Supporting Information for:

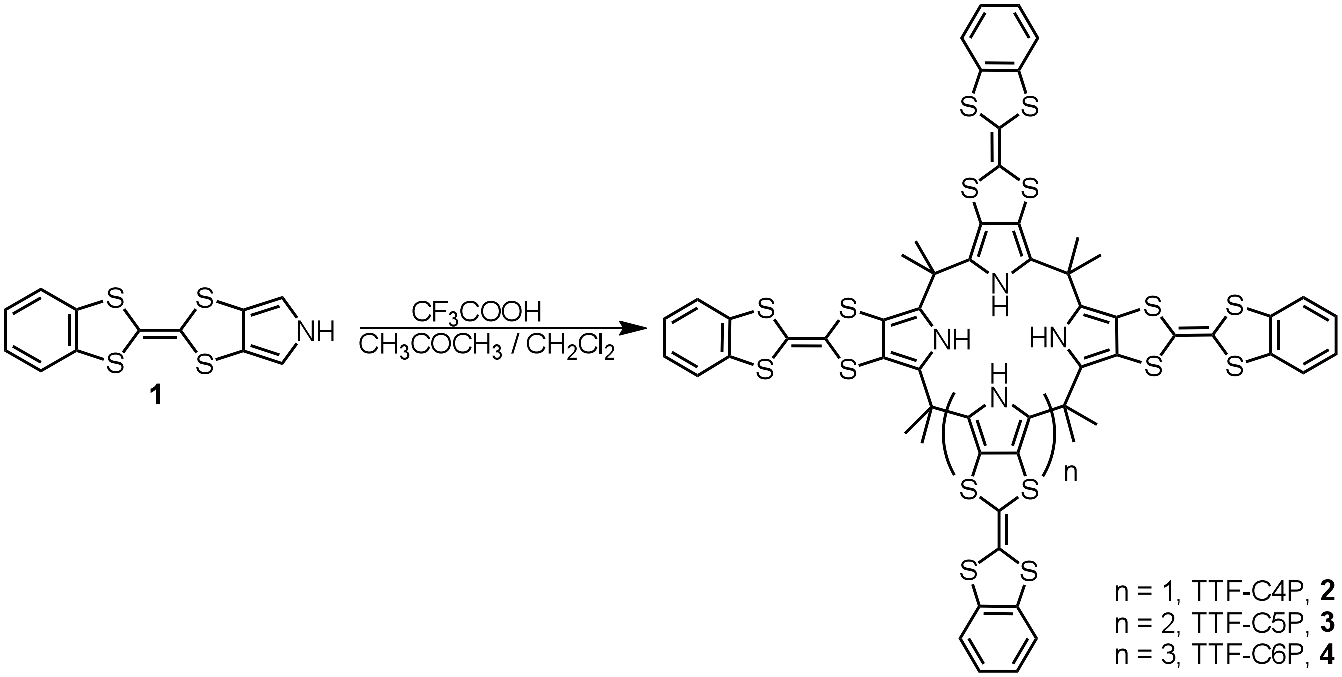
Synthesis and Recognition Properties of Higher Order Tetrathiafulvalene (TTF) Calix[n]pyrroles (n = 4–6).

Jung Su Park, Christopher Bejger, Karina R. Larsen, Kent A. Nielsen, Atanu Jana, Vincent M. Lynch, Jan O. Jeppesen, Dongho Kim, and Jonathan L. Sessler

Contents

I. Synthesis and Characterization

1. Synthesis of benzo-TTF-calix[n]pyrroles (n = 4, 5, and 6; compounds **2, 3,** and **4**, respectively) and corresponding spectroscopic characterization data.



Compound **1** was prepared using the procedure described previously.[S1] In accord with the above Scheme, compound **1** (1.00 g, 3.34 mmol) was dissolved in a mixture of Me_2CO (350 mL) and CH_2Cl_2 (350 mL) and degassed with argon for 30 min before trifluroacetic acid (TFA, 3 mL) was slowly added. The mixture was then stirred for two days at room temperature, after which triethylamine (6 mL) was slowly added. After removal of the solvent, a solid yellow residue was obtained. This solid was dried *in vacuo* and purified by column chromatography (silica gel, CH_2Cl_2 / hexanes = 2:1, eluent) to give fractions corresponding to compounds **2**, **3** and **4**. After removal of solvent, these products were obtained in the form of yellow solids.

For **2**: Yield (0.24 g, 0.18 mmol, 21%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.19 (br s, 4H, N-H), 7.09 (dd, J = 5.8, 3.2 Hz, 8H, benzene C-H), 6.95 (dd, J = 5.8, 3.2 Hz, 8H, benzene C-H) 1.60 (s, 24H, CH_3); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 136.4, 127.0, 125.5, 121.6, 118.1, 115.7, 112.2, 30.9, 27.6; HR-MALDI-MS calcd. for $C_{60}H_{44}N_4S_{16}$ 1331.9098; found 1331.9121 (M^+).
This previously reported compound[S1] (see main text) was further characterized by X-ray diffraction analysis in the form of its chloride anion complex (vide infra).

For **3**: Yield (0.19 g, 0.12 mmol, 17%). **Mp:** 236-238º C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.66 (br s, 5H, N-H), 7.09 (dd, $J = 5.9, 3.2$ Hz, 10H, benzene C-H), 7.06 (dd, $J = 5.9, 3.2$ Hz, 10H, benzene C-H) 1.57 (s, 30H, CH_3); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 136.3, 127.3, 125.8, 121.8, 119.8, 116.5, 110.7, 30.9, 27.9; HR-MALDI-MS calcd. for $C_{75}H_{55}N_5S_{20}$ 1664.8872; found 1664.8840 (M^+). This compound was independently prepared at The University of Texas and Yonsei University and subject to elemental analysis; calcd (%) for $C_{75}H_{55}N_5S_{20}$: C, 54.02; H, 3.32; N, 4.20; S, 38.46; found (Atlantic Microlab): C, 53.96; H, 3.51; N, 4.13; S, 38.68; found (Sogang University Organic Research Center): C, 54.11; H, 3.27; N, 4.12; S, 37.98.

This compound was further characterized by X-ray diffraction analysis in the form of its chloride anion complex (vide infra).

For **4**: Yield (0.08 g, 0.039 mmol, 7%). **Mp:** 248-250º C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.66 (br s, 6H, N-H), 7.15 (dd, $J = 5.9, 3.2$ Hz, 12H, benzene C-H), 7.05 (dd, $J = 5.9, 3.2$ Hz, 12H, benzene C-H) 1.53 (s, 36H, CH_3); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 136.4, 127.2, 125.8, 121.8, 119.2, 116.6, 110.1, 31.6, 28.0; HR-MALDI-MS calcd. for $C_{90}H_{66}N_6S_{24}$ 1997.8646; found 1997.8663 (M^+). This compound was independently prepared at The University of Texas and Yonsei University and subject to elemental analysis; calcd (%) for $C_{90}H_{66}N_6S_{24}$: C, 54.02; H, 3.32; N, 4.20; S, 38.46; found (Atlantic Microlab): C, 53.41 and 53.57; H, 3.27 and 3.22; N, 4.08 and 4.10; S, 37.97; found (Sogang University Organic Research Center): C, 53.89; H, 3.25; N, 4.22; S, 38.41.

This compound was further characterized by X-ray diffraction analysis in the form of its chloride anion complex (vide infra).

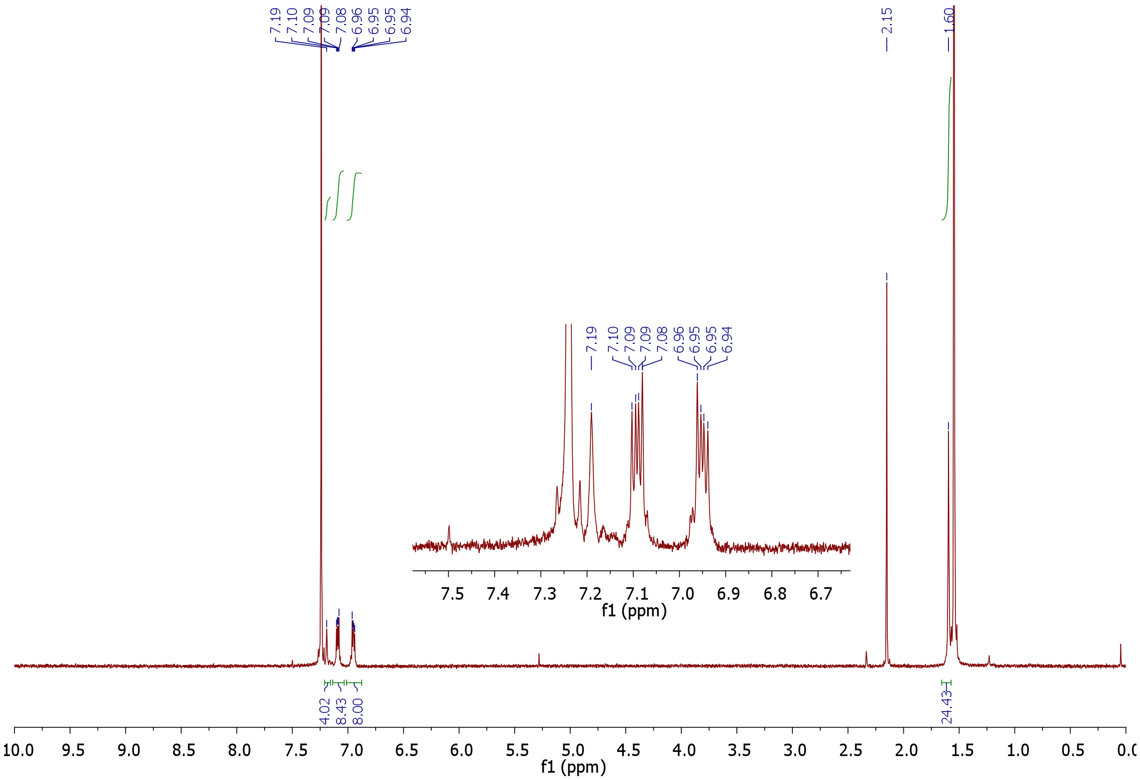
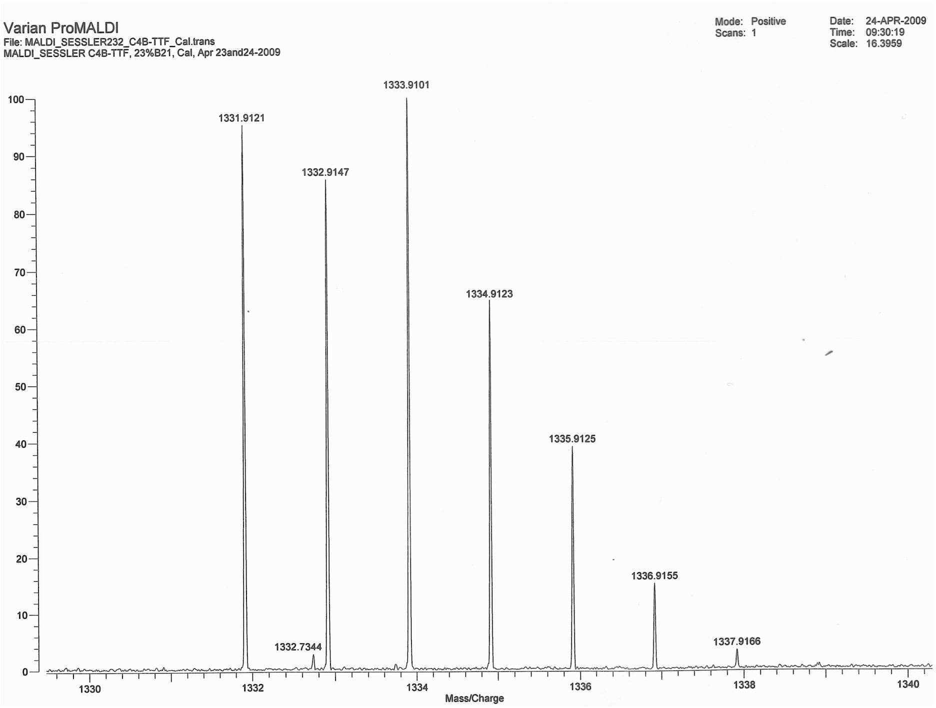


Figure S1. ^{1}H NMR spectrum (400 MHz) of **2** recorded in $CDCl_3$ at 302 K. The peak at 2.15 ppm is residual acetone.

Figure S2. HR MALDI mass spectrum of **2**



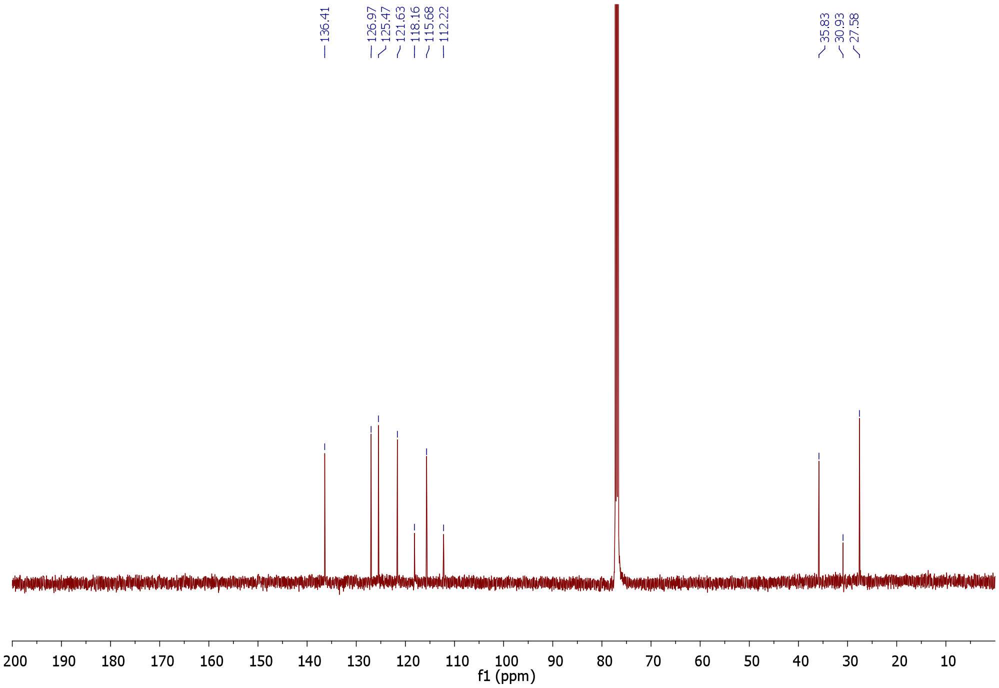


Figure S3. ^{13}C NMR spectrum (125 MHz) of **2** recorded in CDCl$_3$ at 300 K.

