Supplementary Material (ESI) for Chemical Communications

Electronic Supporting Information

One-pot Synthesis of Carbazoles by Palladium-catalyzed *N*-Arylation and Oxidative Coupling

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Table 1: Investigation of the reaction conditions in oxidative coupling.

Entry	cat. (mol %)	Oxidant (equiv)	*T* (°C)	Time (h)	Yield (%)[a]	
					5a	**4a**
1	100	—	80	3	78	6
2	20	O_2	80	11	85	—
3	5	O_2	80	24	85	trace

[a] HPLC yield (absolute calibration curve method).[7]

After the completion of the reaction, the oxide of the phosphine ligand **3** was observed. Dicyclohexyl(2',4',6'-triisopropylbiphenyl-2-yl)phosphine oxide: ^{31}P NMR (202 MHz, $CDCl_3$) δ 46.8 (the chart shown in p S39). ^{31}P NMR for the ligand **3**: –11.5 (see: X. Huang, K. W. Anderson, D. Zim, L. Jiang, A. Klapars, S. L. Buchwald, *J. Am. Chem. Soc*., 2003, **125**, 6653).

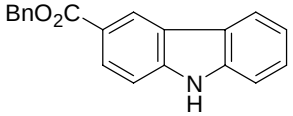
Experimental Section

Typical Procedure for the One-Pot Reaction by Palladium-catalyzed *N*-Arylation and Oxidative Coupling (Table 3, entry 6): Toluene (0.4 mL) was added to a flask containing 4-methylphenyl triflate **1b** (48.0 mg, 0.20 mmol), 4-methoxycarbony aniline **2d** (33.3 mg, 1.1 equiv), $Pd(OAc)_2$ (10 mol%), ligand **3** (15 mol%) and Cs_2CO_3 (1.2 equiv) under argon atmosphere. The mixture was stirred at 100 °C for 1.5 h, then stirred at room temperature for 5 min. AcOH (1.6 mL) was added to the mixture and an oxygen balloon was connected to the reaction vessel, then the reaction mixture was stirred at 80 °C for 22.5 h. After cooling, the reaction mixture was diluted with ethyl acetate, washed with saturated $NaHCO_3$, dried over $MgSO_4$, and concentrated *in vacuo*. The crude material was purified by flash chromatography with hexane/ethyl acetate (15:1) to afford the desired carbazole **10** (37.4 mg, 78 % yield).

Materials: ^{1}H NMR and ^{13}C NMR spectral data of diphenylamine **4a**[1] and carbazoles **5a**[2], **5b**[3], **5c**[3], **5d**[4], **5f**[5], **5g**[3], **6**[6], **7**[7] were in agreement with those previously reported.

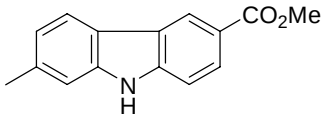
[1] Commercially available – CAS# 122-39-4
[2] Commercially available – CAS# 86-74-8
[3] Yamamoto, M.; Matsubara, S. *Chem lett*. 2007, **36**, 172.
[4] Li, W.-S.; McChesney, J. D.; El-Feraly, F. S. *Phytochemistry* 1991, **30**, 133.
[5] Campeau, L.-C.; Thansandote, P.; Fagnou, K. *Org. Lett*. 2005, **7**, 1857.
[6] Forbes, E. J.; Stacey, M.; Tatlow, J. C.; Wragg, R. T. *Tetrahedron* 1960, **8**, 67.
[7] Witulski, B.; Alayrac, C. *Angew. Chem., Int. Ed*. 2002, **41,** 3281.

Characterization Data for Carbazoles (New Compounds)



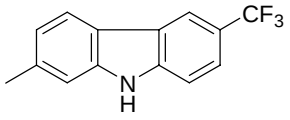
5e

Benzyl 9*H*-Carbazole-3-carboxylate (5e): White solid; IR cm^{-1}: 3289 (NH), 1693 (CO); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.43 (s, 2H, CH_2), 7.27 (m, 1H, Ar), 7.35-7.44 (m, 6H, Ar), 7.51 (d, J = 7.1 Hz, 2H, Ar), 8.10 (d, J = 7.8 Hz, 1H, Ar), 8.16 (dd, J = 7.8, 1.5 Hz, 1H, Ar), 8.35 (s, 1H, NH), 8.84 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 66.5, 110.1, 110.9, 120.3, 120.7, 121.3, 123.0, 123.1, 123.3, 126.6, 127.6, 128.1, 128.2 (2C), 128.6 (2C), 136.5, 139.9, 142.4, 167.2; m.p. = 154-155^{o}C; HRMS (EI): *m/z* calcd for $C_{20}H_{15}NO_2$ (M^+) 301.1103; found: 301.1100.



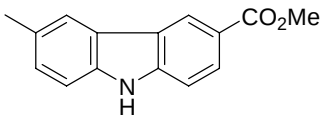
8

Methyl 7-Methyl-9*H*-carbazole-3-carboxylate (8): Yellow white solid; IR cm^{-1}: 3336 (NH), 2949 (CH), 1697 (CO); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.52 (s, 3H, CH_3), 3.97 (s, 3H, CO_2Me), 7.12 (d, J = 8.0 Hz, 1H, Ar), 7.22 (s, 1H, Ar), 7.37 (d, J = 8.5 Hz, 1H, Ar), 7.98 (d, J = 8.0 Hz, 1H, Ar), 8.10 (dd, J = 8.5, 1.5 Hz, 1H, Ar), 8.24 (s, 1H, NH), 8.76 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 51.9, 110.0, 111.0, 120.3, 121.0, 121.3, 121.9, 122.5, 123.2, 127.0, 136.9, 140.4, 142.3, 168.0; m.p. = 210-212^{o}C; HRMS (EI): *m/z* calcd for $C_{15}H_{13}NO_2$ (M^+) 239.0946; found: 239.0942.



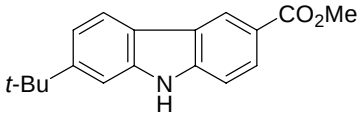
9

3-Trifluoromethyl-7-methyl-9*H*-carbazole (9): Brown solid; IR cm^{-1}: 3392 (NH), 2928 (CH); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.52 (s, 3H, CH_3), 7.10 (d, J = 8.0 Hz, 1H, Ar), 7.20 (s, 1H, Ar), 7.39 (d, J = 8.5 Hz, 1H, Ar), 7.60 (d, J = 8.5 Hz, 1H, Ar), 7.94 (d, J = 8.0 Hz, 1H, Ar), 8.03 (s, 1H, NH), 8.27 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 110.5, 111.0, 117.5 (q), 120.2, 120.5, 121.9, 122.0 (q), 122.1, (d) 123.1, 125.3 (q), 137.2, 140.4, 140.9; m.p. = 202-203^{o}C; HRMS (FAB): *m/z* calcd for $C_{14}H_{10}F_3N$ (M^+) 249.0765; found: 249.0768.



10

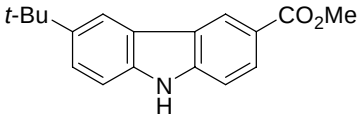
Methyl 6-methyl-9*H*-carbazole-3-carboxylate (10): Yellow white solid; IR cm^{-1}: 3295 (NH), 2948-2851 (CH), 1684 (CO); ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.49 (s, 3H, CH_3), 3.90 (s, 3H, CO_2Me), 7.29 (d, J = 8.3 Hz, 1H, Ar), 7.44 (d, J = 8.3 Hz, 1H, Ar), 7.53 (d, J = 8.5 Hz, 1H, Ar), 7.99 (d, J = 8.5 Hz, 1H, Ar), 8.75 (s, 1H, Ar), 8.75 (s, 1H, Ar), 11.58 (s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 21.0, 51.7, 110.7, 111.1, 119.5, 120.3, 122.0, 122.2, 122.5, 126.4, 127.7, 128.3, 138.5, 142.7, 166.9; m.p. = 233-235^{o}C; HRMS (FAB): *m/z* calcd for $C_{15}H_{13}NO_2$ (M^+) 239.0946; found: 239.0945.



