Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information for:

From Tetrabenzoheptafulvalene to sp^2 Carbon Nano-rings

Kwan Yin Cheung, Shuaijun Yang, Qian Miao*

Department of Chemistry, the Chinese University of Hong Kong, Shatin, New Territories, Hong Kong, China

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1. Synthesis

General: The reagents and starting materials employed were commercially available and used without any further purification unless otherwise noted or made following reported methods as indicated. Anhydrous and O_2-free diethyl ether, THF and toluene were purified by an Advanced Technology Pure-Solv PS-MD-4 system. NMR spectra were recorded on a Bruker AVANCE III 400MHz spectrometer (^{1}H NMR: 400 MHz, ^{13}C NMR: 100 MHz). Chemical shift values (δ) are expressed in parts per million using residual solvent protons (^{1}H NMR, δH = 7.26 for $CDCl_3$; δH = 5.32 for CD_2Cl_2, ^{13}C NMR, δC = 77.16 for $CDCl_3$; δC = 53.84 for CD_2Cl_2) as internal standard. Mass spectra were recorded on Thermo Finnigan MAT 95 XL spectrometer or Bruker Autoflex speed MALDI-TOF. X-ray crystallography data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer. UV-vis absorption spectra were recorded on a Varian CARY 1E UV-vis spectrophotometer. Fluorescence spectra were taken on a Hitachi F-4500 spectrofluorometer. Melting points, without correction, were measured using a Nikon Polarized Light Microscope ECLIPSE 50i POL equipped with an INTEC HCS302 heating stage.

Compound **5**

A mixture of 8*H*-dibenzo[3,4:6,7]cyclohepta[1,2-*c*]furan-8-one (**4**)[1] (1.28 g, 5.2 mmol) and Lawesson's reagent (2.1 g, 5.2 mmol) in 50 ml of anhydrous toluene was heated to reflux under N_2 for 1 day. Then the reaction mixture was concentrated under reduced pressure, and the crude product was purified by column chromatography on silica gel with hexane/dichloromethane 6/1 to 5/1 (V/V) as eluent. **5** (817 mg, 60%) was collected as dark magenta solid. mp: 160–162 ℃. ^{1}H NMR ($CDCl_3$) δ (ppm): 7.84 (s, 2H), 7.76 (d, ^3J = 8 Hz, 2H), 7.44–7.50 (m, 4H), 7.31 (dd, ^3J = 7.2 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ (ppm): 246.1, 150.0, 140.2, 130.9, 129.6, 127.7, 126.3, 125.8, 124.5. HRMS (EI^+): calcd. for $C_{17}H_{10}OS$ ($[M]^+$): 262.0447, found: 262.0451.

Compound **7**

A mixture of 10-bromo-5H-dibenzo[a,d]cyclohepten-5-one (**6**)[2] (560 mg, 2.0 mmol) and Lawesson's reagent (794 mg, 2.0 mmol) in 30 ml of anhydrous toluene was heated to reflux under N_2 for 1 day. Then the reaction mixture was concentrated under reduced pressure, and the crude product was purified by column chromatography on silica gel with hexane/dichloromethane 6/1 (V/V) as eluent. **7** (365 mg, 62%) was collected as dark magenta solid. mp: 129-130 ℃. ^{1}H NMR ($CDCl_3$) δ (ppm): 7.94 (dd, ^3J = 8 Hz, ^4J = 0.8 Hz, 1H), 7.82 (d, ^3J = 8 Hz, ^4J = 1.2 Hz, 1H), 7.78 (dd, ^3J = 8 Hz, ^4J = 1.2 Hz, 1H), 7.67 (s, 1H), 7.52 (td, ^3J = 8 Hz, ^4J = 1.2 Hz, 1H), 7.47 (td, ^3J = 7.6 Hz, ^4J = 1.6 Hz, 1H), 7.38–7.45 (m, 2H), 7.28 (d, ^3J = 7.6 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ (ppm): 239.8, 149.9, 149.4, 134.2, 130.5, 130.3, 130.1, 129.8, 129.4, 120.0, 128.9, 128.4, 127.9, 125.0. (14 instead of 15 signals were observed likely because one signal overlaps with another.) HRMS (EI^+): calcd. for $C_{15}H_9BrS$ ($[M]^+$): 301.9583, found: 301.9580.

Compound **8**

To a solution of **7** (365 mg, 1.2 mmol) in 15 ml of chloroform was added 1 ml of 80% hydrazine hydrate. The resulting solution was stirred at room temperature for 15 minutes, and then washed with water and brine, dried with anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude hydrazone was directly used in the next step without further

1. T. Sasaki, K. Kanamatsu, K. Iizuka and I. Ando, *J. Org. Chem.*, 1976, **41**, 1425–1429.
2. J. H. Rupard, T. De Paulis, A. Janowsky and H. E. Smith, *J. Med. Chem.*, 1989, **32**, 2261–2268.

purification. A suspension of the crude hydrazone, anhydrous sodium sulfate (173 mg, 1.2 mmol), and red HgO (525 mg, 2.4 mmol) in 15 ml of anhydrous Et_2O was stirred in a round-bottom flask, which was attached to a drying tube of anhydrous $CaCl_2$. To this suspension was added 1 ml of a freshly prepared solution of concentrated KOH in ethanol. The reaction mixture was then stirred overnight at room temperature. The suspension was then filtered through a pad of Celite and washed with diethyl ether. Crude diazo compound was obtained as red solid after revomal of solvent under reduced pressure and was used directly in the next step without further purification. To a solution of **5** (281 mg, 1.1 mmol) in 10 ml anhydrous THF was added a solution of the crude diazo compound in 10 ml anhydrous THF. The mixture was stirred at room temperature overnight in a round-bottom flask, which was attached to a drying tube of anhydrous $CaCl_2$. After concentrated under reduced pressure, **8** (470 mg, 83%) was obtained as pale green solid by trituration with ethanol followed by filtration and wash with ethanol. mp: 270-273 ℃. ^{1}H NMR ($CDCl_3$) δ (ppm): 7.92–7.96 (m, 2H), 7.78–7.84 (m, 4H), 7.61 (d, ^3J = 7.6 Hz, 1H), 7.29 (d, ^3J = 7.2 Hz, 1H), 6.96-7.19 (m, 10H), 6.90 (d, ^3J = 7.2 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ (ppm): 142.1, 138.8, 135.2, 135.0, 134.7, 134.5, 134.4, 13.2, 133.8, 133.2, 133.0, 132.4, 131.2, 130.9, 130.8, 130.8, 129.3, 128.4, 128.2, 128.1, 128.1, 127.7, 127.5, 127.2, 126.9, 126.9, 125.3, 124.7, 123.5, 74.3, 73.6. HRMS (EI^+): calcd. for $C_{32}H_{19}BrOS$ ($[M]^+$): 530.0335, found: 530.0329.

Compound **9**

A mixture of **8** (150 mg, 0.28 mmol) and *t*-BuOK (100 mg, 0.89 mmol) in 10 ml of anhydrous THF was heated to reflux under an atmosphere of N_2 overnight. Then the reaction mixture was quenched with water and extracted with CH_2Cl_2. The organic layer was washed with brine, dried with anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel with hexane/dichloromethane 1/1 (V/V) as eluent. **9** (77 mg, 61%) was collected as white solid. mp: 319-321 ℃. ^{1}H NMR (CD_2Cl_2) δ (ppm): 7.42-7.46 (m, 4H), 7.25-7.30 (m, 4H), 7.08-7.15 (m, 8H), 6.19 (s, 2H). ^{13}C NMR ($CDCl_3$) δ (ppm): 158.2, 141.0, 132.1, 130.0, 128.8, 127.8, 127.3, 95.6, 69.5. HRMS (EI^+): calcd. for $C_{32}H_{18}OS$ ($[M]^+$): 450.1073, found: 450.1077.

Compound **10**

A mixture of **9** (50 mg, 0.11 mmol), tricyclohexylphosphine tetrafluoroborate (61 mg, 0.17 mmol) and K_2CO_3 (61 mg, 0.44 mmol) in 2 ml of anhydrous toluene was heated to reflux under N_2 for 2 days. Then the reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel with hexane/ CH_2Cl_2 1/1 to 2/3 (V/V) as eluent. **10** (42 mg, 90%) was collected as white solid. mp: not melt when heated up to 380 ℃. ^{1}H NMR (CD_2Cl_2) δ (ppm): 7.28 (dd, ^3J = 7.2 Hz, ^4J = 0.8 Hz, 4H), 7.23 (dd, ^3J = 7.2 Hz, ^4J = 0.8 Hz, 4H), 7.12 (td, ^3J = 7.6 Hz, ^4J = 1.6 Hz, 4H), 7.06 (td, ^3J = 7.6 Hz, ^4J = 1.6 Hz, 4H), 5.98 (s, 2H). ^{13}C NMR ($CDCl_3$) δ (ppm): 157.4, 144.0, 142.5, 131.0, 128.5, 127.9, 127.0, 125.5, 94.5. HRMS (EI^+): calcd. for $C_{32}H_{18}O$ ($[M]^+$): 418.1352, found: 418.1351.

$Fe(CO)_3$(**10**)

A mixture of **10** (133 mg, 0.32 mmol) and $Fe_2(CO)_9$ (140 mg, 0.38 mmol) in 5 ml of anhydrous toluene was heated to reflux under N_2 overnight. Then the reaction mixture was filtered through a pad of Celite and the filtrate was concentrated under reduced pressure and purified by column chromatography on silica gel with hexane/dichloromethane 4/1 to 3/1 (V/V) as eluent. $Fe(CO)_3$(**10**) (39 mg, 22%) was collected as orange solid. mp: not melt when heated up to 380 ℃. ^{1}H NMR (CD_2Cl_2) δ (ppm): 7.61 (dd, ^3J = 7.6 Hz, ^4J = 1.2 Hz, 2H), 7.44 (d, ^3J = 7.6 Hz, 2H), 7.06-7.27 (m, 12H), 5.93 (s, 2H). ^{13}C NMR (CD_2Cl_2) δ (ppm): 211.7,

210.5, 146.4, 145.1, 144.7, 144.3, 142.7, 133.6, 133.3, 130.3, 129.3, 127.9, 127.9, 127.7, 127.3, 126.7, 126.0, 108.1, 69.1. HRMS (EI^+): calcd. for $C_{35}H_{18}FeO_4$ ($[M]^+$): 558.0549, found: 558.0547.

Compound **12**

A mixture of dibenzo-1,5-cyclooctadiene-3,7-diyne (**11**)[3] (238 mg, 1.19 mmol) and 8*H*-dibenzo[3,4:6,7]cyclohepta[1,2-*c*]furan-8-one (**4**) (645 mg, 2.62 mmol) in 12 ml anhydrous THF was heated to reflux under an atmosphere of N_2 overnight. The reaction mixture was then concentrated under reduced pressure and purified by column chromatography on silica gel with hexane/CH_2Cl_2/Et_2O 3/2/1 to 2/2/1 (V/V/V) as eluent. *Syn*-**12** (113 mg, 14%) and *anti*-**12** (603 mg, 73%) were obtained as light yellow solid.

