Selective Deprotonation of Tetra[3,4]thienylene in the Presence of *n*-BuLi

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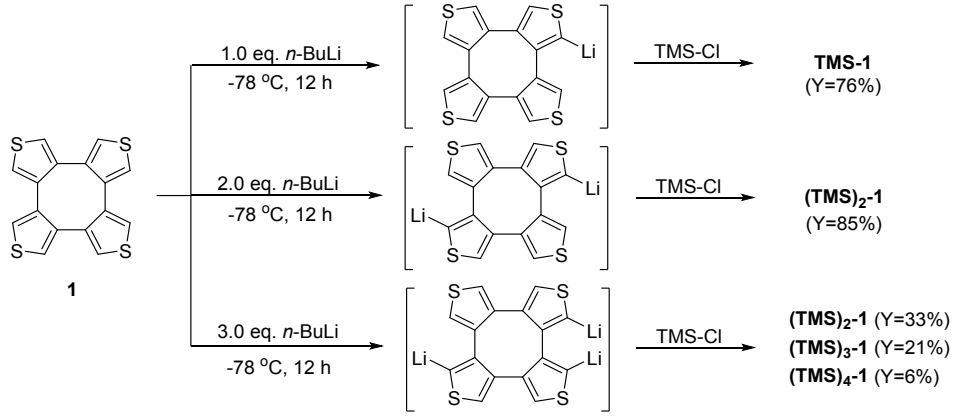
Experimental Section

Materials and general methods Unless otherwise noted, all starting materials were commercially available and were used without further purification. Ether, tetrahydrofuran (THF) and hexane for use were freshly distilled from sodium/benzophenone prior to use. Concentration of *n*-BuLi (hexane) was determined by titration with N-pivaloyl-o-toluidine.[S1] Column chromatography was carried out on silica gel (300-400 mesh). Analytical thin-layer chromatography was performed on glass plates of Silica Gel GF-254 with detection by UV. Tetra[3,4]thienylene (**1**) and 2,2'-dibrome-5,5'-trimethylsilyl-3,3'-bithiophene (**2**) was synthesized by the reported procedure, respectively.[S2, S3] All reactions were carried out under a dry and oxygen free argon atmosphere in slight positive pressure by using Schlenk techniques or under an argon atmosphere in glovebox. The argon in the glovebox was constantly circulated through a copper/molecular sieve catalyst unit. The oxygen and moisture concentrations in the glovebox atmosphere were monitored by an O_2/H_2O Combi-Analyzer to ensure both were always below 1 ppm. ^1H and ^{13}C NMR spectra were obtained using C_6D_6 (dried over fresh potassium chips in the glovebox) or $CDCl_3$ as solvent and recorded on 400 MHz or 300 MHz for ^1H and 100 MHz or 75 MHz for ^{13}C spectrometer at room temperature.

X-ray crystallographic analysis The X-ray crystallographic analyses were performed using crystals of compounds **3** and **(TMS)$_2$-1** with the size 0.41×0.25×0.23 mm and 0.39×0.22×0.18 mm, respectively. The intensity data were collected with the ω scan mode (207K) on a diffractometer with CCD detector using Mo Kα radiation (λ = 0.71073 Å). The data were corrected for Lorentz and polarisation effects and absorption corrections were performed using SADABS program.[S4] The crystal structures were solved using the SHELXTL program and refined using full matrix least squares.[S5] The positions of hydrogen atoms were calculated theoretically and included in the final cycles of refinement in a riding model along with attached carbons. Further details are in the deposited CIFs. Slow evaporation of solutions of **3** in dry hexane and **(TMS)$_2$-1** in $CHCl_3$-CH_3OH (6:1 v/v) were employed for growing single crystals.

Typical procedure for the separated preparations of TMS-1, $(TMS)_2$-1 and $(TMS)_3$-1 by using different equivalents of n-BuLi.



Scheme 1S. Formation of Li-**1** to Li_4-**1** from **1** in the presence of *n*-BuLi, and their quenching products.

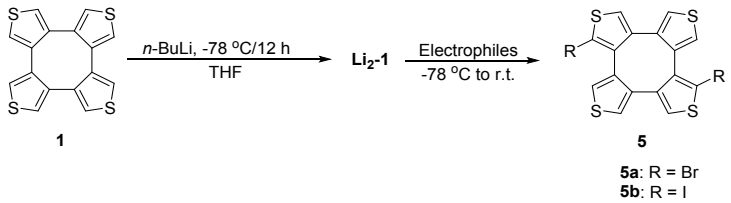
n-BuLi was added dropwise at -78 °C to a solution of **1** (50 mg, 0.15 mmol) in dry THF (5 mL). The reaction mixture was kept at -78 °C for 12 h. TMS-Cl was added and allowed to warm to room temperature slowly overnight. The reaction mixture was quenched with saturated $NaHCO_3$, extracted with dichloromethane (3×10 mL), and finally dried over anhydrous $MgSO_4$. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel via gradient elution using petroleum ether as eluent.

n-BuLi (0.06 mL, 0.15 mmol, 2.40 M in hexane, 1.0 equiv) and TMS-Cl (0.03 mL, 0.23 mmol) were used to yield **TMS-1** (white solid) 46 mg (76% yield); m.p. 194-196 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39 (s, 1H), 7.22 (s, 1H), 7.18-7.15 (br, 5H), 0.1 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.64, 139.45, 139.06, 138.33, 137.83, 137.62, 137.50, 137.43, 128.82, 125.52, 124.28, 123.77, 123.70, 123.42, 0.47; HRMS (EI^+): m/z calcd for [$C_{19}H_{16}S_4Si$] 399.9904, found 399.9909.

n-BuLi (0.12 mL, 0.31 mmol, 2.51 M in hexane, 2.0 equiv) and TMS-Cl (0.05 mL, 0.38 mmol) were used to yield **$(TMS)_2$-1** in 85%.

n-BuLi (0.19 mL, 0.46 mmol, 2.40 M in hexane, 3.1 equiv) and TMS-Cl (0.08 mL, 0.61 mmol) were used to yield **$(TMS)_3$-1** (21%), **$(TMS)_2$-1** (33%) and **$(TMS)_4$-1** (6%) at same time.

Typical procedure for the preparation of 5a and 5b from 1.



Scheme 2S. The synthetic routes to **5a** and **5b**.

Synthesis of 5a.

n-BuLi (0.12 mL, 0.31 mmol, 2.51 M in hexane) was added dropwise at -78 °C to a solution of **1** (50 mg, 0.15 mmol) in dry THF (5 mL). After 12 h, $Br_2Cl_4C_2$ (123.9 mg, 0.38 mmol)was added and allowed to warm to room temperature slowly overnight. The reaction mixture was quenched with water, extracted with dichloromethane (3×20 mL), and finally dried over anhydrous $MgSO_4$. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel via gradient elution using the solvent (petroleum ether) as eluent to yield **5a** as white solid, 58.3 mg (79% yield); m.p. > 300 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, J = 4.0 Hz, 2H), 7.16 (d, J = 4.0 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.38, 137.17, 136.11, 134.23, 126.98, 124.77, 124.21, 111.93; HRMS (EI^+): m/z calcd for [$C_{16}H_6S_4Br_2$] 483.7719, found 483.7712.

Synthesis of 5b.

n-BuLi (0.12 mL, 0.31 mmol, 2.51 M in hexane) was added dropwise at -78 °C to a solution of **1** (50 mg, 0.15 mmol) in dry THF (5 mL). After 12 h, I_2 (96.6 mg, 0.38 mmol) was added and allowed to warm to room temperature slowly overnight. The reaction mixture was quenched with saturated sodium thiosulfate, extracted with dichloromethane (3×20 mL), and finally dried over anhydrous $MgSO_4$. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel via gradient elution using the solvent (petroleum ether) as eluent to yield **5b** as white solid, 53.0 mg (60% yield); m.p. > 300 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40 (s, 2H), 7.34 (s, 2H), 7.19 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.46, 137.08, 136.88, 136.15, 129.42, 127.08, 124.63; HRMS (EI^+): m/z calcd for [$C_{16}H_6S_4I_2$] 579.7442, found 579.7444.

