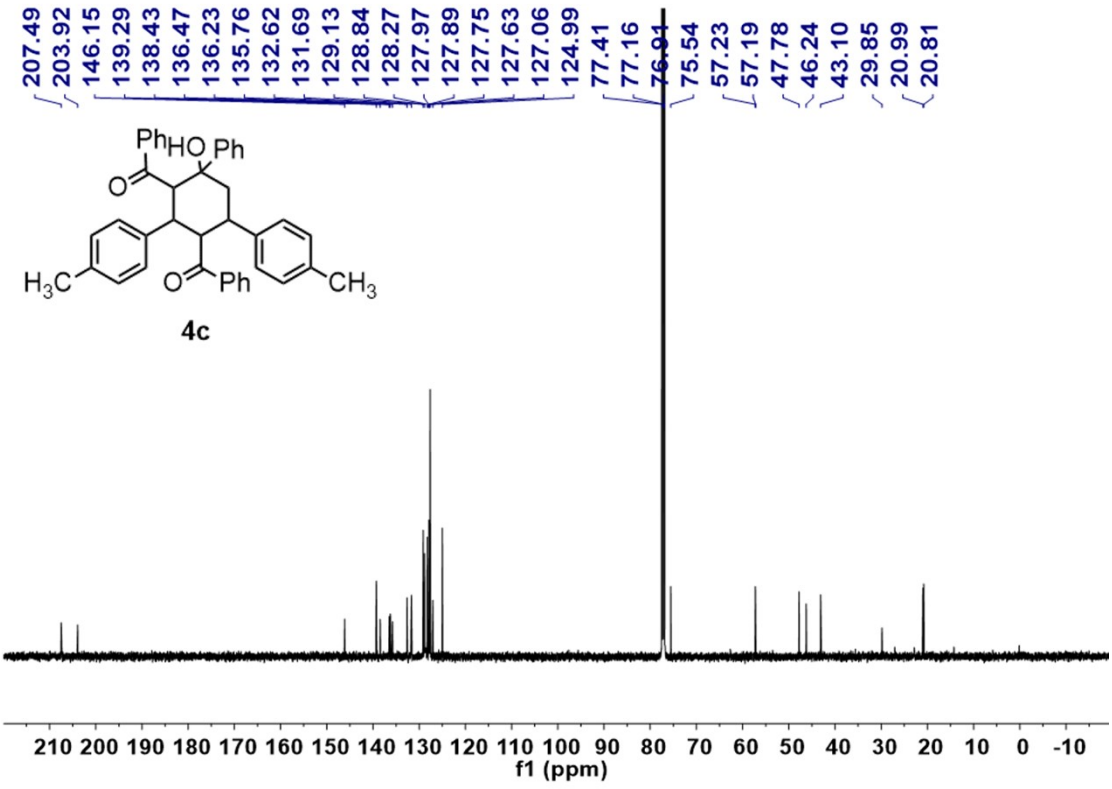


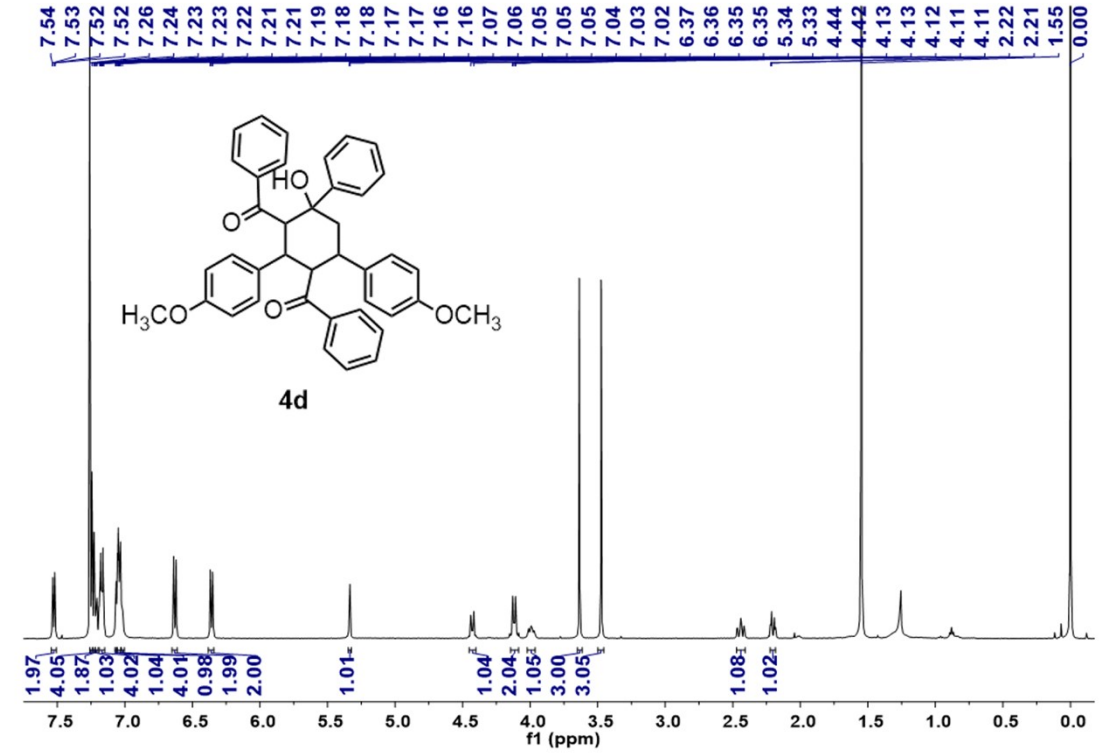
^{1}H NMR of **4c**



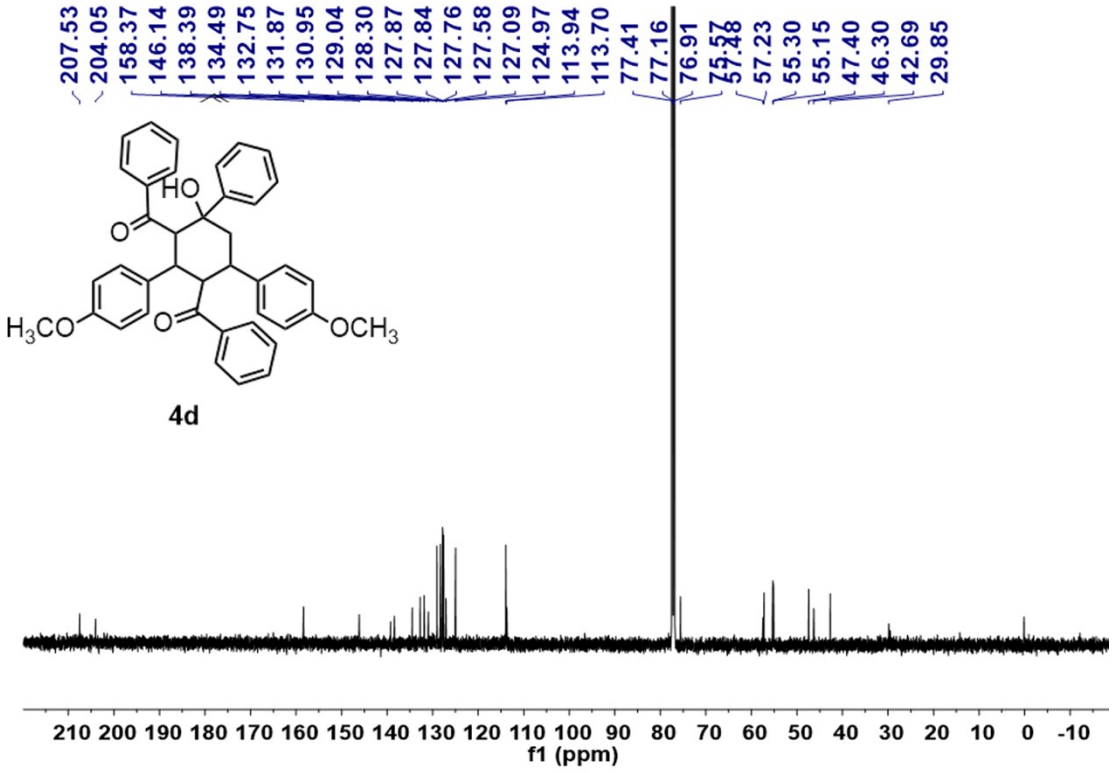
^{13}C NMR of **4c**



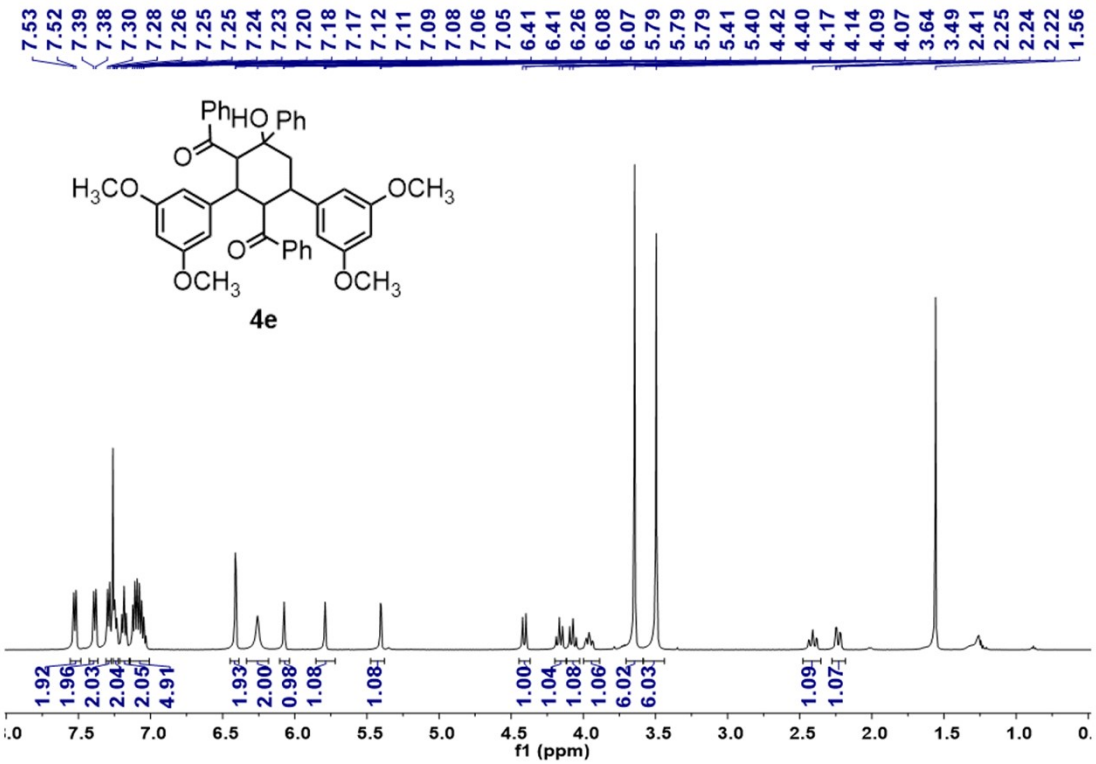
^{1}H NMR of **4d**



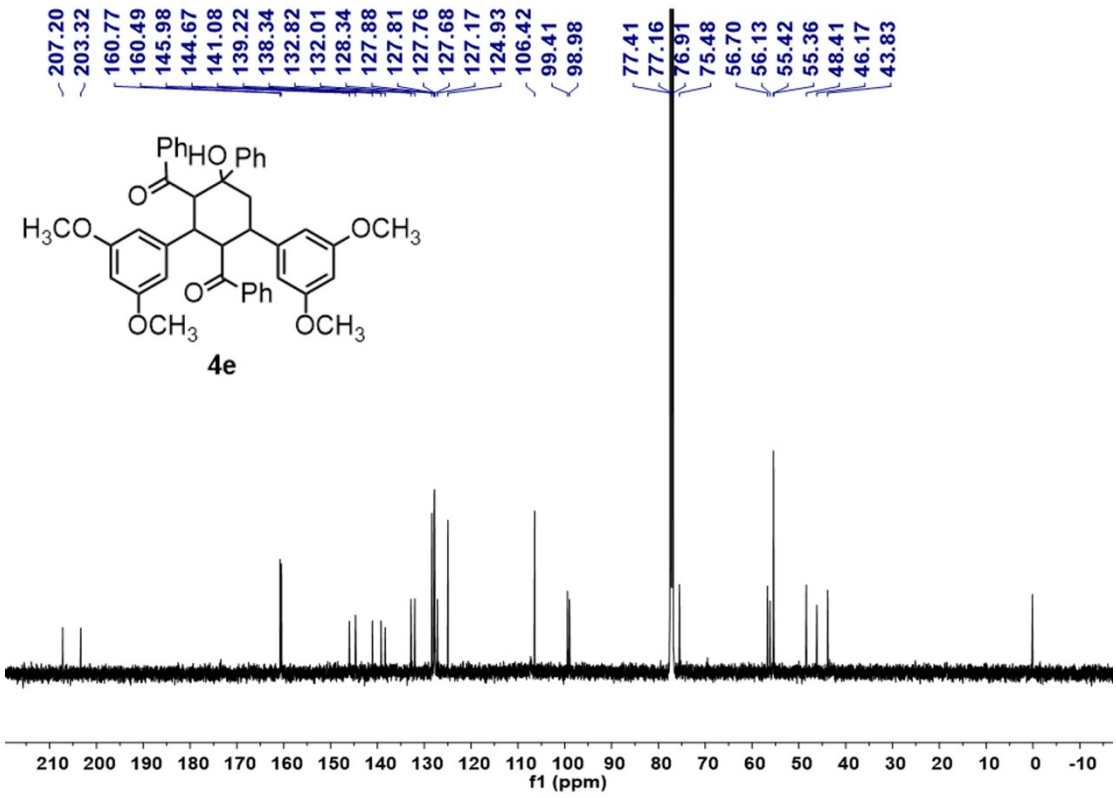