Syn-**12**: mp: 365-369 ℃. ^{1}H NMR ($CDCl_3$) δ (ppm): 7.82 (dd, ^3J = 8.4 Hz, ^4J = 1.2 Hz, 4H), 7.51–7.58 (m, 8H), 7.16 (td, ^3J = 8 Hz, ^4J = 1.2 Hz, 4H), 6.90 (td, ^3J = 8.4 Hz, ^4J = 1.2 Hz, 4H), 6.56 (d, ^3J = 7.2 Hz, 4H), 5.94 (s, 4H). ^{13}C NMR ($CDCl_3$) δ (ppm): 191.8, 151.4, 149.2, 137.4, 133.9, 131.8, 130.8, 130.6, 129.4, 128.6, 124.2, 91.4. HRMS (MALDI-TOF): calcd. for $C_{50}H_{28}O_4$ ($[M+Na]^+$): 715.1880, found: 715.1860.

Anti-**12**: mp: 371-373 ℃. ^{1}H NMR ($CDCl_3$) δ (ppm): 8.14 (dd, ^3J = 8 Hz, ^4J = 1.2 Hz, 2H), 8.09 (dd, ^3J = 8 Hz, ^4J = 1.2 Hz, 2H), 7.64 (td, ^3J = 7.6 Hz, ^4J = 1.2 Hz, 2H), 7.54–7.58 (m, 6H), 7.44-7.48 (m, 4H), 7.30 (td, ^3J = 8 Hz, ^4J = 1.2 Hz, 2H), 6.99 (td, ^3J = 7.6 Hz, ^4J = 1.2 Hz, 2H), 6.90 (d, ^3J = 7.2 Hz, 2H), 6.18 (s, 2H), 6.12 (d, ^3J = 7.2 Hz, 2H), 4.73 (s, 2H). ^{13}C NMR ($CDCl_3$) δ (ppm): 194.7, 194.1, 152.4, 151.4, 151.4, 147.8, 139.0, 138.5, 135.4, 133.0, 132.2, 131.9, 131.0, 130.8, 130.4, 130.0, 129.5, 129.1, 128.8, 128.5, 128.3, 126.4, 125.3, 124.1, 92.0, 89.3. HRMS (EI^+): calcd. for $C_{50}H_{28}O_4$ ($[M]^+$): 692.1982, found: 692.1984.

Compound **3**

To a suspension of zinc powder (506 mg, 7.7 mmol) in 13 ml anhydrous THF cooled with an ice bath was added $TiCl_4$ (0.4 ml, 3.6 mmol) under an atmosphere of N_2. The reaction mixture was then refluxed for 3 hours and then cooled with an ice bath. A suspension of *syn*-**12** (149 mg, 0.22 mmol) in 10 ml anhydrous THF was added into the reaction mixture as cooled with the ice bath. The resulting mixture was then heated to reflux overnight. The reaction was quenched with an aqueous solution of $NaHCO_3$, extracted with CH_2Cl_2, and the organic layer was dried with anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel with hexane/CH_2Cl_2 3/1 (V/V) as eluent.

3 (27 mg, 20%) was obtained as white solid. mp: not melt when heated up to 380 ℃. ^{1}H NMR (CD_2Cl_2) δ (ppm): 7.58 (dd, ^3J = 7.6 Hz, ^4J = 0.8 Hz, 4H), 7.40-7.42 (m, 4H), 7.30–7.33 (m, 8H), 7.23 (td, ^3J = 7.6 Hz, ^4J = 1.6 Hz, 4H), 7.15 (td, ^3J = 7.6 Hz, ^4J = 1.6 Hz, 4H), 6.83 (s, 4H). ^{13}C NMR (CD_2Cl_2) δ (ppm): 143.2, 143.0, 141.4, 139.0, 137.4, 137.2, 131.7, 128.5, 128.2, 127.5, 127.5, 127.1, 125.7. HRMS (EI^+): calcd. for $C_{50}H_{28}$ ($[M]^+$): 628.2186, found: 628.2179.

13 (14 mg, 10%) was obtained as white solid. mp: not melt when heated up to 380 ℃. ^{1}H NMR (CD_2Cl_2) δ (ppm): 7.80 (dd, ^3J = 7.6 Hz, ^4J = 1.2 Hz, 4H), 7.42–7.44 (m, 4H), 7.18–7.34 (m, 16H), 6.85 (s, 4H). ^{13}C NMR (CD_2Cl_2) δ (ppm): 143.0, 142.1, 139.5, 137.2, 136.5, 132.6, 129.5, 128.3, 127.6, 127.3, 126.2, 125.5, 76.1. HRMS (MALDI-TOF): calcd. for $C_{50}H_{28}O$ ($[M+H]^+$): 645.2213, found: 645.2229.

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3. S. Chaffins, M. Brettreich and F. Wudl, *Synthesis*, **2002**, 1191–1194.

2. X-ray crystallography

X-ray crystallography data were collected on a Bruker AXS Kappa ApexII Duo Diffractometer.

Table S1 Summary of Crystal Structures of **10**, $Fe(CO)_3$(**10**), *syn*-**12**•$CHCl_3$, **3**•CH_2Cl_2 and **13**•CH_2Cl_2.

	10	$Fe(CO)_3$(**10**)	*syn*-**12**•$CHCl_3$	**3**•CH_2Cl_2	**13**•CH_2Cl_2
Space Group	P 2_1/n	P-1	P 2_1 2_1 2_1	P 2_1/n	P 2_1/n
Unit Cell Lengths (Å)	a = 12.737(3) b = 10.827(3) c = 15.738(4)	a = 8.8576(7) b = 9.5150(7) c = 15.8743(12)	a = 10.5417(4) b = 14.8356(5) c = 24.1274(8)	a = 12.2233(2) b = 20.4397(3) c = 15.6120(2)	a = 12.3541(3) b = 20.5251(6) c = 15.5930(4)
Unit Cell Angles (°)	α = 90.00 β = 105.708(6) γ = 90.00	α = 79.571(2) β = 74.479(2) γ = 86.413(2)	α = 90 β = 90 γ = 90	α = 90 β = 11.7594(9) γ = 90	α = 90 β = 11.5622(16) γ = 90
Cell Volume ($Å^3$)	2089.27	1267.66	3773.34	3622.59	3677.21
R factor	0.0997	0.0504	0.0536	0.0569	0.0675

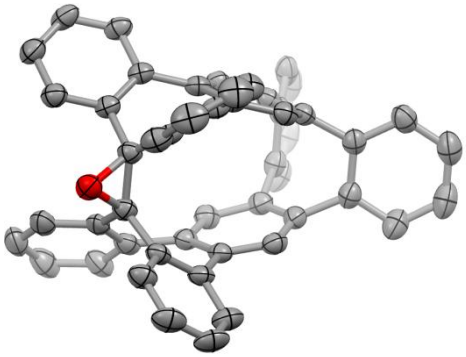


Figure S1 Structure of **13** in the crystal of **13**•CH_2Cl_2 (Hydrogen atoms are removed for clarity and carbon atom positions shown as 50% probability ellipsoids.)

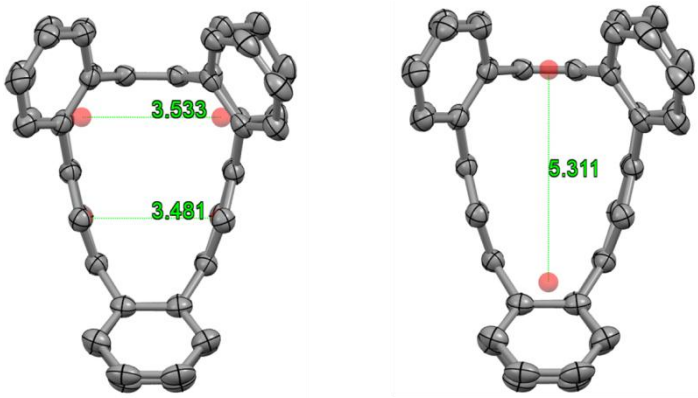


Figure S2 Crystal structure of **3** indicating the dimensions (Hydrogen atoms and crystallized CH_2Cl_2 molecule are removed for clarity and carbon atom positions shown as 50% probability ellipsoids.)

3. Cyclic Voltammetry

The cyclic voltammetry was performed in a solution of CH_2Cl_2 with 0.1M Bu_4NPF_6 as the supporting electrolyte, at a scan rate of $10mVs^{-1}$. Ferrocene/ferrocenium was used as the internal standard. Potentials were referenced to ferrocenium/ferrocene ($FeCp_2^+/FeCp_2^0$).

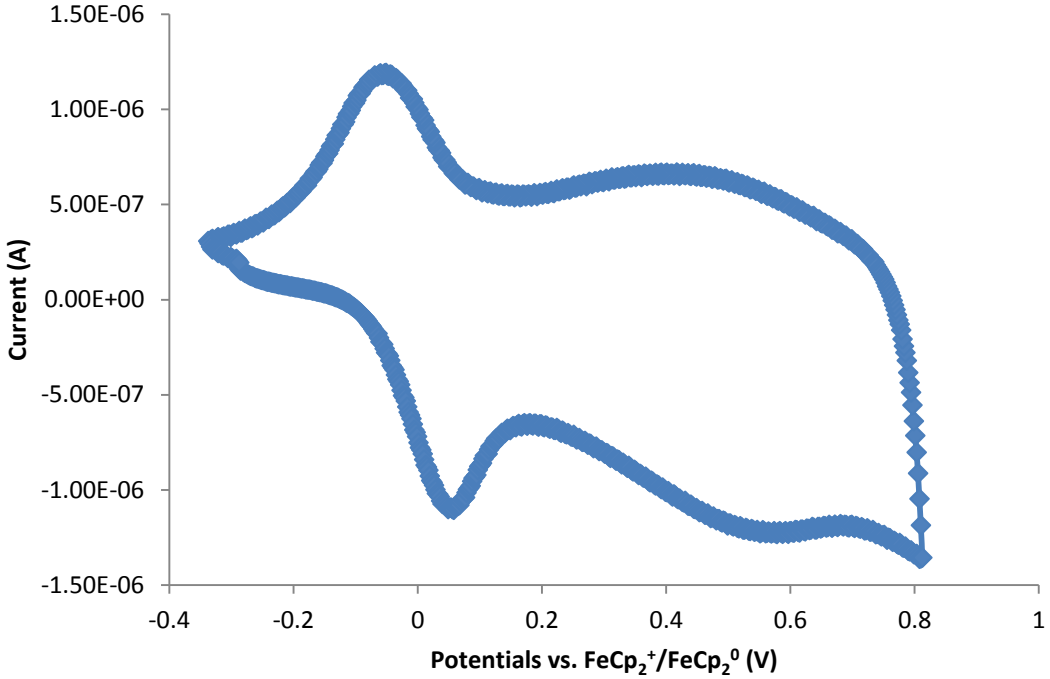


Figure S3 Cyclic voltammogram of **3**

Table S2 Summary of electrochemical potentials and energy levels and HOMO of **3**

