Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

2,6,10-Triphenylenotriyne:

a star-shaped trisaryne synthon

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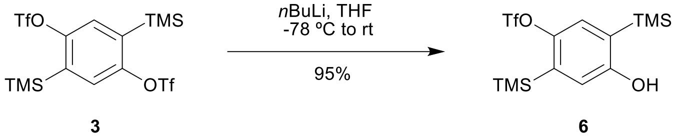
1. Experimental Procedures

1.1. *General methods*

All reactions were carried out under argon using oven-dried glassware. THF, DCM and MeCN were purified in a MBraun SPS-800 Solvent Purificarion System. Finely powdered CsF was dried under vacuum at 100 °C, cooled under argon and stored in a glove-box. $Pd(PPh_3)_4$ was prepared from $PdCl_2$ following a published procedure.[1] 1,4-benzodiyne precursor was prepared following a published procedure[2] and it is also commercially available in ABCR. Other commercial reagents were purchased from ABCR GmbH or Sigma-Aldrich, and were used without further purification. TLC was performed on Merck silica gel 60 F254 and chromatograms were visualized with UV light (254 and 365 nm). Column chromatography was performed on Merck silica gel 60 (ASTM 230-400 mesh). ^{1}H ^{19}F NMR and ^{13}C NMR spectra were recorded at 300, 282 and 75 MHz (Varian Mercury-300 instrument), 500, 470 and 125 MHz (Varian Inova 500) or 750, 705 and 188 MHz (Neo 750 instrument) respectively.

1.2. *Synthesis of the 2,6,10-triphenylenotriyne precursors 2*

Synthesis of 4-hydroxy-2,5-bis(trimethylsilyl)phenyl trifluoromethanesulfonate (6)



To a solution of bistriflate **6** (500 mg, 0,965 mmol) in THF (3.25 mL, 0.3M) at -78 °C, *n*BuLi (0.39 mL, 2.5 M in hexanes) was slowly dropwise added (for over 10 min). The reaction mixture was allowed to warm up to room temperature overnight. Then, volatiles were removed under reduced pressure and the reaction crude was purified by column chromatography (silica gel, 1:1 Hex:DCM) to afford phenol **6** as a white solid (354 mg, 95%). Spectroscopic data were coincident with those described in the literature for this compound.[3]

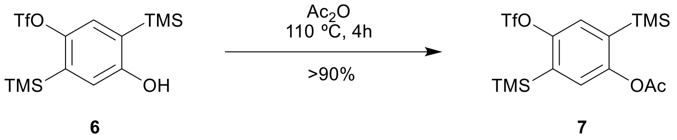
6: ^{1}H NMR (300 MHz, $CDCl_3$), δ: 7.23 (s, 1H), 6.75 (s, 1H), 4.96 (s, 1H), 0.35 (s, 9H), 0.31 (s, 9H) ppm.

Table S1. Optimization of the reaction of bistriflate **3** with *n*BuLi.

Entry[a]	T / °C	*n*BuLi/ mol%	Scale / g	Yield / %
1	rt	110	0.2	46
2	0	110	0.2	51
3	-20	110	0.2	66
4	-78	110	0.2	85
5	-100	110	0.2	85
6	-100	140	0.2	90
7	-100	200	0.2	95
8	-78	200	0.5	95
9	-78	200	1	90
10	-78	200	3	30

[a] All reactions were carried out under Ar atmosphere and in THF (0.3 M). Optimization yields were estimated by ^{1}H NMR directly from the reaction crude.

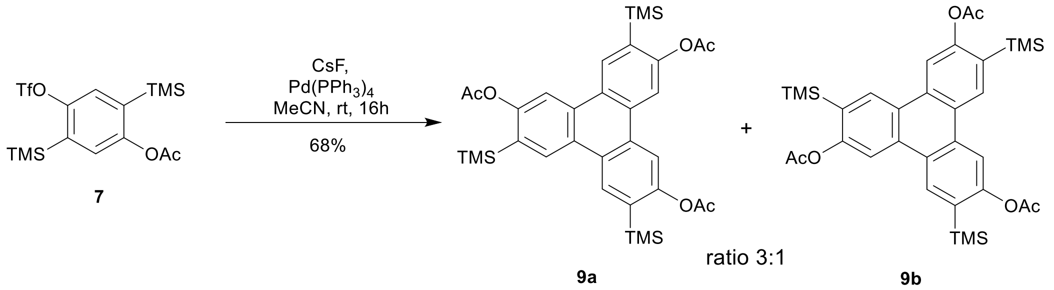
Synthesis of 4-[(trifluoromethyl)sulfonyl)oxy]-2,5-bis(trimethylsilyl)phenyl acetate (7)



In a round bottom flask, phenol **6** (5.7 g, 14.8 mmol) and acetic anhydride (225 mL) were placed. The reaction mixture was stirred and heated at 110 °C for 4 h. Then, the mixture was slowly added over satd. aq. $NaHCO_3$ (1 L) and stirred for 2 h. The resulting aqueous solution was extracted with Et_2O (3 x 100 mL), the combined organic layers were washed with brine (100 mL), dried over anhyd. Na_2SO_4, filtered and the volatiles were removed under reduced pressure to afford acetate **7** as a brown solid (5.7 g, 90%), which was used in the next step without further purification.

7: Brown solid, m.p.: 57-59 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 7.39 (s, 1H), 7.18 (s, 1H), 2.33 (s, 3H), 0.36 (s, 9H), 0.29 (s, 9H) ppm. ^{19}F NMR (282 MHz, $CDCl_3$), δ: -74.4 ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$), δ: 169.4 (C), 153.9 (C), 152.4 (C), 136.2 (C), 135.7 (C), 130.0 (CH), 125.8 (CH), 21.5 (CH_3), -0.9 (CH_3), -1.22 (CH_3) ppm. MS (APCI), m/z: 429 (M^++1, 100). HRMS (APCI), m/z found: 429.0829 (calcd. for $C_{15}H_{24}F_3O_5SSi_2$: 429.0830).

Synthesis of trisacetates 9a and 9b

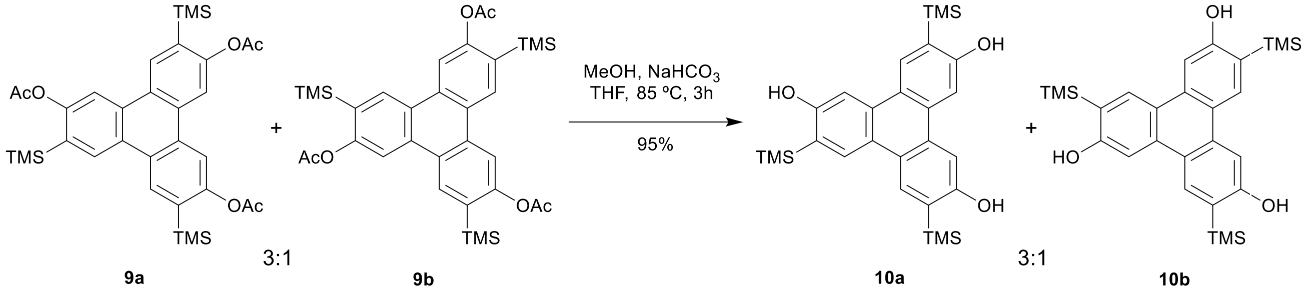


In a Schlenk tube, the benzyne precursor **11** (0.5 g, 1.16 mmol) and $Pd(PPh_3)_4$ (135 mg, 10 mol%) were placed and dissolved in MeCN (15 mL). Then, CsF (530 mg, 3.48 mmol) was added. After stirring for 16 h at rt, the solvent was removed under reduced pressure. The crude reaction product was purified by column chromatography (silica gel, 1:1 Hex:Et_2O) to yield the desired mixture of products (**9a**,**9b**, 3:1 isomeric ratio, 159 mg, 66% yield). A second column chromatography (silica gel, 4:1 Hex:Et_2O) allowed the isolation of pure samples of both isomers.

9a: White solid, m.p.: 256-257 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.76 (s, 2H), 8.61 (s, 1H), 8.25 (s, 1H), 8.18 (s, 2H), 2.44 (s, 3H), 2.42 (s, 6H), 0.44 (s, 9H), 0.43 (s, 9H), 0.43 (s, 9H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$) δ: 169.73 (C), 169.7 (C), 169.6 (C), 155.0 (C), 154.6 (C), 154.5 (C), 132.0 (2C), 131.6 (CH), 131.3 (2C), 131.2 (C), 130.8 (2CH), 130.7 (C), 127.1 (C), 126.9 (C), 126.6 (C), 116.2 (2CH), 115.6 (CH), 21.8 (CH_3), 21.7 ($2CH_3$), -0.6 (CH_3), -0.8 ($2CH_3$) ppm. MS (APCI), m/z: 619 (M^++1, 100), 618 (M^+, 64). HRMS (APCI), m/z found: 619.2361 (calcd. for $C_{33}H_{43}O_6Si_3$: 619.2362).

9b: White solid, m.p.: 321-322 ºC. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.60 (s, 3H), 8.25 (s, 3H), 2.44 (s, 9H), 0.43 (s, 27H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$), δ: 169.7 (C), 155.1 (C), 132.3 (C), 131.4 (CH), 131.2 (C), 126.3 (C), 115.6 (CH), 21.8 (CH_3), -0.6 (CH_3) ppm. MS (APCI), m/z: 619 (M^++1, 100), 618 (M^+, 82). HRMS (APCI), m/z found: 619.2359 (calcd. for $C_{33}H_{43}O_6Si_3$: 619.2362).

Synthesis of trisphenols 10a and 10b

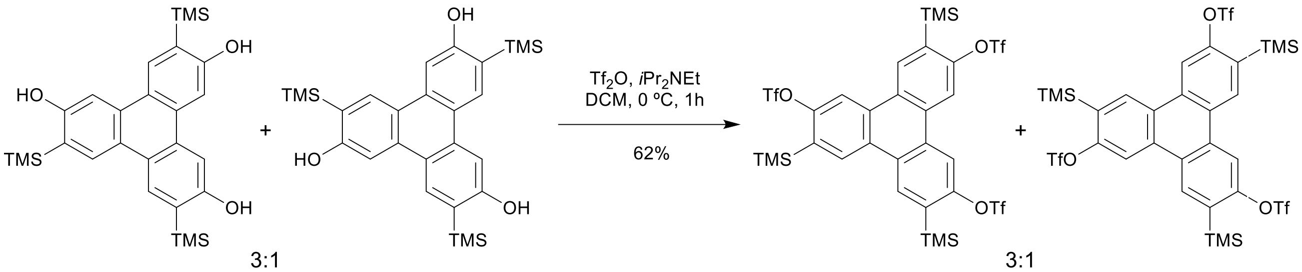


In a round bottom flask, a mixture of isomers **9a** and **9b** (3:1, 160 mg, 0.258 mmol) was dissolved in THF (22 mL, 0.01 M). Then, a 1:1 mixture of satd. aq. $NaHCO_3$ and MeOH (22 mL) was added. The reaction was stirred under reflux for 3 h. Then, the phases were separated, the aqueous layer was acidified to pH=1 (by adding 10% aq. HCl) and extracted with AcOEt (3 x 20 mL). The combined organic layers were dried ($MgSO_4$) and filtered. Volatiles were removed under reduced pressure. The crude residue was dissolved in DCM (60 mL), the solution was dried over $MgSO_4$ and concentrated under reduced pressure, isolating the desired mixture of products **10a** and **10b** (3:1 isomeric ratio, 121 mg, 95% yield). A second column chromatography (silica gel, 9:1 Hex:AcOEt) allowed the isolation of pure samples of both isomers.

