Electronic Supplementary Information

Synthesis and characterization of cyclodiphosphazanes embedded macrocycle tetrabromo-resorcin[4]arene-tetrakis(cyclodiphosphazane) and its tetra-rhodium(I) complex

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Table S1 Crystallographic information for compounds **1** and **2**.

	1	**2**
Empirical formula	$C_{76}H_{115}Br_4N_8O_8P_8 \cdot 1(C_6H_{14})$	$C_{216}H_{304}Br_8Cl_8N_{16}O_{16}P_{16}Rh_8$
Fw	1922.32	5622.41
Cryst. System	Triclinic	Orthorhombic
Space group	P-1	Amm2
a, Å	12.7606(3)	32.9022(9)
b, Å	19.6694(5)	37.7479(10)
c, Å	19.8216(5)	12.5306(4)
α, deg	89.631(2)	90
β, deg	72.434(2)	90
γ, deg	83.770(2)	90
V, A^3	4713.3(2)	15562.9(8)
Z	2	2
$D_{calc,}$ g cm^{-3}	1.355	1.200
μ (Mo Ka), mm^{-1}	1.898	1.637
F(000)	2002.0	5712.0
crystal size, mm	0.271 × 0.091 × 0.072	0.254 × 0.165 × 0.092
T(K)	100	100
*2*θ range, deg	4.168 to 49.998	3.642 to 50
total no. Reflns	49983	87929
no. of indep reflns	16572 [R_{int} = 0.0819]	14156 [R_{int} = 0.1771]
S	1.033	1.024
R_1	0.0582	0.0719
wR_2	0.1445	0.1877

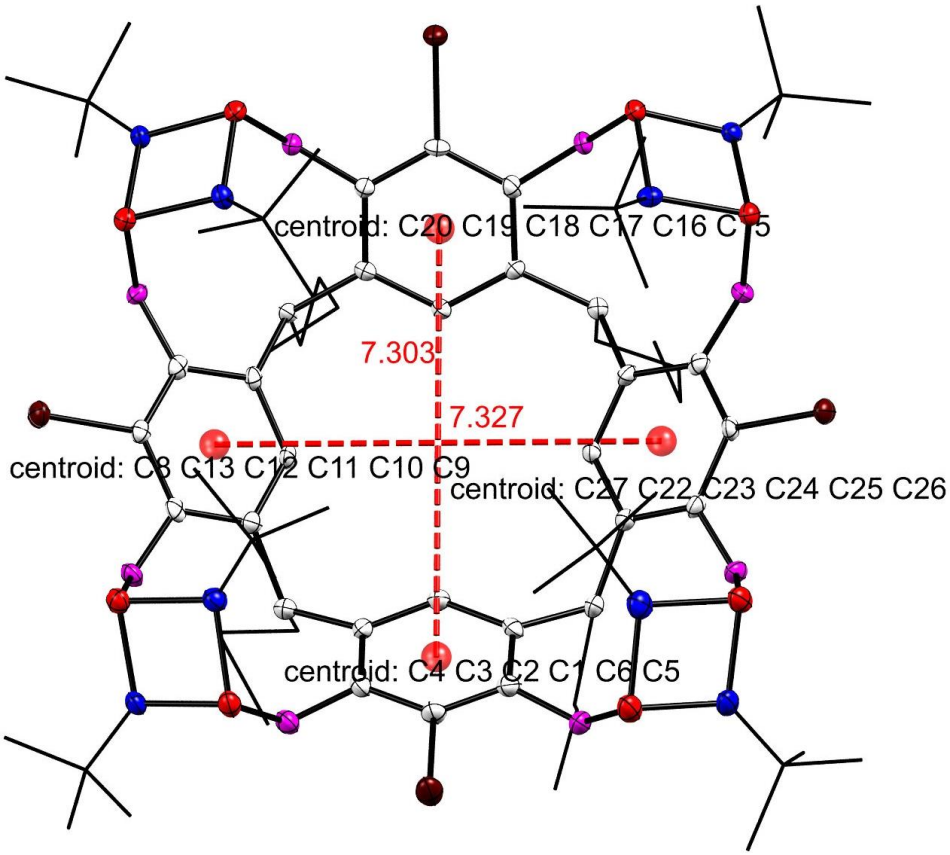

Fig. S1 Molecular structure of **1**(top view). Distances between the centroids of the phenyl groups of resorcin[4]arene core showing C_{4v} *crown* conformation of **1.**

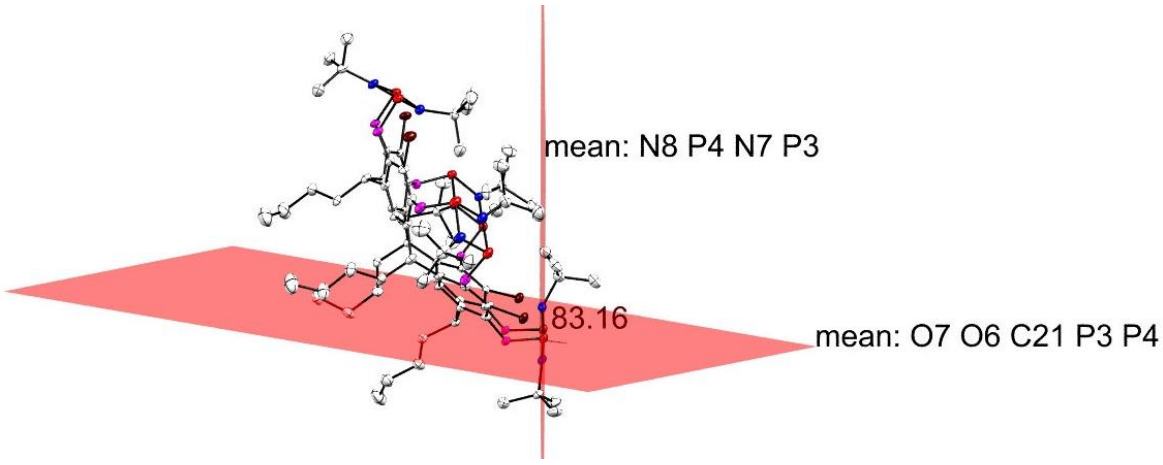

Fig. S2 Molecular structure of **1** showing the angle between planes (O7O6C21P3P4) and (N8P4N7P3).