Compound	Oxidation potential vs Fc+/Fc (V) [a]	HOMO (eV) [c]
	E_{ox1}	
3	0.49	−5.59

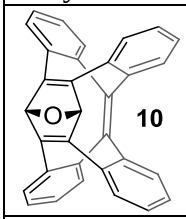
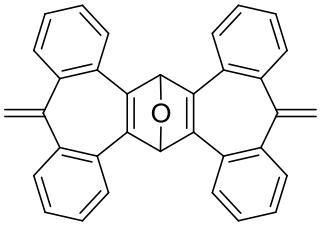
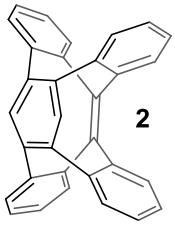
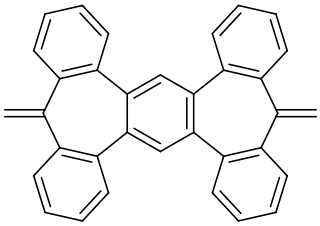
a. Half-wave potential versus ferrocenium/ferrocene for the oxidation wave. *c*. Estimated from HOMO = −5.10 – E_{ox} (eV).[4]

([4]) The commonly used formal potential of the redox couple of ferrocenium/ferrocene (Fc^+/Fc) in the Fermi scale is −5.1 eV, which is calculated on the basis of an approximation neglecting solvent effects using a work function of 4.46 eV for the normal hydrogen electrode (NHE) and an electrochemical potential of 0.64 V for (Fc^+/Fc) versus NHE. See: C. M. Cardona, W. Li, A. E.Kaifer, D. Stockdale, G. C. Bazan, *Adv. Mater.* **2011**, *23*, 2367–2371.

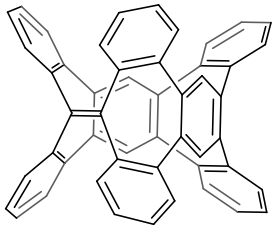
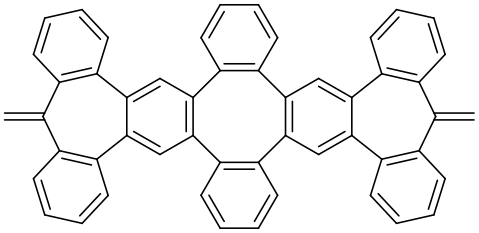
4. Density functional theory (DFT) calculation

Gaussian 09 program[5] was used for the Density Function Theory (DFT) calculation. Molecular geometries were optimized at the B3LYP level of DFT with the 6-31G(d,p) basis set. The fully optimized structures were confirmed to be true minima by vibrational analysis.

Table S3 Summary of energies of stationary points (Hartree). E: electronic energy; ZPE: zero-point energy; H: sum of electronic and themal enthalpies

Compound	**Energy**	**E+ZPE**	**H**
Ethylene	-78.5938075248	-78.542678	-78.538691
 **10**	-1305.39703539	-1304.998691	-1304.976466
	-1384.04881870	-1383.595726	-1383.569831
 **2**	-1230.15069602	-1229.758477	-1229.736669
	-1308.90492267	-1308.456419	-1308.431051

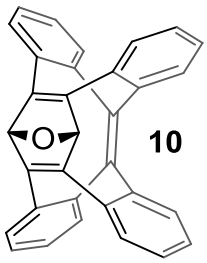
[5] Gaussian 09, Revision B.01, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2009

 3	-1922.25422029	-1921.639147	-1921.604733
	-2000.88413646	-2000.214881	-2000.176516

Optimized Cartesian coordinates at B3LYP/6-31G(d,p) level of theory

Ethylene

C	0.000000	0.000000	-0.664963
C	0.000000	0.000000	0.664971
H	0.000000	0.923177	-1.238159
H	0.000000	-0.923177	-1.238159
H	0.000000	-0.923197	1.238134
H	0.000000	0.923197	1.238134



C	-0.675249	0.000000	-1.063068
C	-1.475554	1.247651	-0.832413
C	-1.475554	-1.247651	-0.832413
C	-1.760104	-1.564146	0.523703
C	-1.760104	1.564146	0.523703
C	-1.207709	-0.674001	1.558519
C	-1.207709	0.674001	1.558519
C	-1.937729	-2.084836	-1.848940
C	-2.473662	-2.733505	0.815479
C	-2.930147	-3.561587	-0.209519
C	-2.663813	-3.236969	-1.540457
C	-1.937729	2.084836	-1.848940
C	-2.473662	2.733505	0.815479
C	-2.663813	3.236969	-1.540457
C	-2.930147	3.561587	-0.209519
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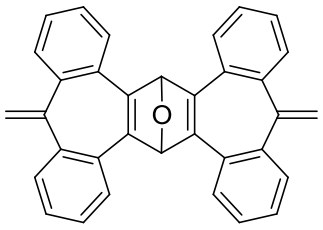

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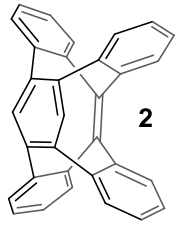

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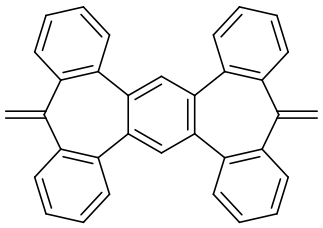

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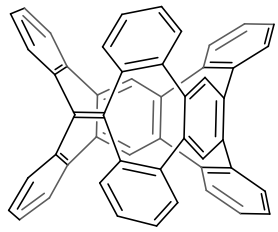

3

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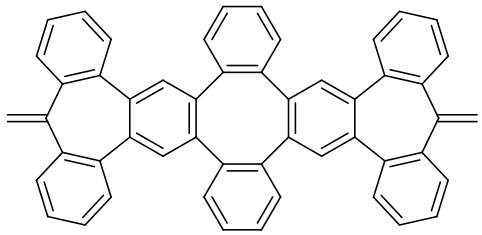

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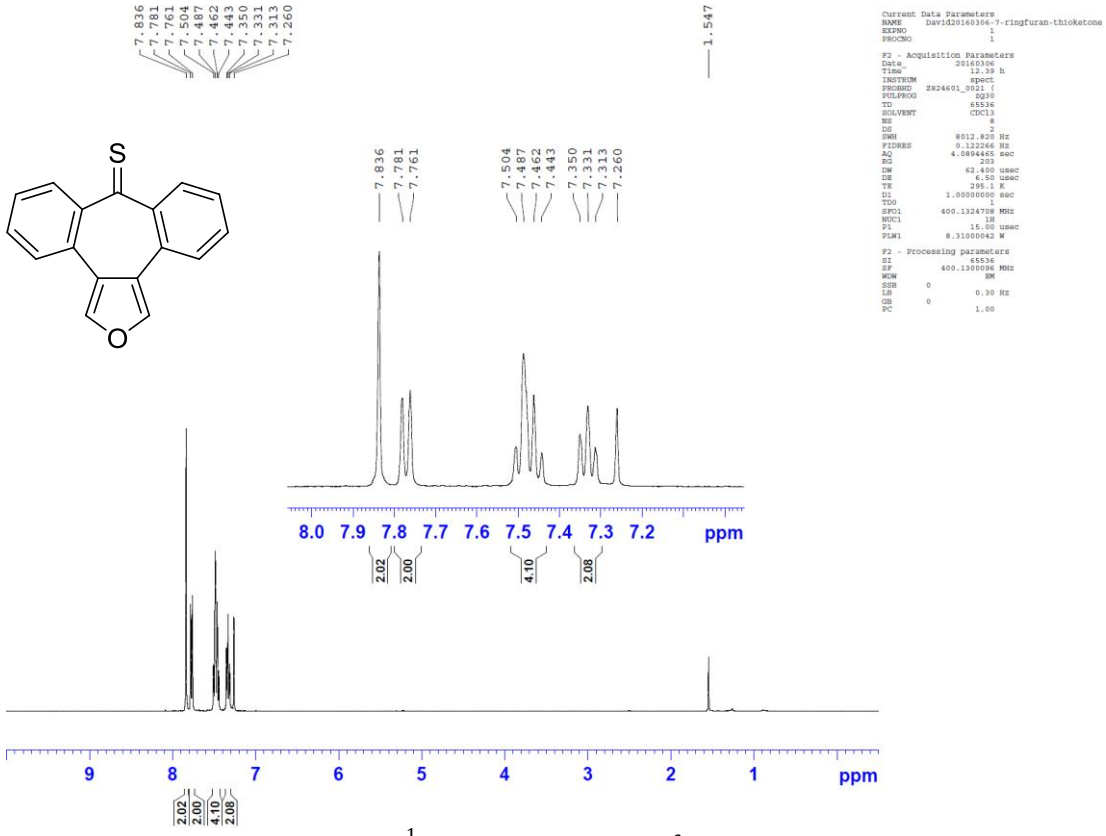
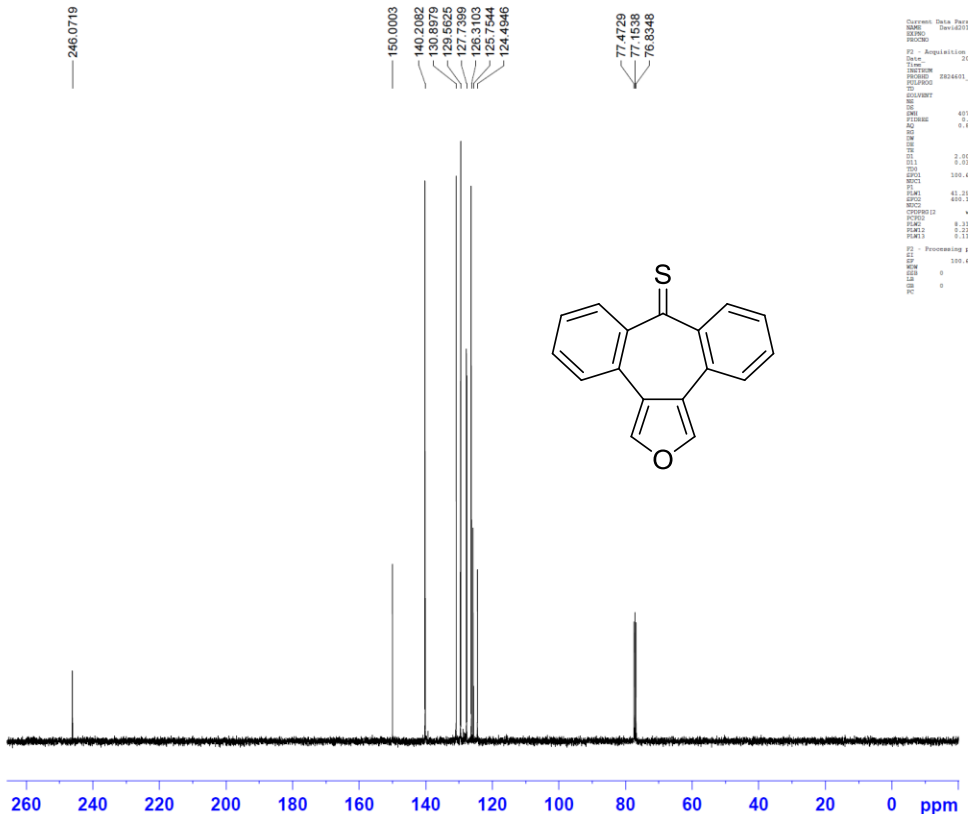
H	-3.090920	6.587487	-0.797389

