Synthesis and cytotoxicity of dibenzo[(γ-aryl)pyridino]aza-17-crown-5 ethers

Le Tuan Anh,[@a] Nguyen T. Thanh Phuong,[a] Truong Hong Hieu,[b] Anatoly T. Soldatenkov,[c] Bui T. Van,[a] Tran T. Thanh Van,[a] Dao T. Nhung,[a] Leonid G. Voskressensky,[c] and Victor N. Khrustalev[c,d]

[a]*Department of Pharmaceutical Chemistry, Faculty of Chemistry, VNU University of Science, 19 Le Thanh Tong, Hoan Kiem, Hanoi 10000, Vietnam.*
E-mail: anhlt@vnu.edu.vn or huschemical.lab@gmail.com
[b]*Department of Biotechnology, Vietnam–Russia Tropical Centre, 58 Nguyen Van Huyen, Hanoi, Vietnam*
[c]*Faculty of Science, Peoples' Friendship University of Russia (RUDN University), 6 Miklukho-Maklaya St., Moscow 117198, Russian Federation.*
[d]*X-Ray Structural Centre, A.N.Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilov St., Moscow 119991, Russian Federation.*

Table of contents

Result from HMBC and HSQC ……2

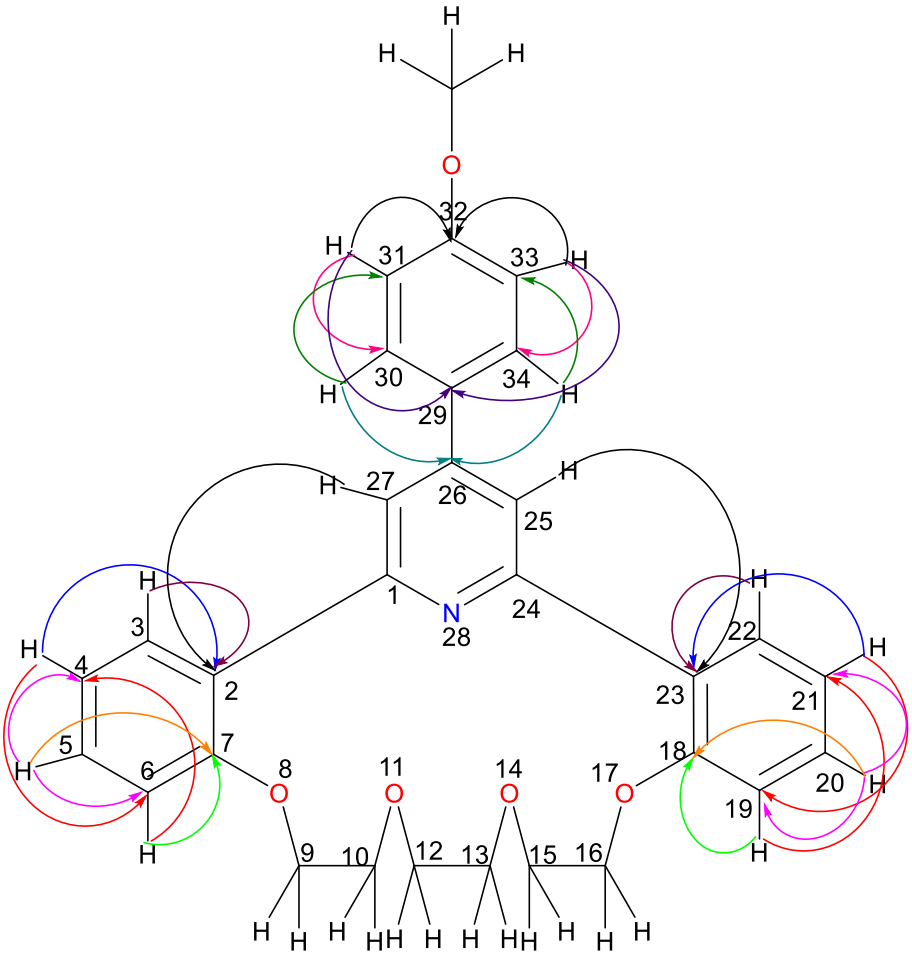
X-ray diffraction experiment ……5

Cytotoxicity assay……..…….…….……..7

References for Experimental Section ……9

Copies of HRMS, ^{1}H NMR, ^{13}C NMR and IR spectra…….……..10

Result from HMBC and HSQC



N_0	Positions	^{1}H-NMR (ppm)	^{13}C-NMR
1.	12,13	3.19	70.58
2.	10,15	3.62	69.56
3.	OCH_3	3.89	55.51
4.	9,16	4.10	71.16
5.	4,21	6.98-7.02	114.52
6.	6,19	6.98-7.02	114.62
7.	5,20	7.12	121.63
8.	3,22	7.35	129.67
9.	30,34	7.64-7.68	128.47
10.	31,33	7.64-7.68	130.76
11.	25, 27	7.72	121.95

12.	2, 23	-	131.65
13.	7,18	-	132.60
14.	29	-	146.77
15.	26	-	156.40
16.	1, 24	-	156.94
17.	32	-	160.43

The HMBC (Heteronuclear Multiple Bond Correlation) experiment gives correlations between carbons and protons that are separated by two, three bonds.

H4	C6
H21	C19
H6	C4
H19	C21
H4	C2
H21	C23
H6	C7
H19	C18
H5	C4,5
H20	C19,21
H5	C7
H20	C18
H3	C2
H22	C23
H31	C30
H33	C34
H30	C31
H34	C33
H31	C29
H33	C29
H30	C26
H34	C26
H31	C32

H33	C32
H27	C2
H25	C23

In aromatic zone showed cross peaks and the assignment of their carbon resonances: H-4 and H-21(σH 6.98-7.02 --> C-2 and C-23, respectively (σC 131.65), C-6 and C-19 (σC 114.62); H-6 and H-19 (σH 6.98-7.02) --> C-4 and C-21 (σC 114.52), C-7 and C-18 (σC 132.60); H-5 and H-20 (σH 7.12) --> C-4 and C-21 (σC 114.52), C-6 and C19 (σC 114.62) and C-7 and C-18 (σC 132.60); H-3 and H-22 (σH 7.35) --> C2 and C23 (σC 131.65), H-31 and H-33 (σH 7.64-7.68) --> C30 and C-34 (σC 128.47), C-29 (σC 146.77), C-32 (σC 160.43), -30 and H-34--> C-26 (σC 156.40), C31 and C-33 (σC 130.76), H-25 and H-27 (σH 7.72) --> C-2 and C-23, respectively (σC 131.65).

X-ray diffraction experiment

The crystal of **3a** ($C_{30}H_{29}NO_5$, M = 483.54) is monoclinic, space group $P2_1/c$, at T = 120 K: a = 10.239(4) Å, b = 10.344(4) Å, c = 23.413(8) Å, β = 98.648(6)°, V = 2451.5(15) Å^3, Z = 4, d_{calc} = 1.310 g/cm^3, $F(000)$ = 1024, μ = 0.089 mm^{-1}. 21314 total reflections (5116 unique reflections, R_{int} = 0.082) were measured on a three-circle Bruker APEX-II CCD diffractometer (λ(MoK$_\alpha$)-radiation, graphite monochromator, φ and ω scan mode, $2\theta_{max}$ = 54.0°) and corrected for absorption (T_{min} = 0.975; T_{max} = 0.980).[1] The structure was determined by direct methods and refined by full-matrix least squares technique on F^2 with anisotropic displacement parameters for non-hydrogen atoms. The hydrogen atoms were placed in calculated positions and refined within riding model with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C) for the methyl group and 1.2U_{eq}(C) for the other groups]. The final divergence factors were R_1 = 0.116 for 3809 independent reflections with $I > 2\sigma(I)$ and wR_2 = 0.242 for all independent reflections, S = 1.031. All calculations were carried out using the SHELXTL program.[2]

Crystallographic data for **3a** have been deposited with the Cambridge Crystallographic Data Center, CCDC 1532287. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

Table 1. Hydrogen bonds for **3a** [Å and °].

D*─H···A*	d(D─H)	d(H···A)	d(D···A)	∠DHA
C9─H9*a*···N28^a	0.99	2.59	3.491(7)	151.9
C12─H12*a*···O17^b	0.99	2.56	3.335(7)	135.1
C12─H12*b*···O8	0.99	2.39	3.013(6)	119.9
C13─H13*b*···O17	0.99	2.55	2.958(6)	104.8
C35─H35*a*···O11^c	0.98	2.55	3.465(7)	156.1

* D – proton donor; A – proton acceptor;

Symmetry transformations used to generate equivalent atoms:

^{a} –x, –y+1, –z; ^{b} –x, y+0.5, –z+0.5; ^{c} x+1, –y+1.5, z+0.5

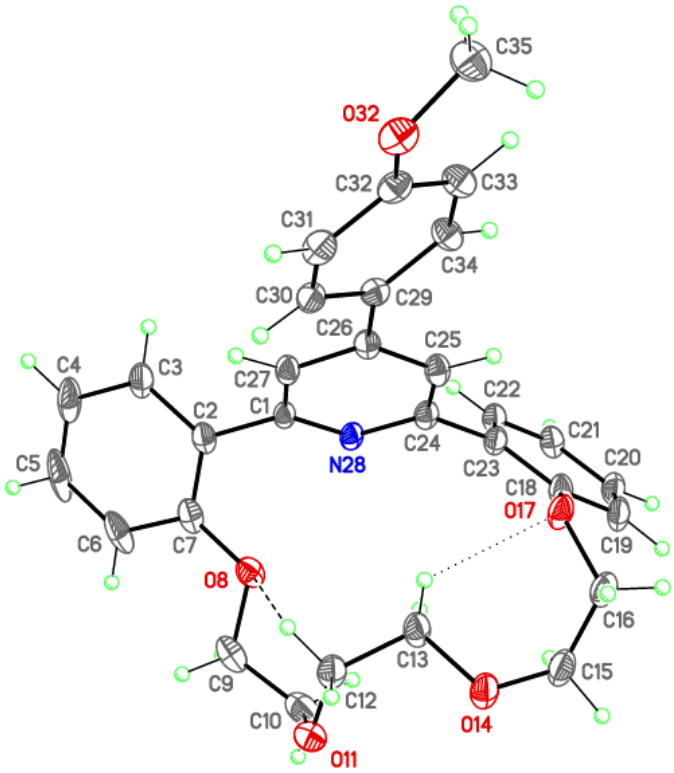


Figure 1. Molecular structure of **3a**. The dashed and dotted lines indicate the intramolecular C─H···O hydrogen bonds.

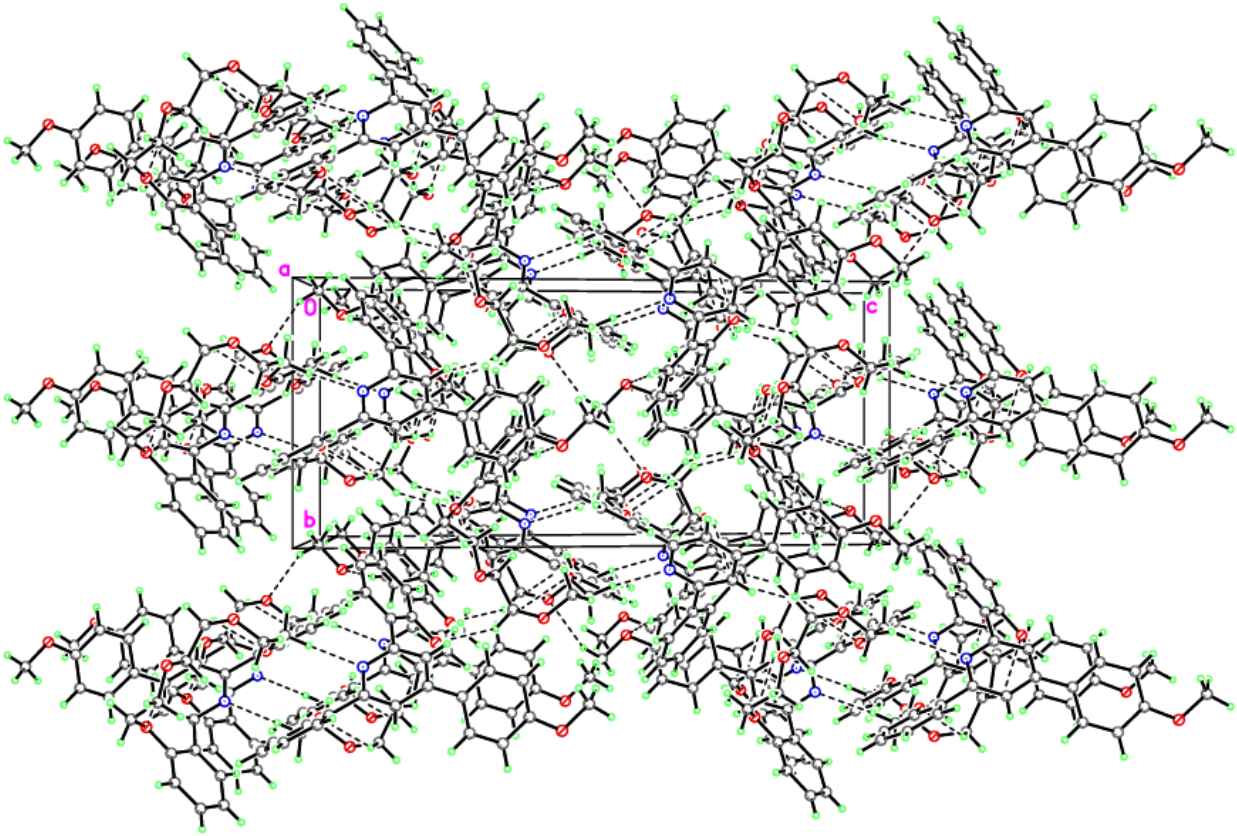


Figure 2. Crystal packing of **3a**. The dashed and dotted lines indicate the intramolecular C─H···O and intermolecular C─H···N and C─H···O hydrogen bonds.

Cytotoxicity assay

Four human cancer cell lines was obtained from the American Type Culture Collection (Manassas, VA) ATCC as FL (human cervix carcinoma), RD (human rhabdosarcoma), Lu1 (human lung adenocarcinoma), HepG2 (human hepatocellular carcinoma). The Vero cell line was initiated from kidney of a normal adult African green monkey. They were grown in DMEM (Dulbecco's Modified Eagle Medium) (Thermo Fisher Scientific, USA) supplemented with L-glutamine, Sodium pyruvate, $NaHCO_3$, PSF (Penicillin- Streptomycin sulfate - Fungizone); NAA (Non-Essential Amino Acids); 10% BCS (Bovine Calf Serum). All incubations were performed at 37ºC for 72 hours in a CO_2 (5%) incubator with the plates capped in the normal fashion

The MTT is based on the protocol described by Skehan & CS (1990) and Likhiwitayawuid & CS (1993).[3] This method has worldwide application and is recommended by National Cancer Institute (NCI) and College of Medicine, University of Illinois at Chicago for routine drug screening.

Table 1. Cytotoxicity tests performed on compounds **3 a-g** in human cancer cell lines.

№	**Comp.**	**Conc.** ***(µg/ml)***	**Cell line, cell survival (%)**				**Concl.**
			HepG2	**Lu1**	**RD**	**FL**	
	DMSO	-	100	100	100	100	
	Taxol (+)	5	1,34±0,8	3,66±0,9	0,68±0,3	3,87±1,1	Positive
1	**3 a**	10	100	99.06±1.5	100	100	Negative
2	**3 b**	10	**0**	**0**	**0**	**0**	Positive with 04 cell lines
3	**3 c**	10	**0**	**0**	**0**	**0**	Positive with 04 cell lines
4	**3 d**	10	**20.07±0.8**	**38.25±0.4**	**39.26±0.7**	64.09±0.8	Positive with 03 cell lines

5	**3 e**	10	67,61±1,4	64,44±1,5	83,27±1,2	59,3±2,3	Negative
6	**3 f**	10	58,45±0,8	72,89±2,8	**44,83±1,1**	**13,46±1,6**	Positive with 02 cell lines: RD, FL
7	**3 g**	10	60,85±1,4	69,47±2,6	**50,09±2,8**	**41,71±3,1**	Positive with 02 cell lines: RD, FL

Table 2. Results of IC_{50} test

Entry	**Comp.**	**Cell lines** IC_{50} ***(µg/ml)***				**Conclusion**
		HepG2	**Lu1**	**RD**	**FL**	
	Taxol (+)	**0.29**	**0.31**	**0.25**	**0.33**	Positive
1	**3 b**	**2.607**	**2.664**	**2.56**	**1.392**	Positive with 04 cell lines
2	**3 c**	**7.957**	**6.946**	**6.887**	**6.742**	Positive with 04 cell lines
3	**3 d**	**6.586**	**7.904**	**7.528**	-	Positive with 03 cell lines
4	**3 f**	-	-	**8.881**	**8.346**	Positive with 02 cell lines: RD, FL
5	**3 g**	-	-	**10.0**	**9.505**	Positive with 02 cell lines: RD, FL

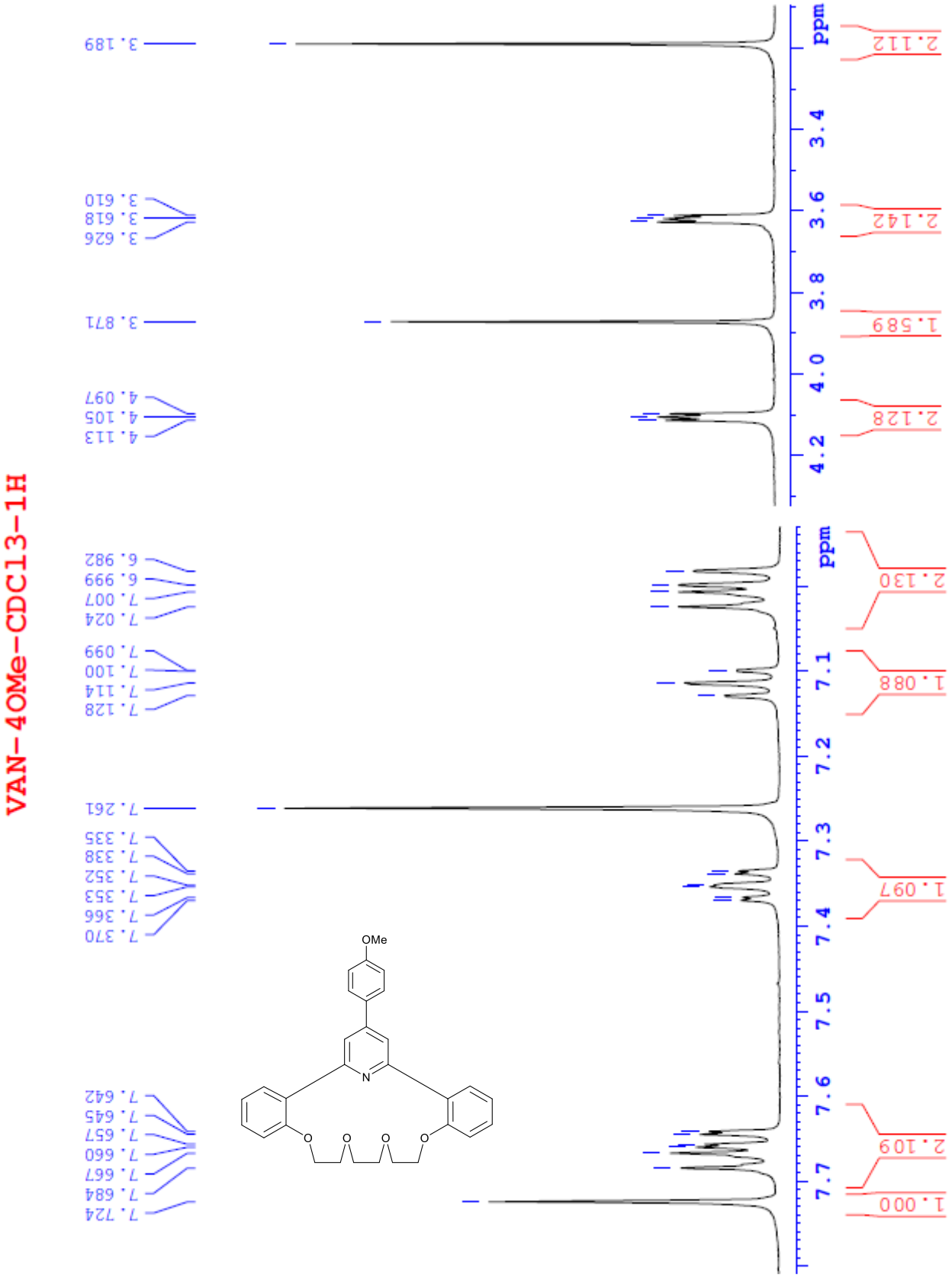
Table 3. Results of test on cytotoxicity on the Vero cell

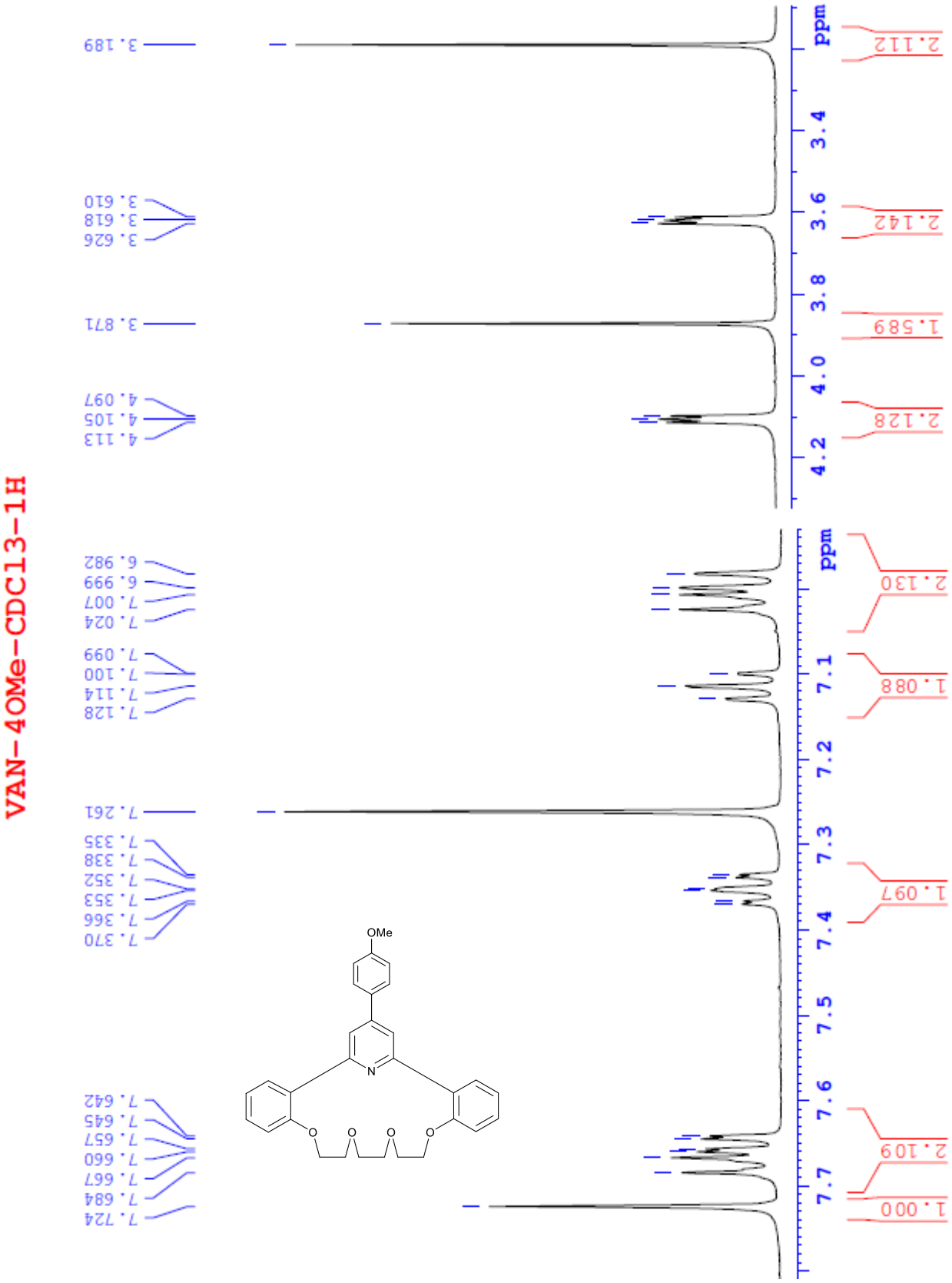
Entry	Compound	Conc. *(μg/ml)*	Vero cell line, cell survival (%)	Conclusion
	DMSO	-	100	
	Taxol (+)	5	**32.24±1.7**	
1	**3 b**	10	71,60±2,3	Negative
		5	86.55±0.7	Negative
2	**3 c**	10	65.86±0.8	Negative
		5	92.22±1.5	Negative
3	**3 d**	10	80.92±1.3	Negative