NMR, IR spectra and HRMS data

NMR spectra of 3

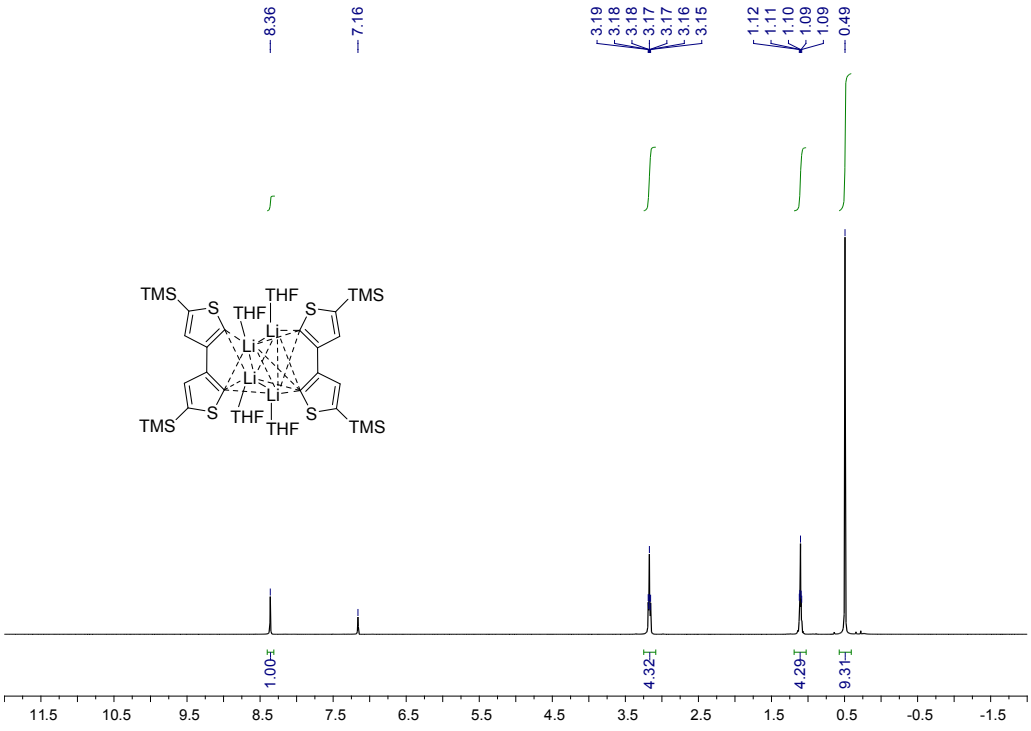


Figure S1. ^{1}H NMR (400 MHz, C_6D_6) spectrum of **3**

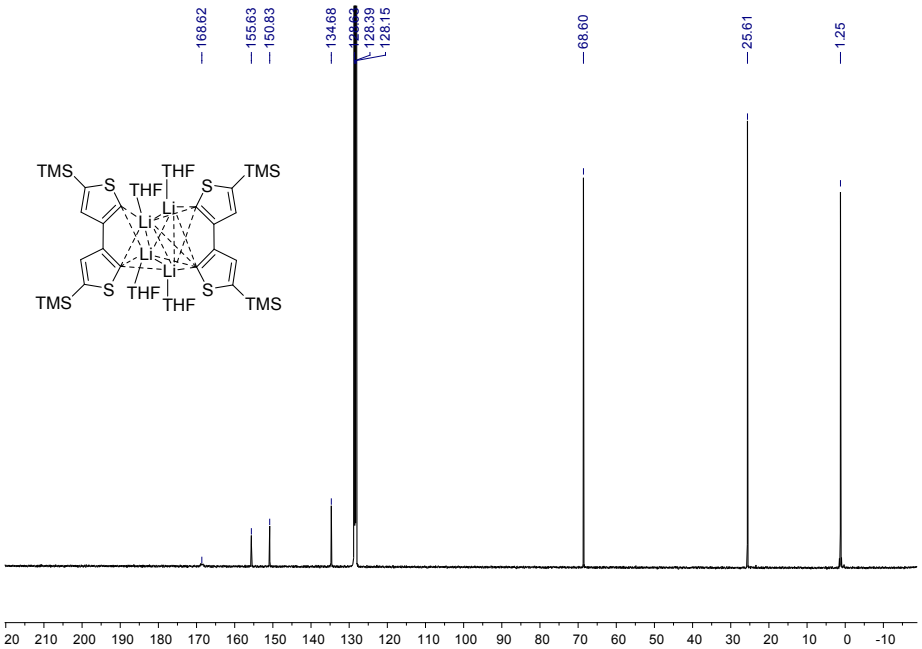


Figure S2. ^{13}C NMR (100 MHz, C_6D_6) spectrum of **3**

NMR, IR spectra and HRMS data of TMS-1

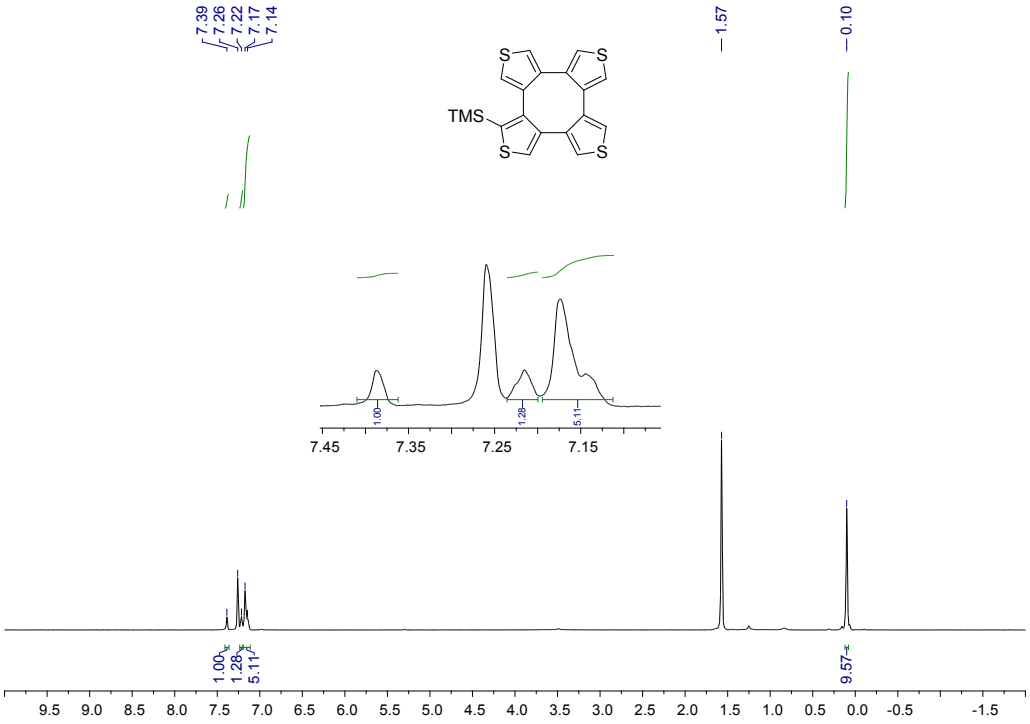


Figure S3. ^{1}H NMR (300 MHz, $CDCl_3$) spectrum of **TMS-1**

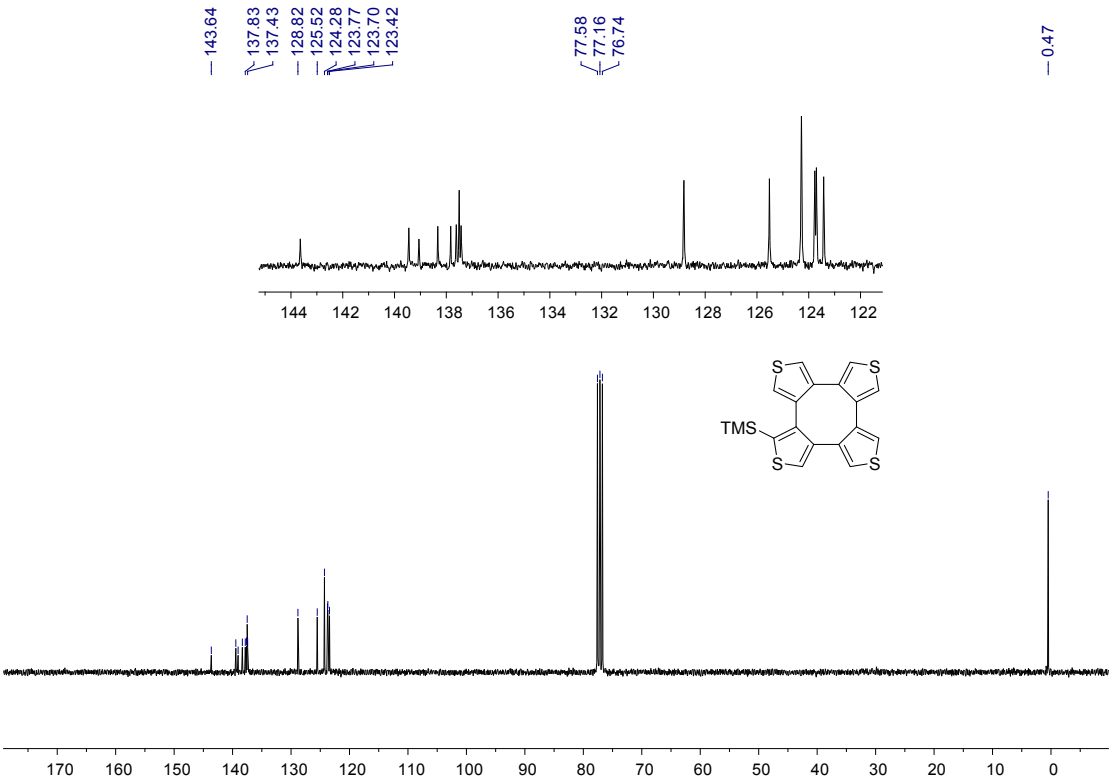


Figure S4. ^{13}C NMR (75 MHz, $CDCl_3$) spectrum of **TMS-1**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

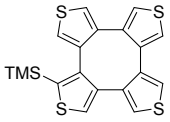
Instrument: Waters Micromass GCT Premier

Sample Serial Number: LL-3-28B-col-PTLC-PP

Operator: Li, Guangping

Date: 2016/07/20

Operation Mode: EI-Positive



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; N: 0-2; O: 0-4; S: 0-4; Si: 0-1

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
399.9909	399.9904	1.3	13.0	C19H16S4Si
399.9909	399.9925	-4.0	18.5	C22H10NOS3
399.9909	399.9898	2.8	14.0	C19H12O4S3
399.9909	399.9922	-3.3	18.5	C21H10NO2S2Si
399.9909	399.9891	4.5	23.5	C25H6NOS2
399.9909	399.9909	0.0	29.0	C27N2O3

