

RSC Advances

Rational Synthesis of Bis(hexyloxy)- Tetra(hydroxy)-Triphenylenes and their Derivatives.

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SUPPORTING INFORMATION

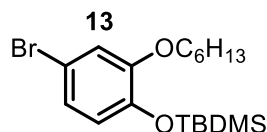
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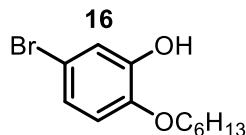
I. General Synthetic Procedures

General Methods: Chemicals were purchased from Aldrich, Strem, Acros, TCI America, or Cambridge Isotope Labs and used as received. Solvents were dried using an Innovative Technologies SPS-400-5 solvent purification system. Thin layer chromatography (TLC) was performed on alumina-backed sheets coated with silica gel 60 F254. TLC plates were visualized using a UV/Vis lamp and/or by staining with iodine or *p*-anisaldehyde solution. Column chromatography was performed using glass columns over Dynamic Absorbents 60 Å, 32-63 μm silica gel. Melting points were determined on a Mettler Toledo Mel-Temp II melting point apparatus and are uncorrected. UV/Vis spectroscopy was recorded on a Varian Cary 100 Bio UV-Visible spectrophotometer. All ¹H and ¹³C NMR spectra were recorded with a Varian Mercury (300 MHz and 75 MHz, respectively) or Varian Unity Plus (400 MHz and 100 MHz, respectively) spectrometer using residual solvent as the internal standard. All chemical shifts are quoted using the δ scale and all coupling constants are expressed in Hertz (Hz). Accurate mass EI/GCMS and ESI/APCI mass spectrometric analysis of compounds **1-6**, **9-10**, **13-14**, **16-18**, **20-23**, and **26** were performed at the UC Riverside Mass Spec Facility. Differential scanning calorimetry was recorded with a TA DSC Q20 equipped with a TA refrigerated cooling system 90. Compounds **7**, **8**, **11**, **12**, **15**, **19**, and **24** were prepared according to literature procedures¹⁻⁷.

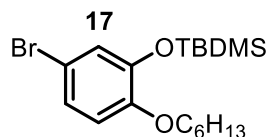
II. Synthesis of Compounds



Compound 13: Under an inert atmosphere, **Compound 12** (1.5g, 5.49 mmol) was dissolved in dimethylformamide (5.5 mL) and diisopropylethylamine (1.5 mL, 8.79 mmol) was added dropwise. The solution was stirred for 15 minutes, tertbutyldimethylsilylchloride (1.2 g, 8.24 mmol) was added, and the solution stirred overnight. Water was added (20 mL) and the crude product extracted with hexanes (3x15mL). The combined organic extracts were washed with brine (30mL), dried over MgSO₄, and concentrated under reduced pressure. The crude material was purified by column chromatography, eluting with 10% dichloromethane in hexanes, to afford the pure product (1.5 g, 70%) as a colorless oil. EI/GCMS (m/z) [M]⁺ calculated for C₁₈H₃₁O₂SiBr, 386.1271; found 386.1282. ¹H NMR (CDCl₃, 300 MHz): δ 7.03-6.92 (m, 2H), 6.73 (d, *J* = 8.5 Hz, 1H), 3.92 (t, *J* = 6.45 ppm, 2H), 1.87-1.78 (m, 2H), 1.58-1.43 (m, 2H), 1.43-1.30 (m, 4H), 1.03 (s, 9H), 0.94 (t, *J* = 5.7, 3H), 0.18 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 151.41, 144.15, 123.23, 122.00, 116.06, 113.44, 68.61, 31.58, 29.21, 2580, 25.64, 25.55, 22.60, 18.31, 14.03, -4.70 ppm.



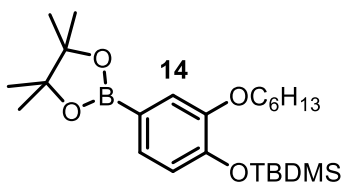
Compound 16: To a solution of **15** (2.3 g, 8.01 mmol) in dichloromethane (52mL) was added meta-chloroperoxybenzoic acid (2.6 g, 14.9 mmol) in small portions, and the solution stirred at 40°C overnight. A 2M solution of ammonia in methanol (12.1 mL) was added, and the mixture was stirred for 2 hours. Saturated sodium bicarbonate was added (30 mL), and the product was extracted with diethyl ether (3 x 30mL). The combined organic layers were washed with saturated sodium bicarbonate (100mL) and brine (100 mL), dried over MgSO₄, and concentrated in vacuo to afford the pure product, isolated as an off-white solid (2.2 g, 99%). Mp = 41.8-42.6°C. EI/GCMS (m/z) [M]⁺ calculated for C₁₂H₁₇O₂Br, 272.0406; found 272.0339. ¹H NMR (CDCl₃, 300 MHz): δ 7.08 (d, *J* = 2.4 Hz, 1H), 6.95 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.71 (d, *J* = 8.8 Hz, 1H), 5.67 (s, 1H), 4.02 (t, *J* = 6.7, 2H), 1.86-1.77 (m, 2H), 1.49-1.40 (m, 2H), 1.38-1.30 (m, 4H), 0.92 (t, *J* = 7.3 Hz, 3H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 146.86, 145.49, 122.97, 117.97, 113.25, 112.96, 69.42, 31.77, 29.32, 25.88, 22.83, 14.28 ppm.



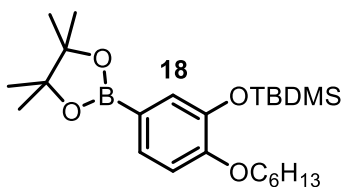
Compound 17: Under an inert atmosphere, Compound **16** (541 mg, 1.98 mmol) was dissolved in dimethylformamide (2 mL) and diisopropylethylamine (0.54 mL, 3.17 mmol) was added dropwise. The solution was stirred for 15 minutes, tertbutyldimethylsilylchloride (448 mg, 2.97 mmol) was added, and the solution stirred overnight. Water was added (10 mL) and the crude product extracted with hexanes (3x10mL). The combined organic extracts were washed with brine (20mL), dried over MgSO₄, and concentrated under reduced pressure. The crude material was subjected to high vacuum to remove volatiles and afford the pure product (725 mg, 95%) as a yellow oil. EI/GCMS (m/z) [M]⁺ calculated for C₁₈H₃₁O₂SiBr, 386.1271; found 386.1258. ¹H NMR (CDCl₃, 300 MHz): δ 6.96 (d, *J* = 2.5 Hz, 1H), 6.91 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.70 (d, *J* = 8.5, 1H), 3.90 (t, *J* = 7.0 Hz, 2H), 1.85-1.76 (m, 2H), 1.52-1.42 (m, 2H), 1.36-1.28 (m, 4H), 1.00 (s, 9H), 0.89 (t, *J* = 7.6 Hz, 3H), 0.15 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 150.03, 145.83, 124.37, 124.01, 113.93, 111.94, 68.61, 31.61, 29.28, 25.78, 25.62, 22.61, 18.33, 14.04, -4.70 ppm.

General procedure to prepare aryl pinacolboranes from aryl halides.

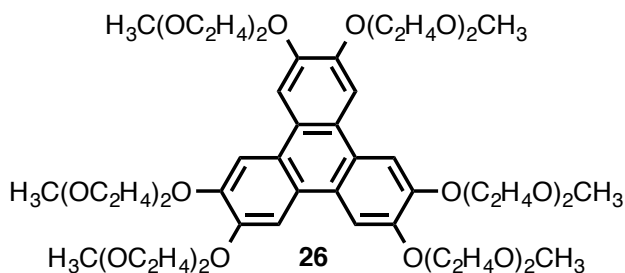
A mixture of aryl halide, bis(acetonitrile)dichloropalladium (II) (1 mol%), and Sphos Buchwald ligand (4 mol %) was prepared in a pressure flask, and immediately subjected to a vacuum/N₂ cycle (3x). To the solids was added dry 1,4-dioxane (1.7M with respect to aryl halide), and dry triethylamine (1.5 equivalents) under N₂. Last, pinacolborane (1.5 equivalents) was added quickly and the flask capped tightly. The mixture was stirred at 100°C until the reaction mixture darkened and thickened (around 3 hours). The mixture was allowed to cool, diluted with diethyl ether, and filtered over a pad of Celite. The filtrate was concentrated under reduced pressure, and the crude material purified by column chromatography.



Compound 14: Reaction scale: Compound **13** (1.5 g, 3.87 mmol). The product eluted from the column with 20% dichloromethane in hexanes, and was isolated as a yellow oil (1.28 g, 76%). ESI/APCI (m/z) [MH]⁺ calculated for C₂₄H₄₄¹¹BO₄Si, 435.3096; found 435.3110. ¹H NMR (CDCl₃, 300 MHz): δ 7.32-7.27 (m, 1H), 6.35 (d, *J* = 7.9 Hz, 1H), 3.99 (t, *J* = 6.9, 1H), 1.90-1.76 (m, 2H), 1.51-1.45 (m, 2H), 1.34 (s, 12H), 1.31-1.18 (m, 4H), 1.01 (s, 9H), 0.91 (t, *J* = 6.9 Hz, 3H), 0.17 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 150.20, 147.94, 128.00, 120.63, 118.48, 83.53, 68.33, 31.65, 29.48, 25.84, 25.70, 25.67, 24.86, 22.62, 18.42, 14.07, -4.61 ppm.



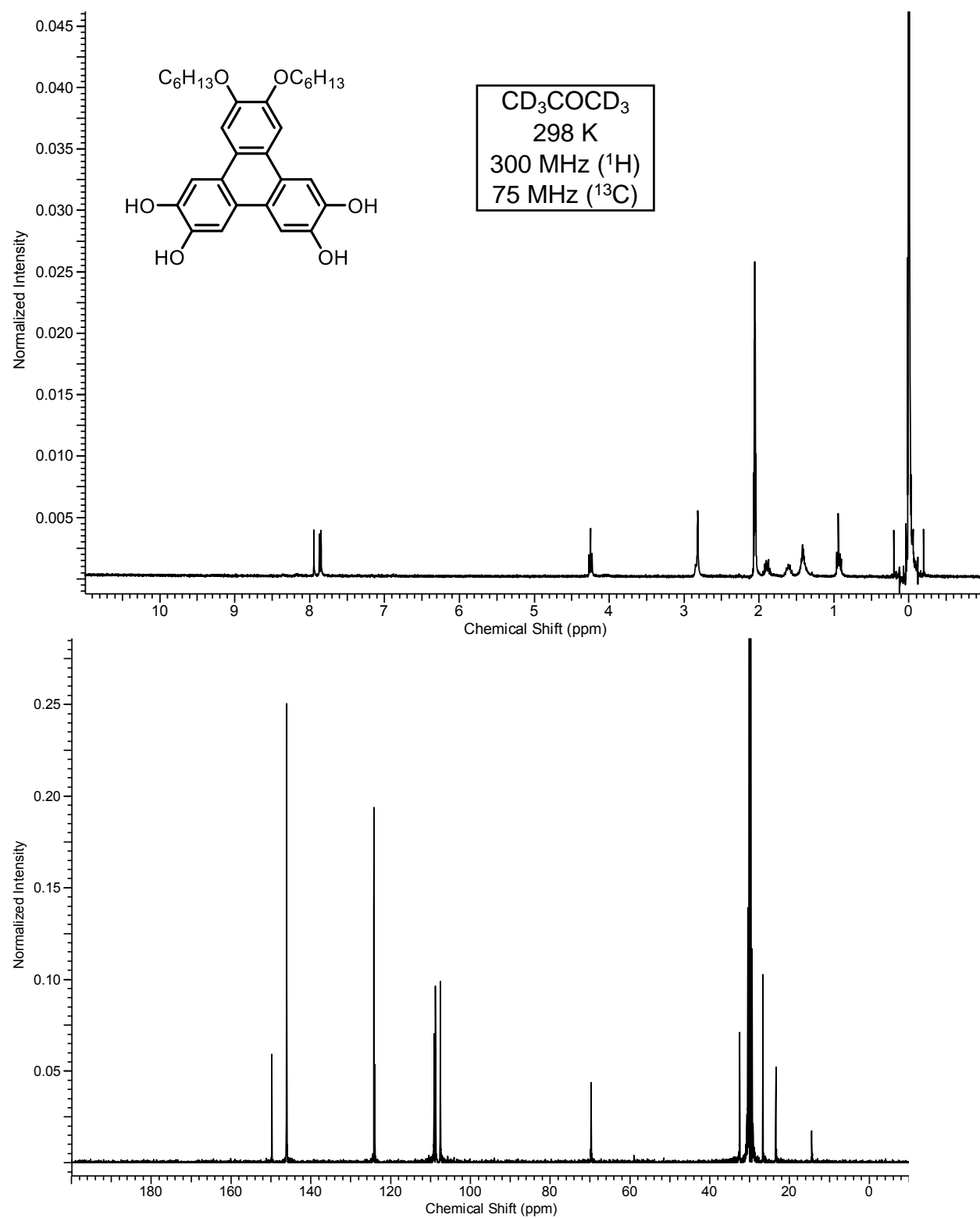
Compound 18: Reaction Scale: Compound **17** (355 mg, 0.916 mmol). The product eluted from the column with 30% dichloromethane in hexanes, and was isolated as a yellow solid (312 mg, 78%). Mp = 69.2 - 71.1°C. ESI/APCI (m/z) [MH]⁺ calculated for C₂₄H₄₄¹¹BO₄Si, 435.3096; found 435.3104. ¹H NMR (CDCl₃, 300 MHz): δ 7.38 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.27-7.24 (m, 1H), 6.84 (d, *J* = 8.2 Hz, 1H), 3.96 (t, *J* = 6.7 Hz, 2H), 1.87-1.76 (m, 2H), 1.52-1.42 (m, 2H), 1.33 (s, 12H), 1.30-1.23 (m, 4H), 1.01 (s, 9H), 0.91 (t, *J* = 6.6 Hz, 3H), 0.17 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 159.29, 144.16, 129.20, 126.90, 111.85, 83.38, 68.15, 31.62, 29.28, 25.78, 25.73, 25.71, 24.81, 22.59, 18.36, 14.03, -4.59 ppm.



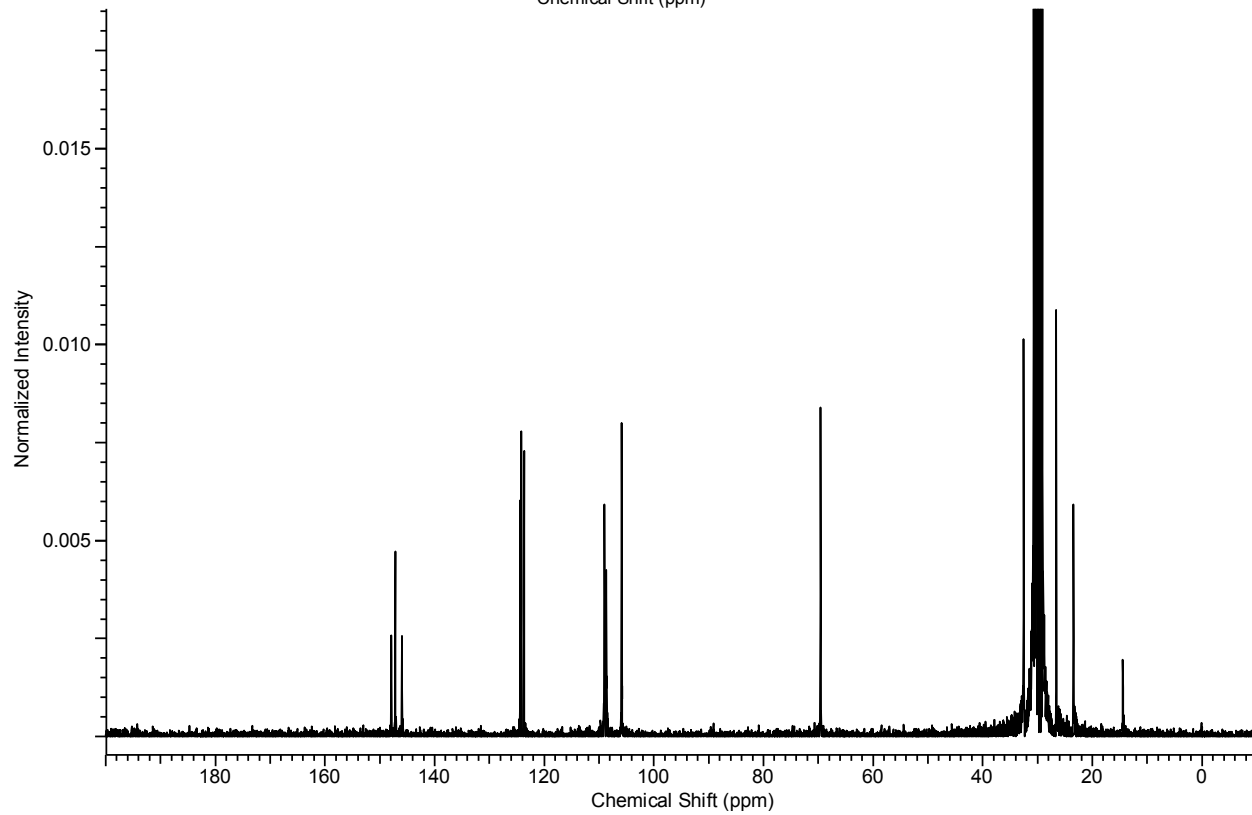
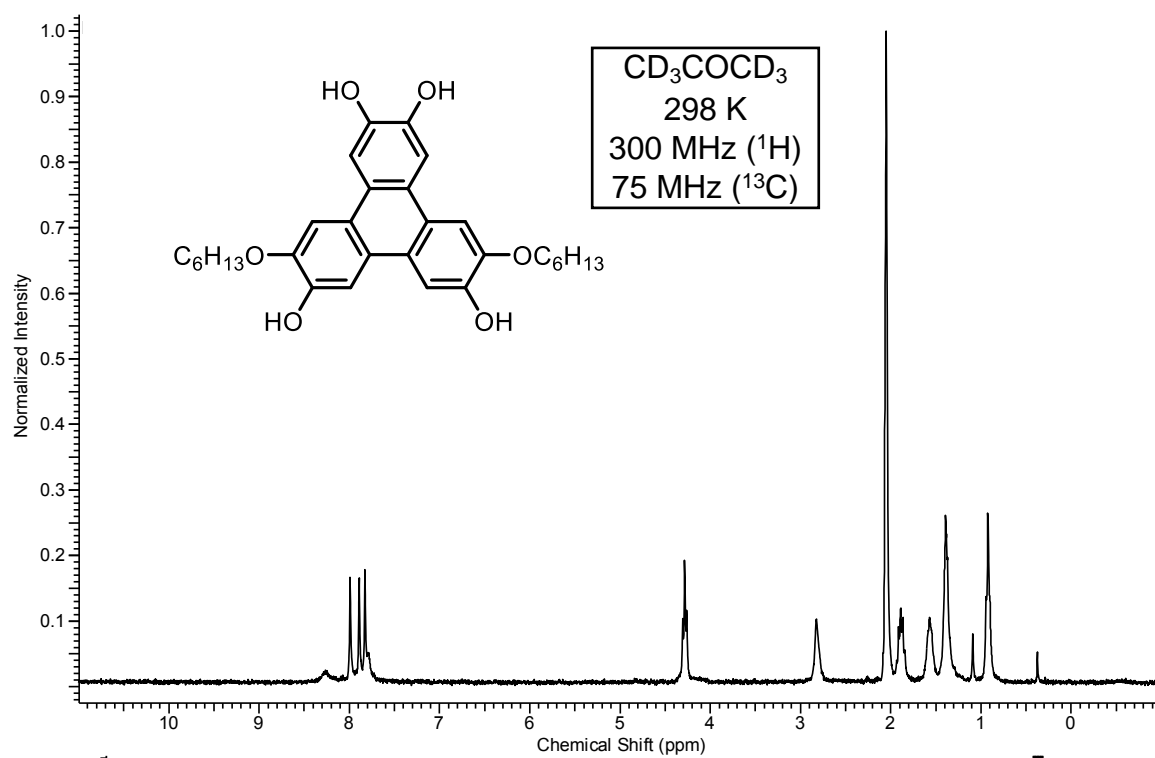
Compound 26: Hexakis(monomethyl di(ethylene glycol)) triphenylene: To a mixture of 2,3,6,7,10,11-hexahydroxytriphenylene (105 mg, 0.324 mmol), potassium carbonate (447 mg, 3.24 mmol), and catalytic 18-C-6 was added dimethylformamide (3.2 mL) and di(ethylene glycol) monomethyl ether tosylate (710 mg, 2.59 mmol) under nitrogen. The reaction solution was stirred at 80°C overnight. The solution was allowed to cool, and water was added. Excess di(ethylene glycol) monomethyl ether tosylate was extracted from the aqueous layer with diethyl ether (2x). The combined ethereal extracts were washed with aqueous hydrochloric acid (1M), and the aqueous layers combined. The product was extracted from the aqueous phase with ethyl acetate (3x), and the ethyl acetate phase washed with brine, dried over MgSO₄, and concentrated under reduced pressure to afford analytically pure product (183 mg, 30%). The product was isolated as a dark oil which gradually solidified. ESI/APCI (m/z) [MNa]⁺ calculated for C₄₈H₇₂O₁₈Na, 959.4611; found 959.4634. ¹H NMR (300 MHz, CDCl₃): δ 7.88 (s, 6H), 4.42 (t, *J* = 4.6 Hz, 12H), 4.01 (t, *J* = 4.4 Hz, 12H), 3.80-3.83 (m, 12H), 3.61-3.63 (m, 12H), 3.41 (s, 18H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 148.41, 123.62, 107.61, 71.76, 70.54, 69.64, 68.89, 58.81 ppm.