H	-1.857610	5.044670	0.677713
H	3.856630	3.747143	0.676738
H	5.635251	4.603812	-0.799923
H	5.194064	4.882942	-3.240645
H	2.943681	4.349414	-4.141579
H	0.380365	-2.523801	-4.335495
H	-1.427812	-2.109841	-4.337079
H	1.427812	2.109841	-4.337079
H	-0.380365	2.523801	-4.335495

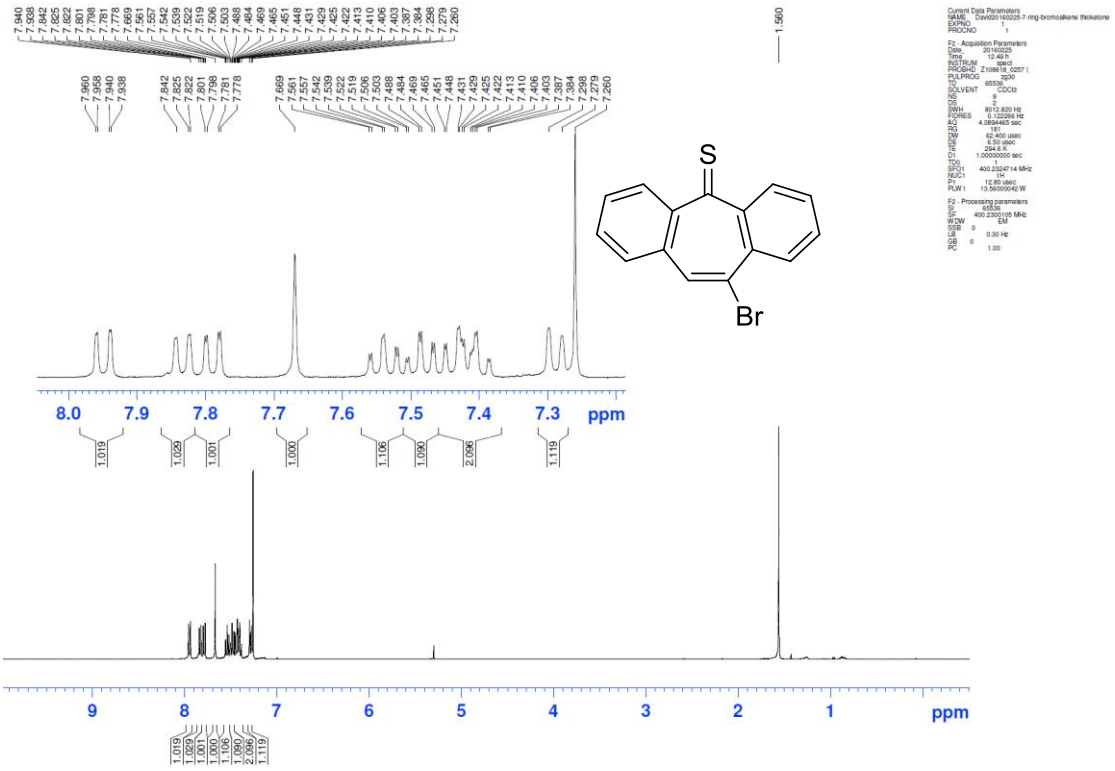
