Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

ESI

Synthesis of Hydroxylated Azacalix[1]arene[3]pyridines from Hydrolysis of High Valent Arylcopper Complexes and Conversion to A Double Azacalix[1]arene[3]pyridine Host Molecule

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1. General Information

The ^{1}H and ^{13}C spectra were recorded on a 400 MHz spectrometer. Chemical shifts are reported in ppm with either tetramethylsilane or the residual solvent resonance used as an internal standard. Infrared spectra were recorded using a FT-IR spectrometer with KBr discs in the 4000-400 cm^{-1} region. Melting points are uncorrected. Flash column chromatography was performed on silica gel (100-200 mesh or 200-300 mesh).

2. General procedure for the reaction of arylcopper(II) complexes 2a-f with KOH: synthesis of hydroxylated azacalix[1]arene[3]pyridines 4a-f.

To a solution of arylcopper(II) complexes **2a-f** (0.2 mmol) in DMSO (15 mL) was added an aqueous solution of KOH (1.6 M, 0.5 mL) while stirring. After reaction at ambient temperature for 1 h, the mixture was poured into a mixed solution of a saturated aqueous solution of EDTA (50 mL) and brine (50 mL), and extracted with DCM (3 × 75 mL). The combined organic layer was washed again with brine (2 × 100 mL), dried with anhydrous Na_2SO_4. After removal of solvent, the residue was chromatographed on a silica gel column (100-200 mesh) using a mixture of petroleum ether and ethyl acetate (5 : 1) as mobile phase to give pure products **4a-f** in 92% - 99% yields.

4a (87 mg, 99%): white solid; mp 206℃, (lit. 207-208℃[1]); ^{1}H NMR (400 MHz, $CDCl_3$) 7.44 (t, *J* = 8.0 Hz, 2H)， 7.17 (t, *J* = 7.7 Hz, 1H), 6.79 (d, *J* = 7.4 Hz, 2H), 6.70-6.67 (m, 1H), 6.57 (d, *J* = 7.6 Hz, 2H), 6.12-6.08 (m, 4H), 3.26 (s, 6H), 3.14 (s, 6H). R_f = 0.29 (Petroleum Ether (60 – 90 °C) : Ethyl Acetate = 3 : 1).

4b (83mg, 92%): white solid; mp 183℃, (lit. 183-185℃[2]); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 1H), 6.59-6.57 (m, 4H), 6.11-6.06 (m, 4H), 5.39 (s, 1H), 3.25 (s, 6H), 3.12 (s, 6H), 2.19 (s, 3H); IR (KBr) ν 2895, 1579, 1472, 1418, 1156, 769 cm^{-1}. R_f = 0.34 (Petroleum Ether (60 – 90 °C) : Ethyl Acetate = 3 : 1).

4c (87mg, 93%): white solid; mp 182℃,(lit. 181-182℃[2]); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (t, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 7.7 Hz, 1H), 6.59 (d, *J* = 7.7 Hz, 2H), 6.38 (s, 2H), 6.12-6.07 (m, 4H), 5.03 (br, 1H), 3.71 (s, 3H), 3.25 (s, 6H), 3.13 (s, 6H); IR(KBr)ν 2895, 1579, 1471, 1360, 1155, 769 cm^{-1}. R_f = 0.32 (Petroleum Ether (60 – 90 °C) : Ethyl Acetate = 3 : 1).

4d (92mg, 99%): white solid; mp 286℃,(lit. 286-287℃[2]); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.41 (m, 3H), 7.11 (s, 2H), 6.65 (d, *J* = 7.7 Hz, 2H), 6.16-6.10 (m, 4H), 3.25 (s, 6H), 3.12 (s, 6H); IR(KBr)ν 2895, 2222, 1577, 1473, 1420, 1156 cm^{-1}. R_f = 0.33 (Petroleum Ether (60 – 90 °C) : Ethyl Acetate = 3 : 1).

4e (91mg, 96%): white solid; mp 201℃, (lit. 200-201℃[2]); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.39 (m, 3H), 6.77 (s, 2H), 6.63 (d, *J* = 7.7 Hz, 2H), 6.12 (d, *J* = 8.0 Hz, 2H), 6.08 (d, *J* = 8.0 Hz, 2H), 3.26 (s, 6H), 3.11 (s, 6H); IR(KBr)ν

1893, 1576, 1472, 1365, 1155, 769 cm^{-1}. R_f = 0.34 (Petroleum Ether (60 – 90 ^{o}C) : Ethyl Acetate = 3 : 1).

4f (90mg, 95%): white solid; mp 186℃ , (lit. 184-185℃ [2]); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.40 (m, 2H), 7.22 (t, *J* = 7.7 Hz, 1H), 6.73 (s, 2H), 6.65 (d, *J* = 8.0 Hz, 1H), 6.58 (d, *J* = 8.0 Hz, 1H), 6.13-6.05 (m, 4H), 5.57(br, 0.5H), 3.25 (m, 6H), 3.12-3.11 (m, 6H); IR(KBr)ν 1577, 1473, 1277, 1153, 901, 768 cm^{-1}. R_f = 0.35 (Petroleum Ether (60 – 90 ^{o}C) : Ethyl Acetate = 3 : 1).

3. Synthesis of 5

Method A. To a mixture of **4a** (87 mg, 0.2 mmol) and $CuCl_2{\cdot}2H_2O$ (6.8 mg, 0.04 mmol) in CH_3CN (10 mL) was added with TMEDA (12 L, 0.08 mmol). After reaction at room temperature for 24 h, the resulting mixture was poured into a mixed solution of a saturated aqueous solution of EDTA (50 mL) and brine (50 mL), and extracted with DCM (3 × 75 mL). The combined organic layer was washed consecutively with an aqueous solution of sodium hydrosulfite (100 mL) and brine (2 × 100 mL), dried with anhydrous Na_2SO_4. After removal of solvent, the residue was chromatographed on a silica gel column (100- 200 mesh) using a mixture of petroleum ether and ethyl acetate (1 : 2) as mobile phase to give pure product **5** (61 mg, 70%).

