Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Liquid Crystalline Macrocyclic Azacalix[4]pyridine and Its Complexes with Zinc Ion: Conformational Change from Saddle to Flattened Shape

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1. General Information

NMR spectra were recorded using a JEOL ECX-400 at 400 MHz for ^{1}H NMR and at 100 MHz for ^{13}C NMR in $CDCl_3$ or in 1,1,2,2,-tetrachloroethane for complexes **1**·$ZnCl_2$. Chemical shifts of ^{1}H and ^{13}C NMR signals were quoted to $CDCl_3$ (7.26 ppm for C^1HCl_3 in $CDCl_3$, 77.2 ppm for $^{13}CDCl_3$, respectively). Abbreviations are used in the description of NMR data as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, br = broad), coupling constant (*J*, Hz). High-resolution electrospray ionization (ESI) mass spectra were obtained on a Thermo Fisher Exactive Mass Spectrometer. Infrared spectra were recorded using a Nicolet-6700 FT-IR spectrometer with KBr pellets in the 4000-400 cm^{-1} region. Elemental analysis was recorded on Thermo Quest CE Instruments flash EA1112 analyser.

Liquid crystal behaviors of the samples were characterized with a polarized optical microscope (POM) equipped with a Mettler-Toledo FP82 HT hot-stage. Differential scanning calorimetry (DSC) thermograms were obtained on a TA Instruments Q100. V9.9 (build 303). One-dimensional (1D) X-ray diffraction (XRD) experiments were carried out on a high-flux small angle X-ray scattering (SAXS) instrument (SAXSess, Anton Paar) equipped with Kratky block-collimation system and a PANalytical PW3830 sealed-tube X-ray generator (Cu Kα) with the wavelength λ being 0.1542 nm. The diffraction patterns were recorded on an imaging-plate with the q range ($q = 4\pi\sin\theta/\lambda$) covering from 0.06 to 29 nm^{-1}. The d-spacing is given by $2\pi/q$. In addition, to study the temperature dependence on the phase evolution, a temperature control unit (Anton Paar TCS300) was utilized in conjunction with the SAXSess. Two-dimensional (2D) XRD experiments were carried out on a Bruker D8Discover diffractometer with a GADDS as the 2D detector. In all XRD experiments, the diffraction peak positions were calibrated with silver behenate and the background scattering was subtracted from the sample pattern.

Reconstruction of relative electron density distribution in real space was carried out based on the XRD data by using the formula for 2D Fourier transformation:

$$\rho(x,y) - \rho_0 = \sum_{hk} F(hk)\exp[-2\pi(hx+ky)]$$

where x and y are the fractional coordinates of a point in the unit cell and ρ_0 is the average electron density. $F(hk)$ is the complex structure factor and carried out by the function $|F(hk)| = \sqrt{I(hk)}$

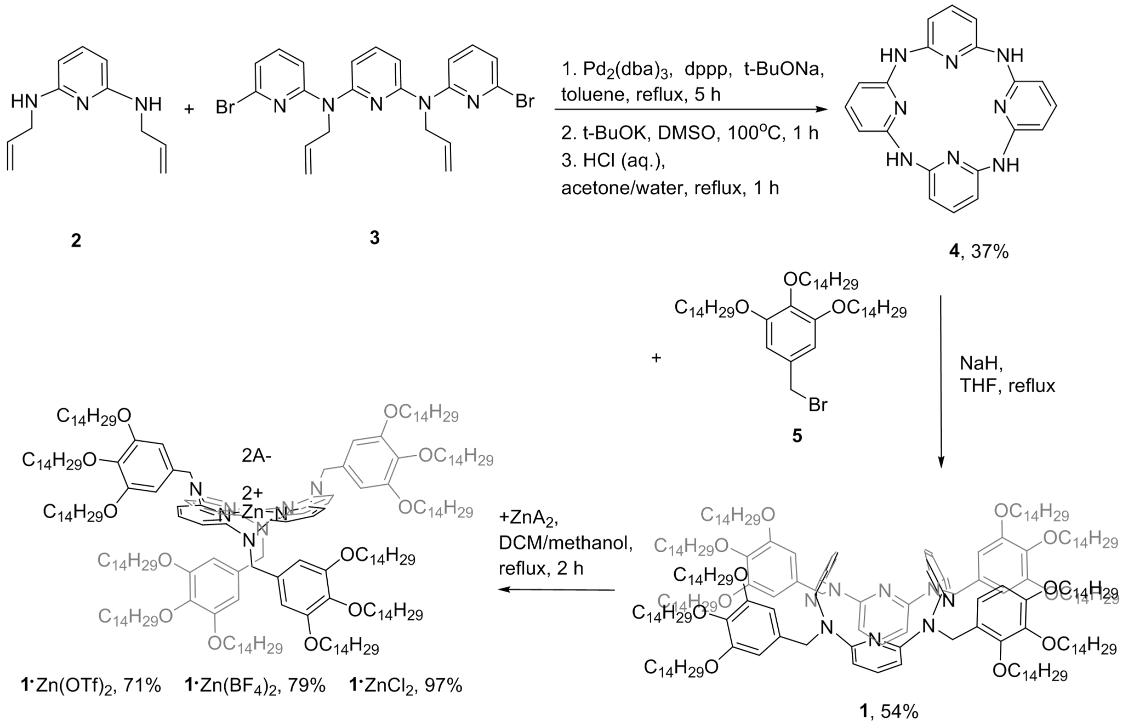
where $I(hk)$ is the diffraction intensities. As a consequence of the 2D lattices in the case of the Cr phase of **1** being centrosymmetric, the structure factors are real and given by $F(hk) = \pm|F(hk)|$ with corresponding possible phase of 0 (+) or π (–). Then the electron density can be expressed as:

$$\rho(x, y) - \rho_0 = \sum_{hk} \pm \sqrt{I(hk)} \cos[-2\pi(hx + ky)]$$

Considering the Cr phase of **1**, the first six clear peaks on the equator of the 2D XRD pattern can be indexed as (10), (20), (02), (22), (40), (04). The electron density profiles were calculated using the suitable phase combinations of "+ – – + + –" for the corresponding reflections.

2. Synthesis

Compounds **2-4**[1] and **5**[2] were synthesized according to literatures.



Scheme S1 Synthesis of *N*-substituted azacalix[4]pyridine derivative **1** and its zinc complexes.

Synthesis of N-[3,4,5-tris(tetradecyloxy)benzyl]-bridged calix[4]pyridine 1. To a solution of **4** (74 mg, 0.2 mmol) in dry THF (50 mL) at room temperature was added NaH (115 mg, 4.8 mmol) slowly and the mixture was heated to reflux. After10 h, compound **5** (1.62g, 2 mmol) was added slowly and the reaction was refluxed for another 12 h. The reaction mixture was then cooled down to room temperature and water (1 mL) was added slowly. The solvent was removed under reduced pressure, and the residue was dissolved in DCM (50 mL). The organic solution was washed with brine (3 × 50 mL) and dried over with anhydrous $MgSO_4$. After removal of solvent, the residue was chromatographed on a silica gel column (Silicycle® Flash P60) with a mixture of petroleum ether and ethyl acetate as the mobile phase. Compound **1** was obtained and recrystallized from the mixture of ethyl ester and methanol at -20 °C to give white amorphous solid (354 mg, 54%): IR (KBr, cm^{-1}) ν 2920, 2851, 1592, 1466, 1436, 1381, 1331, 1220, 1116; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.30 (t, 4H, *J* = 8.0 Hz), 6.55 (s, 8H), 6.38 (d, 8H, *J* = 5.2 Hz), 5.10 (d, 4H, *J* = 16.8 Hz), 4.51 (d, 4H, *J* = 17.6 Hz), 3.87 (t, 24H, *J* = 6.2 Hz), 1.76-1.68 (m, 24H), 1.44 - 1.41 (m, 24H), 1.38 - 1.20 (m, 240H), 0.88 (t, 36H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 159.0, 153.4, 138.9, 137.1, 135.0, 105.2, 73.5, 69.3, 54.2, 32.1, 30.6, 29.95, 29.89, 29.72, 29.69, 29.6, 26.40, 26.36, 22.9, 14.3. HRMS (FTMS - ESI) calcd. for $C_{216}H_{376}N_8O_{12}$: $[M+H]^+$ 3275.91306; found: 3275.91433; $[M+2H]^{2+}$ 1638.46017; found: 1638.45877. Anal. Calcd. for $C_{216}H_{376}N_8O_{12}$: C, 79.16; H, 11.56; N, 3.42; found: C, 78.96; H, 11.60; N, 3.51.