11

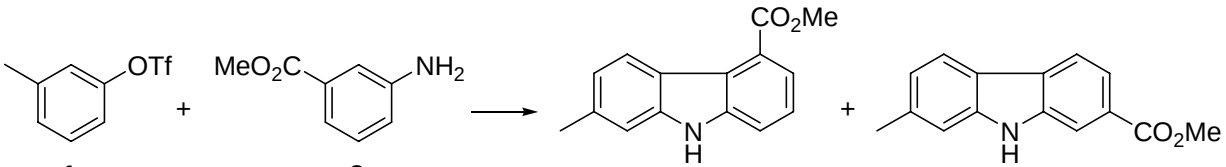
Methyl 7-*tert*-Butyl-9*H*-carbazole-3-carboxylate (11): White solid; IR cm^{-1}: 3282 (NH), 2968 (CH_3), 1695 (CO); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43 (s, 9H, *t*-Bu), 3.97 (s, 3H, CO_2Me), 7.37 (dd, J = 8.3, 1.7 Hz, 1H, Ar), 7.41 (d, J = 8.5 Hz, 1H, Ar), 7.46 (d, J = 1.7 Hz, 1H, Ar), 8.03 (d, J = 8.3

Hz, 1H, Ar), 8.10 (dd, *J* = 8.5, 1.7 Hz, 1H, Ar), 8.19 (s, 1H, NH), 8.77 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.7 (3C), 35.2, 51.9, 107.4, 110.0, 118.5, 120.1, 121.0, 121.3, 122.7, 123.2, 127.0, 140.2, 142.5, 150.5, 168.0; m.p. = 224-228^{o}C; HRMS (FAB): *m/z* calcd for $C_{18}H_{19}NO_2$ (M^+) 281.1416; found: 281.1414.



12

Methyl 6-*tert*-Butyl-9*H*-carbazole-3-carboxylate (12): White solid; IR cm^{-1}: 3331 (NH), 2955 (CH), 1694 (CO); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.45 (s, 9H, *t*-Bu), 3.98 (s, 3H, CO_2Me), 7.39 (d, *J* = 8.5 Hz, 1H, Ar), 7.40 (d, *J* = 8.5 Hz, 1H, Ar), 7.54 (dd, *J* = 8.5, 2.0 Hz, 1H, Ar), 8.11 (dd, *J* = 8.5, 1.7 Hz, 1H, Ar), 8.14 (s, 1H, Ar), 8.22 (s, 1H, NH), 8.83 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.9 (3C), 34.8, 51.9, 110.1, 110.4, 116.8, 121.2, 122.7, 123.1, 123.4, 124.6, 127.2, 138.0, 142.7, 143.5, 167.9; m.p. = 216-217^{o}C; HRMS (FAB): *m/z* calcd for $C_{18}H_{19}NO_2$ (M^+) 281.1416; found: 281.1414.



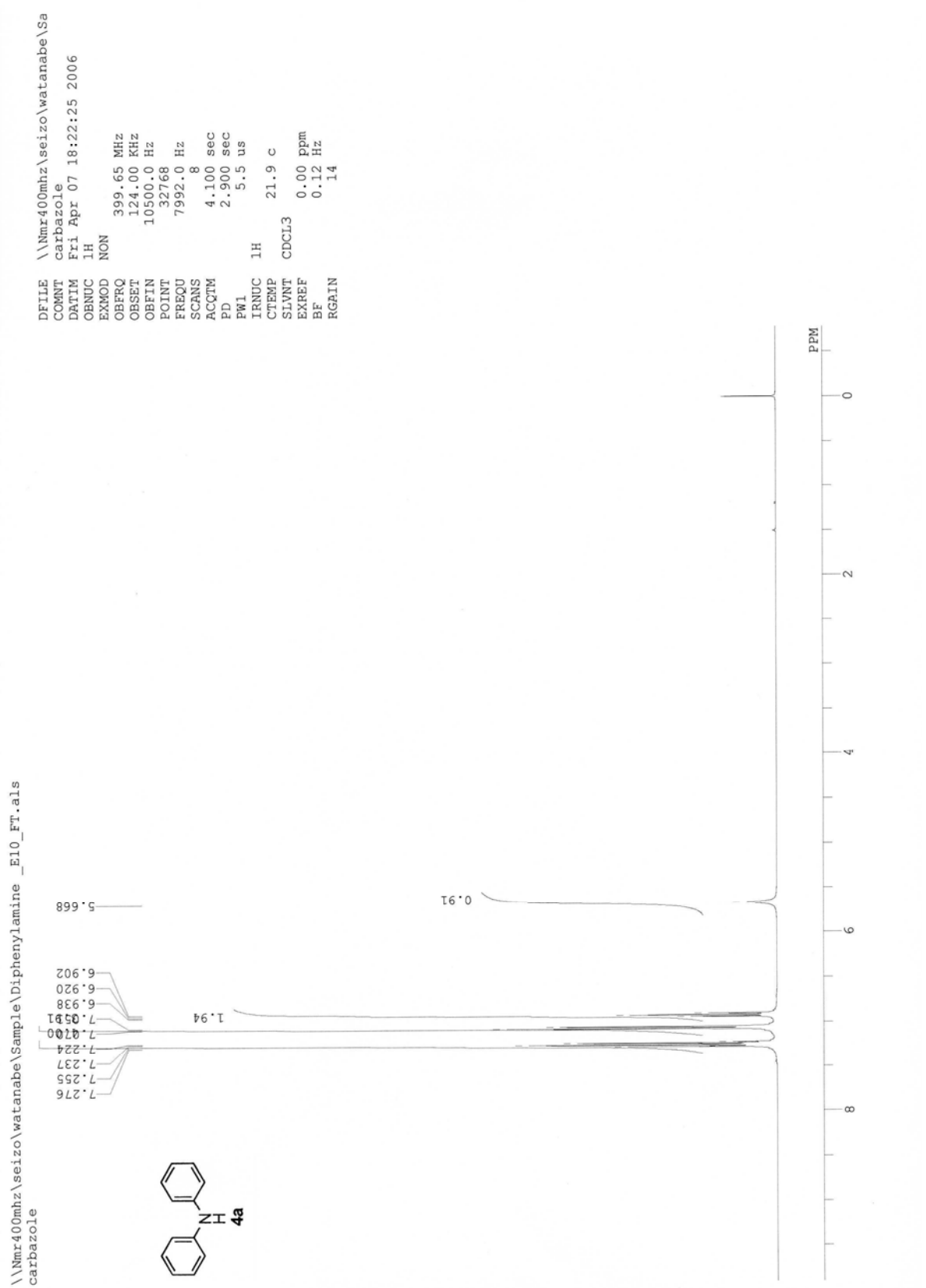
1c + **2e** → **13** + **14**

13/**14** = 86/14
(60% yield)

Methyl 7-Methyl-9*H*-carbazole-4-carboxylate (13) and Methyl 7-Methyl-9*H*-carbazole-2-carboxylate (14). By a procedure identical with that described for the synthesis of **10**, triflate **1c** (48.0 mg, 0.20 mmol) and aniline **2e** (33.3 mg, 1.1 equiv) were converted into a regioisomeric mixture of **13** and **14** (28.5 mg, 60% yield; **13**/**14** = 86:14), which was separated by HPLC.

Compound **13**: white solid; IR cm^{-1}: 3402 (NH), 2950-2860 (CH), 1702 (CO); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.49 (s, 3H, CH_3), 4.05 (s, 3H, CO_2Me), 7.07 (d, *J* = 8.3 Hz, 1H, Ar), 7.12 (s, 1H, Ar), 7.36 (dd, *J* = 8.1, 7.6 Hz, 1H, Ar), 7.49 (d, *J* = 8.1 Hz, 1H, Ar), 7.84 Hz (d, *J* = 7.6 Hz, 1H, Ar), 8.12 (s, 1H, NH), 8.71 (d, *J* = 8.3 Hz, 1H, Ar); ^{13}C NMR (100 Hz, $CDCl_3$): δ 21.9, 52.1, 110.4, 114.8, 119.6, 121.4, 122.0, 122.6, 124.1, 124.7, 125.2, 137.2, 140.3, 140.8, 168.6; m.p. = 220-221^{o}C; HRMS (FAB): *m/z* calcd for $C_{15}H_{13}NO_2$ (M^+) 239.0946; found: 239.0942.

