Supporting Information

The synthesis and application of *o*-carborane-based macrocyclic arenes

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Table of Contents

1. NMR Spectra and HR-MS (ESI) of New Compounds

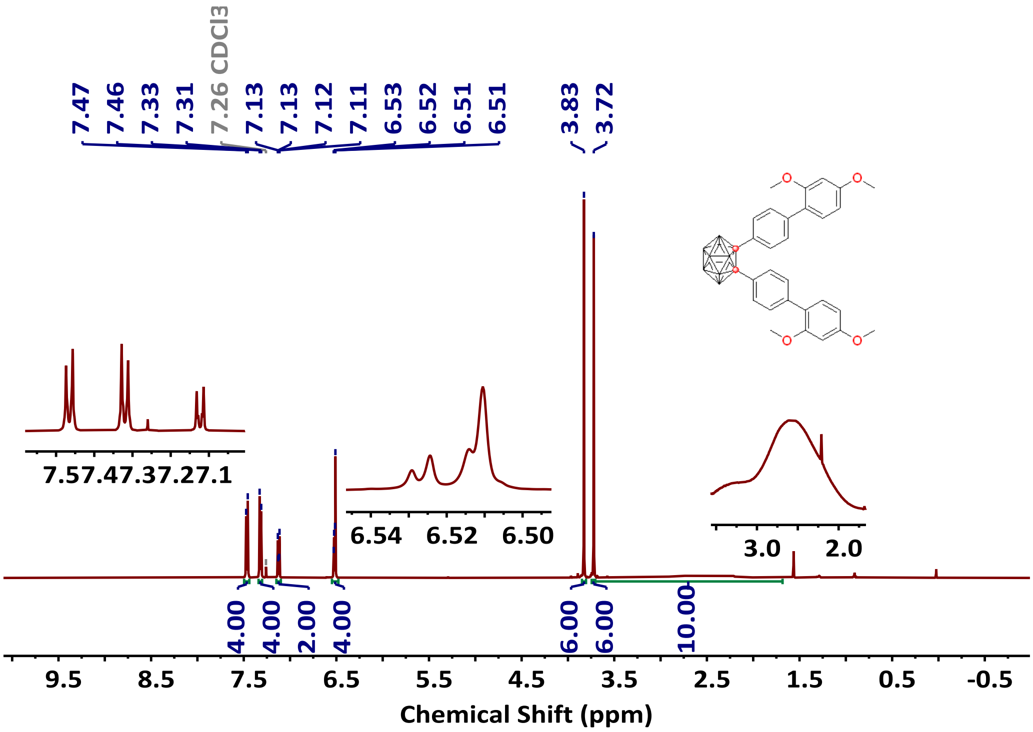

Figure S1. ^{1}H NMR spectrum of **2a** (500 MHz, $CDCl_3$).

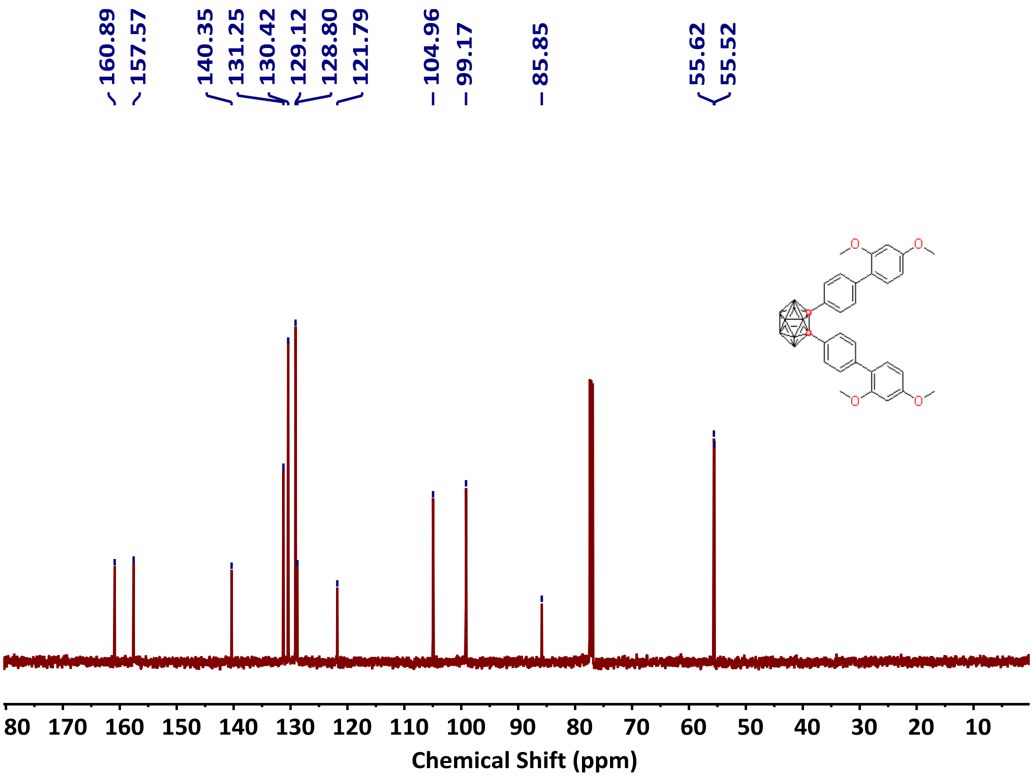

Figure S2. ^{13}C NMR spectrum of **2a** (126 MHz, $CDCl_3$).

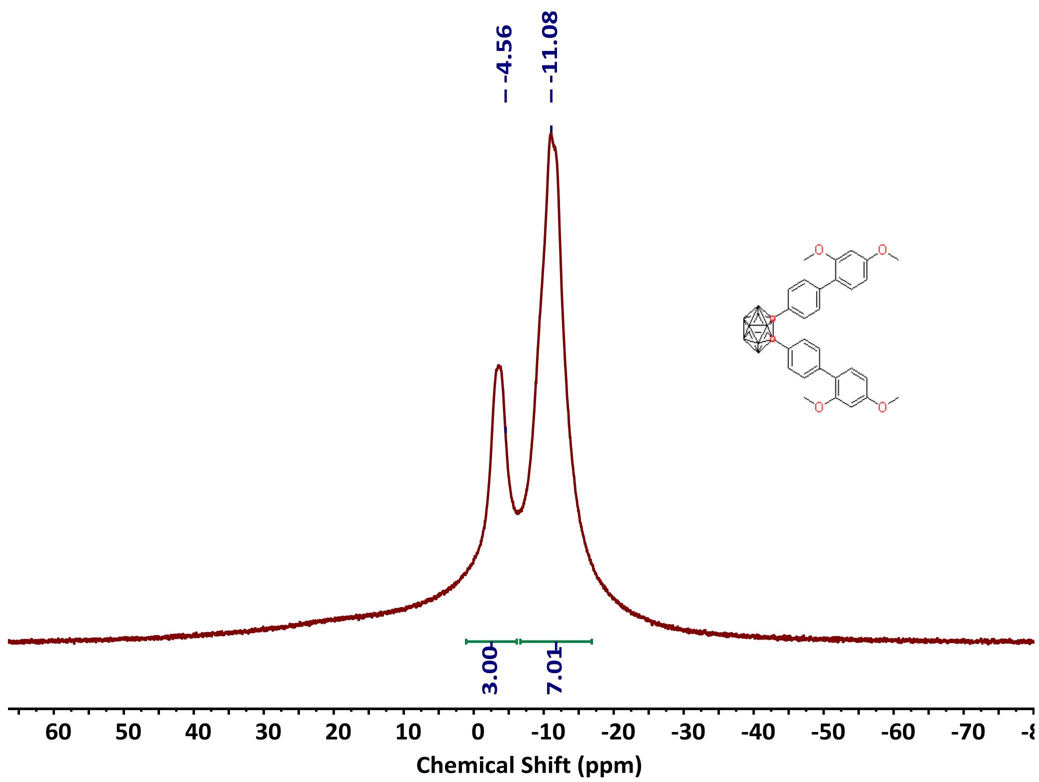

Figure S3. ^{11}B NMR spectrum of **2a** (160 MHz, $CDCl_3$).

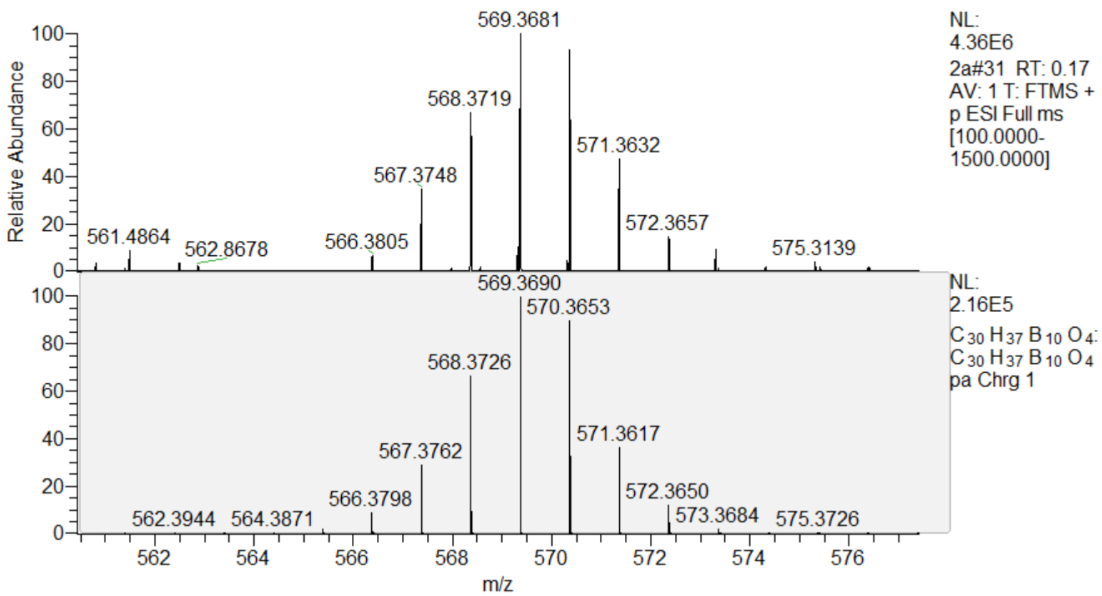

Figure S4. HR-MS (ESI) spectra of **2a**.

