Supporting Information

Regioselectively Switchable Alkyne Cyclotrimerization Catalyzed by the System of Ni(II)/Bidentate *P*-Ligand/Zn with ZnI_2 as Additive

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1. General Considerations

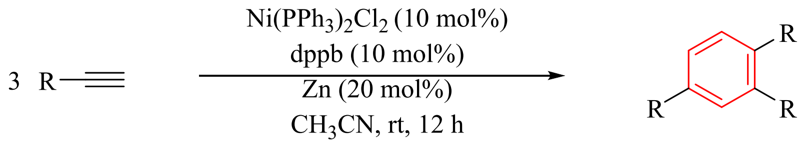
All manipulations were performed using standard Schlenk techniques and all reactions performed on a parallel reactor (WATTECS WP-TEC-1020) under argon atmosphere with oven-dried glassware. Acetonitrile, toluene, THF, DMF and dioxane were dried and distilled by the standard method. Zinc powder was activated with 30% dilute hydrochloric acid and stored in a dry box. Unless otherwise stated, starting materials were purchased from reagent suppliers (Innochem, Aldrich, Alfa, and so on) and used without further purification. Reactions were monitored by thin layer chromatography (TLC) and GC-MS analysis. Products on TLC were visualized by exposure to ultraviolet light (254 or 365 nm). GC-MS analysis was performed on a gas chromatography mass spectrometry (SHIMADZU GCMS-QP2010-Ultra) using a Rxi-5Sil MS column (30 m × 0.32 mmID, 0.25 μm df) with *n*-dodecane as an internal standard. Column chromatography was performed on silica gel (200–300 mesh) and the solvent eluents used were noted in brackets. All yields referred to were isolated yields (average of two runs) of compounds estimated to be >95% pure as determined by ^{1}H NMR.

^{1}H, ^{13}C and ^{19}F spectra were recorded on a Bruker AVANCE 400 (400 MHz), Bruker AVANCE III 400HD (400 MHz) and Bruker AVANCE III 500WB (500 MHz) spectrometer. Chemical shifts (δ) are recorded in ppm and coupling constants (*J*) are given in Hertz (Hz). Multiplicities are abbreviated as follows: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; dd = doublet of doublet; dt = doublet of triplet. High-resolution mass spectra (HRMS) were recorded by National Center for Mass Spectrometry in Beijing, Institute of Chemistry Chinese Academy of Sciences, on Thermo Fisher Scientific Exactive GC Orbitrap mass spectrometer.

Solvent abbreviation: petroleum ether = PE; dichloromethane = DCM; ethyl acetate = EA; tetrahydrofuran = THF; dimethylformamide = DMF; triethylamine = TEA

2. General Procedures

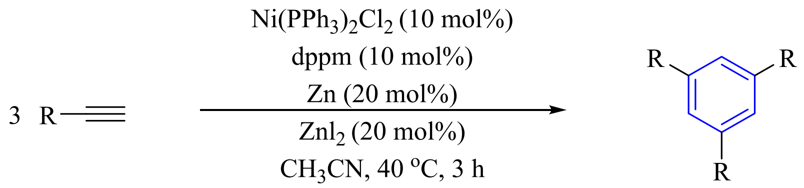
2.1 Synthesis of 1,2,4-trisubstituted benzenes 2 (Condition A)



1 **2**

An oven-dried 25-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%, 0.1 mmol, 66 mg), dppb (10 mol%, 0.1 mmol, 43 mg) and zinc dust (20 mol%, 0.2 mmol, 13 mg) was evacuated and backfilled with argon, with the operation being repeated twice. Anhydrous CH_3CN (2 mL) was added and the mixture stirred at 80 °C for about 7 minutes until the solution turned yellow. The mixture was allowed to cool to ambient temperature (20-25 °C) immediately and a solution of alkyne **1** (1 mmol) in anhydrous CH_3CN (2 mL) was slowly added via syringe. The reaction mixture was stirred on WATTECS WP-TEC-1020 parallel reactor at room temperature for 12 hours until the reaction was complete (monitored by TLC and GC-MS). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (eluents noted in brackets) to afford the desired product **2**. The spectroscopic characterizations of known products were compared with published data.

2.2 Synthesis of 1,3,5-trisubstituted benzenes 3 (Condition B)

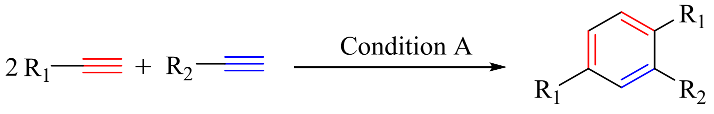


1 **3**

An oven-dried 25-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%, 0.1 mmol, 66 mg), dppm (10 mol%, 0.1 mmol, 39 mg), zinc dust (20 mol%, 0.2 mmol, 13 mg) and anhydrous ZnI_2 (20 mol%, 0.2 mmol, 64 mg) was evacuated and backfilled with argon, with the operation being repeated twice. A solution of alkyne **1** (1 mmol) in anhydrous CH_3CN (2 mL) was slowly added via syringe. The reaction mixture was stirred on WATTECS WP-TEC-1020 parallel reactor at 40 °C for 3 hours until the reaction was complete (monitored by TLC and GC-MS). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (eluents noted in brackets) to afford the desired product **3**. The spectroscopic characterizations of known products were compared with published data.

2.3 General procedure for cross-cyclotrimerization of alkynes

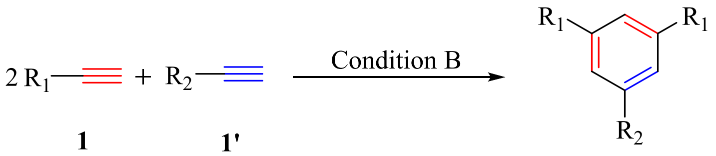
2.3.1 Synthesis of 1,2,4-heterotrisubstituted benzenes 5 (Condition A)



1 **1'** **5**

An oven-dried 25-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%), dppb (10 mol%) and zinc dust (20 mol%) was evacuated and backfilled with argon, with the operation being repeated twice. Anhydrous CH_3CN (2 ml) was added and the mixture stirred at 80 ^oC for about 7 minutes until the solution turned yellow. The mixture was allowed to cool to room temperature (25 ^oC) immediately. A mixture of alkyne **1** (0.9 mmol) and alkyne **1'** (0.3 mmol) in anhydrous CH_3CN (2 mL) was slowly added via syringe. The reaction mixture was stirred on WATTECS WP-TEC-1020 parallel reactor at room temperature for 12 hours until the reaction was complete (monitored by TLC and GC-MS). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (eluents noted in brackets) to afford the desired product **5**. The spectroscopic characterizations of known products were compared with published data.

2.3.2 Synthesis of 1,3,5-trisubstituted benzenes 6 (Condition B)

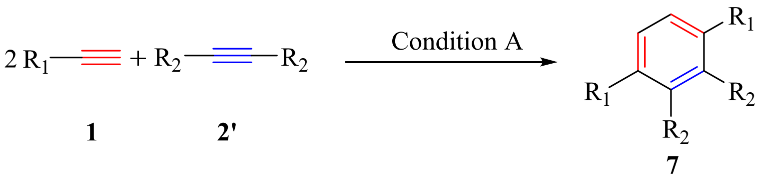


1 **1'** **6**

An oven-dried 25-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%), dppm (10 mol%), zinc dust (20 mol%) and anhydrous ZnI_2 (20 mol%) was evacuated and backfilled with argon, with the operation being repeated twice. A mixture of alkyne **1**

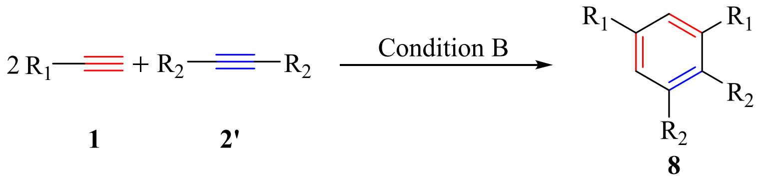
(0.9 mmol) and alkyne **1'** (0.3 mmol) in anhydrous CH_3CN (2 mL) was slowly added via syringe. The reaction mixture was stirred on WATTECS WP-TEC-1020 parallel reactor at 40 ^{o}C for 3 hours until the reaction was complete (monitored by TLC and GC-MS). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (eluents noted in brackets) to afford the desired product **6**. The spectroscopic characterizations of known products were compared with published data.

2.3.3 Synthesis of 1,2,3,4-tetrasubstituted benzenes 7 (Condition A)



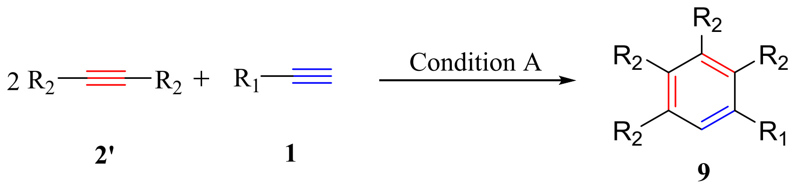
The procedure to synthesize products **7** was the same as the procedure 2.3.1 except that a mixture of alkyne **1** (0.9 mmol) and alkyne **2'** (0.3 mmol) was used in place of the previous alkyne combination.

2.3.4 Synthesis of 1,2,3,5-tetrasubstituted benzenes 8 (Condition B)



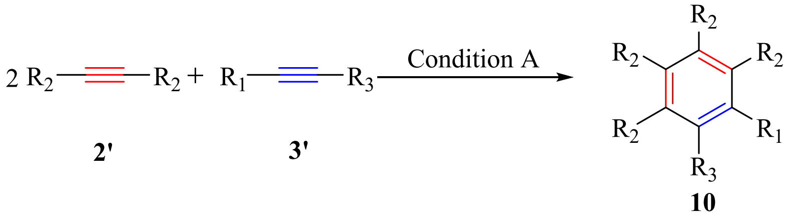
The procedure to synthesize products **8** was the same as the procedure 2.3.2 except that a mixture of alkyne **1** (0.9 mmol) and alkyne **2'** (0.3 mmol) was used in place of the previous alkyne combination.

2.3.5 Synthesis of 1,2,3,4,5-pentasubstituted benzenes 9 (Condition A)



The procedure to synthesize products **9** was the same as the procedure 2.3.1 except that a mixture of alkyne **2'** (0.9 mmol) and alkyne **1** (0.3 mmol) was used in place of the previous alkyne combination.

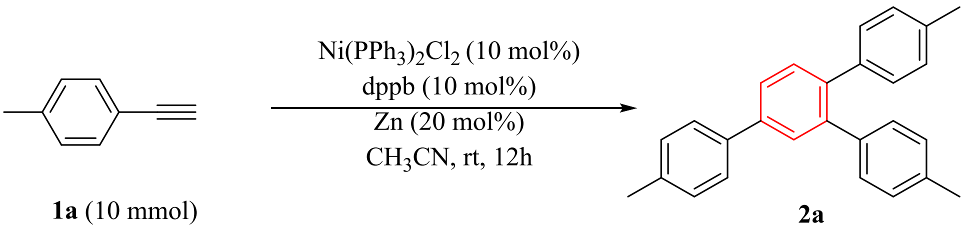
2.3.6 Synthesis of 1,2,3,4,5,6-hexasubstituted benzenes 10 (Condition A)



The procedure to synthesize products **10** was the same as the procedure 2.3.1 except that a mixture of alkyne **2'** (0.9 mmol) and alkyne **3'** (0.3 mmol) was used in place of the previous alkyne combination.

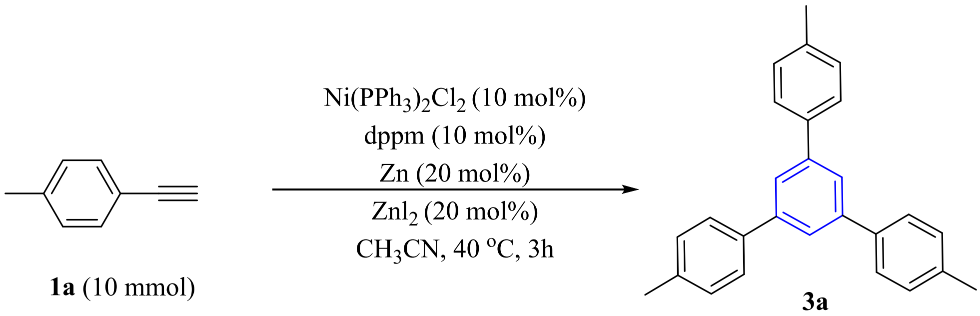
2.4 General procedure for 10 mmol-scale reaction

2.4.1 Synthesis of 2a (Condition A)



An oven-dried 100-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%), dppb (10 mol%) and zinc dust (20 mol%) was evacuated and backfilled with argon, with the operation being repeated twice. Anhydrous CH_3CN (20 mL) was added and the mixture stirred at 80 ^{o}C (oil bath) for about 7 minutes until the solution turned yellow. The mixture was allowed to cool to room temperature (25 ^{o}C) immediately and a solution of alkyne **1a** (10 mmol) in anhydrous CH_3CN (20 mL) was slowly added via syringe. The reaction mixture was stirred at room temperature for 12 hours until the reaction was complete (monitored by TLC). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (PE/EA = 10:1) to afford the corresponding product **2a** (1.05 g, 90%).

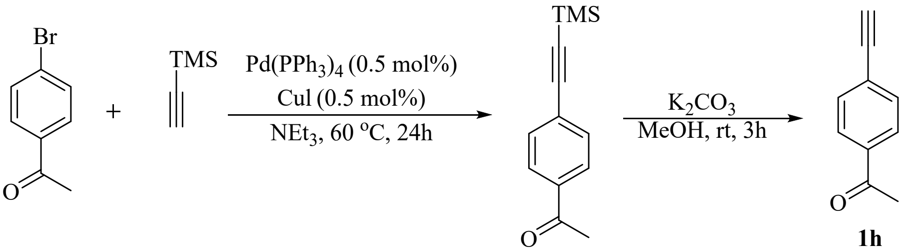
2.4.2 Synthesis of 3a (Condition B)



An oven-dried 100-mL flask charged with $Ni(PPh_3)_2Cl_2$ (10 mol%), dppm(10 mol%), zinc dust (20 mol%) and anhydrous ZnI_2 (20 mol%) was evacuated and backfilled with argon, with the operation being repeated twice. A solution of alkyne **1a** (10 mmol) in anhydrous CH_3CN (20 mL) was slowly added via syringe. The reaction mixture was stirred at 40 °C for 3 hours until the reaction was complete (monitored by TLC). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (PE/EA = 10:1) to afford the corresponding product **3a** (0.95 g, 82%).

3. Synthesis of Starting Materials

3.1 Synthesis of 4-ethynylacetophenone 1h.

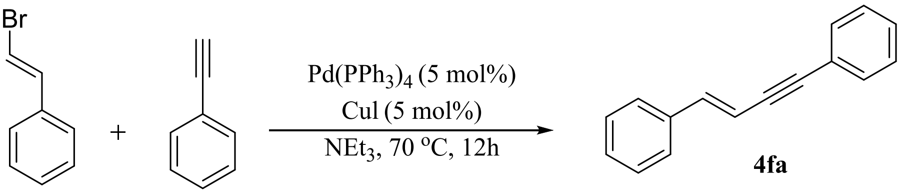


1h

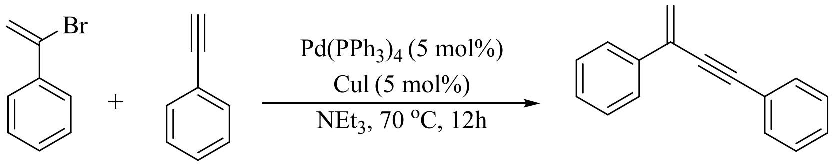
Step 1: An oven-dried 100-mL flask charged with $Pd(PPh_3)_4$ (57.8 mg, 0.5 mol%), CuI (9.5 mg, 0.5 mol%) and 4-bromoacetophenone (1.99 g, 10 mmol) was evacuated and backfilled with argon, with the operation being repeated twice. A solution of trimethylsilylacetylene (1.56 ml, 11 mmol) in triethylamine (60 ml) was slowly added via syringe. The reaction mixture was performed at 60 ^{o}C for 24 hours until the reaction was complete (monitored by TLC). After complete conversion the reaction mixture was allowed to cool to room temperature and washed with brine (25 ml). The aqueous layer was extracted with ether (2 x 50 ml) and the combined organic layers were dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (PE / EA = 10:1) to afford the corresponding product 1-(4-((trimethylsilyl)ethynyl)phen yl)ethan-1-one (brown oil, 2.15 g, 99%).

Step 2: An oven-dried 100-mL flask was charged with K_2CO_3 (4.14 g, 30 mmol), 1-(4-((trimethylsilyl)ethynyl)phenyl)ethan-1-one (2.15 g, 9.9 mmol) and methanol (60 ml) under argon. The reaction mixture was stirred at room temperature until the reaction was complete (monitored by GCMS). The solvent was removed under reduced pressure and the residue was dissolved with water (50 ml). The aqueous layer was extracted with ether (3 x 50 ml) and the combined organic layers were dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (PE / EA = 10:1) to afford the corresponding product **1h** (white solid, 1.22 g, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 7.6 Hz, 2H), 7.56 (d, J = 8.0 Hz, 2H), 3.24 (s, 1H), 2.59 (s, 3H).

3.2 Synthesis of 4fa and 4fb.



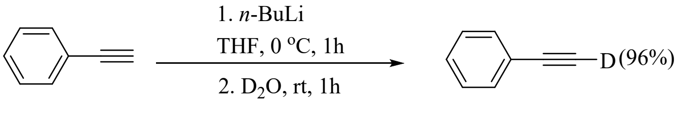
(*E*)-but-1-en-3-yne-1,4-diyldibenzene (4fa). An oven-dried 100-mL flask charged with Pd(PPh$_3$)$_4$ (1.156 g, 5 mol%), CuI (0.19 g, 5 mol%) was evacuated and backfilled with argon, with the operation being repeated twice. A mixture of (E)-(2-bromovinyl)benzene (3.77 g, 20 mmol) and ethynylbenzene (2.09 g, 21 mmol) in triethylamine (50 ml) was slowly added via syringe. The reaction mixture was performed at 70 °C for 12 hours until the reaction was complete (monitored by GC-MS). The solvent was removed under reduced pressure and the residue was filtered through a silica-gel pad using ethyl acetate as eluent. The ethyl acetate was evaporated under reduced pressure and the residue was purified by silica-gel column chromatography (PE/DCM = 10 : 1) to give the desired product **4fa** (white solid, 3.42 g, 84%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.53–7.49 (m, 2H), 7.46–7.44 (m, 2H), 7.39–7.30 (m, 6H), 7.08 (d, *J* = 16.2 Hz, 1H), 6.42 (d, *J* = 16.2 Hz, 1H).



4fb

But-3-en-1-yne-1,3-diyldibenzene (4fb). The procedure to synthesize products **4fb** was the same as the procedure to prepare compound **4fa** except that (1-bromovinyl)benzene (3.77 g, 20 mmol) was used instead of (E)-(2-bromovinyl)benzene. The residue was purified by silica-gel column chromatography (PE / DCM = 10 : 1) to give the desired product **4fb** (yellow liquid, 3.21 g, 79%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.76–7.72 (m, 2H), 7.57–7.53 (m, 2H), 7.42–7.32 (m, 6H), 6.00 (d, *J* = 0.9 Hz, 1H), 5.78 (d, *J* = 0.8 Hz, 1H).

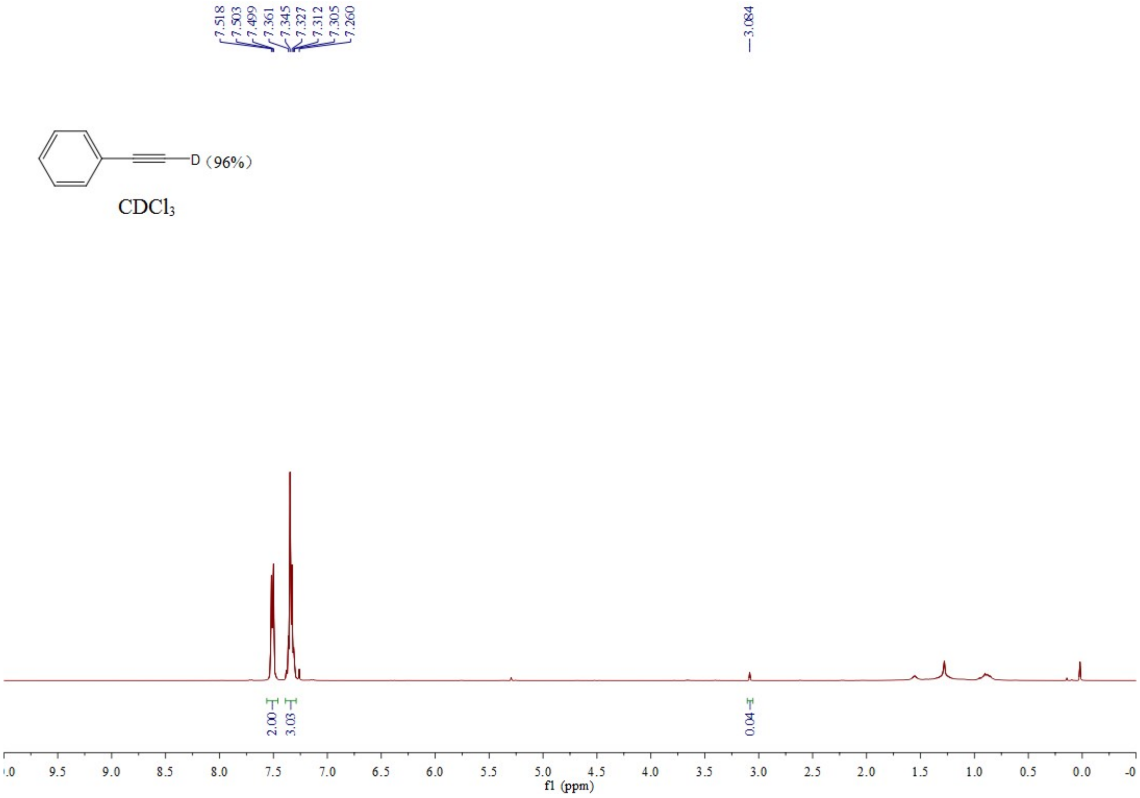
3.3 Synthesis of d-phenylacetylene 1f-[D].

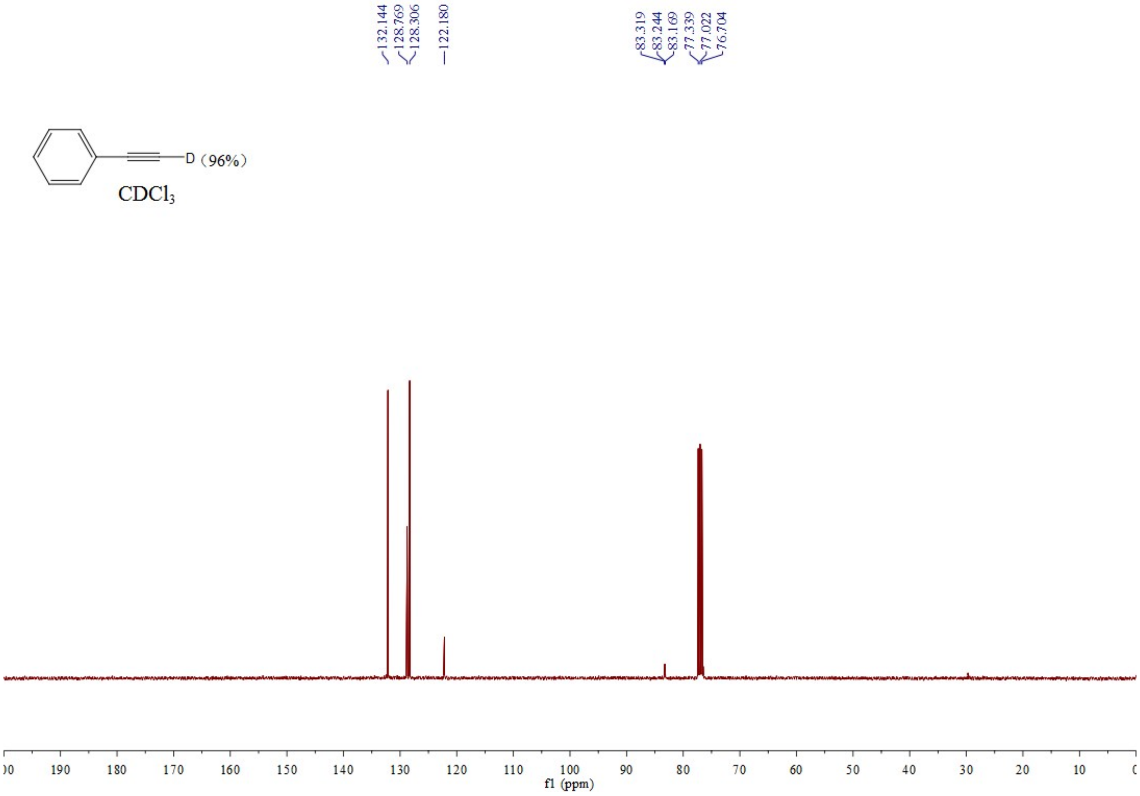


1f **1f-[D]**

An oven-dried 100-mL flask charged with a solution of phenylacetylene **1f** (1.53 g, 15 mmol) in THF (30 ml) under argon atmosphere. *n*-BuLi 1.6 M in hexane (10.3 ml, 16.5 mmol) was slowly added via syringe and the reaction mixture was stirred at 0 ^{o}C for 1 hour until total consumption of starting material. The resulting mixture was quenched with D_2O (4 ml, 100%-D) and stirred at room temperature for 1 hour until the reaction was complete (monitored by GC-MS). The mixture was extracted with diethyl ether (2 x 30 ml), and the combined organic layers was dried over anhydrous Na_2SO_4 and evaporated to dryness to give a pale yellow liquid d-phenylacetylene **1f-[D]** (1.31 g, 85% yield, 96% D). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54–7.48 (m, 2H), 7.39–7.29 (m, 3H), 3.08 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.14, 128.77, 128.31, 122.18, 83.32, 83.24, 83.17. MS (EI, m/z, rel.%): 103 (M^+, 100%), 77 (M^+–26, 66%).

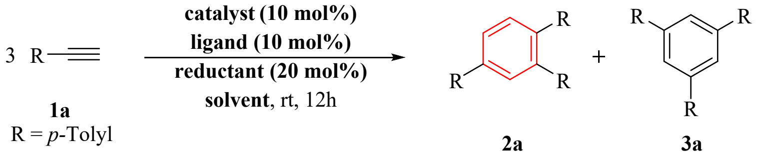
^{1}H NMR and ^{13}C NMR spectra of 1f-[D]



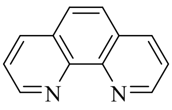


4. Optimization of Reaction Conditions

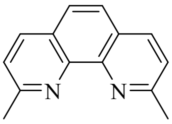
Table S1. Optimization of reaction conditions for the synthesis of **2a**[a]



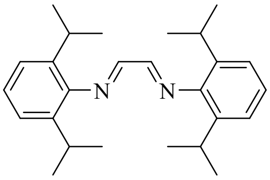
Entry	Catalyst	Ligand	Reductant	Solvent	(2a+3a) Yield [%][b]	(2a : 3a)
1	$NiCl_2$	–	Zn	CH_3CN	trace	–
2	**C1**	–	Zn	CH_3CN	34	27 : 73
3	$Ni(PPh_3)_2Cl_2$	–	Zn	CH_3CN	56	69 : 31
4	$Ni(dppm)Cl_2$	–	Zn	CH_3CN	40	43 : 57
5	$Ni(dppe)Cl_2$	–	Zn	CH_3CN	46	46 : 54
6	$Ni(dppp)Cl_2$	–	Zn	CH_3CN	49	49 : 51
7	$Ni(dppb)Cl_2$	–	Zn	CH_3CN	41	71 : 29
8	$Ni(dppf)Cl_2$	–	Zn	CH_3CN	trace	–
9	$Ni(PPh_3)_2Cl_2$	–	Zn	dioxane	26	47 : 53
10	$Ni(PPh_3)_2Cl_2$	–	Zn	toluene	trace	–
11	$Ni(PPh_3)_2Cl_2$	–	Zn	THF	41	44 : 56
12	$Ni(PPh_3)_2Cl_2$	–	Zn	DMF	29	52 : 48
13	$Ni(PPh_3)_2Cl_2$	–	Mg	CH_3CN	trace	–
14	$Ni(PPh_3)_2Cl_2$	–	Fe	CH_3CN	trace	–
15	$Ni(PPh_3)_2Cl_2$	–	NaH	CH_3CN	trace	–
16	$Ni(PPh_3)_2Cl_2$	–	$NaBH_4$	CH_3CN	70	28 : 72
17	$Ni(PPh_3)_2Cl_2$	–	$LiAlH_4$	CH_3CN	73	81 : 19
18	**$Ni(PPh_3)_2Cl_2$**	**L6**	**Zn**	**CH_3CN**	**94(91)**	**97 : 3**
19	$Ni(PPh_3)_2Cl_2$	**L10**	Zn	CH_3CN	77	87 : 13
20	$Ni(PPh_3)_2Cl_2$	**L11**	Zn	CH_3CN	78	87 : 13
21	$Ni(PPh_3)_2Cl_2$	**L12**	Zn	CH_3CN	74	85 : 15



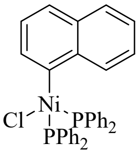
L10



L11



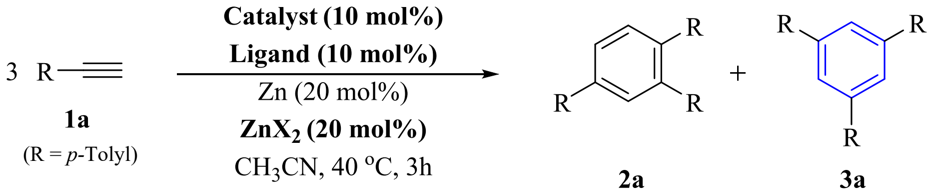
L12



C1

[a] Conditions: All reactions were run with 10 mol% catalyst, 10 mol% ligand and 4 mL solvent on an 1 mmol scale at room temperature for 12 hours in the same as the procedure 2.1 (Condition A). [b] Total yields and the regioisomer ratio of **2a:3a** were determined by GC-MS analysis using dodecane as an internal standard, and isolated yields in parenthesis.

Table S2. Optimization of conditions for the selective synthesis of **3a**[a]


3 R—≡

1a

(R = *p*-Tolyl)

Catalyst (10 mol%)

Ligand (10 mol%)

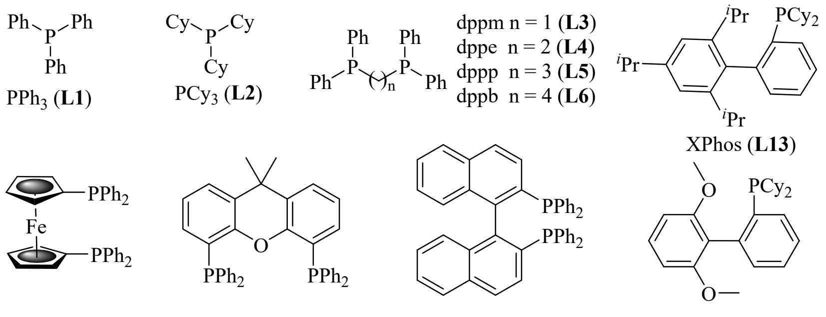
Zn (20 mol%)

ZnX_2 (20 mol%)

CH_3CN, 40 °C, 3h

2a + **3a**

Entry	Catalyst	Ligand	Additive	(**2a** + **3a**) Yield [%][b]	**2a : 3a**
1	$Ni(PPh_3)_2Cl_2$	-	$ZnCl_2$	78	14 : 86
2	$Ni(PPh_3)_2Cl_2$	-	$ZnBr_2$	76	14 : 86
3	$Ni(PPh_3)_2Cl_2$	-	ZnI_2	80	13 : 87
4	$Ni(PPh_3)_2Cl_2$	-	$ZnSO_4$	66	75 : 25
5	$Ni(PPh_3)_2Cl_2$	-	MgI_2	80	75 : 25
6	$Ni(dppm)Cl_2$	-	ZnI_2	67	40 : 60
7	$Ni(dppe)Cl_2$	-	ZnI_2	49	34 : 66
8	$Ni(dppp)Cl_2$	-	ZnI_2	59	24 : 76
9	$Ni(dppb)Cl_2$	-	ZnI_2	60	28 : 72
10	$Ni(dppf)Cl_2$	-	ZnI_2	trace	-
11[c]	$Ni(PPh_3)_2Cl_2$	**L1**	ZnI_2	76	22 : 78
12[c]	$Ni(PPh_3)_2Cl_2$	**L2**	ZnI_2	65	43 : 57
13	**$Ni(PPh_3)_2Cl_2$**	**L3**	**ZnI_2**	**91(85)**	**6 : 94**
14	$Ni(PPh_3)_2Cl_2$	**L4**	ZnI_2	85	10 : 90
15	$Ni(PPh_3)_2Cl_2$	**L5**	ZnI_2	78	18 : 82
16	$Ni(PPh_3)_2Cl_2$	**L6**	ZnI_2	44	23 : 77
17	$Ni(PPh_3)_2Cl_2$	**L7**	ZnI_2	81	17 : 83
18	$Ni(PPh_3)_2Cl_2$	**L8**	ZnI_2	90	23 : 77
19	$Ni(PPh_3)_2Cl_2$	**L9**	ZnI_2	86	19 : 81
20	$Ni(PPh_3)_2Cl_2$	**L13**	ZnI_2	90	23 : 77
21	$Ni(PPh_3)_2Cl_2$	**L14**	ZnI_2	69	35 : 65
22[d]	$Ni(PPh_3)_2Cl_2$	**L3**	ZnI_2	80	2 : 98
23[e]	$Ni(PPh_3)_2Cl_2$	**L3**	ZnI_2	88	18 : 82
24[f]	$Ni(PPh_3)_2Cl_2$	**L3**	ZnI_2	82	17 : 83


PPh_3 (**L1**)

PCy_3 (**L2**)

dppm n = 1 (**L3**)

dppe n = 2 (**L4**)

dppp n = 3 (**L5**)

dppb n = 4 (**L6**)

XPhos (**L13**)

dppf (**L7**)

Xantphos (**L8**)

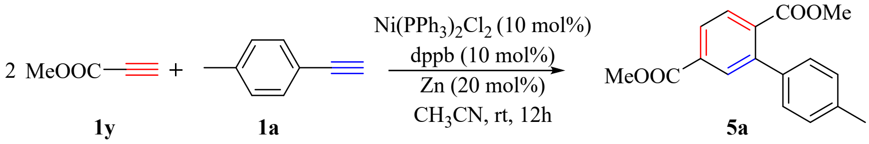
(±)-BINAP (**L9**)

SPhos (**L14**)

[a] Conditions: All reactions were run with 10 mol% catalyst, 10 mol% ligand, 20 mol% addtitive

and 2 mL CH_3CN on an 1 mmol scale at 40 °C for 3 hours as described in the procedure 2.2 (Condition B). [b] Total yields and the ratio of **2a:3a** were determined by GC-MS analysis using dodecane as an internal standard, and isolated yields in parenthesis. [c] Ligand (20 mol%). [d] 25 °C. [e] 55 °C. [f] 70 °C.

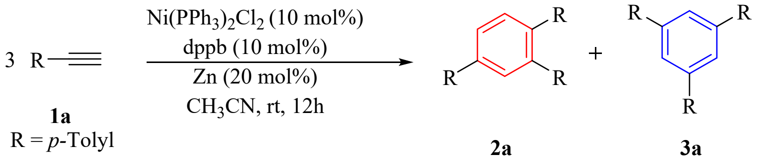
Table S3. Optimizing the ratio of starting materials for synthesis of **5a**[a]



Entry	Ratio of **1y:1a**	Yield [%][b]
1	0.5 : 1	22
2	1 : 1	36
3	2 : 1	45
4	***3 : 1***	***56***
5	4 : 1	53
6	5 : 1	38

[a] Conditions: All reactions were run with $Ni(PPh_3)_2Cl_2$ (0.1 mmol, 65.7 mg), dppb (0.1 mmol, 43 mg), Zn (0.2 mmol, 13 mg), CH_3CN (4 mL), and a mixture of **1y** and **1a** at room temperature for 12 hours under the Condition A. [b] Isolated yields.

Table S4. Optimizing activation time at 80 °C for Condition A[a]



Entry	**Activation time [min]**	**(2a+3a) Yield [%][b]**	**2a : 3a**
1	0	48	79 : 21
2	2	78	90 : 10
3	3	85	90 : 10
4	5	90	91 : 9
5	***7***	***94***	***97 : 3***
6	10	81	94 : 6
7	15	63	84 : 16

[a] Conditions: All reactions were run with 10 mol% $Ni(PPh_3)_2Cl_2$, 10 mol% dppb, 20 mol% Zn and 4 mL CH_3CN on an 1 mmol scale at room temperature for 12 hours as described in the procedure 2.1 (Condition A). [b] Total yields and the ratio of **2a:3a** were determined by GC-MS analysis using dodecane as an internal standard.

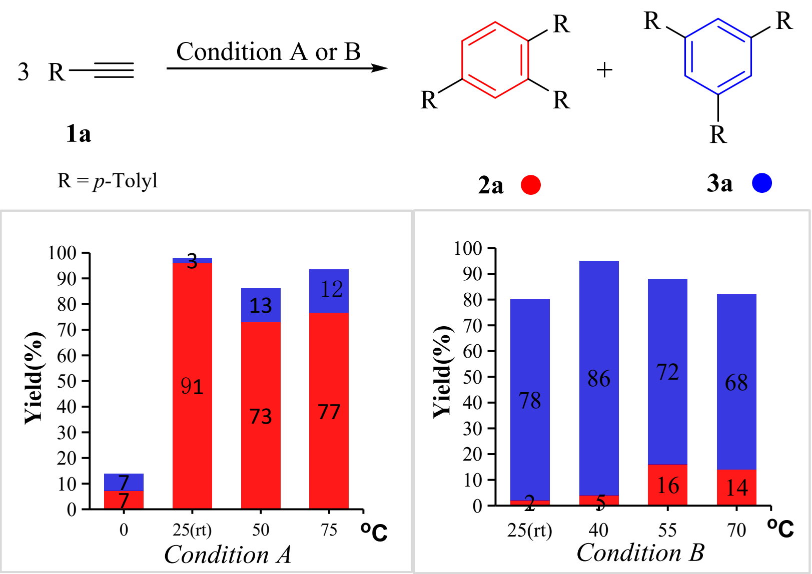
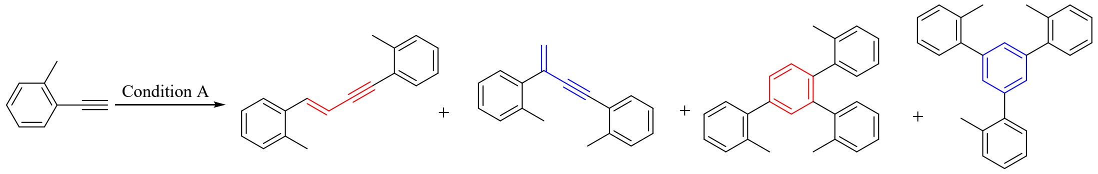


Figure S1. The effect of reaction temperatures on the catalytic efficiency and selectivity. All reactions were run under standard Condition A or Condition B. The total heights of the bars were the total yields of **2a** and **3a**. The product fraction corresponding to 1,2,4-isomer (**2a**, red block) and 1,3,5-isomer (**3a**, blue block) were plotted.

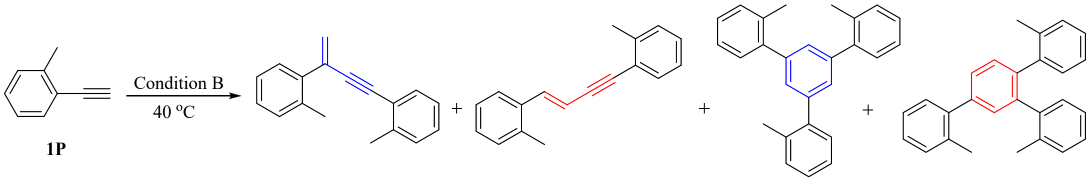
5. Control Experiments

5.1 The formation of head-to-head and head-to-tail dimers using the sterically hindered substrate



1p **4pa** (43%) **4pb** (16%) **2p** (20%) **3p** (12%)

The hindered substrate **1p** (1 mmol) was subjected to standard Condition A (procedure 2.1). After 12 hours, the mixture was analyzed by GC-MS and dodecane was used as the internal standard. **4pa** as the main product was isolated in 43% yield.

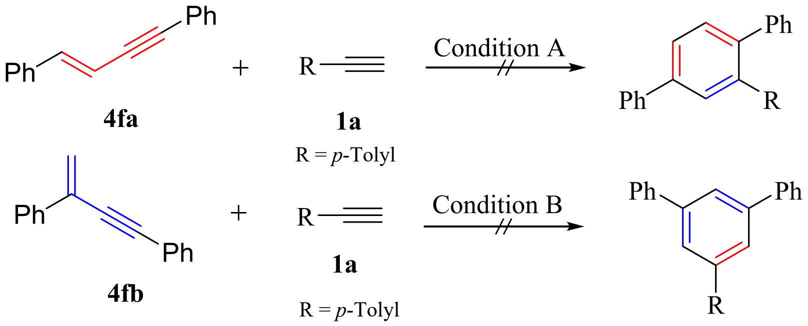


1P

4pa (14%) **3p** (22%) **2p** (14%)

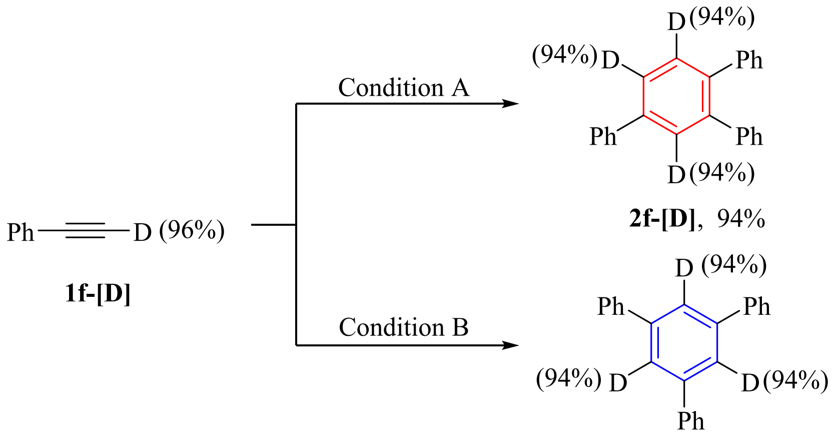
The hindered substrate **1p** (1 mmol) was subjected to standard Condition B (procedure 2.2) at 40 °C for 3 hours. The product fraction of dimers and trimers were determined by GC-MS analysis using dodecane as an internal standard. **4pb** as the main product was isolated in 38% yield.

5.2 Knowing about the intermediate property of dimers in the reaction



The **4fa** (0.3 mmol) and **4fb** (0.3 mmol) without steric hindrance were subjected to standard Conditions A and B in the presence of the third alkyne **1a** (0.3 mmol), respectively, As a result, the corresponding heterotrisubstitued benzenes were not observed.

5.3 Deuterium labeling experiments



1f-[D]

2f-[D], 94%

3f-[D], 96%

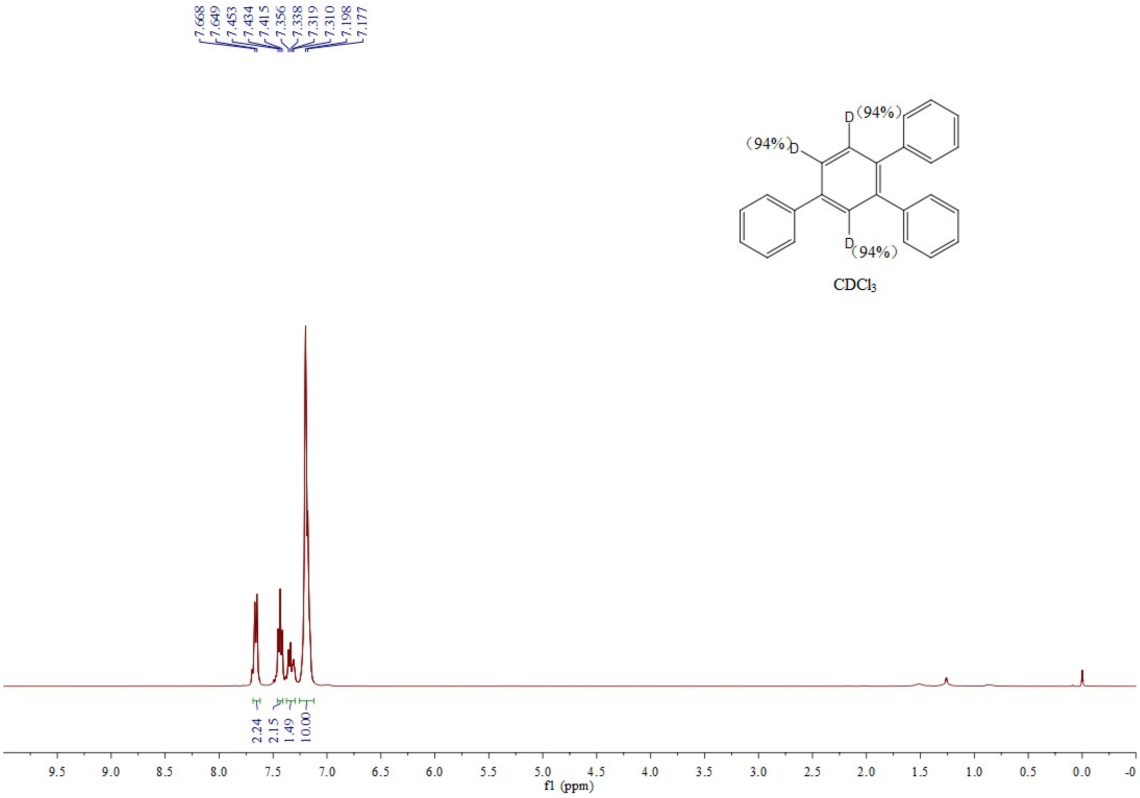
The **1f-[D]** (1 mmol) was subjected respectively to standard Conditions A and B until the reaction was complete (monitored by GC-MS). The resulting mixture was filtered through a pad of silica using ethyl acetate as eluent and concentrated under reduced pressure. The residue was purified by silica-gel column chromatography (PE/DCM = 10:1) to afford the desired product **2f-[D]** and **3f-[D]**, respectively.

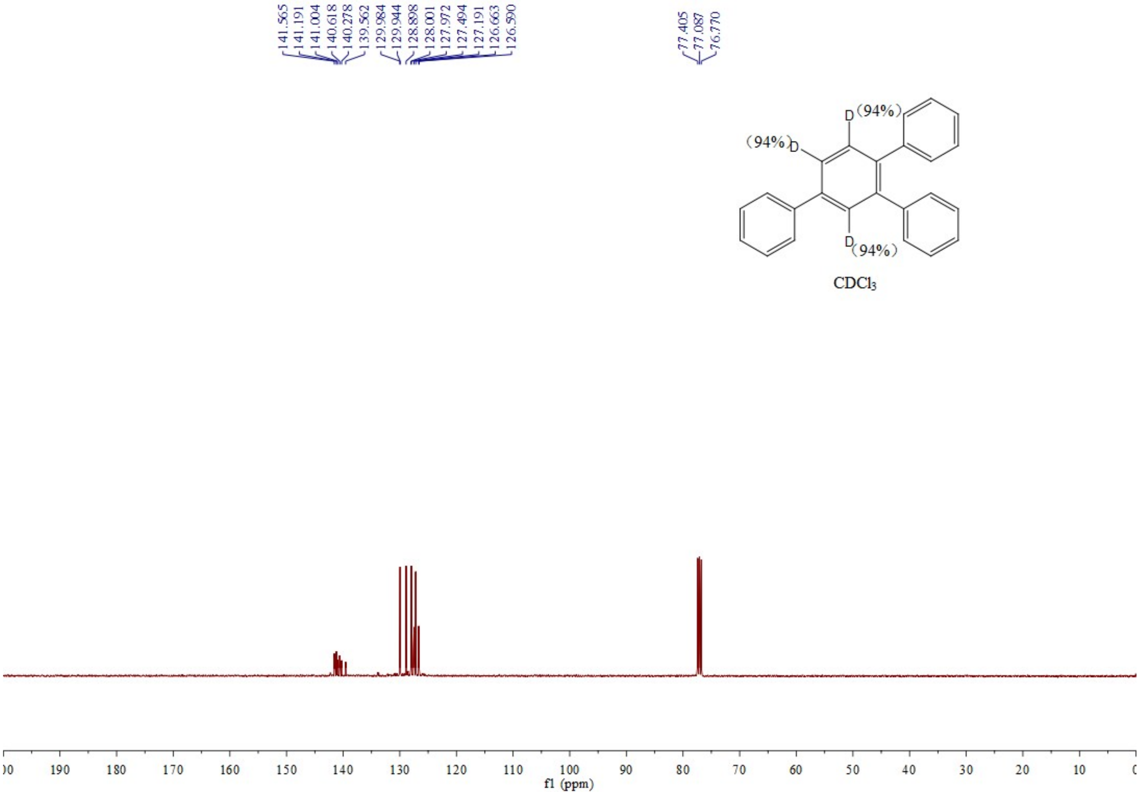
1,2,4-Triphenyl-3,5,6-trideuterobenzene (**2f-[D]**). Yield: 94%. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, J = 7.7 Hz, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.36–7.31 (m, 1H), 7.25–7.13 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.56, 141.19, 141.00, 140.62, 140.28, 139.56, 129.98, 129.94, 128.90, 128.00, 127.97, 127.49, 127.19, 126.66, 126.59; MS (EI, m/z, rel.%): 309 (M^+, 100%), 292 (M^+–17, 18%).

1,3,5-Triphenyl-2,4,6-trideuterobenzene (**3f-[D]**). Yield: 96%. White solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.72 (d, J = 7.1 Hz, 6H), 7.50 (t, J = 7.5 Hz, 6H), 7.43–7.39 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.22, 141.14, 128.87, 127.56, 127.37; MS (EI, m/z, rel.%): 309 (M^+, 100%), 291 (M^+–18, 11%).

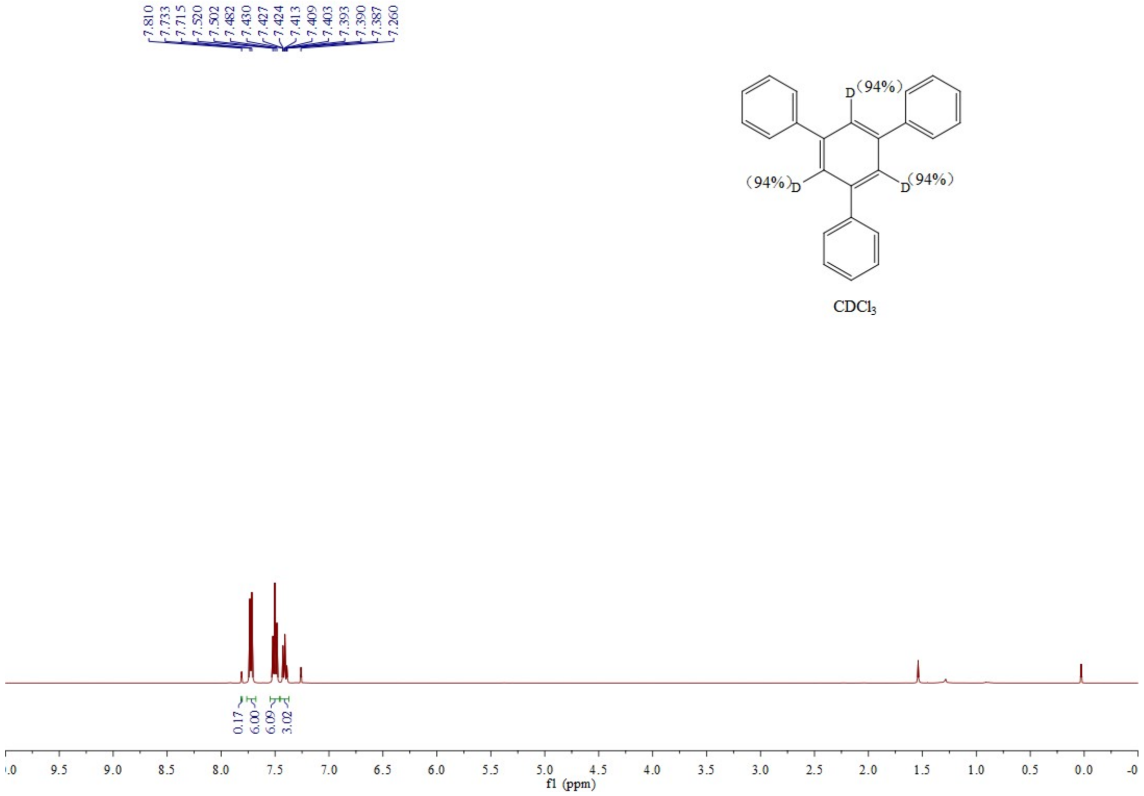
^{1}H and ^{13}C NMR spectra of 2f-[D] and 3f-[D]

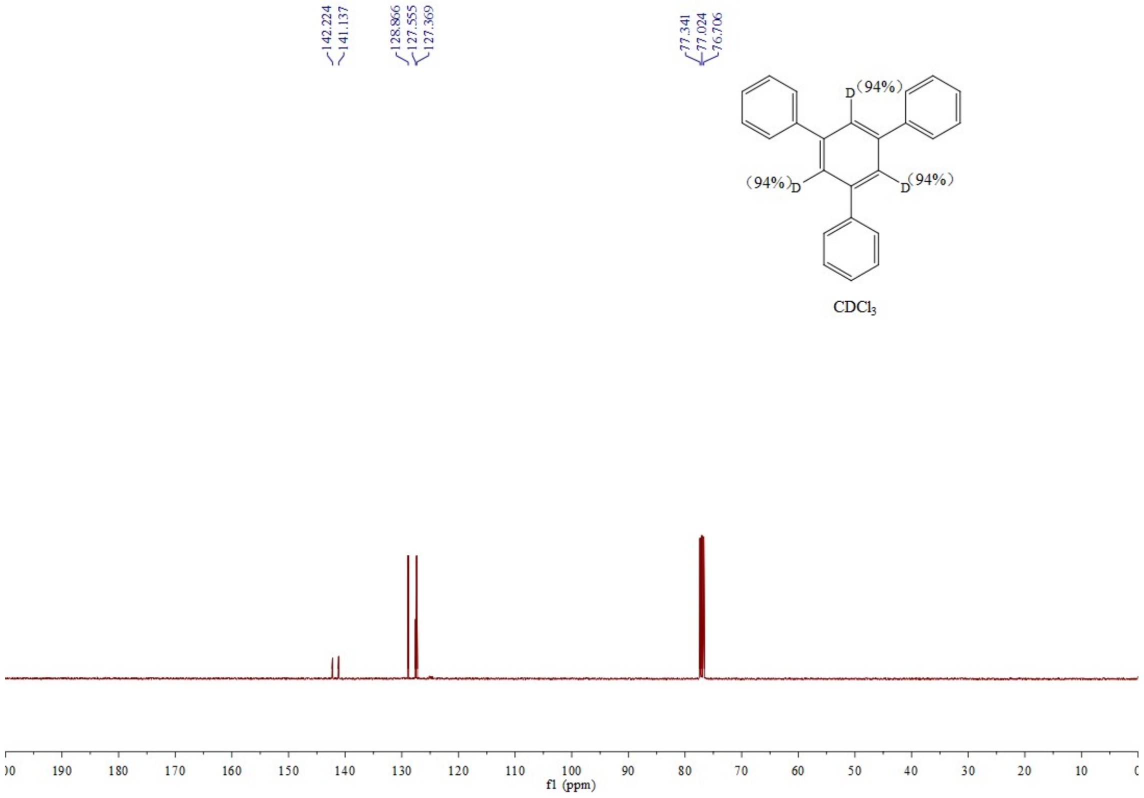
1,2,4-Triphenyl-3,5,6-trideuterobenzene (**2f-[D]**)





1,3,5-Triphenyl-2,4,6-trideuterobenzene (**3f-[D]**)





6. Characterization Data of the Products

1,2,4-Tris(4-tolyl)benzene (2a, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 91% yield, mp 121–123 °C (lit.[30] 122–123 °C). The regioisomer ratio of **2a:3a** was 97:3. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59–7.52 (m, 4H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.24–7.21 (m, 2H), 7.08–7.00 (m, 8H), 2.37 (s, 3H), 2.293 (s, 3H), 2.290 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.83, 140.07, 139.19, 138.80, 138.41, 137.85, 137.15, 136.13, 136.04, 131.12, 129.76, 129.72, 129.55, 129.28, 128.70, 128.67, 126.97, 125.73, 21.15; MS (EI, m/z, rel.%): 348 (M^+, 100%), 333 (M^+–15, 19%), 318 (M^+–30, 19%), 303 (M^+–45, 11%), 151 (M^+–197, 12%).

1,2,4-Tris(4-methoxyphenyl)benzene (2b, Table 2).[1] Isolated by column chromatography (*n*-pentane : EA = 10 : 1) to afford a white solid in 98% yield, mp 115–116 °C (lit.[7] 116–117 °C). The regioisomer ratio of **2b:3b** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64–7.58 (m, 4H), 7.46 (d, *J* = 7.9 Hz, 1H), 7.14 (dd, *J* = 11.2, 8.6 Hz, 4H), 7.02 (d, *J* = 8.5 Hz, 2H), 6.81 (dd, *J* = 8.3, 4.4 Hz, 4H), 3.87 (s, 3H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.28, 158.39, 158.32, 140.45, 139.64, 138.52, 134.21, 133.80, 133.28, 131.04, 130.97, 130.92, 128.95, 128.13, 125.40, 114.31, 113.49, 113.47, 55.37, 55.21, 55.20; MS (EI, m/z, rel.%): 396 (M^+, 100%), 381 (M^+–15, 11%), 281 (M^+–115, 10%), 207 (M^+–189, 13%).

1,2,4-Tris[4-(phenyl)phenyl]benzene (2c, Table 2).[2] Isolated by column chromatography (PE : DCM = 5 : 1) to afford a yellow solid in 93% yield, mp 263–264 °C (lit.[2] 170–172 °C). The regioisomer ratio of **2c:3c** was 94:6. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, *J* = 7.8 Hz, 3H), 7.73 (t, *J* = 9.0 Hz, 3H), 7.68 (d, *J* = 7.4 Hz, 2H), 7.63–7.61 (m, 5H), 7.54–7.38 (m, 11H), 7.33 (dd, *J* = 10.8, 8.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.68, 140.65, 140.64, 140.49, 140.37, 140.10, 139.99, 139.46, 139.40, 139.29, 139.19, 131.28, 130.37, 130.32, 129.39, 128.86, 128.77, 127.62, 127.51, 127.42, 127.29, 127.09, 127.00, 126.99, 126.74, 126.70, 126.16; MS (EI, m/z, rel.%): 534 (M^+, 100%), 443 (M^+–91, 26%).

1,2,4-Tris(4-aminophenyl)benzene (2d, Table 2).[3] Isolated by column chromatography ($CHCl_3$: MeOH = 10 : 1) to afford a yellow solid in 82% yield, mp

183–184 °C. The regioisomer ratio of **2d:3d** was 94:6. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, *J* = 1.9 Hz, 1H), 7.52–7.50 (m, 2H), 7.47 (s, 1H), 7.40 (d, *J* = 7.9 Hz, 1H), 7.01 (dd, *J* = 10.6, 8.4 Hz, 4H), 6.76 (d, *J* = 8.5 Hz, 2H), 6.58 (dd, *J* = 8.4, 3.8 Hz, 4H), 3.66 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.81, 144.81, 144.71, 140.56, 139.61, 138.37, 132.43, 132.00, 131.24, 130.84, 130.79, 128.51, 127.97, 124.77, 115.43, 114.76; MS (ESI, m/z, rel.%): 352.3 ($[M+H]^+$, 100%).