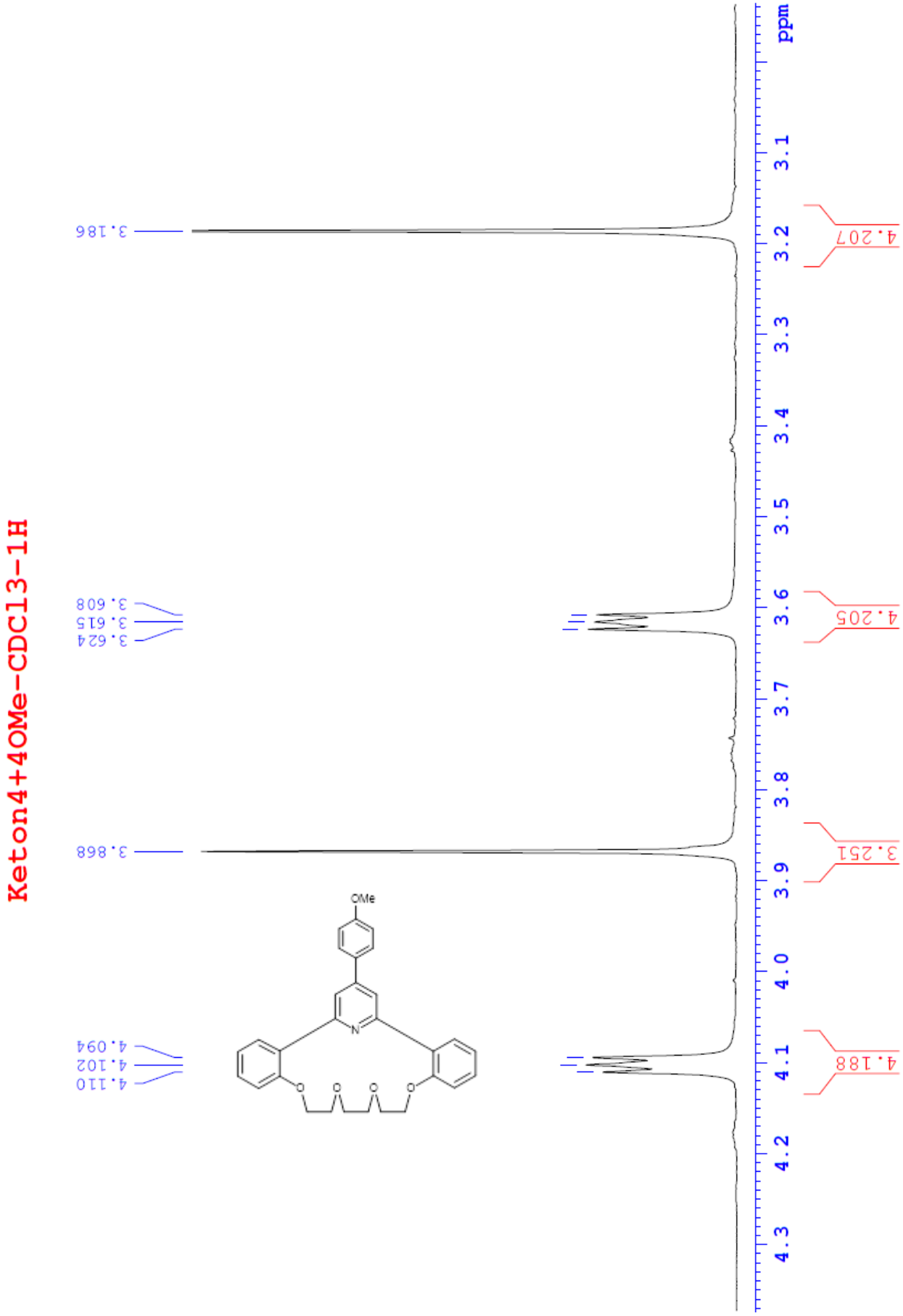
References

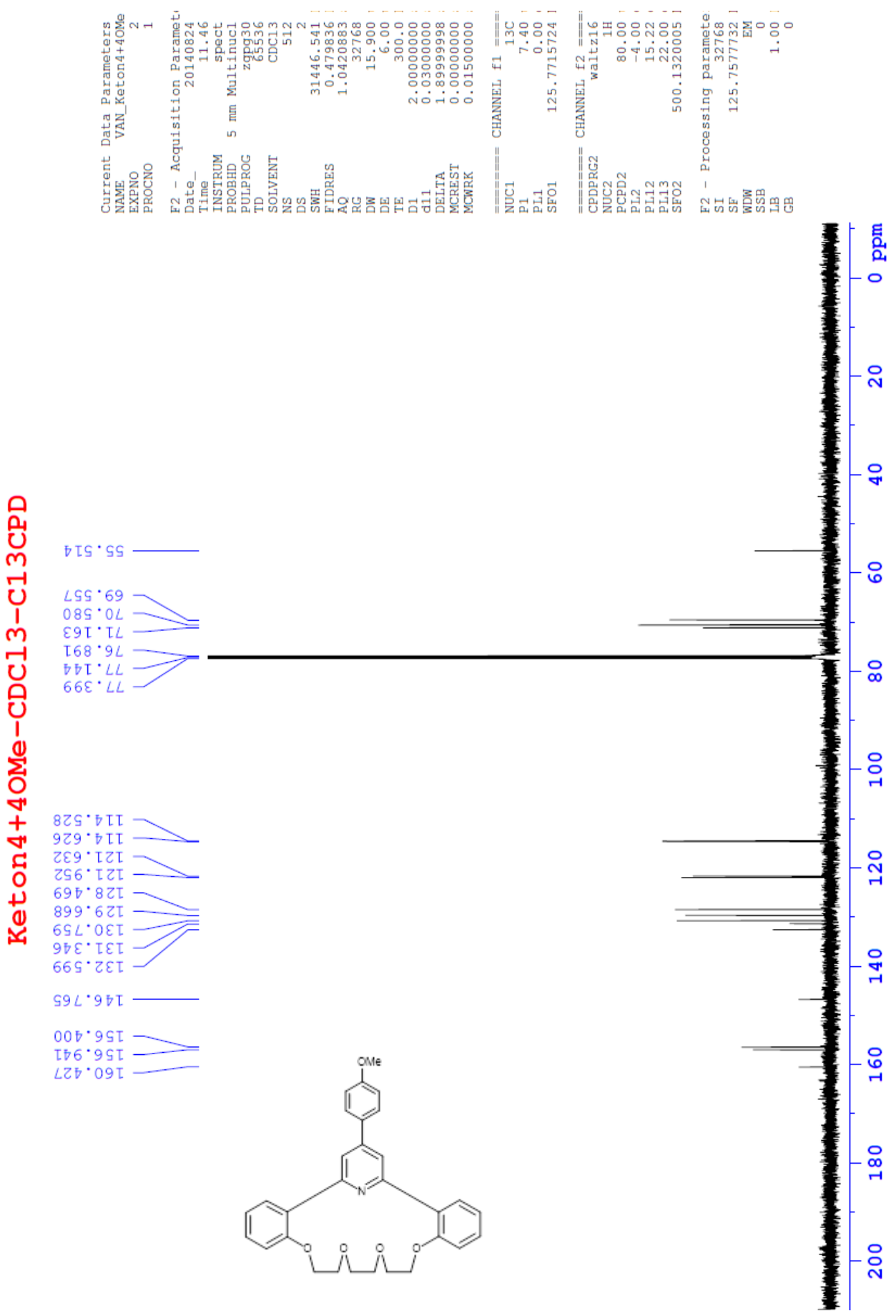
1. G. M. Sheldrick, *SADABS, v. 2.03*, Bruker/Siemens Area Detector Absorption Correction Program; Bruker AXS Inc., Madison, WI, **2003**.
2. G. M. Sheldrick, *Acta Cryst.* **2015**, *C71*, 3-8.
3. Skehan P., Storeng R., Scudiero D., Monks A., McMahon J., Vistica D., Warren J.T., Bokesch H., Kenney S., Boyd M.R. New colorimetric cytotoxicity assay for anticancer-drug screening. J Natl Cancer Inst. **1990**, 82(13),1107–1112.
4. Likhitwitayawuid K., Angerhofer C.K., Cordell G.A., Pezzuto J.M., Ruangrungsi N. Cytotoxic and antimalarial bisbenzylisoquinoline alkaloids from Sephania erecta. *Journal of Natural Products*. **1993**, 56 (1) pp. 30-38.

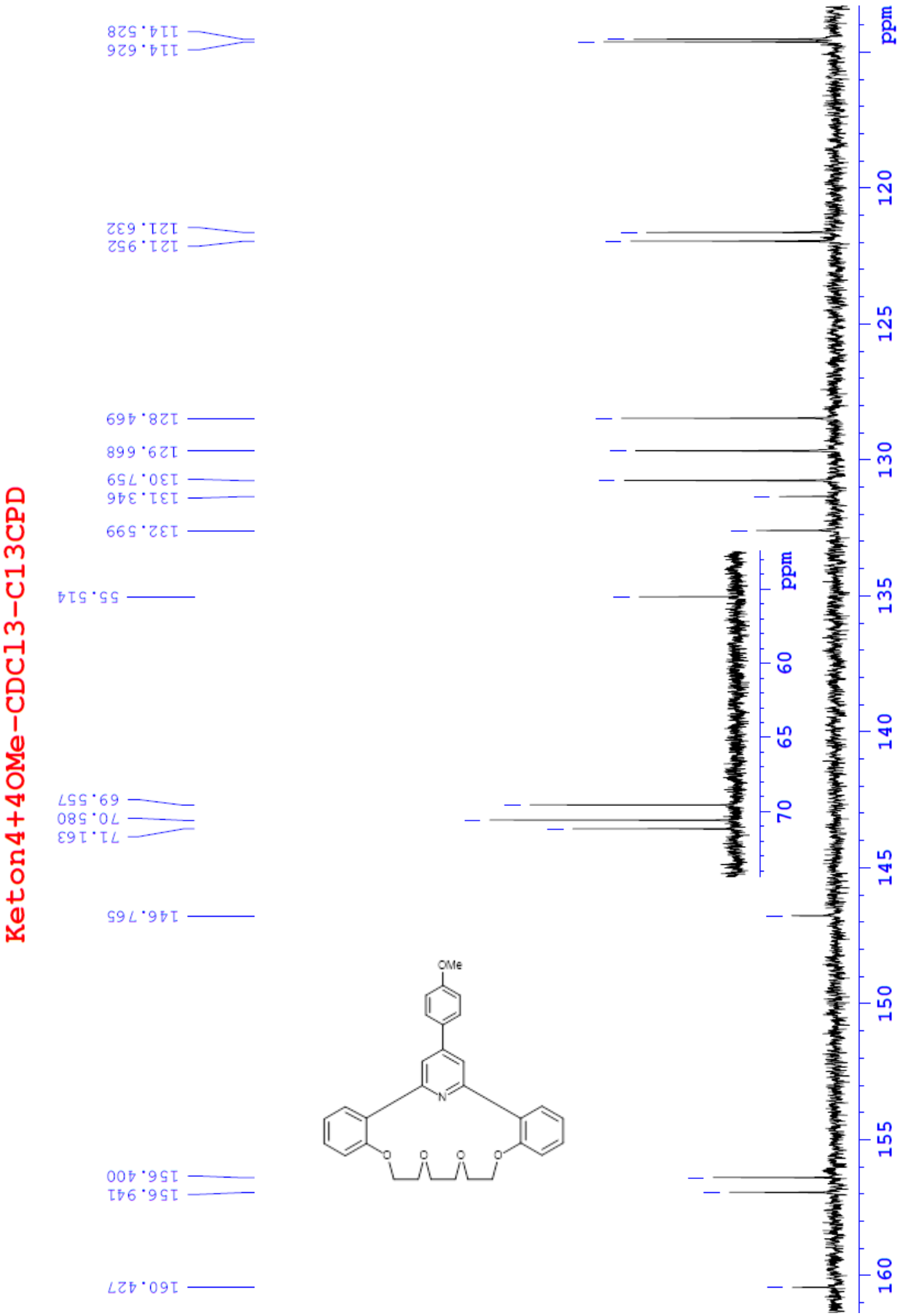
Copies of HRMS, 1H NMR and 13C NMR spectra

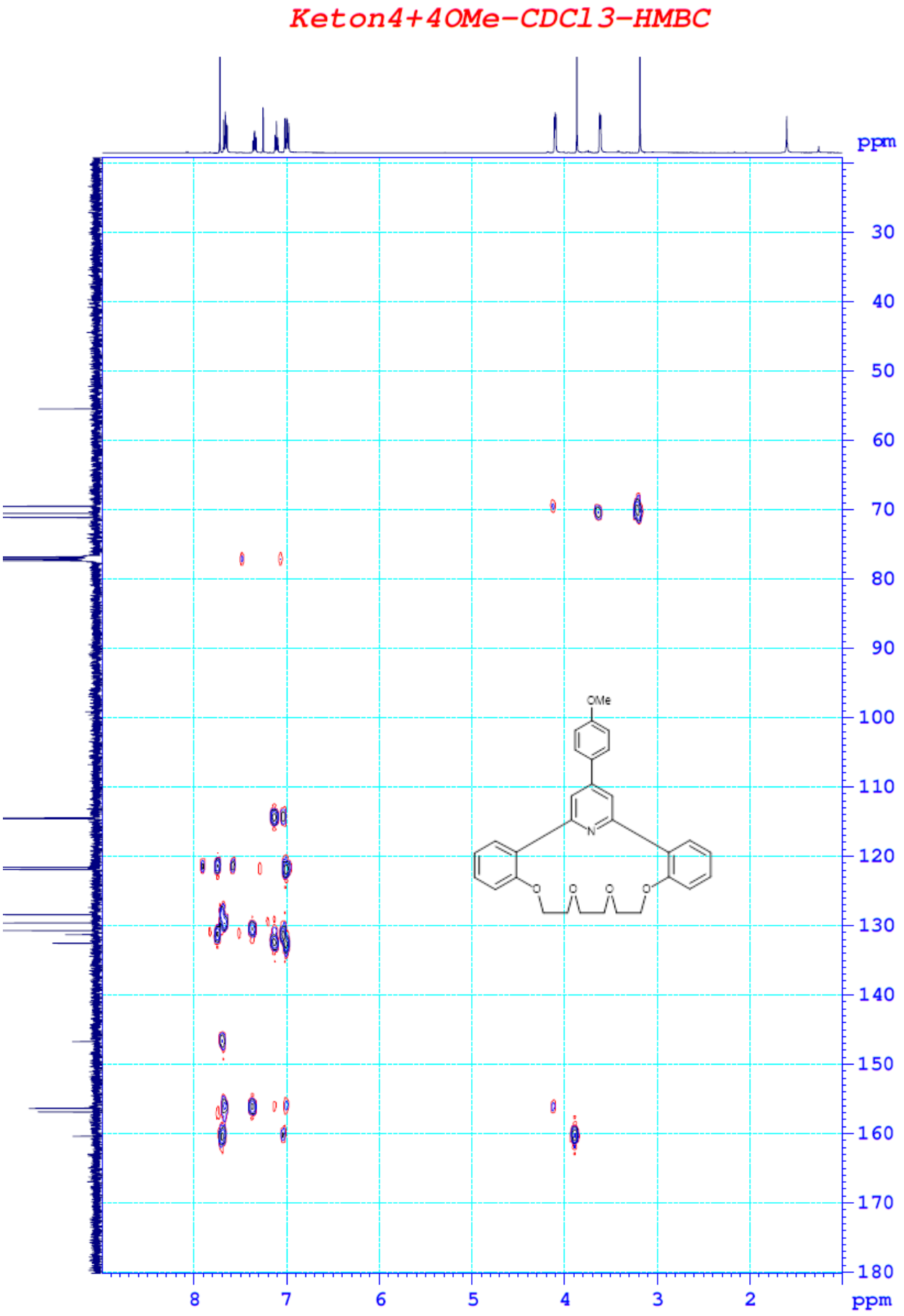


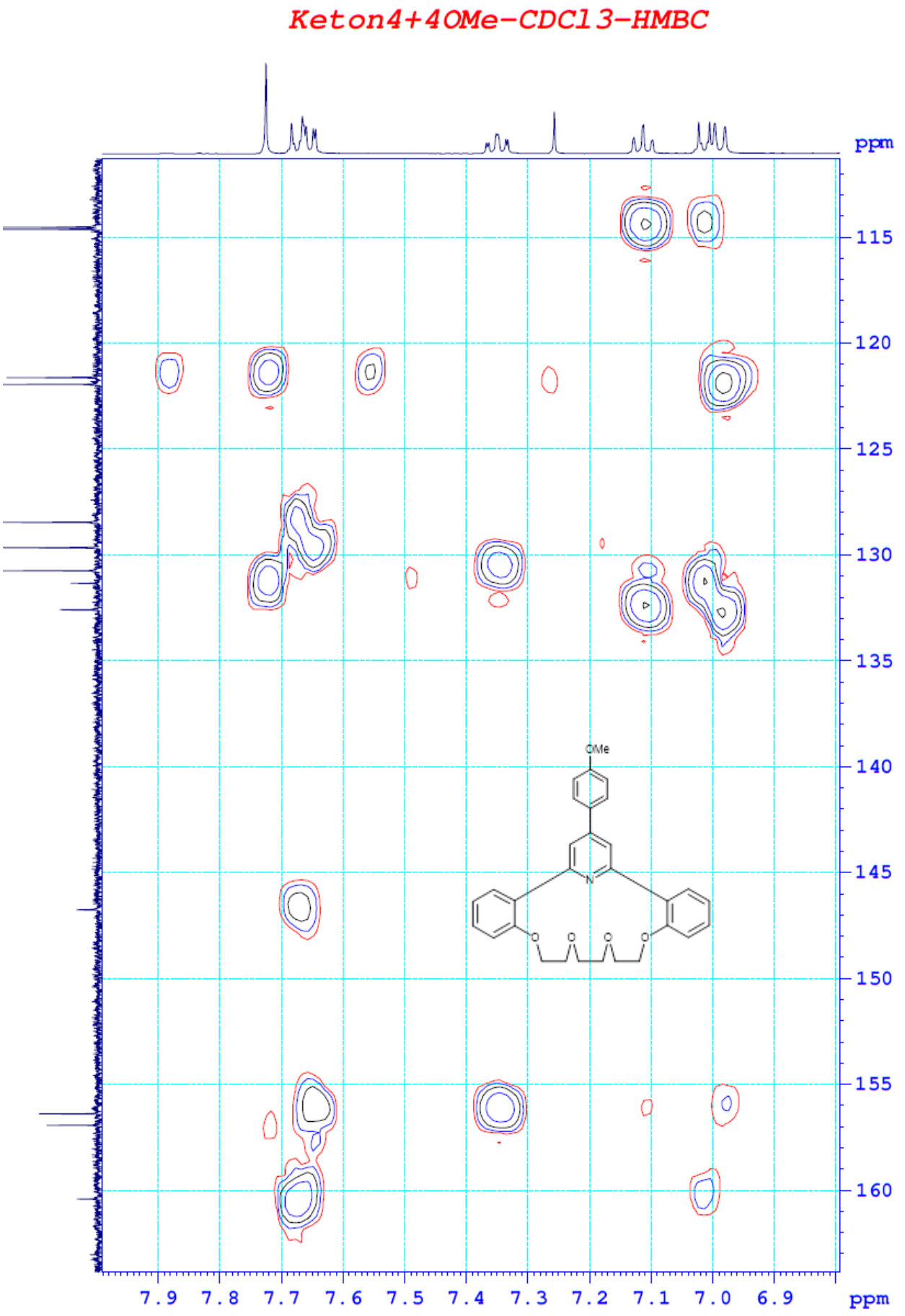

VAN-4OMe-CDC13-1H
3.189
3.610
3.618
3.626
3.871
4.097
4.105
4.113
6.982
6.999
7.007
7.024
7.099
7.100
7.114
7.128
7.261
7.335
7.338
7.352
7.353
7.366
7.370
7.642
7.645
7.657
7.660
7.667
7.684
7.724
OMe
N
O
O
O
O
ppm
4.2
4.0
3.8
3.6
3.4
2.112
2.142
1.589
2.128
ppm
7.7
7.6
7.5
7.4
7.3
7.2
7.1
2.130
1.088
1.097
2.109
1.000

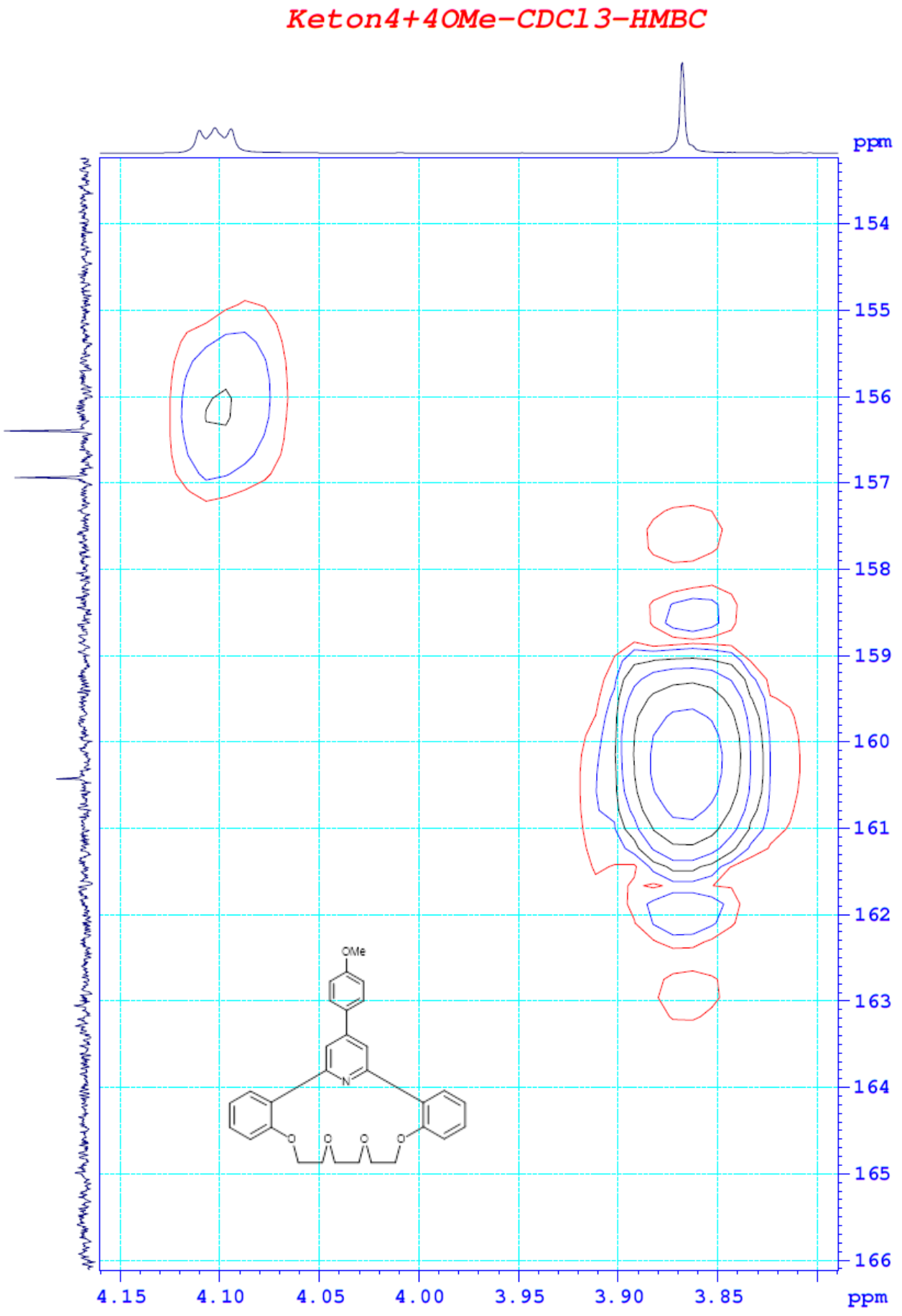

Keton4+4OMe-CDCl3-1H
ppm
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
4.1
4.2
4.3
3.186
3.608
3.615
3.624
3.868
4.094
4.102
4.110
4.207
4.205
3.251
4.188
OMe
N
O

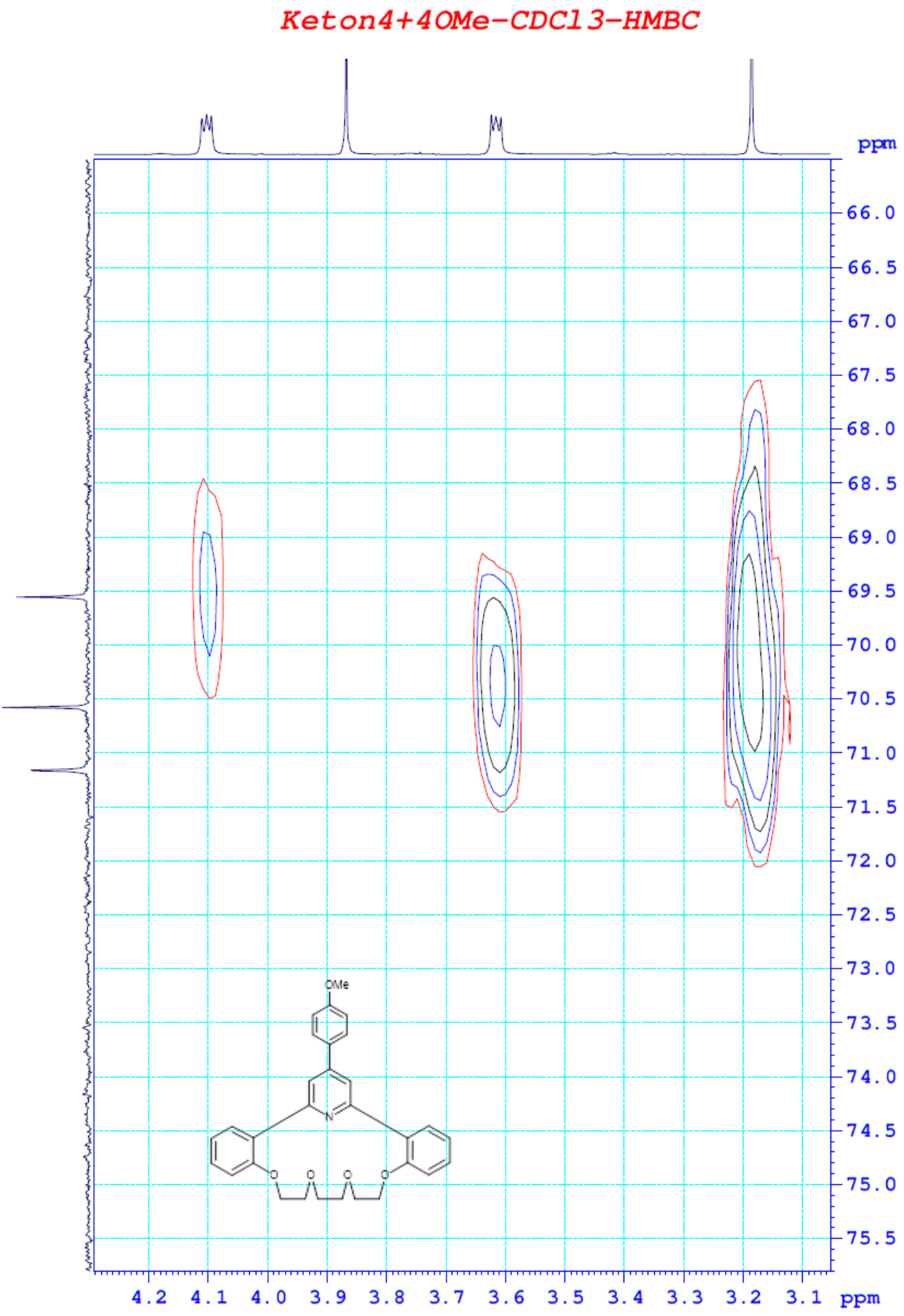

Keton4+4OMe-CDC13-C13CPD
Current Data Parameters
NAME VAN_Keton4+4OMe
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140824
Time 11.46
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 2
SWH 31446.541
FIDRES 0.479836
AQ 1.0420883
RG 32768
DW 15.900
DE 6.00
TE 300.0
D1 2.00000000
d11 0.03000000
DELTA 1.89999998
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ========
NUC1 13C
P1 7.40
PL1 0.00
SFO1 125.7715724
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00
PL2 -4.00
PL12 15.22
PL13 22.00
SFO2 500.1320005
F2 - Processing parameters
SI 32768
SF 125.7577732
WDW EM
SSB 0
LB 1.00
GB 0
55.514
69.557
70.580
71.163
76.891
77.144
77.399
114.528
114.626
121.632
121.952
128.469
129.668
130.759
131.346
132.599
146.765
156.400
156.941
160.427
OMe
N
O
O
O
O
0 ppm
20
40
60
80
100
120
140
160
180
200

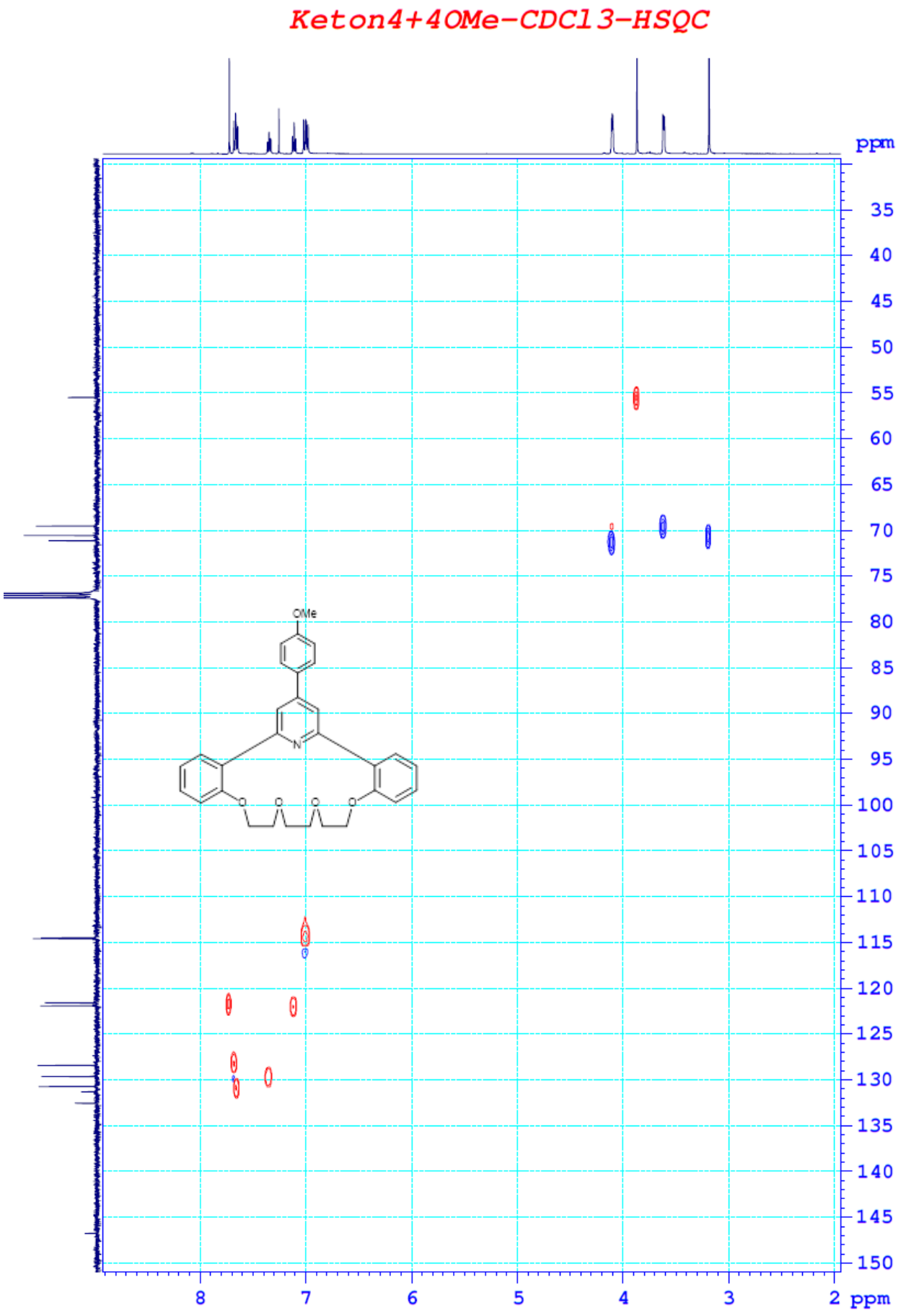

Keton4+4OMe-CDCl3-C13CPD
114.528
114.626
121.632
121.952
128.469
129.668
130.759
131.346
132.599
55.514
69.557
70.580
71.163
146.765
156.400
156.941
160.427
OMe
N
O
ppm
120
125
130
135
140
145
150
155
160
60
65
70