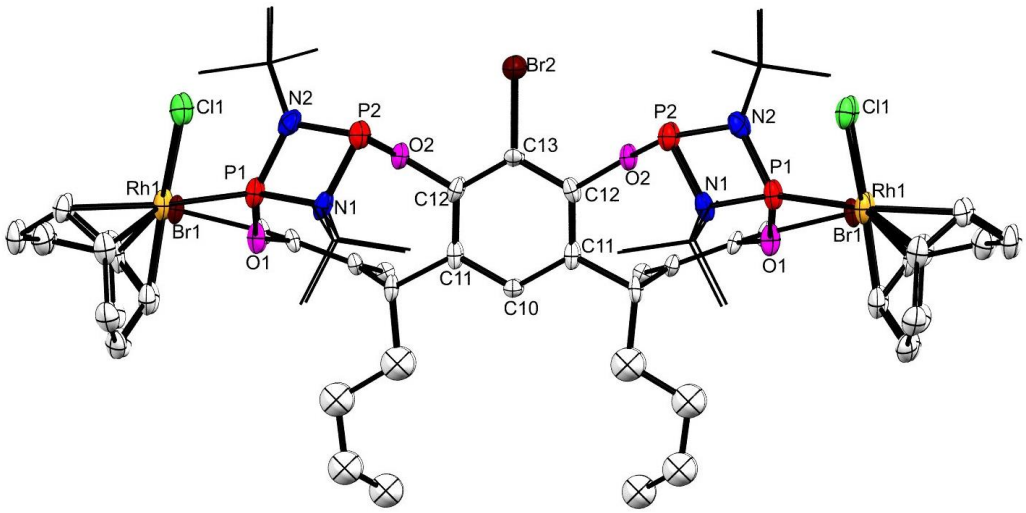

Fig. S3 Molecular structure of **2**. Depicting the structural arrangements (front view).

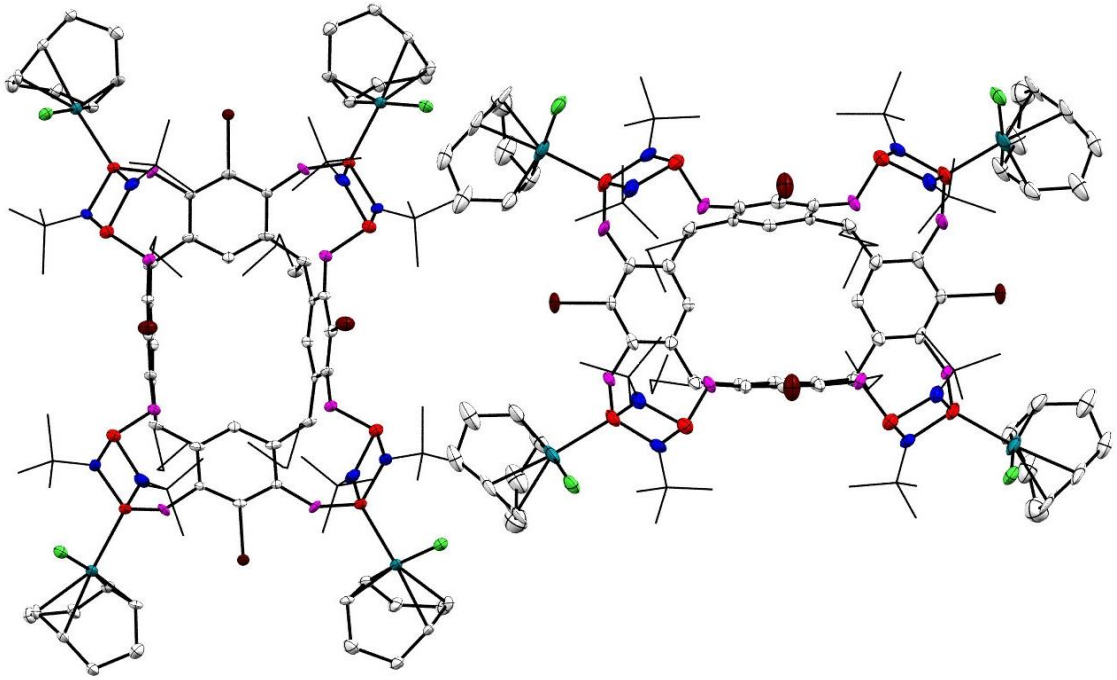

Fig. S4 Molecular structure of **2**(top view). Selected bond lengths (Å) and bond angles (°): Rh1−P1 2.237(3), Rh1−Cl1 2.370(4), P1−O1 1.646(9), P1−N1 1.707(9), O1−P1−N1 108.0(5), P1−Rh1−Cl1 90.74(13), O1−P1−Rh1 111.4(3).

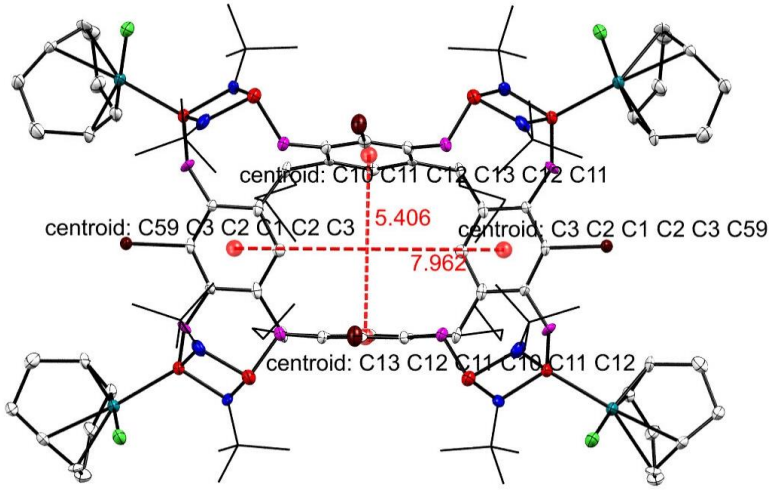

Fig. S5 Molecular structure of **2**(top view). Distances between the centroids of the phenyl groups of resorcin[4]arene showing C_{2v} *boat* conformation of **2.**