General procedure for synthesis of N-[3,4,5-tris(tetradecyloxy)benzyl]-bridged calix[4]pyridine zinc salts. Compound **1** (98 mg, 0.03 mmol) and zinc salt (0.033 mmol) were added in mixture of dry DCM (5 mL) and methanol (5 mL) at room temperature and the reaction mixture was heated to reflux. After 2 h, the solvent was removed under reduced pressure, and the residue was dissolved in mixture of DCM (5 mL) and acetone (25 mL). After partial removal of solvent under reduced pressure without heating, white precipitate was filtered and washed with 5 mL acetone to afford pure **1**·ZnA_2.

N-[3,4,5-tris(tetradecyloxy)benzyl]-bridged calix[4]pyridine zinc triflate (1·Zn(OTf)₂): white solid (77 mg, yield: 71%); IR (KBr, cm^{-1}) ν 2923, 2852, 1592, 1468, 1439, 1332, 1223, 1115, 1030; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.60 (t, 4H, *J* = 8.2 Hz), 6.86 (d, 8H, *J* = 8.4Hz), 6.44 (s, 8H), 5.04 (s, 8H), 3.89 (t, 8H, *J* = 6.6 Hz), 3.74 (t, 16H, *J* = 5.4 Hz), 1.78 - 1.60 (m, 24H), 1.52 - 1.20 (m, 264H), 0.88 (t, 36H, *J* = 6.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 155.4, 153.6, 142.0, 137.2, 130.5, 120.2 (q, *J* = 320 Hz), 111.0, 105.4, 73.5, 69.1, 58.2, 32.1, 30.6, 29.9, 29.8, 29.6, 26.43, 26.35, 22.9, 14.3. HRMS (FTMS - ESI) calcd. for $C_{218}H_{376}F_6N_8O_{18}S_2Zn$: $[M-2OTf]^{2+}$ 1669.41691;

found: 1669.41370. Anal. Calcd. for $C_{218}H_{376}F_6N_8O_{18}S_2Zn$: C, 71.92; H, 10.41; N, 3.08; found: C, 72.07; H, 10.46; N, 3.05.

N-[3,4,5-tris(tetradecyloxy)benzyl]-bridged calix[4]pyridine zinc tetrafluroborate (1·Zn(BF_4)$_2$): white solid (84 mg, yield: 79%); IR (KBr, cm^{-1}) ν 2921, 2852, 1592, 1467, 1437, 1331, 1222, 1116; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.64 (t, 4H, *J* = 8.0 Hz), 6.91 (d, 8H, *J* = 8.0 Hz), 6.49 (s, 8H), 5.02 (s, 8H), 3.89 (t, 8H, *J* = 6.2 Hz), 3.74 (t, 16H, *J* = 5.4 Hz), 1.75 - 1.60 (m, 24H), 1.52 - 1.17 (m, 264H), 0.88 (t, 36H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 155.4, 153.6, 142.2, 137.3, 130.6, 111.4, 105.7, 73.5, 69.1, 58.0, 32.1, 30.6, 29.95, 29.89, 29.8, 29.6, 26.43, 26.35, 22.9, 14.3. HRMS (FTMS - ESI) calcd. for $C_{216}H_{376}B_2F_8N_8O_{12}Zn$: $[M-2BF_4]^{2+}$1669.41691; found: 1669.41553. Anal. Calcd. for $C_{216}H_{376}B_2F_8N_8O_{12}Zn$: C, 73.78; H, 10.78; N, 3.19; found: C, 74.16; H, 10.83; N, 3.26.

N-[3,4,5-tris(tetradecyloxy)benzyl]-bridged calix[4]pyridine zinc chloride (1·$ZnCl_2$): white solid (99 mg, yield: 97%); IR (KBr, cm^{-1}) ν 2922, 2852, 1592, 1467, 1439, 1380, 1332, 1222, 1115; ^1H NMR(400 MHz, $CDCl_2CDCl_2$, 140 ºC) δ 7.77 (t, 4H, *J* = 7.8Hz), 6.98 (d, 8H, *J* = 8.0Hz), 6.59 (br, 8H), 4.99 (s, 8H), 4.93 (t, 8H, *J* = 6.4 Hz), 3.80 (s, 16H), 1.79 - 1.63 (m, 24H), 1.52 - 1.27 (m, 264H), 0.94 (m, 36H); ^{13}C NMR (100 MHz, $CDCl_2CDCl_2$, 140 ºC) δ 155.9, 153.8, 142.8, 139.2, 129.8, 111.8, 108.0, 73.6, 70.1, 57.7, , 31.9, 30.6, 29.8, 29.72, 29.66, 29.6, 29.3, 26.34, 26.28, 22.6, 14.9. HRMS (FTMS - ESI) calcd. for $C_{216}H_{376}Cl_2N_8O_{12}Zn$: $[M-2Cl]^{2+}$ 1669.41691; found: 1669.41425.

3. Phase behaviors of azacalix[4]pyridine derivative 1

During cooling and subsequently heating at a rate faster than 2 °C/min, two exothermic processes are detected upon cooling. The first transition takes place at around 42 °C, while the second one appears at around 23 °C. Upon heating, two endothermic processes observed possess the onset temperatures very close to that of cooling, implying an enantiotropic phase transition behavior. However, when as much a slow heating rate as 0.5 °C/min was applied, a distinct exothermic process was observed right after the endothermic process at around 50 °C. This outcome reflects a “melt/recrystallization” process in which a new ordered phase with a higher melting temperature at *ca.* 70 °C was generated. After the "recrystallized" **1** was cooled to low temperatures, re-heating the samples only gave the isotropic transition at *ca.* 70 °C, indicating that the phase structure resulted from "melt/recrystallization" is the most stable in an entire low temperature region (Fig. S1).

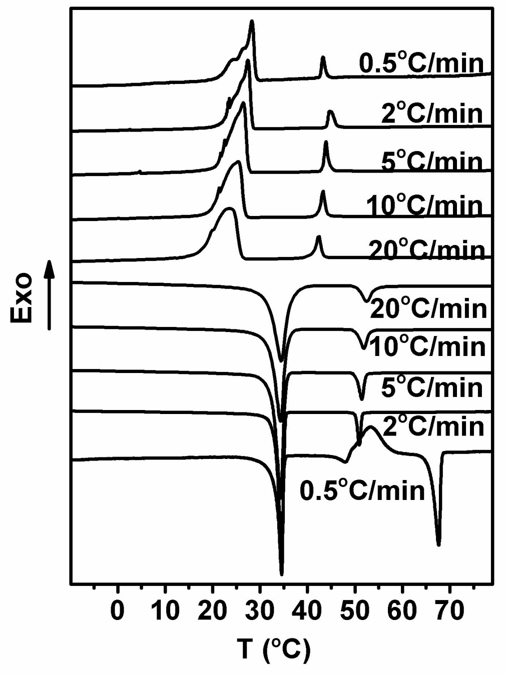


Fig. S1 DSC traces of 1 during heating and cooling cycles with different rates (0.5-20 °C/min).