Figure S5. HRMS data of **TMS-1**

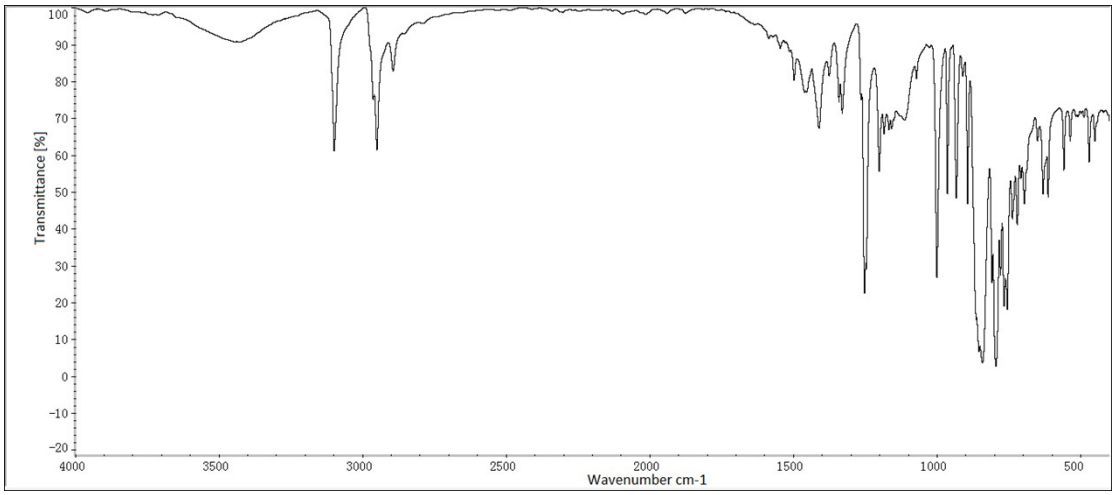


Figure S6. IR spectrum of **TMS-1**

NMR, IR spectra and HRMS data of $(TMS)_2$-1

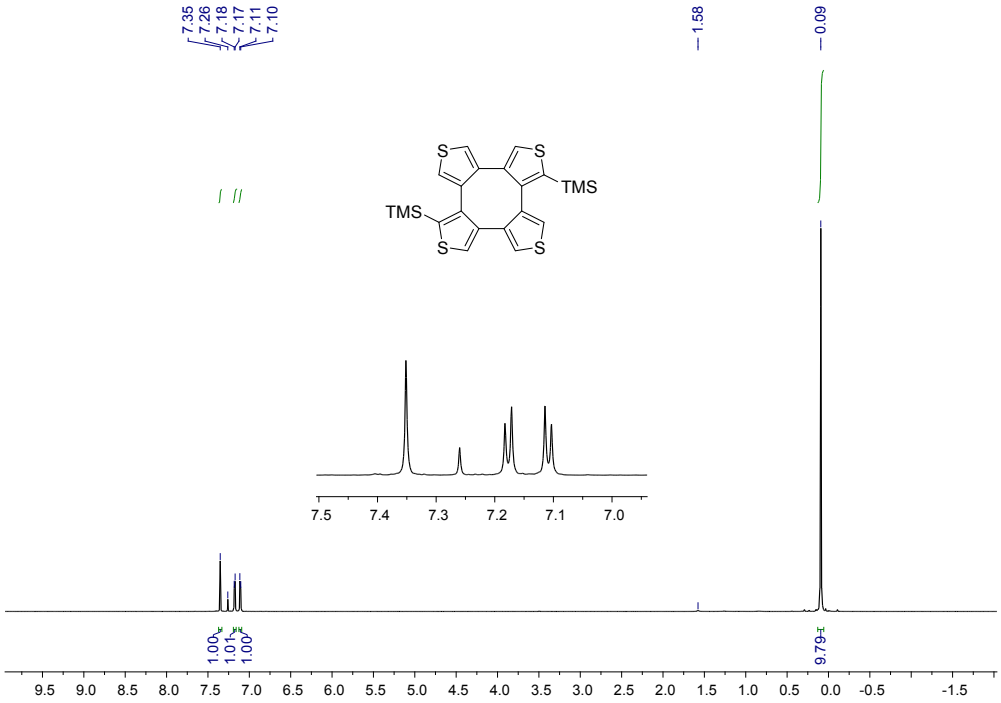


Figure S7. ^{1}H NMR (300 MHz, $CDCl_3$) spectrum of **$(TMS)_2$-1**

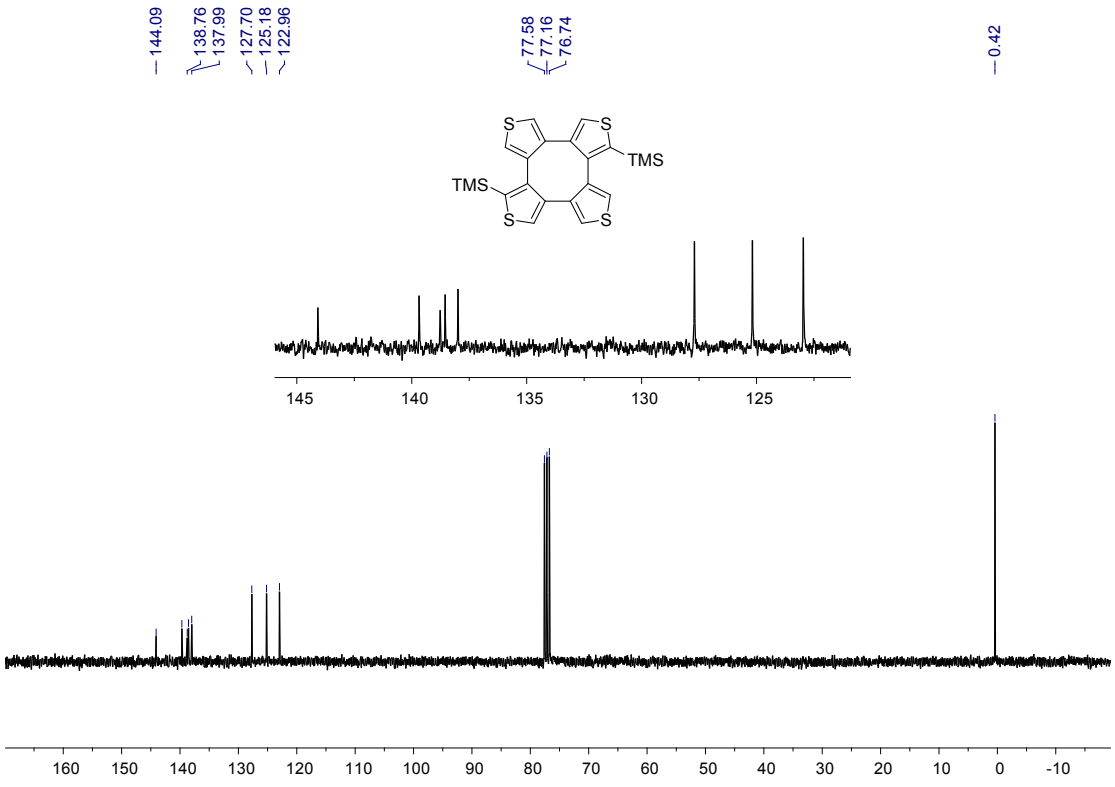
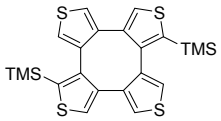


Figure S8. ^{13}C NMR (75 MHz, $CDCl_3$) spectrum of **$(TMS)_2$-1**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Thermo Fisher Scientific LTQ FT Ultra

Card Serial Number : M152129



Sample Serial Number: LL-2-115-pp

Operator : HUAQIN Date: 2014/10/10

Operation Mode: MALDI-FT_DHB

Elemental composition search on mass 473.04

m/z= 468.04-478.04

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
473.0367	473.0372	-1.02	12.5	$C_{22}H_{25}S_4Si_2$
	473.0361	1.30	23.0	$C_{29}H_{15}NS_3$
	473.0359	1.79	23.0	$C_{28}H_{15}ONS_2Si$
	473.0357	2.27	23.0	$C_{27}H_{15}O_2NSSi_2$
	473.0379	-2.53	28.5	$C_{31}H_9O_2N_2S$
	473.0386	-3.90	37.5	$C_{39}H_5$
	473.0346	4.60	33.5	$C_{34}H_5O_2N_2$
	473.0390	-4.85	18.0	$C_{24}H_{19}O_2NS_2Si_2$

Figure S9. HRMS data of **$(TMS)_2$-1**

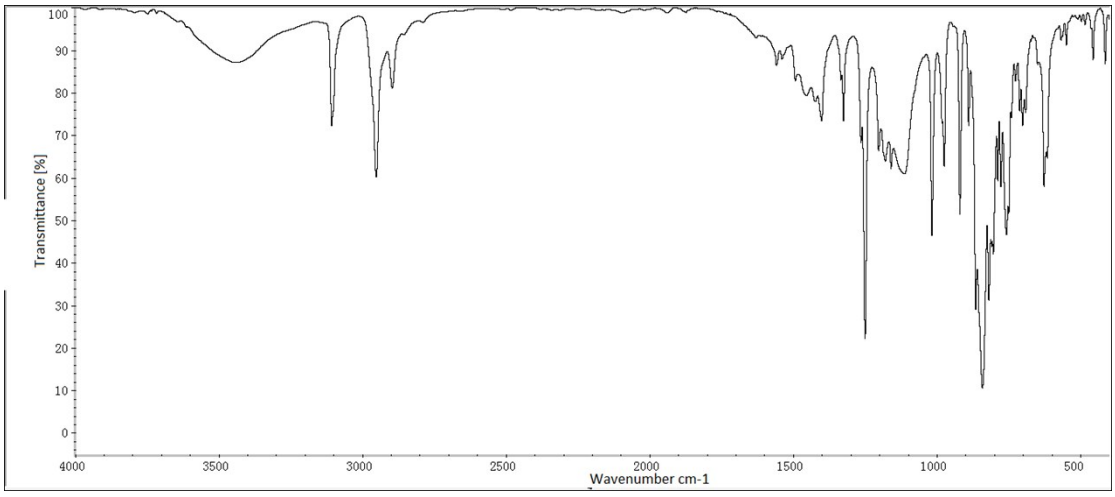


Figure S10. IR spectrum of **$(TMS)_2$-1**

NMR, IR spectra and HRMS data of $(TMS)_3$-1

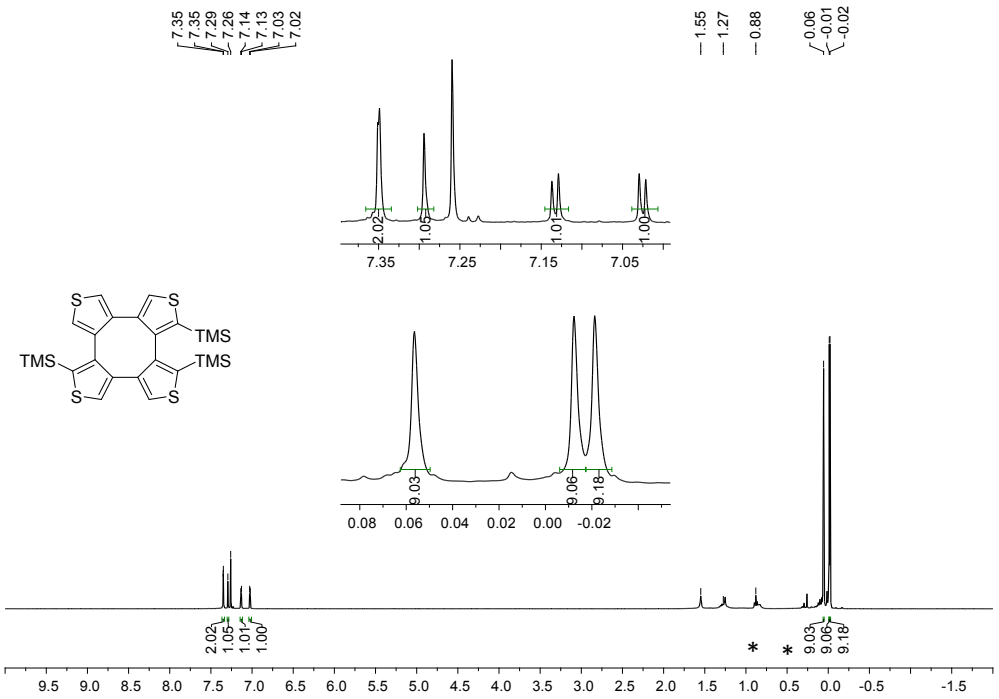


Figure S9. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **$(TMS)_3$-1** (∗ impurity from pentane)