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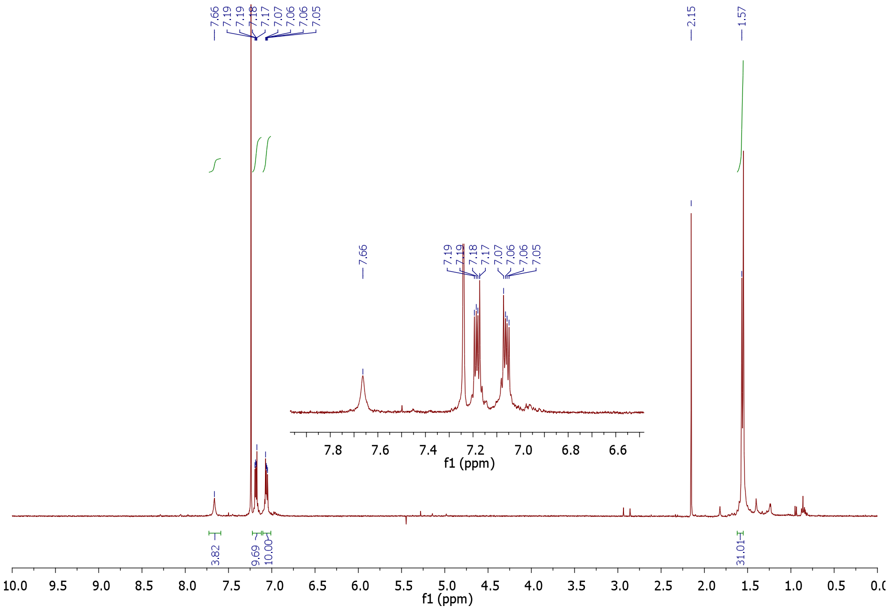


Figure S4. ^{1}H NMR spectrum (300 MHz) of **3** recorded in $CDCl_3$ at 302 K. The peak at 2.15 ppm is residual acetone.

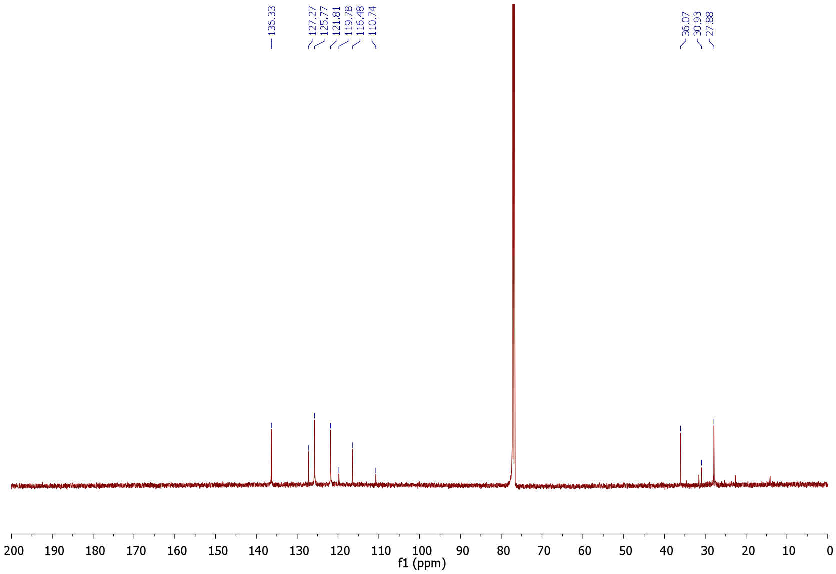


Figure S5. ^{13}C NMR spectrum (125 MHz) of **3** recorded in $CDCl_3$ at 300 K.

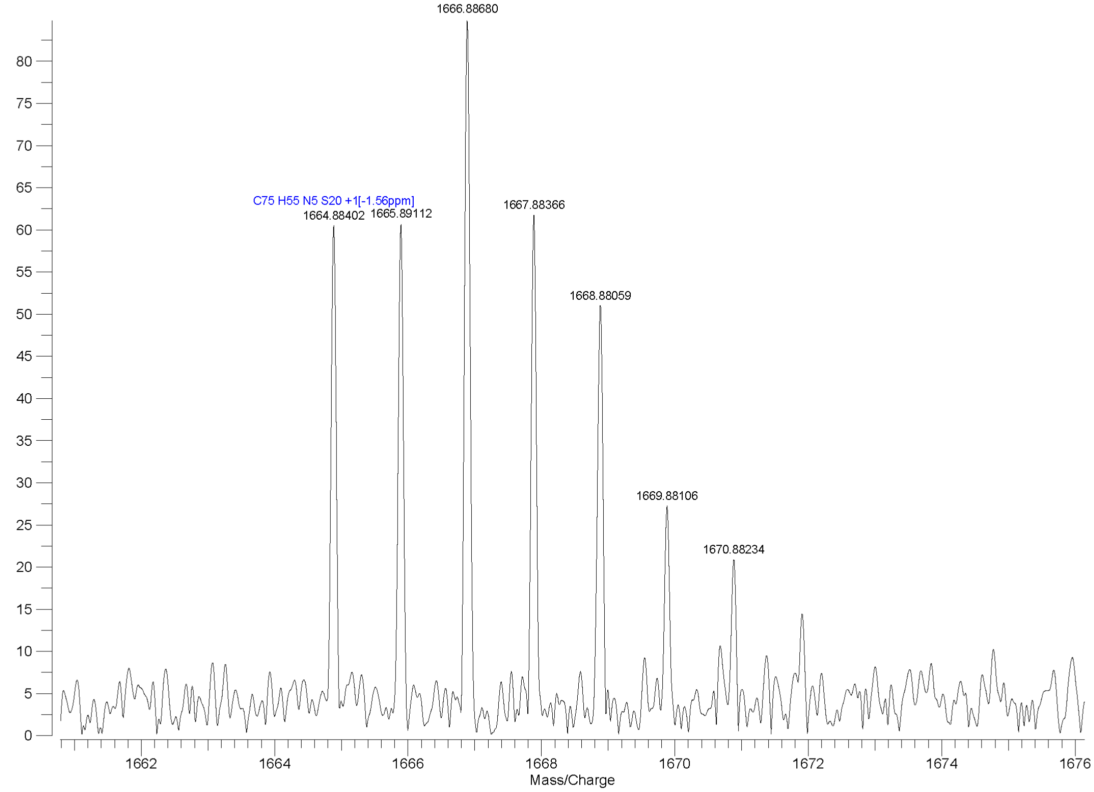
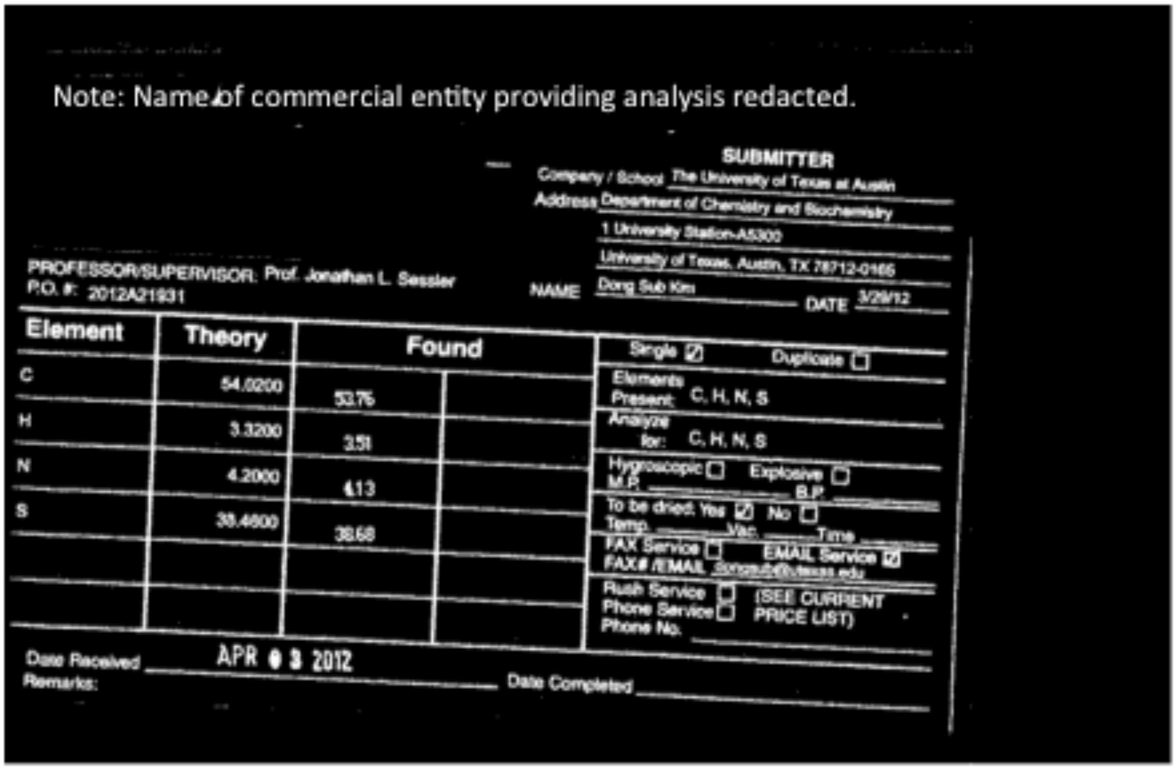


Figure S6. HR MALDI mass spectrum of **3**.



[redacted]

Note: Name of commercial entity providing analysis redacted.

SUBMITTER
Company / School The University of Texas at Austin
Address Department of Chemistry and Biochemistry
1 University Station-A5300
University of Texas, Austin, TX 78712-0165
NAME Dong Sub Kim DATE 3/29/12

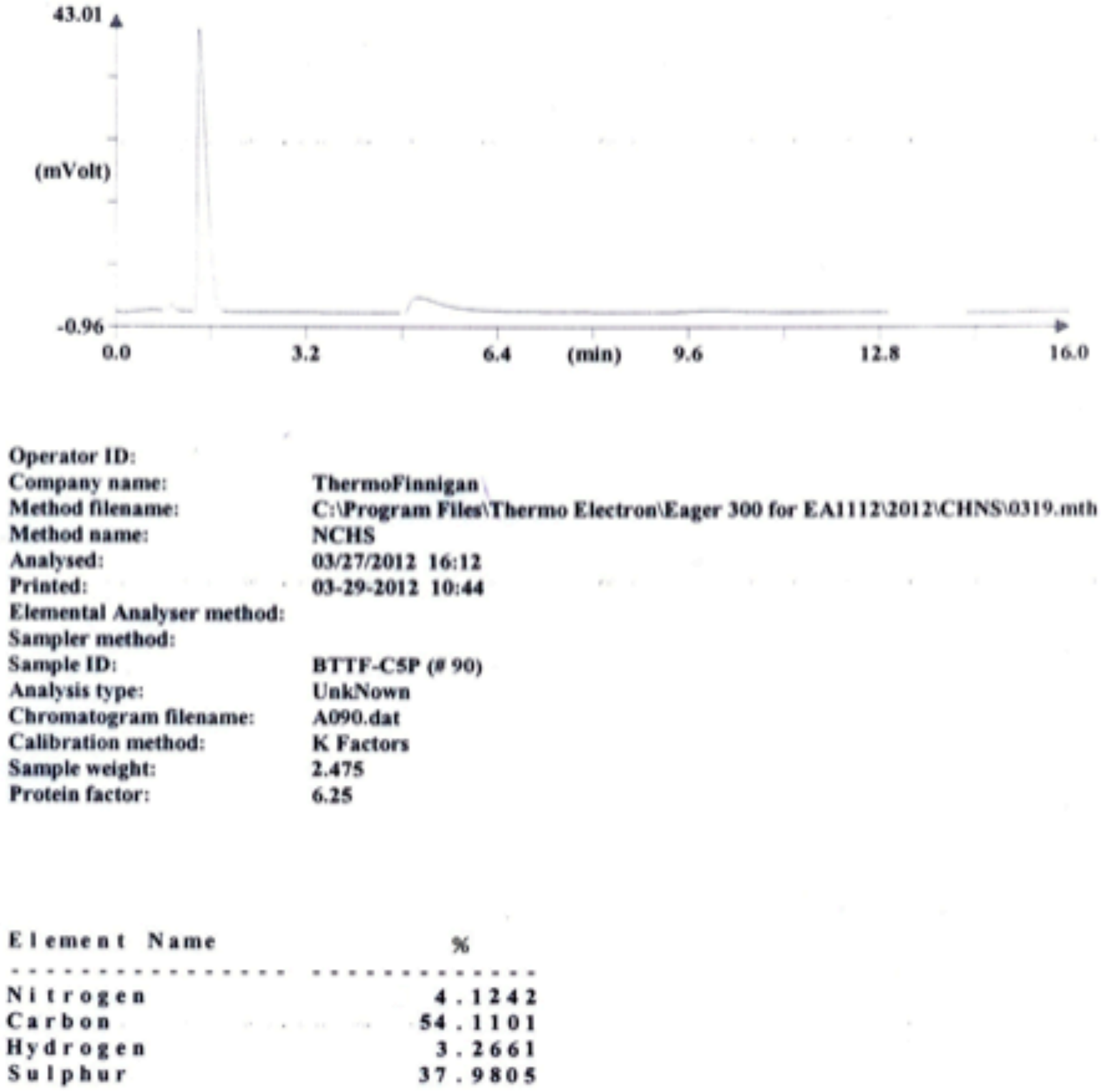
PROFESSOR/SUPERVISOR: Prof. Jonathan L. Sessler
P.O. #: 2012A21931

Element	Theory	Found	
C	54.0200	53.76	
H	3.3200	3.51	
N	4.2000	4.13	
S	38.4600	38.68	

Single ☑ Duplicate ☐
Elements Present: C, H, N, S
Analyze for: C, H, N, S
Hygroscopic ☐ Explosive ☐
M.P. ____ B.P. ____
To be dried: Yes ☑ No ☐
Temp. ____ Vac. ____ Time ____
FAX Service ☐ EMAIL Service ☑
FAX# /EMAIL [illegible]@utexas.edu
Rush Service ☐ (SEE CURRENT
Phone Service ☐ PRICE LIST)
Phone No. ____

Date Received APR 03 2012
Remarks: ____ Date Completed ____

Note: Name of commercial entity providing analysis redacted.