Table S1 Diffractions of the Col$_h$ of **1**

hkl	**100**	**110**	**200**	**210**	**001**
d_{exp}^{a}**/nm**	3.09	1.79	1.55	1.17	0.63
d_{cal}^{b}**/nm**	3.09	1.78	1.55	1.17	0.63

[a] experimental value observed in Fig. 3c. [b] calculated value based on $a = b = 3.57$ nm and $c = 0.63$ nm.

Table S2 Diffractions of the Cr of **1**

hkl	**110**	**003**	**200**	**020**	**004**	**220**	**400**	**040**	**600**
d_{exp}^{a}/nm	4.30	3.55	3.51	2.73	2.68	2.17	1.75	1.36	1.16
d_{cal}^{b}/nm	4.31	3.54	3.51	2.73	2.67	2.16	1.76	1.37	1.17

hkl	**350**	**060**	**800**	**080**	**0018**	**4018**	**880**	**8018**
d_{exp}^{a}/nm	1.00	0.89	0.86	0.67	0.59	0.56	0.53	0.49
d_{cal}^{b}/nm	0.99	0.91	0.88	0.68	0.59	0.55	0.54	0.49

[a] experimental value observed in Fig. 3d. [b] calculated value based on a = 7.02 nm, b = 5.46 nm and c = 10.62 nm.

Computer simulation

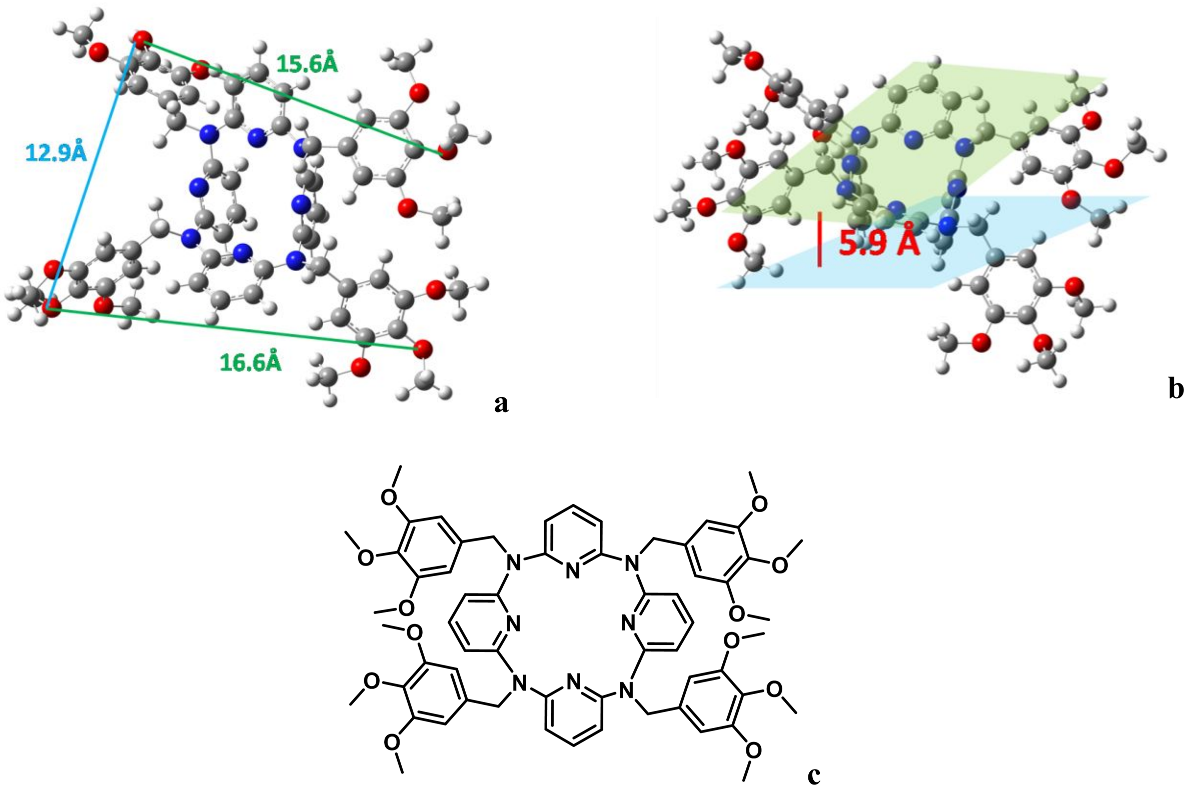


Fig. S2 (a) Top view, (b) side view of optimized structure and (c) molecular structure of N-(3,4,5-tris(methoxy)benzyl)-bridged calix[4]pyridine (at rb3lyp/3-21g* level).

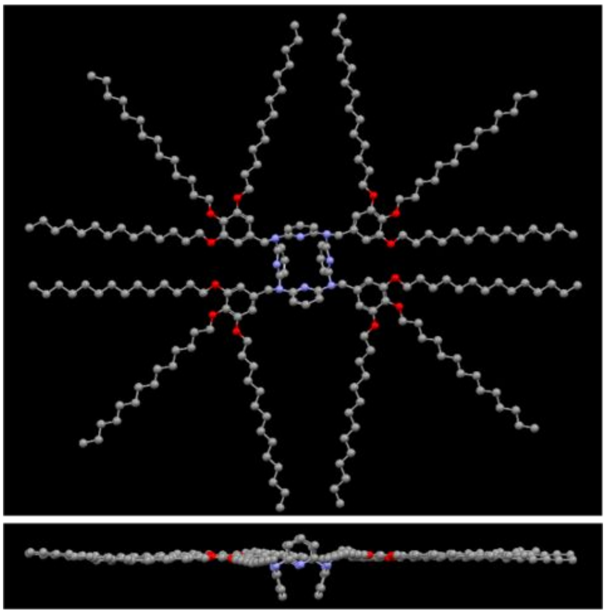


Fig. S3 Top view and side view of optimized structure of **1** (forcefield: COMPASS).

4. Phase behaviors of coordination complexes of 1 with zinc salts

DSC traces

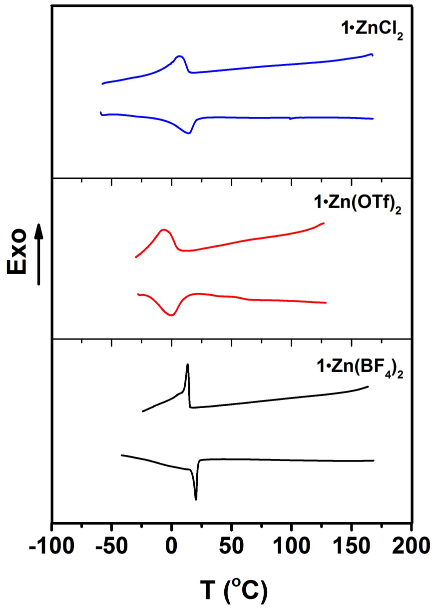


Fig. S4 DSC traces of **1**·ZnCl$_2$, **1**·Zn(BF$_4$)$_2$,(c) **1**·Zn(OTf)$_2$ and **1**·ZnCl$_2$ (from bottom to top) measured upon cooling and subsequently heating at a rate of 10 °C/min.