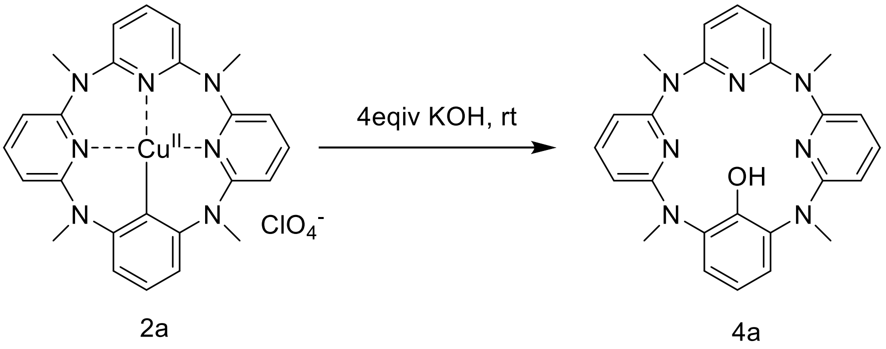
Method B. To a solution of arylcopper(II) complex **2a** (117 mg, 0.2 mmol) in DMSO (15 mL) was added an aqueous solution of KOH (1.6 M, 0.5 mL) while stirring. After reaction at ambient temperature for 1 h, an aqueous solution of HCl (2 M, 0.5 mL), CH_3CN (30 mL) and TMEDA (120 L, 0.8 mmol) was added consecutively. The resulting mixture was kept stirring for another 24 h, and then was poured into a mixed solution of a saturated aqueous solution of EDTA (100 mL) and brine (100 mL), and extracted with DCM (3 × 125 mL). The combined organic layer was washed with an aqueous solution of sodium hydrosulfite (100 mL) and then with brine (2 × 100 mL), dried with anhydrous Na_2SO_4. After removal of solvent, the residue was chromatographed on a silica gel column (100- 200 mesh) using a mixture of petroleum ether and ethyl acetate (1 : 2) as mobile phase to give pure product **5** (60 mg, 68%) as while solid: mp >300 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, *J* = 8.0 Hz, 4H), 7.00 (d, *J* = 8.0 Hz, 2H), 6.99 (s, 4H), 6.53 (d, *J* = 8.6 Hz, 4H), 6.13 (d, *J* = 8.6 Hz, 4H), 5.55 (s, 2H), 3.25 (s, 12H), 3.21 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 159.0, 148.8, 139.2, 137.4, 135.7, 132.5, 124.3, 119.7, 96.5, 95.6, 38.5, 36.4; IR (KBr) ν 3454, 2901, 1575, 1472, 1361, 1154 cm^{-1}. HRMS (ESI-ion trap) calcd for $C_{50}H_{48}N_{14}O_2$: $[M+H]^+$ 877.4163. Found: 877.4140. Elemental analysis calcd (%) for $C_{50}H_{48}N_{14}O_2{\cdot}H_2O$: C, 67.02; H, 5.74; N, 21.88. Found: C, 67.15; H, 5.60; N, 21.79. R_f = 0.28 (Petroleum Ether (60 – 90 ^{o}C) : Ethyl Acetate = 1 : 1). A high quality single crystal suitable for X-ray diffraction analysis was obtained by the diffusion of vapor of a mixture of *n*-pentane and *n*-hexane into a solution of **5** in DCM at room temperature.

4. Preparation of Single Crystals of Host-Guest Complexes.

Single crystals of the complexes between **5** and propane-1,3-diamine and butane-1,4-diamine were cultivated by evaporation of the solvent of a solution of **5** and an equimolar 1,3-diamine and butane-1,4-diamine in a mixture of DCM and *n*-heptane.

5. Solvent Effect on the Hydrolysis of Arylcopper(II) Complex 2a

Table S1. Solvent Effect on the Hydrolysis of Arylcopper(II) Complex 2a[a]

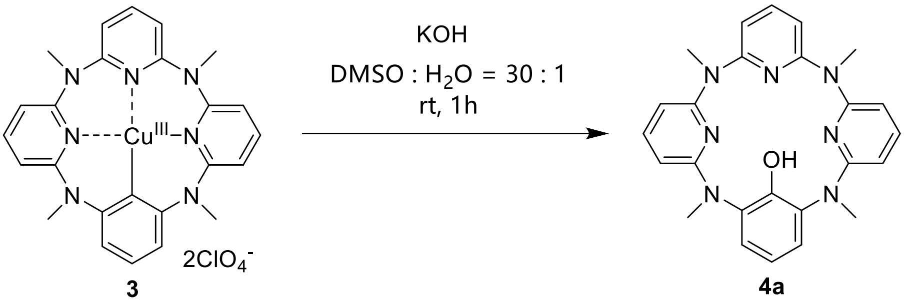


entry	Solvent	time	Yield of 4a (%)
1	DMSO	30 min	>99
2	DMF	30 min	95
3	Methanol : DCM = 1 : 1	120 h	N.R.[b]
4	Acetonitrile	120 h	N.R.[b]
5	sulfolane	120h	Trace

[a] The reaction was conducted by mixing a solution of arylcopper(II) complexes **2a** (0.2 mmol) in DMSO (15 mL) with an aqueous solution of KOH (1.6 M, 0.5 mL). [b] No reaction.

6. Reaction of Arylcopper(III) Complex 3 with KOH

Table S2. Reaction of Arylcopper(III) Complex 3 with KOH

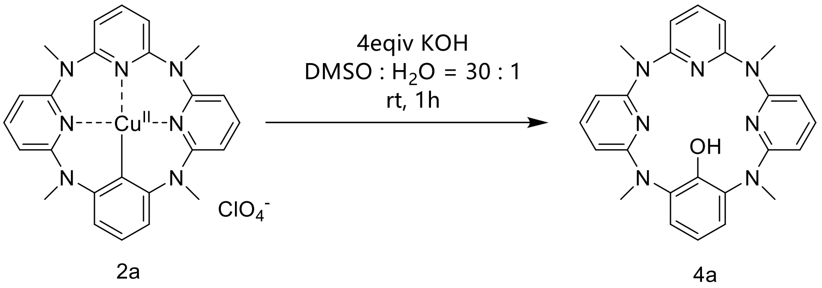


entry	KOH (equiv)	Yield 4 (%)
1	1	0
2	2	32
3	3	64
4	4	59-64[a]
5	10	58

[a] A few experiments were performed, and chemical yields were in the range of 59% - 64%.

7. Effect of Additive on the Reaction of Arylcopper(II) Complex 2a

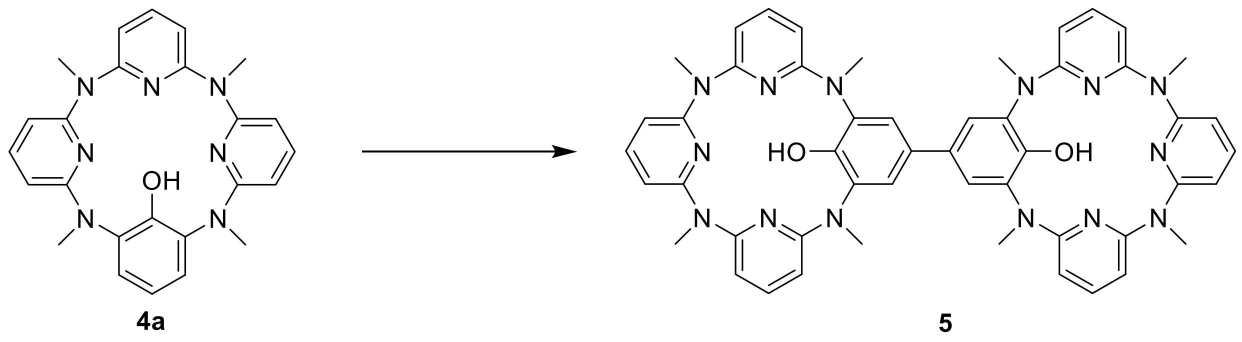
Table S3. Effect of Additive on the Reaction of Arylcopper(II) Complex 2a



entry	additive	Equiv	Yield of 4a (%)
1	-	-	>99
2	TEMPO	1	97
3	TEMPO	10	87
4	NHPI	2	96
5	H_2O_2	2	97