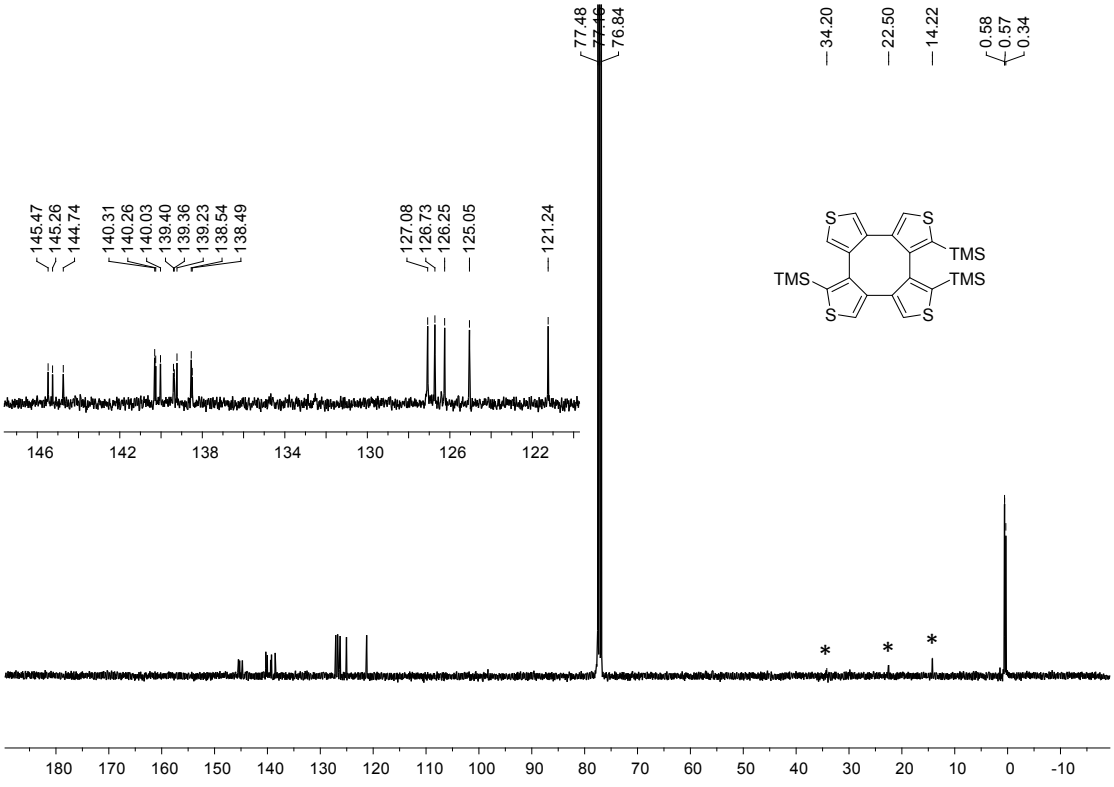
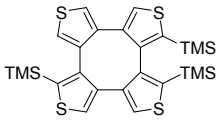


Figure S10. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **$(TMS)_3$-1** (∗ impurity from pentane)

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Waters Micromass GCT Premier

Sample Serial Number: LL-3-26B-col-2



Operator: Li, Guangping

Date: 2016/07/01

Operation Mode: EI-Positive

Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; O: 0-4; S: 0-4; Si: 0-3

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
544.0697	544.0695	0.4	13.0	C25H32S4Si3
544.0697	544.0691	1.1	14.0	C26H28O3S4Si
544.0697	544.0688	1.7	14.0	C25H28O4S3Si2
544.0697	544.0708	-2.0	38.0	C42H12Si

Figure S11. HRMS data of **(TMS)$_3$-1**

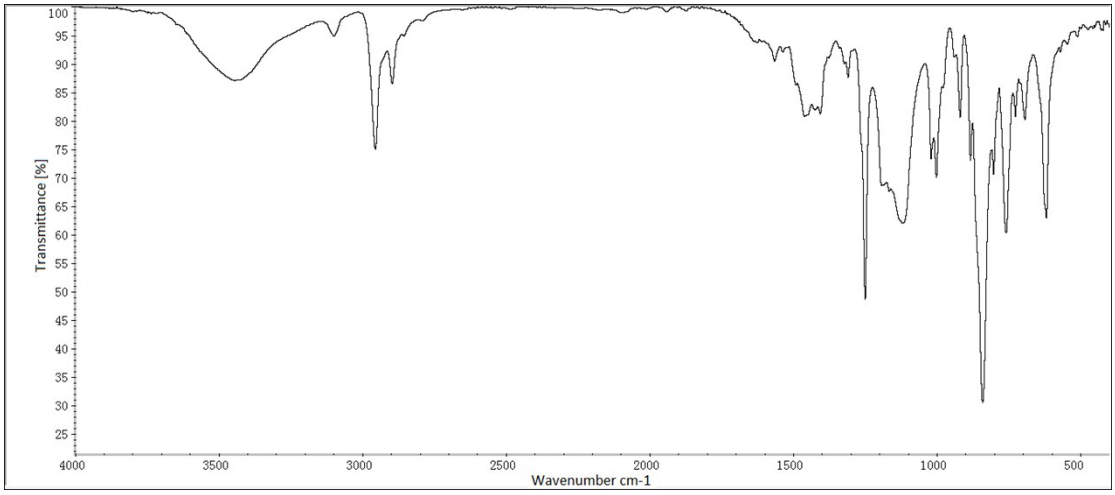


Figure S12. IR spectrum of **(TMS)$_3$-1**

NMR, IR spectra and HRMS data of 4a

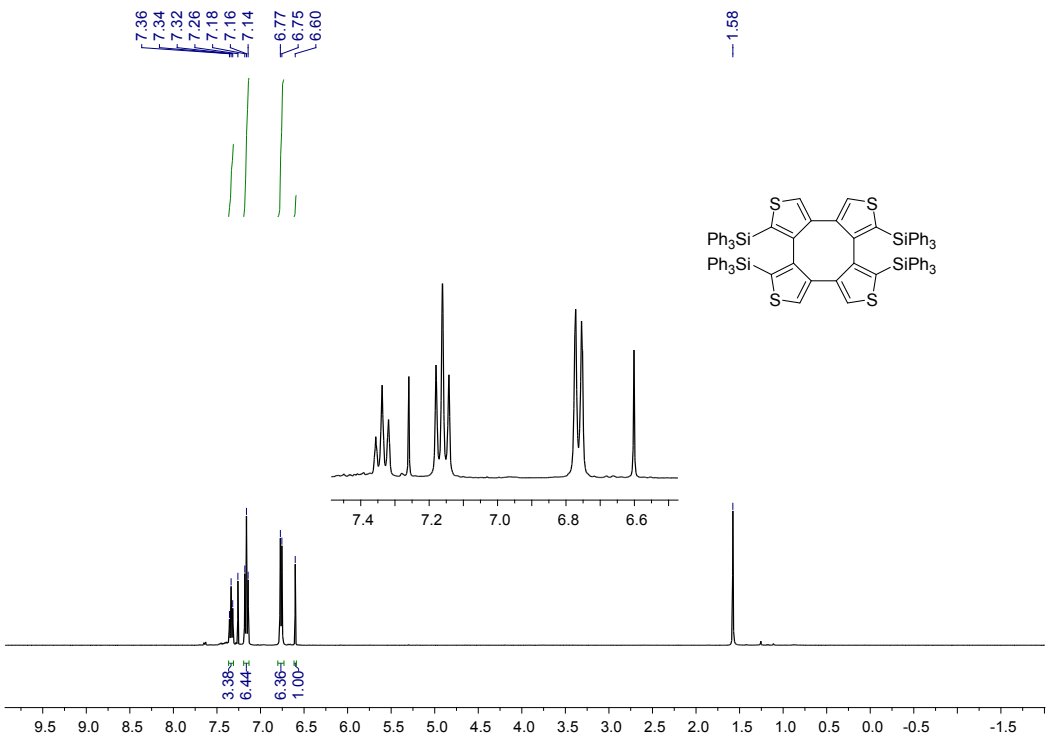


Figure S13. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **4a**

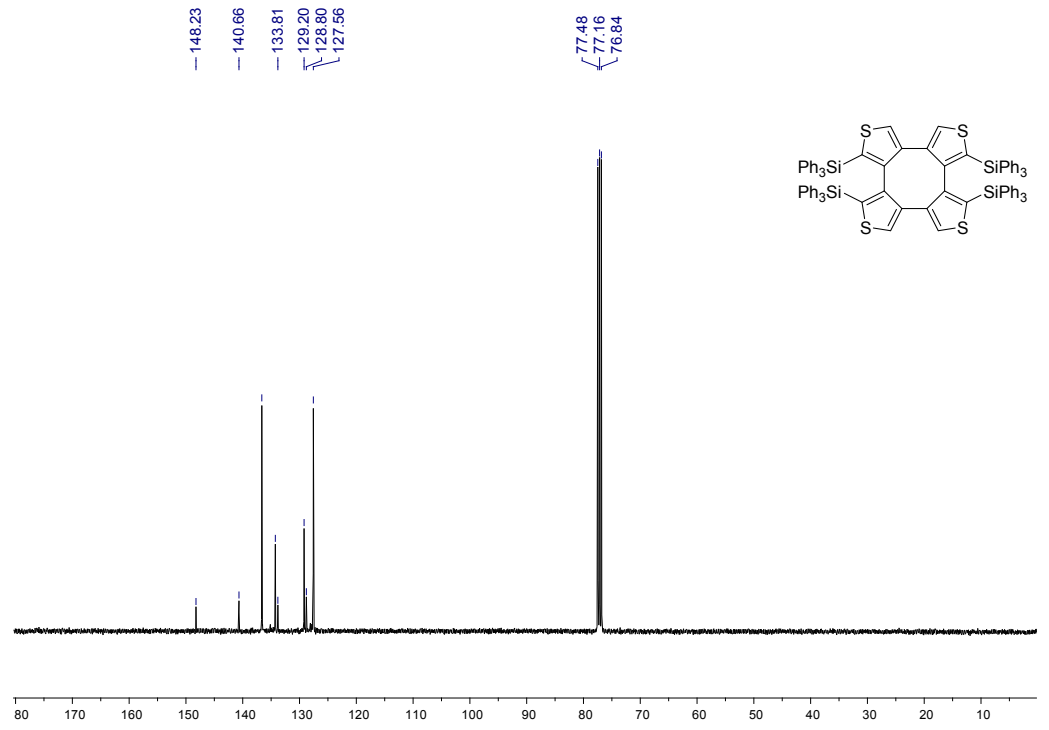
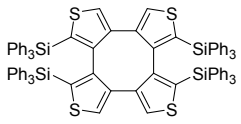