1D XRD experimental results

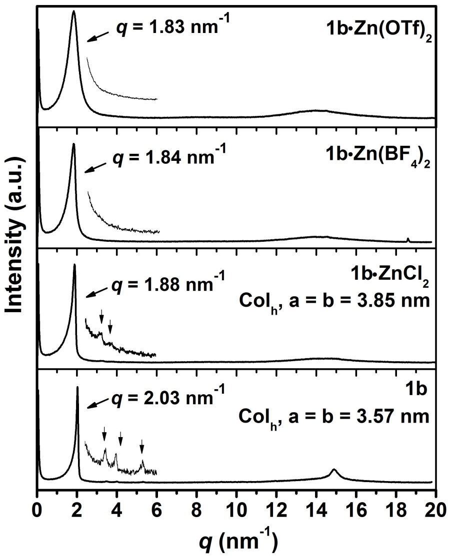


Fig. S5 1D XRD profiles of **1** and its complexes with zinc salts at room temperature. The samples were quenched from molten state.

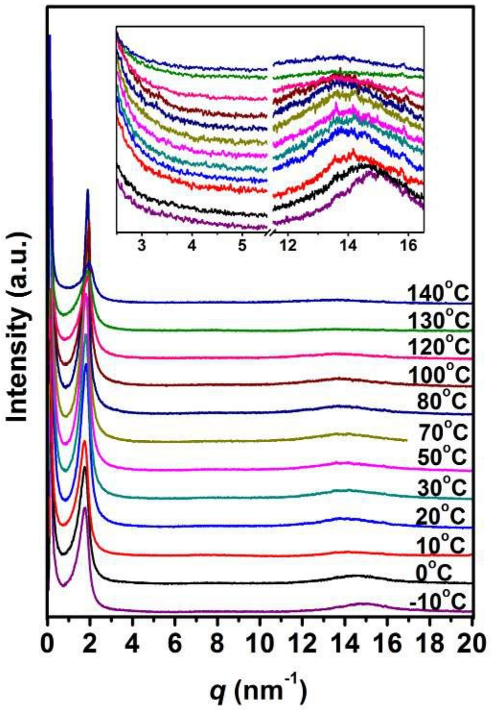


Fig. S6 Set of 1D XRD profiles of **1**·Zn(BF_4)$_2$ recorded upon heating.

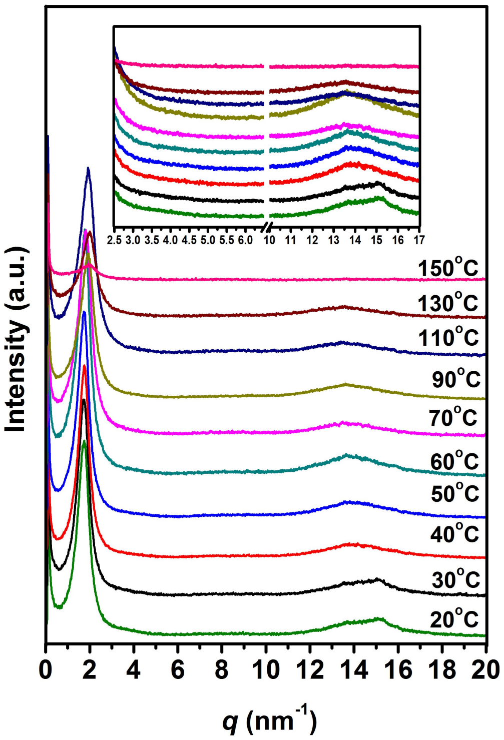
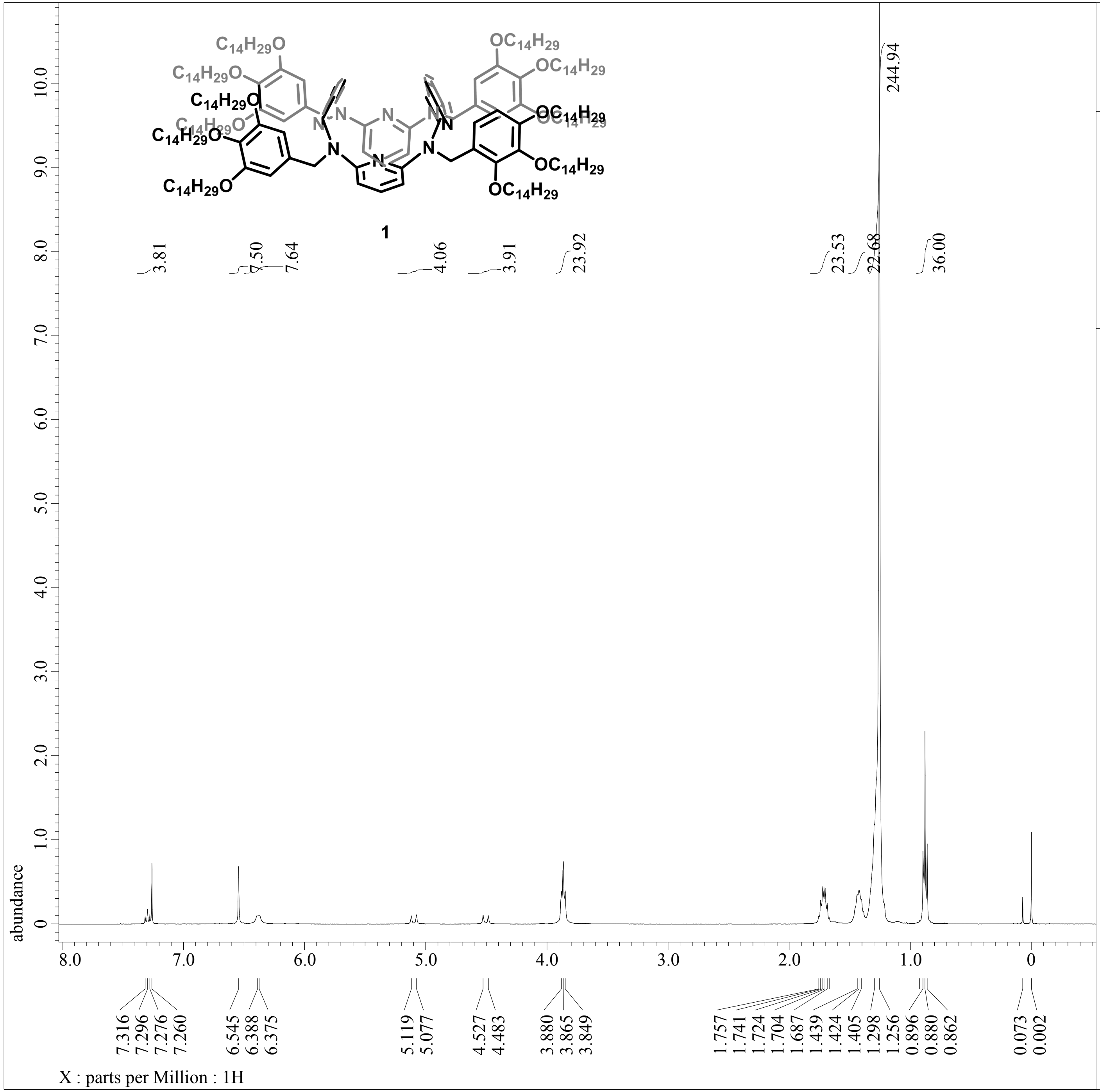


Fig. S7 Set of 1D XRD profiles of **1**·Zn(OTf)$_2$ recorded upon heating.