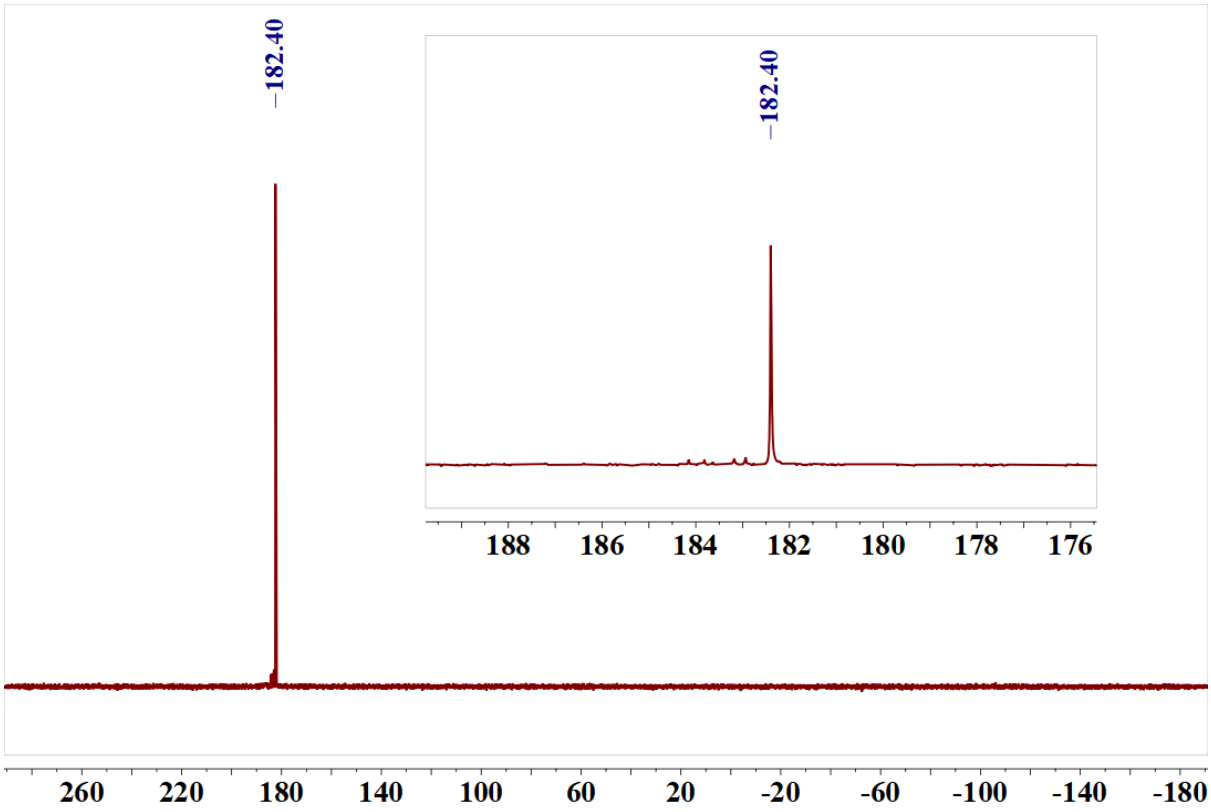

Fig. S6 $^{31}P\{^{1}H\}$ NMR spectrum of **1** in $CDCl_3$ (162 MHz).

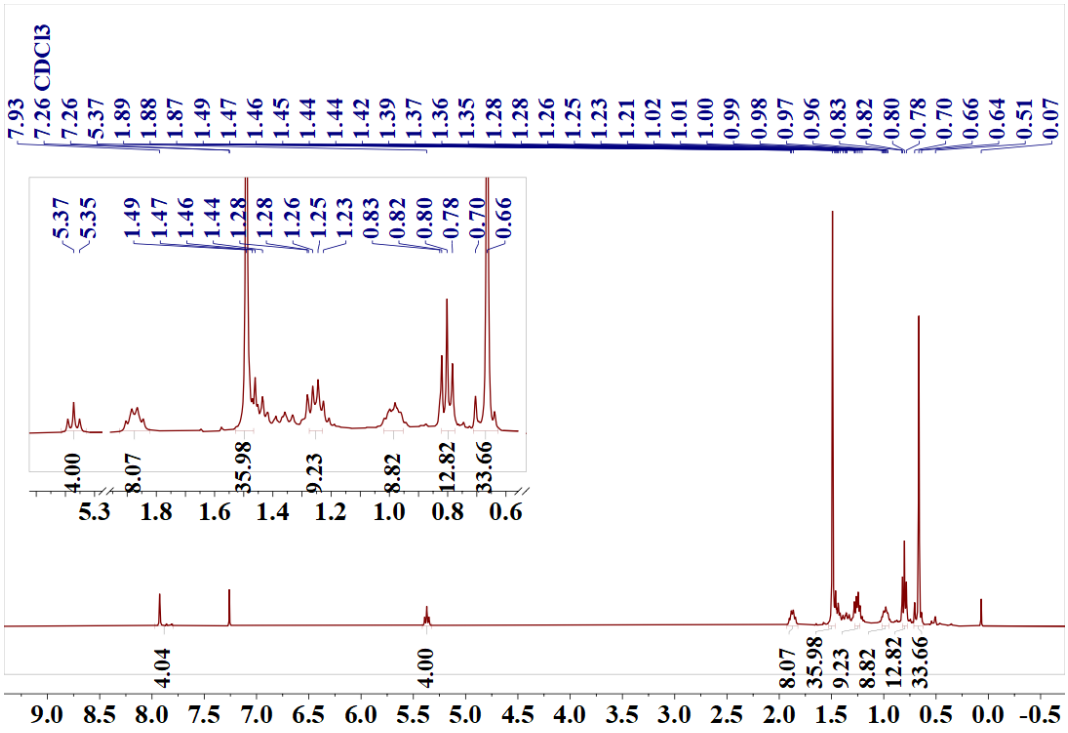

Fig. S7 ^{1}H NMR spectrum of **1** in $CDCl_3$ (400 MHz).

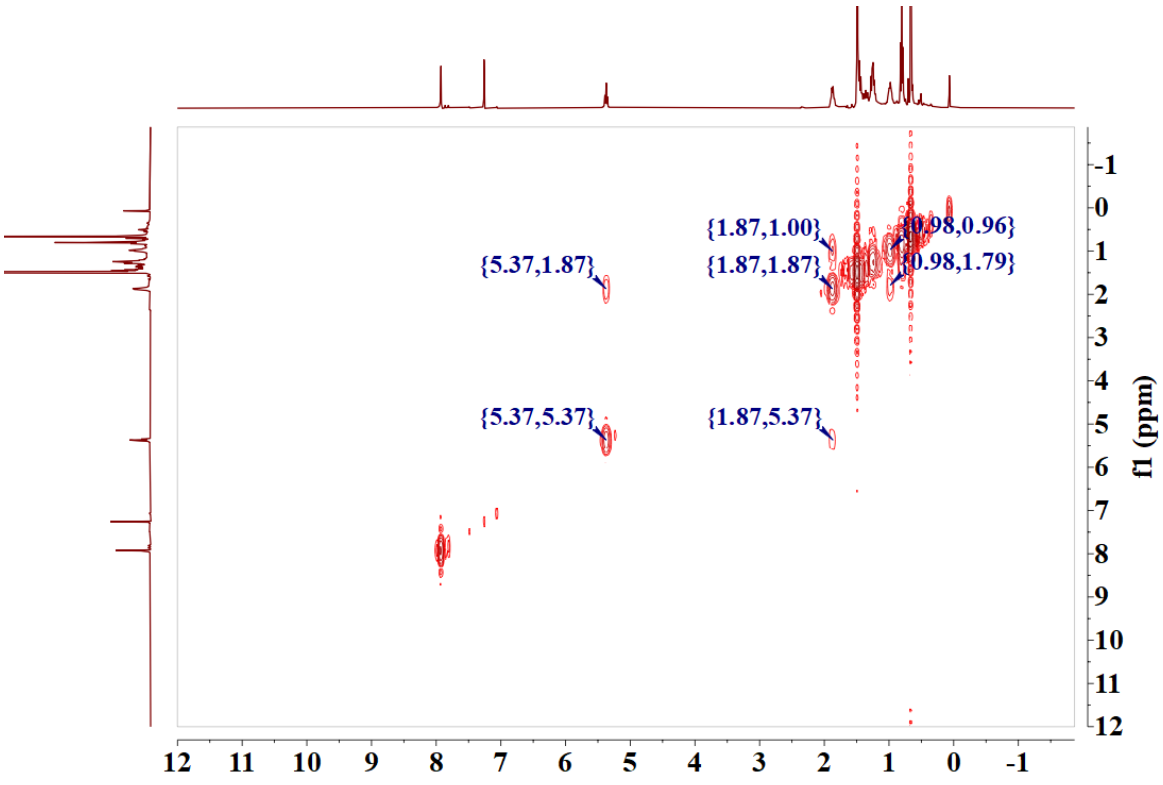

Fig. S8 ^{1}H-^{1}H COSY NMR spectrum of **1** showing the correlation between 5.37, 1.87 ppm peaks and 1.87, 0.98 ppm peaks.