III. ^1H and ^{13}C NMR Spectra

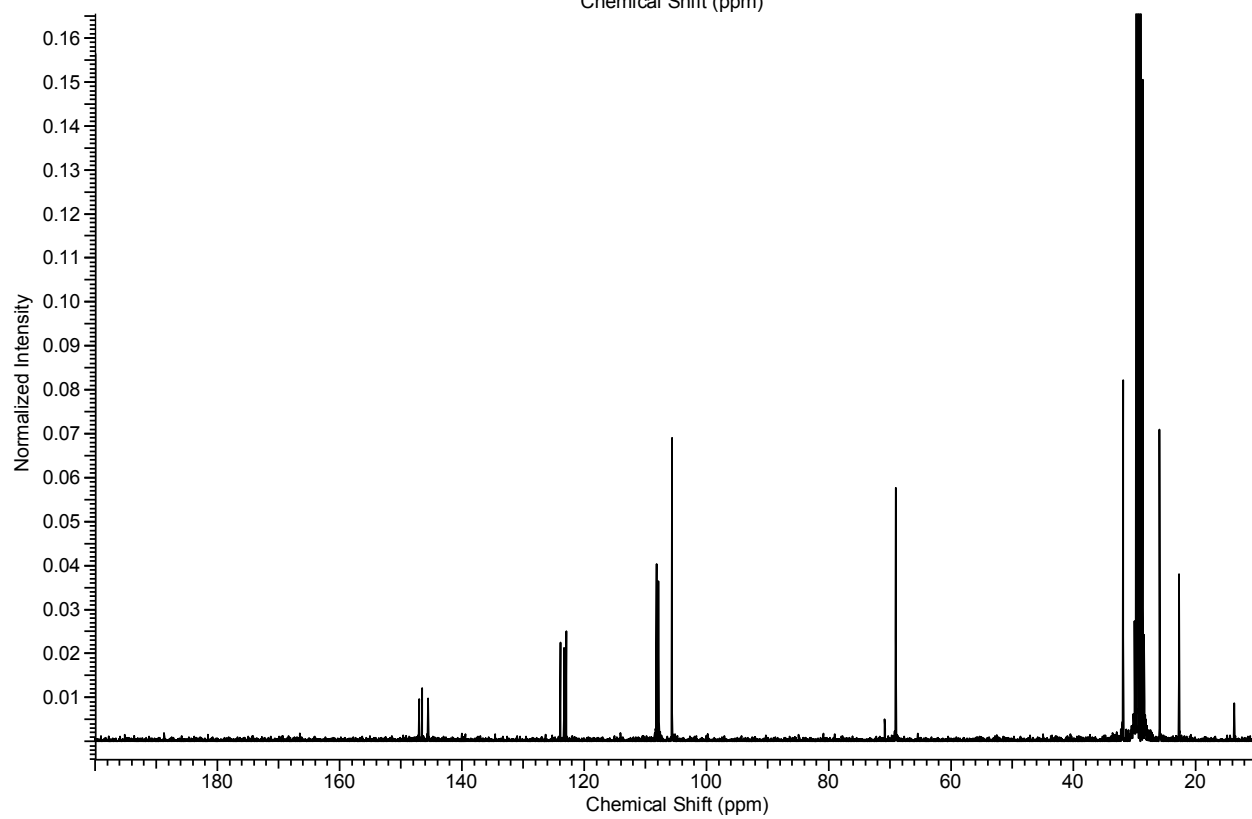
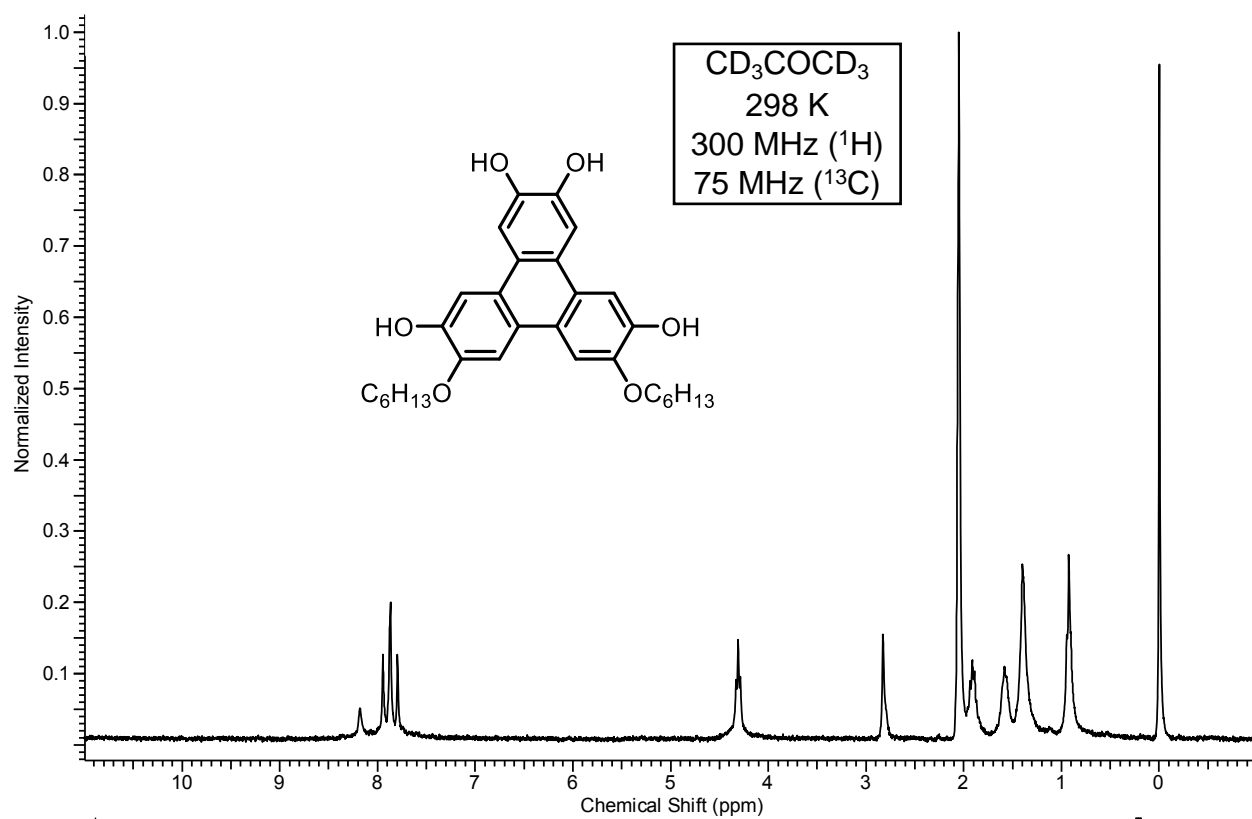
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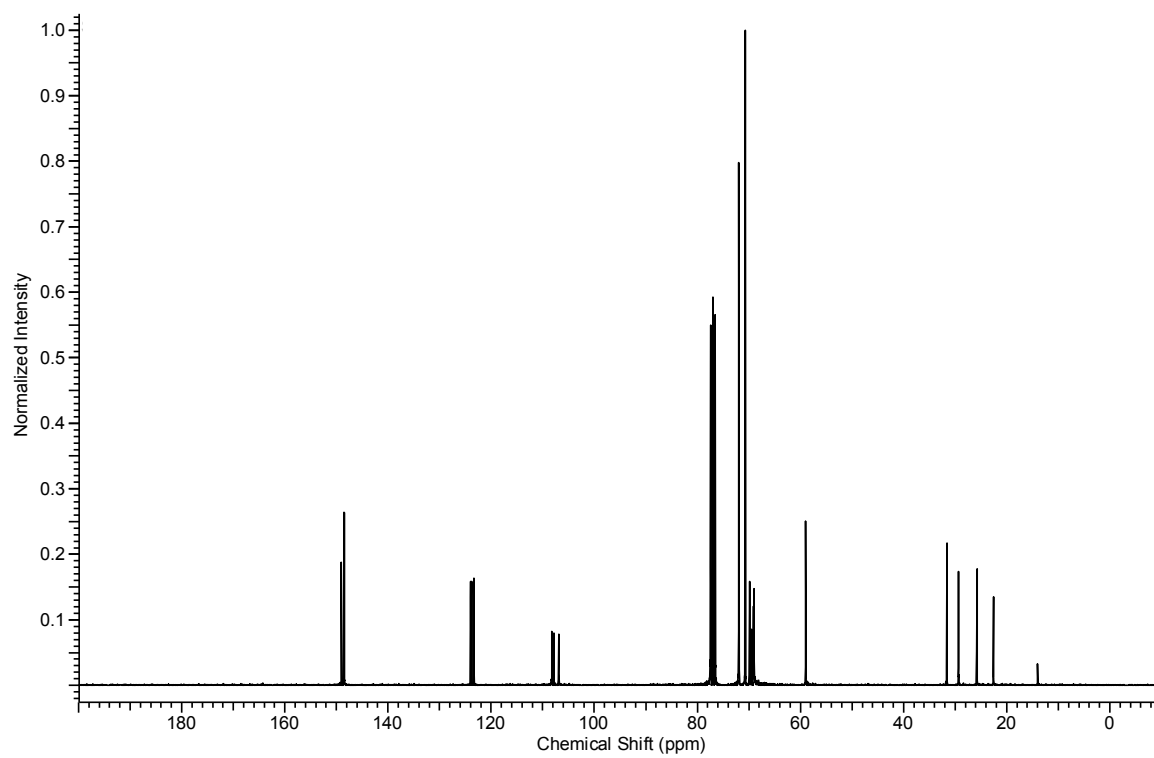
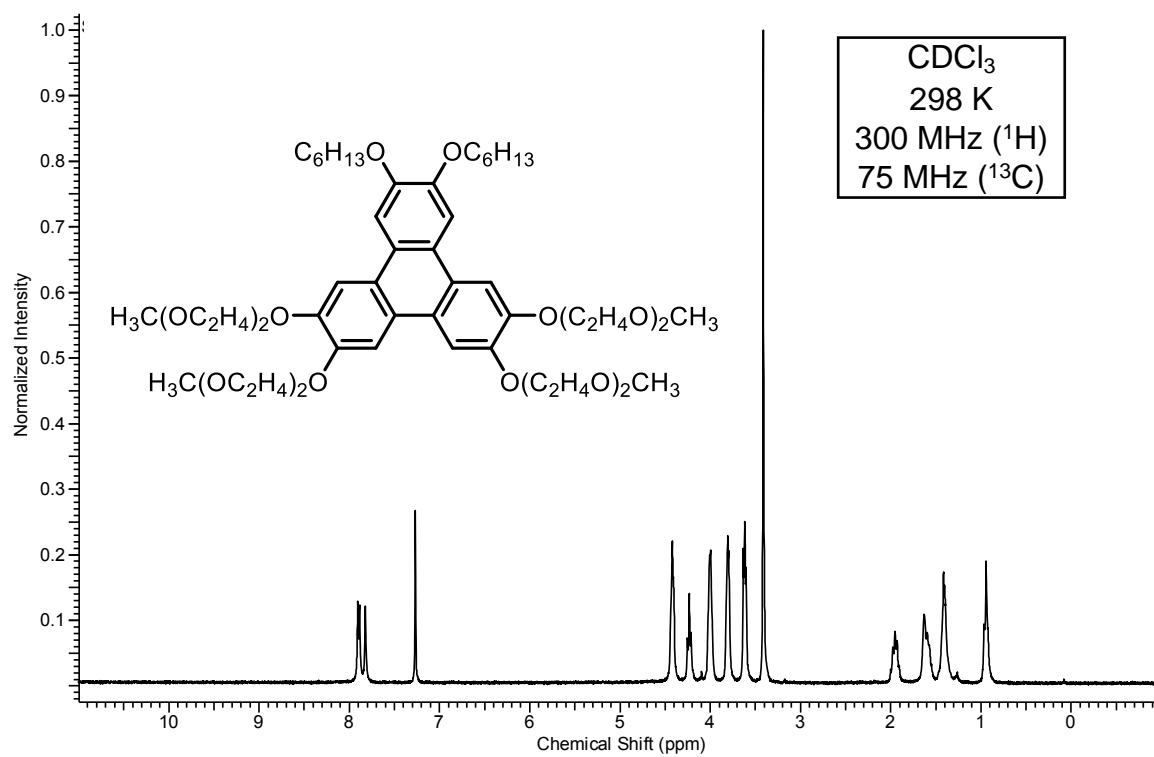
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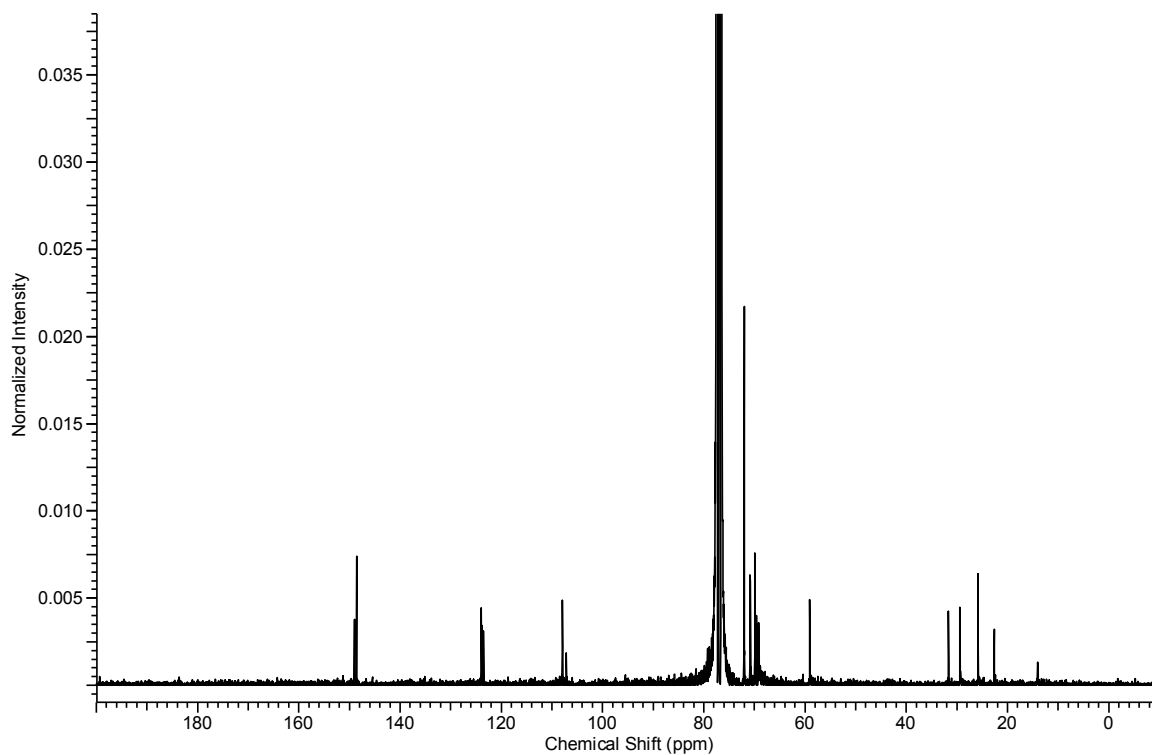
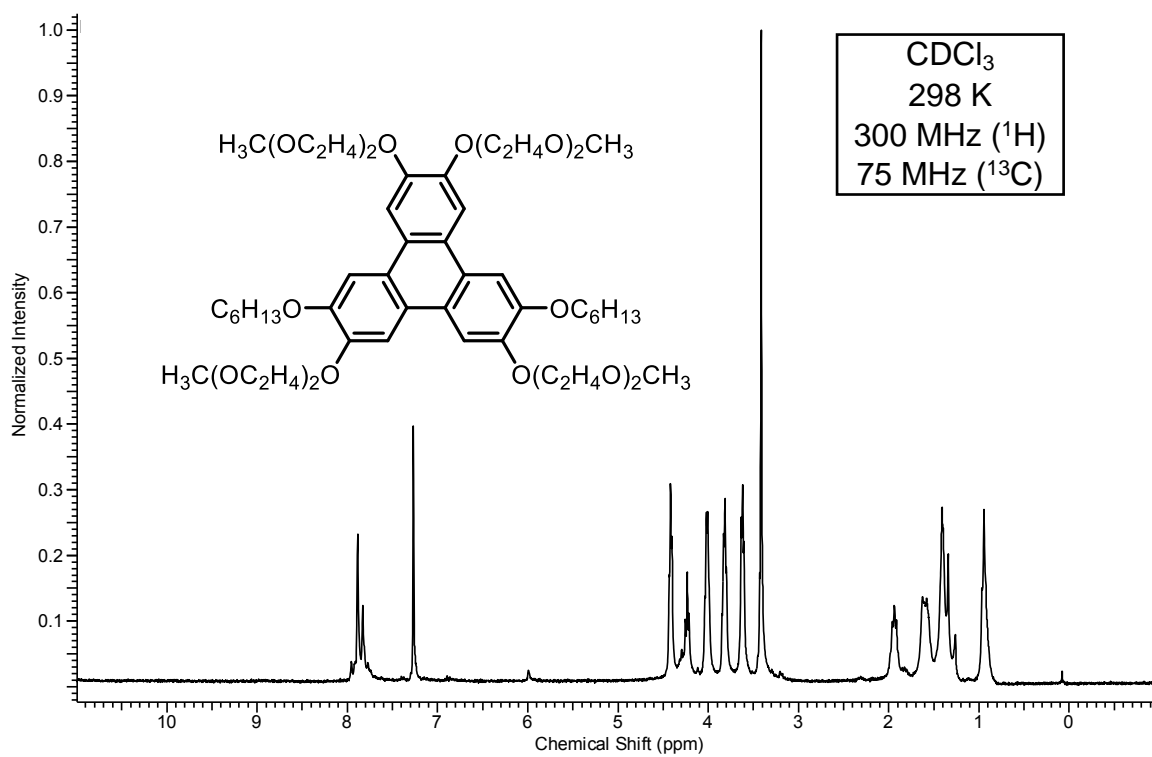
Compound 3



