Supporting Information for

Synthesis and Crystal Structure of Sulphur-Bridged Molecular Hoop Consisting of 5,7,12,14-Tetrathiapentacene

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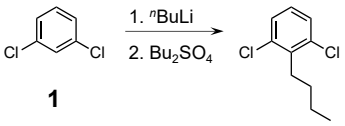
1. Experimental Procedures

General Information

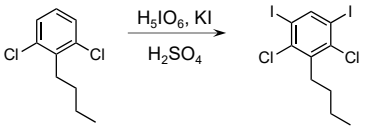
All reagents were commercially sourced. Melting points were determined using a Yanaco MP-500P micro melting point apparatus. ^{1}H (600 MHz) and ^{13}C NMR (150 MHz) spectra were reported on a Bruker AVANCE 600 instrument. The following abbreviations were used to explain the multiplicities: singlet (s); doublet (d); triplet (t); double doublet (dd); multiplet (m). Absorption spectra were recorded on a JASCO V-560 instrument. IR spectra of KBr-pressed pellet samples were measured employing a JASCO FT/IR Spectrometer. MS spectra were determined on a Thermo Scientific, Exactive Plus Orbitrap Mass spectrometer for ionization. Only relatively intense peak or structurally diagnostic mass spectral fragment ion peaks were reported. The diffraction data for TC[2]TTP, **6′**, and **6″** at 120 or 100 K were collected using a Rigaku XtaLAB Synergy-S system with multilayer mirror-monochromatized Cu*K*a radiation (λ = 1.54184 Å) at Kitasato University. Raw frame data were integrated using CrysAlisPro.[1] The crystal structures were solved employing SHELXT[2] and refined using full-matrix least squares in *F*2 (SHELXL).[3] Theoretical calculation was performed at the Research Center for Computational Science at Okazaki using the Gaussian 16 program (Revision C. 01).[4] Cyclic voltammetry was recorded on a HOKUTO DENKO HZ-5000 automatic polarization system. The CV cell consisted of Pt working electrode, Pt wire counter electrode, and an $Ag/AgNO_3$ reference electrode. The measurement was carried out in CH_2Cl_2 and benzonitrile solution of sample with a concentrate 0.1 M *n*-$Bu_4N{\cdot}PF_6$ as a supporting electrolyte. All redox potentials were measured against Ag/Ag^+ and converted to vs. Fc/Fc^+.



1 **2**

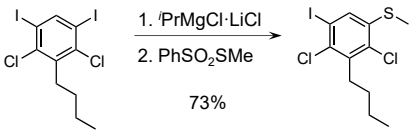
Synthesis of 2-butyl-1,3-dichlorobenzene (2): To a 200 mL 3-necked recovery flask was added 1,3-dichlorobenzene (3.9 mL, 34 mmol) and THF (46 mL), and the mixture was cooled at −78 °C. A 1.6 M hexane solution of *n*-BuLi (21 mL, 34 mmol)was slowly added and stirred at −78 °C for 2 h. The THF (37 mL) solution of dibutyl sulfate (7.4 mL, 37 mmol) was added to the resultant colorless suspension of the lithium salt. The reaction mixture was stirred for 12 h and was then quenched with H_2O, extracted with hexane and dried over $MgSO_4$. The organic solution was evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using hexane as eluent to give 2-butyl-1,3-dichlorobenzene (4.2 g, 21 mmol) as a colorless oil in 61% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 (d, *J* = 7.8 Hz, 2H), 7.02 (t, *J* = 8.4 Hz, 1H), 2.90 (t *J* = 8.4 Hz, 2H), 1.58-1.54 (m, 2H), 1.47-1.41 (m, 2H), 0.96 (t, *J* = 7.8 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.9, 135.4,

128.2, 127.4, 31.2, 30.5, 22.9, 14.0. HRMS (ESI, positive mode): *m/z* calcd for $C_{10}H_{12}Cl_2$: [M] 202.0311; found 202.0311. Analytical data was found to match literature data.[5]



2 **3**

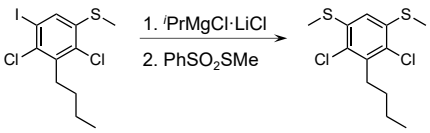
Synthesis of 3-butyl-2,4-Dichloro-1,5-diiodobenzene (3): To a 300 mL recovery flask was added periodic acid (2.1 g, 9.3 mmol) and 20 mL of concentrated sulfuric acid. To the colorless solution was slowly added KI (3.3 g, 28 mmol) at 0 °C, and 2-butyl-1,3-dichlorobenzene (3.8 g, 19 mmol) was quickly added. After stirring at ambient temperature for 12 h, ice was added to the reaction mixture. The generated solid was collected via filtration, and purified by column chromatography on silica gel using hexane as the eluent. After evaporation of the solvent, the residue was recrystallized by MeOH/EtOH at 0 °C to afford 3-butyl-2,4-dichloro-1,5-diiodobenzene (4.6 g, 10 mmol) as a colorless needles in 54% yield. Mp = 67–59 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.23 (s, 1H), 3.10 (t, *J* = 7.8 Hz, 2H), 1.56-1.51 (m, 2H), 1.50-1.41 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.9, 141.3, 139.4, 98.0, 35.7, 29.9, 22.9, 13.9. UV-vis (CH_2Cl_2, $c = 1.7\times10^{-5}$ M) λ_{max} (ε) 255 (6831, sh), 248 (14894, sh), 236 (25816) nm, IR (KBr) 2949, 2924, 2866, 1465, 1453, 1390, 1343, 1192, 1178, 1103, 1079, 929, 865, 727, 581 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{10}H_{10}Cl_2I_2$ [M] 453.8243; found 453.8246.



3 **4**

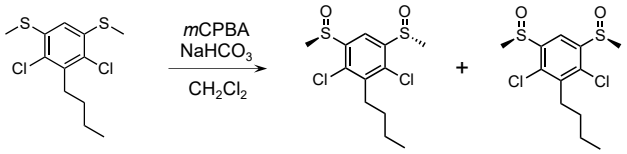
Synthesis of (3-butyl-2,4-dichloro-5-iodophenyl)(methyl)sulphane (4): To a 100 mL second necked recovery flask was added 3-butyl-2,4-dichloro-1,5-diiodobenzene (2.4 g, 5.3 mmol) and dry THF (10 mL) under argon atmosphere, and the mixture was cooled to −78 °C. The THF solution of *i*-PrMgCl·LiCl (1.3 M, 4.9 mL, 6.3 mmol) was slowly added and stirred for 24 h. The prepared magnesium reagent was added to a solution of prepared *S*-methylbenzenesulphonothioate (1.1 g, 5.8 mmol)[6] in dry THF at −78 °C. The resulting solution was stirred at ambient temperature for 2 h and was then quenched with sat. NH_4Claq., extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4. The organic solution was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel using hexane as the eluent to afford (3-butyl-2,4-dichloro-5-iodophenyl)(methyl)sulphane (1.5 g, 3.9 mmol) as a colorless solid in 73% yield. Mp = 44–46 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 (s, 1H), 3.01 (t, *J* = 7.8 Hz, 2H), 2.44 (s, 3H), 1.55-1.49 (m, 2H),

1.47-1.41 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 140.3, 139.1, 134.9, 132.7, 132.3, 97.7, 33.8, 30.1, 22.9, 15.7, 14.0. UV-vis (CH_2Cl_2, c = 1.0×10^{-5} M) λ_{max} (ε) 314 (2529, sh), 303 (3182, sh), 266 (14561), 238 (29623) nm, IR (KBr) 2952, 2924, 2871, 2857, 1543, 1525, 1464, 1455, 1433, 1417, 1395, 1351, 1323, 1252, 1186, 1104, 1071, 930, 911, 846, 733, 613 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{11}H_{13}Cl_2IS$ [M] 373.9154; found 373.9154.