8. Screening of the Conditions for the Synthesis of 5

Table S4. Screening of the Conditions for the Synthesis of 5

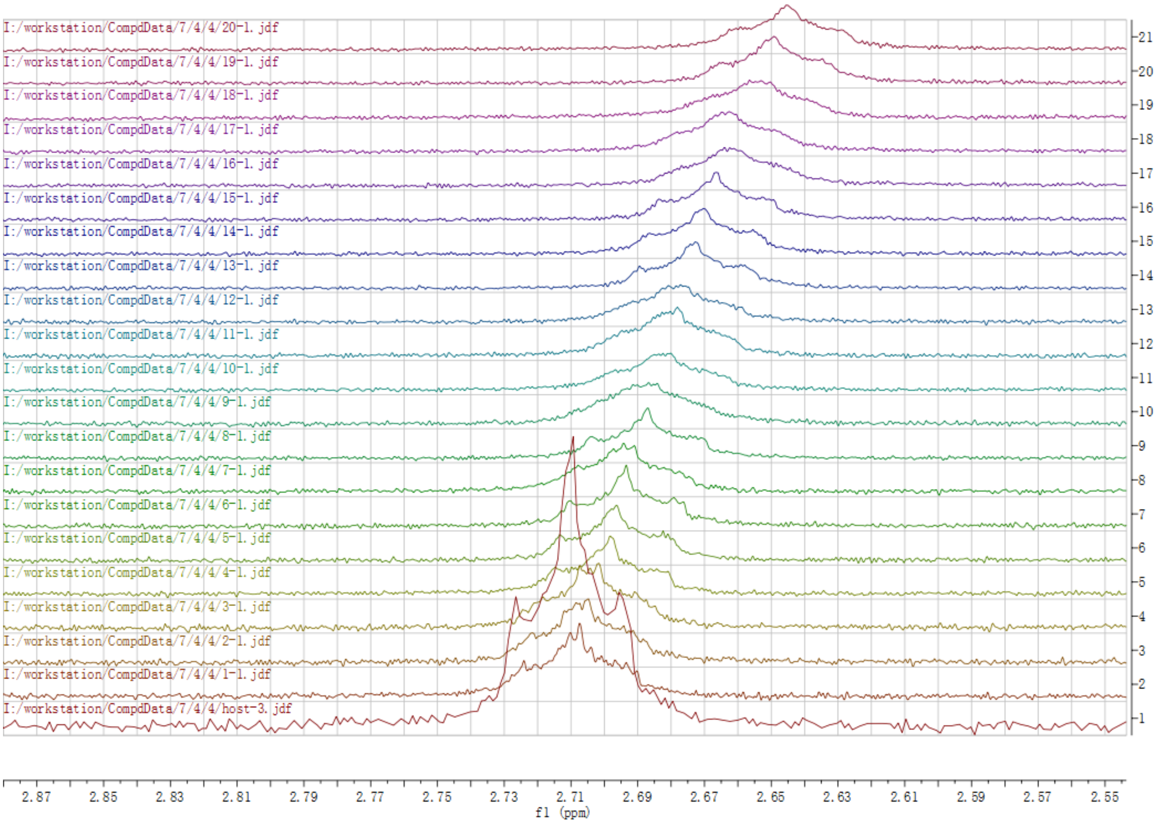


Entry	**Oxidant**	**Equiv of Oxidant**	**Ligand**[a]	**Solvent**	**Temperature**	**Time**	**Yield of 5(%)**
1	$FeCl_3$	2	-	Ethanol	rt	24h	N.R.[b]
2	$FeCl_3$	2	-	Ethanol	50°C	24h	N.R.[b]
3	$FeCl_3$	2	-	Ethanol	Reflux	24h	N.R.[b]
4	$FeCl_3$	2	1,10-Phenanthroline	Ethanol	Reflux	24h	N.R.[b]
5	$FeCl_3$	2	-	Acetonitrile	Reflux	24h	N.R.[b]
6	$FeCl_3$	4	-	Acetonitrile	Reflux	24h	N.R.[b]
7	$FeCl_3$	4	1,10-Phenanthroline	Acetonitrile	Reflux	24h	N.R.[b]
8	$CuCl_2$	2	TMDEA	DCE	Reflux	24h	P.M.[c]
9	$CuCl_2$	2	TMEDA	Ethanol	Reflux	24h	P.M.[c]
10	$CuCl_2$	2	TMEDA	Acetonitrile	Reflux	24h	P.M.[c]
11	$CuCl_2$	2	TMEDA	Acetonitrile	rt	24h	80
12	$CuCl_2$	1	TMEDA	Acetonitrile	rt	24h	73
13	$CuCl_2$	0.2	TMEDA	Acetonitrile	rt	24h	70

[a] The ratio of oxidant over ligand is 1 to 2. [b] No Reaction. [c] A mixture of very polar and unseparable by-products.

9. ^{1}H-NMR Titration of 5 with Butane-1,4-diamine

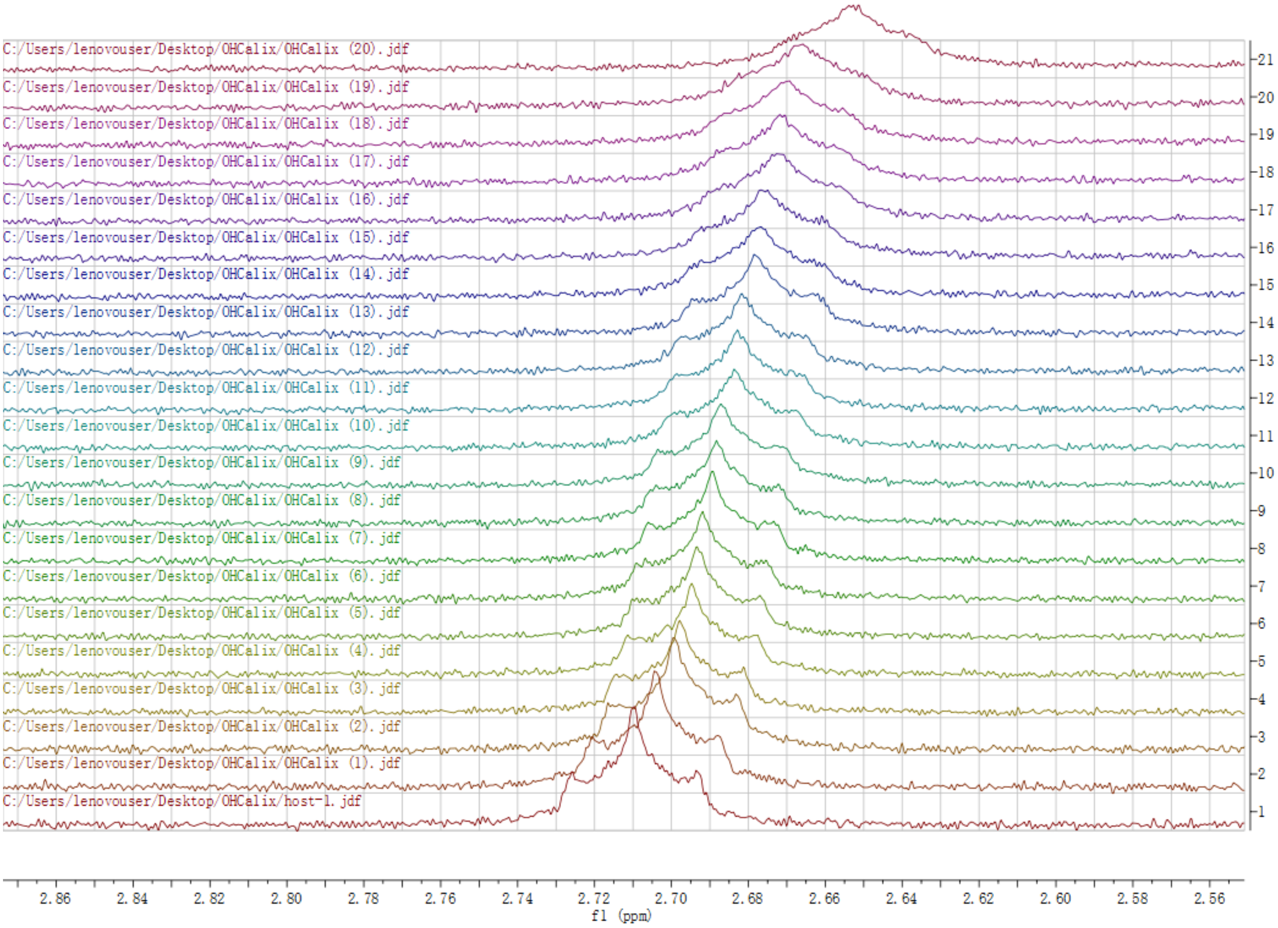
Figure S1. ^{1}H-NMR Titration of 5 with Butane-1,4-diamine



Condition: Host **5** (0 – 3 equiv) was added gradually into the $CDCl_3$ solution of butane-1,4-diamine (1.0 mM) at 20℃.