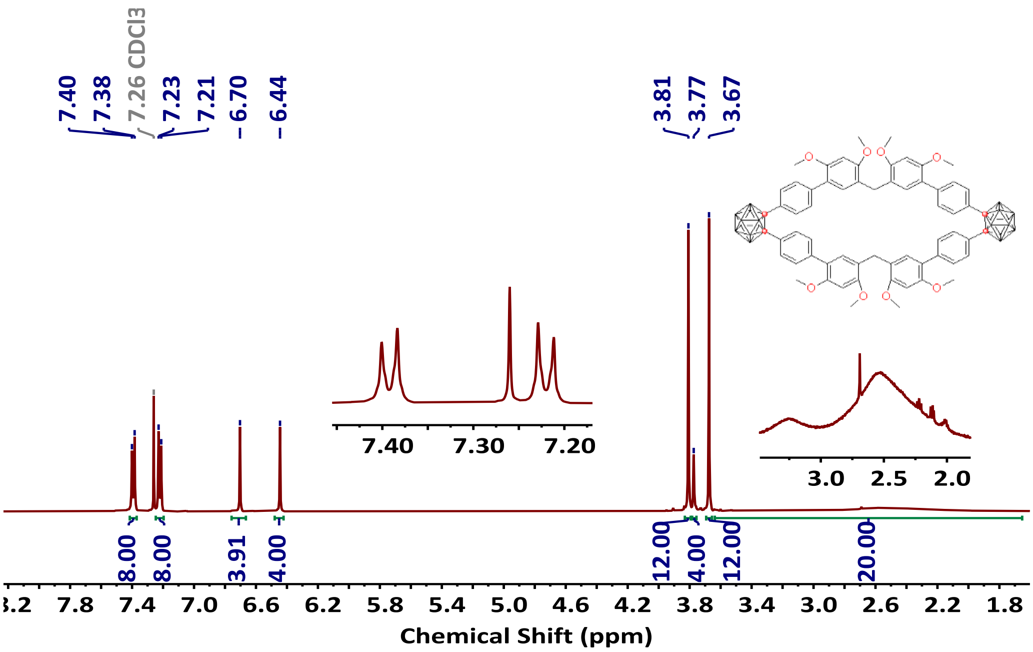

Figure S5. ^{1}H NMR spectrum of **3a** (500 MHz, $CDCl_3$).

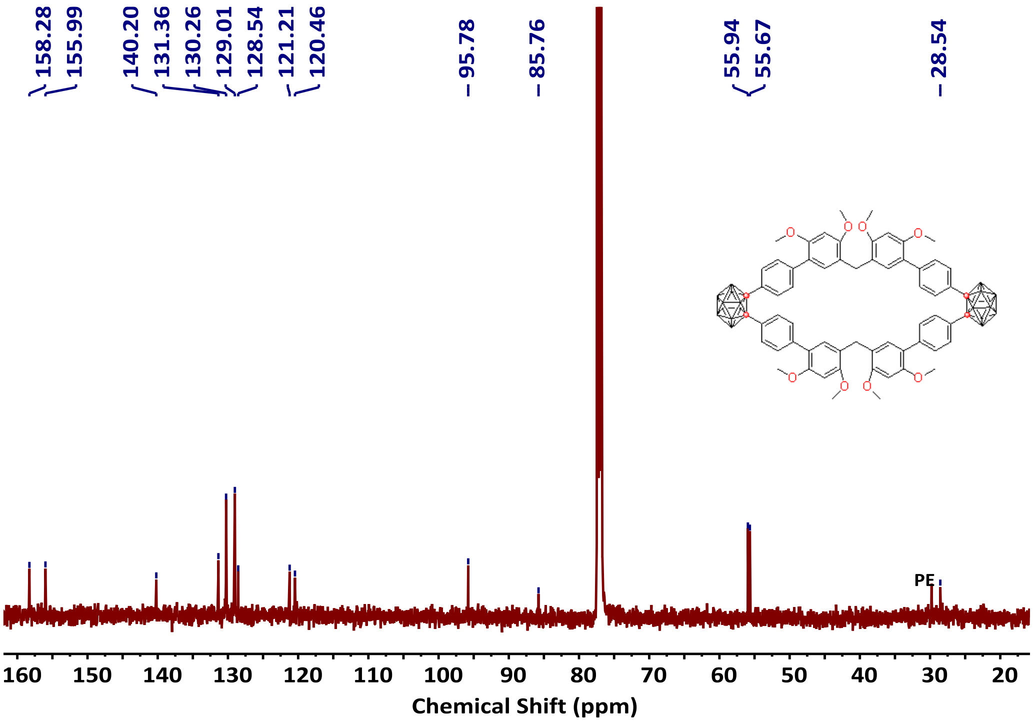

Figure S6. ^{13}C NMR spectrum of **3a** (126 MHz, $CDCl_3$).

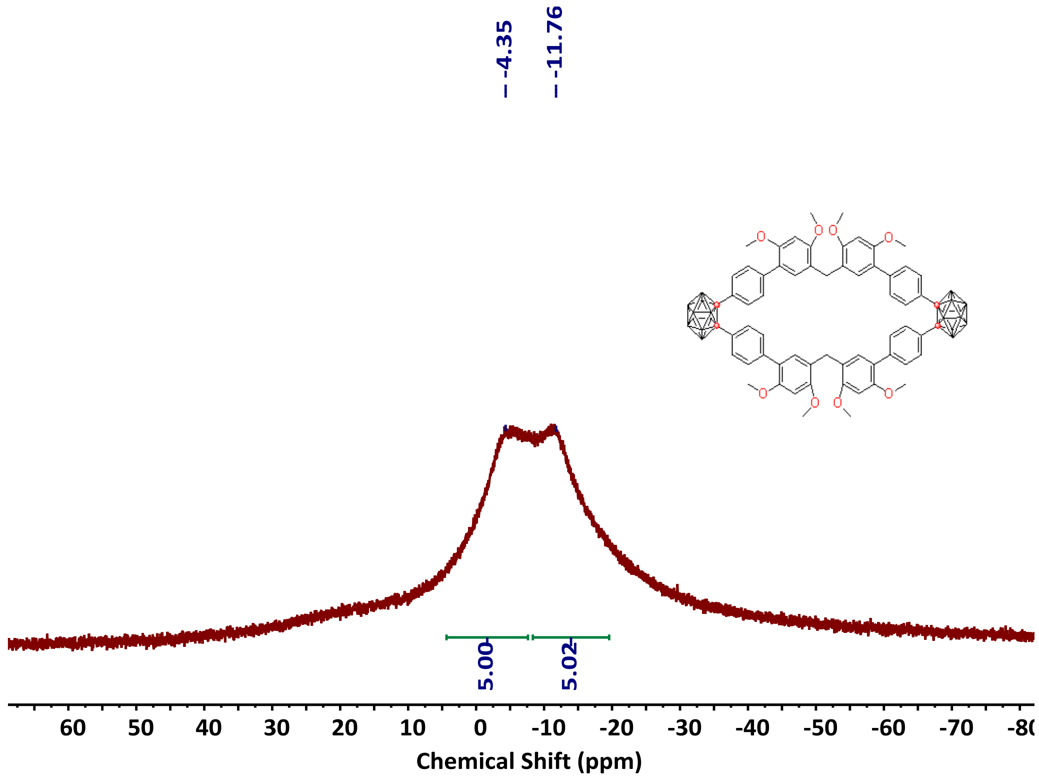

Figure S7. [11]B NMR spectrum of **3a** (160 MHz, $CDCl_3$).

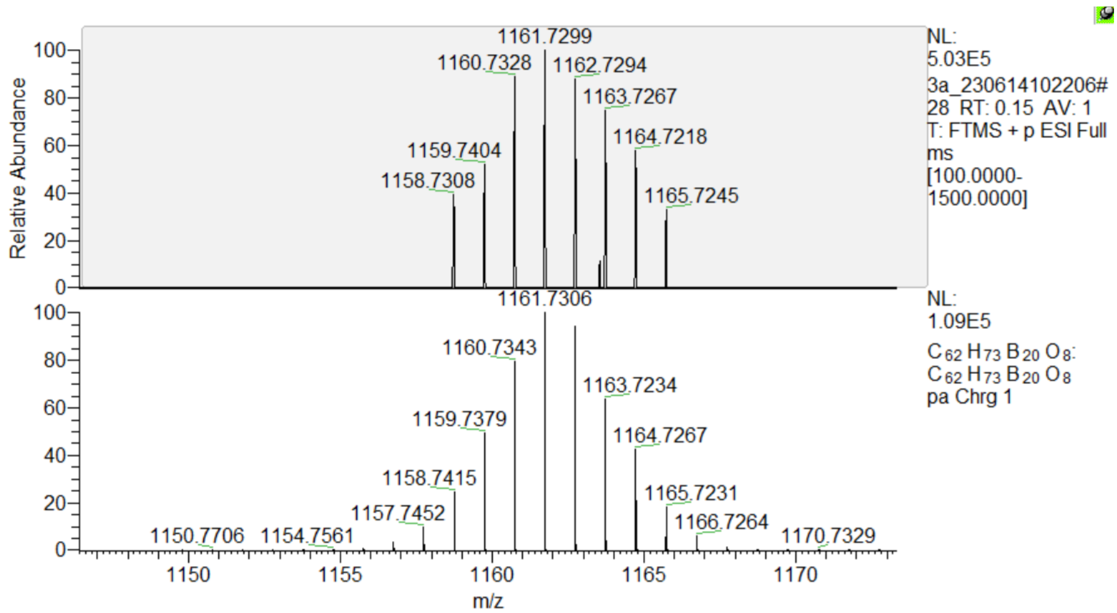

Figure S8. HR-MS (ESI) spectra of **3a**.