3. NMR Spectra



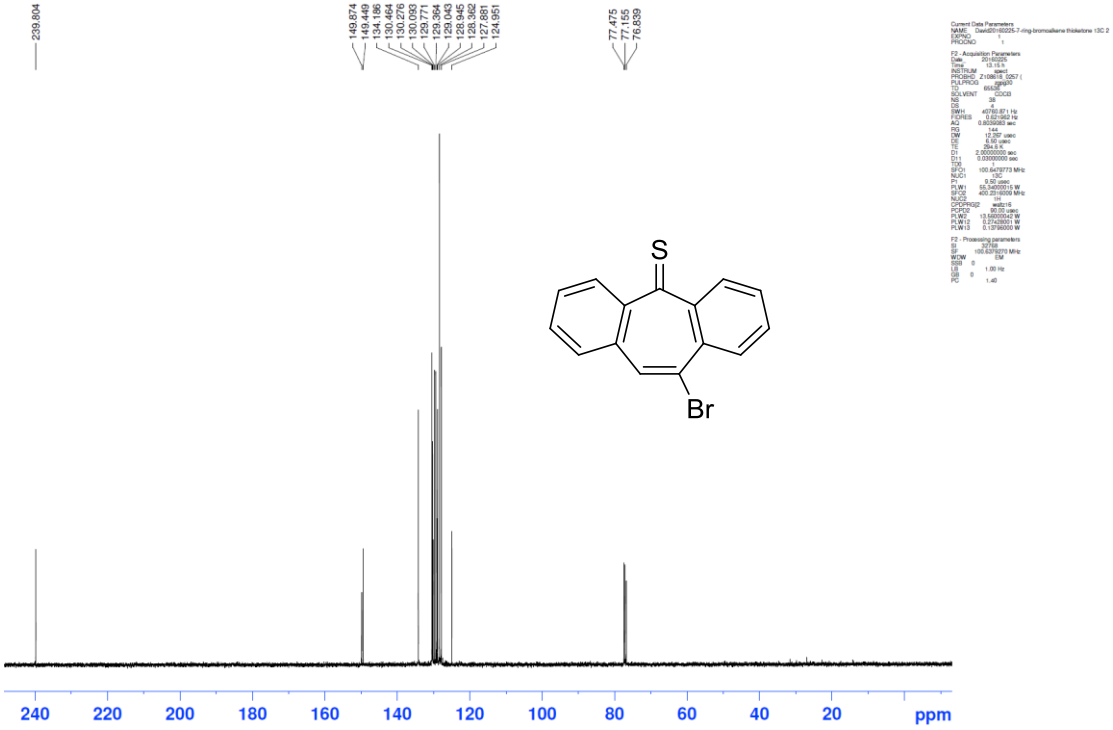
^{1}H NMR spectrum of **5**



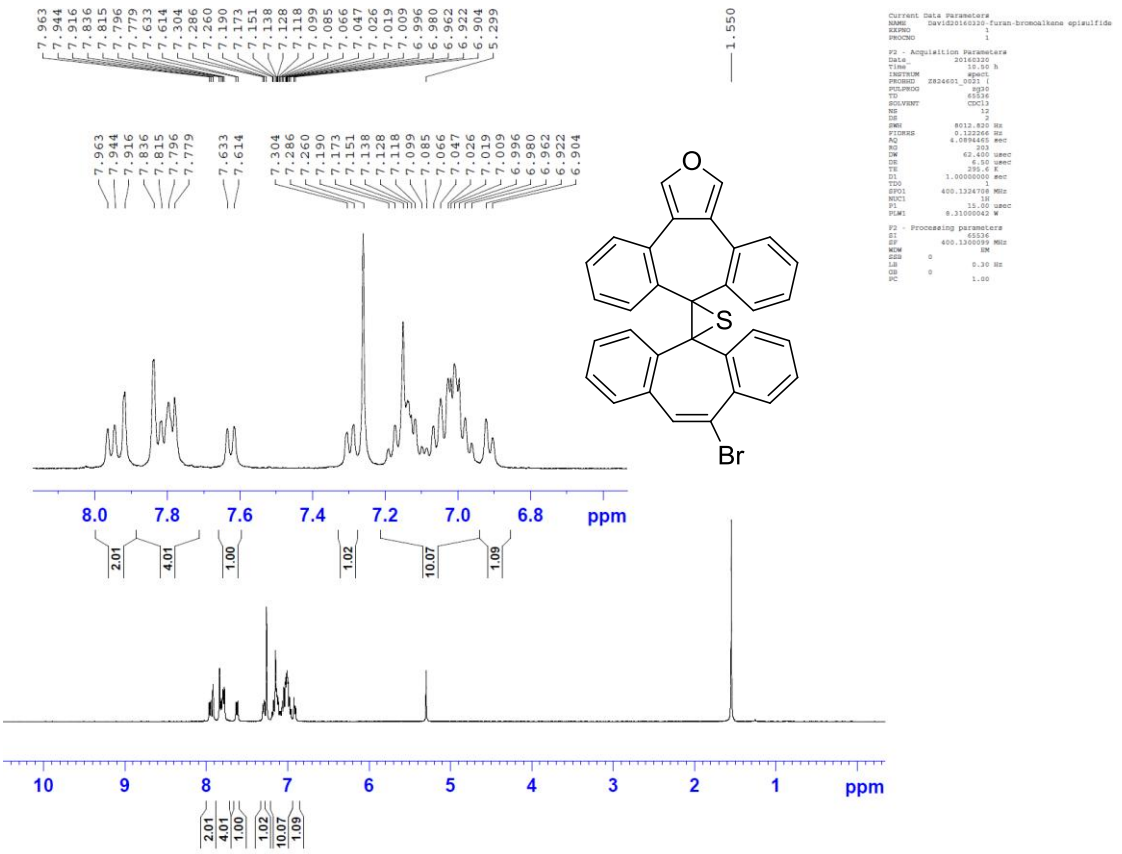
^{13}C NMR spectrum of **5**



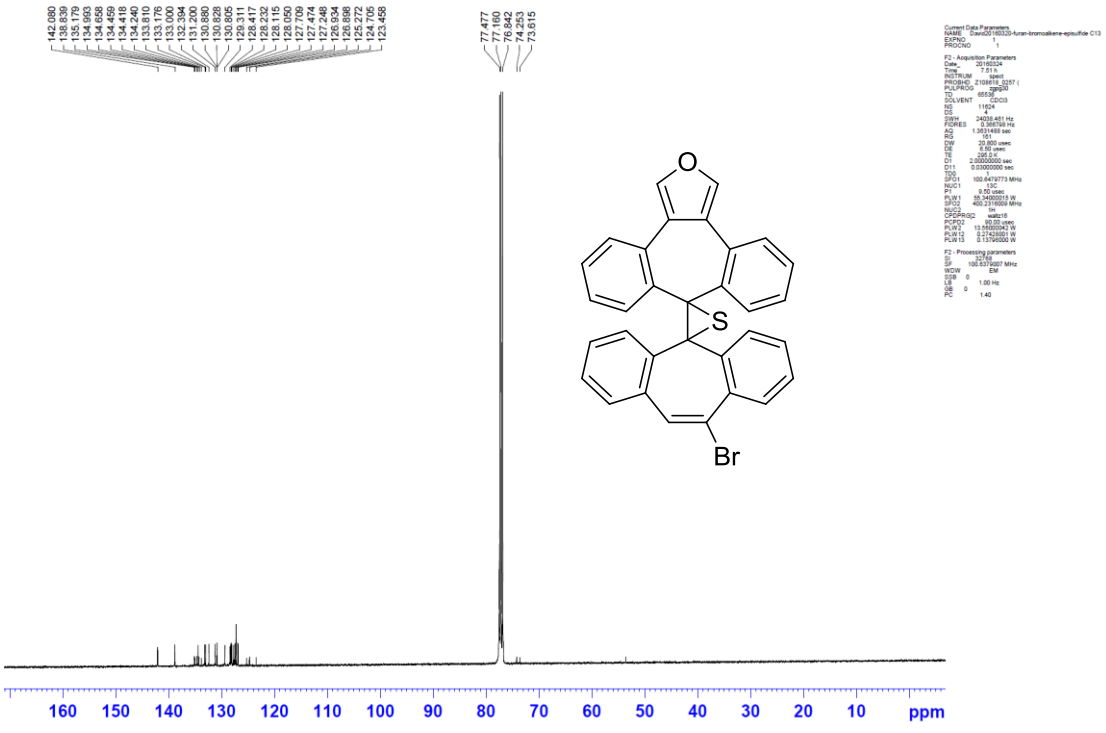
^{1}H NMR spectrum of **7**



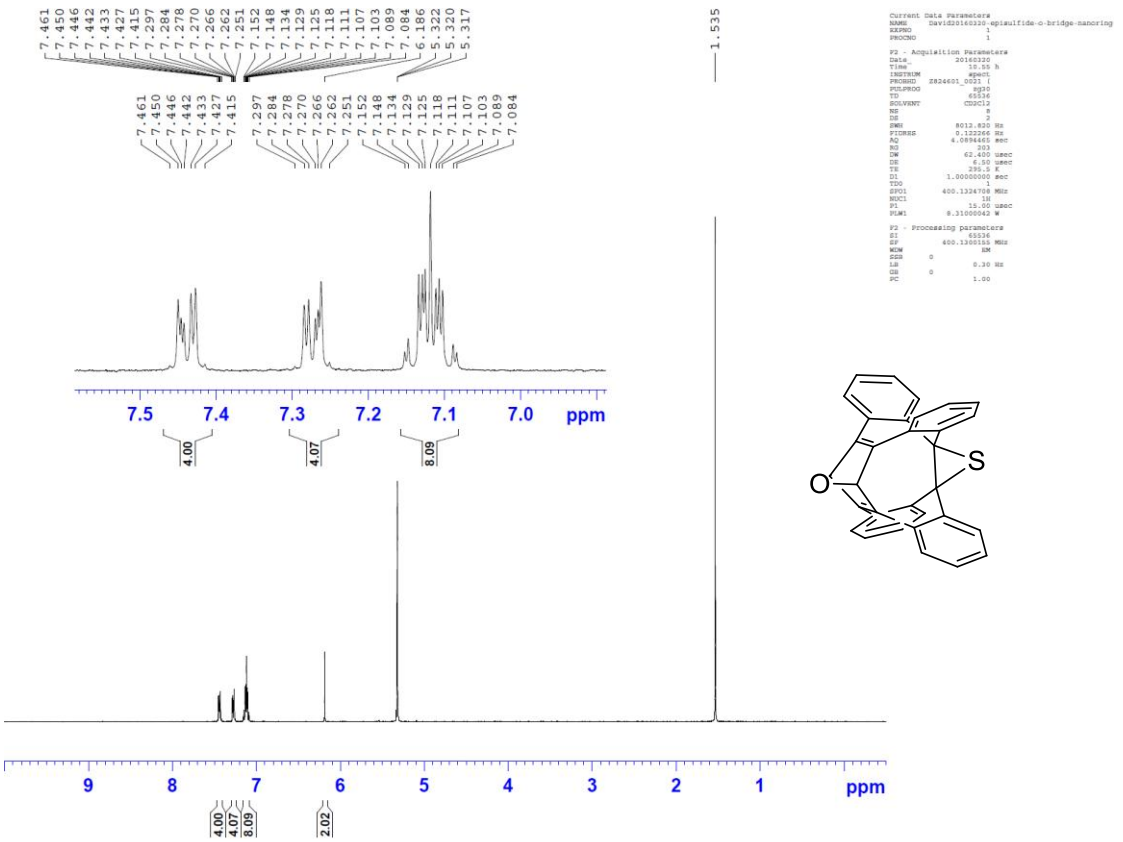
^{13}C NMR spectrum of **7**