1,2,4-Tris(4-dimethylaminophenyl)benzene (2e, Table 2).[4] Isolated by column chromatography (PE : EA = 7 : 1) to afford a yellow solid in 78% yield, mp 200–201 °C. The regioisomer ratio of **2e:3e** was 95:5. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 8.5 Hz, 3H), 7.54 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.43 (d, *J* = 7.9 Hz, 1H), 7.13 (dd, *J* = 12.4, 8.7 Hz, 4H), 6.83 (d, *J* = 8.7 Hz, 2H), 6.66 (dd, *J* = 8.4, 5.3 Hz, 4H), 3.01 (s, 6H), 2.950 (s, 6H), 2.946 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.90, 149.04, 148.93, 140.51, 139.49, 138.12, 130.92, 130.61, 130.55, 130.52, 130.06, 129.15, 128.53, 127.66, 124.53, 112.83, 112.18, 40.64, 40.61; MS (EI, m/z, rel.%): 435 (M^+, 100%), 420 (M^+–15, 6%), 405 (M^+–30, 10%), 210 (M^+–225, 7%).

1,2,4-Triphenylbenzene (2f, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 96% yield, mp 98–99 °C (lit.[30] 93–94 °C). The regioisomer ratio of **2f:3f** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68–7.63 (m, 4H), 7.50 (d, *J* = 7.9 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.24–7.16 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.56, 141.19, 141.06, 140.66, 140.41, 139.62, 131.12, 129.95, 129.91, 129.45, 128.86, 127.96, 127.93, 127.46, 127.18, 126.63, 126.55, 126.15; MS (EI, m/z, rel.%): 306 (M^+, 100%), 289 (M^+–17, 24%), 228 (M^+–78, 15%), 215 (M^+–91, 13%).

1,2,4-Tris(4-methoxycarbonylphenyl)benzene (2g, Table 2).[5] Isolated by column chromatography (PE : EA = 4 : 1) to afford a white solid in 92% yield, mp 224–225 °C (lit.[5] 224–225 °C). The regioisomer ratio of **2g:3g** was 99:1. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 8.07 (d, *J* = 8.2 Hz, 2H), 7.83 (dd, *J* = 7.9, 5.8 Hz, 4H), 7.73–7.68 (m, 4H), 7.51 (d, *J* = 7.9 Hz, 1H), 7.20 (dd, *J* = 11.0, 8.3 Hz, 4H), 3.86 (s, 3H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 166.62, 166.61, 145.61, 145.27, 144.40, 140.32, 139.79, 139.44, 131.17, 130.08, 129.94, 129.87, 129.51, 129.31, 129.24, 129.21, 128.85, 128.78, 127.01, 126.88, 52.00, 51.93; MS (EI, m/z, rel.%): 480 (M^+, 100%), 449 (M^+−31, 30%), 302 (M^+−178, 21%), 209 (M^+−271, 8%).

1,2,4-Tris(4-acetylphenyl)benzene (2h, Table 2).[1] Isolated by column chromatography (*n*-pentane : EA = 2 : 1) to afford a yellow solid in 91% yield, mp 258–259 °C (lit.[5] 258–259 °C). The regioisomer ratio of **2h:3h** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.76 (dd, *J* = 8.2, 5.6 Hz, 4H), 7.69–7.62 (m, 4H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.22–7.17 (m, 4H), 2.56 (s, 3H), 2.50 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.64, 197.57, 145.71, 145.37, 144.56, 140.22, 139.93, 139.36, 136.32, 135.71, 135.64, 131.29, 130.05, 129.99, 129.43, 129.07, 128.31, 128.26, 127.25, 127.05, 26.68, 26.60, 26.59; MS (EI, m/z, rel.%): 432 (M^+, 54%), 417 (M^+−15, 89%).

1,2,4-Tris(4-formylphenyl)benzene (2i, Table 2).[5] Isolated by column chromatography (PE : EA = 2 : 1) to afford a yellow solid in 75% yield, mp 216–217 °C (lit.[5] 216–217 °C). The regioisomer ratio of **2i:3i** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.06 (s, 1H), 9.98 (s, 1H), 9.97 (s, 1H), 7.99 (d, *J* = 8.3 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 2H), 7.79–7.73 (m, 6H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.34 (dd, *J* = 11.3, 8.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.71, 146.91, 146.60, 146.58, 145.82, 140.18, 140.04, 139.44, 135.69, 135.07, 131.36, 130.51, 130.44, 129.65, 129.61, 129.54, 127.97, 127.74, 127.36; MS (EI, m/z, rel.%): 390 (M^+, 100%), 362 (M^+−28, 15%).

1,2,4-Tris(4-nitrophenyl)benzene (2j, Table 2).[6] Isolated by column chromatography (PE : EA = 2 : 1) to afford a yellow solid in 48% yield, mp 260–262 °C. The regioisomer ratio of **2j:3j** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37–8.35 (m, 2H), 8.17–8.14 (m, 4H), 7.84–7.80 (m, 3H), 7.71 (d, *J* = 1.8 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.37–7.32 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.68, 147.19, 147.14, 146.79, 146.51, 145.90, 139.68, 139.40, 138.86, 131.56, 130.62, 130.55, 129.60, 127.95, 127.88, 124.37, 123.76, 123.71; HRMS (EI) calcd for $C_{24}H_{15}N_3O_6$ 441.0955, found 441.0952.

1,2,4-Tris(4-fluorophenyl)benzene (2k, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 92% yield, mp 140–141 °C (lit.[7] 138–140 °C). The regioisomer ratio of **2k:3k** was 97:3. ^{1}H NMR (400

MHz, $CDCl_3$): δ 7.64–7.57 (m, 4H), 7.47 (d, *J* = 7.9 Hz, 1H), 7.18–7.10 (m, 6H), 6.95 (td, *J* = 8.7, 3.4 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.89, 163.13, 163.09, 161.44, 160.68, 160.64, 140.06, 139.66, 138.54, 137.17, 137.14, 136.81, 136.77, 136.52, 136.49, 131.41, 131.37, 131.33, 131.29, 131.09, 129.19, 128.72, 128.64, 126.18, 115.89, 115.68, 115.17, 115.14, 114.96, 114.93; ^{19}F NMR (500 MHz, $CDCl_3$): δ -115.17, -115.64, -115.77; MS (EI, m/z, rel.%): 360 (M^+, 100%), 338 (M^+–22, 15%), 264 (M^+–96, 7%), 159 (M^+–201, 7%).

1,2,4-Tris(4-chlorophenyl)benzene (2l, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 98% yield, mp 157–158 °C (lit.[2] 159–161 °C). The regioisomer ratio of **2l:3l** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62–7.56 (m, 4H), 7.47–7.41 (m, 3H), 7.25–7.21 (m, 4H), 7.11–7.06 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.87, 139.64, 139.43, 139.08, 138.68, 138.56, 133.84, 133.10, 133.01, 131.18, 131.10, 131.06, 129.14, 129.10, 128.44, 128.40, 128.35, 126.39; MS (EI, m/z, rel.%): 410 ($[M+2]^+$, 100%), 408 (M^+, 100%), 372 (M^+–36, 14%), 338 (M^+–70, 79%), 302 (M^+–106, 45%), 169 (M^+–239, 25%), 151 (M^+–257, 52%).

1,2,4-Tris(4-bromophenyl)benzene (2m, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 91% yield, mp 161–162 °C (lit.[2] 158–160 °C). The regioisomer ratio of **2m:3m** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63–7.56 (m, 4H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.41–7.38 (m, 4H), 7.03 (t, *J* = 8.7 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.86, 139.84, 139.70, 139.52, 139.13, 138.55, 132.06, 131.43, 131.40, 131.38, 131.37, 131.16, 129.06, 128.68, 126.39, 122.02, 121.32, 121.24; MS (EI, m/z, rel.%): 545 ($[M+6]^+$, 31%), 543 ($[M+4]^+$, 95%), 541 ($[M+2]^+$, 100%), 539 (M^+, 33%), 382 (M^+–157, 49%), 302 (M^+–237, 34%).

1,2,4-Tris(3-methylphenyl)benzene (2n, Table 2).[1] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a yellow oil in 97% yield. The regioisomer ratio of **2n:3n** was 91:9. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.73–7.69 (m, 2H), 7.59 (s, 1H), 7.56–7.53 (m, 2H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 7.5 Hz, 1H), 7.19–7.09 (m, 6H), 7.01 (t, *J* = 8.3 Hz, 2H), 2.49 (s, 3H), 2.35 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 141.70, 141.30, 141.20, 140.56, 140.24, 139.72, 138.61, 137.64, 137.59, 131.06, 130.63, 130.57, 129.28, 128.79, 128.24, 127.84, 127.63, 127.62, 127.30, 127.24, 127.14, 127.08, 125.91, 124.16, 21.34, 21.18; MS (EI, m/z, rel.%): 348 (M^+, 100%), 333 (M^+–15, 27%), 348 (M^+–30, 25%), 303 (M^+–45, 15%), 239 (M^+–109, 7%), 151 (M^+–197, 11%).

1,2,4-Tris(3-fluorophenyl)benzene (2o, Table 2).[1] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a yellow oil in 99% yield. The regioisomer ratio of **2o:3o** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62–7.59 (m, 2H), 7.47–7.45 (m, 1H), 7.43–7.32 (m, 3H), 7.21–7.14 (m, 2H), 7.07–7.02 (m, 1H), 6.94–6.86 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.55, 163.81, 163.80, 162.11, 161.364, 161.355, 143.24, 143.17, 142.92, 142.84, 142.56, 142.49, 140.00, 139.98, 139.70, 139.67, 138.92, 138.90, 131.17, 130.48, 130.40, 129.68, 129.63, 129.60, 129.54, 129.26, 126.60, 125.64, 125.61, 125.60, 125.57, 122.80, 122.77, 116.83, 116.76, 116.61, 116.54, 114.64, 114.43, 114.17, 114.06, 113.98, 113.95, 113.85, 113.77; ^{19}F NMR (500 MHz, $CDCl_3$): δ -112.59, -113.613, -113.21; MS (EI, m/z, rel.%): 360 (M^+, 100%), 340 (M^+–20, 18%), 318 (M^+–42, 5%), 264 (M^+–96, 9%), 159 (M^+–201, 12%).

1,2,4-Tris(2-fluorophenyl)benzene (2q, Table 2).[7] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a white solid in 92% yield, mp 101–102 °C. The regioisomer ratio of **2q:3q** was 97:3. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, J = 9.6 Hz, 2H), 7.54–7.47 (m, 2H), 7.33–7.25 (m, 1H), 7.22–7.10 (m, 6H), 6.98 (t, J = 7.5 Hz, 2H), 6.91 (td, J = 9.0, 4.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.19, 160.85, 160.82, 158.73, 158.40, 158.37, 135.91, 135.512, 135.506, 135.10, 131.93, 131.92, 131.88, 131.84, 131.37, 131.34, 130.89, 130.88, 130.84, 129.36, 129.28, 129.17, 129.15, 129.09, 129.07, 128.79, 128.62, 128.61, 128.54, 128.51, 128.43, 128.30, 124.54, 124.50, 123.67, 123.66, 116.36, 116.14, 115.54, 115.32; ^{19}F NMR (500 MHz, $CDCl_3$): δ -115.17, -115.64, -115.77; MS (EI, m/z, rel.%): 360 (M^+, 100%), 340 (M^+–20, 94%), 318 (M^+–42, 33%), 264 (M^+–96, 20%), 159 (M^+–201, 56%).

1,2,4-Tris(2-thienyl)benzene (2r, Table 2).[26] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a white solid in 96% yield, mp 80–81 °C. The regioisomer ratio of **2r:3r** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 1.7 Hz, 1H), 7.62 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.38 (d, *J* = 3.5 Hz, 1H), 7.32 (d, *J* = 5.0 Hz, 2H), 7.27 (dd, *J* = 5.9, 5.1 Hz, 1H), 7.11 (dd, *J* = 5.0, 3.7 Hz, 1H), 7.01 (dd, *J* = 4.9, 3.7 Hz, 1H), 6.99–6.96 (m, 2H), 6.92–6.91 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.29, 142.27, 142.25, 134.17, 133.96, 132.98, 131.41, 128.53, 128.20, 127.42, 127.09, 127.03, 126.24, 126.12, 125.38, 123.67; MS (EI, m/z, rel.%): 324 (M^+, 100%), 291 (M^+–33, 23%), 279 (M^+–45, 26%), 258 (M^+–66, 11%), 245 (M^+–79, 13%).

1,2,4-Tris(3-thienyl)benzene (2s, Table 2).[1] Isolated by column chromatography (PE : DCM = 5 : 1) to afford a white solid in 99% yield, mp 129–130 °C. The regioisomer ratio of **2s:3s** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 1.9 Hz, 1H), 7.60 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.52–7.49 (m, 2H), 7.44 (dd, *J* = 5.0, 1.4 Hz, 1H), 7.41 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.21 (ddd, *J* = 9.2, 5.0, 3.0 Hz, 2H), 7.14 (dd, *J* = 3.0, 1.3 Hz, 1H), 7.09 (dd, *J* = 3.0, 1.3 Hz, 1H), 6.83 (ddd, *J* = 10.8, 5.0, 1.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.04, 141.70, 141.65, 135.82, 135.07, 134.22, 130.64, 128.97, 128.86, 128.30, 126.37, 126.30, 125.52, 124.87, 124.73, 123.01, 122.87, 120.53; MS (EI, m/z, rel.%): 324 (M^+, 100%), 290 (M^+–34, 32%), 279 (M^+–45, 17%), 258 (M^+–66, 17%), 245 (M^+–79, 13%).

1,2,4-Tris(4-pyridyl)benzene (2t, Table 2). Isolated by column chromatography ($CHCl_3$: TEA = 10 : 1) to afford a yellow solid in 94% yield, mp 253–254 °C. The regioisomer ratio of **2t:3t** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (dd, *J* = 4.5, 1.6 Hz, 2H), 8.52 (td, *J* = 5.3, 1.6 Hz, 4H), 7.79 (dd, *J* = 8.0, 1.9 Hz, 1H), 7.69 (d, *J* = 1.8 Hz, 1H), 7.62–7.53 (m, 3H), 7.10 (ddd, *J* = 10.0, 4.5, 1.6 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.52, 149.87, 149.82, 148.02, 147.73, 146.93, 139.03, 138.77, 138.43, 131.35, 129.08, 127.48, 124.49, 124.41, 121.59; MS (EI, m/z, rel.%): 309 (M^+, 100%), 281 (M^+–43, 28%); HRMS (EI) calcd for $C_{21}H_{15}N_3$ 309.1260, found 316.1258.

1,2,4-Tris(1-cyclohexenyl)benzene (2w, Table 2).[7] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a colorless oil in 90% yield. The regioisomer ratio of **2w:3w** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.14 (d, *J* = 1.9 Hz, 1H), 7.05 (d, *J* = 7.9 Hz, 1H), 6.13–6.11 (m, 1H), 5.68–5.66 (m, 2H), 2.43–2.40 (m, 2H), 2.24–2.13 (m, 10H), 1.80 – 1.62 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.50, 141.04, 140.65, 139.83, 139.14, 136.36, 128.46, 125.80, 125.76, 125.32, 124.22, 122.85, 29.69, 29.62, 29.56, 27.40, 25.89, 25.77, 25.74, 23.32, 23.12, 22.24, 22.23, 22.22; MS (EI, m/z, rel.%): 318 (M^+, 100%), 275 (M^+–43, 100%), 261 (M^+–57, 38%), 233 (M^+–85, 16%), 195 (M^+–123, 17%), 165 (M^+–153, 16%), 81 (M^+–237, 16%).

(E)-1,4-bis(trimethylsilyl)but-3-en-1-yne (4xa, Table 2).[8] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a yellow oil in 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.51 (d, *J* = 19.3 Hz, 1H), 5.97 (d, *J* = 19.3 Hz, 1H), 0.19 (s, 9H), 0.08 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.08, 123.42, 105.40, 94.81, -0.00, -1.62; MS (EI, m/z, rel.%): 196 (M^+, 12%), 181 (M^+–15, 87%), 155 (M^+–41, 21%), 123 (M^+–73, 19%), 97 (M^+–99, 14%), 73 (M^+–123, 100%).

Benzene-1,2,4-tricarboxylic acid trimethyl ester (2y, Table 2).[4] Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow oil in 99% yield. The regioisomer ratio of **2y:3y** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32 (d, *J* = 1.5 Hz, 1H), 8.10 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 3.87 (s, 3H), 3.85 (s, 3H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.47, 166.70, 165.21, 136.16, 132.37, 132.18, 131.54, 130.15, 128.82, 52.81, 52.75, 52.52; MS (EI, m/z, rel.%): 252 (M^+, 4%), 221 (M^+–31, 100%).

1,2,4-Triacetylphenylbenzene (2z, Table 2).[9] Isolated by column chromatography (PE : EA = 2 : 1) to afford a white solid in 90% yield, mp 75–76 °C. The regioisomer ratio of **2z:3z** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.04 (d, *J* = 7.9 Hz, 1H), 7.52 (d, *J* = 7.9 Hz, 1H), 2.59 (s, 3H), 2.53 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.82, 200.18, 196.38, 143.98, 138.75, 138.39, 131.32, 127.73, 127.69, 29.16, 28.25, 26.71; MS (EI, m/z, rel.%): 204 (M^+, 1%), 189 (M^+–15, 100%), 161 (M^+–43, 3%).

1,3,5-Tris(4-tolyl)benzene (3a, Table 3).[10] Isolated by column chromatography (PE : EA = 10 : 1) to afford a white solid in 85% yield, mp 174–175 ^{o}C (lit.[16] 173–175 ^{o}C). The regioisomer ratio of **3a:2a** was 94:6. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (s, 3H), 7.67 (d, J = 7.8 Hz, 6H), 7.35 (d, J = 7.7 Hz, 6H), 2.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.19, 138.44, 137.27, 129.55, 127.20, 124.59, 21.14; MS (EI, m/z, rel.%): 348 (M^+, 100%), 333 (M^+–15, 2%), 318 (M^+–30, 3%), 303 (M^+–45, 3%), 151 (M^+–197, 3%).

1,3,5-Tris(4-methoxyphenyl)benzene (3b, Table 3).[10] Isolated by column chromatography (*n*-pentane : EA = 10 : 1) to afford a white solid in 97% yield, mp 140–141 ^{o}C (lit.[16] 136–139 ^{o}C). The regioisomer ratio of **3b:2b** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (s, 3H), 7.64 (d, J = 8.8 Hz, 6H), 7.02 (d, J = 8.7 Hz, 6H), 3.88 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.35, 141.87, 133.89, 128.38, 123.87, 114.29, 55.39; MS (EI, m/z, rel.%): 396 (M^+, 100%), 381 (M^+–15, 11%), 207 (M^+–189, 22%).

1,3,5-Tris[4-(phenyl)phenyl]benzene (3c, Table 3).[11] Isolated by column chromatography (PE : DCM = 5 : 1) to afford a yellow solid in 89% yield, mp 233–234 ^{o}C (lit.[11] 232–234 ^{o}C). The regioisomer ratio of **3c:2c** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (s, 3H), 7.82 (d, J = 8.3 Hz, 6H), 7.74 (d, J = 8.3 Hz, 6H), 7.68 (d, J = 7.3 Hz, 6H), 7.49 (t, J = 7.6 Hz, 6H), 7.39 (t, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.00, 140.67, 140.52, 140.03, 128.87, 127.75, 127.64, 127.45, 127.11, 125.03; MS (EI, m/z, rel.%): 534 (M^+, 100%), 267 (M^+–267, 15%).

1,3,5-Tris(4-aminophenyl)benzene (3d, Table 3).[3] Isolated by column chromatography ($CHCl_3$: MeOH = 10 : 1) to afford a yellow solid in 94% yield, mp 227–228 ^{o}C. The regioisomer ratio of **3d:2d** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (s, 3H), 7.51 (d, J = 8.5 Hz, 6H), 6.78 (d, J = 8.5 Hz, 6H), 3.74 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.96, 140.07, 130.02, 126.30, 121.01, 113.46; MS (ESI, m/z, rel.%): 352.2 ($[M+H]^+$, 100%).

1,3,5-Tris(4-dimethylaminophenyl)benzene (3e, Table 3).[12] Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow solid in 92% yield, mp 231–232 ^{o}C. The regioisomer ratio of **3e:2e** was 99:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ

7.65 (s, 3H), 7.62 (d, *J* = 8.7 Hz, 6H), 6.86 (d, *J* = 8.6 Hz, 6H), 3.02 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.03, 142.05, 129.87, 127.96, 122.62, 112.83, 40.67; MS (EI, m/z, rel.%): 435 (M^+, 100%), 419 (M^+–16, 12%), 208 (M^+–227, 13%).

1,3,5-Triphenylbenzene (3f, Table 3).[10] Isolated by column chromatography (PE : EA = 10 : 1) to afford a white solid in 99% yield, mp 175–176 °C (lit.[10] 176.7–178.4 °C). The regioisomer ratio of **3f:2f** was 98:2. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (s, 3H), 7.72 (d, *J* = 7.2 Hz, 6H), 7.50 (t, *J* = 7.5 Hz, 6H), 7.41 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.38, 141.18, 128.88, 127.58, 127.39, 125.21; MS (EI, m/z, rel.%): 306 (M^+, 100%), 289 (M^+–17, 12%), 228 (M^+–78, 8%), 215 (M^+–91, 2%).

1,3,5-Tris(4-methoxycarbonylphenyl)benzene (3g, Table 3).[10] Isolated by column chromatography (PE : EA = 4 : 1) to afford a white solid in 78% yield, mp 171–172 °C (lit.[10] 170.4–172.1 °C). The regioisomer ratio of **3g:2g** was 95:5. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (d, *J* = 8.3 Hz, 6H), 7.86 (s, 3H), 7.77 (d, *J* = 8.3 Hz, 6H), 3.96 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.86, 145.00, 141.65, 130.28, 129.52, 127.31, 126.07, 52.22; MS (EI, m/z, rel.%): 480 (M^+, 100%), 449 (M^+–31, 76%), 302 (M^+–178, 13%), 209 (M^+–271, 20%).

1,3,5-Tris(4-acetylphenyl)benzene (3h, Table 3).[13] Isolated by column chromatography (*n*-pentane : EA = 2 : 1) to afford a yellow solid in 89% yield, mp 254–255 °C (lit.[31] 256 °C). The regioisomer ratio of **3h:2h** was 95:5. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 8.4 Hz, 6H), 7.87 (s, 3H), 7.80 (d, *J* = 8.3 Hz, 6H), 2.67 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.62, 145.13, 141.67, 136.51, 129.14, 127.56, 126.17, 26.74; MS (EI, m/z, rel.%): 432 (M^+, 48%), 417 (M^+–15, 100%); HRMS (EI) calcd for $C_{30}H_{24}O_3$ 432.1720, found 432.1718.

1,3,5-Tris(4-formylphenyl)benzene (3i, Table 3).[14] Isolated by column chromatography (PE : EA = 2 : 1) to afford a yellow solid in 90% yield, mp 234–235 °C (lit.[32] 230–232 °C). The regioisomer ratio of **3i:2i** was 97:3. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.10 (s, 3H), 8.02 (d, *J* = 8.2 Hz, 6H), 7.91 (s, 3H), 7.87 (d, *J* = 8.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.69, 146.28, 141.60, 135.80, 130.42, 127.98, 126.47; HRMS (EI) calcd for $C_{27}H_{18}O_3$ 390.1250, found 390.1245.

1,3,5-Tris(4-fluorophenyl)benzene (3k, Table 3).[15] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 78% yield, mp 241–242 °C (lit.[16] 234–236 °C). The regioisomer ratio of **3k:2k** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (s, 3H), 7.64 (dd, *J* = 8.6, 5.5 Hz, 6H), 7.17 (t, *J* = 8.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.94, 161.48, 141.56, 137.05, 137.02, 128.95, 128.87, 124.87, 115.90, 115.68; ^{19}F NMR (500 MHz, $CDCl_3$): δ -115.02; MS (EI, m/z, rel.%): 360 (M^+, 100%), 338 (M^+–22, 8%), 264 (M^+–96, 5%), 159 (M^+–201, 4%).

1,3,5-Tris(4-chlorophenyl)benzene (3l, Table 3).[10] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 87% yield, mp 244–245 °C (lit.[16] 243–246 °C). The regioisomer ratio of **3l:2l** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (s, 3H), 7.60 (d, *J* = 7.2 Hz, 6H), 7.45 (d, *J* = 7.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.46, 139.20, 133.95, 129.11, 128.58, 125.05; MS (EI, m/z, rel.%): 410 ($[M+2]^+$, 100%), 408 (M^+, 99%), 372 (M^+–36, 4%), 338 (M^+–70, 20%), 302 (M^+–106, 26%), 169 (M^+–239, 20%), 151 (M^+–257, 34%).

1,3,5-Tris(4-bromophenyl)benzene (3m, Table 3).[15] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 85% yield, mp 255–256 °C (lit.[16] 255–256 °C). The regioisomer ratio of **3m:2m** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (s, 3H), 7.61 (d, *J* = 7.8 Hz, 6H), 7.53 (d, *J* = 7.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.53, 139.63, 132.07, 128.91, 125.00, 122.13; MS (EI, m/z, rel.%): 545 ($[M+6]^+$, 31%), 543 ($[M+4]^+$, 95%), 541 ($[M+2]^+$, 100%), 539 (M^+, 33%), 382 (M^+–157, 7%), 302 (M^+–237, 17%).

1,3,5-Tris(3-methylphenyl)benzene (3n, Table 3).[15] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a white solid in 94% yield, mp 106–107 °C (lit.[16] 110–111 °C). The regioisomer ratio of **3n:2n** was 91:9. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.80 (s, 3H), 7.56 (s, 3H), 7.53 (d, *J* = 7.7 Hz, 3H), 7.38 (t, *J* = 7.6 Hz, 3H), 7.23 (d, *J* = 7.5 Hz, 3H), 2.46 (s, 9H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 142.31, 141.02, 138.60, 128.71, 128.27, 128.03, 124.90, 124.32, 21.26; MS (EI, m/z, rel.%): 348 (M^+, 100%), 333 (M^+–15, 2%), 348 (M^+–30, 3%), 303 (M^+–45, 3%), 239 (M^+–109, 4%), 174 (M^+–174, 5%).

1,3,5-Tris(3-fluorophenyl)benzene (3o, Table 3).[16] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a white solid in 82% yield, mp 173–174 °C (lit.[16] 169–171 °C). The regioisomer ratio of **3o:2o** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (s, 3H), 7.48–7.37 (m, 9H), 7.13–7.08 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.48, 162.04, 143.01, 142.93, 141.44, 141.42, 130.47, 130.39, 125.51, 122.98, 122.95, 114.71, 114.50, 114.38, 114.16; ^{19}F NMR (500 MHz, $CDCl_3$): δ -112.69; MS (EI, m/z, rel.%): 360 (M^+, 100%), 338 (M^+–22, 7%), 264 (M^+–96, 7%), 159 (M^+–201, 6%).

1,3,5-Tris(2-methylphenyl)benzene (3p, Table 3).[10] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a white solid in 75% yield, mp 129–130 °C (lit.[16] 129–130 °C). The regioisomer ratio of **3p:2p** was 96:4. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.36–7.24 (m, 15H), 2.38 (s, 9H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 141.73, 141.55, 135.46, 130.34, 129.79, 128.49, 127.29, 125.77, 20.34; MS (EI, m/z, rel.%): 348 (M^+, 100%), 333 (M^+–15, 14%), 318 (M^+–30, 5%), 303 (M^+–45, 5%), 257 (M^+–91, 33%).

1,3,5-Tris(2-fluorophenyl)benzene (3q, Table 3).[27] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a white solid in 90% yield, mp 121–122 °C. The regioisomer ratio of **3q:2q** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (s, 3H), 7.45 (td, *J* = 7.7, 1.5 Hz, 3H), 7.29–7.21 (m, 3H), 7.16–7.07 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.12, 158.65, 136.25, 130.95, 130.91, 129.33, 129.25, 129.10, 129.07, 129.04, 128.75, 128.62, 124.48, 124.44, 116.31, 116.09; ^{19}F NMR (500 MHz, $CDCl_3$): δ -117.65; MS (EI, m/z, rel.%): 360 (M^+, 100%), 338 (M^+–22, 9%), 318 (M^+–42, 4%), 264 (M^+–96, 3%), 159 (M^+–201, 8%).

1,3,5-Tris(2-thienyl)benzene (3r, Table 3).[10] Isolated by column chromatography (*n*-pentane : DCM = 5 : 1) to afford a yellow solid in 92% yield, mp 156–157 °C (lit.[10] 155.6–157.1 °C). The regioisomer ratio of **3r:2r** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (s, 3H), 7.42 (dd, *J* = 3.6, 1.1 Hz, 3H), 7.34 (dd, *J* = 5.1, 1.1 Hz, 3H), 7.13 (dd, *J* = 5.1, 3.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

143.55, 135.72, 128.10, 125.40, 123.88, 122.79; MS (EI, m/z, rel.%): 324 (M^+, 100%), 279 (M^+–45, 3%), 258 (M^+–66, 4%), 245 (M^+–79, 5%).

1,3,5-Tris(3-thienyl)benzene (3s, Table 3).[16] Isolated by column chromatography (PE : DCM = 5 : 1) to afford a white solid in 89% yield, mp 129–130 °C (lit.[16] 129–130 °C). The regioisomer ratio of **3s:2s** was 92:8. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (s, 3H), 7.55 (dd, J = 2.9, 1.4 Hz, 3H), 7.48 (d, J = 1.4 Hz, 1H), 7.46 (d, J = 1.4 Hz, 2H), 7.44 (dd, J = 4.9, 2.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.17, 137.01, 126.52, 126.42, 123.70, 120.85; MS (EI, m/z, rel.%): 324 (M^+, 100%), 290 (M^+–34, 8%), 279 (M^+–45, 4%), 258 (M^+–66, 4%), 245 (M^+–79, 5%).

1,3,5-Tris(4-pyridyl)benzene (3t, Table 3).[17] Isolated by column chromatography ($CHCl_3$: TEA = 10 : 1) to afford a white solid in 89% yield. The regioisomer ratio of **3t:2t** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, J = 4.7 Hz, 6H), 7.92 (s, 3H), 7.61 (d, J = 5.5 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.58, 147.41, 140.44, 126.37, 121.83; MS (EI, m/z, rel.%): 309 (M^+, 100%), 281 (M^+–43, 5%).

1,3,5-Tributylbenzene (3u, Table 3).[9] Isolated by column chromatography (*n*-pentane : DCM = 500 : 1) to afford a yellow oil in 93% yield. The regioisomer ratio of **3u:2u** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.85 (s, 3H), 2.59 (t, J = 7.6 Hz, 6H), 1.67–1.59 (m, 6H), 1.40 (dq, J = 14.6, 7.3 Hz, 6H), 0.97 (t, J = 7.3 Hz, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.69, 125.86, 35.72, 33.82, 22.56, 14.01; MS (EI, m/z, rel.%): 246 (M^+, 41%), 204 (M^+–42, 100%), 161 (M^+–85, 35%), 147 (M^+–99, 65%), 119 (M^+–127, 30%), 105 (M^+–141, 44%), 91 (M^+–155, 30%).

1,3,5-Tricyclopropylbenzene (3v, Table 3).[1] Isolated by column chromatography (*n*-pentane : DCM = 10 : 1) to afford a colorless oil in 97% yield. The regioisomer ratio of **3v:2v** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.61 (s, 3H), 1.85 (tt, J = 8.4, 5.2 Hz, 3H), 0.94 (ddd, J = 8.4, 6.4, 4.4 Hz, 6H), 0.70 (dt, J = 6.5, 4.5 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.88, 120.37, 15.38, 8.95; MS (EI, m/z, rel.%): 198 (M^+, 68%), 183 (M^+–15, 20%), 169 (M^+–29, 17%), 157 (M^+–41, 43%), 141 (M^+–57, 45%), 129 (M^+–69, 100%), 115 (M^+–83, 49%).

1,3,5-Tris(1-cyclohexenyl)benzene (3w, Table 3).[9] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a colorless oil in 81% yield. The regioisomer ratio of **3w:2w** was 82:18. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (s, 3H), 6.09 (ddd, *J* = 5.5, 3.8, 1.6 Hz, 3H), 2.42 (ddd, *J* = 6.2, 5.1, 2.2 Hz, 6H), 2.20 (qd, *J* = 6.2, 2.5 Hz, 6H), 1.81–1.75 (m, 6H), 1.69–1.63 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.62, 137.26, 124.55, 120.47, 27.75, 25.87, 23.14, 22.23; MS (EI, m/z, rel.%): 318 (M^+, 100%), 275 (M^+–43, 8%), 264 (M^+–54, 13%), 237 (M^+–81, 23%), 195 (M^+–123, 8%), 165 (M^+–153, 16%), 141 (M^+–177, 20%), 81 (M^+–237, 31%).

1,3,5-Tris(trimethylsilyl)benzene (3x, Table 3).[3] Isolated by column chromatography (PE : DCM = 10 : 1) to afford a yellow oil in 92% yield. The regioisomer ratio of **3x:2x** was 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (s, 3H), 0.29 (s, 27H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.87, 139.36, -0.00; MS (EI, m/z, rel.%): 294 (M^+, 10%), 279 (M^+–15, 100%), 132 (M^+–162, 11%), 73 (M^+–221, 20%).

Benzene-1,3,5-tricarboxylic acid trimethyl ester (3y, Table 3).[9] Isolated by column chromatography (PE : EA = 5 : 1) to afford a white solid in 80% yield, mp 146–147 °C (lit.[33] 144 °C). The regioisomer ratio of **3y:2y** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.78 (s, 3H), 3.94 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.30, 134.47, 131.17, 77.38, 52.54; MS (EI, m/z, rel.%): 252 (M^+, 14%), 221 (M^+–31, 100%).

1,3,5-Triacetylphenylbenzene (3z, Table 3).[9] Isolated by column chromatography (PE : EA = 2 : 1) to afford a white solid in 90% yield, mp 160–161 °C (lit.[34] 160.5–161.7 °C). The regioisomer ratio of **3z:2z** was 91:9. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.69 (s, 3H), 2.70 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.60, 137.94, 131.72, 26.81; MS (EI, m/z, rel.%): 204 (M^+, 12%), 189 (M^+–15, 85%), 161 (M^+–43, 14%).

(E)-1,4-bis(2-methylphenyl)but-1-en-3-yne (4pa, Scheme 2).[28] Isolated by column chromatography (n-pentane : DCM = 10 : 1) to afford a white solid in 43% yield, mp 56–57 °C (lit.[28] 54–57 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.39 (m, 1H), 7.37 (d, *J* = 7.4 Hz, 1H), 7.18 (d, *J* = 16.1 Hz, 1H), 7.13–7.03 (m, 6H), 6.25 (d, *J* = 16.1 Hz, 1H), 2.40 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.15, 138.69, 135.86, 135.39, 131.94, 130.62, 129.52, 128.51, 128.30, 126.31, 125.65, 125.01, 123.26, 109.40, 93.12, 90.43, 20.83, 19.88; MS (EI, m/z, rel.%): 232 (M^+, 100%), 217 (M^+–15, 88%), 202 (M^+–30, 67%), 115 (M^+–117, 38%).

2,4-Bis(2-methylphenyl)but-1-en-3-yne (4pb, Scheme 2).[29] Isolated by column chromatography (*n*-pentane : DCM = 10 : 1) to afford a yellow oil in 38% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.6 Hz, 1H), 7.37–7.32 (m, 1H), 7.24–7.18 (m, 5H), 7.16–7.10 (m, 1H), 5.87 (d, *J* = 1.8 Hz, 1H), 5.54 (d, *J* = 1.8 Hz, 1H), 2.51 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.16, 139.43, 135.55, 131.98, 131.88, 130.41, 129.43, 128.79, 128.33, 127.88, 125.89, 125.52, 124.99, 122.99, 93.28, 89.60, 20.72, 20.31; MS (EI, m/z, rel.%): 232 (M^+, 24%), 217 (M^+–15, 100%), 202 (M^+–30, 62%), 115 (M^+–117, 26%).

2,5-Dimethyl 4'-methyl[1,1'-biphenyl]-2,5-dicarboxylate (5a, Table 4). Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow oil in 56% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06–8.02 (m, 2H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.22 (s, 4H), 3.94 (s, 3H), 3.68 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.70, 166.24, 142.42, 137.46, 137.27, 134.89, 132.37, 131.76, 129.64, 128.99, 128.16, 127.87, 52.39, 52.21, 21.21; MS (EI, m/z, rel.%): 284 (M^+, 100%), 253 (M^+–31, 96%); HRMS (EI) calcd for $C_{17}H_{16}O_4$ 284.1043, found 316.1040.

Methyl 4,4''-dimethyl-[1,1':4',1''-terphenyl]-2'-carboxylate (5b, Table 4).[18] Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow oil in 51% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 1.8 Hz, 1H), 8.02 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.04 (s, 8H), 3.94 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.02, 145.00, 140.64, 137.82, 137.76, 136.77, 136.44, 131.90, 130.74, 129.63, 129.53, 128.95, 128.75, 128.24, 52.09, 21.13, 21.11; MS (EI, m/z, rel.%): 316 (M^+, 100%), 301 (M^+–15, 23%), 257 (M^+–59, 45%), 242 (M^+–74, 99%); HRMS (EI) calcd for $C_{22}H_{20}O_2$ 316.1458, found 316.1455.

(4,4''-dimethyl-[1,1':3',1''-terphenyl]-5'-yl)trimethylsilane (6a, Table 4). Isolated by column chromatography (PE : DCM = 10 : 1) to afford a colorless oil in 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (t, *J* = 1.6 Hz, 1H), 7.69 (d, *J* = 1.6 Hz, 2H), 7.56 (d, *J* = 8.1 Hz, 4H), 7.29 (d, *J* = 8.0 Hz, 4H), 2.43 (s, 6H), 0.36 (s, 9H);

^{13}C NMR (100 MHz, CDCl$_3$): δ 141.47, 141.01, 138.77, 137.06, 130.76, 129.48, 127.24, 126.56, 21.10, -1.02; MS (EI, m/z, rel.%): 330 (M$^+$, 39%), 315 (M$^+$–15, 100%); HRMS (EI) calcd for C$_{23}$H$_{26}$Si 330.1798, found 330.1796.

(4'-methyl-[1,1'-biphenyl]-3,5-diyl)bis(trimethylsilane) (6b, Table 4). Isolated by column chromatography (PE : DCM = 10 : 1) to afford a colorless oil in 70% yield. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.70 (d, *J* = 1.0 Hz, 2H), 7.65 (s, 1H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 9.3 Hz, 2H), 2.42 (s, 3H), 0.33 (s, 18H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 139.92, 139.70, 139.19, 136.87, 136.85, 132.74, 129.42, 127.30, 21.09, -1.04; MS (EI, m/z, rel.%): 312 (M$^+$, 34%), 297 (M$^+$–15, 100%); HRMS (EI) calcd for C$_{19}$H$_{28}$Si$_2$ 312.1724, found 312.1721.

Dimethyl [1,1':4',1''-terphenyl]-2',3'-dicarboxylate (7a, Table 4).[19] Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow oil in 67% yield. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.79 (s, 2H), 7.24–7.23 (m, 6H), 7.15–7.12 (m, 4H), 3.94 (s, 6H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 167.83, 143.45, 139.60, 131.25, 130.77, 129.63, 128.15, 127.41, 52.68; MS (EI, m/z, rel.%): 346 (M$^+$, 94%), 315 (M$^+$–31, 100%), 287 (M$^+$–59, 14%).

Tetramethyl benzene-1,2,3,4-tetracarboxylate (7b, Table 4).[20] Isolated by column chromatography (PE : EA = 2 : 1) to afford a white solid in 82% yield, mp 122–123 °C (lit.[35] 129–131 °C). ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.99 (s, 2H), 3.90 (s, 6H), 3.89 (s, 6H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 166.84, 165.28, 133.76, 132.81, 131.03, 53.07, 53.01; MS (EI, m/z, rel.%): 310 (M$^+$, 1%), 279 (M$^+$–31, 100%).

Dimethyl [1,1':3',1''-terphenyl]-4',5'-dicarboxylate (8a, Table 4).[21] Isolated by column chromatography (PE : EA = 5 : 1) to afford a yellow solid in 60% yield, mp 76–77 °C (lit.[36] 75–77 °C). ^{1}H NMR (400 MHz, CDCl$_3$): δ 8.23 (d, *J* = 1.2 Hz, 1H), 7.77 (d, *J* = 1.2 Hz, 1H), 7.64 (d, *J* = 7.4 Hz, 2H), 7.49–7.39 (m, 8H), 3.94 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 169.20, 166.22, 142.24, 141.24, 139.33, 139.12, 133.40, 132.67, 129.02, 128.87, 128.64, 128.34, 128.30, 127.98, 127.49, 127.24, 52.65, 52.31; MS (EI, m/z, rel.%): 346 (M$^+$, 46%), 315 (M$^+$–31, 89%).

Tetramethyl benzene-1,2,3,5-tetracarboxylate (8b, Table 4).[22] Isolated by column chromatography (PE : EA = 2 : 1) to afford a white solid in 80% yield, mp

107–108 °C (lit.[37] 105–107 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.82 (s, 2H), 4.00 (s, 3H), 3.98 (s, 3H), 3.94 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.92, 164.68, 164.34, 140.35, 135.10, 131.26, 129.21, 53.00, 52.97, 52.81; MS (EI, m/z, rel.%): 310 (M^+, 1%), 279 (M^+–31, 100%).

Tetramethyl [1,1'-biphenyl]-2,3,4,5-tetracarboxylate (9a, Table 4).[23] Isolated by column chromatography (PE : EA = 2 : 1) to afford a yellow solid in 78% yield, mp 155–156 °C (lit.[38] 151 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (s, 1H), 7.43–7.41 (m, 3H), 7.34–7.32 (m, 2H), 3.95 (s, 3H), 3.92 (s, 3H), 3.88 (s, 3H), 3.62 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.451, 167.446, 165.69, 164.95, 142.19, 138.00, 136.72, 134.10, 133.99, 130.17, 130.11, 128.58, 128.24, 53.21, 53.02, 52.99, 52.57; MS (EI, m/z, rel.%): 386 (M^+, 25%), 355 (M^+–31, 100%), 323 (M^+–63, 12%).

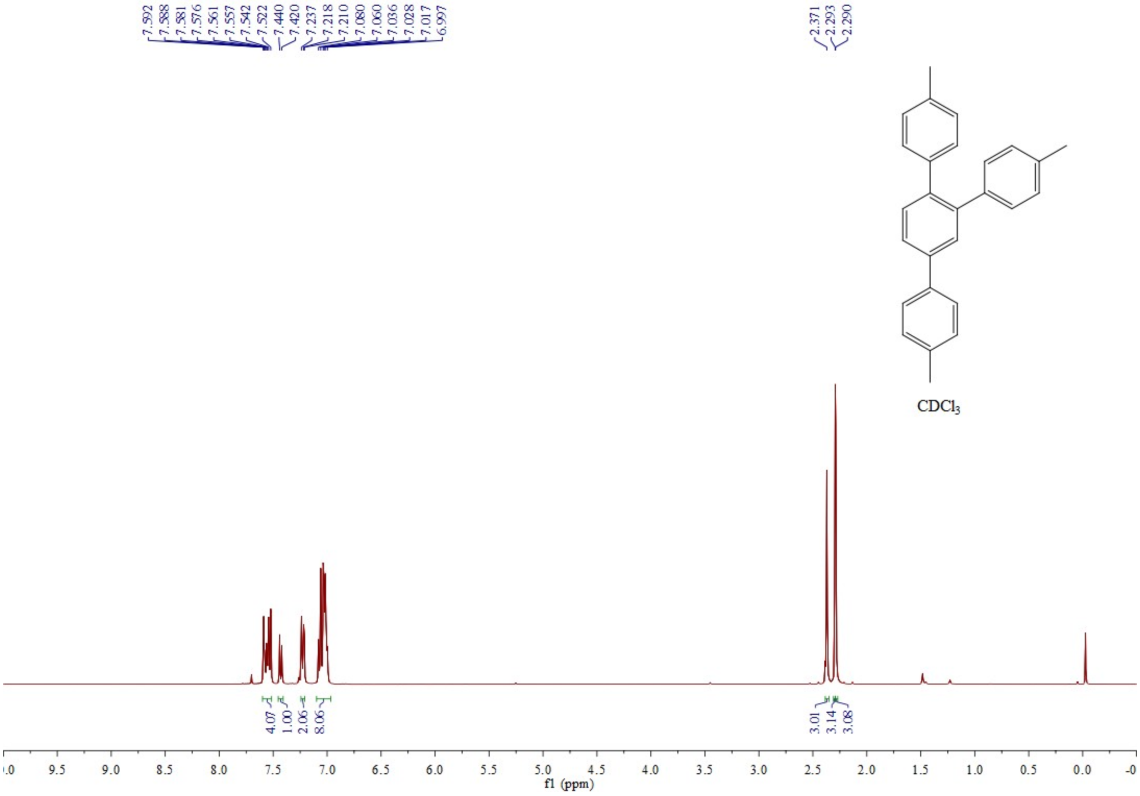
Pentamethyl benzene-1,2,3,4,5-pentacarboxylate (9b, Table 4).[24] Isolated by column chromatography (PE : EA = 2 : 1) to afford a white solid in 85% yield, mp 150–151 °C (lit.[37] 148–150 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.65 (s, 1H), 3.94 (s, 6H), 3.94 (s, 6H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.76, 164.69, 164.11, 138.76, 134.15, 130.22, 130.12, 53.38, 53.19, 53.15; MS (EI, m/z, rel.%): 368 (M^+, 1%), 337 (M^+–31, 100%).

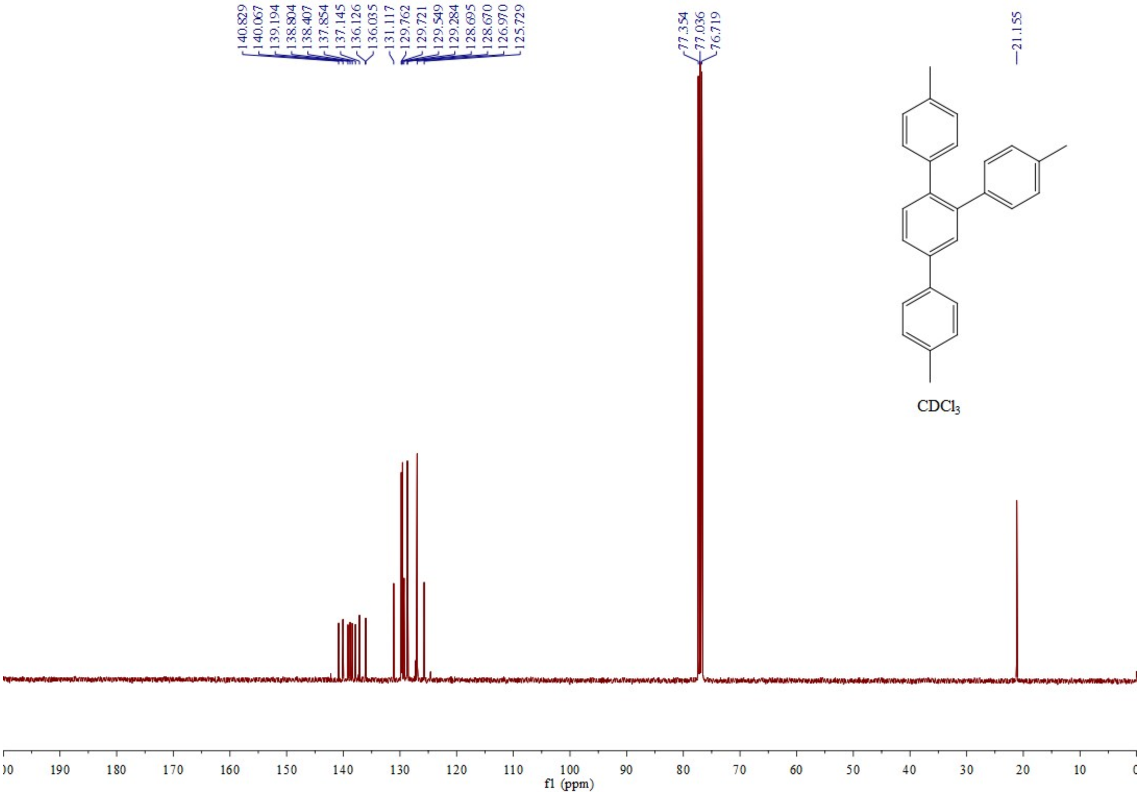
Benzene-1,2,3,4,5,6-hexacarboxylic acid hexamethyl ester (10a, Table 4).[9] Isolated by column chromatography (PE : EA = 1 : 1) to afford a white solid in 99% yield, mp 189–190 °C (lit.[39] 189–190 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.87 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.12, 133.91, 53.45; MS (EI, m/z, rel.%): 426 (M^+, 1%), 395 (M^+–31, 100%), 364 (M^+–62, 4%), 349 (M^+–77, 6%), 293 (M^+–133, 5%).

2-Ethyl-3,4,5,6-tetramethyl [1,1'-biphenyl]-2,3,4,5,6-pentacarboxylate (10b, Table 4).[25] Isolated by column chromatography (PE : EA = 1 : 1) to afford a yellow solid in 72% yield, mp 121–122 °C (lit.[40] 124–125 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39–7.35 (m, 3H), 7.21–7.19 (m, 2H), 3.96 (q, J = 7.1 Hz, 2H), 3.90 (s, 3H), 3.87 (s, 3H), 3.86 (s, 3H), 3.49 (s, 3H), 0.90 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.49, 166.12, 165.99, 165.57, 165.51, 140.65, 136.86, 136.68, 135.91, 132.26, 131.52, 131.27, 128.63, 128.39, 128.15, 61.94, 53.30, 53.23, 53.21, 52.56, 13.43; MS (EI, m/z, rel.%): 458 (M^+, 47%), 427 (M^+-31, 27%), 413 (M^+-45, 22%), 381 (M^+-77, 54%), 335 (M^+-123, 100%).

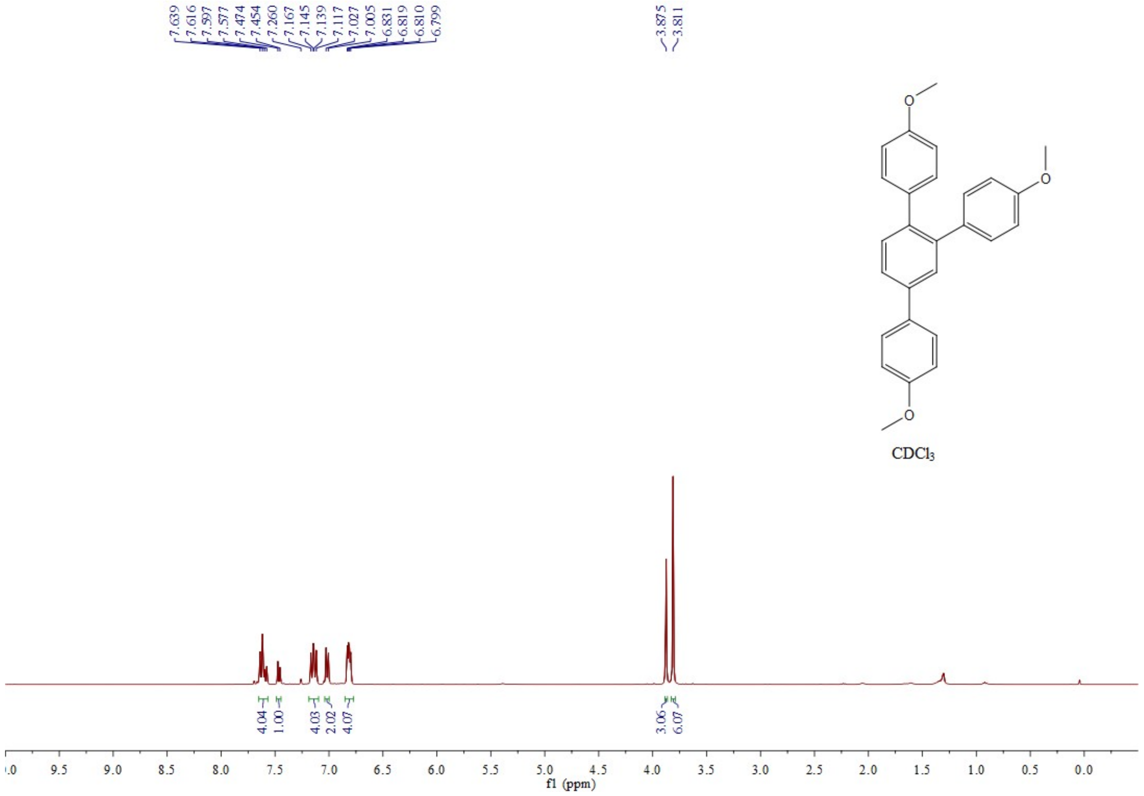
7. Copies of ^{1}H, ^{19}F and ^{13}C NMR Spectra

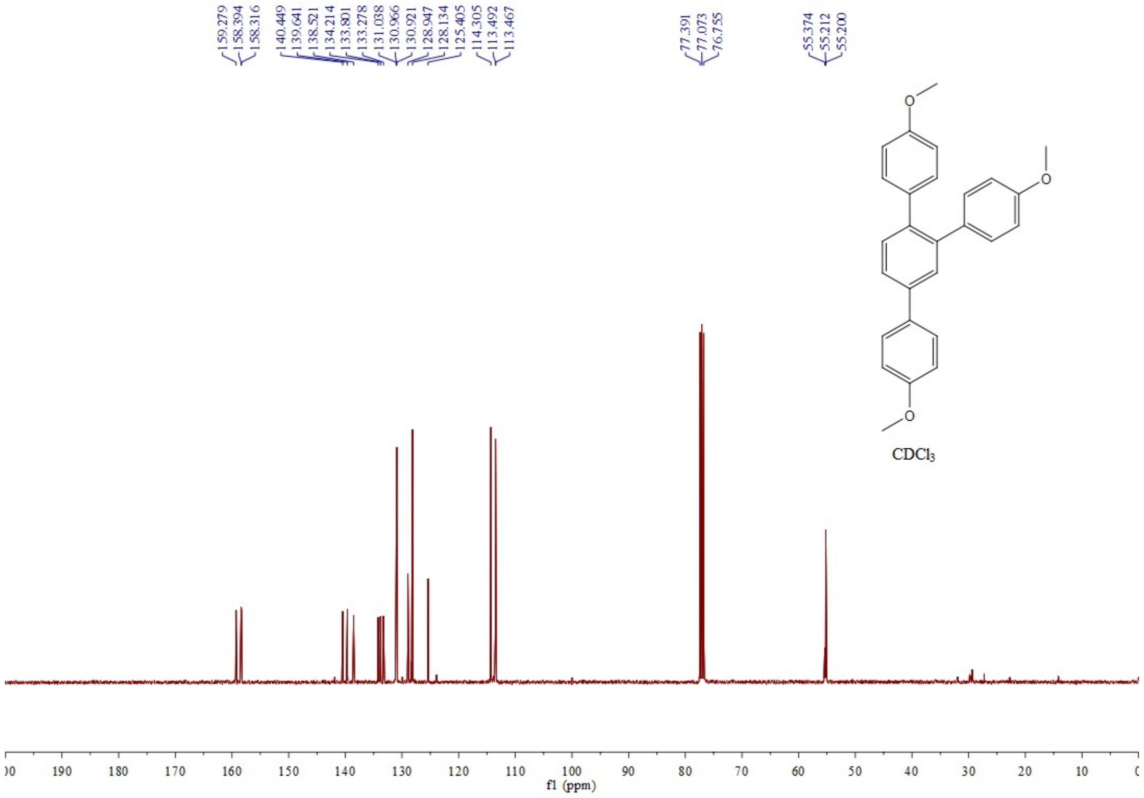
1,2,4-Tris(4-methylphenyl)benzene (2a, Table 2).



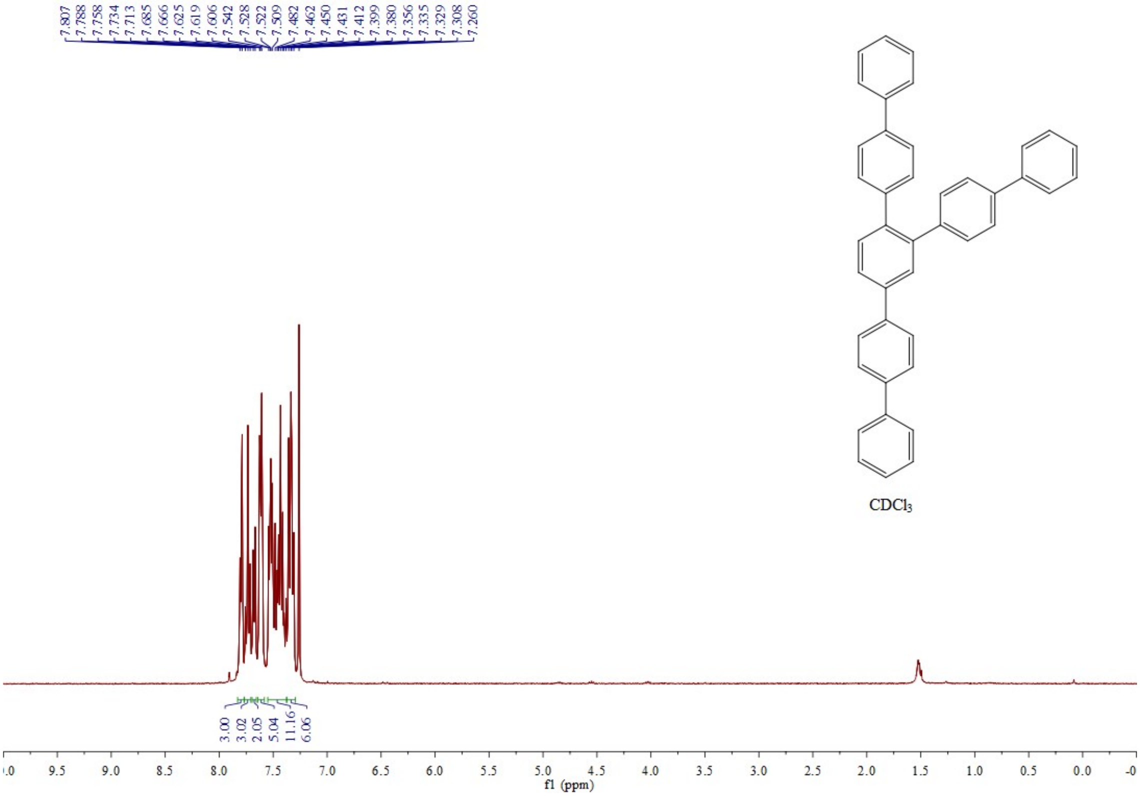


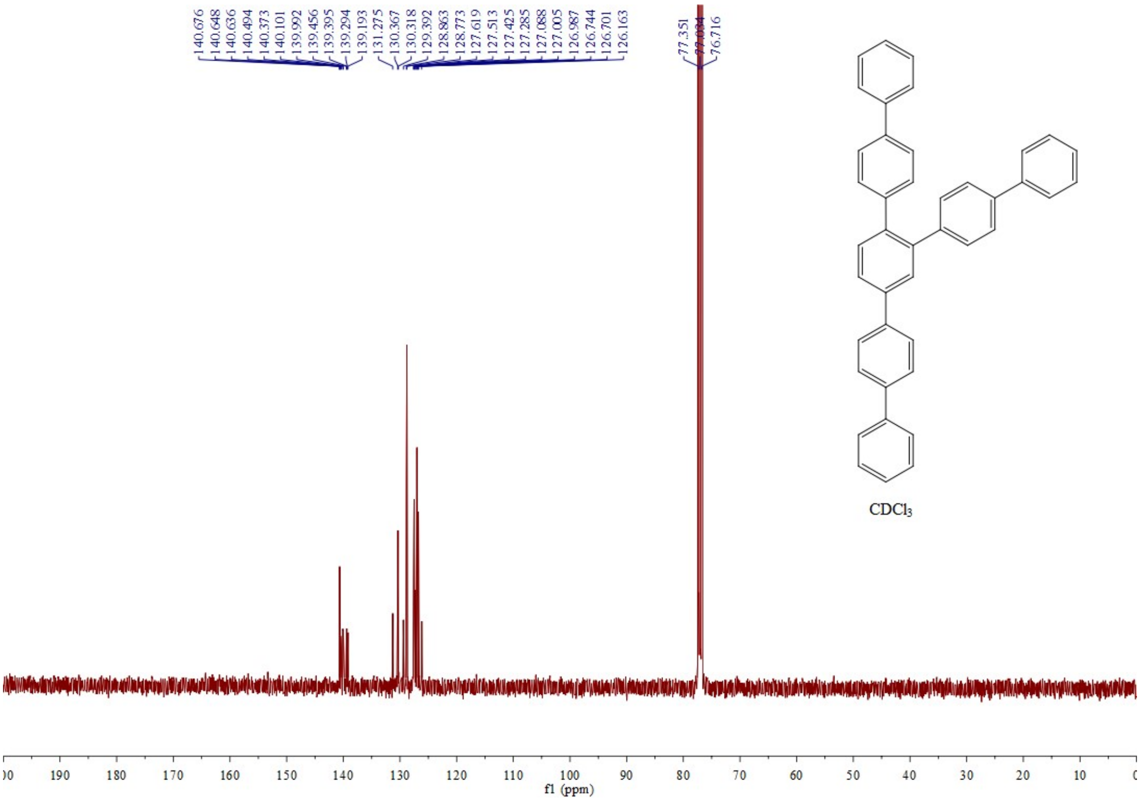
1,2,4-Tris(4-methoxyphenyl)benzene (2b, Table 2).