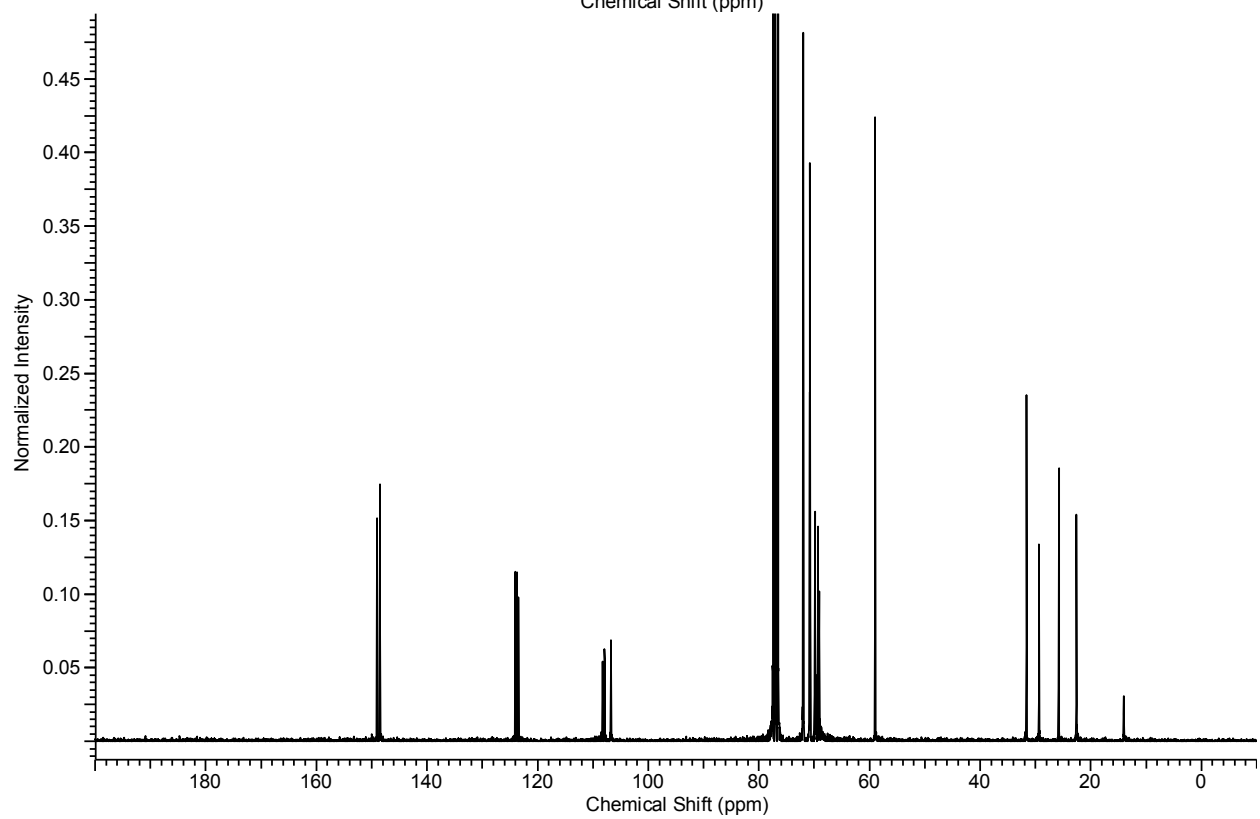
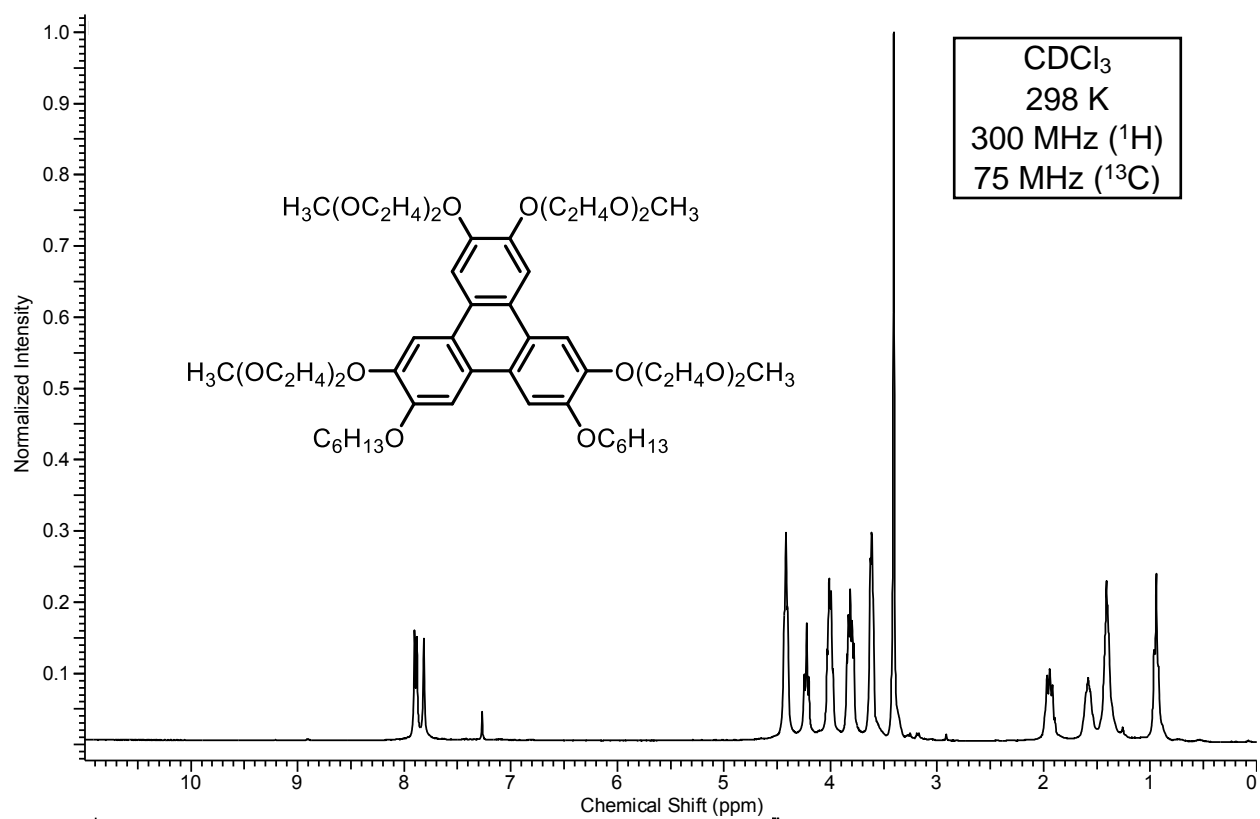
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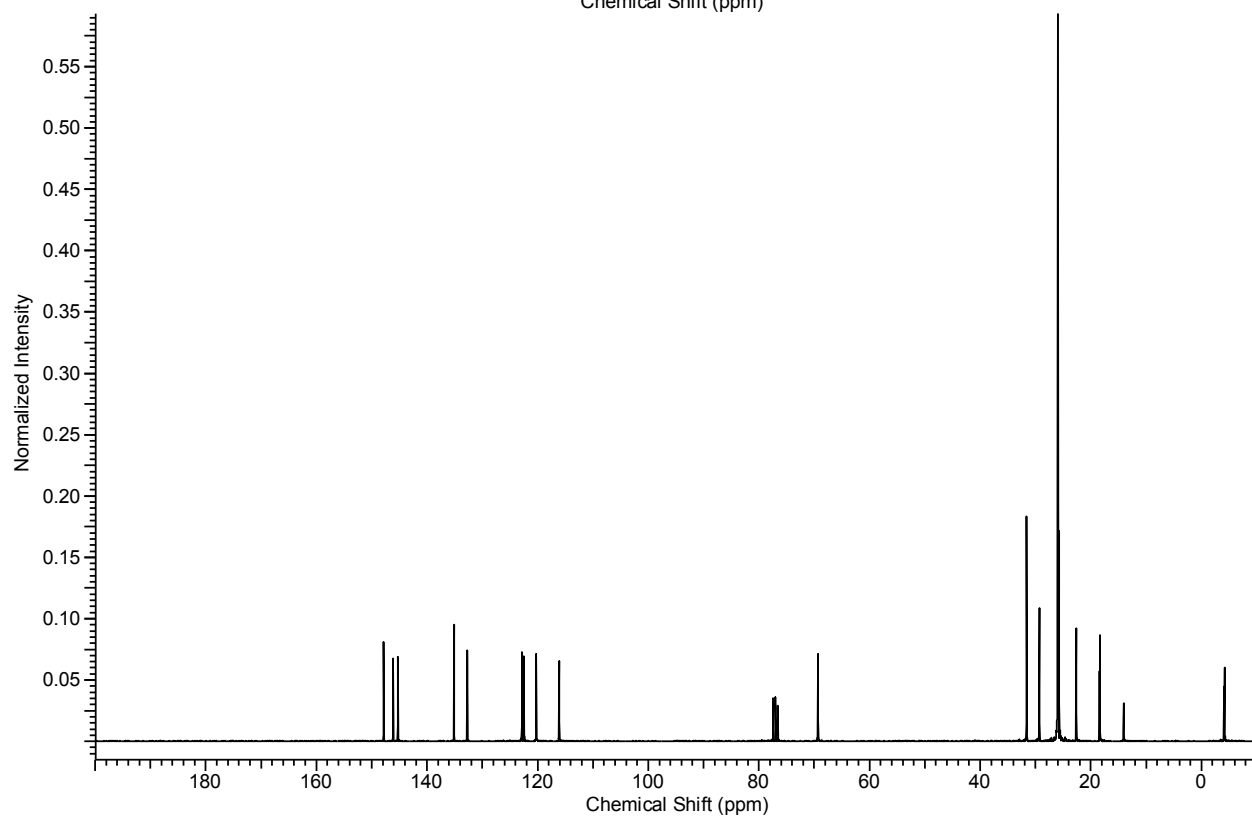
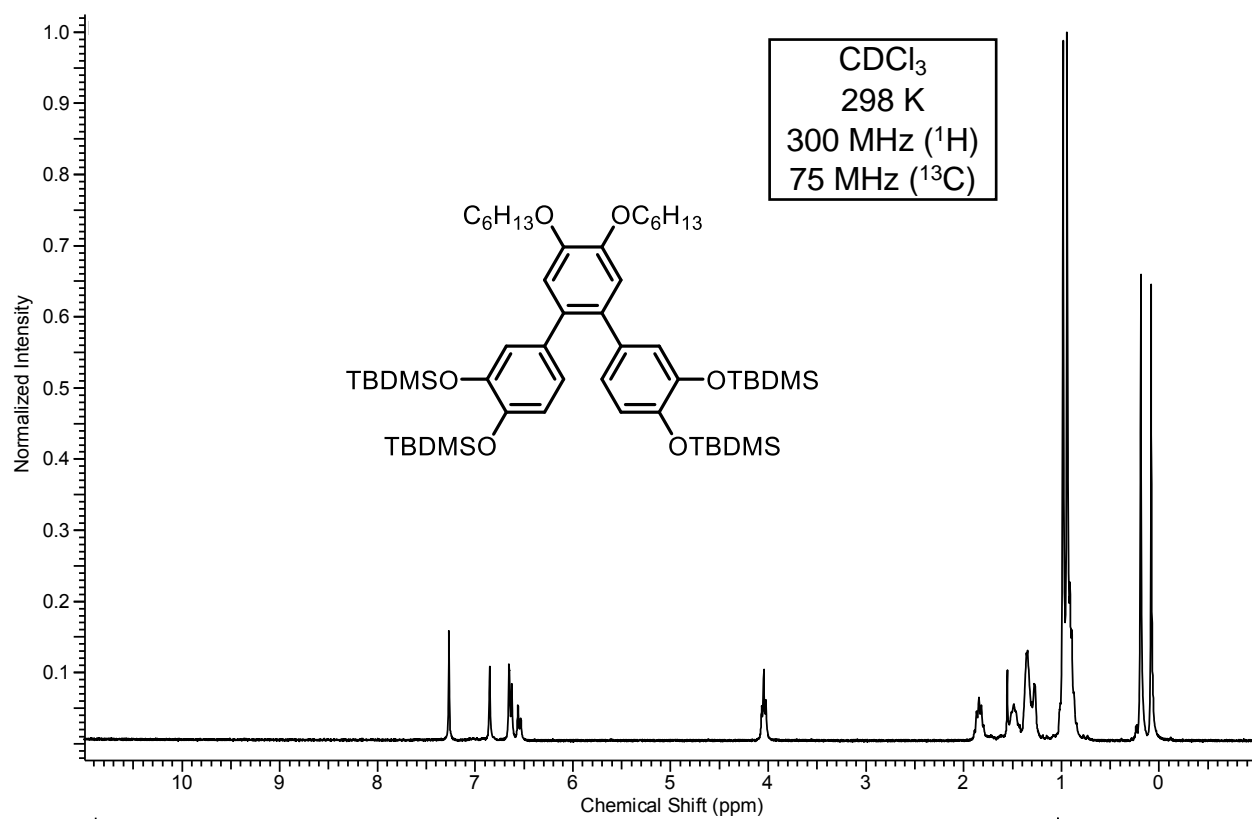
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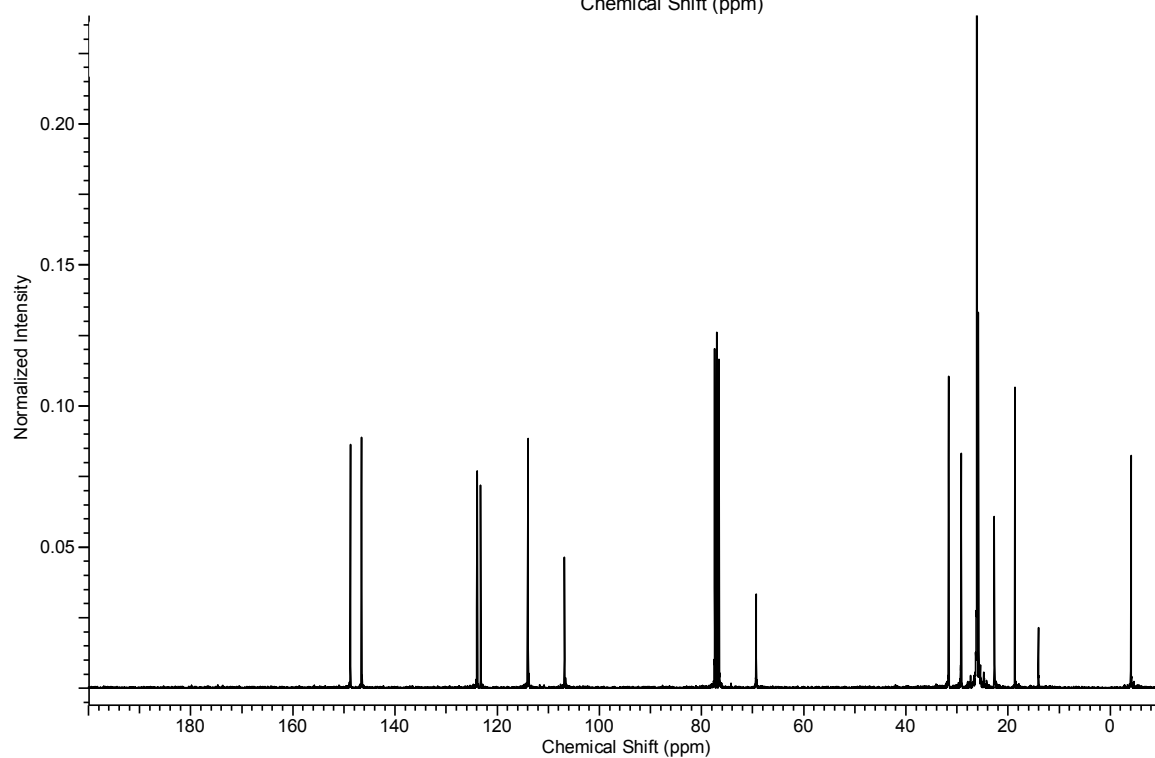
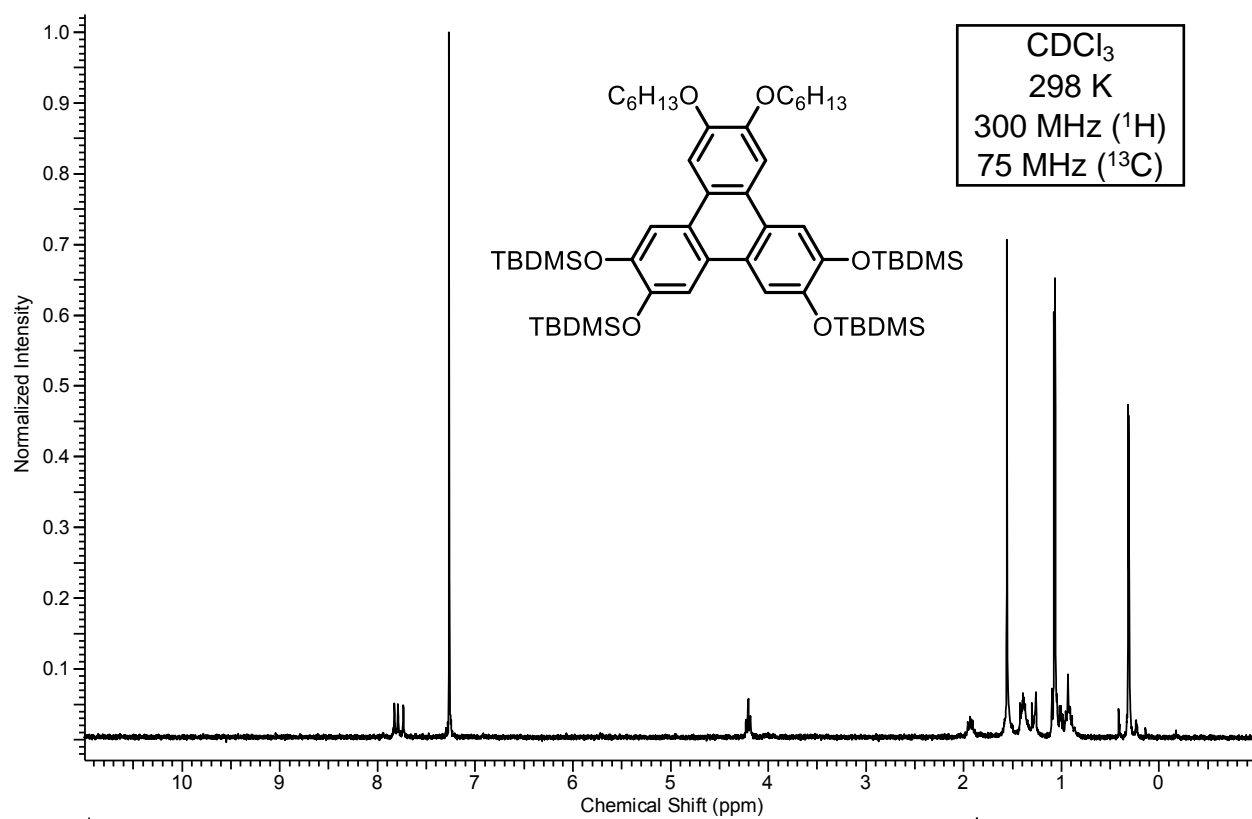
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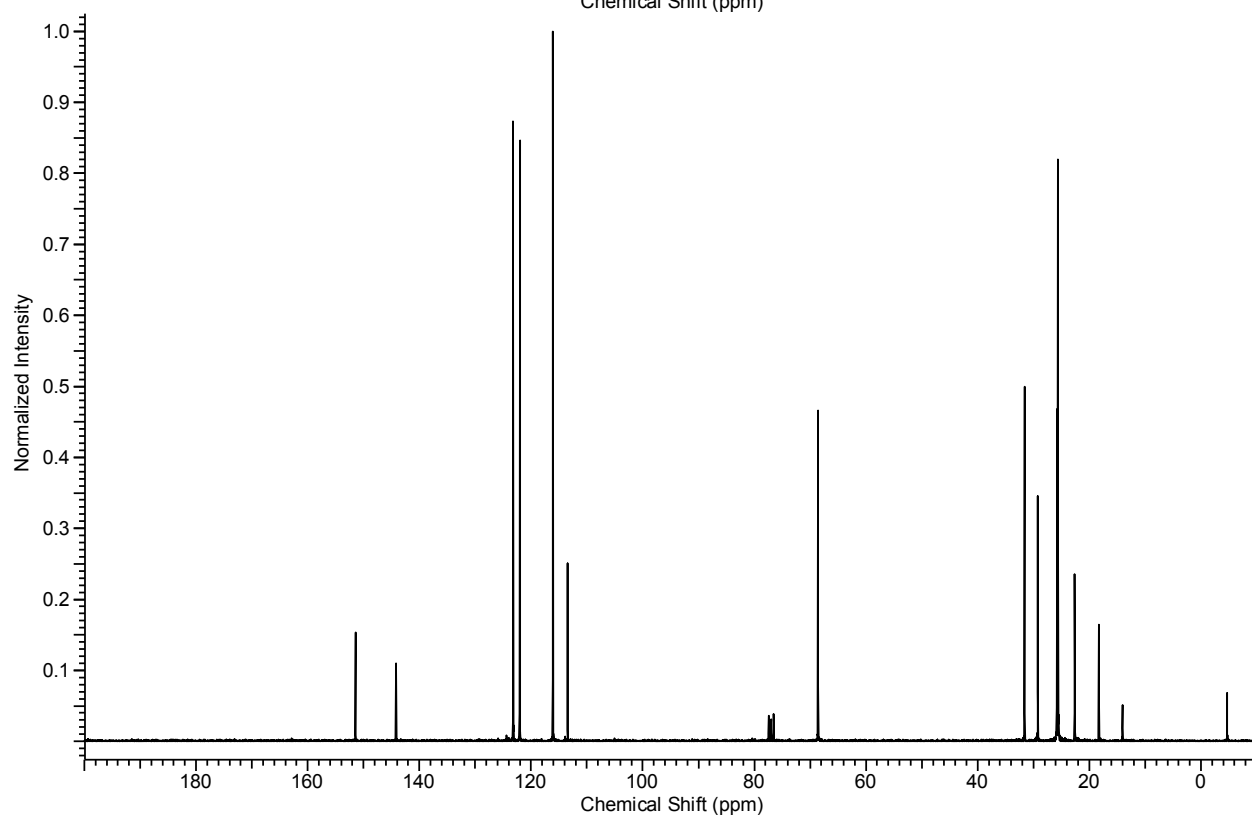
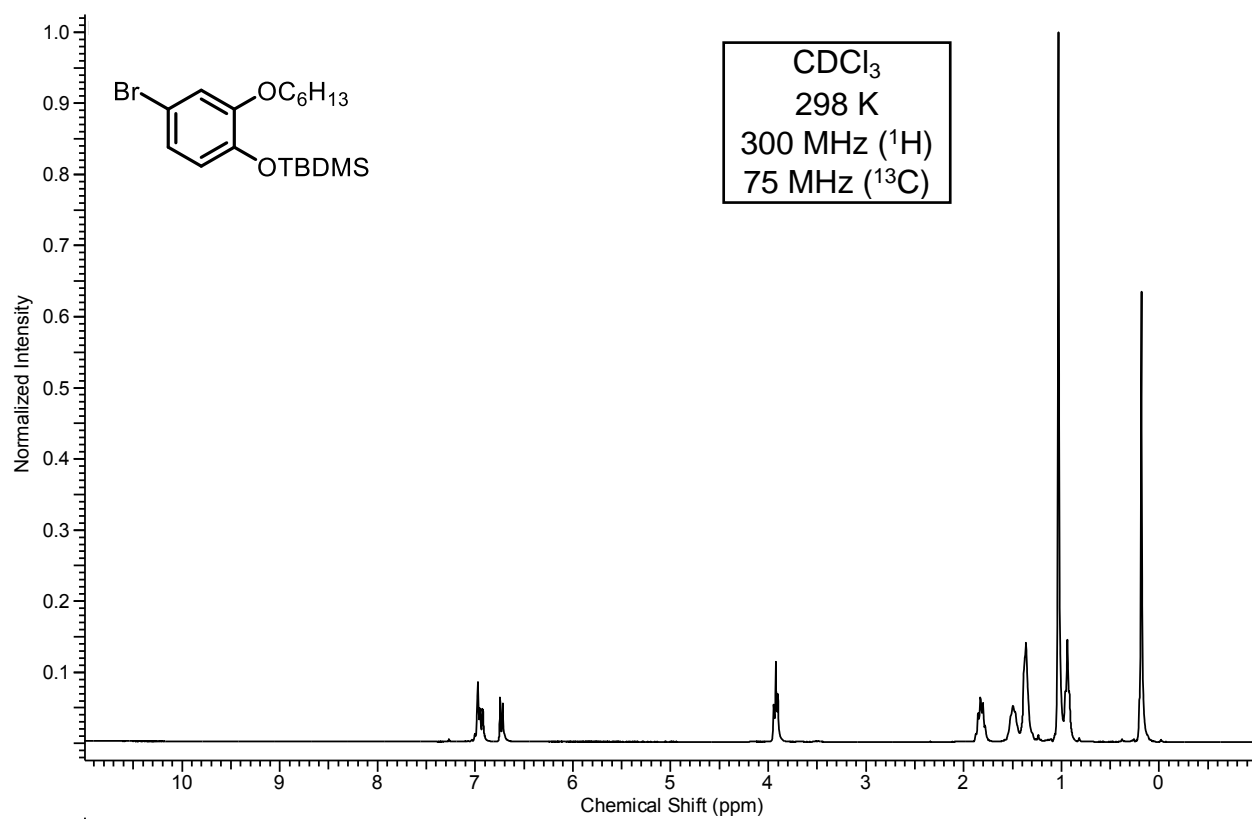
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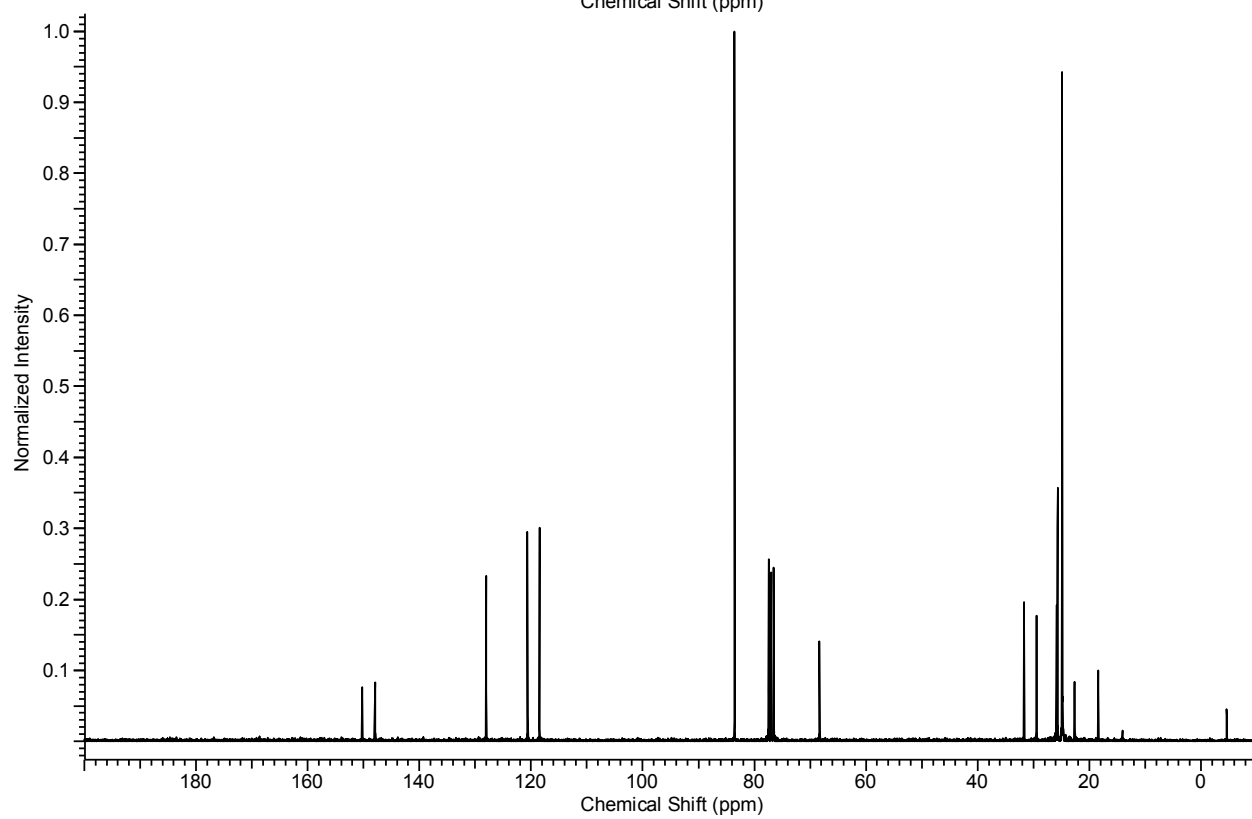