5. References

1. E.-X. Zhang, D.-X. Wang, Z.-T. Huang and M.-X. Wang, *J. Org. Chem.,* 2009, **74**, 8595-8603.
2. T. Ichikawa, M. Yoshio, A. Hamasaki, S. Taguchi, F. Liu, X.-B. Zeng, G. Ungar, H. Ohno, and T. Kato, *J. Am. Chem. Soc.,* **2012**, *134*, 2634-2643.

6. Copies of ^{1}H and ^{13}C NMR Spectra of 1 and 1·ZnA$_2$ (pg. 11-18)



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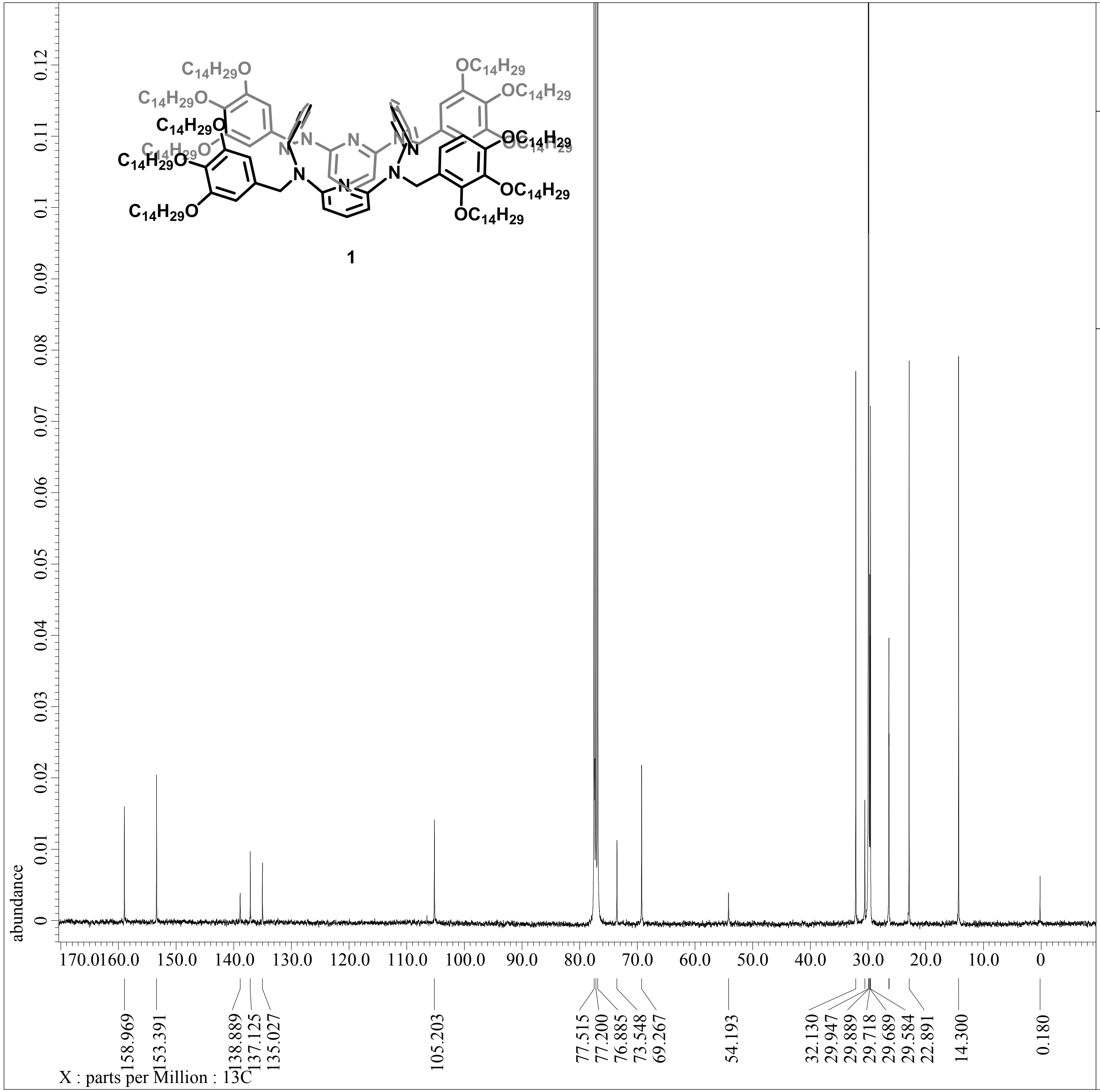

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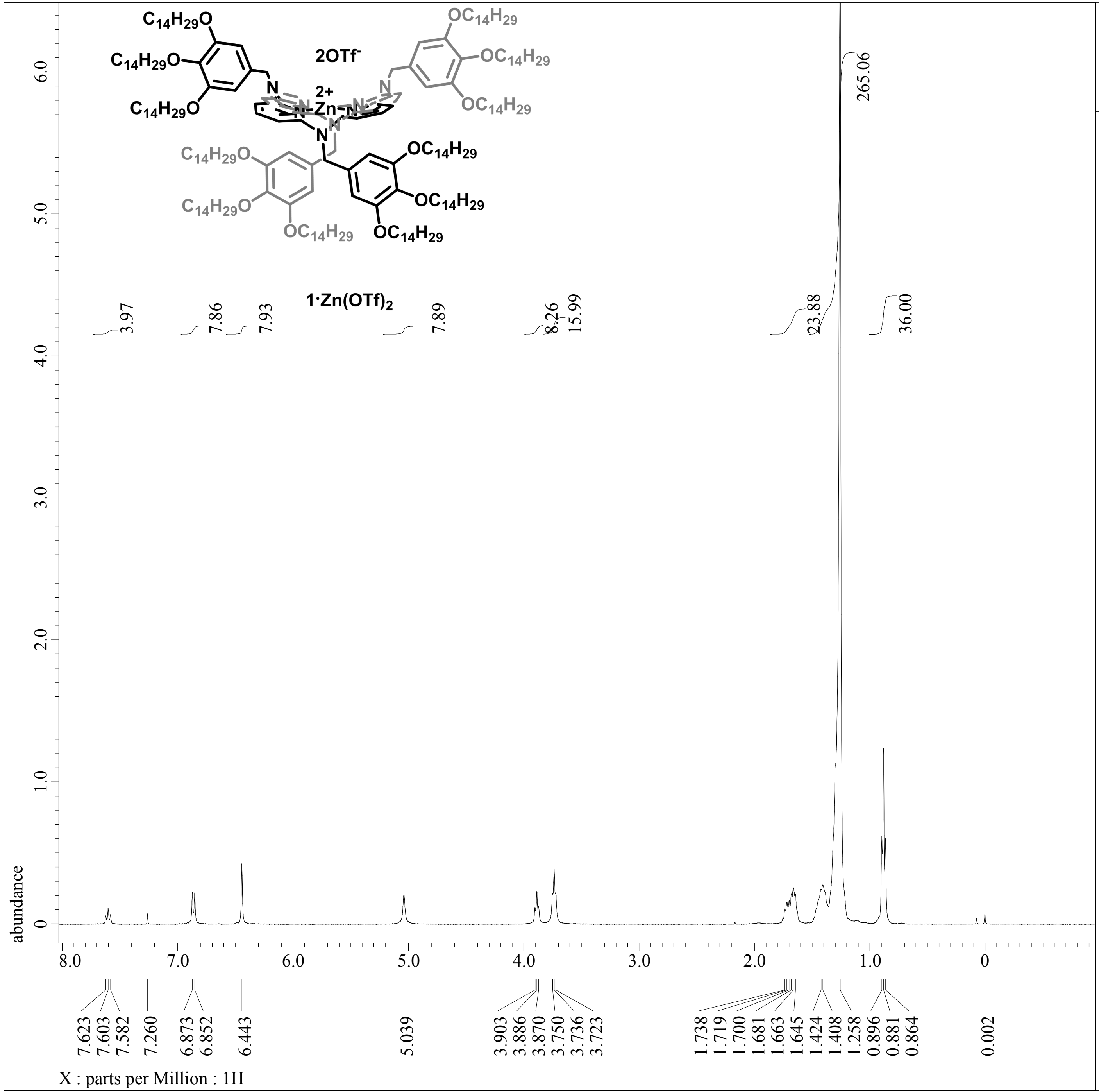