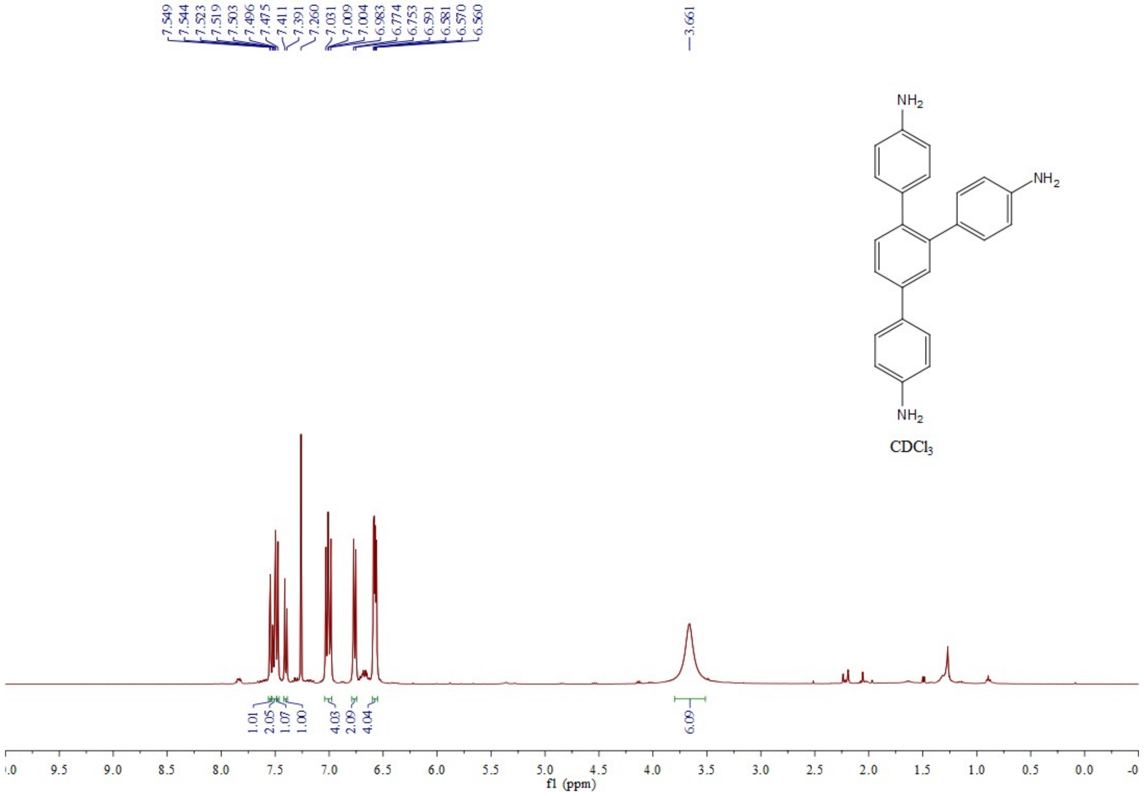


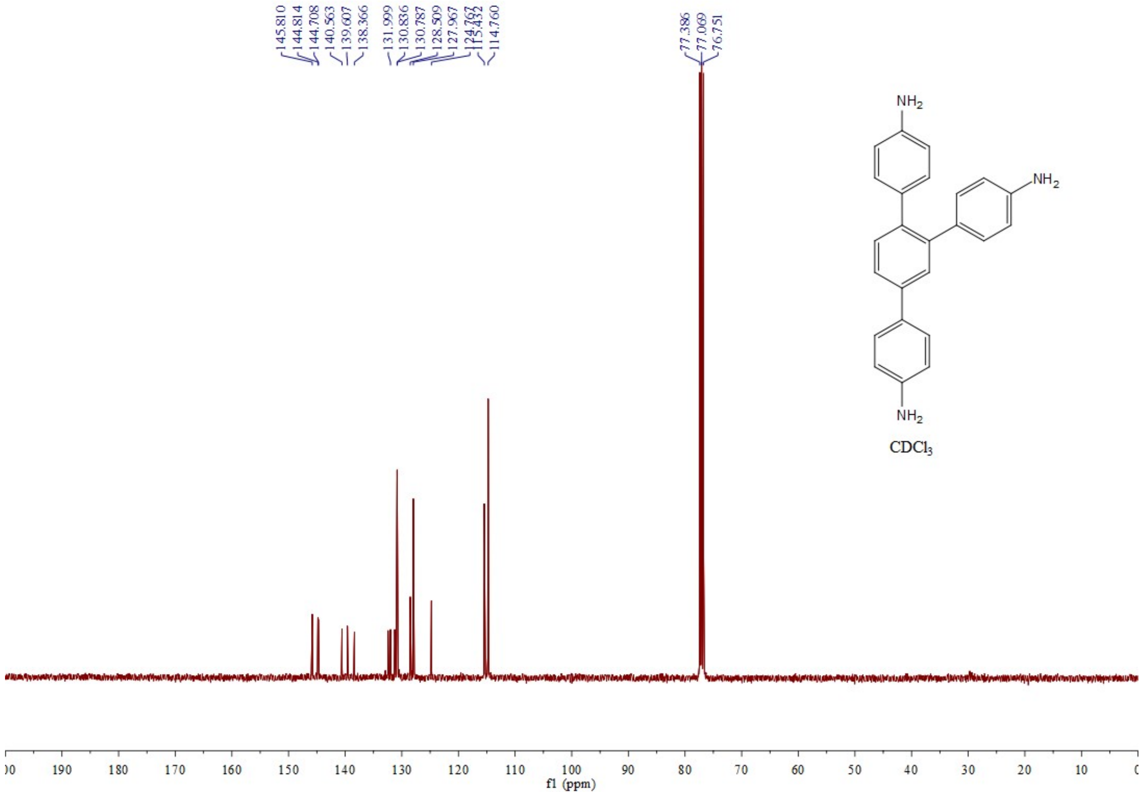
1,2,4-Tris[4-(phenyl)phenyl]benzene (2c, Table 2).



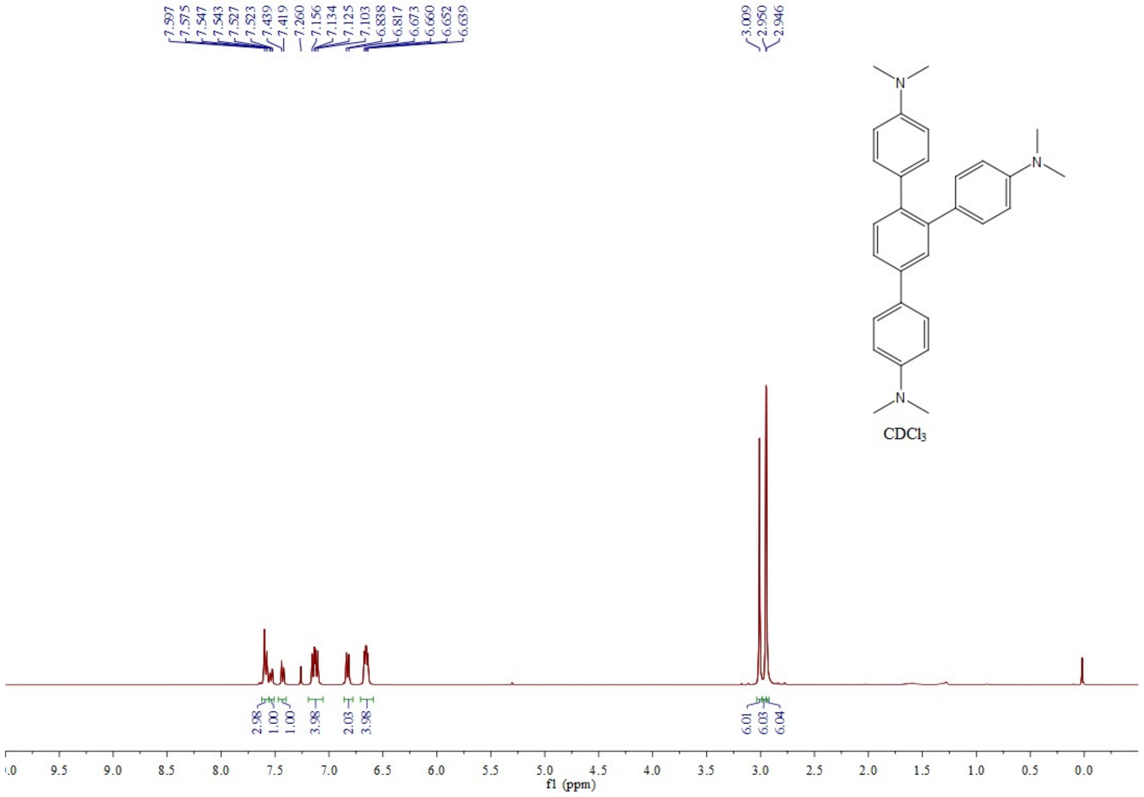


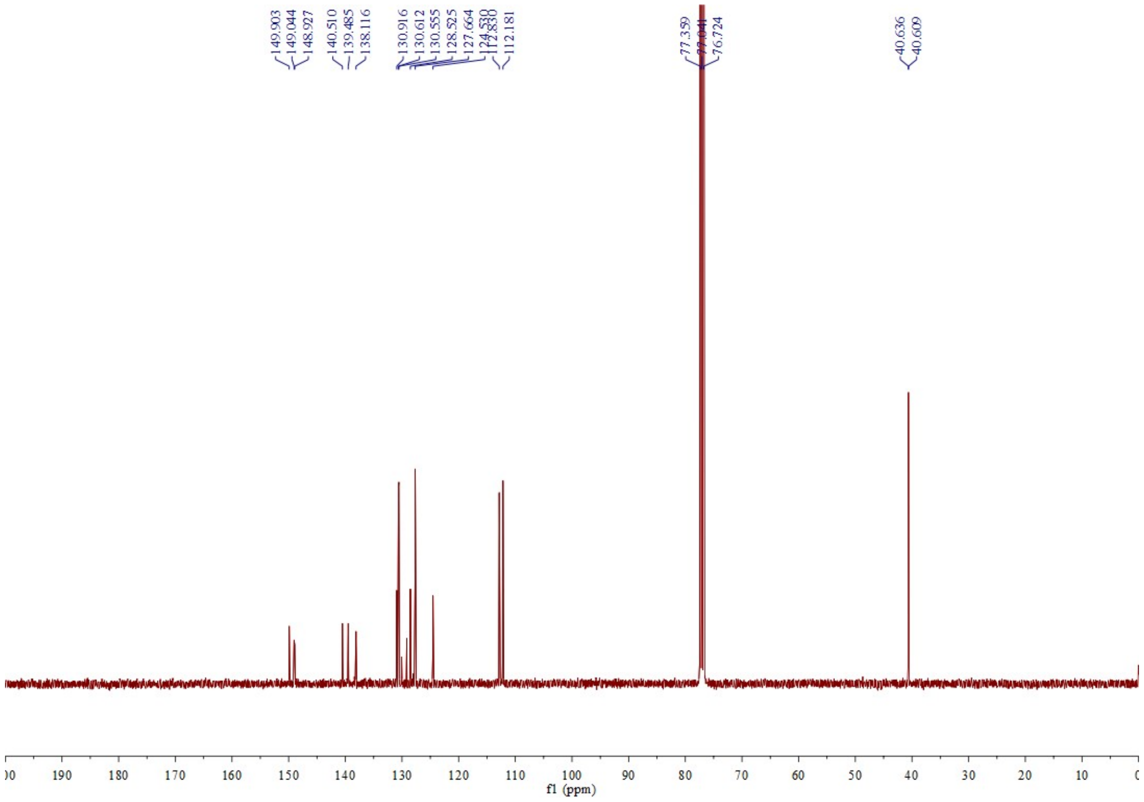
1,2,4-Tris(4-aminophenyl)benzene (2d, Table 2).



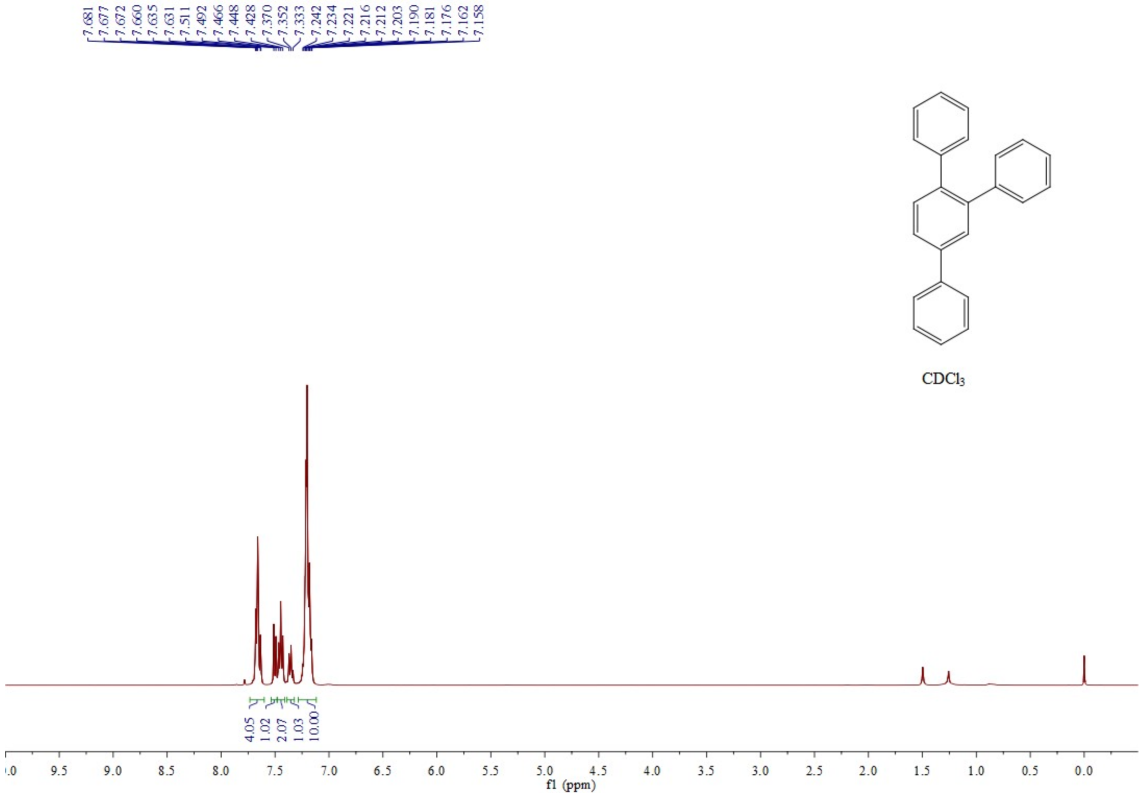


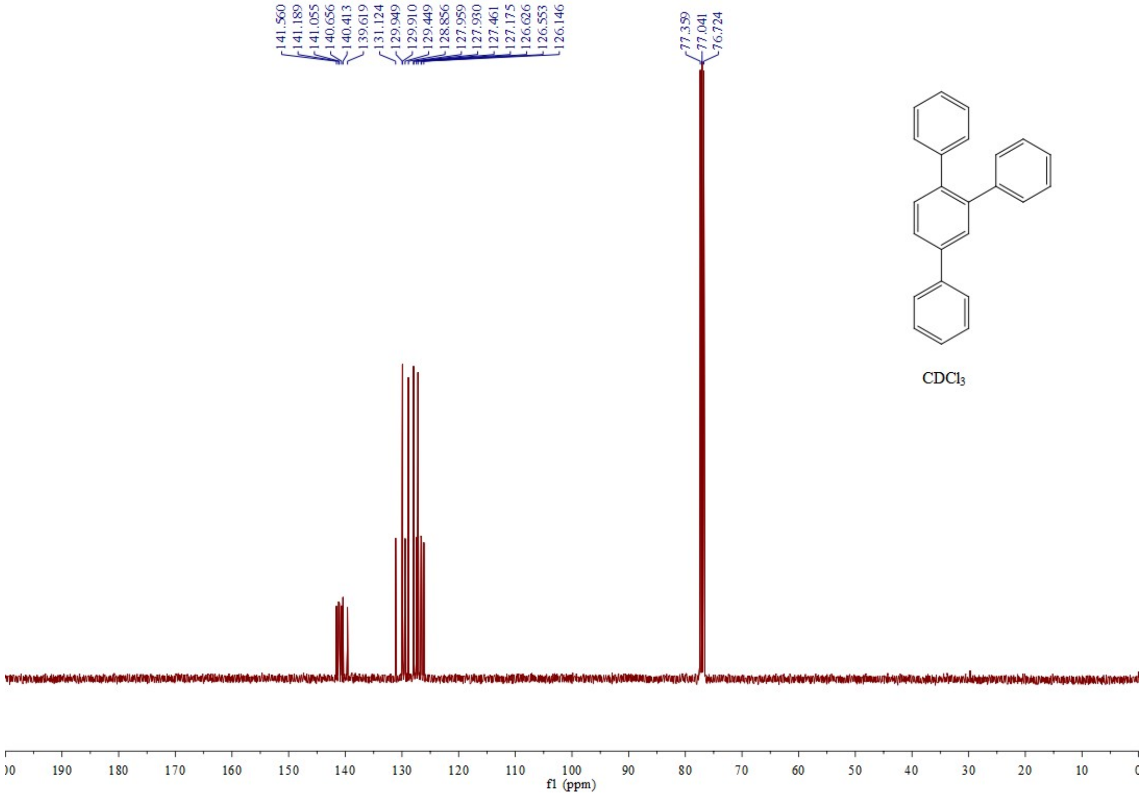
1,2,4-Tris(4-dimethylaminophenyl)benzene (2e, Table 2).



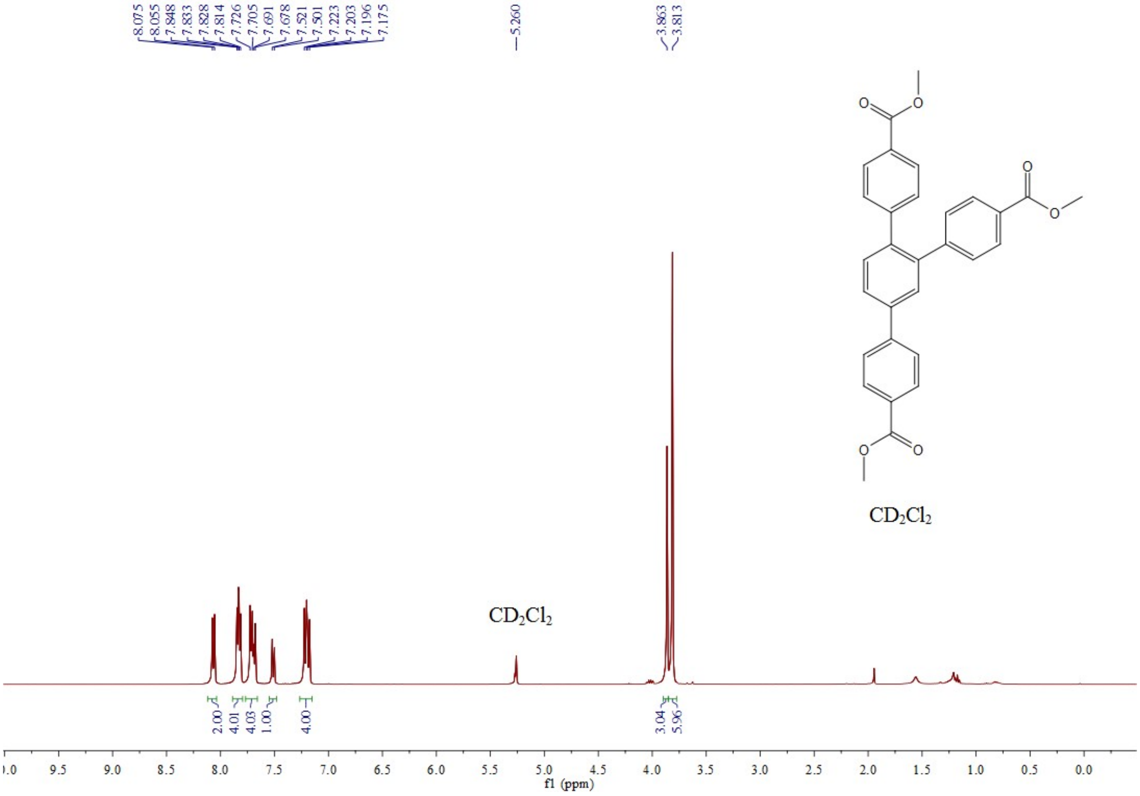


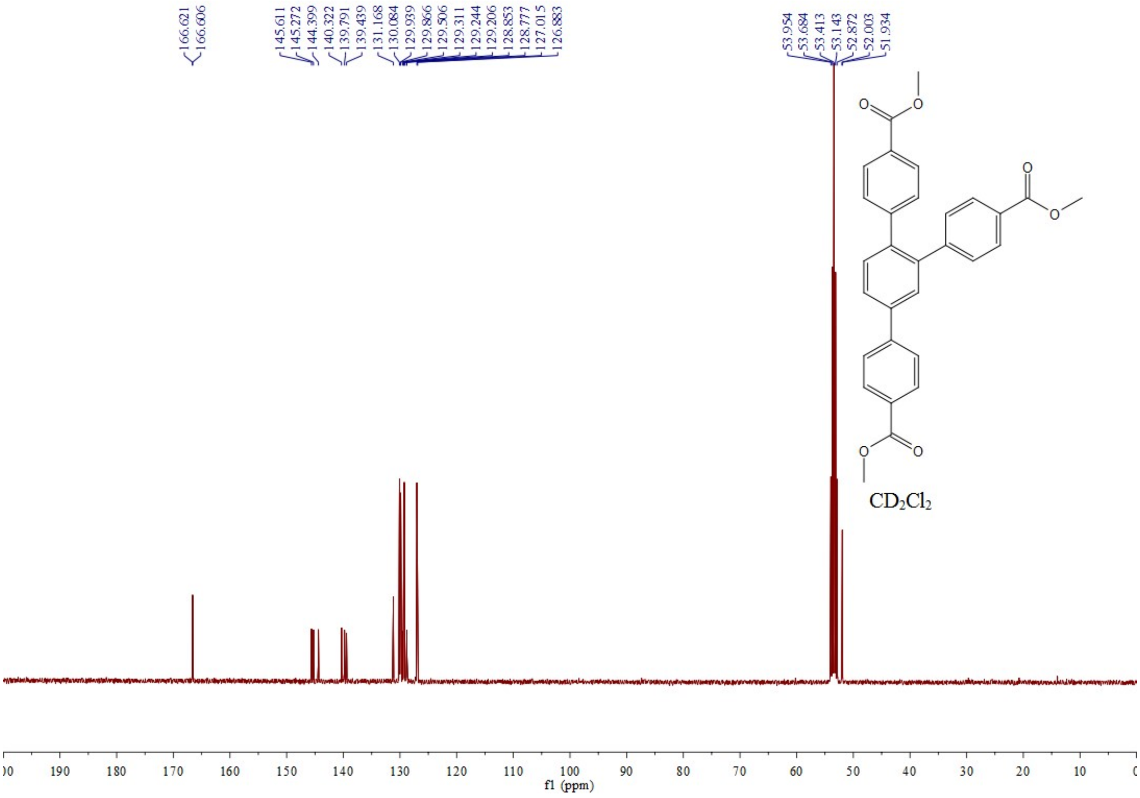
1,2,4-Triphenylbenzene (2f, Table 2).



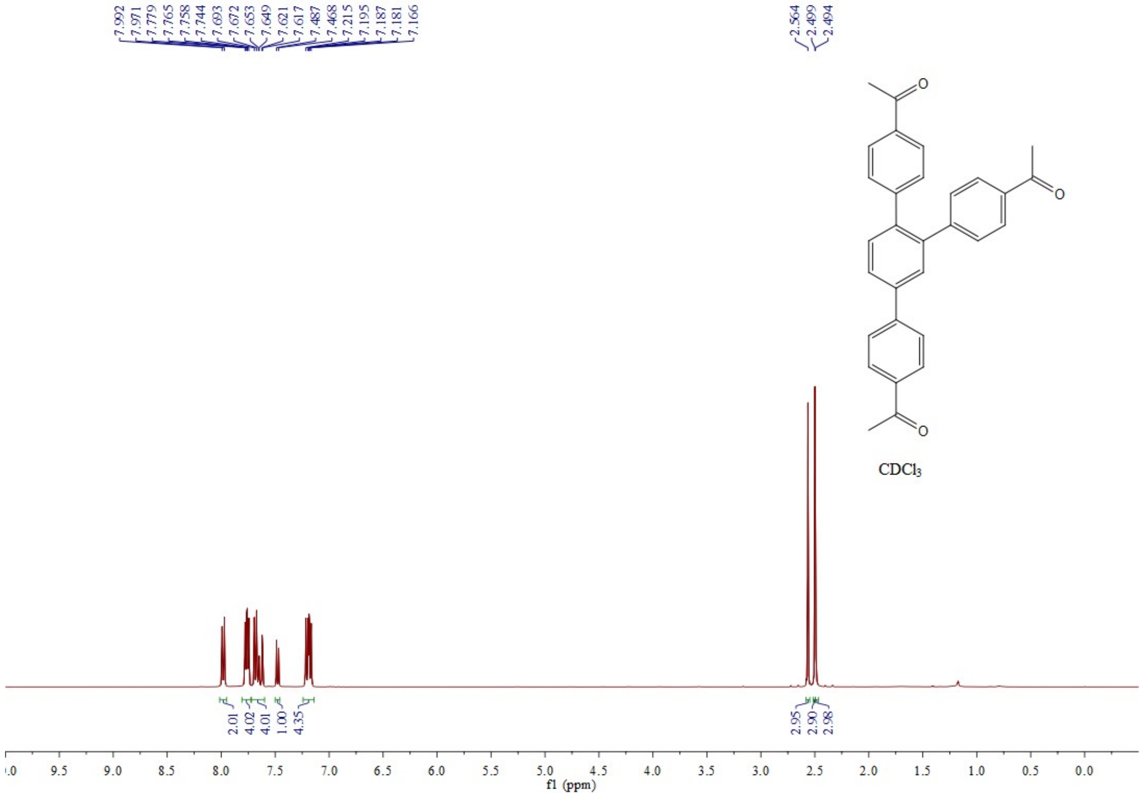


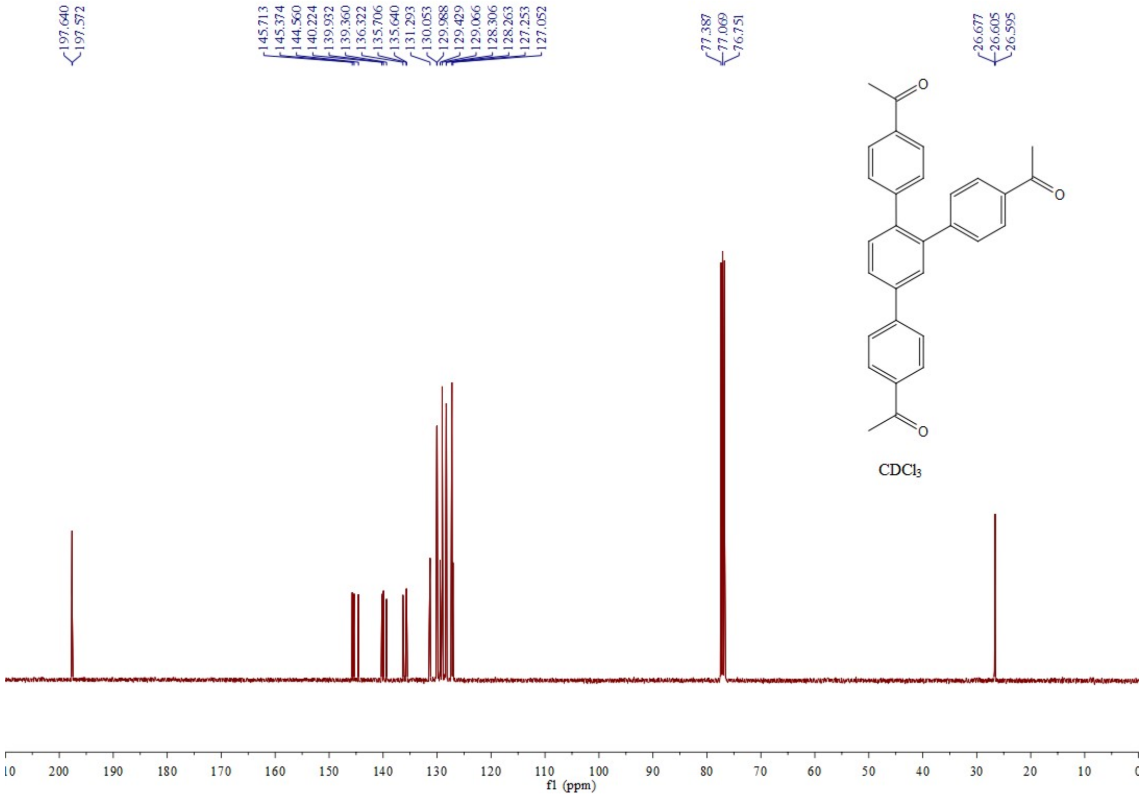
1,2,4-Tris(4-methoxycarbonylphenyl)benzene (2g, Table 2).



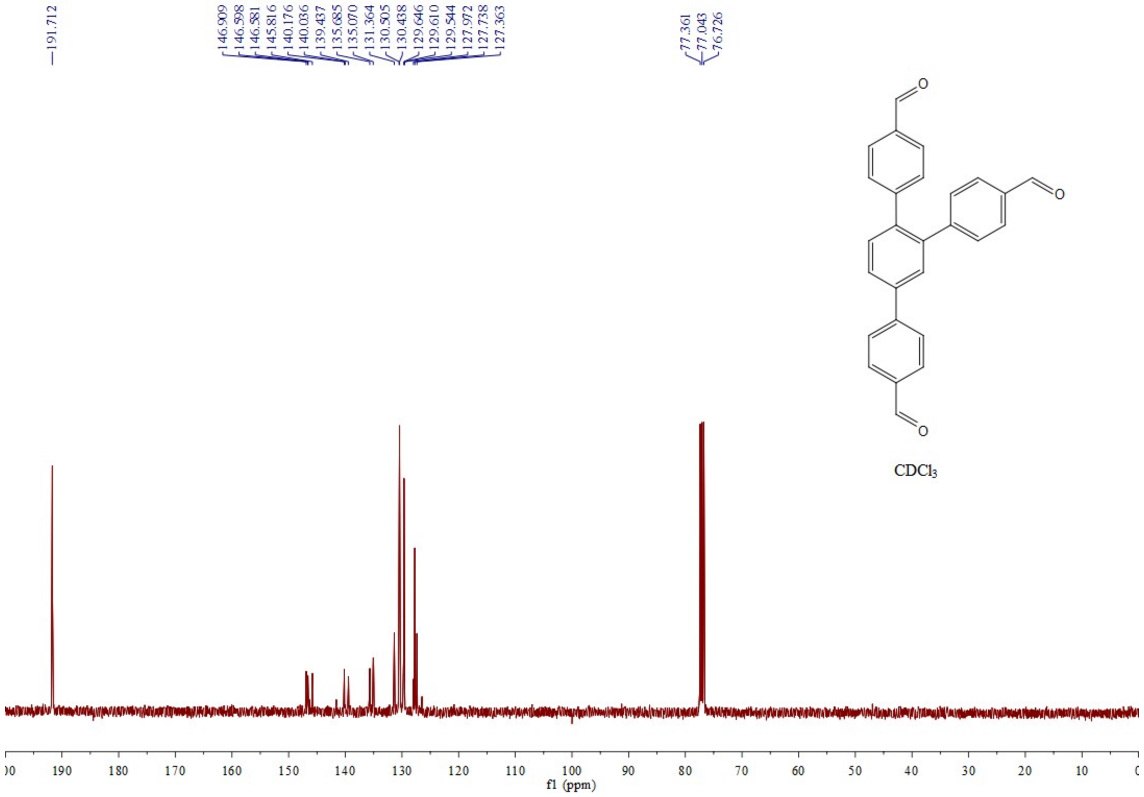


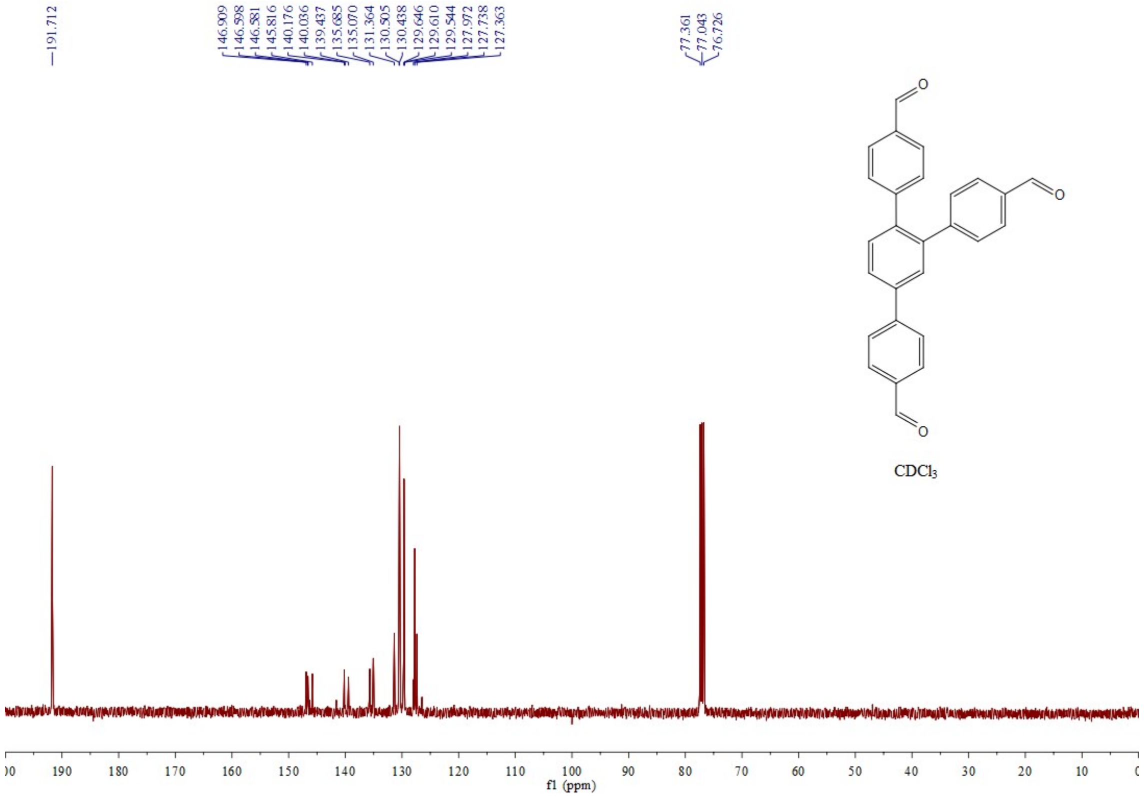
1,2,4-Tris(4-acetylphenyl)benzene (2h, Table 2).



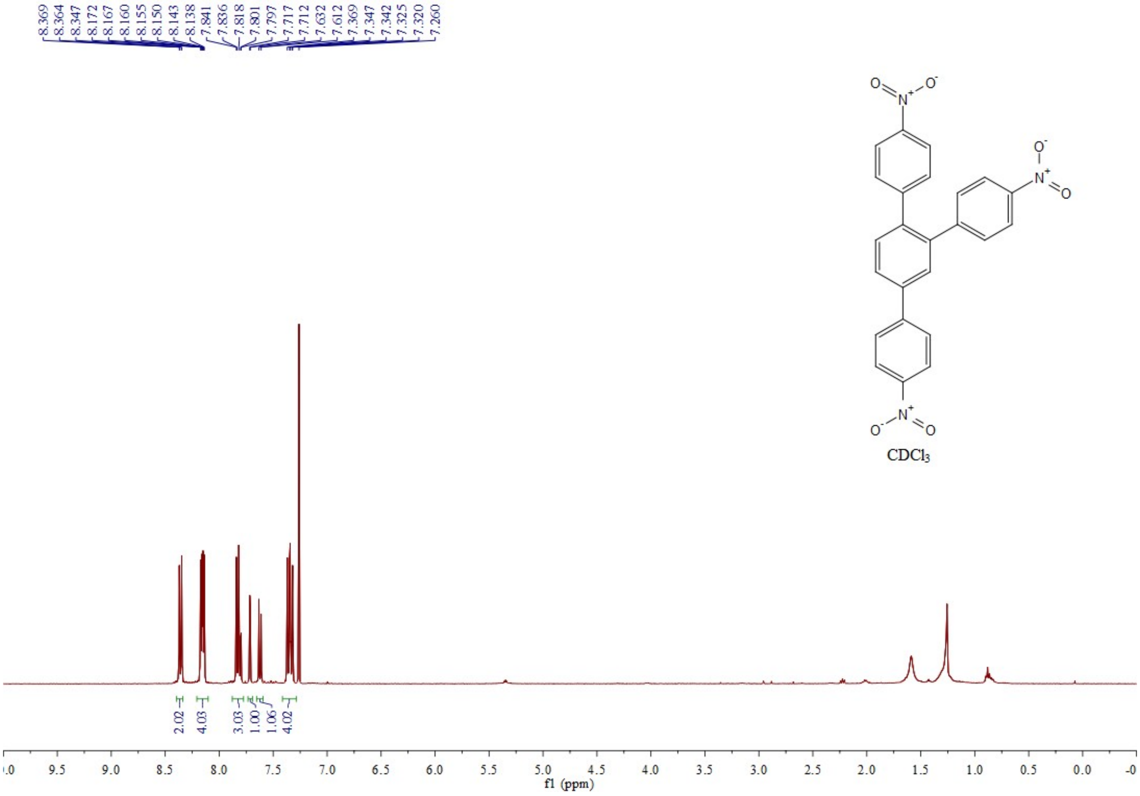


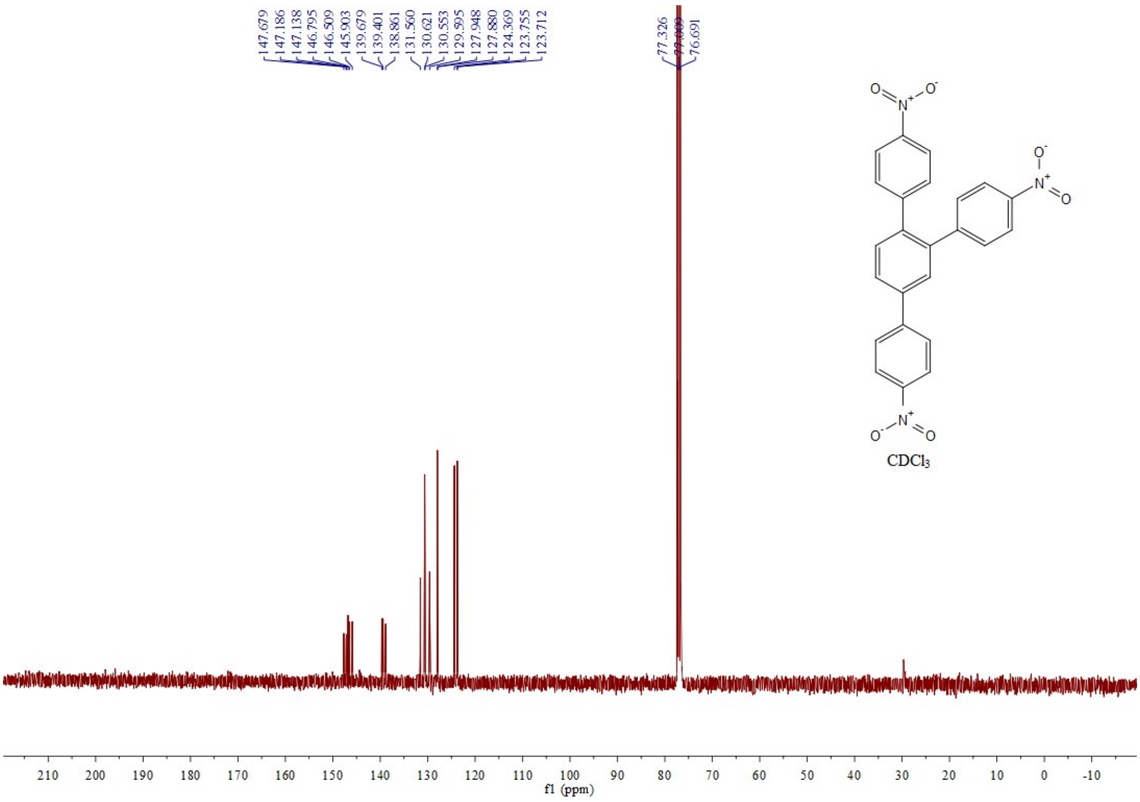
1,2,4-Tris(4-formylphenyl)benzene (2i, Table 2).



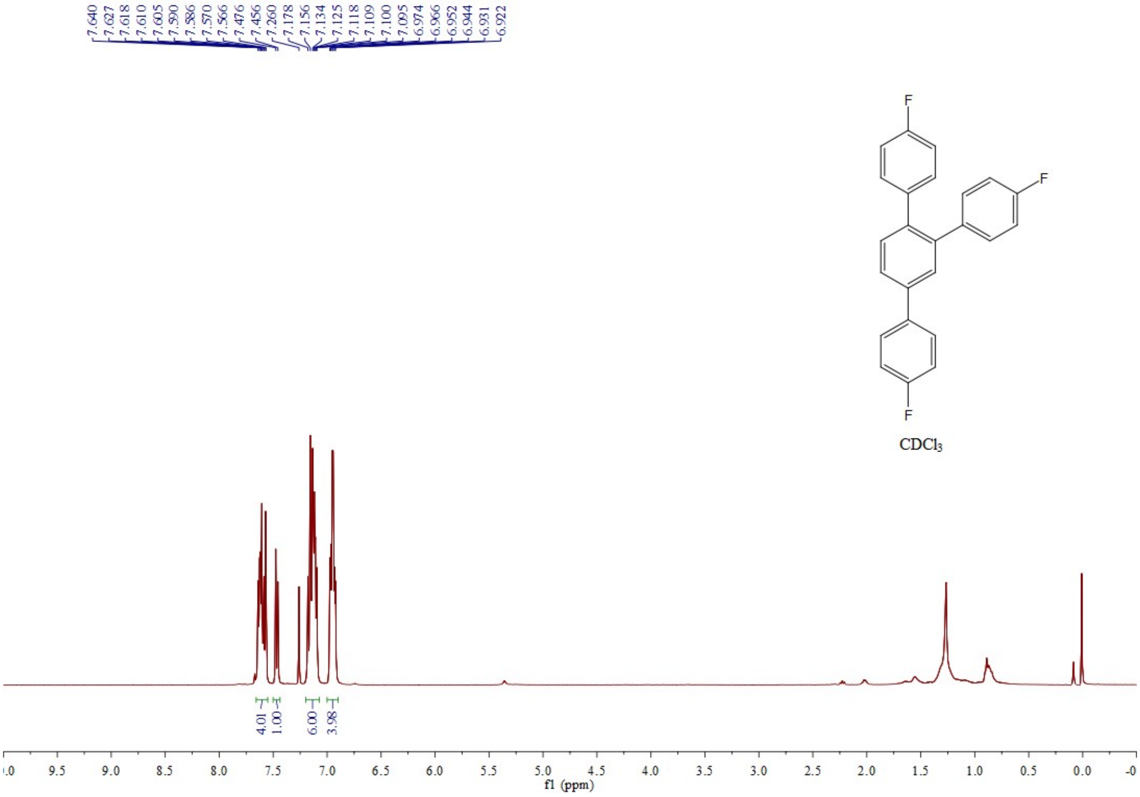


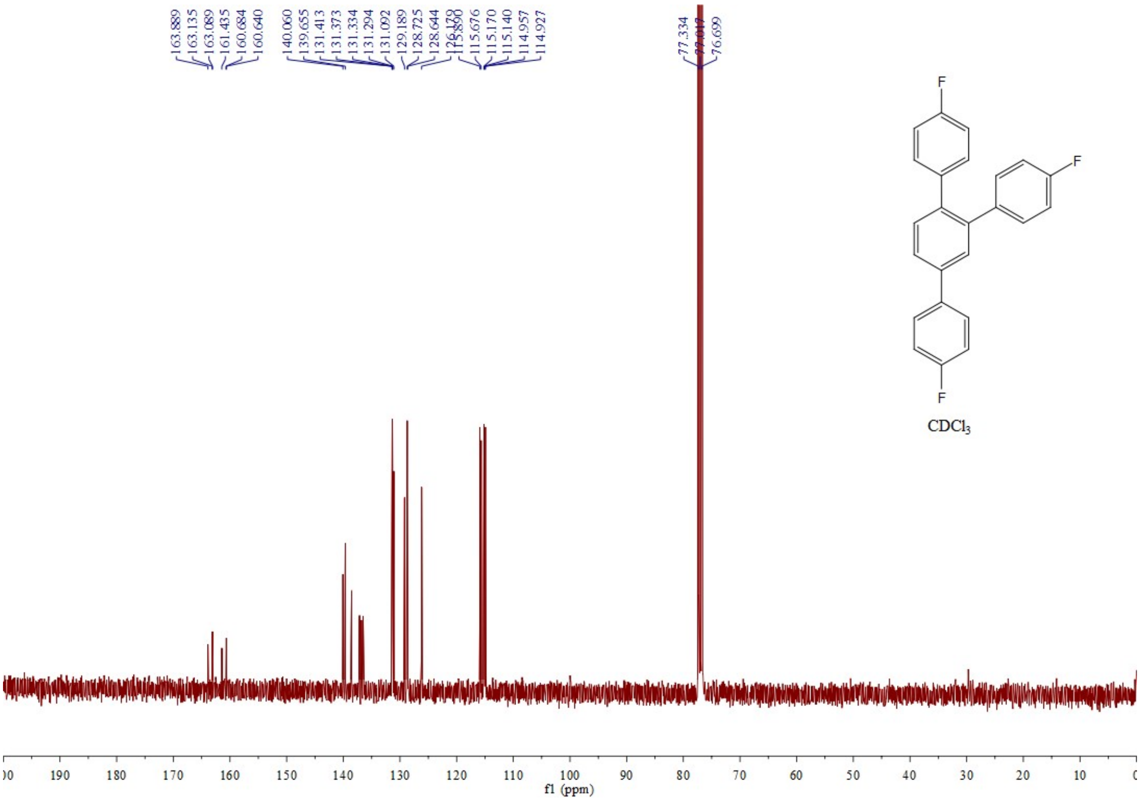
1,2,4-Tris(4-nitrophenyl)benzene (2j, Table 2).



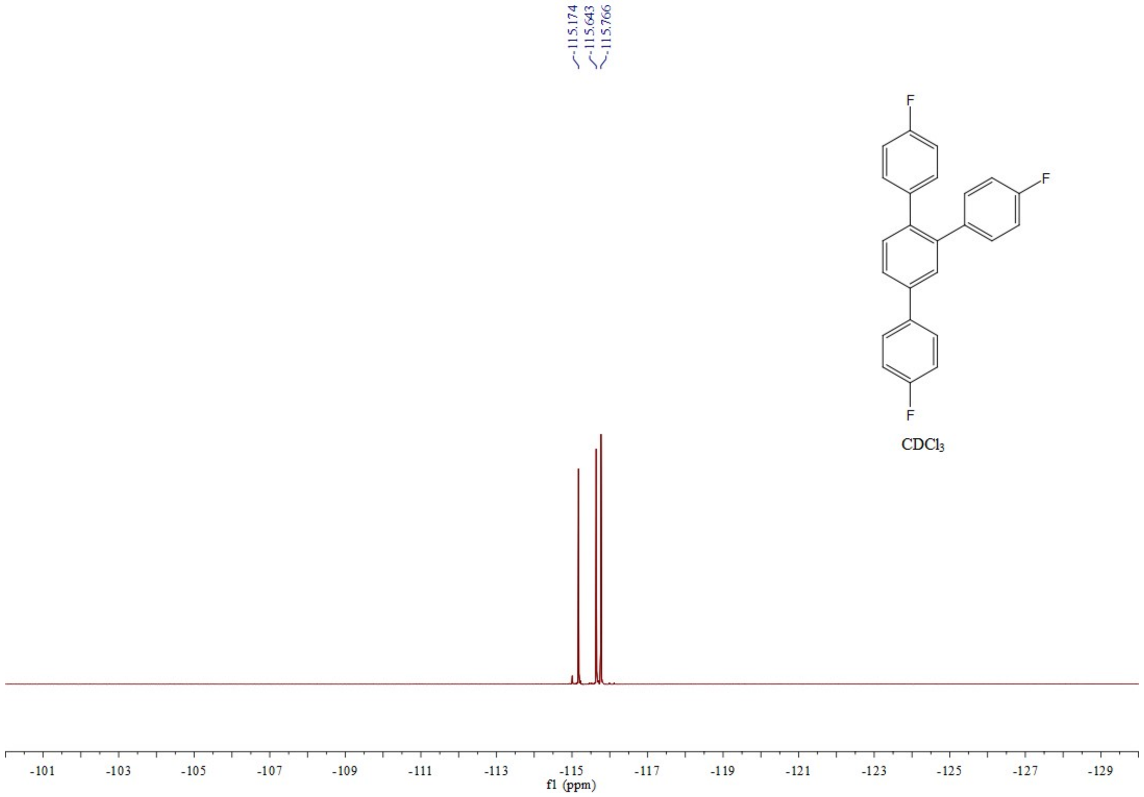


1,2,4-Tris(4-fluorophenyl)benzene (2k, Table 2).

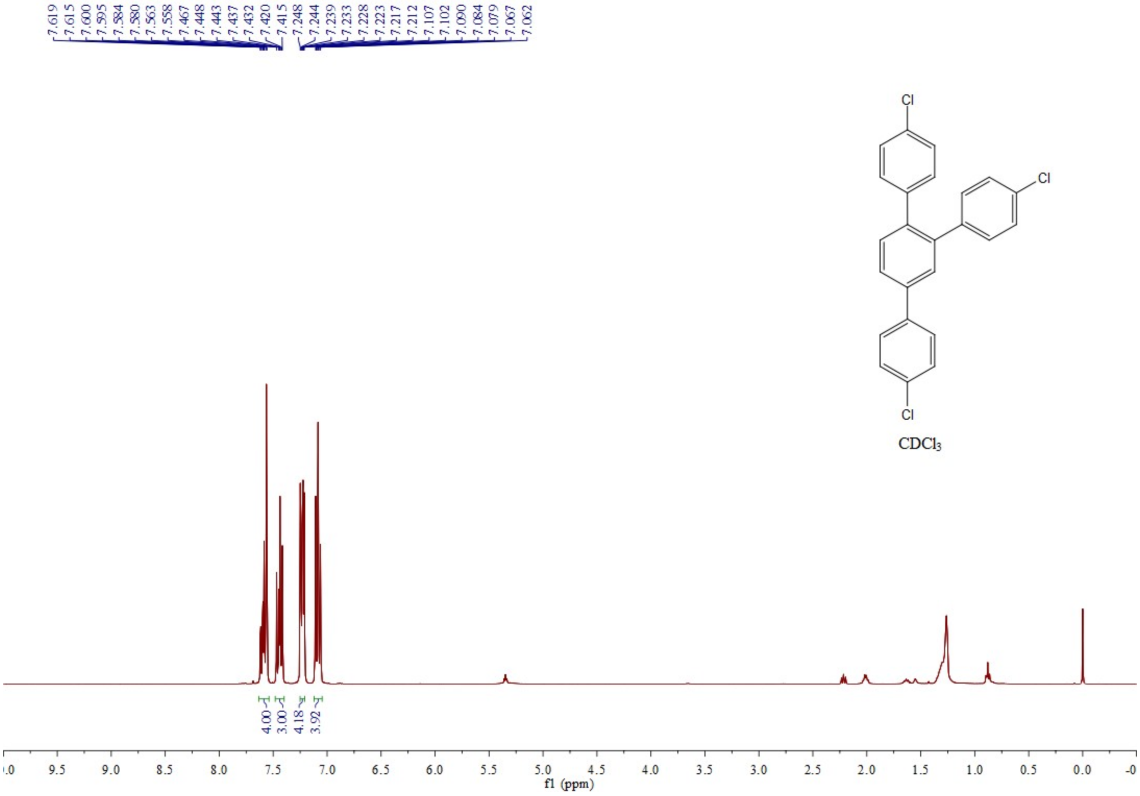


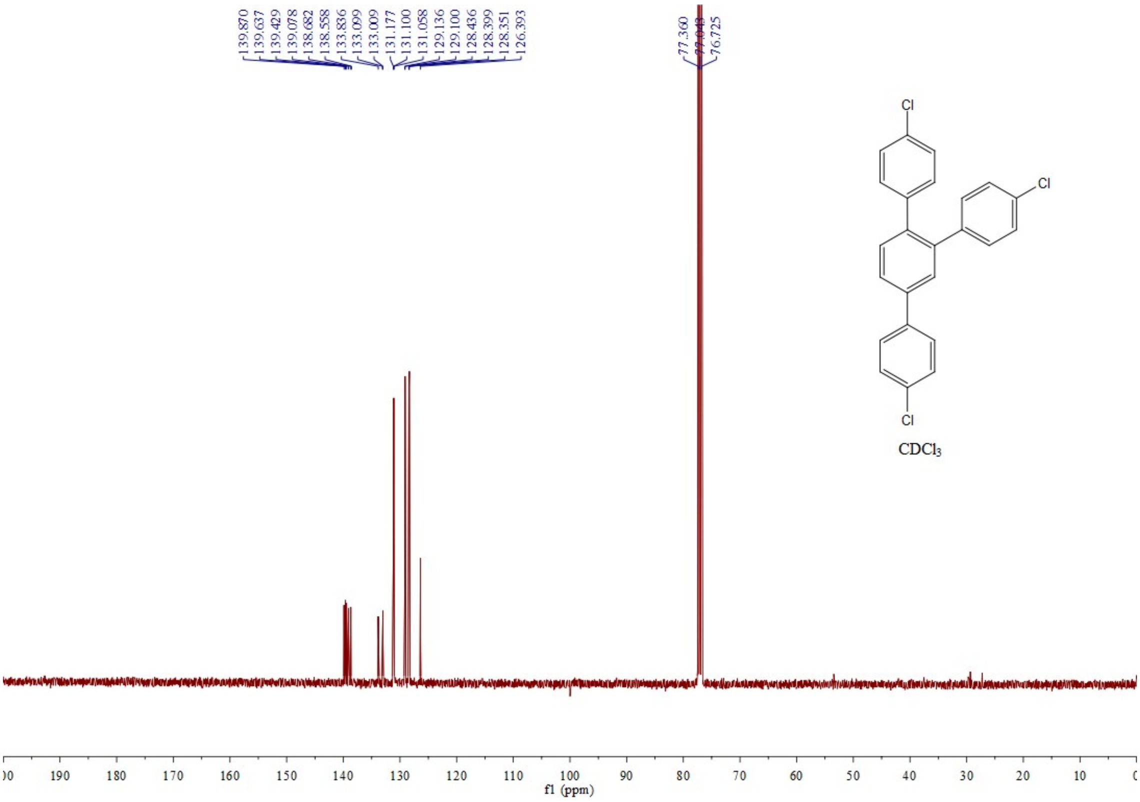


^{19}F NMR spectra of 1,2,4-Tris(4-fluorophenyl)benzene (2k, Table 2).

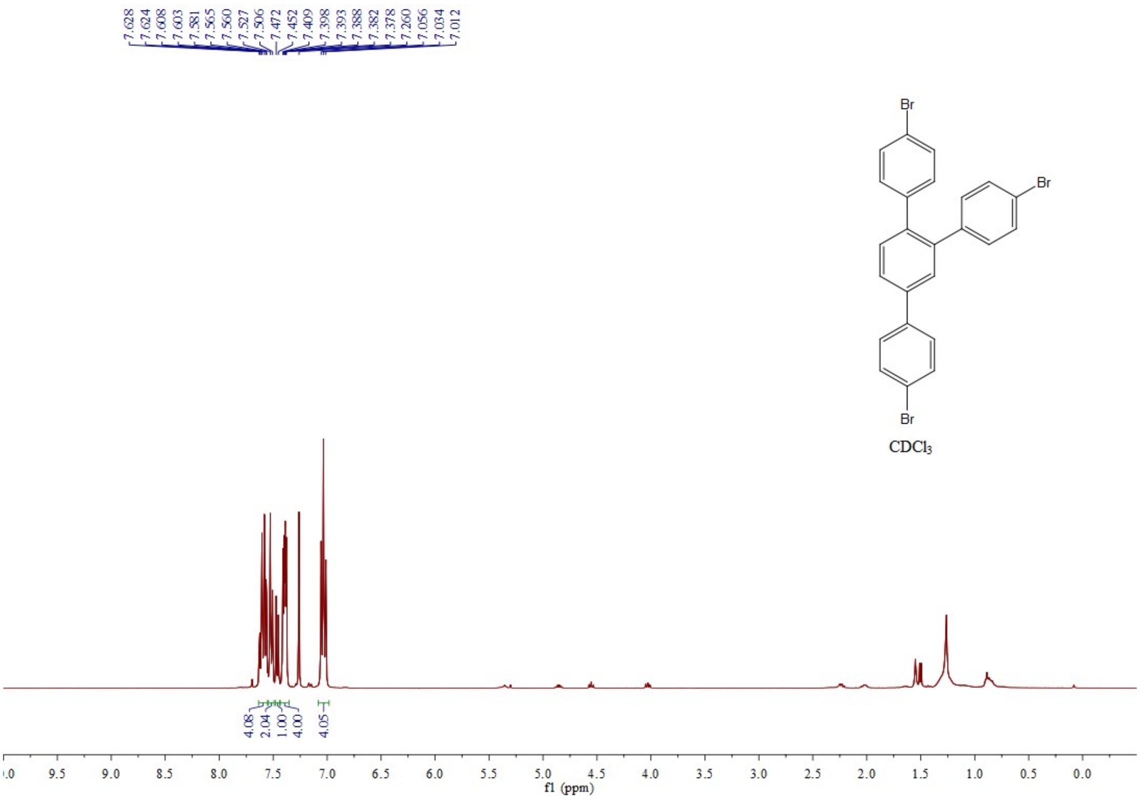


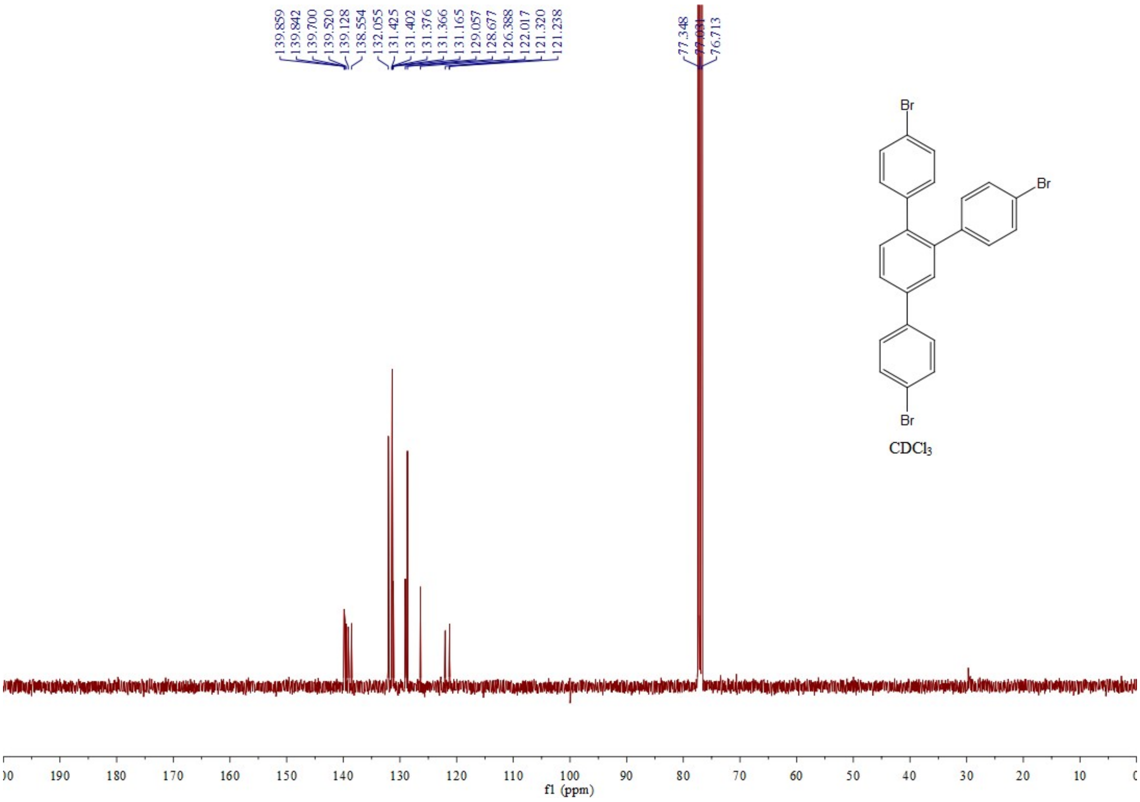
1,2,4-Tris(4-chlorophenyl)benzene (2l, Table 2).