10a: White solid, m.p.: 191-192 ºC. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.69 – 8.54 (s, 2H), 8.48 (s, 1H), 7.77 (s, 1H), 7.51 (s, 2H), 5.45 (s, 1H), 5.36 (s, 2H), 0.47 (s, 9H), 0.46 (s, 9H), 0.43 (s, 9H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$) δ: 159.8 (C), 159.0 (C), 158.7 (C), 132.4 (C), 131.3 (CH), 130.5 (2CH), 127.5 (C), 127.2 (C), 124.6 (C), 124.1 (C), 123.1 (C), 122.6 (C), 108.3 (C), 107.0 (2CH), 106.4 (CH), -0.69 (CH_3) ppm. MS (APCI), m/z: 493 (M^++1, 19). HRMS (APCI), m/z found: 493.2040 (calcd. for $C_{27}H_{37}O_3Si_3$: 493.2045).

10b: White solid, m.p.: 291-292 ºC. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.45 (s, 3H), 7.73 (s, 3H), 5.22 (s, 3H), 0.45 (s, 27H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$), δ: 160.1 (C), 133.5 (C), 131.3 (CH), 125.4 (C), 121.7 (C), 106.4 (CH), -0.58 (CH_3) ppm. MS (APCI), m/z: 493 (M^++1, 100). HRMS (APCI), m/z found: 493.2044 (calcd. for $C_{27}H_{37}O_3Si_3$: 493.2045).

Synthesis of tristriflates 2a and 2b



10a **10b** **2a** **2b**

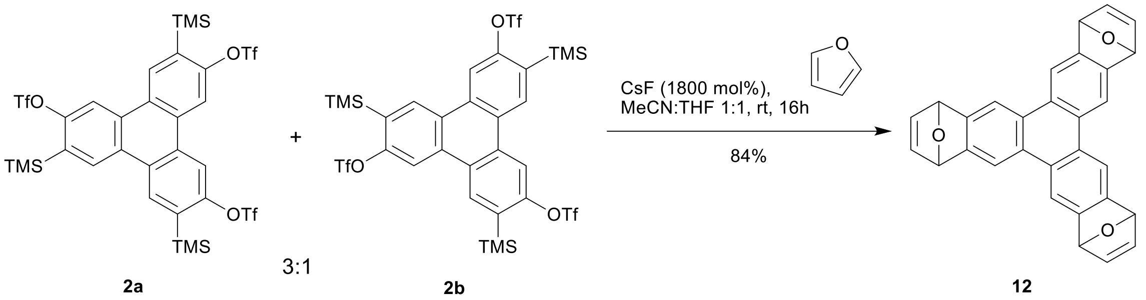
To a solution of triols **10a** and **10b** (80 mg, 0.13 mmol), and *i*Pr$_2$NEt in dry DCM (2 mL, 0.08 M) at 0 °C, Tf$_2$O (161 µL, 0.96 mmol) was dropwise added. After stirring at 0 °C for 1 h, the reaction was quenched with 10 mL of satd. aq. NaHCO$_3$. The resulting mixture was extracted with DCM (3 x 5 mL) and the volatiles were removed under reduced pressure. The reaction crude was purified by column chromatography (SiO$_2$, 1:3 Et$_2$O:Hex) allowing the isolation of a mixture of products **2a** and **2b** (3:1 isomeric ratio, 89.5 mg, 62% yield). A second column chromatography (silica gel, 2.5% Et$_2$O in Hex) allowed the isolation of pure samples of both isomers.

2a: White solid, m.p.: 222-223 ºC. ^{1}H NMR (500 MHz, CDCl$_3$), δ: 8.82 (s, 1H), 8.81 (s, 1H), 8.67 (s, 1H), 8.56 (s, 1H), 8.40 (s, 1H), 8.40 (s, 1H), 0.54 (s, 18H), 0.53 (s, 9H) ppm. ^{19}F NMR (471 MHz, CDCl$_3$), δ: -74.07, -74.14, -74.13 ppm. ^{13}C NMR-DEPT (125 MHz, CDCl$_3$), δ: 155.2 (C), 155.0 (C), 155.0 (C), 134.0 (C), 133.9 (C), 133.0 (C), 132.4 (CH), 132.3 (2xCH), 131.9 (C), 131.6 (C), 131.4 (C), 127.9 (C), 127.8 (C), 127.5 (C) , 122.7 – 114.9 (q, J = 320.3 Hz, CF$_3$), 118.8 (q, J = 320.3 Hz, 2xCF$_3$), 114.0 (2xCH), 113.8 (CH), -0.8 (CH$_3$), -0.9 (CH$_3$) ppm. MS (APCI), m/z: 873 (M$^+$-15, 95), 888 (M$^+$, 86). HRMS (APCI), m/z found: 888.0447 (calcd. for $C_{30}H_{33}F_9O_9S_3Si_3$: 888.0445).

2b: White solid, m.p.: 247-249 ºC. ^{1}H NMR (500 MHz, CDCl$_3$), δ: 8.66 (s, 3H), 8.56 (s, 3H), 0.52 (s, 27H) ppm. ^{19}F NMR (471 MHz, CDCl$_3$), δ: -74.1 ppm. ^{13}C NMR-DEPT (125 MHz, CDCl$_3$), δ: 155.4 (C), 133.1 (C), 132.4 (CH), 132.1 (C), 127.3 (C), 118.8 (q, J = 320.3 Hz, CF$_3$), 113.8 (CH), -0.9 (CH$_3$) ppm. MS (APCI), m/z: 873 (M$^+$-15, 100), 888 (M$^+$, 66). HRMS (APCI), m/z found: 888.0449 (calcd. for $C_{30}H_{33}F_9O_9S_3Si_3$: 888.0445).

1.3. *Reactivity of the 2,6,10-triphenylenotriyne precursors 2*

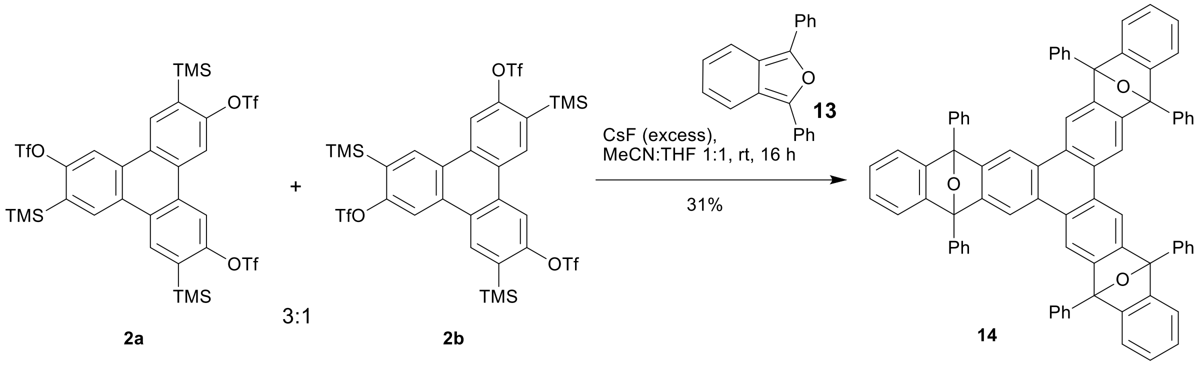
Synthesis of 1,4,7,10,13,16-hexahydro-1,4:7,10:13,16-triepoxytrinaphthylene (12)



Tristriflates **2** (40 mg, 0.045 mmol) and CsF (123 mg, 0.809 mmol) were placed in a Schlenk tube under Ar atmosphere. Then, THF (1 mL), furan (98 µL, 1.349 mmol) and MeCN (1 mL) were subsequently added. The reaction mixture was stirred for 16 h at rt. Then, volatiles were removed under reduced pressure and the crude reaction product was purified by flash chromatography (SiO_2, EtOAc) to give the trisadduct **12** (16 mg, 84% yield) as a mixture of diastereomers.

12: Brown solid, m.p.: gradual decomposition. ^{1}H NMR (750 MHz, $CDCl_3$), δ: 8.47 – 8.40 (m, 6H), 7.08 – 7.03 (m, 6H), 5.92 (s, 6H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$), δ: 146.5 (C), 142.6 (CH), 128.0 (C), 115.0 (CH), 82.5 (CH) ppm. MS (APCI), m/z: 427 (M^++1, 100). HRMS (APCI), m/z found: 427.1329 (calcd. for $C_{30}H_{19}O_3$: 427.1329).

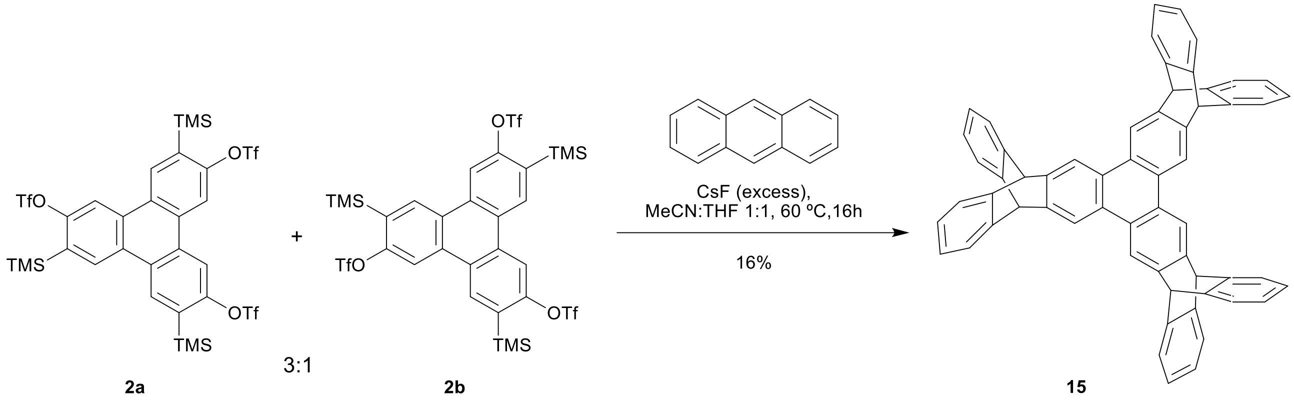
Synthesis of 5,8,13,16,21,24-hexaphenyl-5,8,13,16,21,24-hexahydro-5,24:8,13:16,21-triepoxyanthra[2,3-*j*]heptaphene (14)



Tristriflates **2** (50 mg, 0.057 mmol), 1,3-diphenylisobenzofuran (55 mg, 204 mmol, 360 mol%) and CsF (154 mg, 1.013 mmol, 1800 mol%) were placed in a Schlenk tube under Ar atmosphere. Then, THF (2 mL) and MeCN (2 mL) were subsequently added. The reaction mixture was stirred for 16 h at rt. Then, volatiles were removed under reduced pressure and the crude reaction product was purified by flash chromatography (SiO_2, gradient from Hex to DCM) isolating to give compound **14** (18 mg, 31% yield) as a mixture of diastereomers.