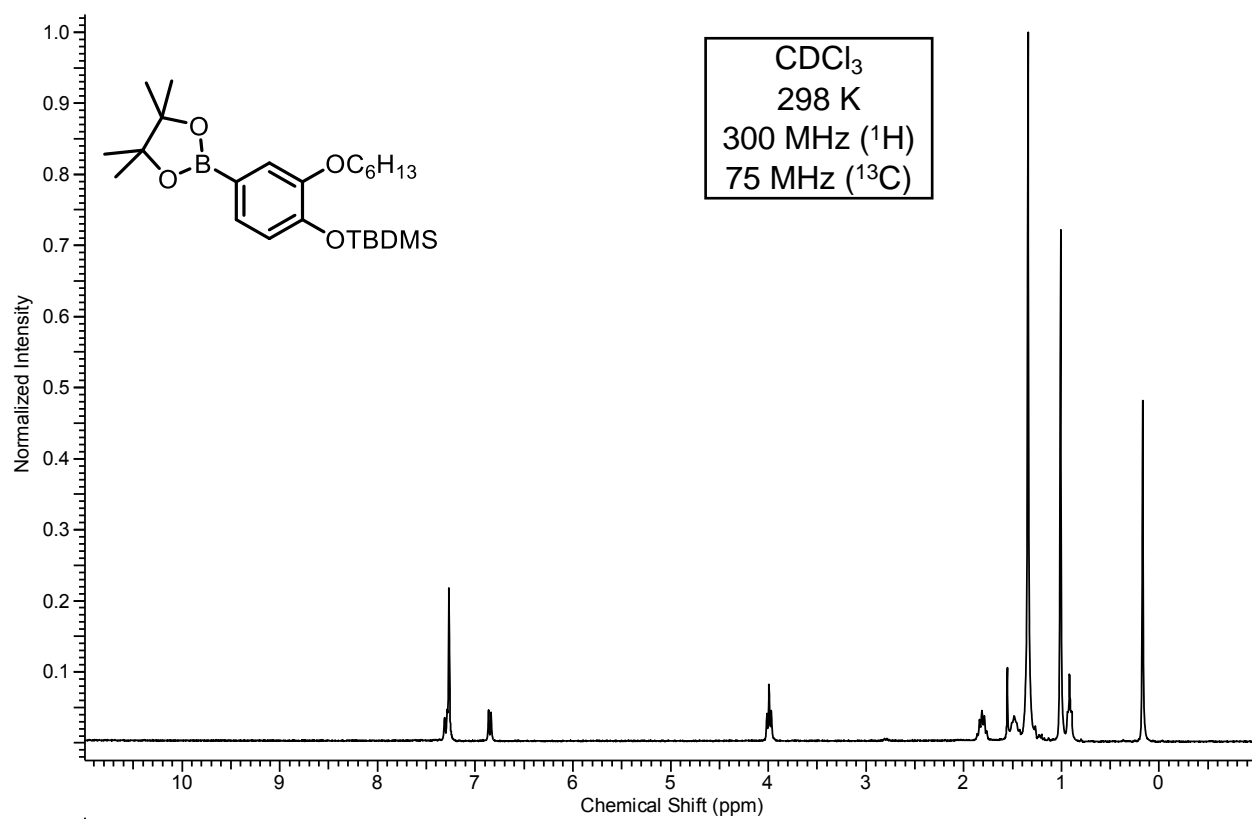
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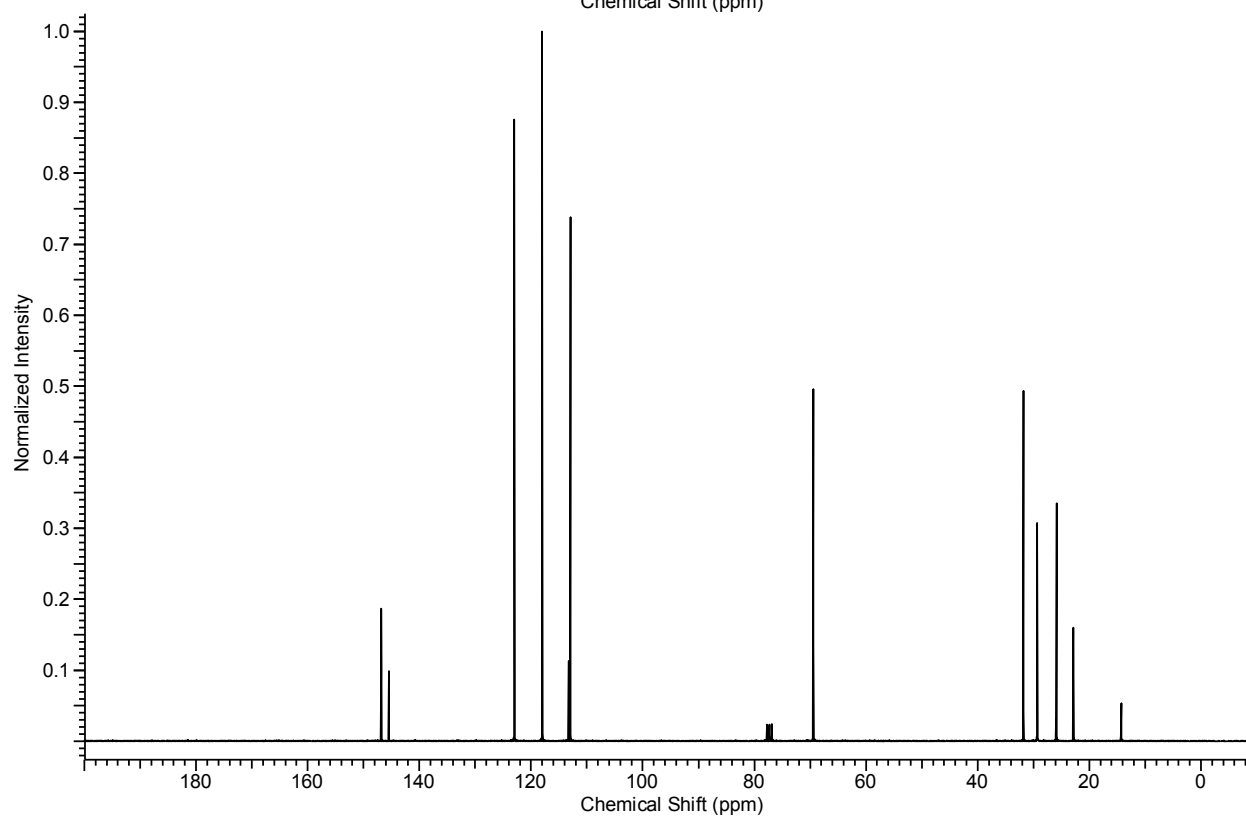
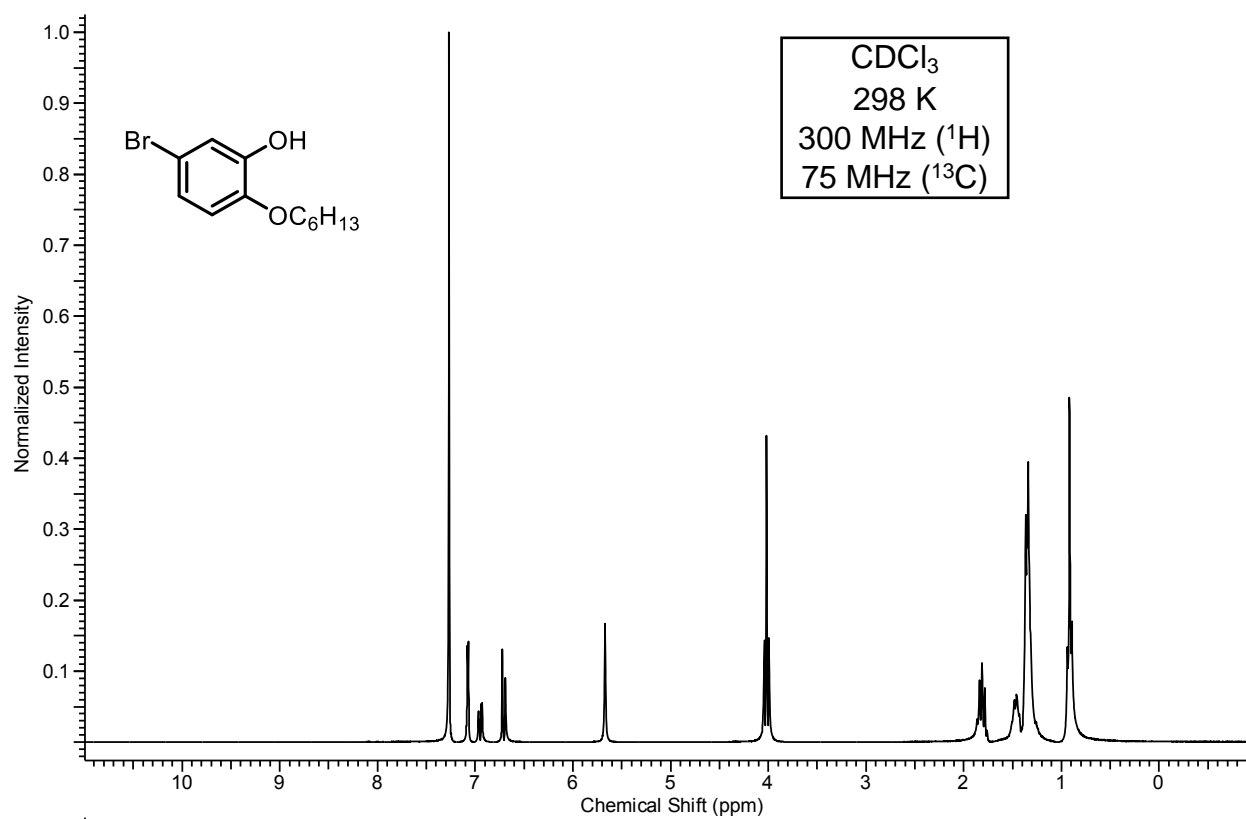
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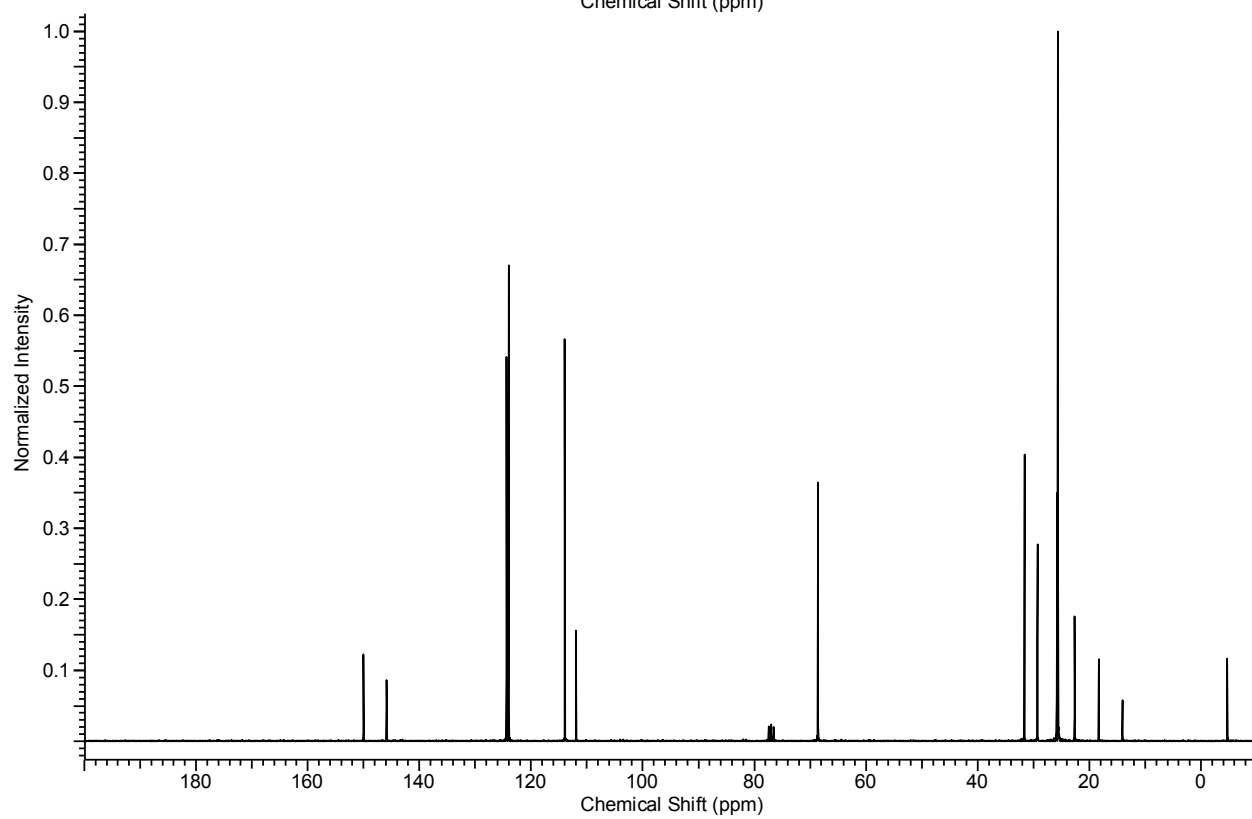
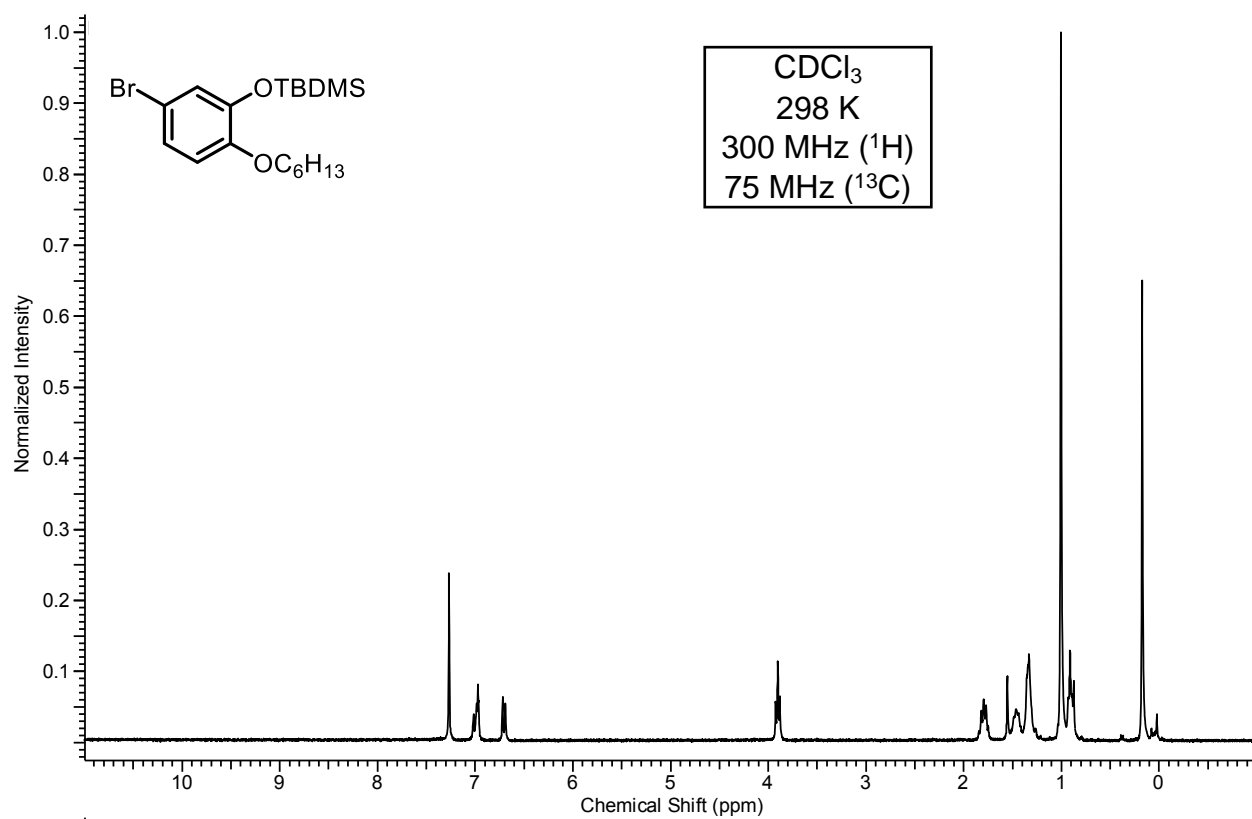
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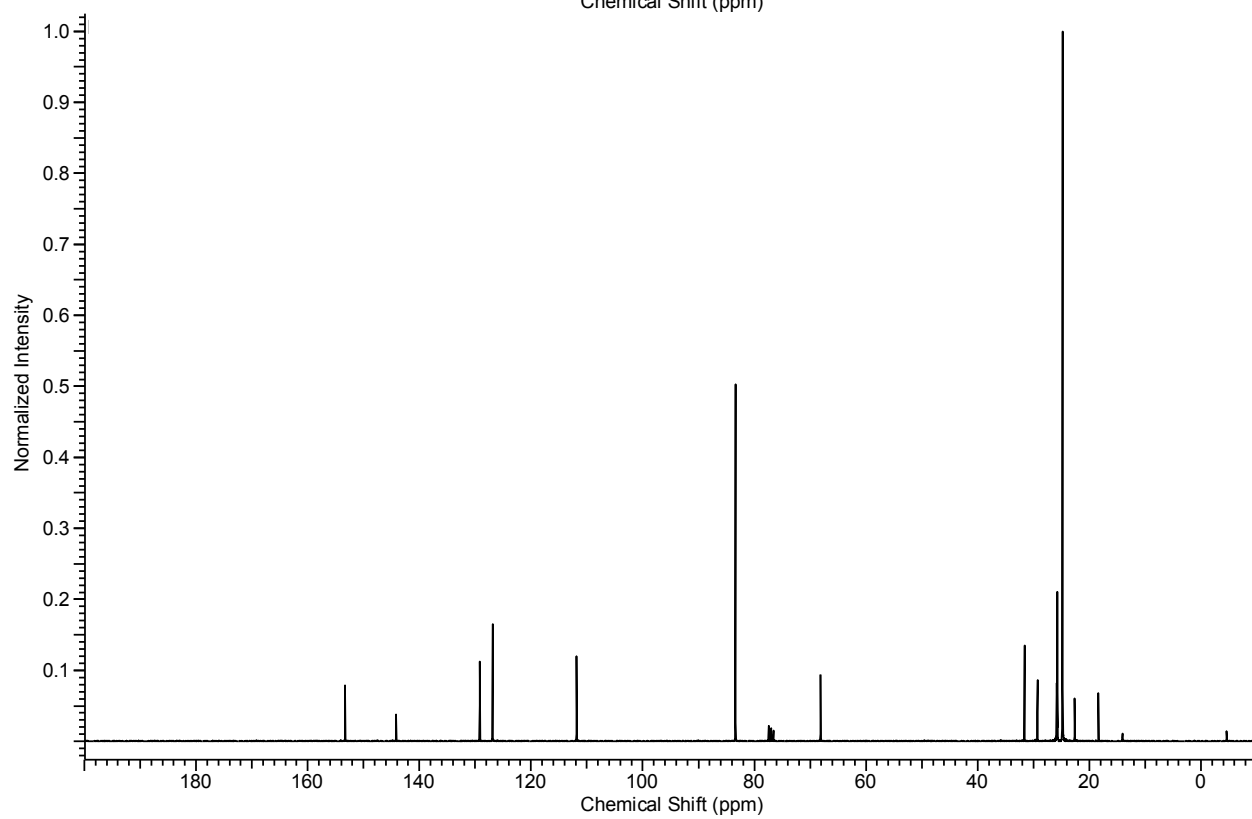
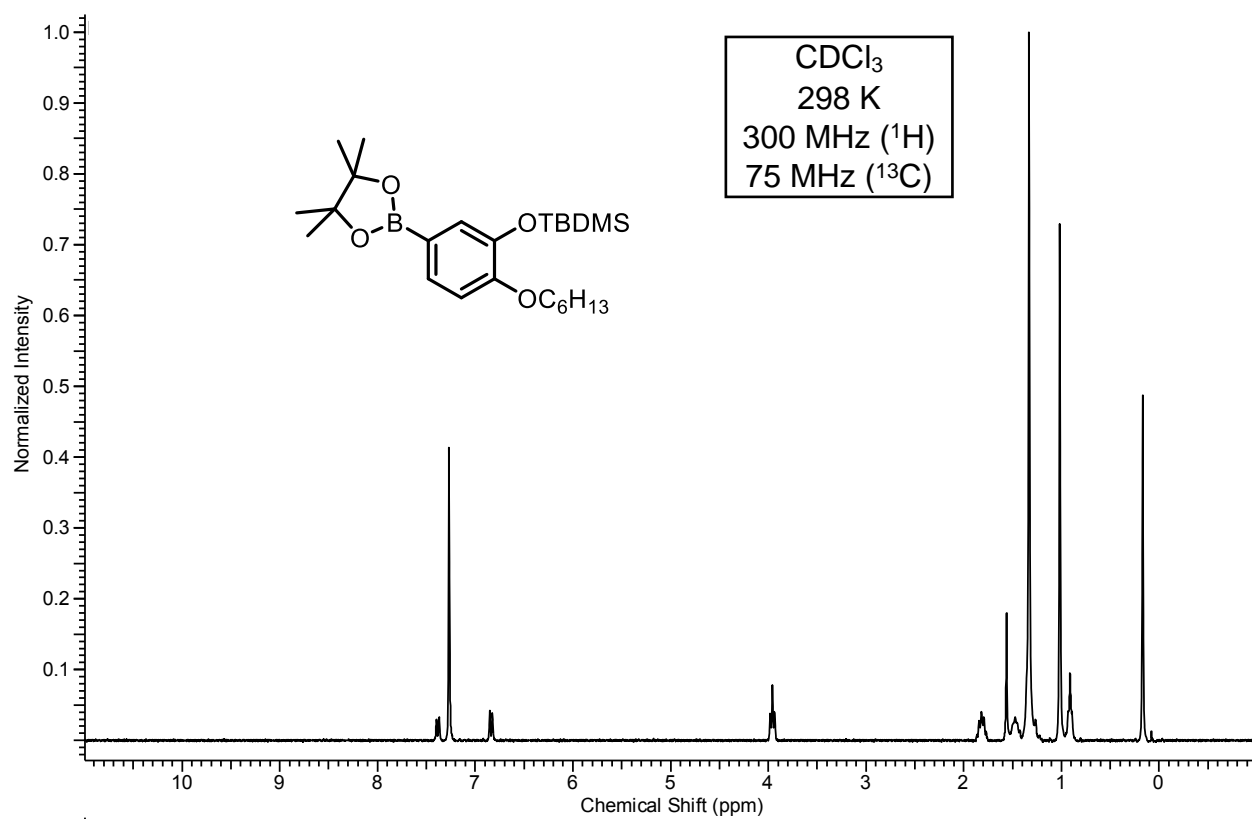
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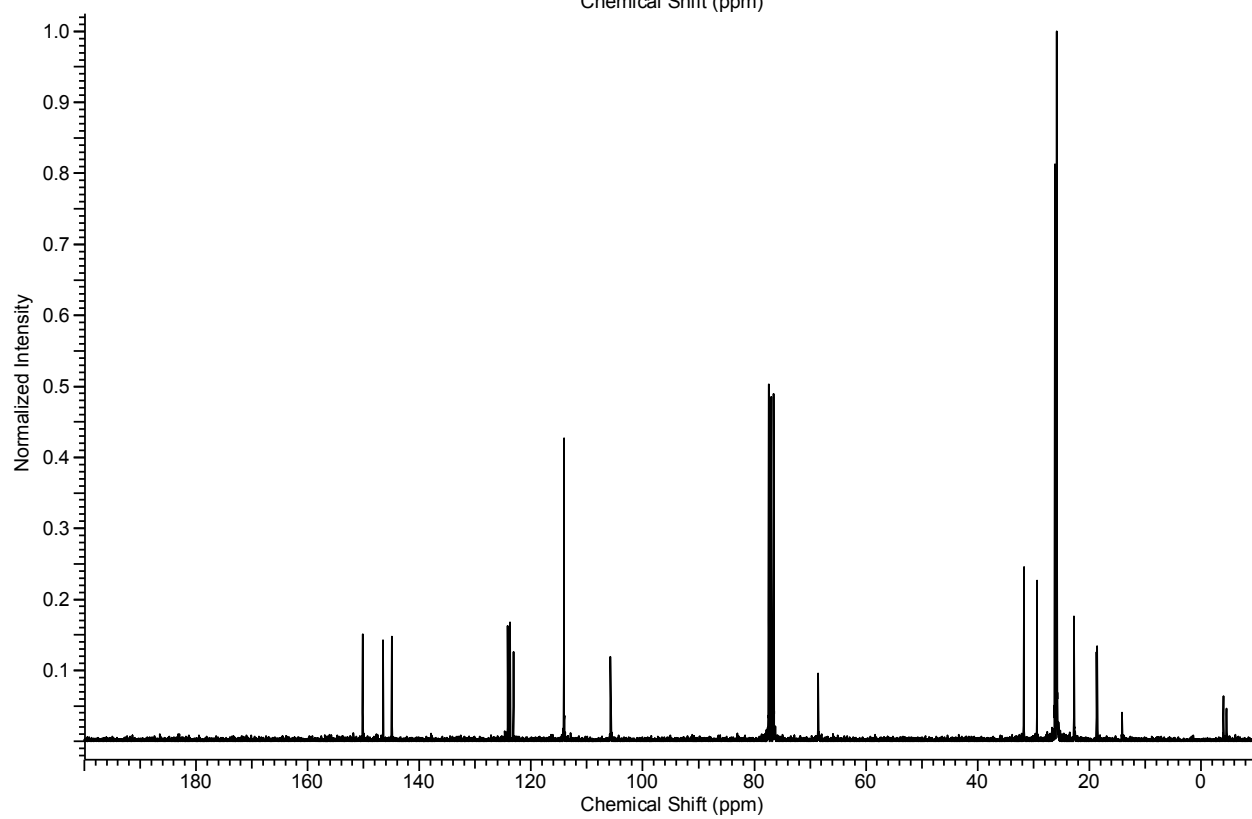
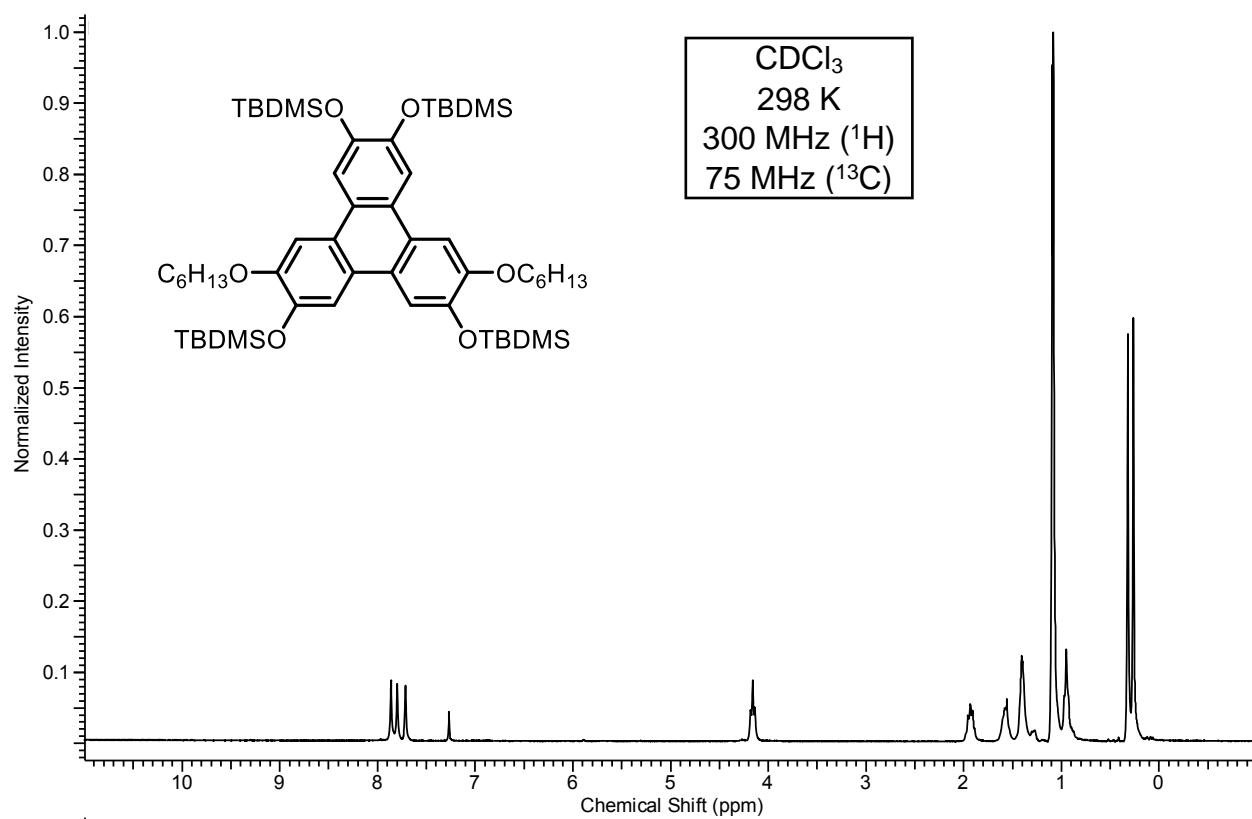
Compound 17