4 **5**

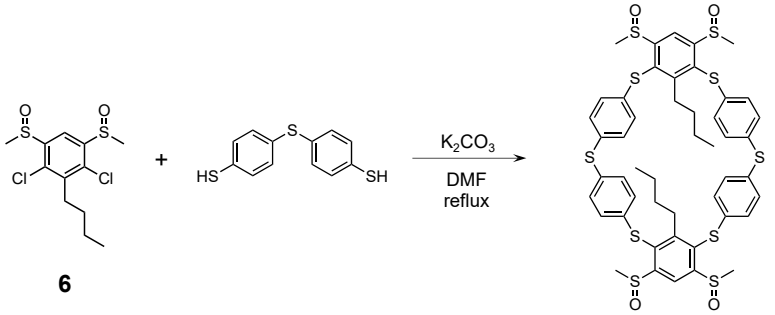
Synthesis of (5-butyl-4,6-dichloro-1,3-phenylene)bis(methylsulphane) (5): To a 100 mL second necked recovery flask was added (3-butyl-2,4-dichloro-5-iodophenyl)(methyl)sulfane (1.4g, 3.8 mmol) and THF (15 mL) under argon atmosphere, and the mixture was cooled to −78 °C. The THF solution of *i*-PrMgCl·LiCl (1.3 M, 3.6 mL, 4.6 mmol) was slowly added and stirred for 48 h. The prepared magnesium reagent was added to a solution of prepared *S*-methylbenzenesulphonothioate (0.8 g, 4.2 mmol) in dry THF at −78 °C. The resulting mixture was stirred at ambient temperature for 2 h and was then quenched with sat. NH_4Claq., extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4. The organic solution was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel using hexane as the eluent to afford (5-butyl-4,6-dichloro-1,3-phenylene)bis(methylsulphane) (0.68 g, 2.3 mmol) as a colorless needles in 60% yield. Mp = 80–82 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.79 (s, 1H), 2.94 (t, J = 7.8 Hz, 2H), 2.48 (s, 6H), 1.56-1.51 (m, 2H), 1.47-1.41 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.6, 137.3, 128.9, 119.6, 32.1, 30.2, 23.0, 15.9, 14.0. UV-vis (CH_2Cl_2, c = 1.4×10^{-5} M) λ_{max} (ε) 318 (563), 305 (300), 273 (8125, sh), 265 (10641, sh), 248 (18996) nm, IR (KBr) 2959, 2919, 2869, 2857, 1537, 1465, 1456, 1441, 1421, 1400, 1376, 1358, 1205, 1192, 1104, 1077, 963, 926, 919, 823, 730, 647, 475 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{12}H_{16}Cl_2S_2$ [M] 294.0065; found 294.0066.



5 **6'** **6"**

Synthesis of 3-butyl-2,4-dichloro-1,5-bis(methylsulphinyl)benzene (6): To a 100 mL recovery flask was added (5-butyl-4,6-dichloro-1,3-pheylene)bis(methylsulfane) (500 mg, 1.69 mmol), *m*CPBA (835 mg, 3.39 mmol), $NaHCO_3$ (285 mg, 3.39 mmol), and CH_2Cl_2 (20 mL). The mixture was stirred at 0 °C for 2 h. Then, $Na_2S_2O_3$aq. was poured into the flask and extracted with CH_2Cl_2 and

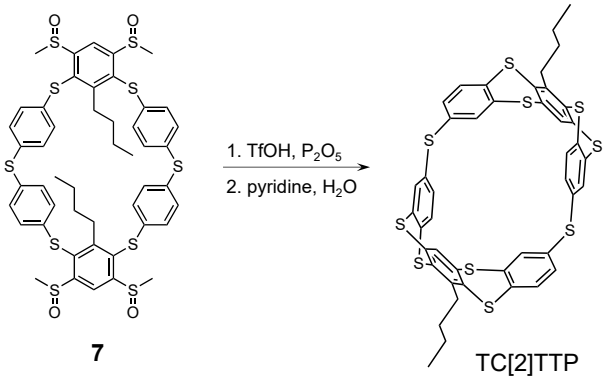
dried over anhydrous $MgSO_4$. After filtration, the organic solution was evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using EtOAc as the eluent to give 3-butyl-2,4-dichloro-1,5-bis(methylsufinyl)benzene (503 mg, 1.54 mmol) as a diastereomeric mixture **6′** and **6″** of colorless needles in 91% yield (isolated yield: **6′** = 45%; **6″** = 46%, respectively). The absolute configuration was determined by single crystal X-ray diffraction analysis. **6′**: ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.56 (s, 1H), 2.98 (t, J = 8.4 Hz, 2H), 2.85 (s, 6H), 1.58-1.53 (m, 2H), 1.51-1.45 (m, 2H), 0.99 (t, J = 7.2 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.0, 141.1, 132.8, 120.5, 41.6, 30.5, 30.2, 22.9, 13.9; **6″**: ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.46 (s, 1H), 2.98 (t, J = 8.4 Hz, 2H), 2.87 (s, 6H), 1.59-1.53 (m, 2H), 1.51-1.45 (m, 2H), 0.99 (t, J = 7.2 Hz, 3H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 145.8, 141.1, 132.7, 120.3, 41.7, 30.5, 30.2, 22.9, 13.9. Mp = 112–115 °C. UV-vis (CH_2Cl_2, c = 1.5×10^{-5} M) λ_{max} (ε) 258 (13783) nm, IR (KBr) 3037, 2956, 2925, 2869, 1717, 1547, 1467, 1455, 1429, 1413, 1386, 1365, 1301, 1284, 1204, 1140, 1065, 946, 930, 911, 841, 681, 448 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{12}H_{16}Cl_2O_2S_2$ [MH] 327.0042; found 327.0041.



6 **7**

Synthesis of 7: To a 100 mL Schlenk tube was added 4,4′-thiobisbenzenethiol (230 mg, 0.917 mmol), 3-butyl-2,4-dichloro-1,5-bis(methylsulphinyl)benzene (300 mg, 0.917 mmol), K_2CO_3 (266 mg, 1.93 mmol), and dry DMF (92 mL) under argon atmosphere. The mixture was stirred at 150 °C for 24 h. After cooling to ambient temperature, the mixture was poured into 10% acetic acid (230 mL). The water phase was extracted with CH_2Cl_2, the organic solution was washed by $NaHCO_3$aq. and dried over $MgSO_4$. The organic phase was evaporated under reduced pressure. The residue was purified by recycling gel permeation chromatography and column chromatography on silica gel using EtOAc as the eluent to obtain **7** (127 mg, 0.126 mmol) as a colorless needle consisting of the mixture of structural isomers in 27% yield. These could not be isolated. Mp = 185–187 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.76 and 8.63 (s×2, 2H), 7.20–7.14 (m, 8H), 6.93–6.79 (m, 8H), 2.93–2.2.76 (m, 16H), 1.48–1.25 (m, 8H), 0.90–0.805 (m, 6H).^{13}C NMR (150 MHz, $CDCl_3$) δ 158.1, 155.8, 155.8, 153.9, 149.7, 148.0, 135.4, 135.3, 134.7, 134.1, 134.0, 133.8, 133.7, 133.1, 132.5, 132.3, 132.2, 132.1, 132.1, 131.2, 131.1, 130.7, 127.7, 127.6, 126.9, 126.7, 126.6, 121.0, 120.8, 120.8, 43.3, 43.3, 42.9, 41.4, 33.4, 31.9, 31.5, 31.5, 23.2, 23.1, 13.9, 13.9. UV-vis (CH_2Cl_2, c = 1.0×10^{-5} M) λ_{max} (ε) 335 (8968, sh), 300 (27834,

sh), 275 (49874, sh) nm, IR (KBr) 2955, 2925, 2868, 1637, 1574, 1474, 1389, 1096, 1061, 1010, 956, 812, 495 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{48}H_{48}O_4S_{10}$ [MH] 1009.0832; found 1009.0833.