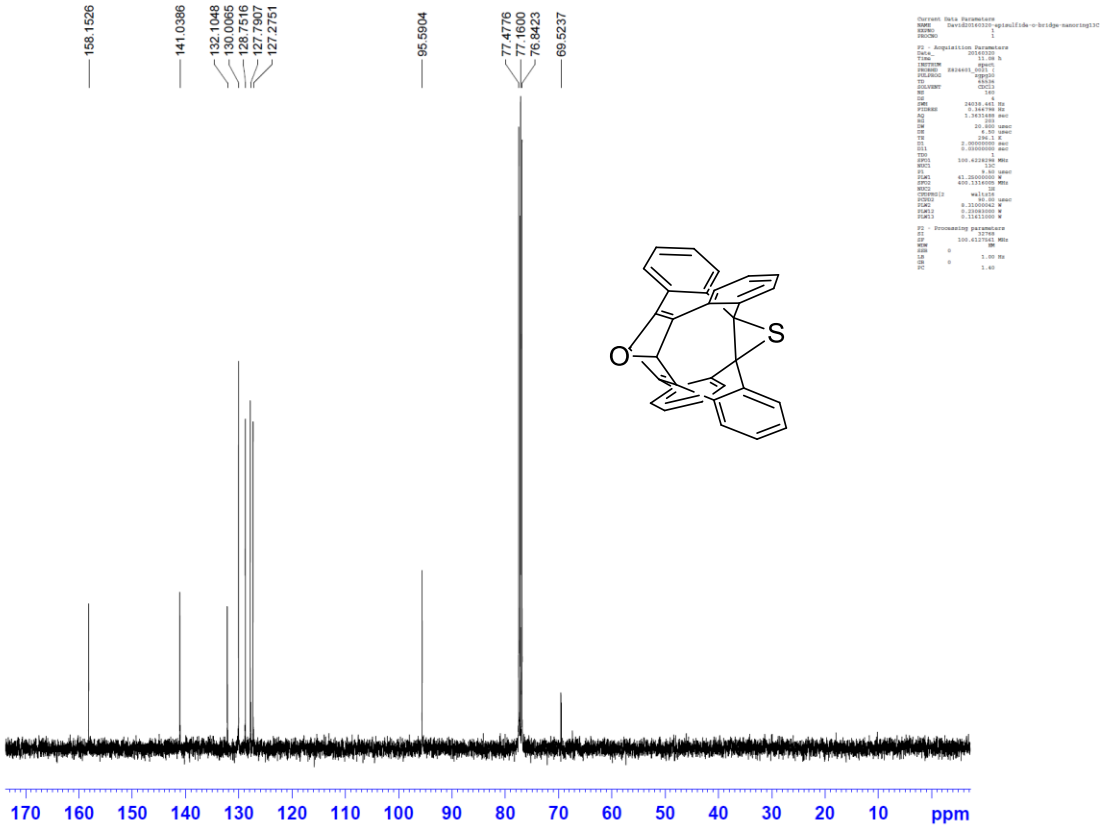
^{1}H NMR spectrum of **8**



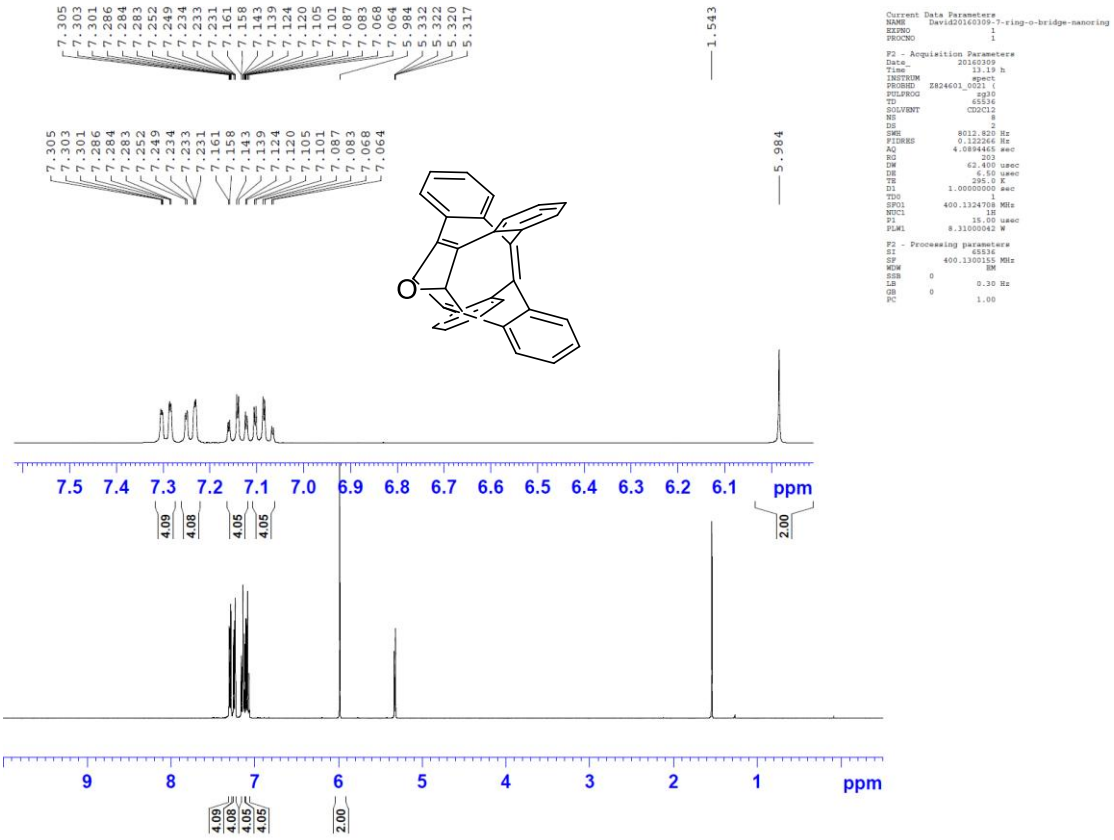
^{13}C NMR spectrum of **8**



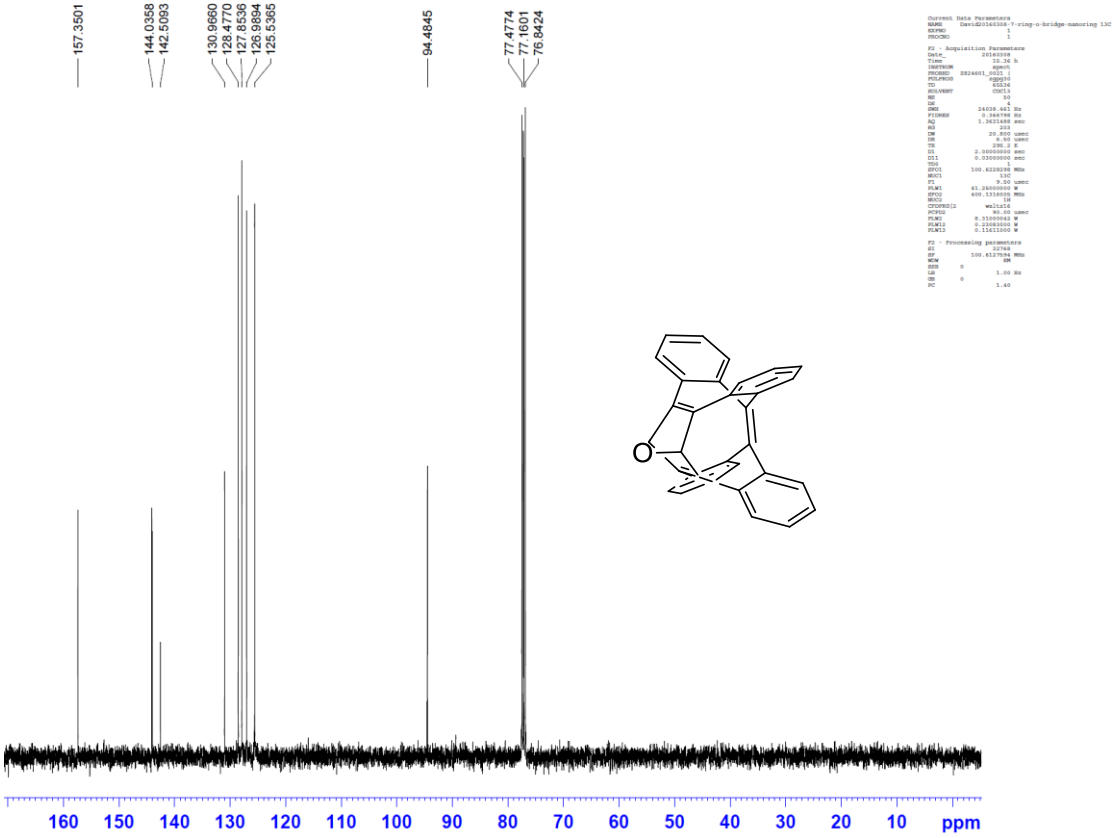
^{1}H NMR spectrum of **9**



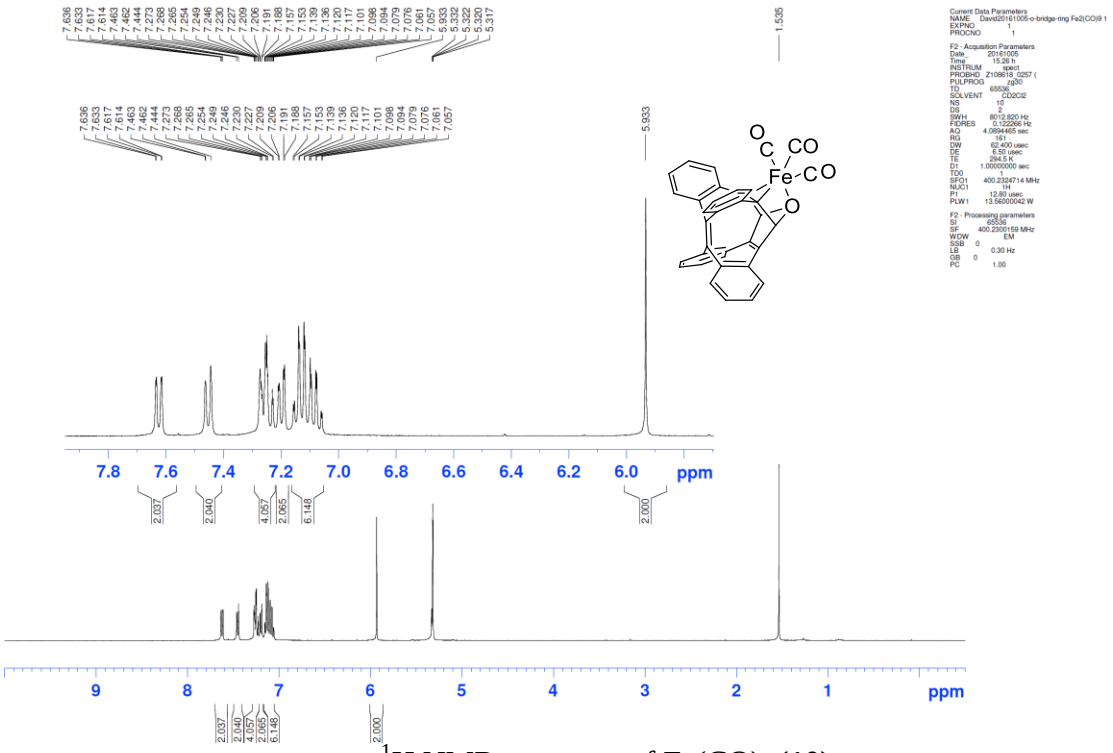
^{13}C NMR spectrum of **9**



