Supporting Information

Oxacalix[2]arene[2]triazine Based Ion-pair Transporters

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1. General information

Reagents for synthesis and analysis were purchased from J&K or Sigma-Aldrich. Egg yolk phosphatidylcholine (EYPC) and a Mini-Extruder used for vesicle preparation was from Avanti Polar Lipids. ^{1}H and ^{13}C NMR spectra were recorded on 300 MHz, 400 MHz 500M spectrometers. Chemical shifts are reported in ppm versus tetramethylsilane with either tetramethylsilane or the residual solvent resonance used as an internal standard. Abbreviations are used in the description of NMR data as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet), coupling constant (*J*, Hz). Melting points are uncorrected. All solvents were dried according to standard procedures prior to use. All other major chemicals were obtained from commercial sources and used without further purification.

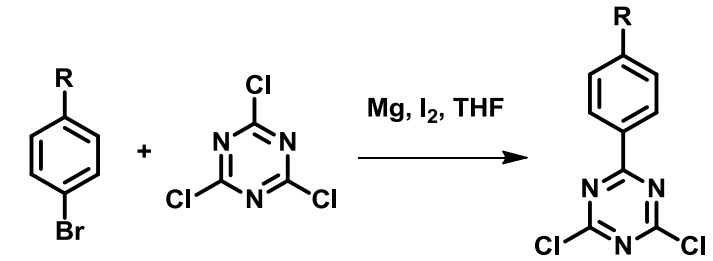
2. Experimental details

General Preparation of lipid lucigenin/CF-containing EYPC LUVs. Egg yolk L-α-phosphatidylcholine (EYPC, 25 mg), was dissolved in a MeOH/$CHCl_3$ (2 ml, 1:1) mixture, the solution was evaporated under reduced pressure on a rotary evaporator (40 °C) to give a thin film, and the resulting thin film was dried under high vacuum for overnight to remove the solvent completely. The lipid film was hydrated in 1.0 mL buffer (10 mM HEPES, 100mM $NaNO_3$ or NaCl, 1mM lucigenin or 50 mM CF, pH = 7.0, for CF pH = 7.4) for 30 min. The LUV suspension was submitted to freeze-thaw for 5 cycles (with liquid nitrogen and 37 °C water bath, respectively), and high-pressure extrusion at room temperature (21 extrusions through 100 nm polycarbonate membrane). The LUV suspension was separated from extravesicular lucigenin dye by size exclusion chromatography (Sephadex G-25, mobile phase: 10mM HEPES, 100mM $NaNO_3$ or NaCl, pH = 7.0).

For preparation of DPPC lucigenin-containing LUVs, DPPC (25 mg) and **3** (0.5 mg) was dissolved in a MeOH/$CHCl_3$ (2 ml, 1:1) mixture, others follow the same procedure.

3. Synthesis

Synthesis of monomers 11



13a R=MeO
13b R=CF_3

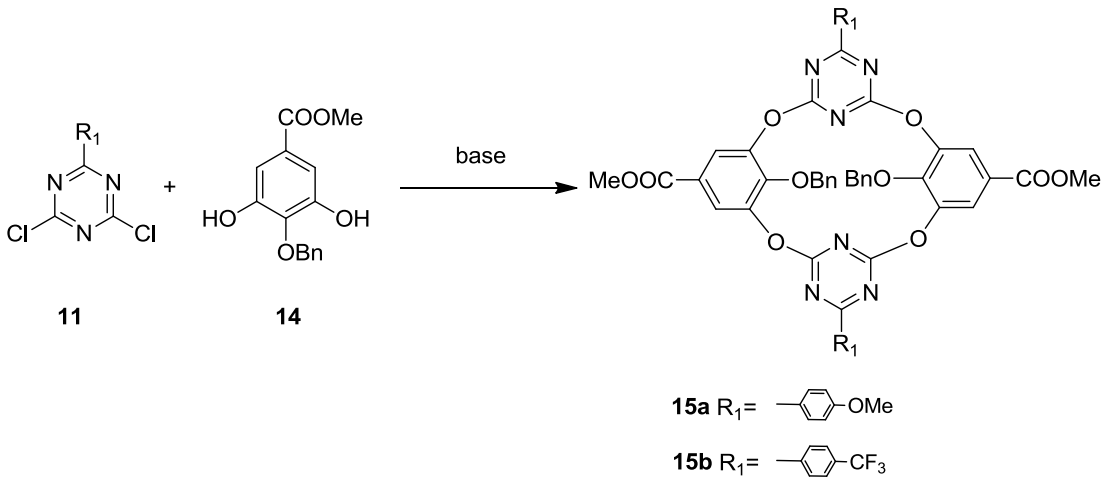
11a R=MeO
11b R=CF_3

In a 500 ml flask, magnesium turnings (2.4 g, 0.1 mol) and iodine (200 mg) were introduced into dried THF (20 ml). **13** (18.7 g, 0.1 mol for **13a**, 22.5 g, 0.1 mol for **13b**) was dissolved in dried THF (100 ml). The solution was added dropwise to the flask. After the complete dissolution of the Mg turnings, the mixture was reacted at 60 °C for 90 minutes under argon atmosphere, to produce the Grignard solution. The Grignard solution was added dropwise to cyanuric chloride (55.3 g, 0.3 mol in 150 ml THF) within 60 minutes. The reactant solution was stirred at 5 °C and under nitrogen atmosphere. After the reaction is finished, the solvent was removed by evaporation under reduced pressure. Diluted hydrochloric acid (500 mL, 1 M) was added to the resulting residue, and the solution was extracted with dichloromethane (3 × 100 mL). The organic solvent was removed under reduced pressure and the residue was washed with isopropanol and filtered. Re-crystallization was carried out to produce pure **11**.

11a (white solid, yield 65%): mp 131-132 ^{o}C ; ^{1}H NMR ($CDCl_3$/300 MHz) δ 8.45 (d, *J* = 9.0 Hz, 2H, ArH), 6.99 (d, *J* = 9.0 Hz, 10H, ArH), 3.91 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$/75 MHz) δ 174.3, 170.3, 165.2, 132.3, 114.5, 55.7 .

11b (white solid, yield 47%): mp 115-117 ^{o}C ^{1}H NMR ($CDCl_3$/300 MHz) δ 8.64 (d, *J* = 8.1 Hz, 2H, ArH), 7.80 (d, *J* = 8.4 Hz, 10H, ArH), ^{13}C NMR ($CDCl_3$/75 MHz) δ 173.5, 172.5, 136-135 (q, *J* = 3. Hz), 130.16, 129.8-118.1 (q, *J* = 3.8 Hz), 1261-125.8 (q, *J* = 270.5 Hz)

Synthesis of 15



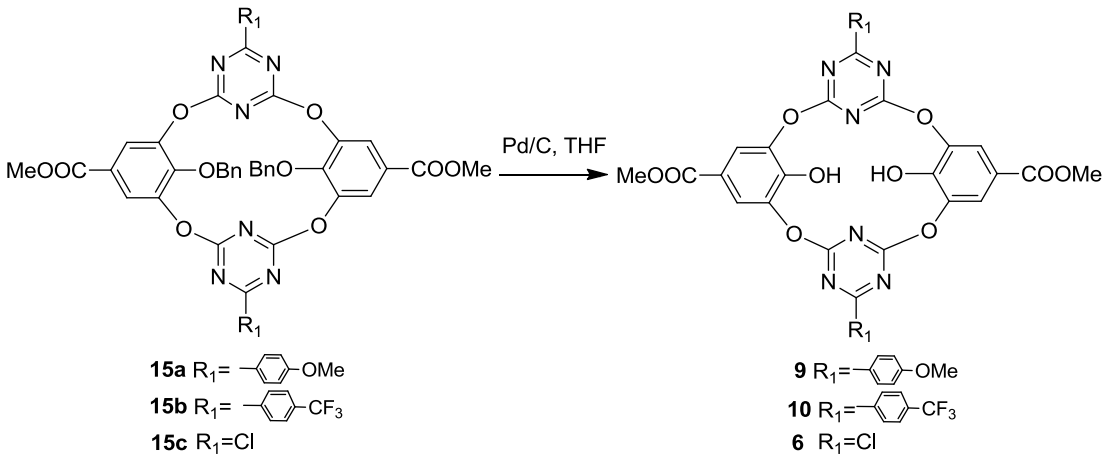
To a well stirred and refluxed solution of K_2CO_3 (310 mg, 1.2 mmol) or DIPEA (310 mg, 1.2 mmol) in reflux acetonitrile (33 mL) was added drop-wise a solution of **11** (*ca.* 254 mg for **11a**, *ca.* 294mg for **11b** 1 mmol) and **14** (274 mg, 1 mmol) in acetonitile (33 mL). After the addition of **11** and **14** (*ca.* 0.5 h), the mixture at reflux was kept stirring for another 3h. After the reaction was finished, the solvent was removed using a rotary evaporator. The residue was dissolved in ethyl acetate (50 mL) and was washed with water (3 × 30 mL). The organic phase was dried with anhydrous Na_2SO_4. After filtration, the filtrate was concentrated. The residue was chromatographed on a silica gel column using a mixture of petroleum ether and ethyl acetate (5 : 1) as an eluent to give pure **15**.

15a (white solid, yield 44%): mp >300 ^{o}C; ^{1}H NMR ($CDCl_3$ / 300 MHz) δ 8.49 (d, *J* = 8.7 Hz, 4H, ArH), 7.63 (s, 4H, ArH), 7.05-6.9 (m, 14H, ArH), 4.91 (s, 4H, CH_2), 3.93 (s, 6H, CH_3), 3.83 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$ / 75 MHz) δ 176.2, 172.2, 165.0, 164.1, 147.1, 145.0, 135.7, 131.5, 128.1, 127.9, 127.6, 126.8, 125.5, 122.1, 114.0, 75.5, 55.5, 52.3; IR (KBr, cm^{-1}) v 1727, 1564, 1525, 1326,1257; MS(ESI) m/z(%) $[M+Na]^+$ 937.2434 (100); Anal. Calcd. for $C_{50}H_{38}N_6O_{12}\cdot 0.5H_2O$: C, 65.00; H, 4.25; N, 9.10 Found: C, 65.10; H, 4.14; N, 9.13.

15b (white solid, yield 39%): mp 282-284 °C; ^{1}H NMR ($CDCl_3$ / 400 MHz) 8.62 (d, *J* = 8.4 Hz, 4H, ArH), 7.80 (d, *J* = 8.4 Hz, 4H, ArH), 7.67 (s, 4H, ArH), 7.05-6.95 (m,

10H, ArH), 4.93 (s, 4H, CH_2), 3.87 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$ / 100 MHz) 175.6, 172.5, 164.9, 146.9, 145.0, 137.5, 137.5, 135.6-134.6(q, *J* = 33.5 Hz), 129.7, 128.2, 128.1, 127.5, 125.9, 125.7-125.5(q, *J* = 3.7 Hz), 125.1-117.5(q, *J* = 274.4 Hz), 122.2; IR (KBr, cm^{-1}) v 1727, 1570, 1535, 1436, 1379, 1320; MS (ESI) m/z(%) $[M+H]^+$ 991.2156 (100); Anal. Calcd. for $C_{50}H_{32}F_6N_6O_{15}$: C, 60.61; H, 3.26; N, 8.48; Found: C, 60.43; H 3.35;N, 8.48.

General synthesis of 6, 9 and 10



To **15** (1 mmol) in 50 ml THF was added Pd/C (10percent, 50 mg). The resulting black suspension was bubbled with N_2 and then placed under 1 atm of H_2. The reaction mixture was stirred at room temperature overnight and filtered through a microfiber filter to remove the catalyst. The filtrate was concentrated under reduced pressure to produce pure **6**, **9** and **10.**

6 (white solid, yield 87%): mp >300 °C; IR (KBr, cm^{-1}) ^1H NMR (Acetone-d_6 / 300 MHz) δ 7.56 (s, 4H, ArH), 3.78 (s, 6H, CH_3); ^{13}C NMR (Acetone-d_6 / 75 MHz) δ 174.2, 173.2, 165.3, 147.0, 141.0, 122.8, 121.8, 52.5; IR (KBr, cm^{-1}) v 1698, 1566, 1518, 1436, 1339, 1258; MS (ESI) m/z(%) $[M+Na]^+$ (100) Anal. Calcd. for $C_{22}H_{12}Cl_2N_6O_{10}\cdot H_2O$: C, 43.87; H, 2.32; N, 13.79; Found: C 44.22; H 2.69;N, 13.37.

9 mp (white solid, yield 94%): 283-284 °C; ^1H NMR (Acetone-d_6 / 300 MHz) δ 8.54 (d, *J* = 8.7 Hz, 4H, ArH), 7.14 (d, *J* = 8.7 Hz, 4H, ArH), 3.95 (s, 6H, CH_3), 3.79 (s, 6H, CH_3); ^{13}C NMR (Acetone-d_6 / 75 MHz) δ 175.8, 172.5, 164.8, 164.2, 146.9, 140.8,

131.1, 127.1, 121.7, 120.7, 114.1, 55.1, 51.4; IR (KBr, cm^{-1}) v 1699, 1566, 1518, 1339, 1258, 1148; MS(ESI) m/z(%) $[M+H]^+$ 733.1522 (100); Anal. Calcd. for $C_{36}H_{26}N_6O_{12}\cdot 2H_2O$: C, 56.11; H, 3.92; N, 10.91; Found: C, 56.42; H 4.07;N, 10.97.

10 (white solid, yield 90%): mp 245-247 °C ; ^{1}H NMR (Acetone-d_6 / 500 MHz) δ 8.76 (d, *J* = 8.1 Hz, 4H, ArH), 7.97 (d, *J* = 8.0 Hz, 4H, ArH), 7.60 (s, 4H, ArH), 3.79 (s, 6H, CH_3); ^{13}C NMR (Acetone-d_6 / 125 MHz) δ 175.8, 173.7, 165.5, 147.7, 141.6, 139.3, 135.0-134.2 (q, 30.9 Hz), 130.5, 128.6-121.5 (q, 274 Hz), 126.7-126.6 (q, 3.7 Hz), 122.6, 52.3; IR (KBr, cm^{-1}) v 1727, 1570, 1535, 1381, 1320; MS(ESI) m/z(%) $[M+H]^+$ 811.1210(100); Anal. Calcd. for $C_{36}H_{20}F_6N_6O_{10}\cdot CH_3COCH_3$: C, 53.92; H, 3.02; N, 9.64; Found: C, 53.95; H 3.07;N, 9.44.

4. Evaluation of the ion transport activity

50 μL lucigenin-loaded vesicles (stock solution) was suspended in 1925μl of the buffer (10 mM HEPES, 100mM NaCl) and placed into a quartz cuvette at 25 °C. The intravesicular lucigenin fluorescence intensity (I_t, λ_{em} = 505 nm, λ_{ex} = 369 nm) was measured over time. DMF or transporters **1-10** (25μl in DMF) at t=50 s (I_0), and triton X-100 (25μl, 10% in water) at 300 s (I_∞) was added, respectively. The fluorescence intensity I_t were normalized to fractional intensities I_f using equation (S1)

$$I_f = (I_t - I_\infty)/(I_0 - I_\infty) \qquad \text{(S1)}$$

Where I_0 is the fluorescence intensity after addition of transporter, anf I_∞ is fluorescence intensity after addition of triton X-100.

The effective concentration EC_{50} and the Hill coefficient n was obtained by Hill equation (S2)

$$Y = Y_\infty + (Y_0 - Y_\infty) / (1 + c / EC_{50})^n \qquad \text{(S2)}$$

Where Y is the I_f value at 120 s, Y_0 is Y in absence of transporter, Y_∞ is Y with excess transporter, and c is the transporter concentration in the cuvette.