7 TC[2]TTP

Synthesis of TC[2]TTP: To a 50 mL second-necked recovery flask was added compound **7** (56 mg, 0.055 mmol), P_2O_5 (8.3 mg, 0.055 mmol), and trifluoromethanesulfonic acid (11 mL) under argon atmosphere. The reaction mixture was stirred at 80 °C for 72 h, and was then poured into pyridine/H_2O mixture (55 mL, 1:1 (v/v)), and was refluxed for 24 h. After cooling to ambient temperature, the pyridine was evaporated under reduced pressure. The precipitate was collected via filtration and purified by column chromatography on silica gel using CS_2 as the eluent to give TC[2]TTP (12 mg, 0.014 mmol) in 24% yield. Mp = 282 °C (decomp.). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 1.8, 8.4 Hz, 4H), 7.38 (d, *J* = 8.4 Hz, 4H), 7.14 (s, 2H), 7.06 (d, *J* = 1.8 Hz, 4H),3.16 (t, *J* = 7.8 Hz, 4H), 1.58-1.51 (m, 8H), 1.06 (t, *J* = 7.2 Hz, 6H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 141.1, 137.2, 136.1, 135.4, 134.5, 133.4, 131.9, 128.5, 126.5, 33.4, 32.4, 23.2, 14.3. UV-vis (CH_2Cl_2, $c = 1.3\times10^{-5}$ M) λ_{max} (ε) 277 (95118) nm, IR (KBr) 2952, 2924, 2856, 1560, 1542, 1446, 1392, 1362, 1250, 1119, 1084, 1038, 875, 810, 714, 679, 589, 466, 445 cm^{-1}, HRMS (ESI, positive mode): *m/z* calcd for $C_{44}H_{32}S_{10}$ [M] 879.9706; found 879.9710.

2. Crystal data for data for TC[2]TTP, 6′, and 6″

Table S1. Crystal data for TC[2]TTP**, 6′**, and **6″**

Compound	TC[2]TTP	TC[2]TTP·2CHCl$_3$	TC[2]TTP·2benzene	TC[2]TTP·toluene
Formula	$C_{44}H_{32}S_{10}$	$C_{46}H_{34}Cl_6S_{10}$	$C_{56}H_{44}S_{10}$	$C_{51.27}H_{40.3}S_{10}$
Formula weight	881.29	1120.03	1037.50	976.91
T [K]	120.15	100.15	100.15	100.15
Crystal system	triclinic	triclinic	triclinic	monoclinic
Space group	*P*-1	*P*-1	*P*-1	$P2_1/c$
a [Å]	10.4234(5)	10.1044(4)	9.6067(3)	13.2924(3)
b [Å]	10.8106(5)	11.5761(4)	11.3115(2)	6.26750(10)
c [Å]	11.3323(3)	12.2229(6)	11.6687(2)	27.5289(7)
α [deg]	93.809(3)	116.082(4)	76.325(2)	90
β [deg]	104.307(4)	106.355(4)	78.743(2)	99.973(2)
γ [deg]	118.489(5)	92.129(3)	88.930(2)	90
V [Å^3]	1061.75(9)	1193.85(10)	1207.84(5)	2258.78(9)
Z	1	1	1	2
D_{calc} [g/cm^3]	1.378	1.558	1.426	1.436
μ [mm^{-1}]	5.055	7.647	4.534	4.811
F(000)	456.0	572.0	540.0	1016.0
Crystal size [mm]	0.024×0.067×0.111	0.093×0.107×0.338	0.131×0.164×0.534	0.042×0.058×0.576
Reflections collected	8961	12478	10752	9764
Independent reflection	3961 [R_{int}= 0.0328]	4462 [R_{int} = 0.0251]	4499 [R_{int}= 0.0311]	4234 [R_{int} = 0.0278]
Parameters	245	318	299	302
Goodness-of-fit on F^2	1.041	1.060	1.096	1.062
R1, wR2 [I>2σ(I)]	0.0327, 0.0828	0.0642, 0.1768	0.0380, 0.0933	0.0332, 0.0860
R1, wR2 [all data]	0.0382, 0.0857	0.0655, 0.1782	0.0391, 0.0942	0.0367, 0.0883
ccdc number	2141641	2153527	2153528	2153529

Table S1. Crystal data for TC[2]TTP**, 6′**, and **6′′**

6′	**6′′**
$C_{24}H_{32}Cl_4O_4S_4$	$C_{24}H_{32}Cl_4O_4S_4$
654.53	654.53
100.15	120.15
Monoclinic	triclinic
$P2_1/n$	P-1
9.1703(2)	9.4018(4)
14.5677(3)	9.7068(5)
22.8030(5)	16.7103(9)
90	79.588(4)
99.171(2)	76.006(4)
90	88.447(4)
3007.32(11)	1455.16(13)
4	2
1.446	1.494
6.416	6.629
1360.0	680.0
0.591×0.311×0.011	0.386×0.135×0.07
21774	13636
5998 [R_{int} = 0.0630]	5453 [R_{int} = 0.0568]
331	331
1.054	1.088
0.0695, 0.1934	0.0466, 0.1213
0.0746, 0.2002	0.0487, 0.1237
2141642	2141643

3. ESI-MS spectra

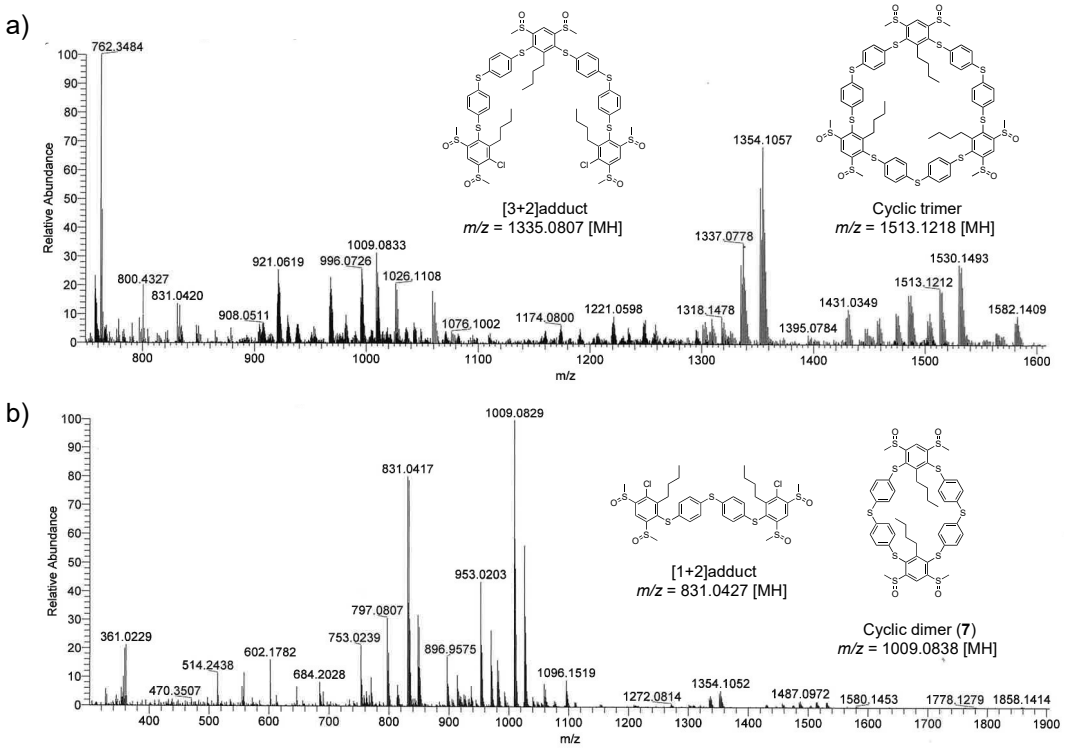


Figure S1. ESI-MS spectra: a) and b) the reaction mixture in macrocyclization.