Compound **14**: white solid; IR: ν = 3402 (NH), 2952 (CH), 1713 (CO); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.54 (s, 3H, CH_3), 3.97 (s, 3H, CO_2Me), 7.10 (d, *J* = 8.1 Hz, 1H, Ar), 7.26 (s, 1H, Ar), 7.92 (d, *J* = 8.1 Hz, 1H, Ar), 7.98 (d, *J* = 8.1 Hz, 1H, Ar), 8.05 Hz (d, *J* = 8.1 Hz, 1H, Ar), 8.12 (s, 1H, NH), 8.13 (s, 1H, Ar); ^{13}C NMR (100 Hz, $CDCl_3$): δ 22.2, 52.1, 111.0, 112.3, 119.6, 120.3, 120.7, 120.8, 121.7, 126.8, 127.3, 137.7, 138.8, 141.3, 167.8; m.p. = 215-216^{o}C; HRMS (FAB): *m/z* calcd for $C_{15}H_{13}NO_2$ (M^+) 239.0946; found: 239.0948.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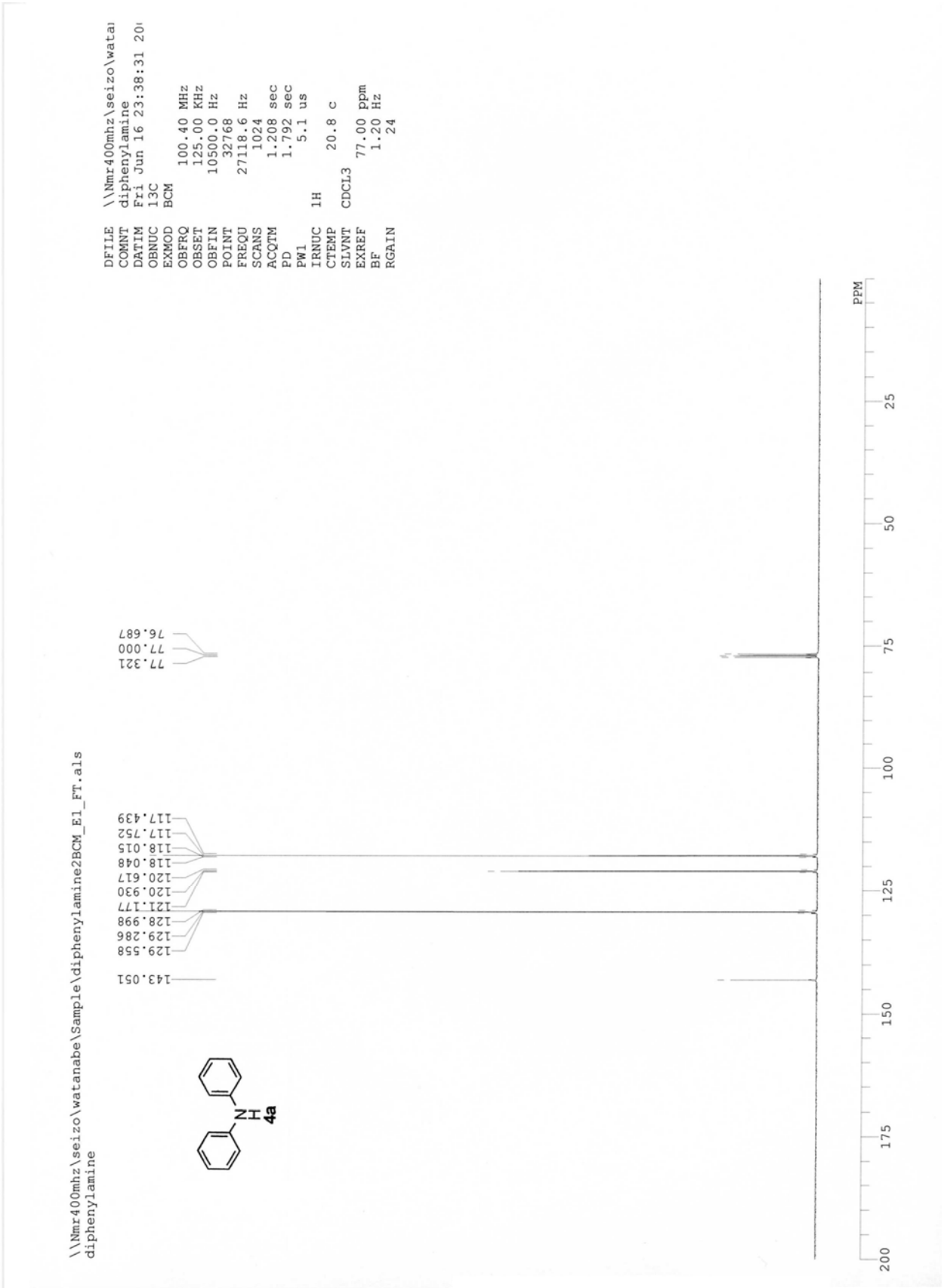
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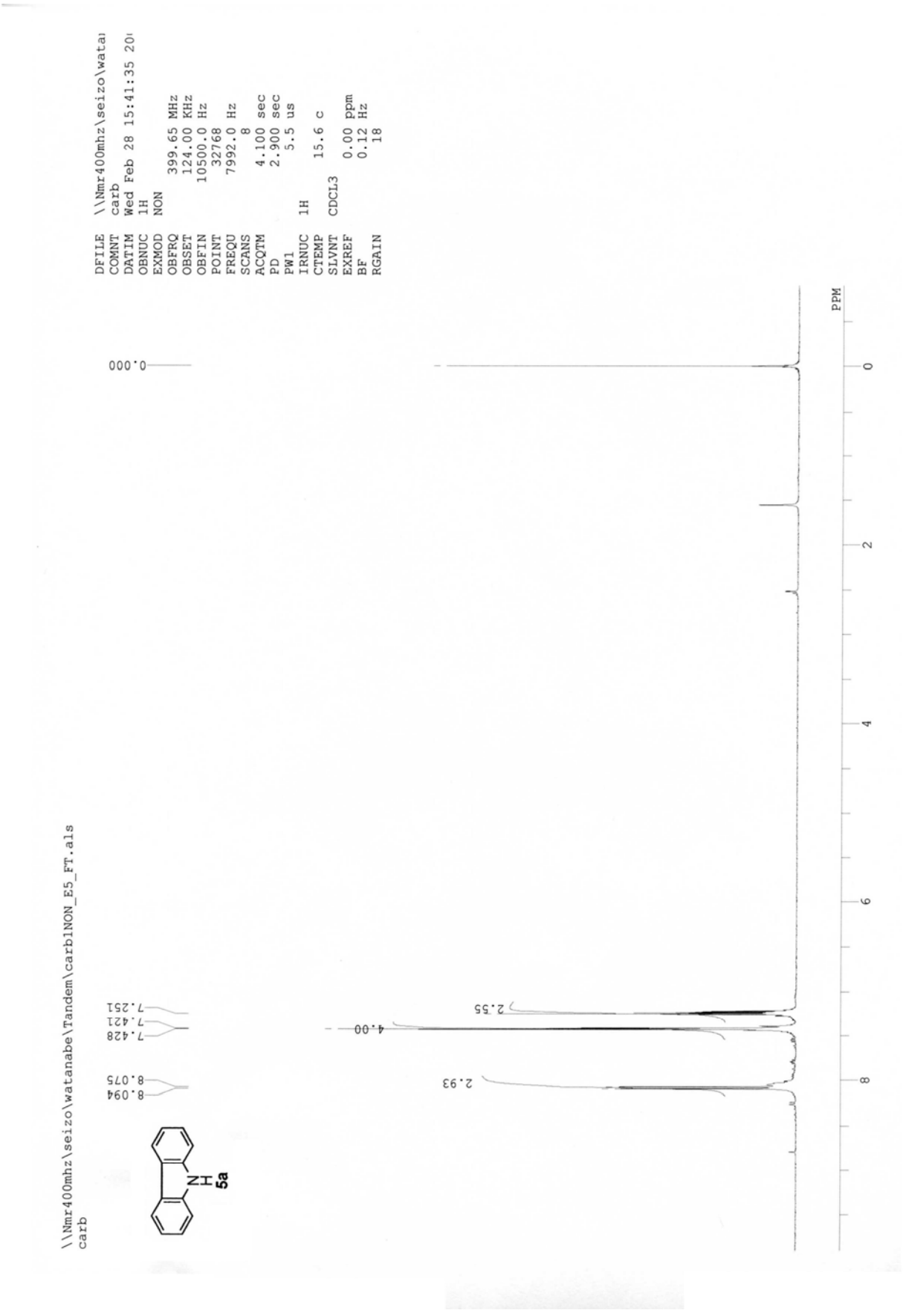