^{13}C NMR of **4d**



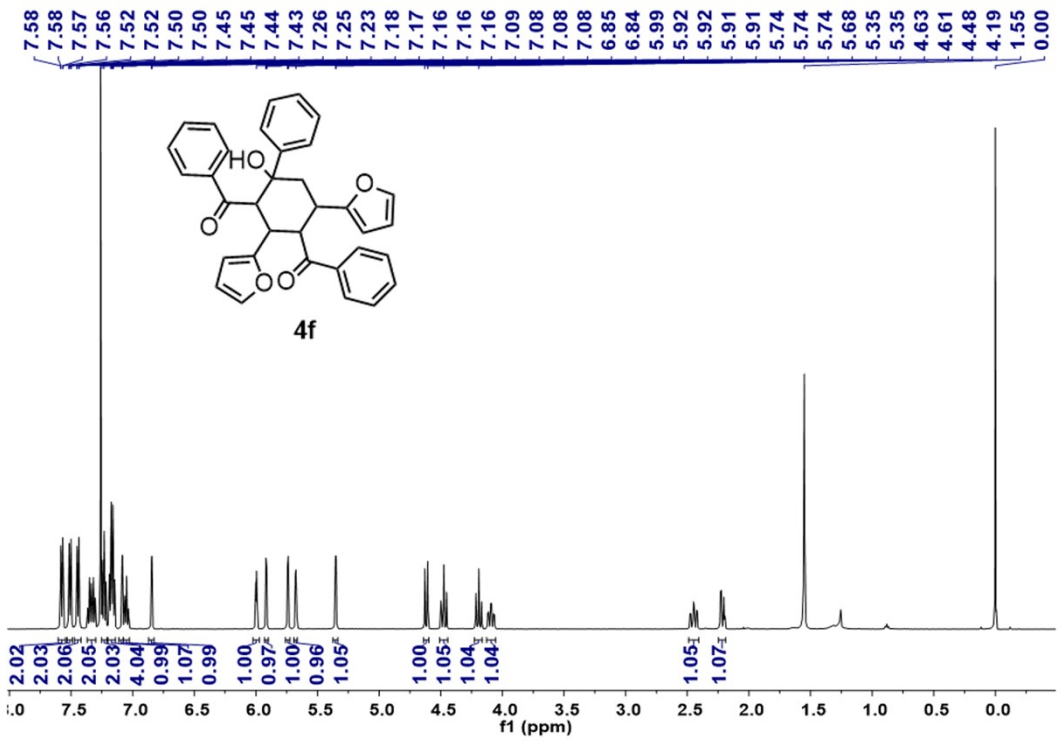
^{1}H NMR of **4e**



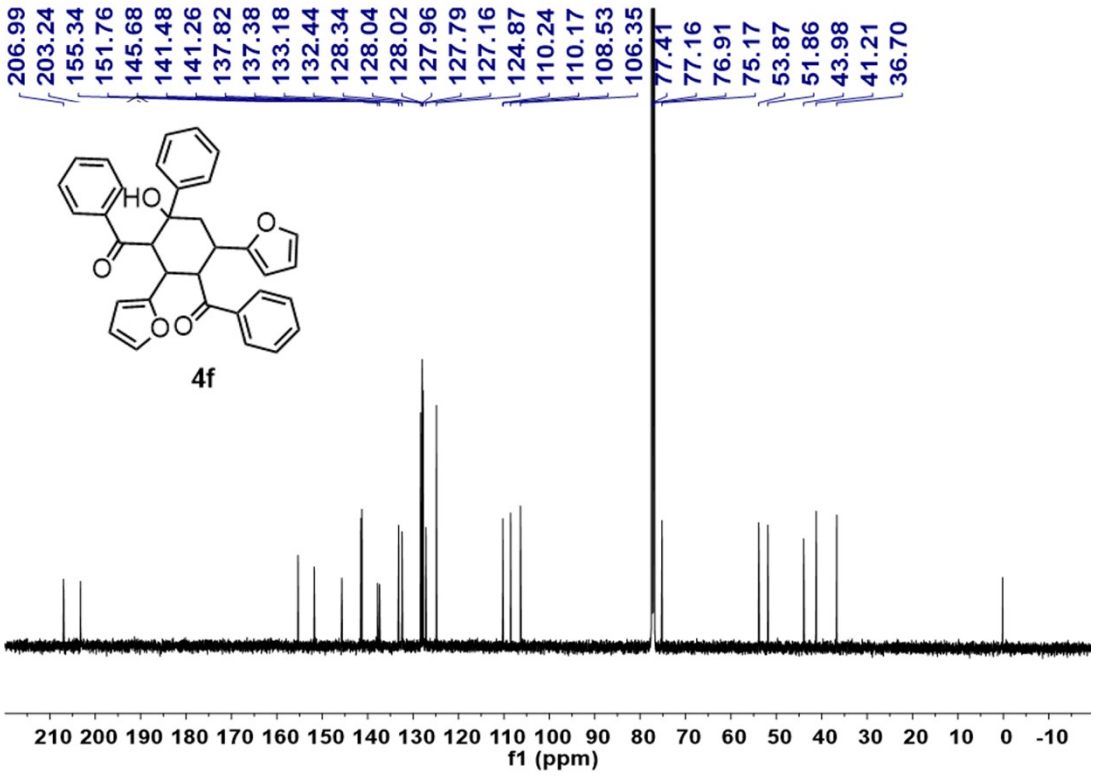
^{13}C NMR of **4e**



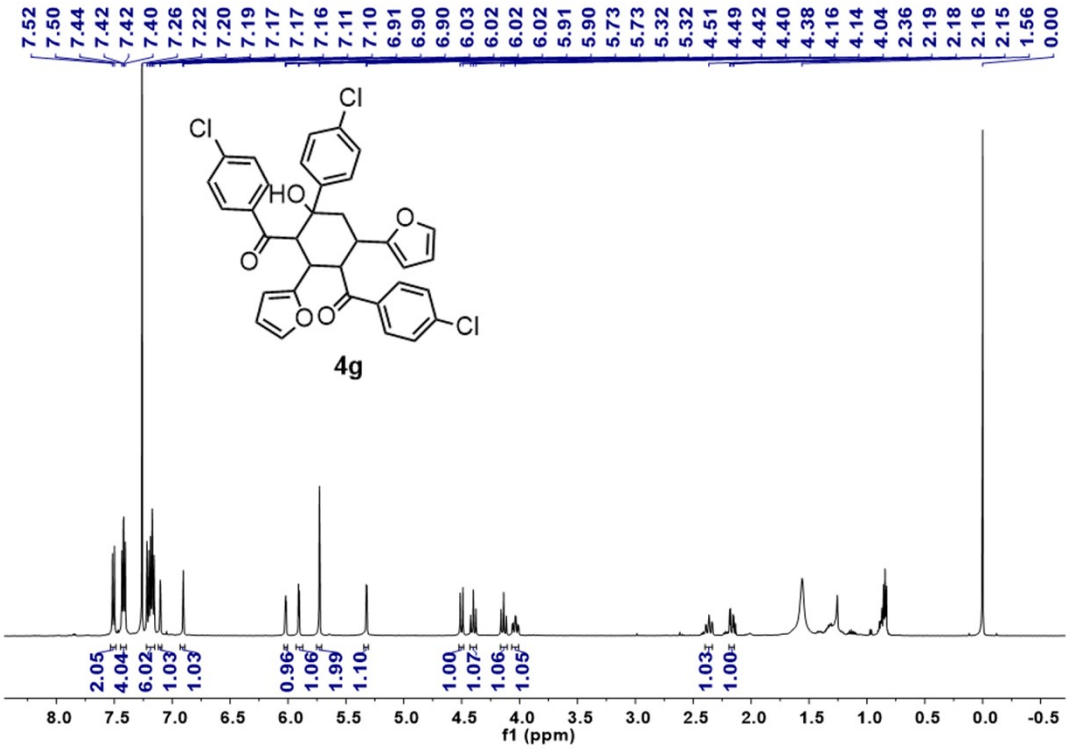
^{1}H NMR of **4f**



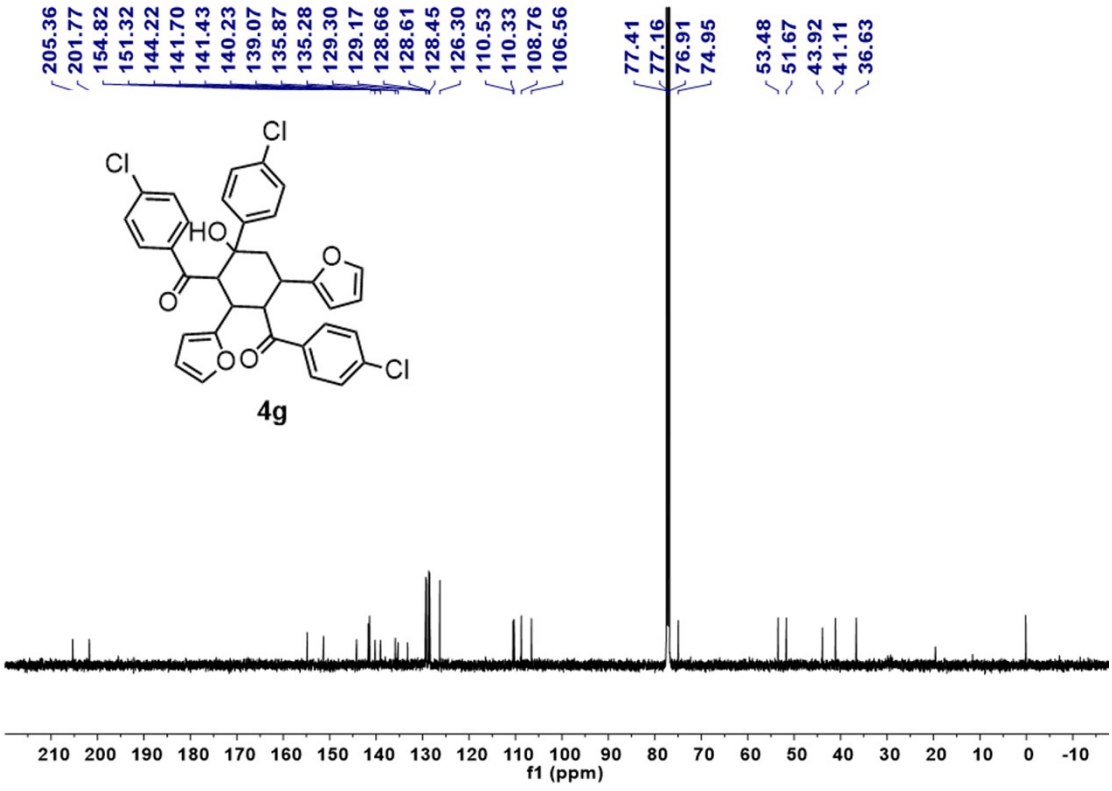