$1\cdot Zn(OTf)_2$

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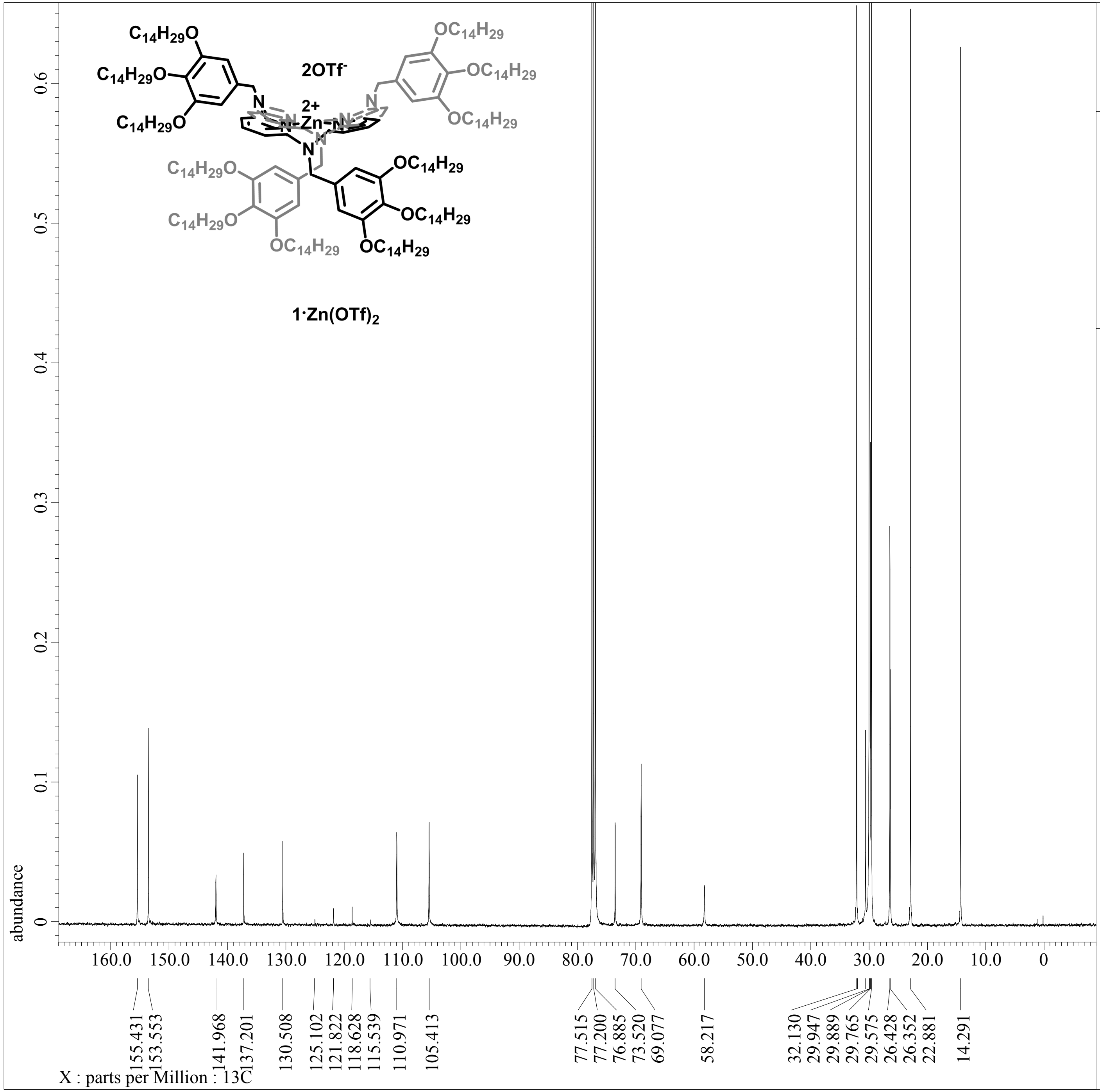

$1\cdot Zn(OTf)_2$

JEOL RESONANCE

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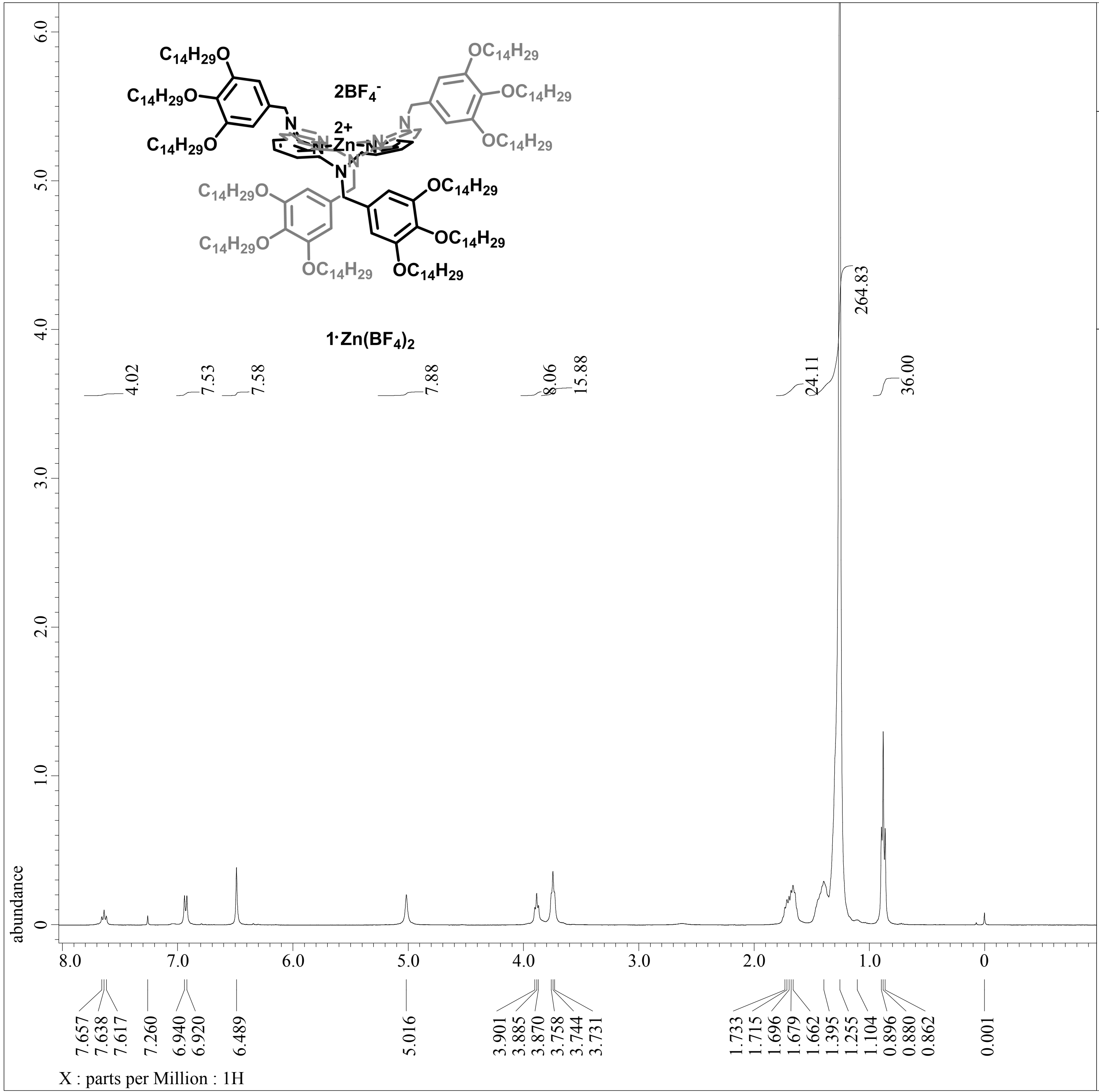
Revision_Time     =  3-JAN-2014 01:06:58
Current_Time      =  3-JAN-2014 01:07:29