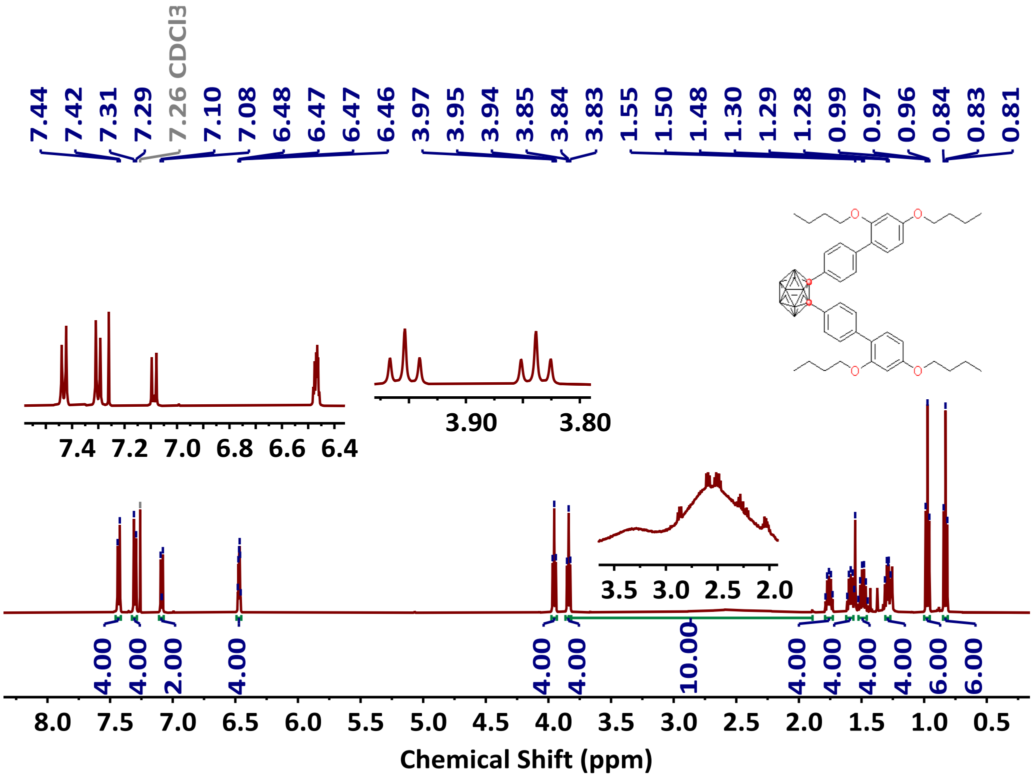

Figure S9. ^{1}H NMR spectrum of **2b** (500 MHz, $CDCl_3$).

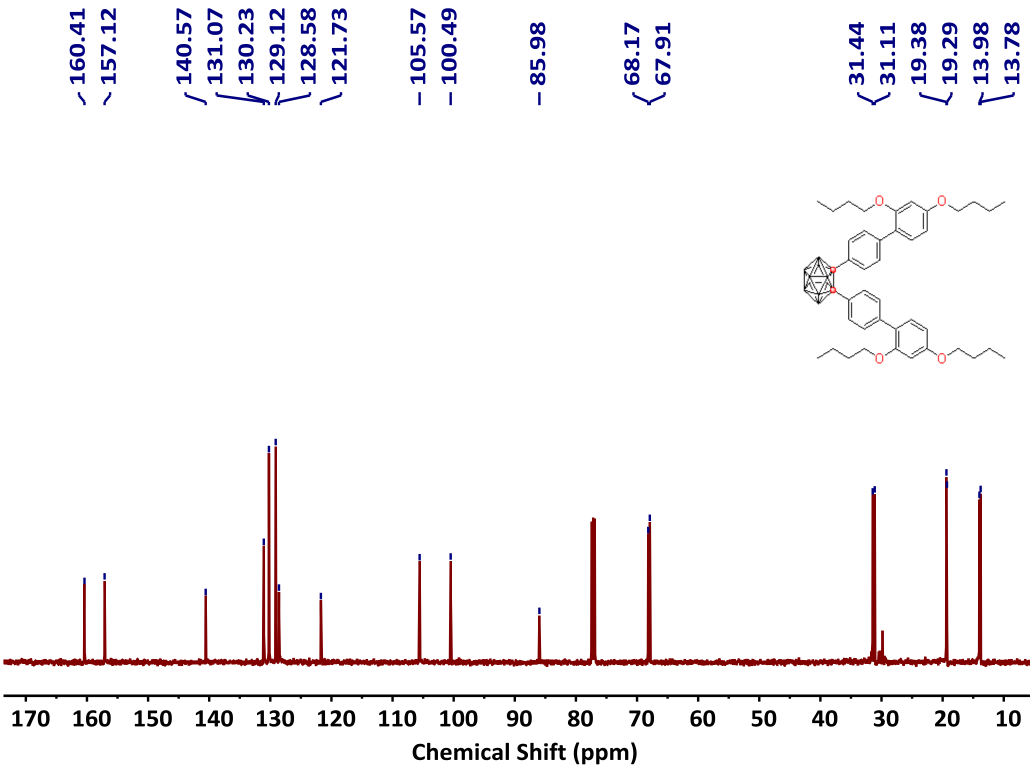

Figure S10. ^{13}C NMR spectrum of **2b** (126 MHz, $CDCl_3$).

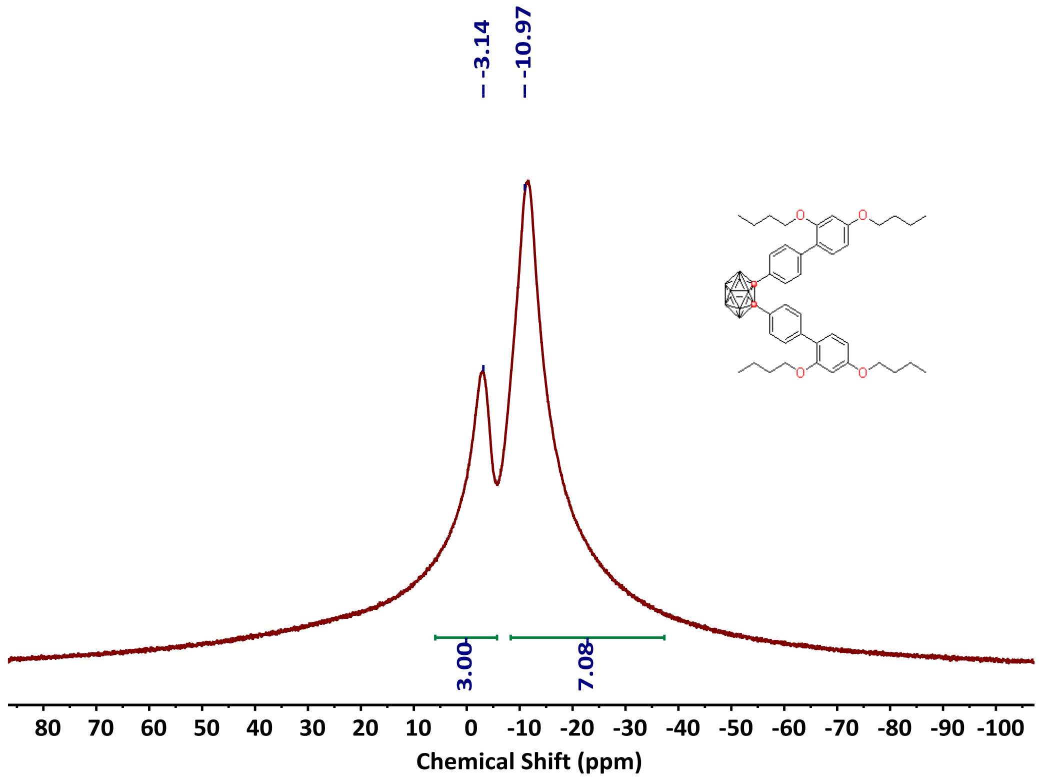

Figure S11. ^{11}B NMR spectrum of **2b** (160 MHz, $CDCl_3$).