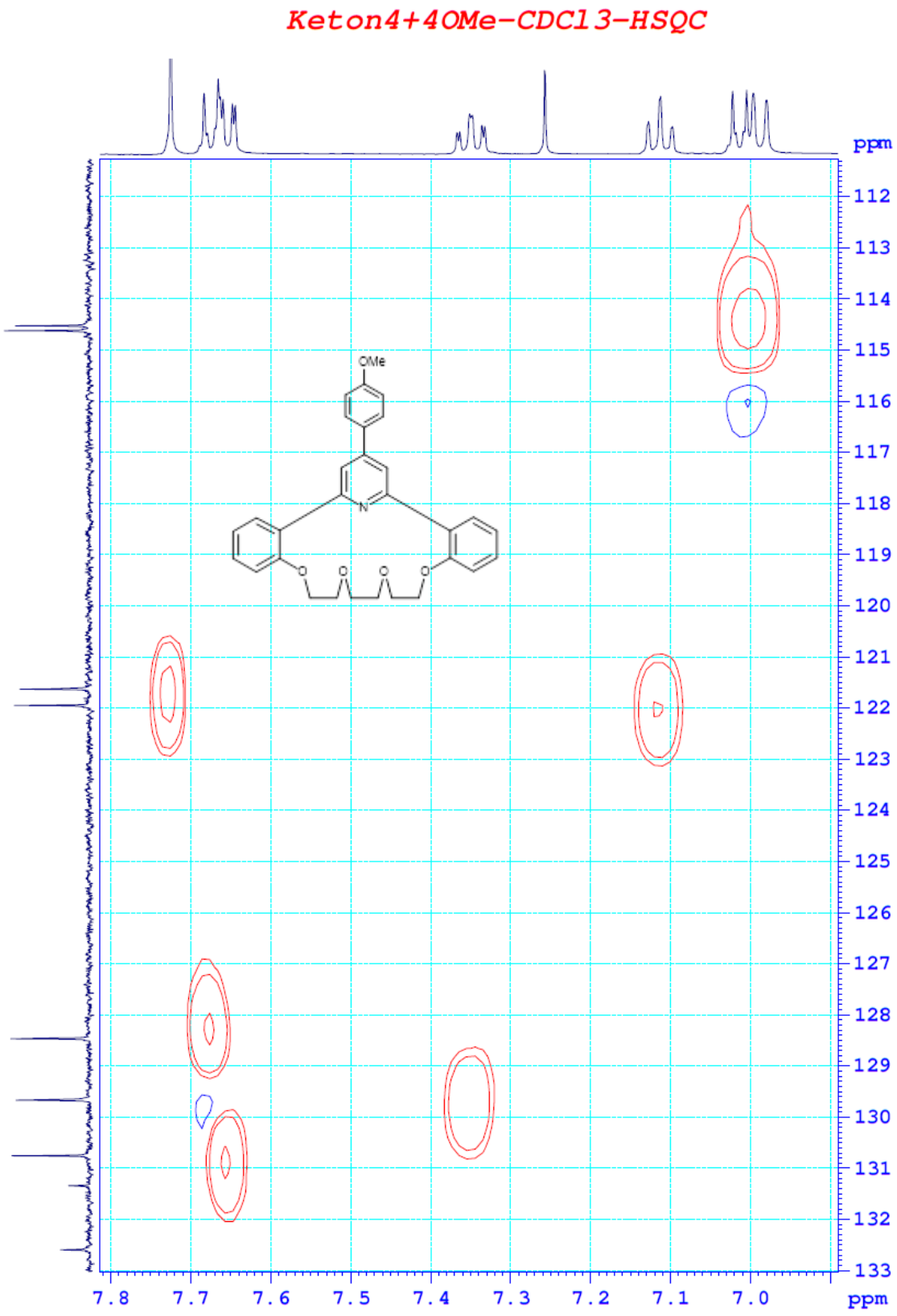

Keton4+4OMe-CDCl3-HMBC
ppm
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
8
7
6
5
4
3
2
ppm
OMe
N
O
O
O
O

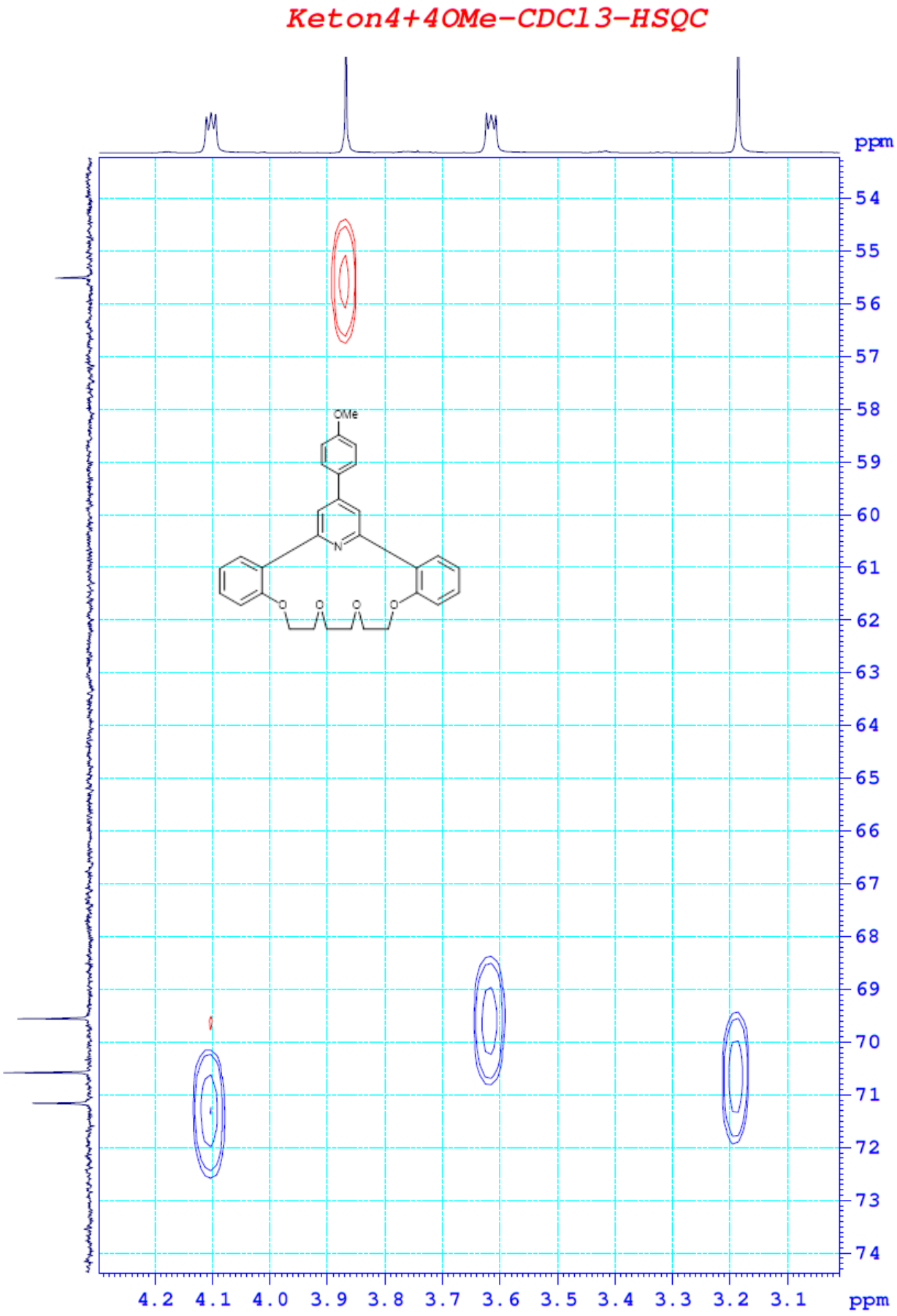

Keton4+4OMe-CDCl3-HMBC
ppm
115
120
125
130
135
140
145
150
155
160
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
ppm
OMe
N
O
O
O
O

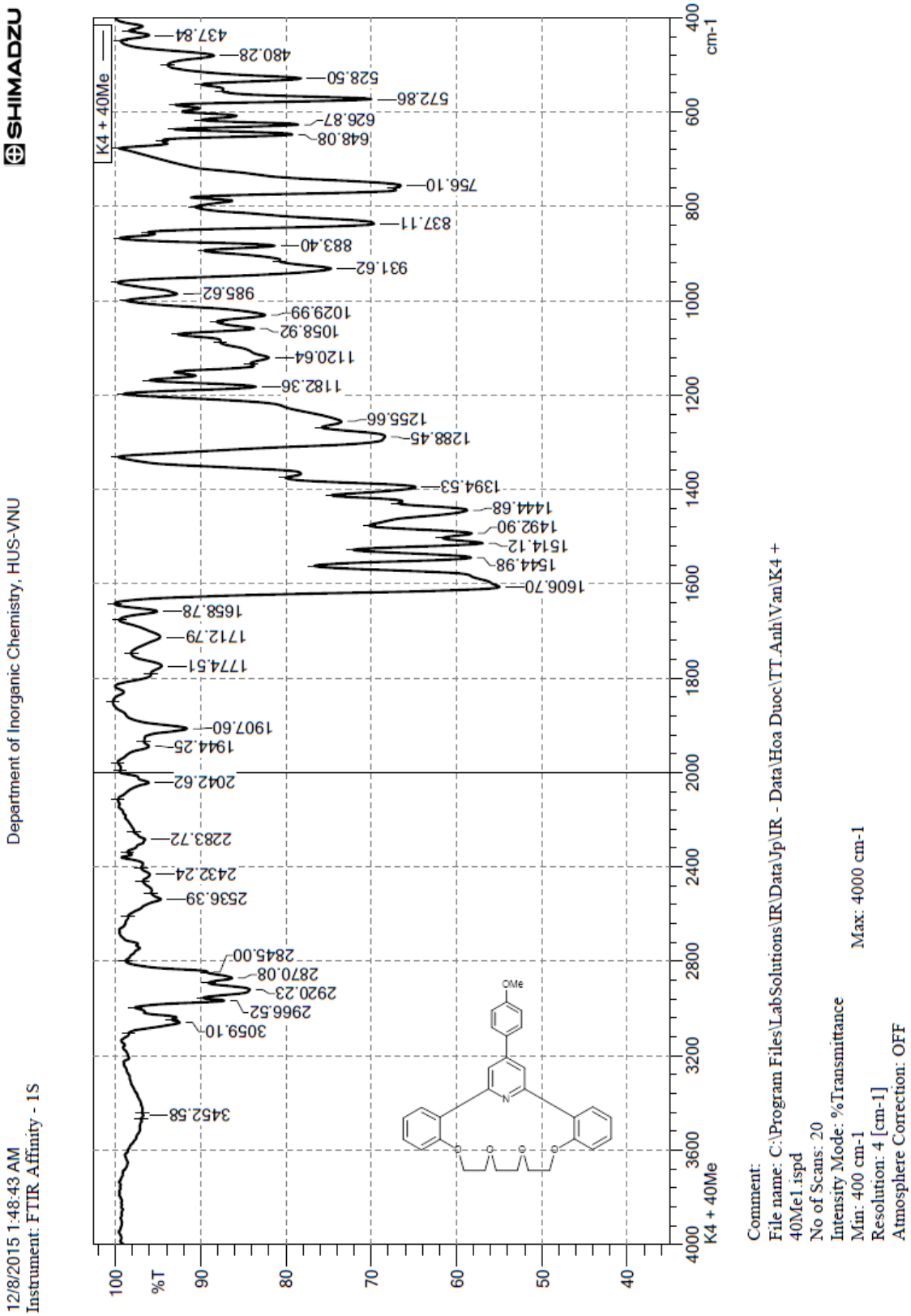

Keton4+4OMe-CDCl3-HMBC
ppm
154
155
156
157
158
159
160
161
162
163
164
165
166
4.15
4.10
4.05
4.00
3.95
3.90
3.85
ppm
OMe
N
O
O
O
O

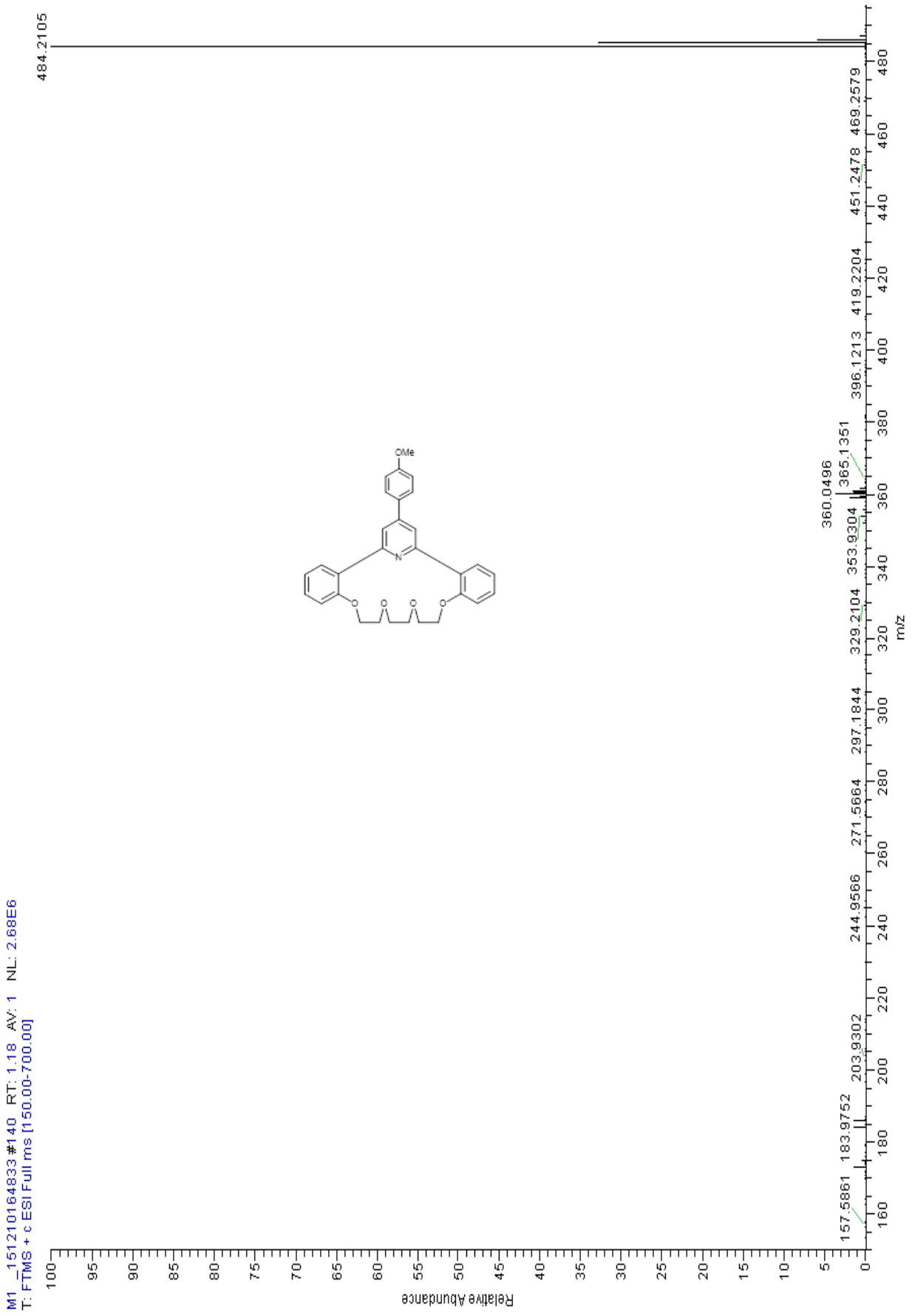

Keton4+4OMe-CDCl3-HMBC
ppm
66.0
66.5
67.0
67.5
68.0
68.5
69.0
69.5
70.0
70.5
71.0
71.5
72.0
72.5
73.0
73.5
74.0
74.5
75.0
75.5
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
ppm
OMe
N
O
O
O
O

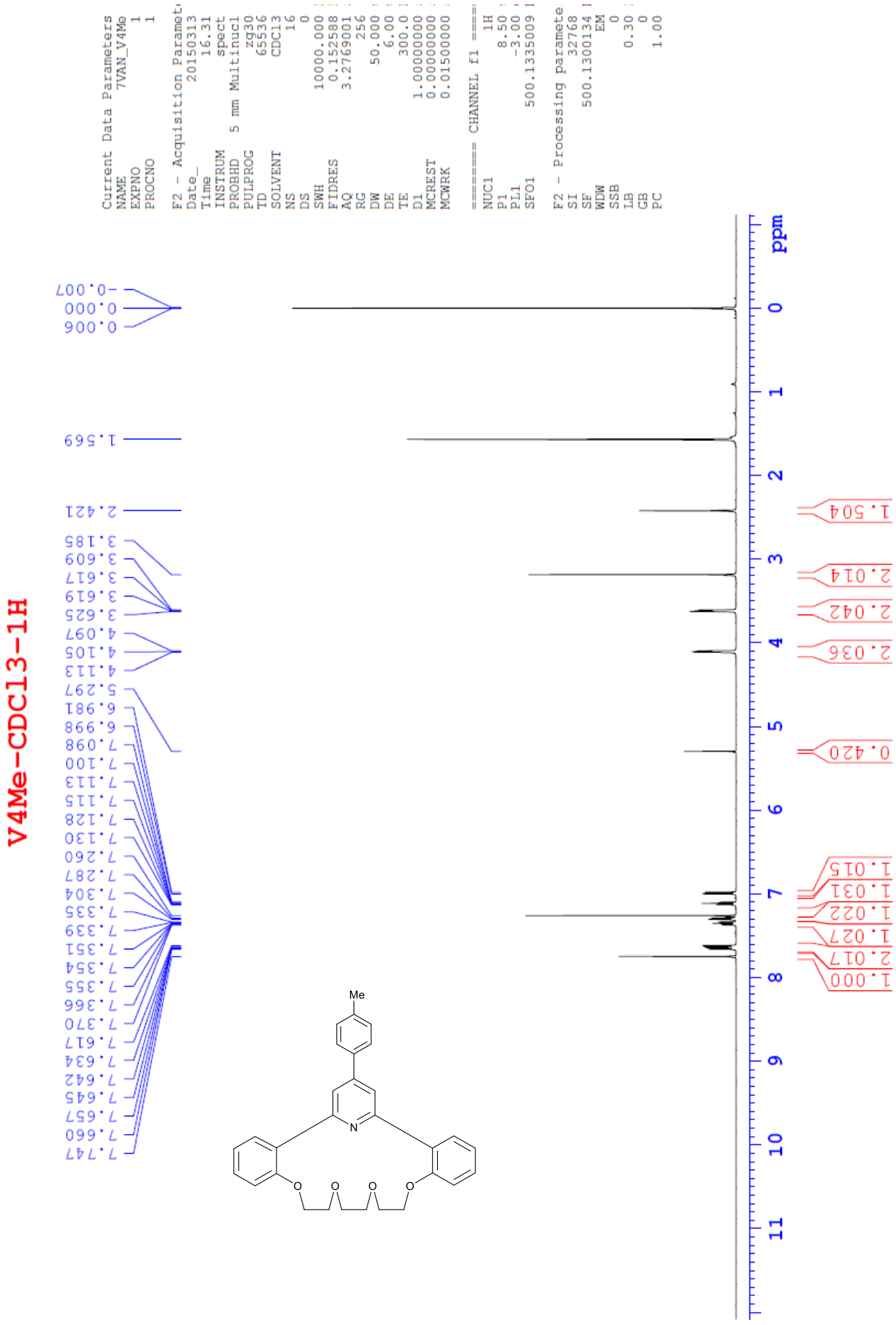

Keton4+4OMe-CDCl3-HSQC
ppm
35
40
45
50
55
60
65
70
75
80
85
90
95
100
105
110
115
120
125
130
135
140
145
150
8
7
6
5
4
3
2 ppm
OMe
N

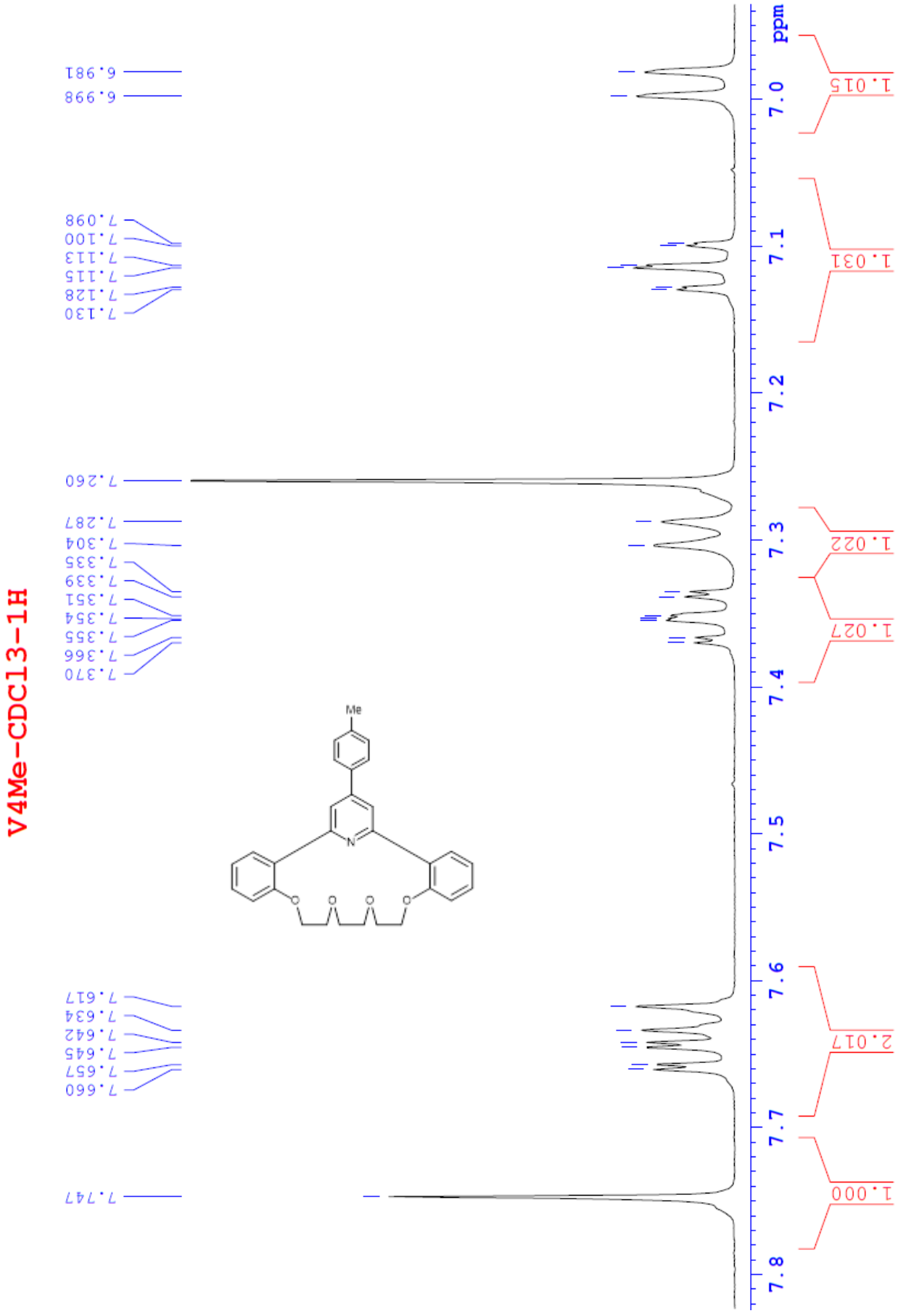

Keton4+4OMe-CDCl3-HSQC
ppm
112
113
114
115
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
131
132
133
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
ppm
OMe
N
O
O
O
O

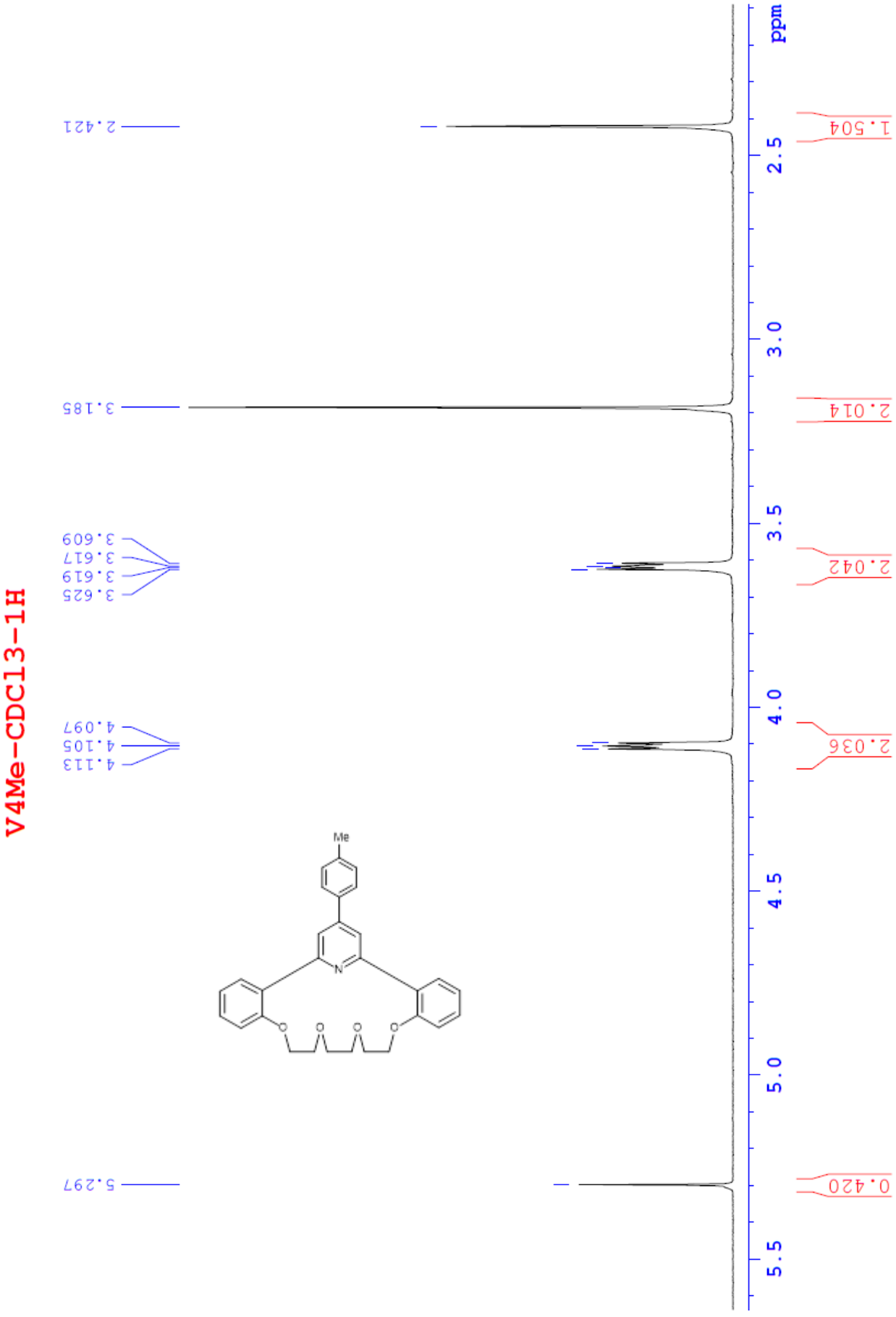

Keton4+4OMe-CDCl3-HSQC
ppm
54
55
56
57
58
59
60
61
62
63
64
65
66
67
68
69
70
71
72
73
74
OMe
N
O
O
O
O
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
ppm