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 25444
Total_Scans       = 25444

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 23.6[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



$1\cdot Zn(BF_4)_2$

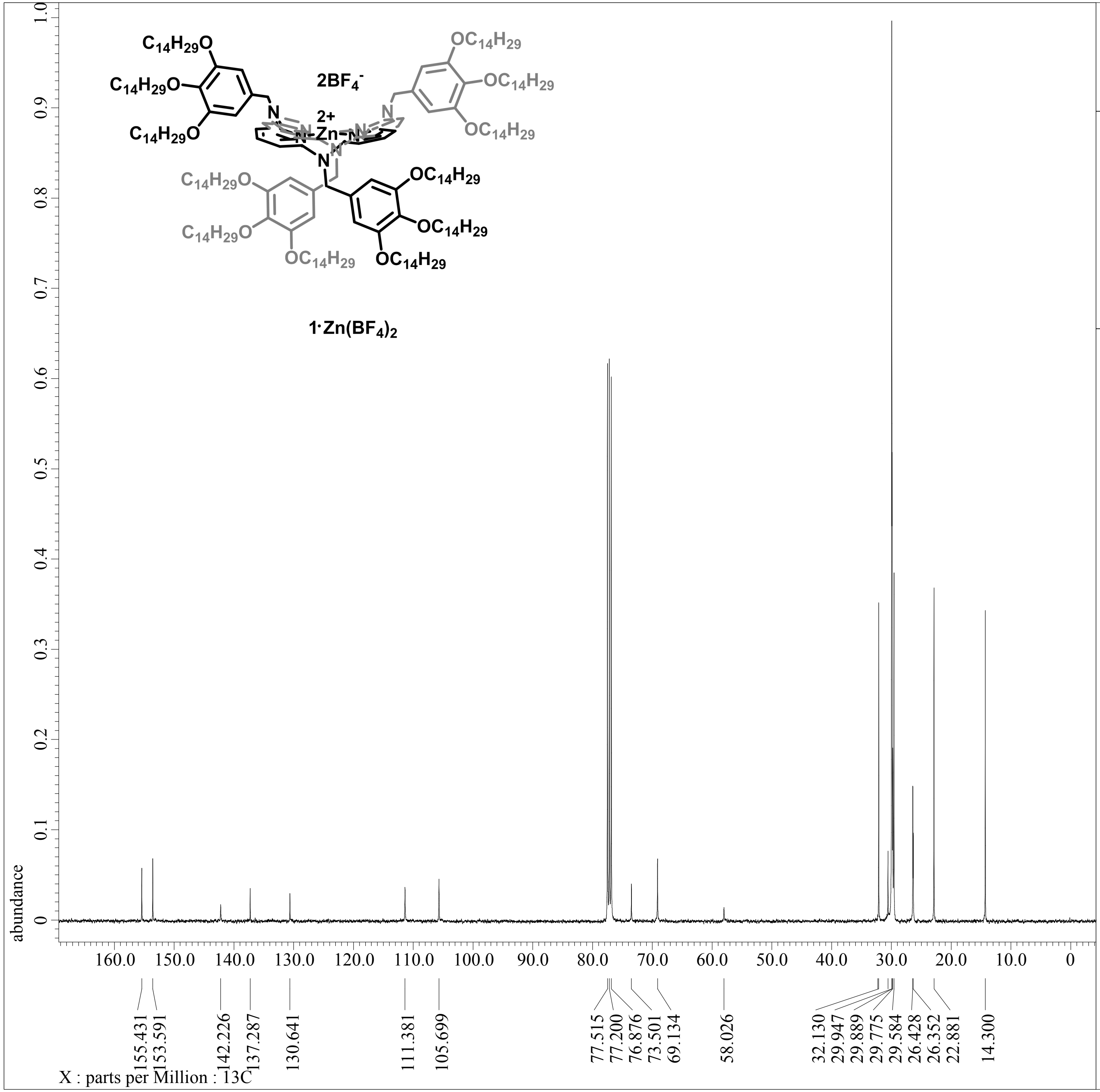
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = F:\far\NMR\LC\far (1)\far\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#703616
Solvent           = CHLOROFORM-D
Creation_Time     = 30-DEC-2013 18:51:48
Revision_Time     = 30-DEC-2013 19:49:48
Current_Time      =  3-JAN-2014 01:00:52

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 22.2[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


1·Zn(BF4)2

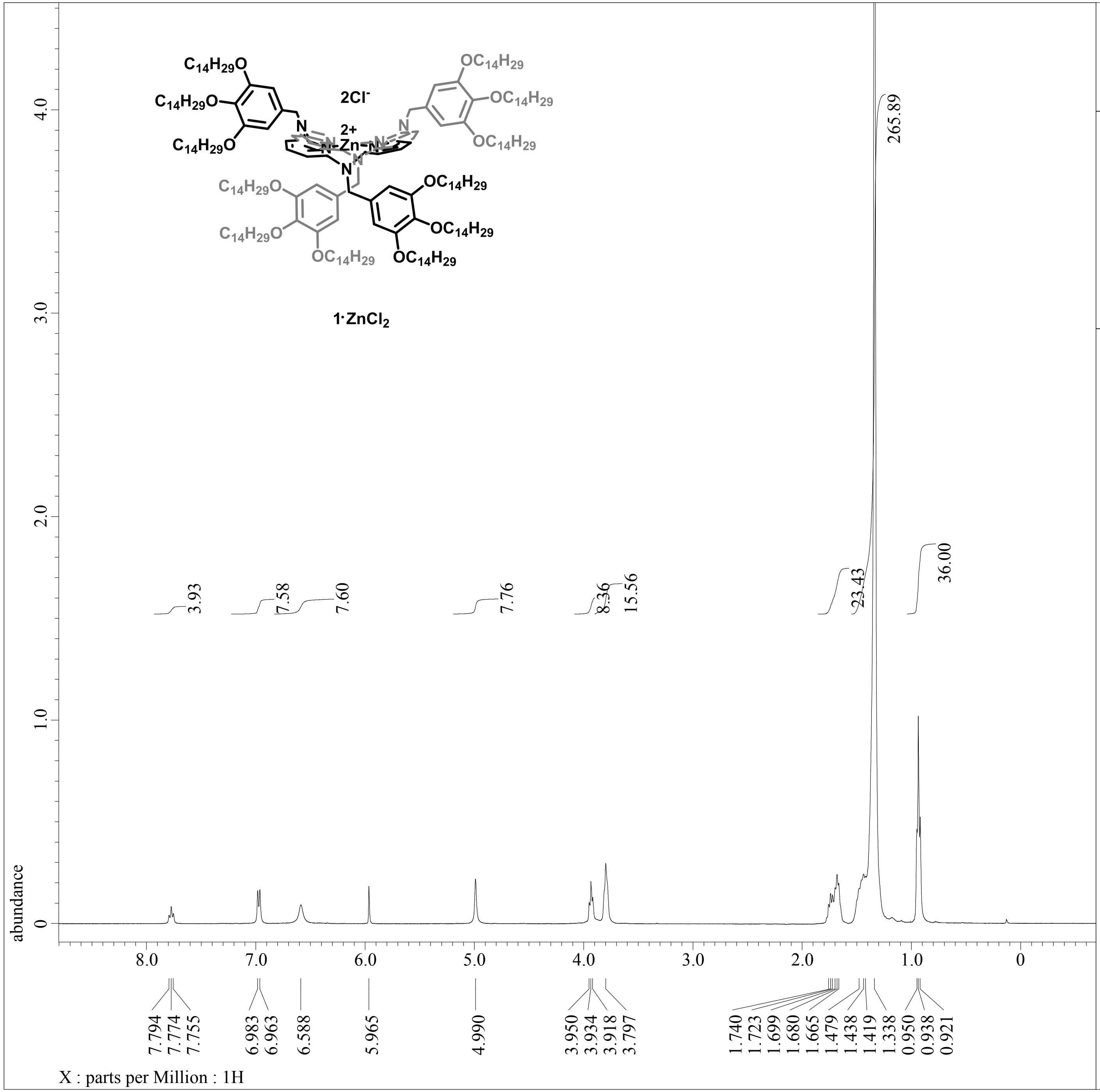
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename           = F:\far\NMR\LC\far (1)\far\
Author             = delta
Experiment         = single_pulse_dec
Sample_Id          = S#715597
Solvent            = CHLOROFORM-D
Creation_Time      = 30-DEC-2013 21:30:59
Revision_Time      =  3-JAN-2014 01:02:41
Current_Time       =  3-JAN-2014 01:02:51