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Printed:	03-29-2012 10:44
Elemental Analyser method:	
Sampler method:	
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Analysis type:	UnkNown
Chromatogram filename:	A090.dat
Calibration method:	K Factors
Sample weight:	2.475
Protein factor:	6.25

Element Name	%
Nitrogen	4.1242
Carbon	54.1101
Hydrogen	3.2661
Sulphur	37.9805

Figure S7. Elemental analysis reports for the benzo-TTF-C5P **3**.

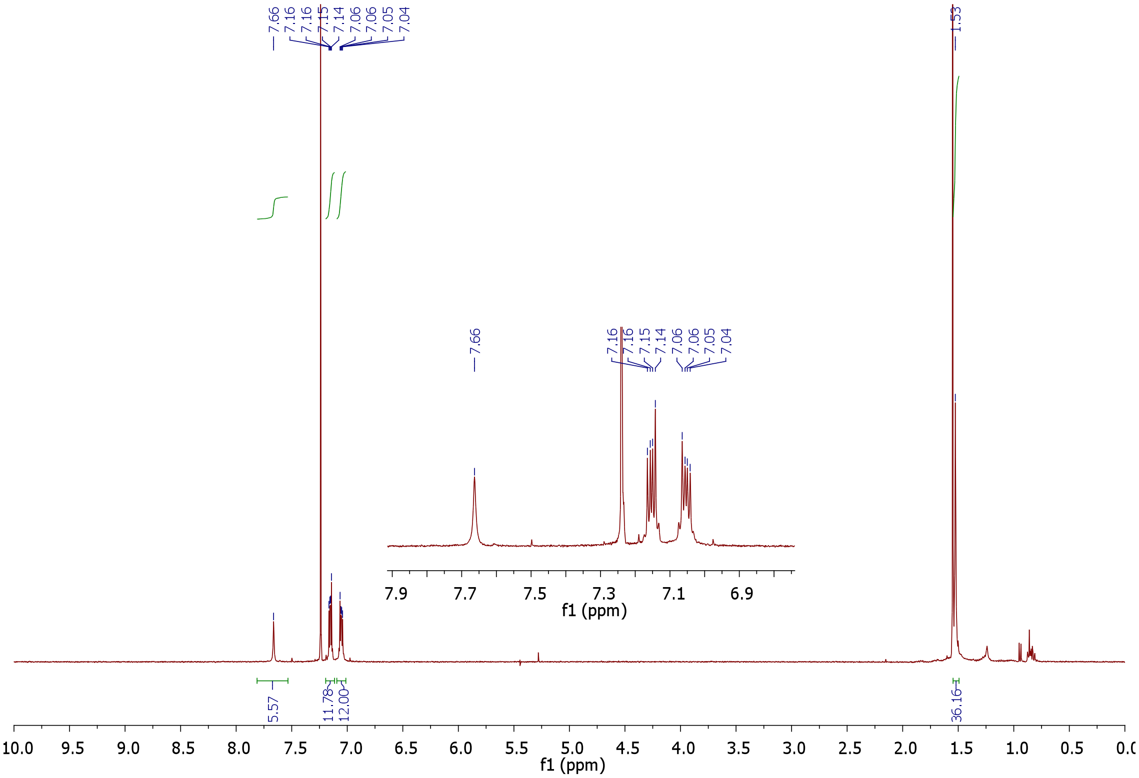


Figure S8. ^{1}H NMR spectrum (400 MHz) of **4** recorded in $CDCl_3$ at 302 K.

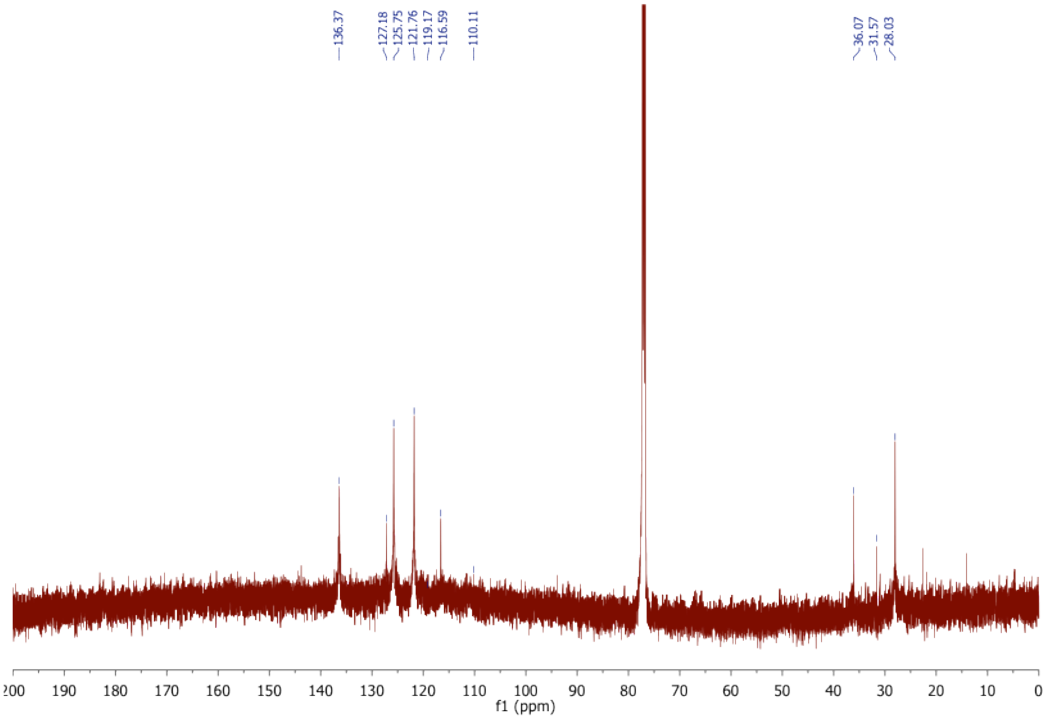


Figure S9. ^{13}C NMR spectrum (125 MHz) of **4** recorded in $CDCl_3$ at 300 K. Note: resolution limited by solubility.

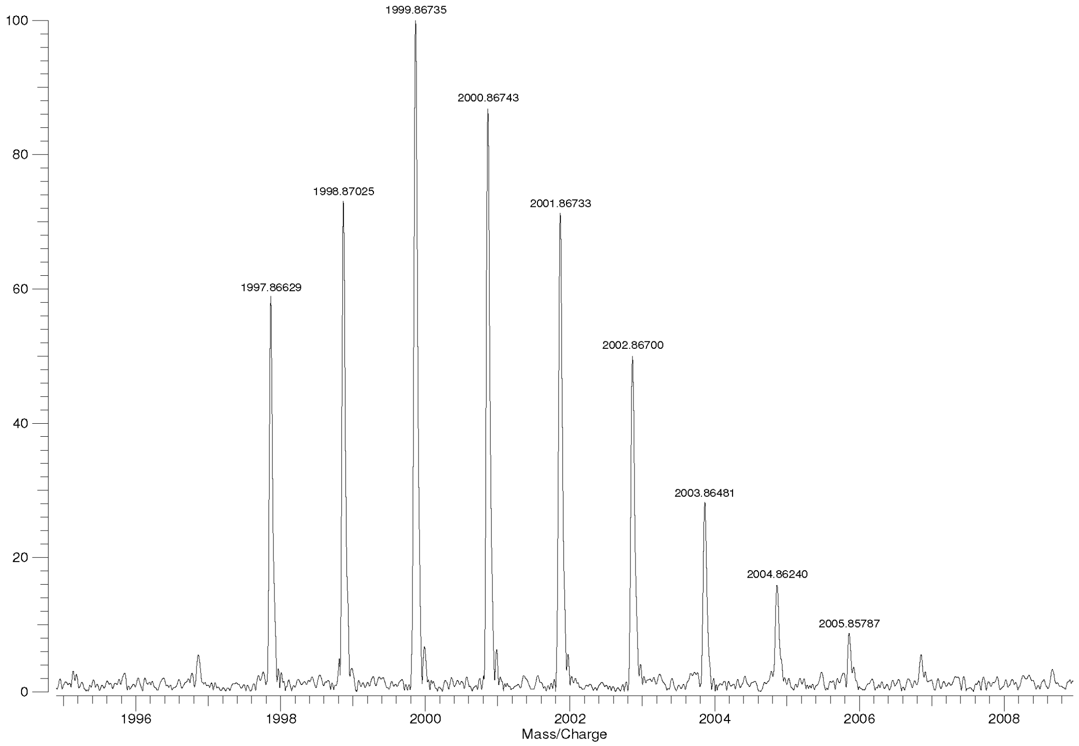
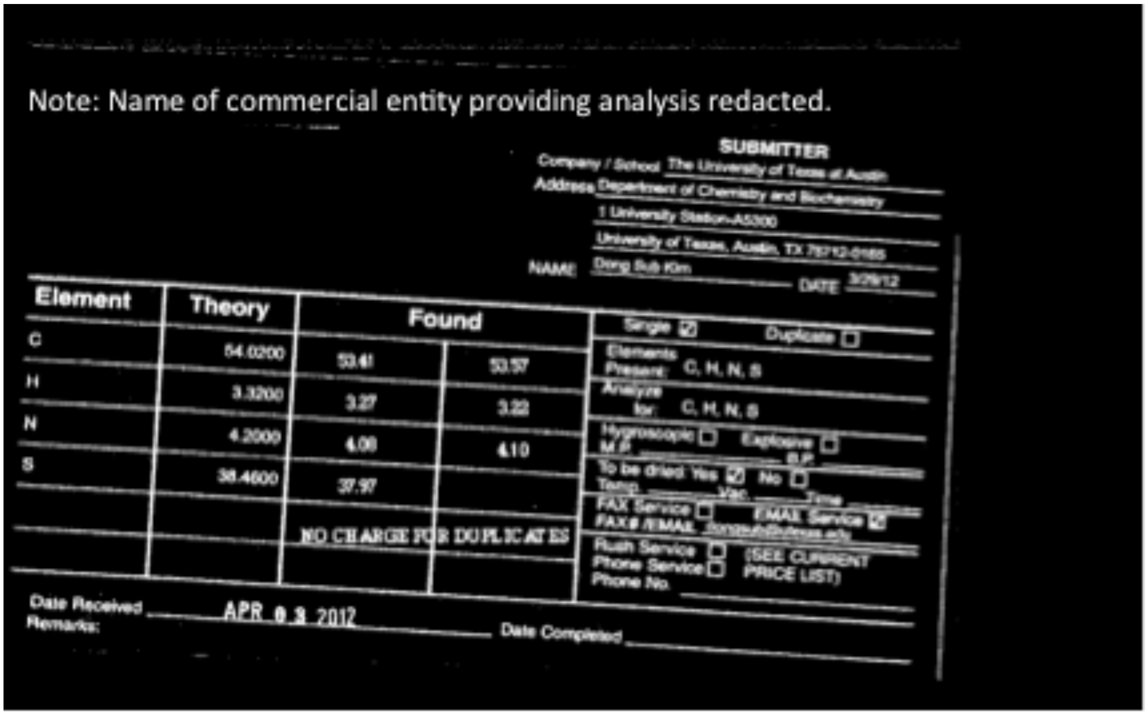


Figure S10. HR MALDI mass spectrum of **4**.



[redacted]

Note: Name of commercial entity providing analysis redacted.