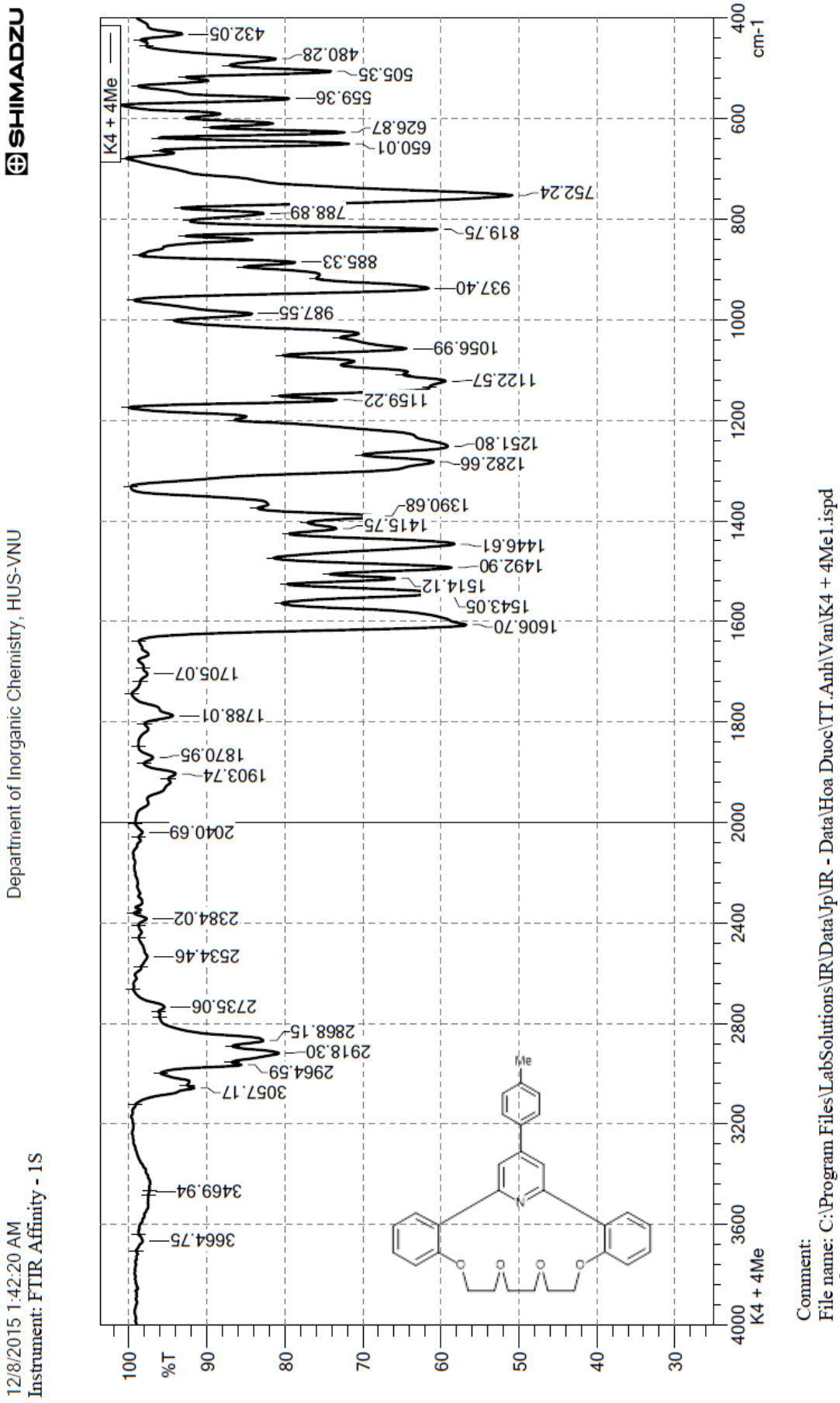

SHIMADZU
Department of Inorganic Chemistry, HUS-VNU
12/8/2015 1:48:43 AM
Instrument: FTIR Affinity - 1S
K4 + 40Me
%T
cm-1
3452.58
3059.10
2966.52
2920.23
2870.08
2845.00
2536.39
2432.24
2283.72
2042.62
1944.25
1907.60
1774.51
1712.79
1658.78
1606.70
1544.98
1514.12
1492.90
1444.68
1394.53
1288.45
1255.66
1182.36
1120.64
1058.92
1029.99
985.62
931.62
883.40
837.11
756.10
648.08
626.87
572.86
528.50
480.28
437.84
OMe
N
Comment:
File name: C:\Program Files\LabSolutions\IR\Data\Jp\IR - Data\Hoa Duoc\TT.Anh\Van\K4 + 40Me1.ispd
No of Scans: 20
Intensity Mode: %Transmittance
Min: 400 cm-1
Max: 4000 cm-1
Resolution: 4 [cm-1]
Atmosphere Correction: OFF

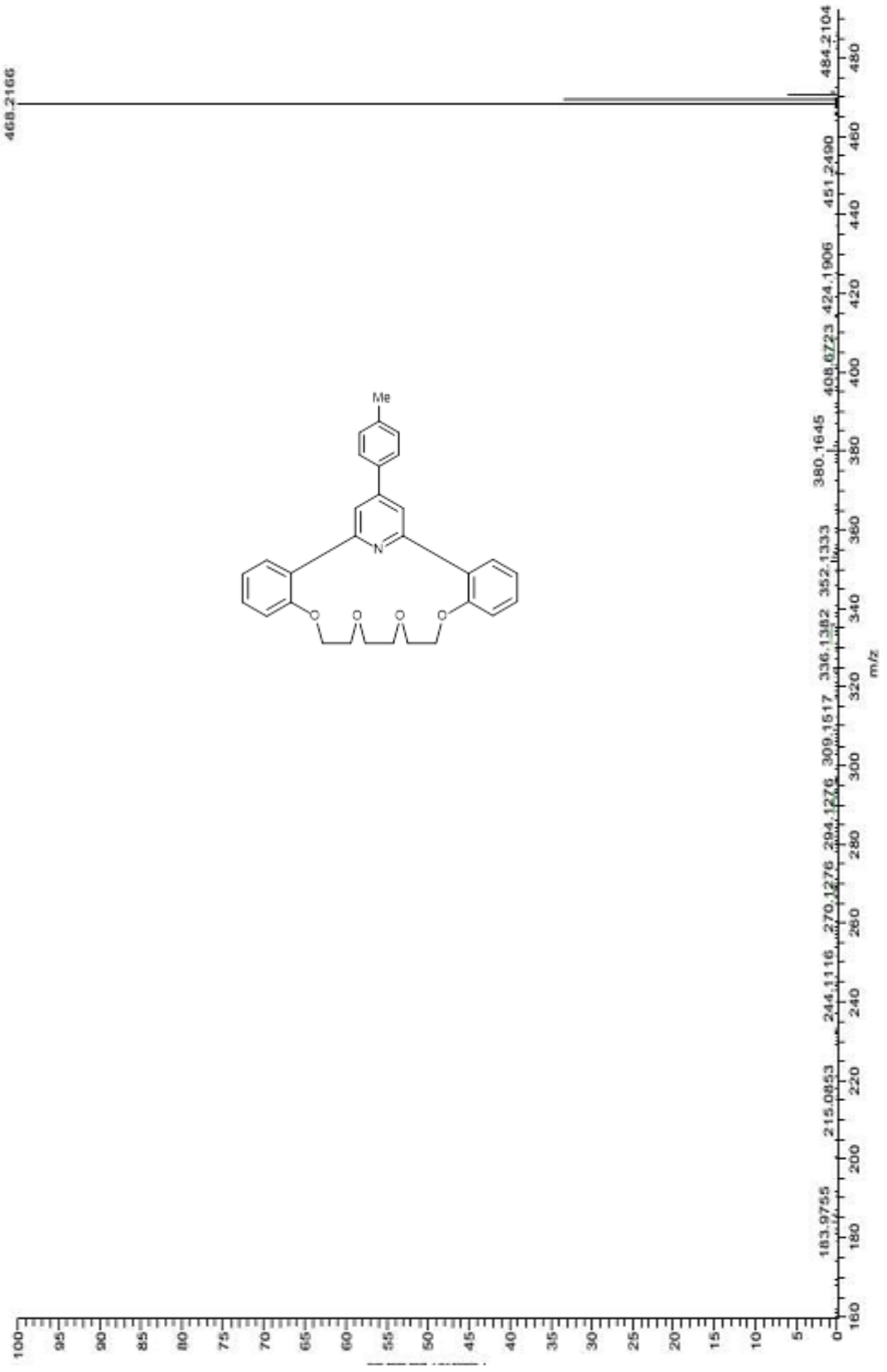

M1 _151210164833 #140 RT: 1.18 AV: 1 NL: 2.68E6
T: FTMS + c ESI Full ms [150.00-700.00]
Relative Abundance
m/z
484.2105
469.2579
451.2478
419.2204
396.1213
365.1351
360.0496
353.9304
329.2104
297.1844
271.5664
244.9566
203.9302
183.9752
157.5861
OMe
N
O

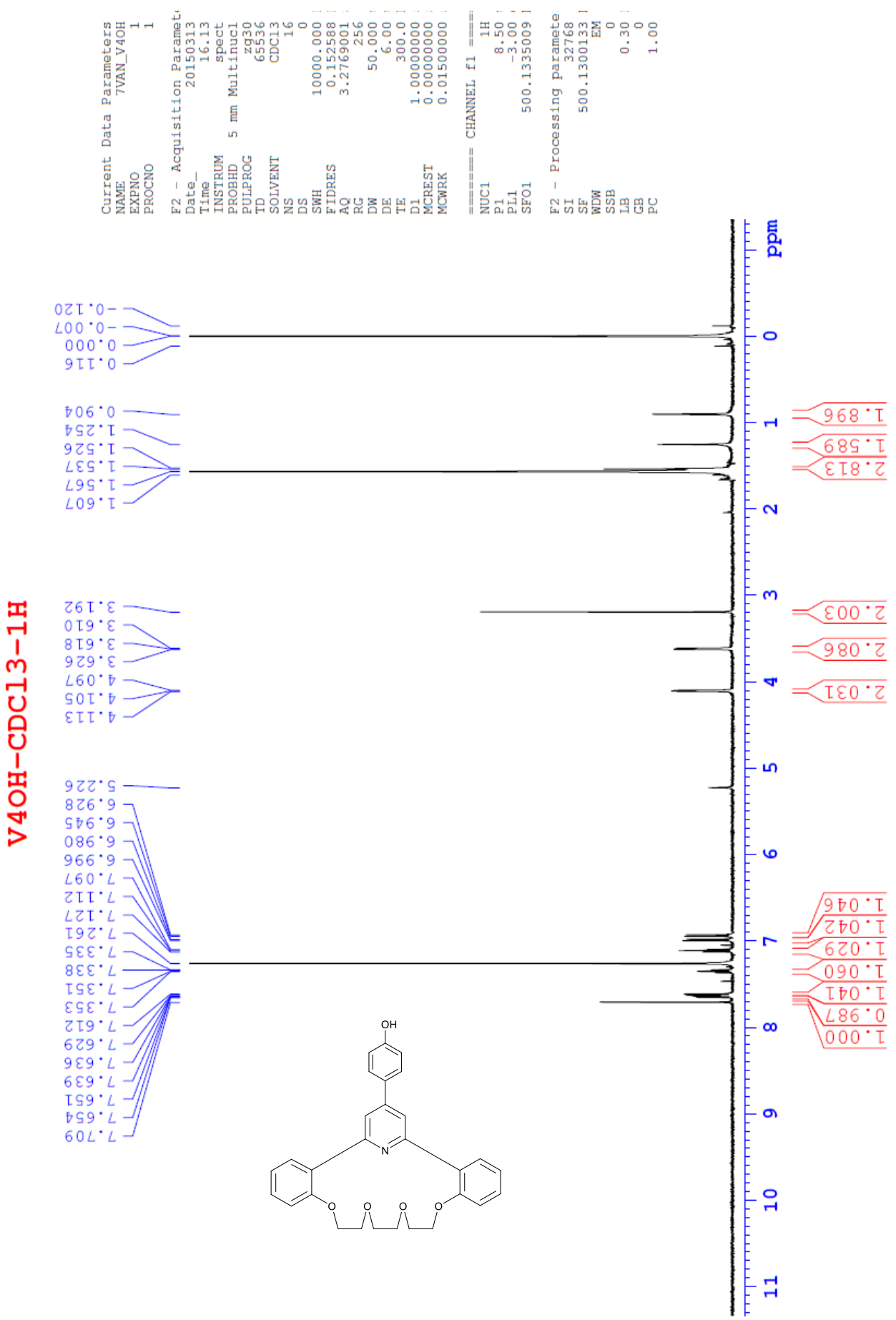

V4Me-CDC13-1H
Current Data Parameters
NAME 7VAN_V4Me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150313
Time 16.31
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2769001
RG 256
DW 50.000
DE 6.00
TE 300.0
D1 1.00000000
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ========
NUC1 1H
P1 8.50
PL1 -3.00
SFO1 500.1335009
F2 - Processing parameters
SI 32768
SF 500.1300134
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
ppm
0 1 2 3 4 5 6 7 8 9 10 11
-0.007
0.000
0.006
1.569
2.421
3.185
3.609
3.617
3.619
3.625
4.097
4.105
4.113
5.297
6.981
6.998
7.098
7.100
7.113
7.115
7.128
7.130
7.260
7.287
7.304
7.335
7.339
7.351
7.354
7.355
7.366
7.370
7.617
7.634
7.642
7.645
7.657
7.660
7.747
1.504
2.014
2.042
2.036
0.420
1.015
1.031
1.022
1.027
2.017
1.000
Me
N
O
O
O
O

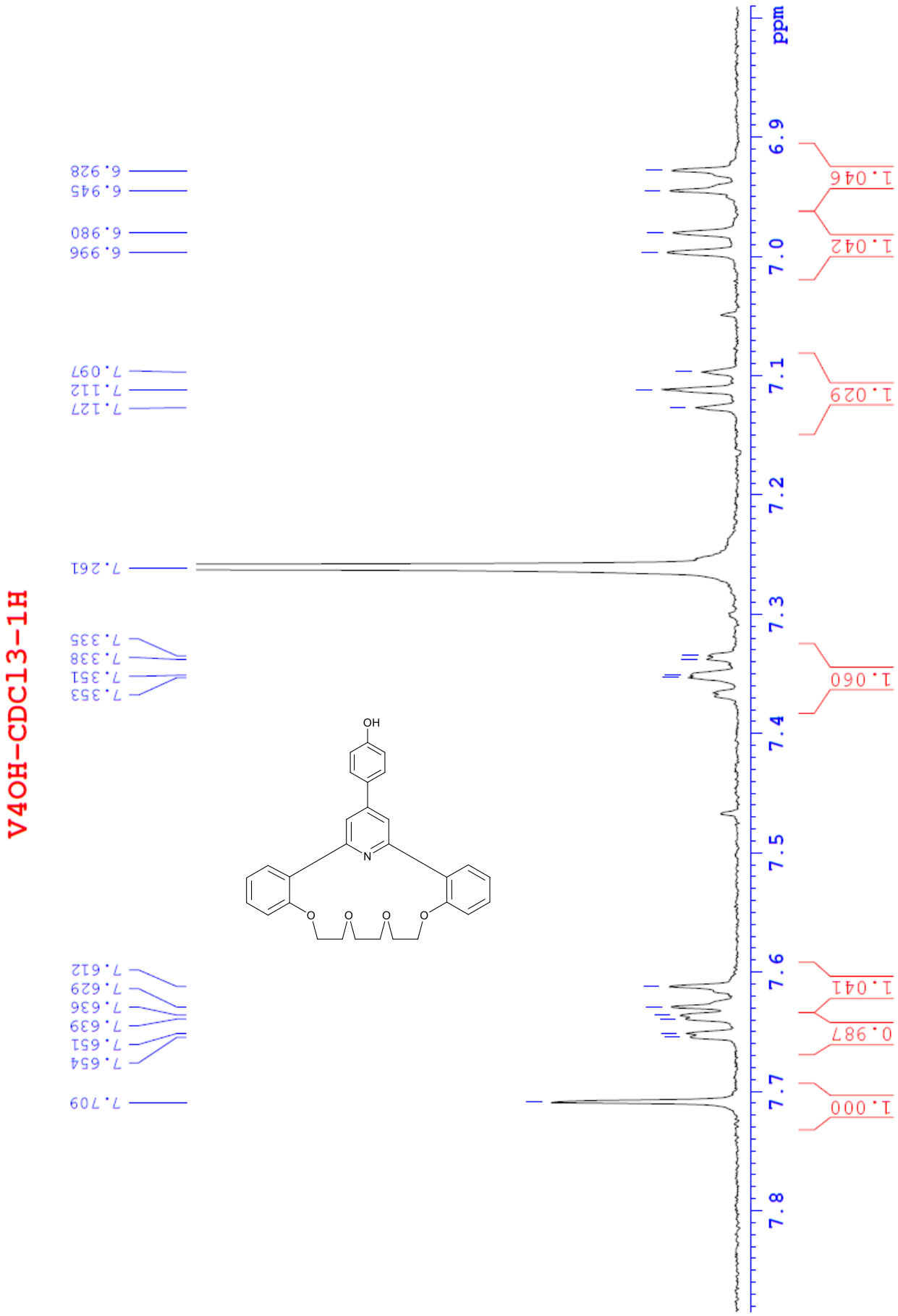

V4Me-CDCl3-1H
ppm
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
1.015
1.031
1.022
1.027
2.017
1.000
6.981
6.998
7.098
7.100
7.113
7.115
7.128
7.130
7.260
7.287
7.304
7.335
7.339
7.351
7.354
7.355
7.366
7.370
7.617
7.634
7.642
7.645
7.657
7.660
7.747
Me
N
O

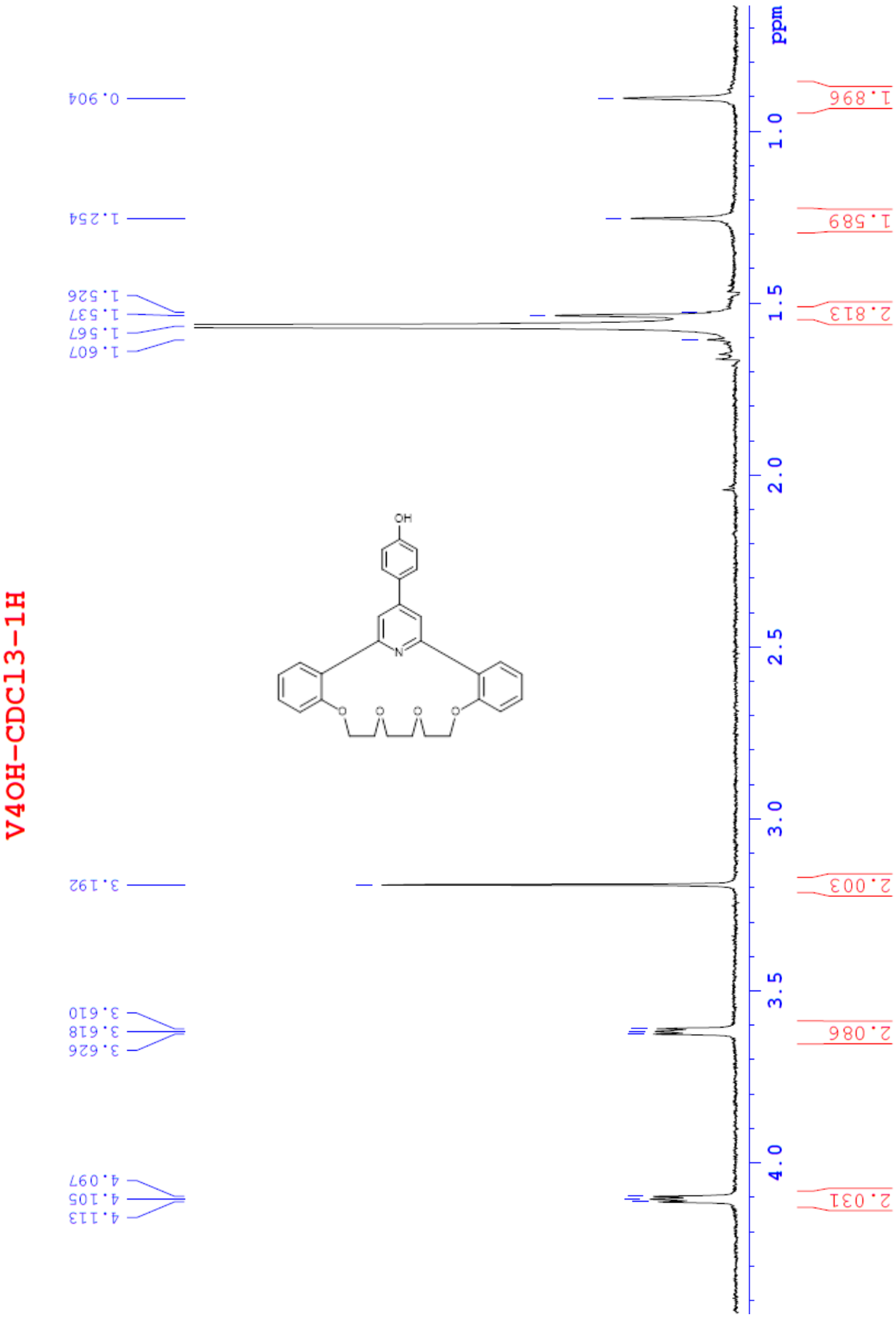

V4Me-CDCl3-1H
ppm
2.5
3.0
3.5
4.0
4.5
5.0
5.5
2.421
3.185
3.609
3.617
3.619
3.625
4.097
4.105
4.113
5.297
1.504
2.014
2.042
2.036
0.420
Me
N
O
O
O
O

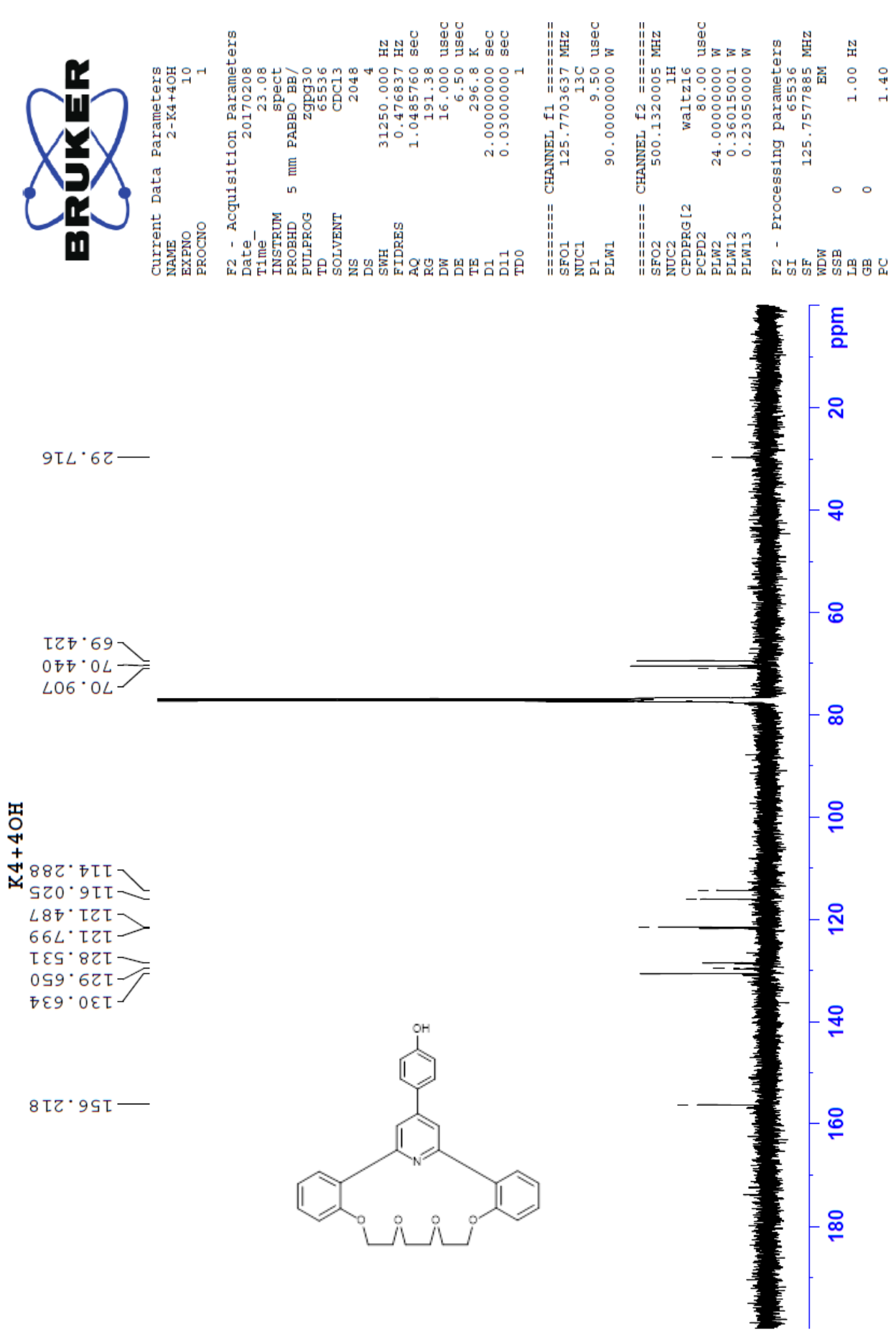

SHIMADZU
Department of Inorganic Chemistry, HUS-VNU
12/8/2015 1:42:20 AM
Instrument: FTIR Affinity - 1S
K4 + 4Me
%T
cm-1
K4 + 4Me
Me
N
3664.75
3469.94
3057.17
2964.59
2918.30
2868.15
2735.06
2534.46
2384.02
2040.69
1903.74
1870.95
1788.01
1705.07
1606.70
1543.05
1514.12
1492.90
1446.61
1415.75
1390.68
1282.66
1251.80
1159.22
1122.57
1056.99
987.55
937.40
885.33
819.75
788.89
752.24
650.01
626.87
559.36
505.35
480.28
432.05
Comment:
File name: C:\Program Files\LabSolutions\IR\Data\Jp\IR - Data\Hoa Duoc\TT.Anh\Van\K4 + 4Me1.ispd
No of Scans: 20
Intensity Mode: %Transmittance
Min: 400 cm-1
Max: 4000 cm-1
Resolution: 4 [cm-1]
Atmosphere Correction: OFF