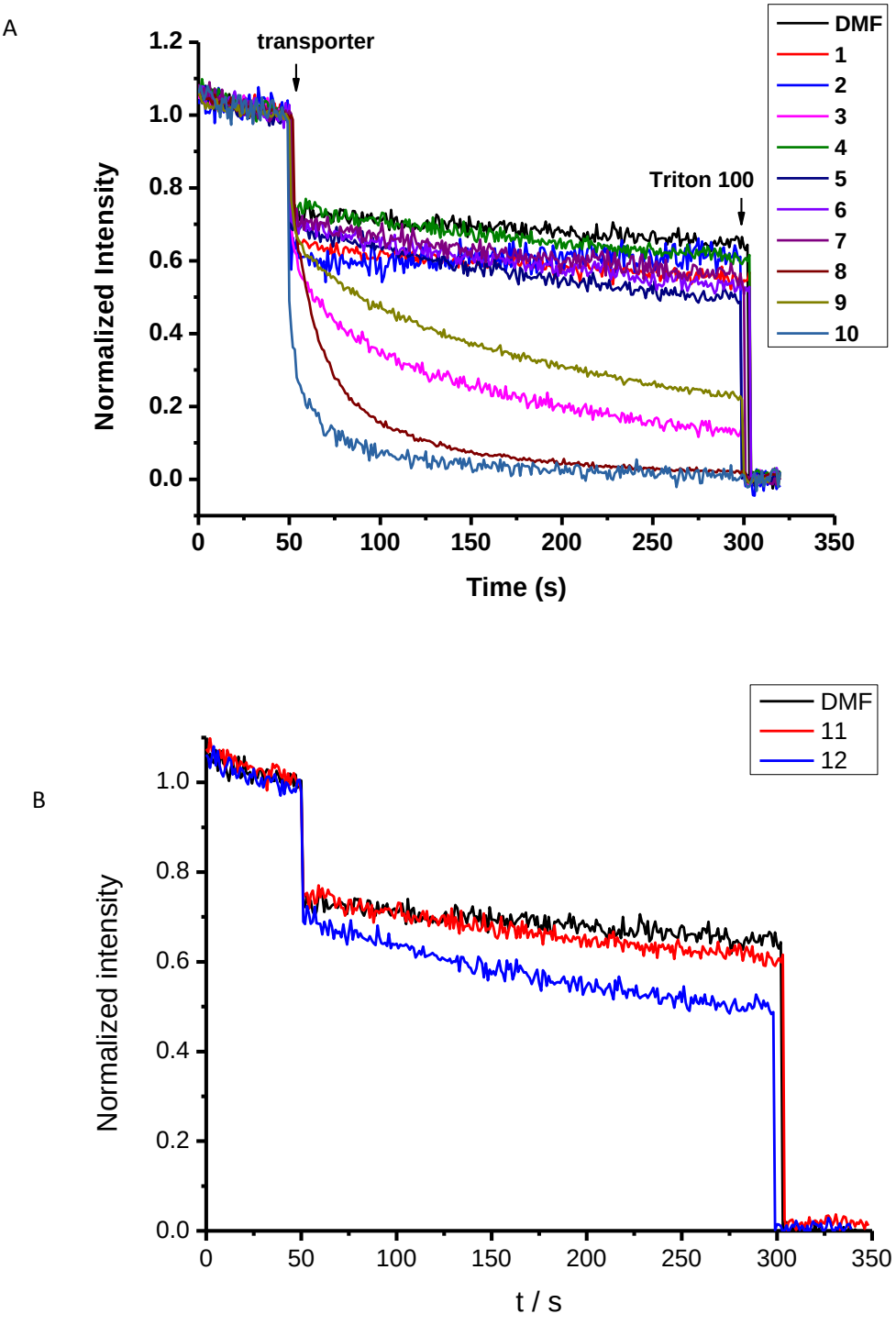
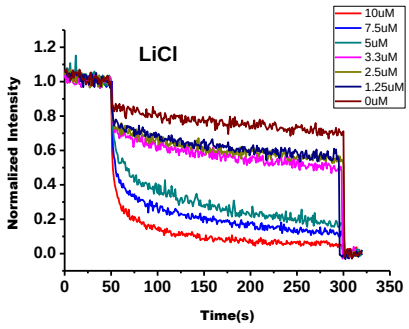
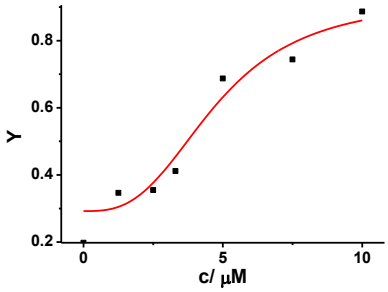
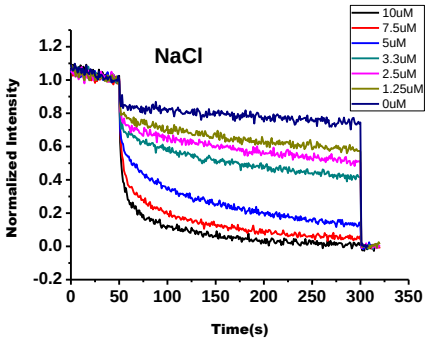


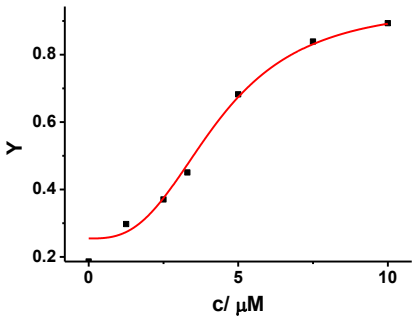
Figure S1. (A) and (B) Evaluation of the ion transport activity of transporters **1-12** by lucigenin⊂EYPC fluorescence assays upon addition of NaCl (with concentration for each compound is 5 μM except for **8** is 7.5 μM).

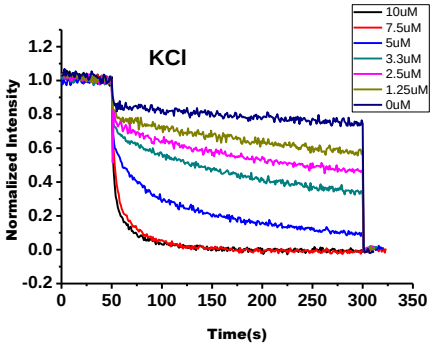
Evaluation of EC_{50} of 3 and the effect of ions

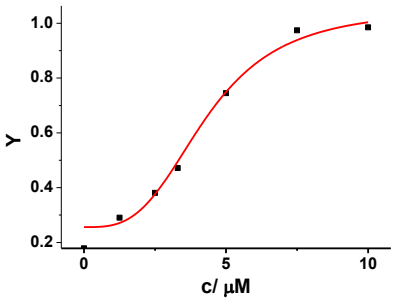


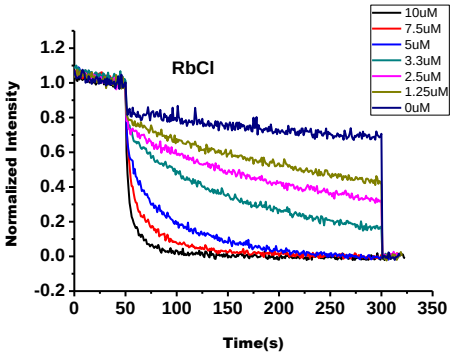


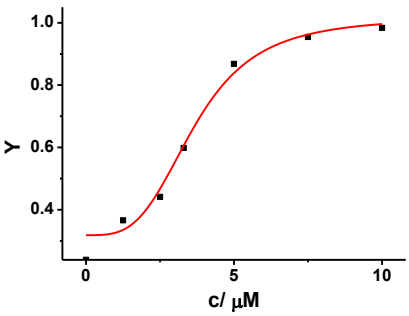












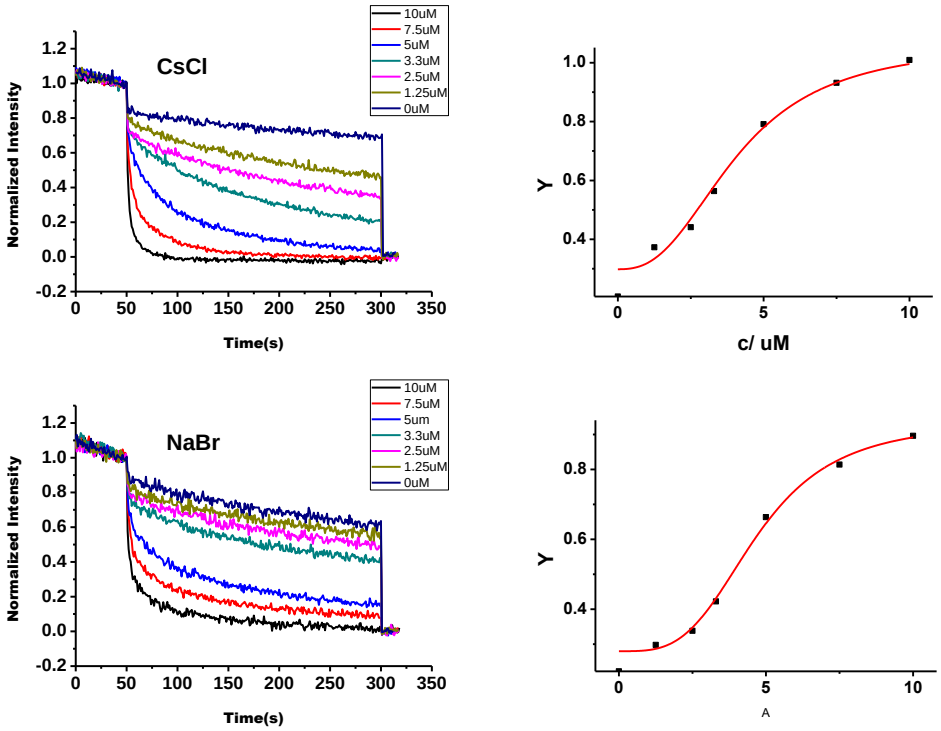
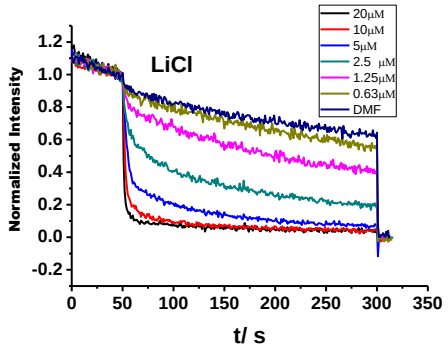
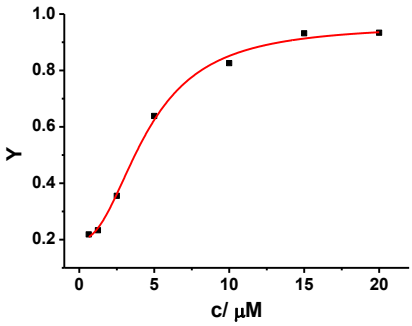
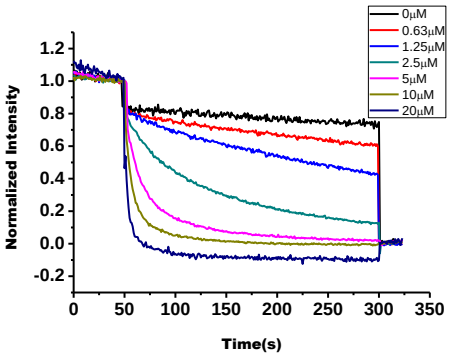


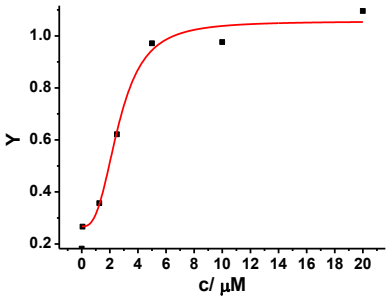
Figure S2. Evaluation of the ion transport activity of **3** in varied concentration by EYPC-LUVs ⊃ Lucigenin fluorescence assay, and Hill plot of the normalized intensities at 120 s.

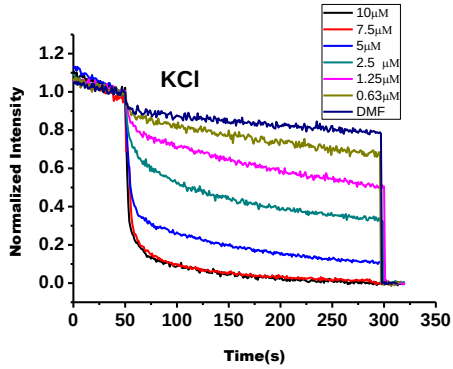
Evaluation of EC_{50} of 8 and the effect of ions