^{13}C NMR of **4f**



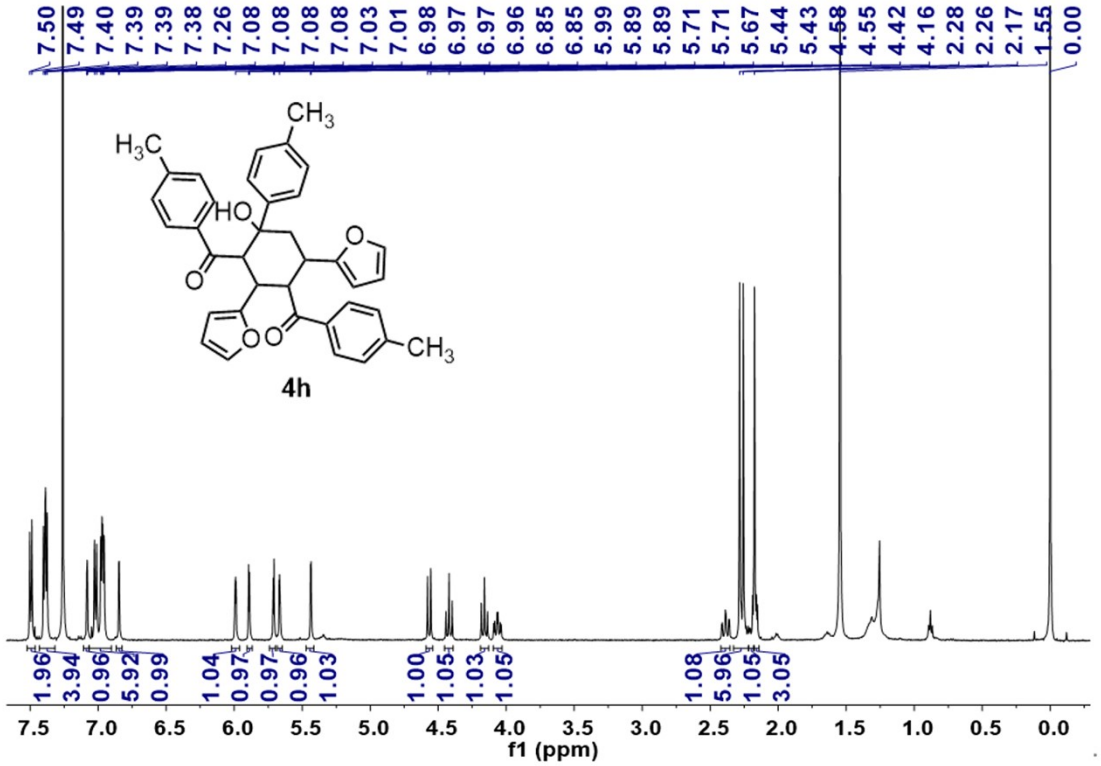
^{1}H NMR of **4g**



^{13}C NMR of **4g**



^{1}H NMR of **4h**



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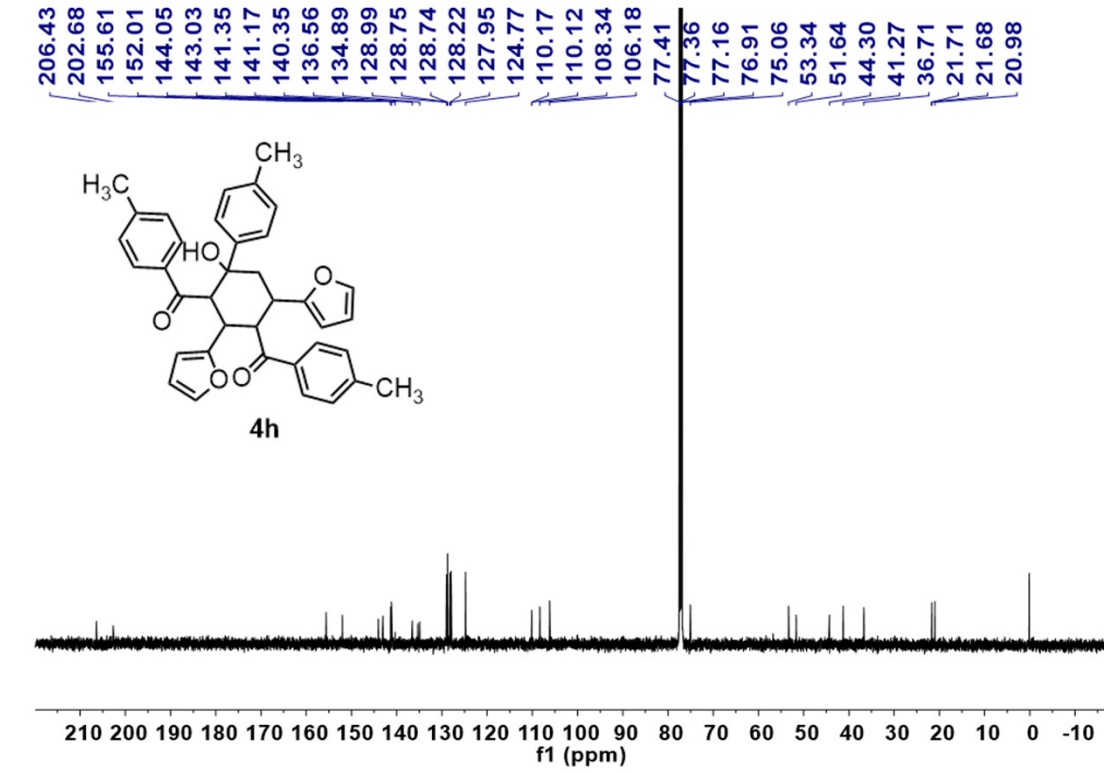
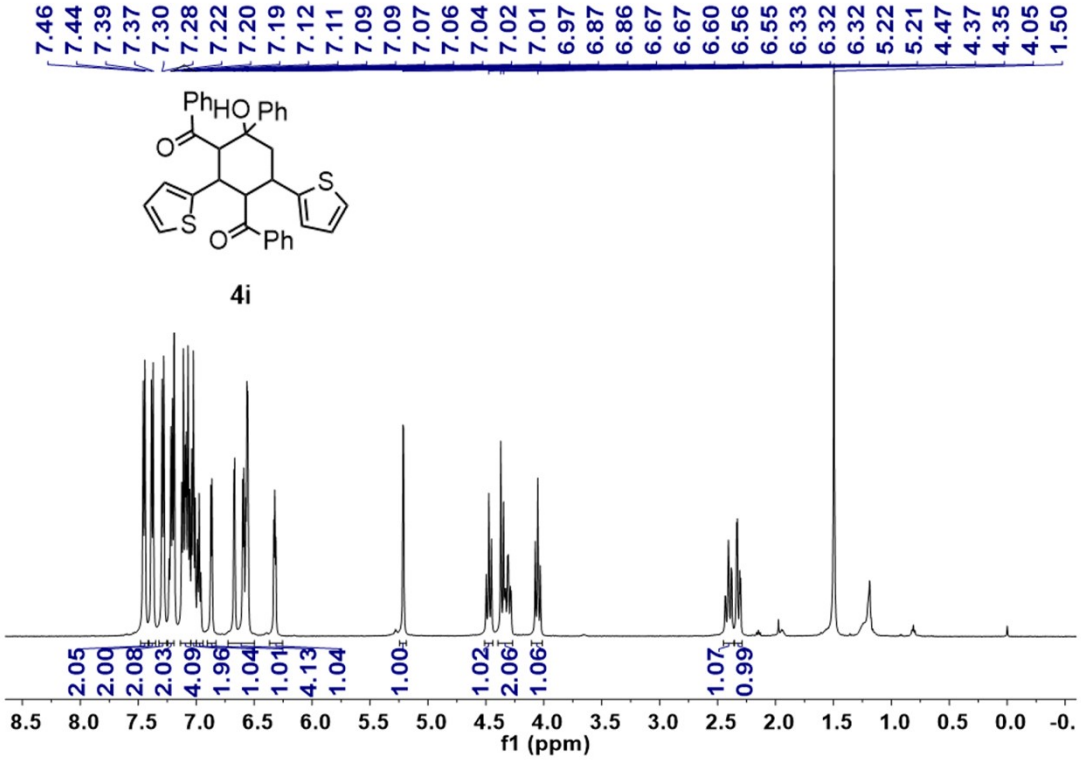