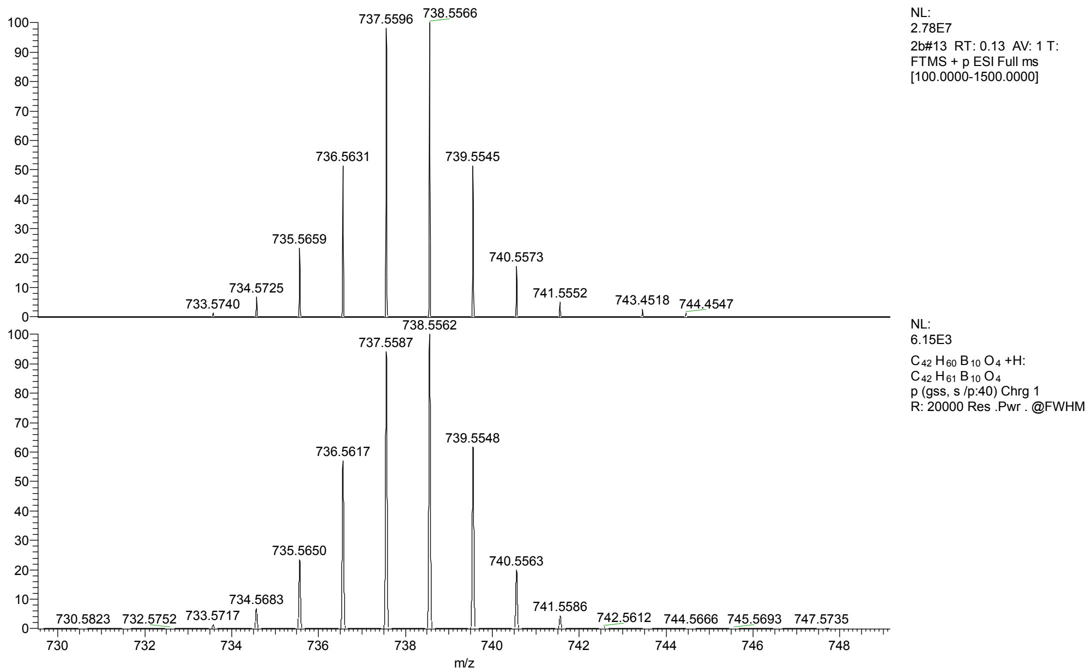

Figure S12. HR-MS (ESI) spectrum of **2b**.

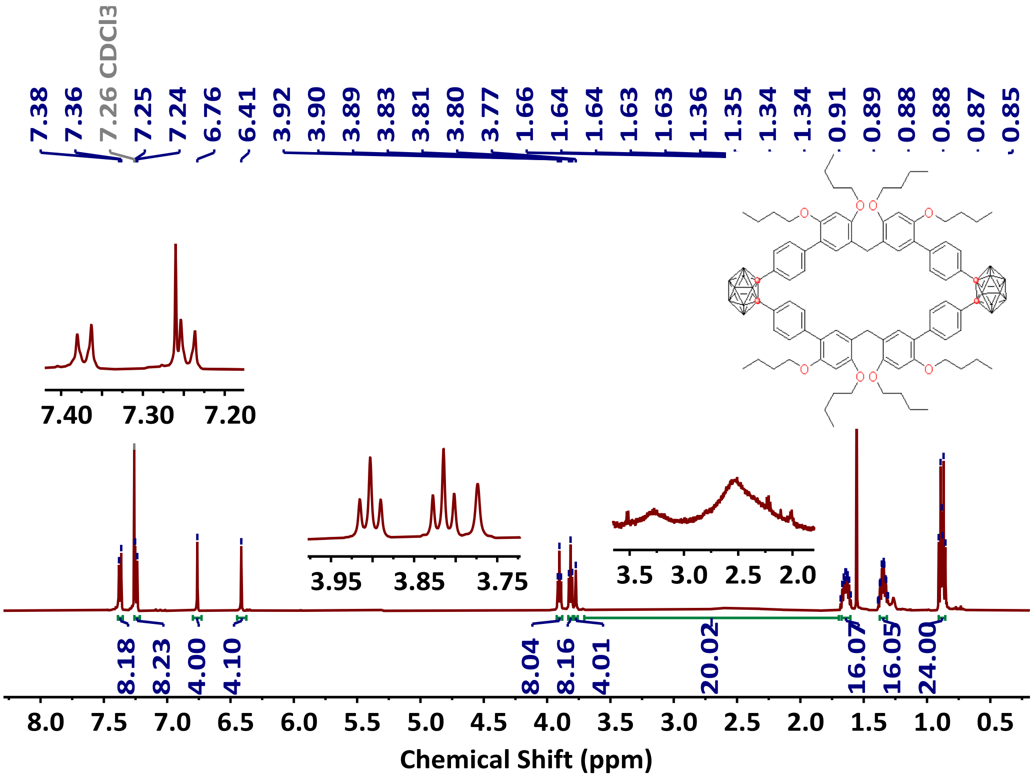

Figure S13. ^{1}H NMR spectrum of **3b** (500 MHz, $CDCl_3$).

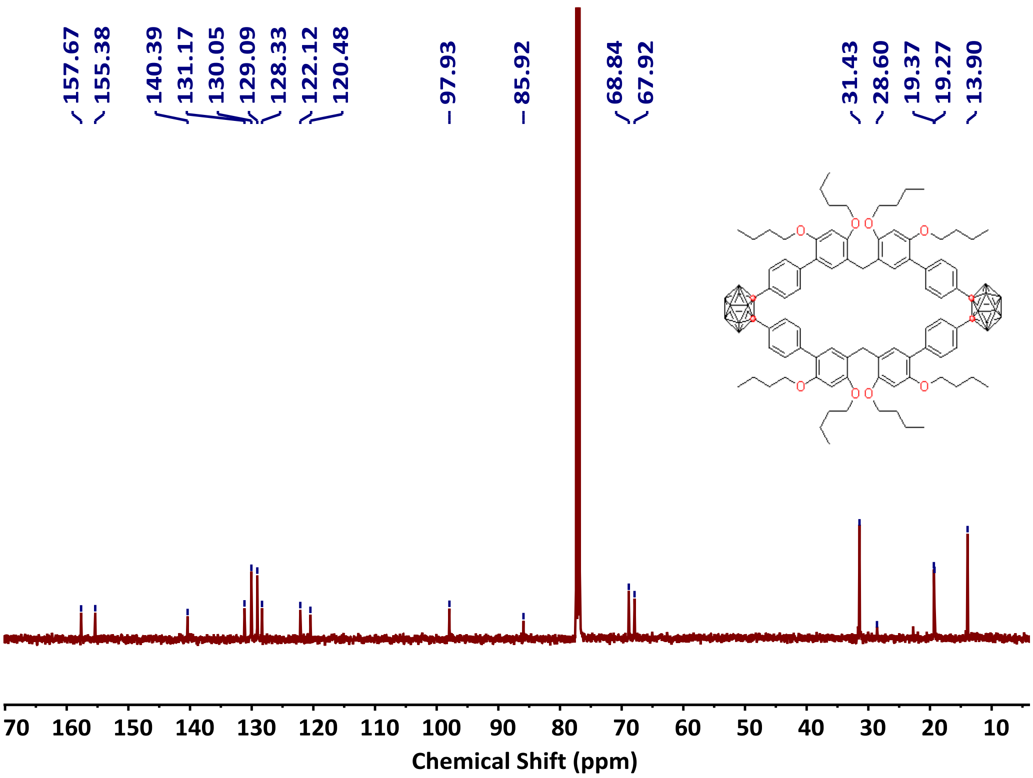

Figure S14. ^{13}C NMR spectrum of **3b** (126 MHz, $CDCl_3$).

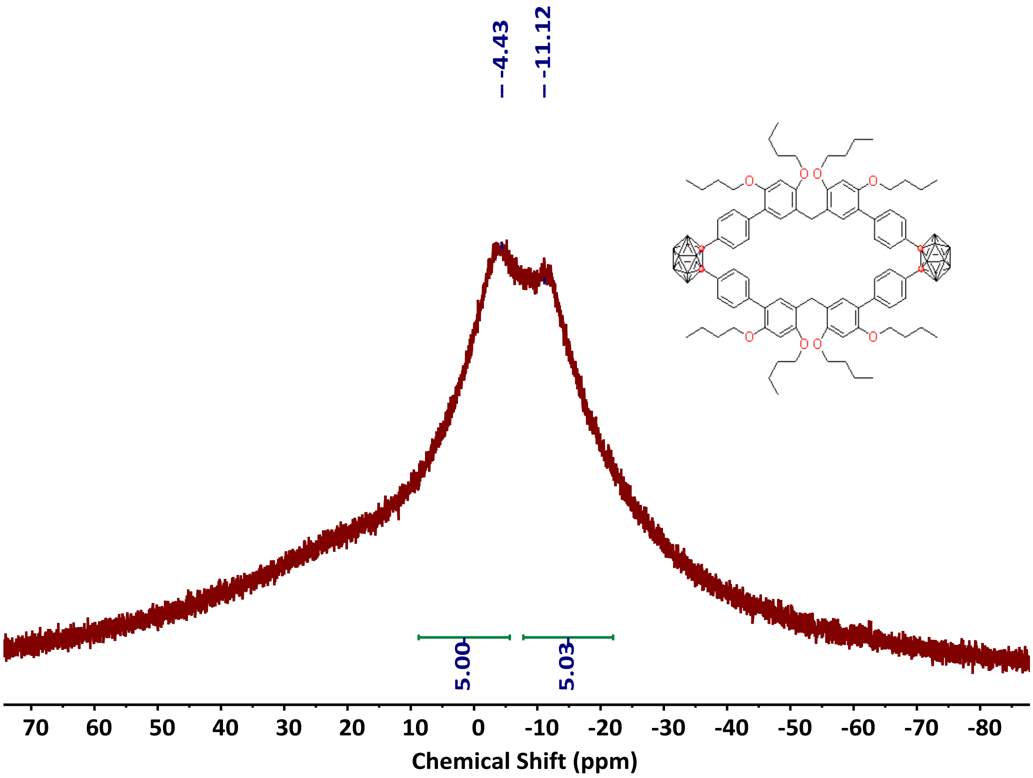

Figure S15. [11]B NMR spectrum of **3b** (160 MHz, $CDCl_3$).