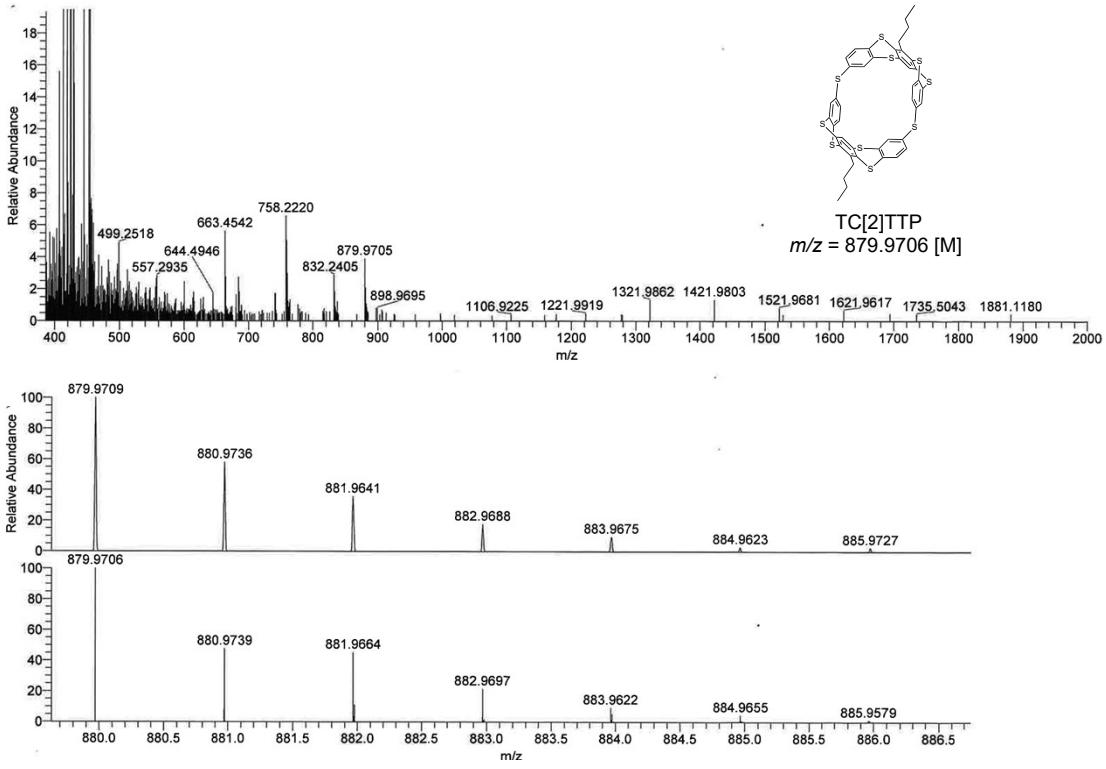


Figure S2. ESI-MS spectrum of TC[2]TTP.

4. Theoretical Calculation

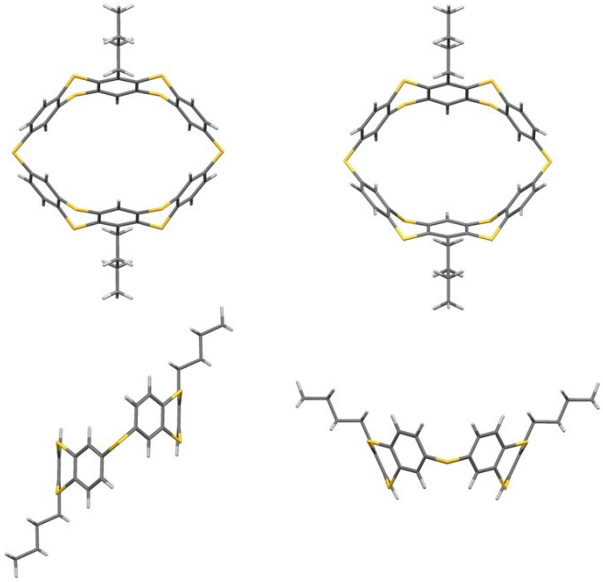


Figure S3. Optimized structures of structural isomer of TC[2]TTP at RB3LYP/6-31G level of theory.

5. ^{1}H and ^{13}C NMR spectra

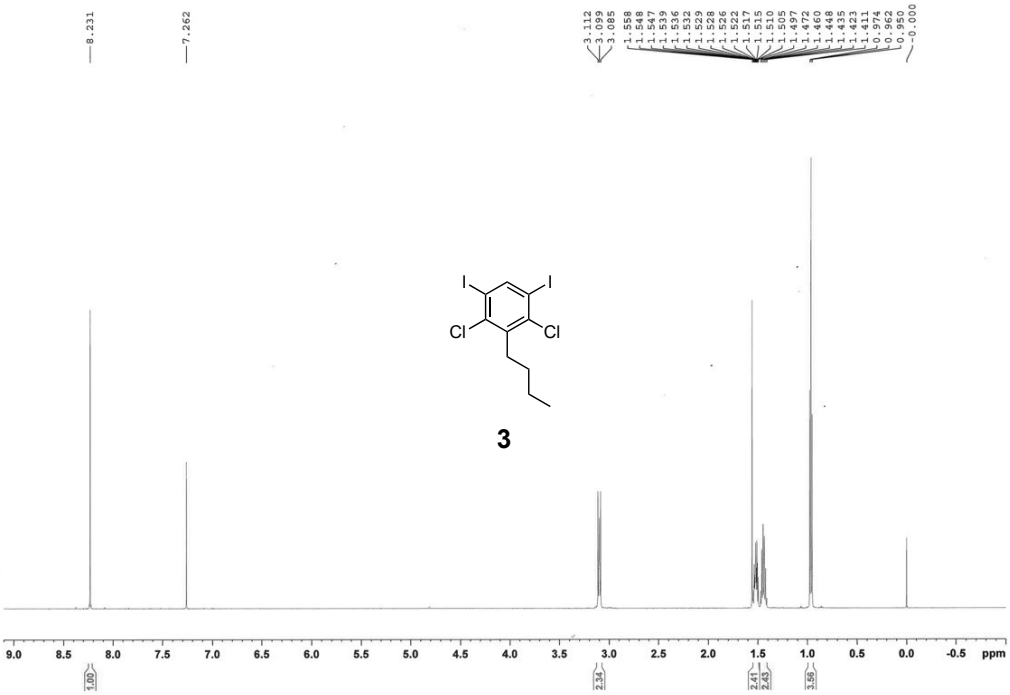


Figure S4. ^{1}H NMR spectrum of **3** (600 MHz, $CDCl_3$).

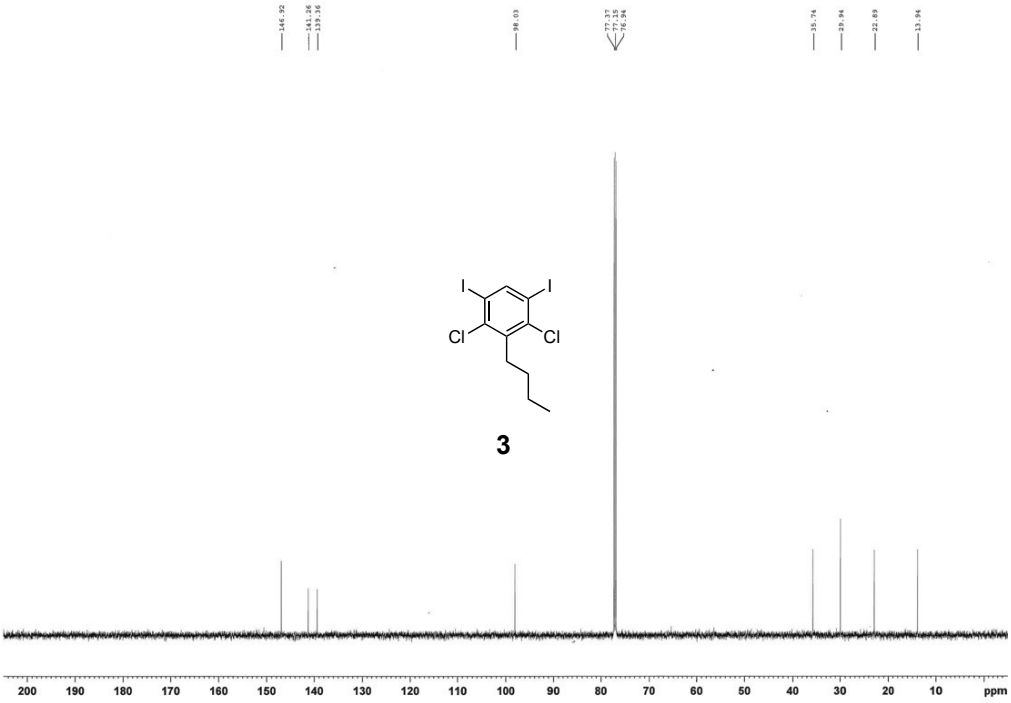


Figure S5. ^{13}C NMR spectrum of **3** (150 MHz, $CDCl_3$).