Figure S14. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **4a**

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Thermo Fisher Scientific LTQ FT Ultra

Card Serial Number : M140045



Sample Serial Number: LL-2-16-PP

Operator : HuaQin Date: 2014/09/01

Operation Mode: MALDI_DHB

Elemental composition search on mass 1360.30

m/z= 1355.30-1365.30

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
1360.2960	1360.2962	-0.21	61.0	$C_{88}H_{64}S_4Si_4$
	1360.2996	-2.69	56.0	$C_{85}H_{68}S_5Si_4$
	1360.3145	-13.60	61.0	$C_{90}H_{64}S_5Si_2$
	1360.2601	26.37	62.0	$C_{88}H_{60}S_5Si_3$
	1360.3324	-26.79	60.0	$C_{88}H_{68}S_3Si_5$
	1360.3358	-29.26	55.0	$C_{85}H_{72}S_4Si_5$
	1360.3391	-31.74	50.0	$C_{82}H_{76}S_5Si_5$
	1360.2452	37.29	57.0	$C_{83}H_{64}S_5Si_5$
	1360.2419	39.76	62.0	$C_{86}H_{60}S_4Si_5$
	1360.3506	-40.18	60.0	$C_{90}H_{68}S_4Si_3$

Figure S15. HRMS data of **4a**

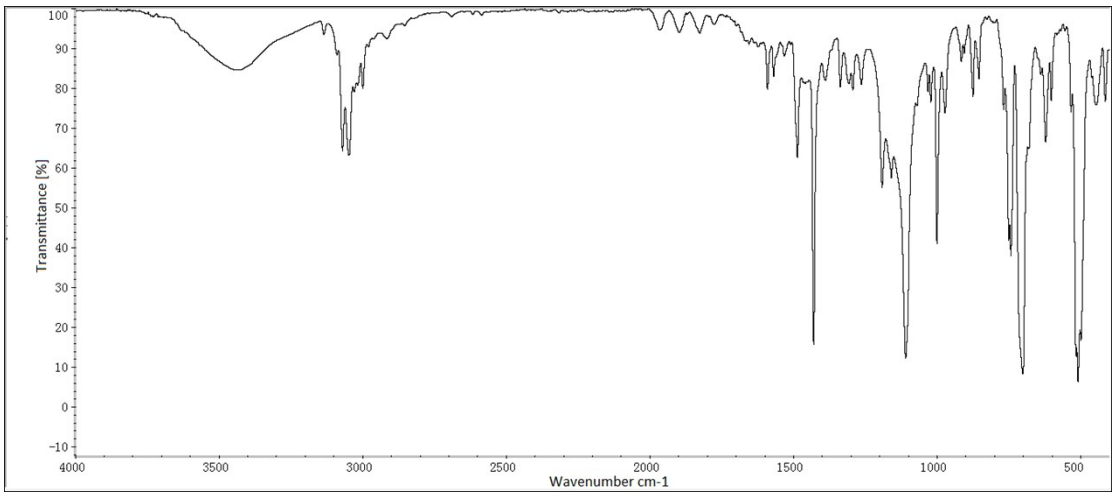


Figure S16. IR spectrum of **4a**

NMR, IR spectra and HRMS data of 4b

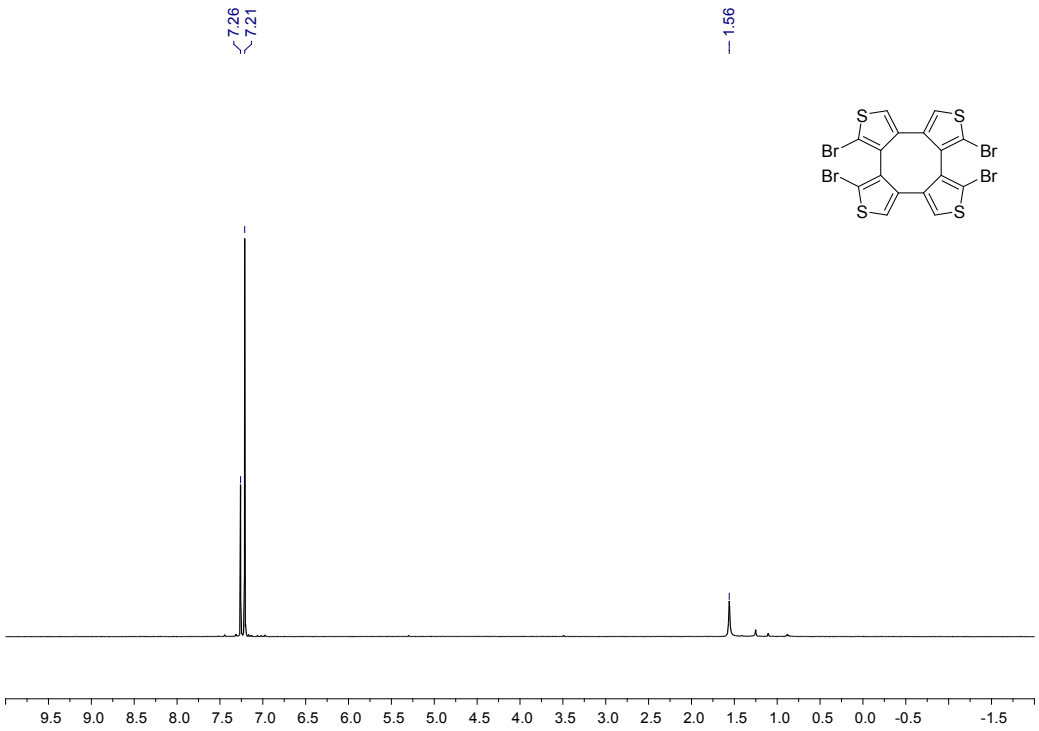


Figure S17. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **4b**

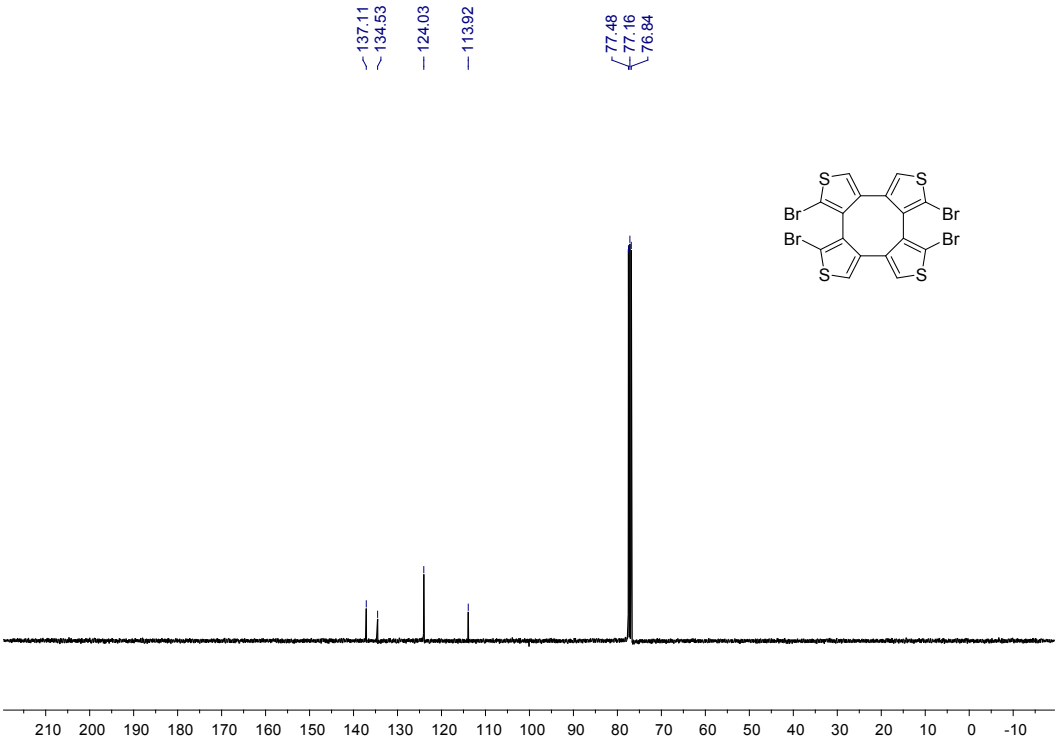


Figure S18. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **4b**

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

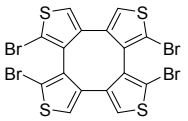
Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T14-06-OS0660

Sample Serial Number: LL-2-14-pp

Operator: Li

Date: 2014/07/14



Elemental Composition Report

Single Mass Analysis
Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
671 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 O: 0-4 S: 0-4 Br: 0-4

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
639.5928	639.5929	-0.1	-0.2	13.0	5546037.5	C16 H4 S4 Br4