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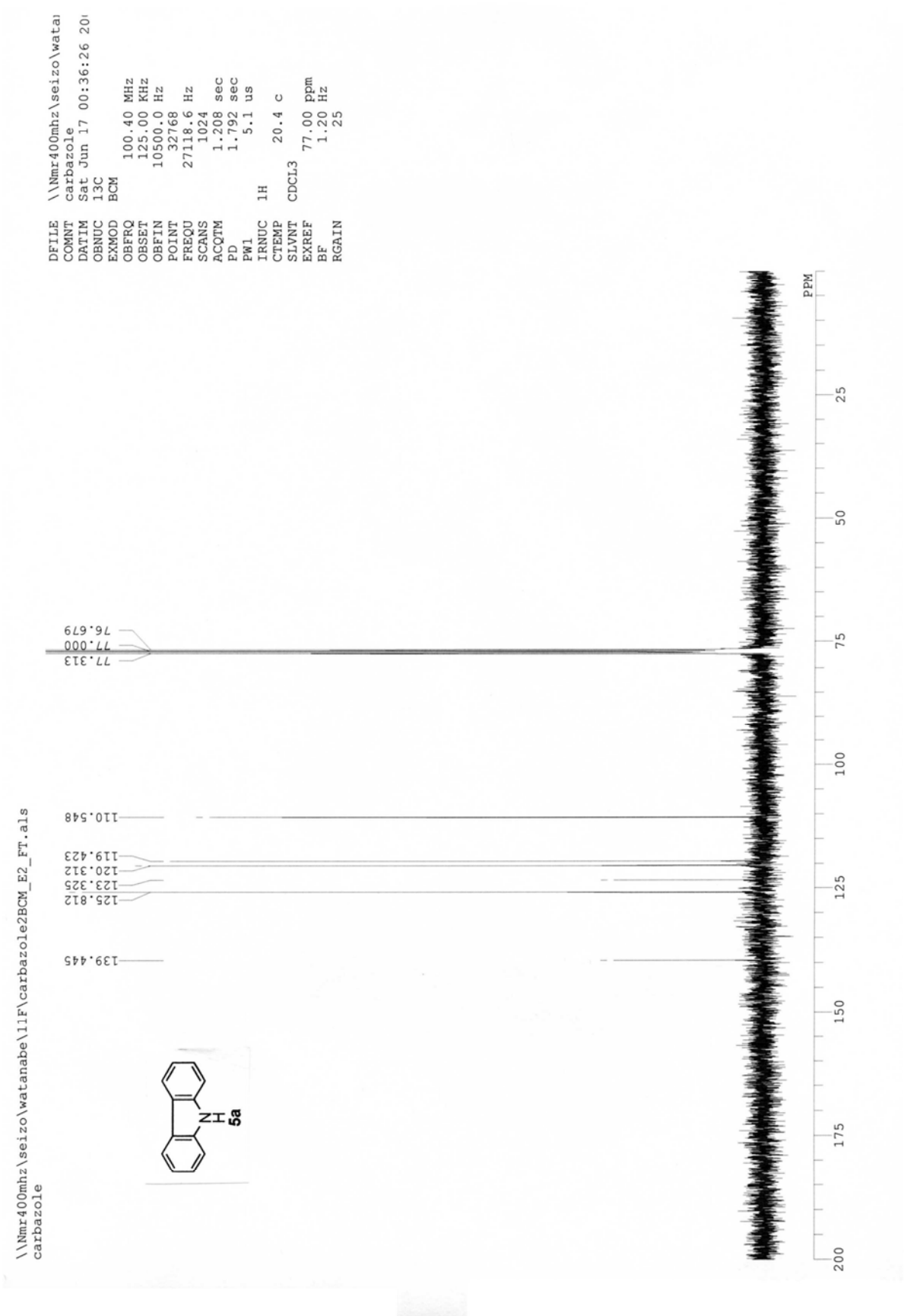
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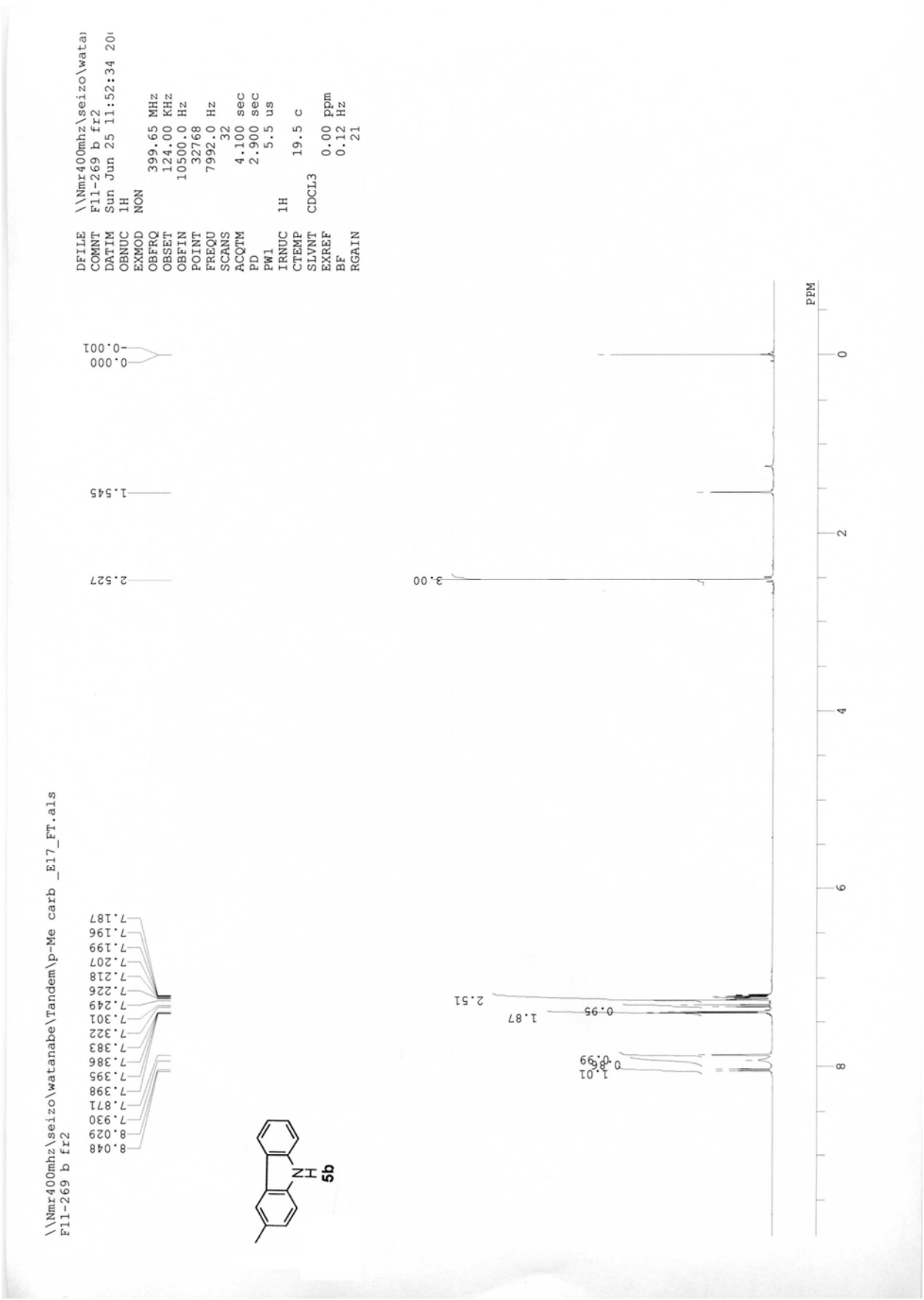
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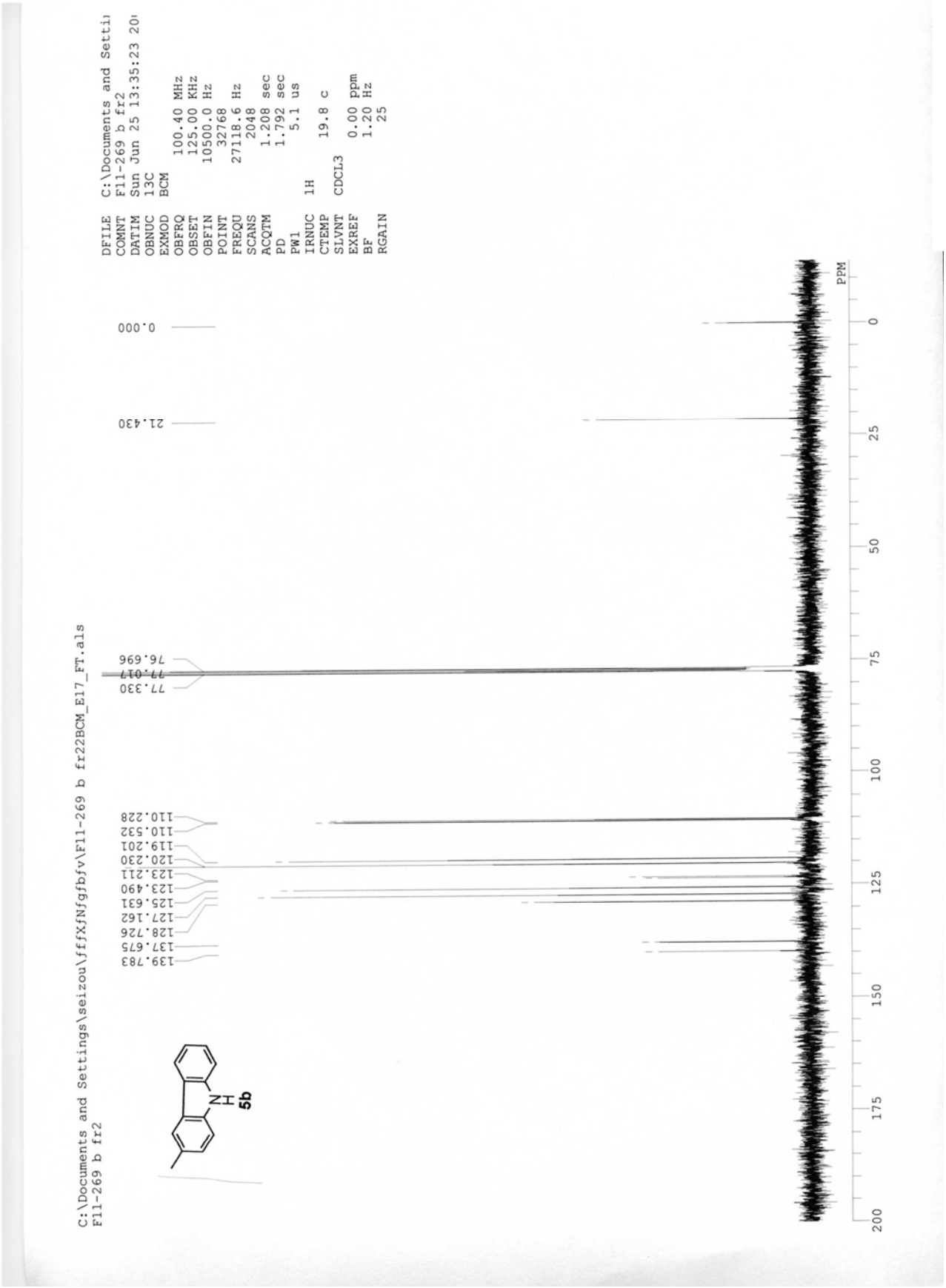