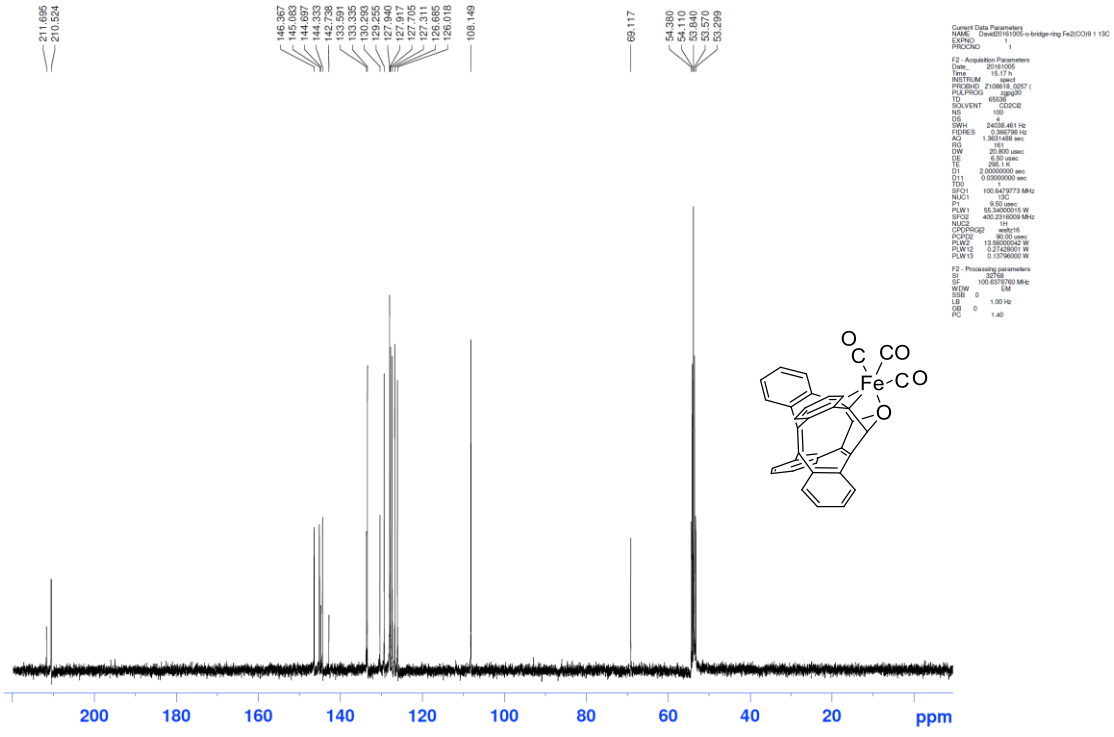
^{1}H NMR spectrum of **10**



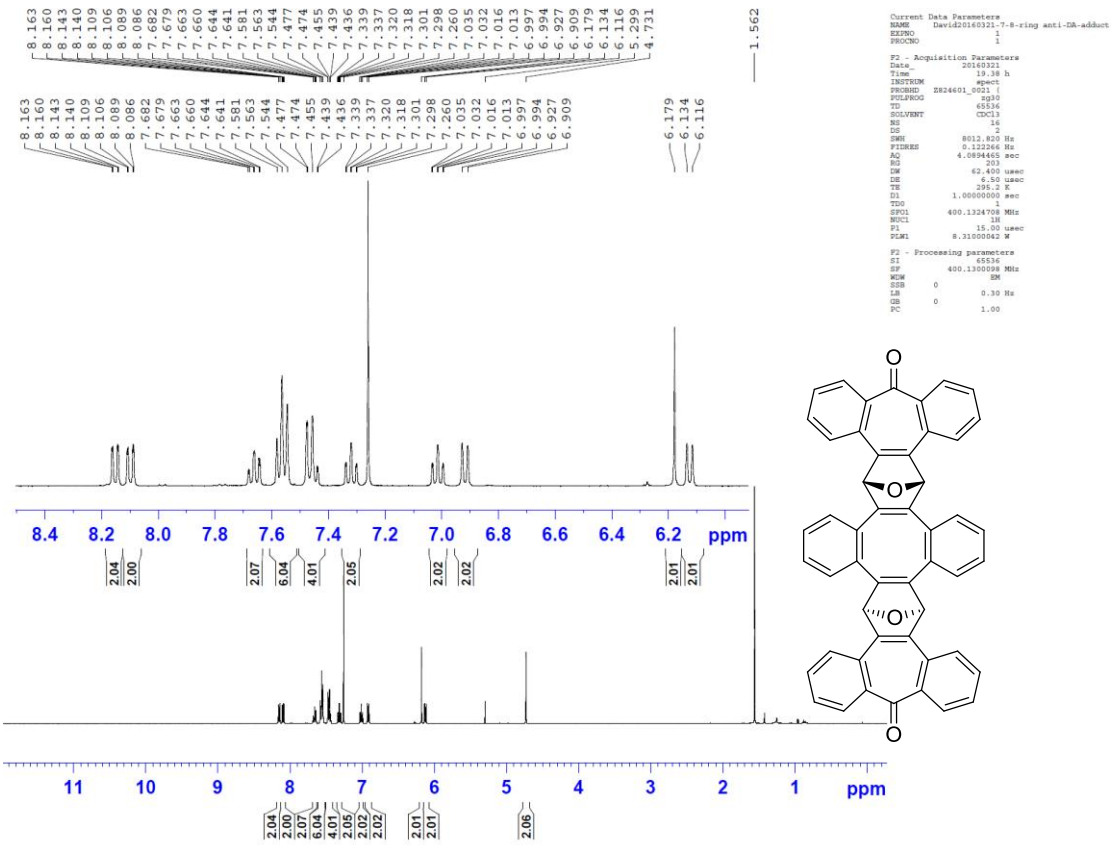
^{13}C NMR spectrum of **10**



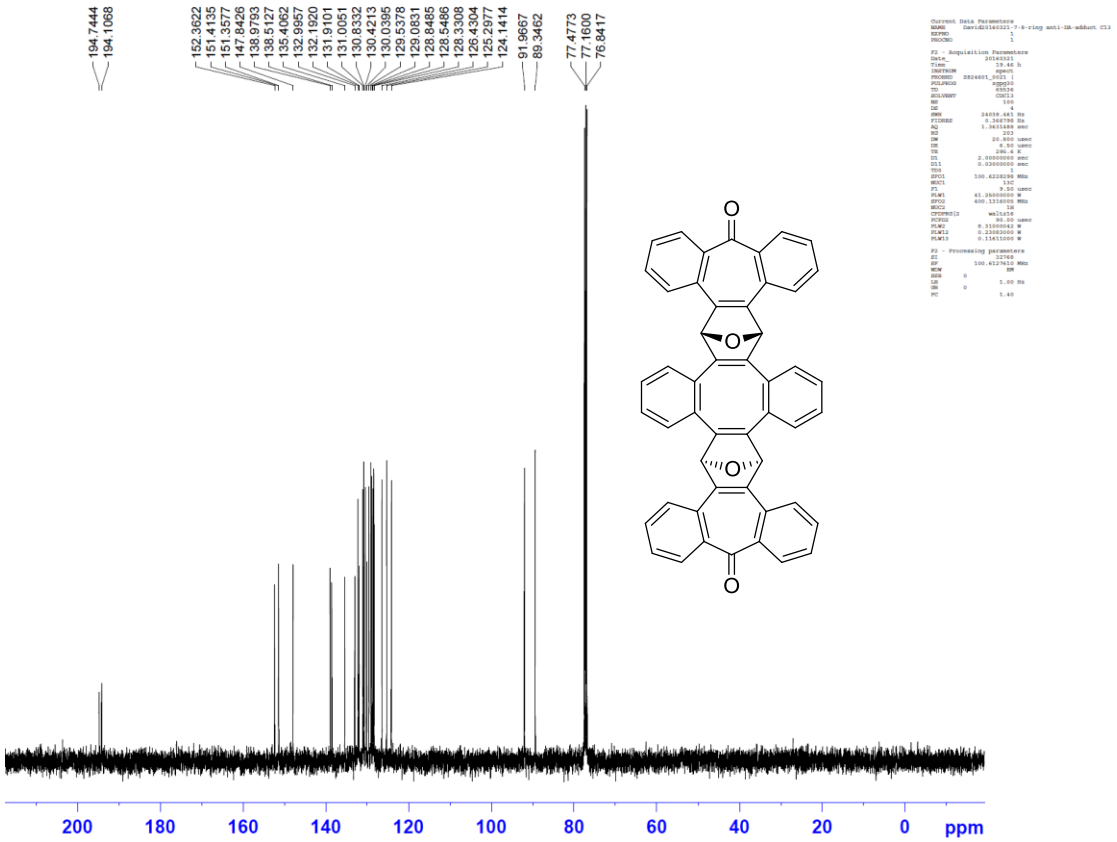
^{1}H NMR spectrum of $Fe(CO)_3$ **(10)**



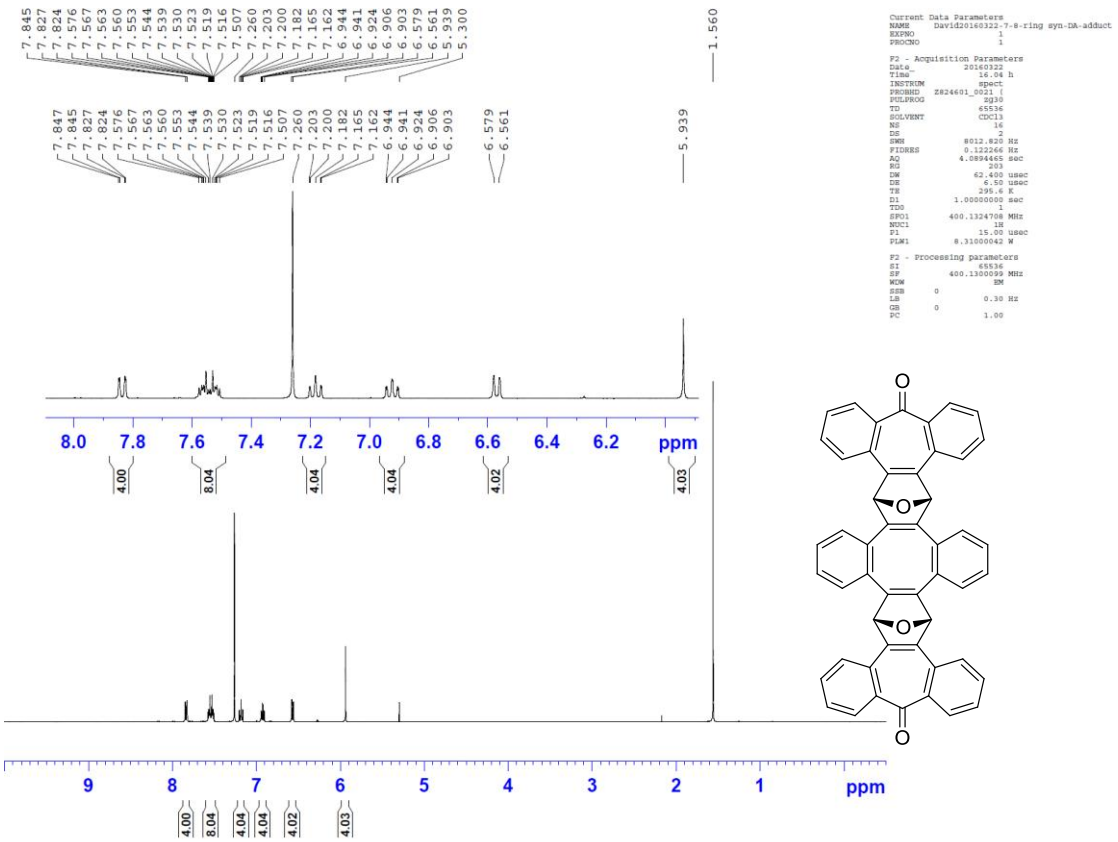
^{13}C NMR spectrum of $Fe(CO)_3$**(10)**