10. ^{1}H-NMR Titration of 4a with Butane-1,4-diamine

Figure S2. ^{1}H-NMR Titration of 4a with Butane-1,4-diamine



Condition: Host **4a** (0 – 6 equiv) was added gradually into the $CDCl_3$ solution of butane-1,4-diamine (1.0 mM) at 20 °C.

11. Reference

[1] B. Yao, D.-X. Wang, Z.-T. Huang and M.-X. Wang, *Chem. Commun.*, 2009, 2899.

[2] H. Zhang, L. Zhao, D.-X. Wang and M.-X. Wang, *Org. Lett.*, 2013, **15**, 3836.

12. Crystal data and structure refinement for X-ray molecular structures

Identification code	**5**	**5**·Propane-1,3-diamine (**5**·C3H10N2)	**5**·Butane-1,4-diamine (**5**·C4H12N2)
Empirical formula	C50 H52 N14 O4	C54.83 H62.12 Cl1.76 N16.83 O2	C58H62N12O2
Formula weight	913.05	1051.26	965.18
Temperature	173.1500 K	173.1500 K	150.00(10) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Monoclinic	Monoclinic	triclinic
Space group	P 1 21/c 1	C 1 2/c 1	P-1
Unit cell dimensions	a = 9.842(2) Å b = 21.609(4) Å c = 25.234(5) Å α= 90°. β= 91.32(3)°. γ = 90°.	a = 28.402(6) Å b = 9.816(2) Å c = 18.722(4) Å α= 90°. β= 90.70(3)°. γ = 90°.	a = 9.6636(3) Å b = 15.1098(6) Å c = 18.1939(7) Å α= 69.768(4)°. β= 84.466(3)°. γ = 84.513(3)°.
Volume	5365.0(19) Å3	5219.2(18) Å3	2475.53(18)
Z	4	4	2
Density (calculated)	1.130 Mg/m3	1.338 Mg/m3	1.2948 Mg/m3
Absorption coefficient	0.075 mm-1	0.173 mm-1	0.084 mm-1
F(000)	1928	2219	1024.4
Crystal size	0.37 x 0.05 x 0.04 mm3	0.279 x 0.183 x 0.112 mm3	0.3 × 0.2 × 0.1
Theta range for data collection	1.614 to 25.199°.	1.434 to 27.500°.	6.56 to 58.9
Index ranges	-11<=h<=11, -25<=k<=25, -30<=l<=29	-36<=h<=22, -11<=k<=12, -24<=l<=24	-12 ≤ h ≤ 12, -19 ≤ k ≤ 20, -24 ≤ l ≤ 24

Reflections collected	37182	18221	21677
Independent reflections	9650 [R(int) = 0.0675]	5976 [R(int) = 0.0413]	11520 [Rint = 0.0259, Rsigma = 0.0499]
Completeness to theta = 25.199°	99.90%	99.60%	99.75%
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	multi-scan
Max. and min. transmission	1.0000 and 0.7660	1.00000 and 0.78949	1.00000 and 0.85030
Refinement method	Full-matrix least-squares on F2	Full-matrix least-squares on F2	Full-matrix least-squares on F2
Data / restraints / parameters	9650 / 0 / 623	5976 / 0 / 386	11520/0/665
Goodness-of-fit on F2	1.208	1.088	1.041
Final R indices [I>2sigma(I)]	R1 = 0.0942, wR2 = 0.2041	R1 = 0.0805, wR2 = 0.2196	R1 = 0.0575, wR2 = 0.1283
R indices (all data)	R1 = 0.1092, wR2 = 0.2128	R1 = 0.0870, wR2 = 0.2259	R1 = 0.0858, wR2 = 0.1452
Extinction coefficient	n/a	n/a	n/a
Largest diff. peak and hole	0.200 and -0.202 e.Å-3	1.921 and -0.489 e.Å-3	1.53 and -0.75 e. Å-3
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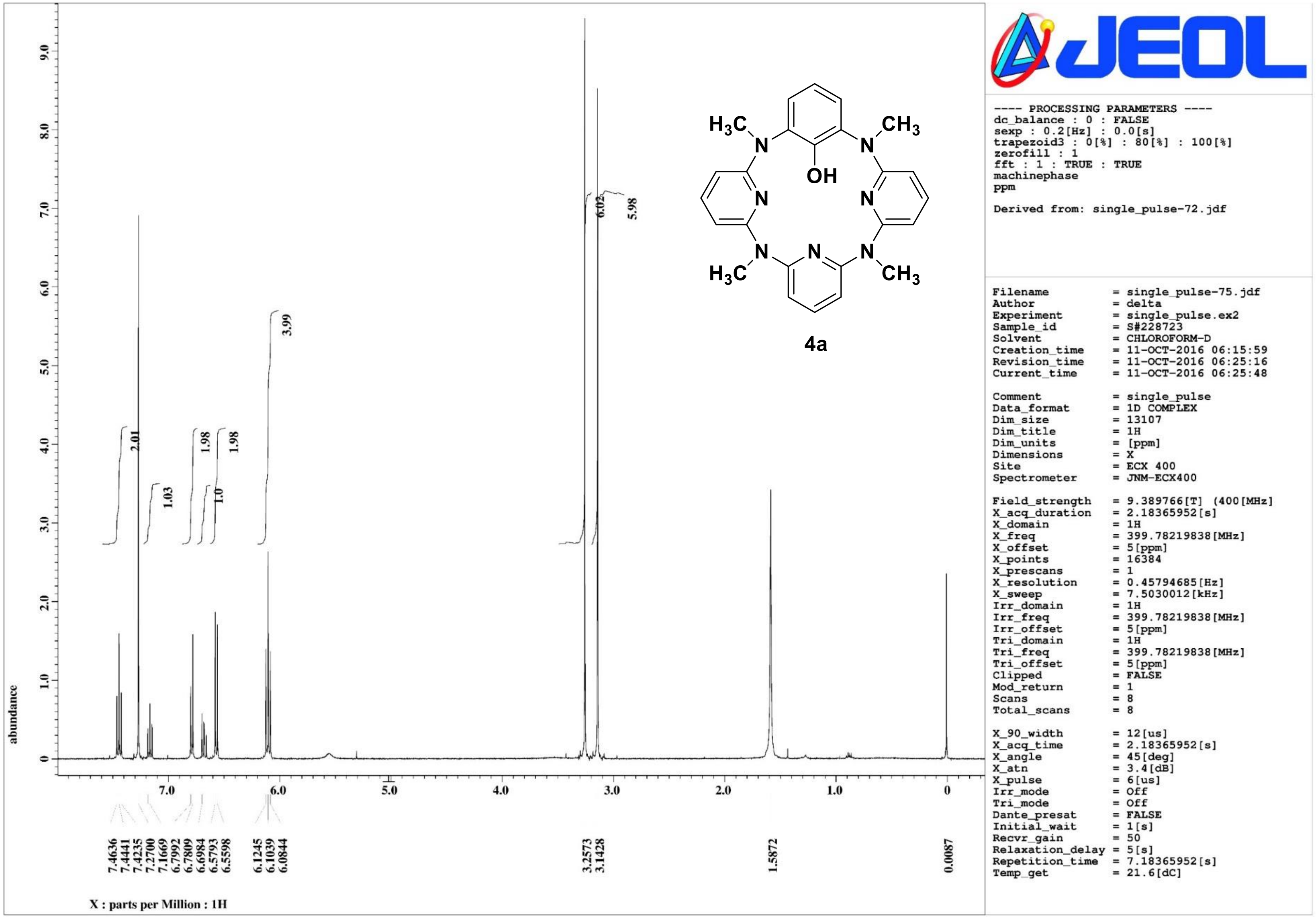
13. Copies of NMR Spectra