^{1}H NMR of **4i**



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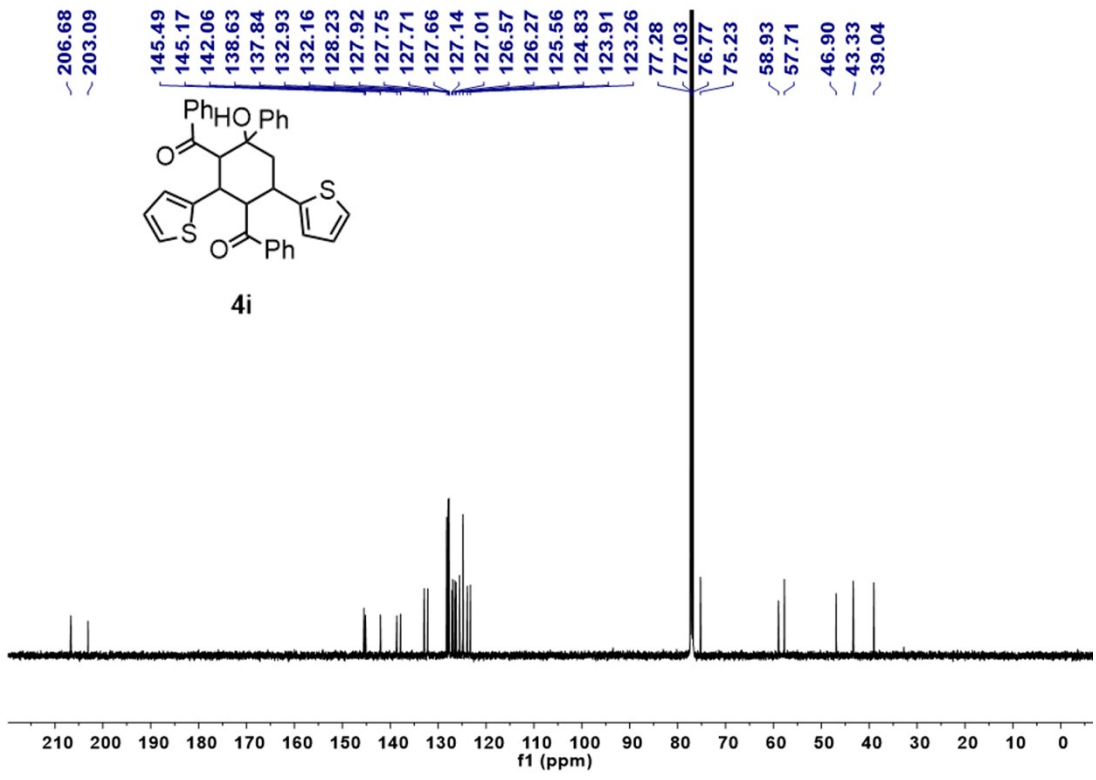
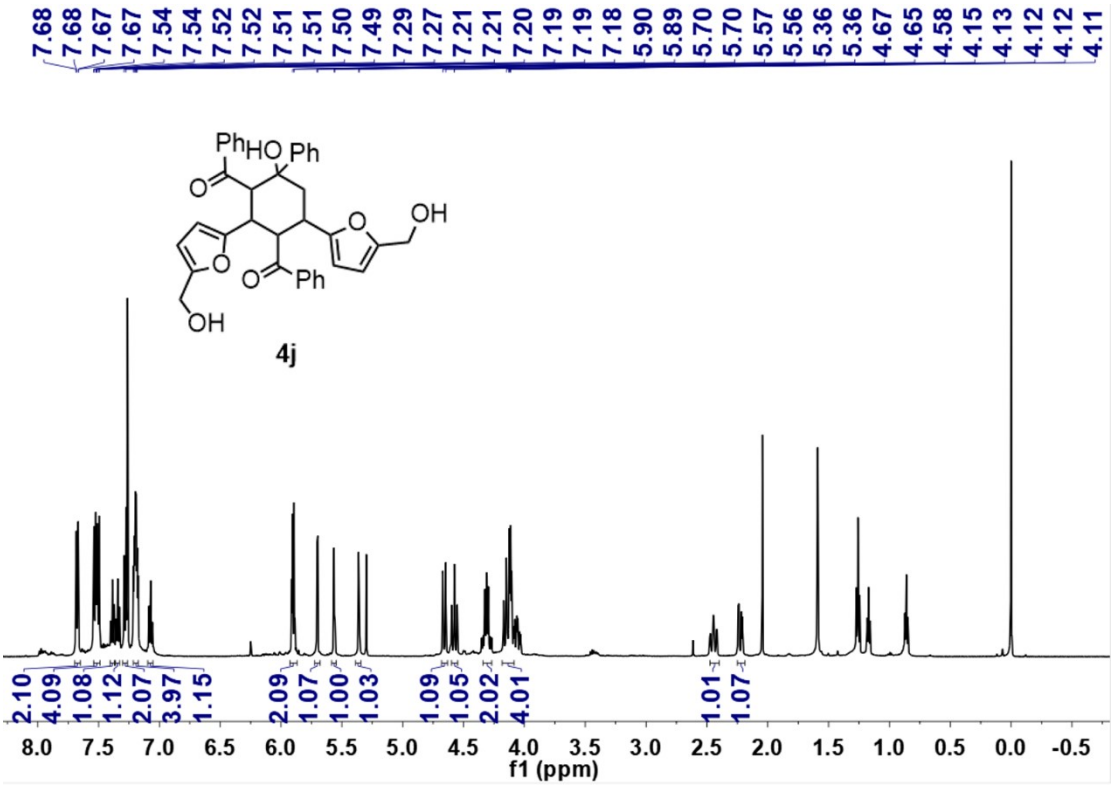
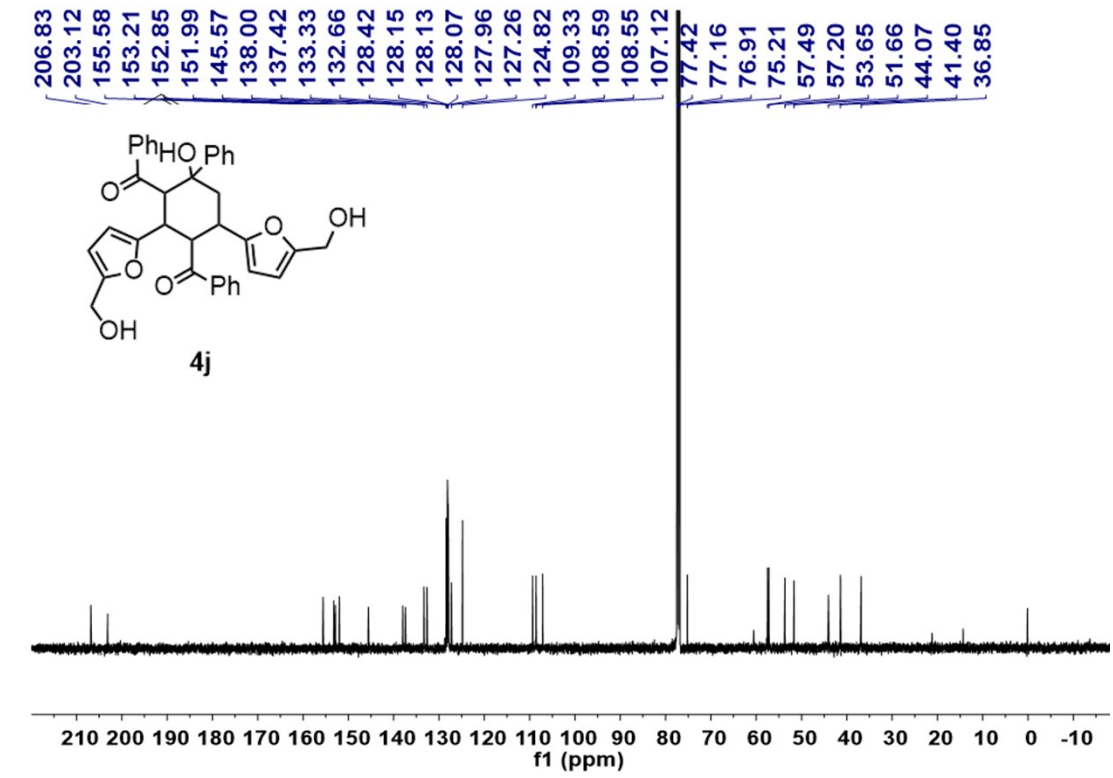


^{1}H NMR of **4j**



^{13}C NMR of **4j**



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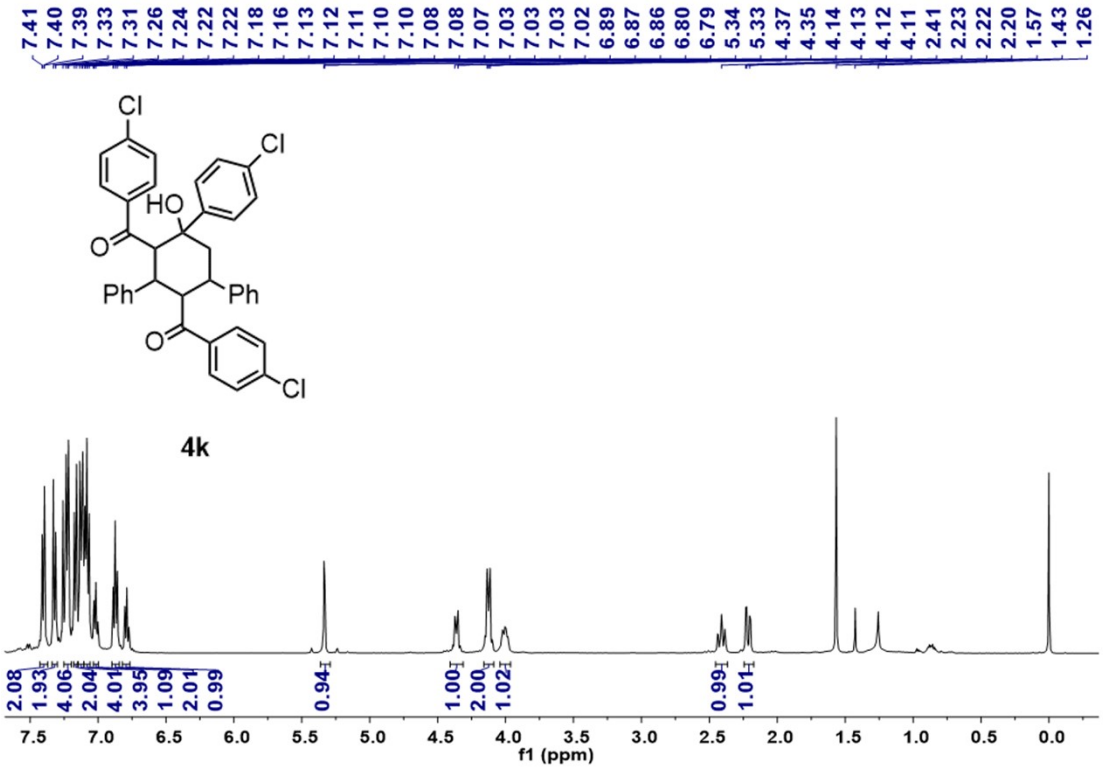
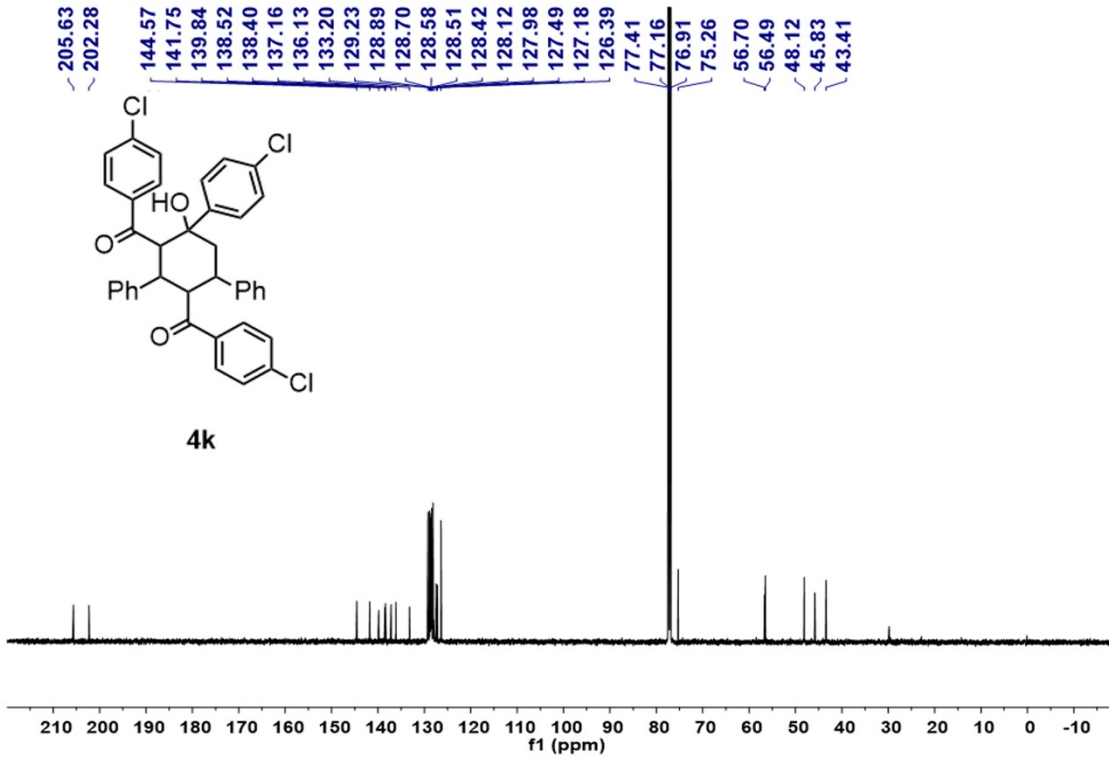
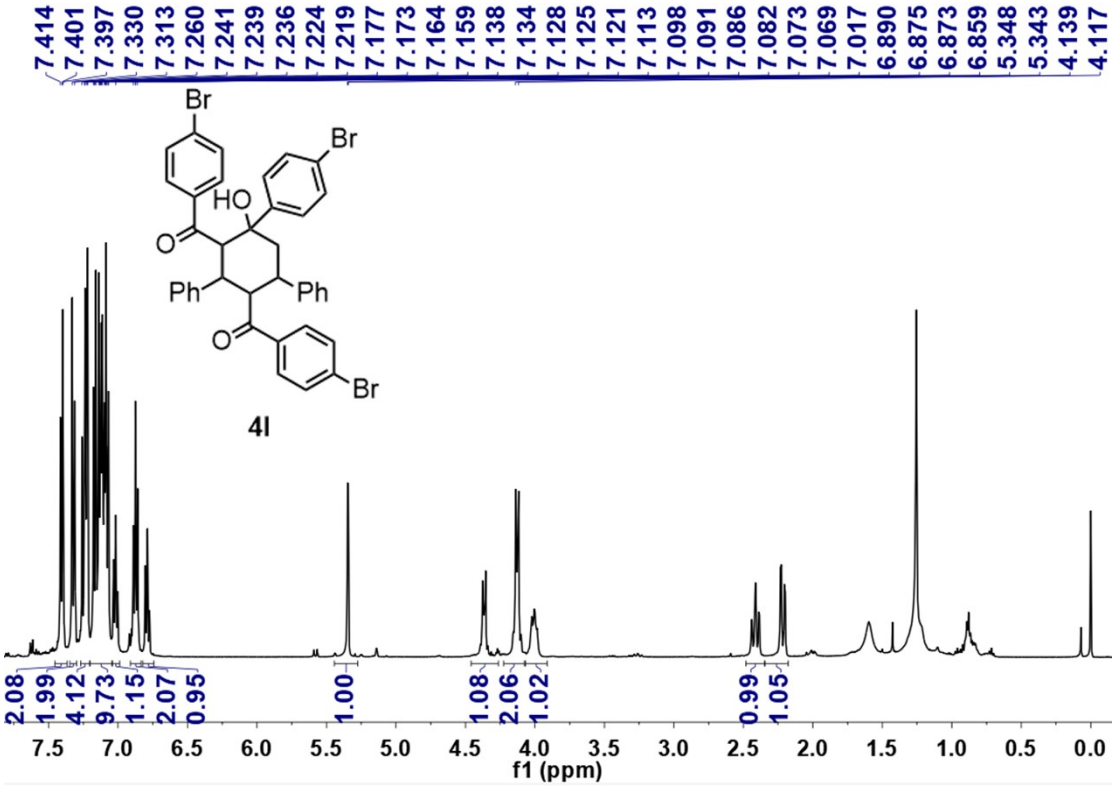