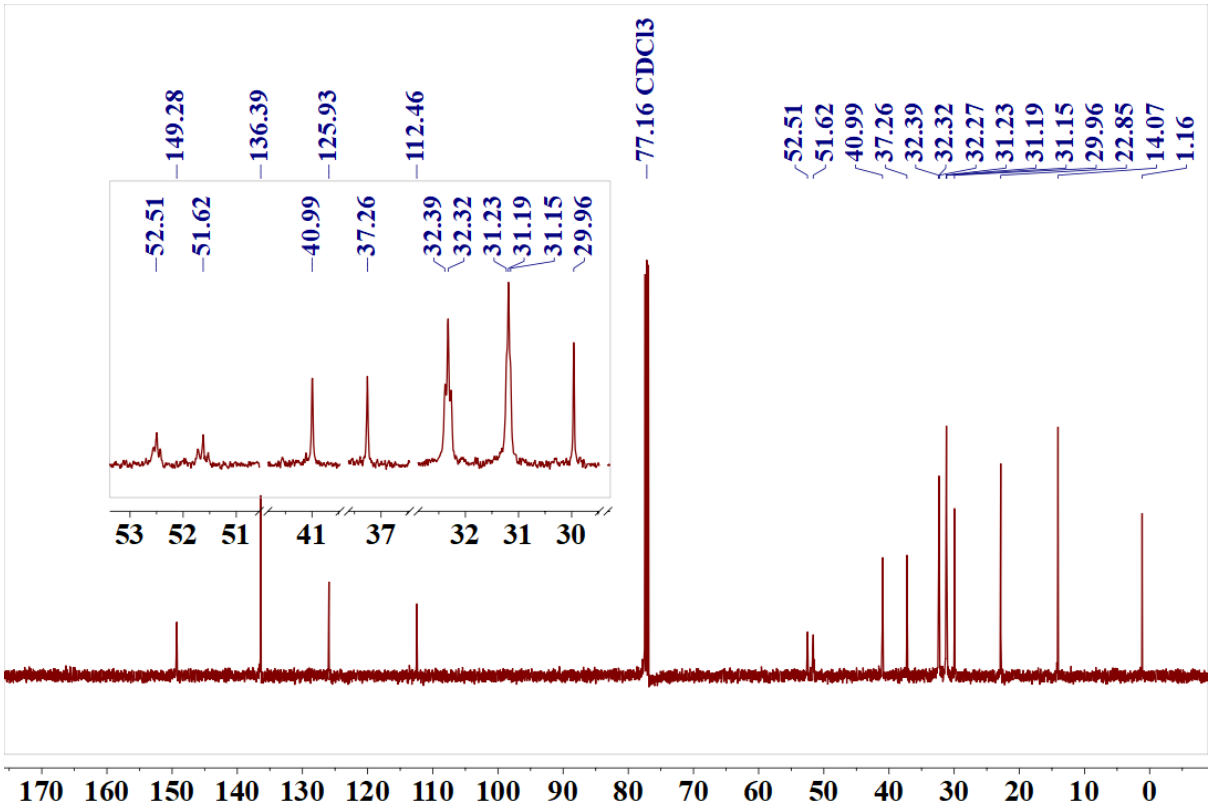

Fig. S9 $^{13}C\{^{1}H\}$ NMR spectrum of **1** in $CDCl_3$(126 MHz).

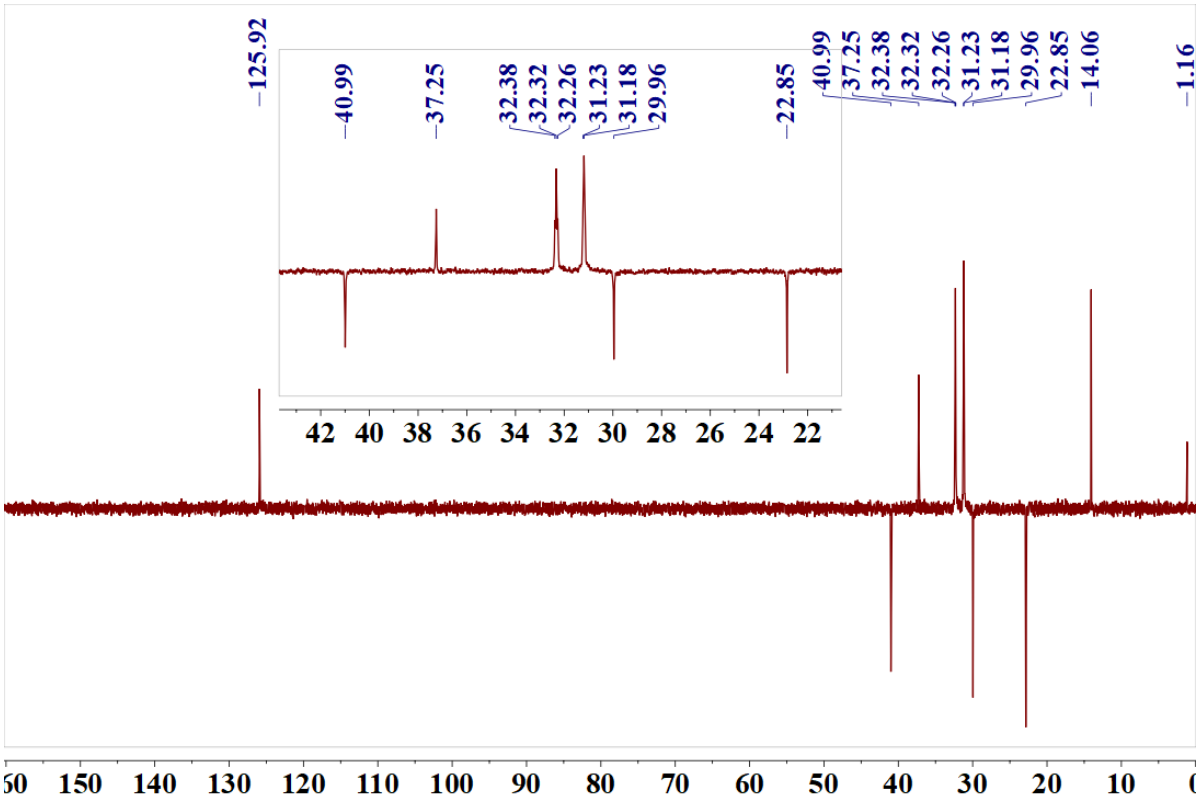

Fig. S10 ^{13}C DEPT–135 NMR spectrum of **1** in $CDCl_3$.