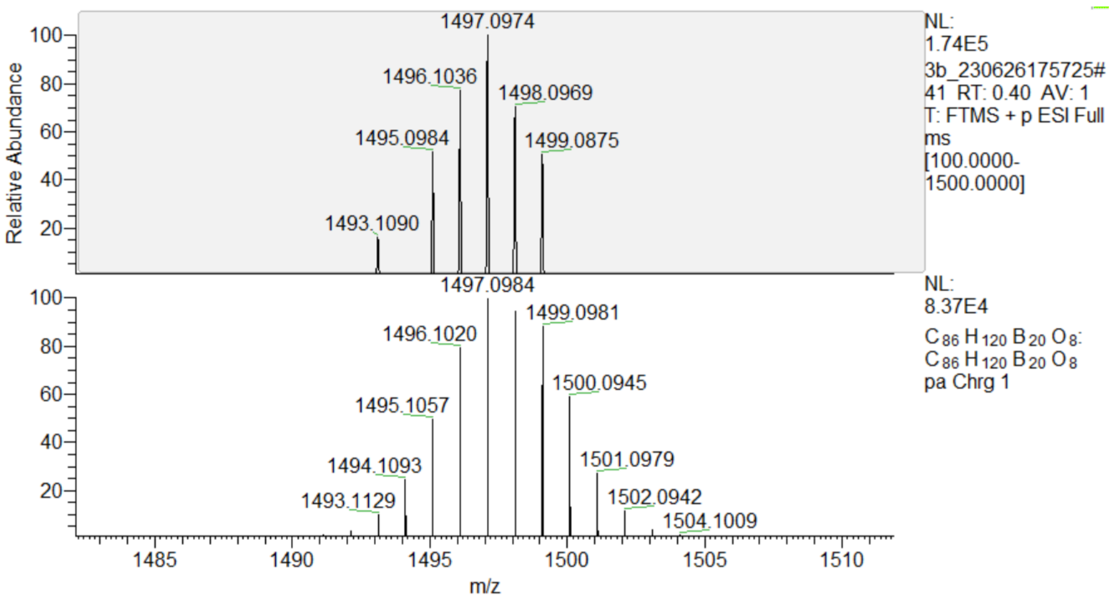

Figure S16. HR-MS (ESI) spectrum of **3b**.

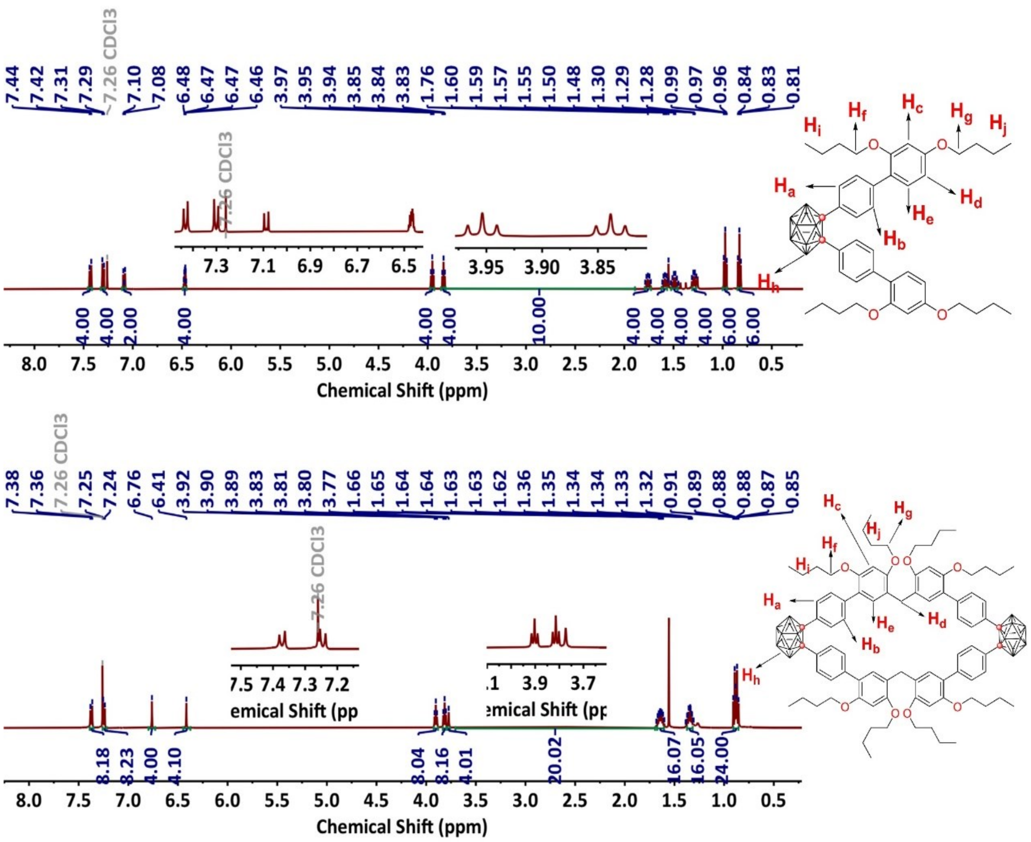

Figure S17. ^{1}H-NMR spectra of **2b** and **3b** in $CDCl_3$.

2. Crystal Data and DFT Calculations

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

Compound	3a	3b
Empirical formula	$C_{62}H_{72}B_{20}O_8$	$C_{86}H_{120}B_{20}O_8$
Formula weight	1161.40	1498.02
Temperature/K	293(2)	293(2)
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	C2/c
a/Å	19.3942(2)	35.4352(3)
b/Å	13.13040(10)	16.80310(10)
c/Å	33.0142(3)	17.1648(2)
α/°	90	90
β/°	96.0910(10)	103.8970(10)
γ/°	90	90
Volume/Å^3	8359.72(13)	9914.43(16)
Z	117	138
ρ_{calc}g/cm^3	1.749	1.740
μ/mm^{-1}	9.344	9.292
F(000)	4329.0	5106.0
Crystal size/mm^3	0.12 × 0.11 × 0.1	0.12 × 0.11 × 0.1
Radiation	CuKα (λ = 1.54184)	CuKα (λ = 1.54184)
2Θ range for data collection/°	4.582 to 153.78	5.854 to 151.948
Reflections collected	58489	48778
Data/restraints/parameters	16746/0/819	10095/152/556
Goodness-of-fit on F^2	1.066	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0732, wR_2 = 0.2255	R_1 = 0.0597, wR_2 = 0.1706
Final R indexes [all data]	R_1 = 0.0899, wR_2 = 0.2367	R_1 = 0.0654, wR_2 = 0.1756
CCDC number	2284988	2284986

DFT Calculations

Density functional theory (DFT) calculations were performed using Gaussian 16(Revision C.01). Calculations were carried out using the generalized B3LYP and 6-31G(d) basis groups with a single crystal as the starting geometrical configuration. Each structure was optimized to meet standard convergence criteria, and the existence of a local minimum was verified by a normal mode frequency calculation. Negative frequencies were not observed.

Table S2. Atomic coordinates of **3a** after geometry optimization.

Atom	X(Å)	Y(Å)	Z(Å)
O	7.46700000	8.71700000	21.85600000
O	11.53800000	1.59000000	16.92100000
O	8.66600000	11.96100000	18.44600000
O	14.51400000	2.42000000	20.64600000
O	8.80300000	6.23400000	13.77000000
O	16.63700000	10.48900000	19.31300000
O	6.94400000	3.37400000	17.22900000
O	12.95200000	12.62500000	21.64500000
C	8.34300000	9.55200000	21.20800000
C	12.42600000	4.59800000	24.74100000
C	11.84100000	2.09500000	18.16100000
C	9.63600000	9.60400000	21.74200000
C	8.94500000	11.17300000	19.51000000
C	12.40300000	3.49600000	22.14200000
C	10.19400000	5.17200000	15.35100000
C	7.99600000	10.30800000	20.10800000
H	7.11900000	10.24800000	19.75000000
C	10.31000000	4.36000000	16.46000000
H	11.17900000	4.21500000	16.81800000
C	13.31500000	2.48600000	20.01500000
C	8.90200000	5.43000000	14.88300000
C	10.24500000	11.24400000	20.03400000
C	10.77400000	2.65900000	18.85000000
C	9.23700000	3.74500000	17.08100000
C	12.40500000	5.18500000	26.10300000
C	13.10600000	2.01100000	18.72200000
H	13.82600000	1.63500000	18.22900000
C	10.18300000	6.86700000	24.31500000
H	9.87600000	5.98500000	24.48700000
C	7.78400000	4.84800000	15.50900000
H	6.90800000	5.03800000	15.19500000
C	11.46700000	3.64300000	24.38300000
H	10.83000000	3.35100000	25.02200000
C	10.55500000	10.46700000	21.12200000

H	11.43500000	10.51700000	21.47700000
C	11.41700000	5.63700000	14.65500000
C	11.10500000	7.44600000	25.18100000
C	15.71600000	8.66200000	14.67400000
C	12.67700000	11.77800000	19.47600000
C	11.44500000	3.12500000	23.11100000
H	10.76600000	2.50100000	22.87800000
C	7.96500000	4.00500000	16.57700000
C	14.53400000	10.67900000	18.30400000
C	10.09000000	8.86900000	22.95500000
C	11.65600000	6.73200000	26.38000000
C	9.70100000	7.53800000	23.21100000
H	9.10200000	7.10000000	22.61700000
C	9.40600000	2.75700000	18.24500000
H	8.77100000	3.01300000	18.95800000
H	9.14500000	1.85800000	17.92600000
C	11.51600000	8.76000000	24.91400000
H	12.15600000	9.17600000	25.48100000
C	6.14100000	8.60900000	21.32400000
H	6.18600000	8.27000000	20.40500000
H	5.71600000	9.49100000	21.32400000
H	5.61700000	7.99300000	21.87400000
C	11.01400000	3.11600000	20.13100000
H	10.29800000	3.51700000	20.61000000
C	13.82400000	6.43400000	13.40300000
C	13.22600000	11.13200000	18.37800000
H	12.66600000	10.99000000	17.62300000
C	15.34500000	10.89900000	19.40800000
C	13.52600000	11.98900000	20.56100000
C	11.56900000	6.92300000	14.12000000
H	10.85100000	7.54100000	14.18500000
C	13.41300000	4.92100000	23.81500000
H	14.10400000	5.52400000	24.06400000
C	12.26400000	3.01700000	20.75700000
C	15.00300000	9.98700000	17.07200000
C	7.32300000	12.02000000	17.97800000
H	7.25300000	12.70200000	17.27700000
H	6.72700000	12.25100000	18.72100000
H	7.06700000	11.14900000	17.61300000
C	13.69300000	5.15100000	13.92900000
H	14.41800000	4.53900000	13.86900000
C	11.00900000	9.44700000	23.85500000
H	11.28400000	10.34600000	23.71800000