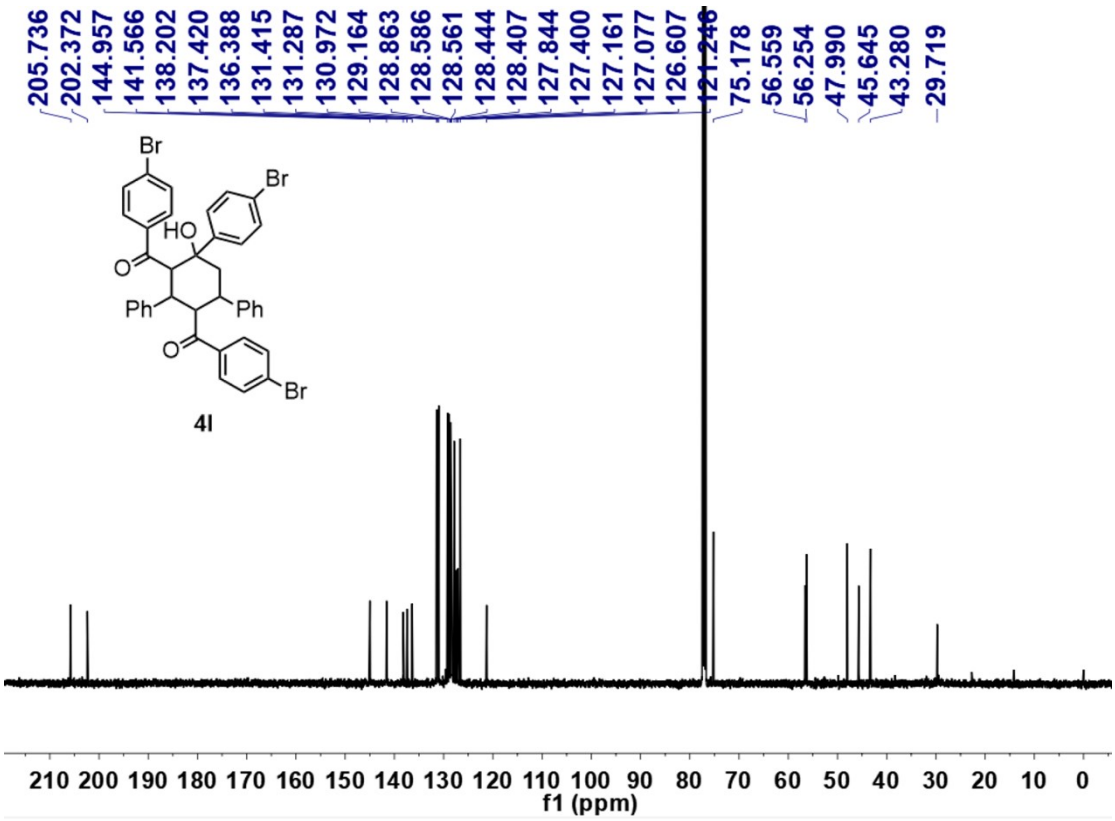
^{13}C NMR of **4k**



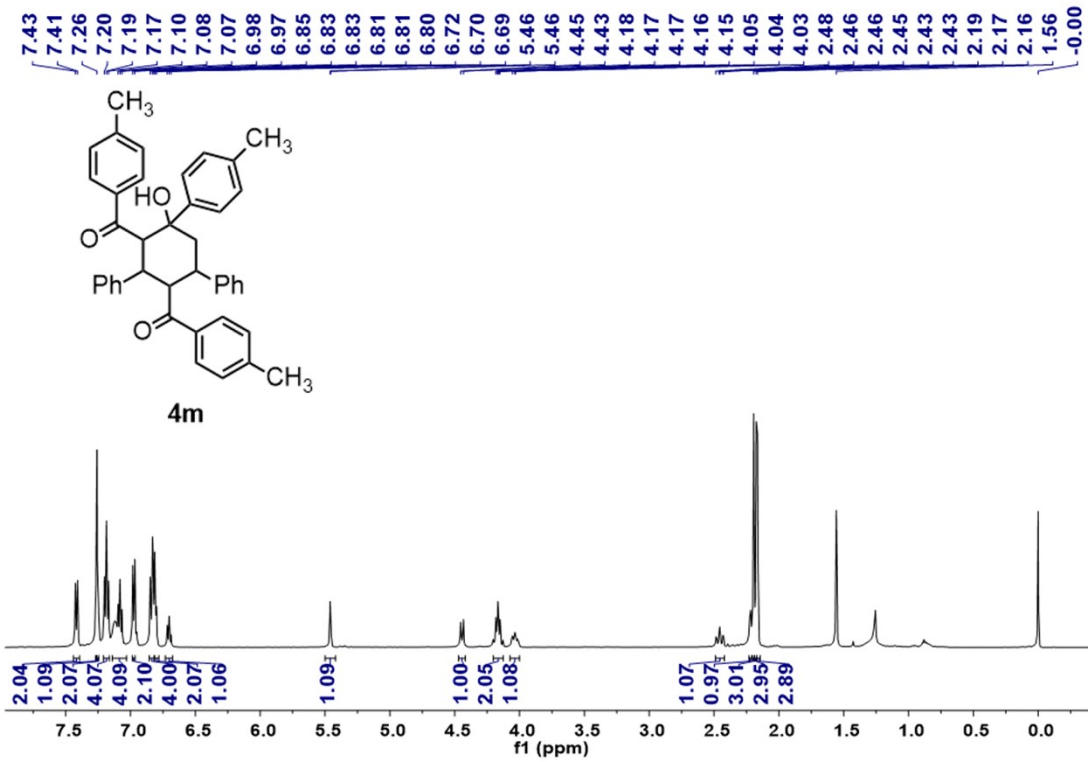
^{1}H NMR of **4l**



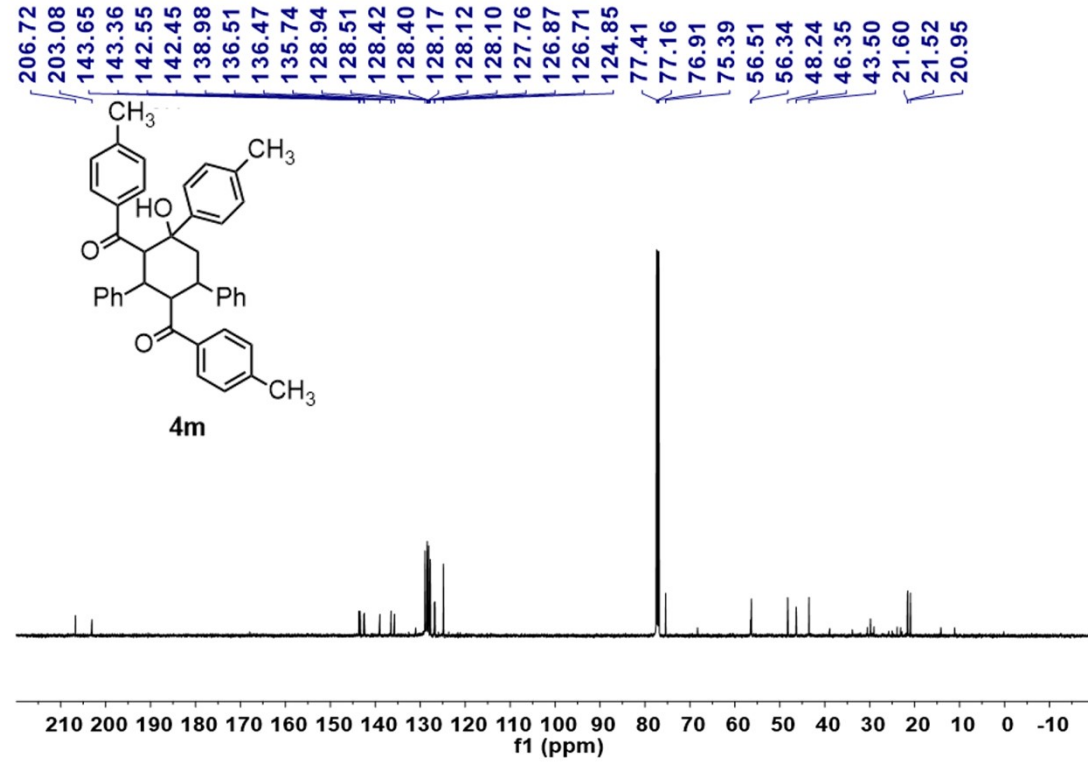
^{13}C NMR of **4l**



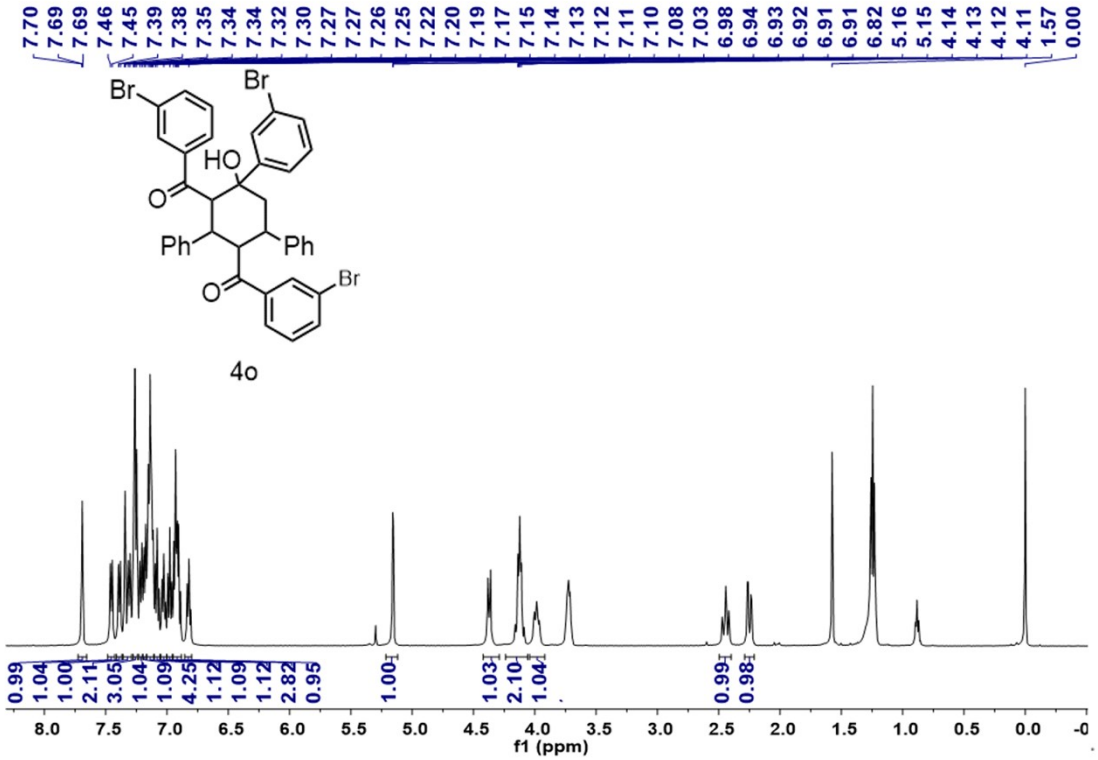
^{1}H NMR of **4m**



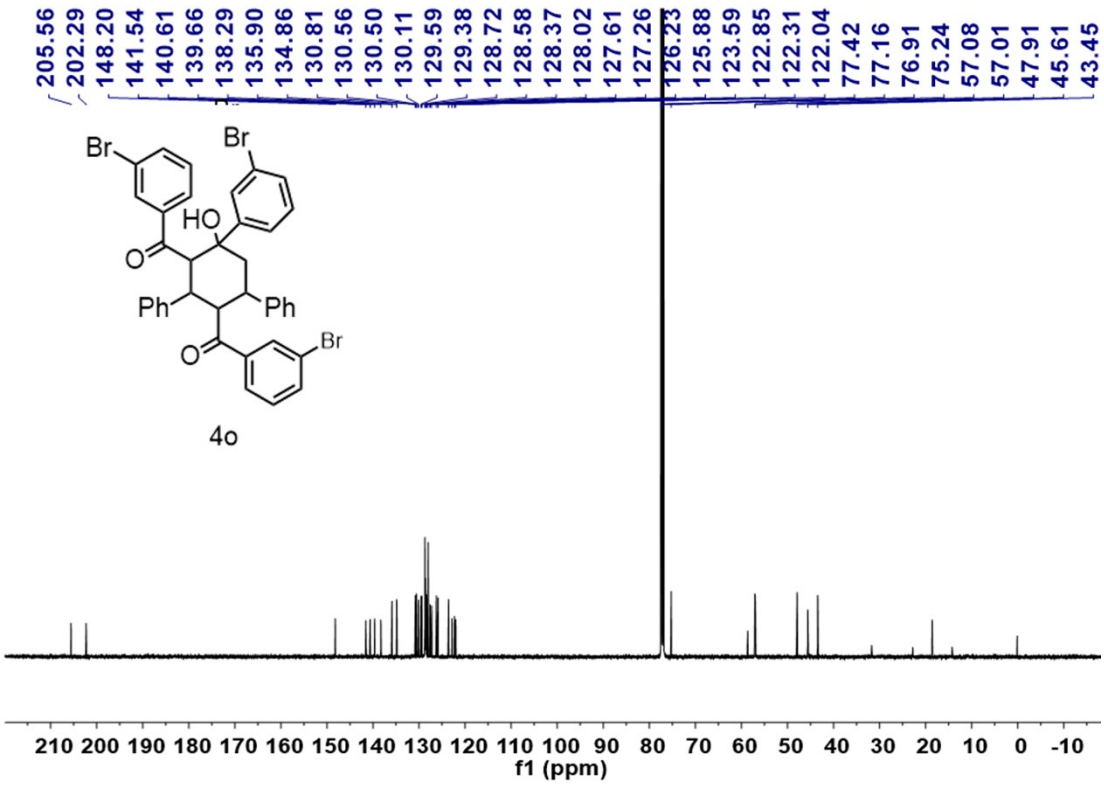
^{13}C NMR of **4m**