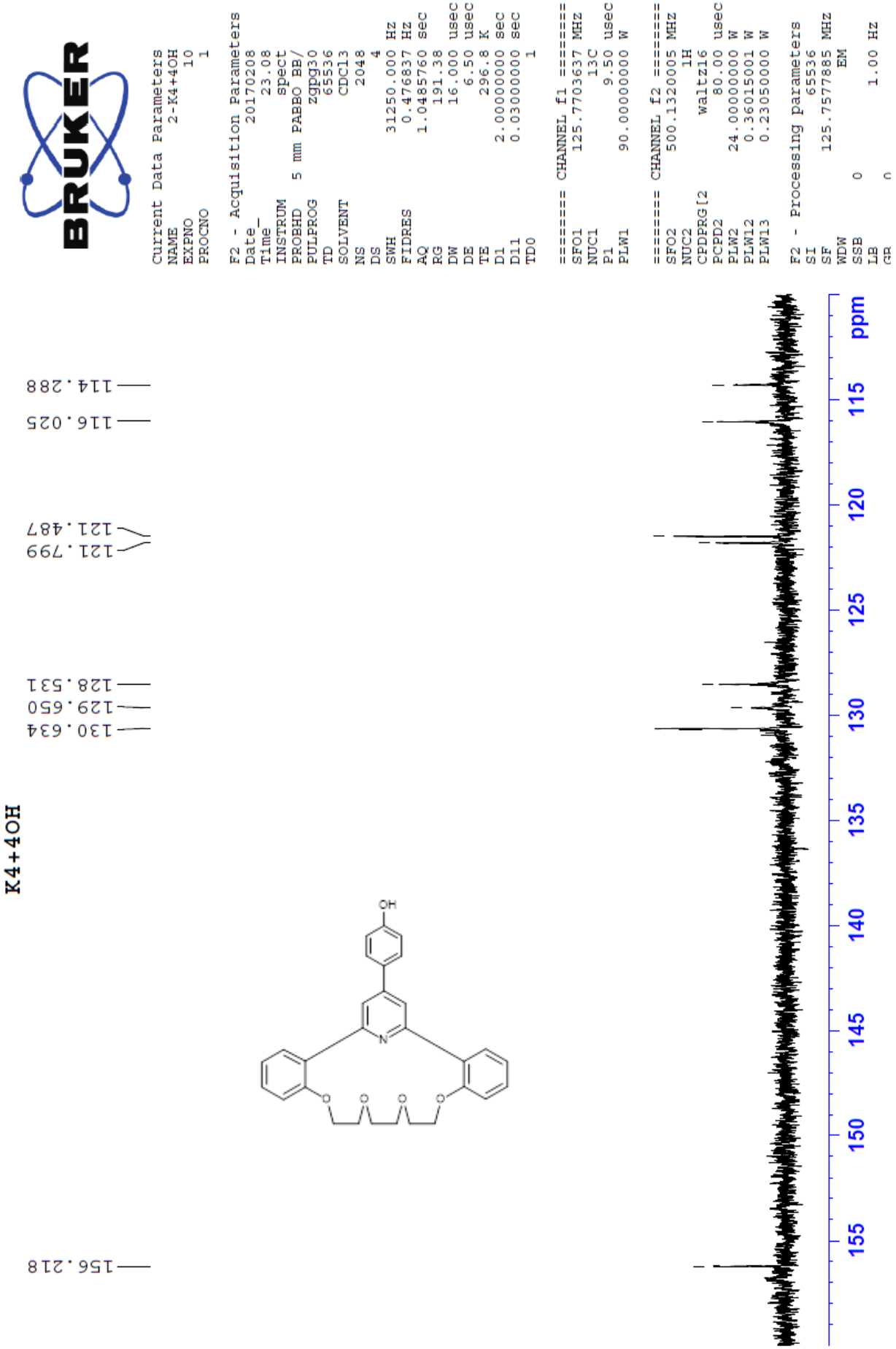

Me
N
O
O
O
O
468.2166
484.2104
451.2490
424.1906
408.6723
380.1645
352.1333
336.1382
309.1517
294.1276
270.1276
244.1116
215.0853
183.9755
m/z

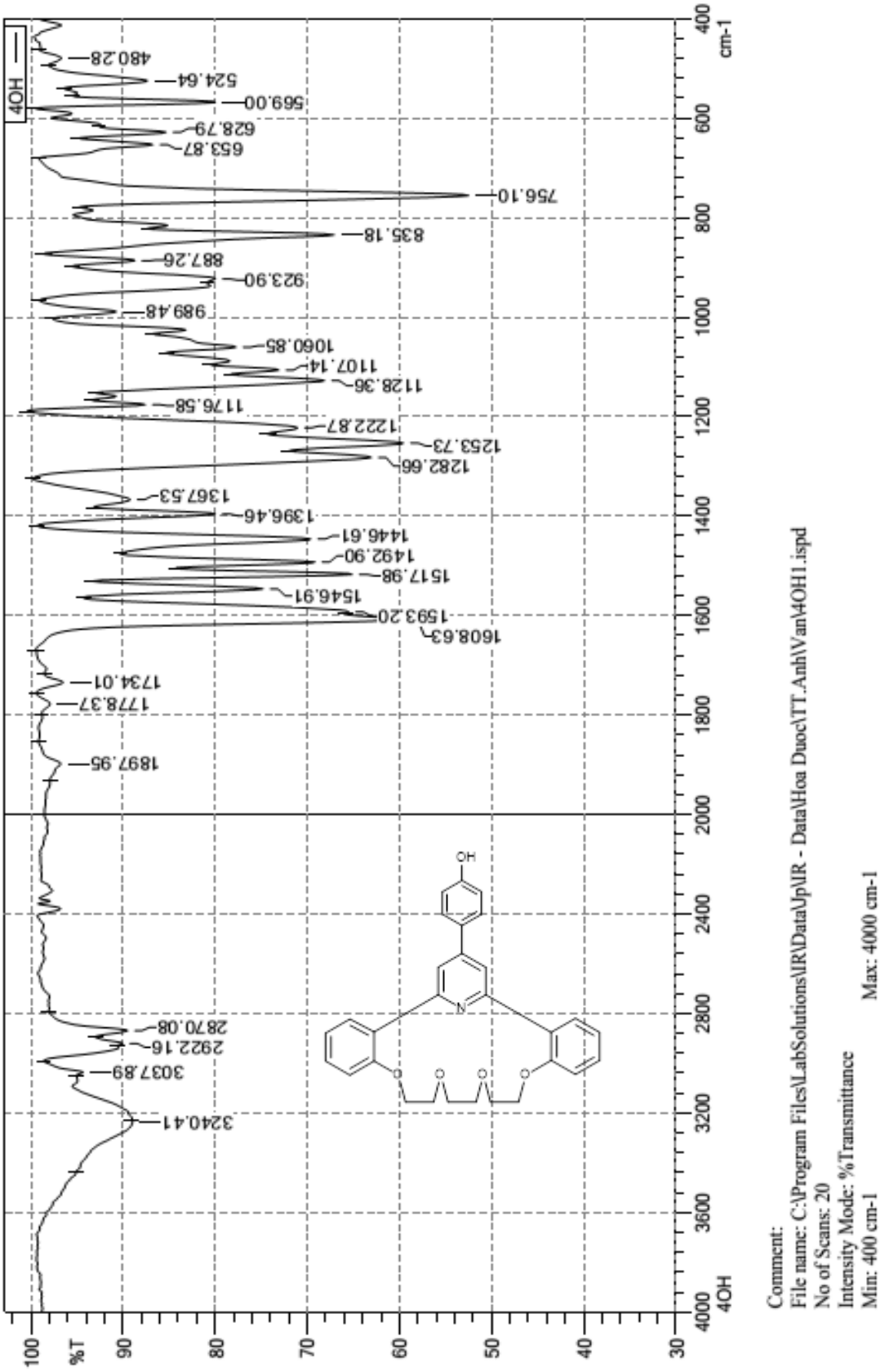

V4OH-CDCl3-1H
ppm
OH
N
O

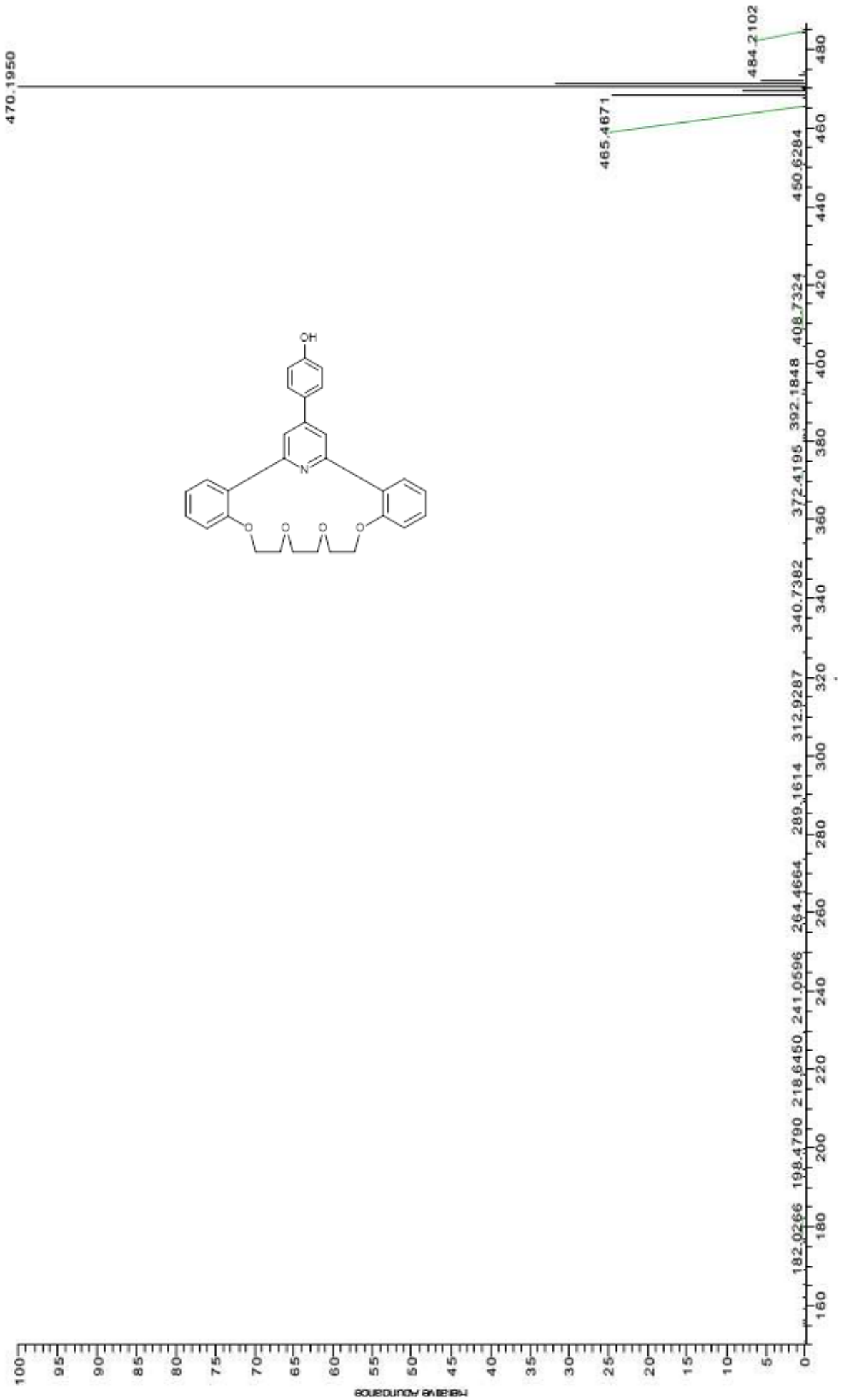

V4OH-CDCl3-1H
ppm
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
6.928
6.945
6.980
6.996
7.097
7.112
7.127
7.261
7.335
7.338
7.351
7.353
7.612
7.629
7.636
7.639
7.651
7.654
7.709
1.046
1.042
1.029
1.060
1.041
0.987
1.000
OH
N
O
O
O
O

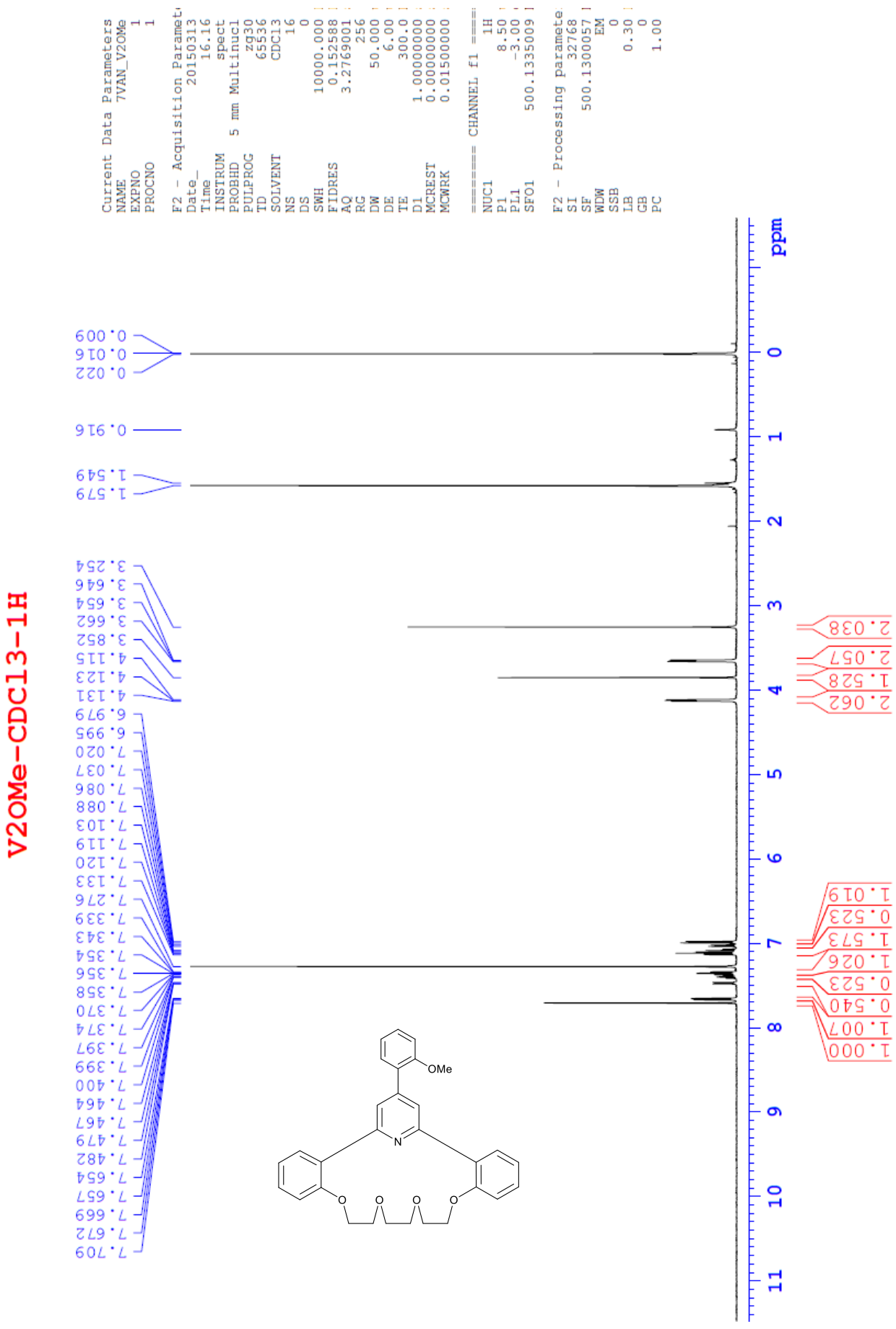

V4OH-CDCl3-1H
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
0.904
1.254
1.526
1.537
1.567
1.607
3.192
3.610
3.618
3.626
4.097
4.105
4.113
1.896
1.589
2.813
2.003
2.086
2.031
OH
N
O

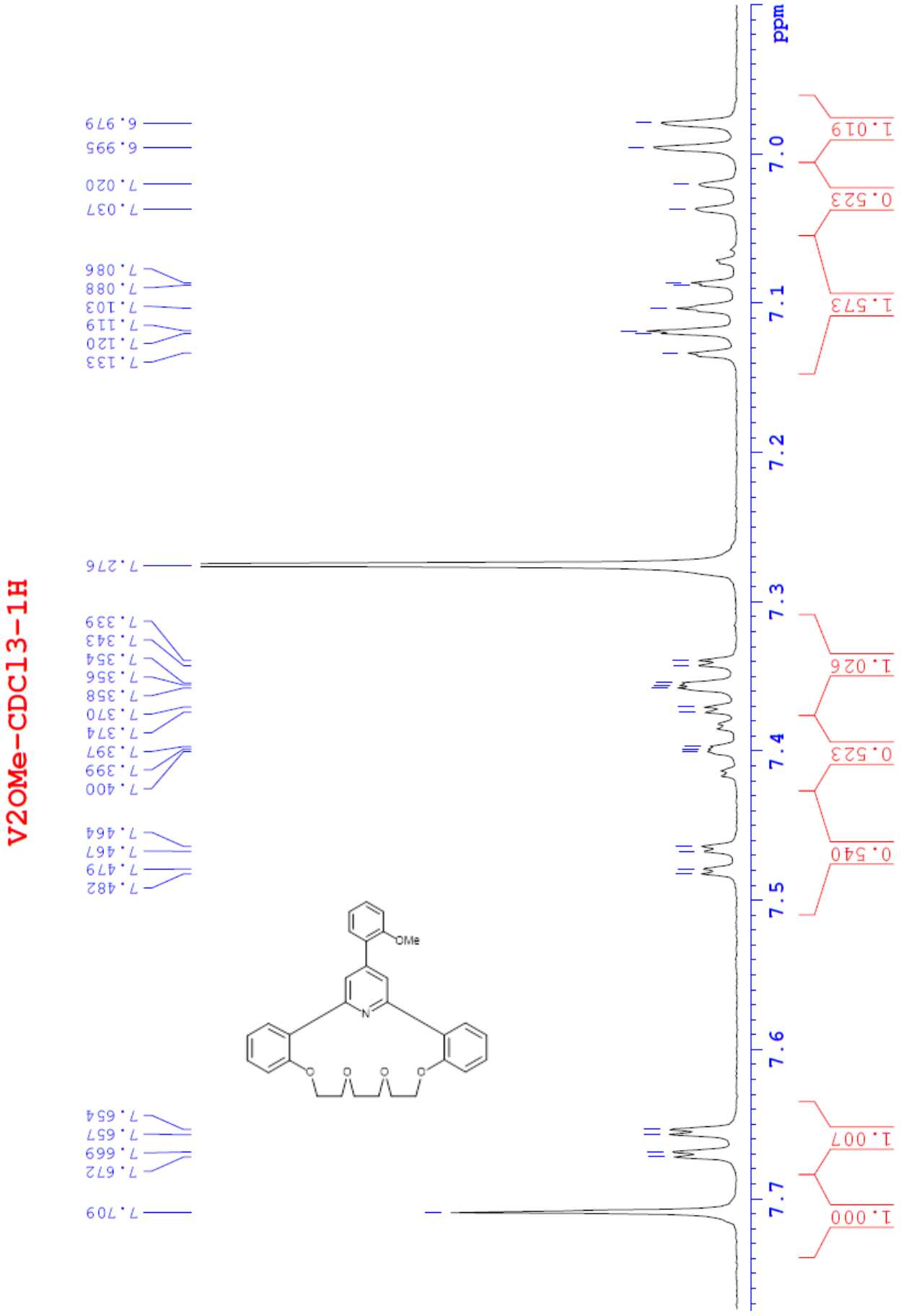

Current Data Parameters
NAME 2-K4+4OH
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170208
Time 23.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.36015001 W
PLW13 0.23050000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
K4+4OH
29.716
69.421
70.440
70.907
114.288
116.025
121.487
121.799
128.531
129.650
130.634
156.218
OH
N
O
ppm
20
40
60
80
100
120
140
160
180

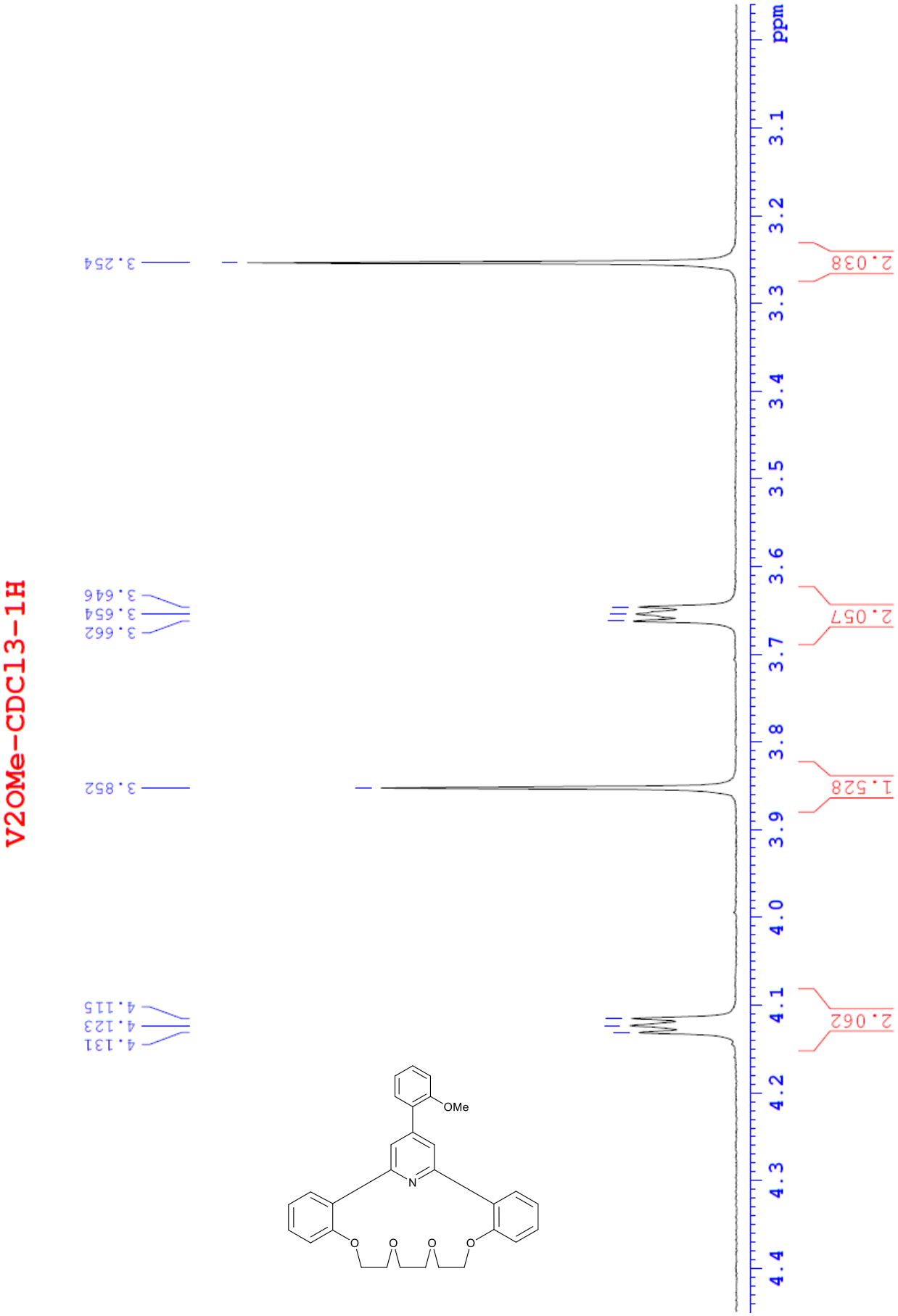

Current Data Parameters
NAME 2-K4+4OH
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170208
Time_ 23.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.04857600 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.36015001 W
PLW13 0.23050000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
K4+4OH
156.218
130.634
129.650
128.531
121.799
121.487
116.025
114.288
ppm
155
150
145
140
135
130
125
120
115
OH
N
O
O
O
O

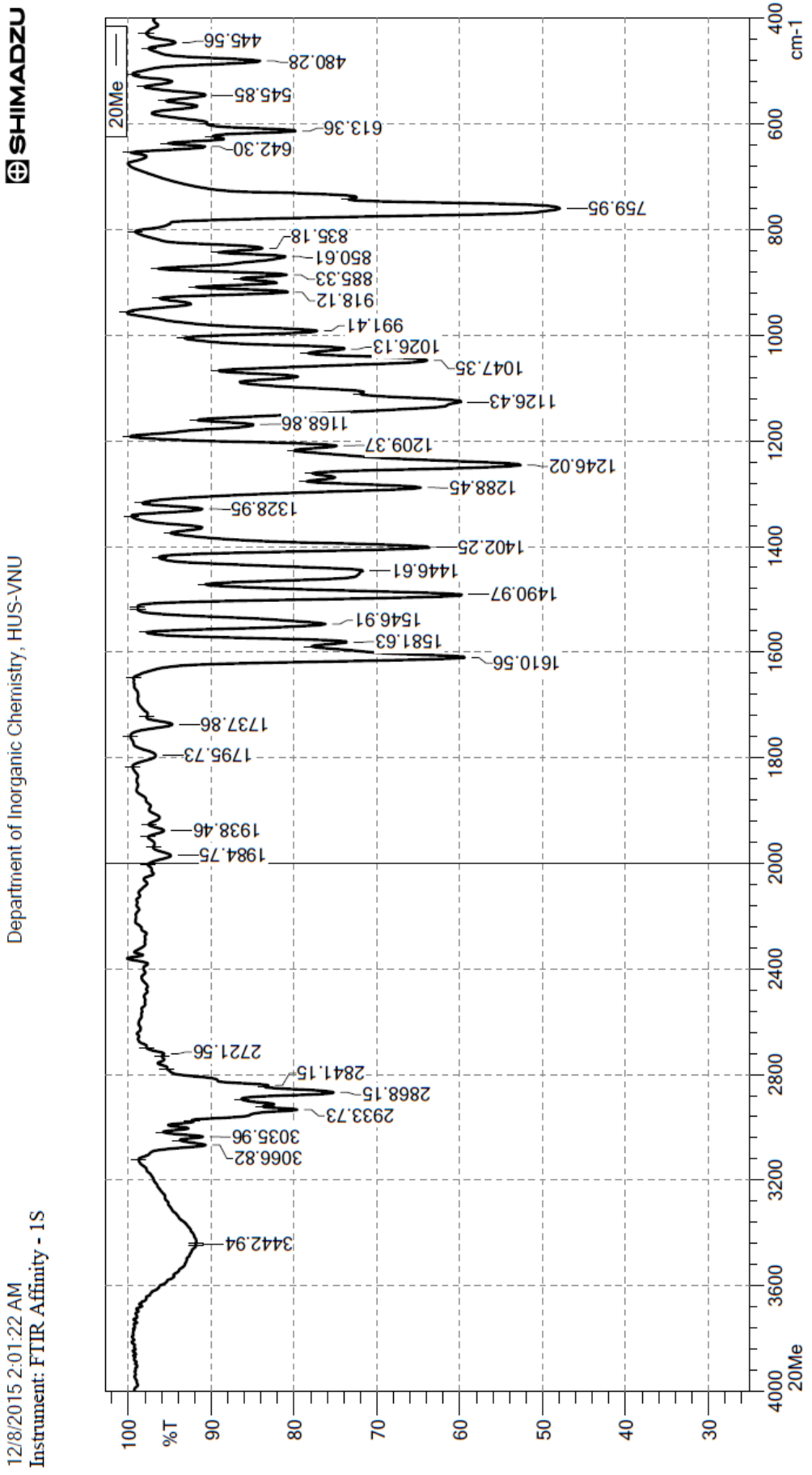

4OH
480.28
524.64
569.00
628.79
653.87
756.10
835.18
887.26
923.90
989.48
1060.85
1107.14
1128.36
1176.58
1222.87
1253.73
1282.66
1367.53
1396.46
1446.61
1492.90
1517.98
1546.91
1593.20
1608.63
1734.01
1778.37
1897.95
2870.08
2922.16
3037.89
3240.41
%T
cm-1
4OH
OH
N
O
Comment:
File name: C:\Program Files\LabSolutions\IR\DataUp\IR - DataHoa Duoc\TT.AnhVan\4OH1.ispd
No of Scans: 20
Intensity Mode: %Transmittance
Min: 400 cm-1
Max: 4000 cm-1