14: White solid, m.p.: 214 ˚C decomposition. ^1H NMR (300 MHz, $CDCl_3$), δ: 8.43 (m, 6H), 8.07 (m, 12H), 7.77 – 7.55 (m, 18H), 7.41 (m, 6H), 7.05 (m, 6H) ppm. ^{13}C NMR-DEPT (75 MHz, $CDCl_3$), δ: 150.0 (C), 149.9 (C), 149.8 (C), 149.7 (C), 148.5 (C), 148.3 (C), 135.3 (C), 135.3 (C), 135.0 (C), 134.9 (C), 128.9 (CH), 128.7 (CH), 128.6 (CH), 128.2 (CH), 128.1 (CH), 128.1 (CH), 127.2 (CH), 127.1 (CH), 126.9 (CH), 126.8 (CH), 126.3 (CH), 126.3 (CH), 120.6 (CH), 120.6 (CH), 120.5 (CH), 115.3 (C), 115.2 (C), 115.0 (C), 90.8 (C), 90.6 (C) ppm. MS (APCI), m/z: 1033 (M^++1, 55), 839 (M^+-194, 100). HRMS (APCI), m/z found: 1033.3679 (calcd. for $C_{78}H_{49}O_3$: 1033.3676).

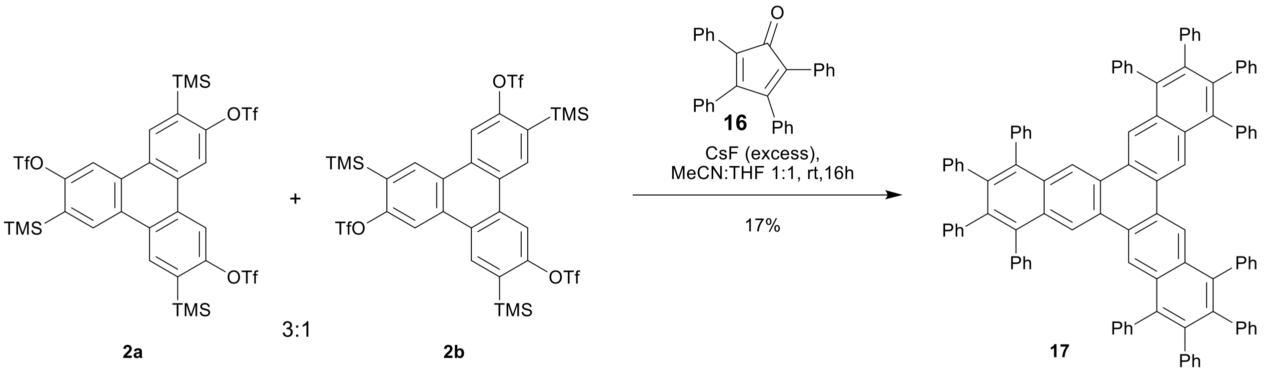
Synthesis of 5,8,13,16,21,24-hexahydro-5,24:8,13:16,21-tris([1,2]benzeno)anthra[2,3-*j*]heptaphene (15)



Tristriflates **2** (50 mg, 0.057 mmol), anthracene (36 mg, 0.202 mmol, 360 mol%) and CsF (154 mg, 1800 mol%) were placed in a Schlenk tube under Ar atmosphere. Then, THF (2 mL) and MeCN (2 mL) were subsequently added. The reaction mixture was stirred for 16h at 60 °C. Then, volatiles were removed under reduced pressure and the crude reaction product was purified by flash chromatography (SiO_2, gradient from Hex to Tol), to give **15** as a white solid (7 mg, 16% yield, m.p.: >350 °C). Spectroscopic data were coincident with those described in the literature for this compound.[4]

15: ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.56 (s, 1H), 7.51 – 7.40 (m, 2H), 7.06 – 6.97 (m, 2H), 5.62 (s, 1H) ppm.

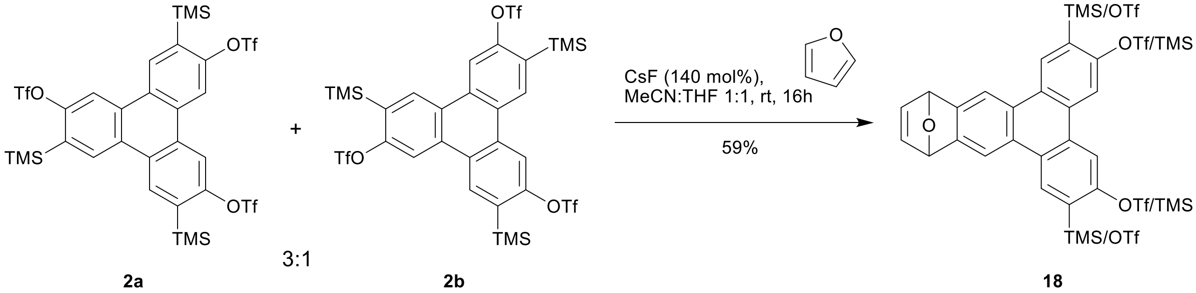
Synthesis of 1,2,3,4,7,8,9,10,13,14,15,16-dodecaphenyltrinaphthylene (17)



Tristriflates **2** (40 mg, 0.045 mmol), tetraphenylcyclopentadienone (**16**, 62 mg, 0,162 mmol, 360 mol%) and CsF (123 mg, 0.778 mmol,1800 mol%) were placed in a Schlenk tube under Ar atmosphere. Then, THF (1 mL) and MeCN (1 mL) were subsequently added. The reaction mixture was stirred for 16 h at rt. Then, volatiles were removed under reduced pressure and the crude reaction product was purified by flash chromatography (SiO_2, gradient from 3:1 DCM:Hex to DCM), isolating **17** as a white solid (10 mg, 17% yield, m.p.: >350 °C). Spectroscopic data were coincident with those described in the literature for this compound.[5]

17: ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.47 (s, 6H), 7.39 – 7.05 (m, 30H), 7.00 – 6.74 (m, 30H) ppm.

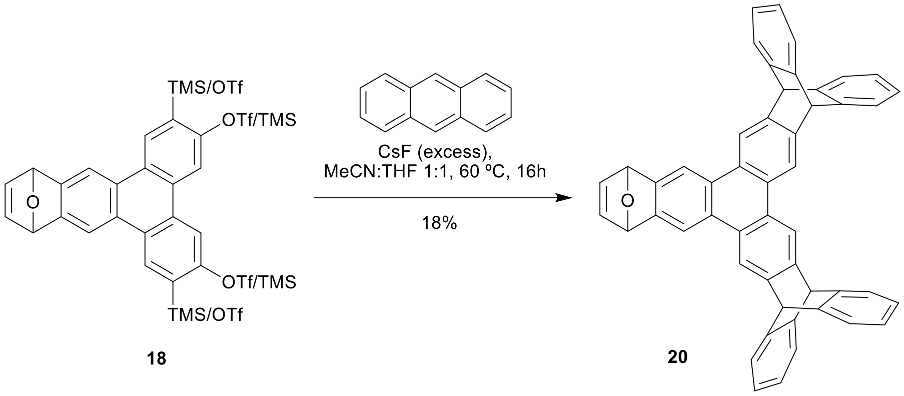
Synthesis of furan monoadducts 18



Tristriflates **2** (100 mg, 0.113 mmol) and CsF (24 mg, 0.158 mmol, 140 mol%) were placed in a Schlenk tube under Ar atmosphere. Then, THF (2 mL), furan (25 µL, 0.345 mmol, 300 mol%) and MeCN (2 mL) were subsequently added. The reaction mixture was stirred for 16 h at rt- Then, volatiles were removed under reduced pressure and the crude reaction product was purified by flash chromatography (SiO_2, gradient from 2:3 DCM:Hex to DCM) to give the furan monoadducts **18** (49 mg, 59% yield).

18: Pale brown solid, m.p.: gradual decomposition. ^{1}H-NMR (300 MHz, $CDCl_3$), 8.89 – 8.19 (singlets), 7.16 – 7.04 (singlets), 5.99 (singlets), 0.57 – 0.52 (singlets). MS (APCI), m/z: 735 (M^++1, 100). HRMS (APCI), m/z found: 735.0789 (calcd. for $C_{30}H_{29}F_6O_7S_2Si_2$: 735.0792).

Synthesis of 5,8,11,14,19,22-hexahydro-5,22:14,19-bis([1,2]benzeno)-8,11-epoxynaphtho[2,3-j]heptaphene (20)



Bistriflates **18** (25 mg, 0.034 mM) and CsF (62mg, 0.4 mmol, 1200 mol%) were placed in a Schlenk tube under Ar atmosphere. Then, THF (0.5 mL), anthracene (18 mg, 0.1 mmol, 300 mol%) and MeCN (0.5 mL) were subsequently added. The reaction mixture was stirred for 16 h at 60 °C. Then, volatiles were removed under reduced pressure and the crude reaction product was purified by column chromatography (SiO_2, 100% DCM) to give the compound **20** (4 mg, 18% yield).

20: White solid, m.p.: gradual decomposition since 220 °C. ^{1}H-NMR (300 MHz, $CDCl_3$), 8.62 (s, 2H), 8.50 (s, 2H), 8.41, (s, 2H), 7.45 (m, 8H), 7.03 (m, 10H), 5.88 (s, 2H), 5.64 (s, 2H), 5.61 (s, 2H) ppm. MS (APCI), m/z: 646.2(M^++1, 100). HRMS (APCI), m/z found: 646.2290 (calcd. for $C_{50}H_{30}O$: 646.2291).