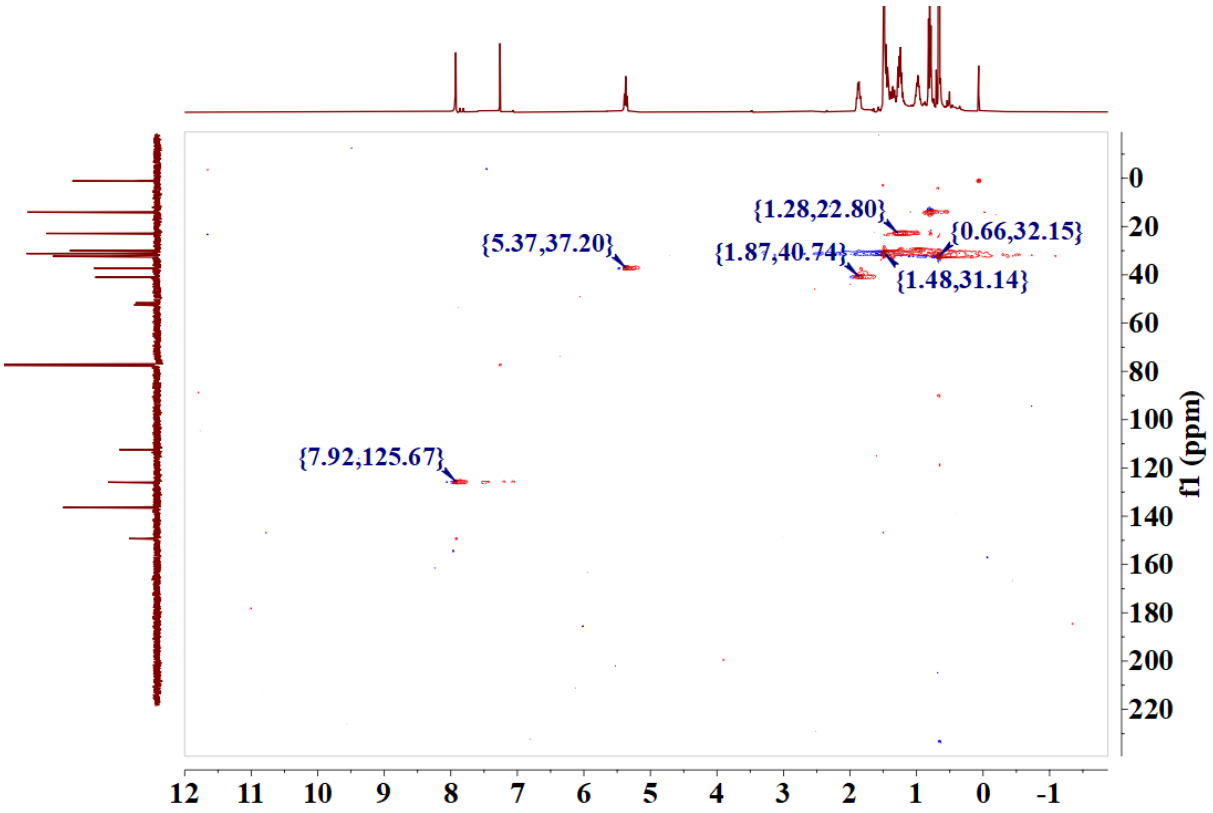

Fig. S11 $^{1}H–^{13}C$ HSQC NMR spectrum of **1** in $CDCl_3$.

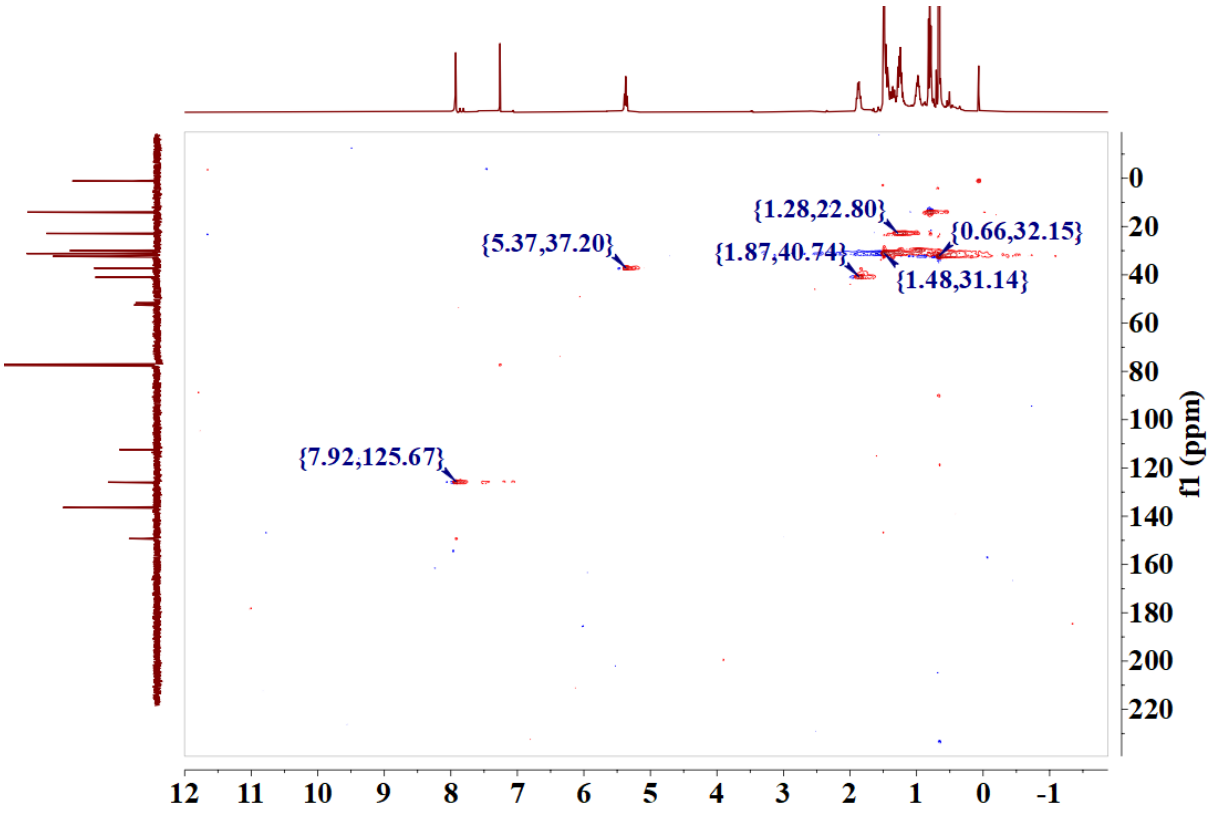

Fig. S12 $^{1}H–^{13}C$ HSQC NMR spectrum of **1** in $CDCl_3$ (Aliphatic region)