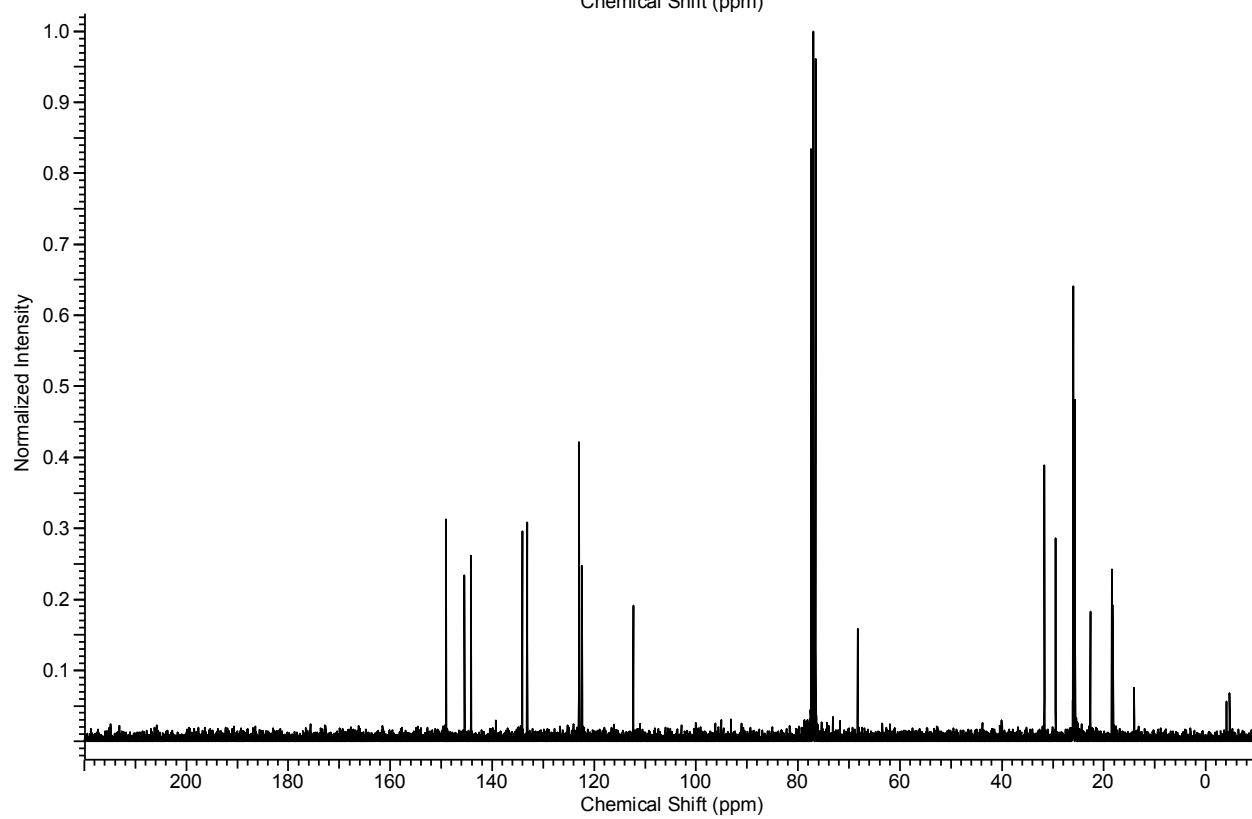
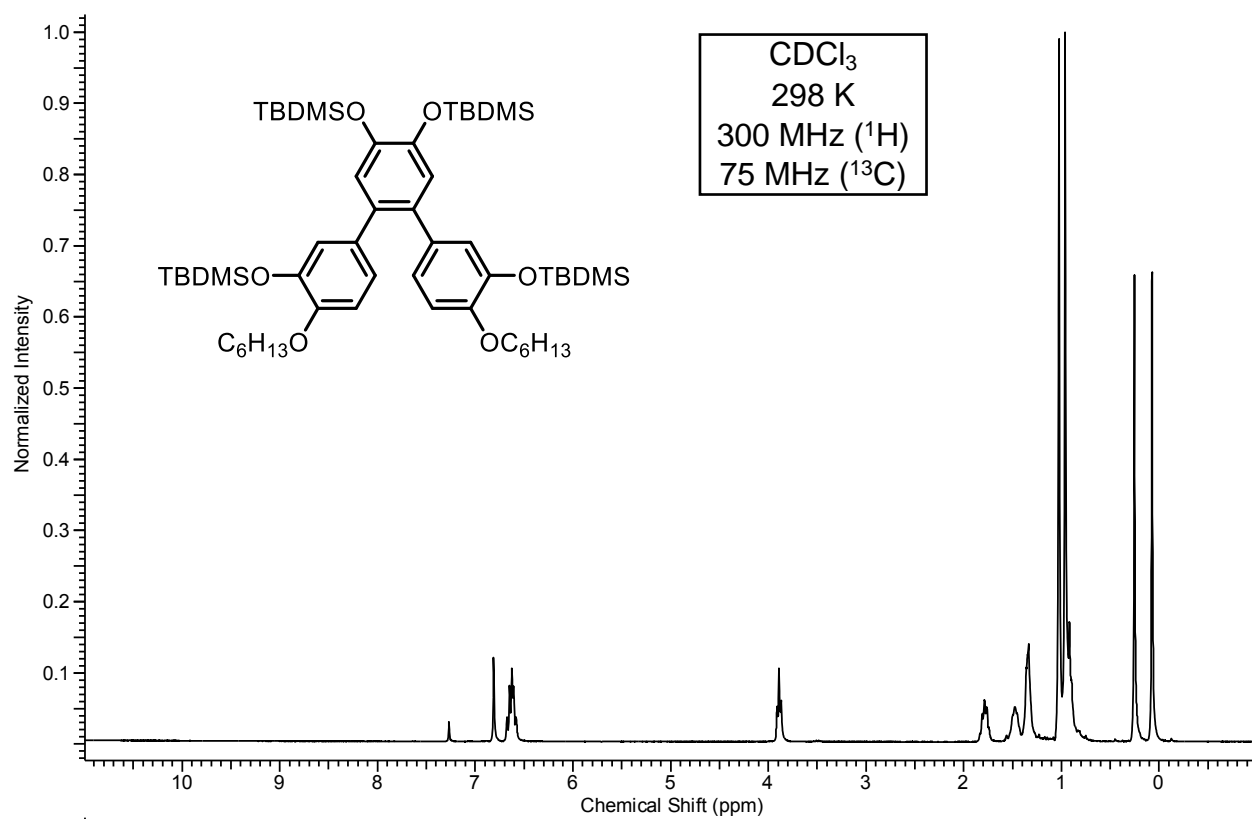
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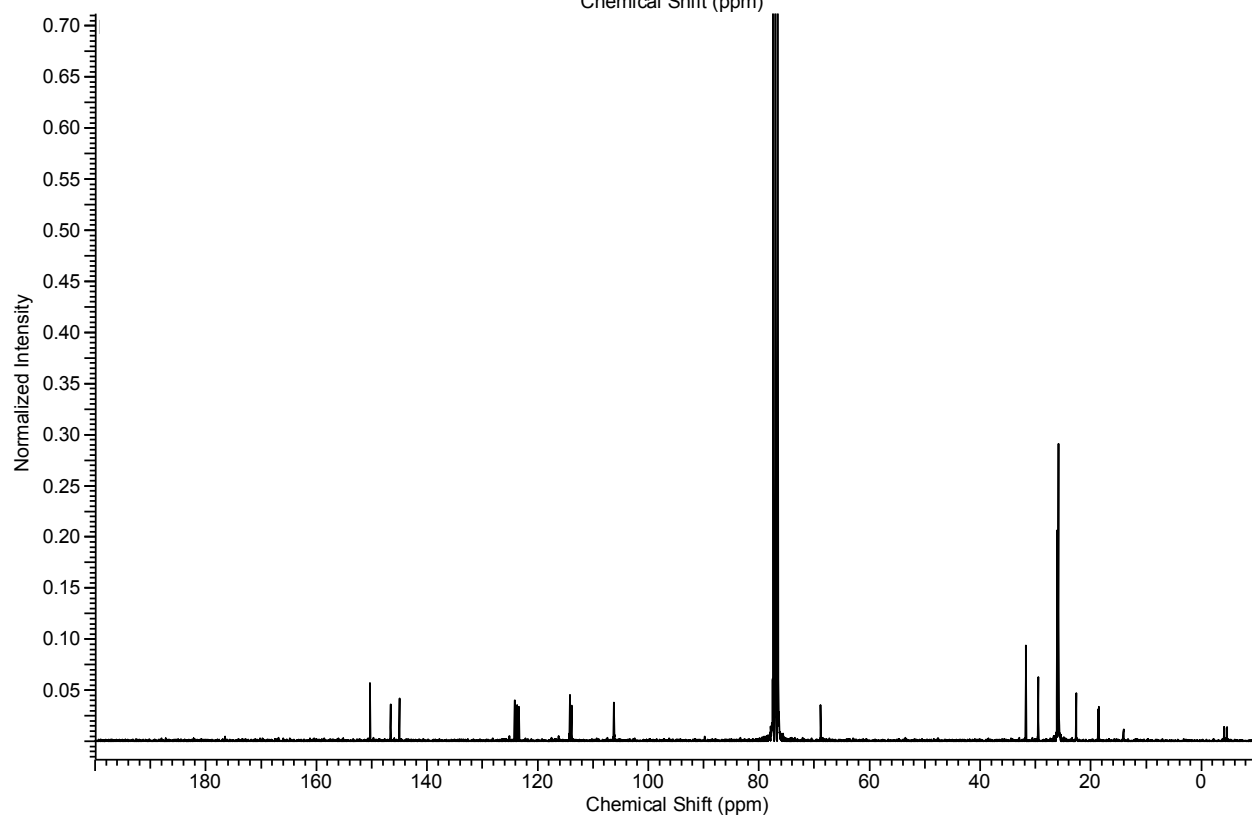
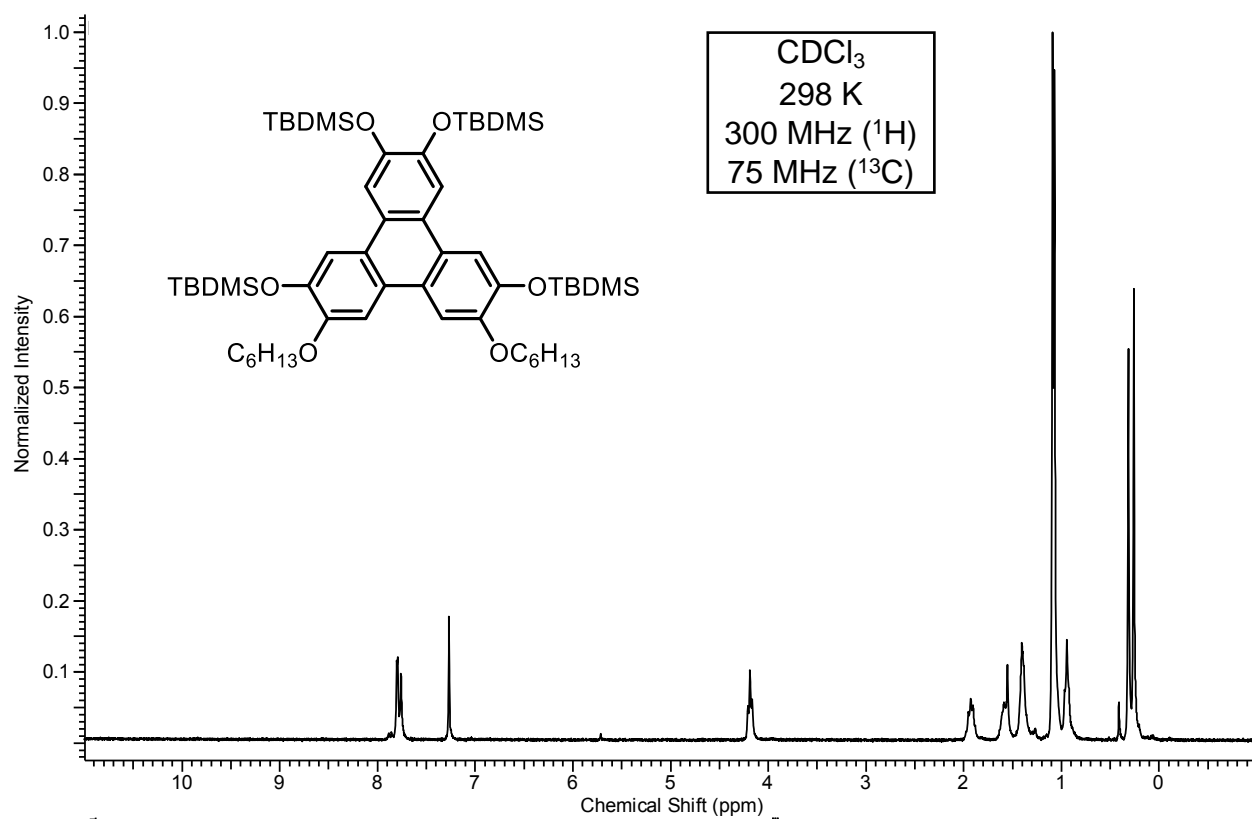
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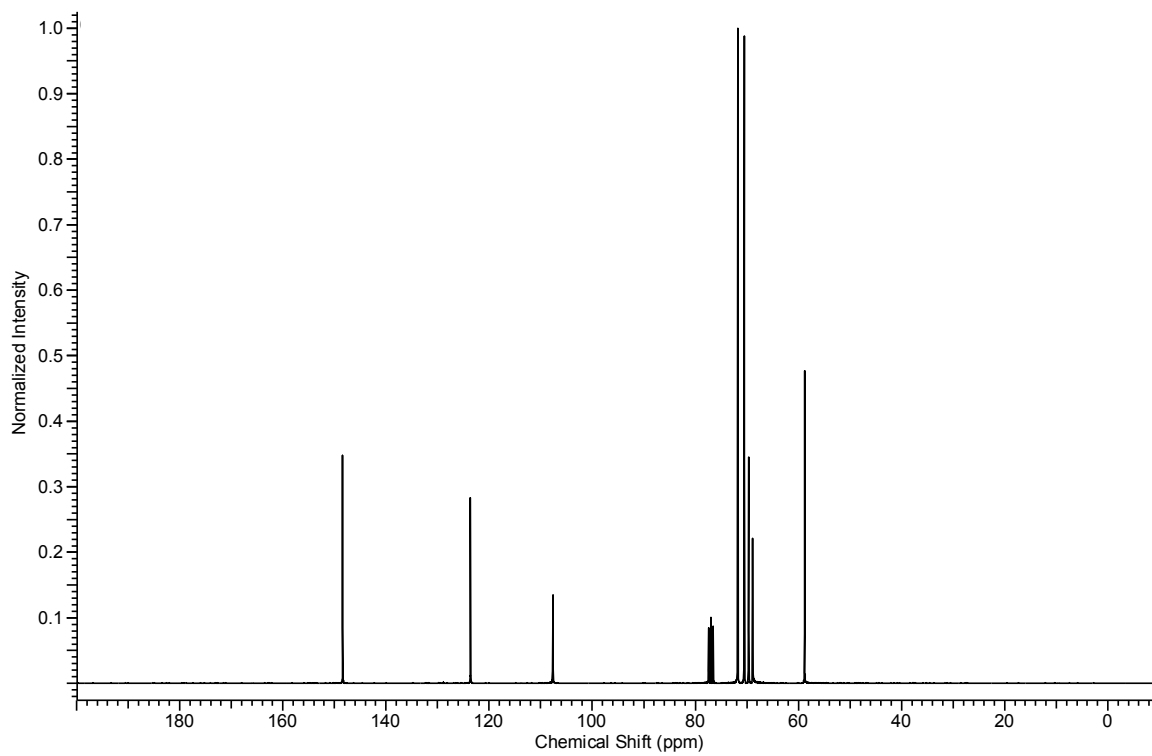
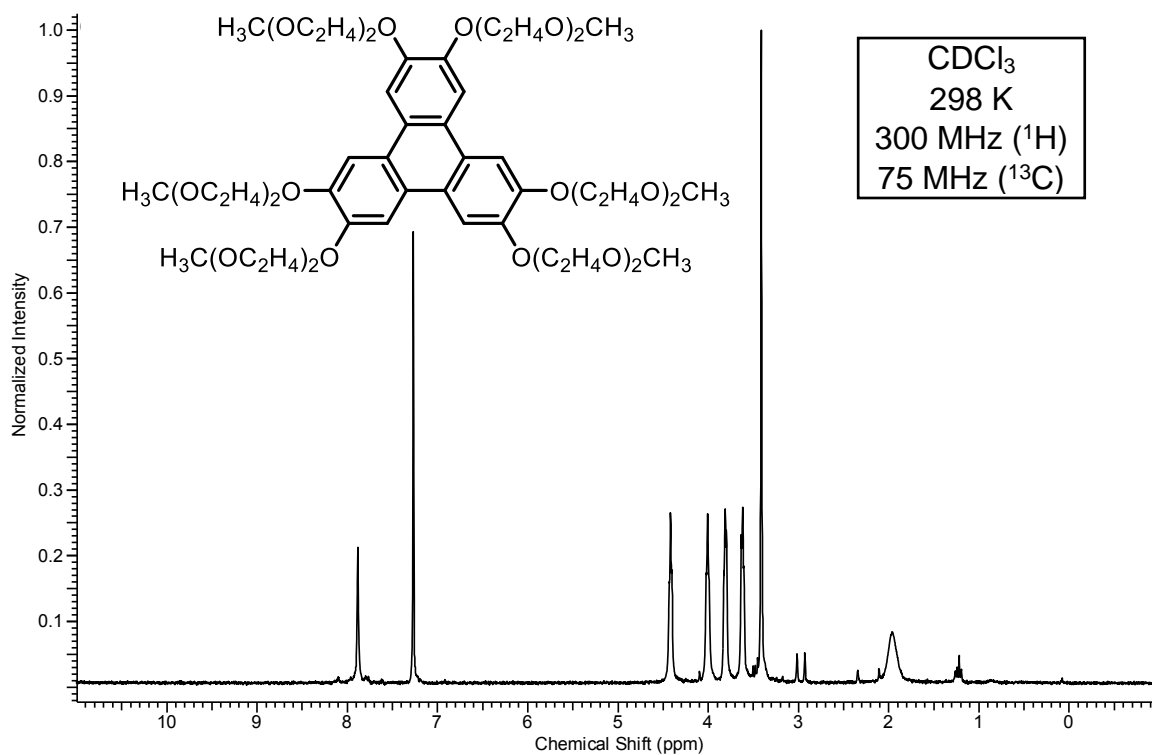
Compound 22