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University of Texas, Austin, TX 78712-0165
NAME Dong Suh Kim
DATE 3/29/12

Element	Theory	Found	
C	54.0200	53.41	53.57
H	3.3200	3.27	3.22
N	4.2000	4.08	4.10
S	38.4600	37.97	
		NO CHARGE FOR DUPLICATES	

Single ☑ Duplicate ☐
Elements Present: C, H, N, S
Analyze for: C, H, N, S
Hygroscopic ☐ Explosive ☐
M.P. ________ B.P. ________
To be dried Yes ☑ No ☐
Temp. ________ Vac. ________ Time ________
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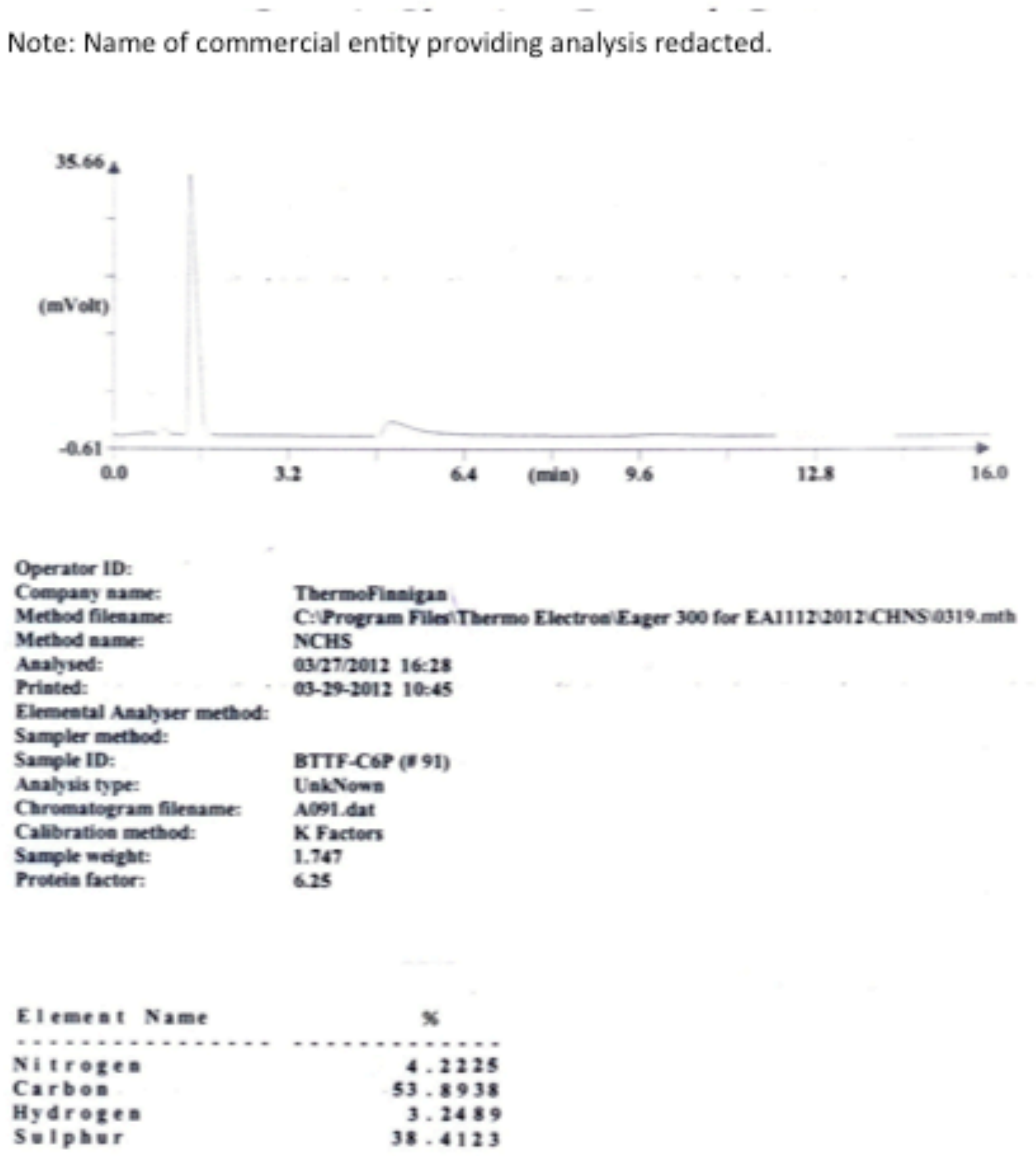
FAX# /EMAIL [illegible]
Rush Service ☐ (SEE CURRENT
Phone Service ☐ PRICE LIST)
Phone No. ________

Date Received APR 03 2012
Remarks: ________ Date Completed ________



Note: Name of commercial entity providing analysis redacted.

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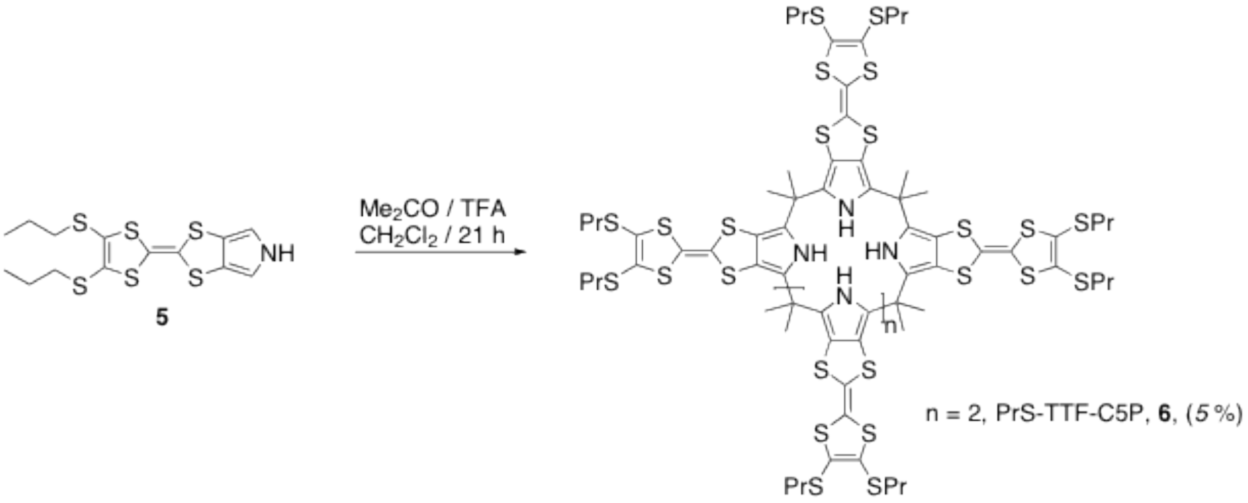
Analysed:	03/27/2012 16:28
Printed:	03-29-2012 10:45
Elemental Analyser method:	
Sampler method:	
Sample ID:	BTTF-C6P (# 91)
Analysis type:	UnkNown
Chromatogram filename:	A091.dat
Calibration method:	K Factors
Sample weight:	1.747
Protein factor:	6.25

Element Name	%
Nitrogen	4.2225
Carbon	53.8938
Hydrogen	3.2489
Sulphur	38.4123

Figure S11. Elemental analysis reports for the benzo-TTF-C6P **4**.

2. Synthesis of bispropylthio functionalized TTF-calix[5]pyrrole (PrS TTF-C5P; **6**) and corresponding spectroscopic characterization data.



A solution of the bispropylthio pyrrolo-TTF **5**[S2] shown in the above scheme (1.18 g, 3.0 mmol) was dissolved in CH_2Cl_2 (600 mL) and Me_2CO (300 mL). The resulting solution was deoxygenated by purging with argon for 15 minutes. At this point, TFA (7 mL) was added to the initial yellow solution. The reaction mixture was stirred for 24 h at room temperature, whereupon an excess of Et_3N (6 mL) was added slowly to neutralize the acid. The reaction mixture was then evaporated near to dryness and CH_2Cl_2 (300 mL) was added. The solution was then washed with plenty of H_2O and the organic part was dried over $MgSO_4$. After evaporation of the solvent, the yellow solid was purified by column chromatography using silica gel (400 mesh). The first yellow band (eluent: hexanes/CH_2Cl_2 2:1) was collected and concentrated to give the known TTF calix[4]pyrrole product (TTF-C[4]P) as a bright yellow solid (0.24 g, 18%). The next eluting yellow fraction was then collected using hexanes/CH_2Cl_2 1:1 as the eluent to give the bisPrS TTF-C5P product **6**, which was further purified via recycling gel permeation chromatography (GPC) using $CHCl_3$ to give pure material (0.070 g, 5%), as judged from standard analytical methods.

Mp: 138-145° C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.99 (t, J = 8 Hz, 30H, CH_3), 1.56 (s, 30H, CH_3), 1.59–1.66 (m, 20H, CH_2), 2.75 (t, J = 8.0 Hz, 20H, SCH_2), 7.64 (bs, 5H, NH); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.1, 23.0, 28.1, 36.1, 38.2, 110.7, 116.6, 120.0, 127.4 (1 signal is missing/overlapping); MALDI-TOF MS (dithranol matrix): m/z 2155.0021 for $[M+1]^+$; (calcd for $C_{85}H_{105}N_5S_{30}$: 2154.999). Elemental analysis (Atlantic Microlab, Inc.): calcd for

$C_{85}H_{105}N_5S_{30}$: C 47.29, H 4.90, N 3.24, S 44.56; found C 47.33, H 4.94, N 3.18, S 44.27.

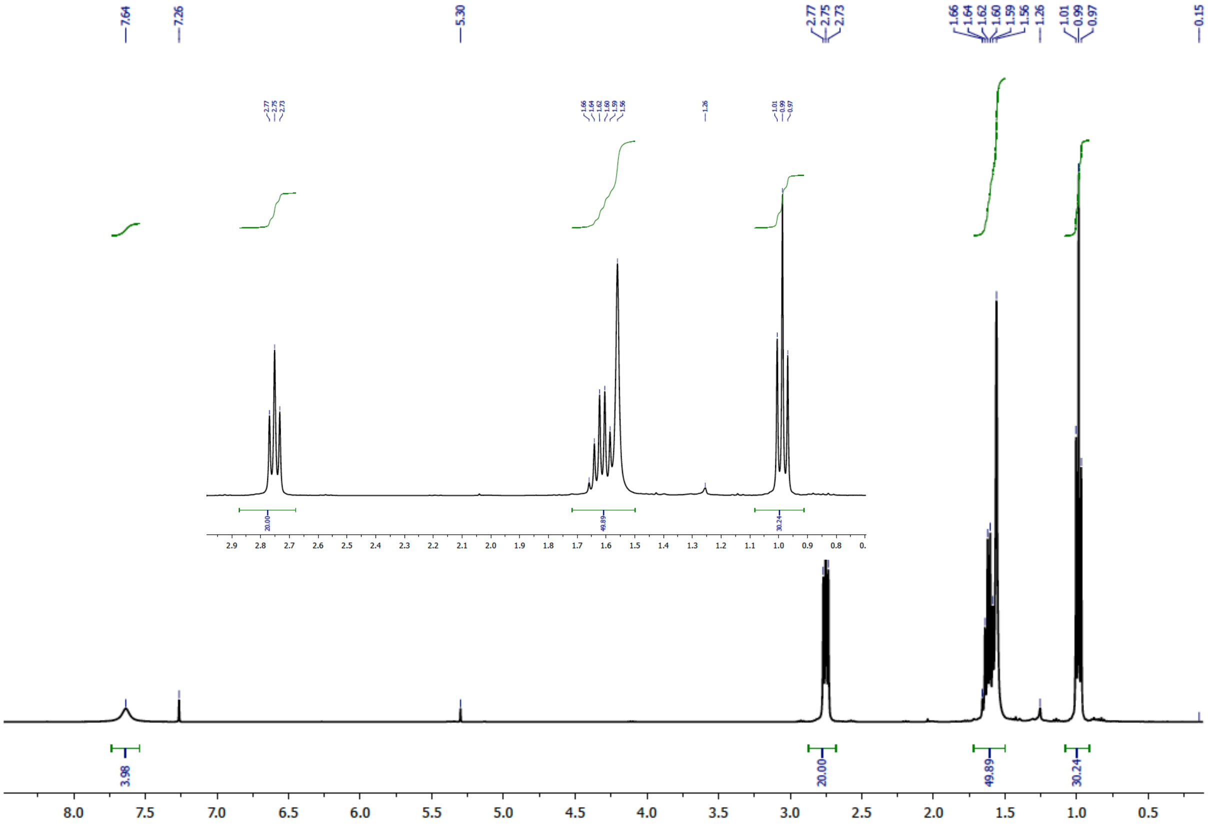


Figure S12. ^{1}H NMR spectrum (400 MHz) of the bispropylthio-TTF-C5P **6** recorded in $CDCl_3$ at 298 K.

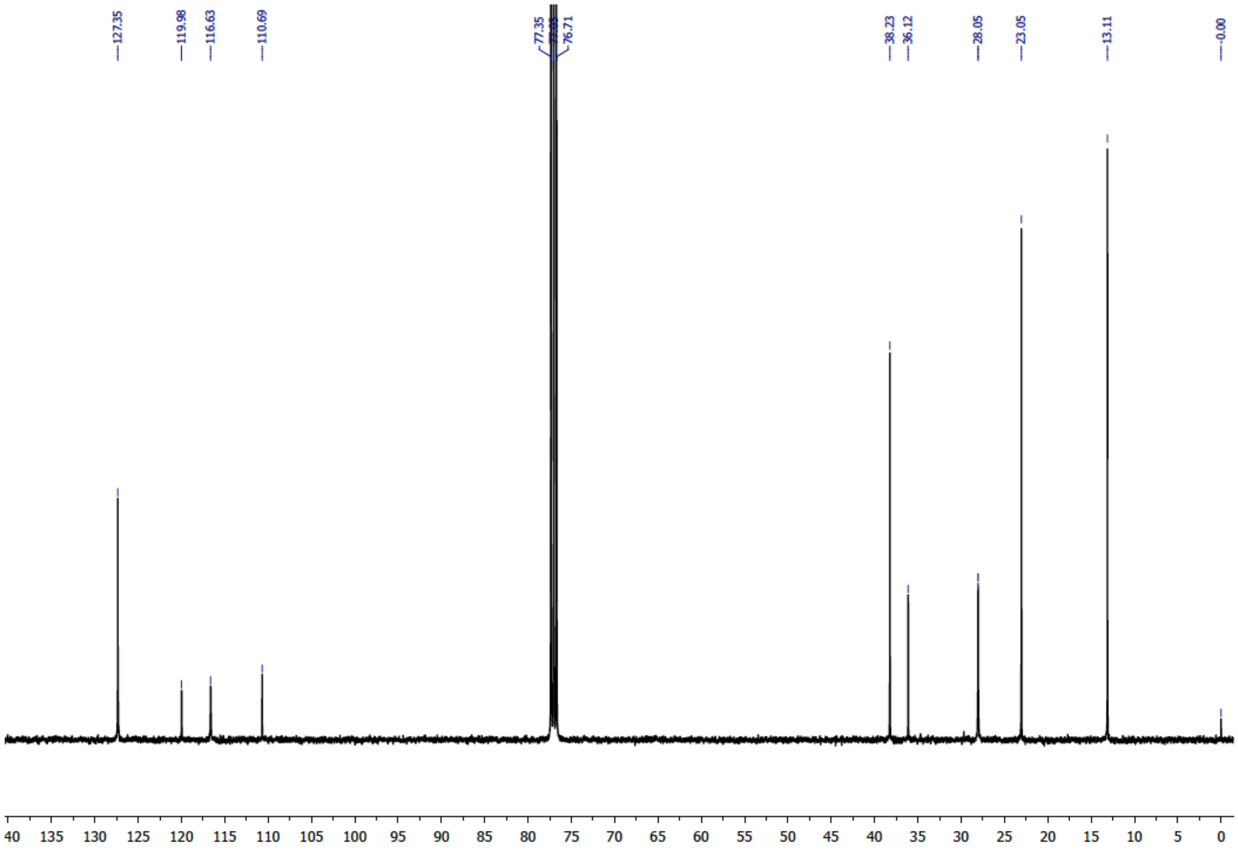


Figure S13. ^{13}C NMR spectrum (100 MHz) of the bispropylthio-TTF-C5P **6** recorded in $CDCl_3$ at 298 K.