Figure S19. HRMS data of **4b**

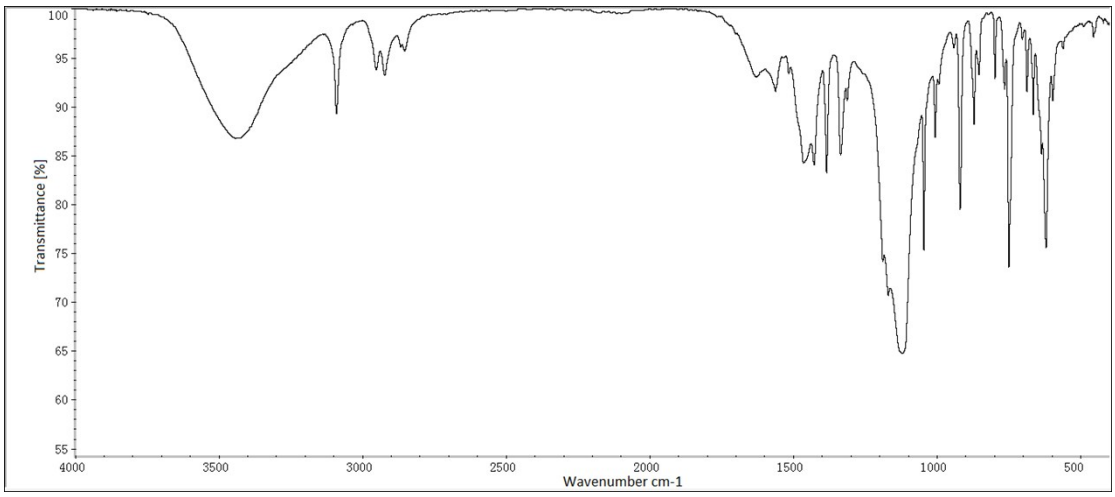


Figure S20. IR spectrum of **4b**

NMR, IR spectra and HRMS data of 4c

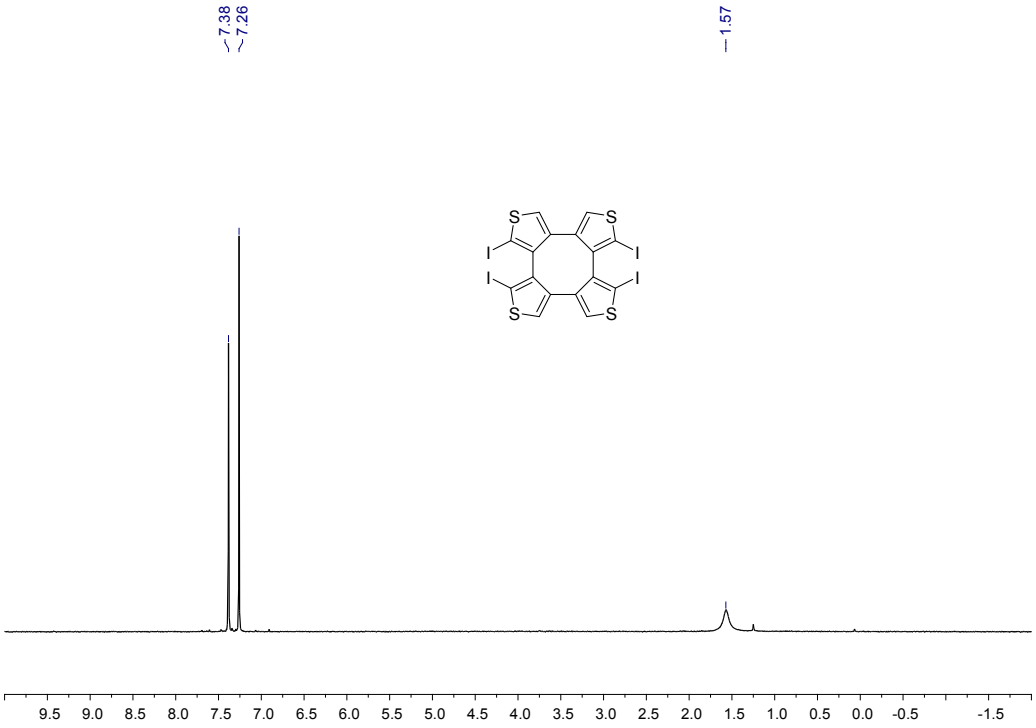


Figure S21. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **4c**

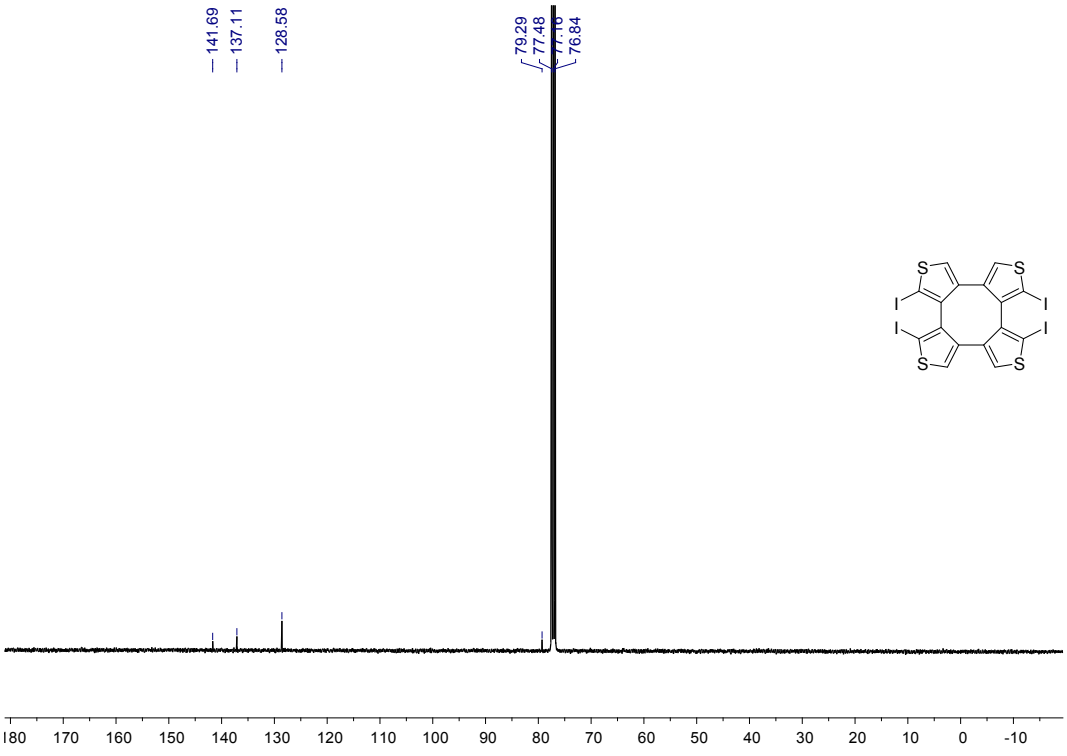


Figure S22. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **4c**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

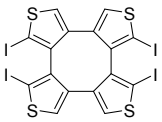
Instrument: Waters Micromass GCT Premier

Sample Serial Number: LL-2-14-col-PP

Operator: Li, Guangping

Date: 2016/07/01

Operation Mode: EI-Positive



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; O: 0-4; S: 0-4; I: 0-4

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
831.5392	831.5375	2.0	13.0	C16H4S4I4
831.5392	831.5386	0.7	18.5	C55H75O4S