C	14.99400000	9.86300000	14.63500000
H	14.73600000	10.23200000	13.79800000
C	14.85000000	11.56900000	20.55200000
H	15.41400000	11.72800000	21.29800000
C	12.74500000	7.31000000	13.50400000
H	12.81800000	8.18600000	13.14600000
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H	14.10300000	4.61700000	21.93900000
C	14.65800000	10.51000000	15.80900000
H	14.17900000	11.33100000	15.76200000
C	15.76800000	8.82700000	17.08800000
H	16.07100000	8.47900000	17.91900000
C	12.58300000	0.99200000	16.16400000
H	12.19900000	0.50100000	15.41000000
H	13.08800000	0.37500000	16.73300000
H	13.18400000	1.69000000	15.82700000
C	11.23800000	12.25000000	19.46000000
H	10.98200000	12.45300000	18.52800000
H	11.17500000	13.09000000	19.98100000
C	16.09900000	8.16700000	15.90900000
H	16.59800000	7.36000000	15.95100000
C	12.50900000	4.76700000	14.53700000
H	12.43600000	3.88600000	14.88600000
C	7.48500000	6.49700000	13.24400000
H	7.54900000	7.14200000	12.51100000
H	6.91600000	6.86400000	13.95400000
H	7.09300000	5.66200000	12.91100000
C	15.10000000	6.79600000	12.67400000
C	16.12100000	7.99300000	13.39700000
C	15.65300000	2.00700000	19.88000000
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H	15.52700000	1.08300000	19.57700000
H	16.45900000	2.06200000	20.43700000
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H	15.77800000	4.52700000	11.80700000
B	16.90600000	8.87100000	12.18800000
H	17.17300000	9.95300000	12.29200000
B	17.77300000	7.59500000	13.11700000
H	18.61000000	7.82000000	13.82700000
B	16.38700000	8.16500000	10.63100000
H	16.32400000	8.78400000	9.69900000
B	15.22000000	6.86700000	10.95700000
H	14.38300000	6.62600000	10.25000000
B	17.75100000	6.12800000	12.14900000
H	18.59100000	5.39200000	12.23100000
B	16.90700000	6.45700000	10.60500000
H	17.19200000	5.93300000	9.65700000
B	17.95300000	7.70600000	11.32800000
H	18.91600000	8.01800000	10.84900000

Table S3. Atomic coordinates of **3b** after geometry optimization.

O	6.55060000	12.10810000	11.60270000
O	11.34530000	8.72390000	9.31870000
O	8.38960000	10.37030000	7.47720000
O	15.95950000	9.74010000	10.35680000
C	9.91700000	12.02170000	10.27580000
H	10.76520000	12.31120000	10.52070000
C	8.85960000	12.27390000	11.15700000
C	9.08090000	13.05190000	12.39510000
C	9.49700000	14.66350000	14.68860000
C	9.77920000	11.37440000	9.07390000
C	15.69520000	12.31310000	9.20320000
C	14.60380000	11.31030000	9.26700000
C	7.59450000	11.82840000	10.75690000
C	18.72960000	15.38390000	8.82380000
C	17.69640000	14.28810000	9.01350000
C	8.48820000	10.95980000	8.70140000
C	13.34560000	11.64210000	8.73420000
H	13.23880000	12.47820000	8.34100000
C	12.25700000	10.79110000	8.76350000
C	10.94260000	11.16430000	8.12720000
H	10.70030000	10.46560000	7.49780000
H	11.07140000	11.97890000	7.61850000
C	7.41350000	11.16260000	9.55500000
H	6.56800000	10.85240000	9.32050000
C	12.44570000	9.54190000	9.33770000
C	9.61180000	15.58570000	15.86980000
C	8.69200000	13.53310000	14.73860000
H	8.28640000	13.29620000	15.54220000
C	10.11010000	14.95270000	13.48320000
H	10.67330000	15.69070000	13.42290000
C	8.48240000	12.75600000	13.61770000
H	7.92450000	12.01470000	13.68150000
C	9.90430000	14.17050000	12.37440000
H	10.33190000	14.39890000	11.58120000
C	15.42120000	13.65580000	9.39460000
H	14.54740000	13.91660000	9.58260000
C	17.98620000	12.94860000	8.81870000
H	18.85840000	12.69640000	8.61860000
C	16.40600000	14.61530000	9.31360000
H	16.18710000	15.50660000	9.46880000
C	14.73040000	10.05650000	9.84120000
C	17.01630000	11.99250000	8.91350000

H	17.24690000	11.10120000	8.78070000
C	13.65790000	9.18230000	9.87230000
H	13.76020000	8.34210000	10.26030000
C	7.09070000	9.93550000	7.02180000
H	6.47820000	10.68710000	6.99080000
H	6.72880000	9.26750000	7.62710000
C	5.21530000	11.81020000	11.15520000
H	5.10520000	10.85240000	11.05870000
H	5.05420000	12.22600000	10.29300000
B	18.67400000	16.68760000	9.94300000
H	17.92390000	16.75750000	10.74480000
C	11.46330000	7.37580000	9.79120000
H	11.75230000	7.36730000	10.71720000
H	12.11210000	6.88840000	9.26010000
B	20.33850000	14.97150000	8.40650000
H	20.66690000	13.93870000	8.22370000
C	16.17240000	8.44950000	10.97590000
H	15.96800000	7.73880000	10.34820000
H	15.59540000	8.35060000	11.75020000
B	9.24030000	17.25340000	15.71630000
H	8.88400000	17.71020000	14.78170000
B	19.28830000	15.83550000	7.28220000
H	18.94530000	15.36170000	6.34930000
B	18.25110000	16.89550000	8.21860000
H	17.22480000	17.12900000	7.89610000
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B	19.27530000	18.10720000	9.05150000
H	18.91570000	19.12290000	9.27220000
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H	4.38480000	13.28600000	12.21400000
H	3.36830000	12.15610000	11.82600000
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H	21.69630000	16.28710000	6.67350000
B	20.93910000	17.77320000	8.59100000
H	21.67420000	18.58440000	8.50650000
B	19.66210000	17.55680000	7.38400000
H	19.56360000	18.22310000	6.51310000
C	10.12580000	6.75380000	9.66540000
H	10.19950000	5.83350000	9.96200000
H	9.53020000	7.20540000	10.28440000
C	17.60630000	8.37280000	11.38980000
H	17.73000000	7.51050000	11.81910000

H	18.13200000	8.35740000	10.57580000
C	8.32820000	8.82440000	3.53680000
H	8.50730000	9.55720000	2.94190000
H	7.55780000	8.34380000	3.22810000
H	9.08620000	8.23640000	3.55400000
C	7.26010000	9.36630000	5.68540000
H	7.06000000	8.42730000	5.82680000
H	6.49340000	9.71740000	5.20600000
C	9.51730000	6.74700000	8.43410000
H	10.10340000	6.29020000	7.80990000
H	9.43770000	7.66550000	8.13230000
C	8.09650000	6.07890000	8.34790000
H	7.68840000	6.30220000	7.50810000
H	7.54570000	6.39930000	9.06700000
H	8.18680000	5.12460000	8.41520000
C	4.42940000	11.75390000	13.38840000
H	4.33760000	10.79280000	13.29180000
H	5.33550000	11.92780000	13.68500000
C	18.17890000	9.31350000	12.21750000
H	17.65100000	9.35610000	13.02970000
H	18.10300000	10.17750000	11.78290000
C	19.61880000	9.13120000	12.61760000
H	20.18610000	9.56570000	11.97780000
H	19.82610000	8.19380000	12.64510000
H	19.76300000	9.51460000	13.48490000
C	8.08060000	9.32880000	4.86110000
H	8.83270000	8.96410000	5.35260000
H	8.29300000	10.26960000	4.75420000
C	3.58250000	12.16120000	14.36270000
H	2.94130000	12.77130000	13.99360000
H	4.08090000	12.60090000	15.05590000
H	3.12840000	11.39950000	14.73000000
O	23.05410000	12.10810000	14.26350000
O	18.25950000	8.72390000	16.54750000
O	21.21510000	10.37030000	18.38900000
O	13.64520000	9.74010000	15.50940000
C	19.68780000	12.02170000	15.59040000
H	18.83950000	12.31120000	15.34560000
C	20.74520000	12.27390000	14.70930000
C	20.52380000	13.05190000	13.47110000
C	20.10770000	14.66350000	11.17770000
C	19.82560000	11.37440000	16.79240000
C	13.90950000	12.31310000	16.66300000