2. X-Ray structure of 20

Cambridge Crystallographic Data Centre (CCDC) 2026785 record contains the supplementary crystallographic data for compound **20**. These data can be obtained free of charge from http://www.ccdc.cam.ac.uk

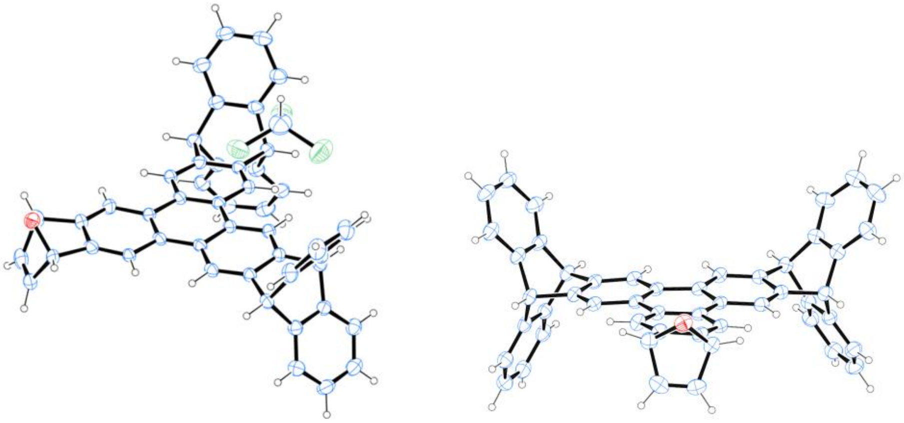


Figure S1

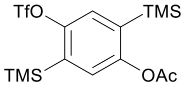
Summary of Data CCDC: 2026785

Formula: $C_{50}H_{30}O$

Unit cell parameters: a 15.3787(5) b 16.0584(6) c 16.3664(6)

Space group: P-1

3. NMR spectra



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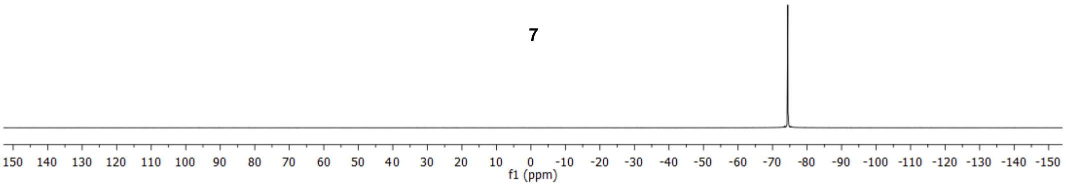
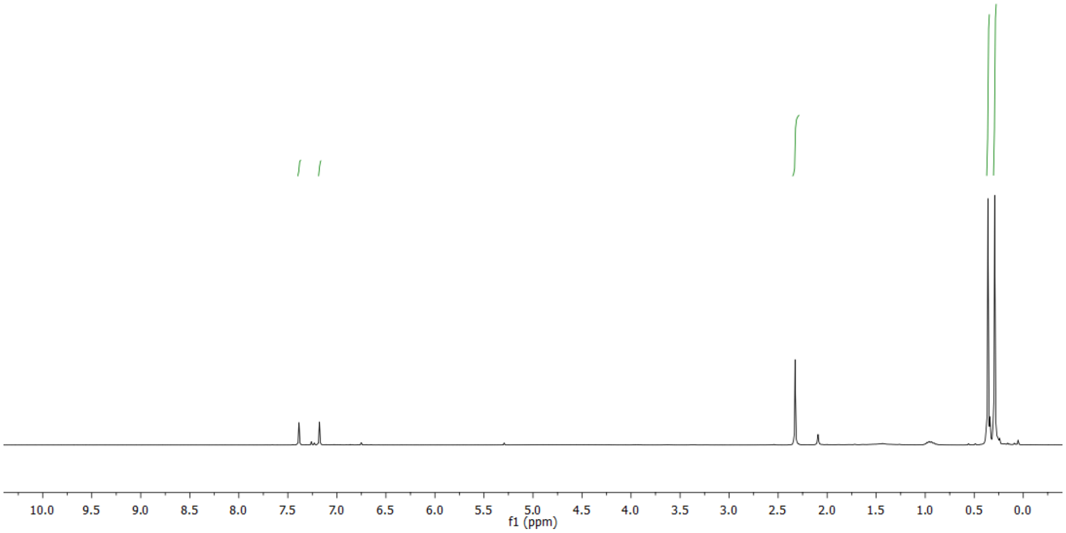
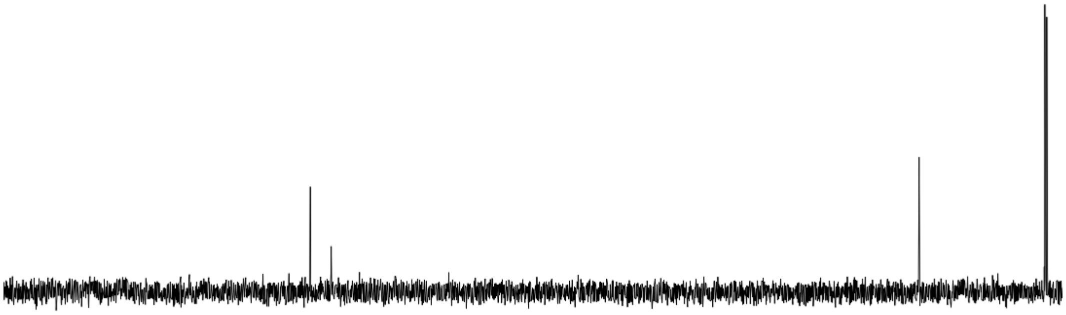
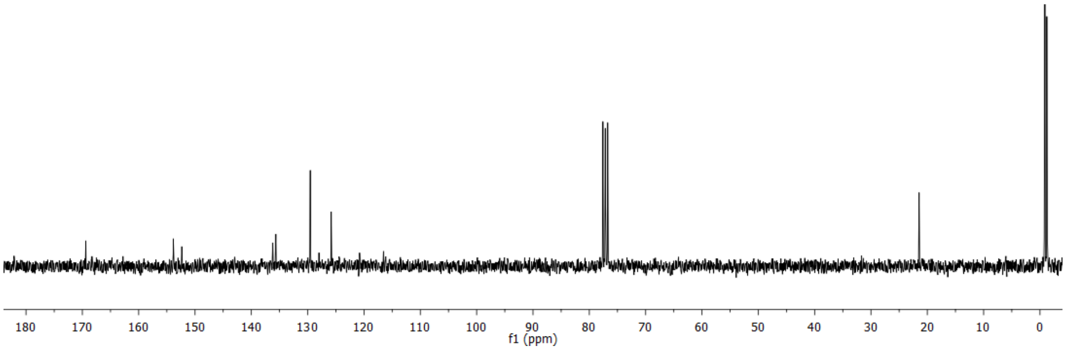
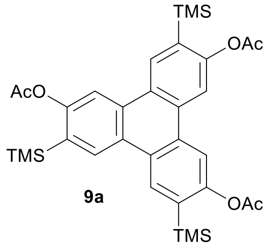