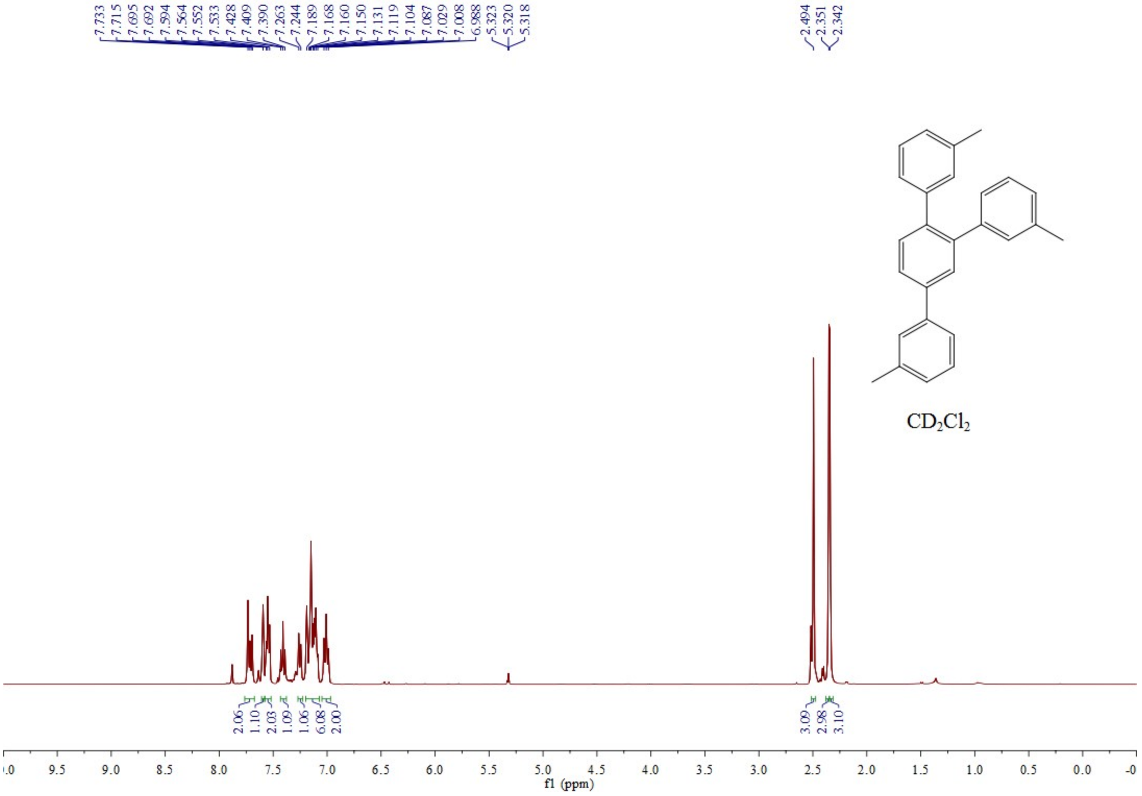


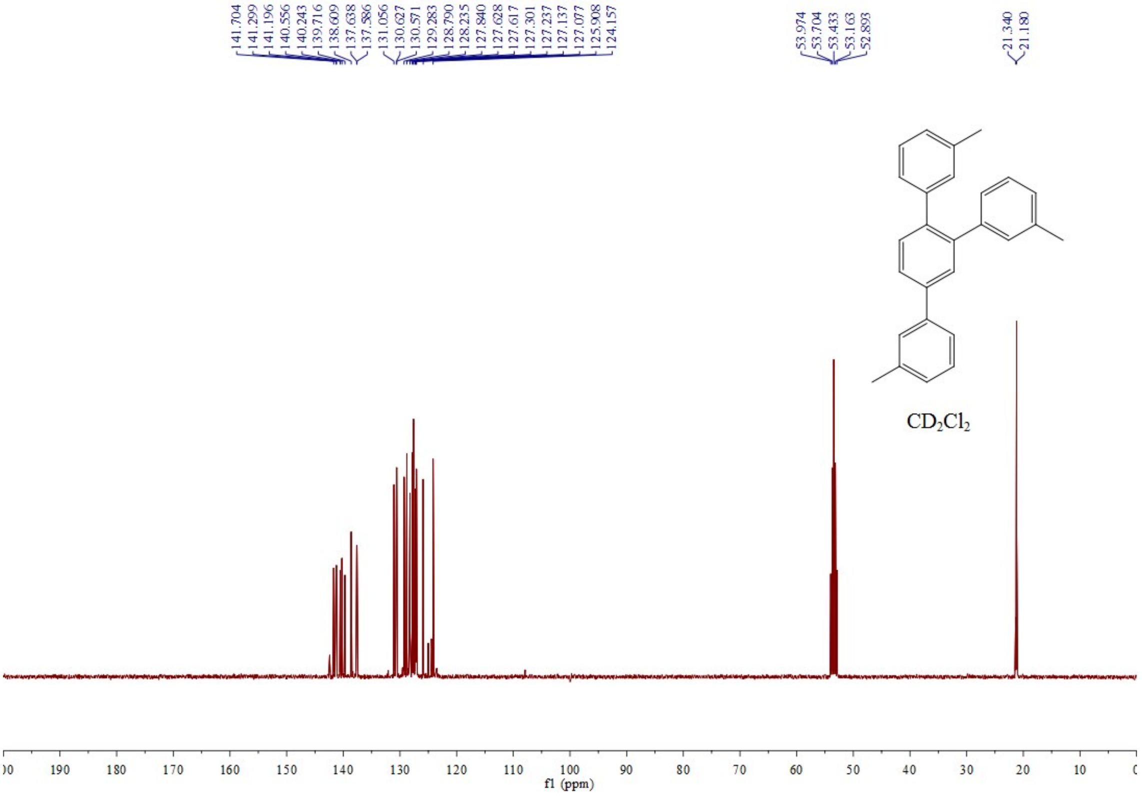
1,2,4-Tris(4-bromophenyl)benzene (2m, Table 2).



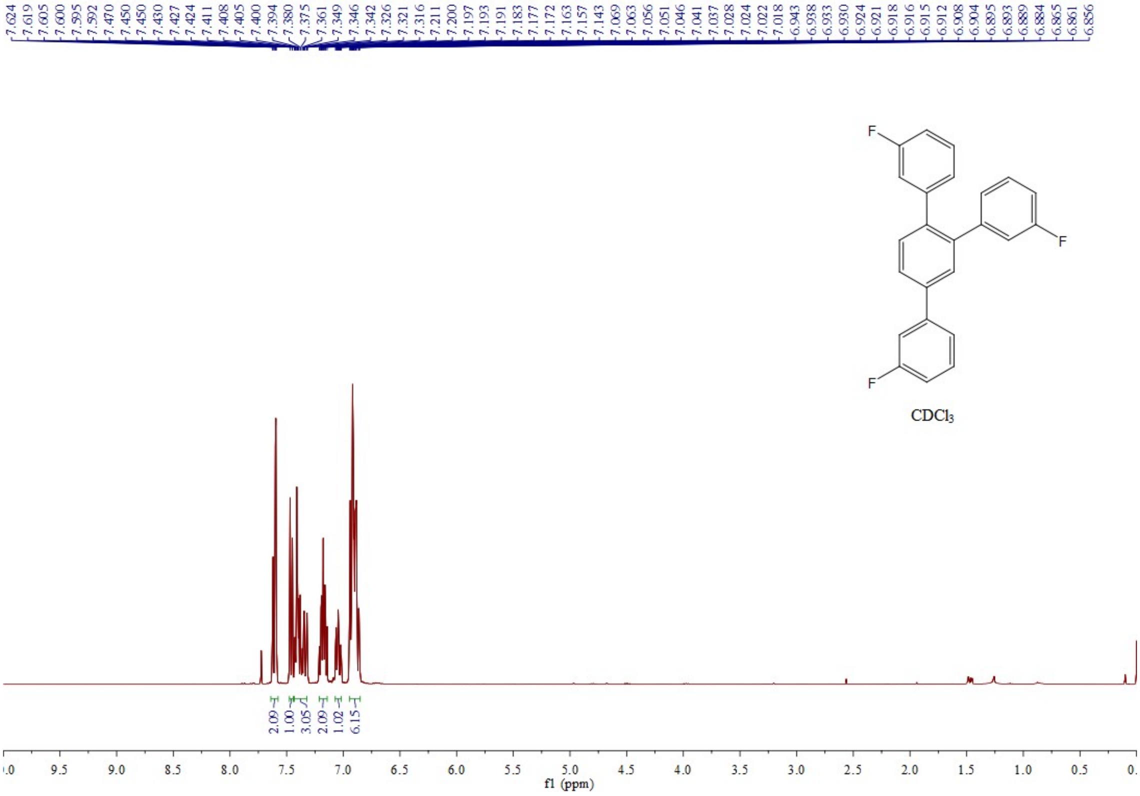


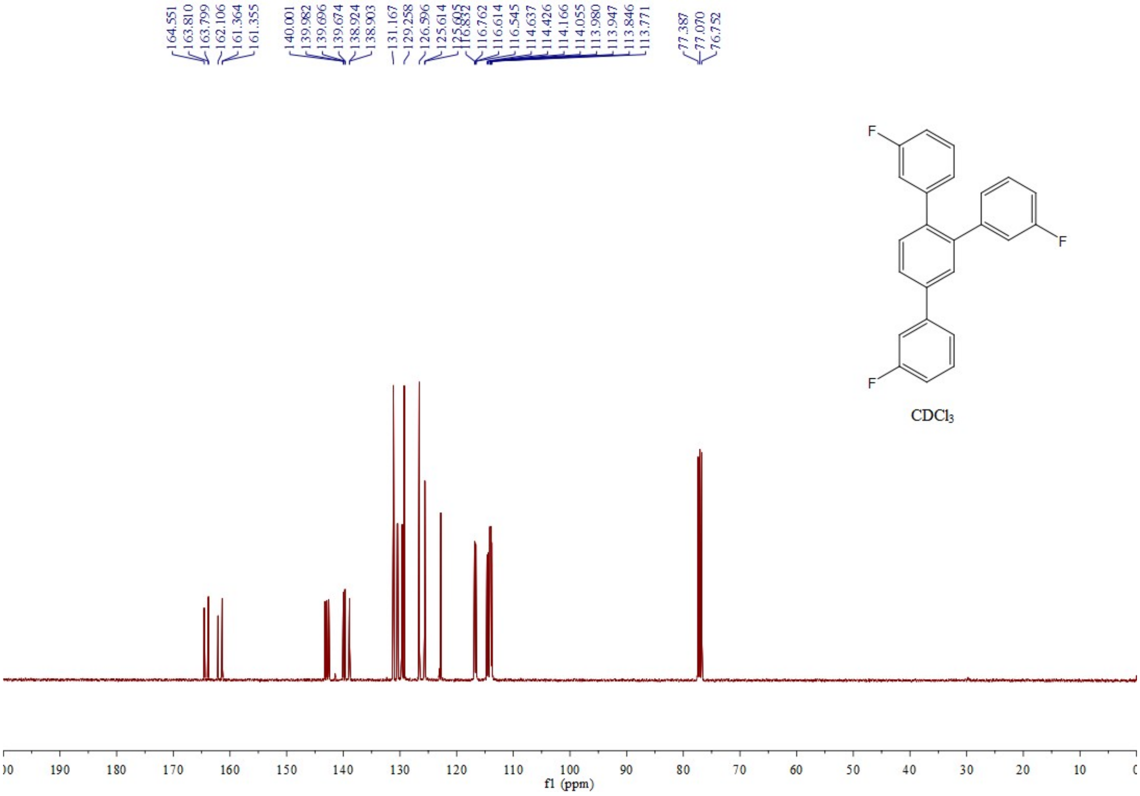
1,2,4-Tris(3-methylphenyl)benzene (2n, Table 2).



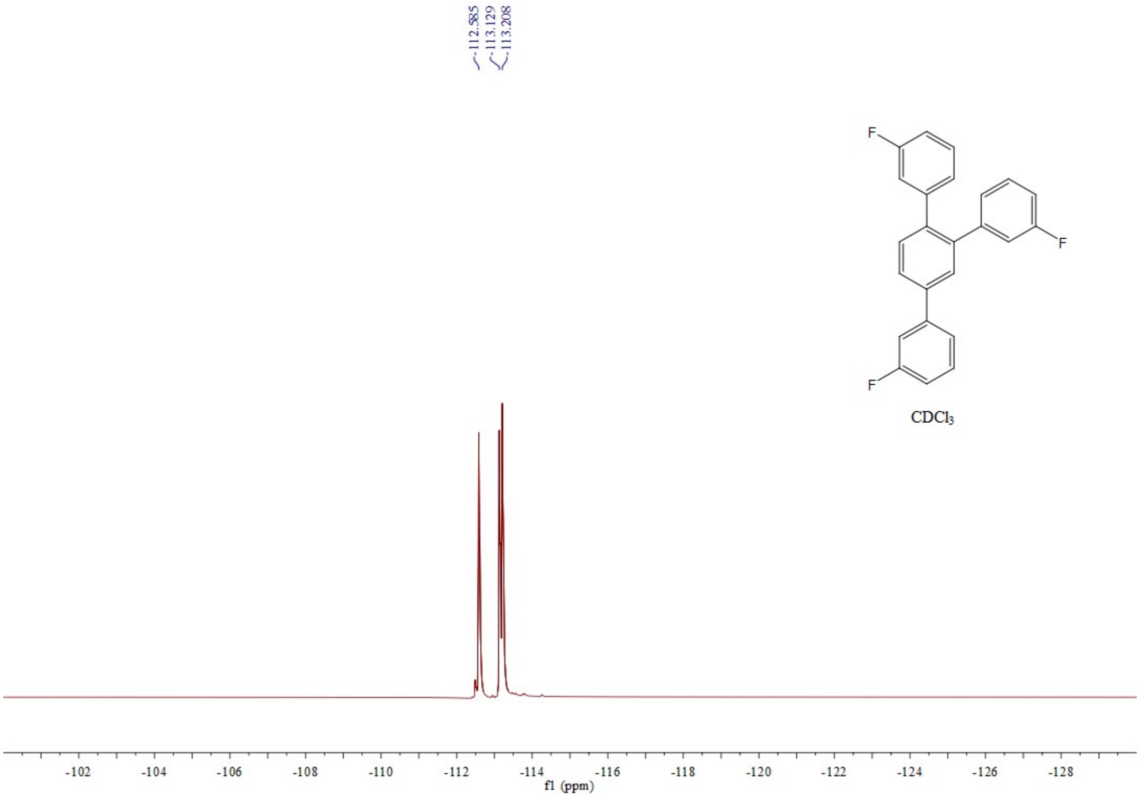


1,2,4-Tris(3-fluorophenyl)benzene (2o, Table 2).

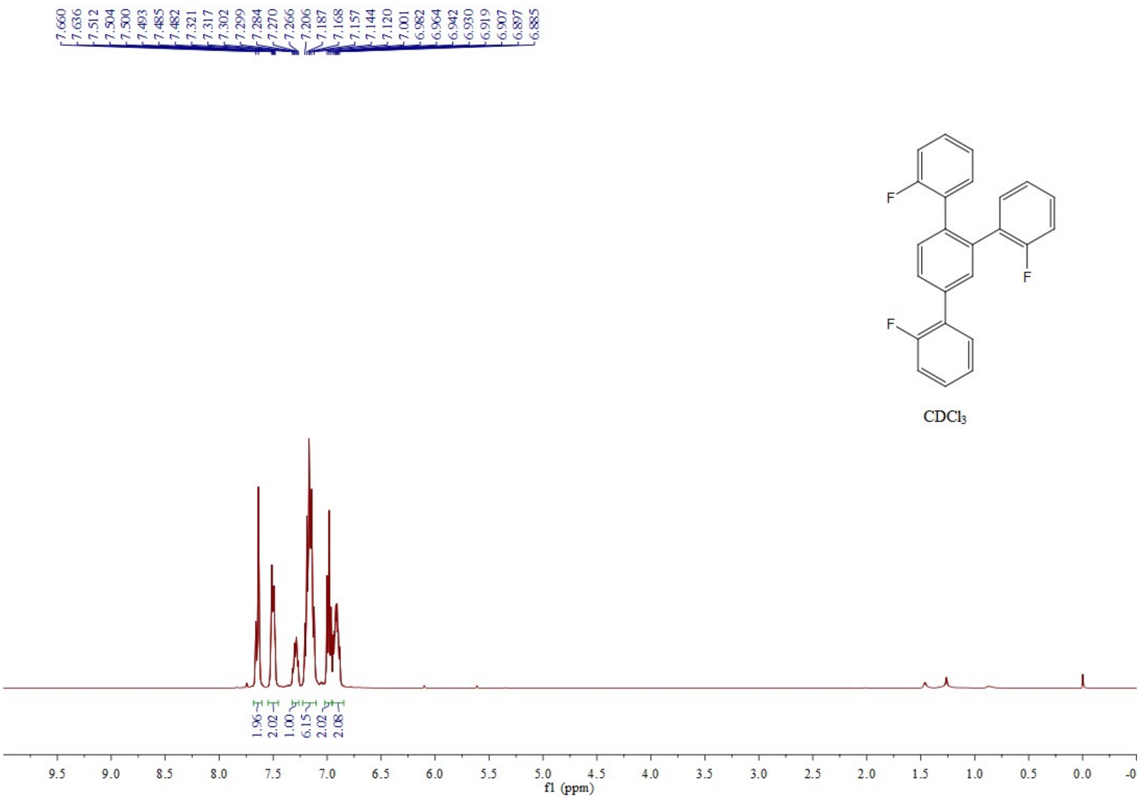


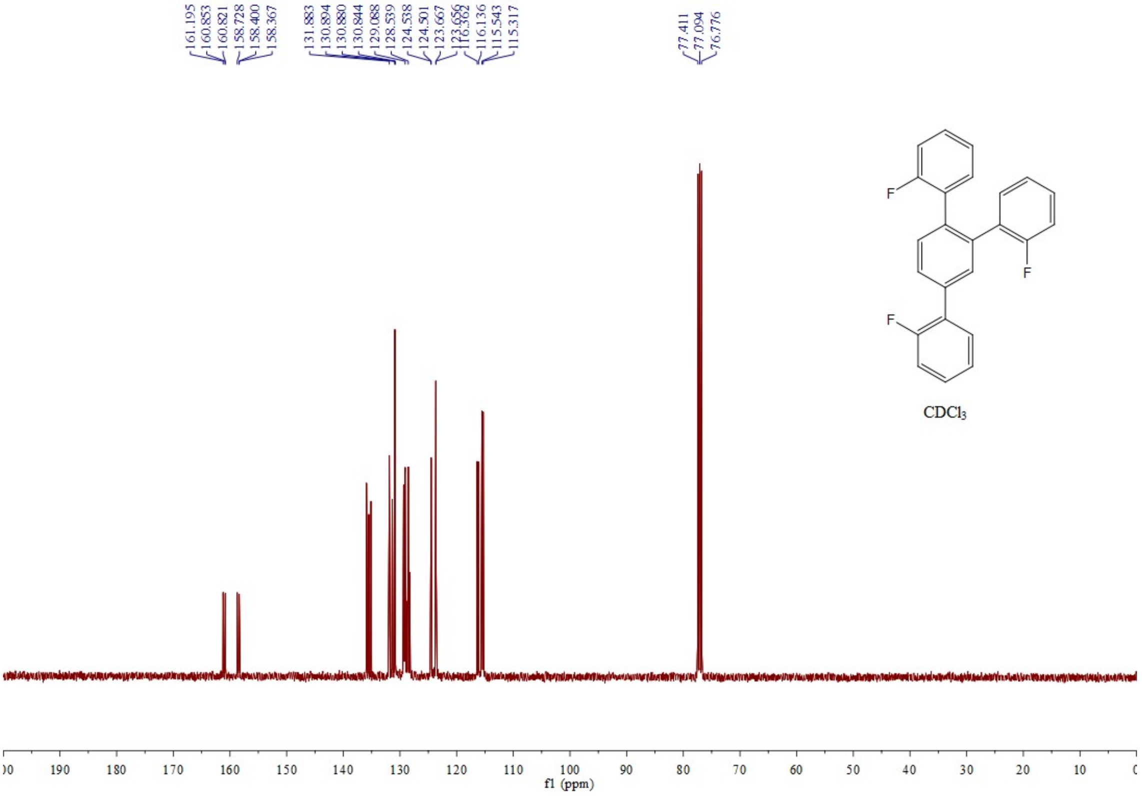


^{19}F NMR spectra of 1,2,4-Tris(3-fluorophenyl)benzene (2o, Table 2).

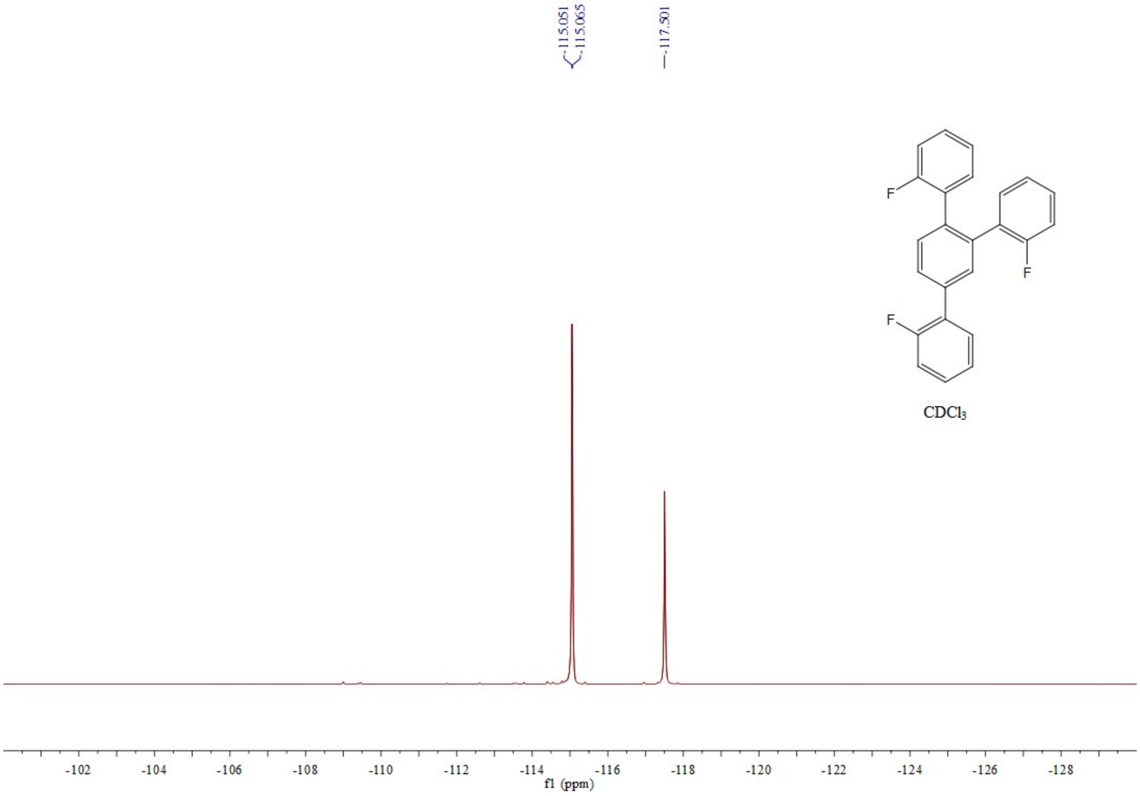


1,2,4-Tris(2-fluorophenyl)benzene (2q, Table 2).

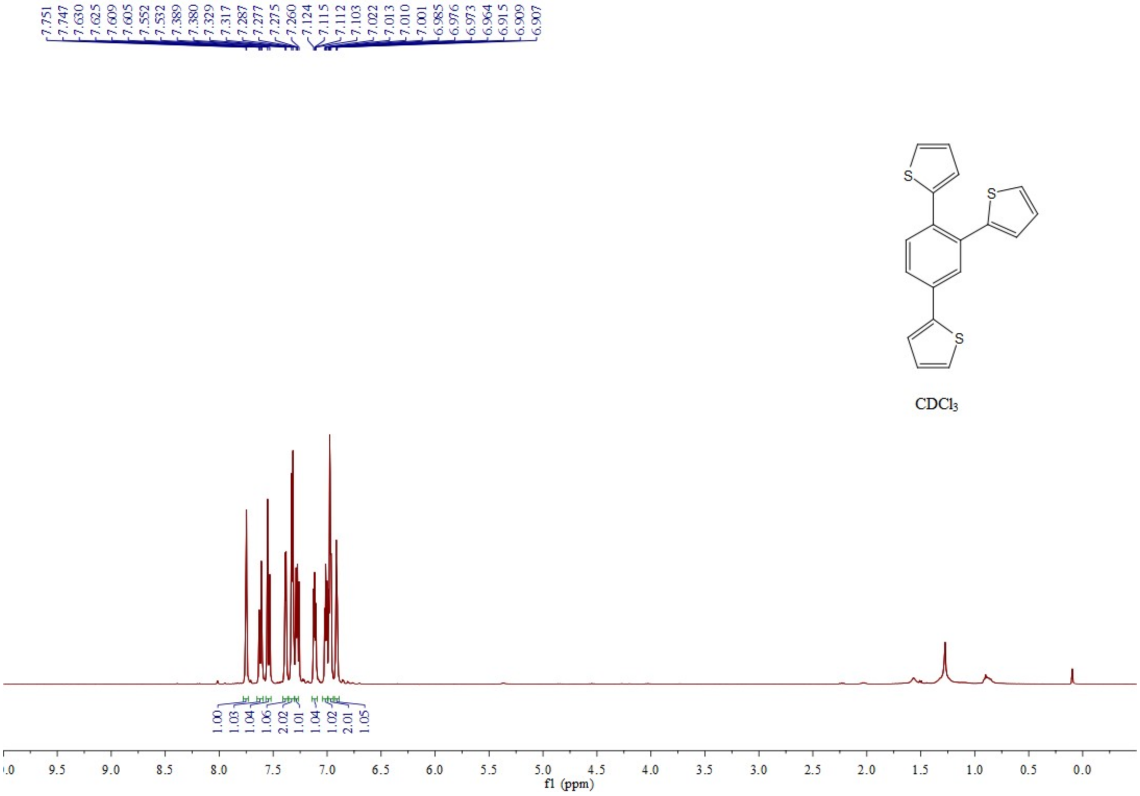


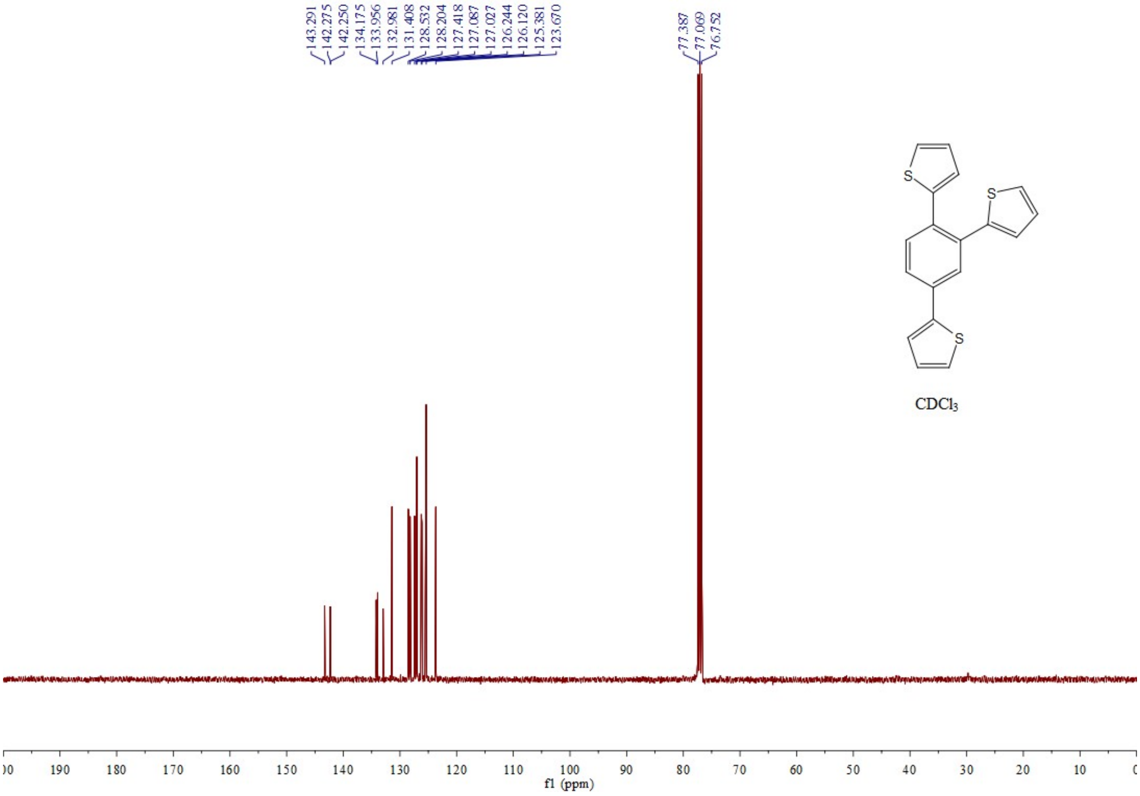


^{19}F NMR spectra of 1,2,4-Tris(2-fluorophenyl)benzene (2q, Table 2).

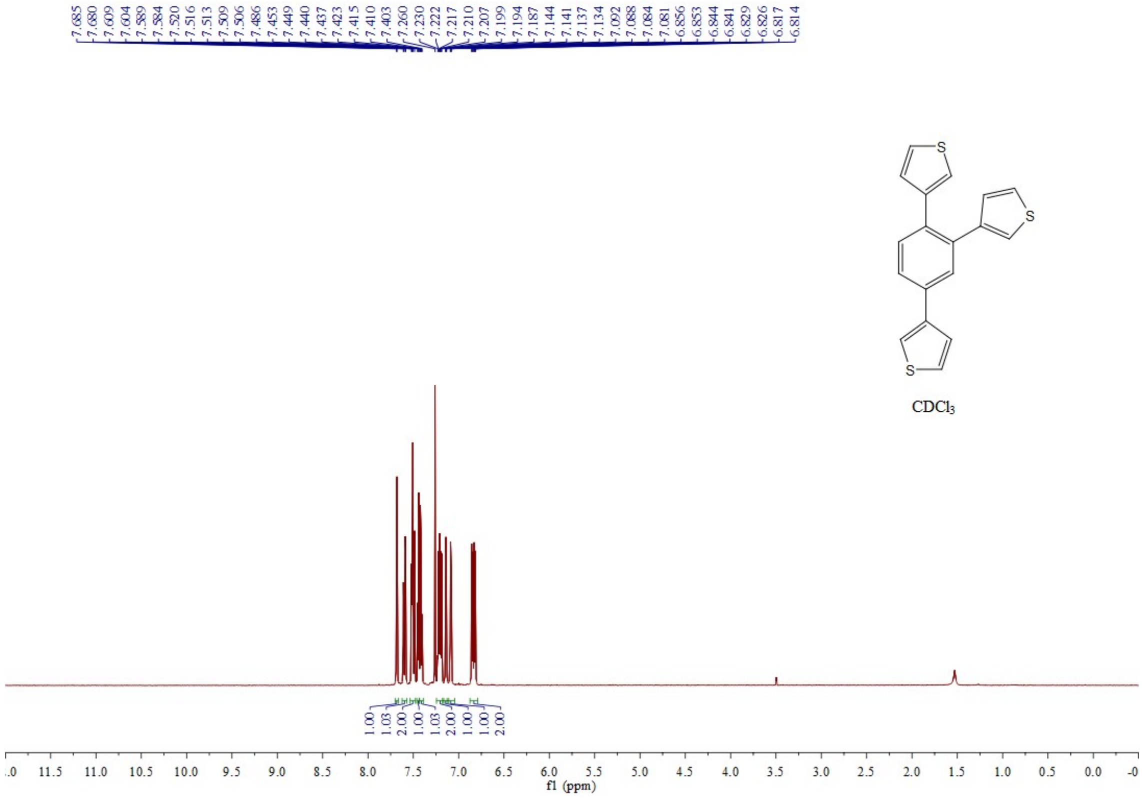


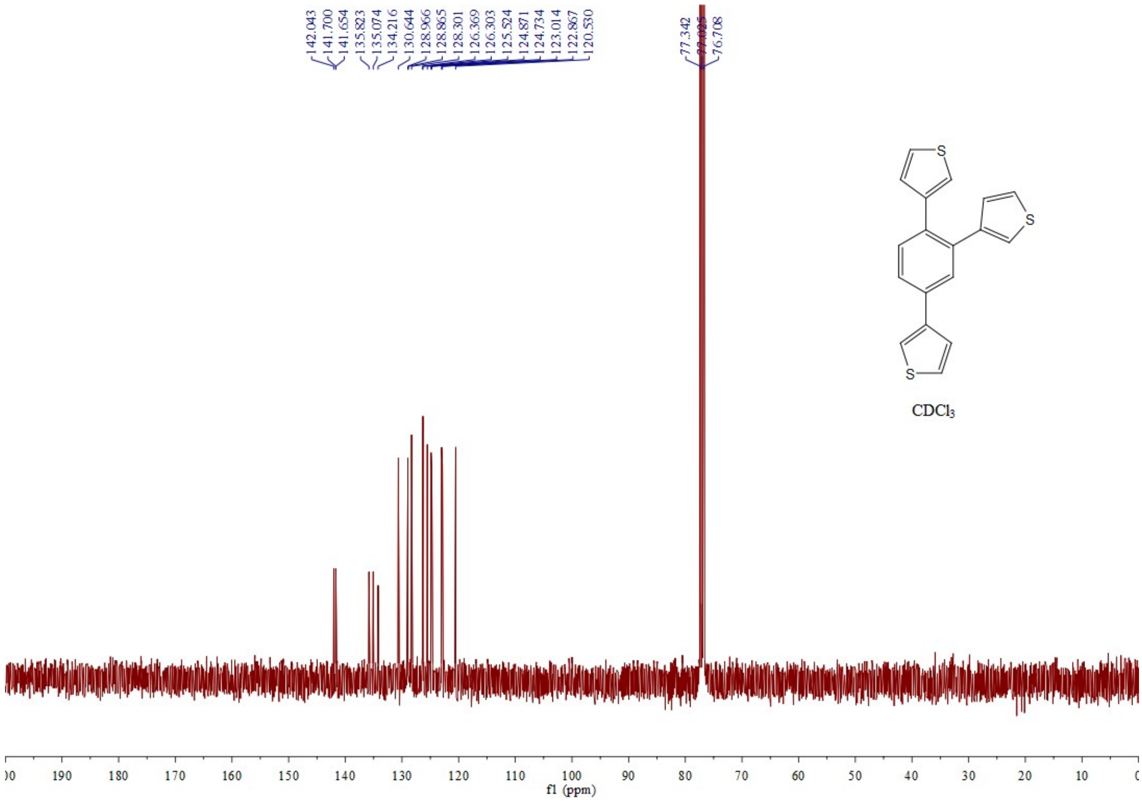
1,2,4-Tris(2-thienyl)benzene (2r, Table 2).



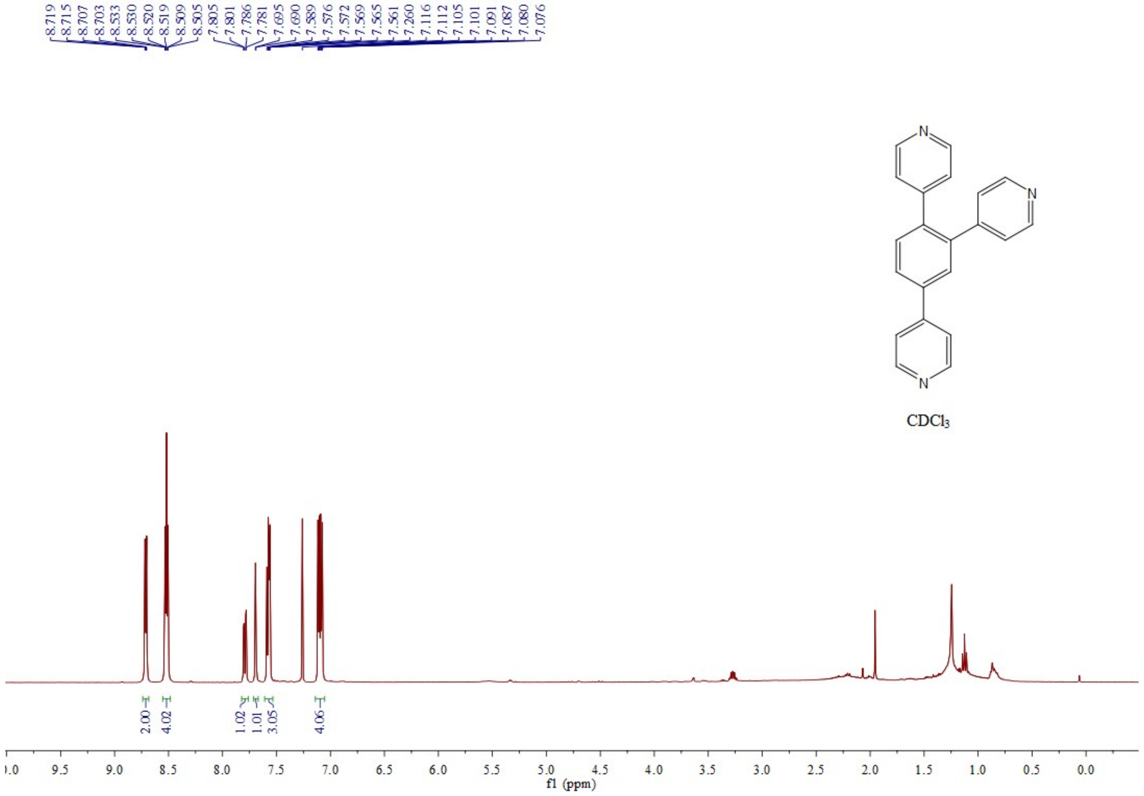


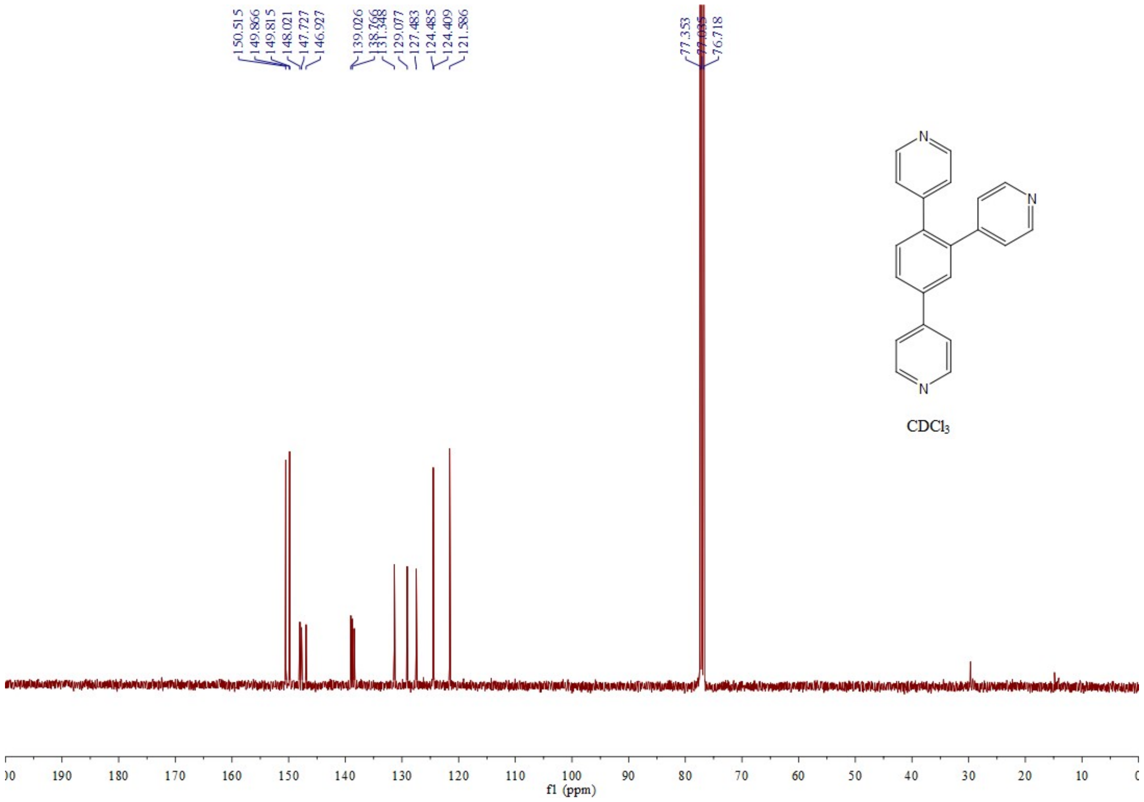
1,2,4-Tris(3-thienyl)benzene (2s, Table 2).



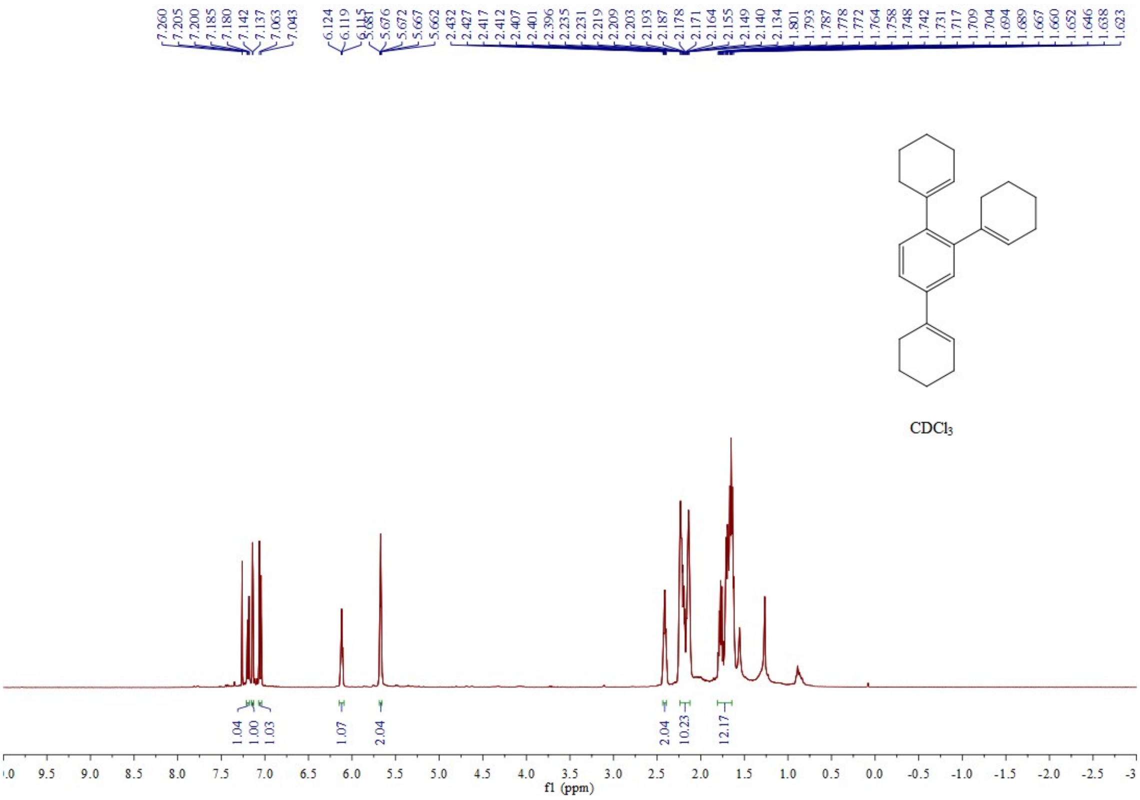


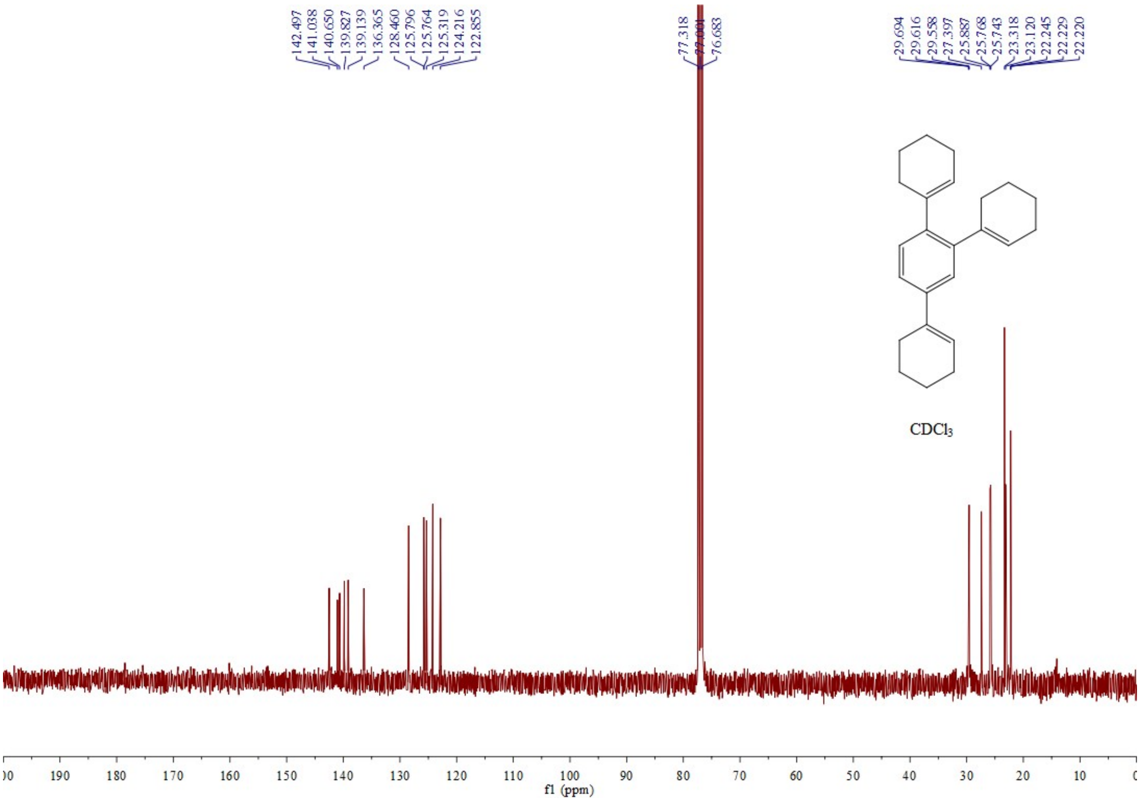
1,2,4-Tris(4-pyridyl)benzene (2t, Table 2).



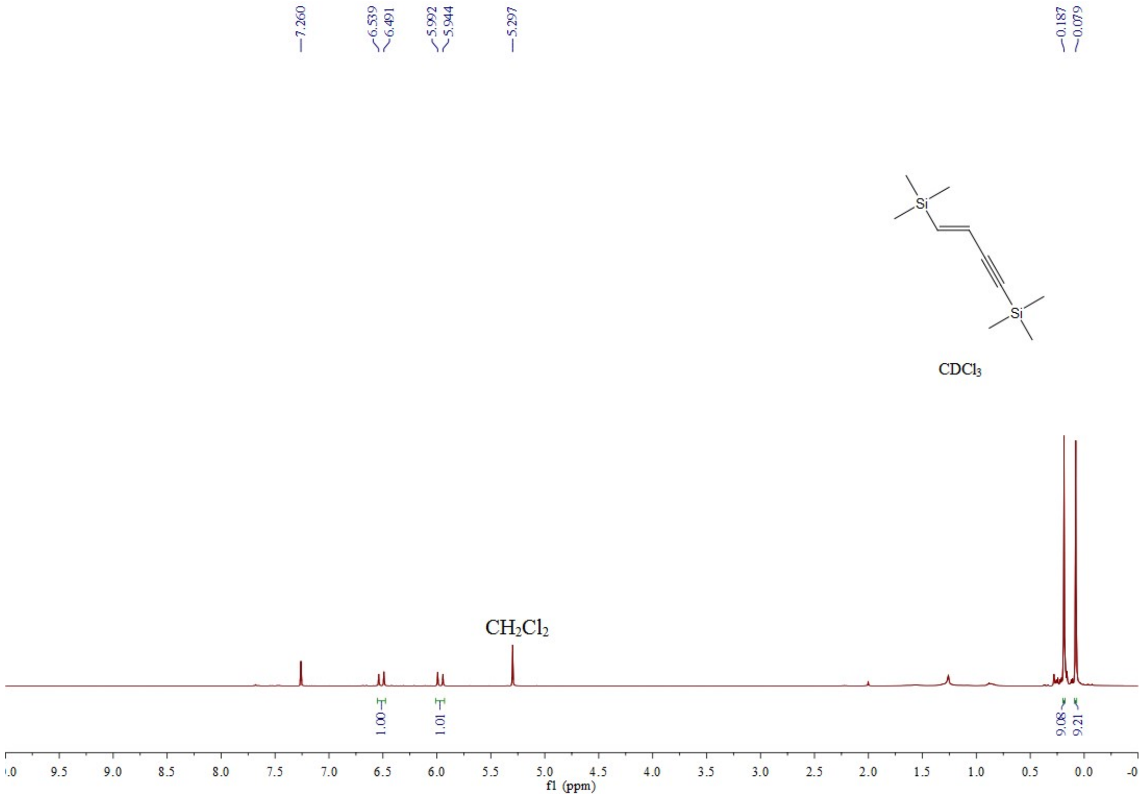


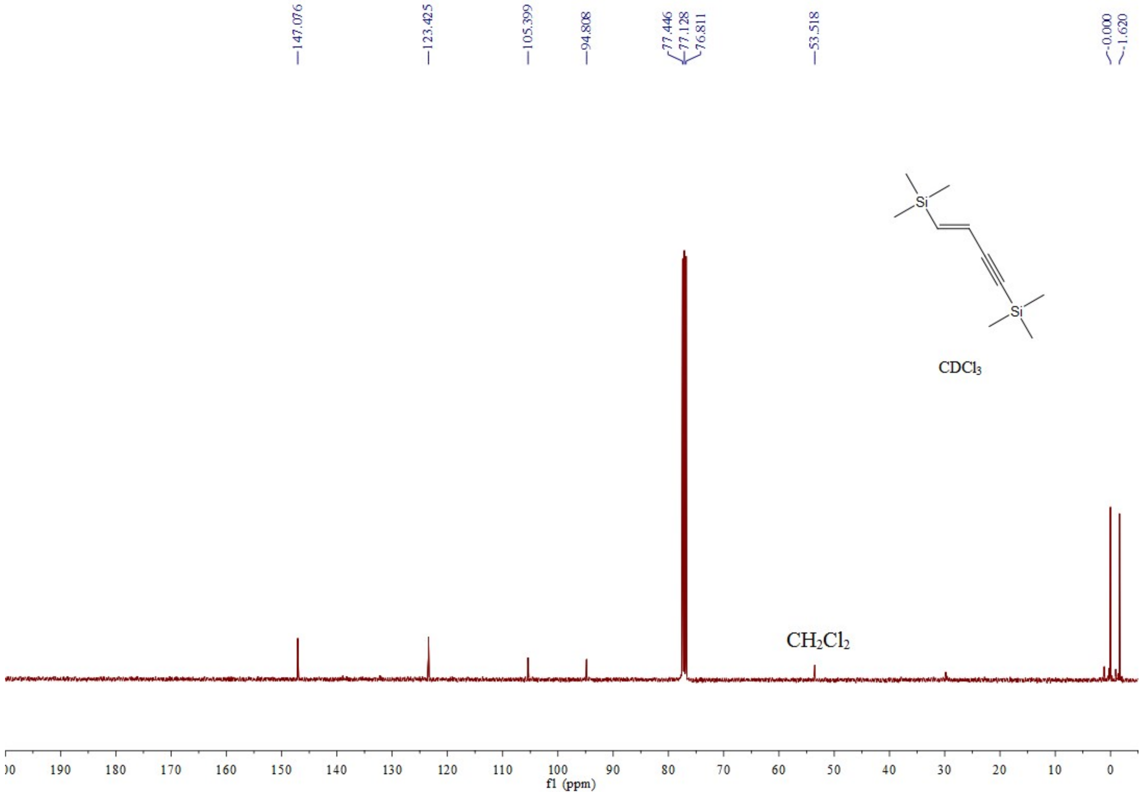
1,2,4-Tris(1-cyclohexenyl)benzene (2w, Table 2).



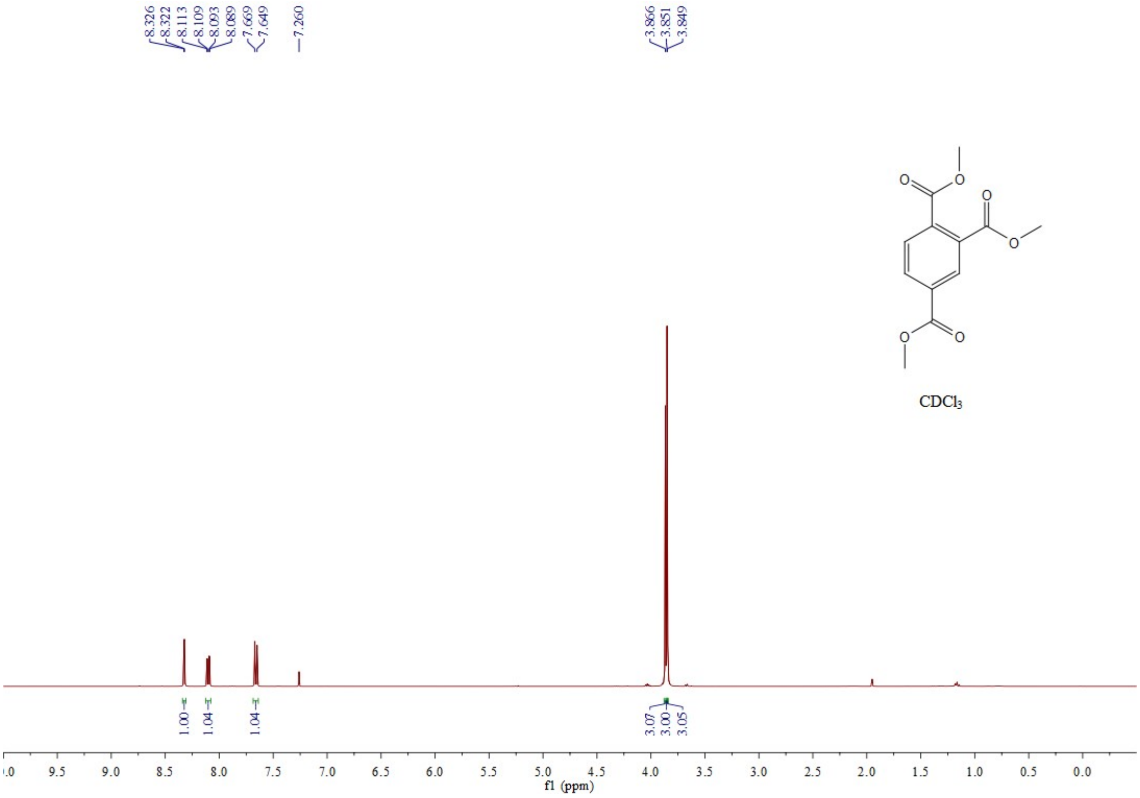


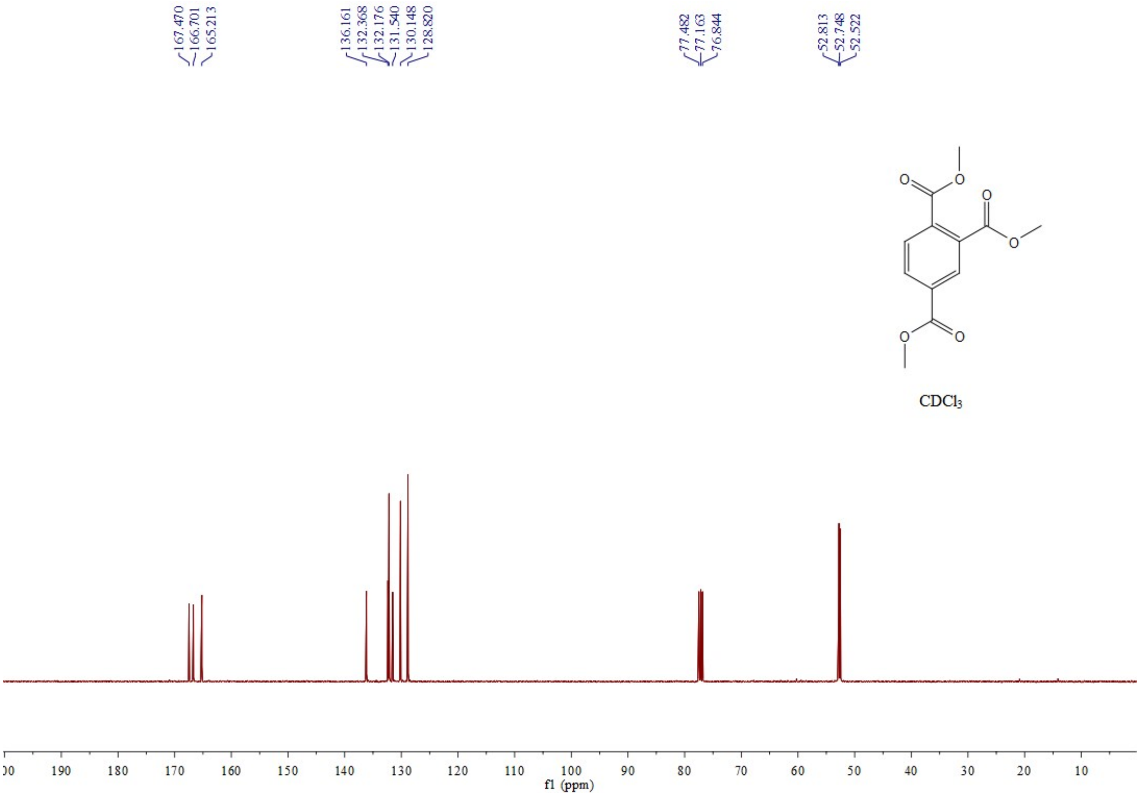
(E)-1,4-bis(trimethylsilyl)but-3-en-1-yne (4xa, Table 2).