C	15.00100000	11.31030000	16.59920000
C	22.01020000	11.82840000	15.10930000
C	10.87510000	15.38390000	17.04240000
C	11.90840000	14.28810000	16.85270000
C	21.11650000	10.95980000	17.16480000
C	16.25910000	11.64210000	17.13210000
H	16.36600000	12.47820000	17.52520000
C	17.34770000	10.79110000	17.10280000
C	18.66220000	11.16430000	17.73910000
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C	12.58840000	11.99250000	16.95270000
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H	17.85240000	7.36730000	15.14900000
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B	10.31640000	15.83550000	18.58400000
H	10.65940000	15.36170000	19.51690000
B	11.35370000	16.89550000	17.64770000
H	12.37990000	17.12900000	17.97010000
B	21.38590000	16.20360000	9.20320000
H	22.41400000	15.96840000	9.51710000
B	10.32940000	18.10720000	16.81480000
H	10.68910000	19.12290000	16.59410000
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B	8.65050000	16.38770000	18.38750000
H	7.90850000	16.28710000	19.19280000
B	8.66570000	17.77320000	17.27520000
H	7.93050000	18.58440000	17.35970000
B	9.94270000	17.55680000	18.48230000
H	10.04110000	18.22310000	19.35310000
C	19.47900000	6.75380000	16.20090000
H	19.40520000	5.83350000	15.90430000
H	20.07450000	7.20540000	15.58180000
C	11.99850000	8.37280000	14.47650000
H	11.87470000	7.51050000	14.04710000
H	11.47270000	8.35740000	15.29040000
C	21.27660000	8.82440000	22.32950000
H	21.09740000	9.55720000	22.92440000
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H	20.51850000	8.23640000	22.31220000
C	22.34470000	9.36630000	20.18080000
H	22.54480000	8.42730000	20.03940000
H	23.11130000	9.71740000	20.66020000
C	20.08740000	6.74700000	17.43210000
H	19.50130000	6.29020000	18.05640000
H	20.16710000	7.66550000	17.73390000
C	21.50820000	6.07890000	17.51830000

H	21.91640000	6.30220000	18.35810000
H	22.05910000	6.39930000	16.79930000
H	21.41790000	5.12460000	17.45110000
C	25.17530000	11.75390000	12.47790000
H	25.26710000	10.79280000	12.57440000
H	24.26930000	11.92780000	12.18130000
C	11.42590000	9.31350000	13.64880000
H	11.95370000	9.35610000	12.83660000
H	11.50180000	10.17750000	14.08330000
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H	9.84180000	9.51460000	12.38130000
C	21.52410000	9.32880000	21.00510000
H	20.77200000	8.96410000	20.51370000
H	21.31170000	10.26960000	21.11200000
C	26.02220000	12.16120000	11.50360000
H	26.66350000	12.77130000	11.87260000
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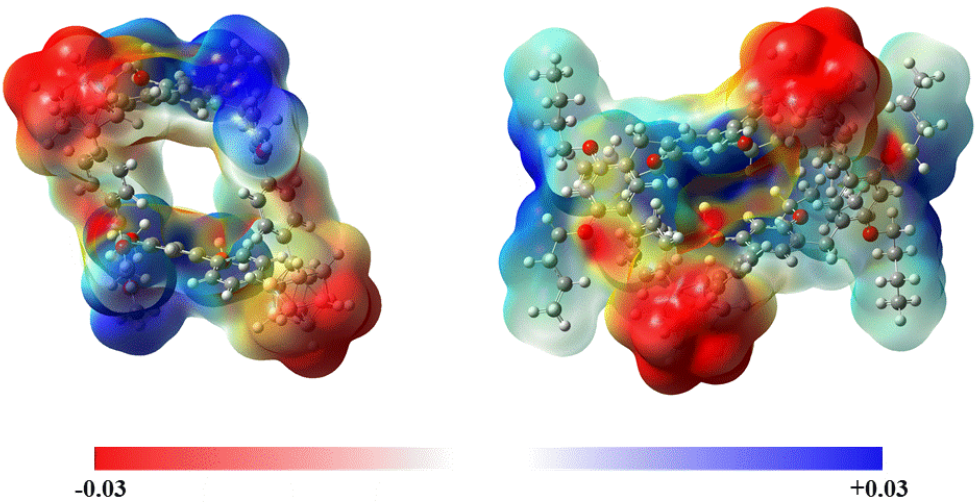

Figure S18. Electrostatic potential maps of **3a**(left) and **3b**(right).

3. Fluorescence Properties

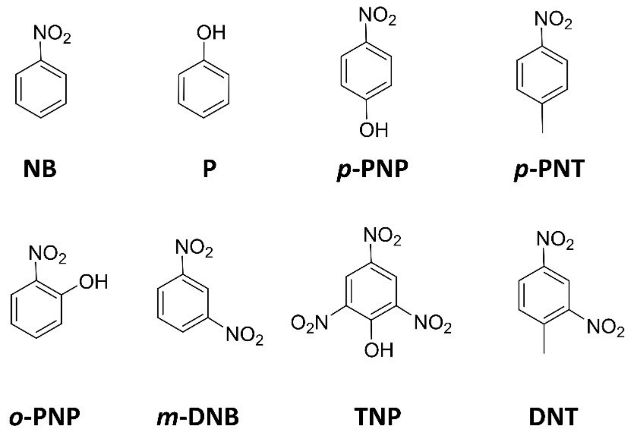

Figure S19. Structure and the abbreviation of guests

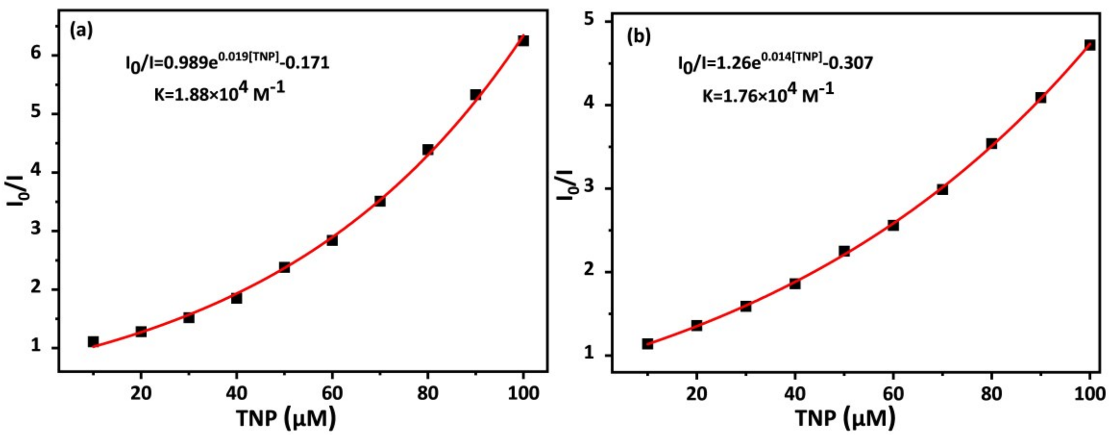

Figure S20. The nonlinear curve-fitting for the quenching constant of (a) **3a** and (b) **3b** with TNP.

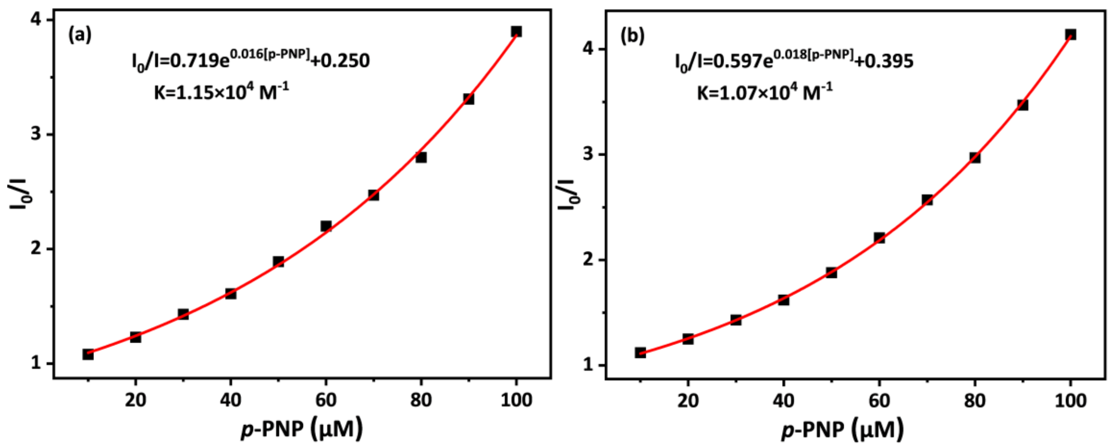

Figure S21. The nonlinear curve-fitting for the quenching constant of (a) **3a** and (b) **3b** with *p*-PNP.