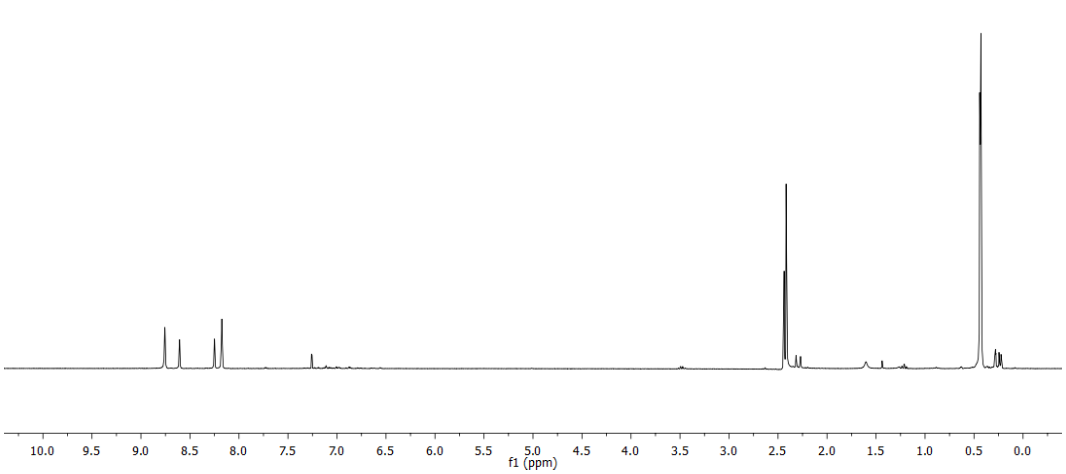


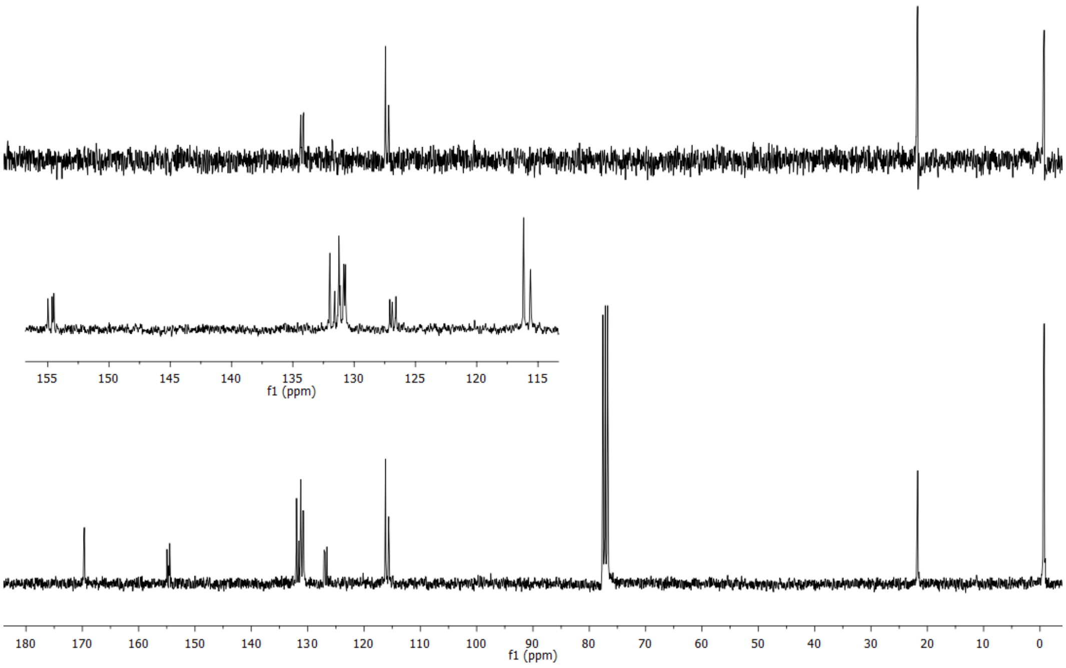


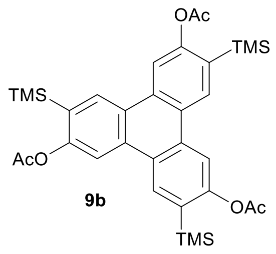



TMS
OAc
AcO
TMS
OAc
TMS

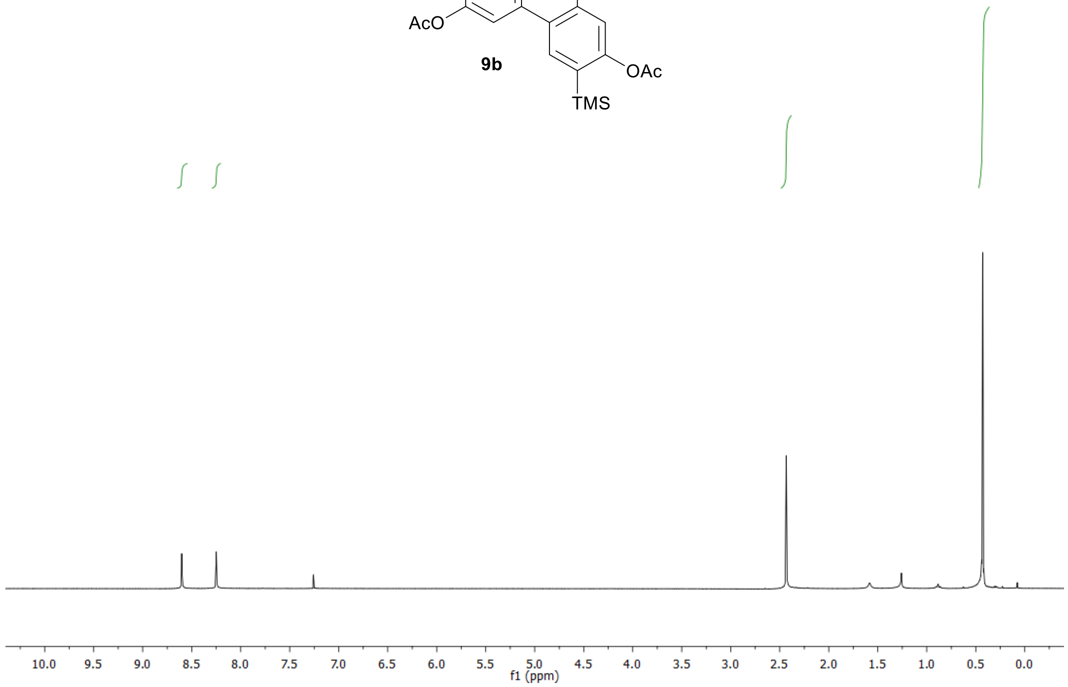
9a

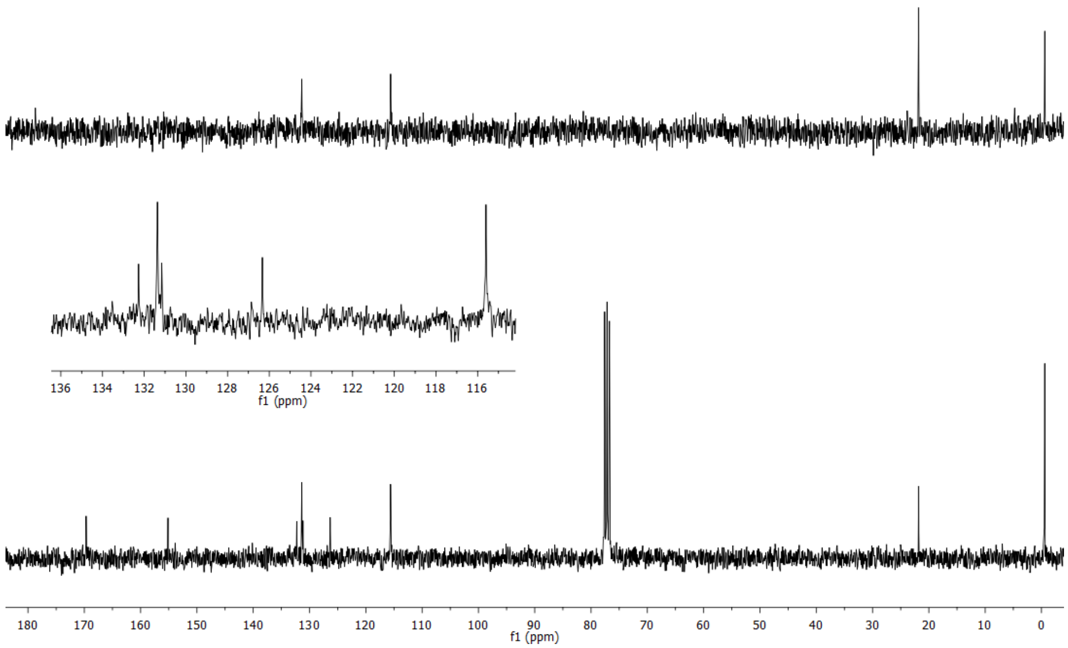

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

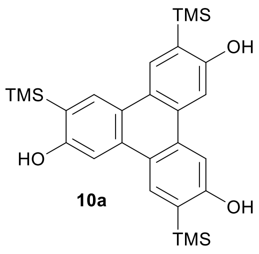

155
150
145
140
135
130
125
120
115
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OAc
TMS
TMS
AcO
OAc
TMS

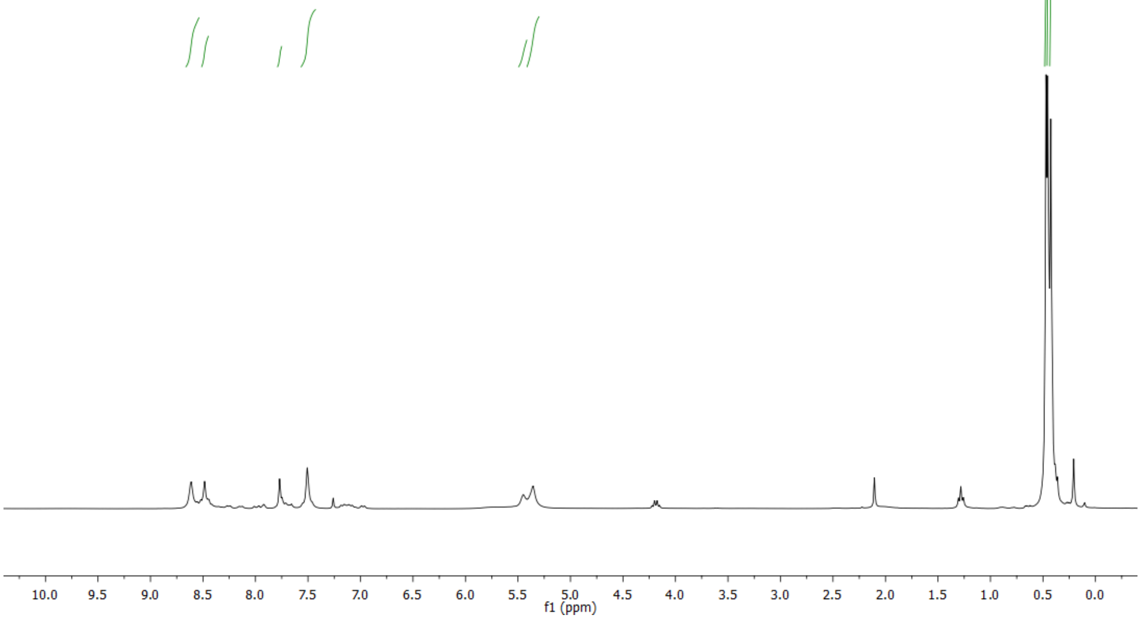
9b

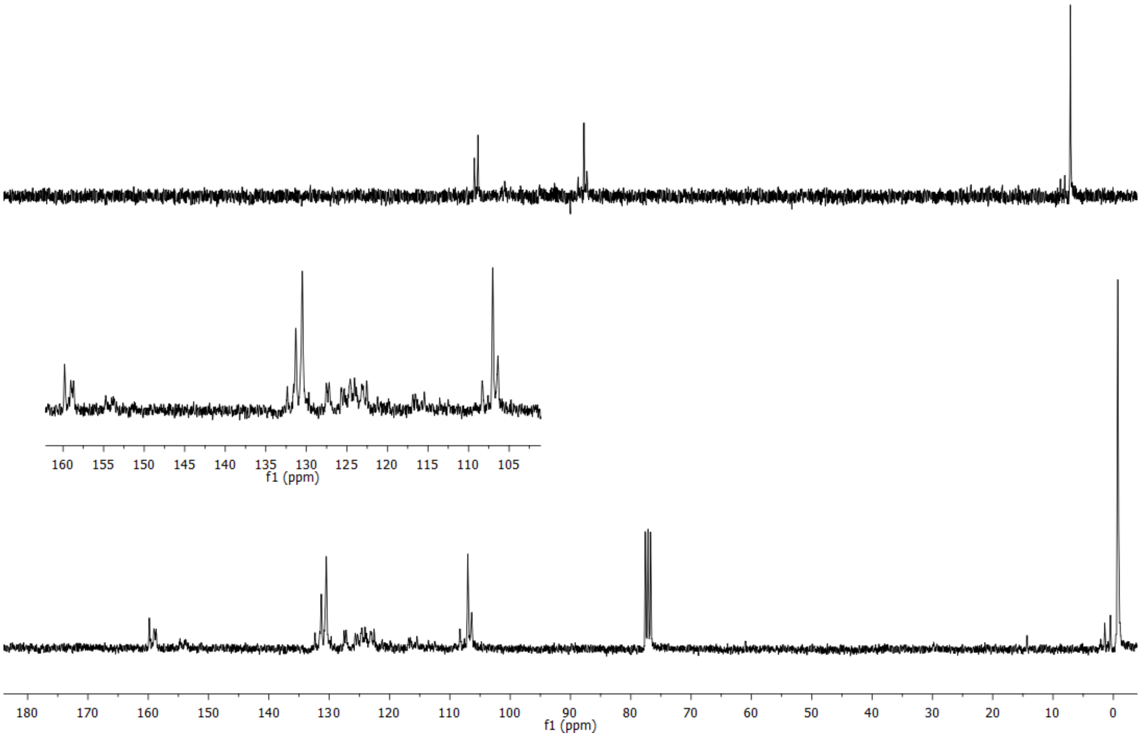

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

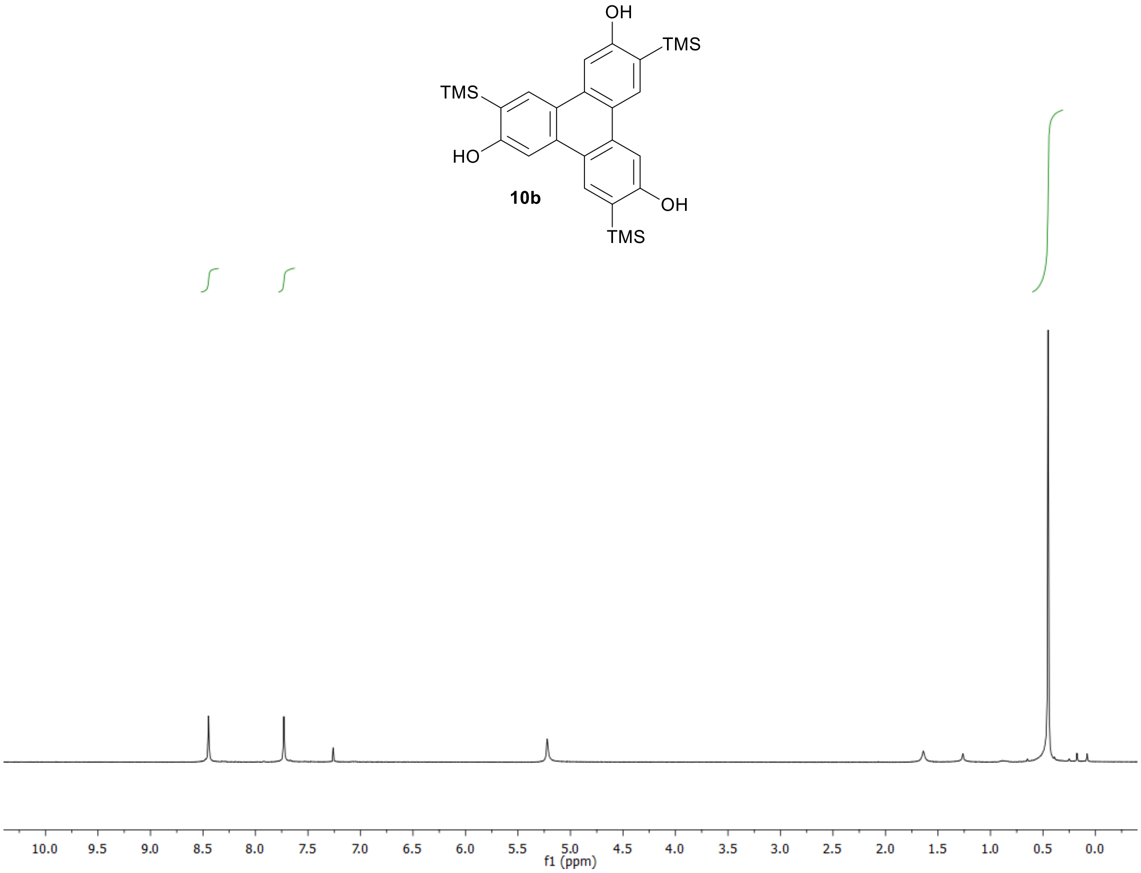

136
134
132
130
128
126
124
122
120
118
116
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