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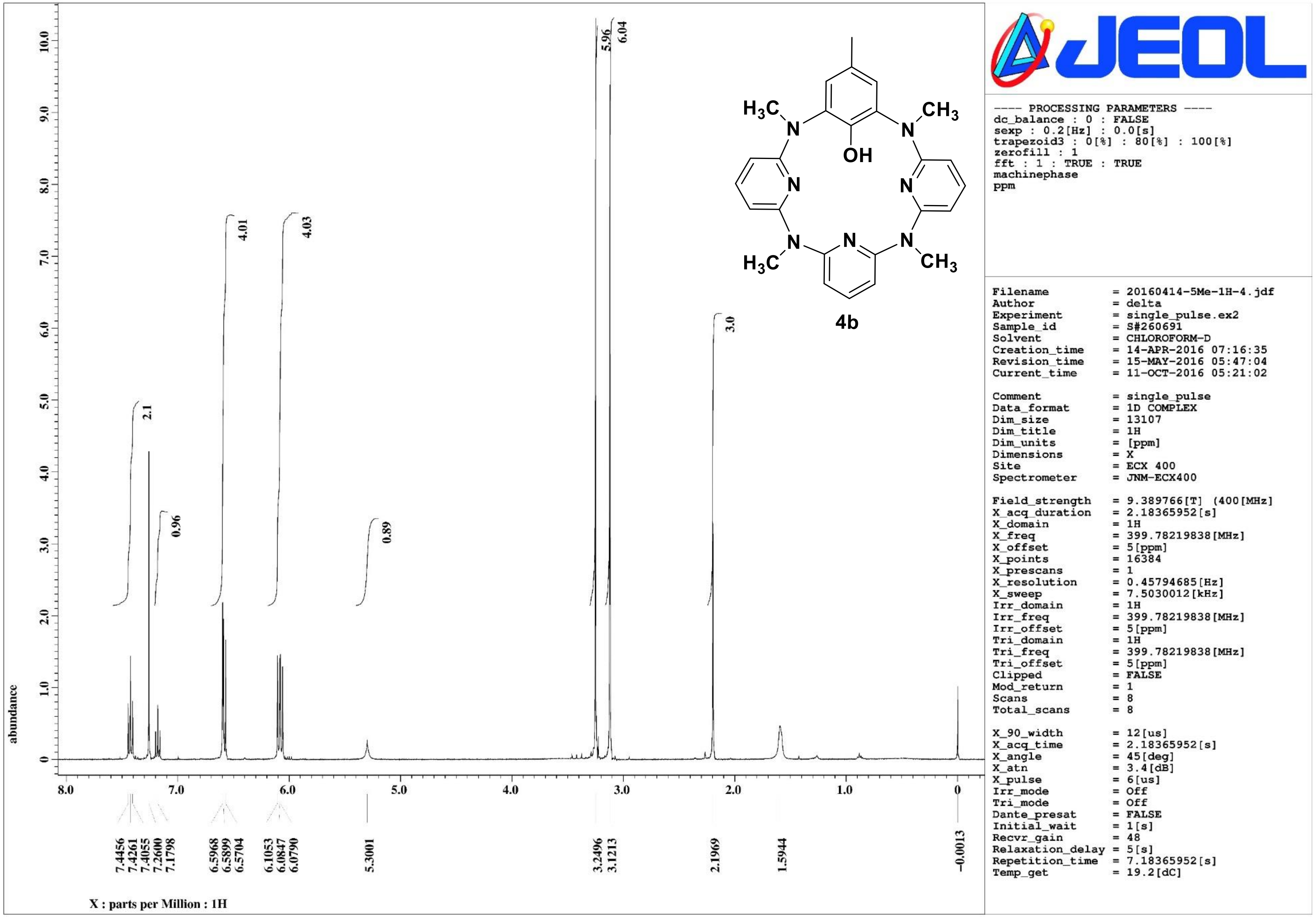

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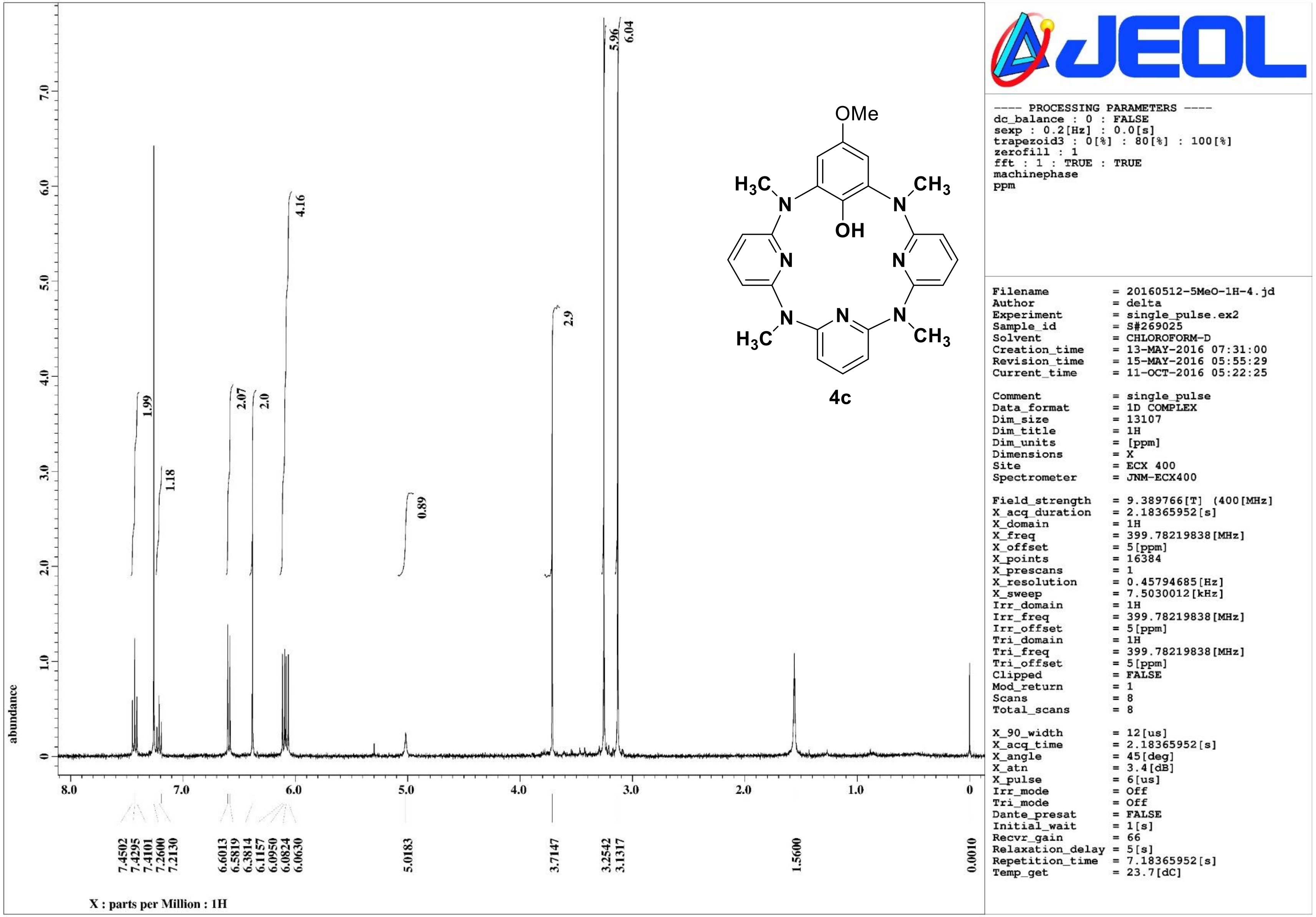