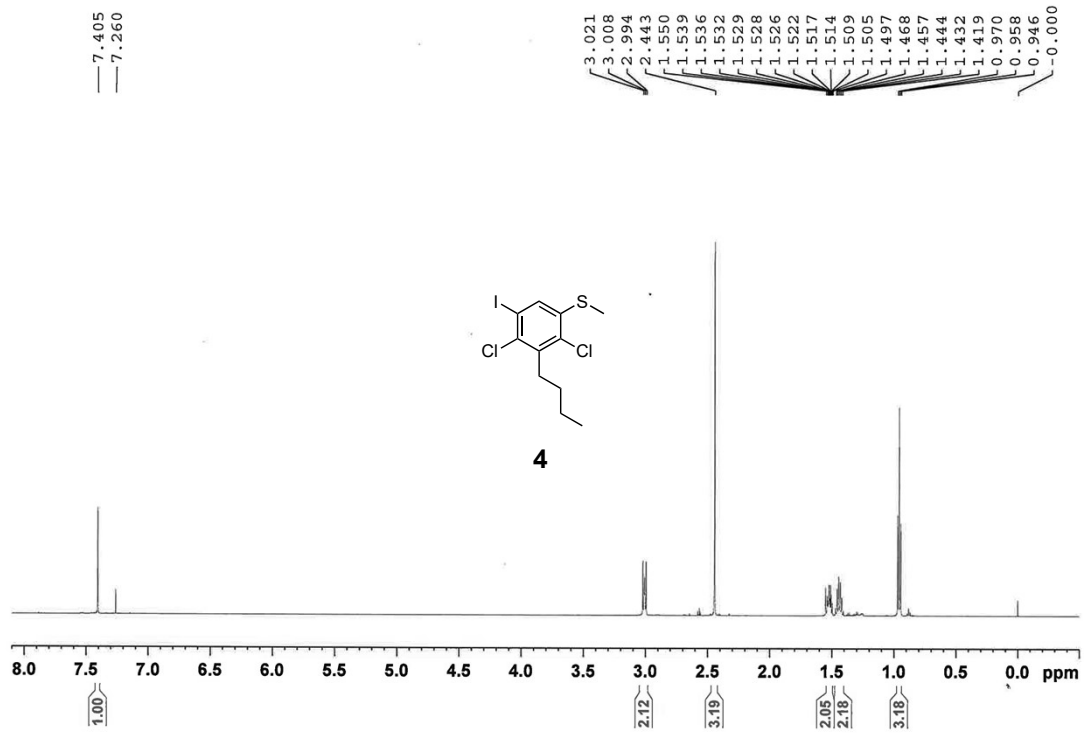


Figure S6. ^{1}H NMR spectrum of **4** (600 MHz, $CDCl_3$).

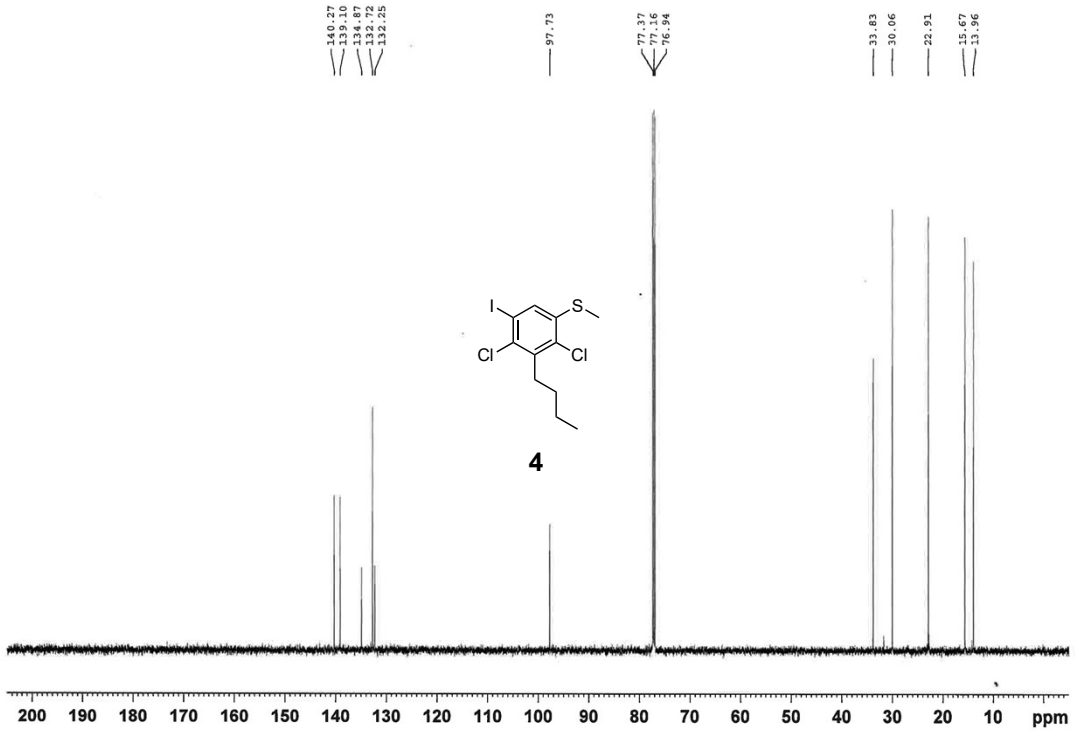


Figure S7. ^{13}C NMR spectrum of **4** (150 MHz, $CDCl_3$).

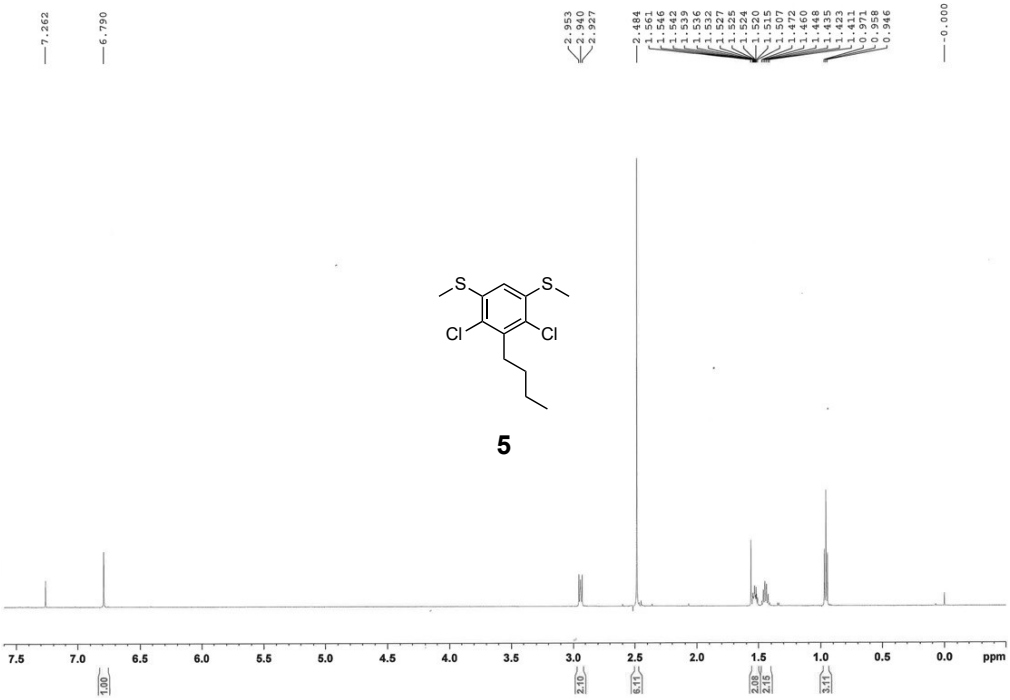


Figure S8. ^{1}H NMR spectrum of **5** (600 MHz, $CDCl_3$).