TMS
OH
TMS
HO
OH
TMS

10a


10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

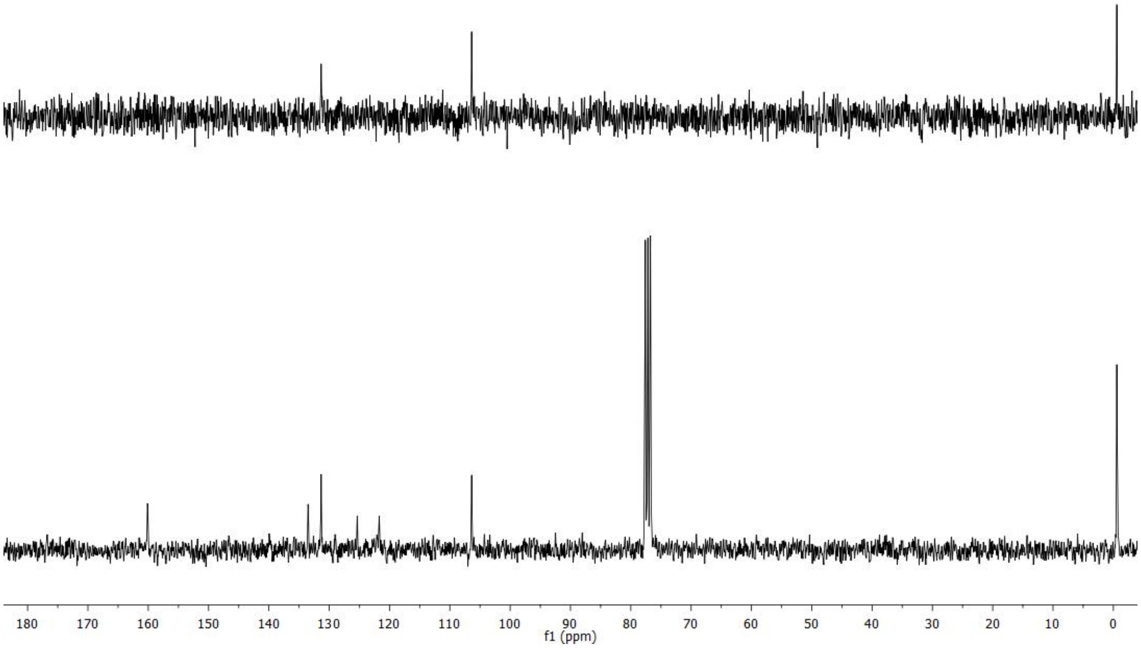

160
155
150
145
140
135
130
125
120
115
110
105
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



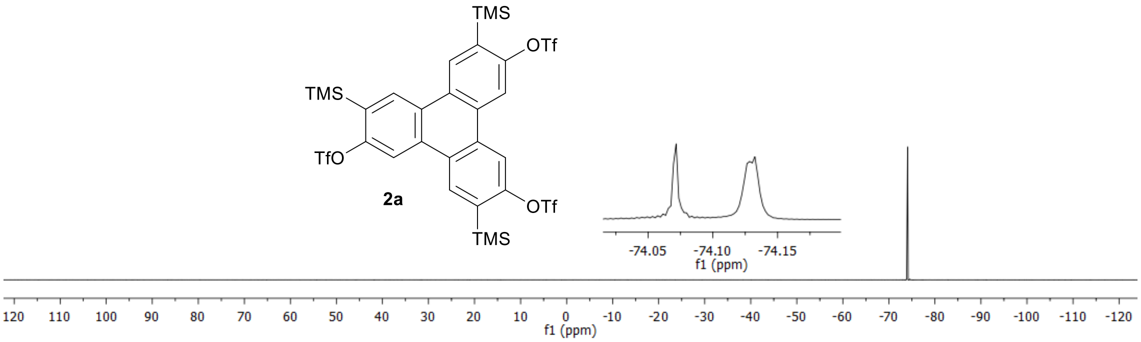
10b

OH
TMS
TMS
HO
OH
TMS

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

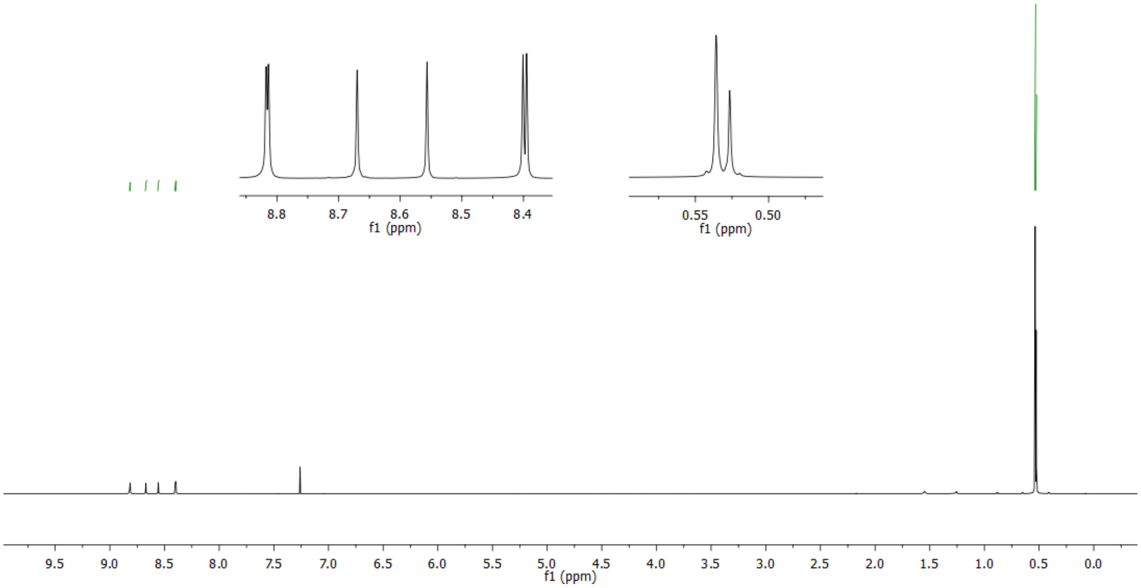


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

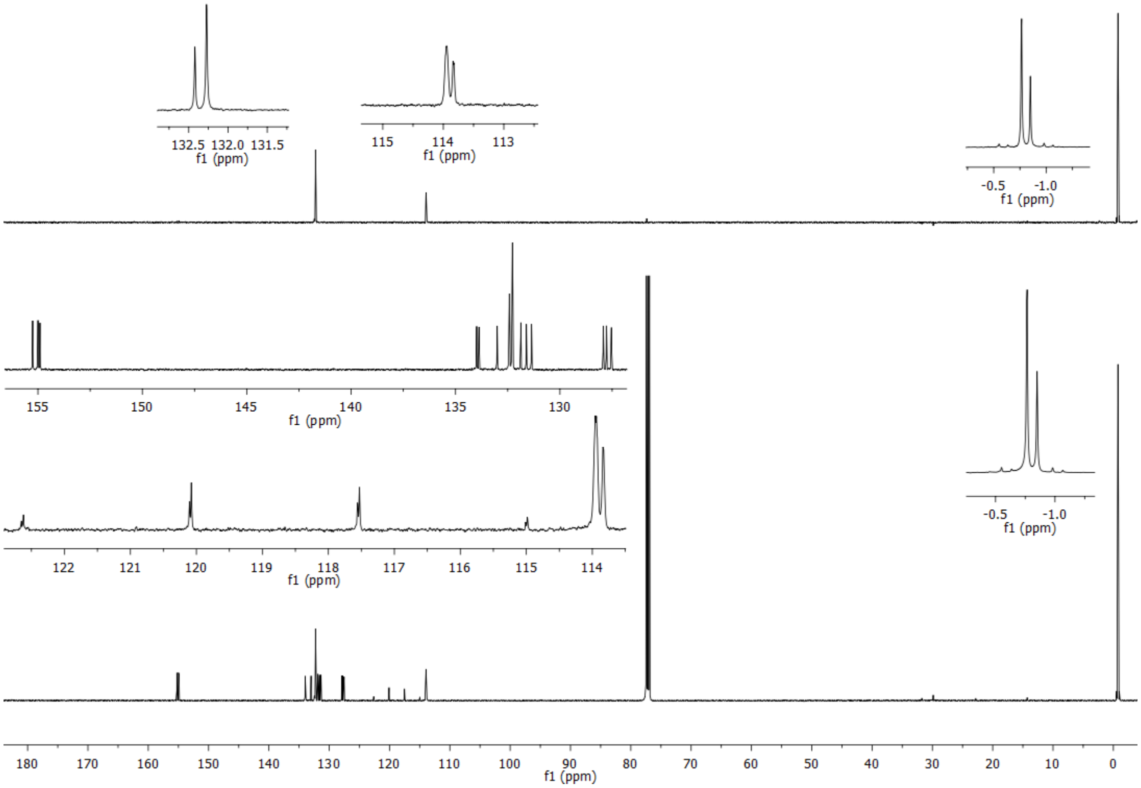


2a

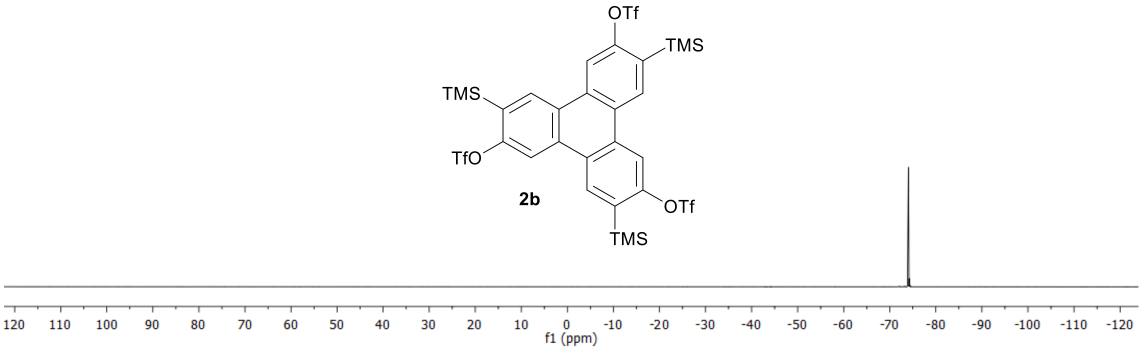
TMS
OTf
TMS
TfO
OTf
TMS
-74.05
-74.10
-74.15
f1 (ppm)
120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120
f1 (ppm)