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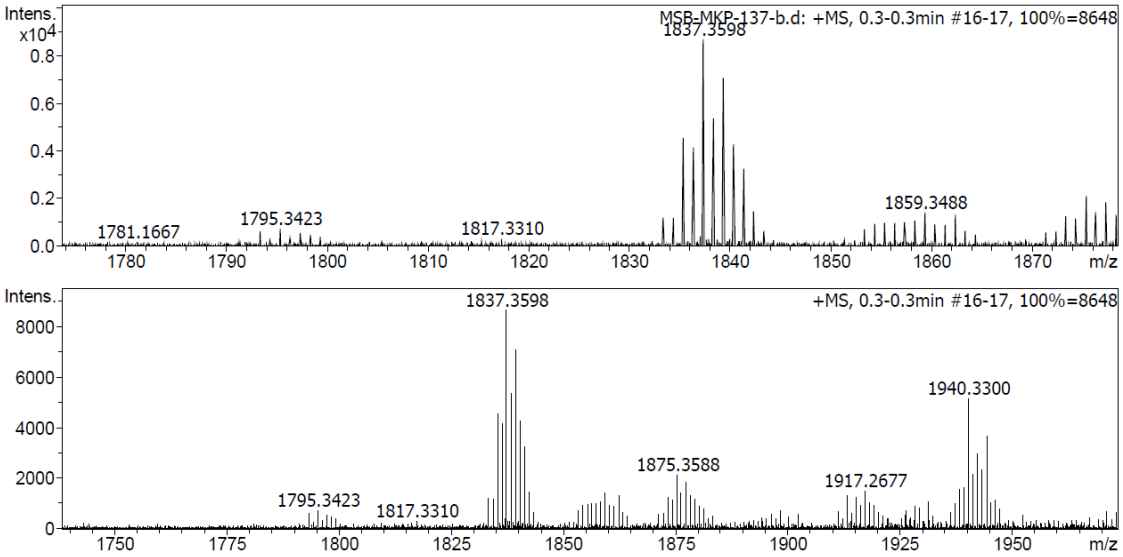
Analysis Info

Analysis Name	D:\Data\SEPT-2016\MSB-MKP-137-b.d	Acquisition Date	9/3/2016 4:07:28 PM
Method	Tune_pos_NAICSI-3000a.m	Operator	MSB IN
Sample Name	MSB-MKP-137-b	Instrument	maXis impact 282001.00081
Comment	C76H116O8N8P8Br4		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	3800 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	2100.0 Vpp	Set Divert Valve	Source



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e¯ Conf	N-Rule
1837.3598	1	C76H117Br4N8O8P8	1833.3623	-0.1	43.9	1	100.00	24.5	even	ok

Fig. S13 HRMS spectrum of **1**.

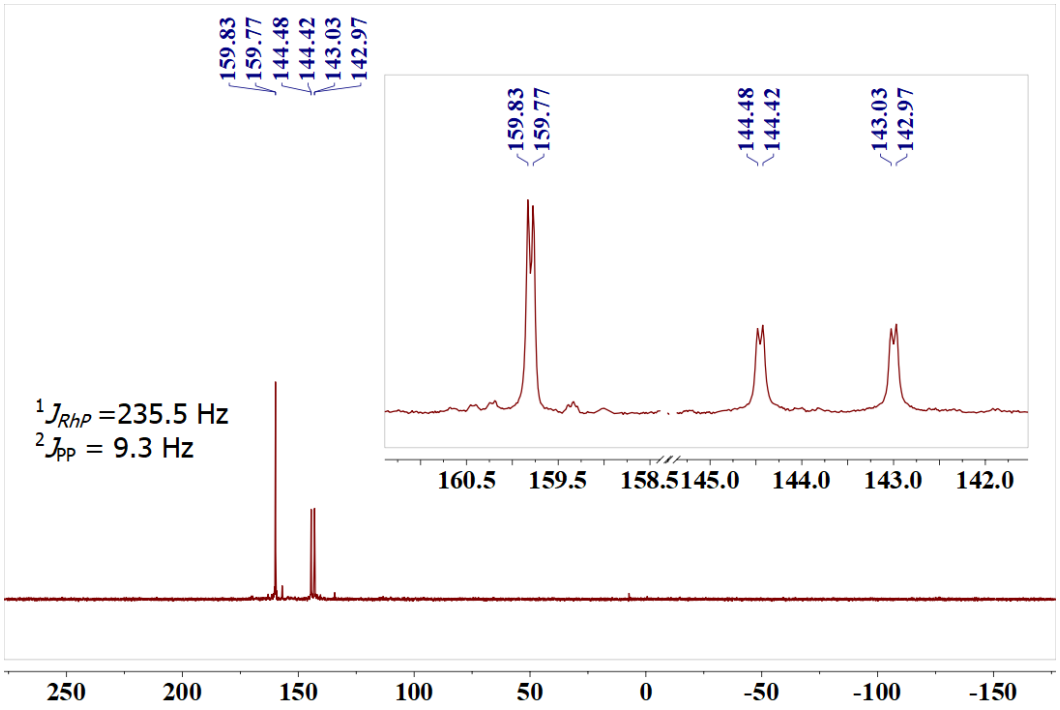


Fig. S14 $^{31}P\{^1H\}$ NMR spectrum of **2** in $CDCl_3$ (162 MHz).