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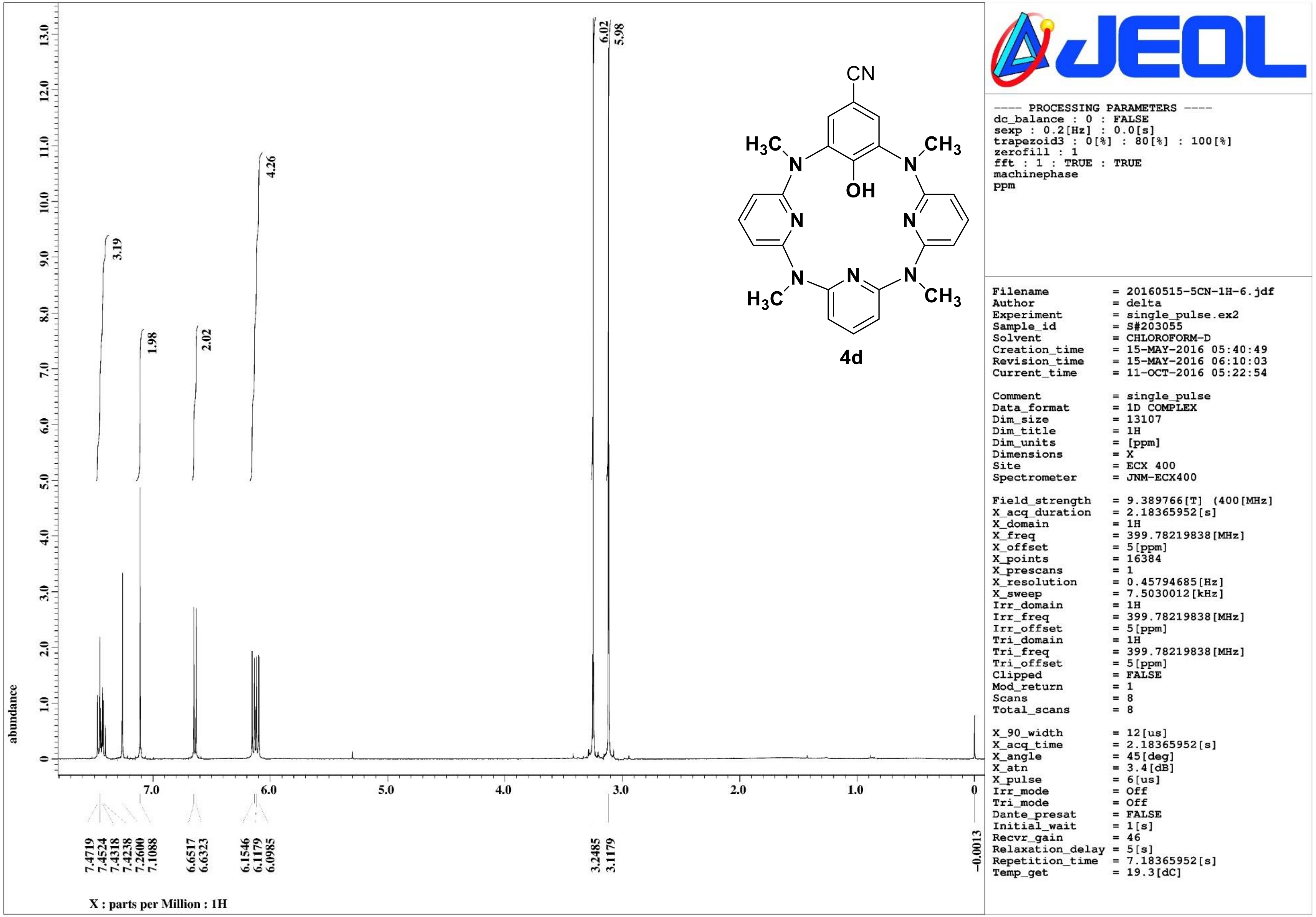

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```

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Filename          = 20160515-5CN-1H-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#203055
Solvent           = CHLOROFORM-D
Creation_time     = 15-MAY-2016 05:40:49
Revision_time     = 15-MAY-2016 06:10:03
Current_time      = 11-OCT-2016 05:22:54