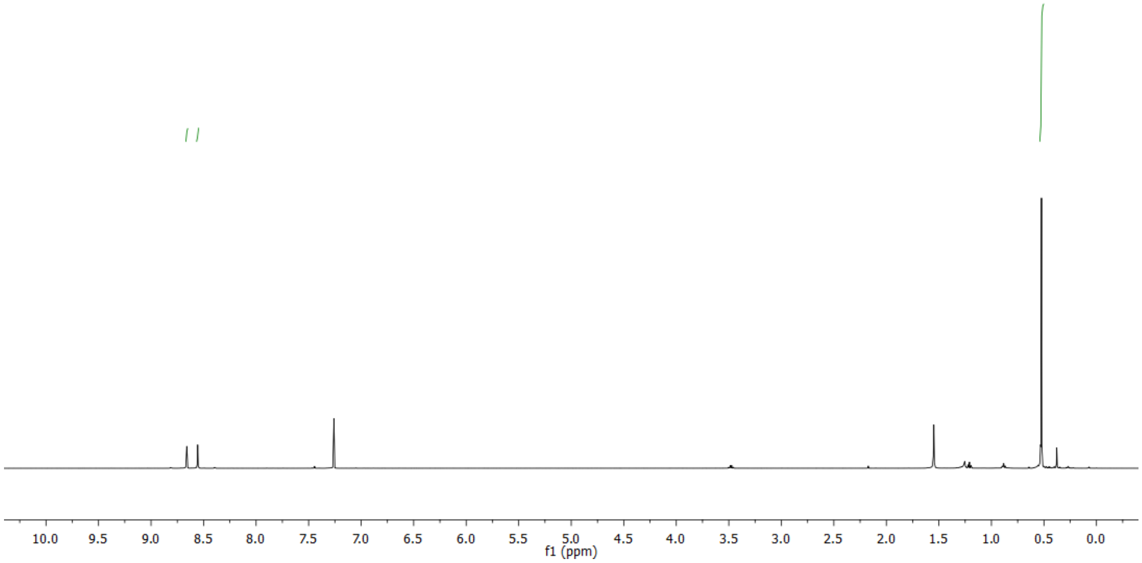
8.8 8.7 8.6 8.5 8.4
f1 (ppm)
0.55 0.50
f1 (ppm)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

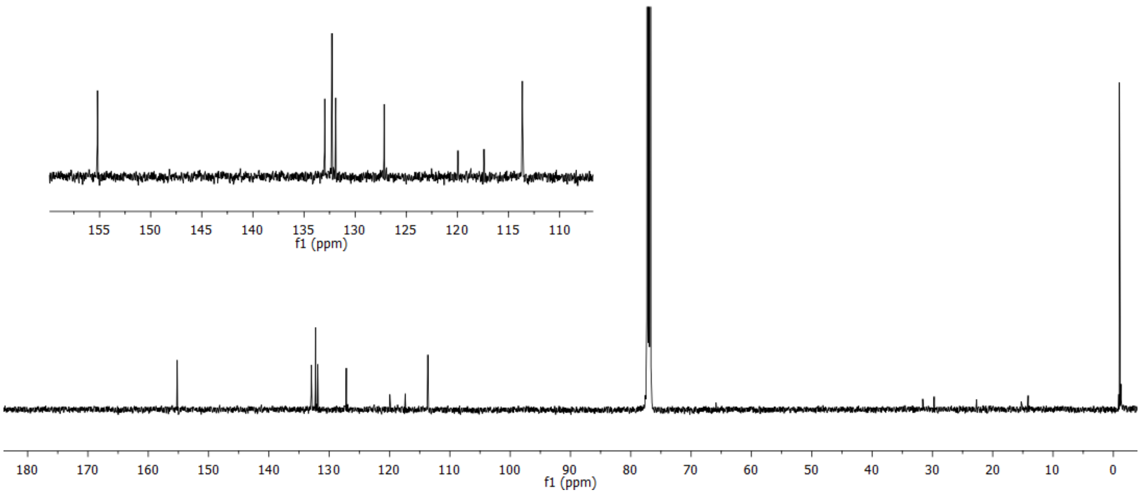


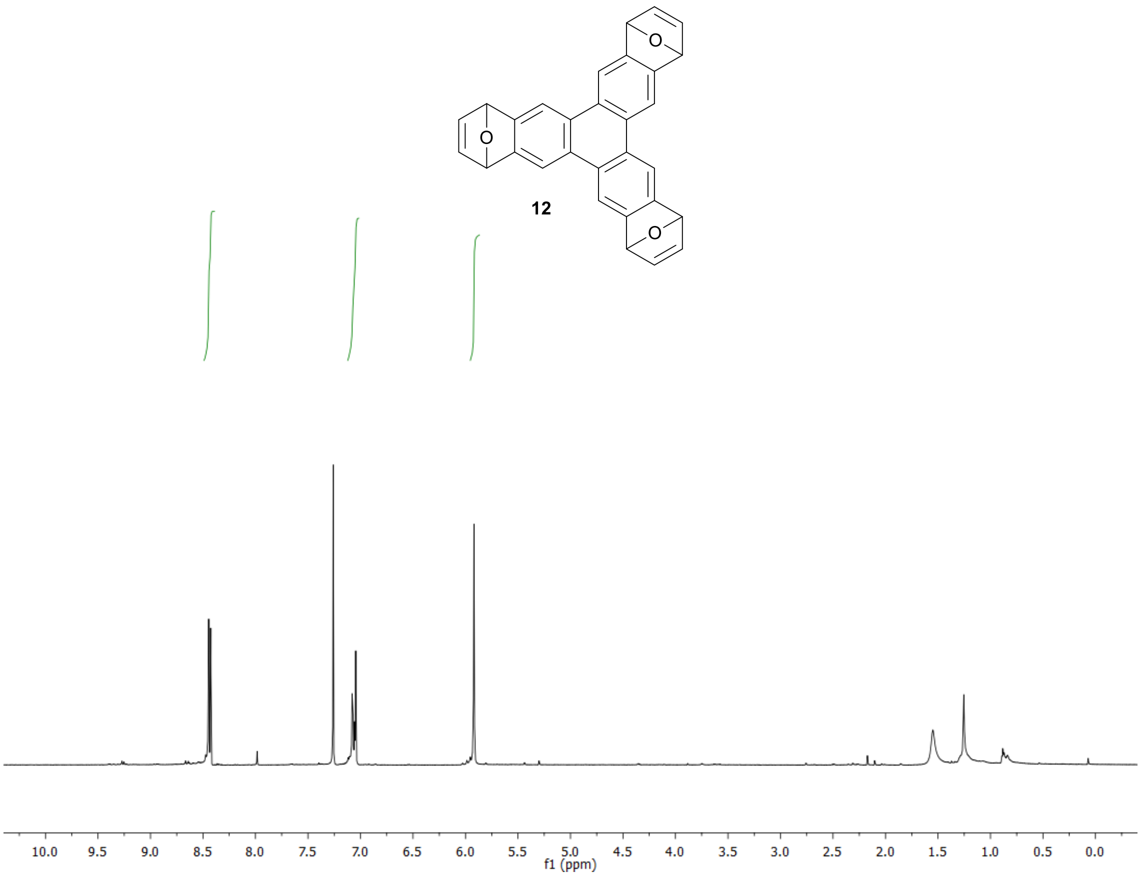
132.5 132.0 131.5
f1 (ppm)
115 114 113
f1 (ppm)
-0.5 -1.0
f1 (ppm)
155 150 145 140 135 130
f1 (ppm)
122 121 120 119 118 117 116 115 114
f1 (ppm)
-0.5 -1.0
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


OTf
TMS
TMS
TfO
OTf
TMS
120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120
f1 (ppm)