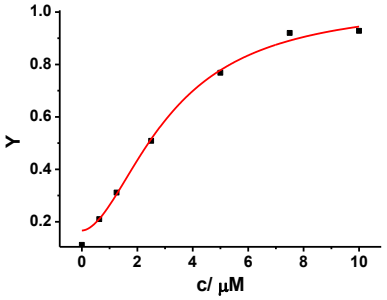


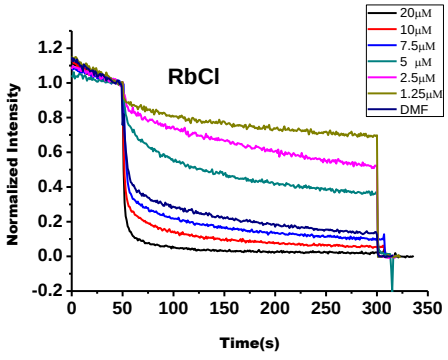


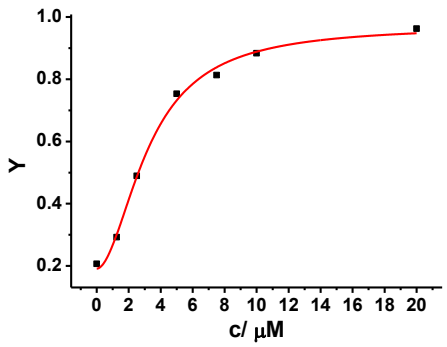












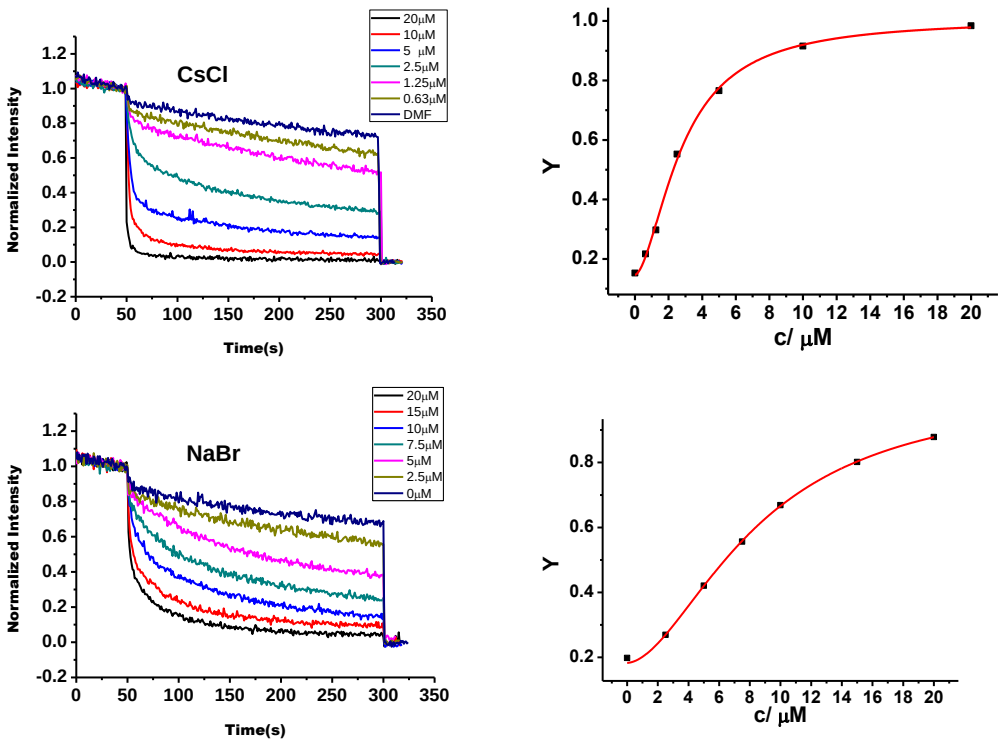
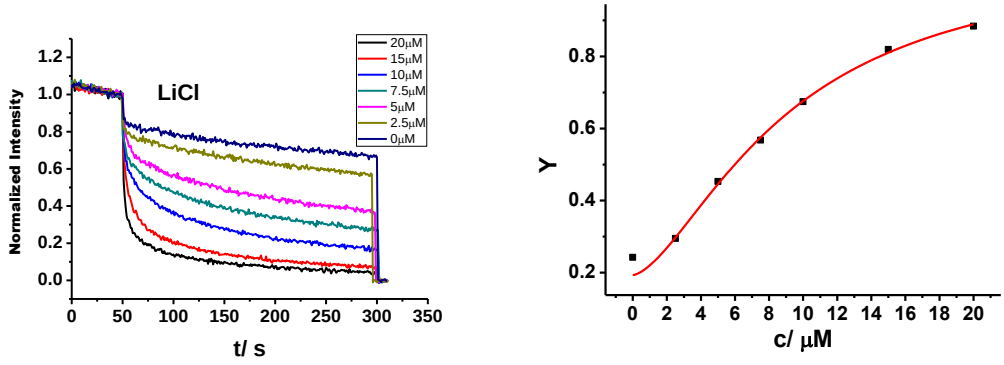
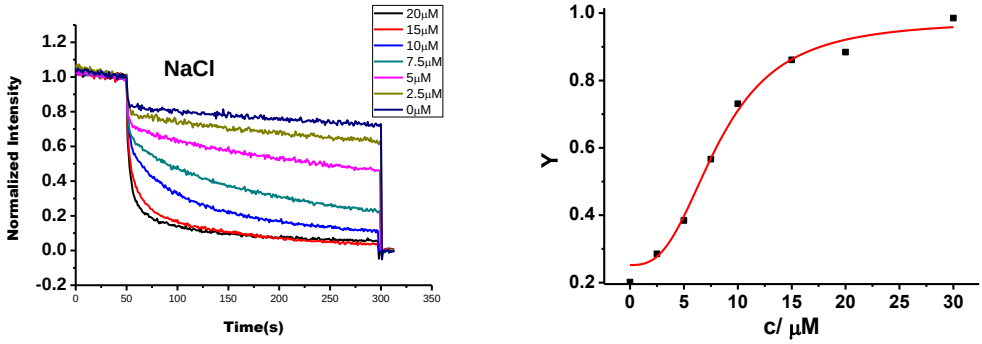
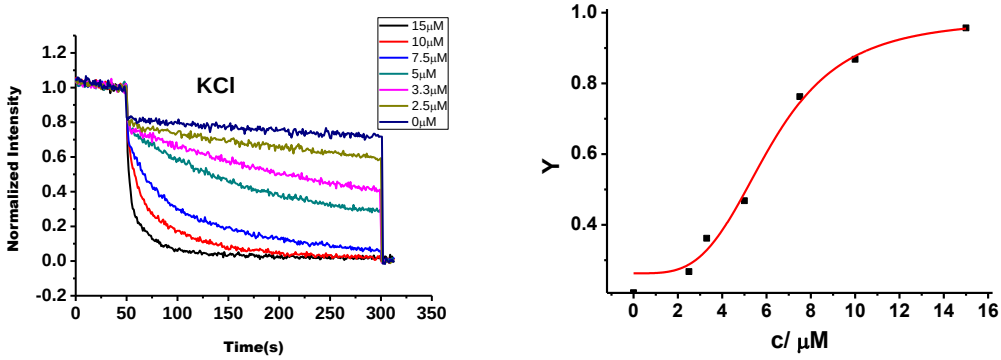


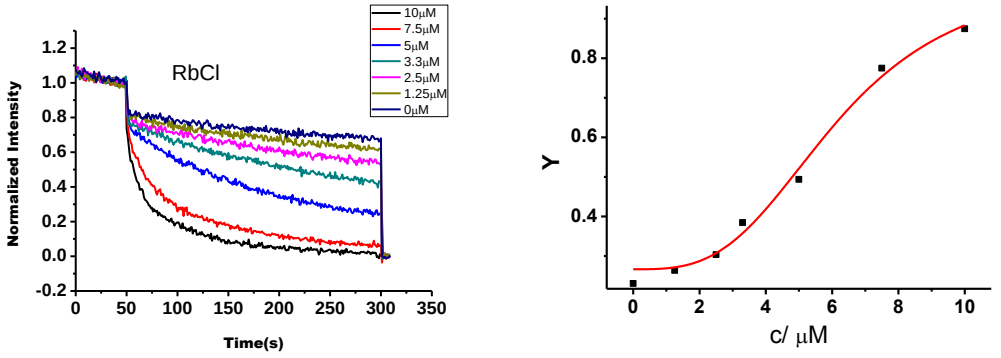
Figure S3. Evaluation of the ion transport activity of **8** in varied concentrations by EYPC-LUVs ⊃ Lucigenin fluorescence assay, and Hill plot of the normalized intensities at 120 s.

Evaluation of EC_{50} of 9 and the effect of ions









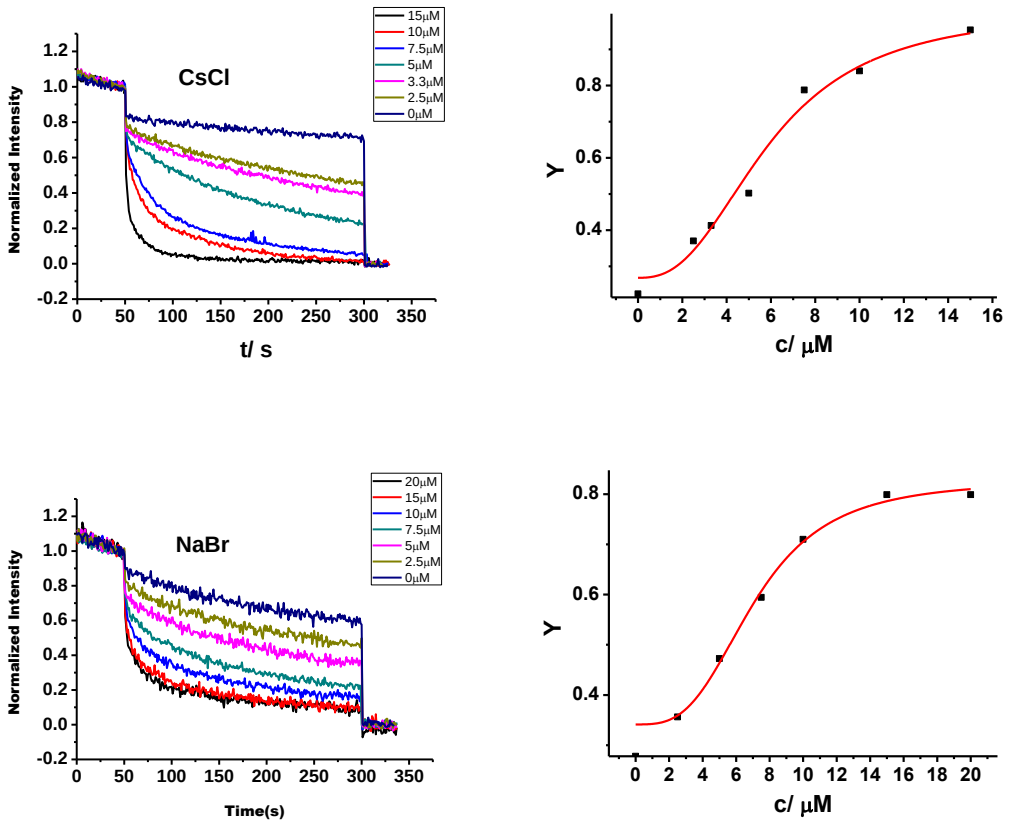
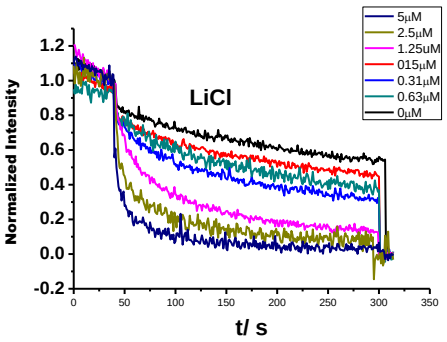
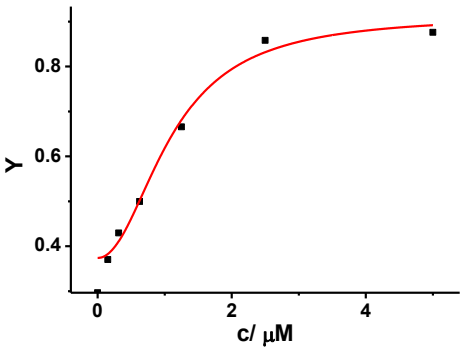
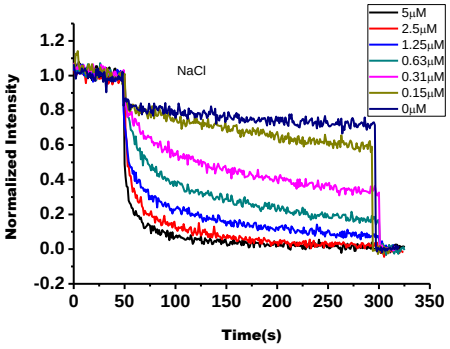


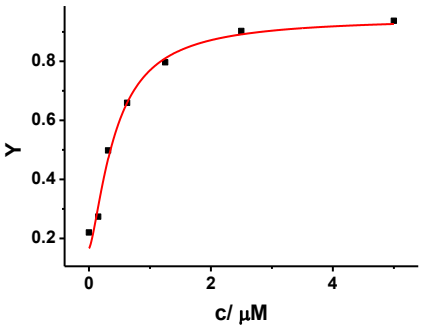
Figure S4 Evaluation of the ion transport activity of **9** in varied concentration by EYPC-LUVs ⊃ Lucigenin fluorescence assay, and Hill plot of the normalized intensities at 120 s.

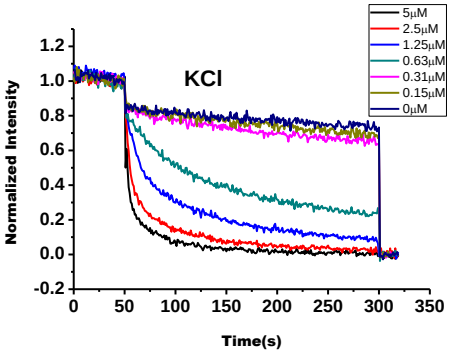
Evaluation of EC_{50} of 10 and the effect of ions

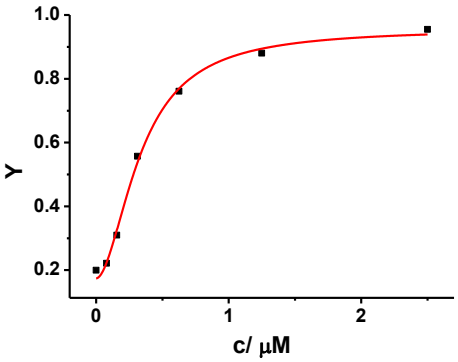


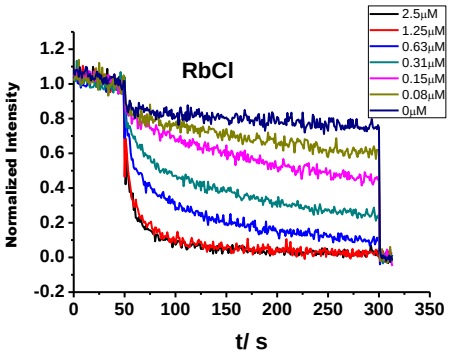


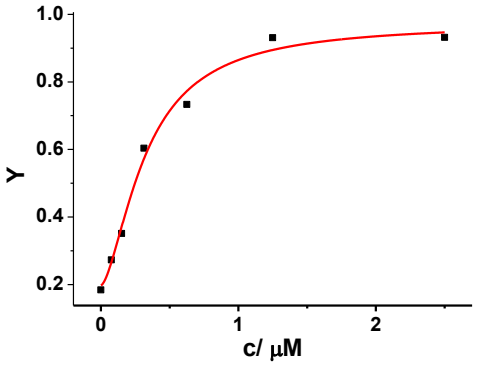












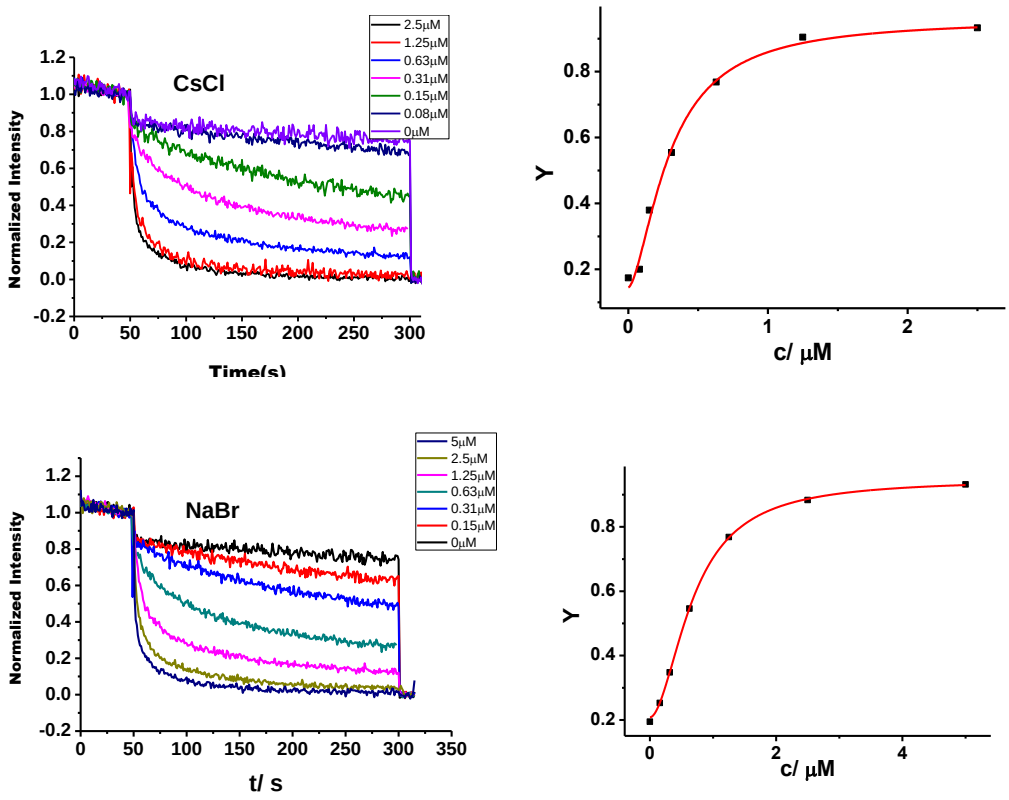


Figure S5. Evaluation of the ion transport activity of **10** in varied concentration by EYPC-LUVs ⊃ Lucigenin fluorescence assay, and Hill plot of the normalized intensities at 120 s.