Compound 23

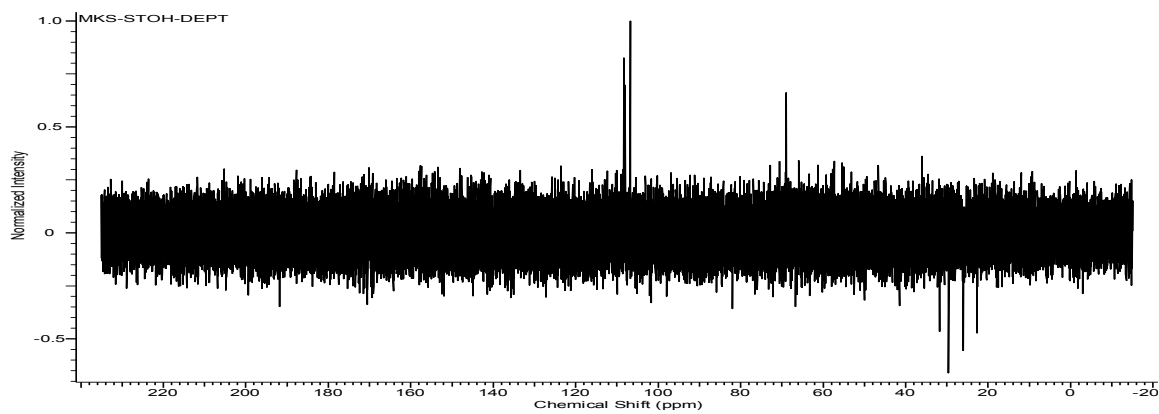


Compound 26

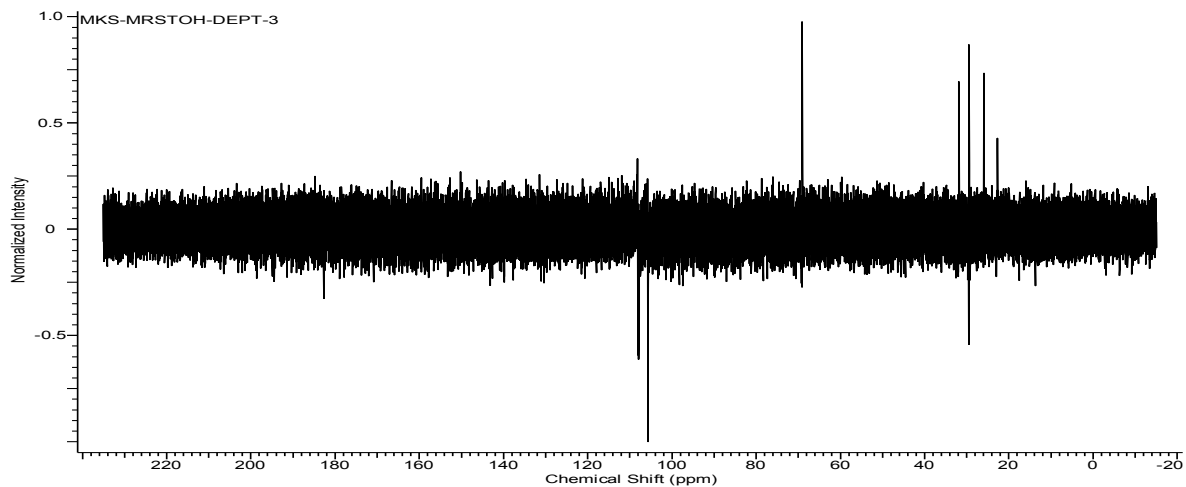


DEPTs

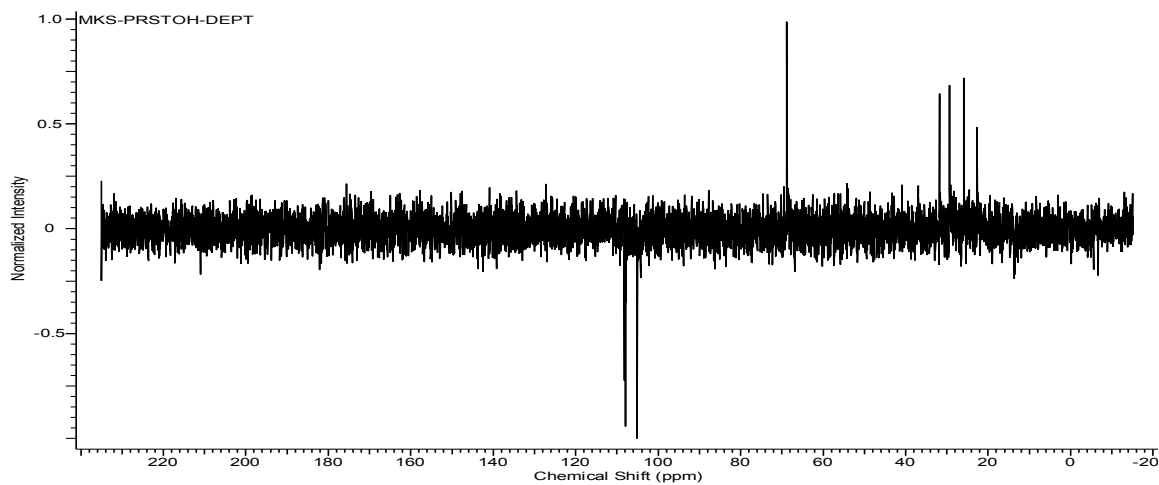
Compound 1 (processed CH up)



Compound 2 (processed CH down)

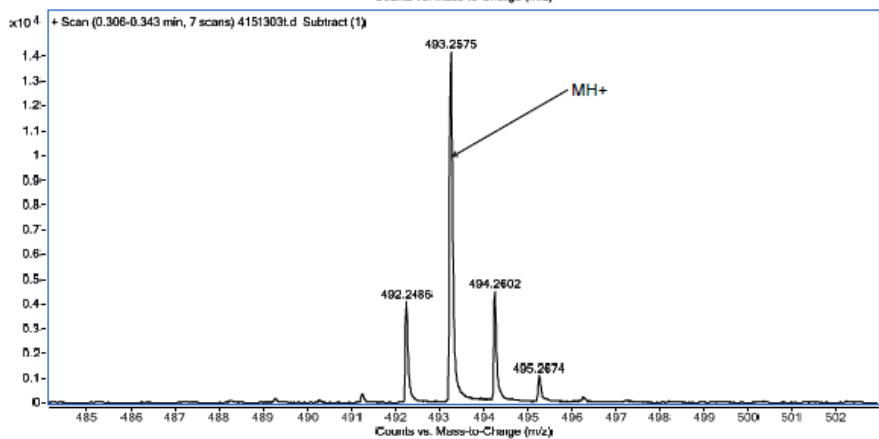
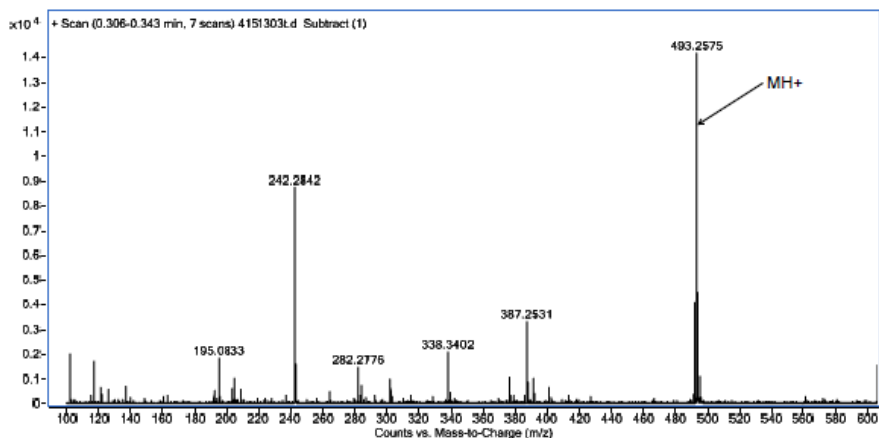


Compound 3 (processed CH down)



IV. Mass Spectrometric Data

Compound 1.

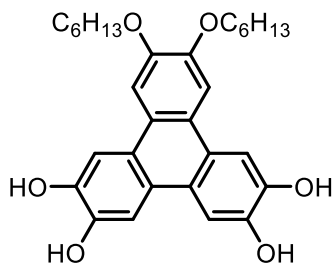


Measured Mass

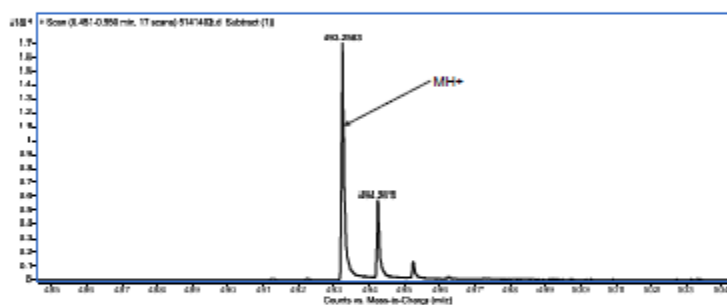
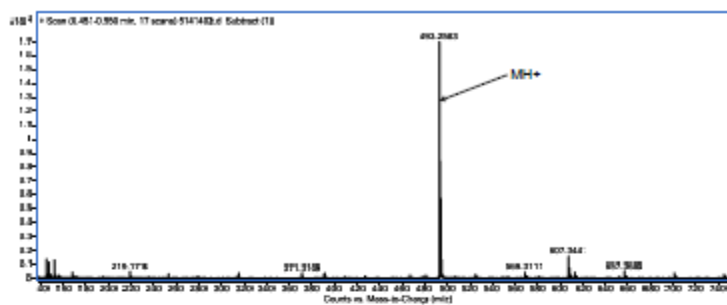
493.2575

Element	Low Limit	High Limit
C	25	35
H	25	45
O	4	8

Formula	Calculated Mass	mDaError	ppmError	RDB
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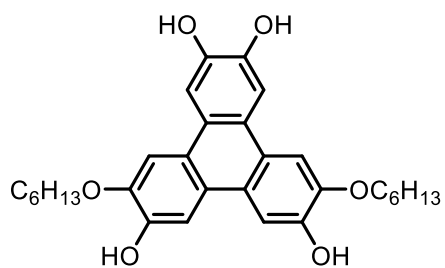


Compound 2

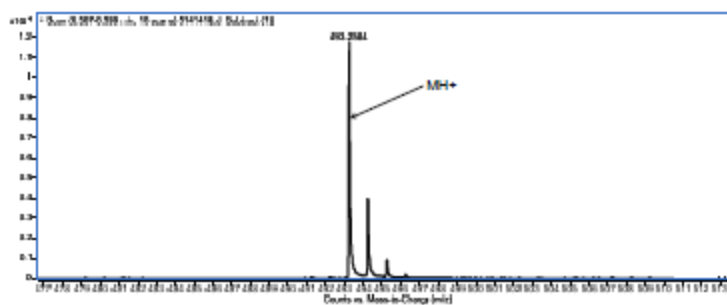
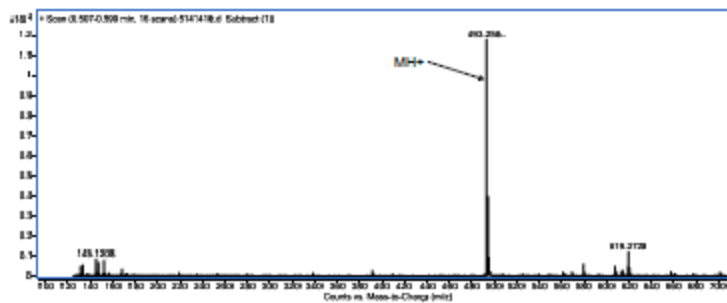