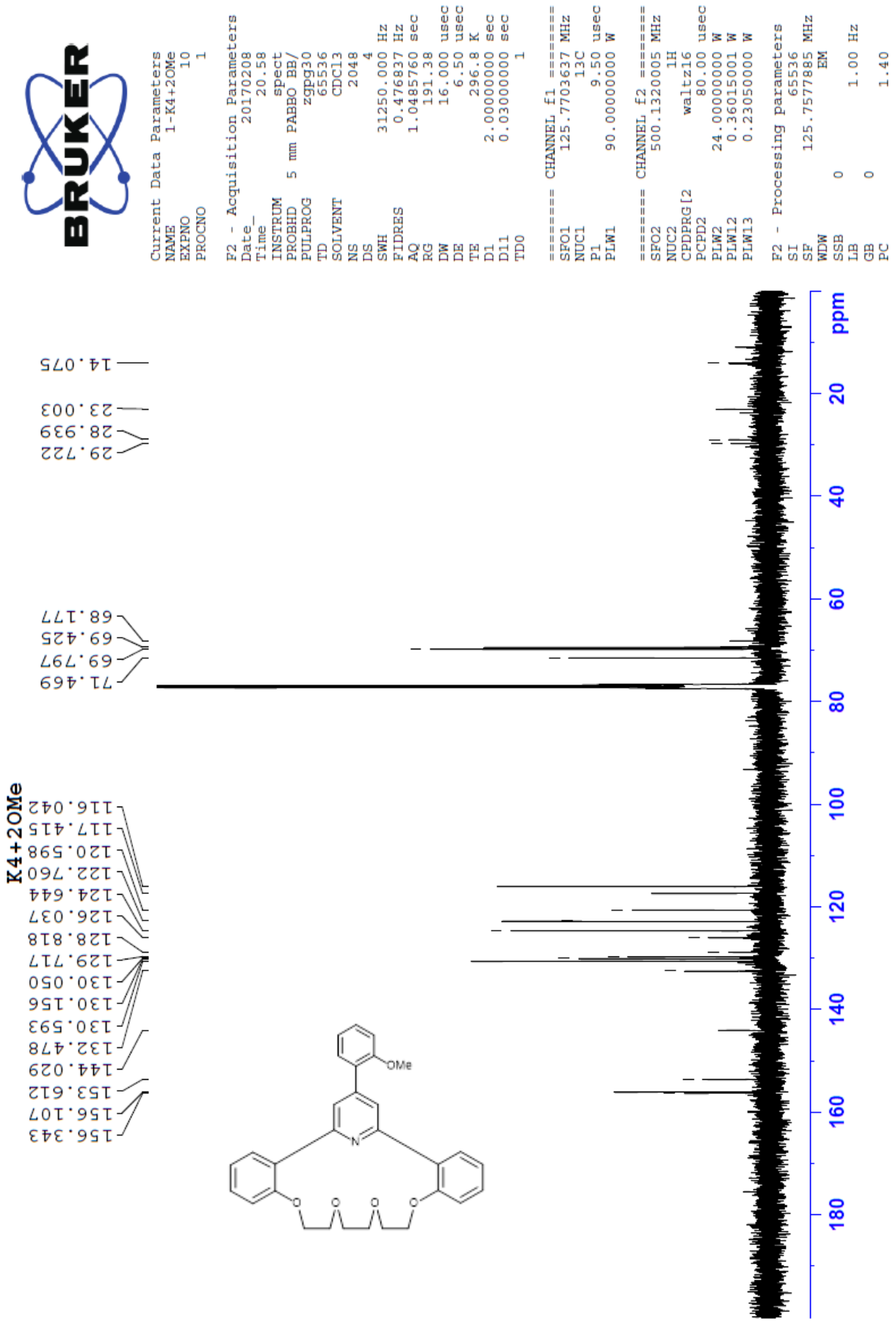

470.1950
484.2102
465.4671
450.6284
408.7324
392.1848
372.4195
340.7382
312.9287
289.1614
264.4664
241.0596
218.6450
198.4790
182.0266
OH
N
O
O
O
O

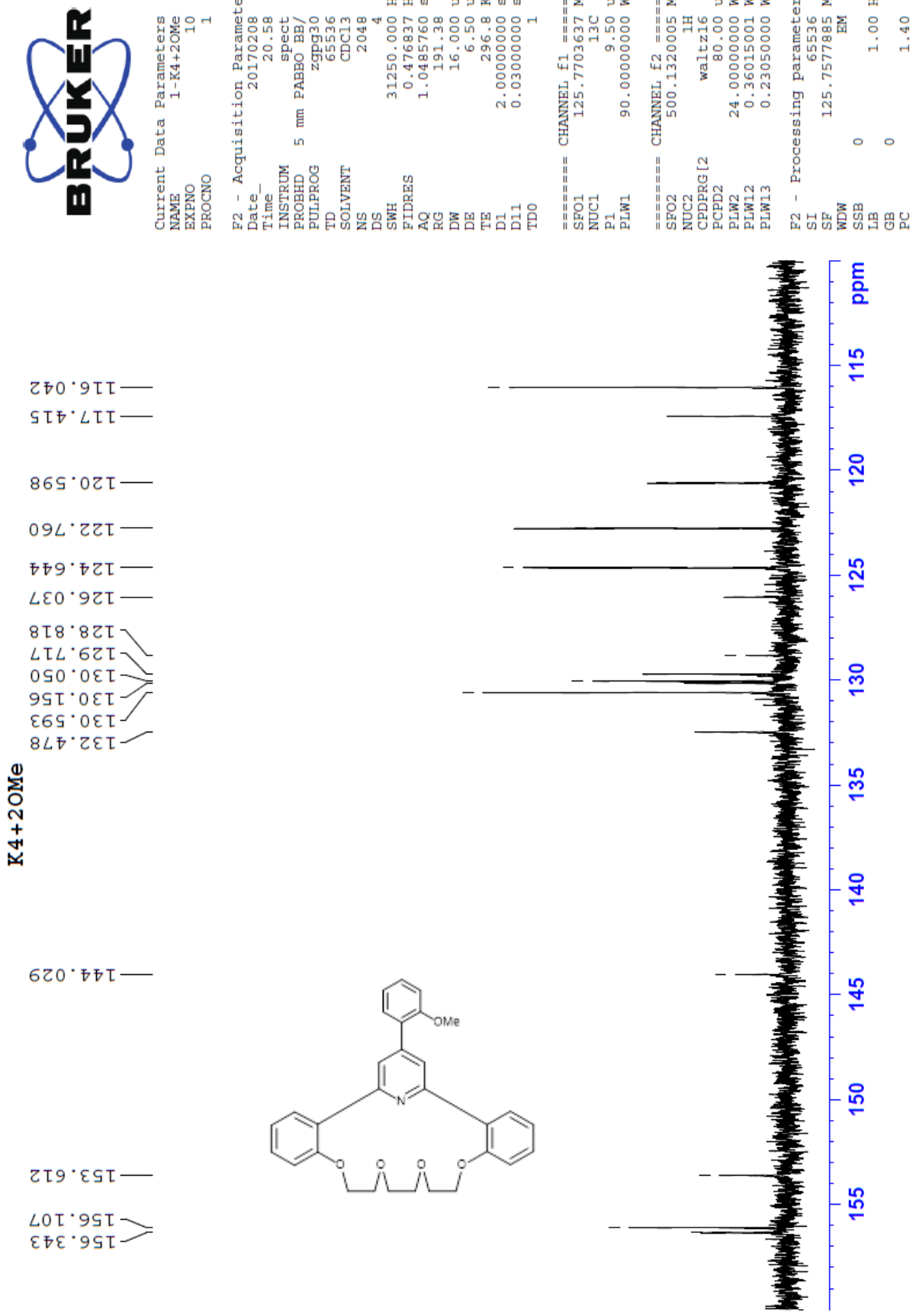

V2OMe-CDC13-1H
Current Data Parameters
NAME 7VAN_V2OMe
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150313
Time 16.16
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2769001
RG 256
DW 50.000
DE 6.00
TE 300.0
D1 1.00000000
MCREST 0.00000000
MCWRK 0.01500000
CHANNEL f1
NUC1 1H
P1 8.50
PL1 -3.00
SFO1 500.1335009
F2 - Processing parameters
SI 32768
SF 500.1300057
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
ppm
OMe
N
O
O
O
O

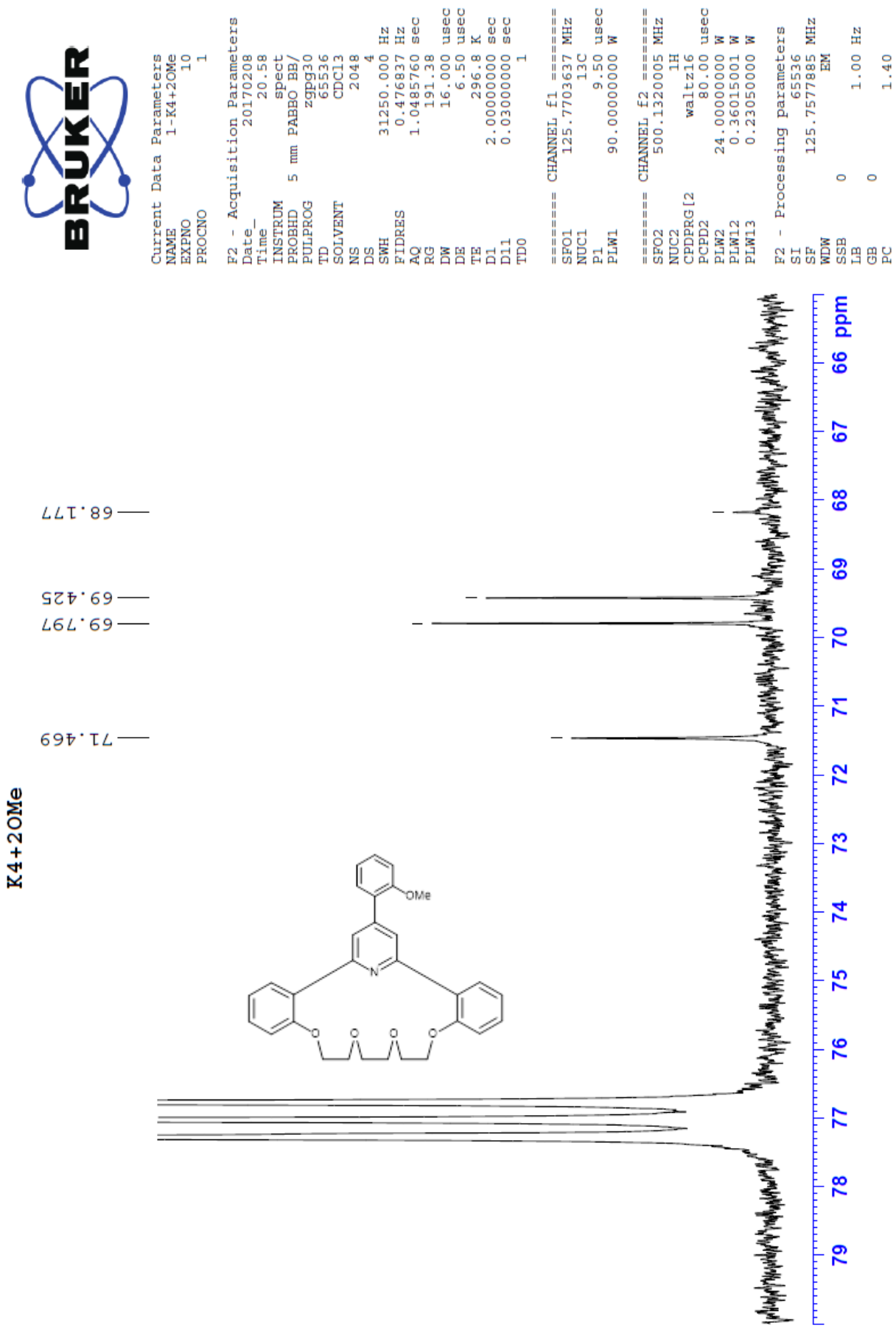

V2OMe-CDCl3-1H
ppm
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
1.019
0.523
1.573
1.026
0.523
0.540
1.007
1.000
6.979
6.995
7.020
7.037
7.086
7.088
7.103
7.119
7.120
7.133
7.276
7.339
7.343
7.354
7.356
7.358
7.370
7.374
7.397
7.399
7.400
7.464
7.467
7.479
7.482
7.654
7.657
7.669
7.672
7.709
OMe
N
O
O
O
O

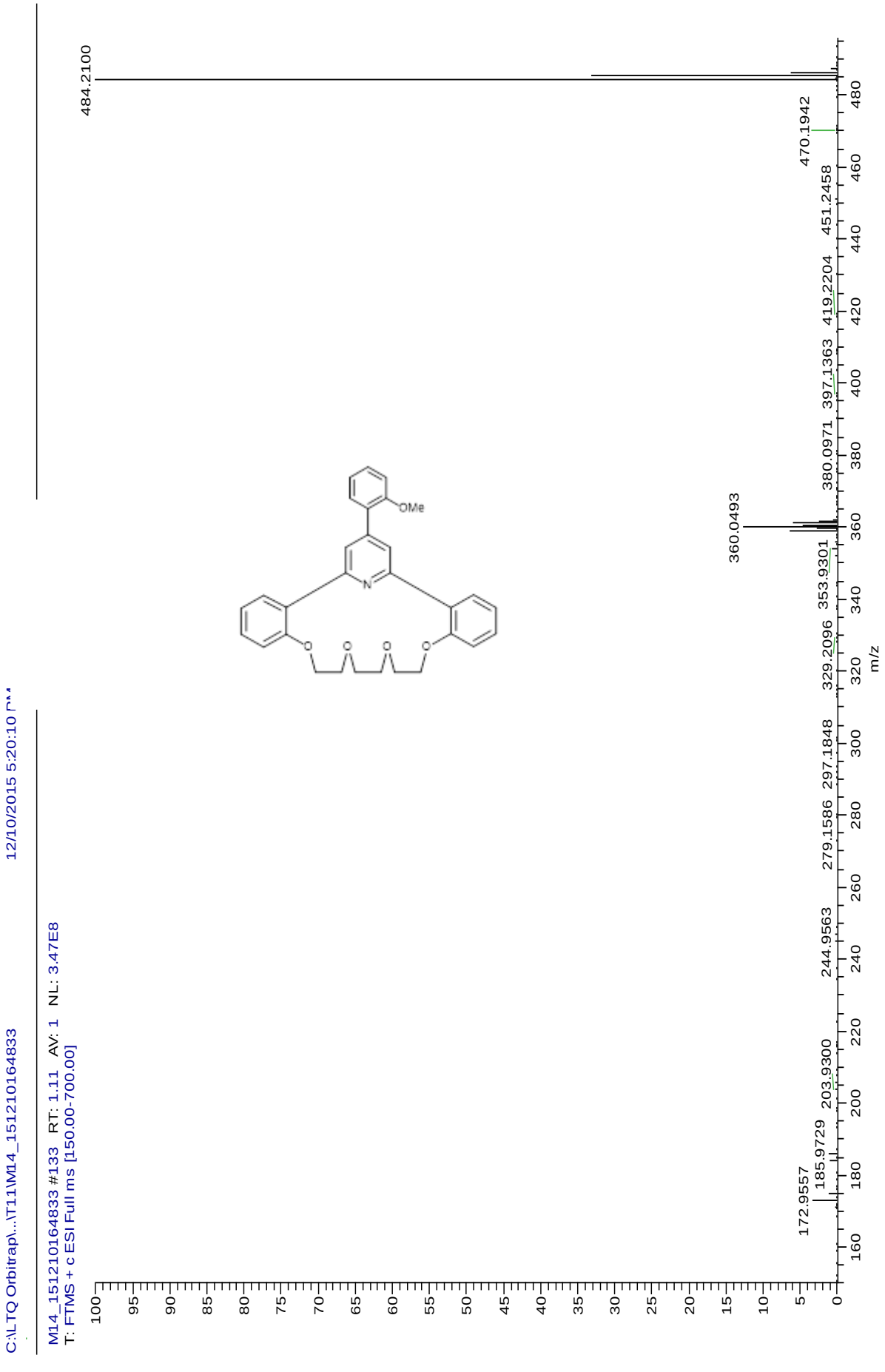

V2OMe-CDCl3-1H
ppm
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
4.1
4.2
4.3
4.4
3.254
3.646
3.654
3.662
3.852
4.115
4.123
4.131
2.038
2.057
1.528
2.062
OMe
N
O
O
O
O

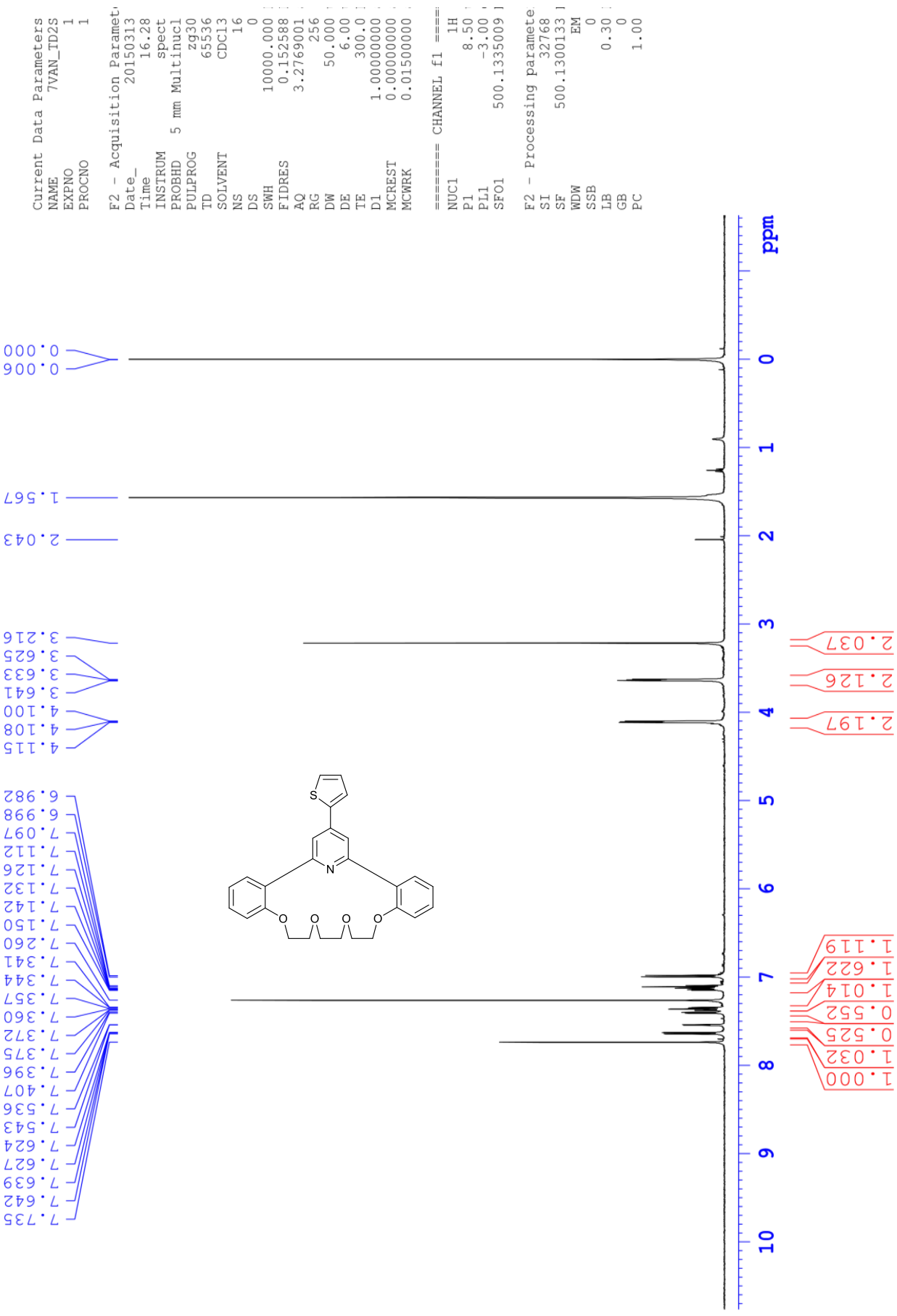

SHIMADZU
Department of Inorganic Chemistry, HUS-VNU
12/8/2015 2:01:22 AM
Instrument: FTIR Affinity - 1S
20Me
%T
cm-1
445.56
480.28
545.85
613.36
642.30
759.95
835.18
850.61
885.33
918.12
991.41
1026.13
1047.35
1126.43
1168.86
1209.37
1246.02
1288.45
1328.95
1402.25
1446.61
1490.97
1546.91
1581.63
1610.56
1737.86
1795.73
1938.46
1984.75
2721.56
2841.15
2868.15
2933.73
3035.96
3066.82
3442.94
Comment:
File name: C:\Program Files\LabSolutions\IR\Data\Jp\IR - Data\Hoa Duoc\TT.Anh\Van\20Me1.ispd
No of Scans: 20
Intensity Mode: %Transmittance
Max: 4000 cm-1
Min: 400 cm-1
Resolution: 4 [cm-1]
Atmosphere Correction: OFF

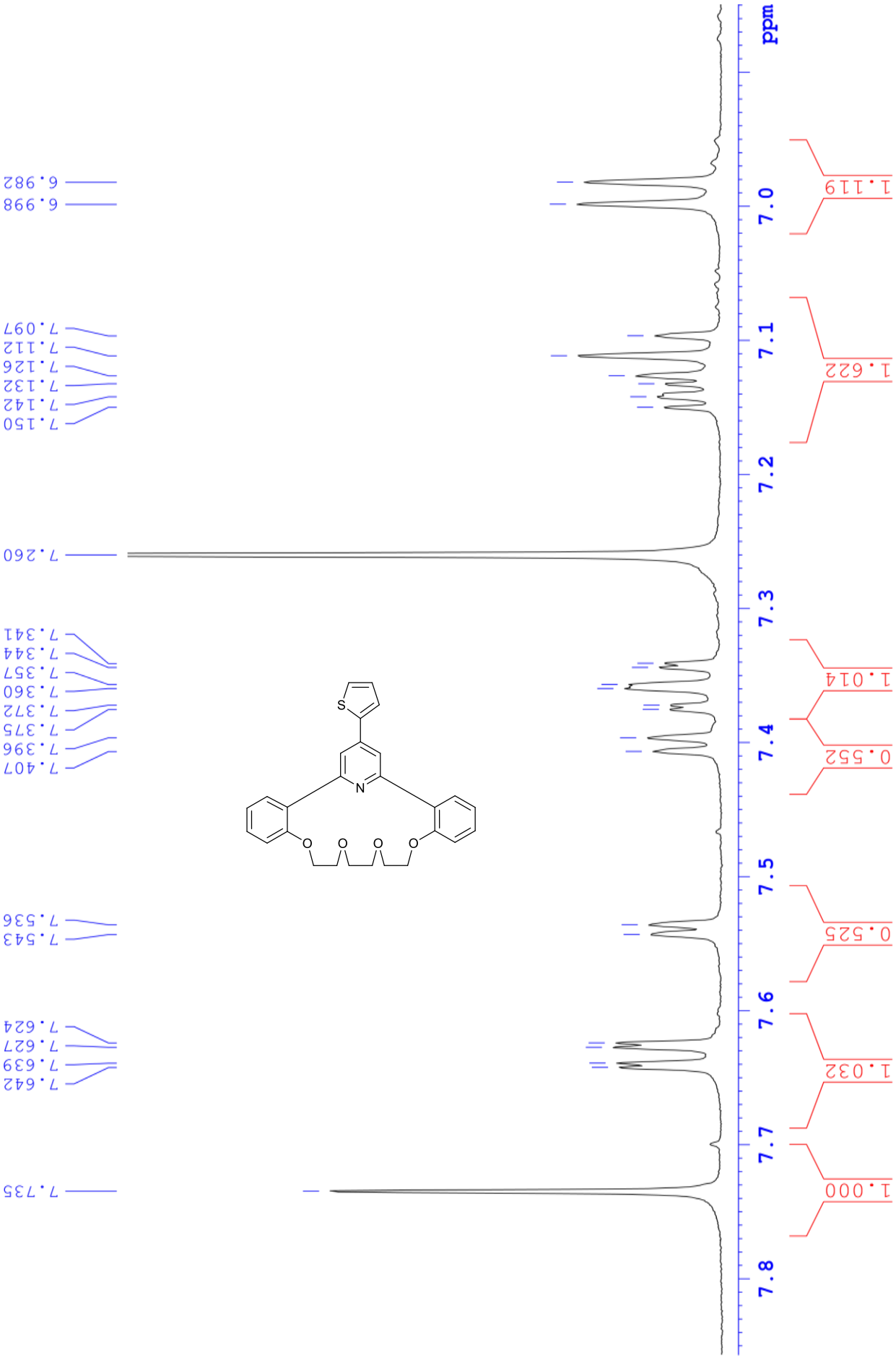

Current Data Parameters
NAME 1-K4+2OMe
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170208
Time_ 20.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2048
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.36015001 W
PLW13 0.23050000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
K4+2OMe
156.343
156.107
153.612
144.029
132.478
130.593
130.156
130.050
129.717
128.818
126.037
124.644
122.760
120.598
117.415
116.042
71.469
69.797
69.425
68.177
29.722
28.939
23.003
14.075
OMe
N
O
ppm
20
40
60
80
100
120
140
160
180

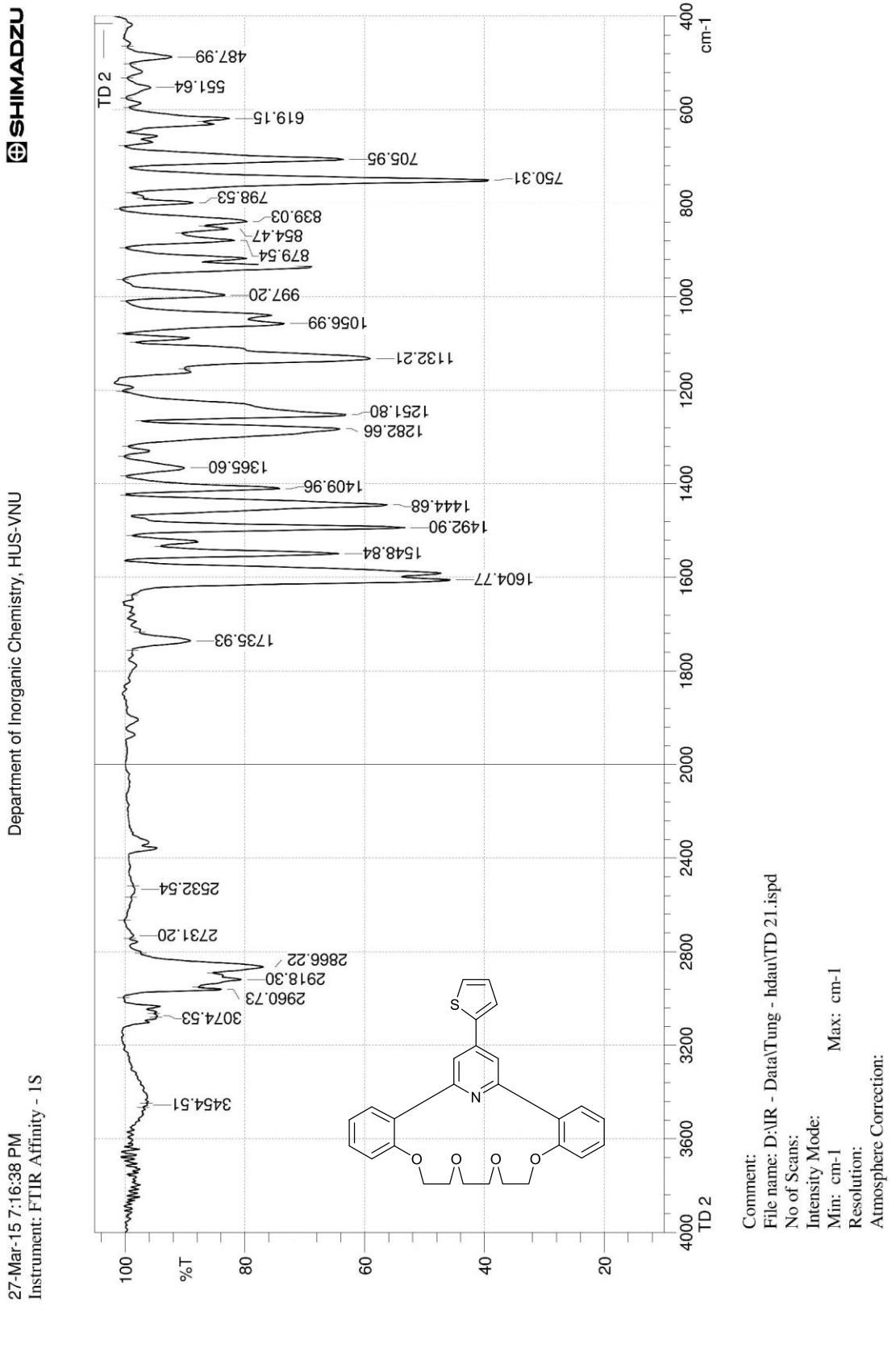

Current Data Parameters
NAME 1-K4+2OMe
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170208
Time 20.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2048
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.36015001 W
PLW13 0.23050000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
K4+2OMe
116.042
117.415
120.598
122.760
124.644
126.037
128.818
129.717
130.050
130.156
130.593
132.478
144.029
153.612
156.107
156.343
OMe
N
O
ppm
115
120
125
130
135
140
145
150
155