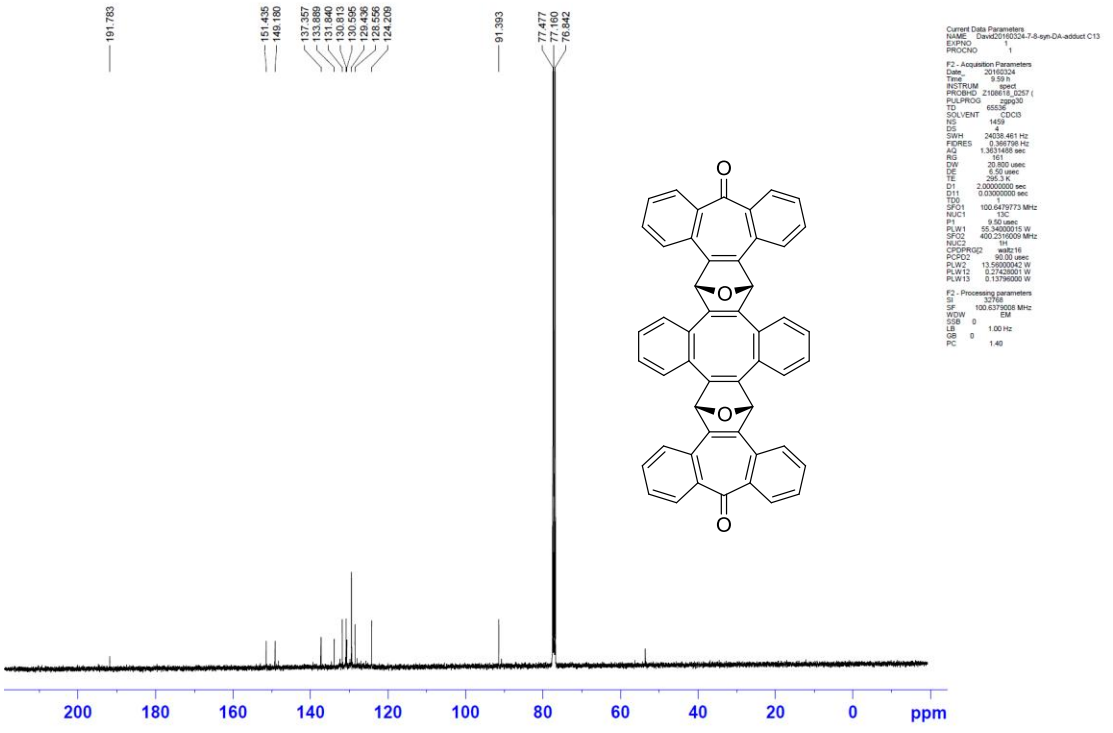
^{1}H NMR spectrum of *anti*-**12**



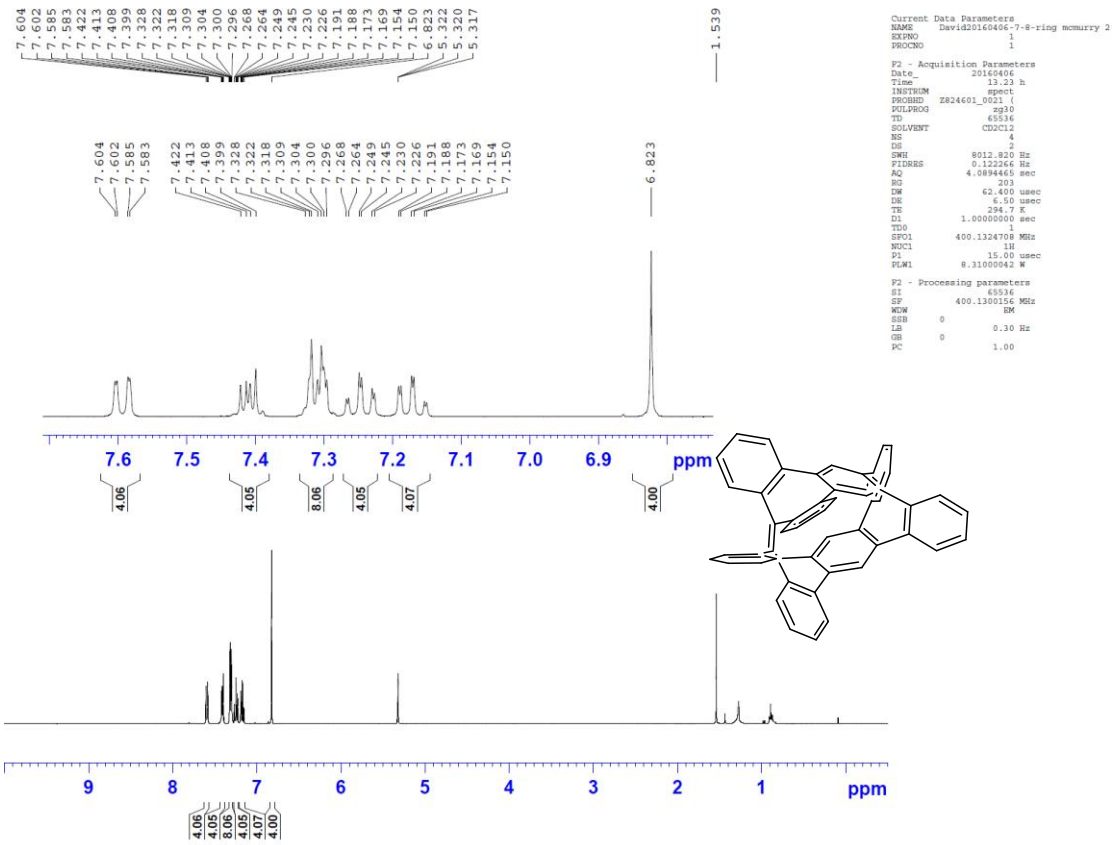
^{13}C NMR spectrum of *anti*-**12**



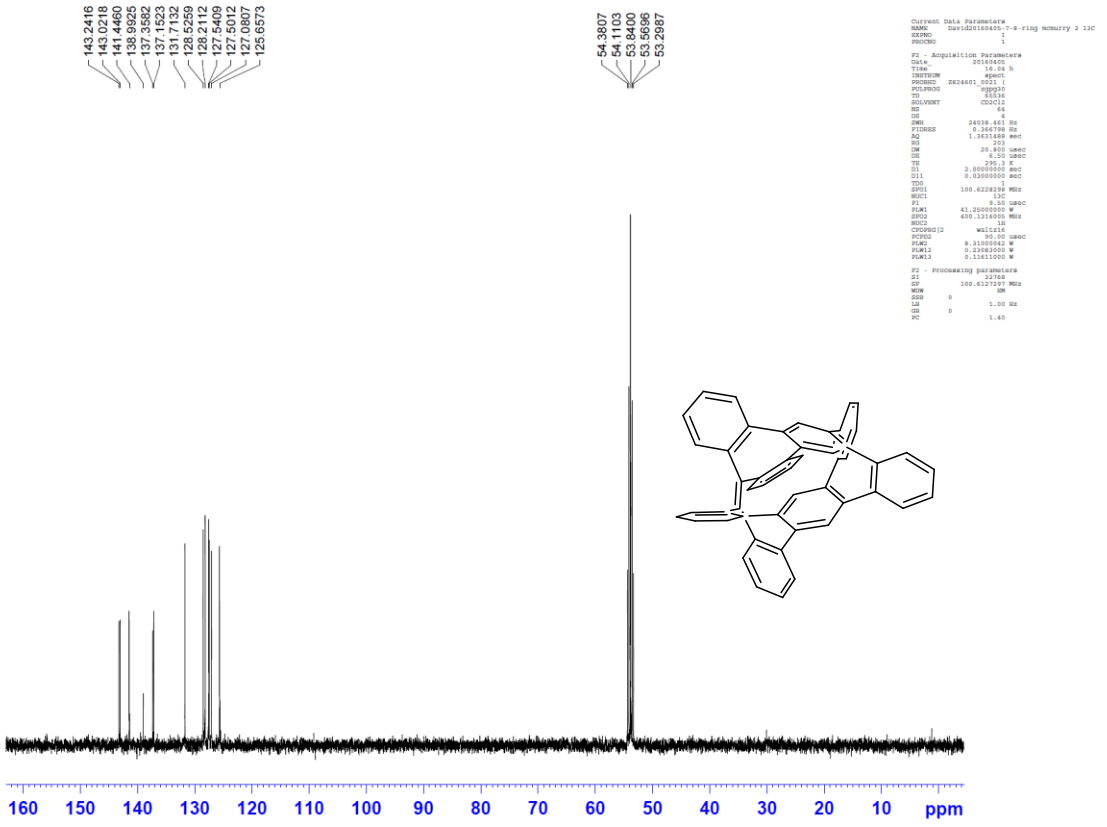
^{1}H NMR spectrum of *syn*-**12**



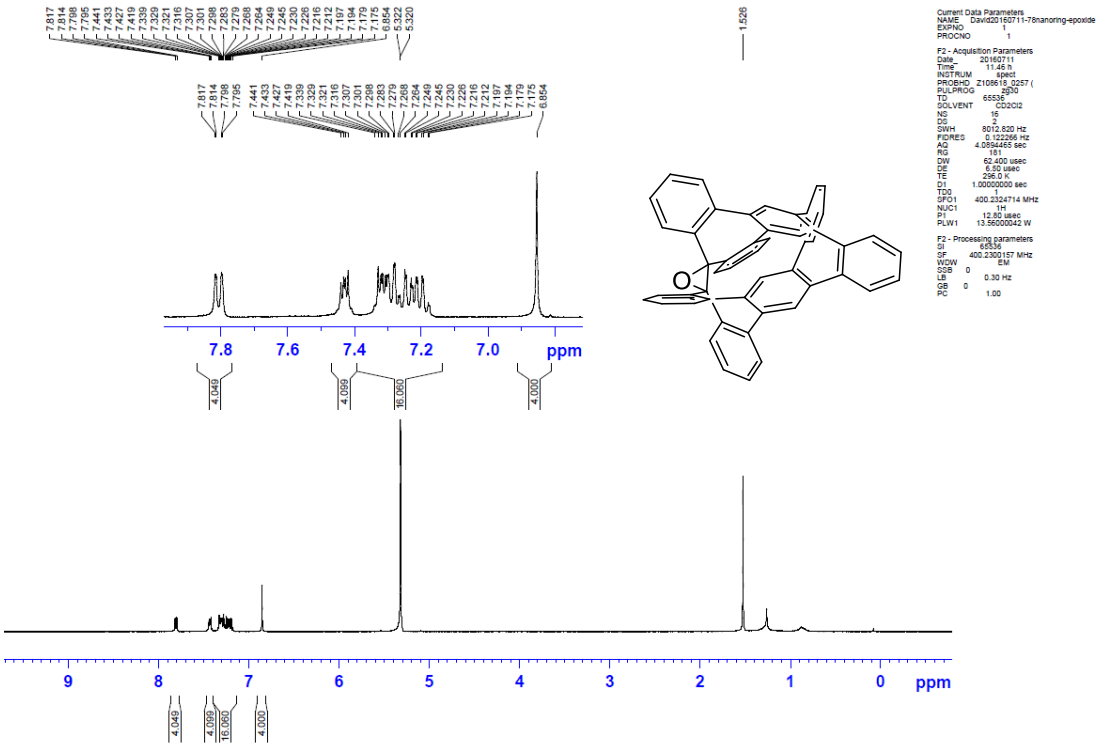
^{13}C NMR spectrum of *syn*-**12**



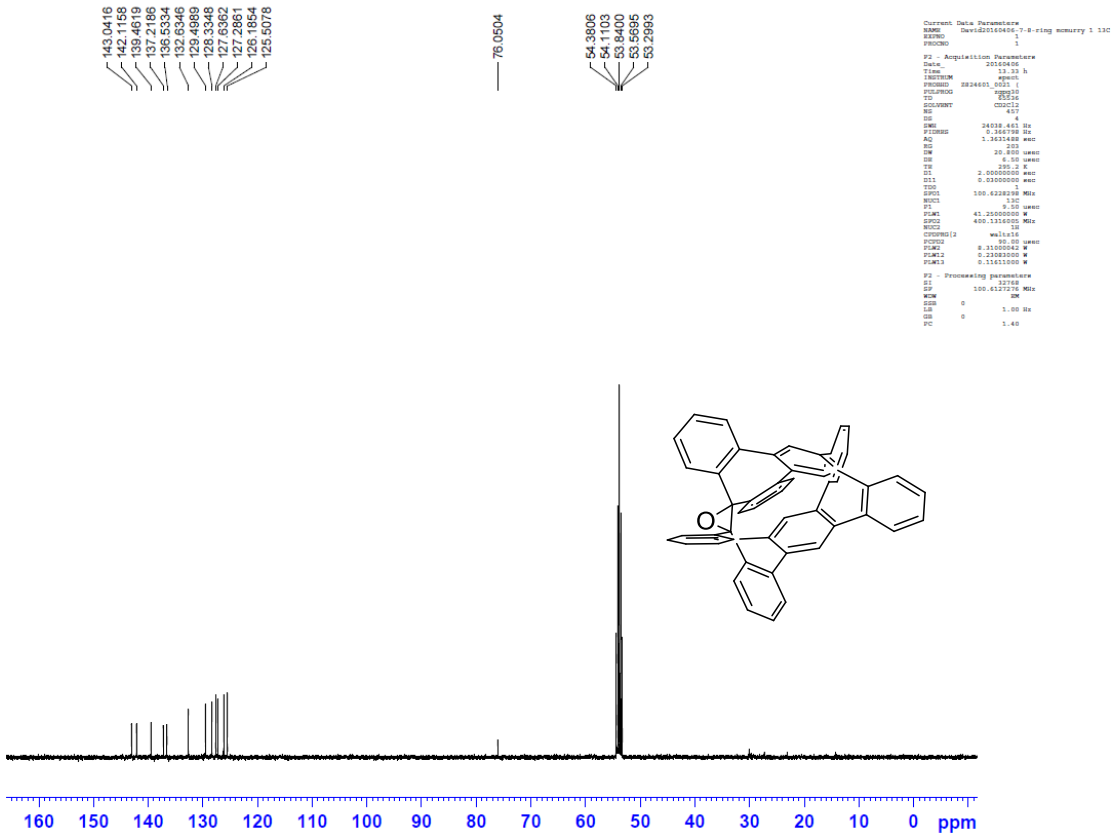
^{1}H NMR spectrum of **3**



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



^{1}H NMR spectrum of **13**



^{13}C NMR spectrum of **13**