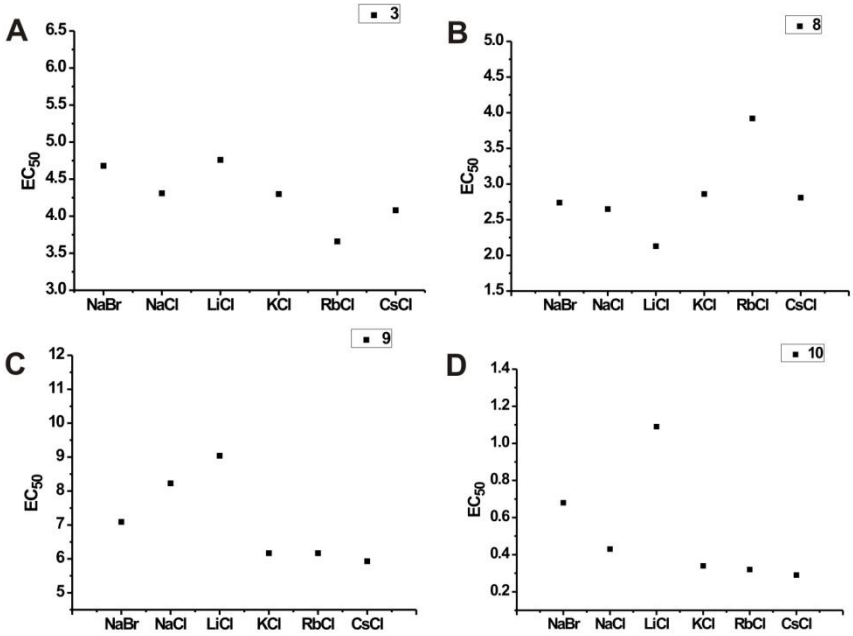


Figure S6. Effect of anions and cations on the effective concentration (EC_{50}) of (A) **3**, (B) **8**, (C) **9**, and (D) **10**.

Table S1 EC_{50} and hill coefficient of **3, 8-10** with various salts

	3		**8**		**9**		**10**	
	EC50	n	*EC50*	n	*EC50*	n	*EC50*	n
NaBr	4.68±0.46	3.4±1.1	2.74±0.45	2.3±0.8	7.09±0.89	3.1±1.4	0.68±0.02	1.9±0.1
NaCl	4.31±0.6	2.9±1.1	2.65±0.62	1.9±0.7	8.23±0.68	2.8±0..7	0.43±0.19	1.5±0.7
LiCl	4.76±1.4	2.9±2.2	2.13±0.20	2.2±0.4	9.04±2.57	1.6±0.9	1.09±0.25	2.1±1.0
KCl	4.30±0.5	3.2±1.2	2.86±0.41	2.1±0.6	6.17±0.53	3.7±1.2	0.34±0.05	2.3±0.8
RbCl	3.66±0.42	3.4±1.3	3.92±0.42	2.3±0.5	6.17±0.95	3.2±1.0	0.32±0.06	1.6±0.6
CsCl	4.08±0.82	2.6±1.4	2.81±0.20	1.7±0.2	5.93±1.12	2.5±1.6	0.29±0.05	1.6±0.5

5. CF release, Cl^-/NO_3^- antiport, and DPPC test

CF release

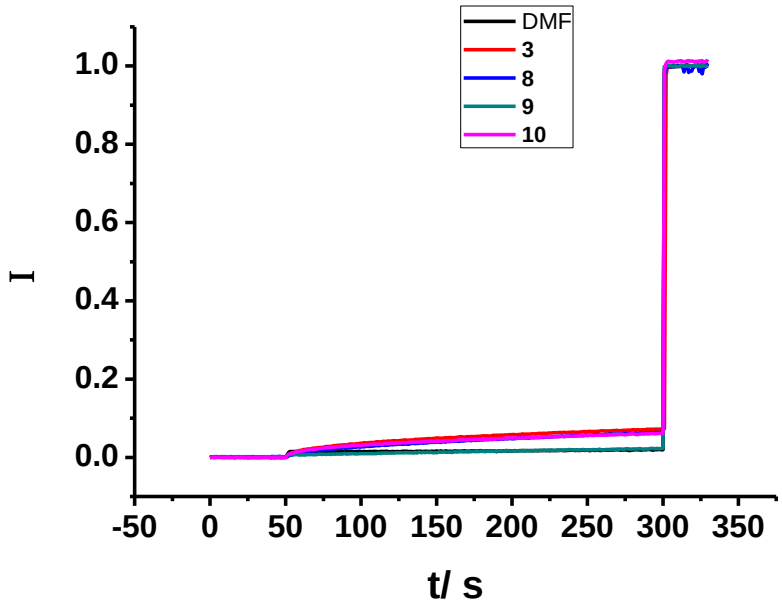


Figure S7. CF leakage experiments with the addition of compounds **3**, **8-10**.

Cl^-/NO_3^- antiport test

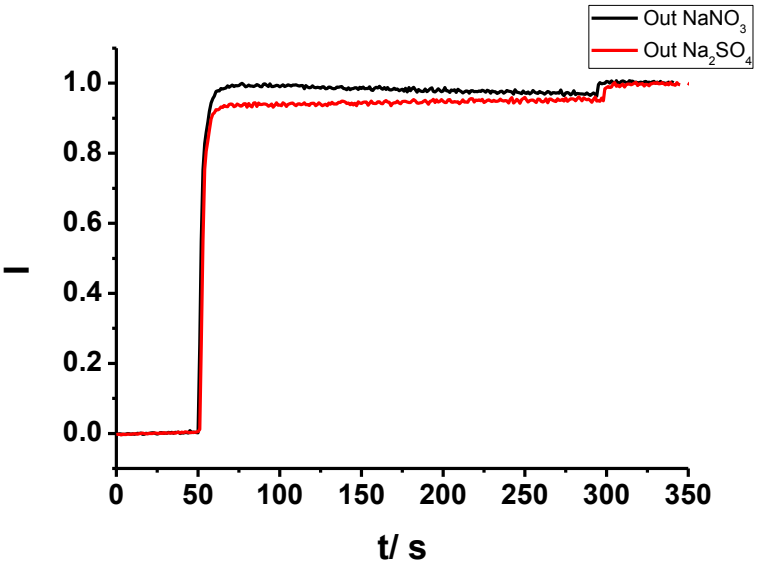


Figure S8. Normalized fluorescence intensity of lucigenin ⊂ EYPC with the addition of the transporter **3** (10 μM): the extravesicular salt is $NaNO_3$ (black line), and Na_2SO_4 (red line), respectively.

DPPC experiment

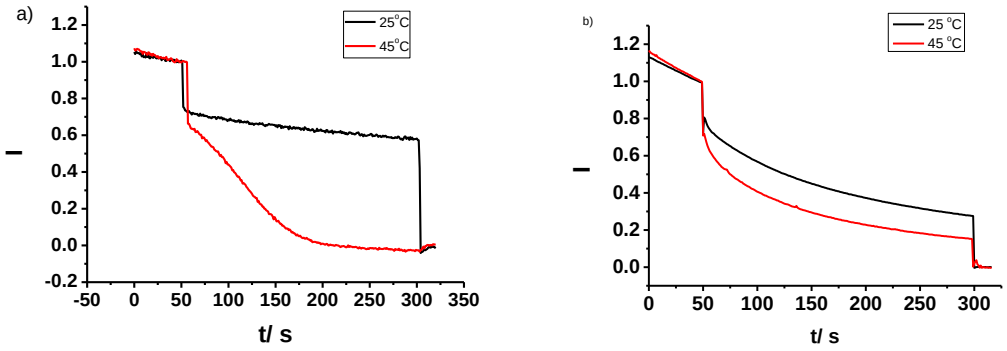


Figure S9. a)NaCl transport by **3** in DPPC vesicles at 25 ℃ and 45 ℃, respectively. The ratio of receptor : lipid is 1:50. b) NaCl transport by **3** in EYPC vesicles at 25 ℃ and 45 ℃, respectively. The ratio of receptor to lipid is 1:50.

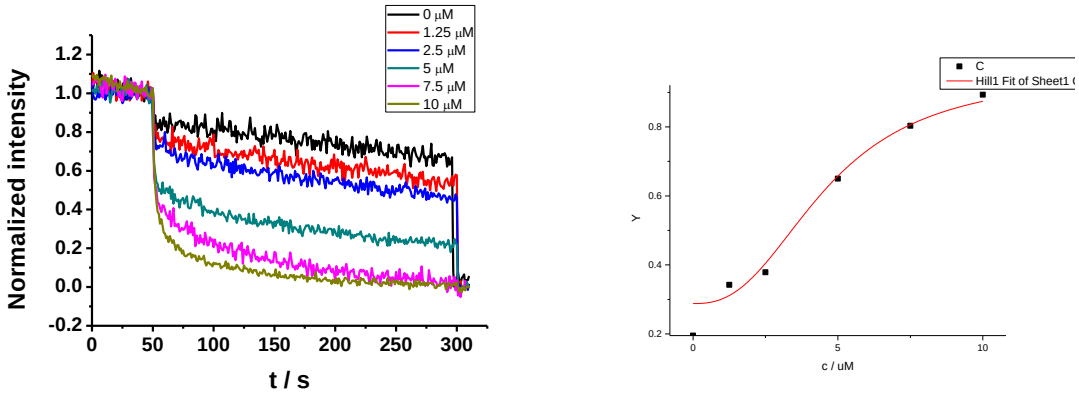


Figure S10. NaCl transport by **3** at pH = 7.6. EC_{50} = 4.59, n = 2.55.

6. Crystal structure and DFT optimization

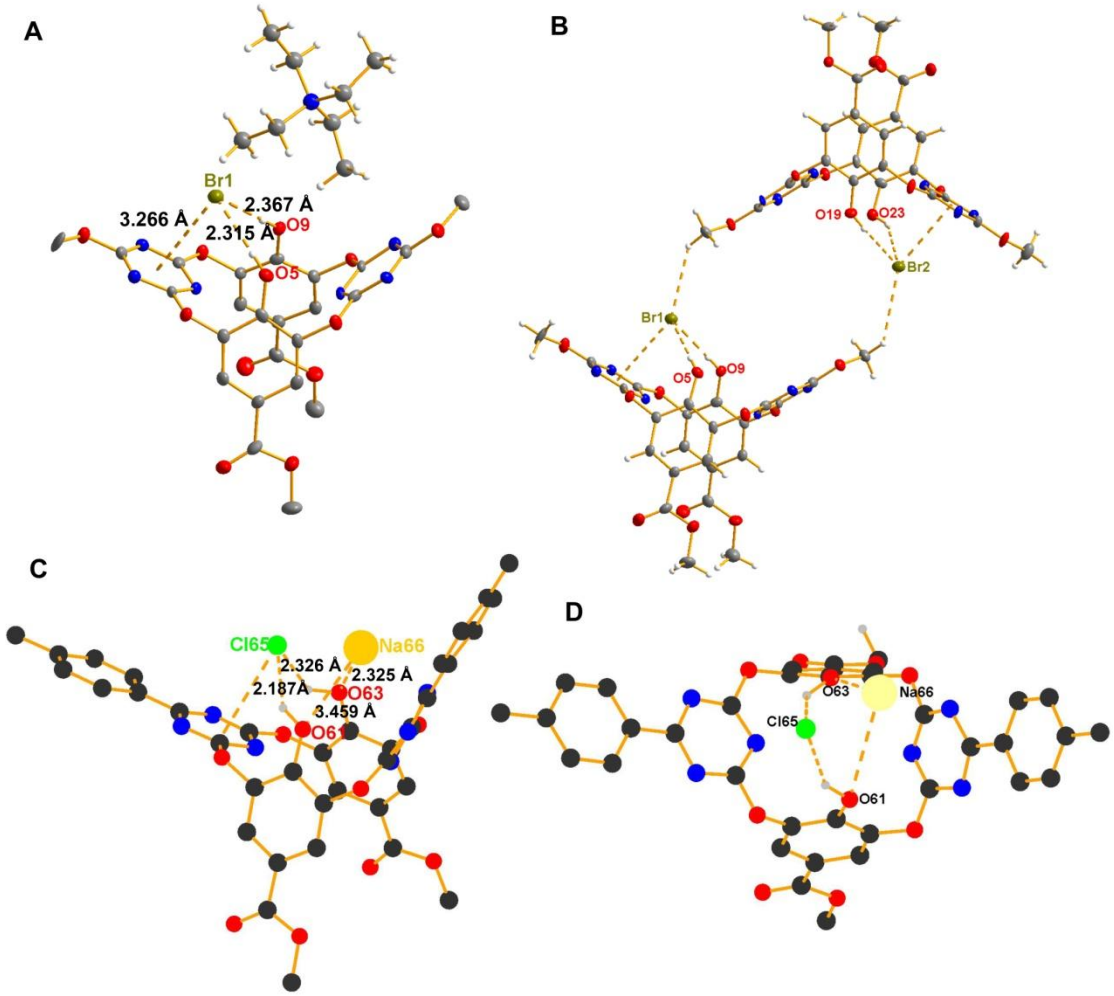


Figure S11. (A) Crystal structure of the complex of **7** and tetraethylammonium bromide (Et_4N^+(**8**·Br^-)), (B) dimer of the complex. DFT optimized structure of the complex of **3** and NaCl (C) side view and (D) top view.

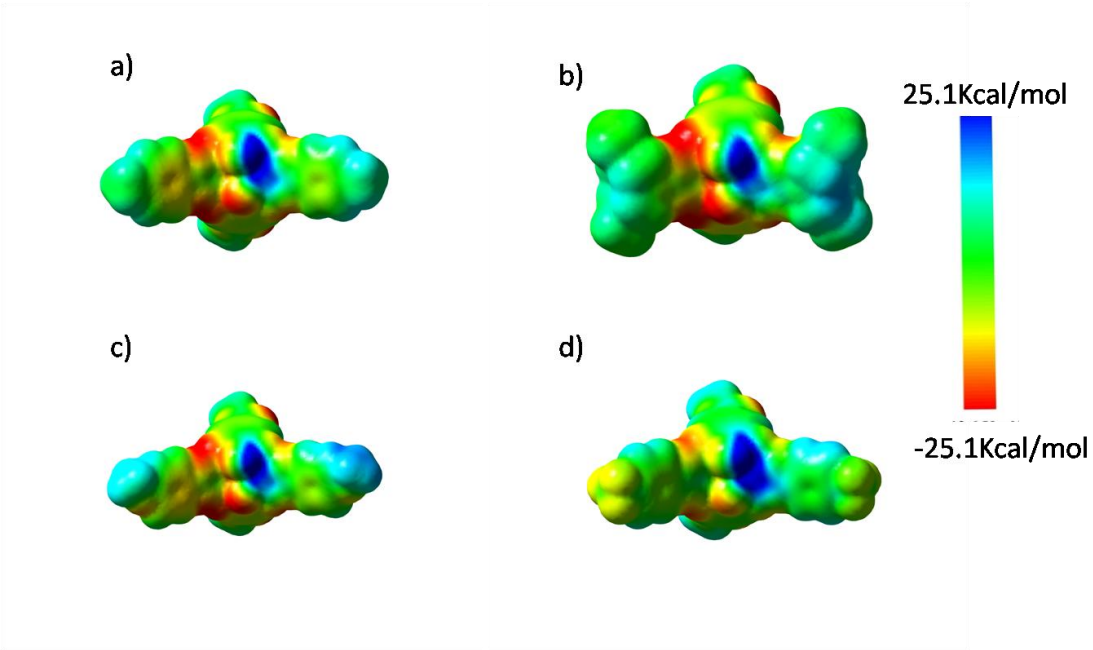


Figure S12. Calculated surface potential of **3** (a), **8** (b), **9** (c), **10** (d), with DFT modeling at B3LYP/6-31G* level.