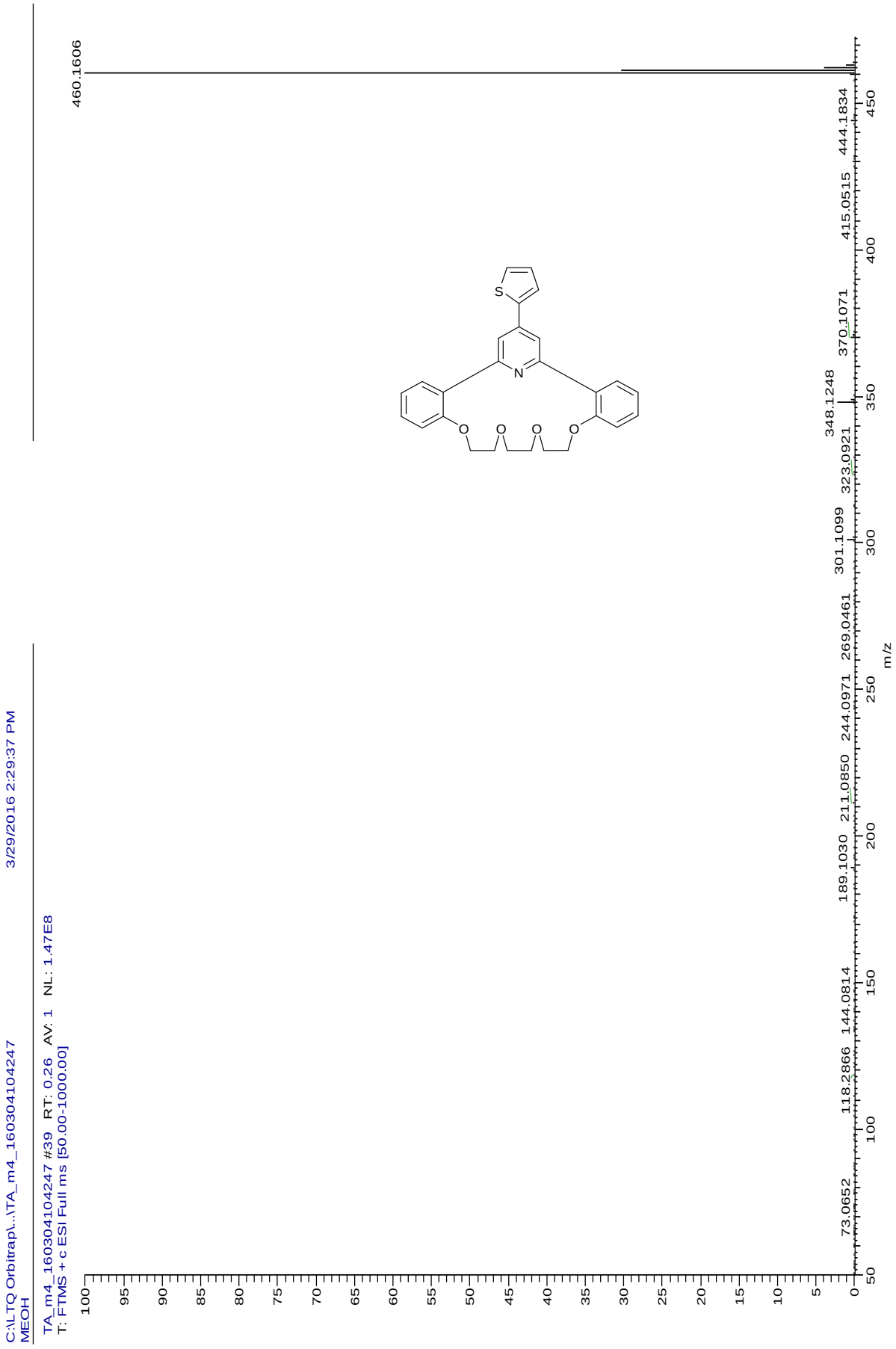

Current Data Parameters
NAME 1-K4+2OMe
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170208
Time 20.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2048
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.36015001 W
PLW13 0.23050000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
K4+2OMe
68.177
69.425
69.797
71.469
OMe
N
O
66 ppm
67
68
69
70
71
72
73
74
75
76
77
78
79

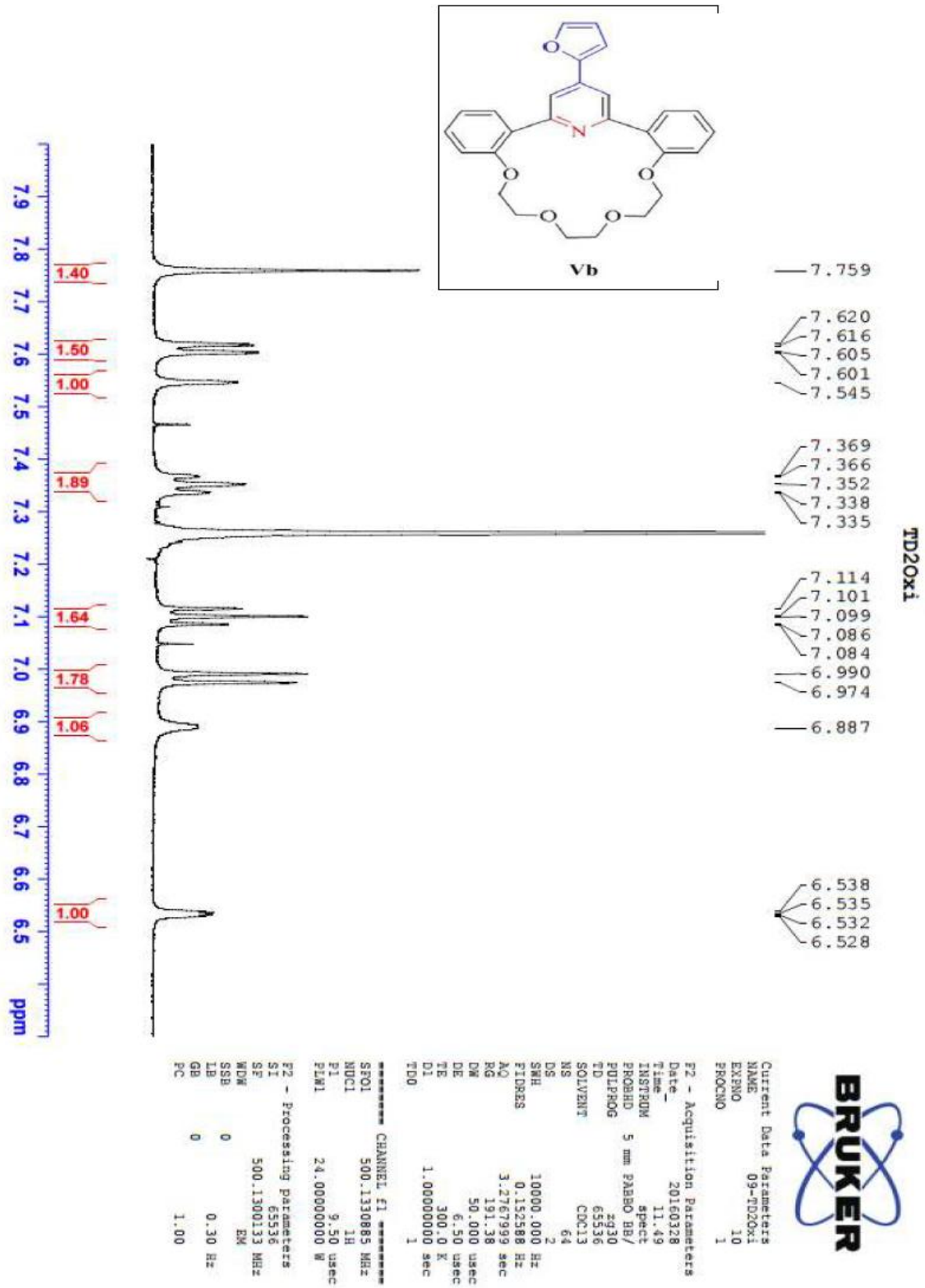

C:\LTQ Orbitrap\...\T11\M14_151210164833
12/10/2015 5:20:10 PM
M14_151210164833 #133 RT: 1.11 AV: 1 NL: 3.47E8
T: FTMS + c ESI Full ms [150.00-700.00]
OMe
N
O
484.2100
470.1942
451.2458
419.2204
397.1363
380.0971
360.0493
353.9301
329.2096
297.1848
279.1586
244.9563
203.9300
185.9729
172.9557
m/z

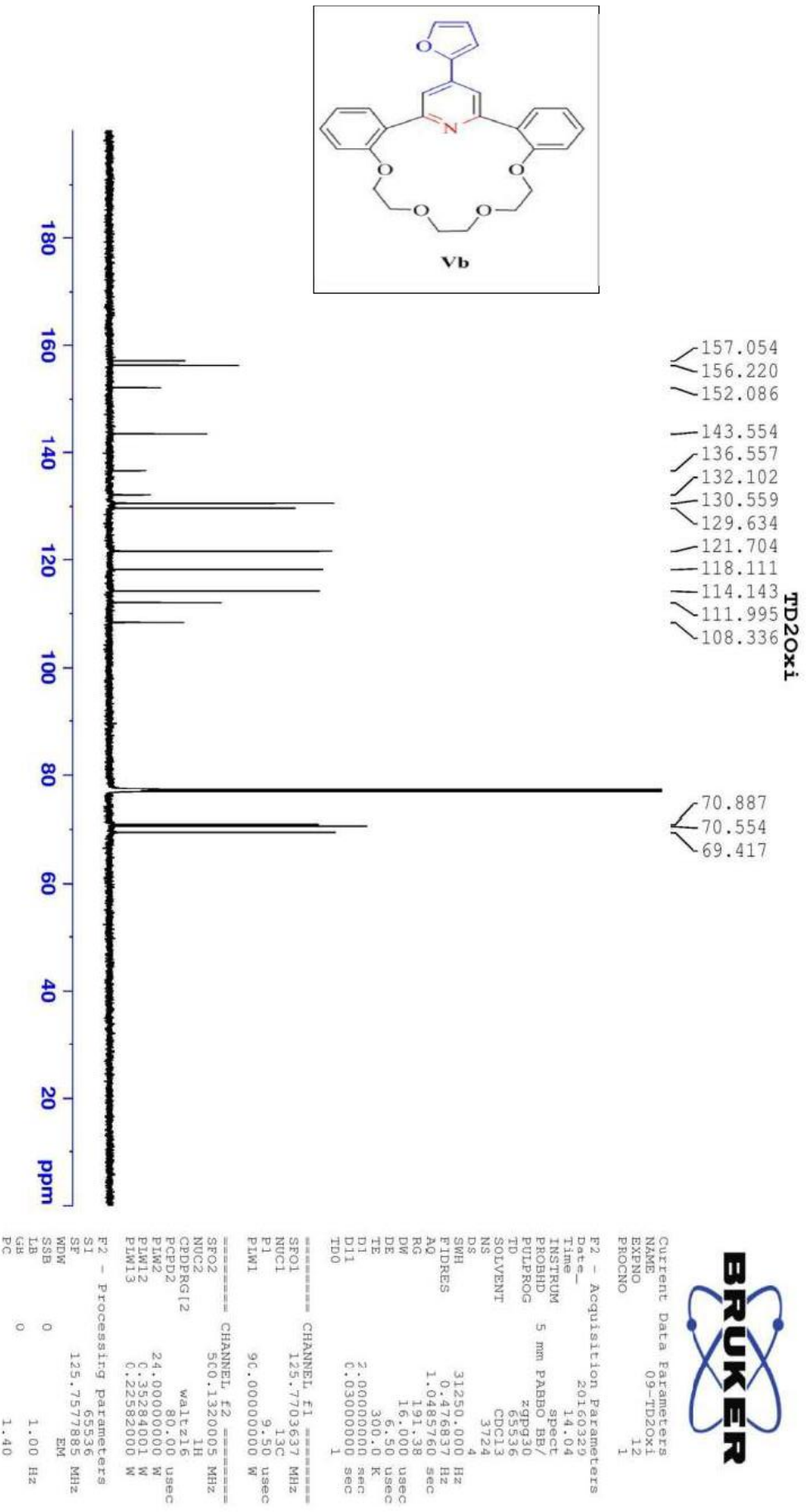

Current Data Parameters
NAME 7VAN_TD2S
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150313
Time 16.28
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2769001
RG 256
DW 50.000
DE 6.00
TE 300.0
D1 1.00000000
MCREST 0.00000000
MCWRK 0.01500000
======== CHANNEL f1 ========
NUC1 1H
P1 8.50
PL1 -3.00
SFO1 500.1335009
F2 - Processing parameters
SI 32768
SF 500.1300133
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
ppm
0.000
0.006
1.567
2.043
3.216
3.625
3.633
3.641
4.100
4.108
4.115
6.982
6.998
7.097
7.112
7.126
7.132
7.142
7.150
7.260
7.341
7.344
7.357
7.360
7.372
7.375
7.396
7.407
7.536
7.543
7.624
7.627
7.639
7.642
7.735
2.037
2.126
2.197
1.000
1.032
0.525
0.552
1.014
1.622
1.119
S
N
O
O
O
O

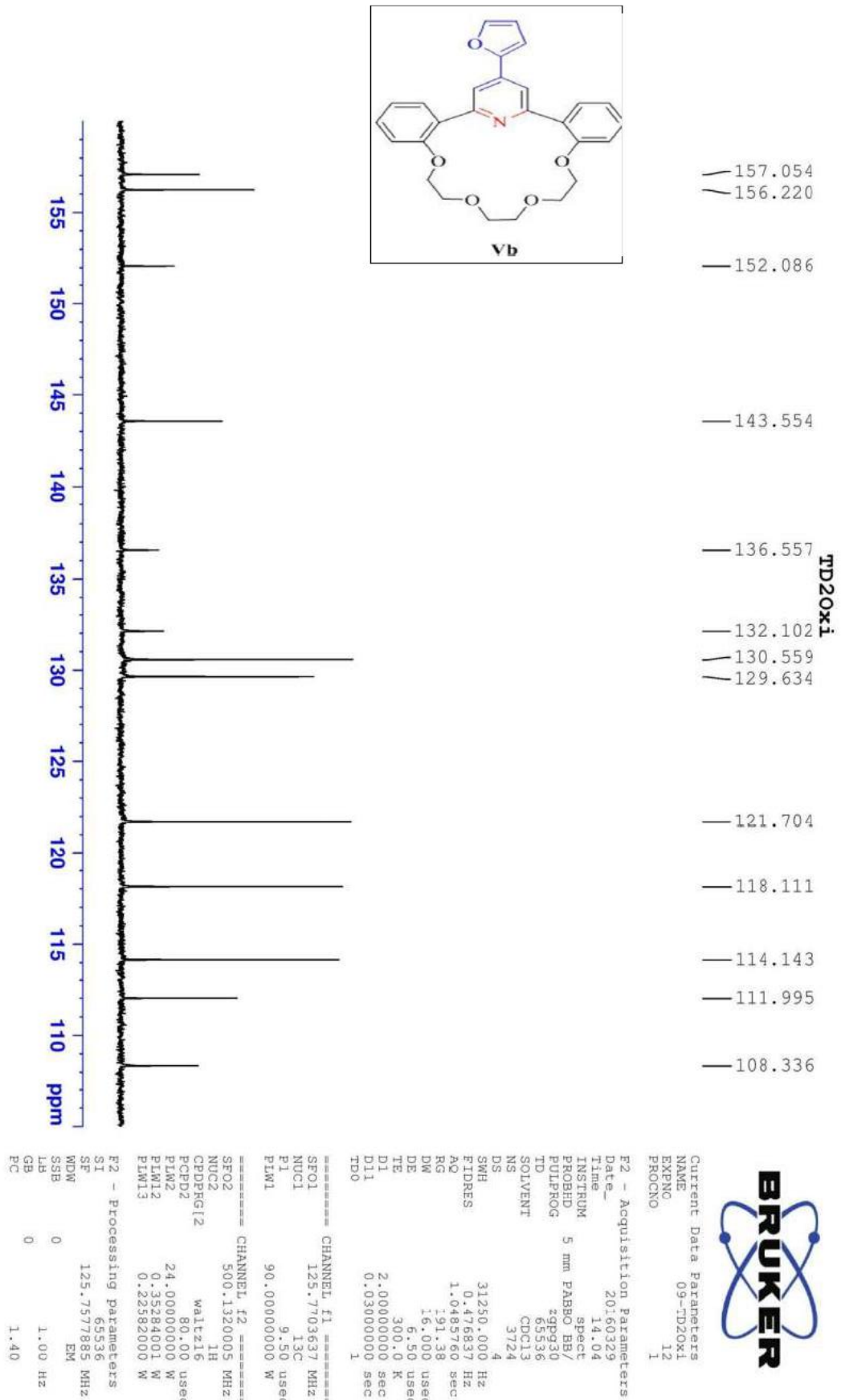

6.982
6.998
7.097
7.112
7.126
7.132
7.142
7.150
7.260
7.341
7.344
7.357
7.360
7.372
7.375
7.396
7.407
7.536
7.543
7.624
7.627
7.639
7.642
7.735
S
N
O
O
O
O
ppm
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
1.119
1.622
1.014
0.552
0.525
1.032
1.000

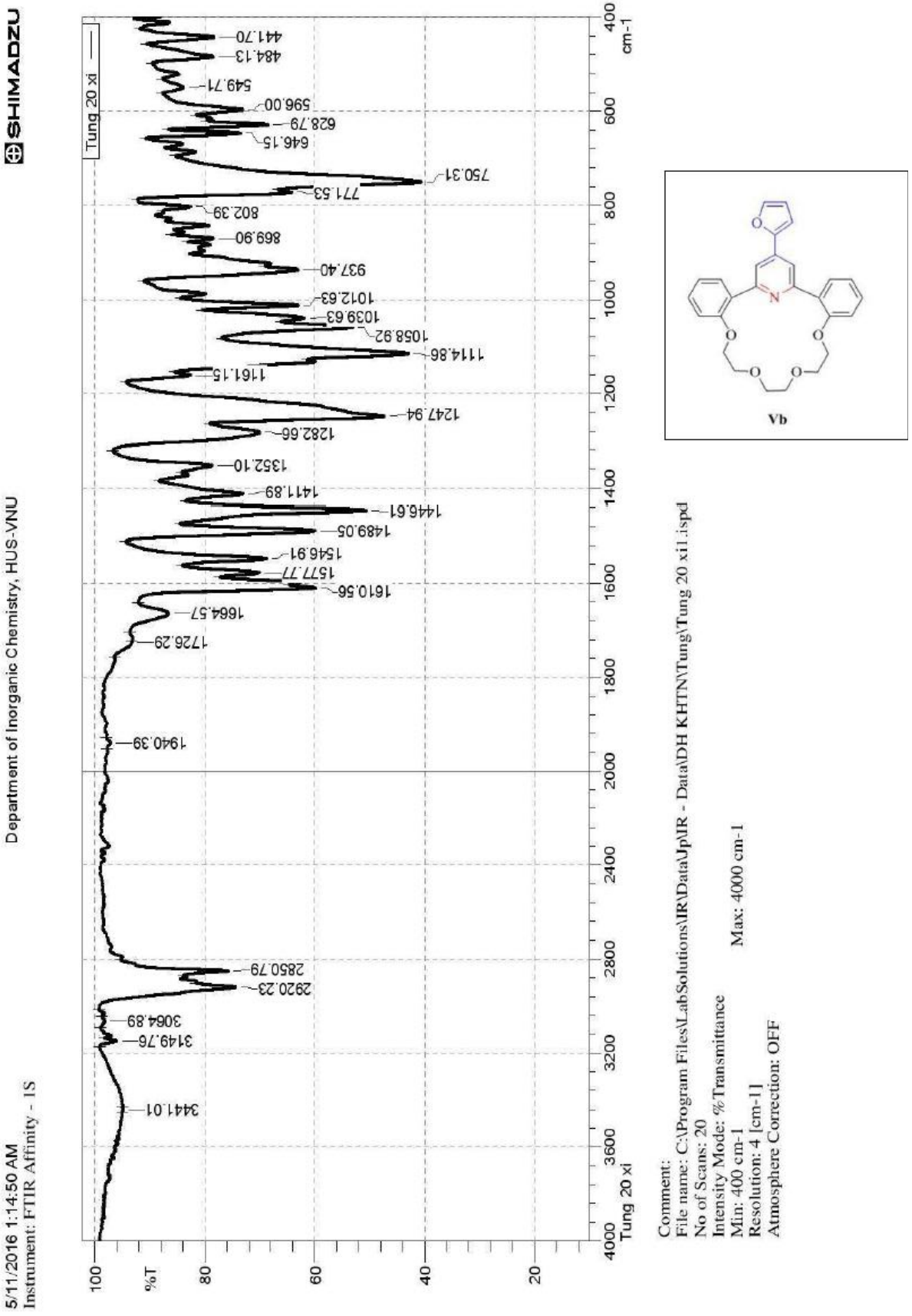

SHIMADZU
Department of Inorganic Chemistry, HUS-VNU
27-Mar-15 7:16:38 PM
Instrument: FTIR Affinity - 1S
TD 2
487.99
551.64
619.15
705.95
750.31
798.53
839.03
854.47
879.54
997.20
1056.99
1132.21
1251.80
1282.66
1365.60
1409.96
1444.68
1492.90
1548.84
1604.77
1735.93
2532.54
2731.20
2866.22
2918.30
2960.73
3074.53
3454.51
S
N
O
O
O
O
%T
100
80
60
40
20
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
400
cm-1
TD 2
Comment:
File name: D:\IR - Data\Tung - hdau\TD 21.ispd
No of Scans:
Intensity Mode:
Min: cm-1 Max: cm-1
Resolution:
Atmosphere Correction:


C:\LTQ Orbitrap\...\TA_m4_160304104247
MEOH
3/29/2016 2:29:37 PM
TA_m4_160304104247 #39 RT: 0.26 AV: 1 NL: 1.47E8
T: FTMS + c ESI Full ms [50.00-1000.00]
S
N
O
O
O
O
460.1606
348.1248
370.1071
415.0515
444.1834
323.0921
301.1099
269.0461
244.0971
211.0850
189.1030
144.0814
118.2866
73.0652
m/z

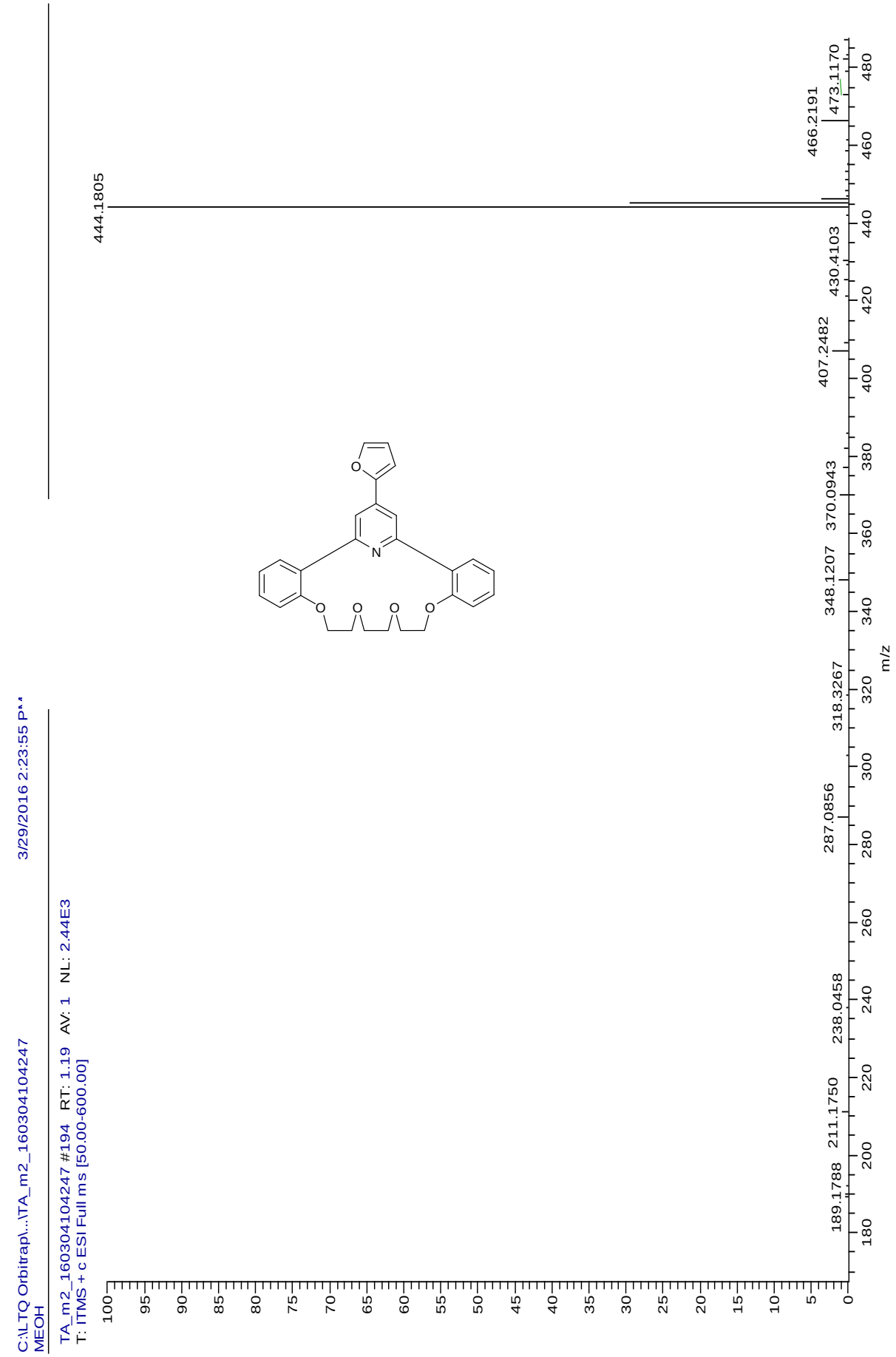

Vb
TD2Oxi
7.759
7.620
7.616
7.605
7.601
7.545
7.369
7.366
7.352
7.338
7.335
7.114
7.101
7.099
7.086
7.084
6.990
6.974
6.887
6.538
6.535
6.532
6.528
1.40
1.50
1.00
1.89
1.64
1.78
1.06
1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 ppm
Current Data Parameters
NAME 09-TD2Oxi
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160328
Time_ 11.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 191.38
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.50 usec
PL1 24.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

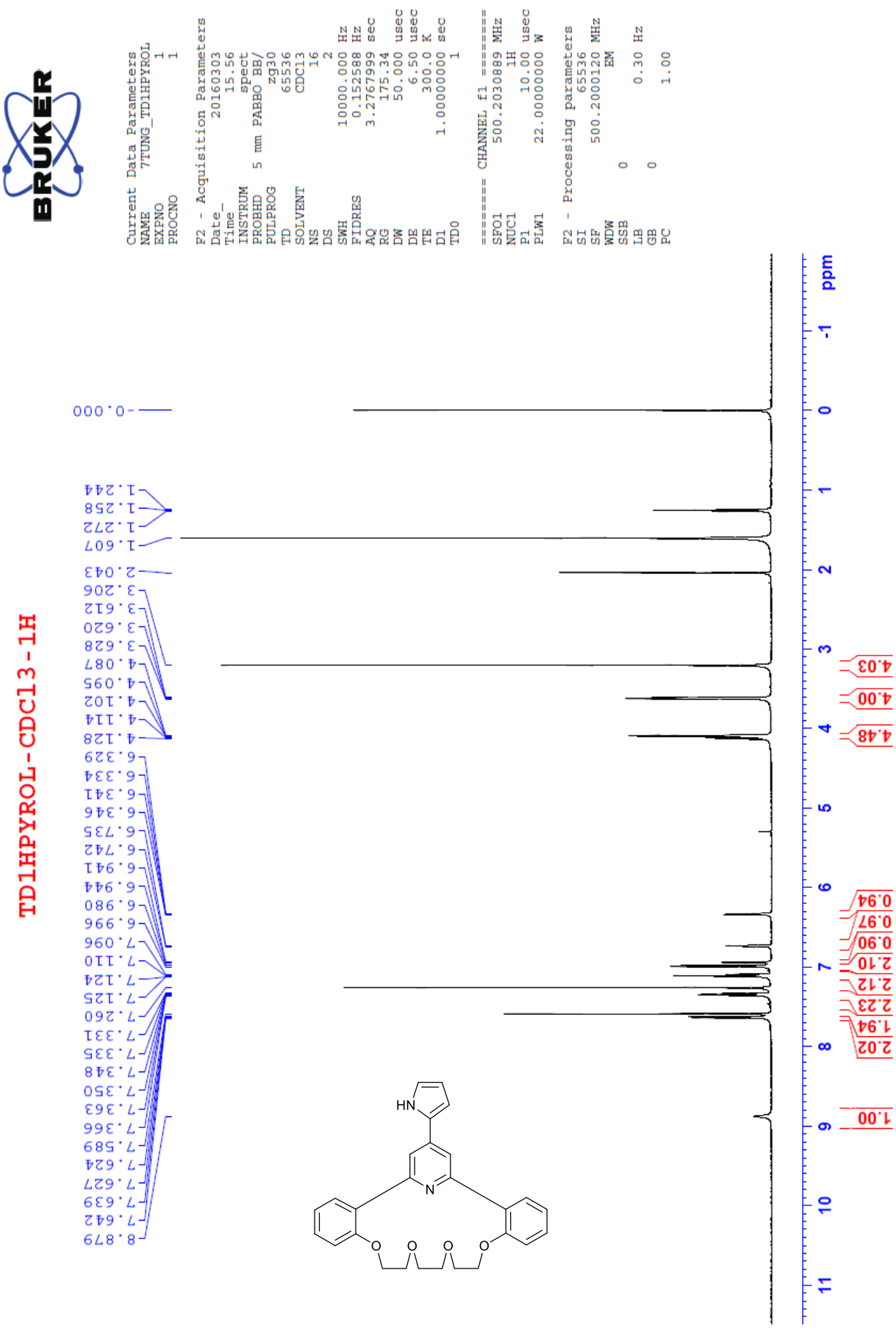

Vb
TD2Oxi
157.054
156.220
152.086
143.554
136.557
132.102
130.559
129.634
121.704
118.111
114.143
111.995
108.336
70.887
70.554
69.417
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME 09-TD2Oxi
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160329
Time 14.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3724
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.35284001 W
PLW13 0.22582000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB
PC 1.40