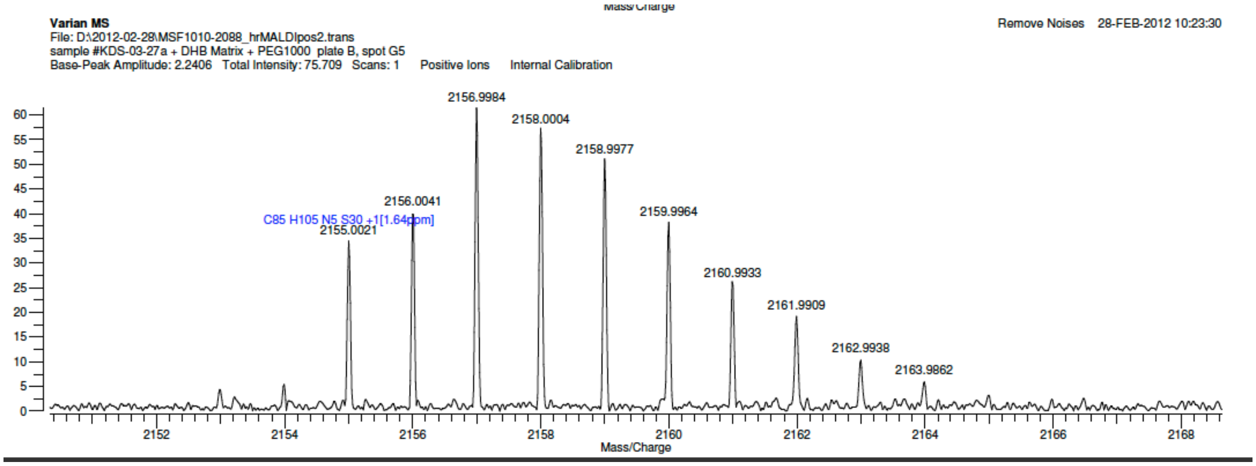
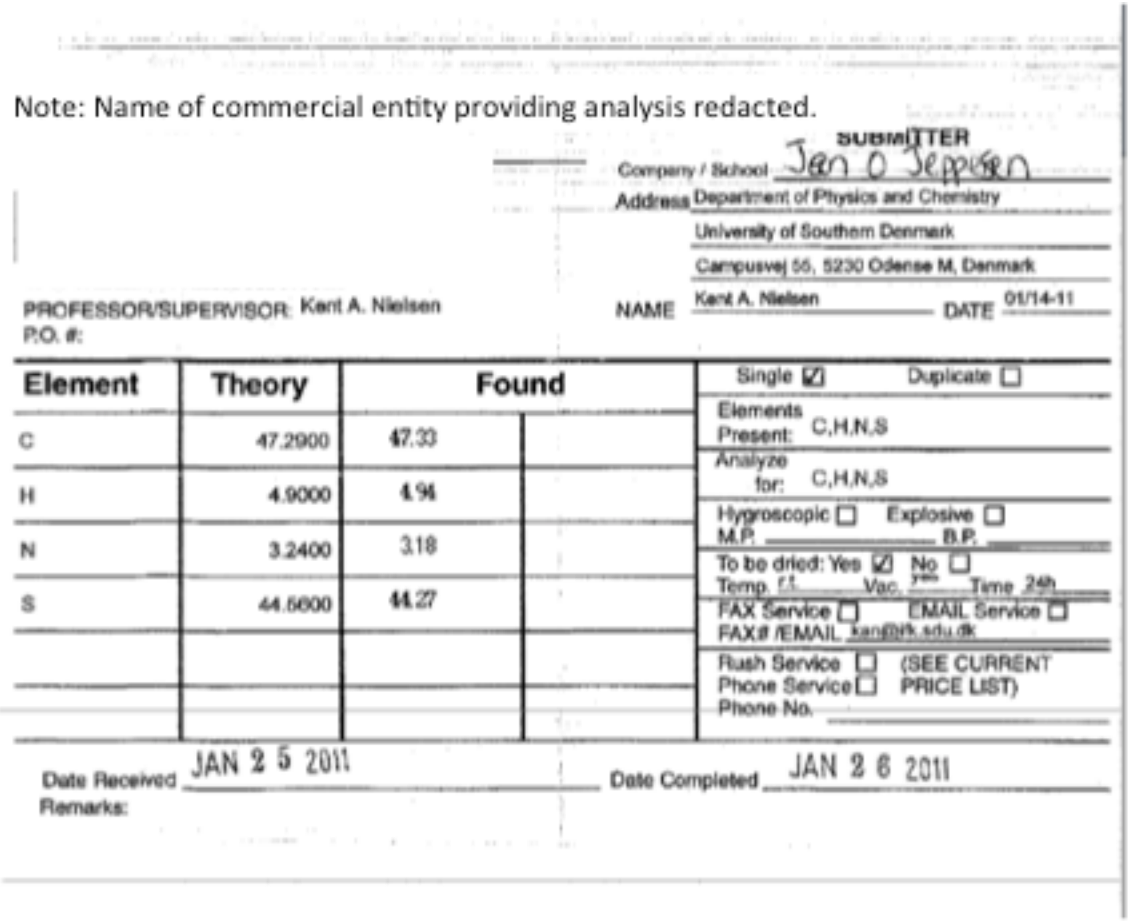


Figure S14. HR MALDI mass spectrum of the bispropylthio-TTF-C5P **6**.



Note: Name of commercial entity providing analysis redacted.

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Campusvej 55, 5230 Odense M, Denmark
NAME Kent A. Nielsen DATE 01/14-11

PROFESSOR/SUPERVISOR: Kent A. Nielsen
P.O. #:

Element	Theory	Found	
C	47.2900	47.33	
H	4.9000	4.94	
N	3.2400	3.18	
S	44.5600	44.27	

Single ☑ Duplicate ☐
Elements Present: C,H,N,S
Analyze for: C,H,N,S
Hygroscopic ☐ Explosive ☐
M.P. B.P.
To be dried: Yes ☑ No ☐
Temp. r.t. Vac. yes Time 24h
FAX Service ☐ EMAIL Service ☐
FAX# /EMAIL kan@ifk.sdu.dk
Rush Service ☐ (SEE CURRENT PRICE LIST)
Phone Service ☐
Phone No.

Date Received JAN 25 2011
Date Completed JAN 26 2011
Remarks:

Figure S15. Elemental analysis report for the bispropylthio-TTF-C5P **6**.

II. X–ray Crystallographic Analyses of 2·TBACl, 3·THACl, and 4·TBACl.

In order to characterize the solid-state conformational properties and binding modes resulting from the interaction between receptors **2**, **3**, and **4** and chloride anion, pentane was diffused into equimolar mixtures of **2**, **3**, and **4** and tetrabutylammonium chloride or tetrahexylammonium chloride in chloroform solution. In this way, yellow, diffraction-quality single crystals were grown from the combination of [**2**+TBACl], [**3**+THACl], and [**4**+TBACl]. Subsequent single crystallographic X–ray diffraction analyses provided evidence for the formation of supramolecular assemblies between the corresponding benzo-TTF-calix[n]pyrroles (**2**, **3**, or **4**) and chloride anion in the solid state that are characterized by an overall 1:1 stoichiometry. The X-ray structures in question are displayed in Figure S14-16.

X–ray crystallographic data for **2·TBACl** were collected on a Rigaku AFC8 diffractometer equipped with a Saturn CCD area detector and a graphite monochromatized MoK_{α} source ($\lambda = 0.71070$ Å). The data were collected at 100 K under a cold nitrogen stream. X-ray crystallographic data for **3·THACl** were collected on Rigaku R-Axis Spider with Image Plate detector using a graphite monochromator with CuK_{α} radiation ($\lambda = 1.5418$Å). The data were collected at 100 K using a Rigaku XStream low temperature device. Data reduction for **2·TBACl** and **3·THACl** was performed using the Rigaku Americas Corporation's Crystal Clear version 1.40.[S3] X-ray crystallographic data for **4·TBACl** were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation ($\lambda = 0.71073$Å). The data were collected at 153 K using an Oxford Cryostream low temperature device. Data reduction for **4·TBACl** was performed using DENZO-SMN.[S4] Details of crystal data, data collections, and structure refinements of **2·TBACl, 3·THACl, and 4·TBACl** are listed in Table S1. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[S5] The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms). All the calculations were carried out with the SHELXTL program.[S6] Further details of the individual structures can be obtained from the Cambridge Crystallographic Data Centre by quoting reference numbers CCDC 868898 for **2·TBACl**, CCDC 868897 for **3·THACl**, and CCDC 868896 for **4·TBACl**.

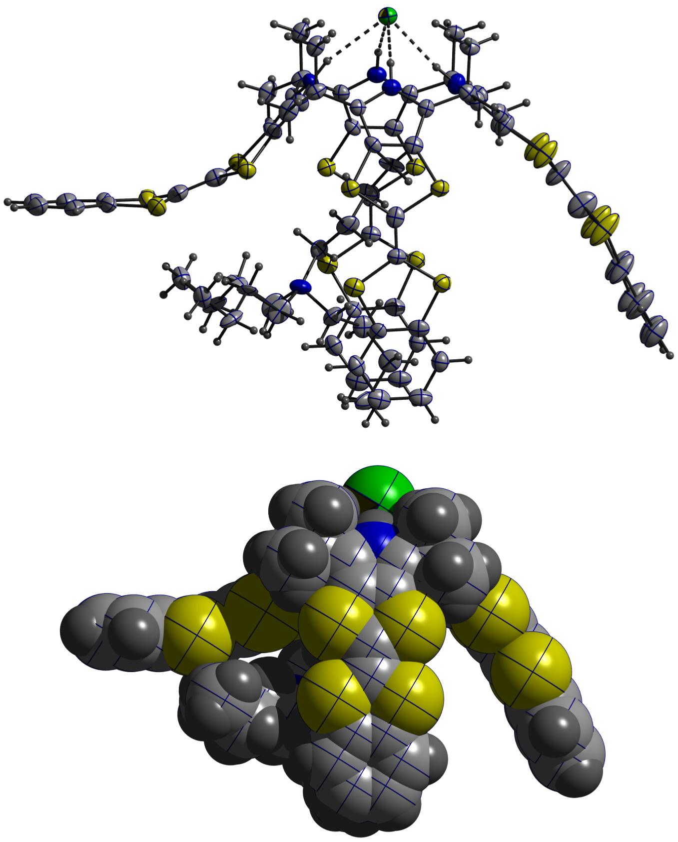


Figure S16. Single crystal X–ray diffraction structure of the TBACl complex of the benzannulated TTF-C4P **2**. Displacement ellipsoids are scaled to the 30% probability level.

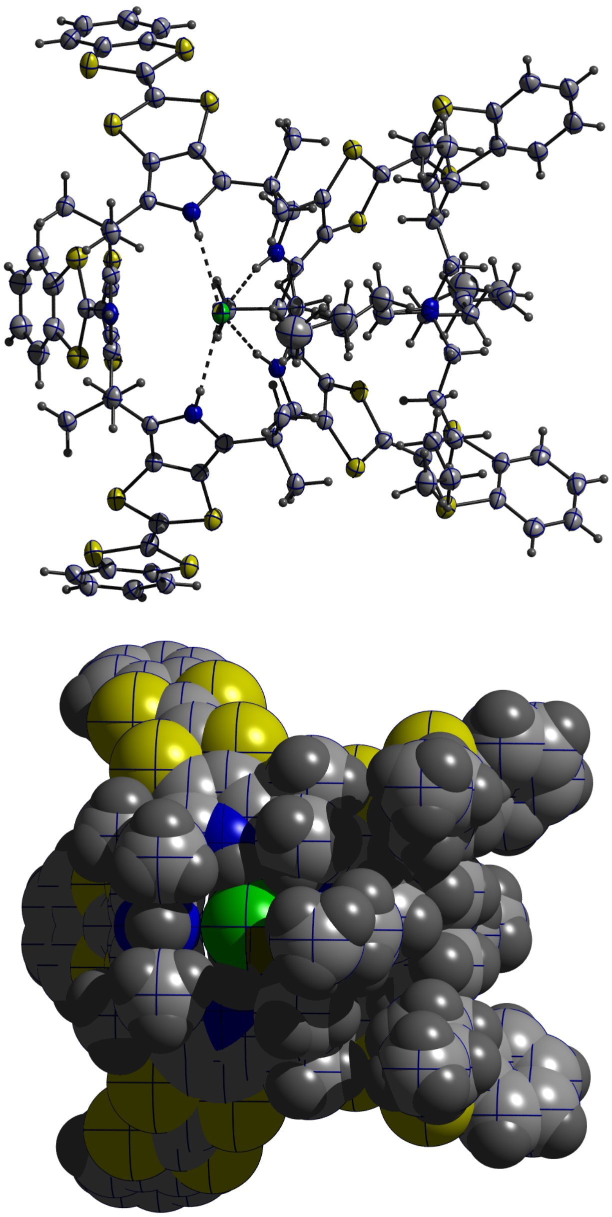


Figure S17. Single crystal X–ray diffraction structure of the THACl complex of the benzannulated TTF-C5P **3**. Displacement ellipsoids are scaled to the 30% probability level