7. Spectroscopic titration

Fitting result of **3** with tetrabutylammonium chloride by Hyperquad2003 program

sigma = 11.4387

,			Value	relative std devn	log beta	standard deviation
Beta	1 , 1	refined	2.3734E+04	0.0619	4.3754	0.0269

Fitting re,sult of **8** with tetrabutylammonium chloride by Hyperquad2003 program

sigma = , 0.9346

,			Value	relative std devn	log beta	standard deviation
Beta	, 1 1	refined	3.5532E+03	0.0086	3.5506	0.0038

Fitting, result of **9** with tetrabutylammonium chloride by Hyperquad2003 program

Sigm,a = 7.5713

,			Value	relative std devn	log beta	standard deviation
Beta	1 1	refined	1.2269E+04	0.0134	4.0888	0.0058

Fitting result of **10** with tetrabutylammonium chloride by Hyperquad2003 program

sigma = 9.6298

			Value	relative std devn	log beta	standard deviation
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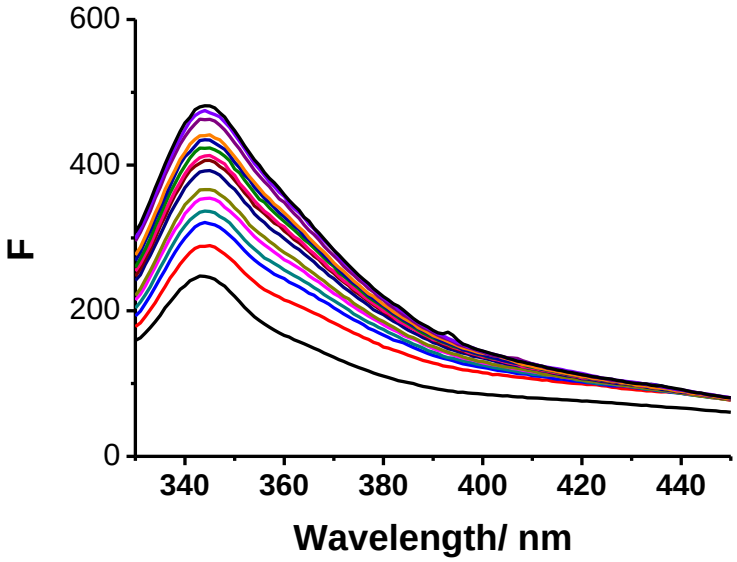


Figure S13. Fluorescence titration of **3** (1.0×10^{-4} M in 2 mL acetonitrile) upon the addition of tetrabutylammonium chloride (0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0, 11.0, 12.0, 13.0, 14.0, 15.0, 16.0 $\times 10^{-3}$ M), respectively. The excitation wavelength was 310 nm and the excitation and emission slits were set at 5nm.

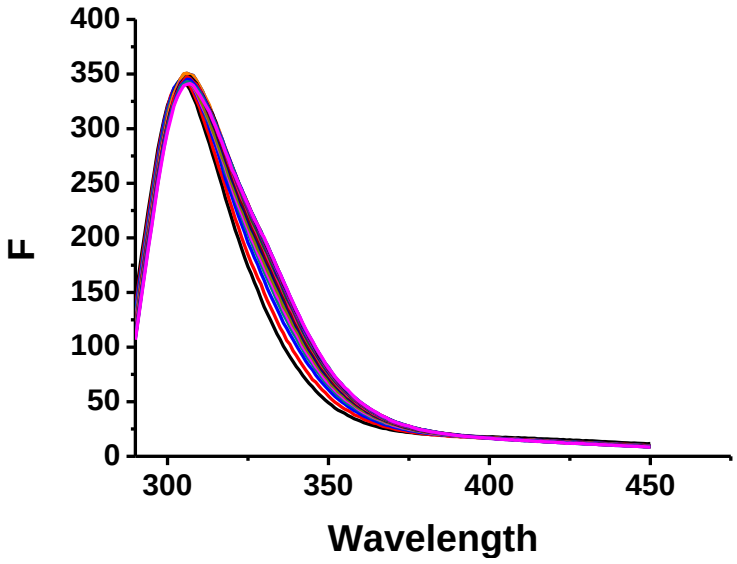


Figure S14. Fluorescence titration of **8** (1.0×10^{-4} M in 2 mL acetonitrile) upon the addition of tetrabutylammonium chloride (0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0, 11.0, 12.0, 13.0, 14.0, 15.0, 16.0, 17.0, 18.0 $\times 10^{-3}$ M), respectively. The excitation wavelength was 278 nm and the excitation and emission slits were set at 5nm.

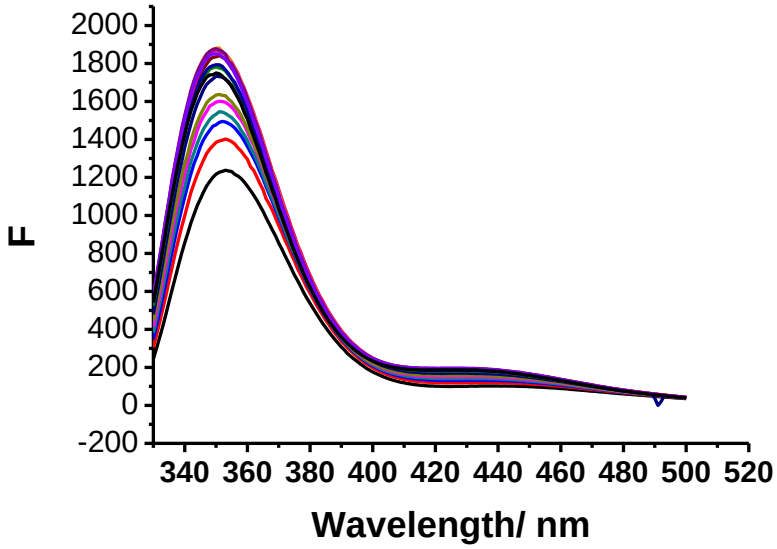


Figure S15. Fluorescence titration of **9** (1×10^{-4} M in 2 mL acetonitrile) upon the addition of tetrabutylammonium chloride (0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, 5.5, 6.0, 6.5, 7.0 $\times 10^{-3}$ M), respectively. The excitation wavelength was 320 nm and the excitation and emission slits were set at 5nm.

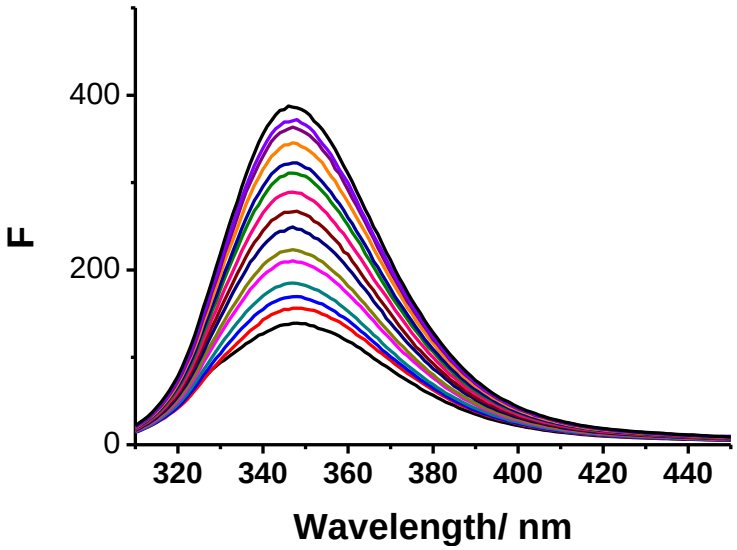


Figure S16 Fluorescence titration of **10** (1×10^{-4} M in 2 mL acetonitrile) upon the addition of tetrabutylammonium chloride (0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0, 11.0, 12.0, 13.0, 14.0, 15.0, 16.0 $\times 10^{-3}$ M), respectively. The excitation wavelength was 296 nm and the excitation and emission slits were set at 5nm.

8. Chemical shifts of the hydroxyl groups of 3, 8-10

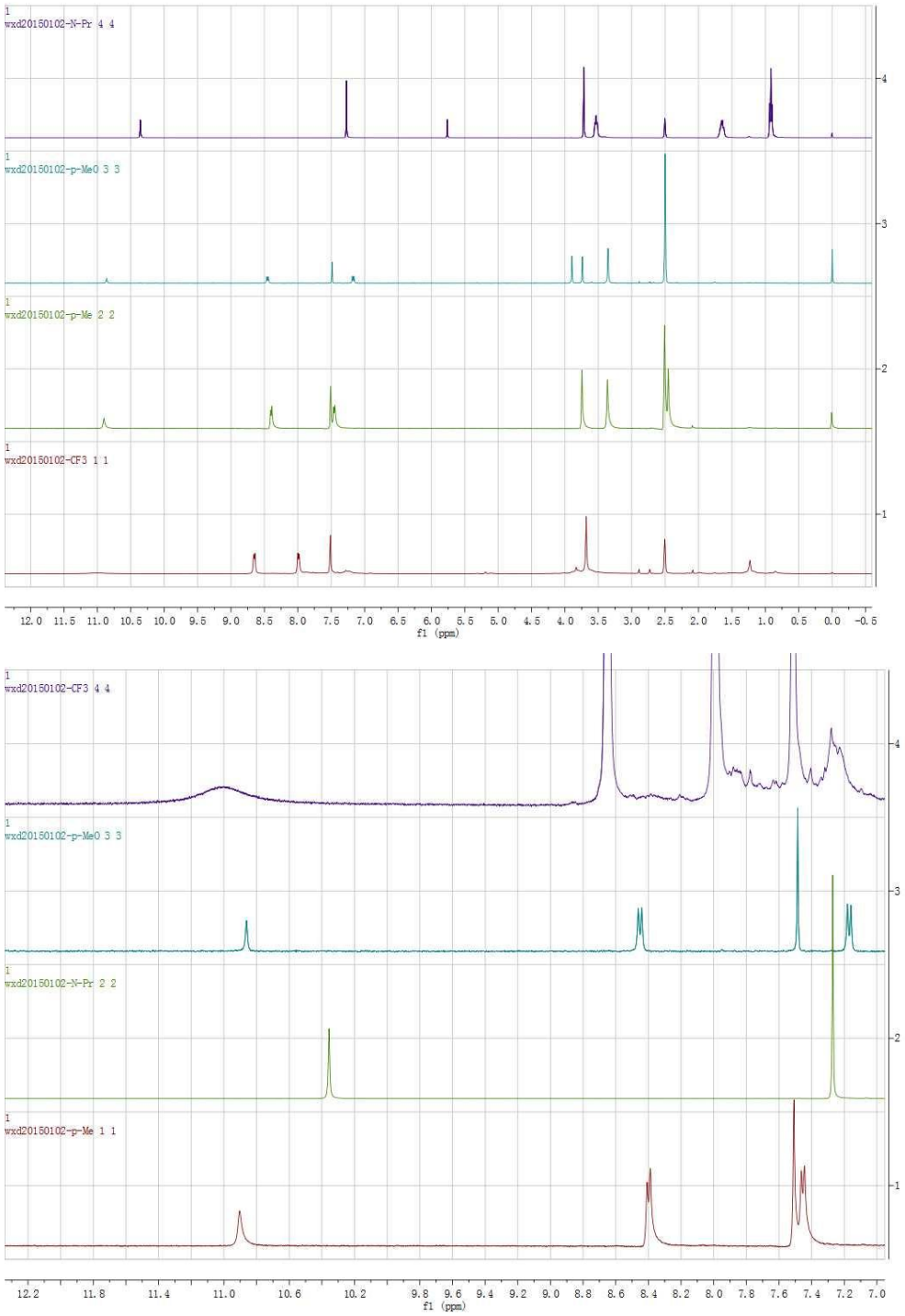
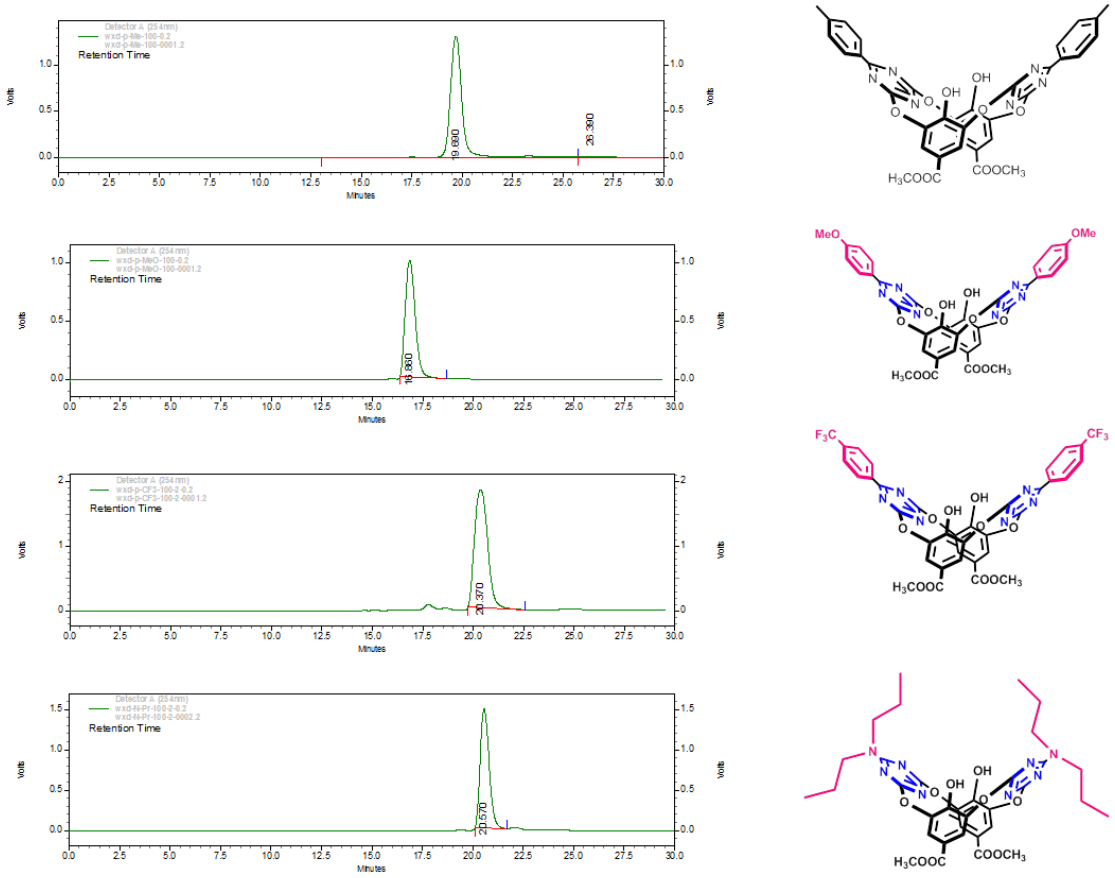