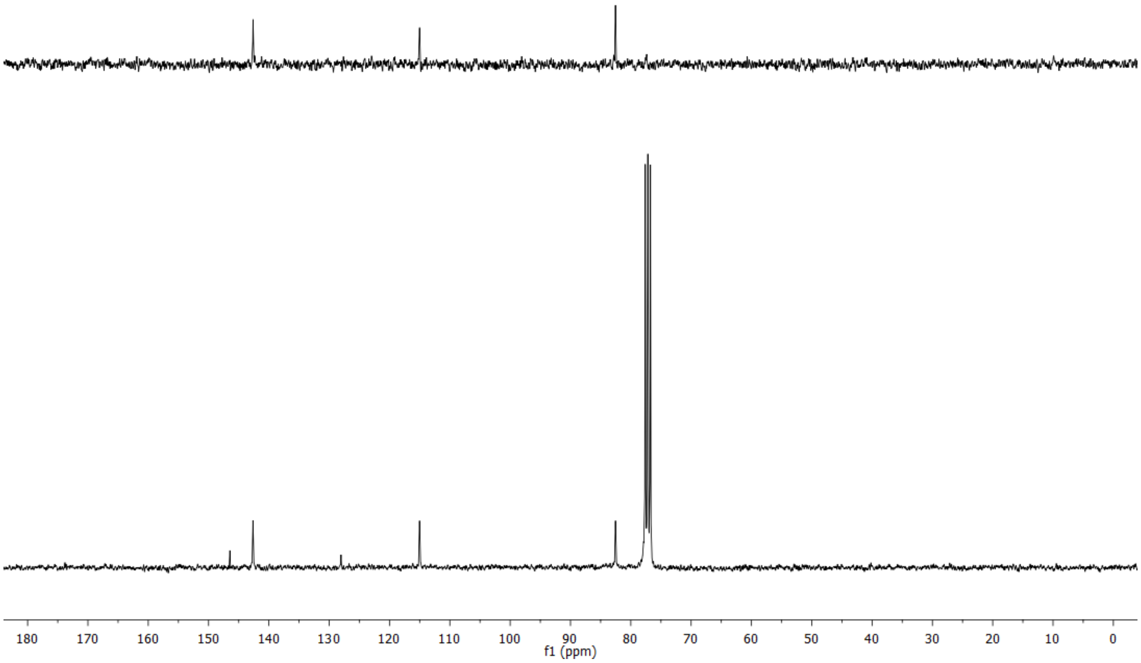
2b

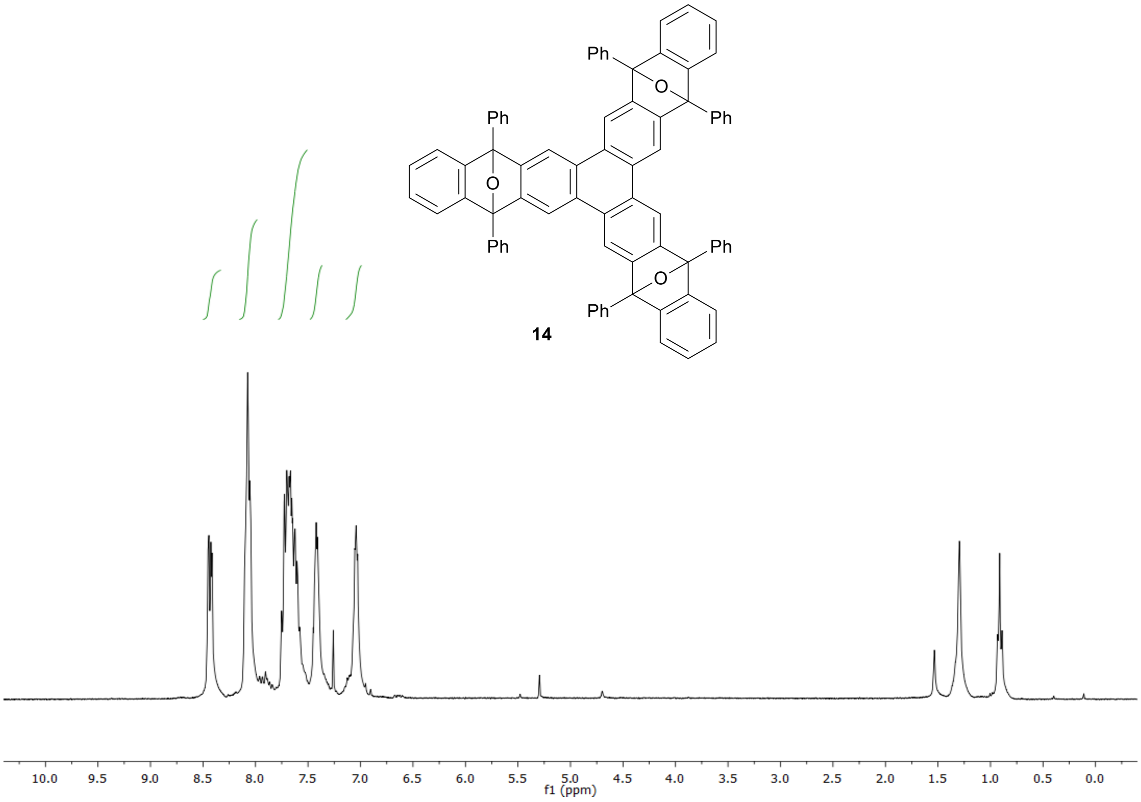

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

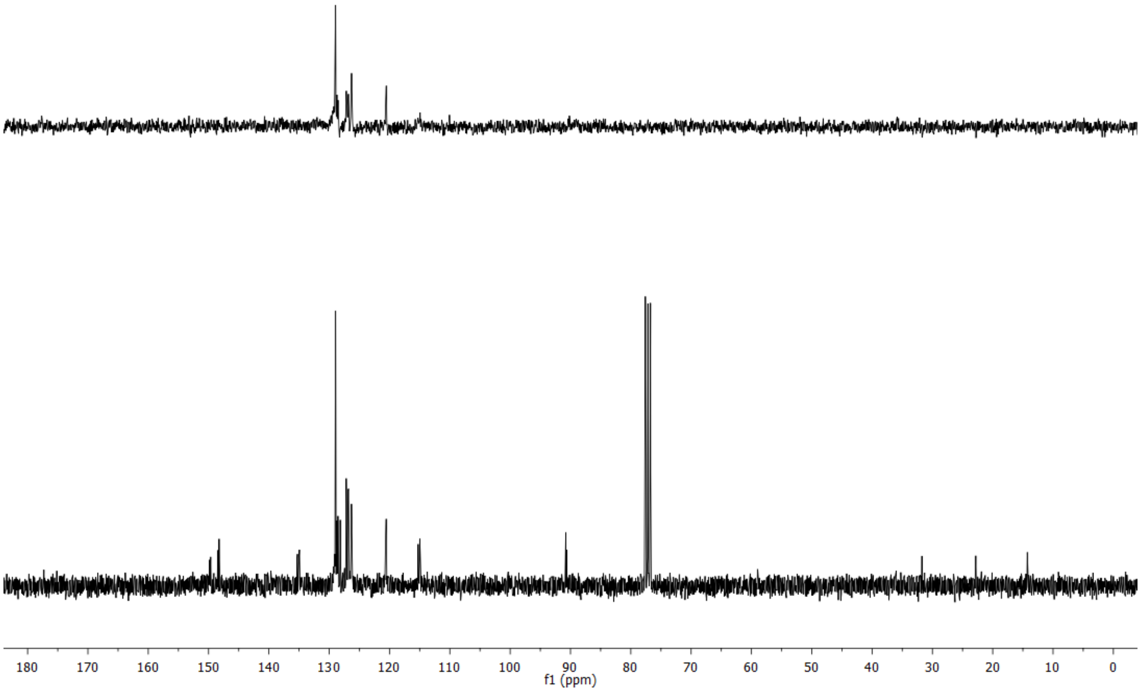

155 150 145 140 135 130 125 120 115 110
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

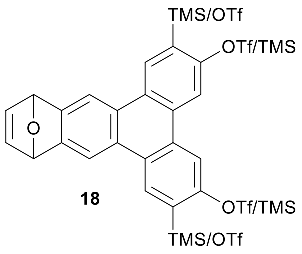

12

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

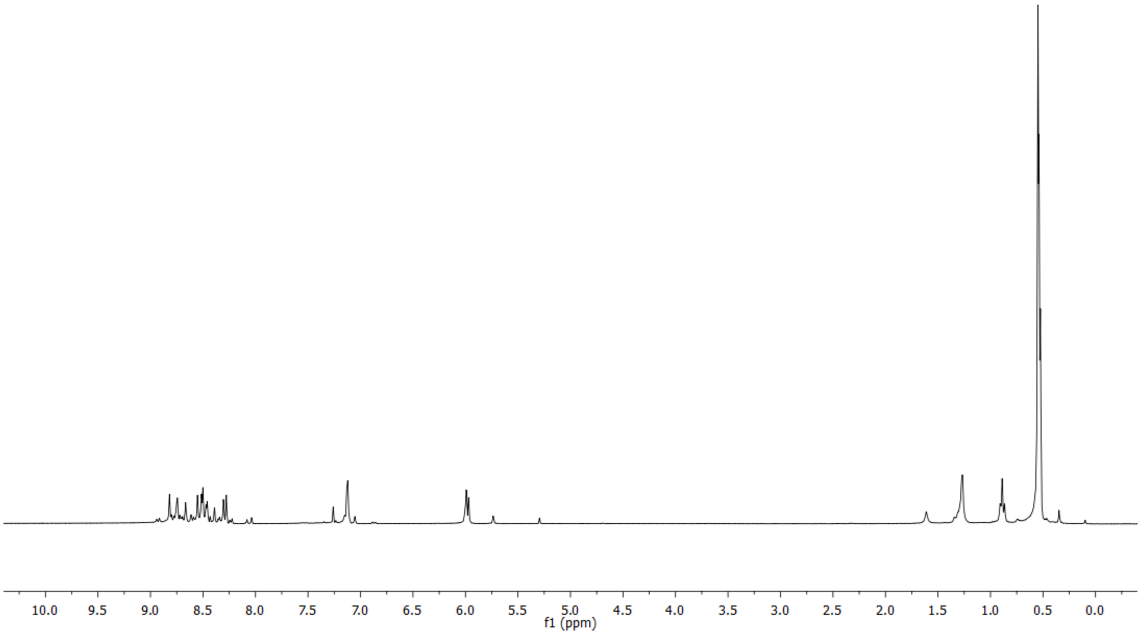

14
Ph
Ph
O
Ph
Ph
O
Ph
O
Ph
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

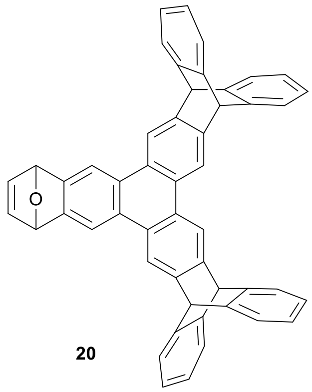


TMS/OTf
OTf/TMS
O
OTf/TMS
TMS/OTf

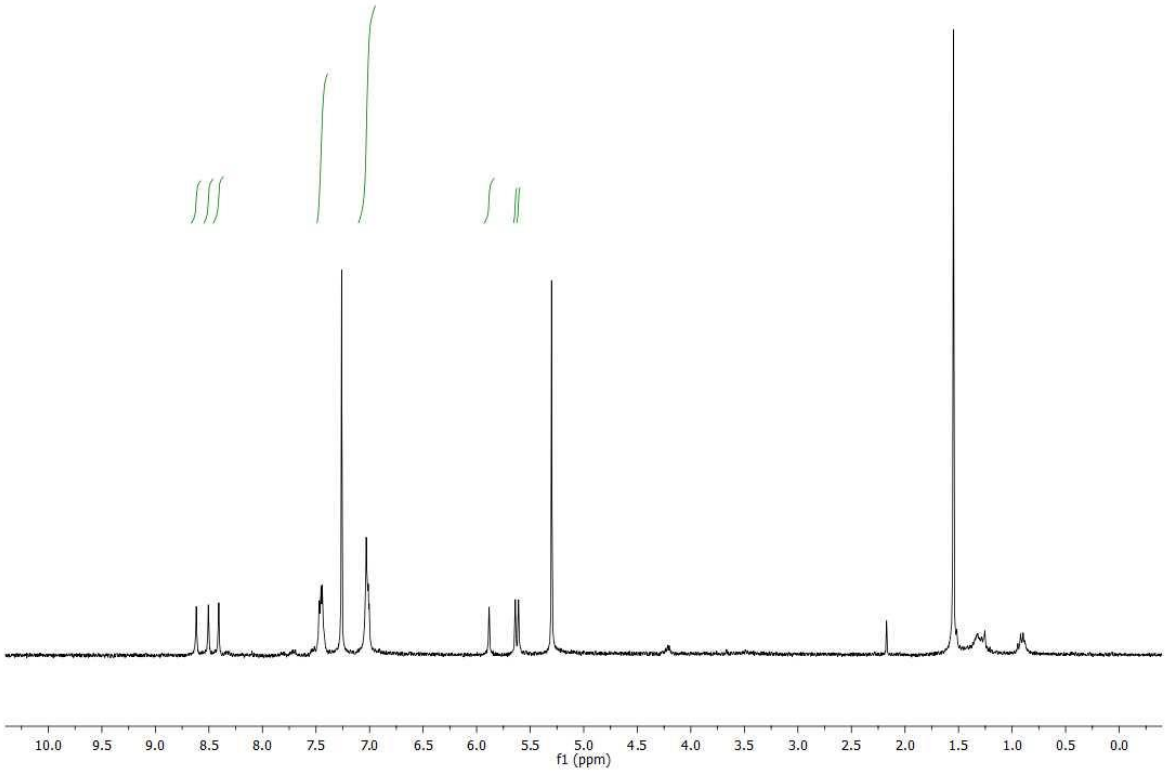
18



10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



20



4. References

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