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
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 19.3[dC]
```
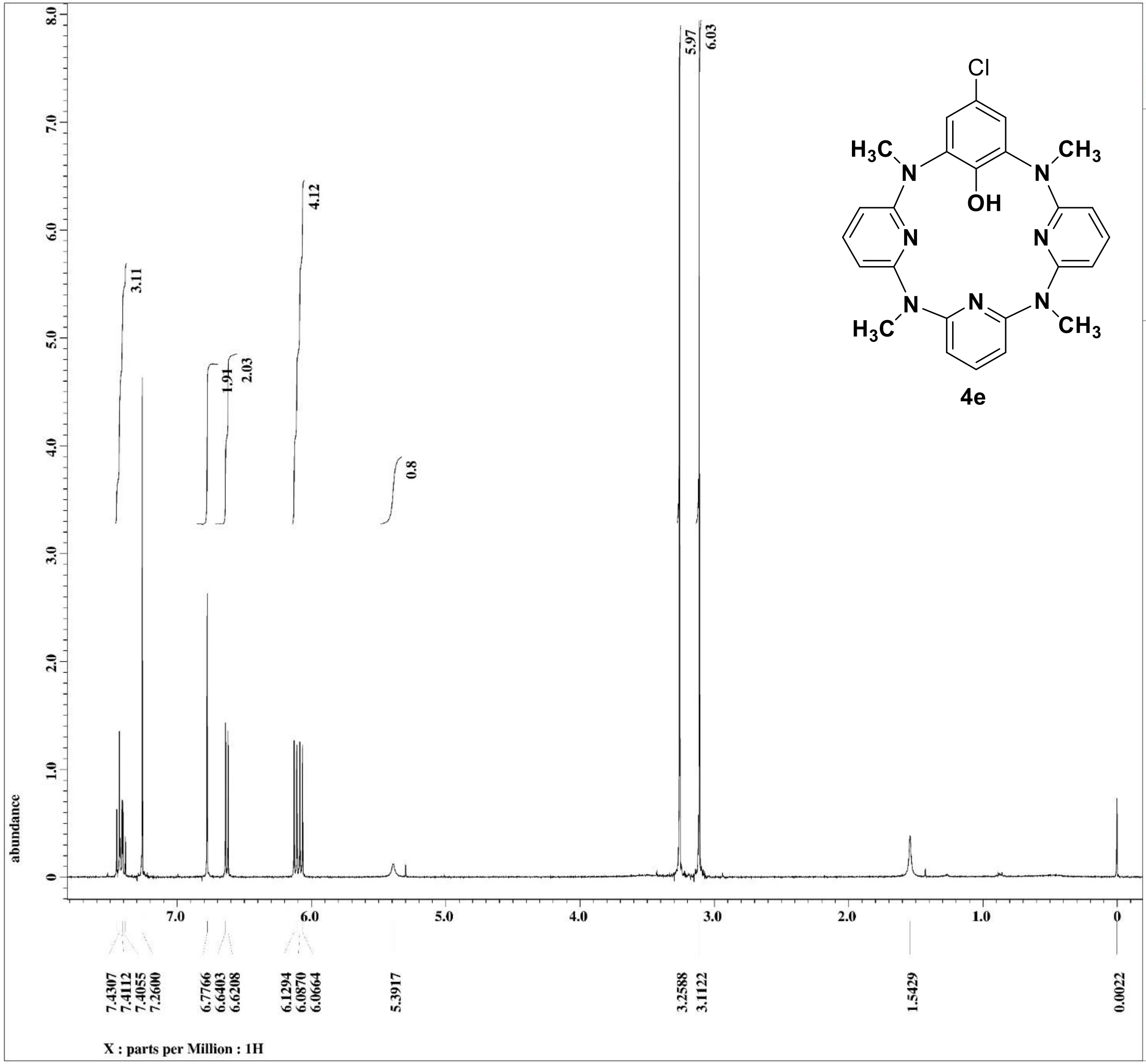
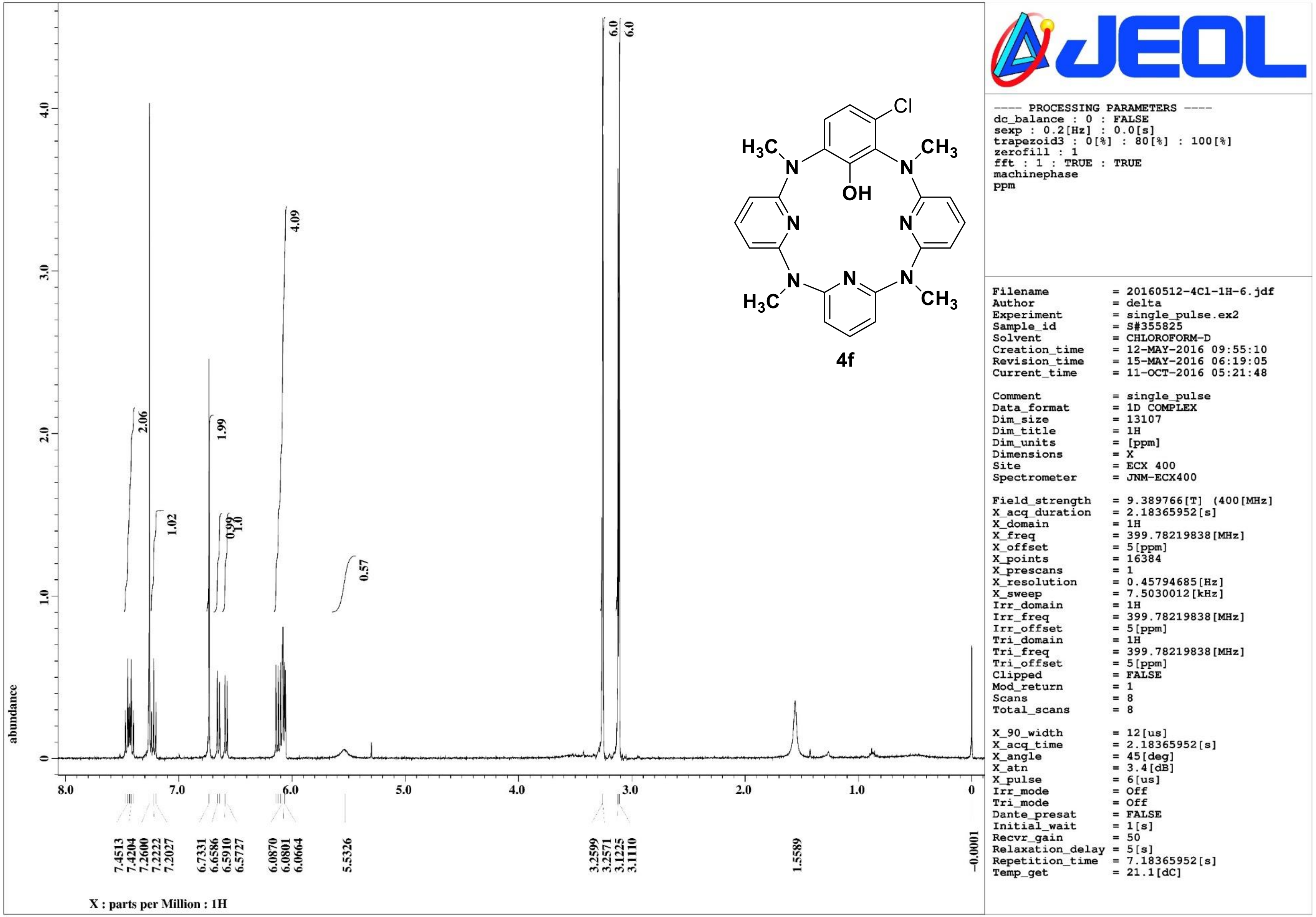



4e

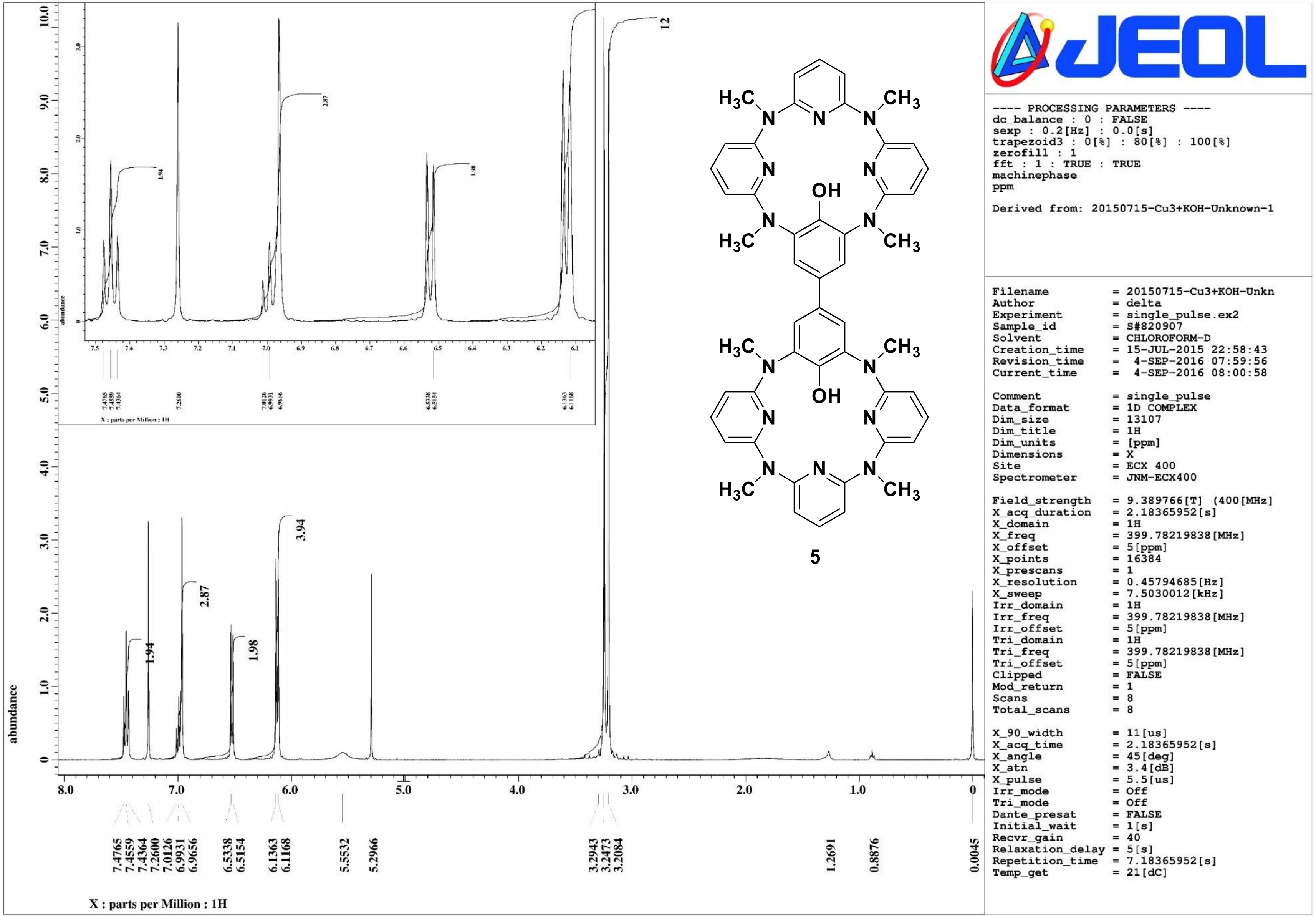
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 20160505-5Cl-1H-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#795174
Solvent           = CHLOROFORM-D
Creation_time     =  5-MAY-2016 22:07:21
Revision_time     = 15-MAY-2016 06:03:14
Current_time      = 11-OCT-2016 05:20:13

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
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 27.8[dC]
```