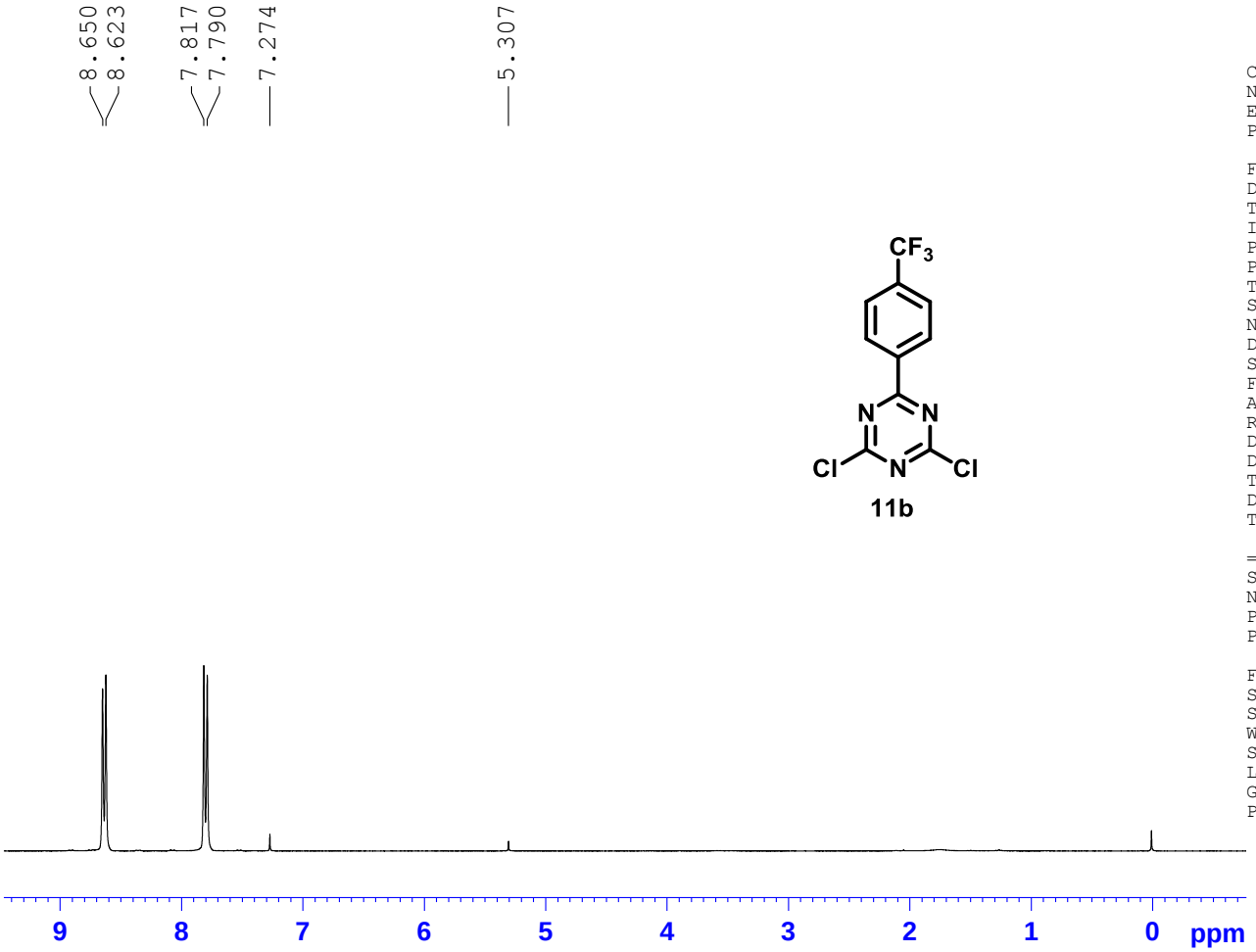
Figure S17. Chemical shifts of the hydroxyl groups of **3**, **8**, **9**, **10** from bottom to top in DMSO-d_6.

9. HPLC determination



10. NMR spectra of the compounds

p-CF3

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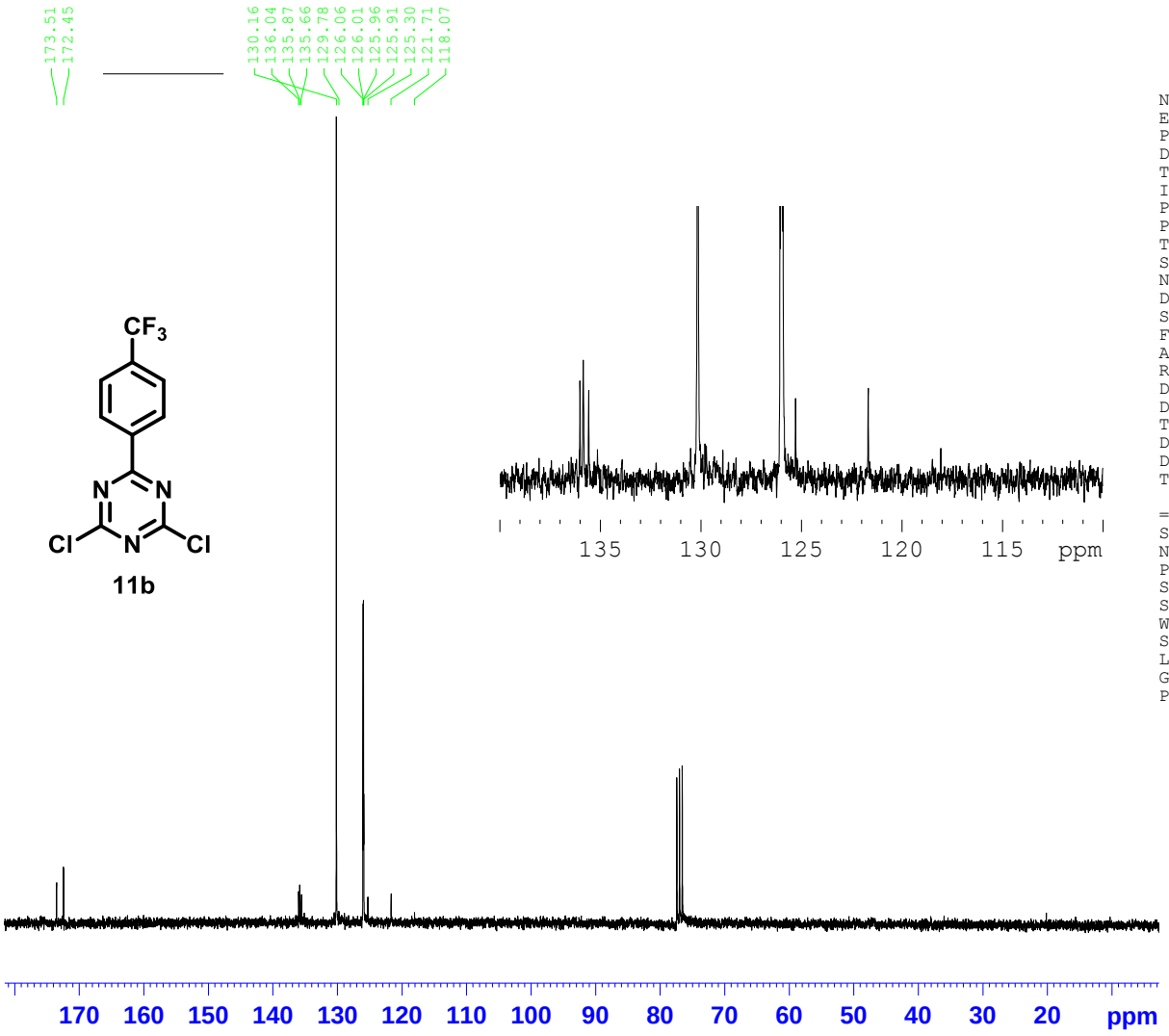
p-CF3



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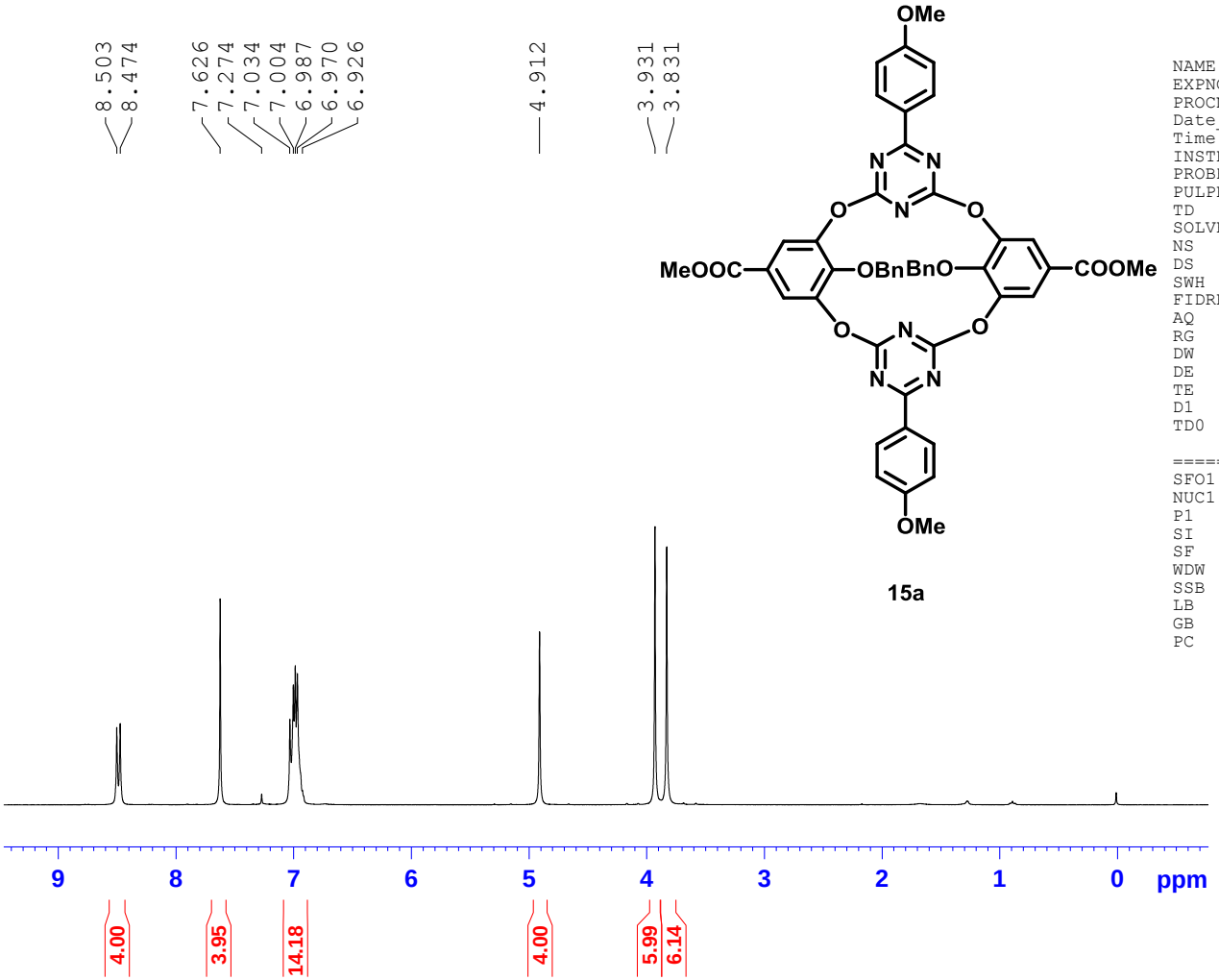


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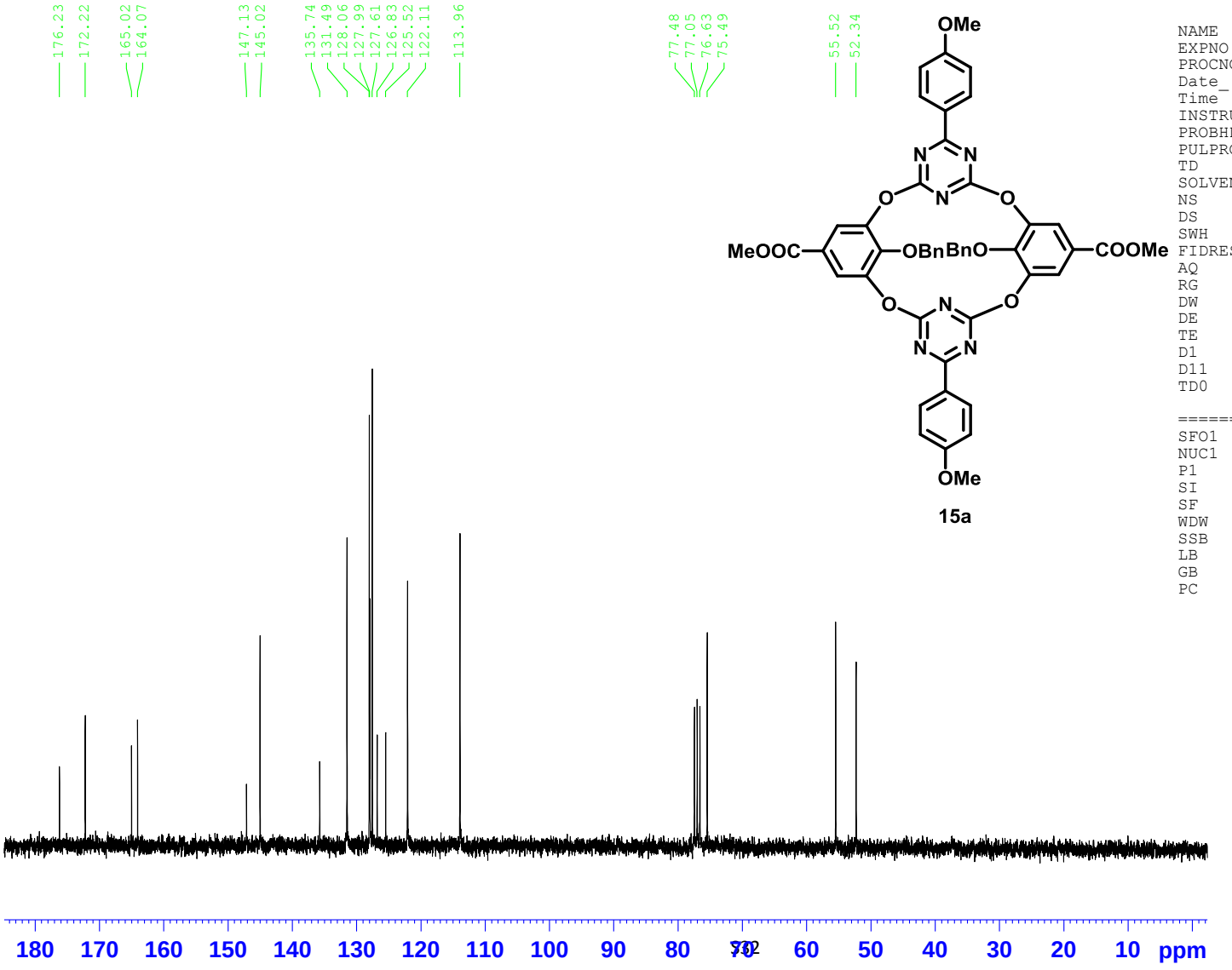