Figure S23. HRMS data of **4c**

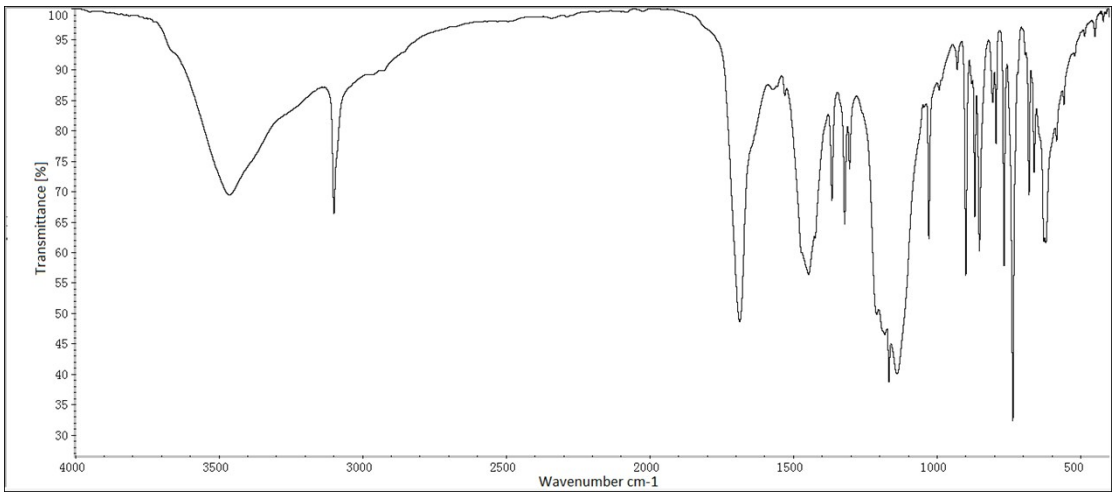


Figure S24. IR spectrum of **4c**

NMR, IR spectra and HRMS data of 4d

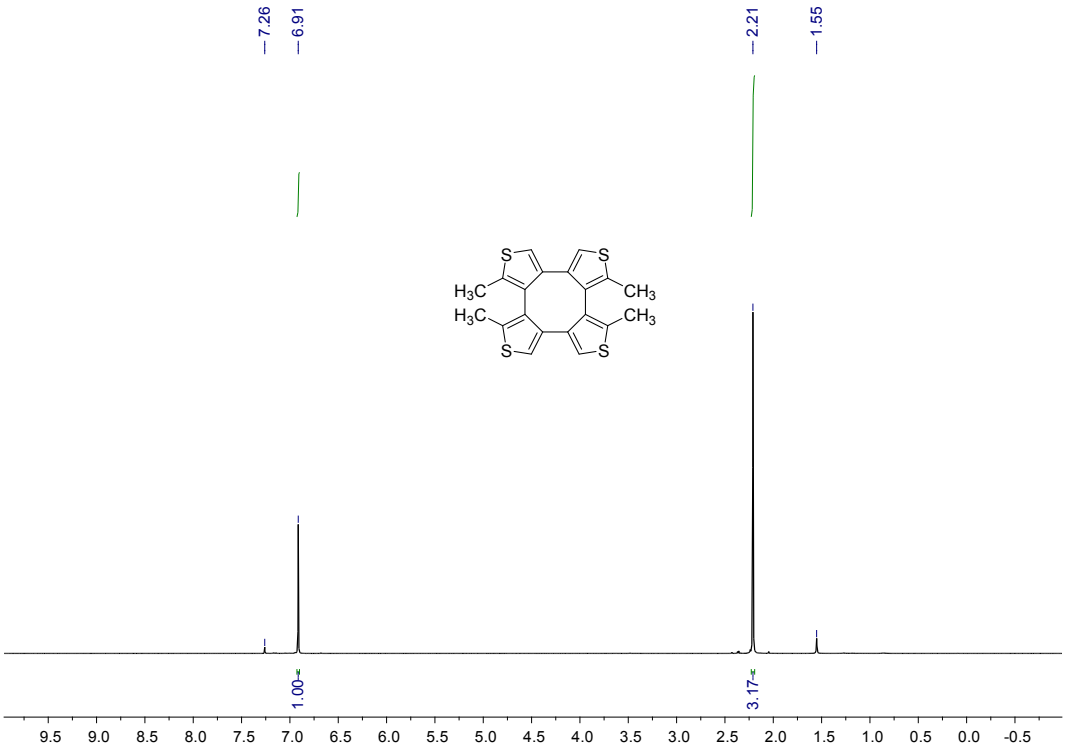


Figure S25. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **4d**

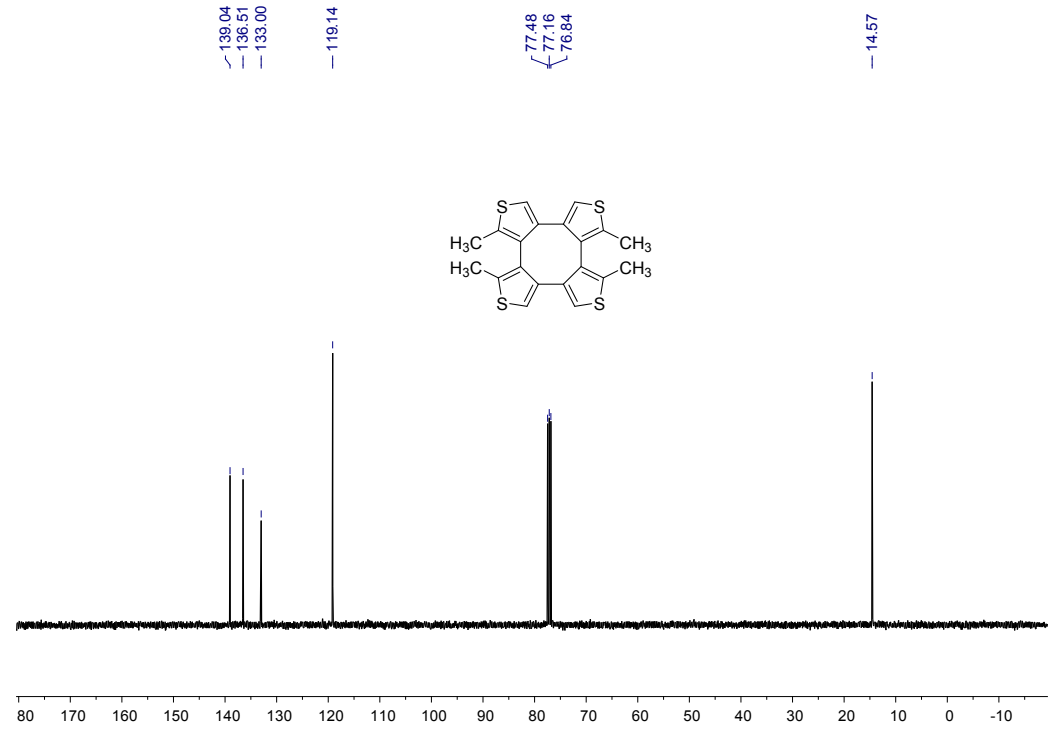
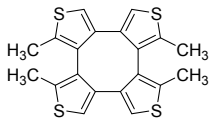


Figure S26. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **4d**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Waters Micromass GCT Premier

Sample Serial Number: LL-2-28-col-PP



Operator: Li, Guangping

Date: 2016/07/01

Operation Mode: EI-Positive

Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; O: 0-4; S: 0-4; Si: 0-3; I: 0-4

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
384.0130	384.0135	-1.3	13.0	C20H16S4
384.0130	384.0133	-0.8	13.0	C19H16OSiS3
384.0130	384.0130	0.0	13.0	C18H16O2Si2S2
384.0130	384.0128	0.5	13.0	C17H16O3Si3S

Figure 27. HRMS data of **4d**

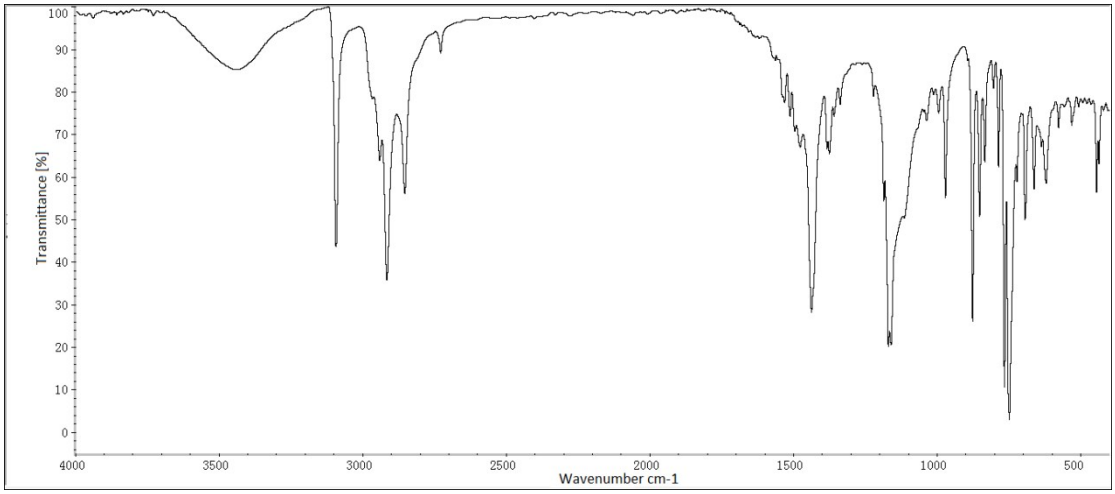


Figure S28. IR spectrum of **4d**

NMR and HRMS data of 5a

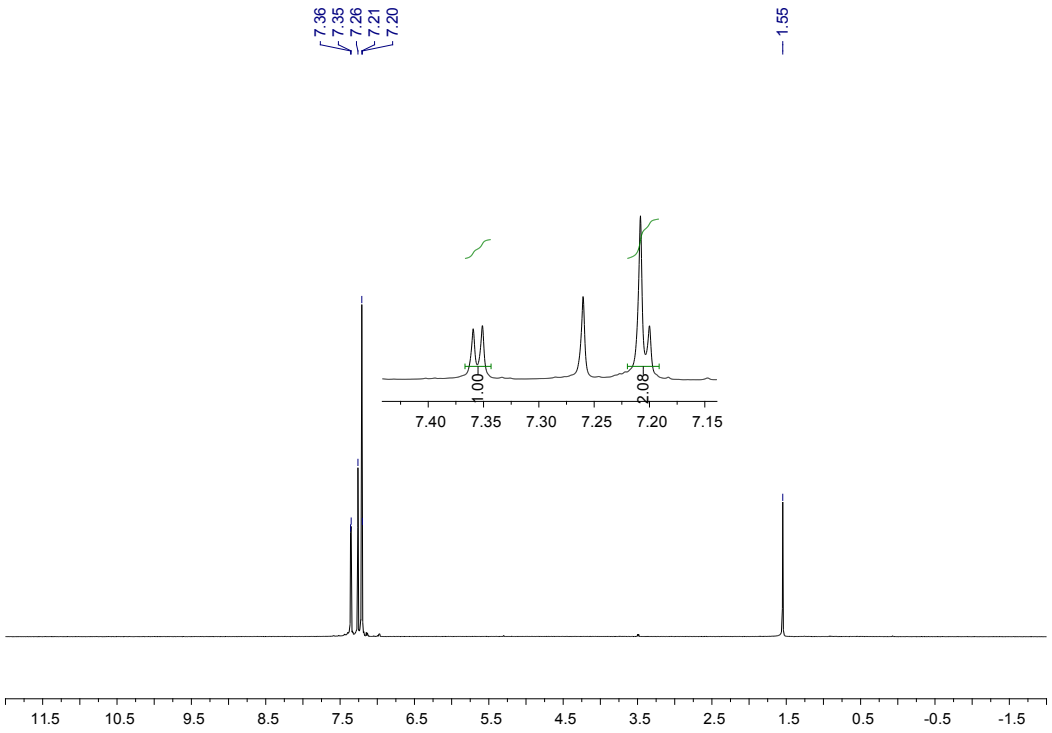


Figure S29. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **5a**

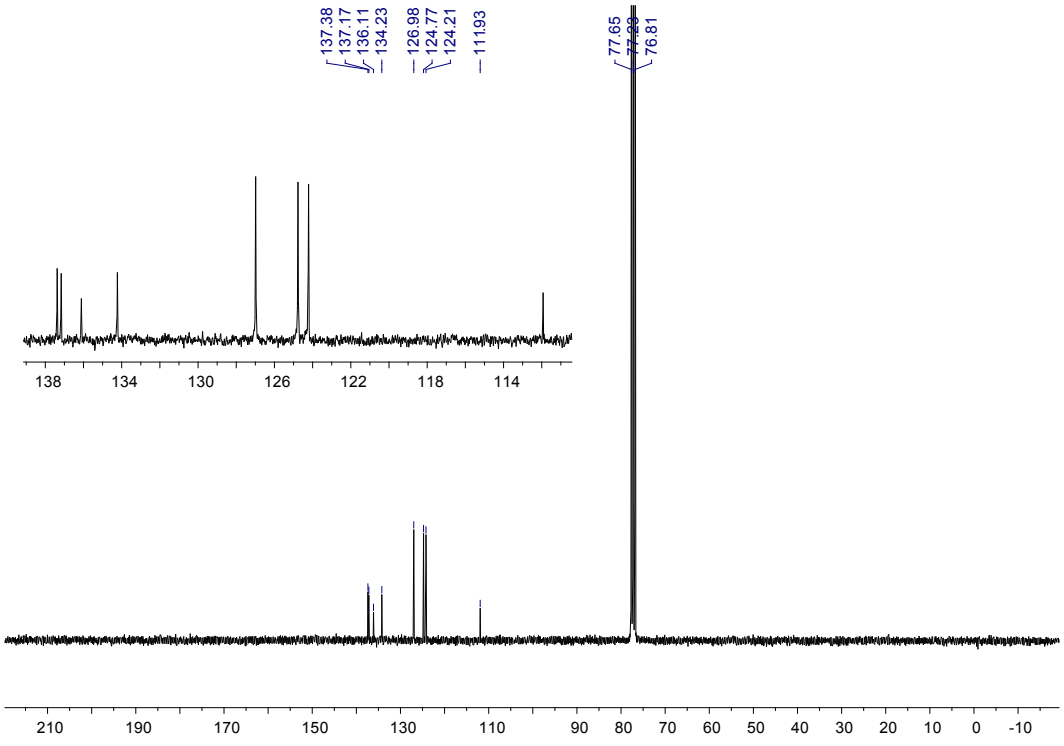


Figure S30. ^{13}C NMR (75 MHz, $CDCl_3$) spectrum of **5a**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Waters Micromass GCT Premier