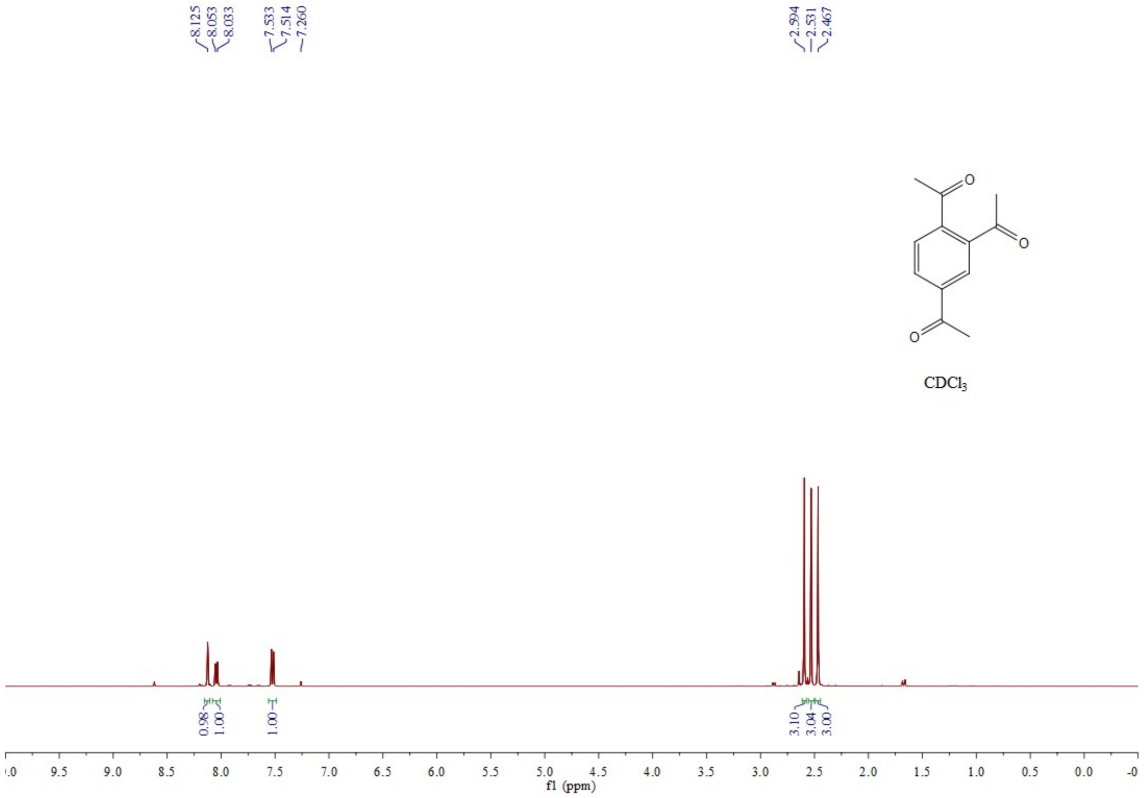


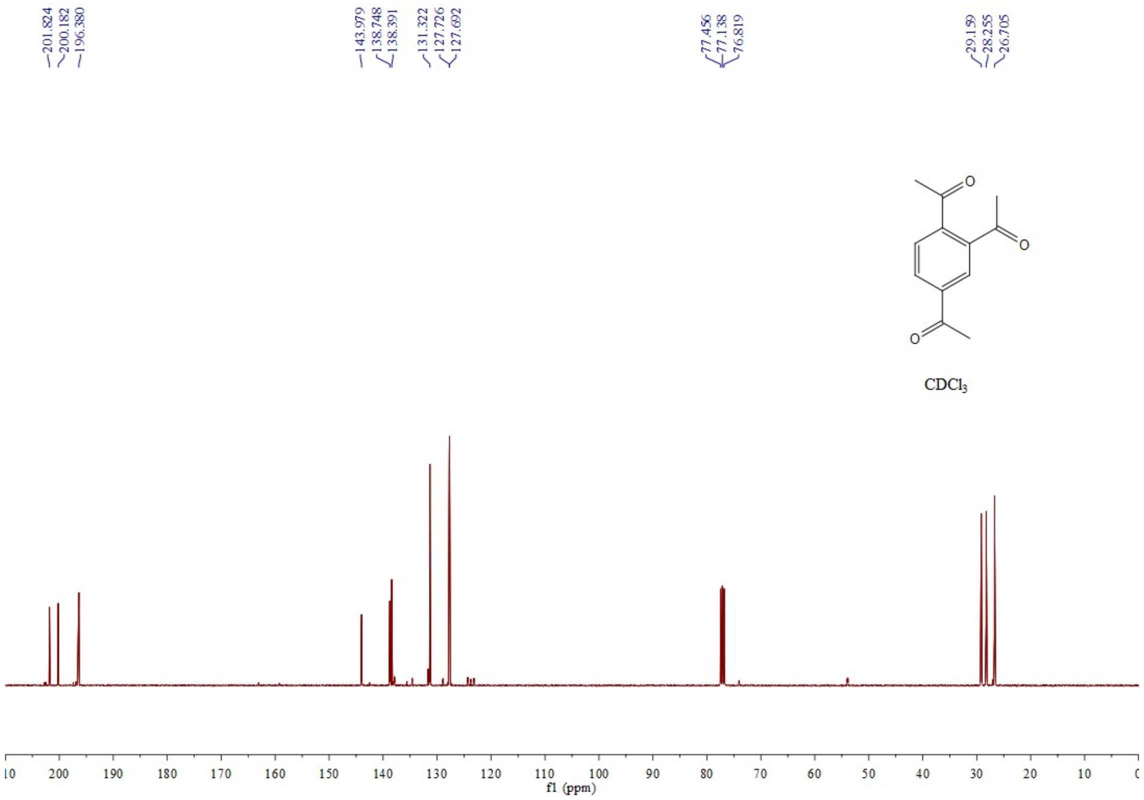
Benzene-1,2,4-tricarboxylic acid trimethyl ester (2y, Table 2).



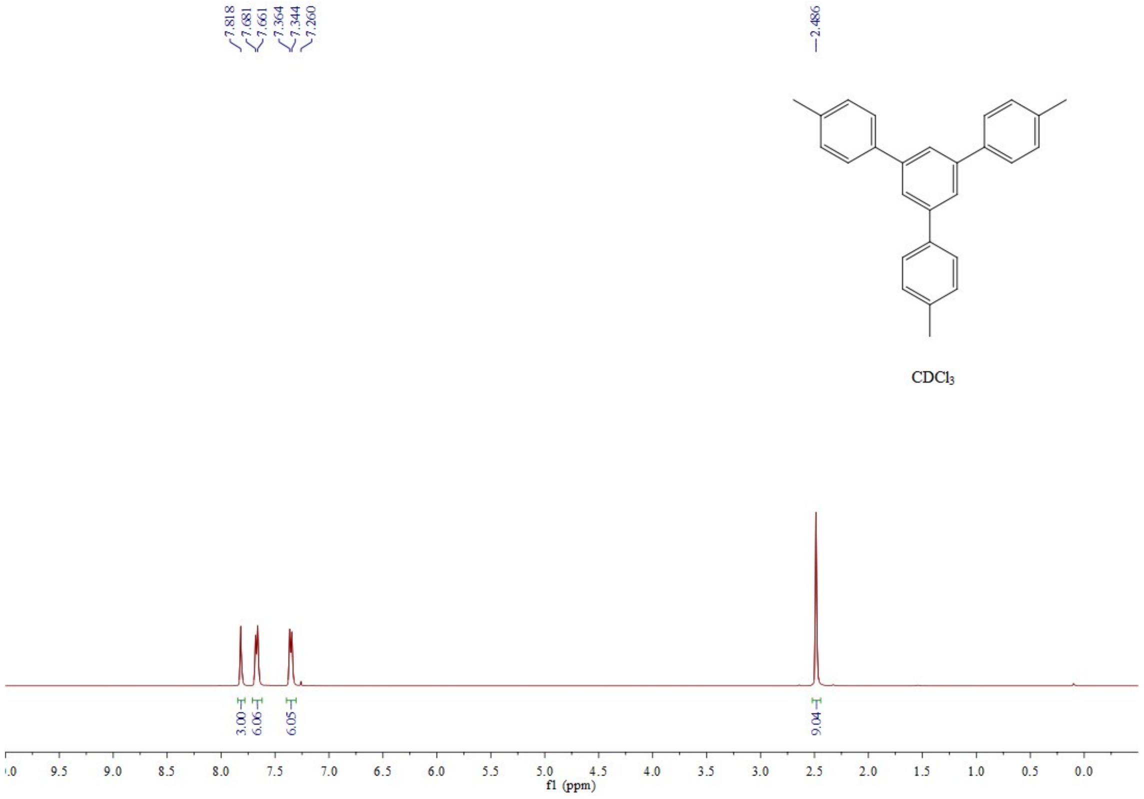


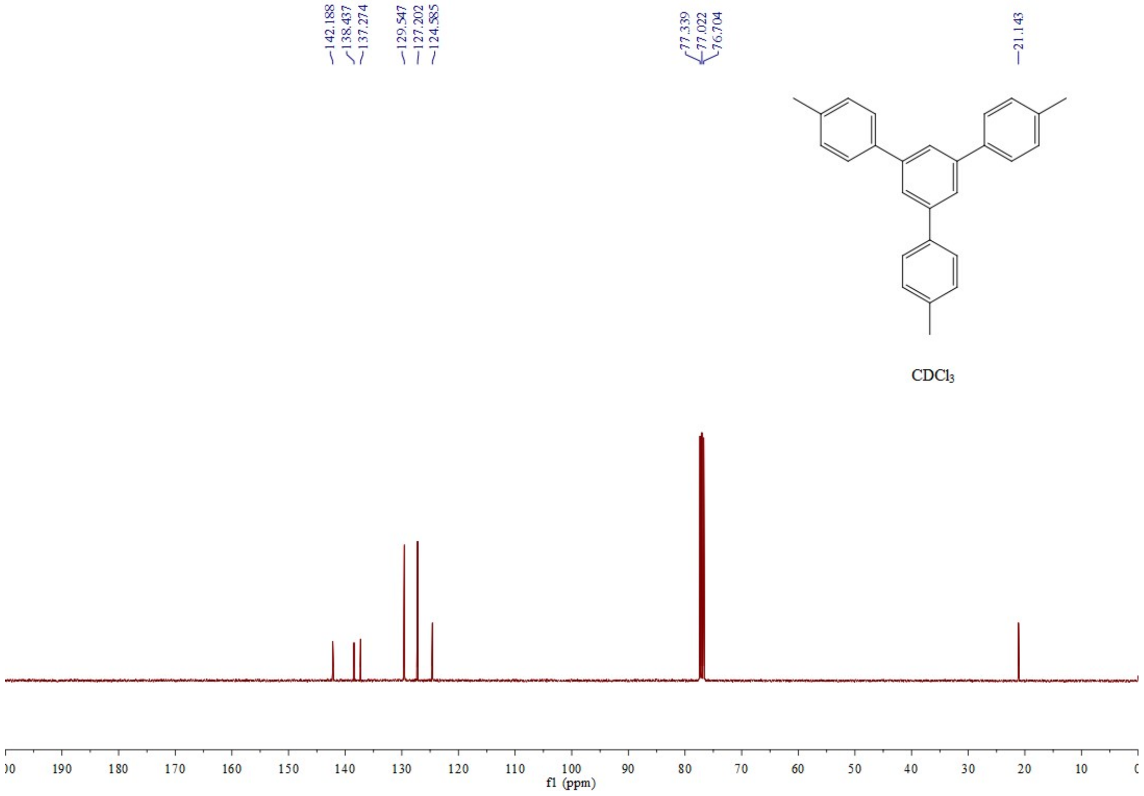
1,2,4-Triacetylphenylbenzene (2z, Table 2).



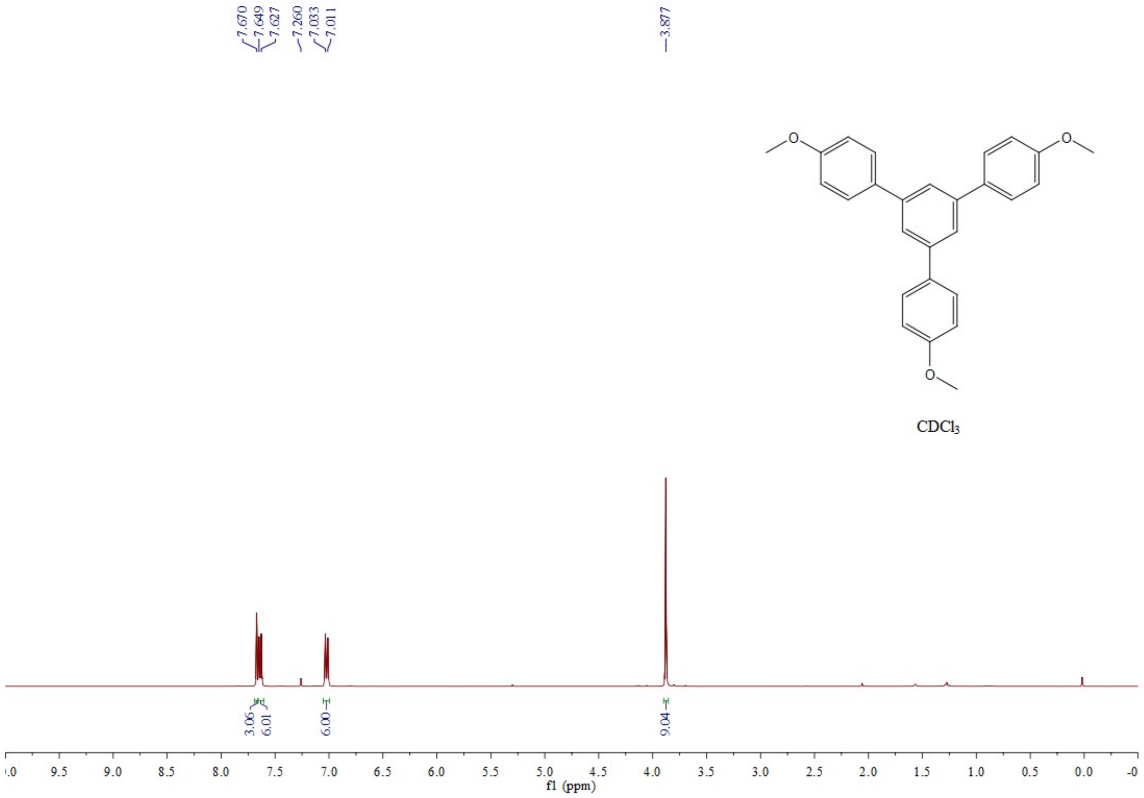


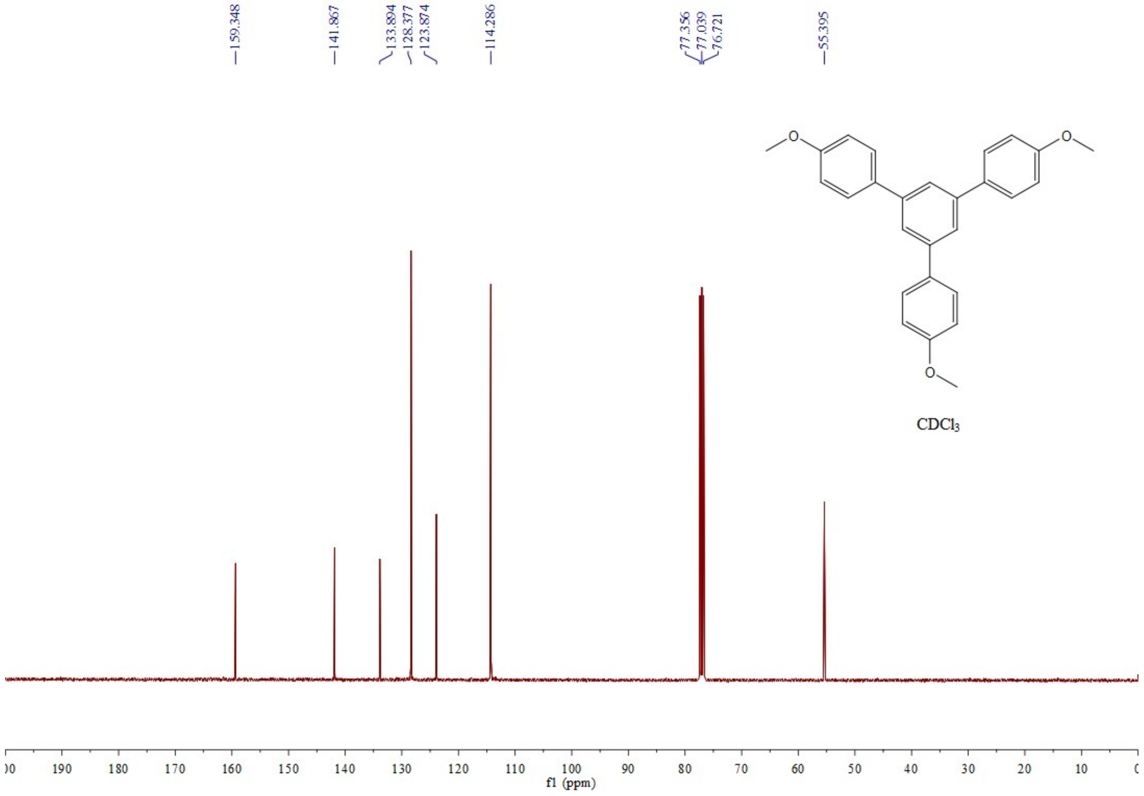
1,3,5-Tris(4-methylphenyl)benzene (3a, Table 3).



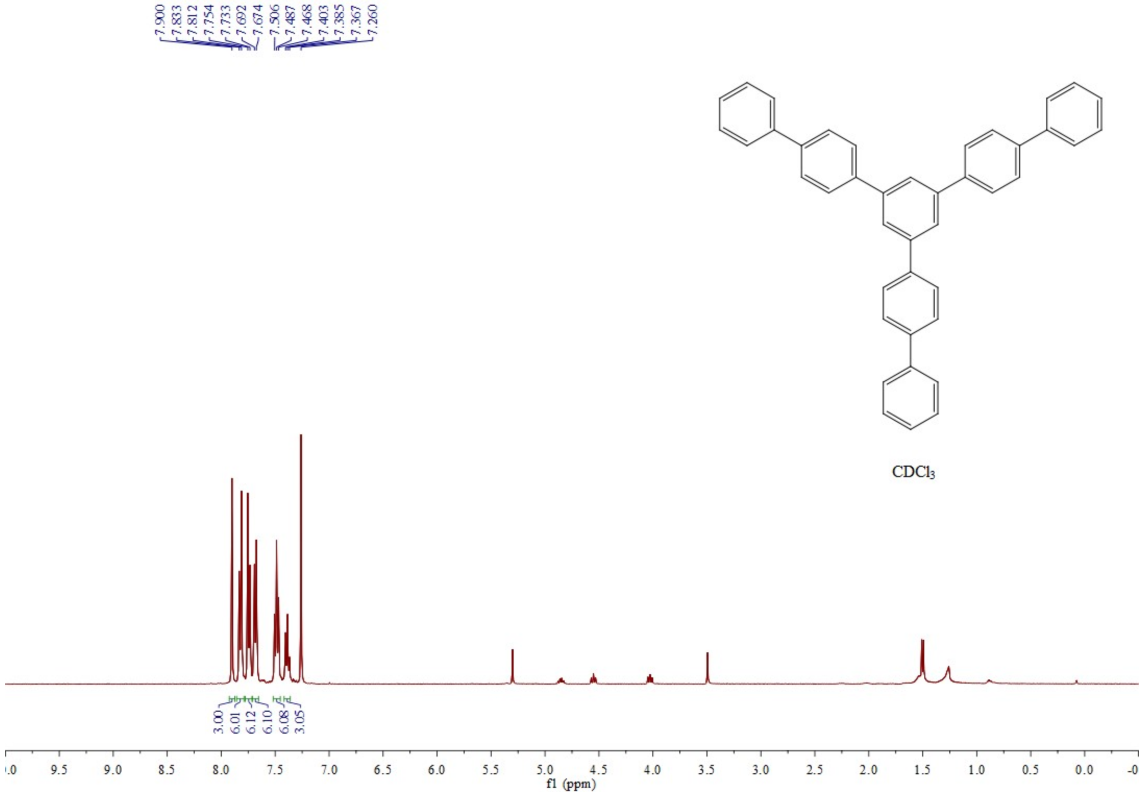


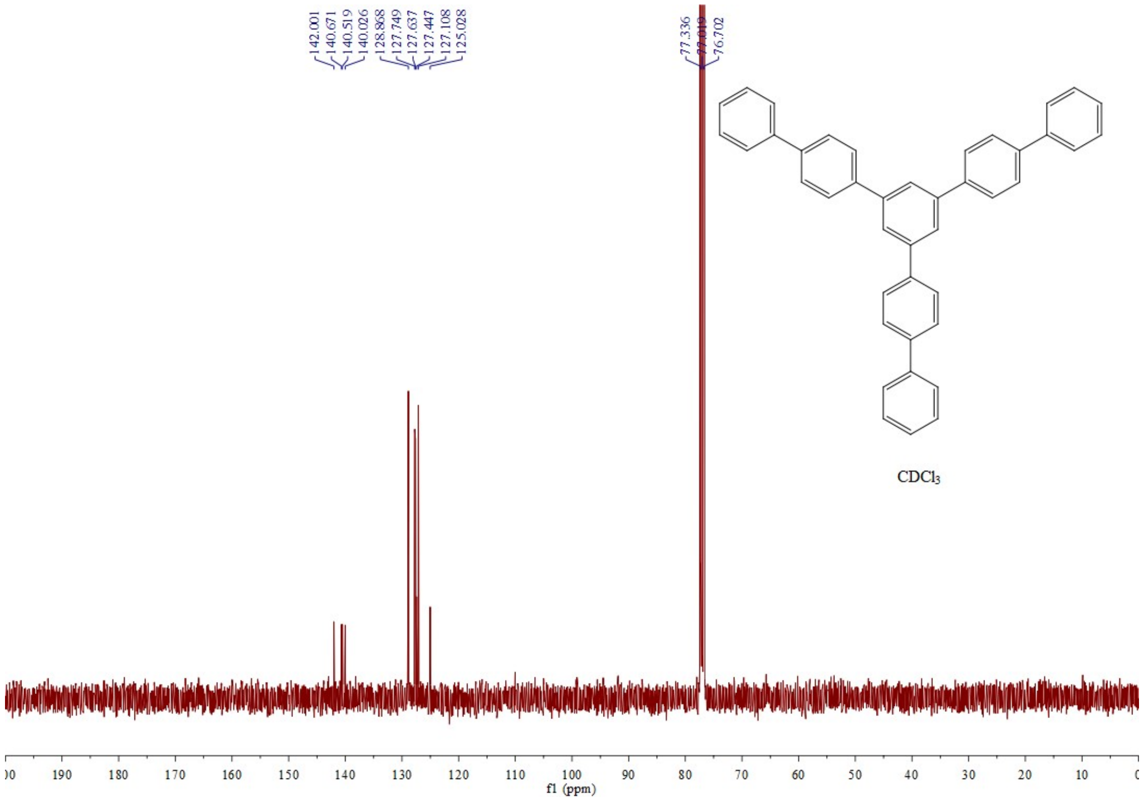
1,3,5-Tris(4-methoxyphenyl)benzene (3b, Table 3).



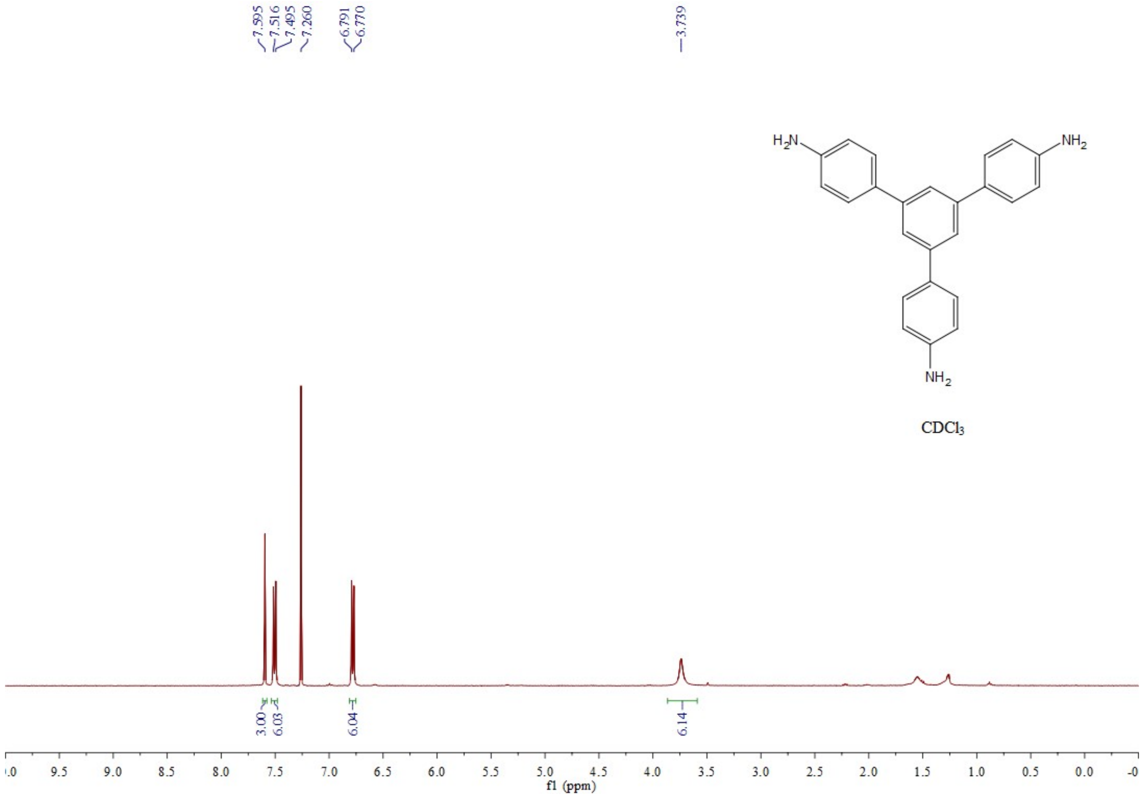


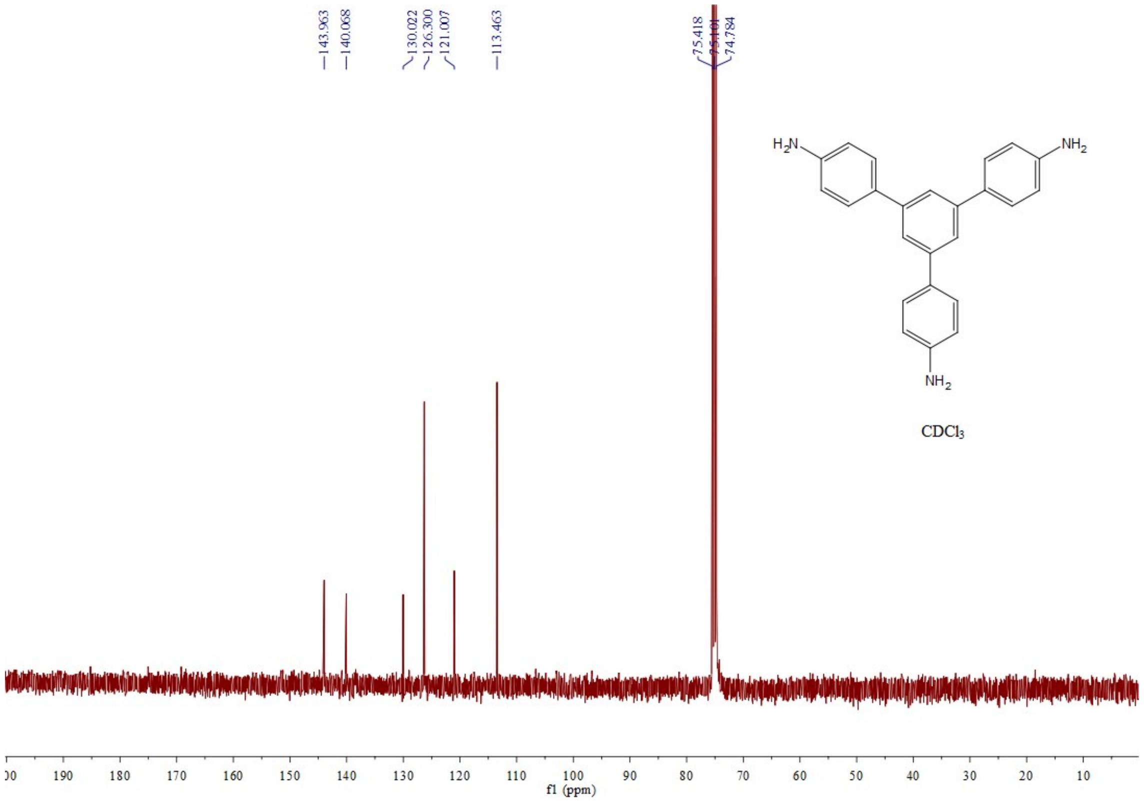
1,3,5-Tris[4-(phenyl)phenyl]benzene (3c, Table 3).



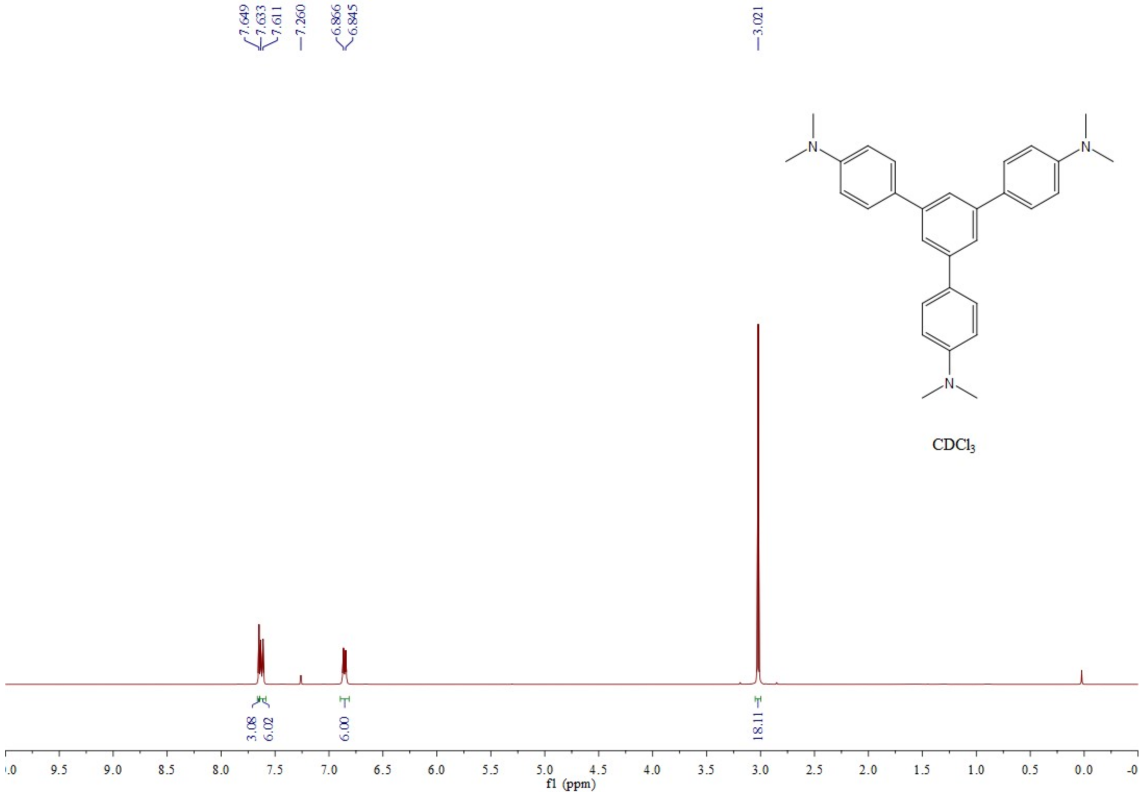


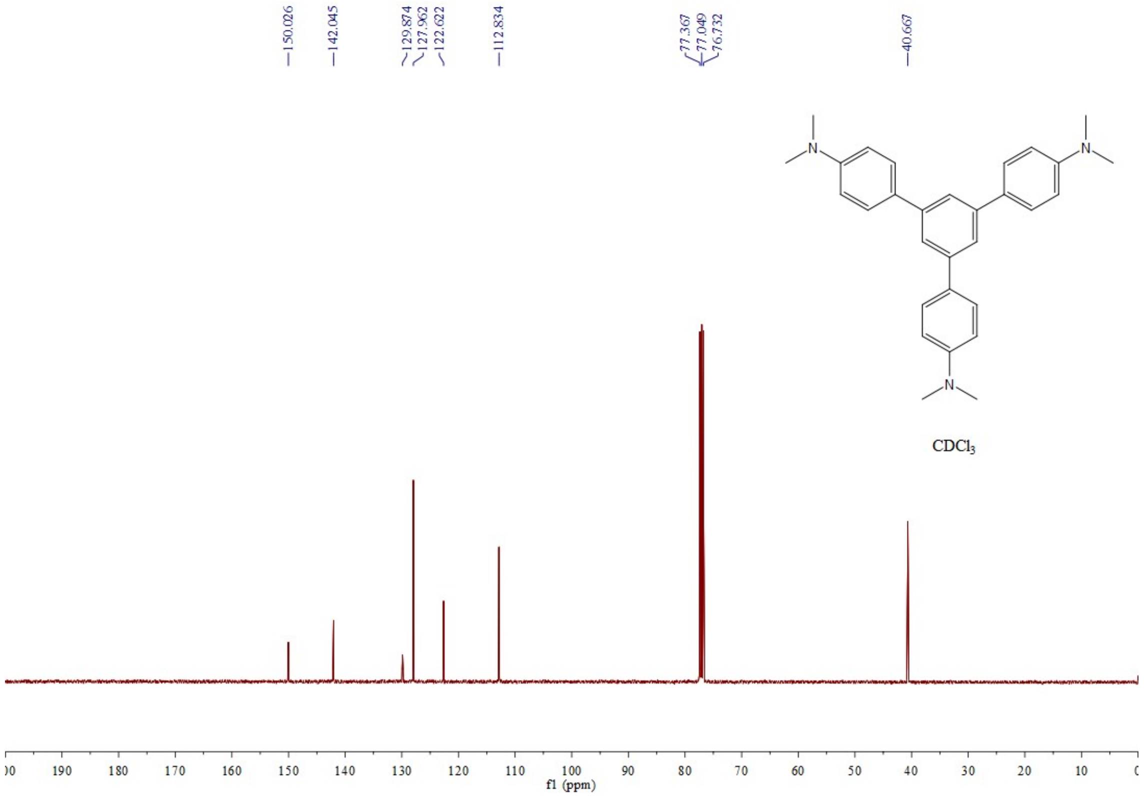
1,3,5-Tris(4-aminophenyl)benzene (3d, Table 3).



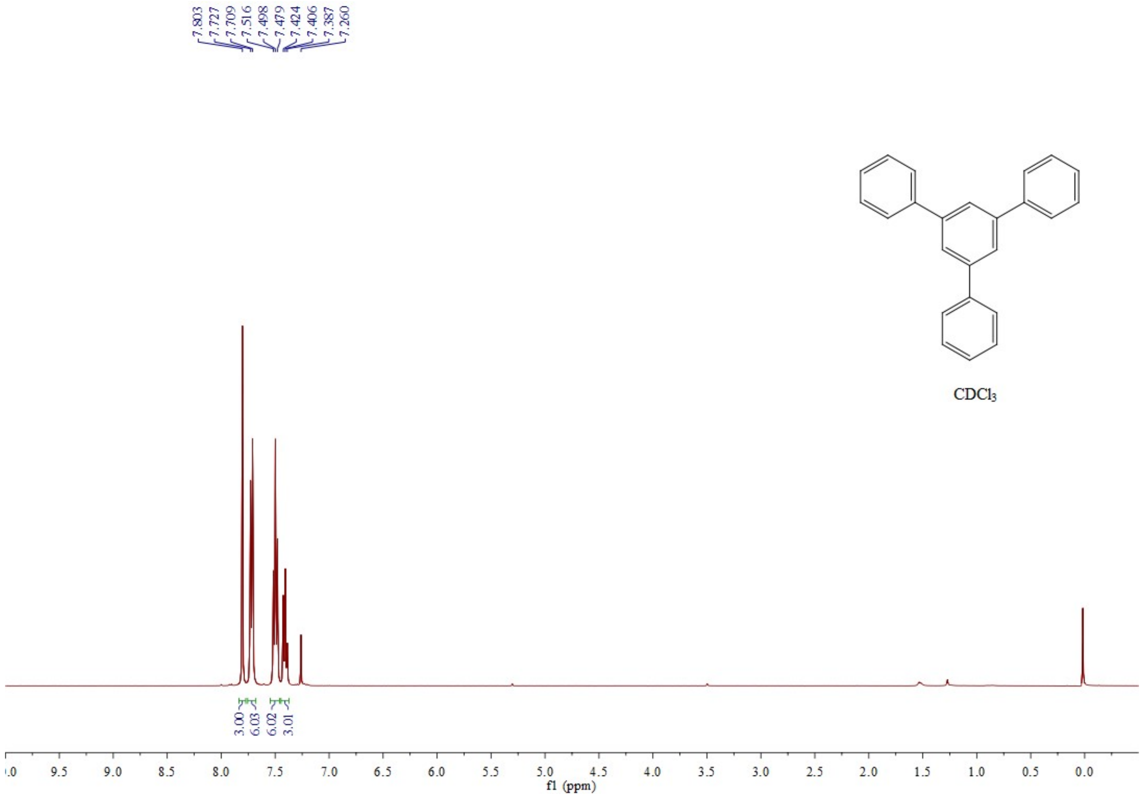


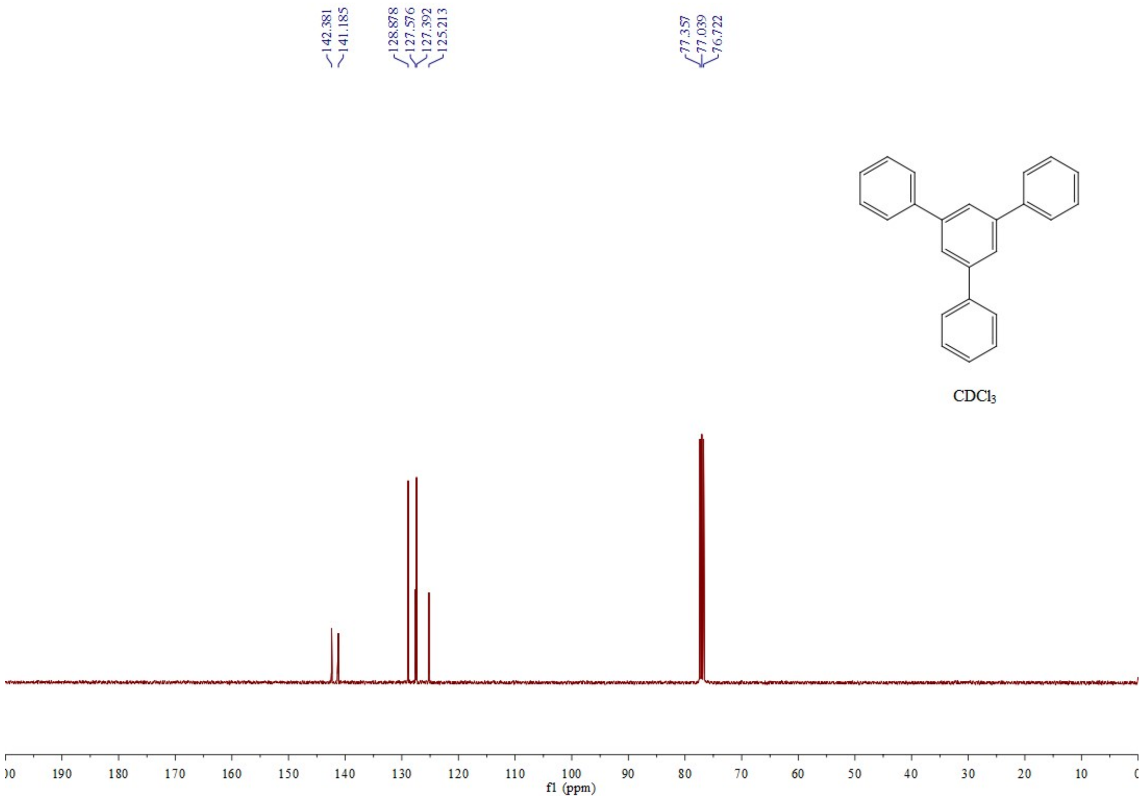
1,3,5-Tris(4-dimethylaminophenyl)benzene (3e, Table 3).



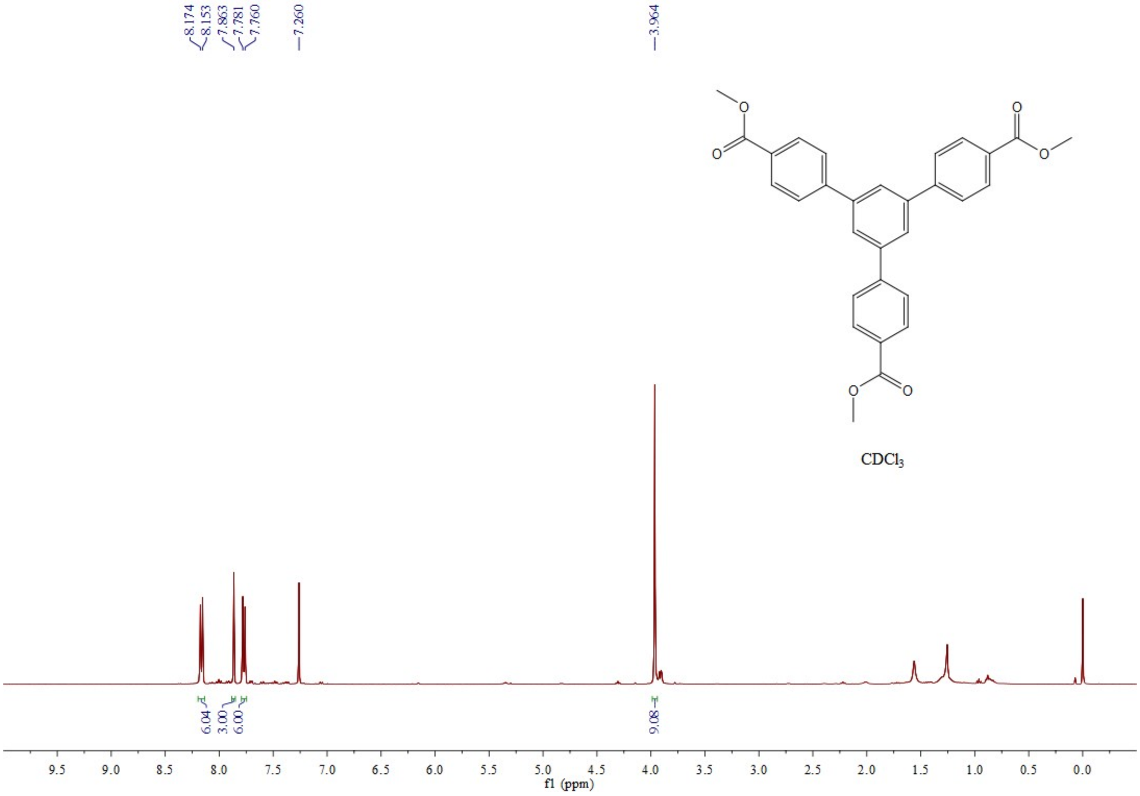


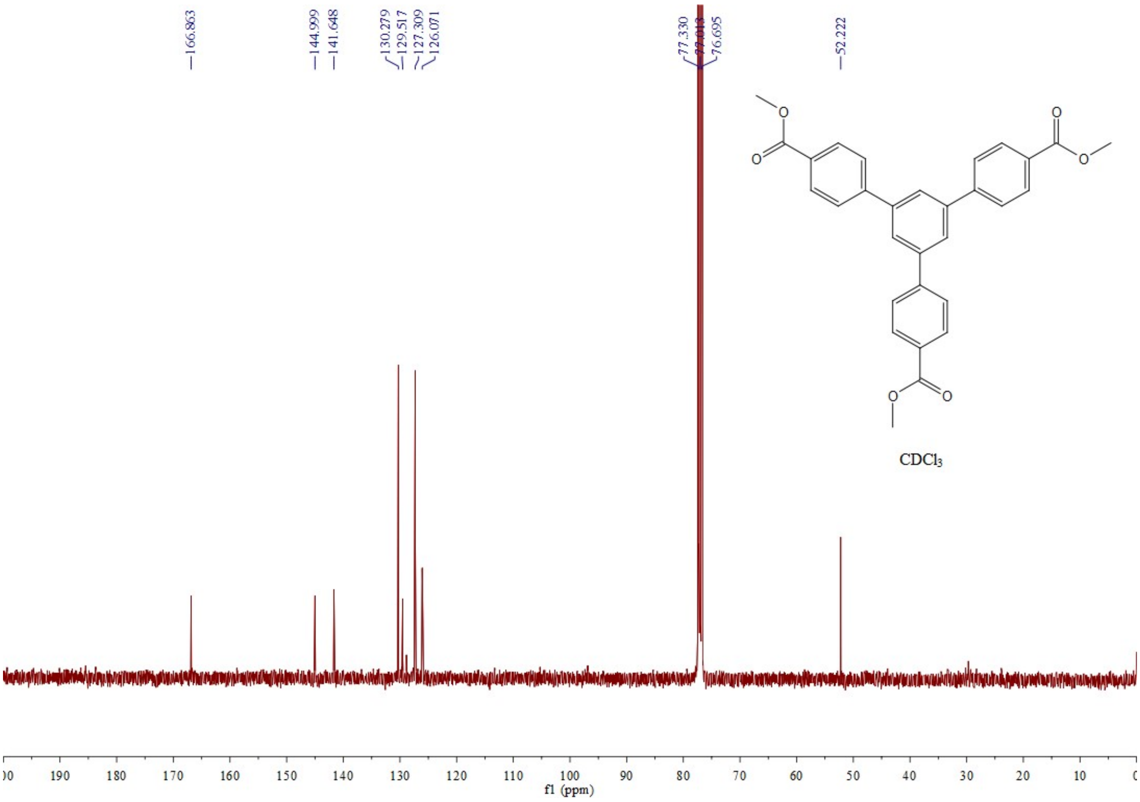
1,3,5-Triphenylbenzene (3f, Table 3).



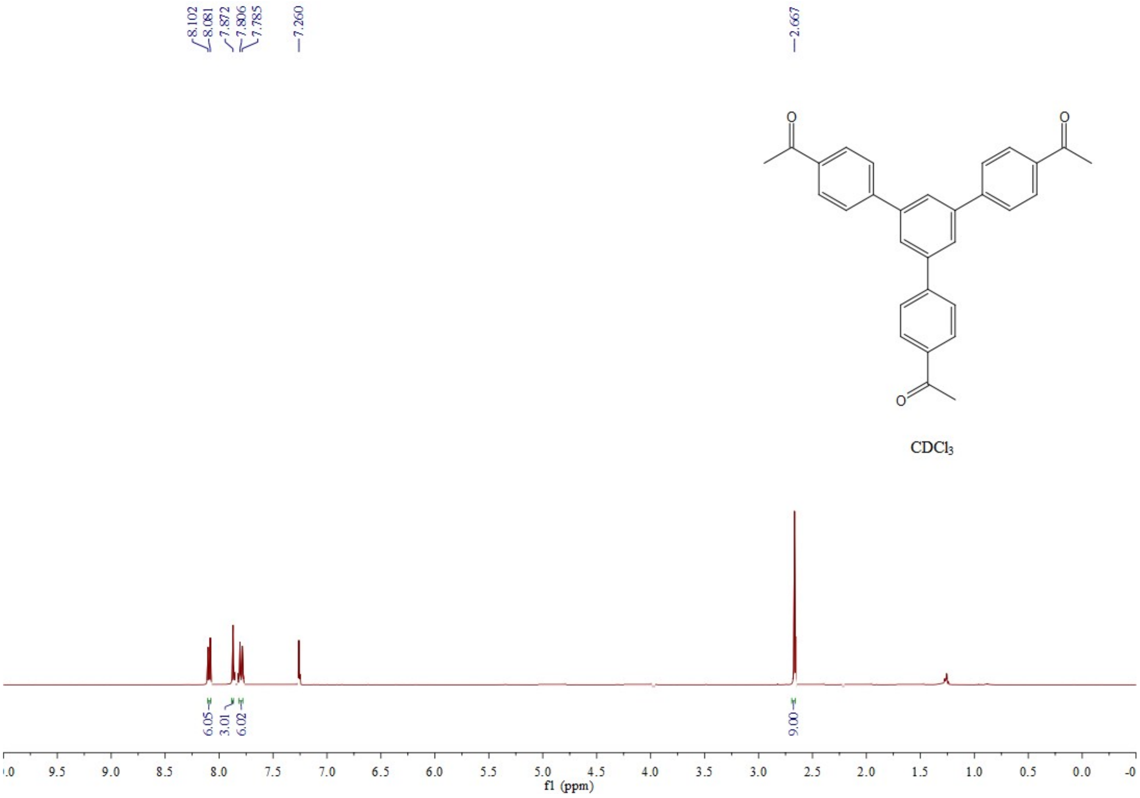


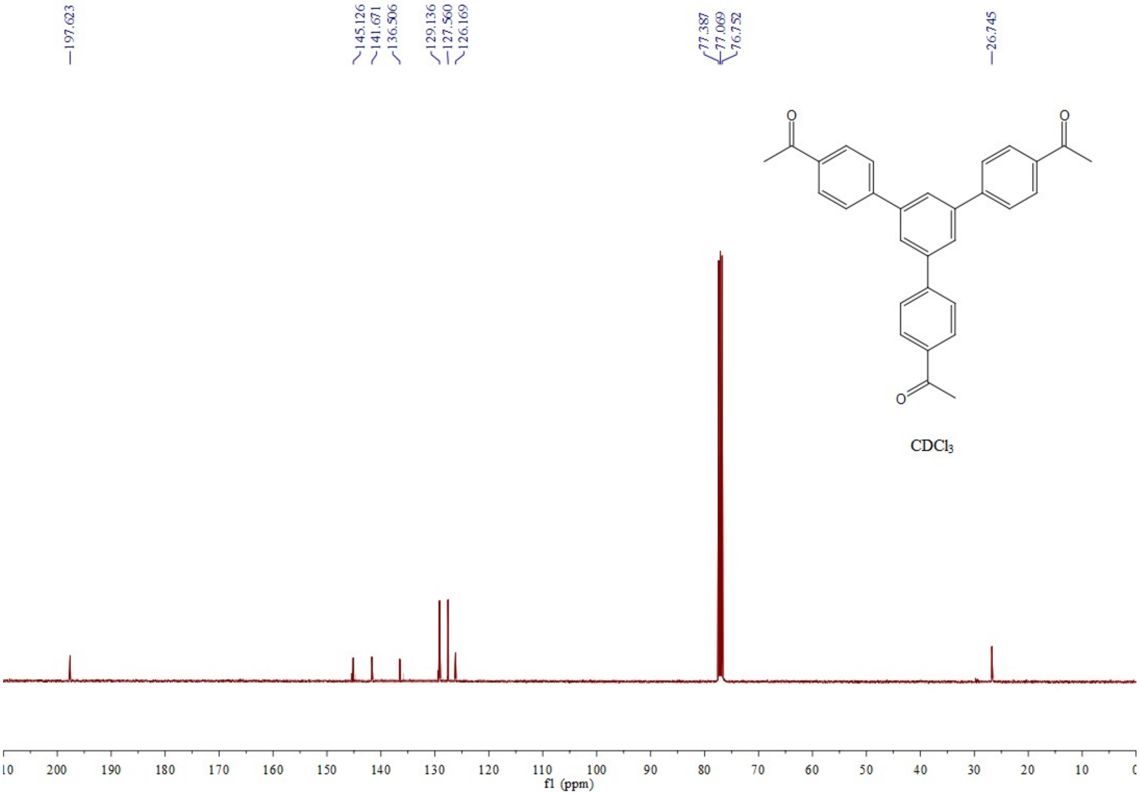
1,3,5-Tris(4-methoxycarbonylphenyl)benzene (3g, Table 3).



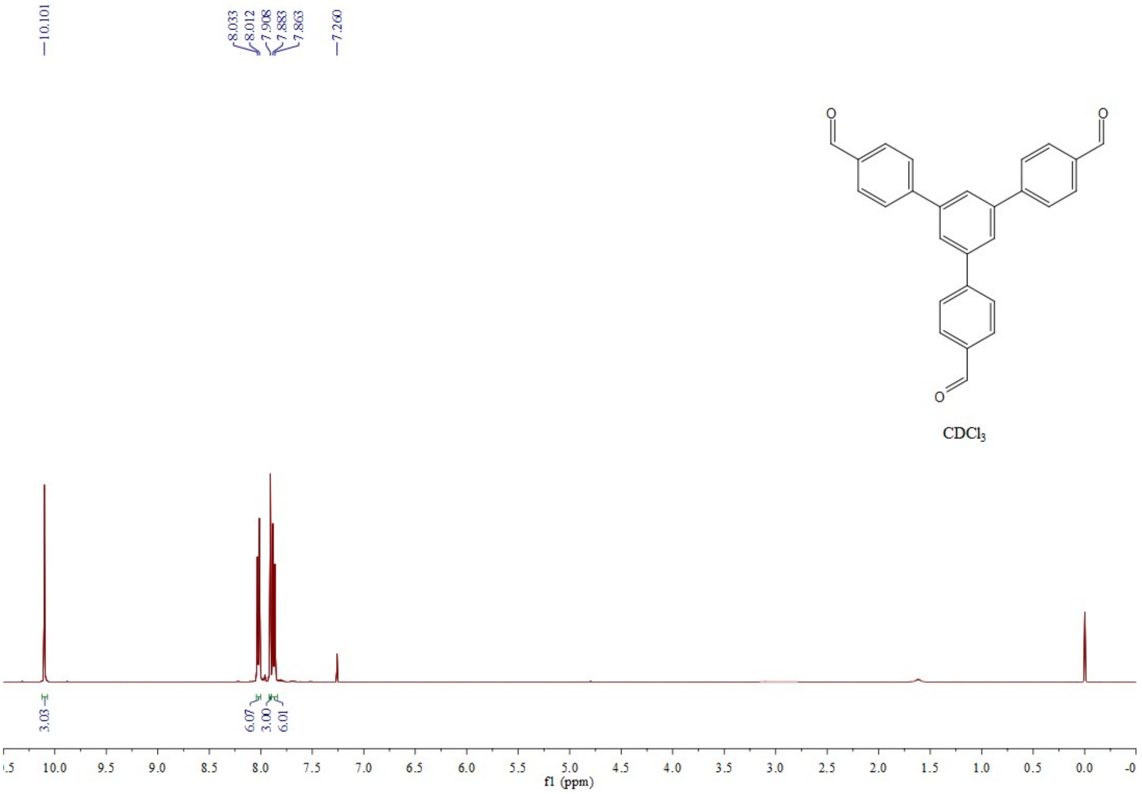


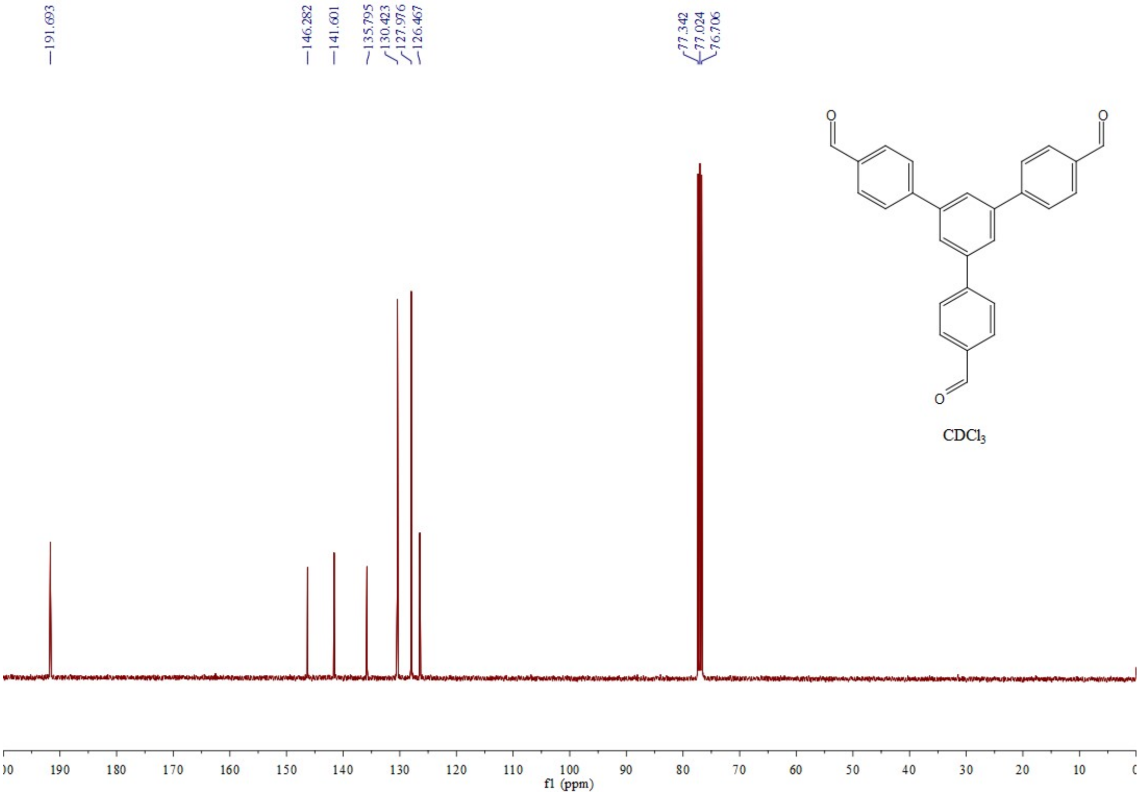
1,3,5-Tris(4-acetylphenyl)benzene (3h, Table 3).