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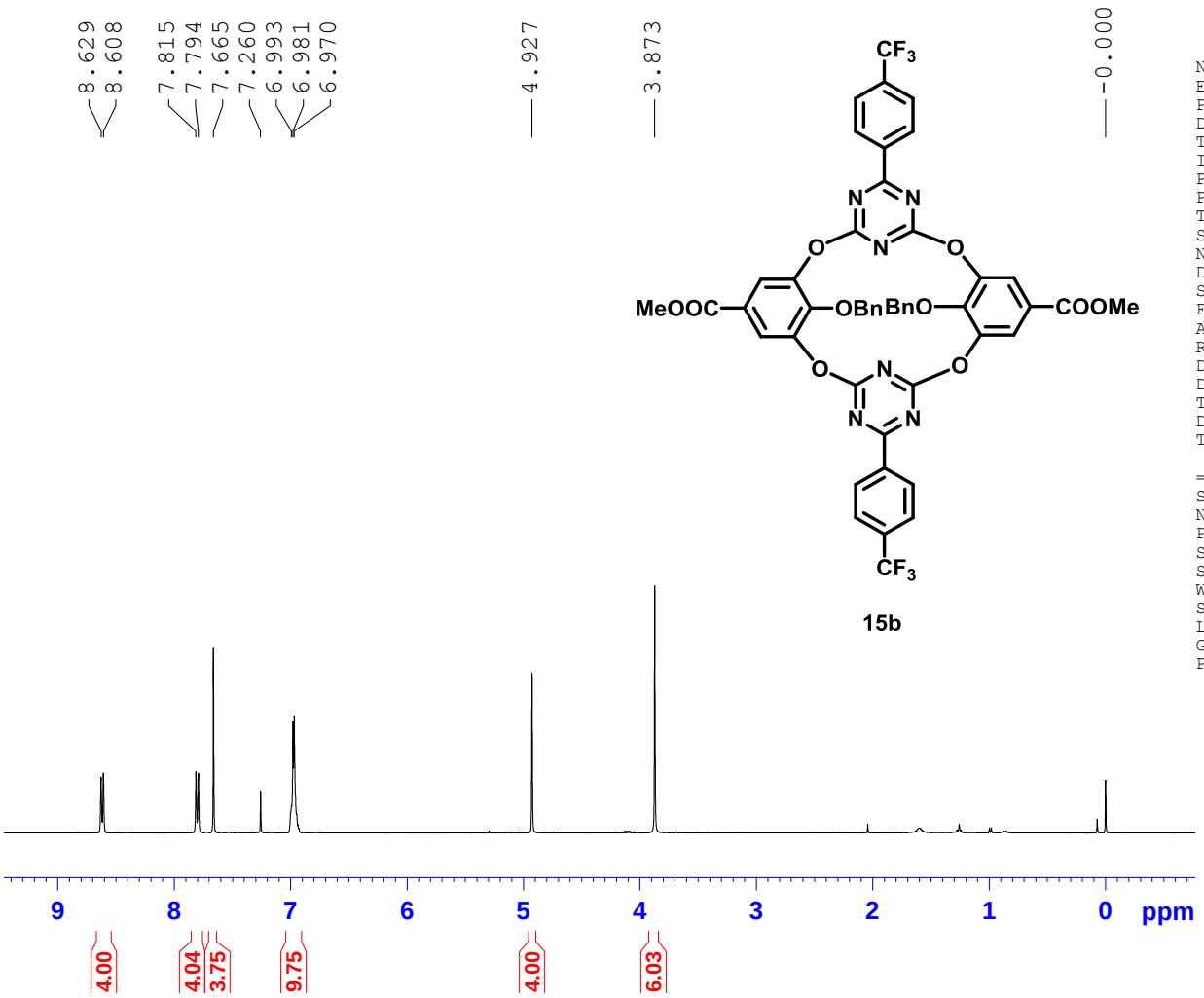

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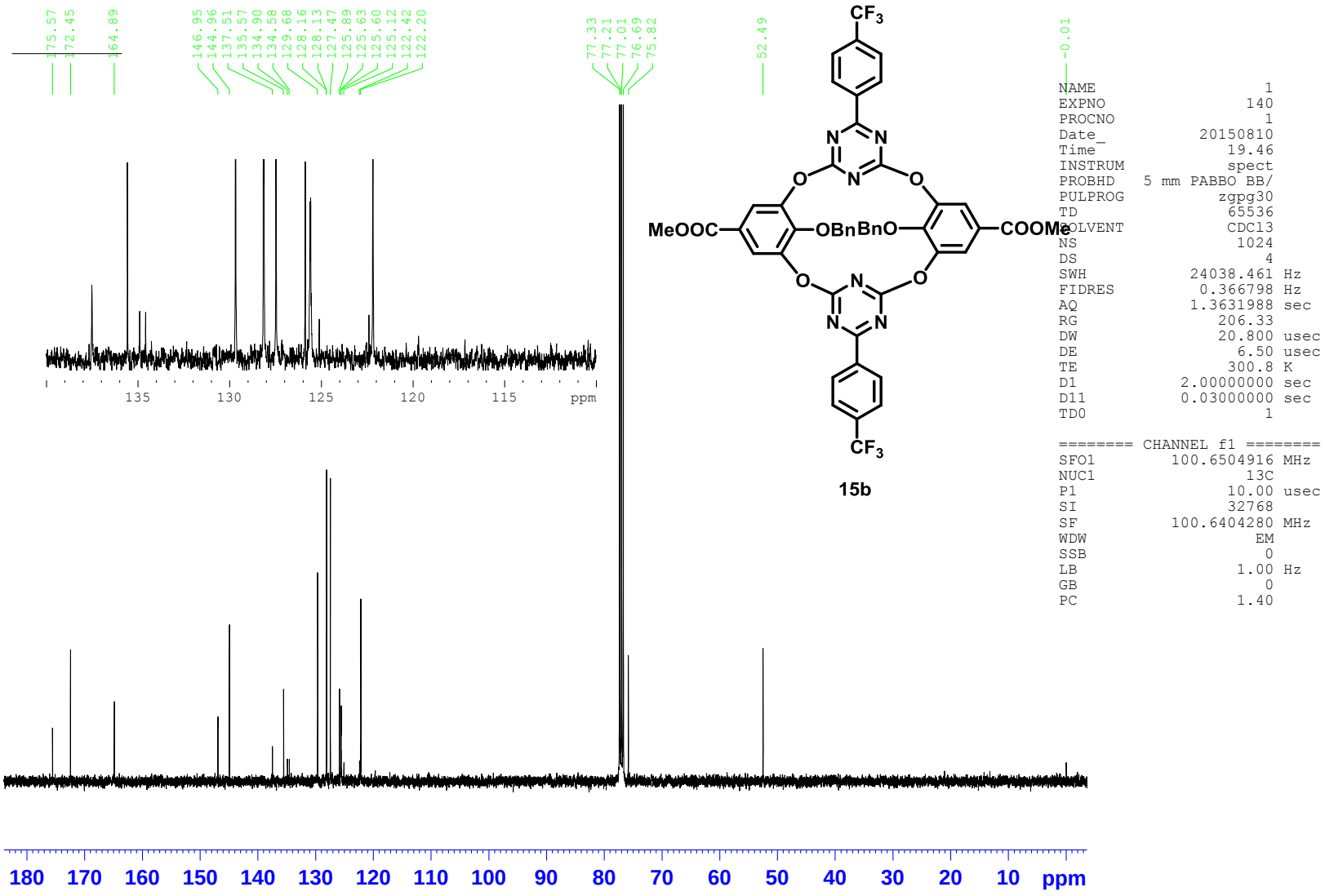
PCF3Bn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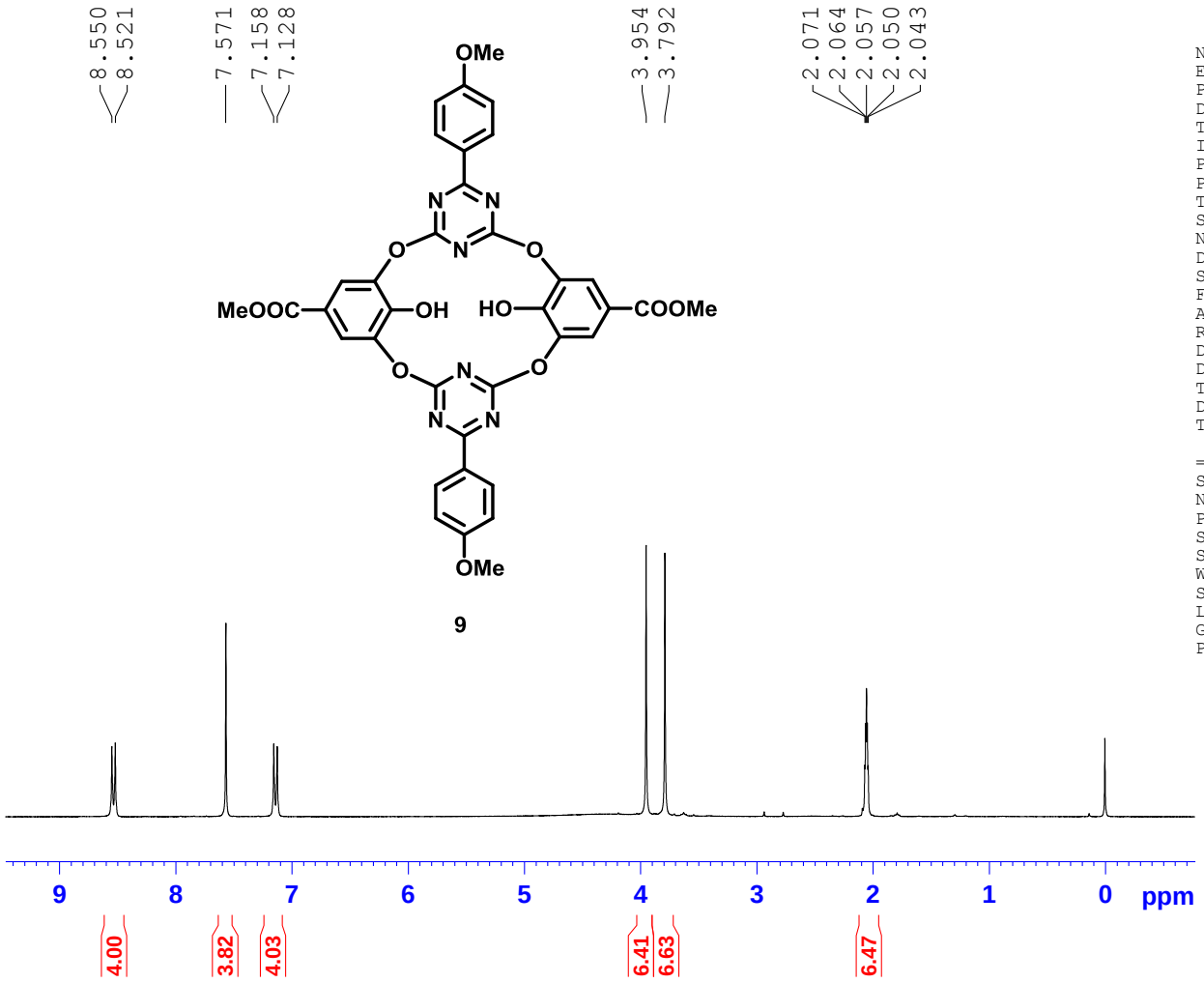
p-MeO OH

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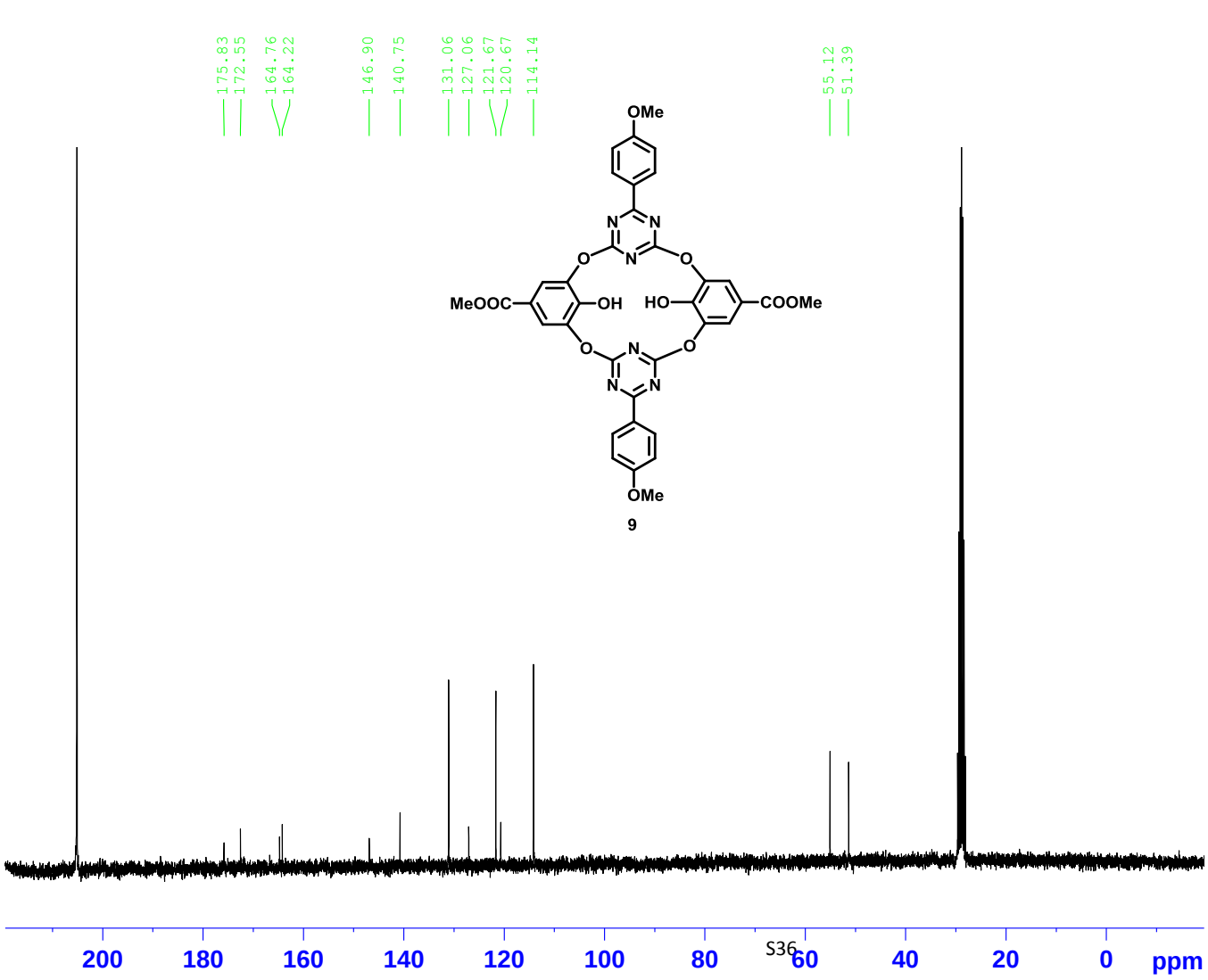