Sample Serial Number: zcm-2-60-c

Operator: Li, Guangping

Date: 2017/01/16

Operation Mode: EI-Positive

Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; Br: 0-2; N: 0-2; S: 0-4; Cl: 0-1; O: 0-4

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
483.7712	483.7719	-1.4	13.0	C16H6S4Br2
483.7712	483.7715	-0.6	9.5	C12H5NO4S2ClBr2
483.7712	483.7697	3.1	4.0	C10H11O2S4ClBr2

Figure S31. HRMS data of **5a**

NMR and HRMS data of 5b

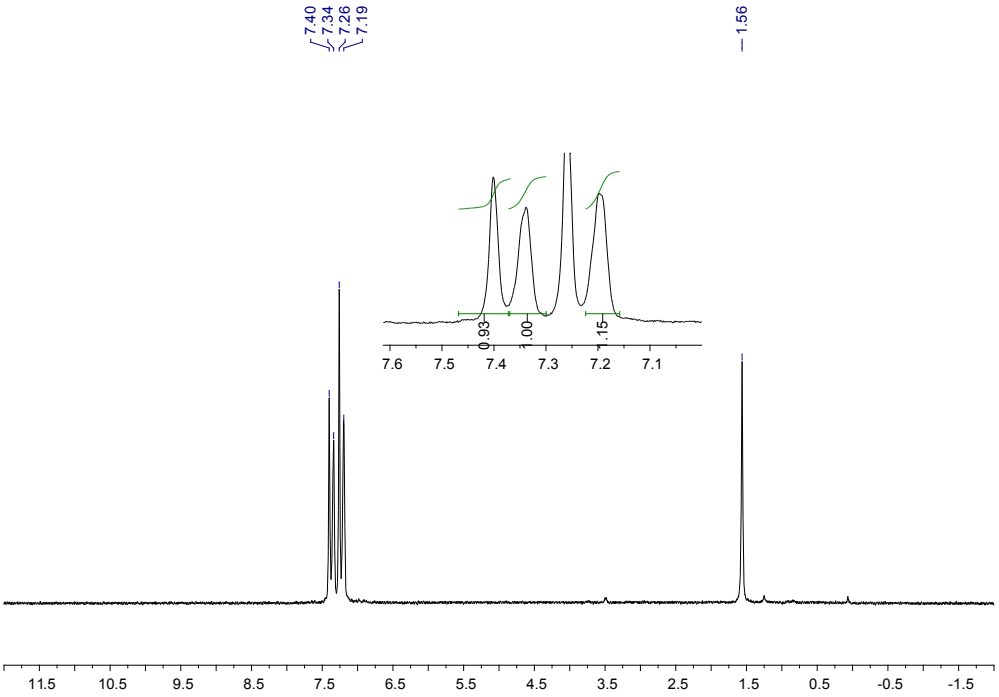


Figure S32. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **5b**

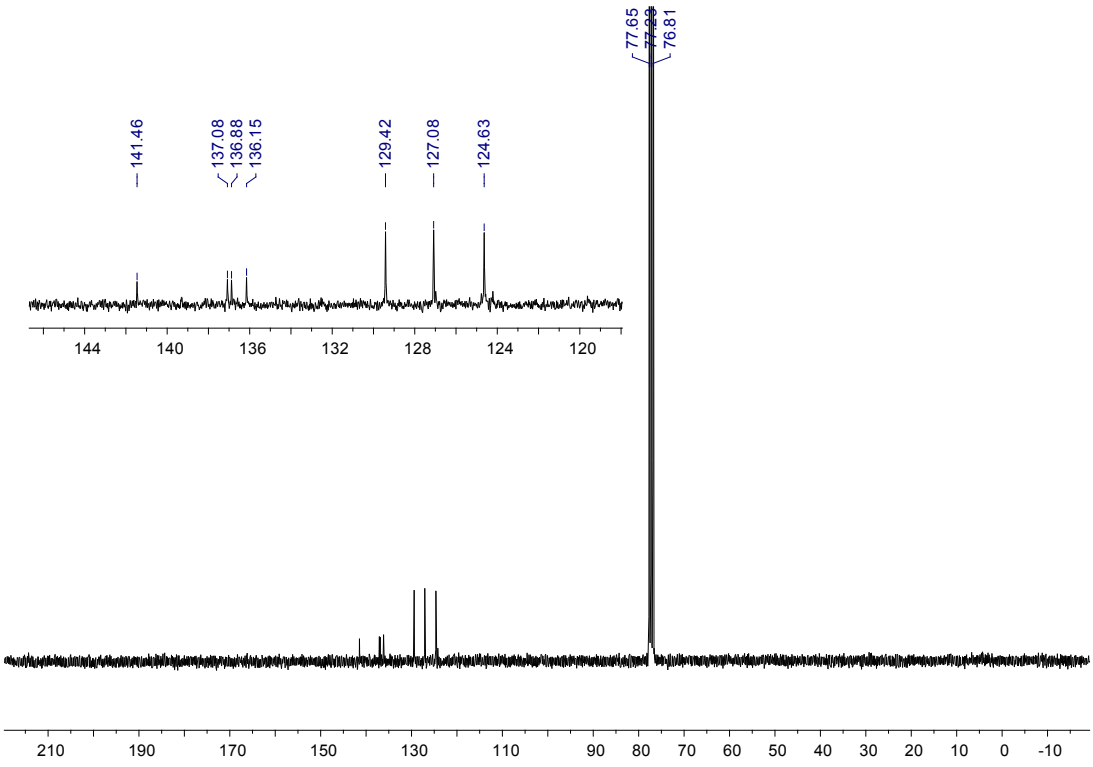


Figure S33. ^{13}C NMR (75 MHz, $CDCl_3$) spectrum of **5b**

National Center for Organic Mass Spectrometry in Shanghai
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Waters Micromass GCT Premier

Sample Serial Number: zcm-2-55-col2-pp

Operator: Li, Guangping

Date: 2017/01/17

Operation Mode: EI-Positive

Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0

C: 0-60; H: 0-80; N: 0-2; S: 0-4; O: 0-6; I: 0-2

Mass	Calc. Mass	Diff (ppm)	DBE	Formula
579.7444	579.7442	0.3	13.0	C16H6S4I2
579.7444	579.7433	1.9	14.0	C15H2O5S2I2
579.7444	579.7460	-2.8	18.5	C18NO2S2I2

Figure S34. HRMS data of **5b**

X-ray crystallographic data

X-ray crystallographic data of 3

Table S1. Crystal data and structure refinement for **3**

Identification code	**3**
Empirical formula	$C_{44}H_{72}Li_4Si_4S_4O_4$
Formula weight	933.37
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Tetragonal, I4(1)/a
Unit cell dimensions	a = 20.763(3) Å alpha = 90 deg.
	b = 20.763(3) Å beta = 90 deg.
	c = 13.7545(18) Å gamma = 90 deg.
Volume	5929.5(17) $Å^3$
Z, Calculated density	1.046 Mg/m^3
Absorption coefficient	0.274 mm^{-1}
F(000)	2000
Crystal size	0.41×0.25×0.23 mm
Theta range for data collection	3.552 to 50 deg.
Limiting indices	-24<=h<=20, -24<=k<=24, -16<=l<=16
Reflections collected / unique	14942/2608 [R(int)=0.0440]
Completeness to theta = 25.0	99.8%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9397 and 0.8960
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2608/44/136
Goodness-of-fit on F^2	1.024
Final R indices [I>2sigma(I)]	R1=0.0612, wR2=0.1603
R indices (all data)	R1=0.1009, wR2=0.1991
Largest diff. peak and hole	0.29 and -0.40 e. $Å^{-3}$

X-ray crystallographic data of $(TMS)_2$-1

Table S6. Crystal data and structure refinement for **$(TMS)_2$-1**

Identification code	**$(TMS)_2$-1**
Empirical formula	$C_{22}H_{24}S_4Si_2$
Formula weight	472.83

Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.5479(13) Å alpha = 90 deg.
	b = 16.600(2) Å beta = 92.242(2) deg.
	c = 13.9079(17) Å gamma = 90 deg.
Volume	2433.3(5) $Å^3$
Z, Calculated density	4, 1.291 Mg/m^3
Absorption coefficient	0.496 mm^{-1}
F(000)	992
Crystal size	0.39×0.22×0.18 mm
Theta range for data collection	1.91 to 25.00 deg.
Limiting indices	-10<=h<=12, -19<=k<=19, -16<=l<=16
Reflections collected / unique	12295 / 4294 [R(int)=0.0261]
Completeness to theta = 25.0	100.0%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9161 and 0.8301
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4294/0/259
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1=0.0447, wR2=0.1207
R indices (all data)	R1=0.0597, wR2=0.1333
Largest diff. peak and hole	0.288 and -0.320 $e.A^{-3}$

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