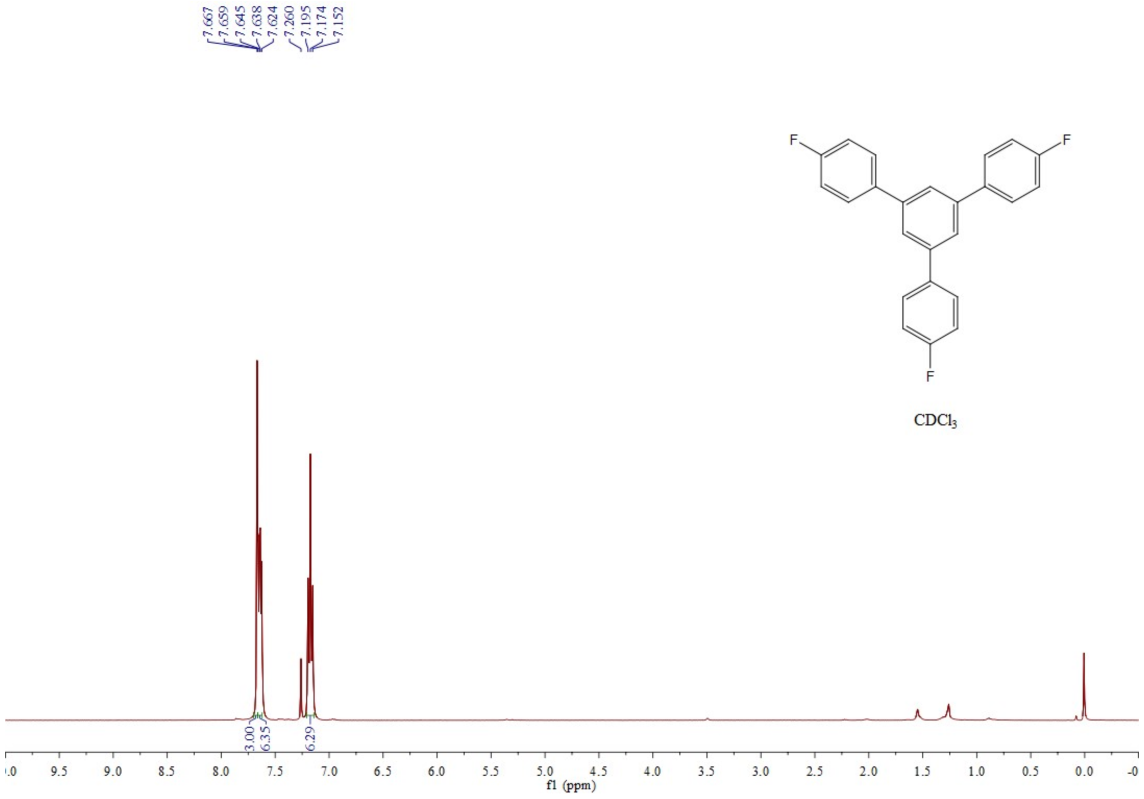


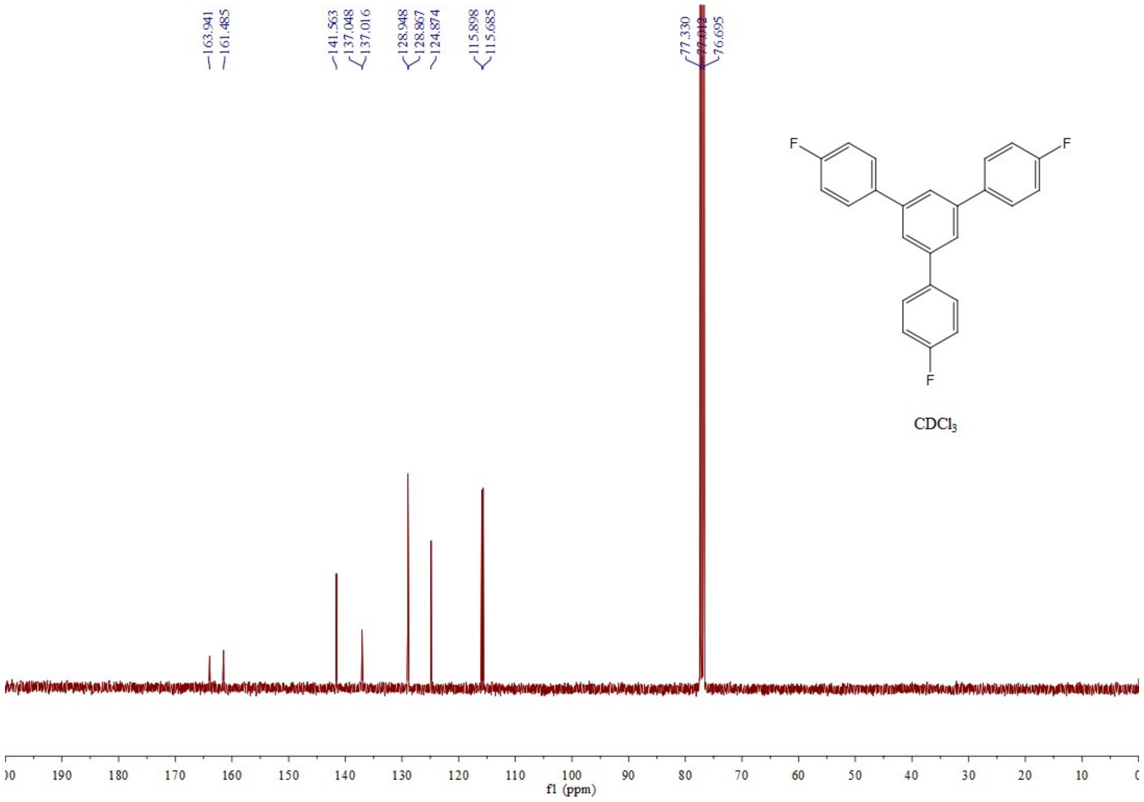
1,3,5-Tris(4-formylphenyl)benzene (3i, Table 3).



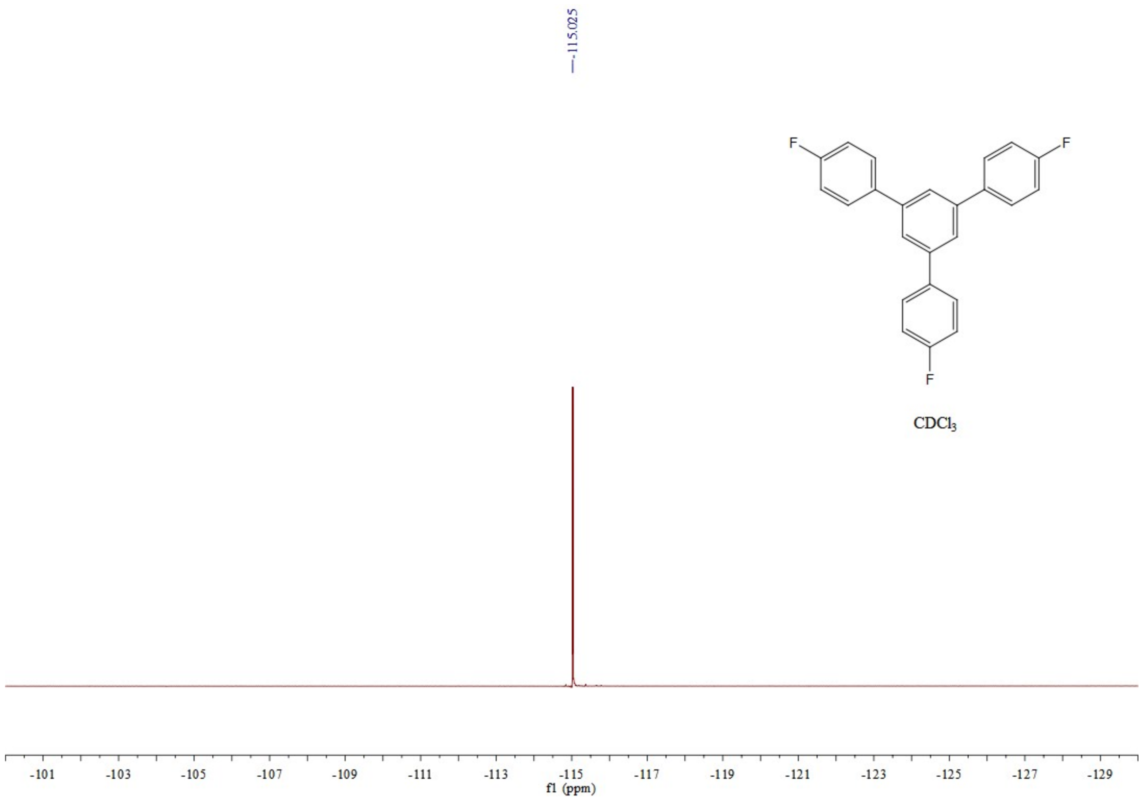


1,3,5-Tris(4-fluorophenyl)benzene (3k, Table 3).

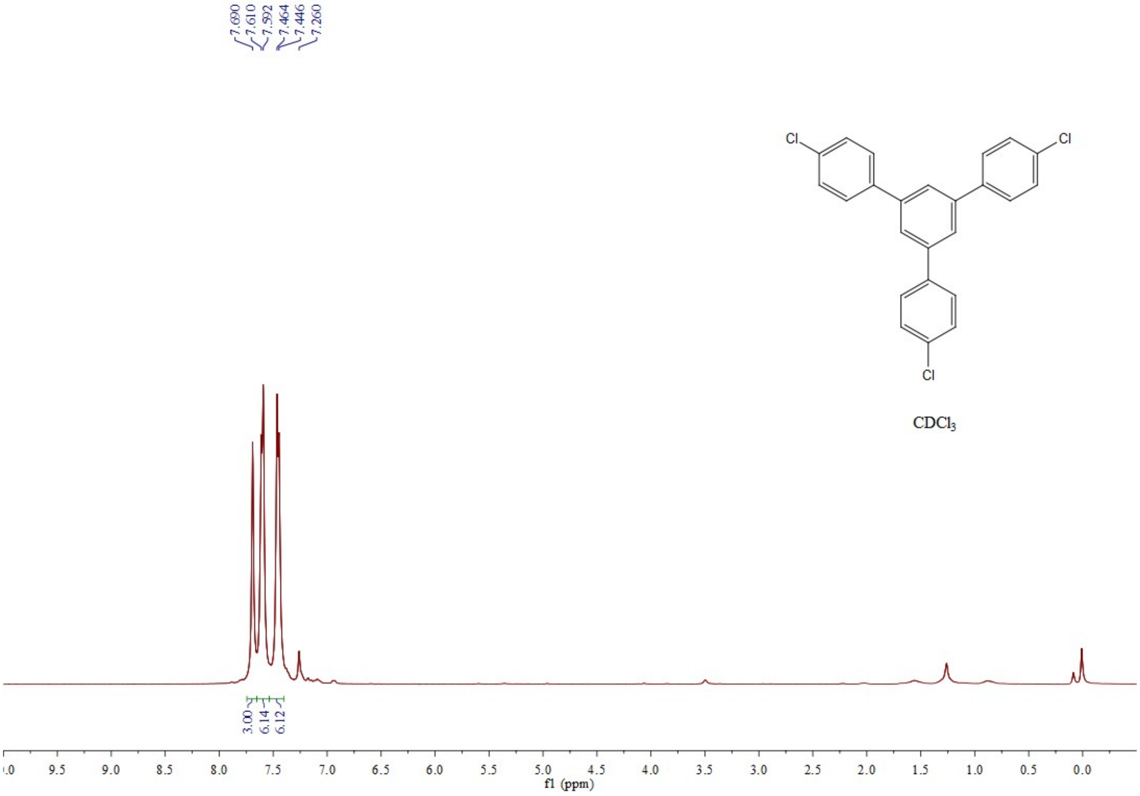


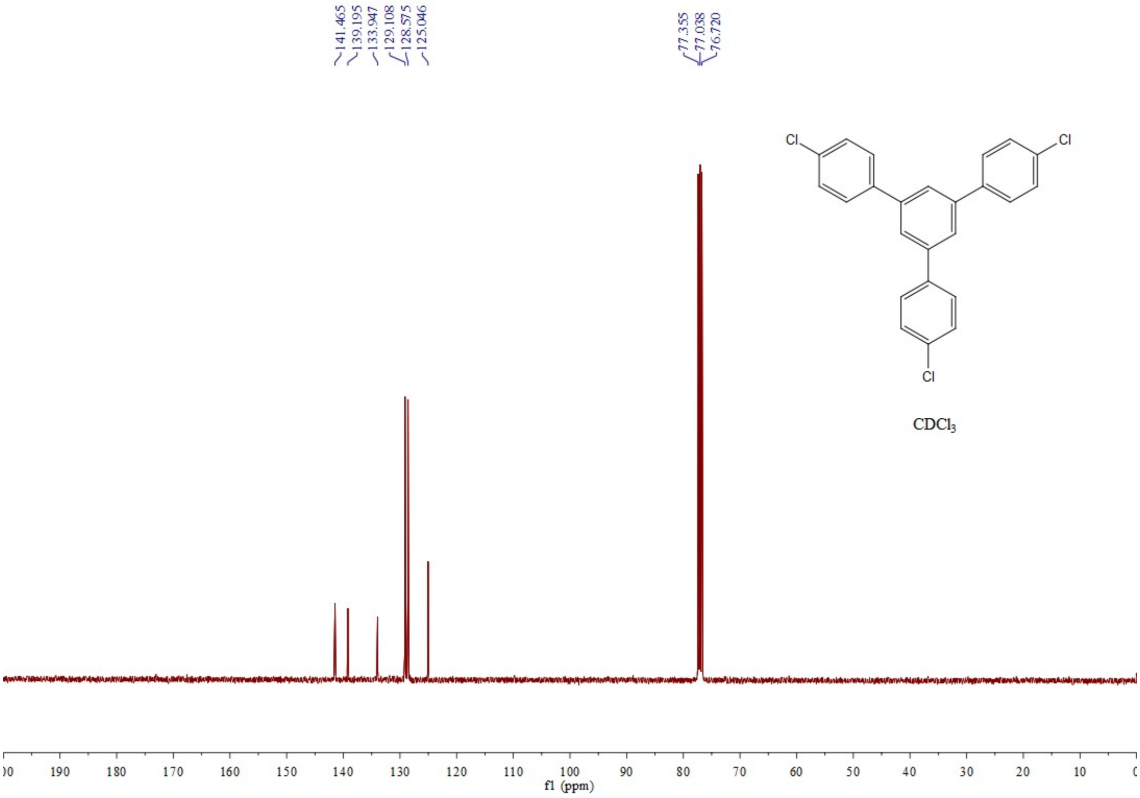


^{19}F NMR spectra of 1,3,5-Tris(4-fluorophenyl)benzene (3k, Table 3).

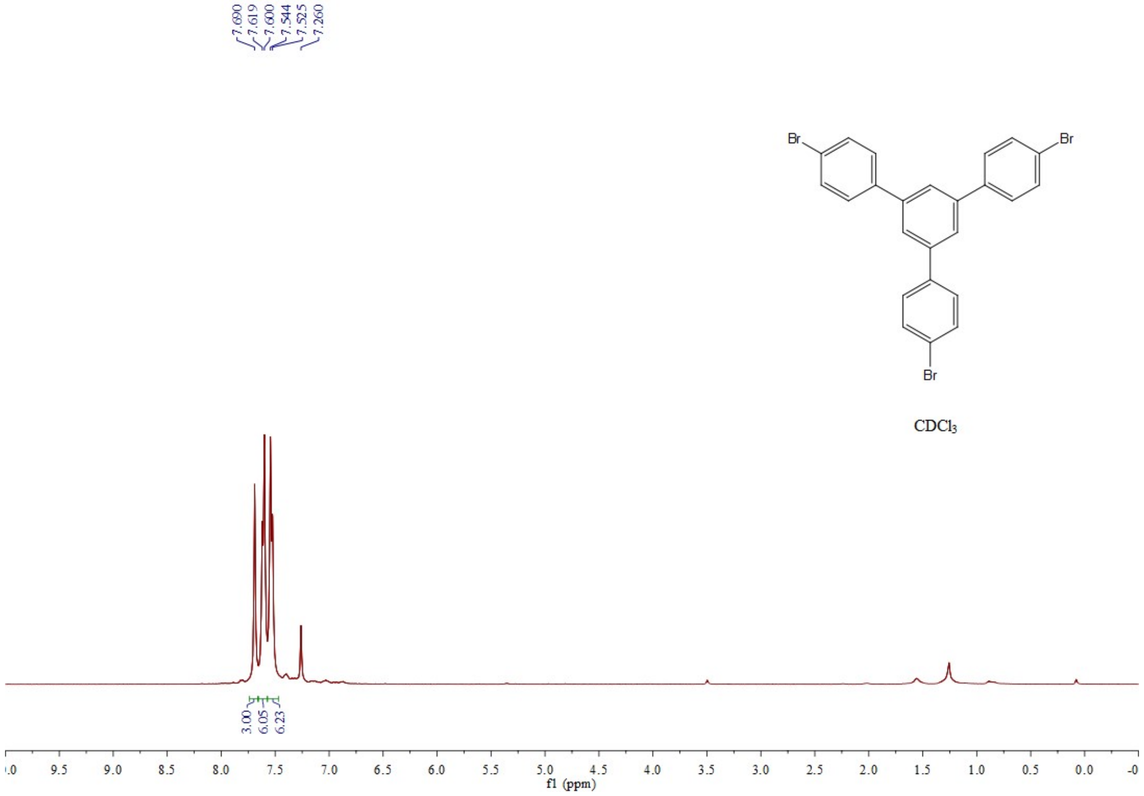


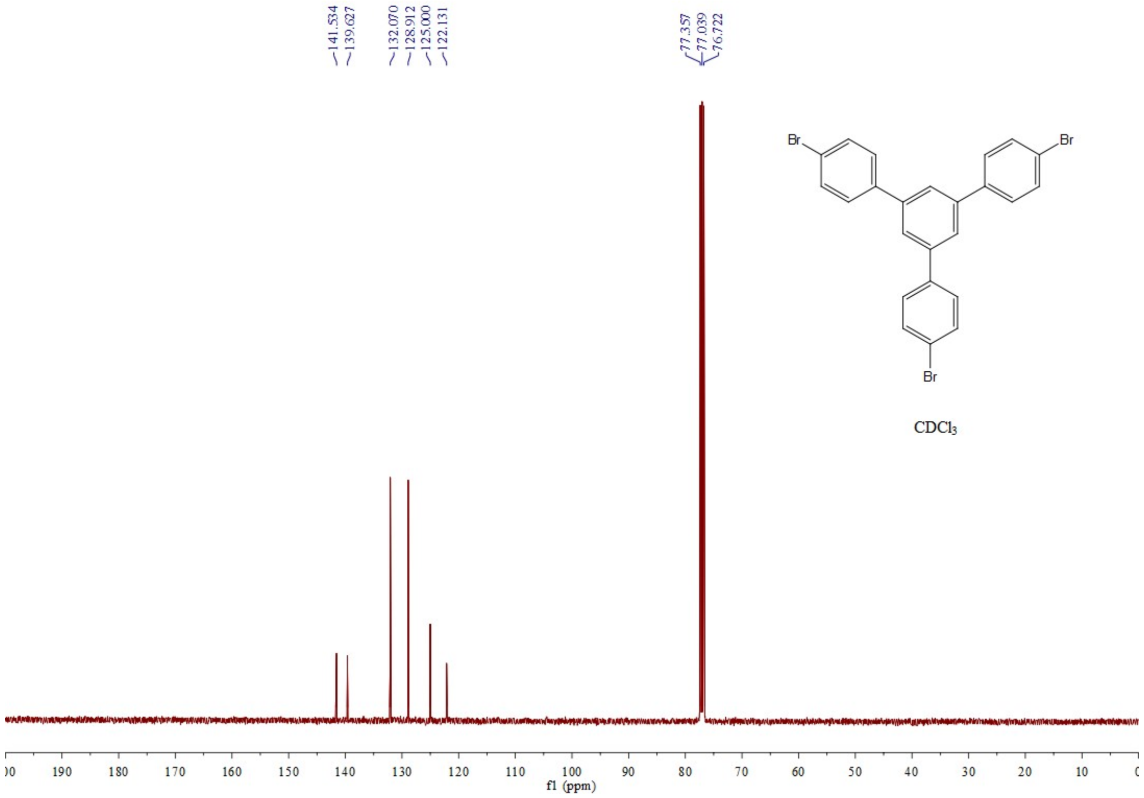
1,3,5-Tris(4-chlorophenyl)benzene (3l, Table 3).



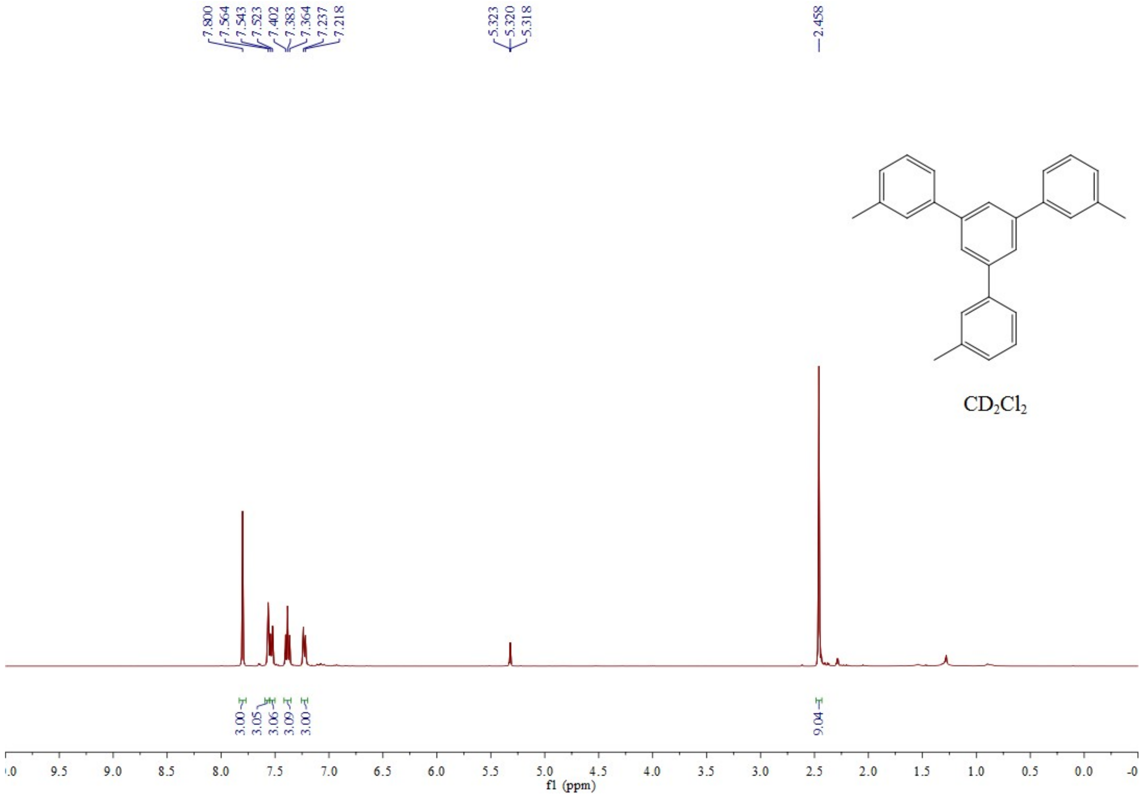


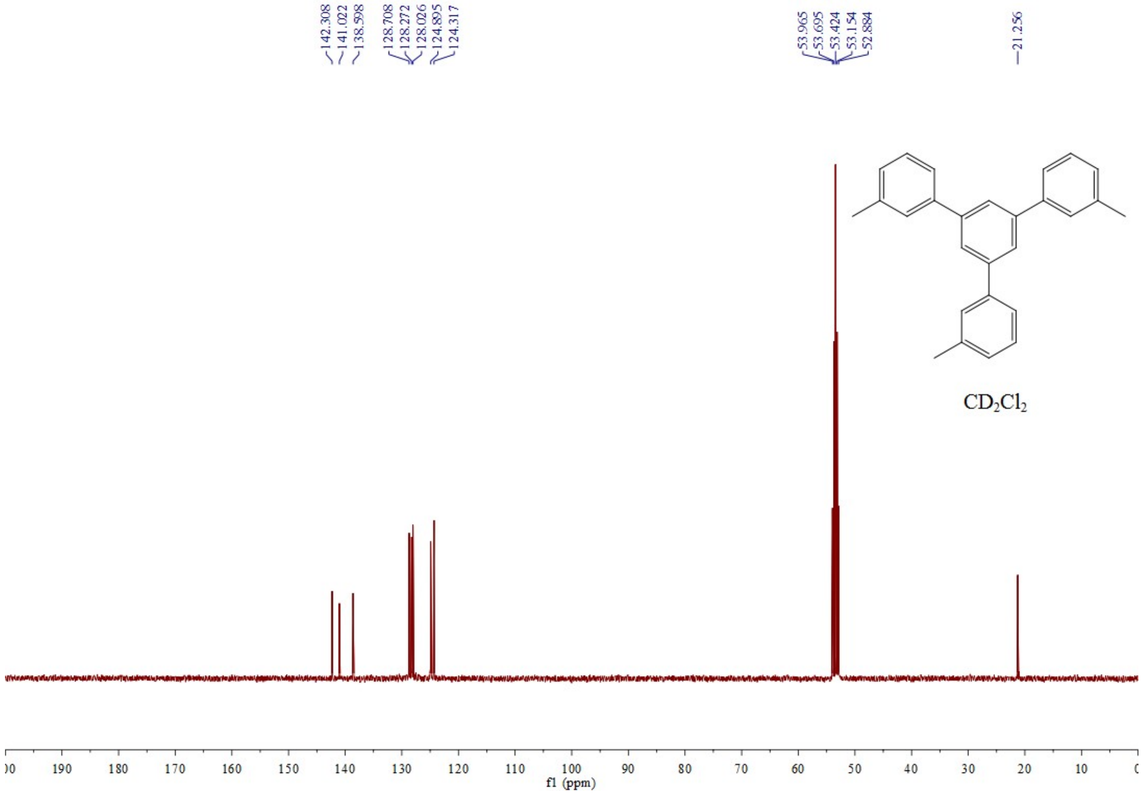
1,3,5-Tris(4-bromophenyl)benzene (3m, Table 3).



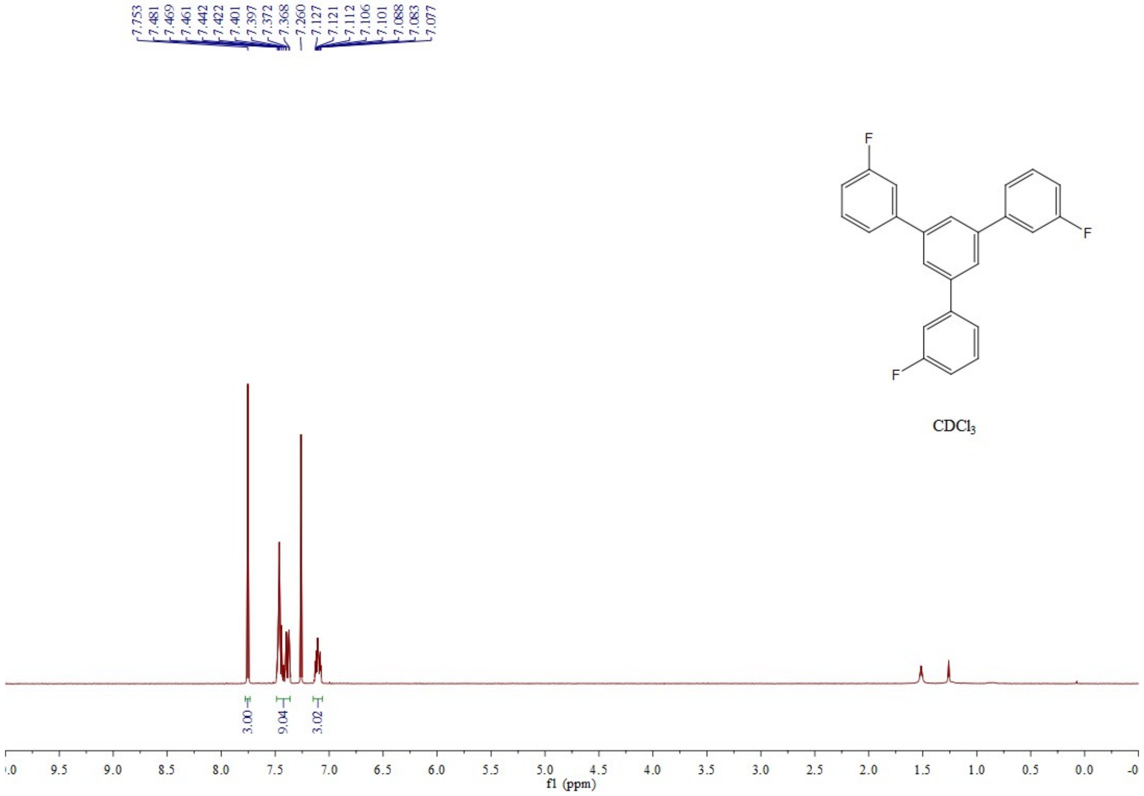


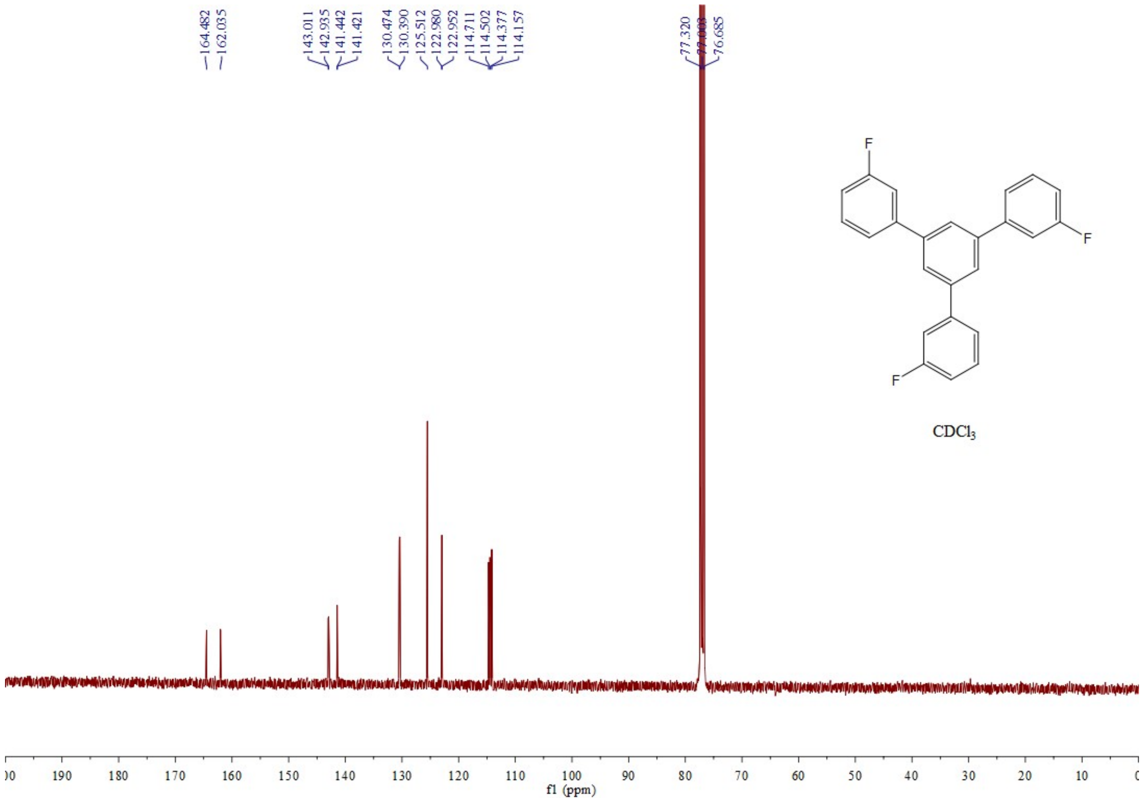
1,3,5-Tris(3-methylphenyl)benzene (3n, Table 3).



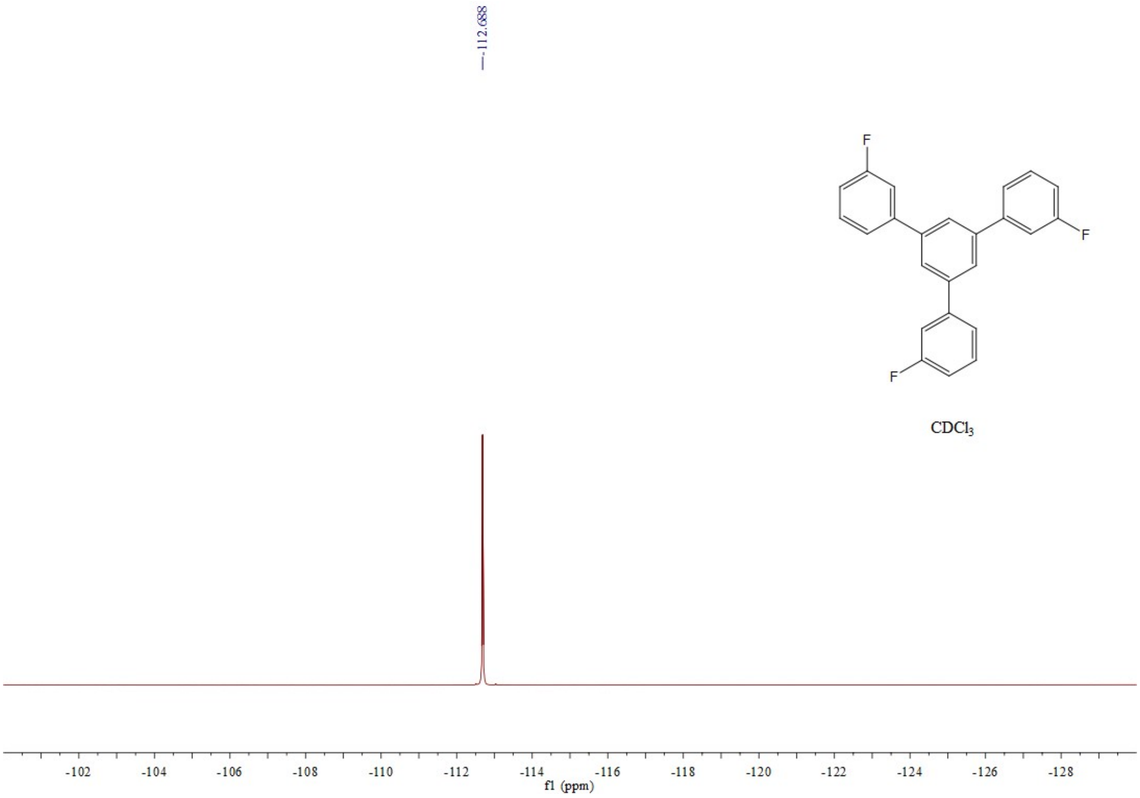


1,3,5-Tris(3-fluorophenyl)benzene (3o, Table 3).

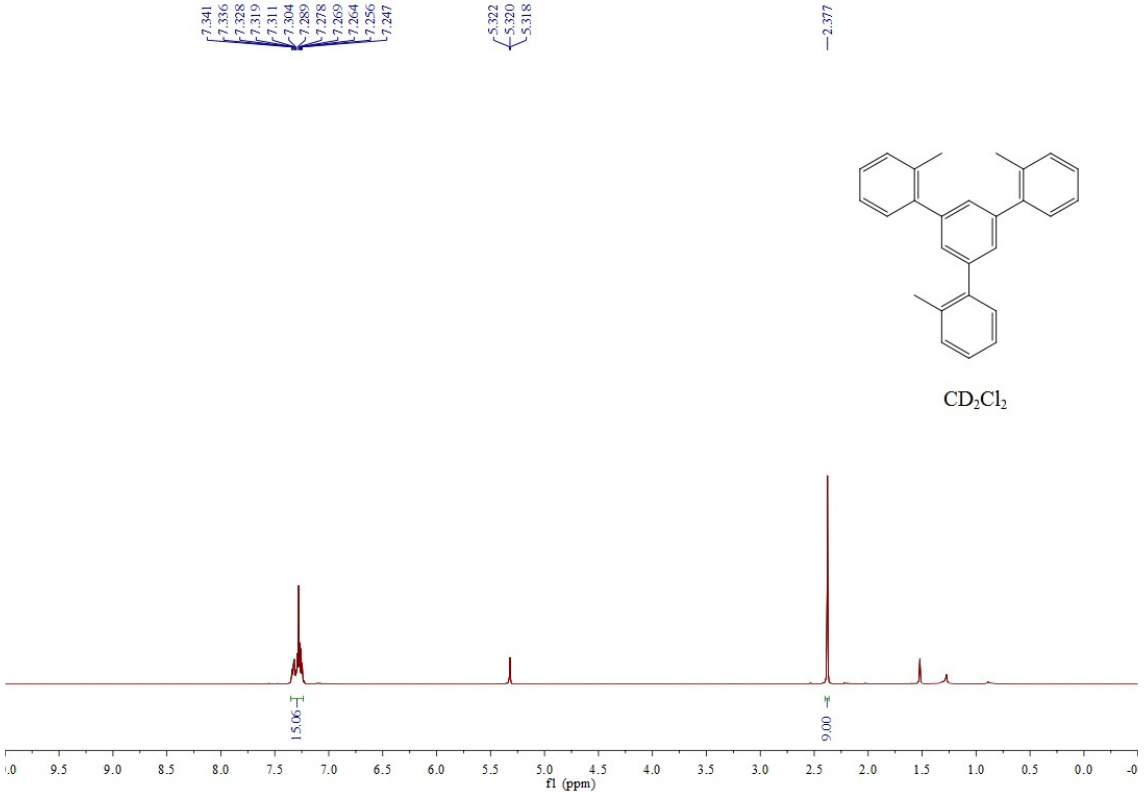


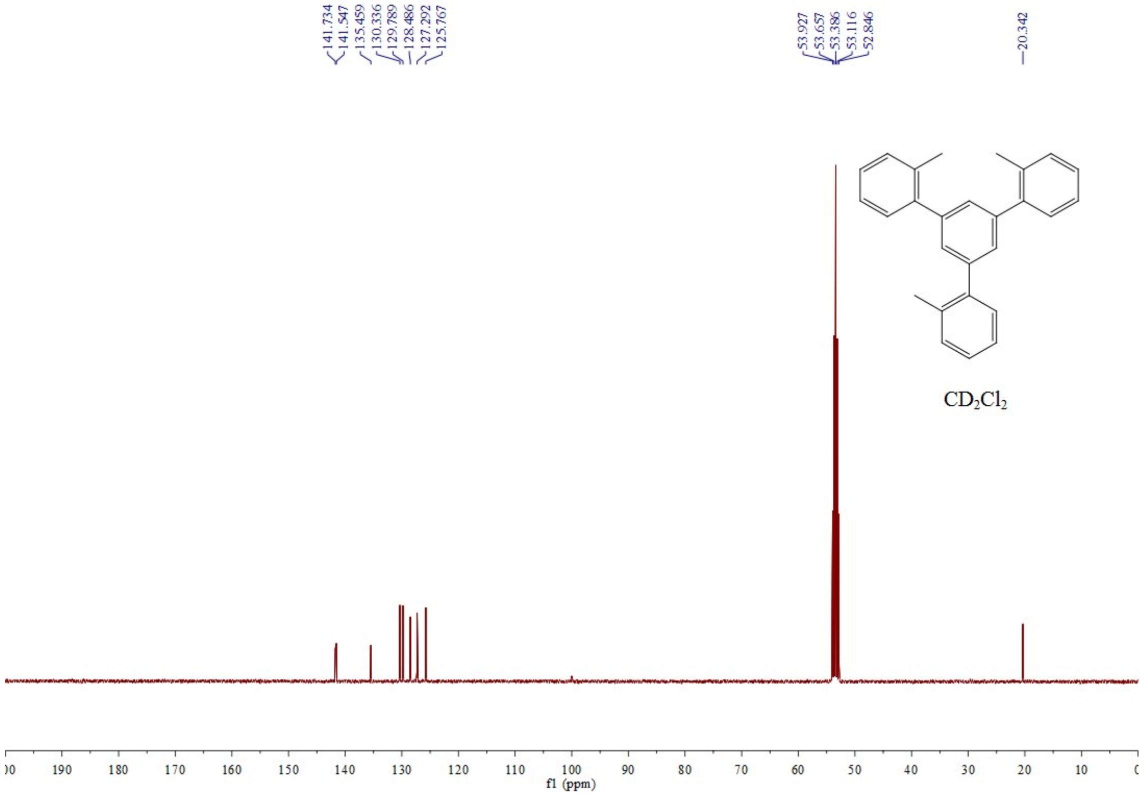


^{19}F NMR spectra of 1,3,5-Tris(3-fluorophenyl)benzene (3o, Table 3).

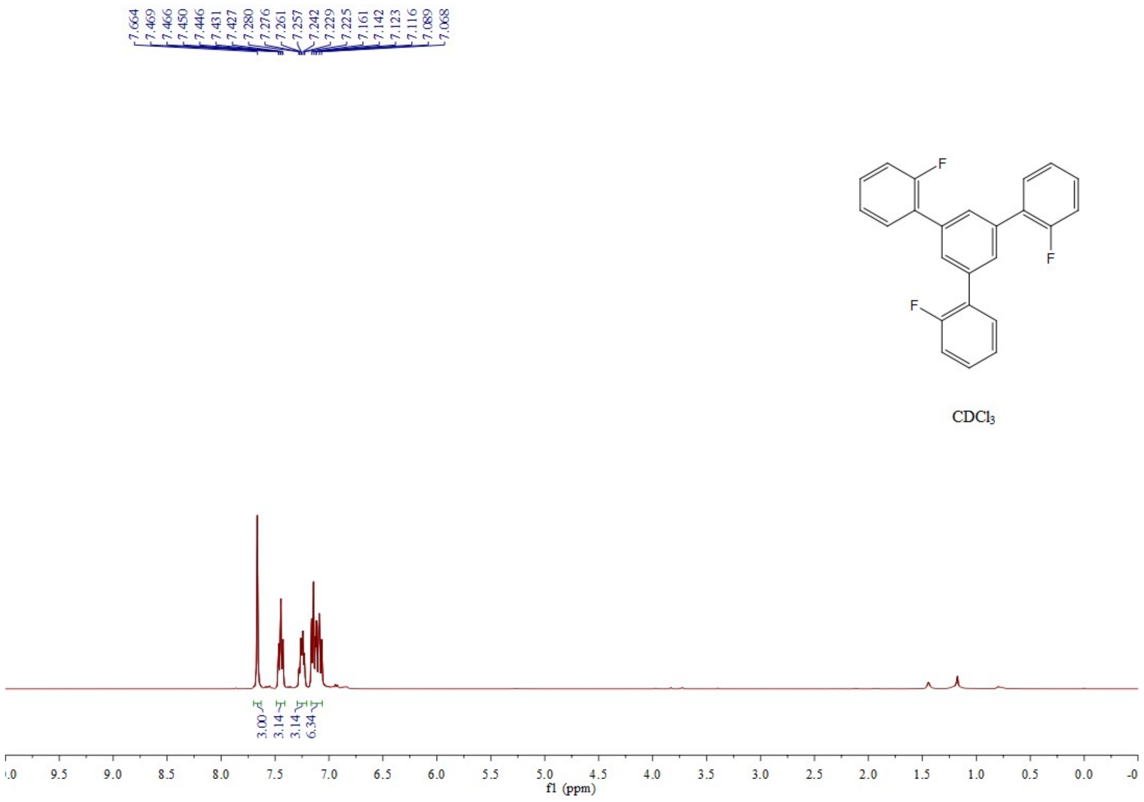


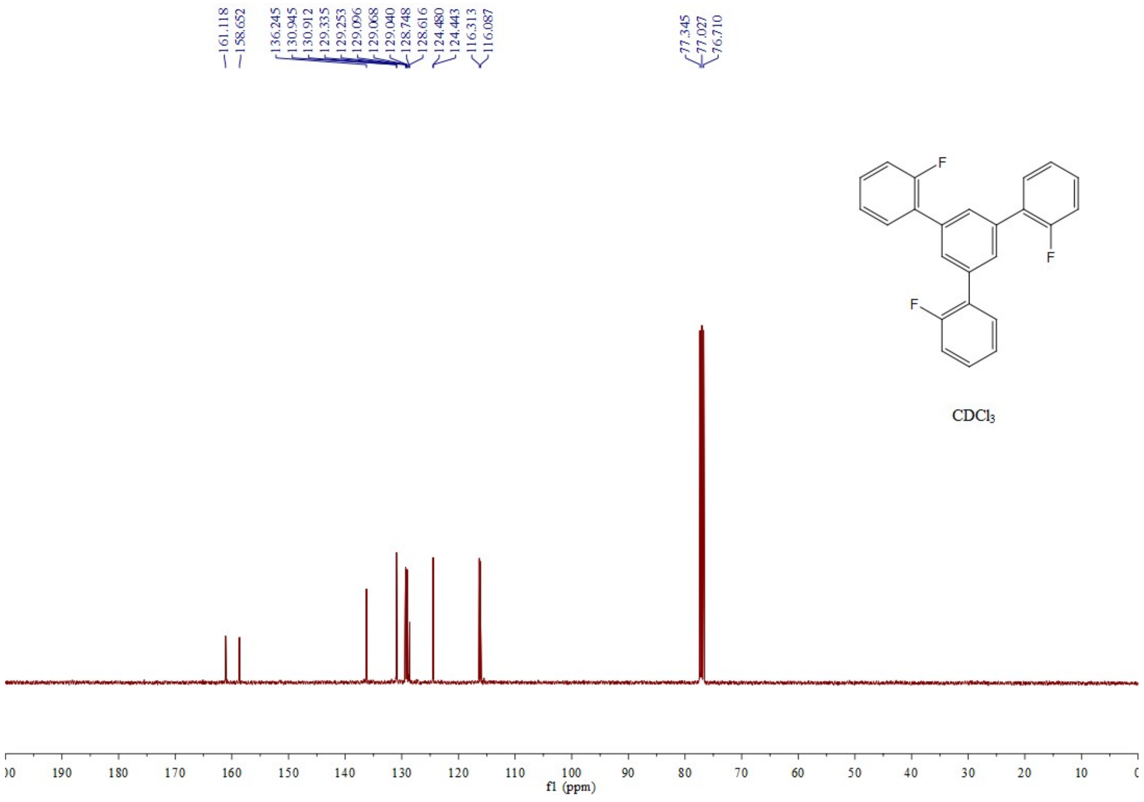
1,3,5-Tris(2-methylphenyl)benzene (3p, Table 3).



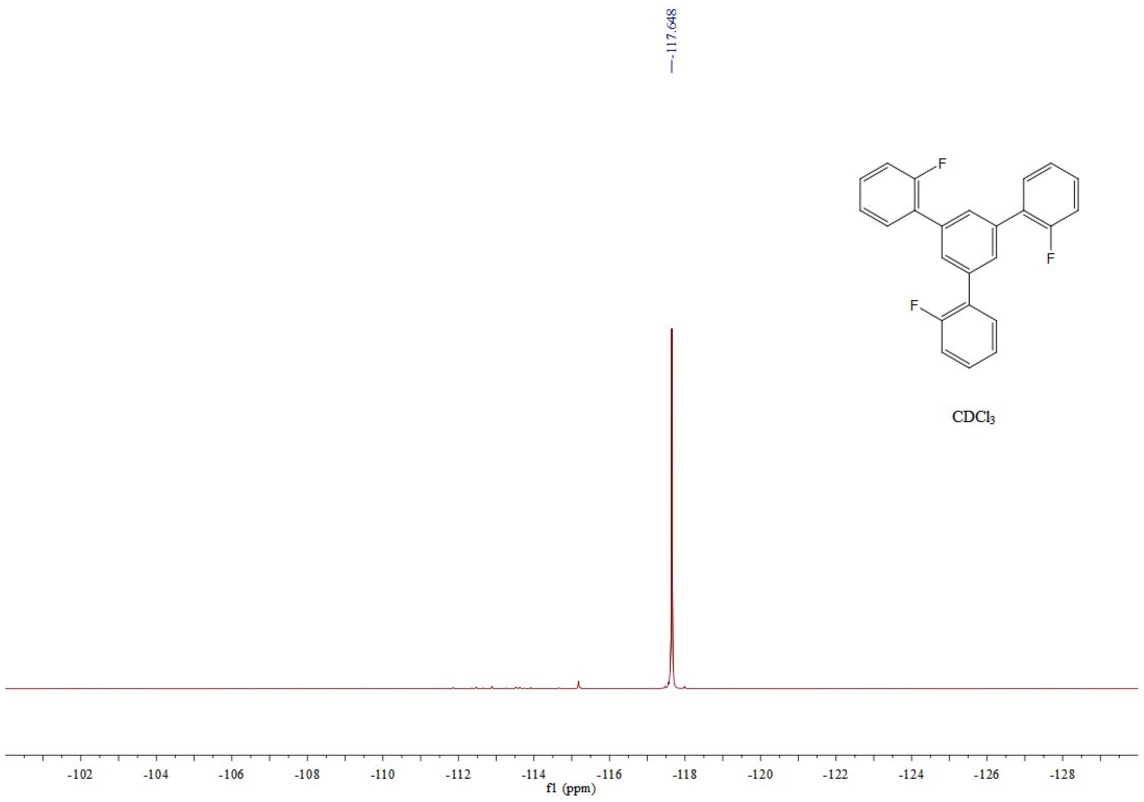


1,3,5-Tris(2-fluorophenyl)benzene (3q, Table 3).

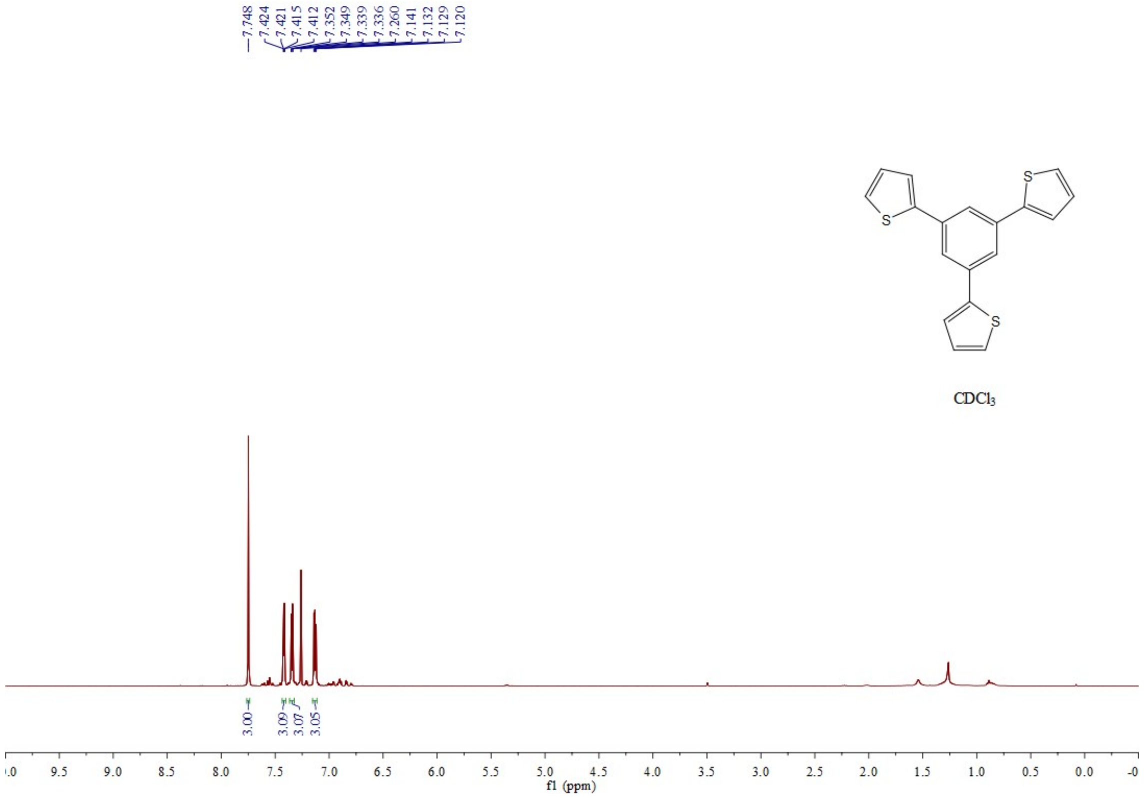


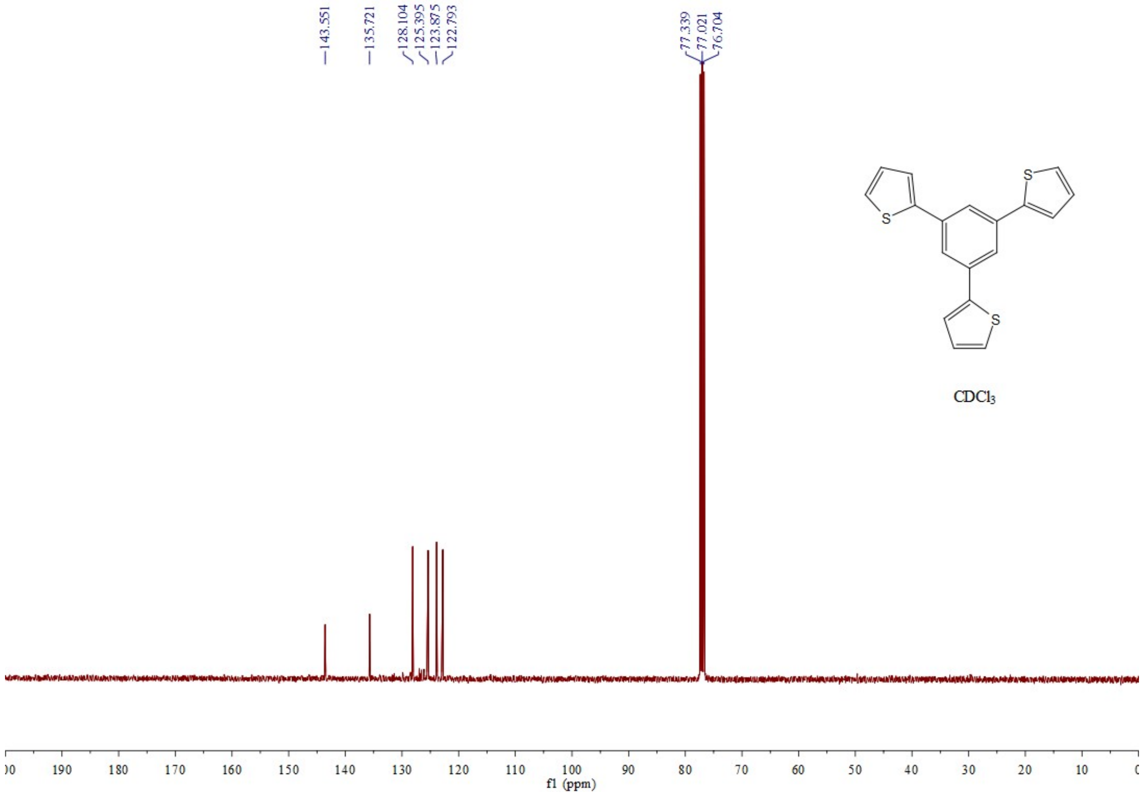


^{19}F NMR spectra of 1,3,5-Tris(2-fluorophenyl)benzene (3q, Table 3).

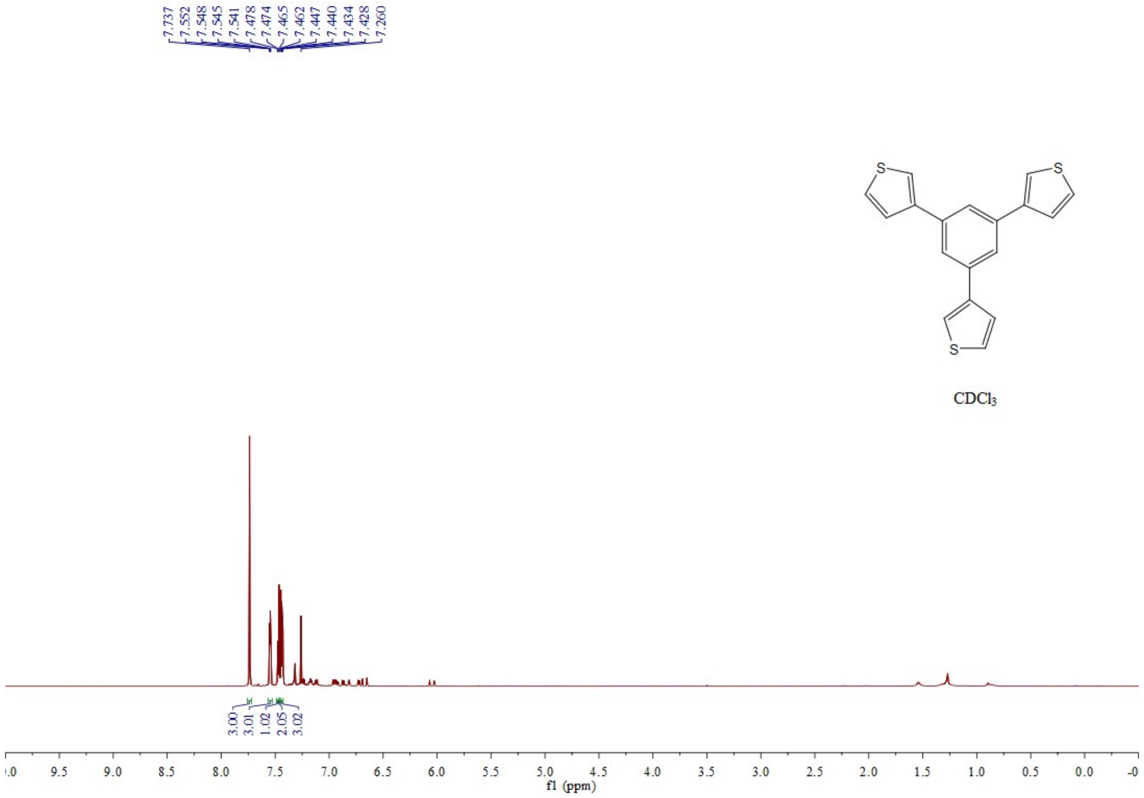


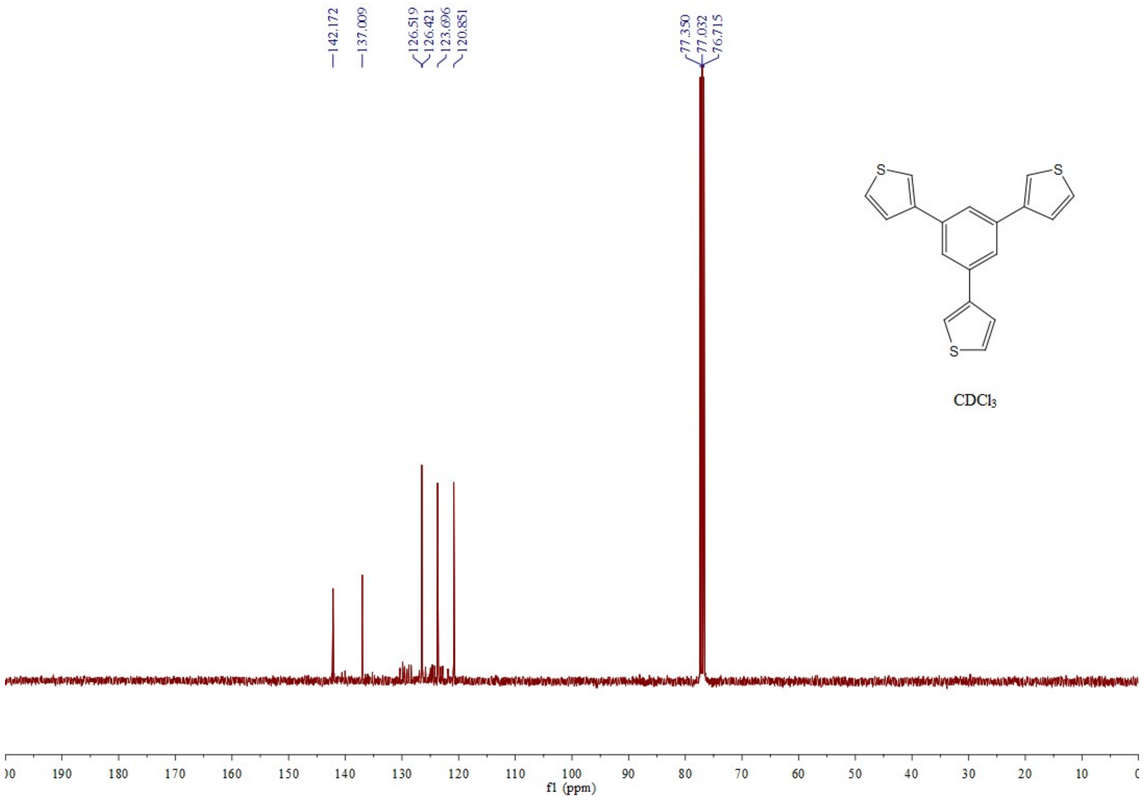
1,3,5-Tris(2-thienyl)benzene (3r, Table 3).