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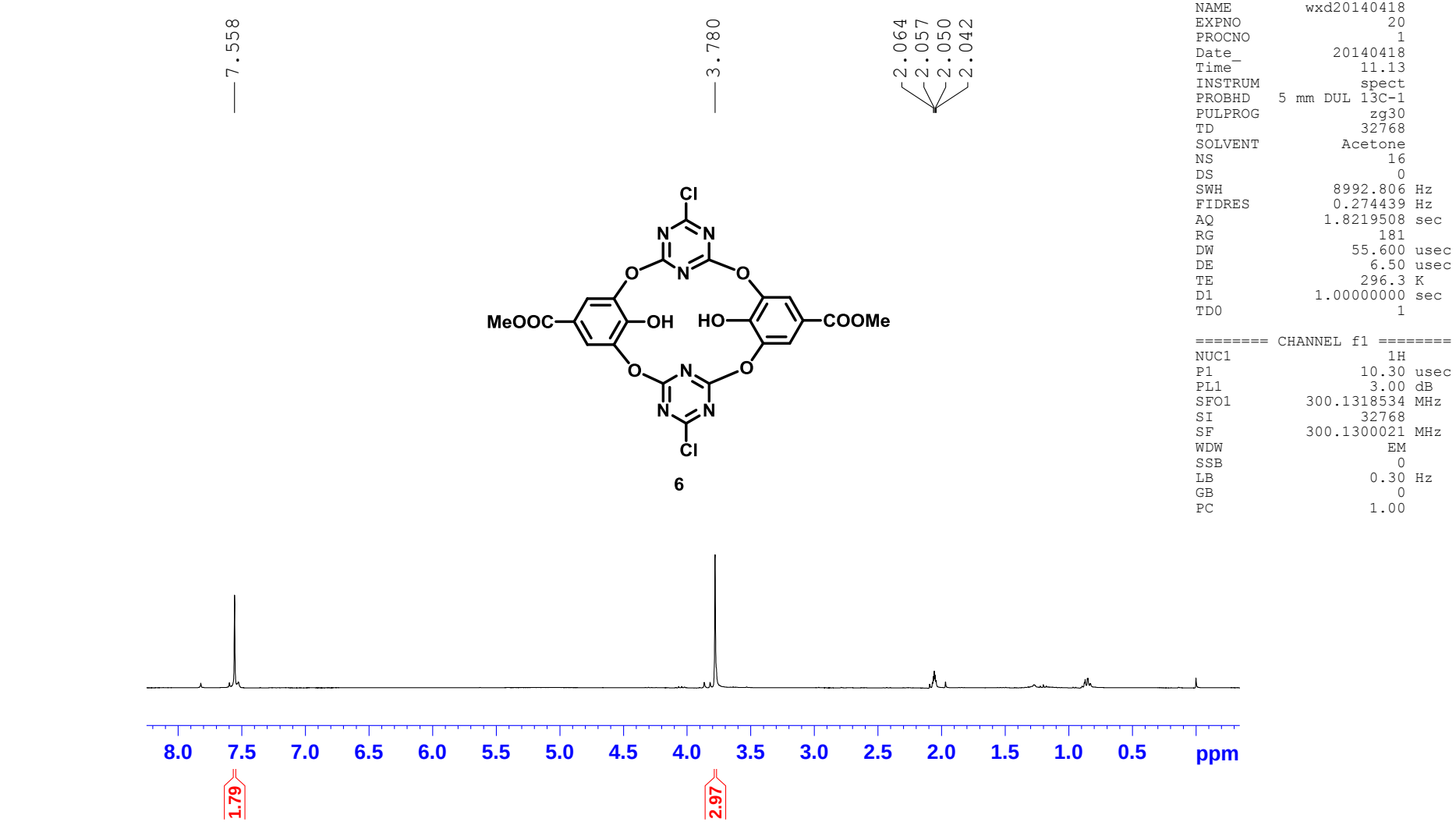


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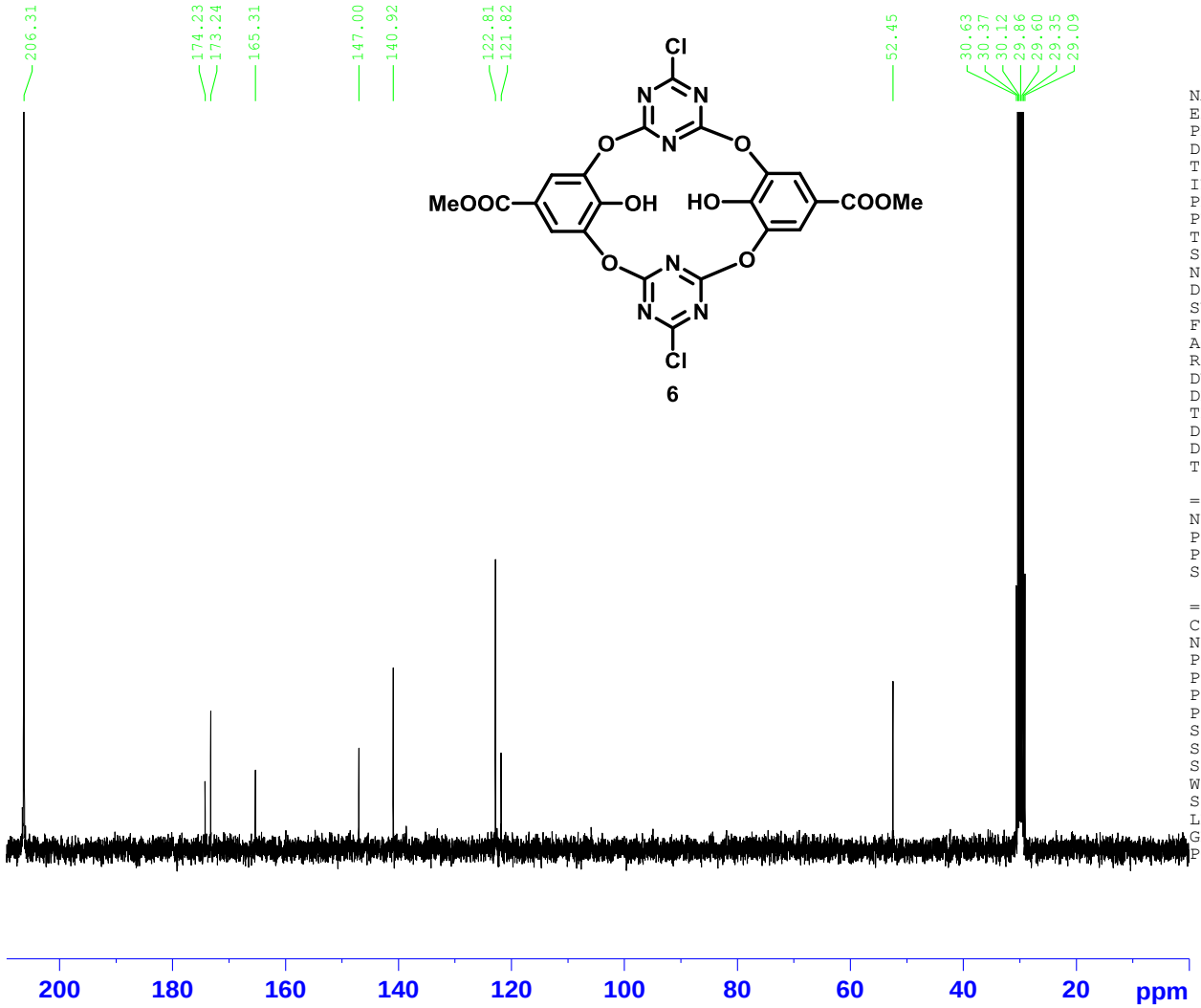


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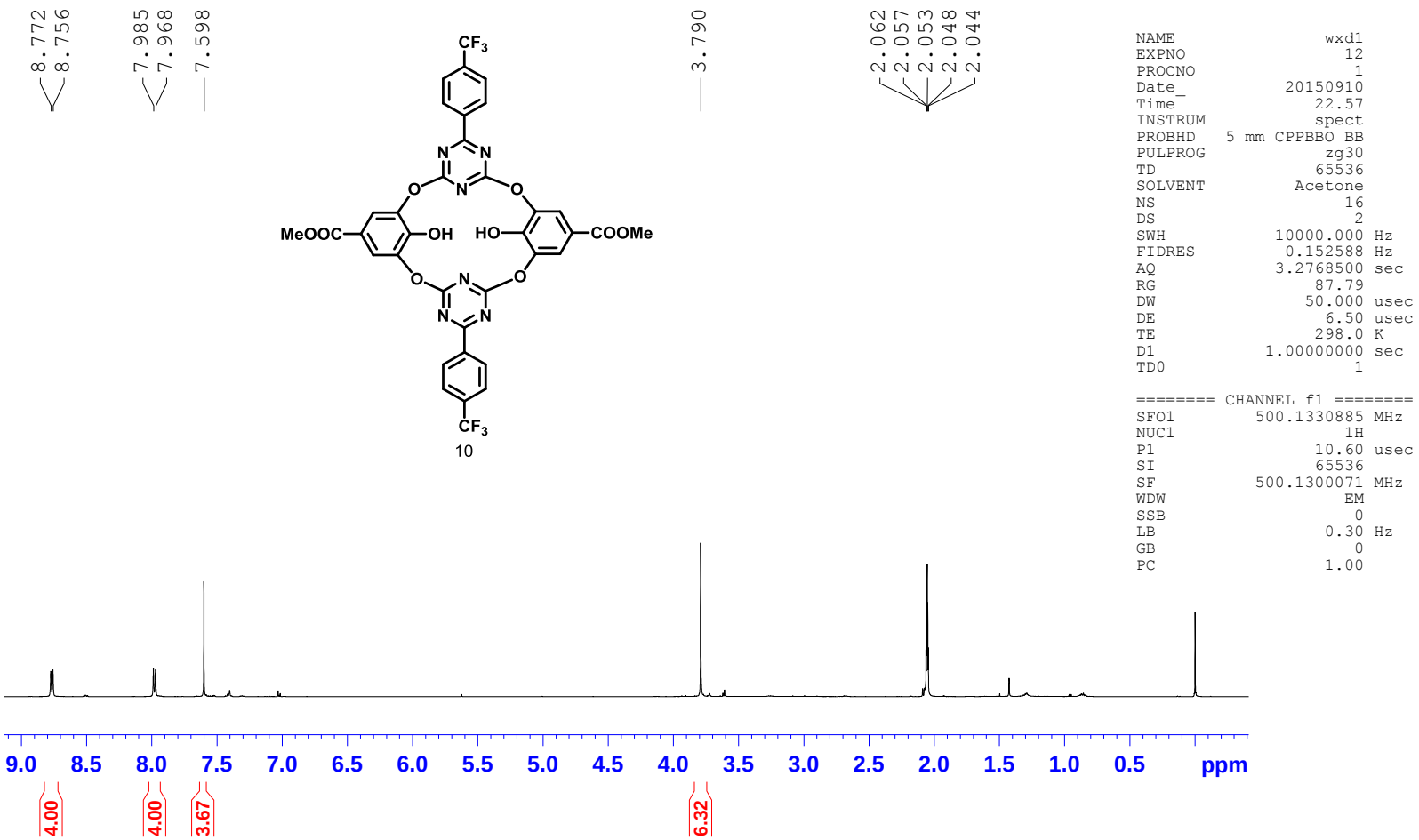
DW                27.800 usec
DE                 6.50 usec
TE                296.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

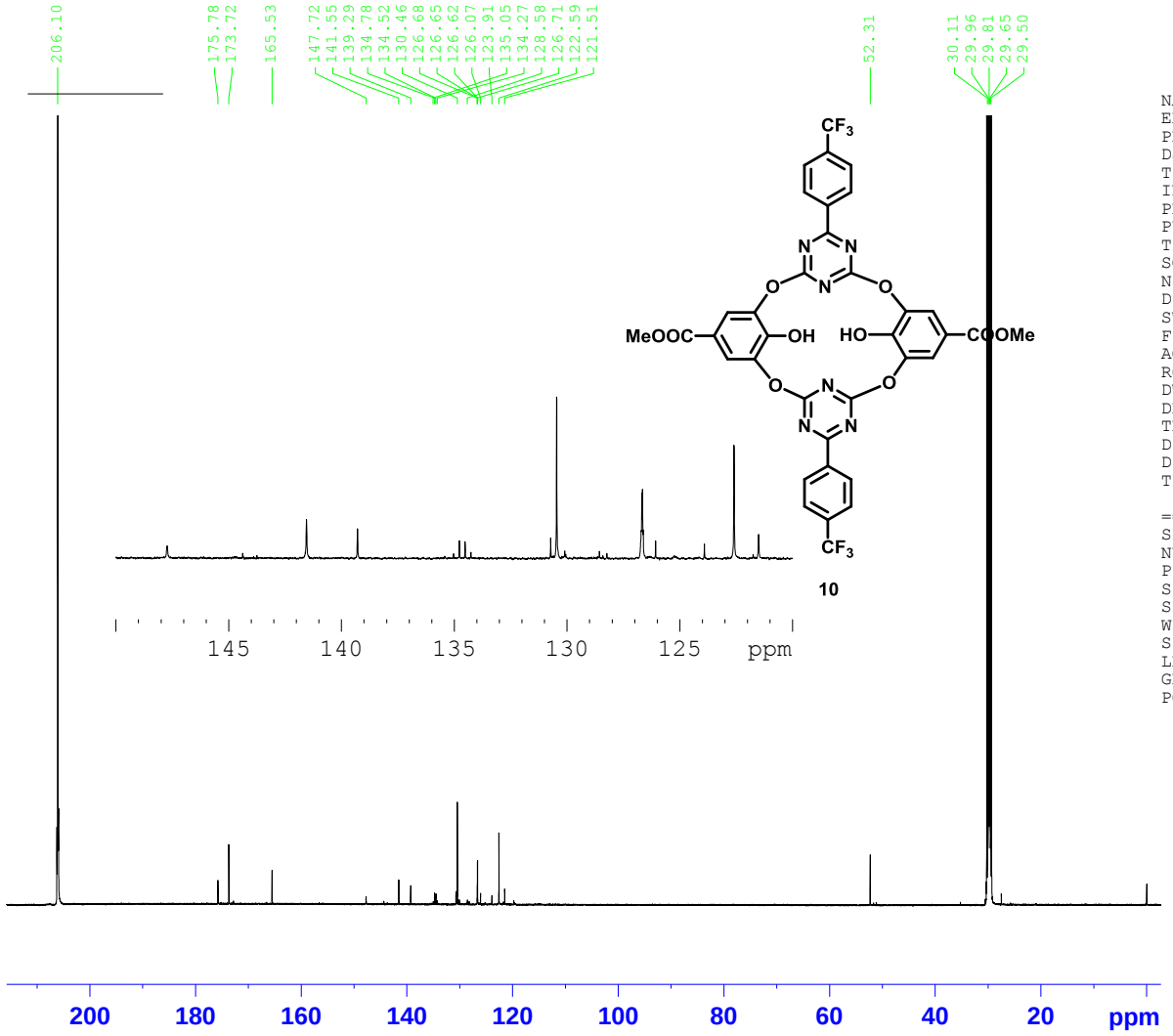
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.74 dB
PL13               23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF            75.4676811 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

pCF3OH


8.772
8.756
7.985
7.968
7.598
3.790
2.062
2.057
2.053
2.048
2.044
CF3
MeOOC
OH
HO
COOMe
CF3
10
NAME wxd1
EXPNO 12
PROCNO 1
Date_ 20150910
Time 22.57
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 87.79
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300071 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.00
4.00
3.67
6.32

pCF3OH



NAME	wxd1
EXPNO	13
PROCNO	1
Date_	20150910
Time	23.02
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	10240
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.89
DW	16.800 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	9.80 usec
SI	32768
SF	125.7576795 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40