Measured Mass		493.2583			
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H	25	45			
O	4	8			
Na	0	1			

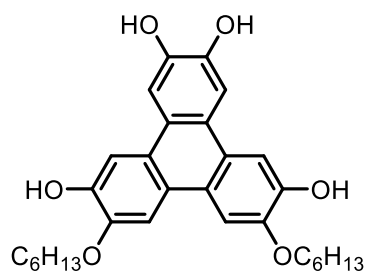
Formula	Calculated Mass	mDaError	ppmError	RDB
C ₃₀ H ₃₇ O ₆	493.2585	-0.2	-0.3	12.5
C ₂₈ H ₃₈ O ₆ Na	493.2561	2.2	4.5	9.5



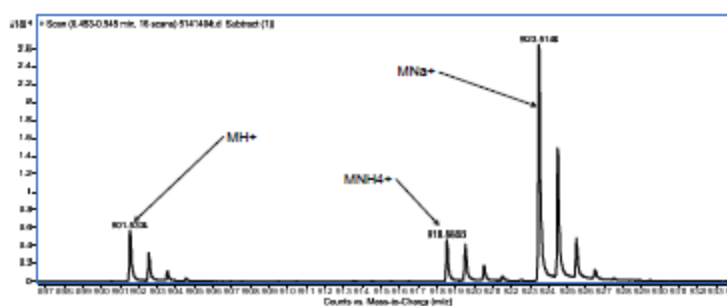
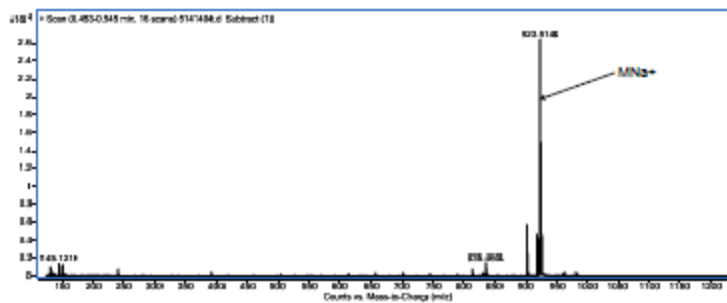
Compound 3



Measured Mass		493.2584			
Element	Low Limit	High Limit			
C	25	35			
H	25	45			
O	4	8			
<u>Formula</u>	<u>Calculated Mass</u>	<u>mDaError</u>	<u>ppmError</u>	<u>RDB</u>	
C30 H37 O6	493.2585	-0.1	-0.1	12.5	

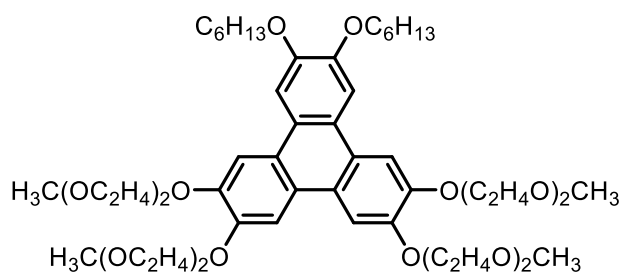


Compound 4

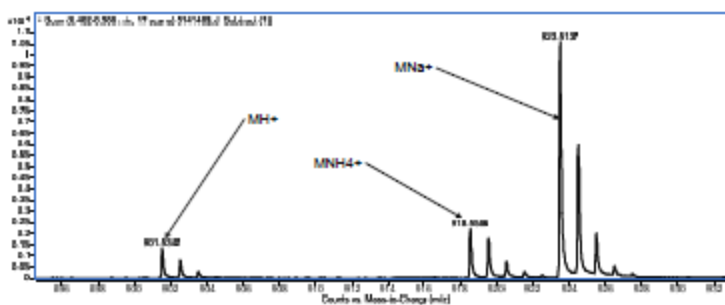
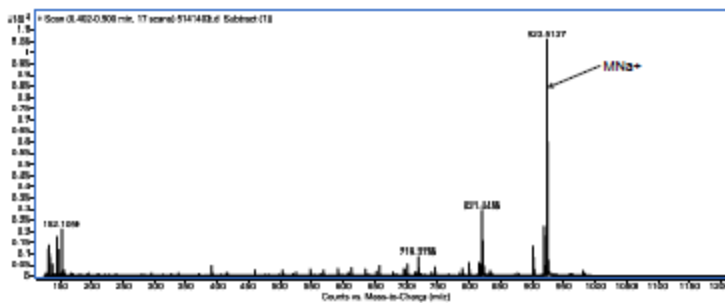


Measured Mass		923.5148			
Element	Low Limit	High Limit			
C	46	56			
H	70	95			
O	5	15			
Na	0	1			

Formula	Calculated Mass	mDaError	ppmError	RDB
C52 H75 O14	923.5151	-0.3	-0.4	15.5
C50 H76 O14 Na	923.5127	2.1	2.2	12.5

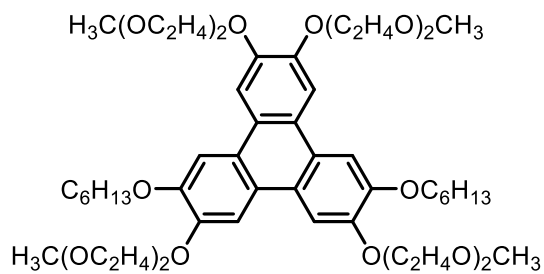


Compound 5

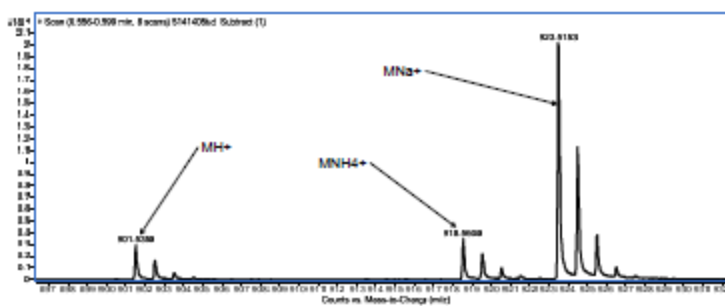
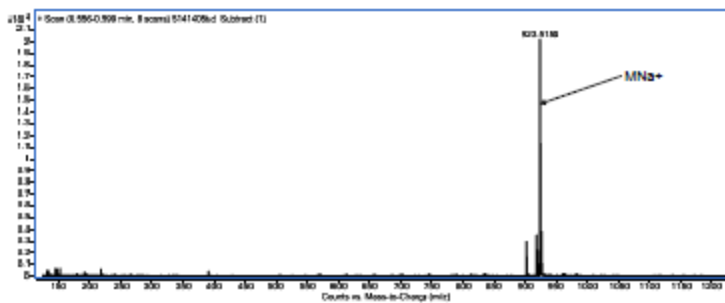


Measured Mass		923.5137			
Element	Low Limit	High Limit			
C	46	56			
H	70	95			
O	5	15			
Na	0	1			

Formula	Calculated Mass	mDaError	ppmError	RDB
C50 H76 O14 Na	923.5127	1.0	1.1	12.5
C52 H75 O14	923.5151	-1.4	-1.6	15.5

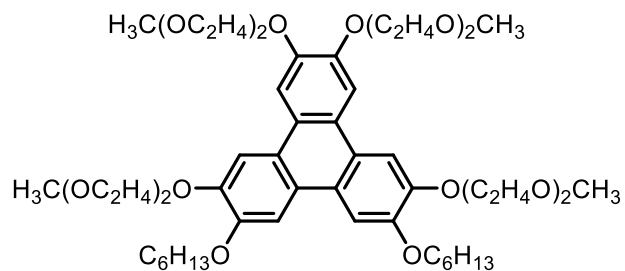


Compound 6

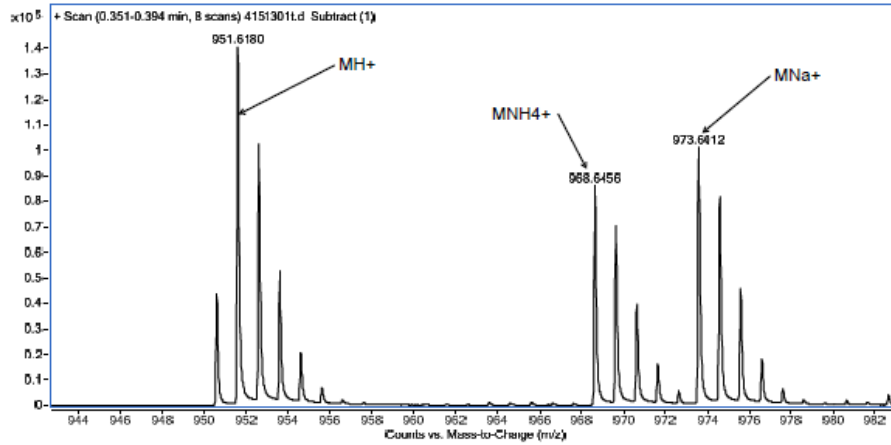
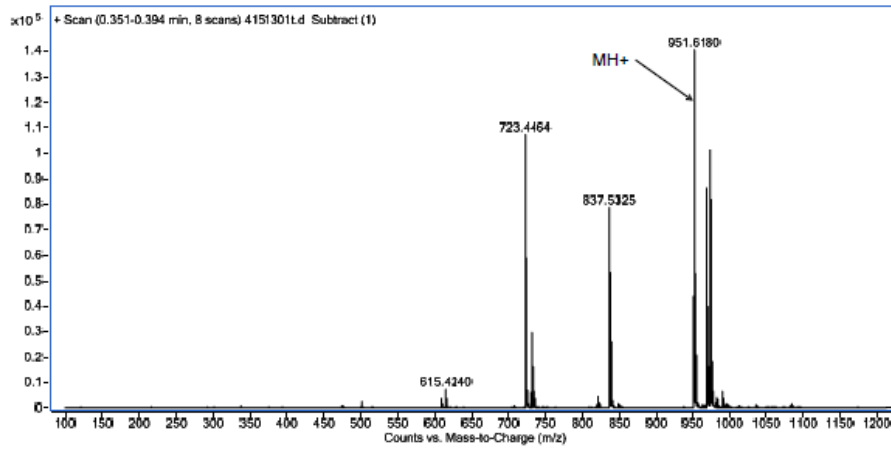


Measured Mass		923.5153		
Element	Low Limit	High Limit		
C	46	56		
H	70	95		
O	5	15		
Na	0	1		

Formula	Calculated Mass	mDaError	ppmError	RDB
C52 H75 O14	923.5151	0.2	0.2	15.5
C50 H76 O14 Na	923.5127	2.6	2.8	12.5



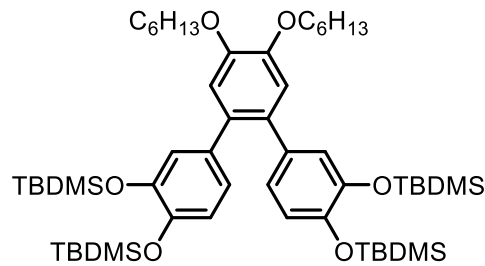
Compound 9



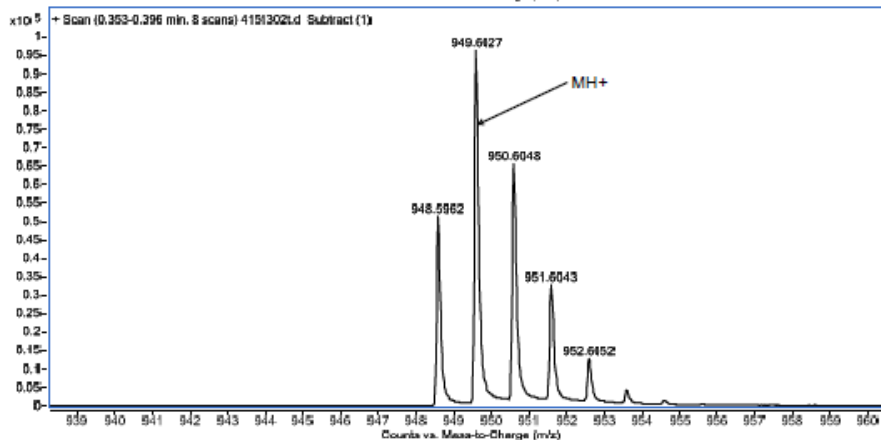
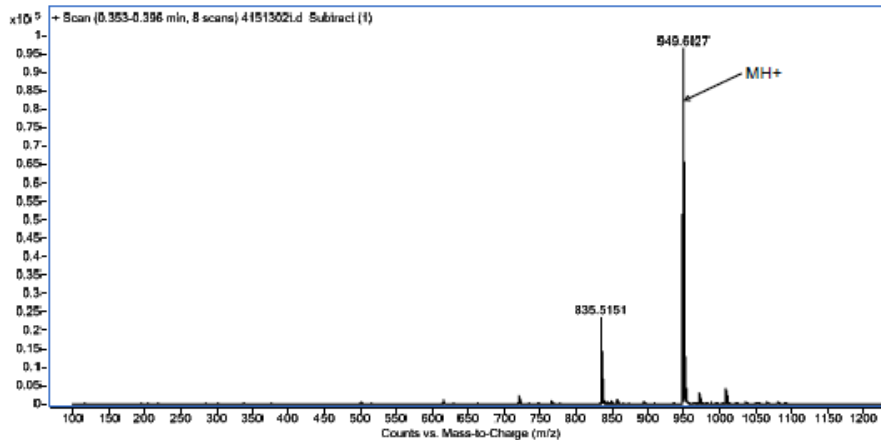
Measured Mass 951.618

Element	Low Limit	High Limit
C	49	59
H	85	105
O	4	8
Si	3	5

Formula	Calculated Mass	mDaError	ppmError	RDB
C ₅₈ H ₉₁ O ₅ Si ₃	951.6169	1.1	1.2	16.5
C ₅₄ H ₉₅ O ₆ Si ₄	951.6200	-2.0	-2.1	11.5



Compound 10

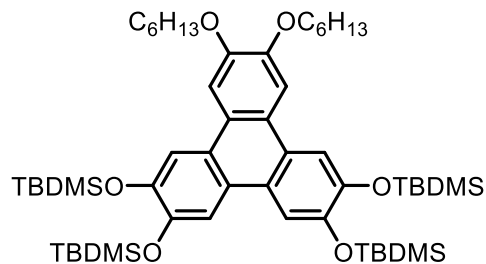


Measured Mass

949.6027

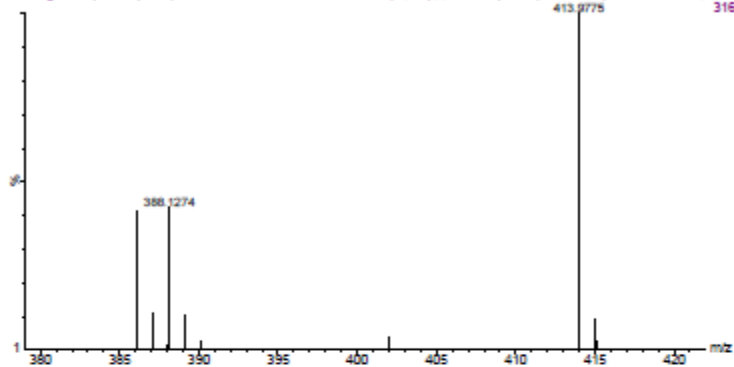
Element	Low Limit	High Limit
C	49	59
H	85	105
O	4	8
Si	3	5

Formula	Calculated Mass	mDaError	ppmError	RDB
C ₅₈ H ₈₉ O ₅ Si ₃	949.6012	1.5	1.5	17.5
C ₅₄ H ₉₃ O ₆ Si ₄	949.6044	-1.7	-1.8	12.5



Compound 13

Northrup(Wesleyan) MR1
 5151401g 1169 (23.554) AM (Cen.1. 80.00. HL5100.0.413.98.1.00): 9m (80. 2x3.00): Cm (1167:1177) TOF MS EI+ 316

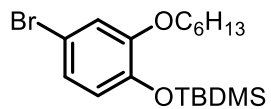


Measured Mass 386.1282

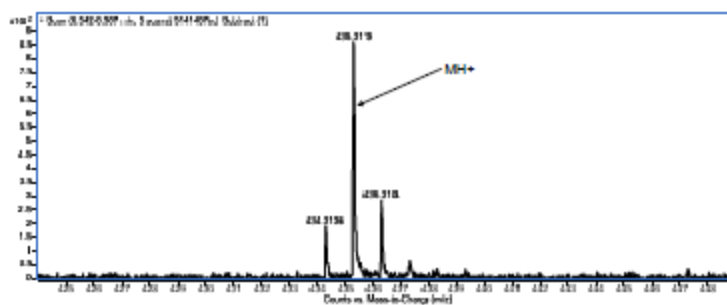
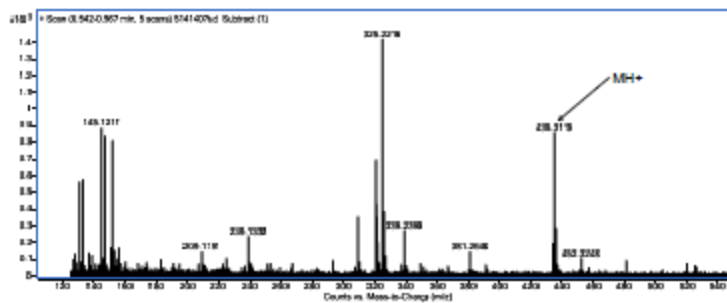
Element	Low Limit	High Limit
C	2	60
H	0	90
N	0	2
O	0	4
Br	0	1
Si	0	1

Formula	Calculated Mass	mDaError	ppmError	RDB
C18 H31 O2 Si Br	386.1271	1.1	2.8	4
C28 H18 O2	386.1301	-1.9	-5.0	20
C23 H18 N2 O4	386.1261	2.1	5.4	16
C22 H27 O Br	386.1240	4.2	10.9	9
C18 H29 N O3 Br	386.1325	-4.3	-11.2	4.5
C26 H18 N2 Si	386.1234	4.8	12.5	20
C24 H22 O3 Si	386.1333	-5.1	-13.1	15
C13 H31 N2 O4 Si Br	386.1231	5.1	13.2	0

Accurate mass done by EI/GCMS for same reasons as sample PR3
 again see expected M+

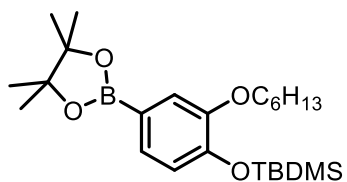


Compound 14

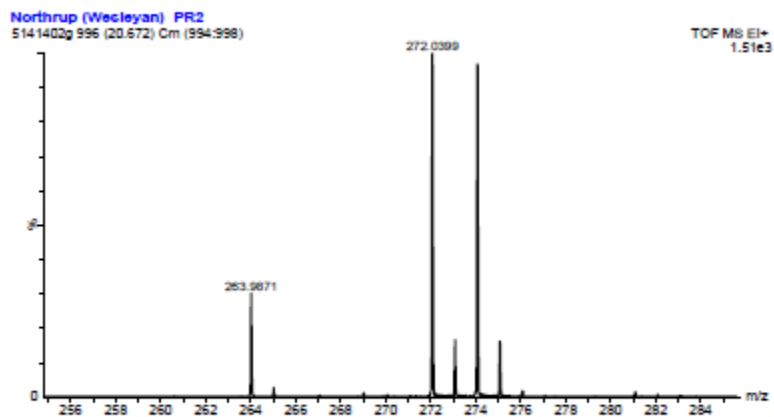


Measured Mass		435.311			
Element	Low Limit	High Limit			
C	19	29			
H	35	55			
O	2	6			
11B	0	1			
Si	0	1			

Formula	Calculated Mass	mDaError	ppmError	RDB
C26 H43 O5	435.3105	0.5	1.1	5.5
C24 H44 11B O4 Si	435.3096	1.4	3.1	4.5



Compound 16

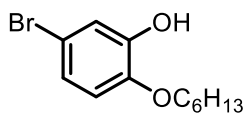


Measured Mass 272.0399

Element	Low Limit	High Limit
C	2	60
H	0	90
N	0	6
O	0	6
Br	0	1

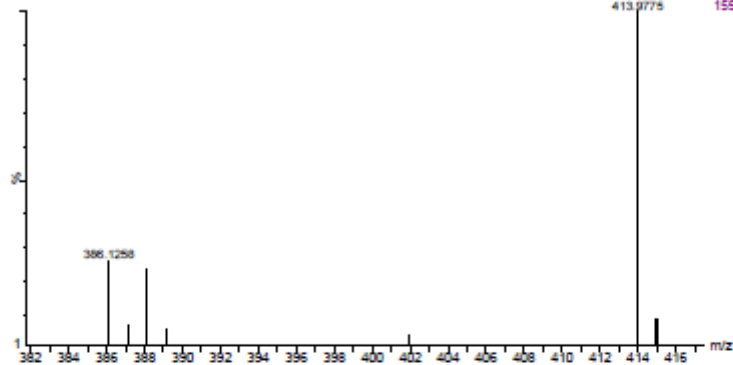
Formula	Calculated Mass	mDaError	ppmError	RDB
C10 H15 N3 O Br	272.0393	0.6	2.2	4.5
C12 H17 O2 Br	272.0406	-0.7	-2.7	4
C11 H6 N5 O4	272.0414	-1.5	-5.6	11.5
C8 H13 N6 Br	272.0380	1.9	7.1	5
C13 H8 N2 O5	272.0428	-2.9	-10.6	11
C20 H4 N2	272.0369	3.0	11.0	20
C7 H17 N2 O4 Br	272.0366	3.3	12.1	0