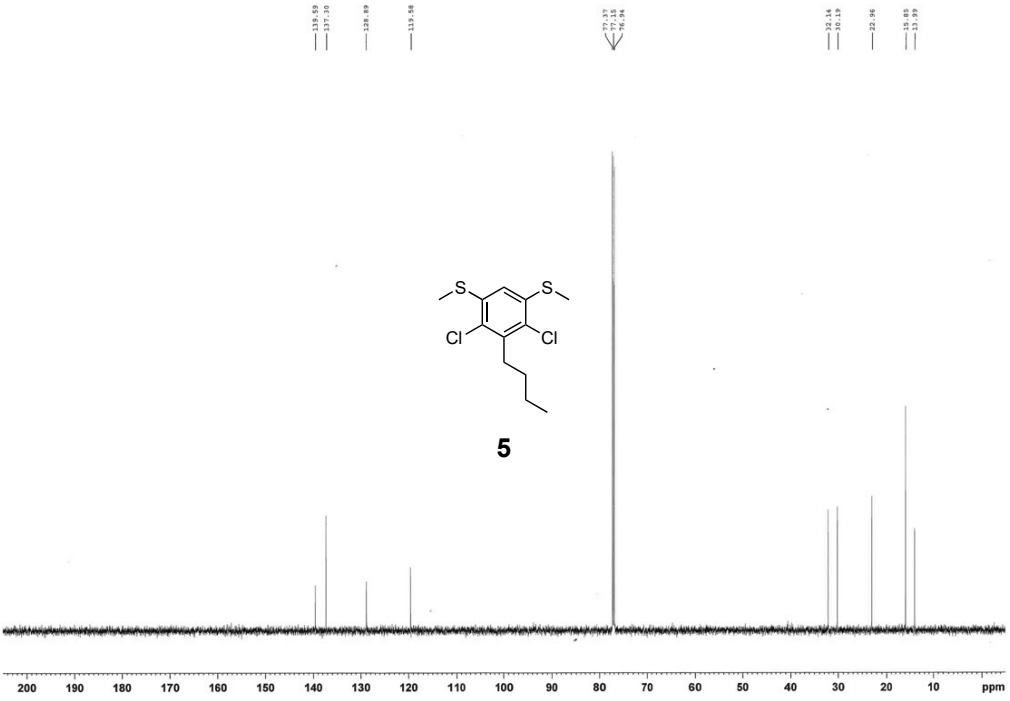


Figure S9. ^{13}C NMR spectrum of **5** (150 MHz, $CDCl_3$).

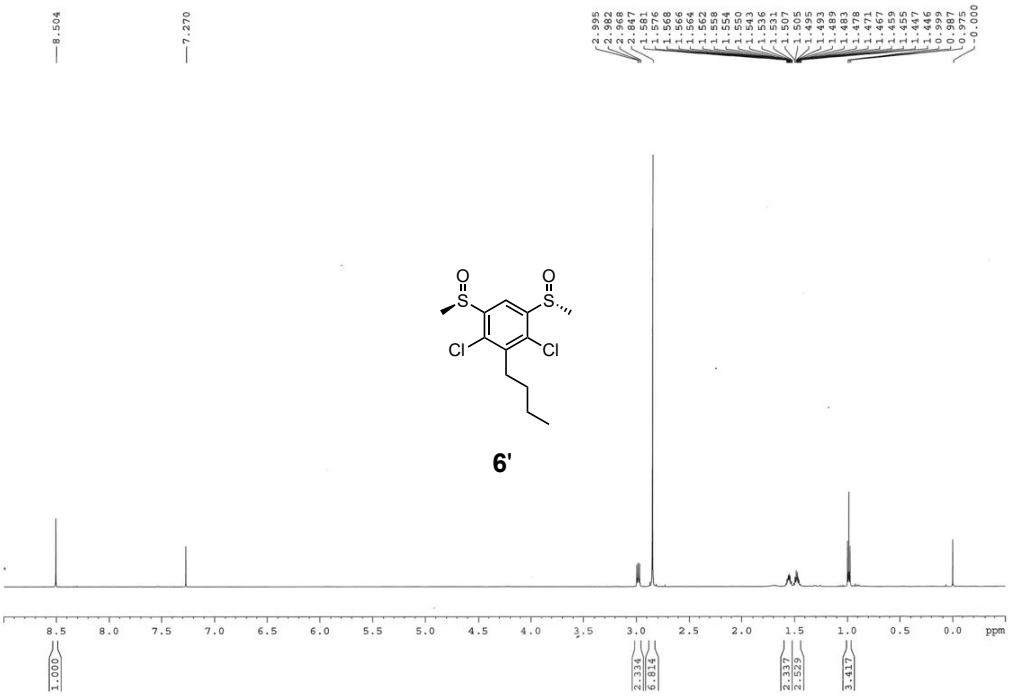


Figure S10. ^{1}H NMR spectrum of **6′** (600 MHz, $CDCl_3$).

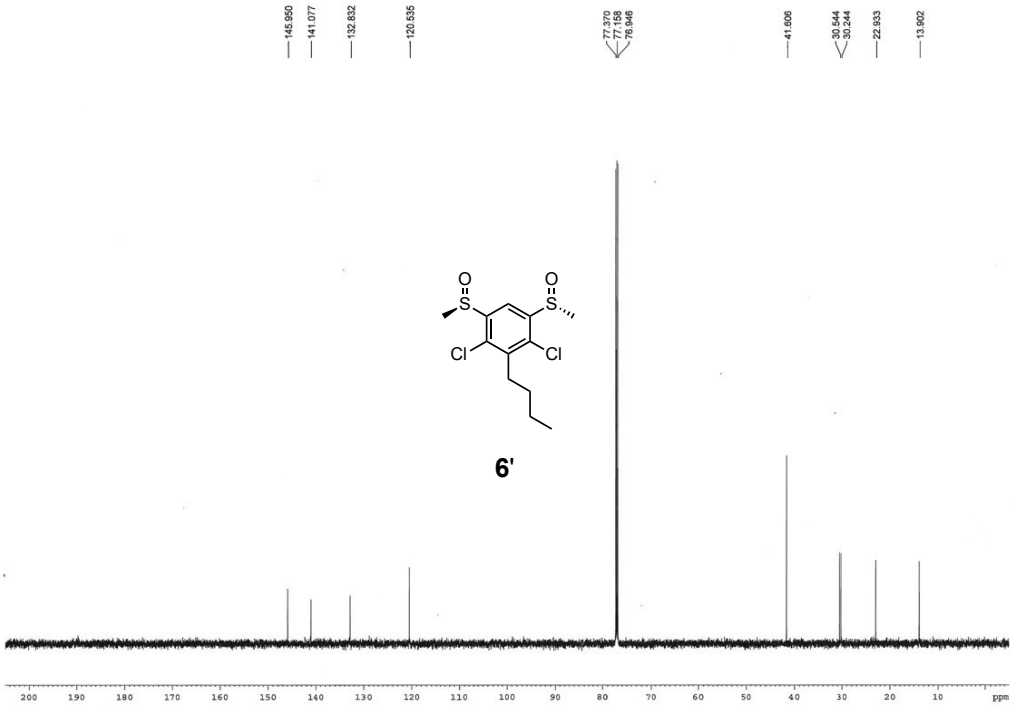


Figure S11. ^{13}C NMR spectrum of **6′** (150 MHz, $CDCl_3$).