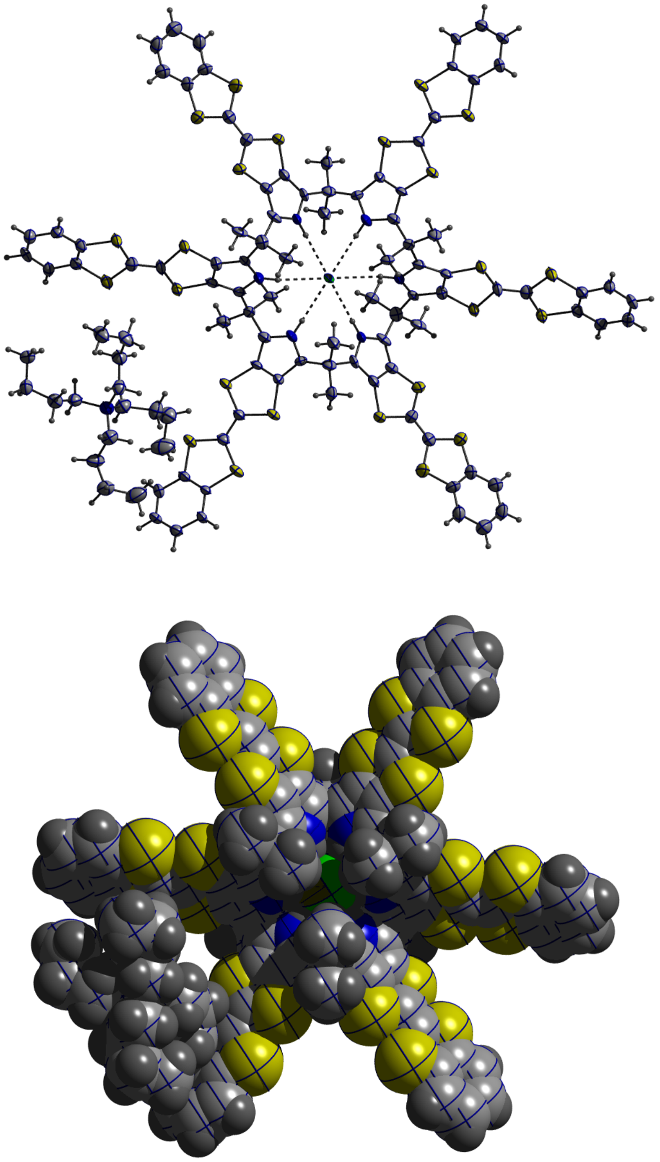


Figure S18. Single crystal X–ray diffraction structure of the TBACl complex of the benzannulated TTF-C6P **4**. Displacement ellipsoids are scaled to the 30% probability level

Table S1. Selected crystal data and refinement parameters

	2	**3**	**4**
Empirical formula	C79 H85 Cl8 N5 S16	C110 H127 Cl3 N6 S20	C110 H110 Cl9 N7 S24
Temperature (K)	100 (2)	93(2)	153(2)
Crystal system	Orthorhombic	Orthorhombic	Monoclinic
Space group	Pnnm	Pnma	C2/c
Crystal color	Yellow	Yellow	Yellow
a (Å)	29.858(6)	28.3045(9)	31.769(2)
b (Å)	14.168(3)	24.6469(8)	20.0818(12)
c (Å)	21.609(4)	17.5652(6)	24.852(2)
α (deg)	90	90	90
β (deg)	90	90	129.585(3)
γ (deg)	90	90	90
Volume ($Å^3$)	9141(3)	12253.8(7)	12219.2(14)
Z	4	4	4
θ range (°)	3.08 to 25.00	6.72 to 70.00	2.03 to 25.00
Completeness to θ	99.7%	88.6%	99.2%
Reflection collected/ Unique	33353 / 8260	10554 / 10554	86545 / 10665
Absorption correction	Semi-empirical		Semi-empirical
Data/restraints/parameters	8260 / 137 / 578	10554 / 1021 / 774	10665 / 438 / 659
Goodness-of-fit on F^2	1.673	1.651	1.099
Final *R* indices [I>2δ(I)]	$R_1 = 0.1431$ $wR_2 = 0.3074$	$R_1 = 0.0874$ $wR_2 = 0.2161$	$R_1 = 0.1086$ $wR_2 = 0.2212$
R indices (all data)	$R_1 = 0.2526$ $wR_2 = 0.3361$	$R_1 = 0.1520$ $wR_2 = 0.2662$	$R_1 = 0.2891$ $wR_2 = 0.2479$
CCDC number	868898	868897	868896

III. UV-Vis Spectrophotometric Titrations.

1.5 mM solutions of the six TBAX (X = Cl^-, Br^-, I^-, CH_3COO^-, $H_2PO_4^-$, and HSO_4^-) used in this study were made up in $CHCl_3$. Depending on the study in question, these solutions contained of **2** (15μM), **3** (20 μM), or **4** (10 μM) so as to avoid dilution errors when they were titrated into to chloroform solutions of either receptors **2** (15μM), **3** (20 μM), or **4** (10 μM). In a similar manner, 30 mM chloroform solution of two test explosives, namely TNB and TNT, were prepared and titrated into to chloroform solutions of either the receptor **3** (0.3 mM) and **4** (0.3 mM). The titrations then consisted of adding aliquots of these stock solutions containing putative substrates (various tetrabutylammonium salts into those containing the receptors). The cell length was 1.0 mm. The resulting UV-Vis spectra were then recorded using a Varian Cary 5000 spectrophotometer at room temperature and the change in spectral signature recorded used to calculate the association constants as detailed below.[S7]

It is to be noted that although only one wavelength was used for the specific binding profiles reproduced below, in general, the analyses were carried out using a variety of wavelengths and concordant results were obtained. The actual values recorded here and in the text were obtained from mathematical curve fitting in accord with standard protocols. They were not deduced by "eyeballing", which may lead to erroneous inferences, such as, e.g., acetate being bound more strongly to receptor **4** than chloride, a conclusion that would be categorically incorrect.

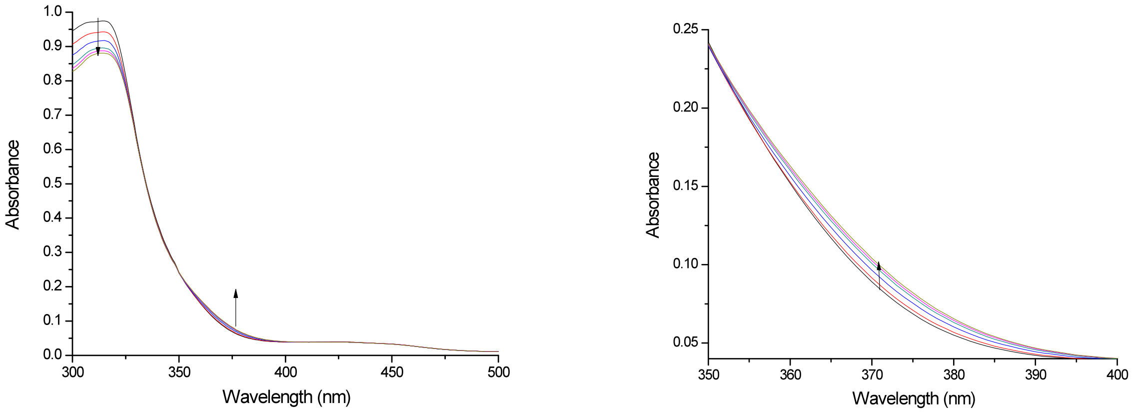


Figure S19. Absorption spectral changes observed for receptors **2** (15 μM) upon progressive additions of TBACl.

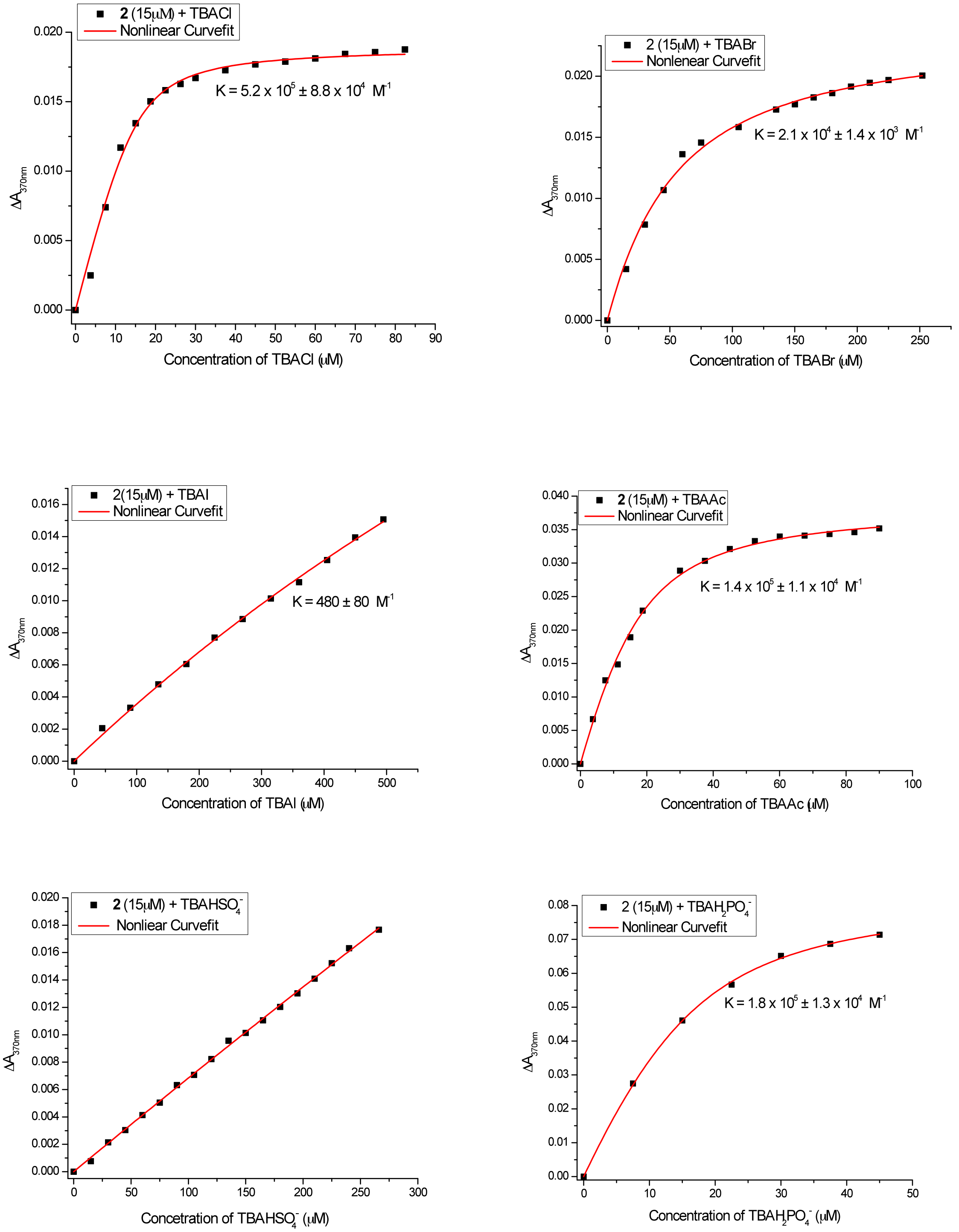


Figure S20. Binding data from titrations of **2** with six tetraalkyammonium salts, namely those of chloride, bromide, iodide, acetate, hydrogen sulfate, and dihydrogen phosphate in $CHCl_3$.

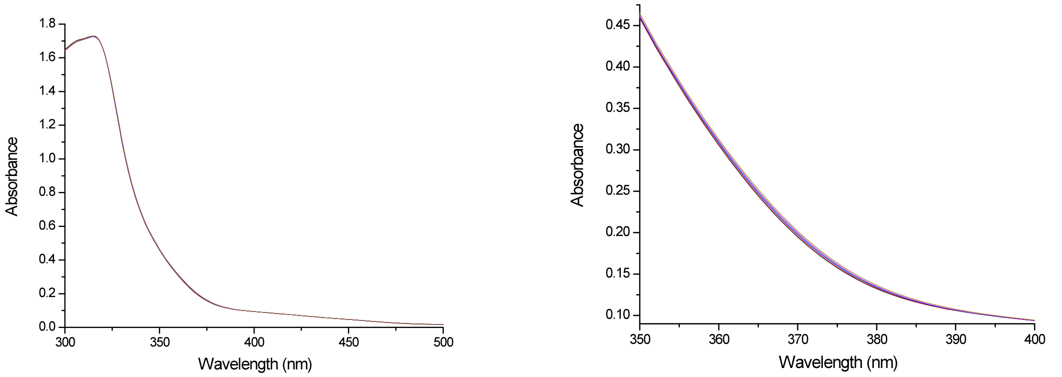
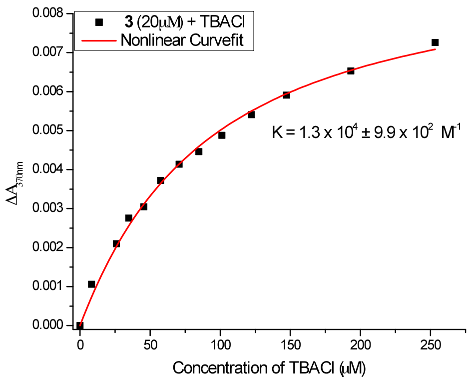
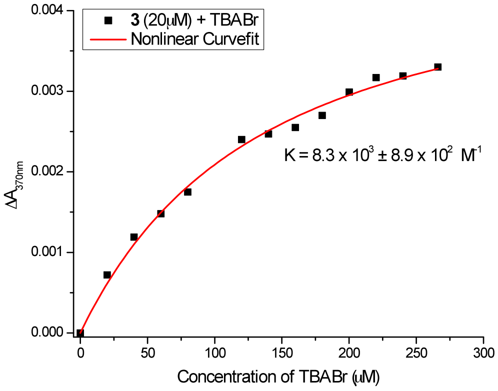
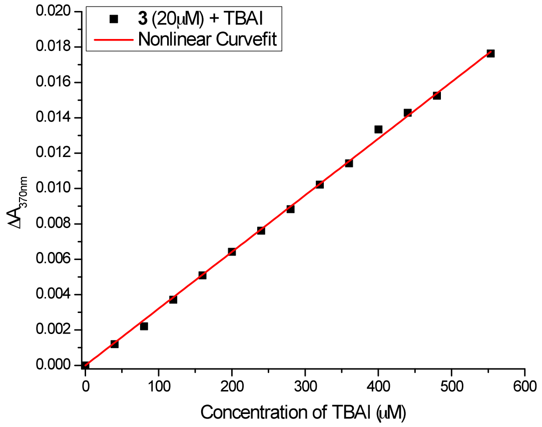
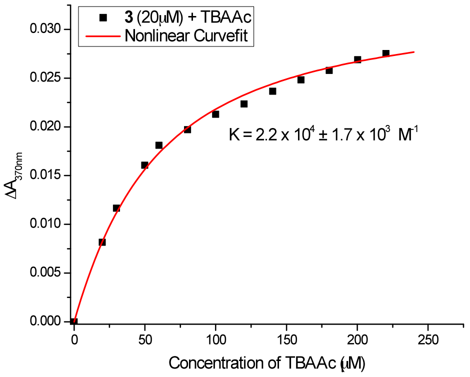


Figure S21. Absorption spectral changes observed for receptors **3** (20 μM) upon progressive additions of TBACl.