Accurate mass done via EI/GCMS and shows expected M+



Compound 17

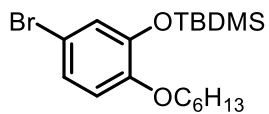
Northrup (Wesleyan) PR3
 5141403g 1167 (23.517) AM (Cen.1. 80.00. Ht.6100.0.413.98.1.00): 6m (90. 2x3.00): Cm (1165:1169) TOF MS ESI-
 155



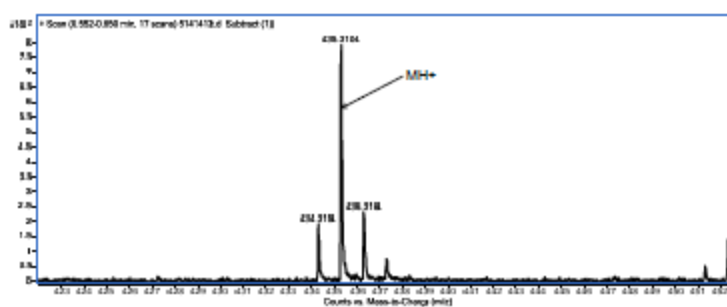
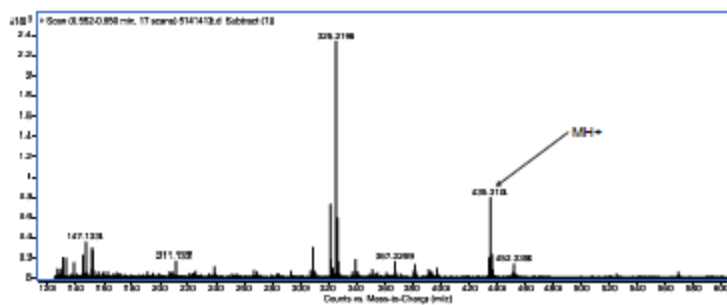
Measured Mass 386.1258

Element	Low Limit	High Limit
C	2	60
H	0	90
N	0	2
O	0	4
Br	0	1
Si	0	1

Formula	Calculated Mass	mDaError	ppmError	RDB
C23 H18 N2 O4	386.1261	-0.3	-0.8	16
C18 H31 O2 Si Br	386.1271	-1.3	-3.4	4
C22 H27 O Br	386.1240	1.8	4.7	9
C26 H18 N2 Si	386.1234	2.4	6.3	20
C13 H31 N2 O4 Si Br	386.1231	2.7	7.0	0
C28 H18 O2	386.1301	-4.3	-11.2	20
C23 H20 N O3 Si	386.1207	5.1	13.2	15.5



Compound 18

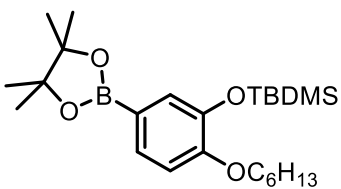


Measured Mass

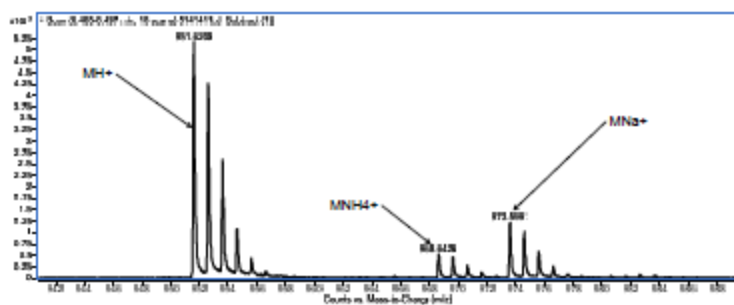
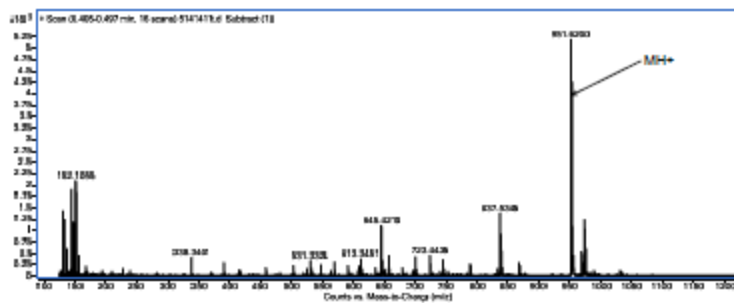
435.3104

Element	Low Limit	High Limit
C	19	29
H	35	55
O	2	6
11B	0	1
Si	0	1

Formula	Calculated Mass	mDaError	ppmError	RDB
C26 H43 O5	435.3105	-0.1	-0.2	5.5
C24 H44 11B O4 Si	435.3096	0.8	1.7	4.5

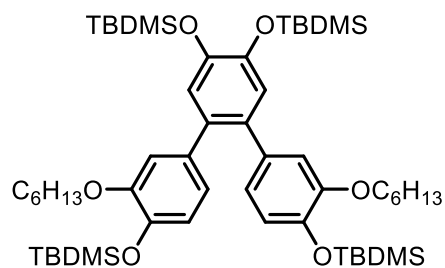


Compound 20

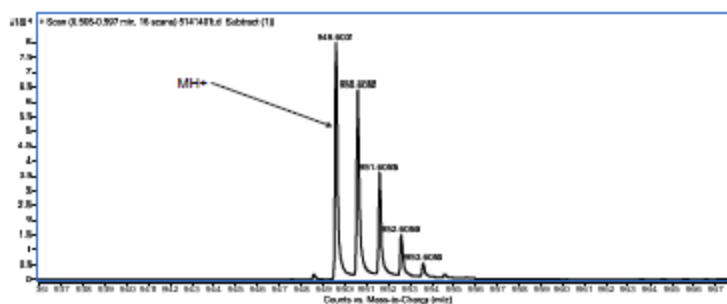
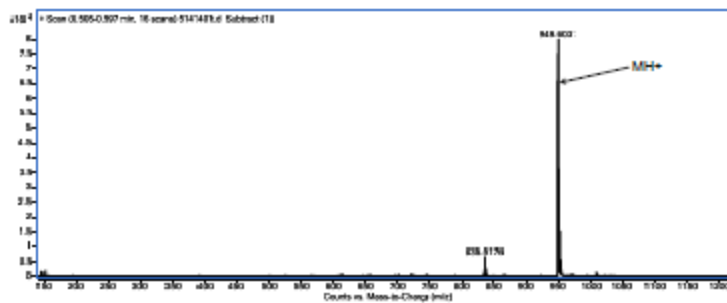


Measured Mass		951.62			
Element	Low Limit	High Limit			
C	49	59			
H	65	105			
O	4	8			
Si	3	5			

Formula	Calculated Mass	mDaError	ppmError	RDB
C54 H95 O6 Si4	951.6200	0.0	0.0	11.5
C58 H91 O6 Si3	951.6169	3.1	3.3	16.5
C50 H99 O7 Si5	951.6232	-3.2	-3.3	6.5

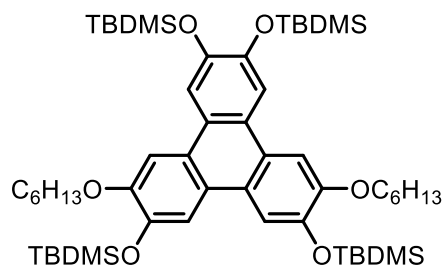


Compound 21

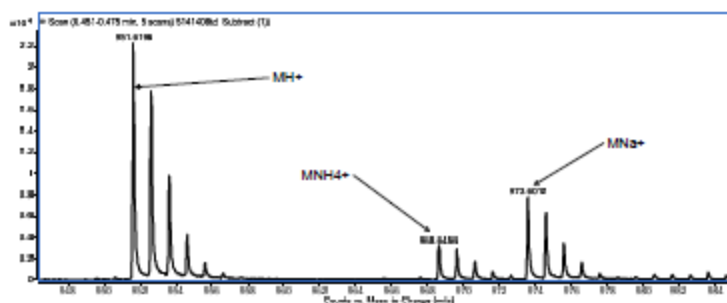
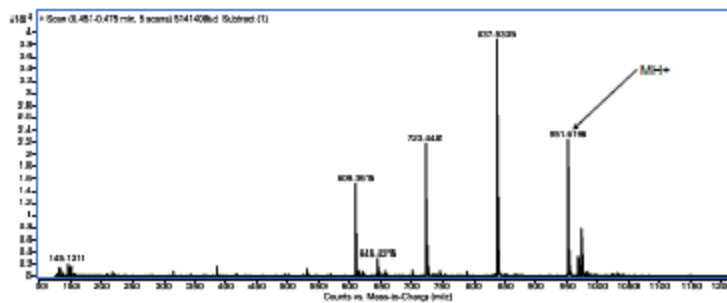


Measured Mass		949.6031			
Element	Low Limit	High Limit			
C	46	56			
H	70	95			
O	5	15			
Si	2	6			

Formula	Calculated Mass	mDaError	ppmError	RDB
C55 H89 O9 Si2	949.6040	-0.9	-0.9	13.5
C54 H93 O6 Si4	949.6044	-1.3	-1.3	12.5
C51 H93 O10 Si3	949.6071	-4.0	-4.2	8.5

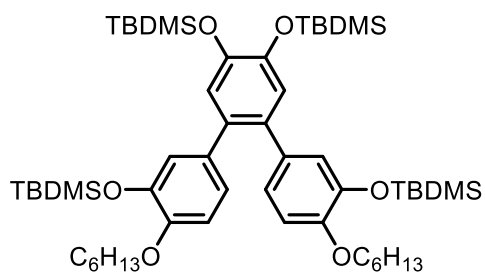


Compound 22

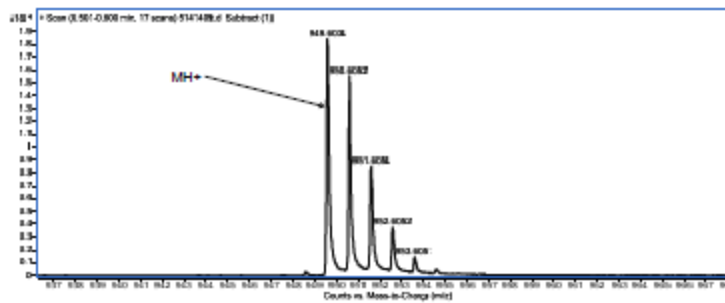
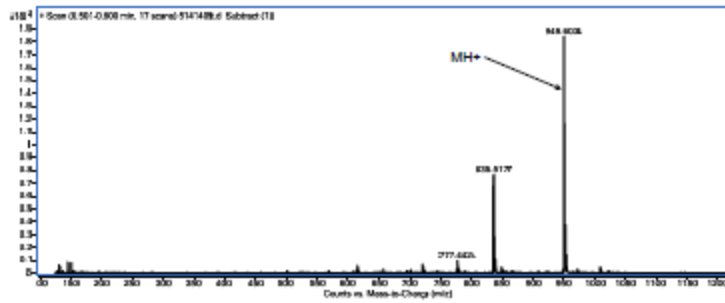


Measured Mass		951.6196			
Element	Low Limit	High Limit			
C	49	59			
H	65	105			
O	4	8			
Si	3	5			

Formula	Calculated Mass	mDaError	ppmError	RDB
C54 H95 O6 Si4	951.6200	-0.4	-0.5	11.5
C58 H91 O6 Si3	951.6169	2.7	2.8	16.5
C50 H99 O7 Si5	951.6232	-3.6	-3.8	6.5

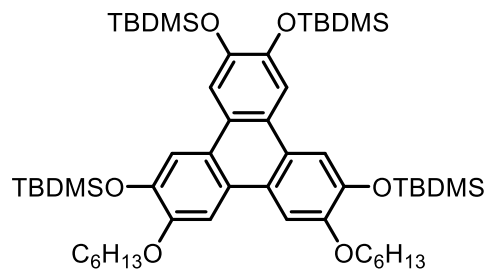


Compound 23



Measured Mass		949.6034			
Element	Low Limit	High Limit			
C	49	59			
H	85	105			
O	4	8			
Si	3	5			

Formula	Calculated Mass	mDaError	ppmError	RDB
C54 H93 O6 Si4	949.6044	-1.0	-1.0	12.5
C58 H89 O6 Si3	949.6012	2.2	2.3	17.5
C50 H97 O7 Si5	949.6075	-4.1	-4.3	7.5



V. UV/Vis & Fluorescence Spectroscopy

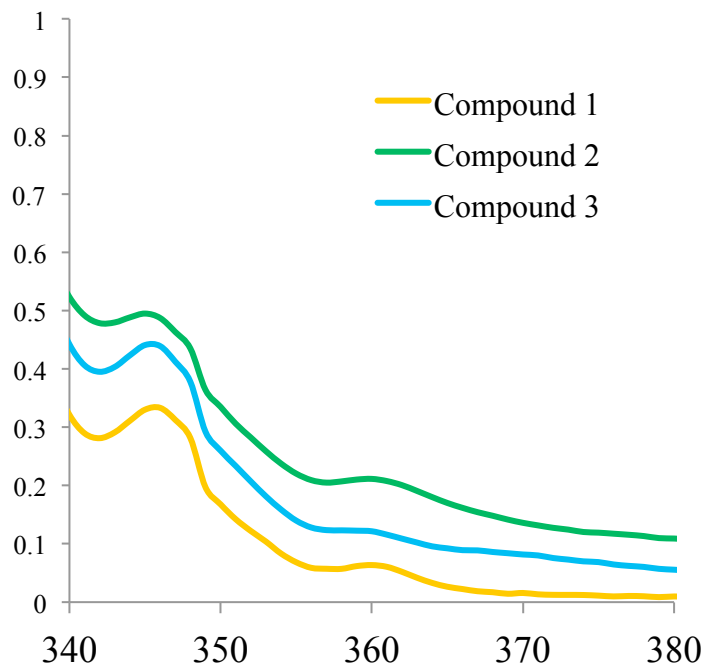


Figure S1: Overlaid UV/Vis spectra of triphenylene tetra-ols **1-3**. All spectra were collected in THF (1.0×10^{-5} M) at 298 K.

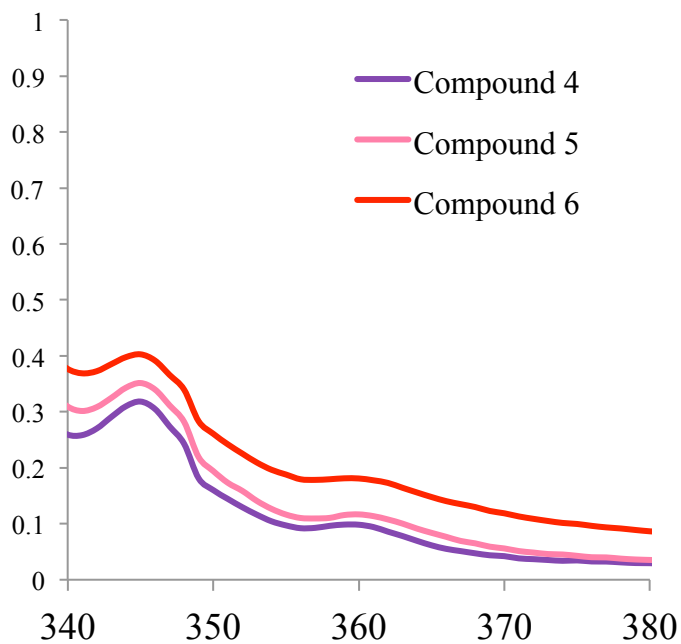


Figure S2: Overlaid UV/Vis spectra of amphiphilic triphenylenes **4-6**. All spectra were collected in THF (1.0×10^{-5} M) at 298 K.

VI. Differential Scanning Calorimetry

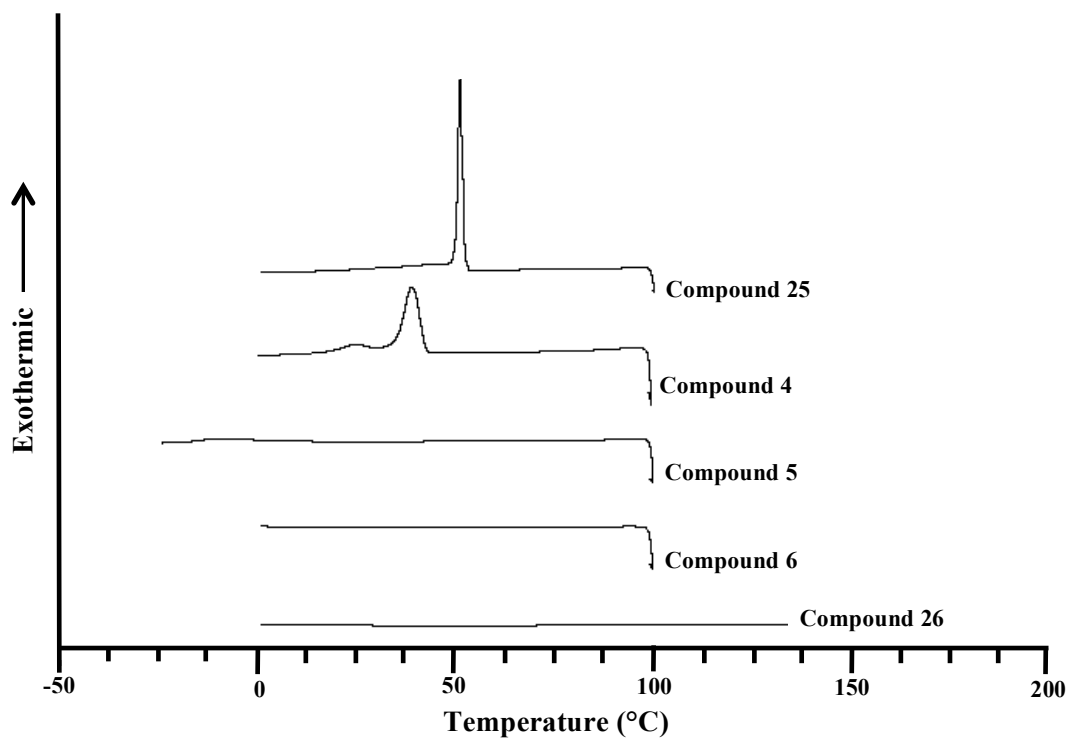


Figure S3. DSC cooling thermal diagram traces for **Compounds 4-6, 25, and 26** at 5°C/min.

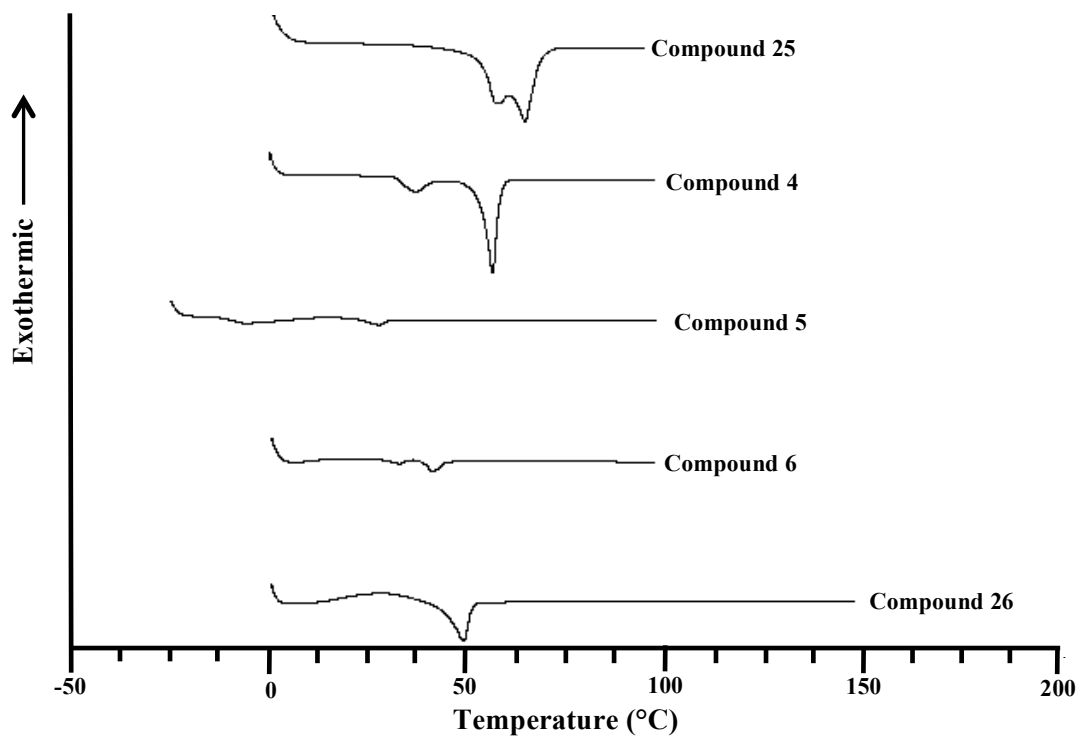


Figure S4. DSC second heating thermal diagram traces for **Compounds 4-6, 25, and 26** at 10°C/min.

VII. Supporting References

- (1) T. Sauer and G. Wegner, *Mol. Cryst. Liq. Cryst.*, 1988, **162**, 97.
- (2) V. Bhalla, H. Singh and M. Kumar, *Org. Lett.*, 2010, **12**, 628.
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