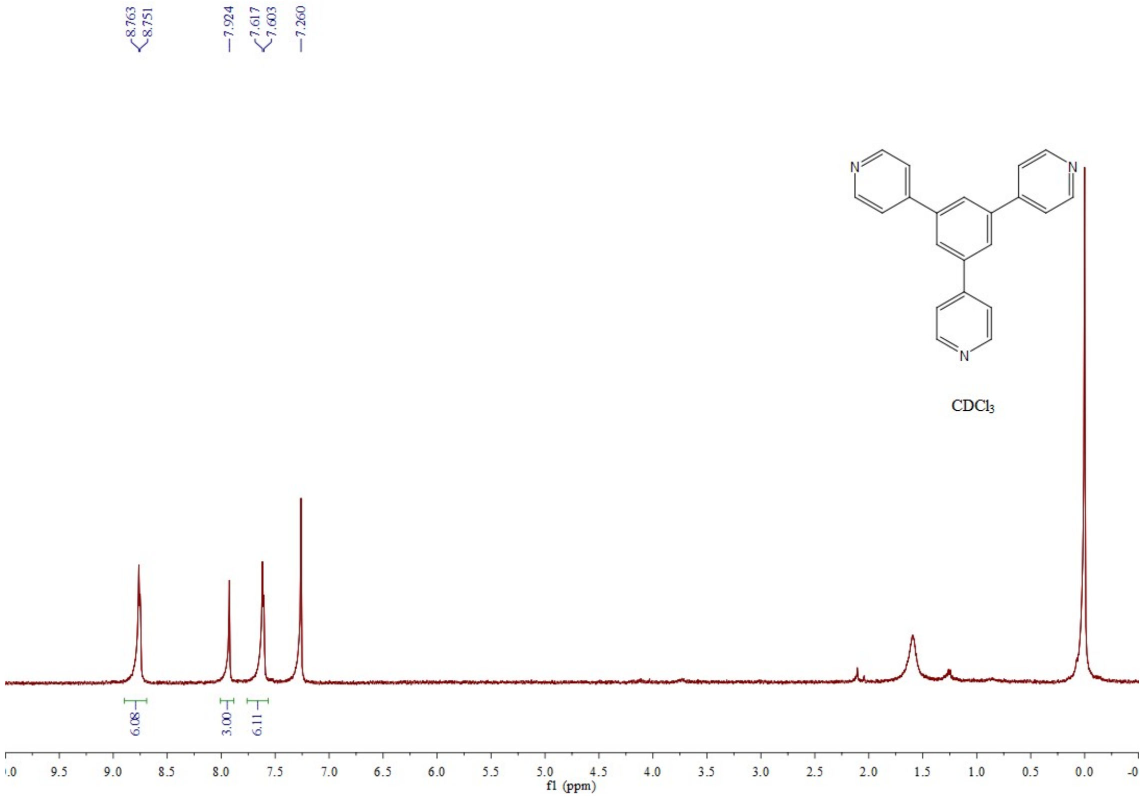


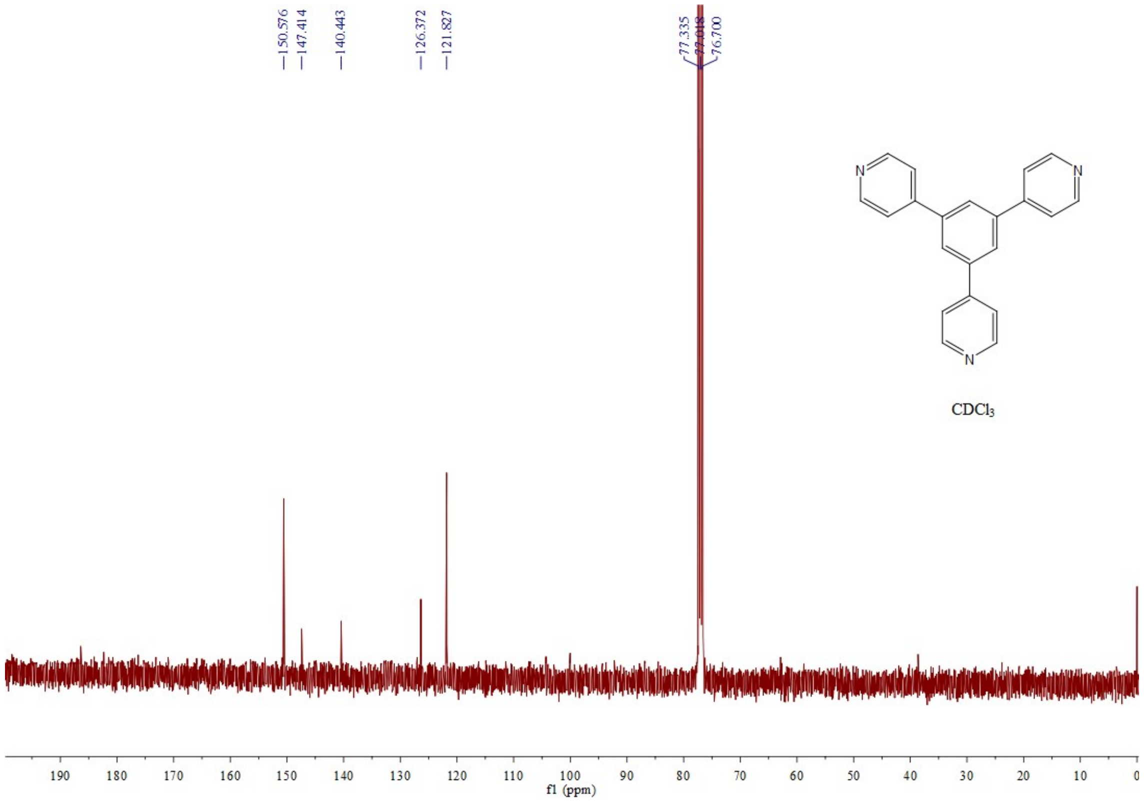
1,3,5-Tris(3-thienyl)benzene (3s, Table 3).



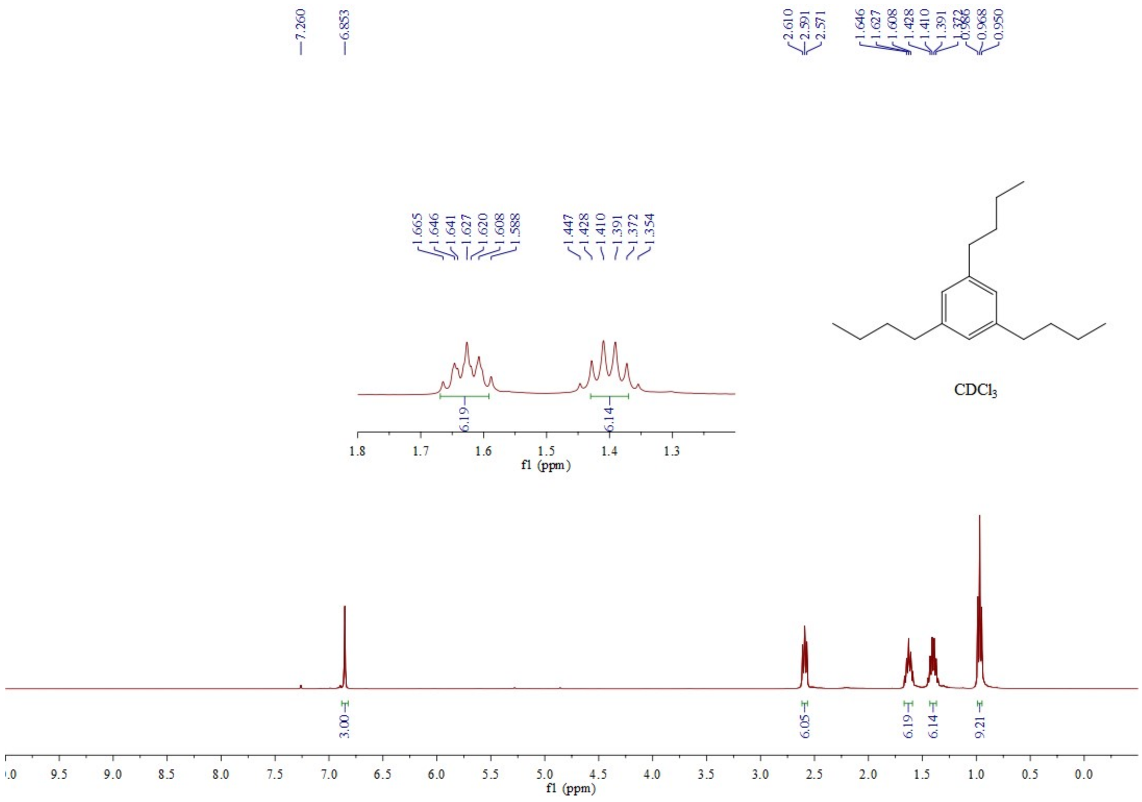


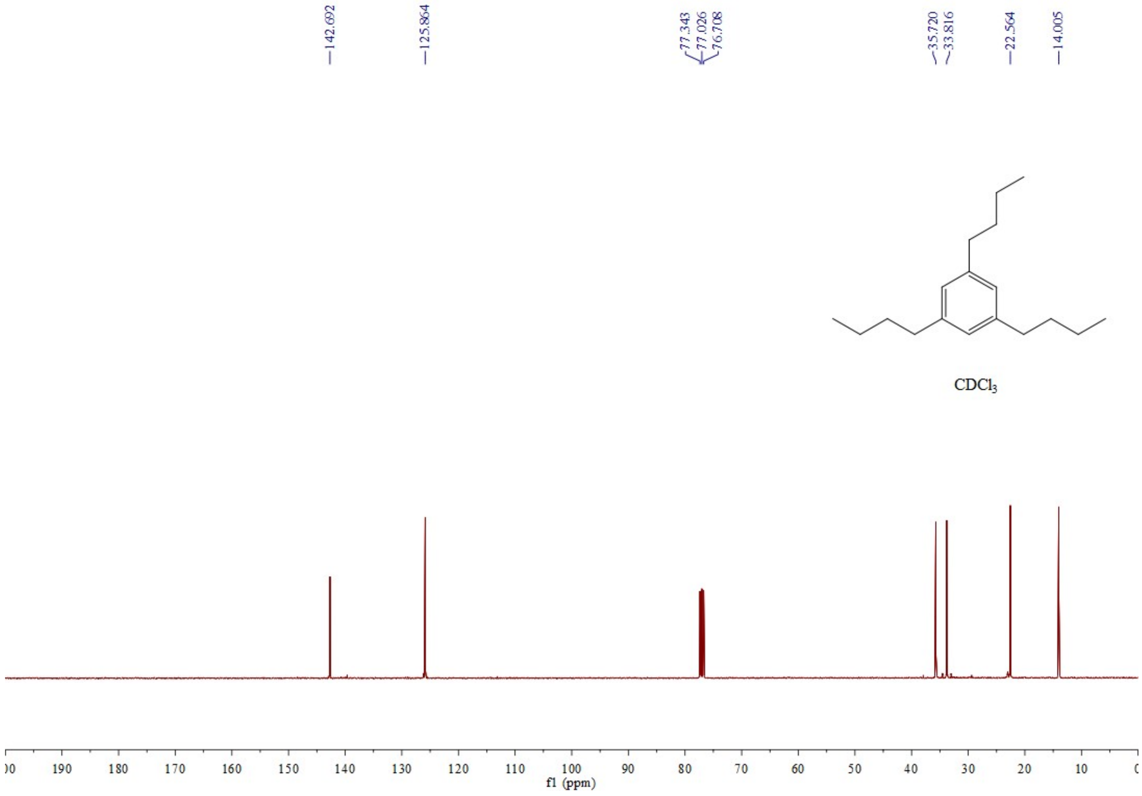
1,3,5-Tris(4-pyridyl)benzene (3t, Table 3).



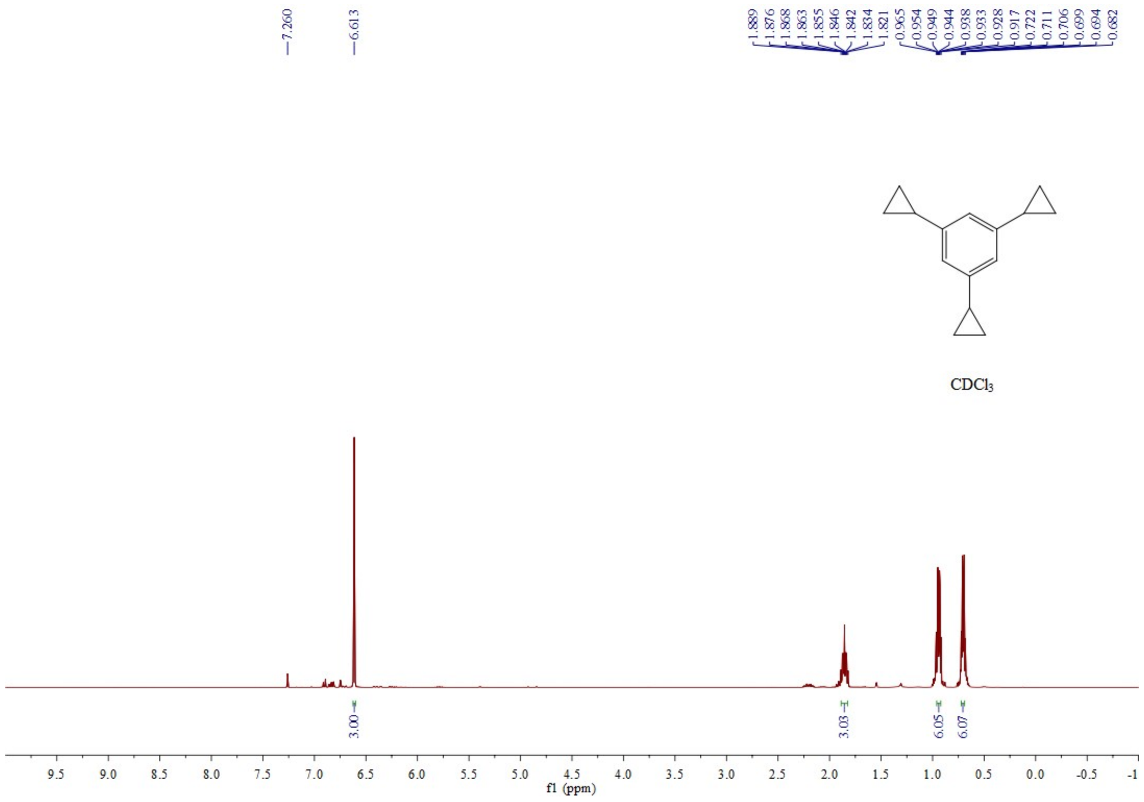


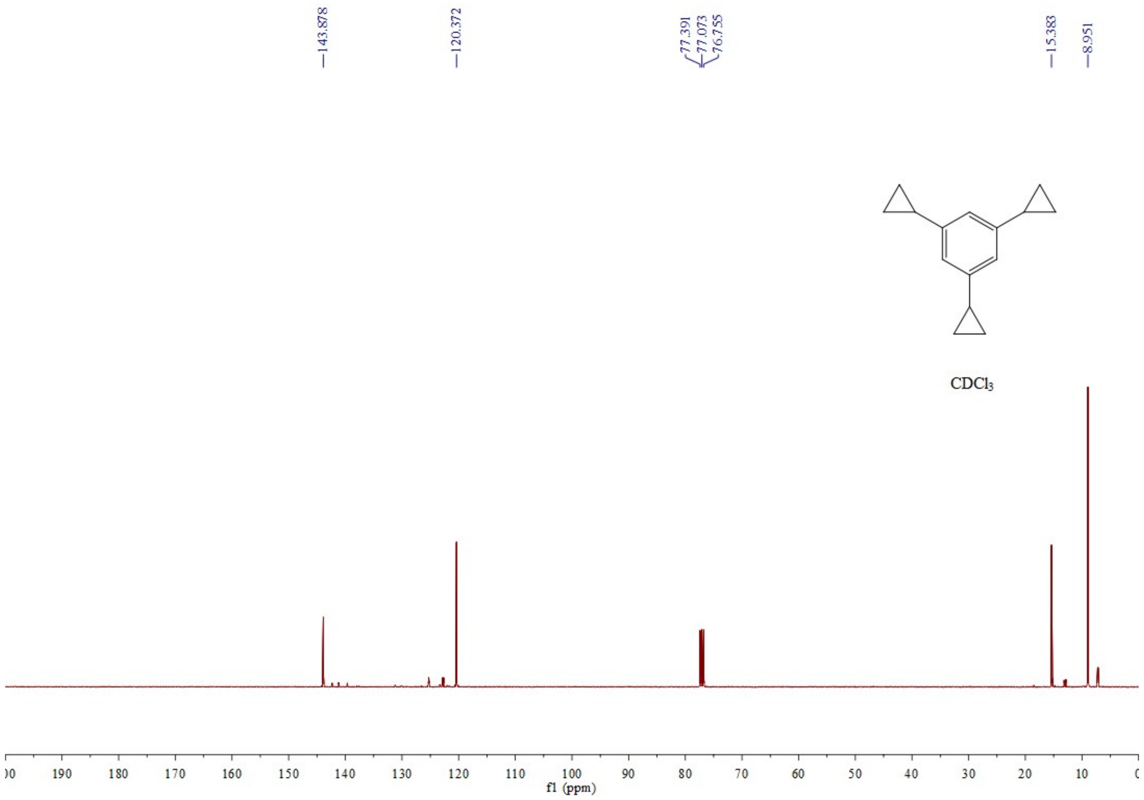
1,3,5-Tributylbenzene (3u, Table 3).



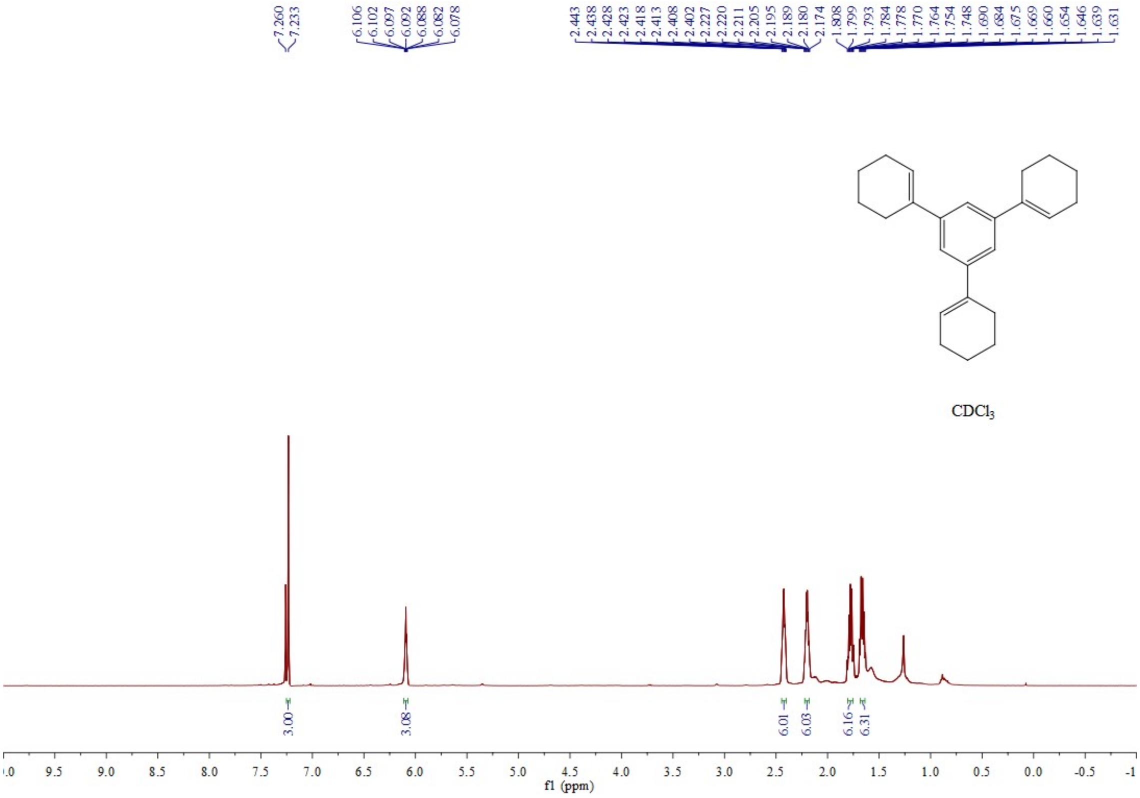


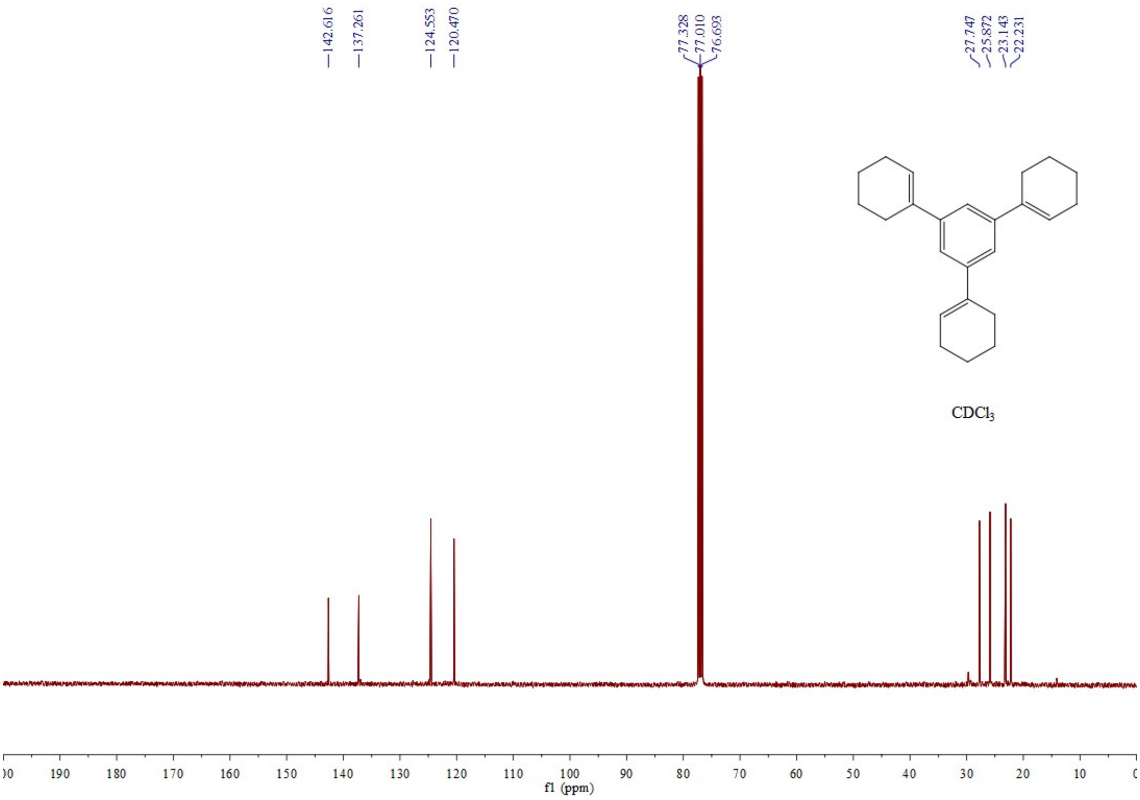
1,3,5-Tricyclopropylbenzene (3v, Table 3).



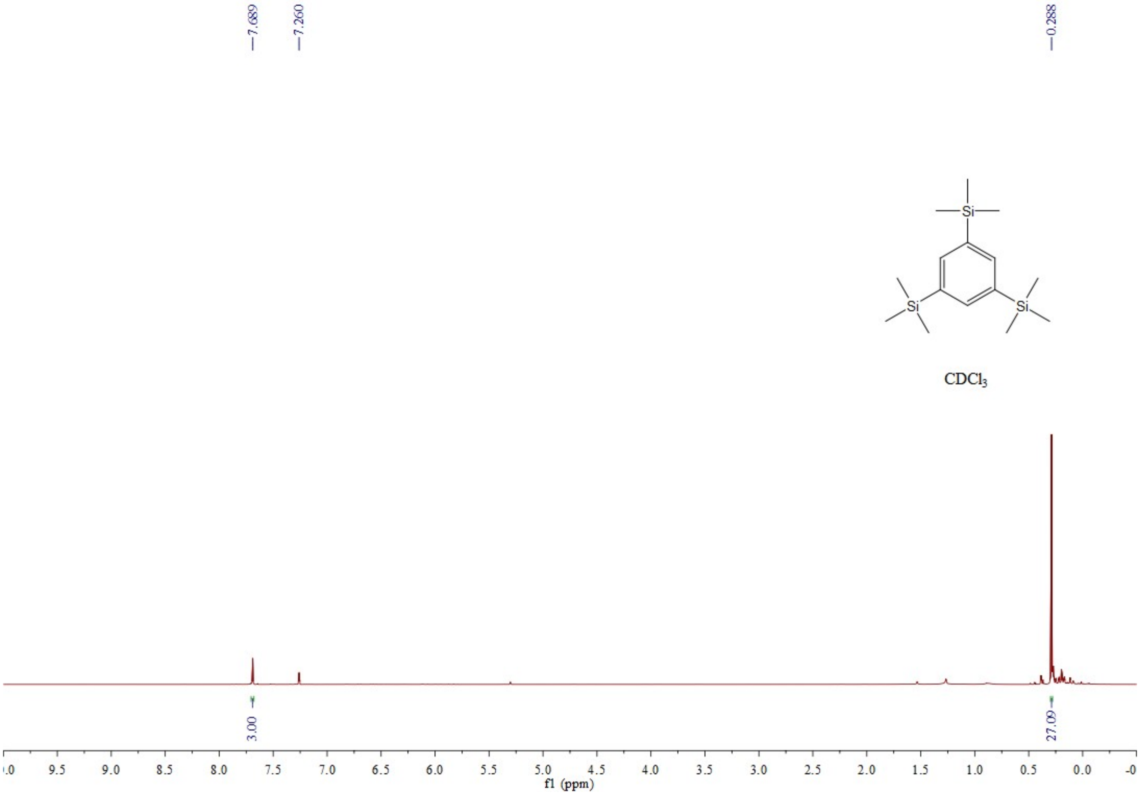


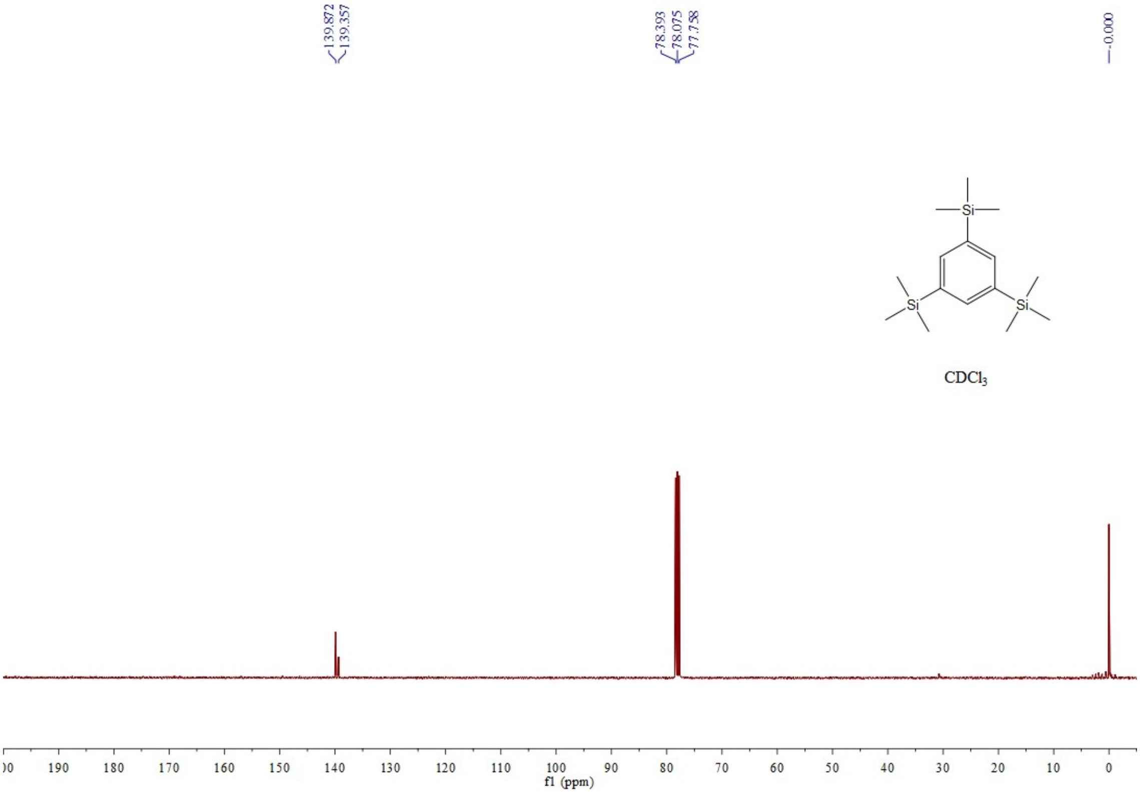
1,3,5-Tris(1-cyclohexenyl)benzene (3w, Table 3).



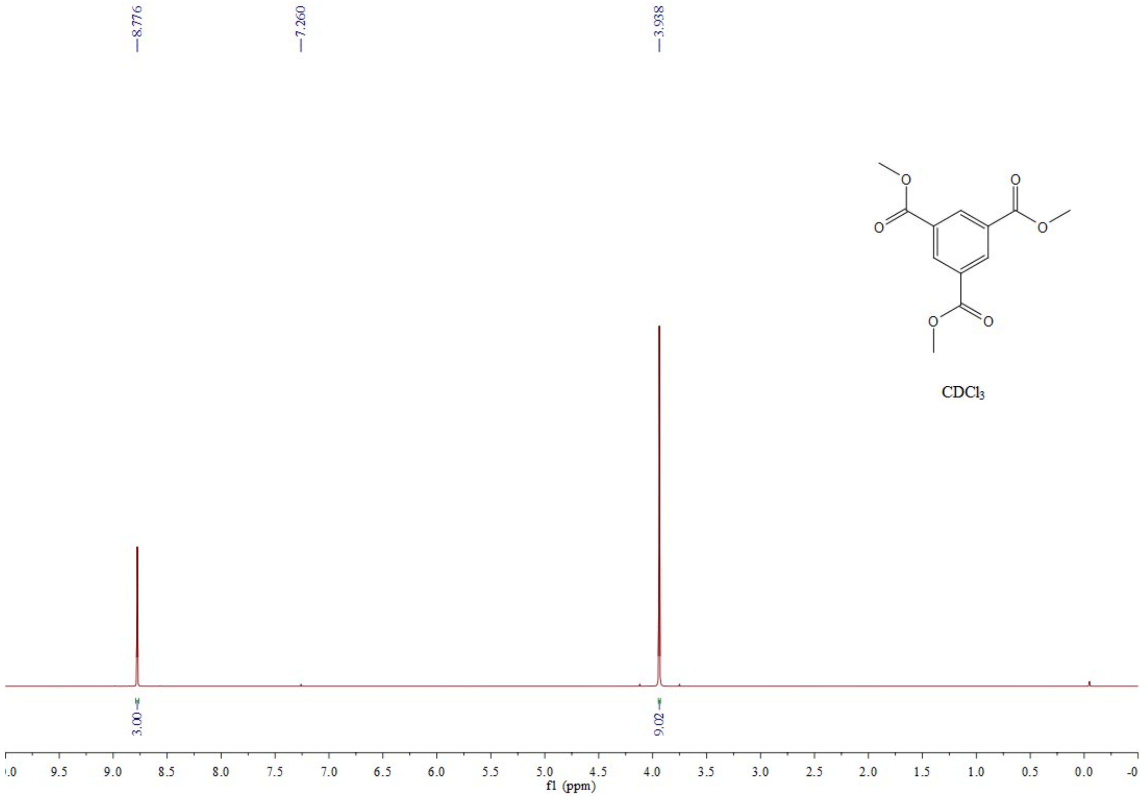


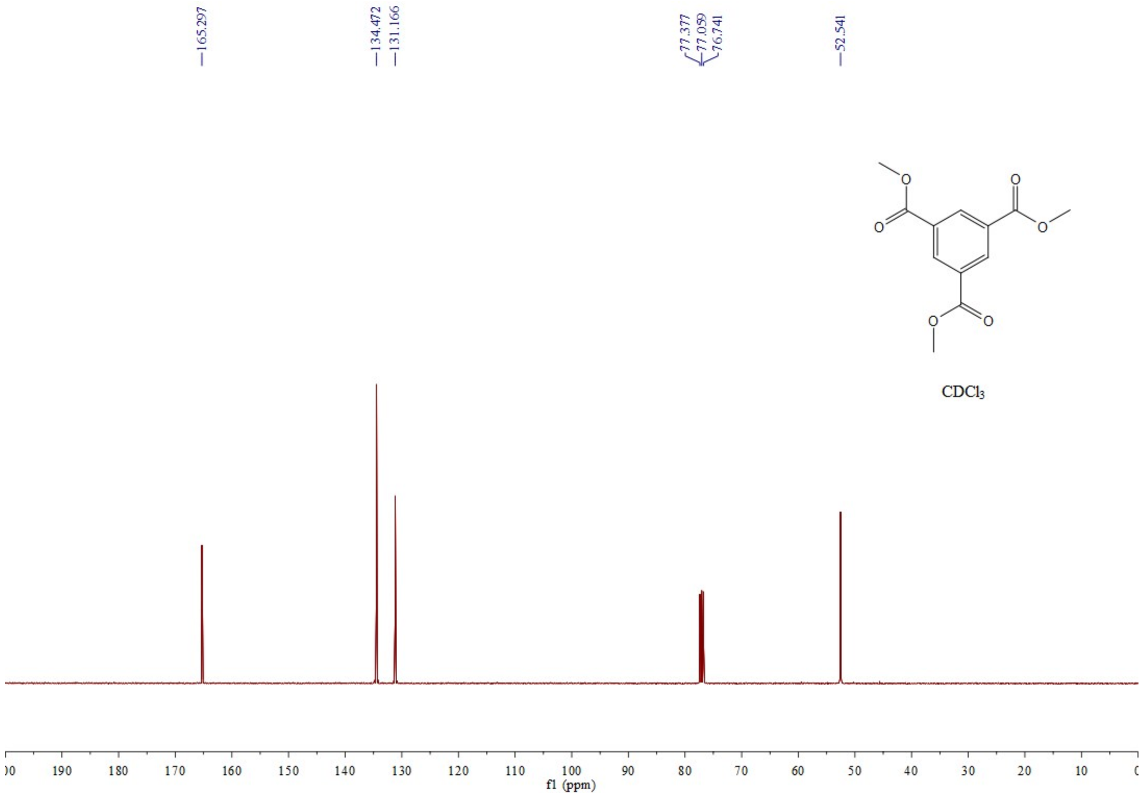
1,3,5-Tris(trimethylsilyl)benzene (3x, Table 3).



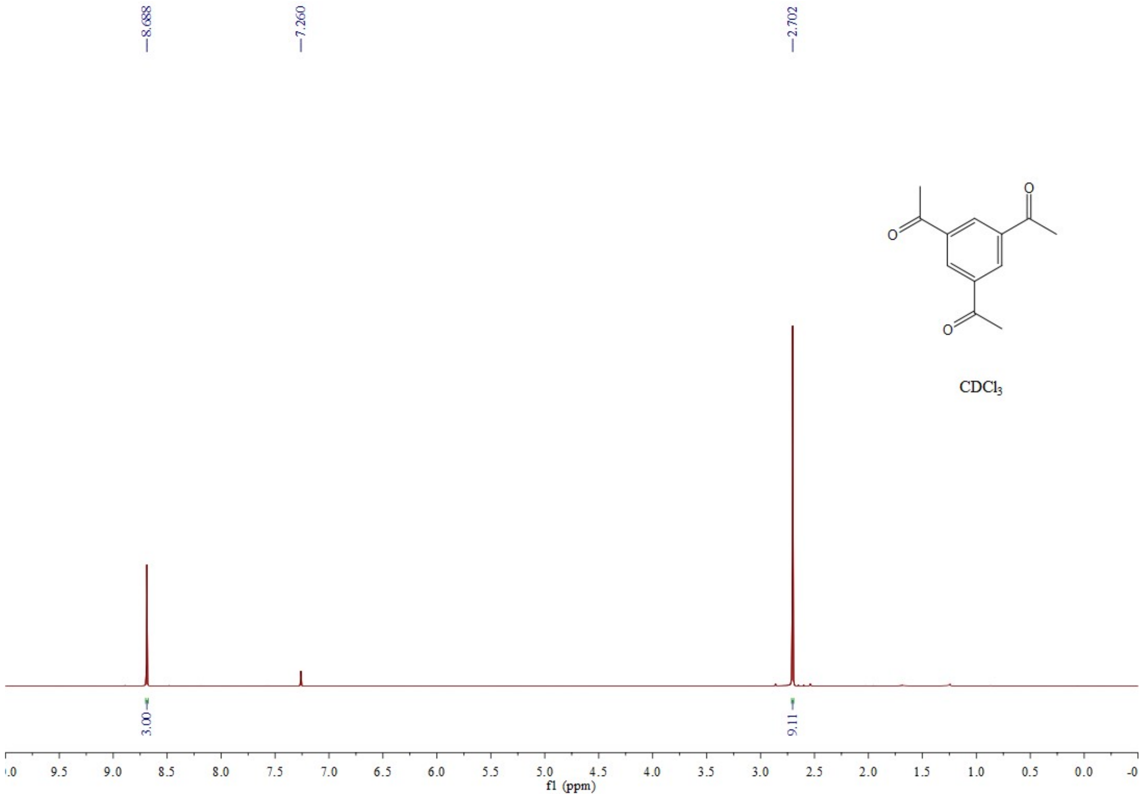


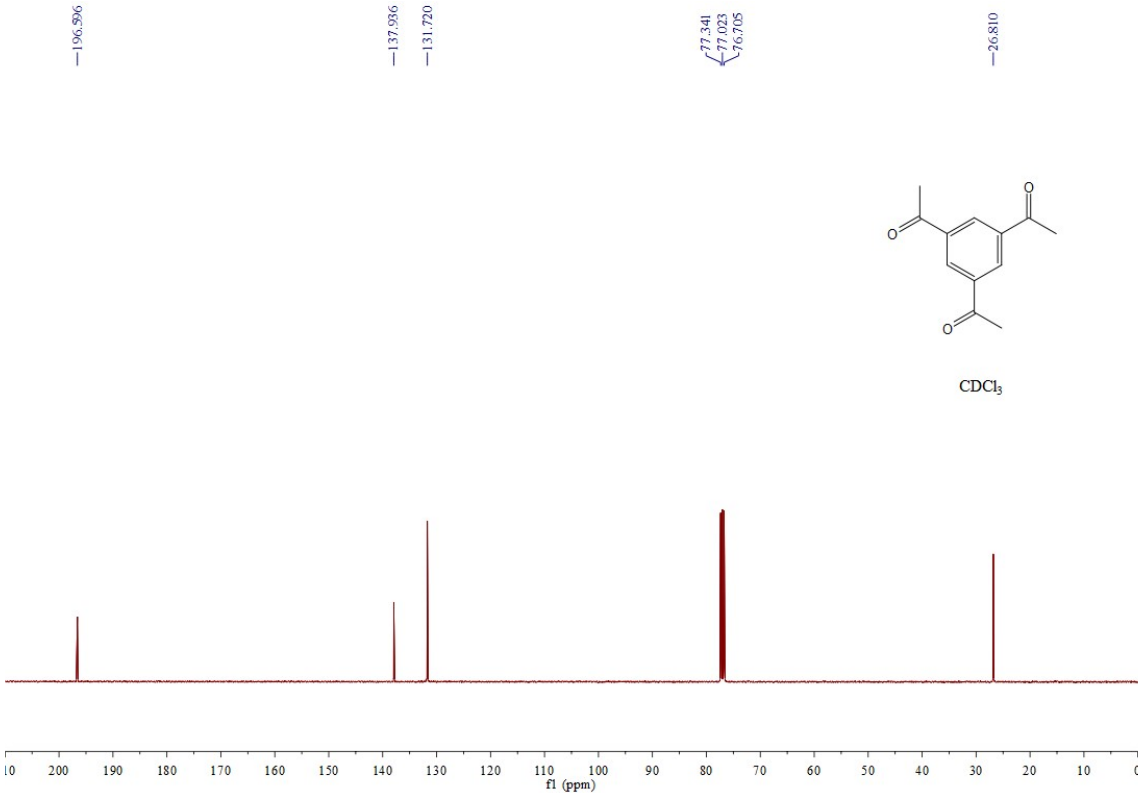
Benzene-1,3,5-tricarboxylic acid trimethyl ester (3y, Table 3).



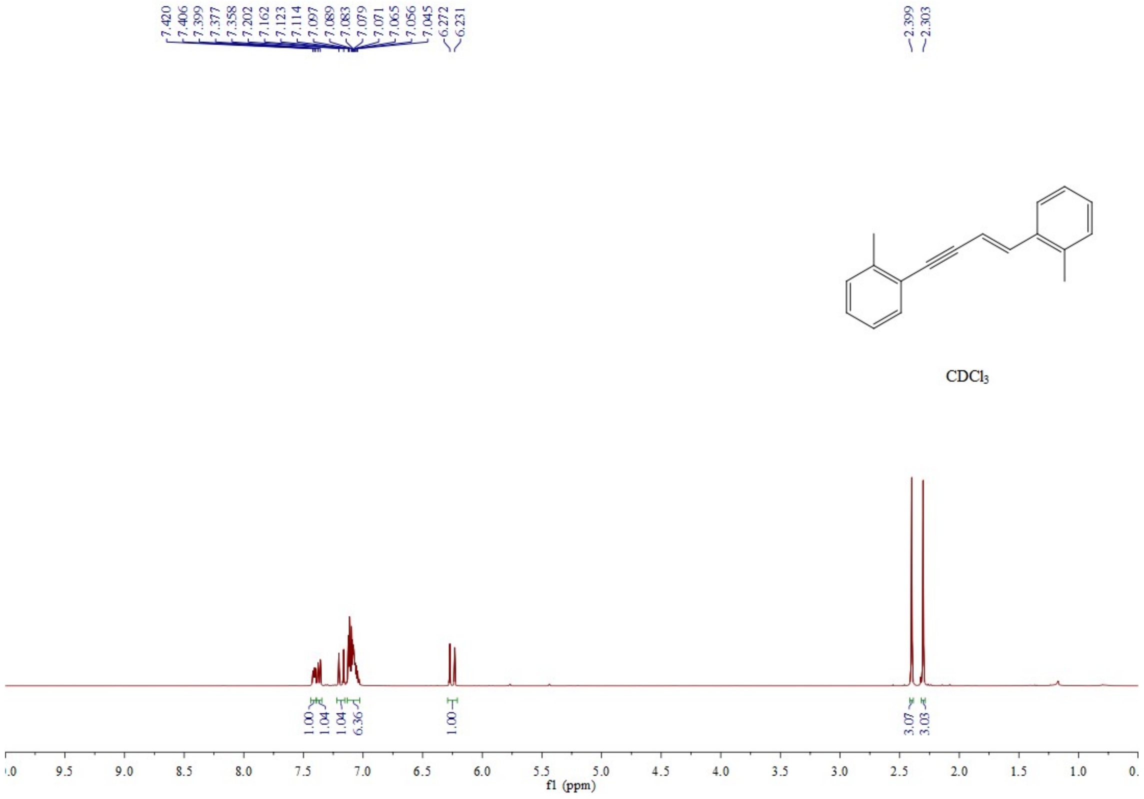


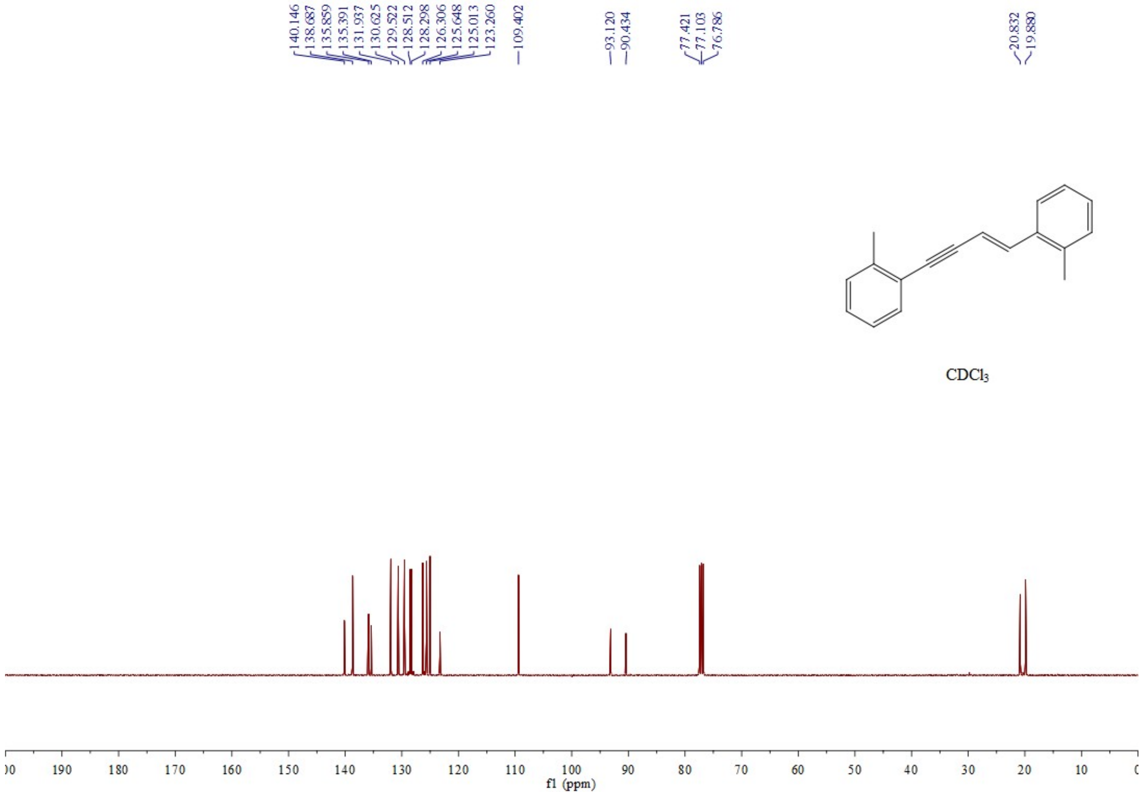
1,3,5-Triacetylphenylbenzene (3z, Table 3).



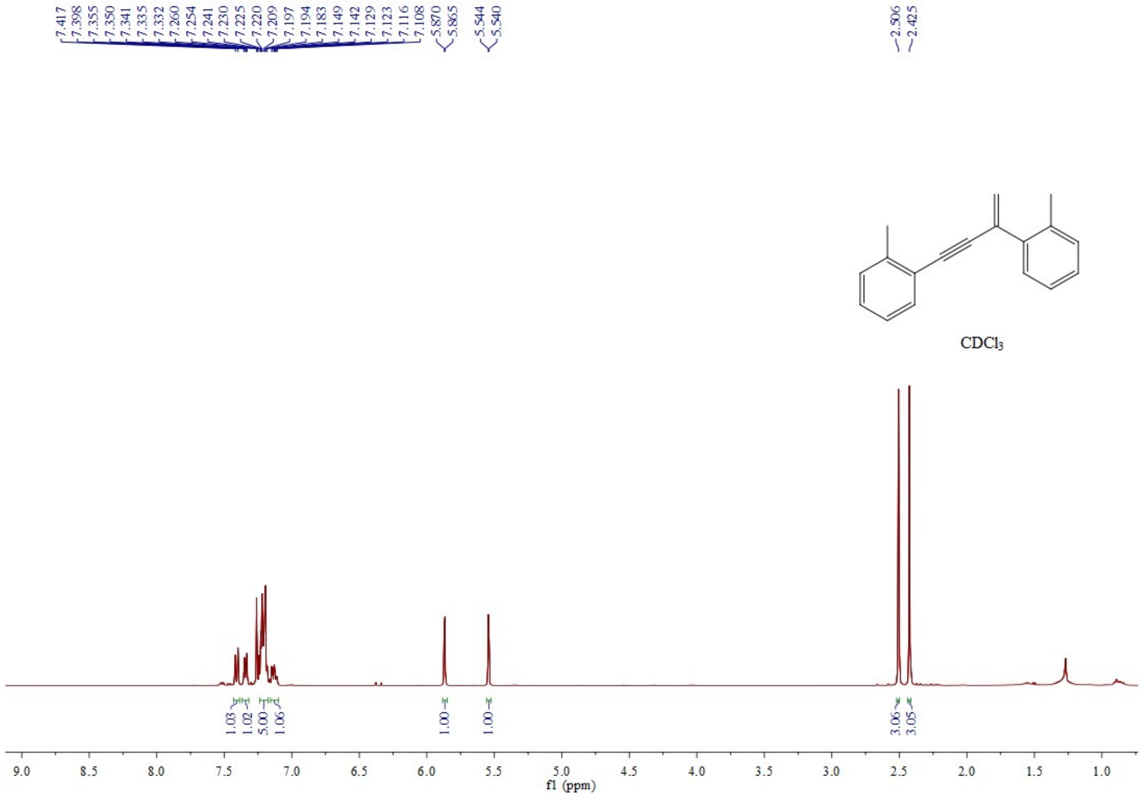


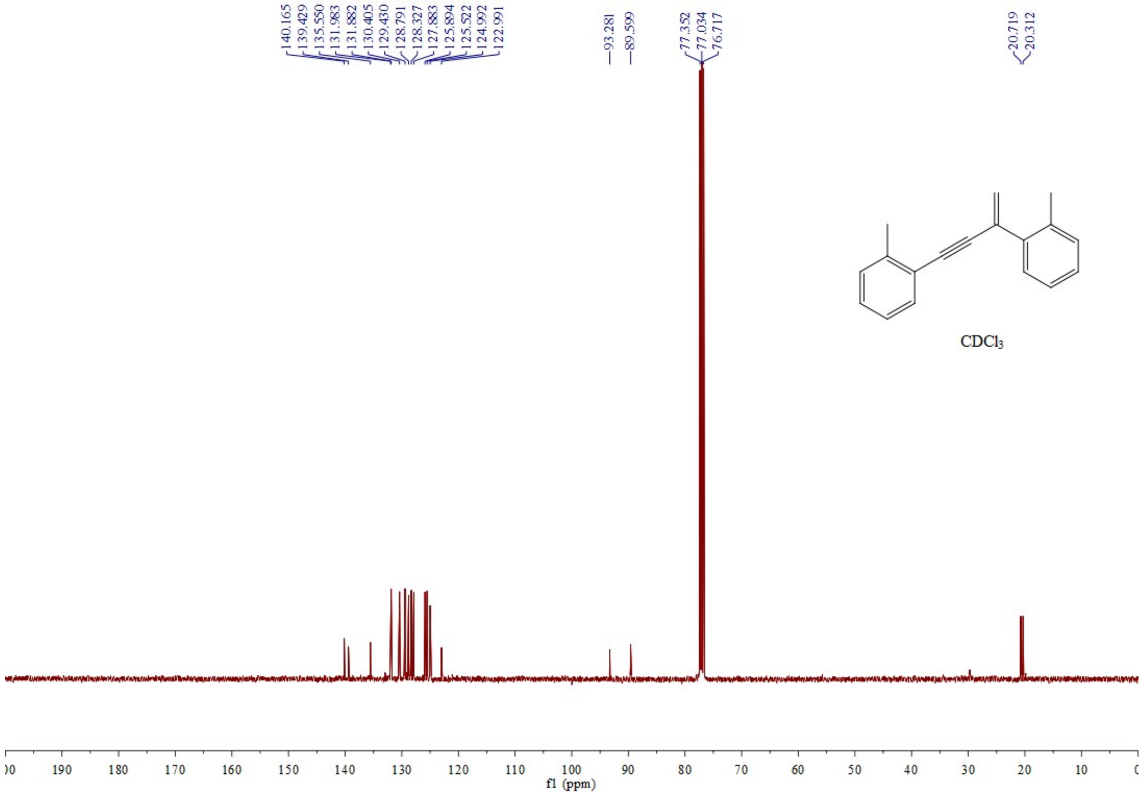
(E)-1,4-bis(2-methylphenyl)but-1-en-3-yne (4pa, Scheme 2).



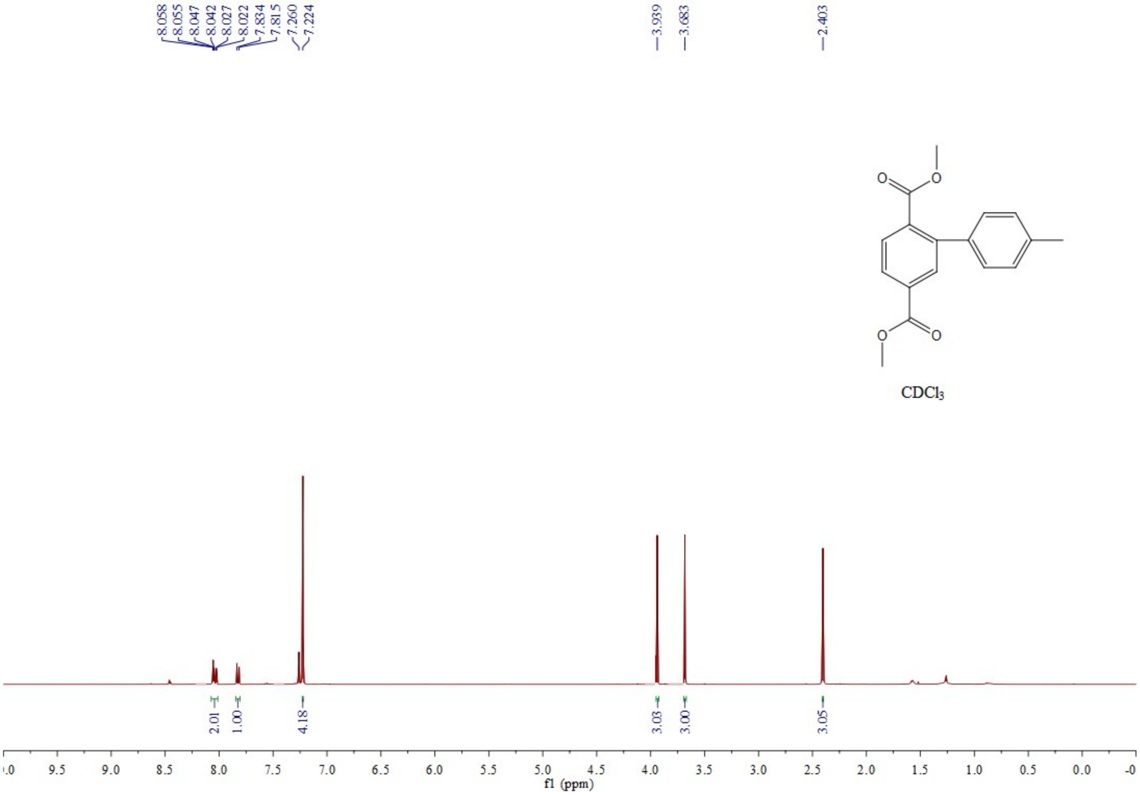


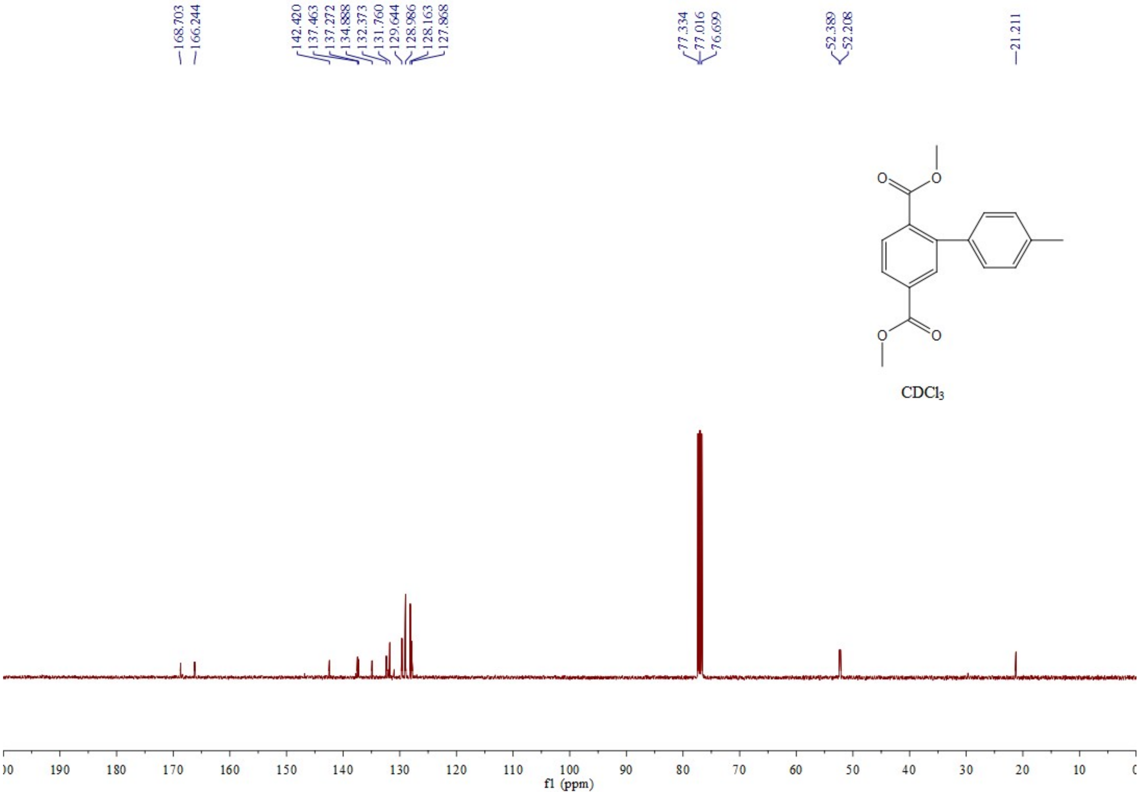
2,4-Bis(2-methylphenyl)but-1-en-3-yne (4pb, Scheme 2).



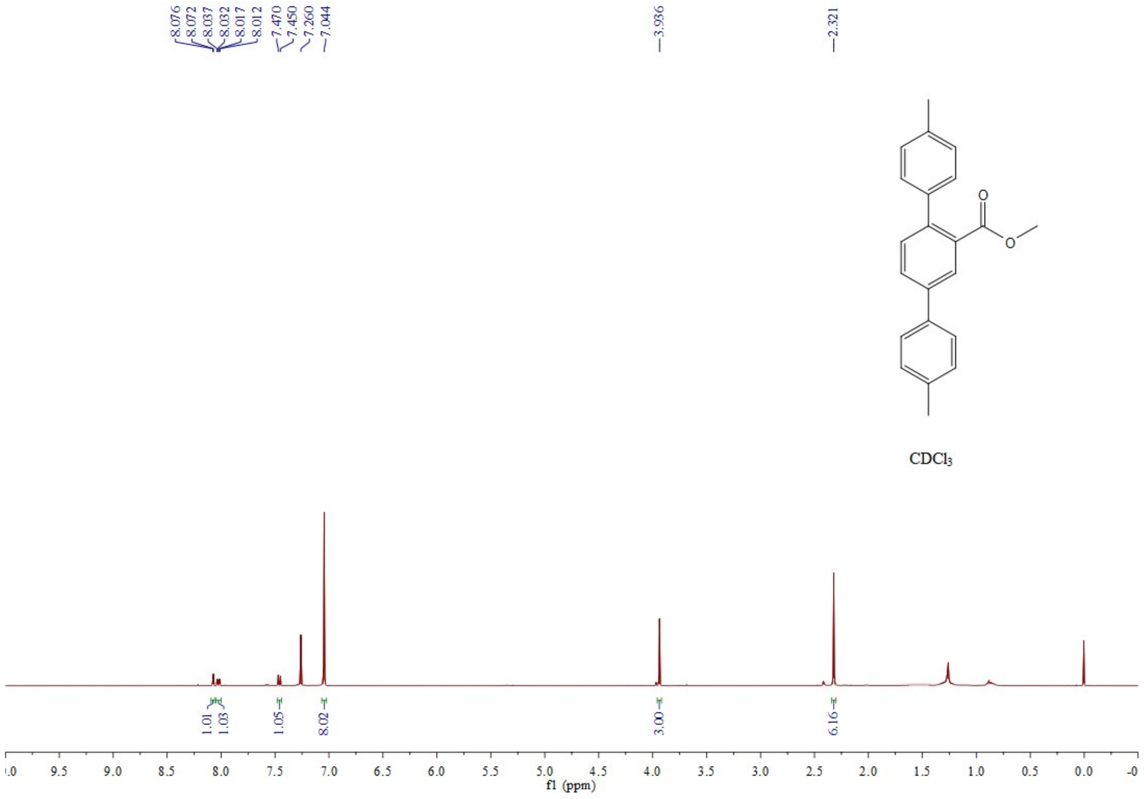


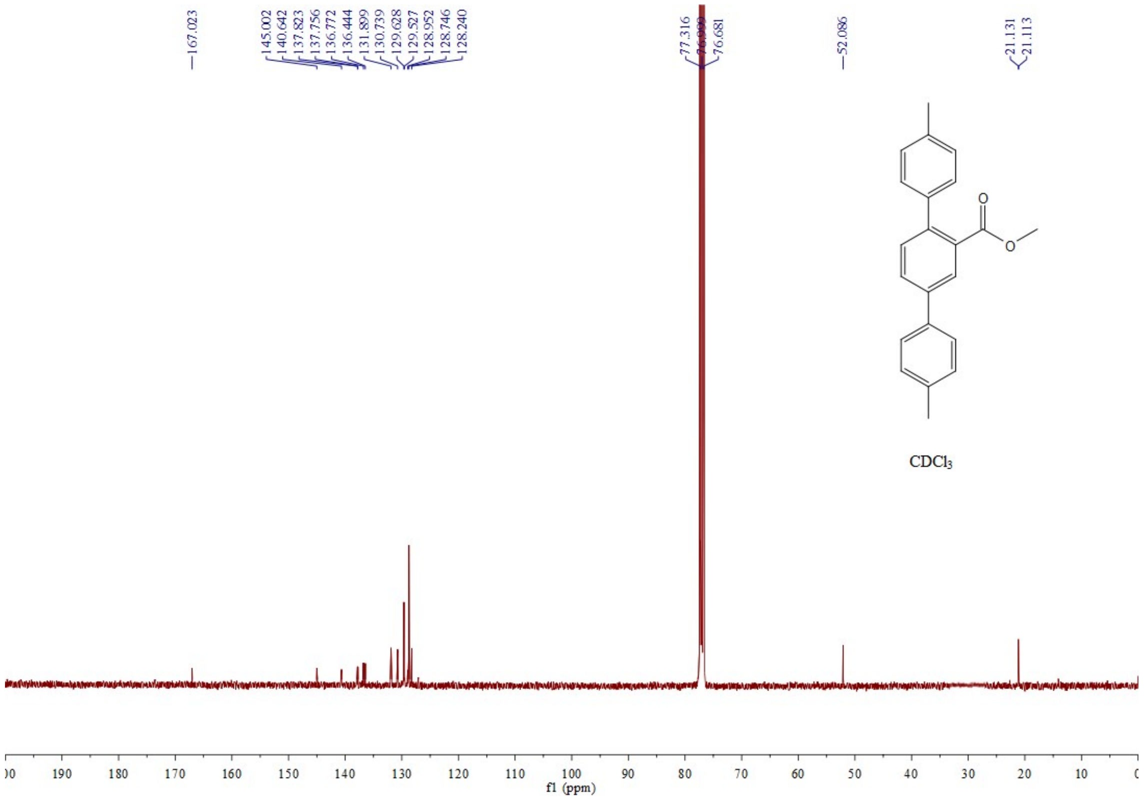
2,5-Dimethyl 4'-methyl[1,1'-biphenyl]-2,5-dicarboxylate (5a, Table 4).