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = 13C
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECX 400
Spectrometer       = JNM-ECX400

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
Irr_Domain         = 1H
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 2732
Total_Scans        = 2732

Relaxation_Delay   = 2[s]
Recvr_Gain         = 54
Temp_Get           = 22.7[dC]
X_90_Width         = 8.75[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 7.8[dB]
X_Pulse            = 2.91666667[us]
Irr_Atn_Dec        = 23.98[dB]
Irr_Atn_Noe        = 23.98[dB]
Irr_Noise          = WALTZ
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```



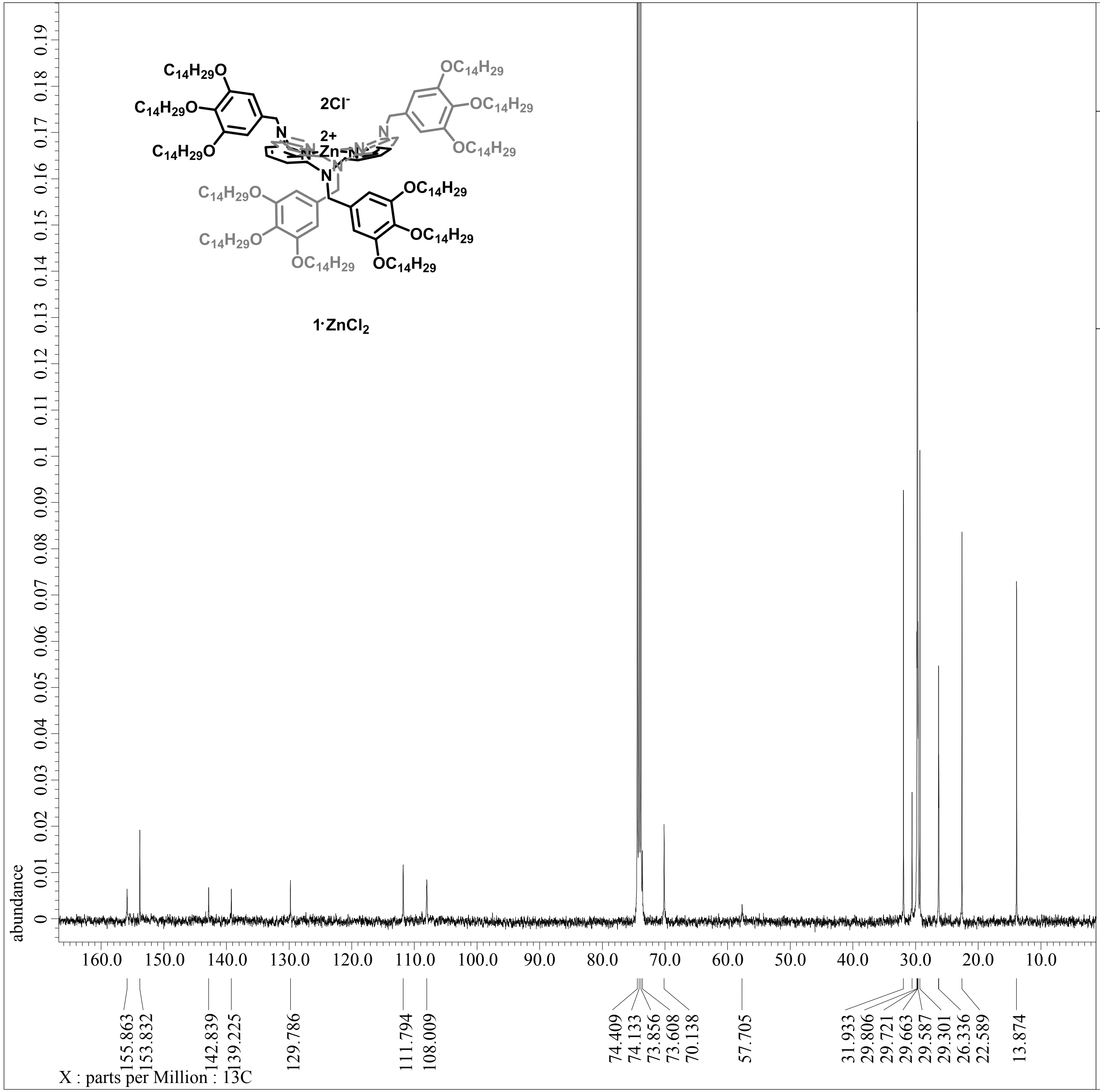
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = F:\far\NMR\LC\far-4-4Bn14-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#337815
Solvent           = TETRACHLOROETHAN
Creation_Time     = 12-JAN-2014 08:42:52
Revision_Time     = 12-JAN-2014 09:42:36
Current_Time      = 12-JAN-2014 18:35:22

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 140[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename           = F:\far\NMR\LC\far-4-4Bn14-
Author             = delta
Experiment         = single_pulse_dec
Sample_Id          = S#339980
Solvent            = TETRACHLOROETHAN
Creation_Time      = 12-JAN-2014 11:25:29
Revision_Time      = 12-JAN-2014 19:20:12
Current_Time       = 12-JAN-2014 19:20:32

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = 13C
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECX 400
Spectrometer       = JNM-ECX400

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
Irr_Domain         = 1H
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 3142
Total_Scans        = 3142

Relaxation_Delay   = 2[s]
Recvr_Gain         = 50
Temp_Get           = 140[dC]
X_90_Width         = 8.75[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 7.8[dB]
X_Pulse            = 2.91666667[us]
Irr_Atn_Dec        = 23.98[dB]
Irr_Atn_Noe        = 23.98[dB]
Irr_Noise          = WALTZ
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```