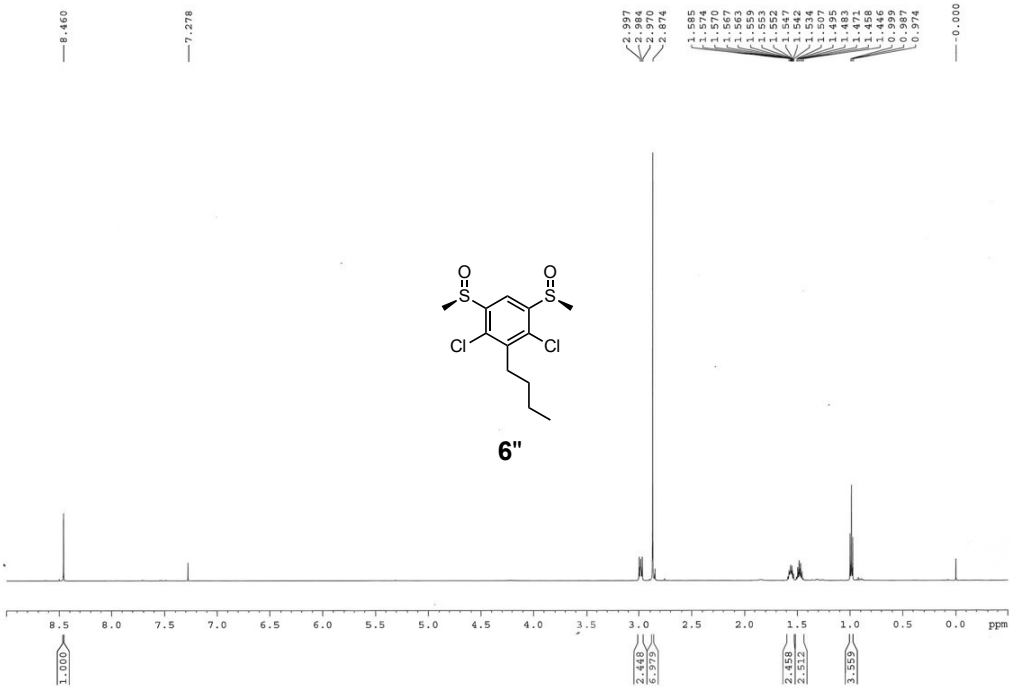


Figure S12. ^{1}H NMR spectrum of **6″** (600 MHz, $CDCl_3$).

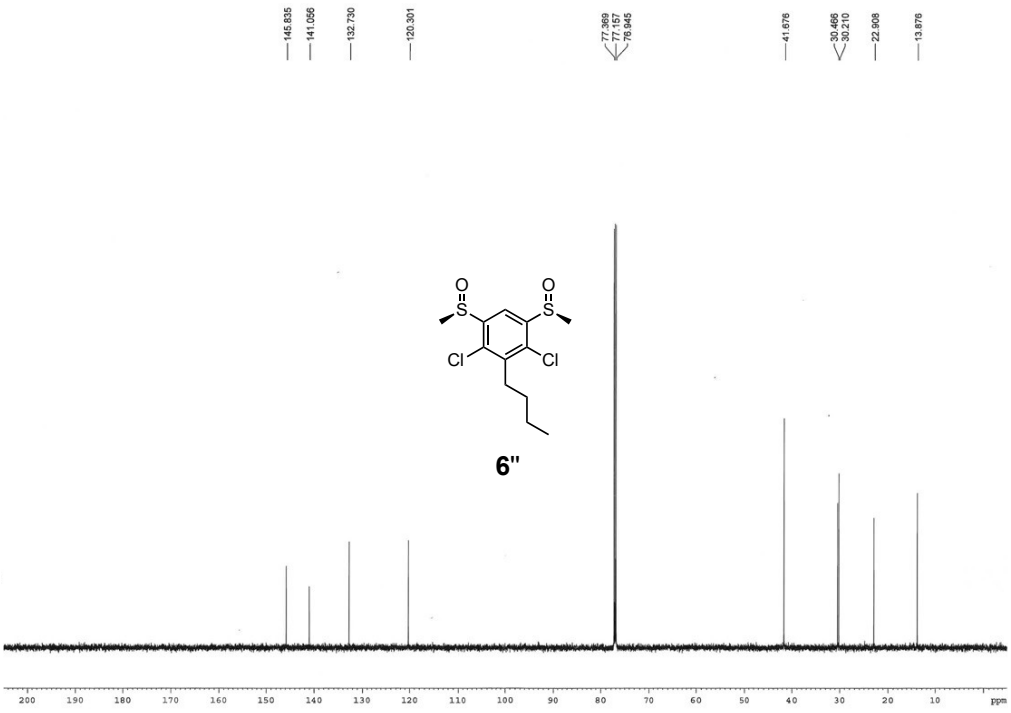


Figure S13. ^{13}C NMR spectrum of **6″** (150 MHz, $CDCl_3$).

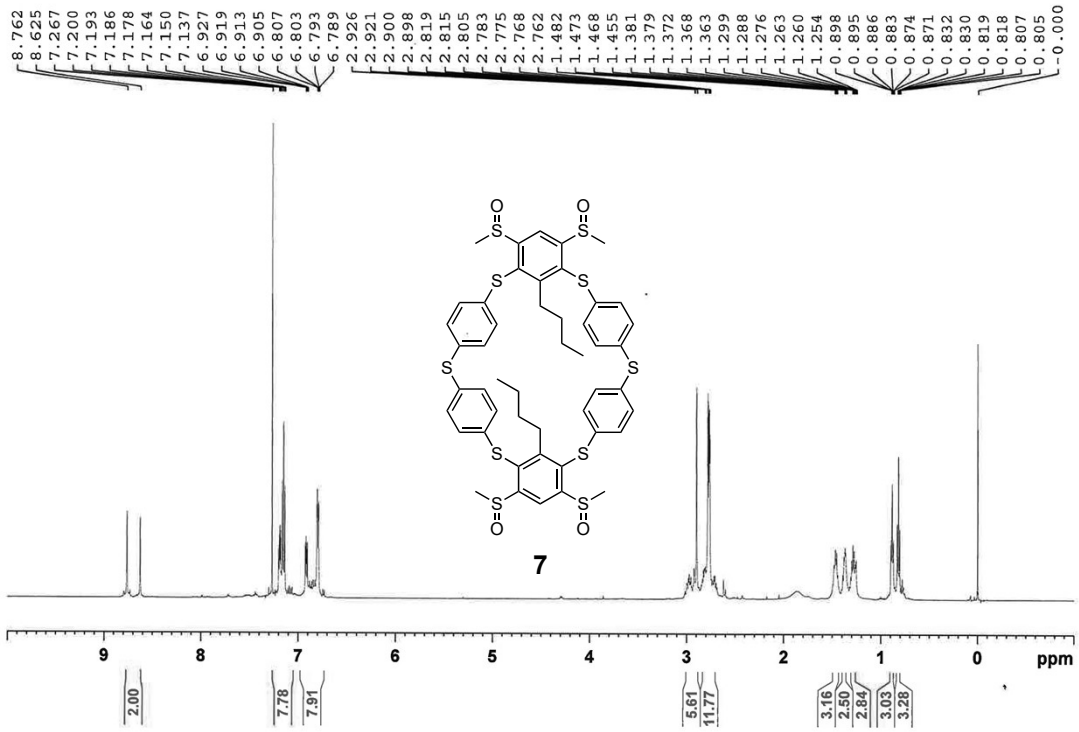


Figure S14. ^{1}H NMR spectrum of **7** (600 MHz, $CDCl_3$).