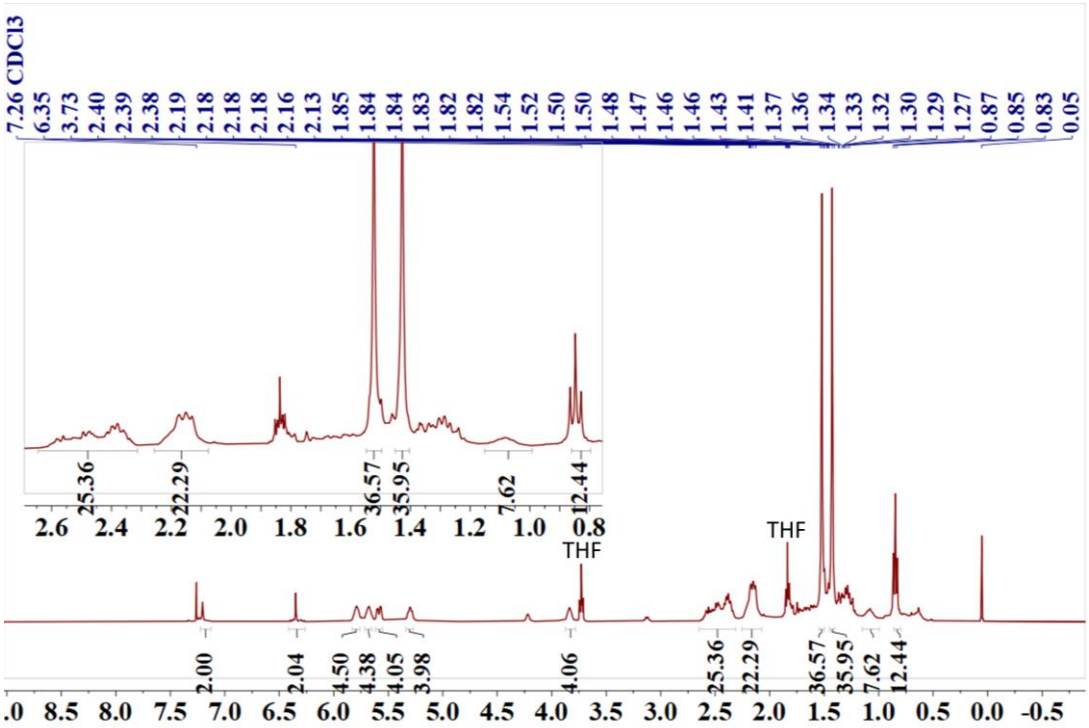


Fig. S15 ^1H NMR spectrum of **2** in $CDCl_3$ (400 MHz).

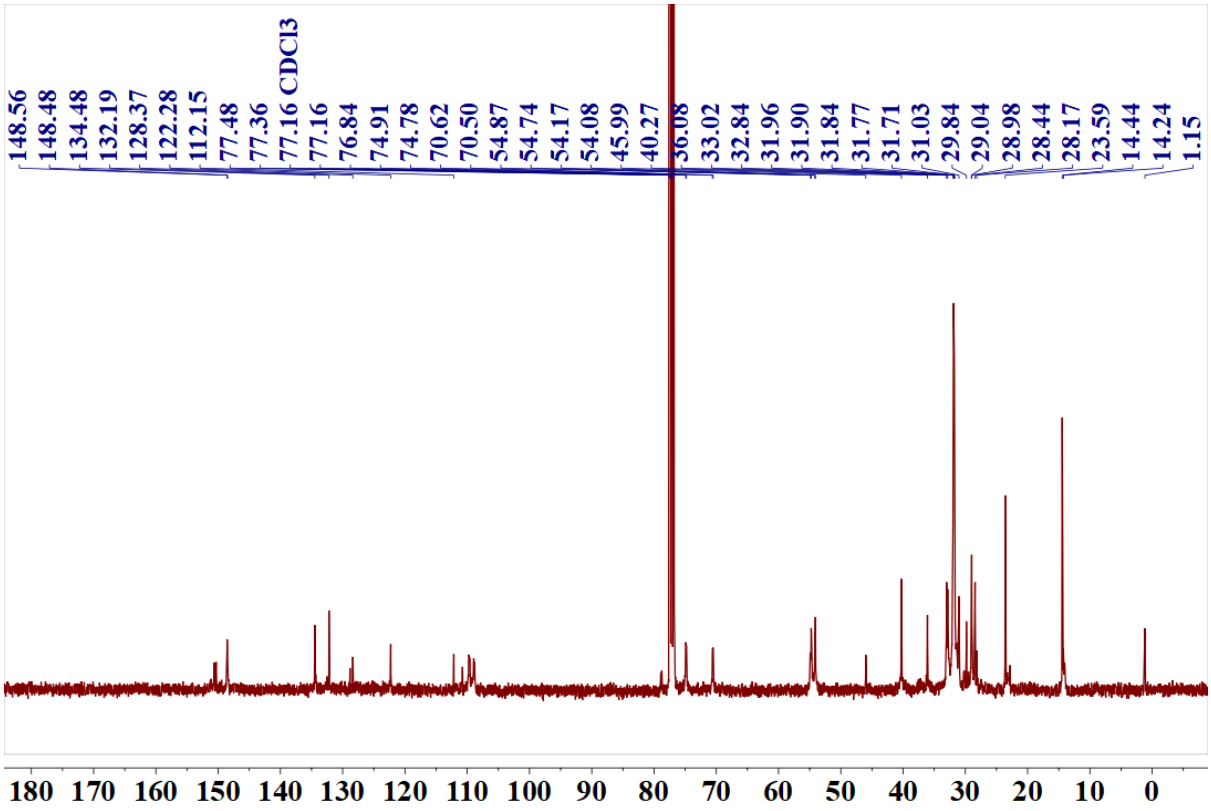


Fig. S16 $^{13}C\{^{1}H\}$ NMR spectrum of **2** in $CDCl_3$ (101 MHz).

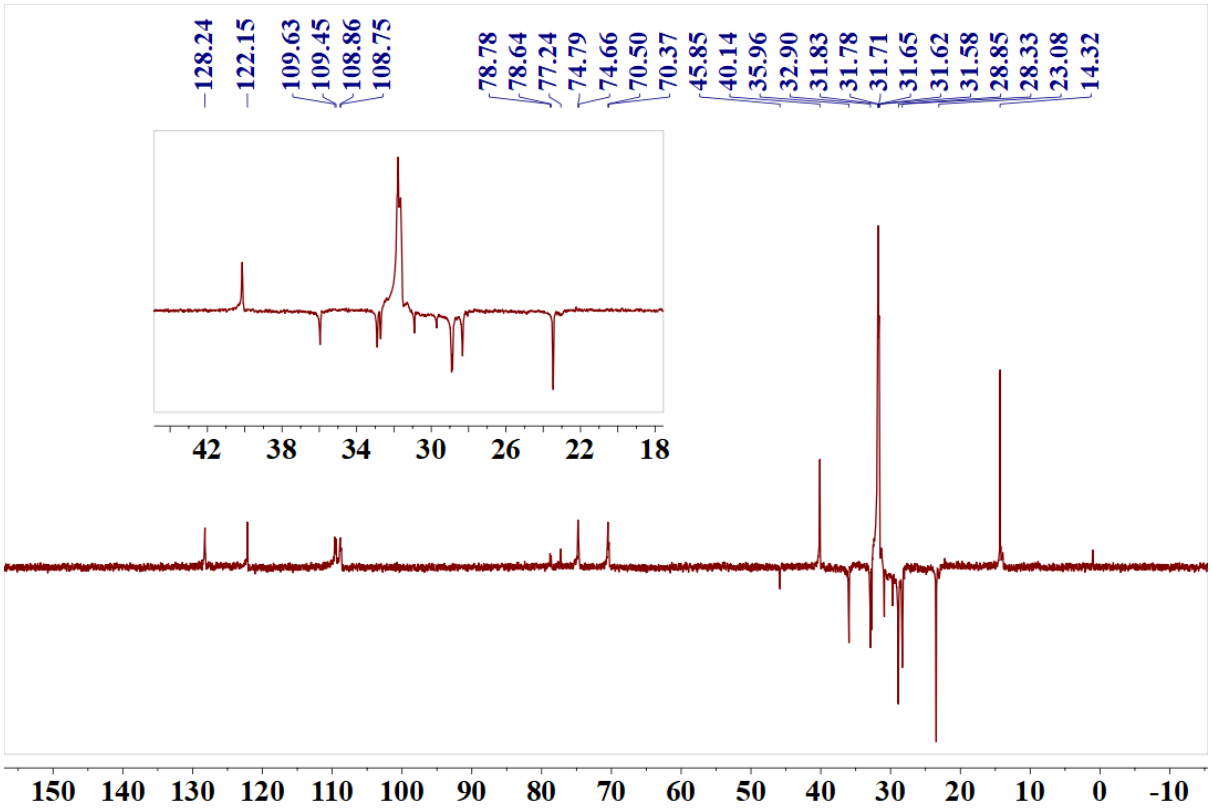


Fig. S17 ^{13}C DEPT-135 NMR spectrum of **2** in $CDCl_3$.