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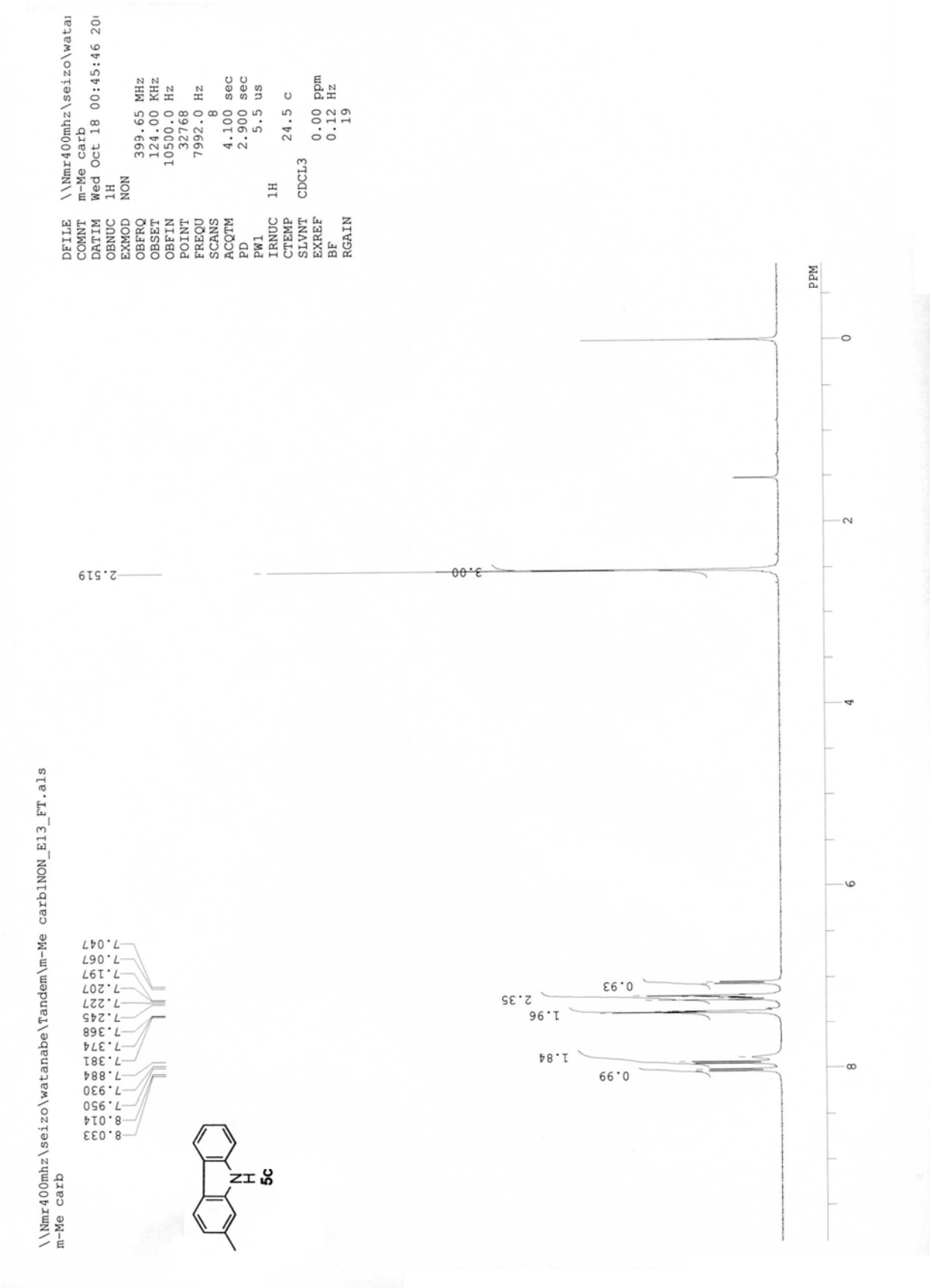
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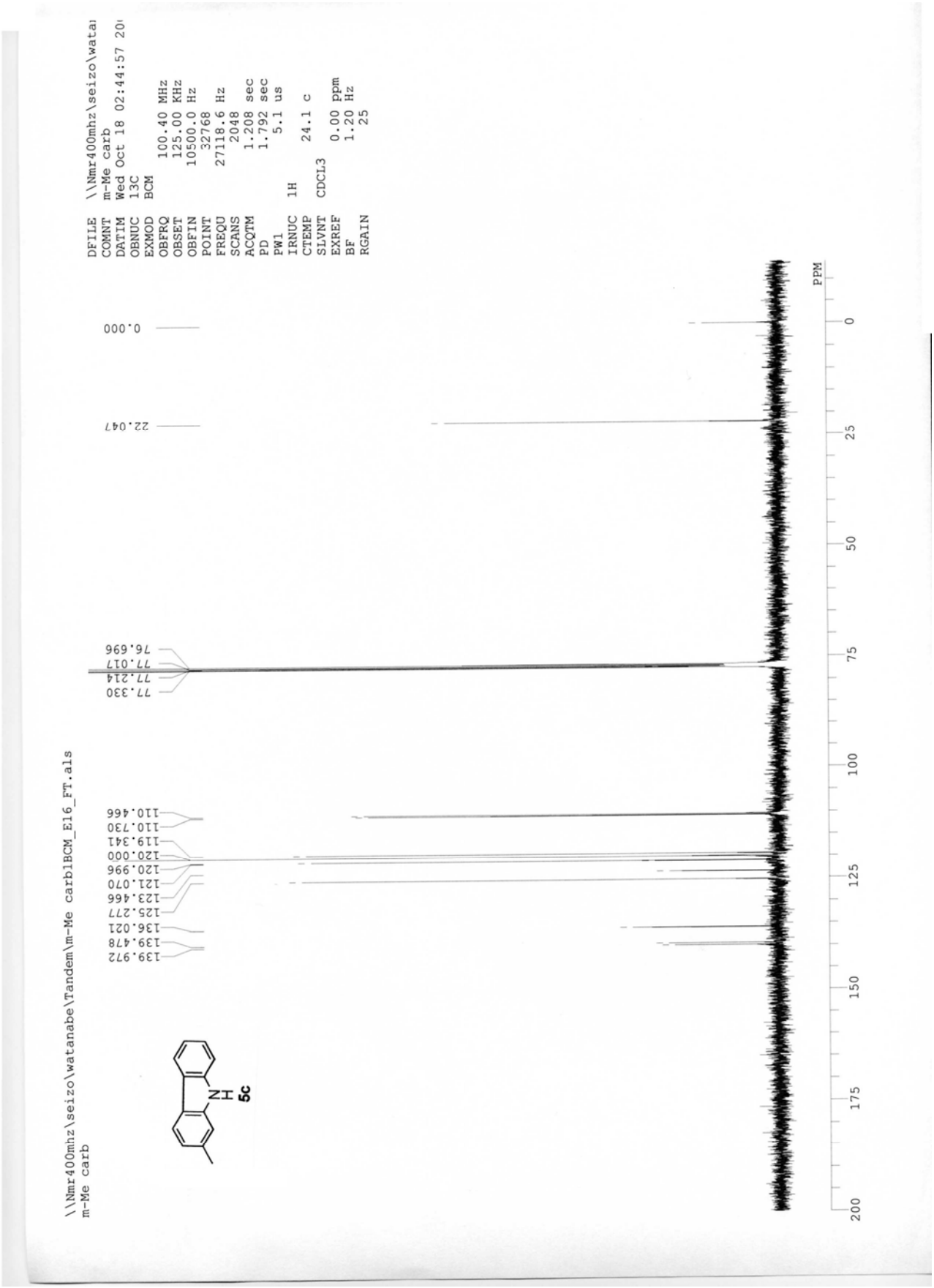
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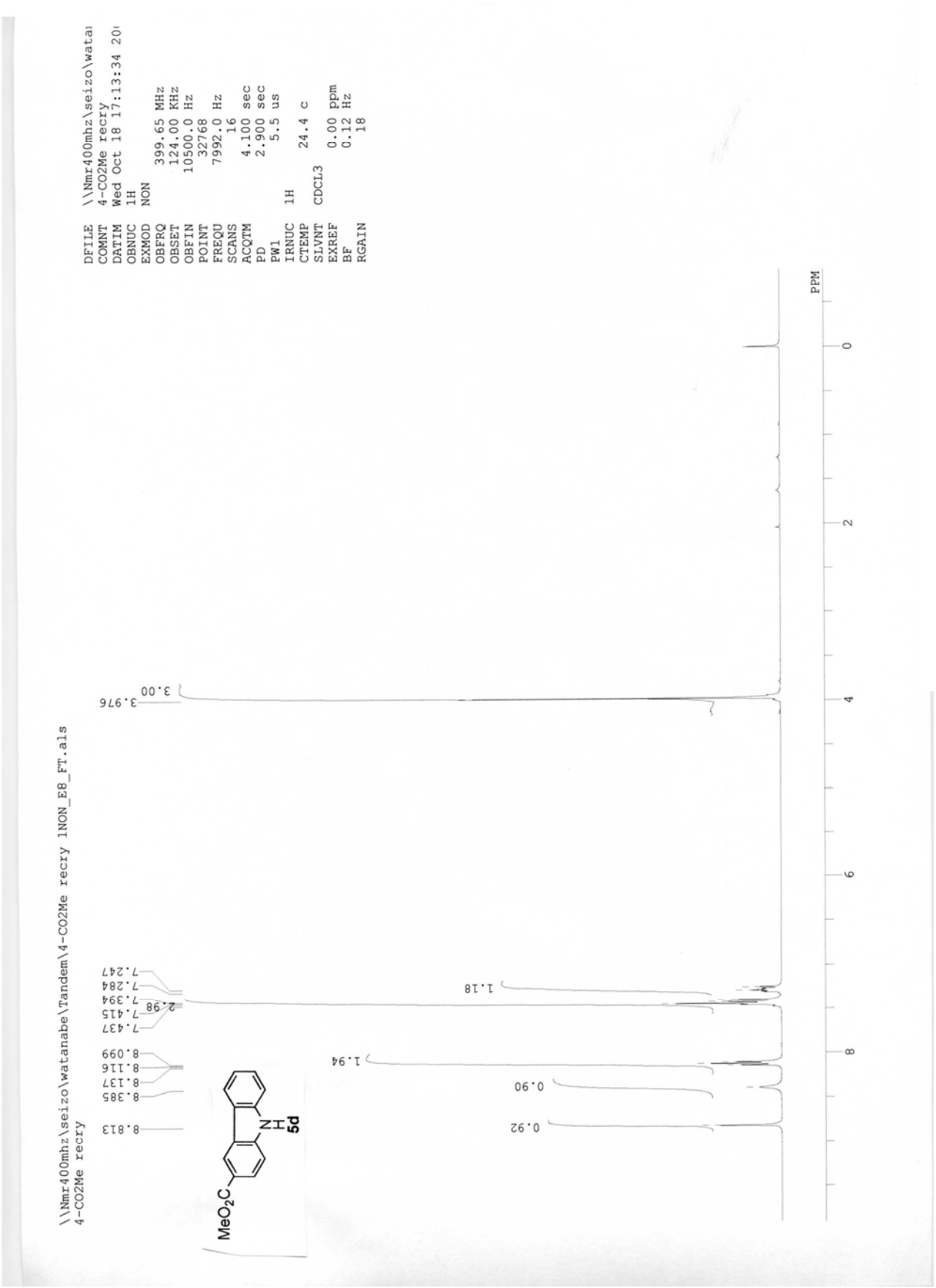