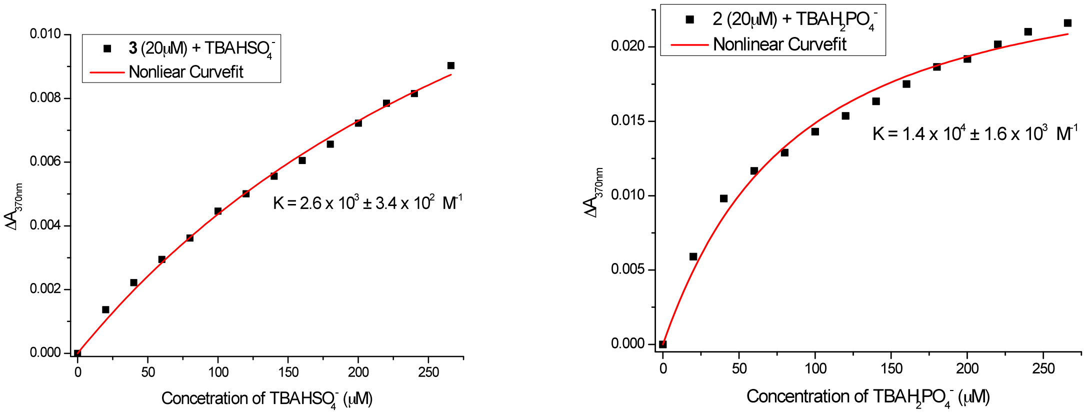


Figure S22. Binding data from titrations of **3** with six tetraalkyammonium salts, namely those of chloride, bromide, iodide, acetate, hydrogen sulfate, and dihydrogen phosphate in $CHCl_3$.

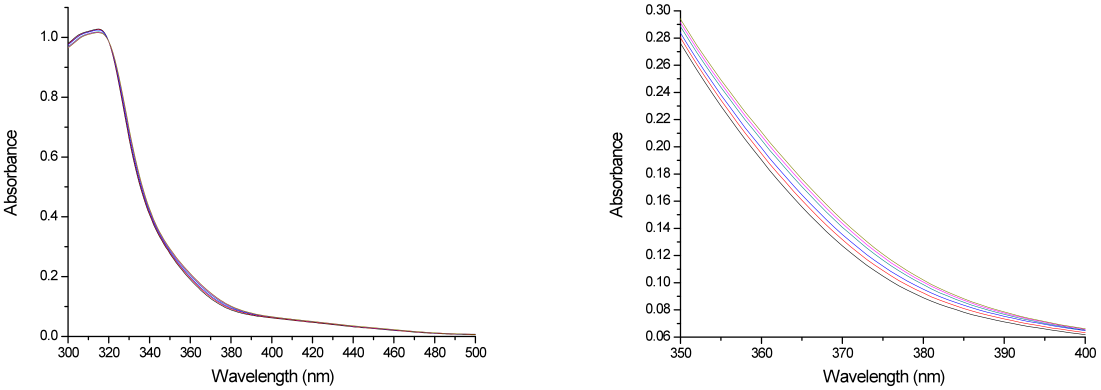


Figure S23. Absorption spectral changes observed for receptors **4** (20 μM) upon progressive additions of TBACl.

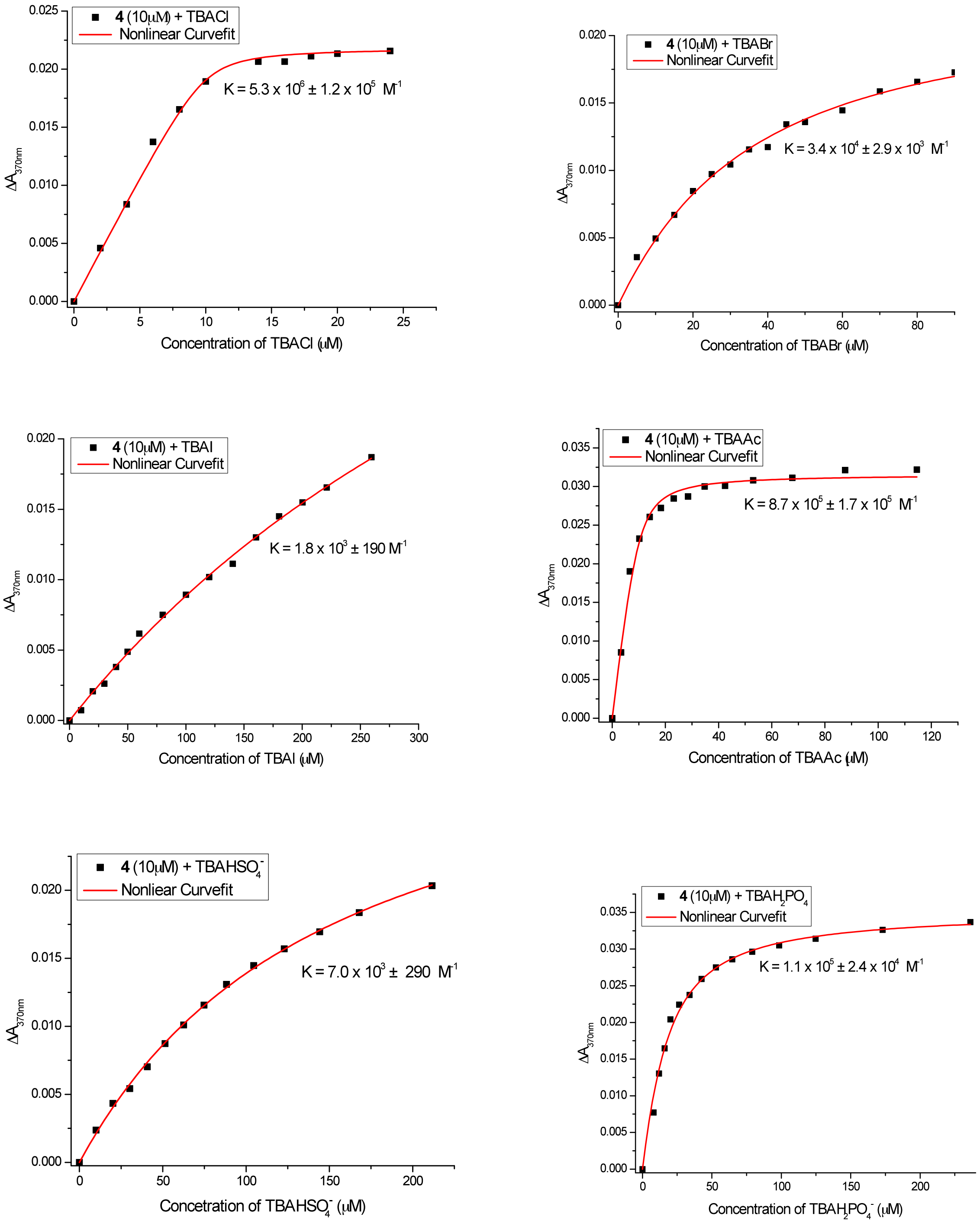


Figure S24. Binding data from titrations of **4** with six tetraalkyammonium salts, namely those of chloride, bromide, iodide, acetate, hydrogen sulfate, and dihydrogen phosphate in $CHCl_3$.

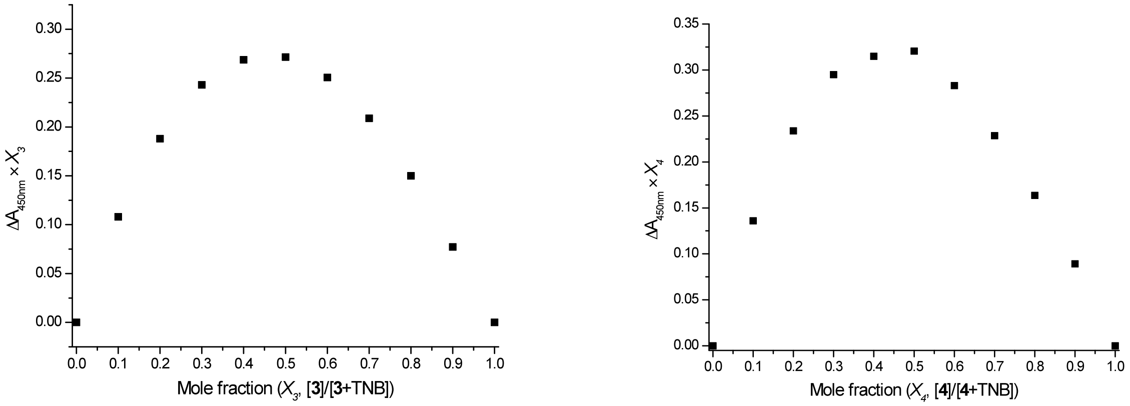


Figure S25. Continuous variation plots for $CHCl_3$ mixtures of [**3**+**TNB**] and [**4**+**TNB**]. These plots were constructed by plotting the product of the change in the absorption at 450 nm and the mole-fractions of the receptor **3** (left) or **4** (right) *vs*. the corresponding mole fractions of **3** and **4**.[S7]

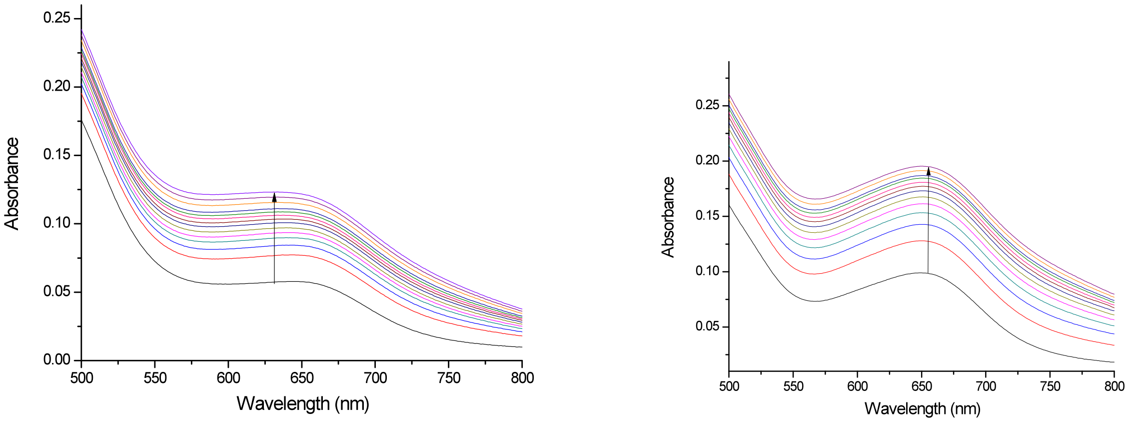


Figure S26. Absorption spectral changes observed for receptors **3** (left) and **4** (right) at 0.3 mM chloroform solution upon progressive additions of TNB.

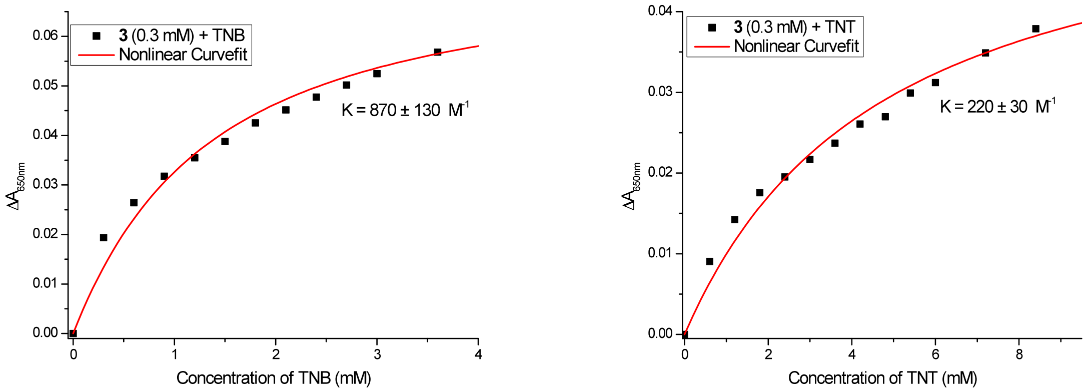


Figure S27. Binding data from titrations of **3** with TNB and TNT in $CHCl_3$.