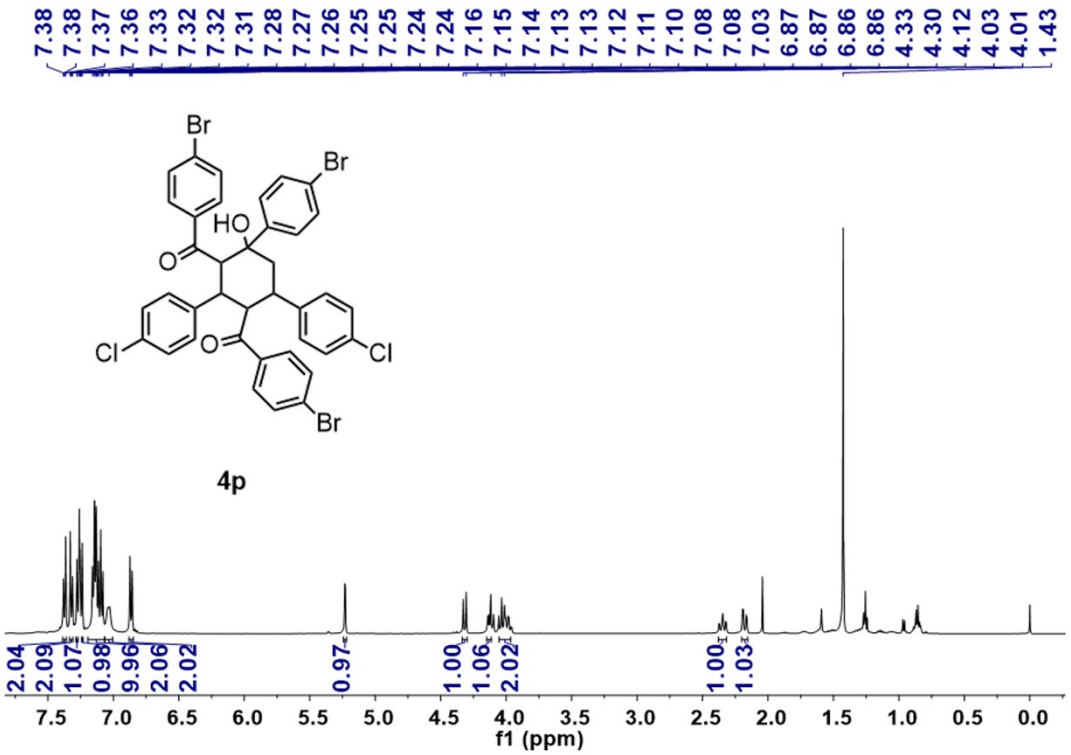
^{1}H NMR of **4o**



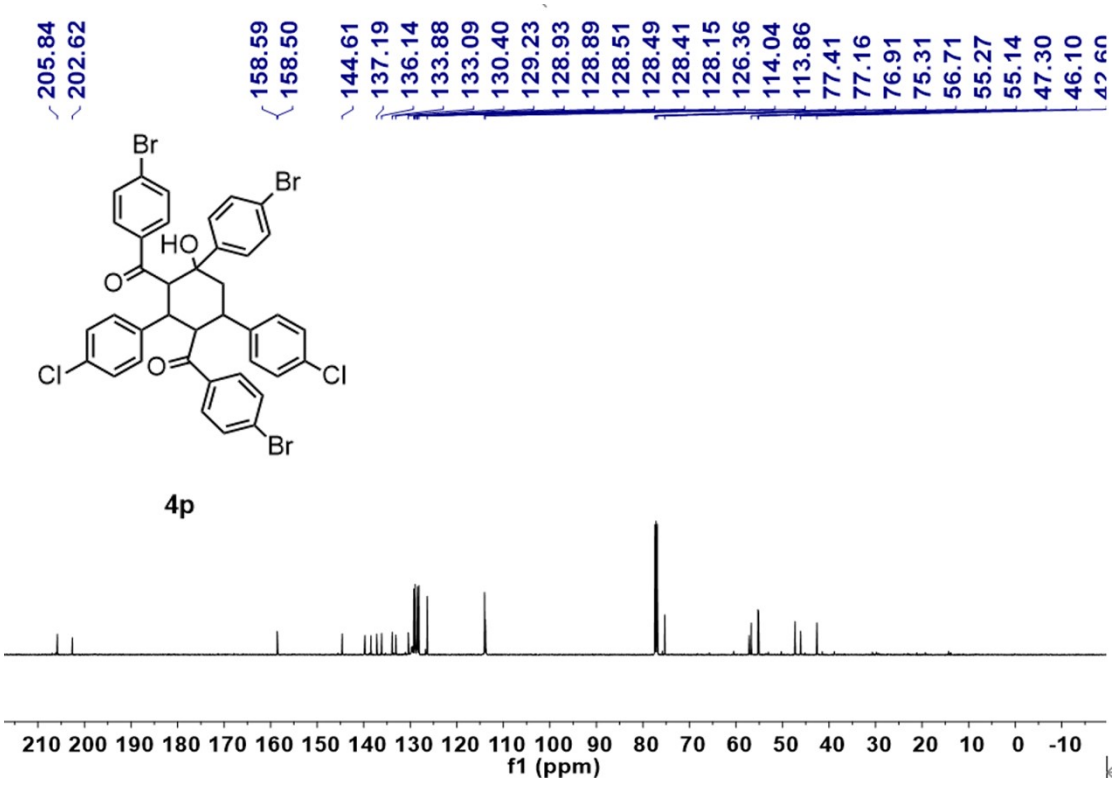
^{13}C NMR of **4o**



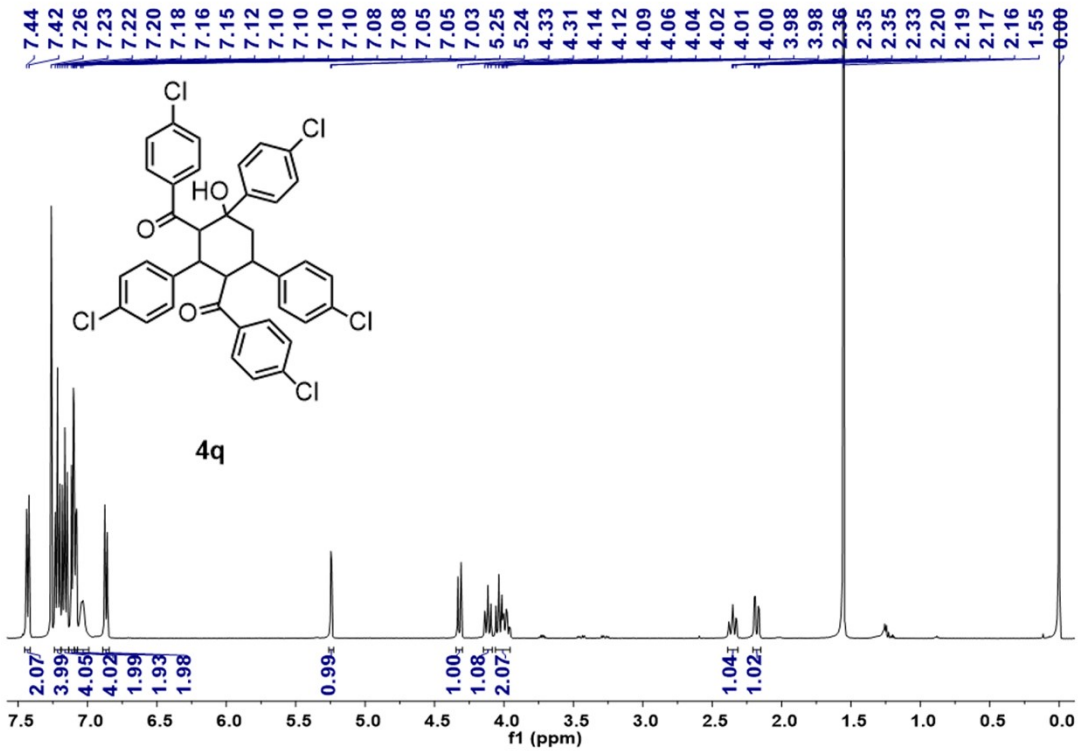
^{1}H NMR of **4p**



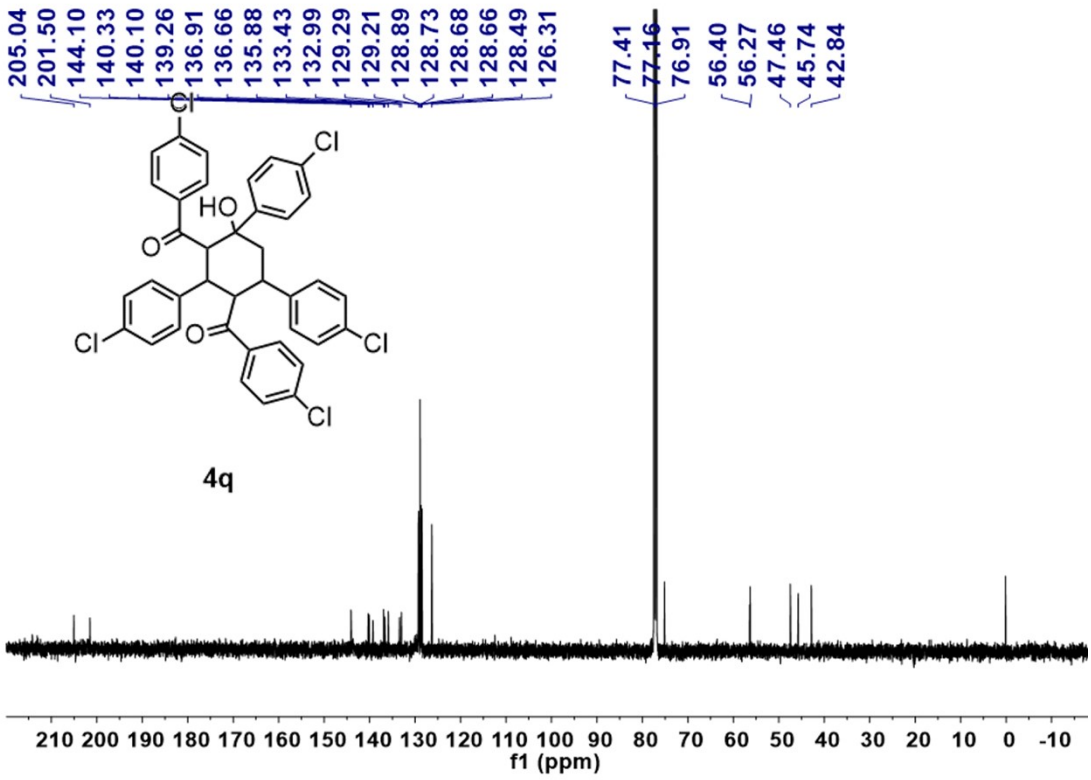
^{13}C NMR of **4p**



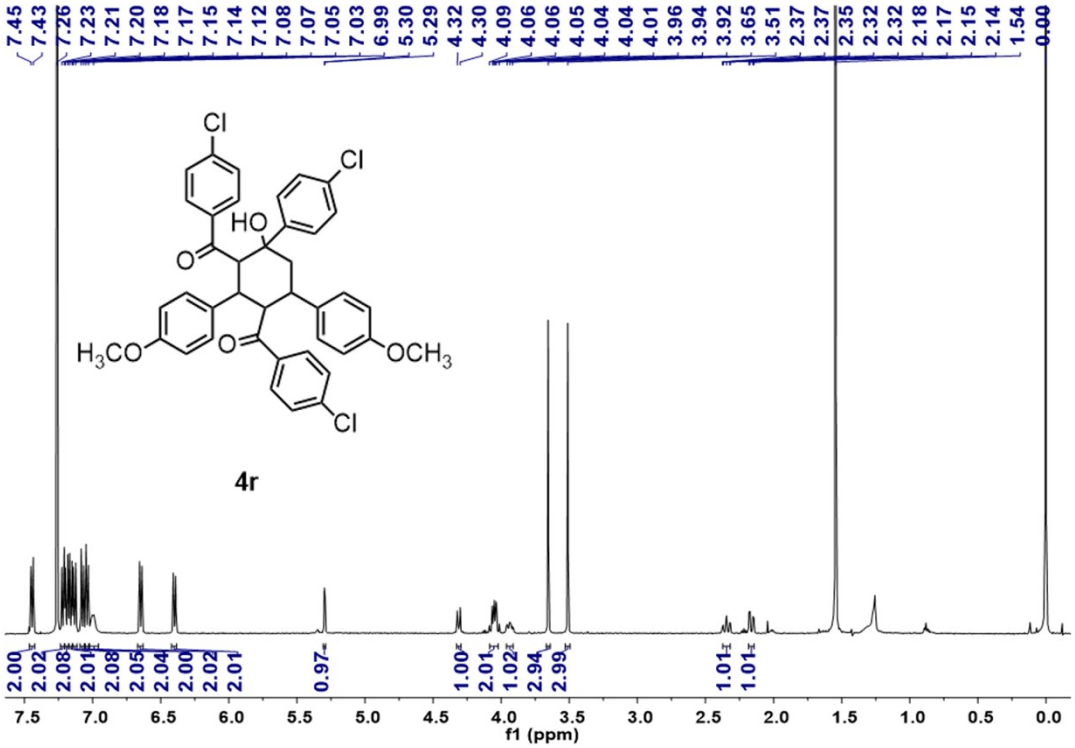