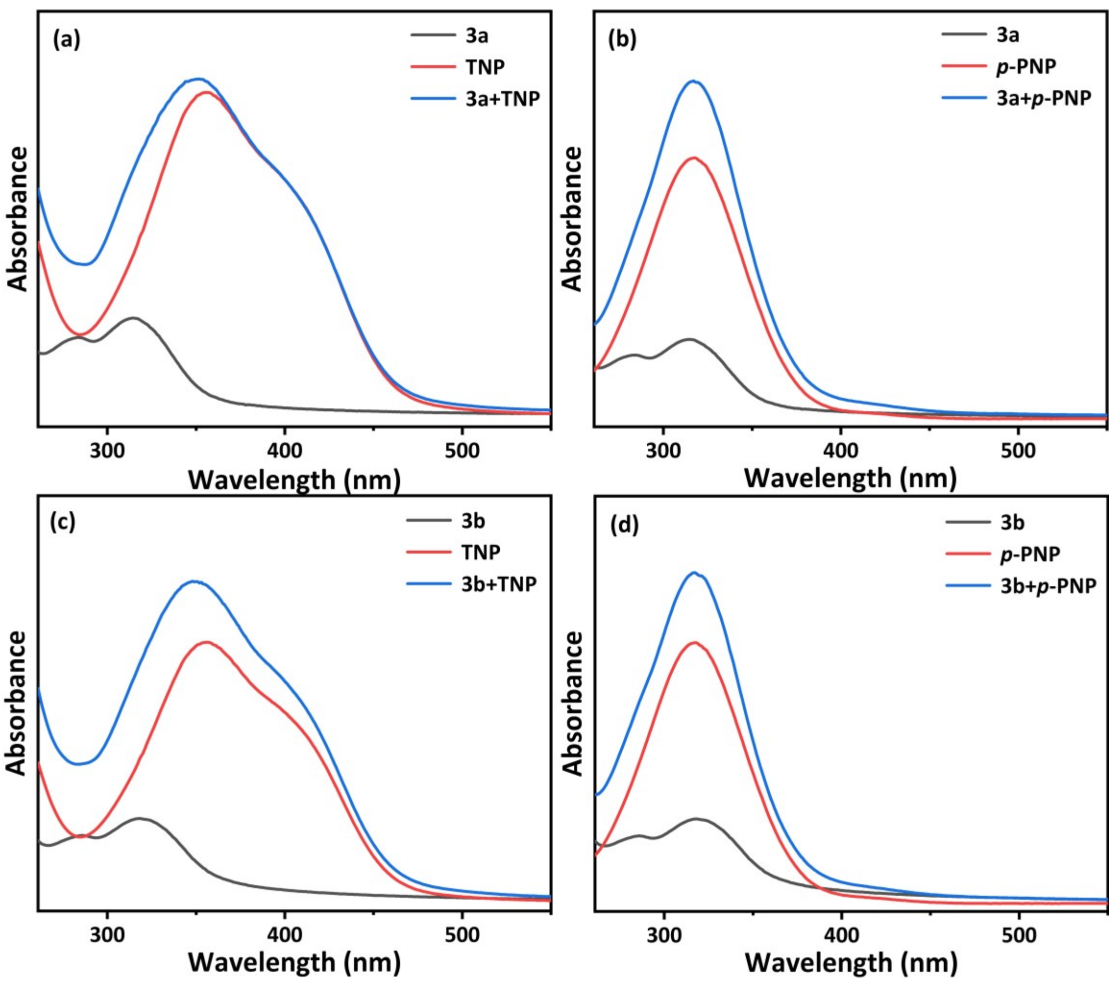

Figure S22. UV-vis spectra of **3a**, **3b**, TNP, *p*-PNP, and mixtures in H_2O/THF (95:5).

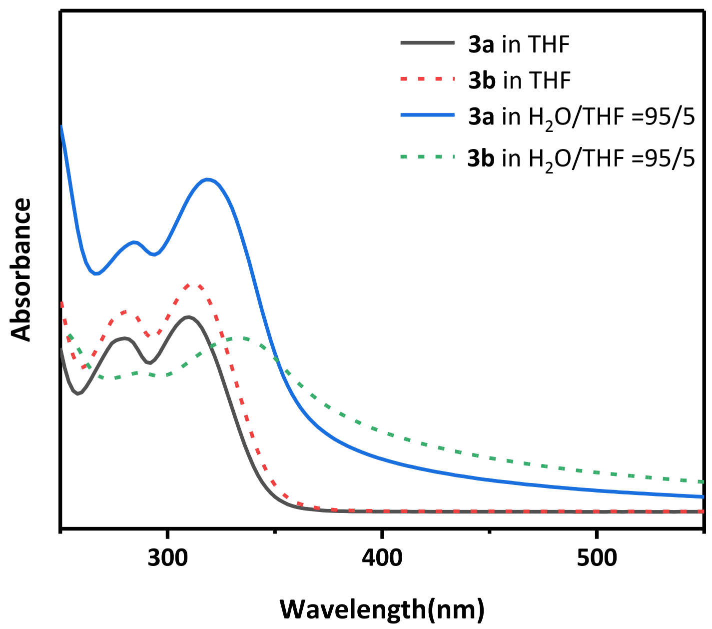

Figure S23. UV-vis spectra of **3a**, **3b** in THF and in H_2O/THF (95:5).

Table S4. UV absorption peak data of **3a**, **3b** in THF and in H_2O/THF (95:5).

Comp.	**λ_{abs}(nm)**	**λ_{abs}(nm)**
3a in THF	280	310
3a in H_2O/THF =95/5	284	318
3b in THF	282	312
3b in H_2O/THF =95/5	287	333

Observation of Figure S23 and datas in Table S4 reveals that the H_2O/THF (95:5) solvent caused red shift in the UV absorption, with the most pronounced red shift being the second UV absorption peak.

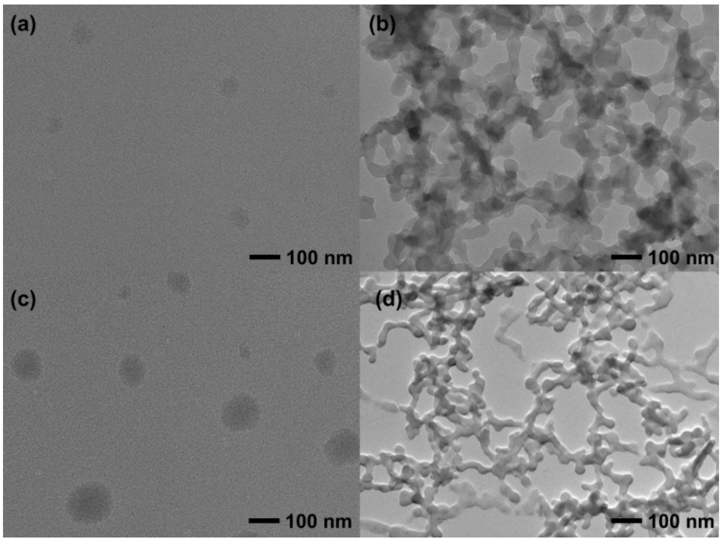

Figure S24. The TEM images of **3a** (a) and **3b** (c)in THF solution and **3a** (b) and **3b** (d)in H_2O/THF (99:1).

Quantum yields determination:

Absolute quantum yields of all compounds in THF/water or in solid state were measured by employing an integrating sphere. The Principle of Absolute Quantum Yield Measurements. The absolute fluorescence quantum yield, η, is, by definition, the ratio of the number of photons emitted to the number of photons absorbed:

$$\eta = \frac{N^{em}}{N^{abs}} \quad \cdots\cdots\cdots\cdots(1)$$

There are two different methods for the measurement of the absolute fluorescence quantum yield: “Direct Excitation” measurements and “Direct & Indirect Excitation” measurements. With “Direct Excitation” measurements one records the scatter and the emission of the sample being directly exited by the radiation from the excitation monochromator only, whereas with “Direct and Indirect Excitation” one also records the emission of the sample while it is in a position where it is only indirectly excited by excitation radiation bouncing within the sphere.

“Direct Excitation” Method

This method only requires two experimental setups, see Figure S25. Note that with the “Direct Excitation” method the emission measurement actually contains the information of both direct and indirect excitation, as photons that pass the sample in the direct excitation beam may still be absorbed after scattering in the sphere.

Figure S25. Two different measurement configurations required for Direct Excitation measurements:(A) reference sample (solvent only) in sample position (1); (B) test sample in position 1 (position 2 remains empty for both measurements.)

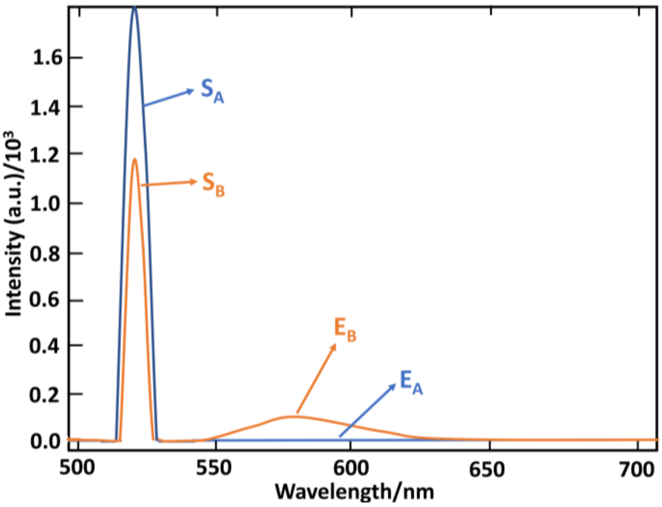

Figure S26. Spectral scans of the excitation scatter region or S-region (peaks on the left) and the emission region (E-region) of the sample and the solvent.

The indices "A" and "B" refer to the experimental setup illustrated in Figure S25. Note that the quantities S_A, S_B, E_A, and E_B refer to the integral of the scans.

The absolute fluorescence quantum yield, calculated with the "Direct Excitation" method is calculated as follows:

$$\eta_{DExc} = \frac{E_B - E_A}{S_A - S_B} \quad \text{..........(2)}$$

$E_A(\lambda)$ and $S_A(\lambda)$, as well as $E_B(\lambda)$ and $S_B(\lambda)$ may be measured in four individual scans. However, it is often convenient to measure these spectra in two scans only. For the calculation of the integrals, the selection of the integral regions, and the final calculation of η_{DExc} use the quantum yield wizard that is supplied with the FLS980 software.

If the sphere background, $E_A(\lambda)$, is sufficiently low the measurement of this region may be omitted to save measurement time. In this case the equation degrades to:

$$\eta_{DExc} = \frac{E_B}{S_A - S_B} \quad \text{..........(3)}$$