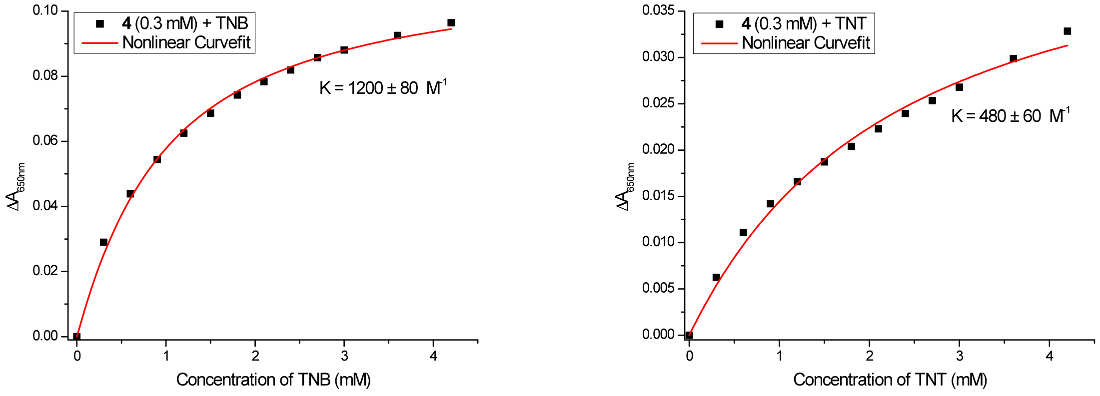
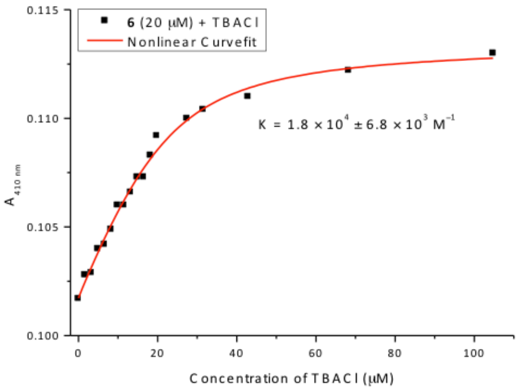
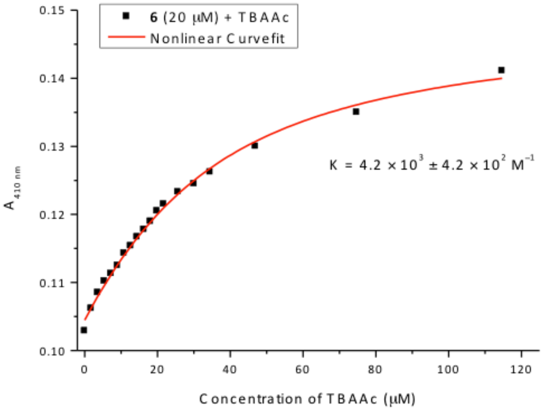


Figure S28. Binding data from titrations of **4** with TNB and TNT in $CHCl_3$.





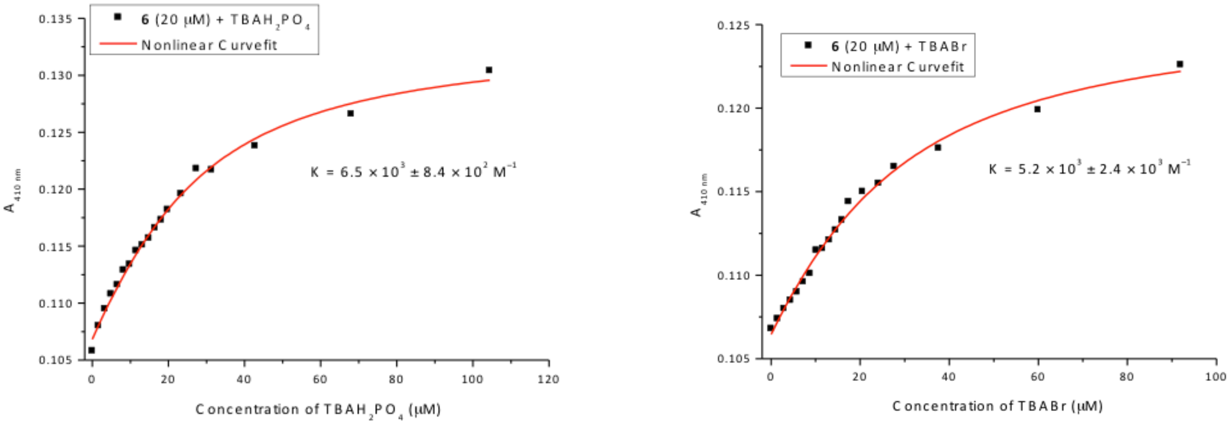


Figure S29. Binding data from titrations of **6** with TBACl, TBAAc (acetate salt), TBAH$_2$PO$_4$ and TBABr in CHCl$_3$.

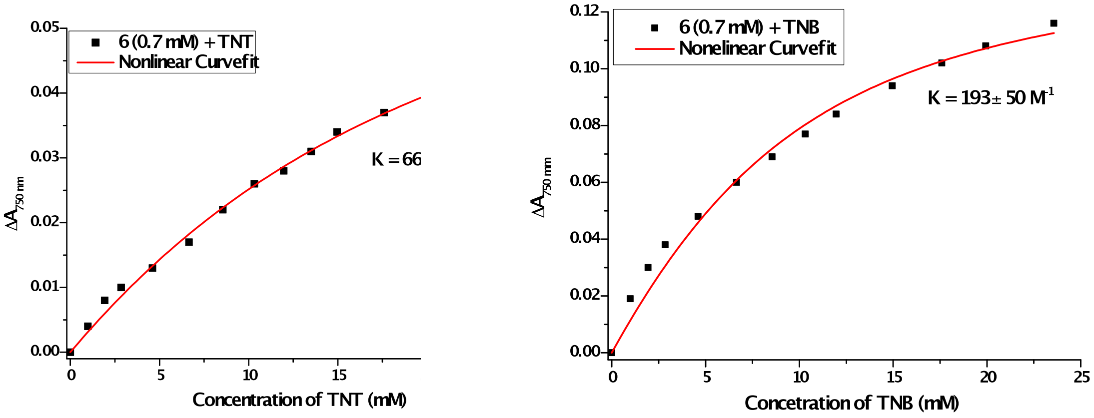


Figure S30. Binding data from titrations of **6** with TNT and TNB in $CHCl_3$.

IV. Cyclic Voltamograms

Cyclic voltammetry (CV) was conducted at ambient temperature in a standard three-electrode cell with a glassy carbon working electrode, a reference electrode (Ag/AgCl), and a counter electrode (Pt wire) (CV-50W voltammetric analyzer, BAS). All CV measurements were performed in 0.2 mM solutions of the compounds (**1, 2, 3,** and **4**) in the presence of 0.4 M tetrahexylammonium hexafluorophosphate (THAPF$_6$) in CHCl$_3$ after bubbling with nitrogen gas for 10 minutes; a scan rate of 100 mVs^{-1} was employed for all measurements. Values are expressed vs. Ag/AgCl. Results are presented in Figure S25–28 and summarized in Figure S29.

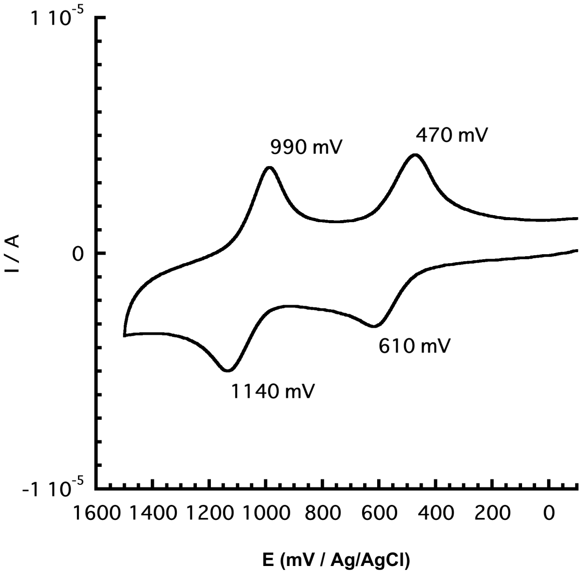


Figure S31. Cyclic voltammogram of a 0.2 mM solution of the pyrrolo-TTF **1** in CHCl$_3$ using 0.4 M TBAPF$_6$ as the supporting electrolyte, Ag/AgCl as the reference electrode, platinum wire as the counter electrode, glassy carbon as the working electrode, and a scan rate of 100 mV/s.

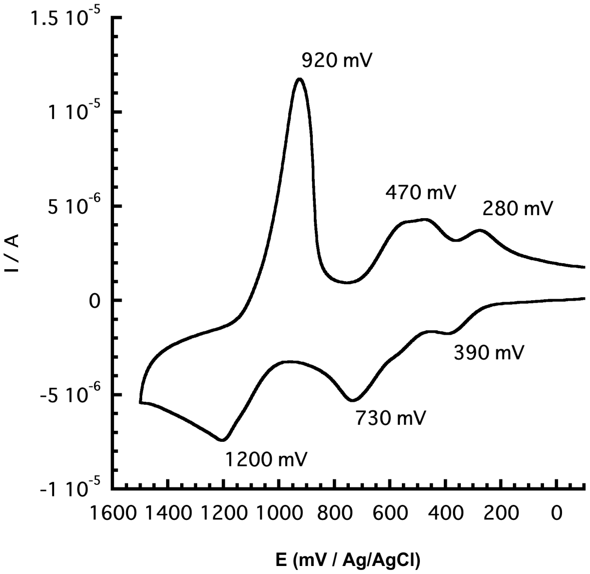


Figure S32. Cyclic voltammogram of a 0.2 mM solution of TTF-C4P **2** in $CHCl_3$ using 0.4 M $TBAPF_6$ as the supporting electrolyte, Ag/AgCl as the reference electrode, platinum wire as the counter electrode, glassy carbon as the working electrode, and a scan rate of 100 mV/s.

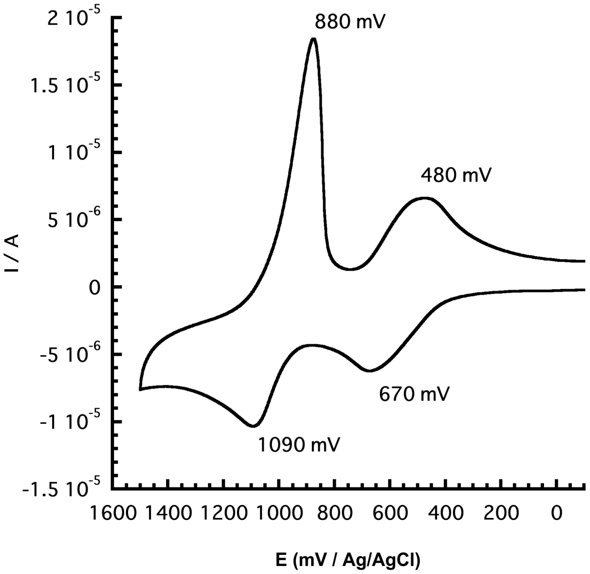


Figure S33. Cyclic voltammogram of a 0.2 mM solution of TTF-C5P **3** in $CHCl_3$ using 0.4 M $TBAPF_6$ as the supporting electrolyte, Ag/AgCl as the reference electrode, platinum wire as the counter electrode, glassy carbon as the working electrode, and a scan rate of 100 mV/s.

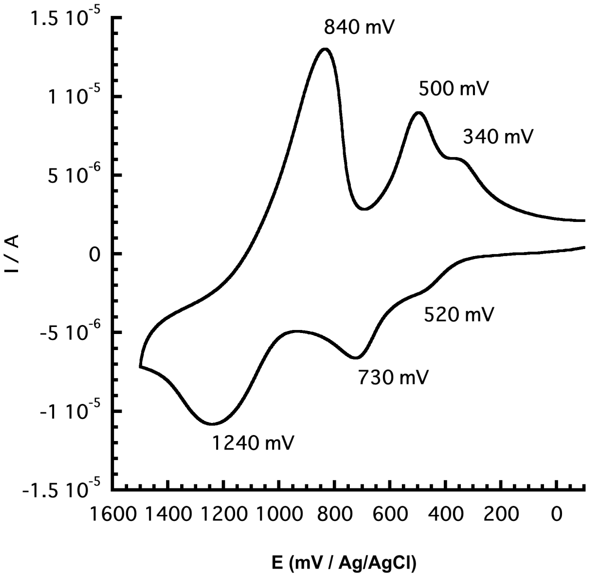


Figure S34. Cyclic voltammogram of a 0.2 mM solution of TTF-C6P **4** in $CHCl_3$ using 0.4 M $TBAPF_6$ as the supporting electrolyte, Ag/AgCl as the reference electrode, platinum wire as the counter electrode, glassy carbon as the working electrode, and a scan rate of 100 mV/s.

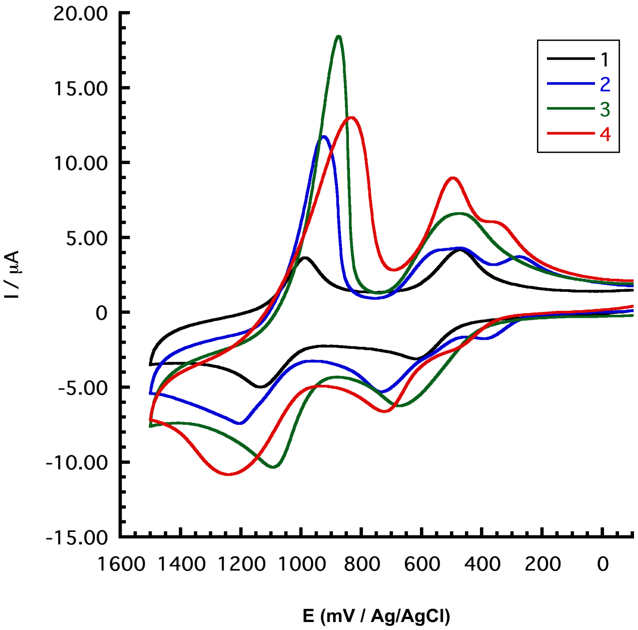


Figure S35. Overlaid cyclic voltammograms of 0.2 mM solutions of **1–4** in $CHCl_3$ using 0.4 M $TBAPF_6$ as the supporting electrolyte, Ag/AgCl as the reference electrode, platinum wire as the counter electrode, glassy carbon as the working electrode, and a scan rate of 100 mV/s.

V. References for SI

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