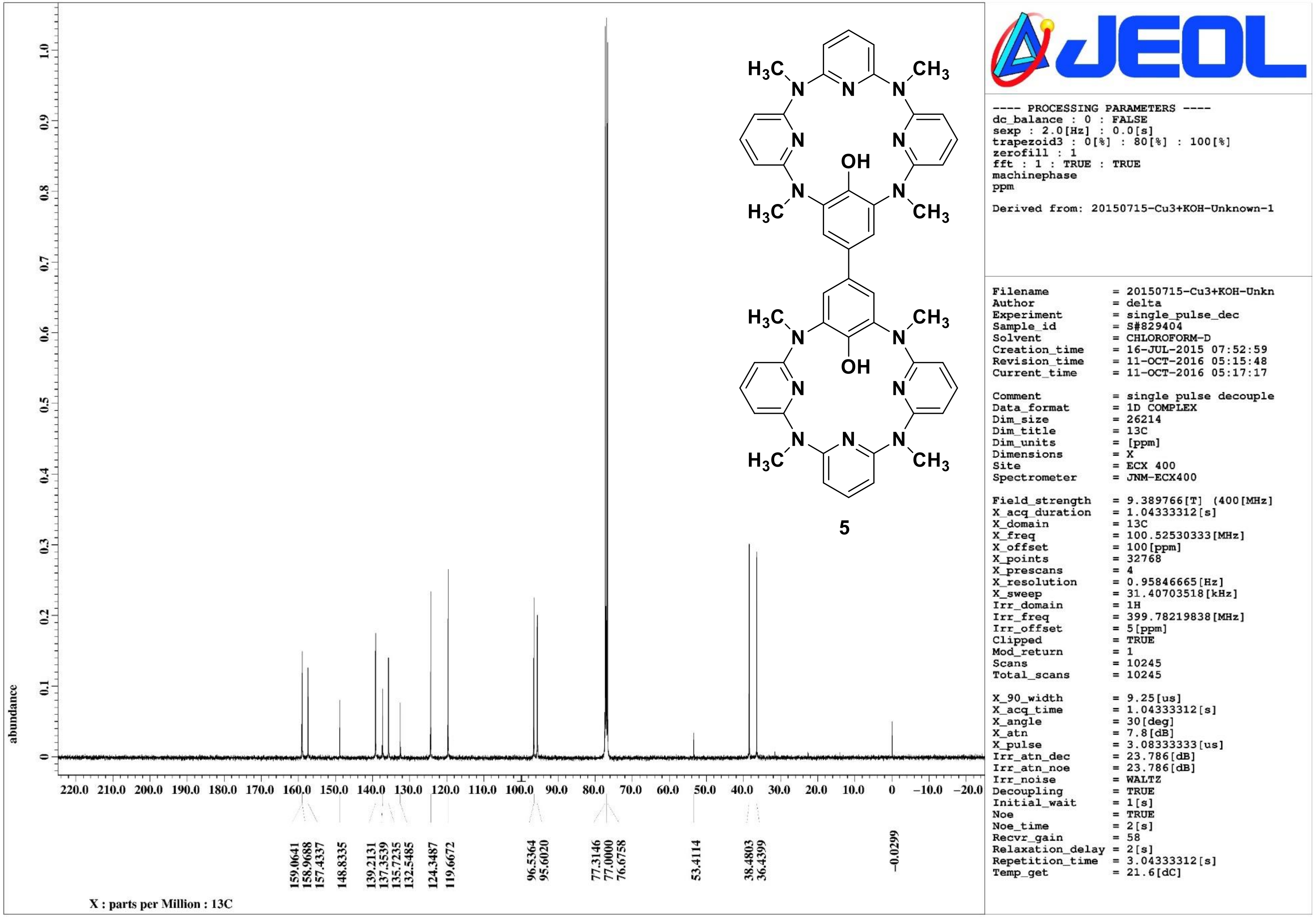


4f

```
---- 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           = 20160512-4Cl-1H-6.jdf
Author             = delta
Experiment         = single_pulse.ex2
Sample_id          = S#355825
Solvent            = CHLOROFORM-D
Creation_time      = 12-MAY-2016 09:55:10
Revision_time      = 15-MAY-2016 06:19:05
Current_time       = 11-OCT-2016 05:21:48

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
Mod_return         = 1
Scans              = 8
Total_scans        = 8

X_90_width         = 12[us]
X_acq_time         = 2.18365952[s]
X_angle            = 45[deg]
X_atn              = 3.4[dB]
X_pulse            = 6[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 50
Relaxation_delay   = 5[s]
Repetition_time    = 7.18365952[s]
Temp_get           = 21.1[dC]
```



```
---- 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

Derived from: 20150715-Cu3+KOH-Unknown-1

Filename          = 20150715-Cu3+KOH-Unkn
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#820907
Solvent           = CHLOROFORM-D
Creation_time     = 15-JUL-2015 22:58:43
Revision_time     =  4-SEP-2016 07:59:56
Current_time      =  4-SEP-2016 08:00:58

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
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 21[dC]
```



5

```
---- 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

Derived from: 20150715-Cu3+KOH-Unknown-1
```

```
Filename           = 20150715-Cu3+KOH-Unkn
Author             = delta
Experiment         = single_pulse_dec
Sample_id          = S#829404
Solvent            = CHLOROFORM-D
Creation_time      = 16-JUL-2015 07:52:59
Revision_time      = 11-OCT-2016 05:15:48
Current_time       = 11-OCT-2016 05:17:17

Comment            = single pulse decouple
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
Mod_return         = 1
Scans              = 10245
Total_scans        = 10245

X_90_width         = 9.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 7.8[dB]
X_pulse            = 3.08333333[us]
Irr_atn_dec        = 23.786[dB]
Irr_atn_noe        = 23.786[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 58
Relaxation_delay   = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 21.6[dC]
```