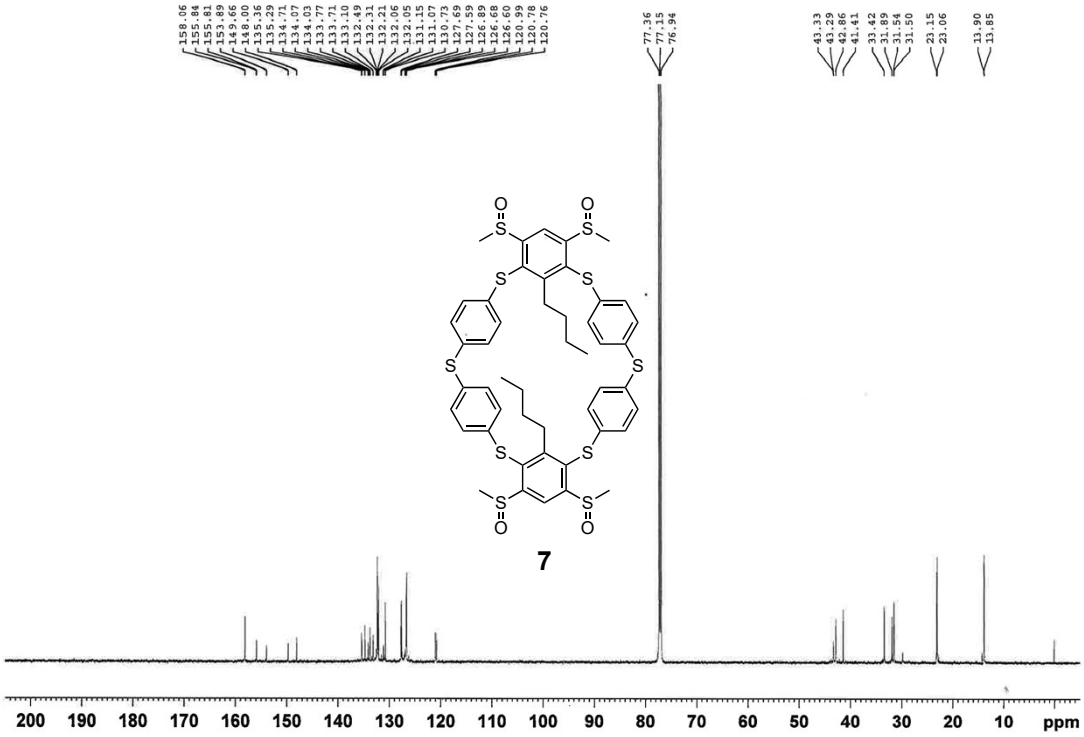


Figure S15. ^{13}C NMR spectrum of **7** (150 MHz, $CDCl_3$).

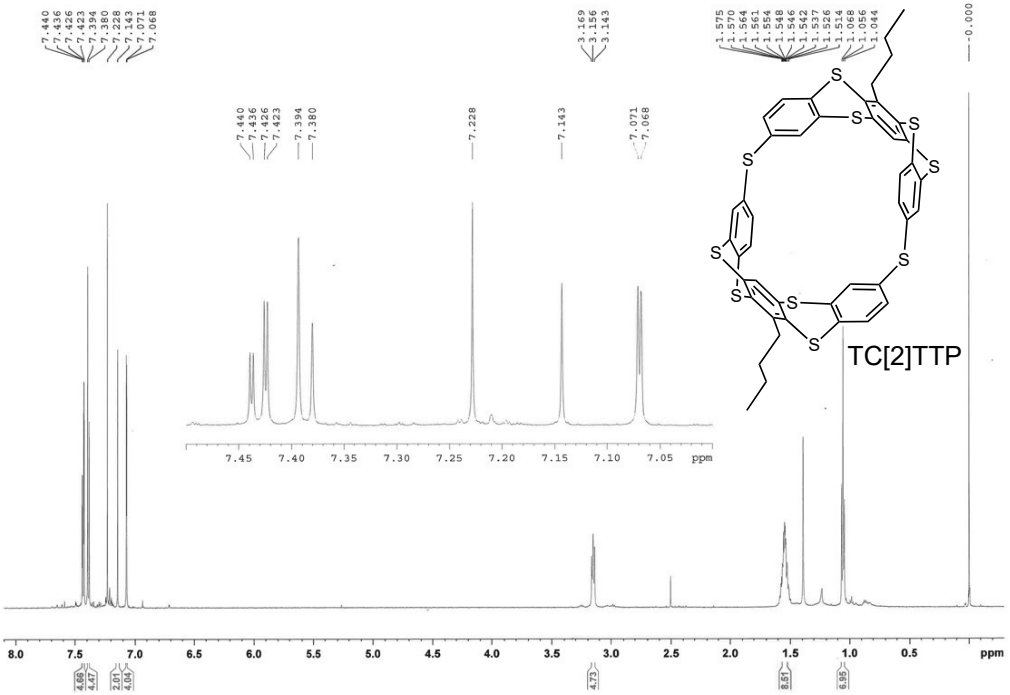


Figure S16. ^{1}H NMR spectrum of TC[2]TTP (600 MHz, $CS_2/CDCl_3$).

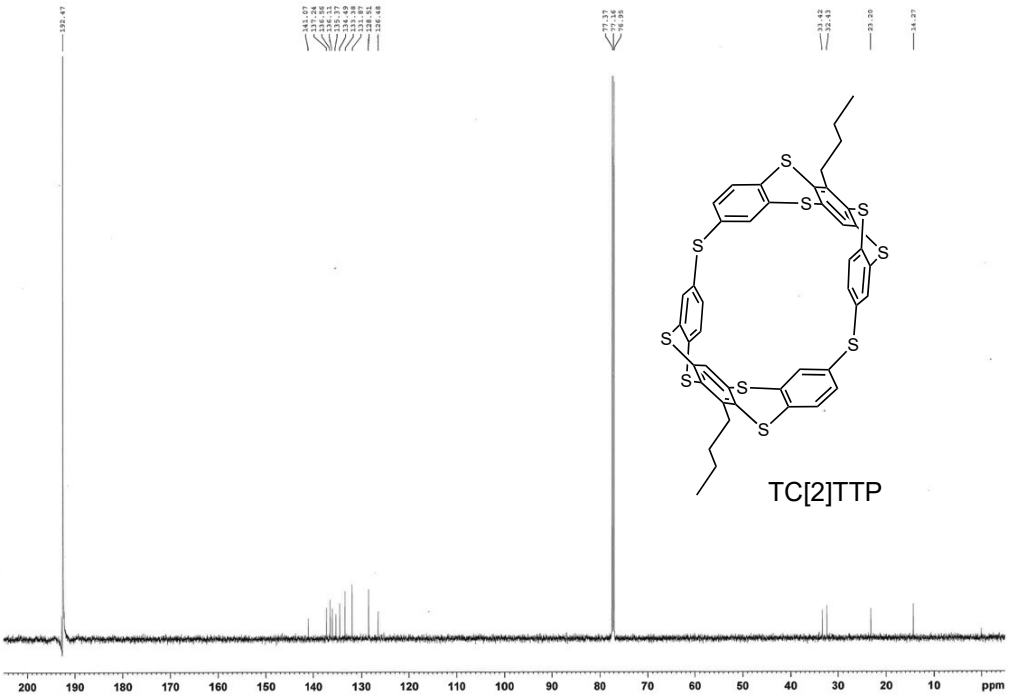


Figure S17. ^{13}C NMR spectrum of TC[2]TTP (150 MHz, $CS_2/CDCl_3$).

6. References

1. *CrysAlisPro: Data collection and Processing Software Package,* Rigaku Oxford Diffraction, Rigaku Corporation, **2019**, Yarton, England.
2. G. M. Scheldrick, *Acta Crystallogr.* **2015**, *A71*, 3-8.
3. G. M. Scheldrick, *Acta Crystallogr.* **2015**, *C71*, 3-8.
4. Gaussian 16, Revision C.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Peterson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gromperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Edigi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyenger, J. Tomasi, M. Cossi, J. M. Millam, M. klene, C. Adamo, R. Cammi,, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, D. J. Fox, Gaussian, Inc., Wallingford Ct, **2016**.
5. N. Weidmann, M. Ketels, P. Knochel, *Angew. Chem. Int. Ed.* **2018**, *57,* 10748-10751, *Angew. Chem.* **2018**, *130*, 10908-10911.
6. K. Fujiki, N. Tanifuji, Y. Sasaki, T. Yokohama, *Synthesis* **2002**, *3*, 343-348.