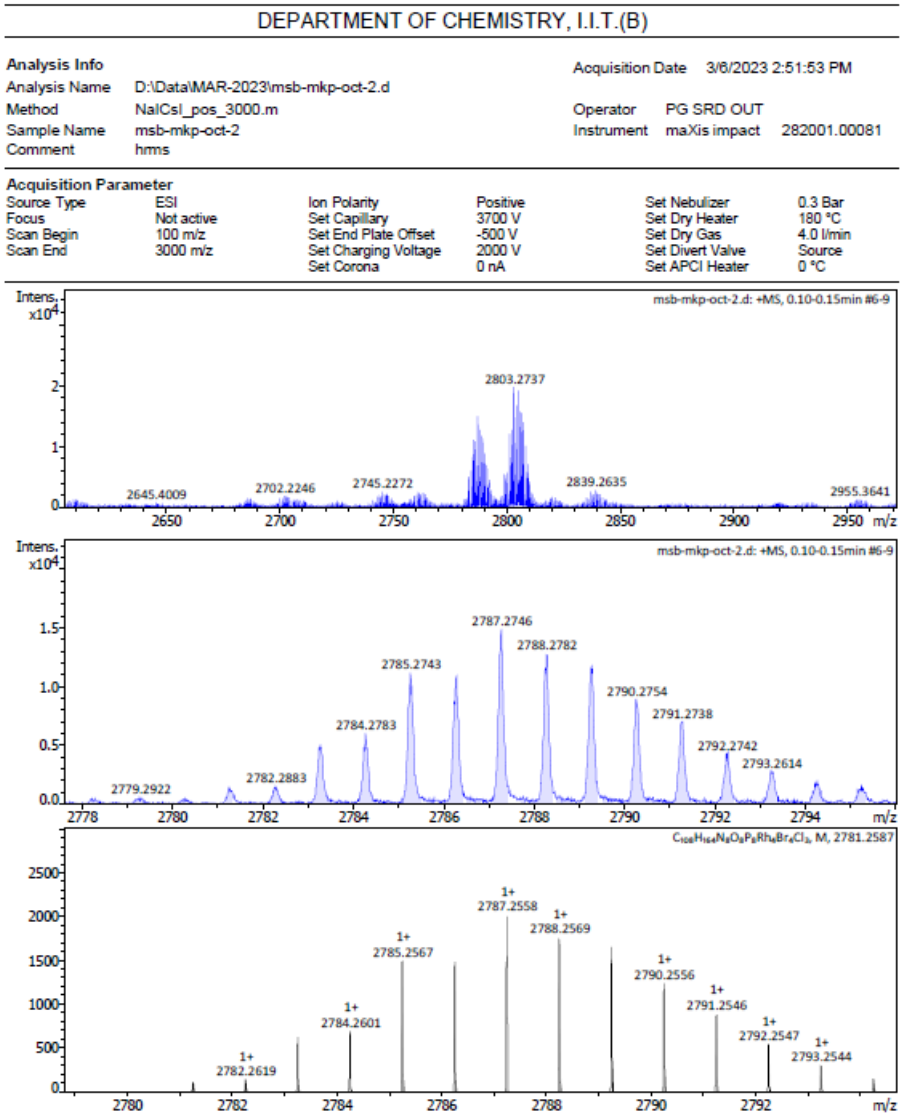


Fig. S18 HRMS spectrum of **2** showing $[M–Cl]^+$.molecular ion peak at 2787.2746.

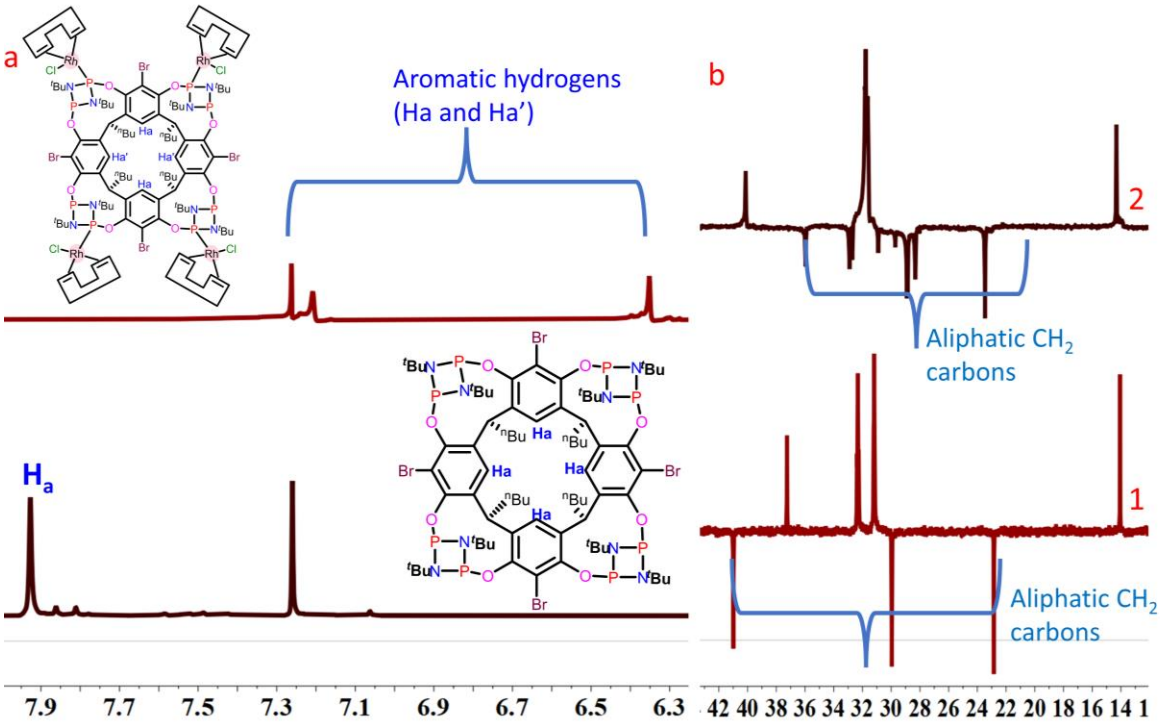


Fig. S19 Stack plots of ^{1}H (**a**: Left portion) and ^{13}C-DEPT (**b**: Right portion) NMR spectra of **1** and **2** showing the changes after complex formation.