^{1}H NMR of **4q**



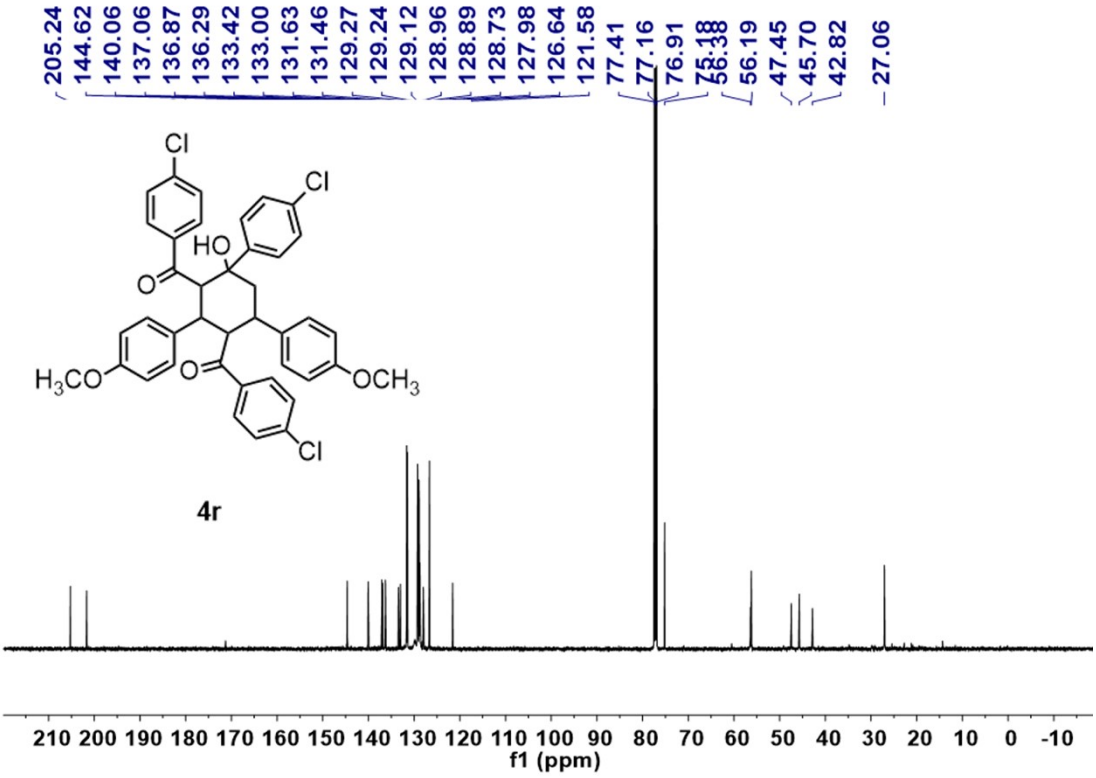
^{13}C NMR of **4q**



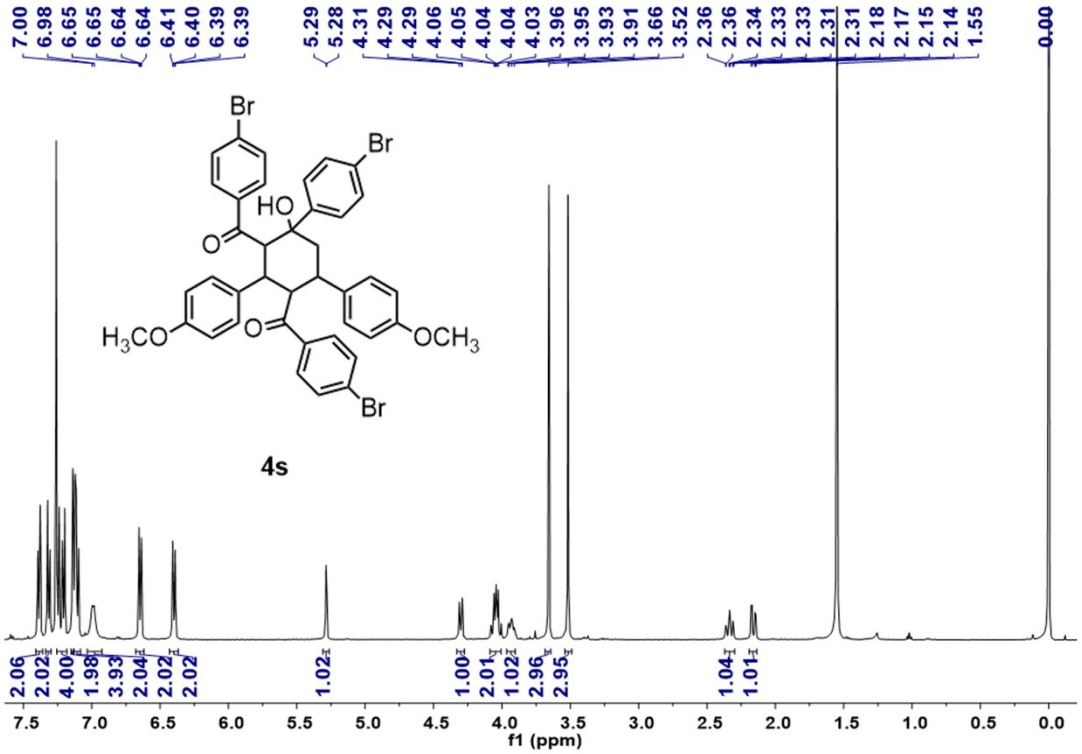
^{1}H NMR of **4r**



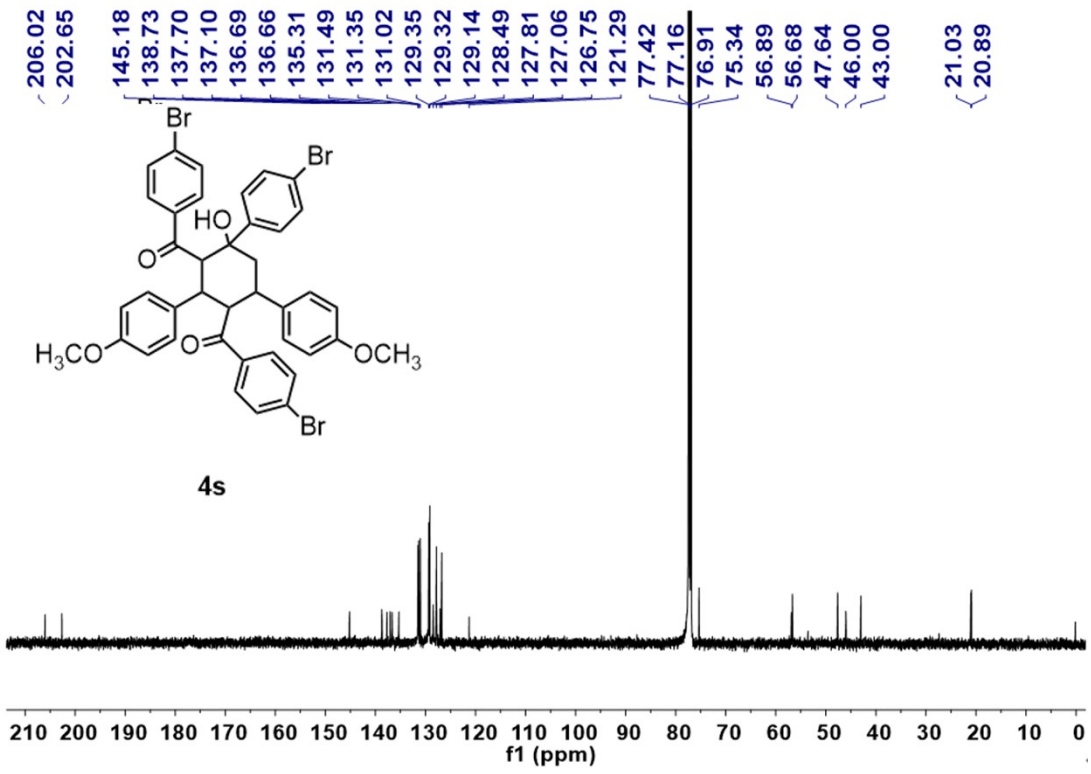
^{13}C NMR of **4r**



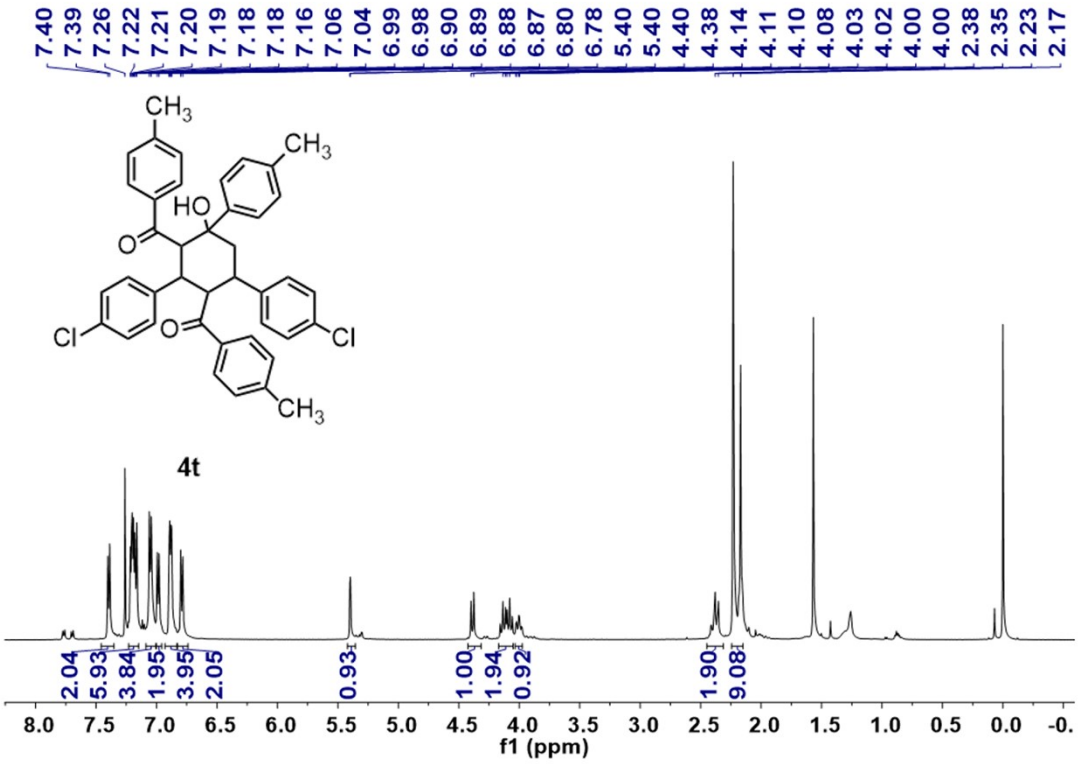
^{1}H NMR of **4s**



^{13}C NMR of **4s**



^{1}H NMR of **4t**