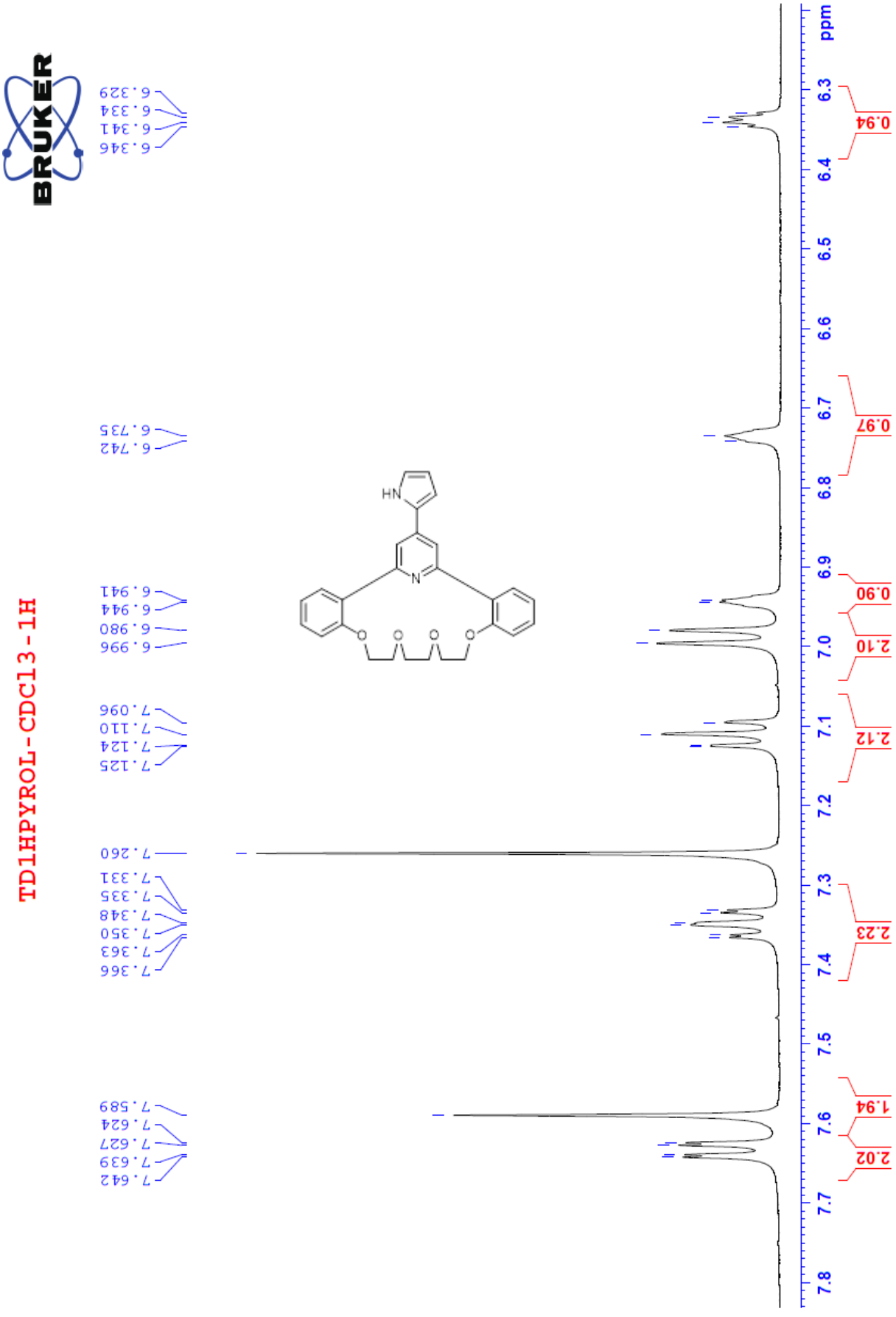

Vb
157.054
156.220
152.086
143.554
136.557
132.102
130.559
129.634
121.704
118.111
114.143
111.995
108.336
TD20xi
155
150
145
140
135
130
125
120
115
110
ppm
Current Data Parameters
NAME 09-TD20xi
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160329
Time 14.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3724
DS 4
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 191.38
DW 16.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.50 usec
PLW1 90.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 24.00000000 W
PLW12 0.35284001 W
PLW13 0.22582000 W
F2 - Processing parameters
SI 65536
SF 125.7577885 MHz
WDW EM
SSB 0 1.00 Hz
LB 0
GB 0
PC 1.40

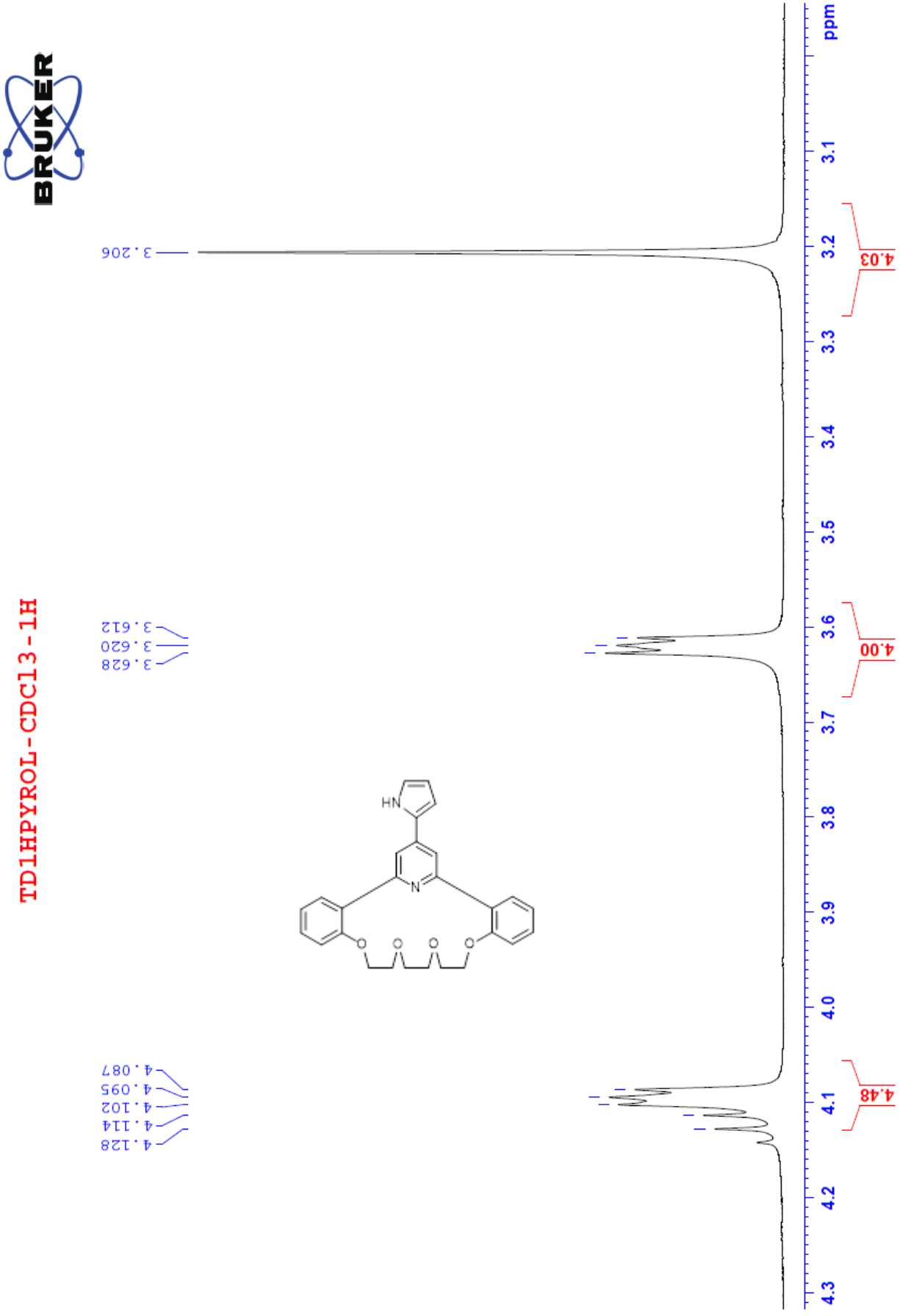

SHIMADZU
Department of Inorganic Chemistry, HUS-VNU
5/11/2016 1:14:50 AM
Instrument: FTIR Affinity - 1S
Tung 20 xi
%T
cm-1
3441.01
3149.76
3064.89
2920.23
2850.79
1940.39
1726.29
1664.57
1610.56
1577.77
1546.91
1489.05
1446.61
1411.89
1352.10
1282.66
1247.94
1161.15
1114.86
1058.92
1039.63
1012.63
937.40
869.90
802.39
771.53
750.31
646.15
628.79
596.00
549.71
484.13
441.70
Tung 20 xi
Comment:
File name: C:\Program Files\LabSolutions\IR\DataJpIR - Data\DH KHTN\Tung\Tung 20 xi1.ispd
No of Scans: 20
Intensity Mode: %Transmittance
Min: 400 cm-1
Max: 4000 cm-1
Resolution: 4 [cm-1]
Atmosphere Correction: OFF

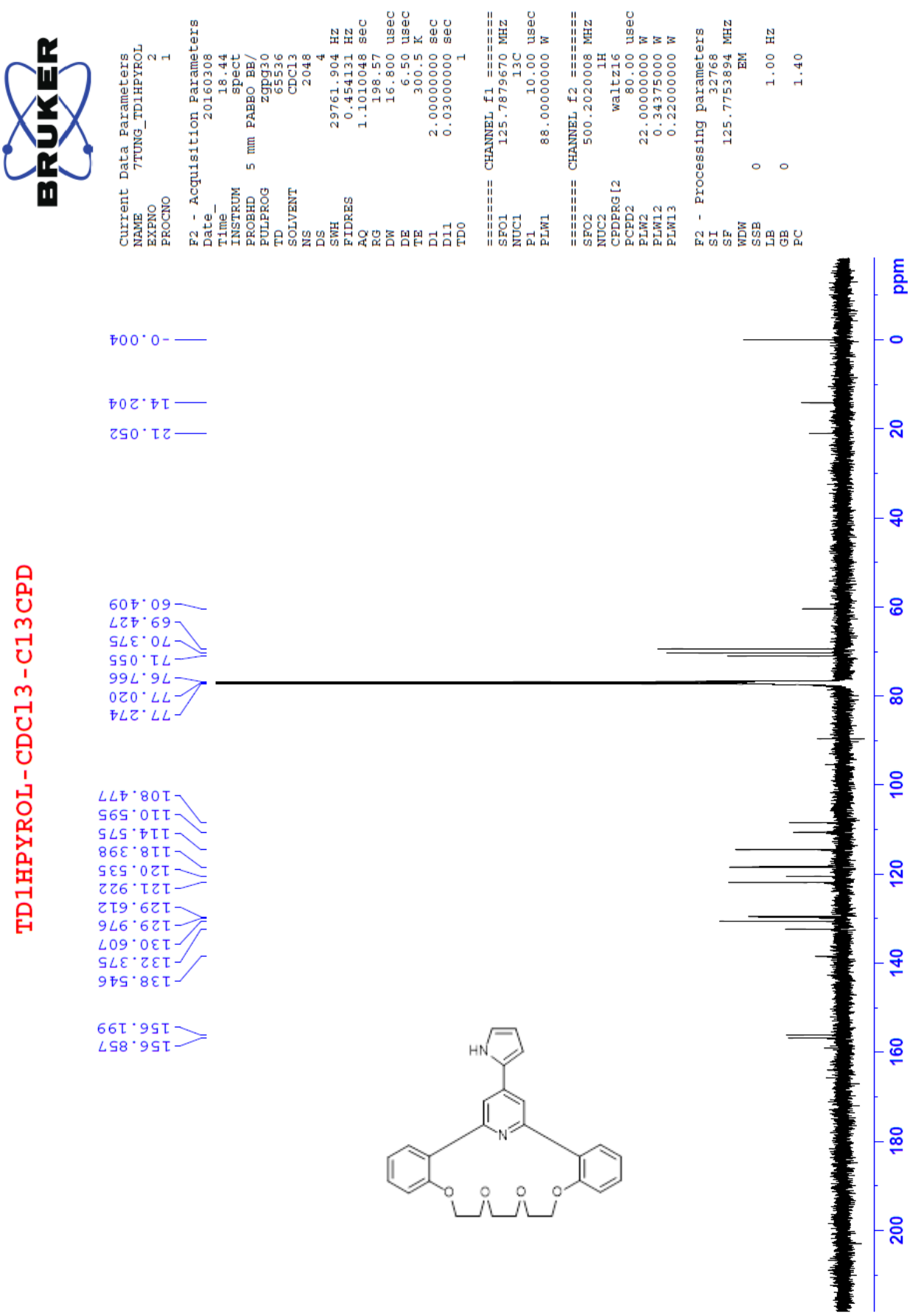
Vb

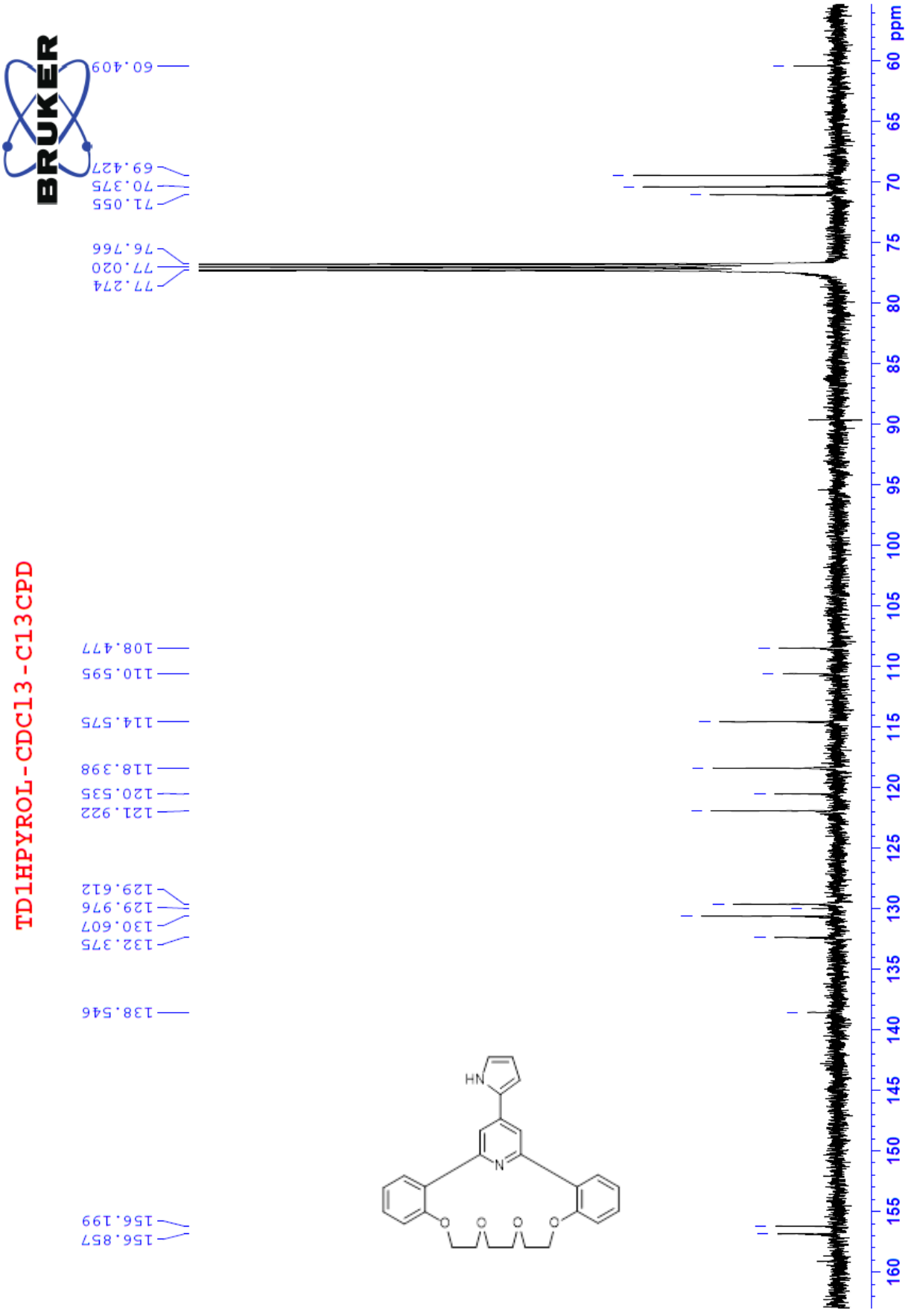

C:\LTQ Orbitrap\...\TA_m2_160304104247
MEOH
3/29/2016 2:23:55 P
TA_m2_160304104247 #194 RT: 1.19 AV: 1 NL: 2.44E3
T: ITMS + c ESI Full ms [50.00-600.00]
444.1805
466.2191
473.1170
407.2482
430.4103
348.1207
370.0943
287.0856
318.3267
189.1788
211.1750
238.0458
m/z
O
N
O
O
O
O

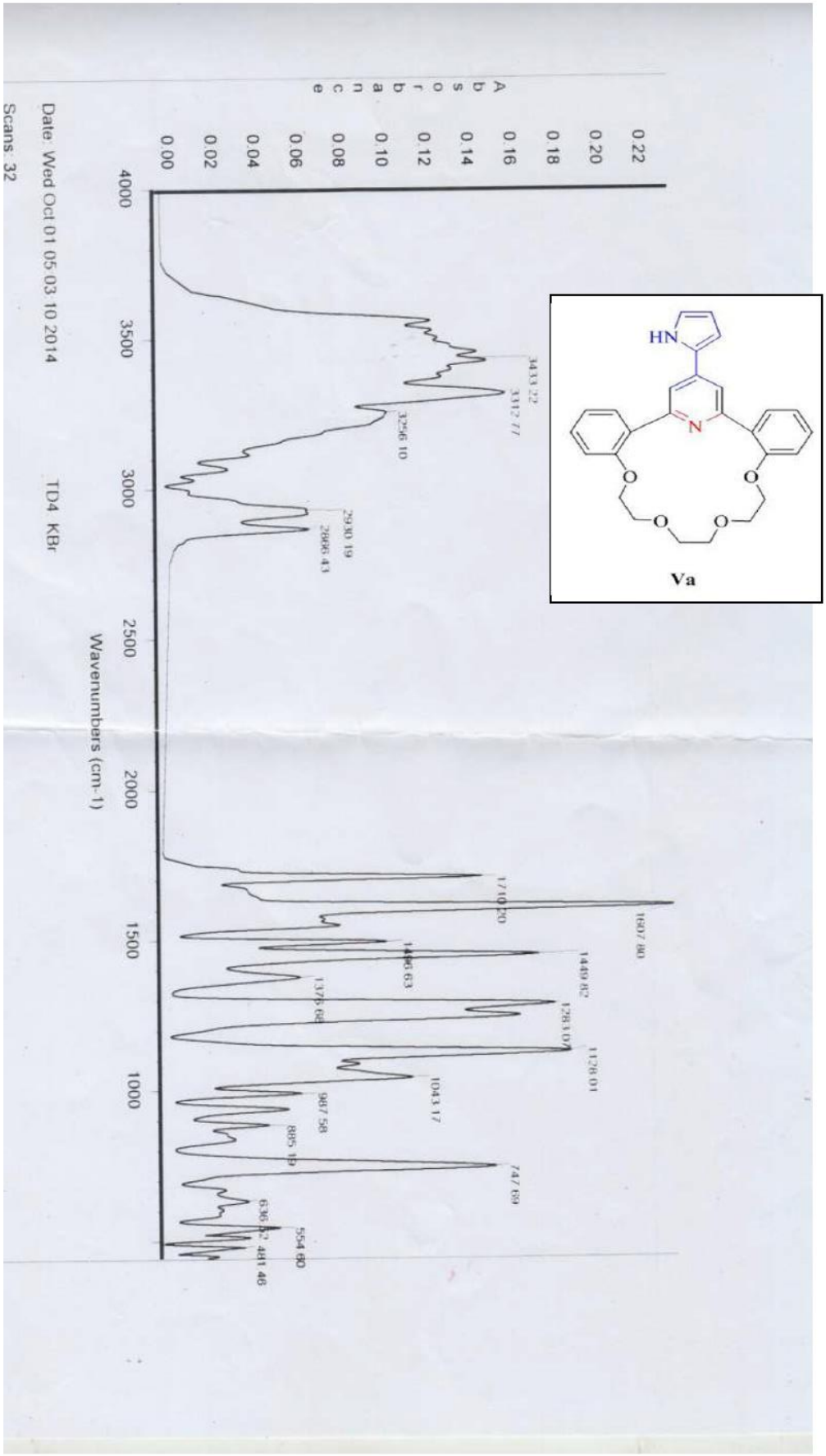

Current Data Parameters
NAME 7TUNG_TD1HPYROL
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160303
Time 15.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 175.34
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.2030889 MHz
NUC1 1H
P1 10.00 usec
PLW1 22.00000000 W
F2 - Processing parameters
SI 65536
SF 500.2000120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TD1HPYROL-CDCl3-1H
ppm
HN
N
O
O
O
O

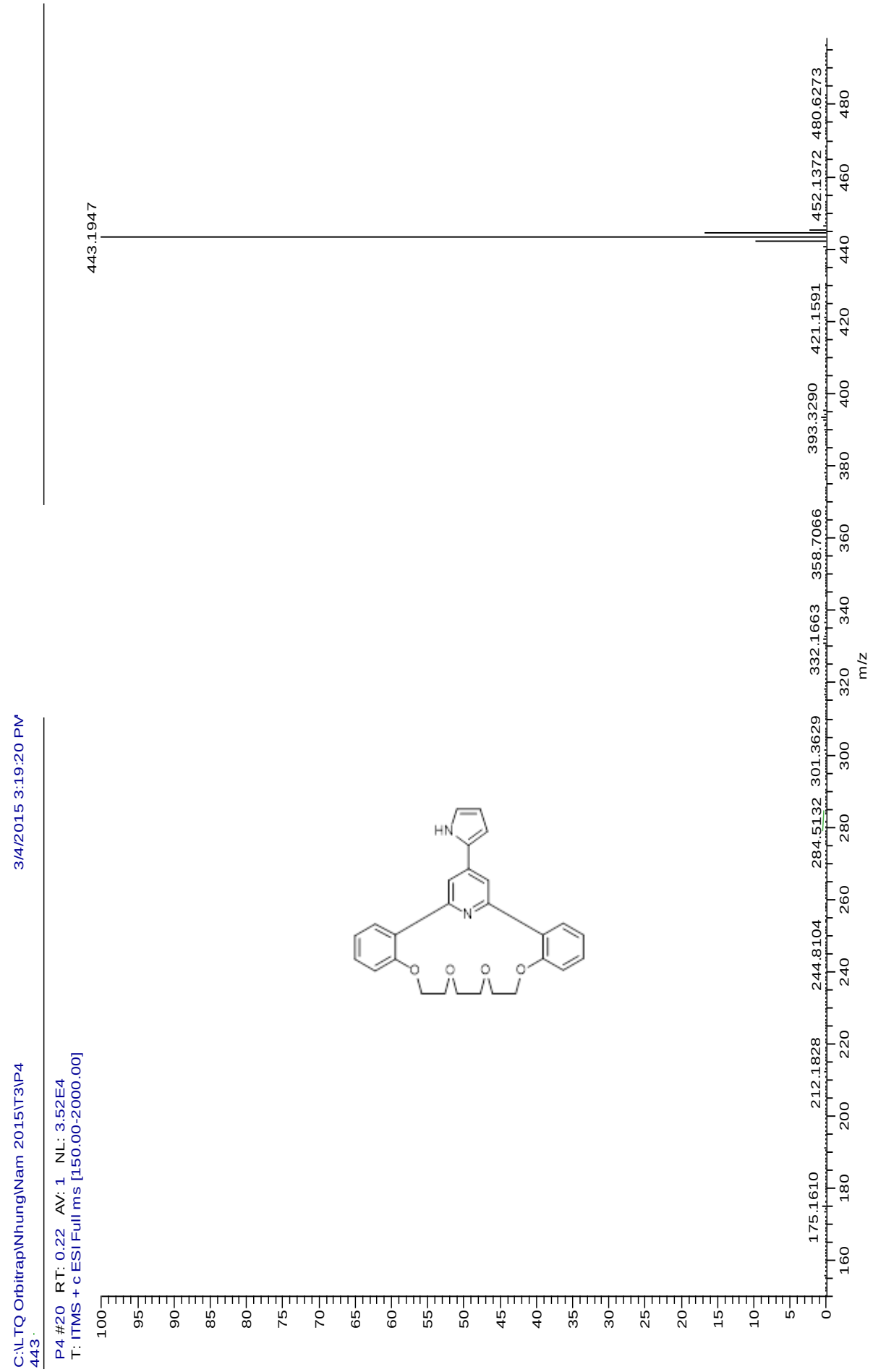

TD1HPYROL-CDCl3-1H
BRUKER
HN
N
O
O
O
O
ppm
6.329
6.334
6.341
6.346
6.735
6.742
6.941
6.944
6.980
6.996
7.096
7.110
7.124
7.125
7.260
7.331
7.335
7.348
7.350
7.363
7.366
7.589
7.624
7.627
7.639
7.642
0.94
0.97
0.90
2.10
2.12
2.23
1.94
2.02


TD1HPYROL-CDCl3-1H
3.206
3.628
3.620
3.612
4.128
4.114
4.102
4.095
4.087
4.03
4.00
4.48
HN
N
ppm
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
4.1
4.2
4.3


Current Data Parameters
NAME 7TUNG_TD1HPYROL
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160308
Time 18.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 198.57
DW 16.800 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7879670 MHz
NUC1 13C
P1 10.00 usec
PLW1 88.00000000 W
======== CHANNEL f2 ========
SFO2 500.2020008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 22.00000000 W
PLW12 0.34375000 W
PLW13 0.22000000 W
F2 - Processing parameters
SI 32768
SF 125.7753894 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TD1HPYROL-CDCl3-C13CPD
-0.004
14.204
21.052
60.409
69.427
70.375
71.055
76.766
77.020
77.274
108.477
110.595
114.575
118.398
120.535
121.922
129.612
129.976
130.607
132.375
138.546
156.199
156.857
HN
N
O
O
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200


TD1HPYROL-CDCl3-C13CPD
60.409
69.427
70.375
71.055
76.766
77.020
77.274
108.477
110.595
114.575
118.398
120.535
121.922
129.612
129.976
130.607
132.375
138.546
156.199
156.857
HN
N
ppm


HN
N
O
O
O
O
Va
Date: Wed Oct 01 05:03:10 2014
TD4 KBr
Scans: 32
Wavenumbers (cm-1)
Absorbance
4000
3500
3000
2500
2000
1500
1000
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3433.22
3312.77
3256.10
2930.19
2866.43
1710.20
1607.80
1465.63
1449.82
1378.68
1283.07
1126.01
1043.17
987.58
885.19
747.69
636.82
554.80
481.46


C:\LTQ Orbitrap\Nhung\Nam 2015\T3\P4
443
3/4/2015 3:19:20 PM
P4 #20 RT: 0.22 AV: 1 NL: 3.52E4
T: ITMS + c ESI Full ms [150.00-2000.00]
443.1947
175.1610
212.1828
244.8104
284.5132
301.3629
332.1663
358.7066
393.3290
421.1591
452.1372
480.6273
m/z
HN
N
O
O
O
O