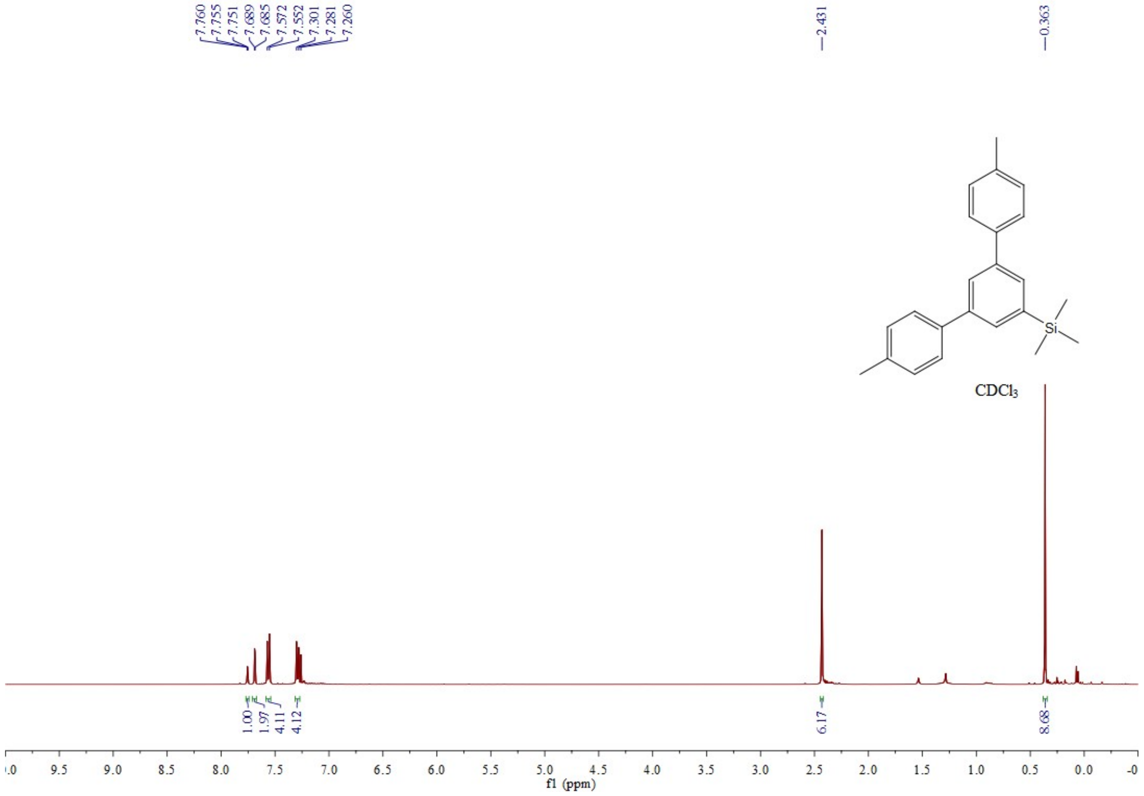


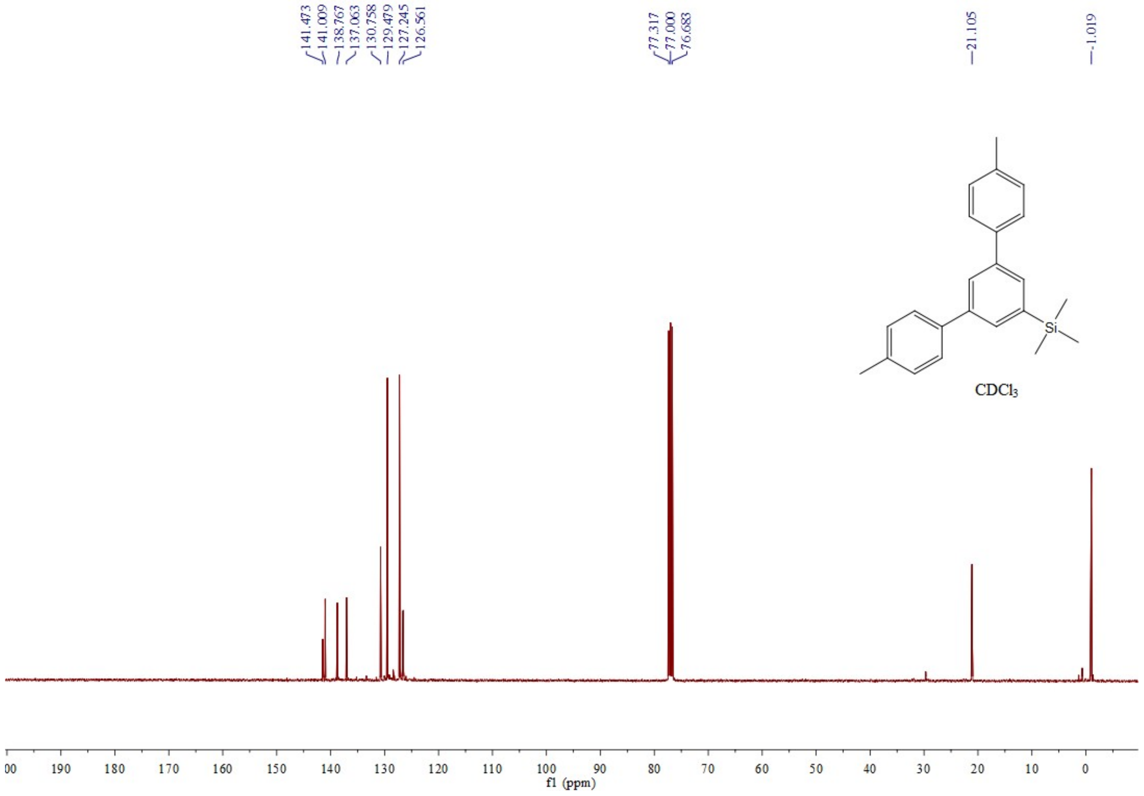
Methyl 4,4''-dimethyl-[1,1':4',1''-terphenyl]-2'-carboxylate (5b, Table 4).



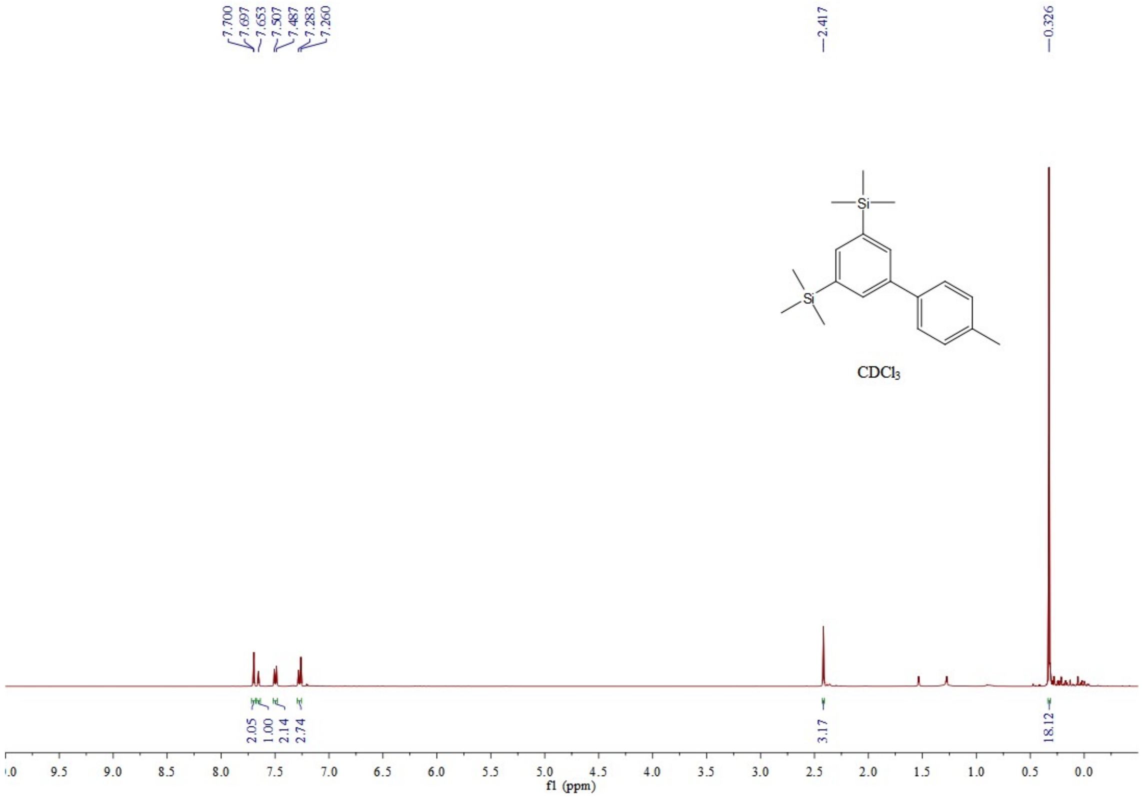


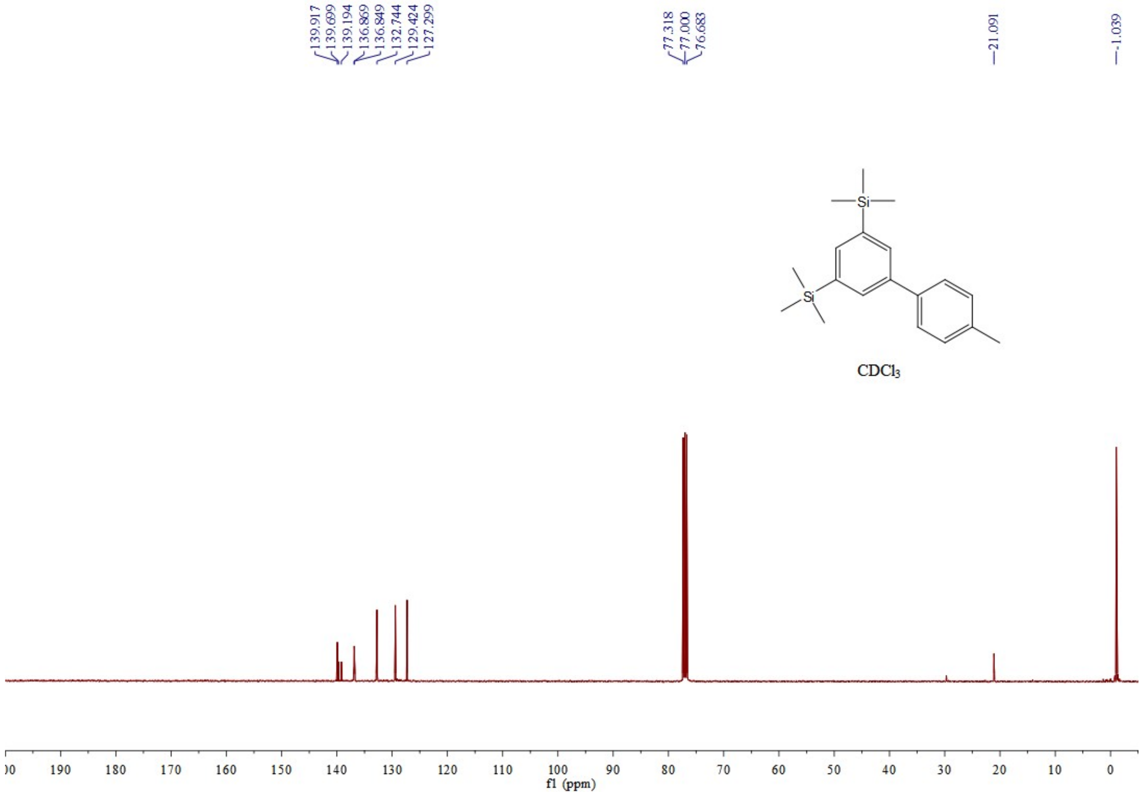
(4,4''-dimethyl-[1,1':3',1''-terphenyl]-5'-yl)trimethylsilane (6a, Table 4).



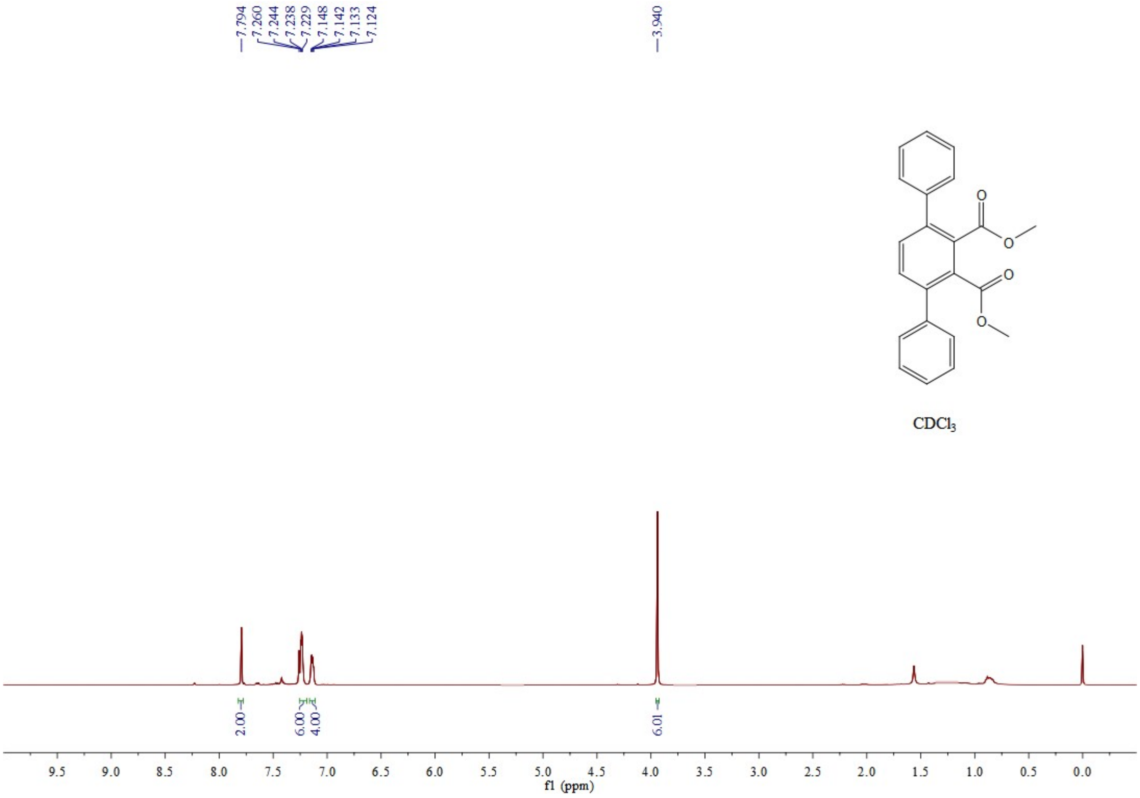


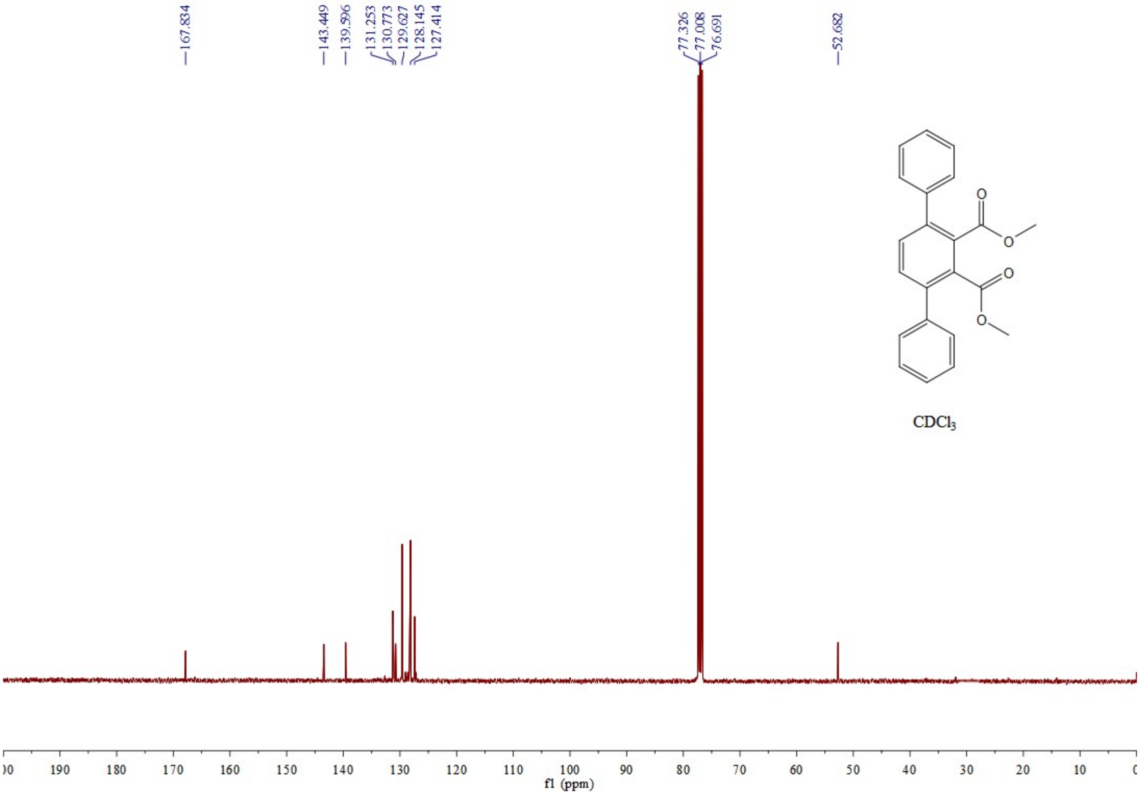
(4'-methyl-[1,1'-biphenyl]-3,5-diyl)bis(trimethylsilane) (6b, Table 4).



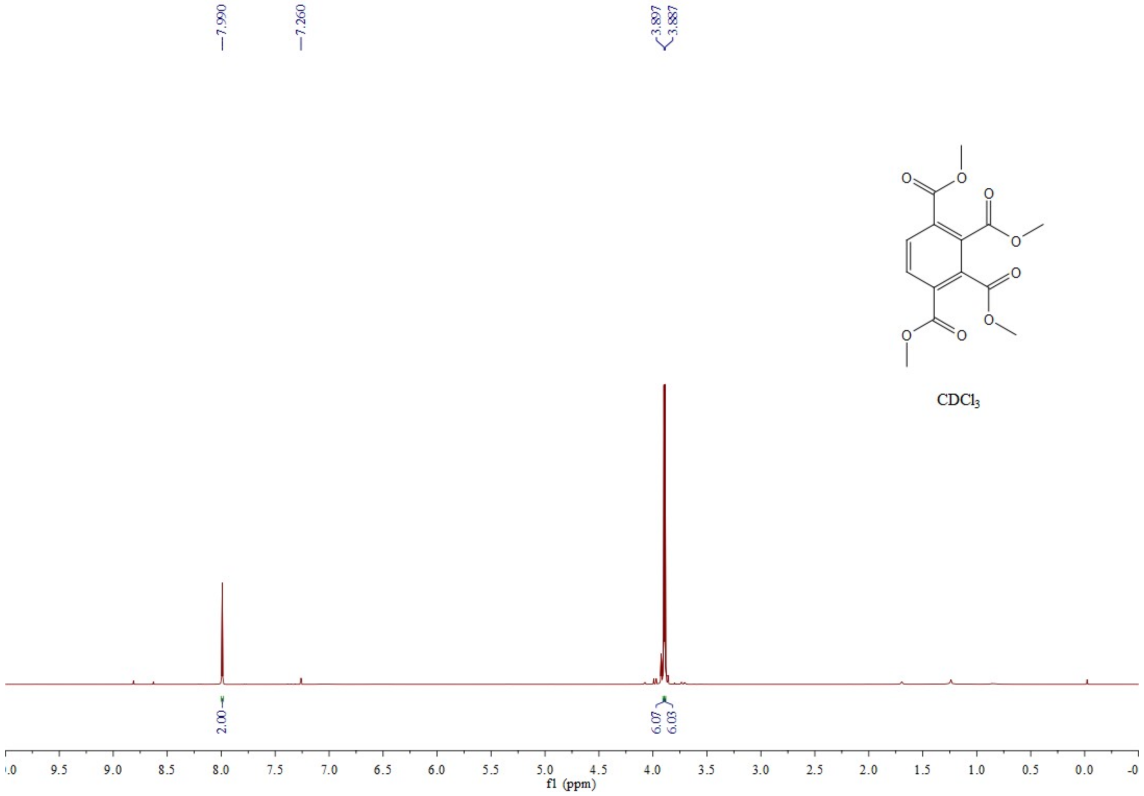


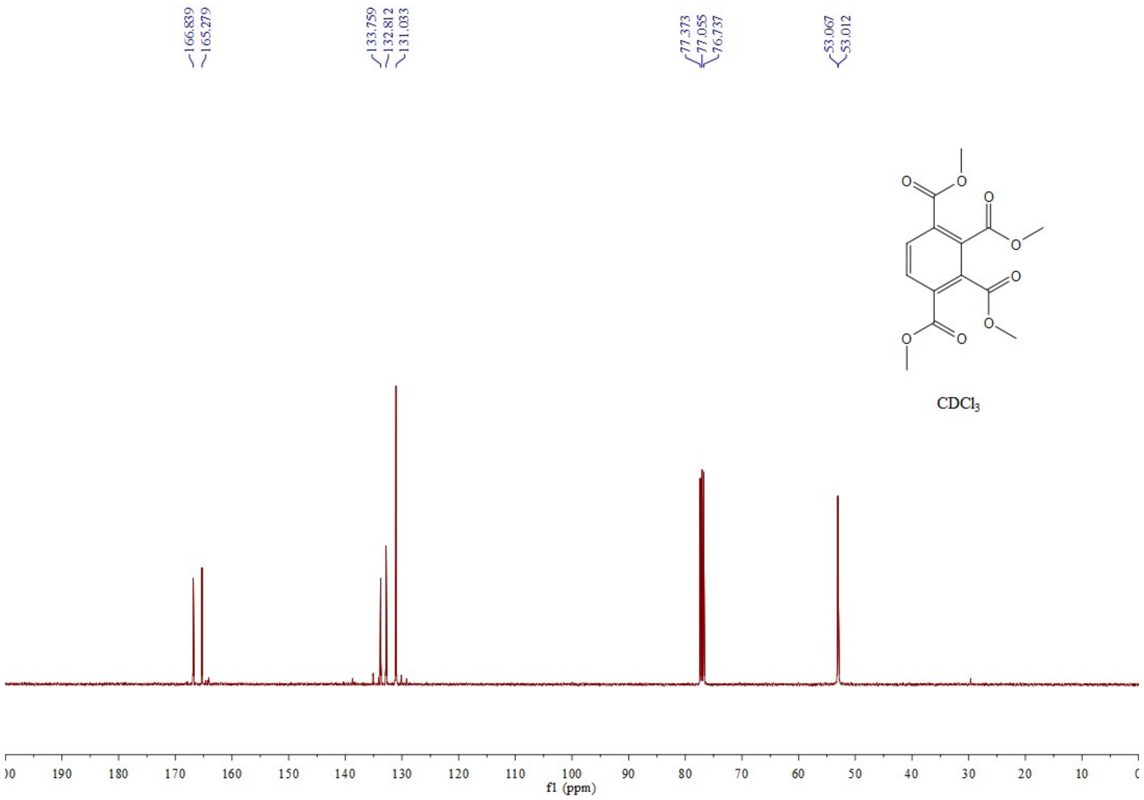
Dimethyl [1,1':4',1''-terphenyl]-2',3'-dicarboxylate (7a, Table 4).



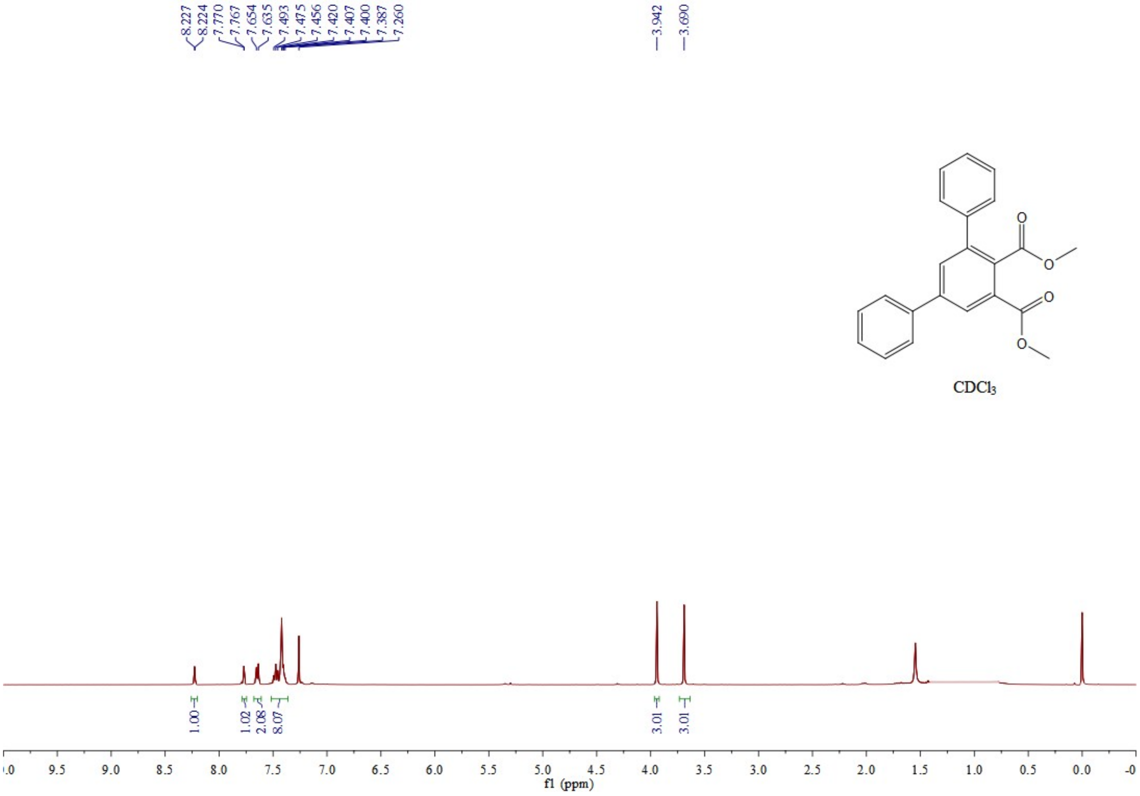


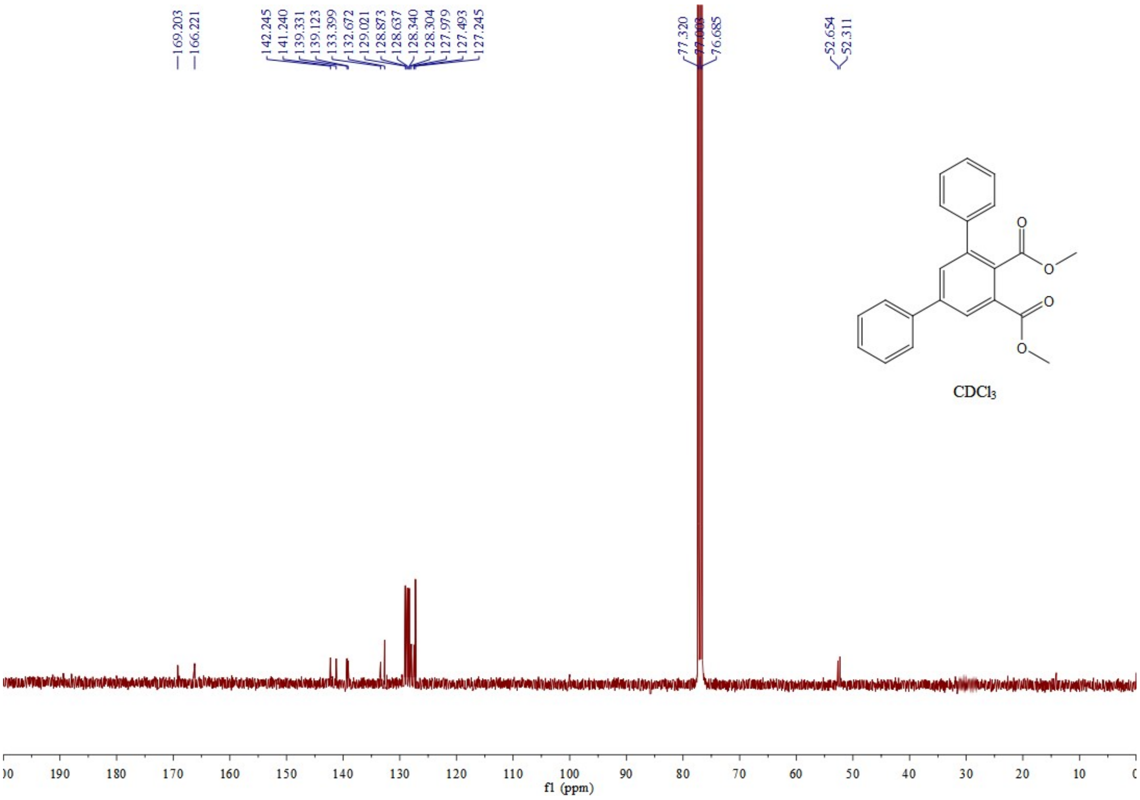
Tetramethyl benzene-1,2,3,4-tetracarboxylate (7b, Table 4).



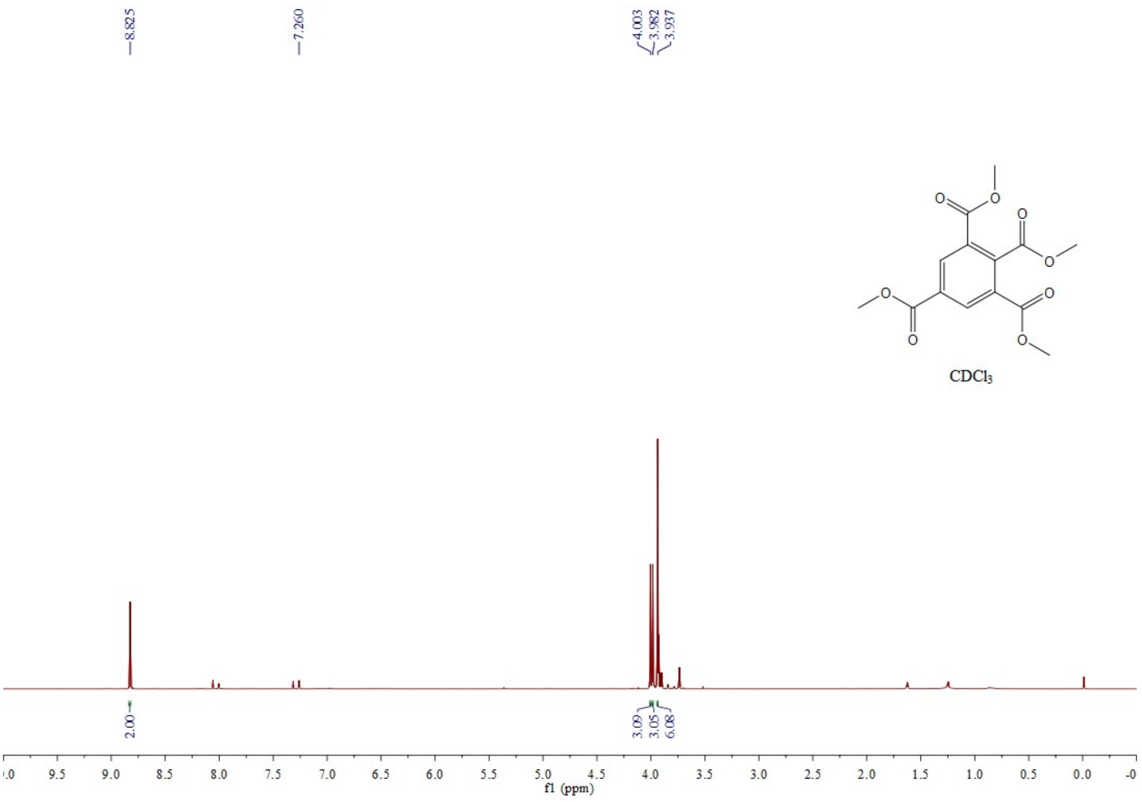


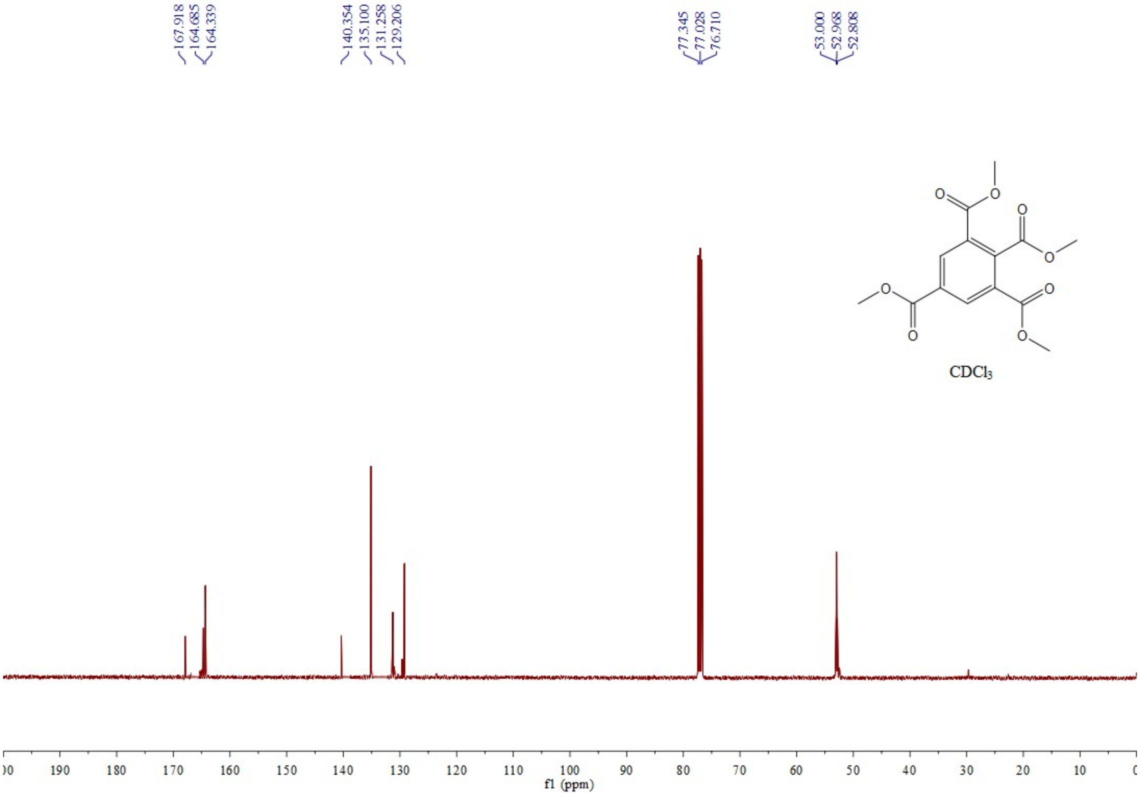
Dimethyl [1,1':3',1''-terphenyl]-4',5'-dicarboxylate (8a, Table 4).



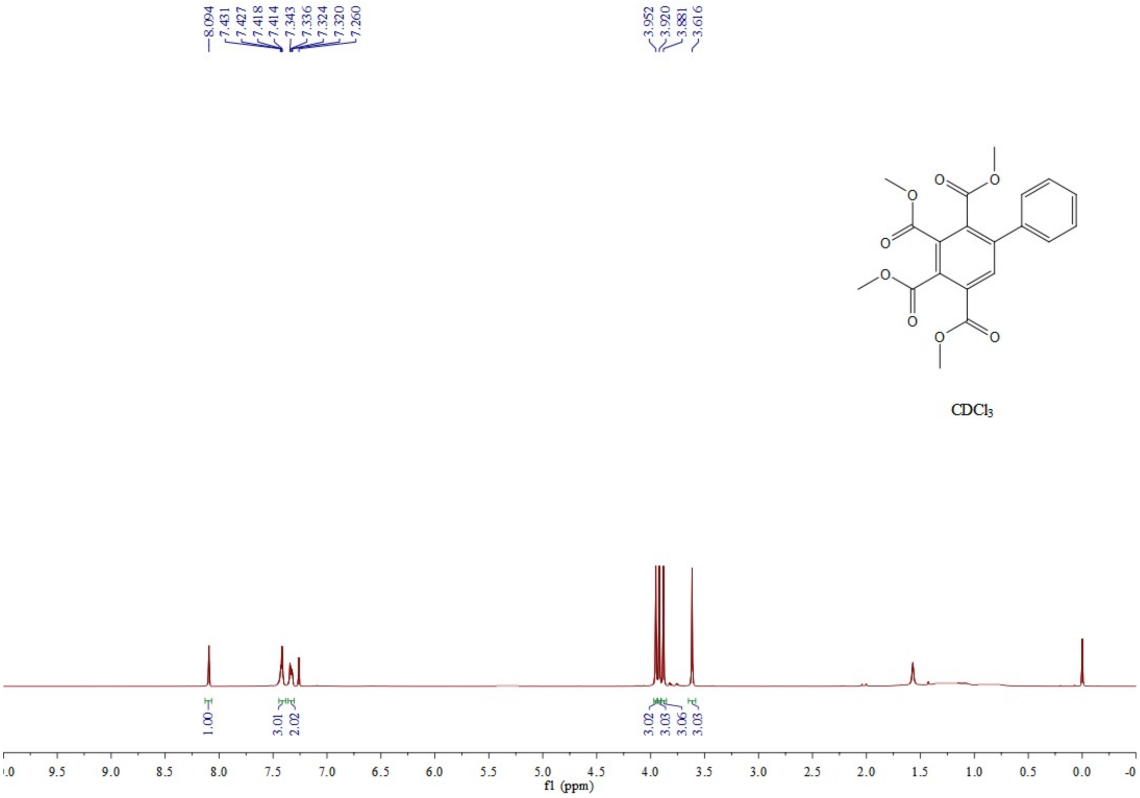


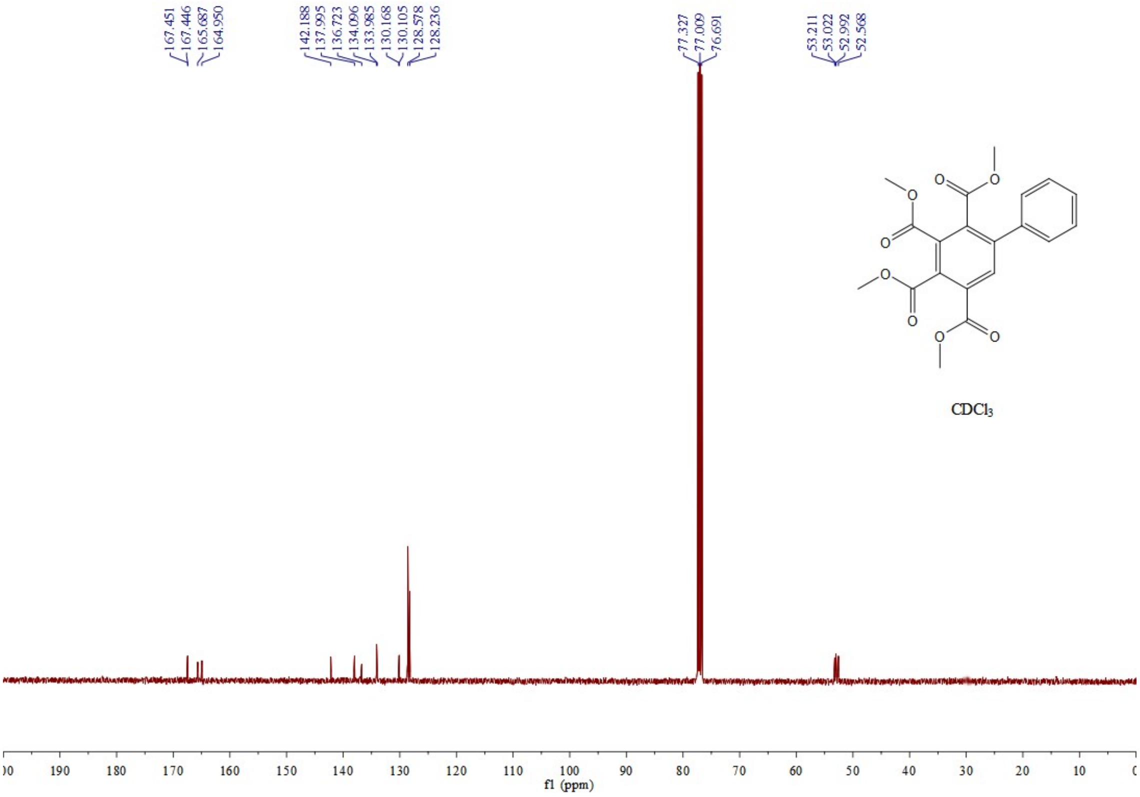
Tetramethyl benzene-1,2,3,5-tetracarboxylate (8b, Table 4).



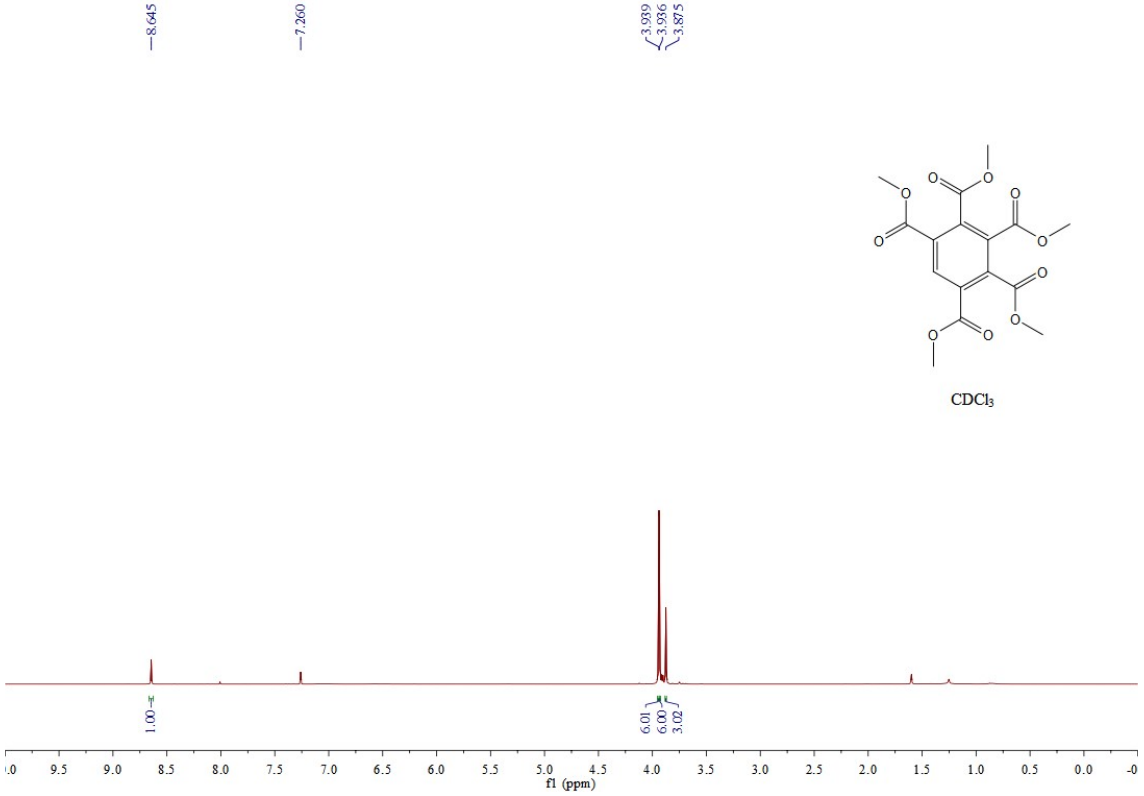


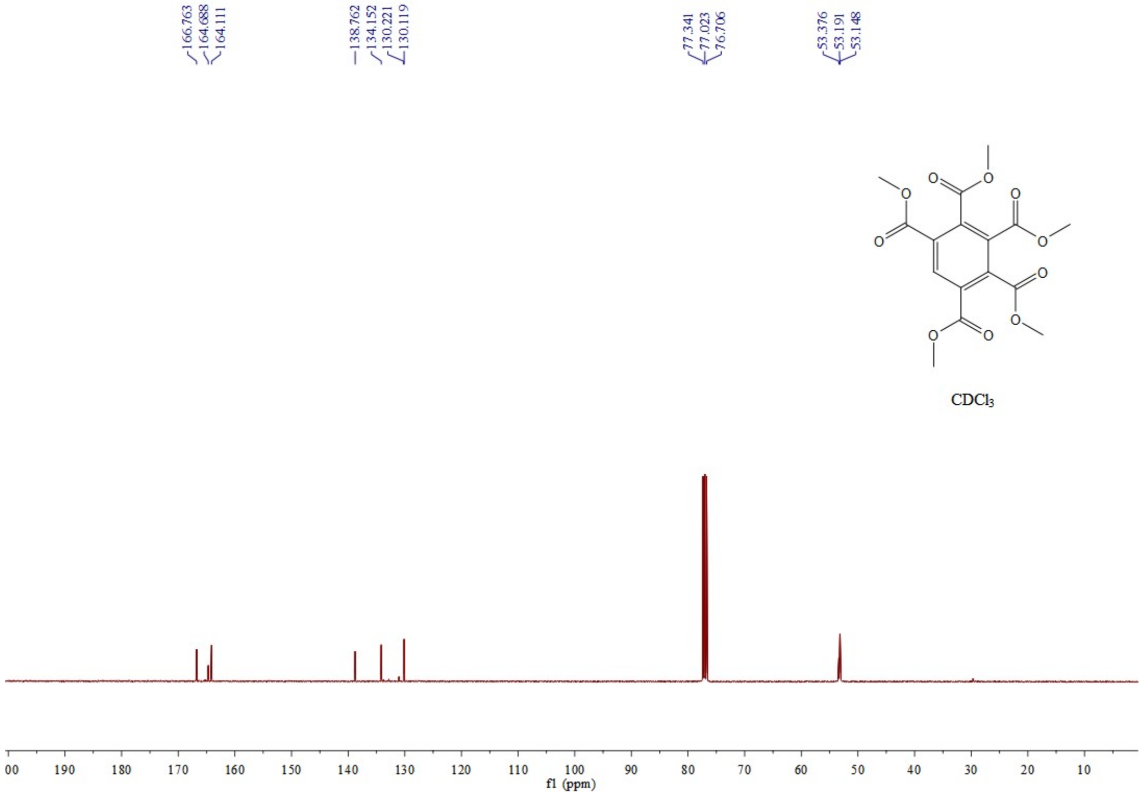
Tetramethyl [1,1'-biphenyl]-2,3,4,5-tetracarboxylate (9a, Table 4).



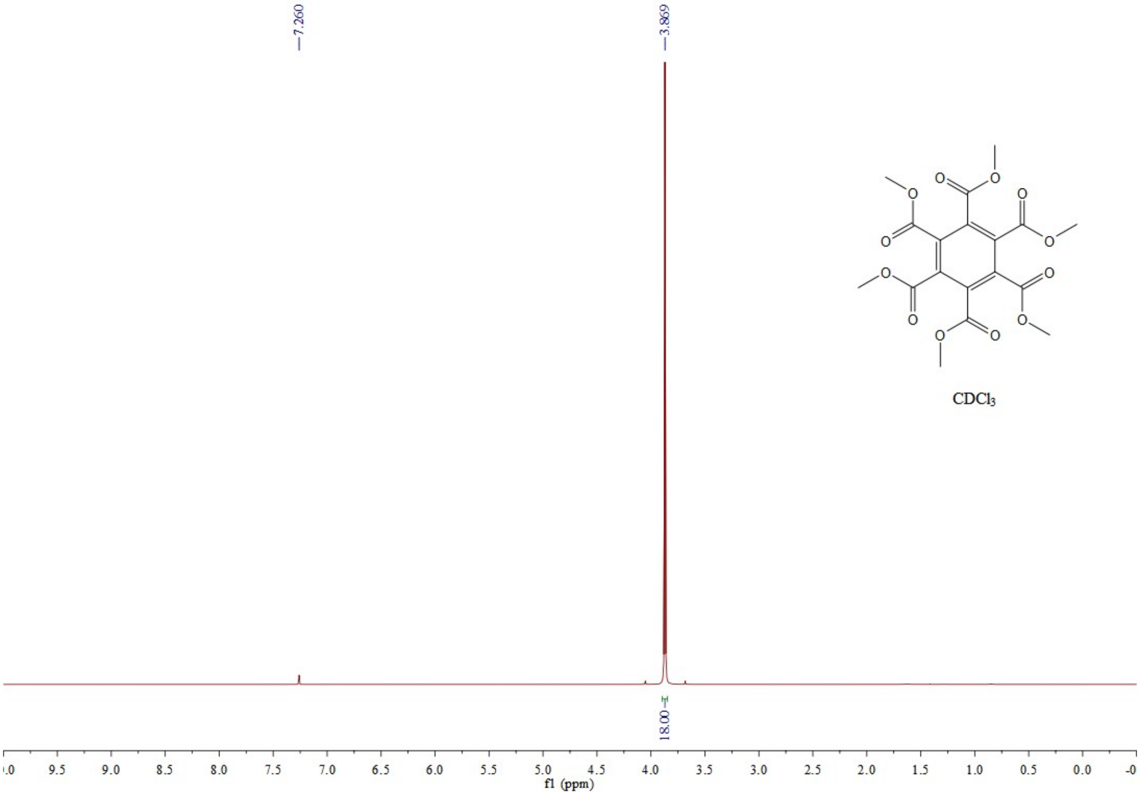


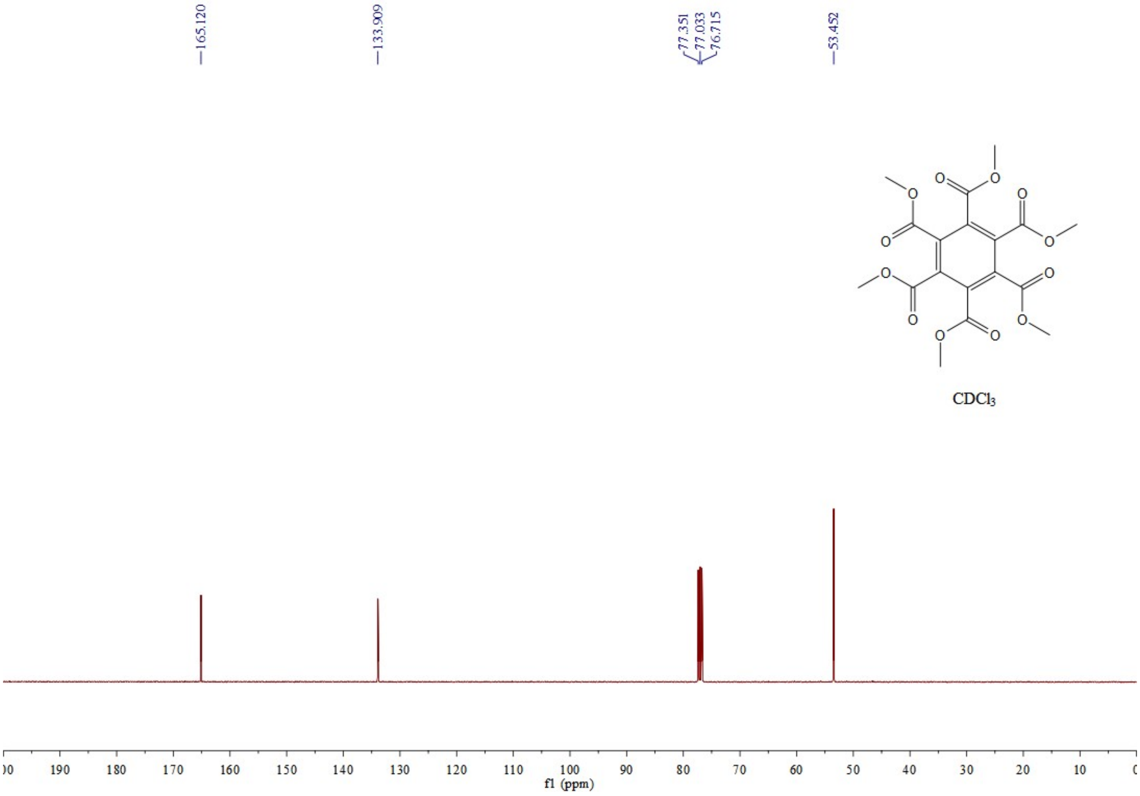
Pentamethyl benzene-1,2,3,4,5-pentacarboxylate (9b, Table 4).



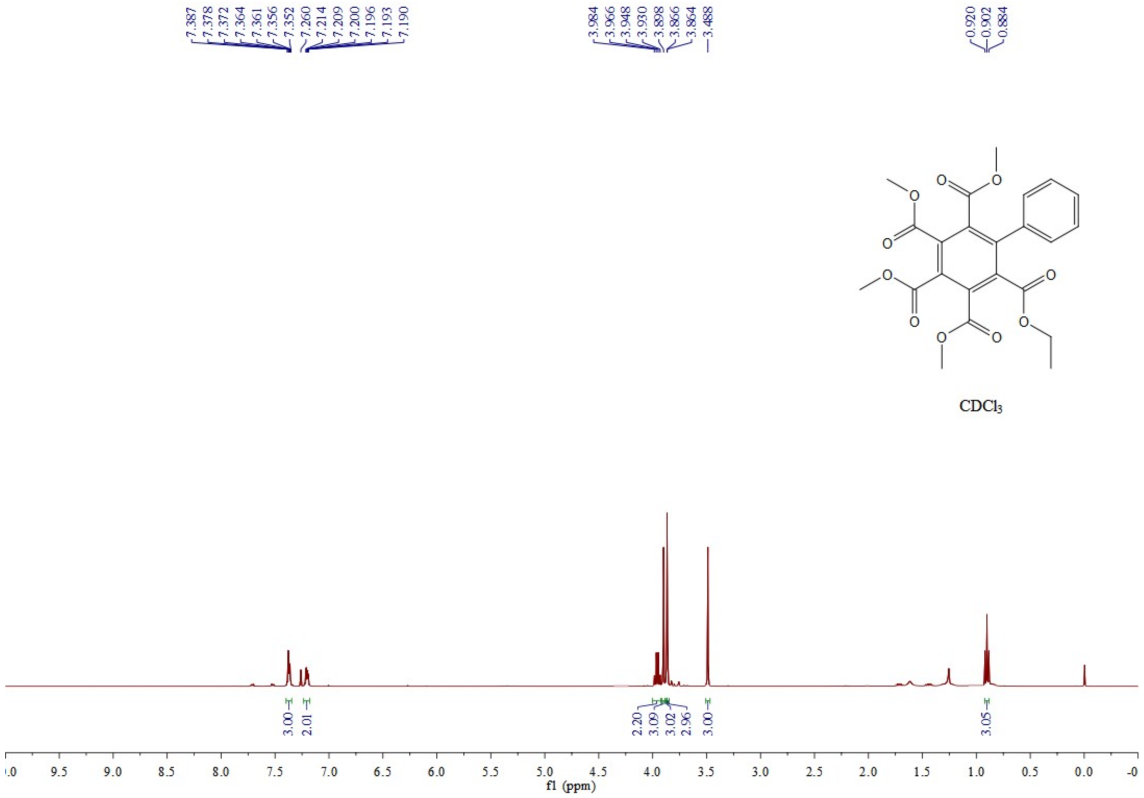


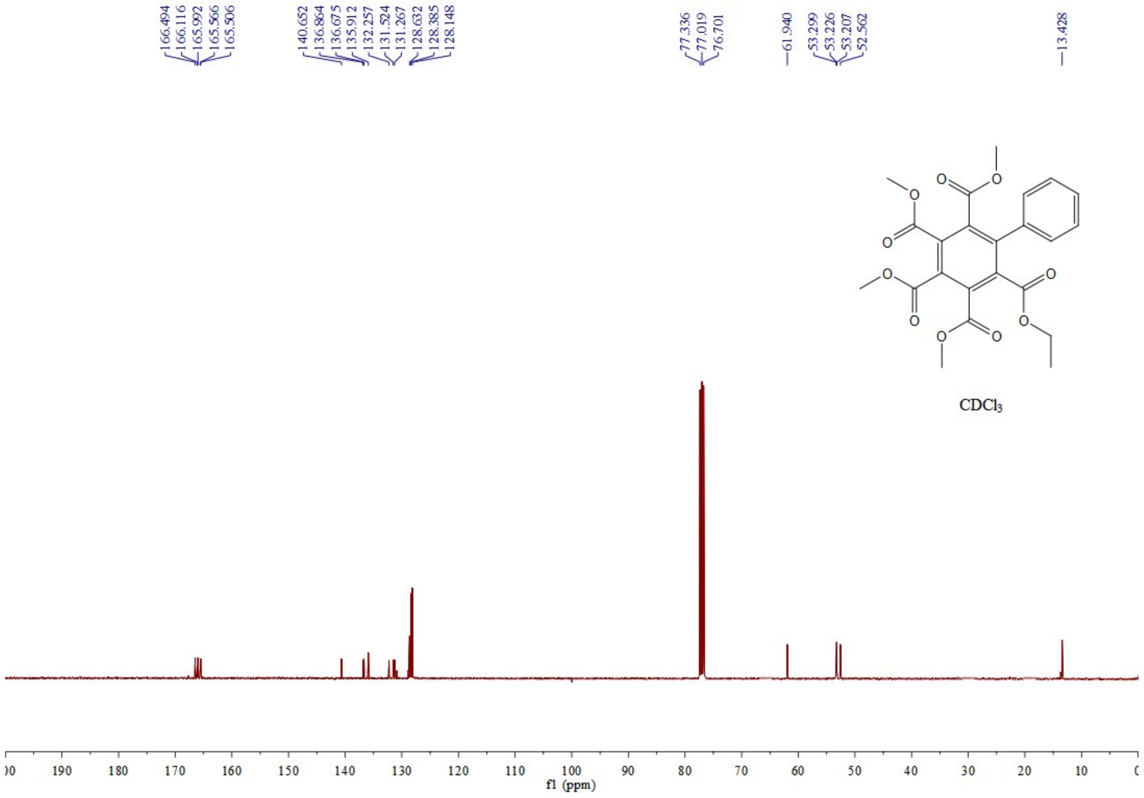
Benzene-1,2,3,4,5,6-hexacarboxylic acid hexamethyl ester (10a, Table 4).





2-Ethyl-3,4,5,6-tetramethyl [1,1'-biphenyl]-2,3,4,5,6-pentacarboxylate (10b, Table 4).





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