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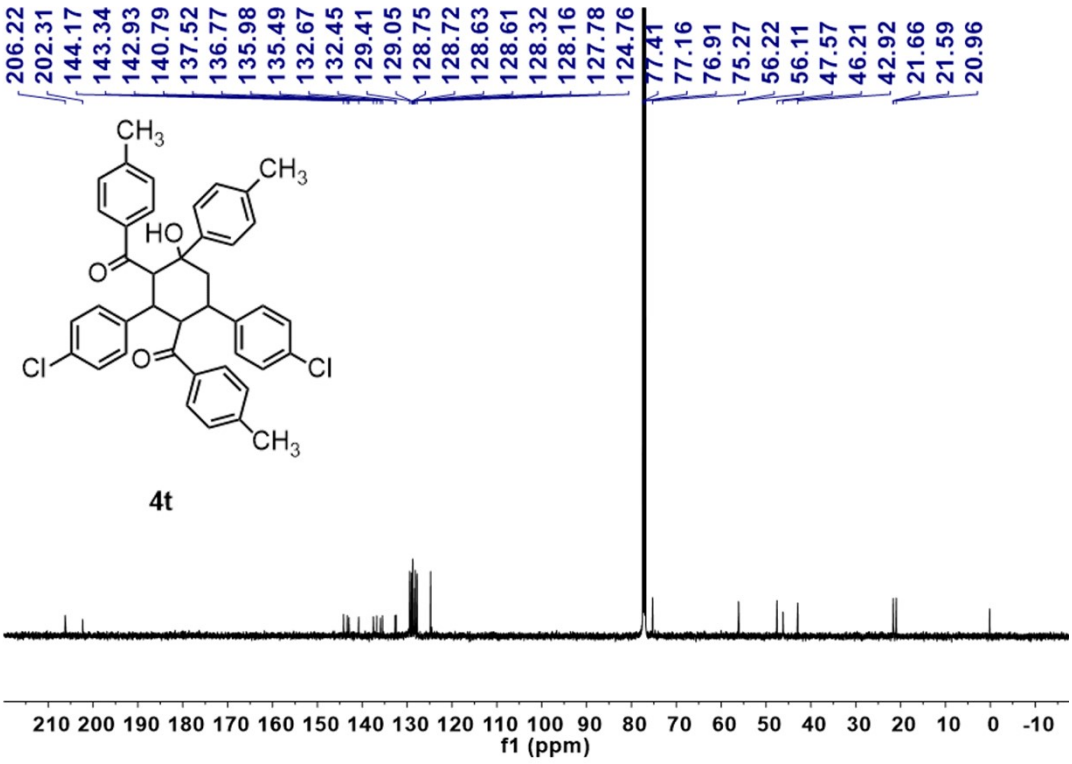
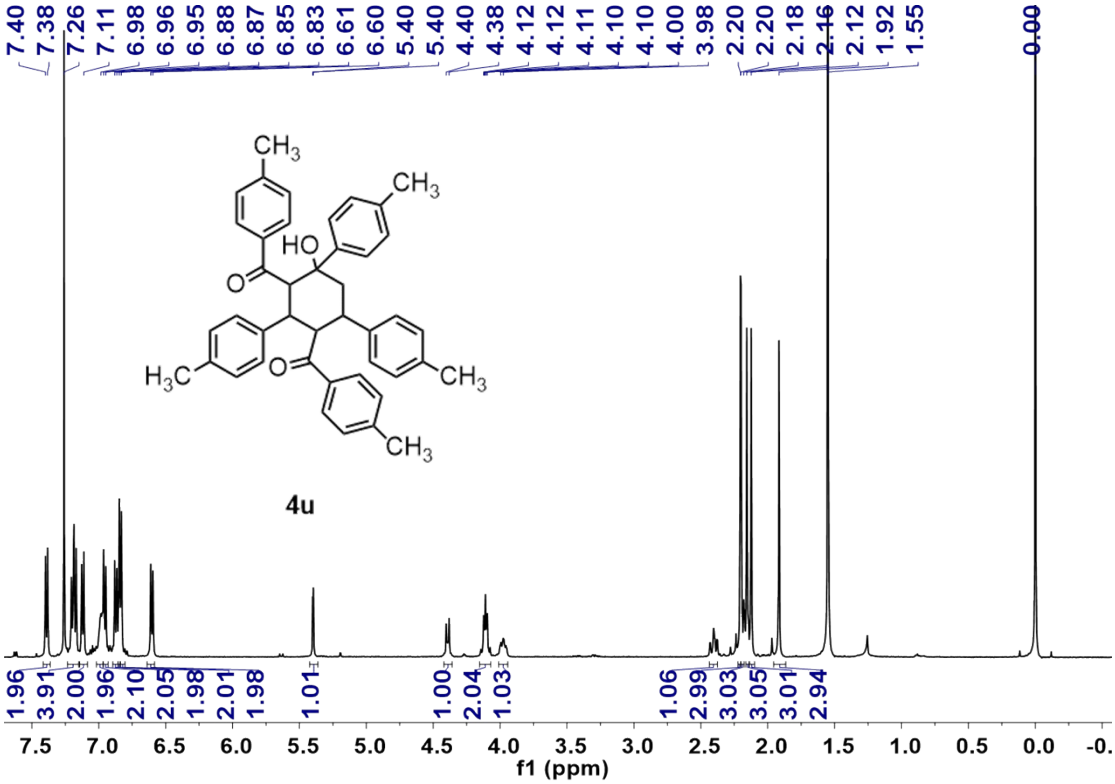


^{1}H NMR of **4u**



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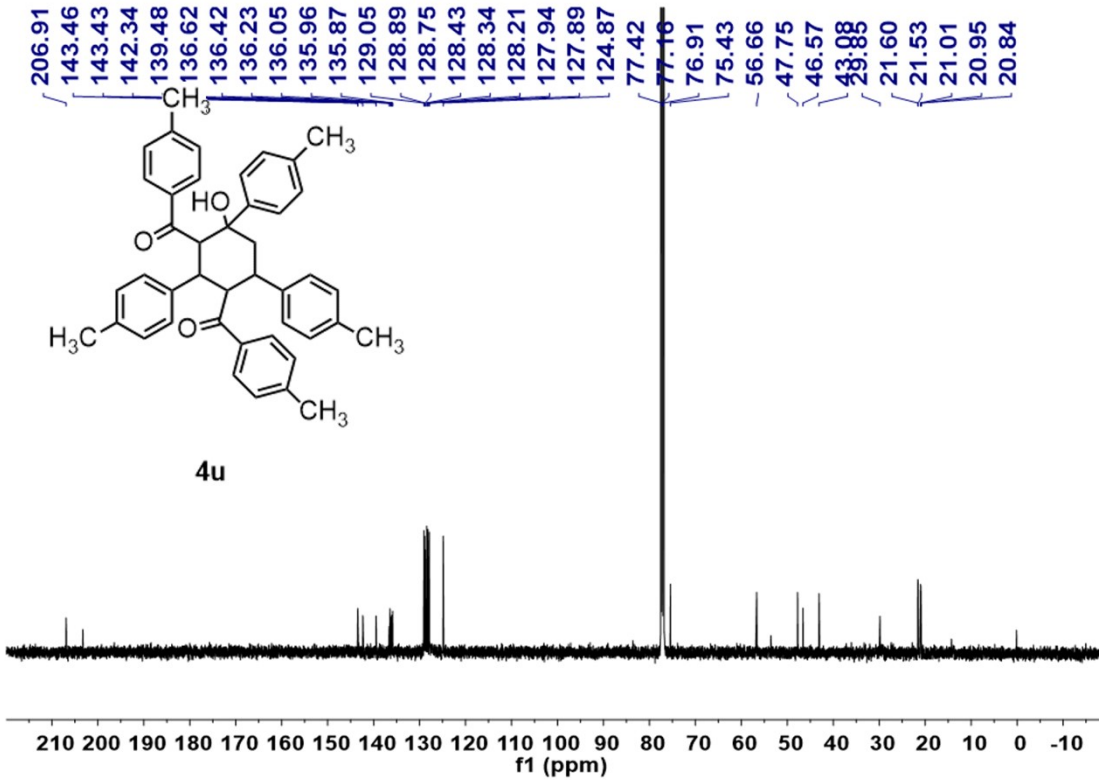

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