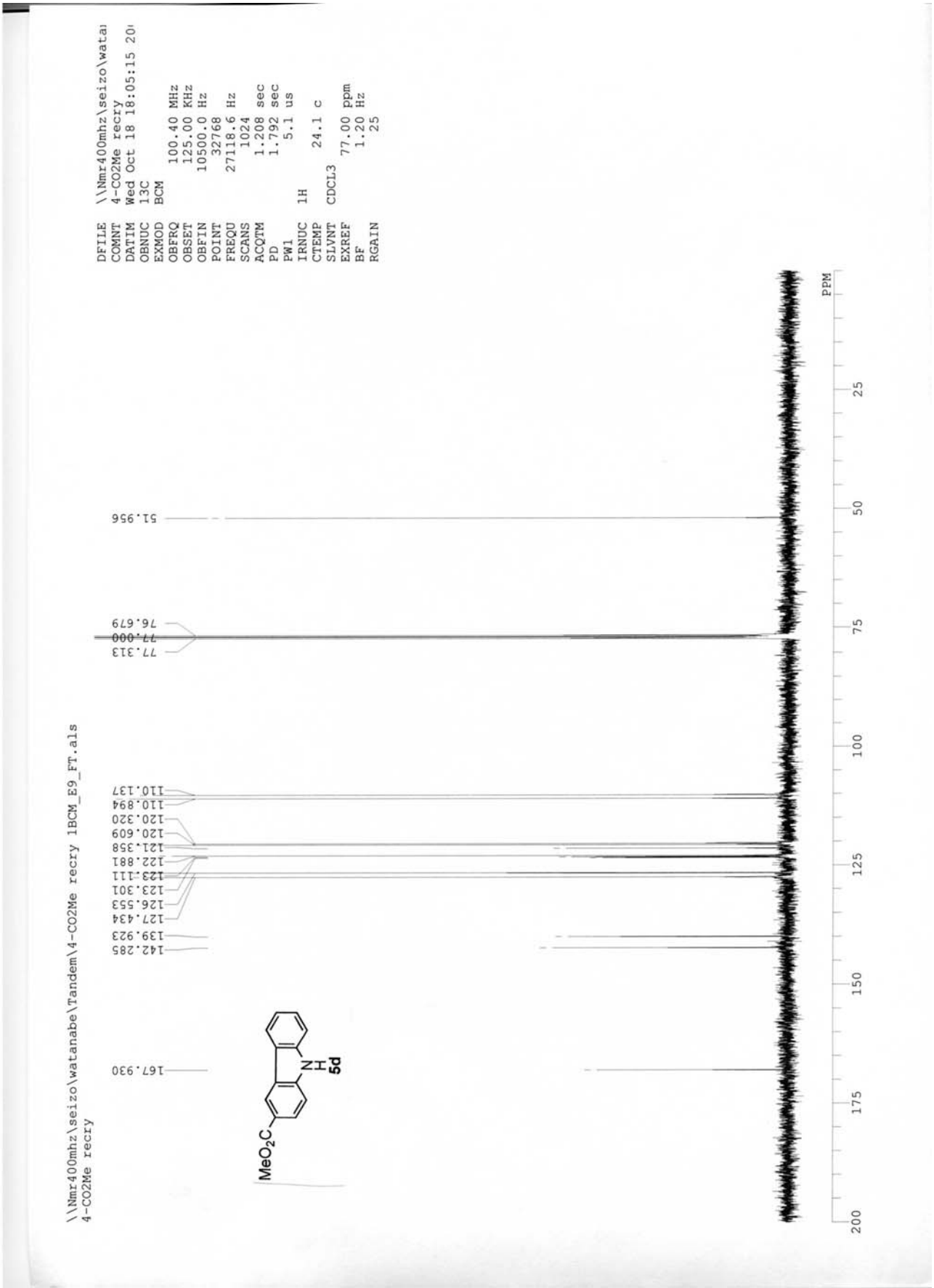
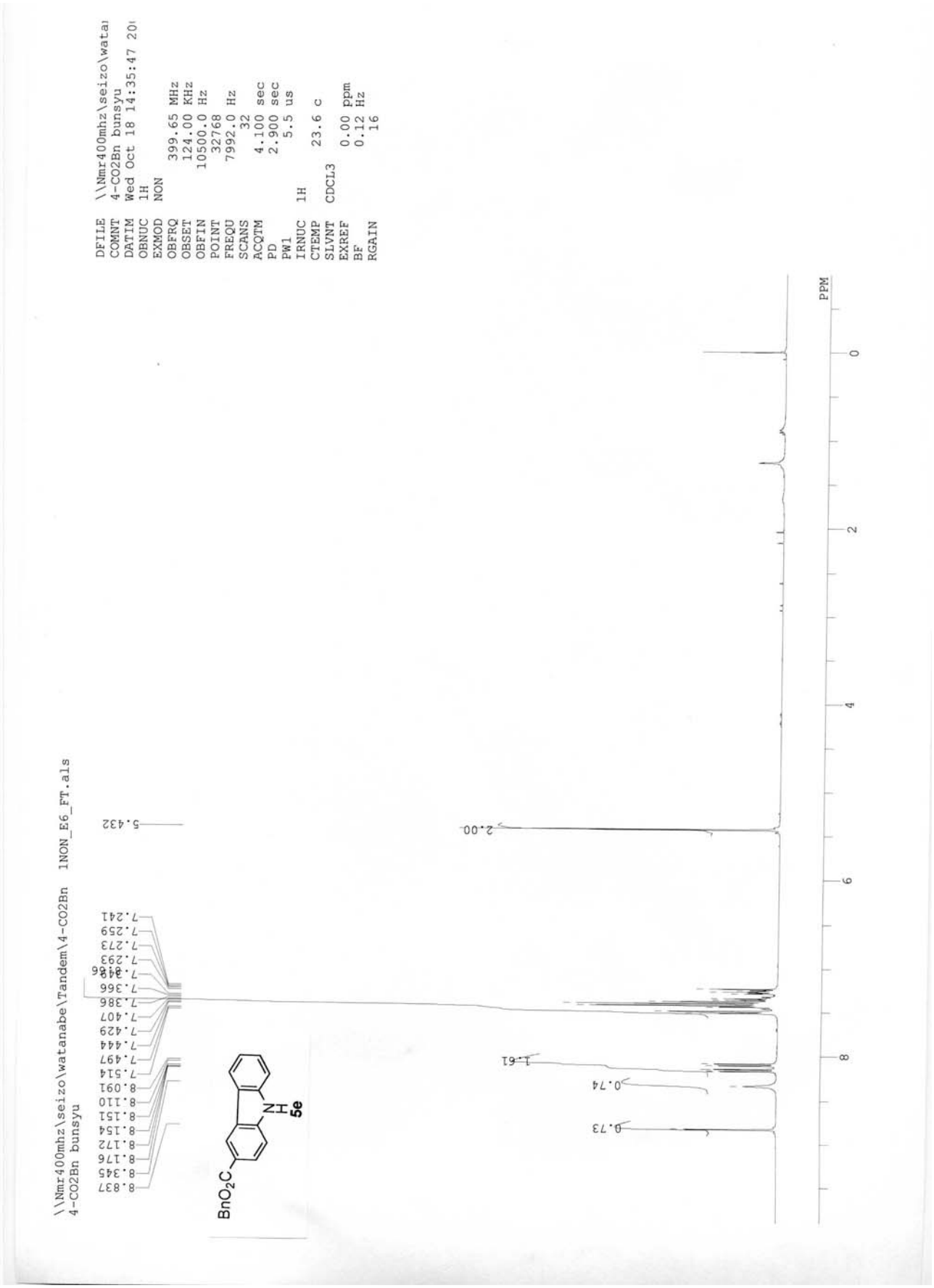
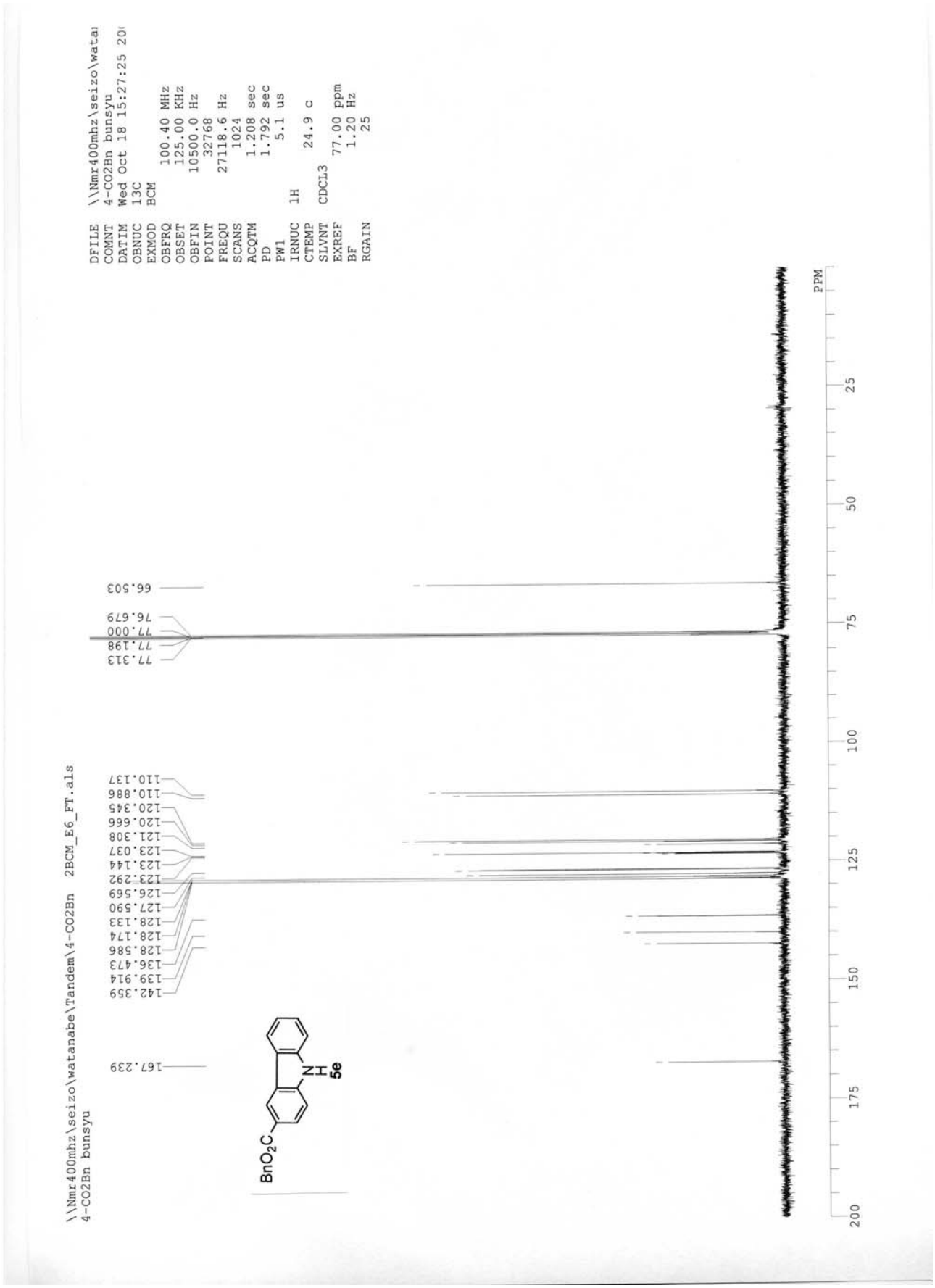
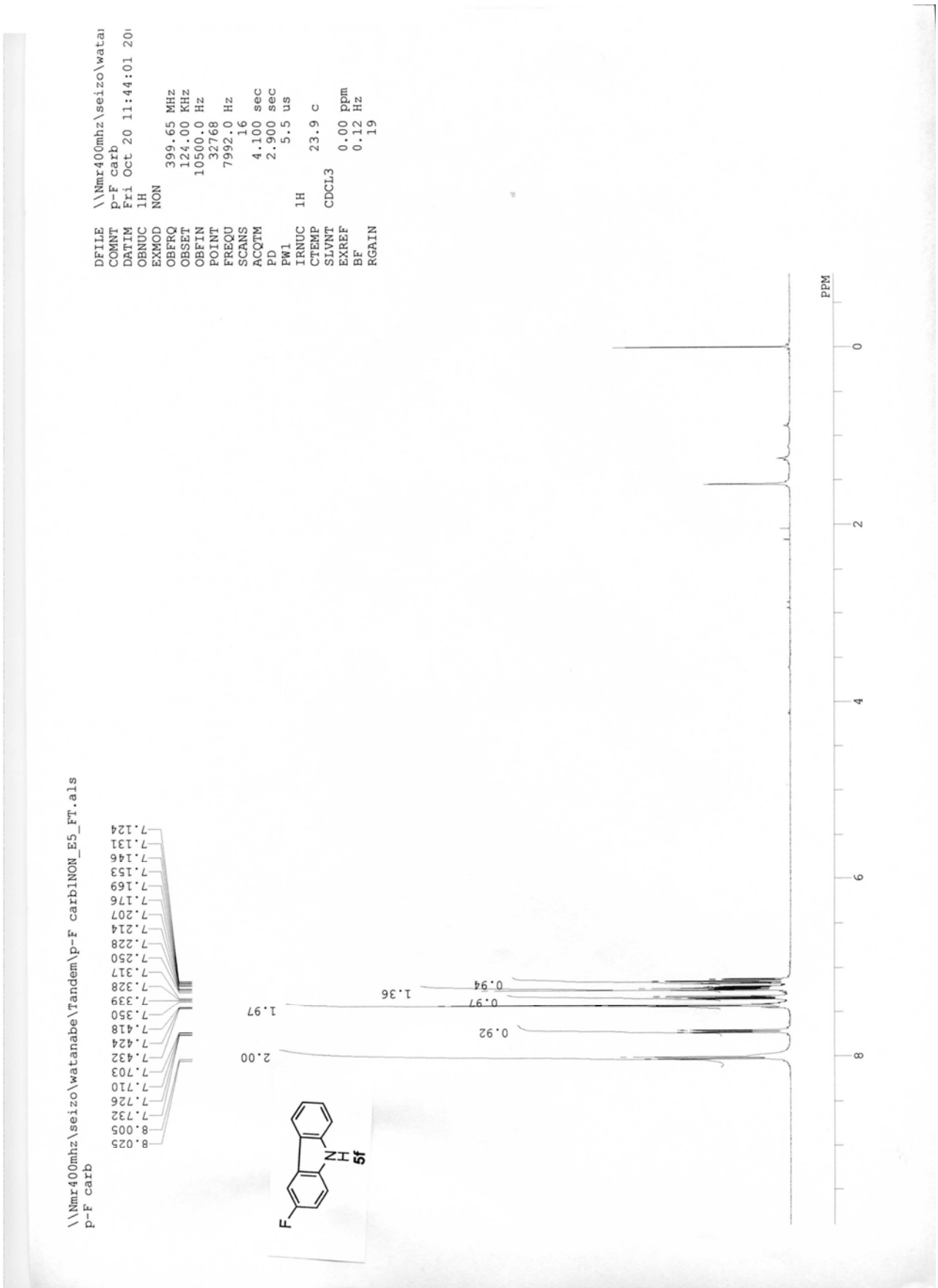
136.021
139.478
139.972
PPM
0
25
50
75
100
125
150
175
200

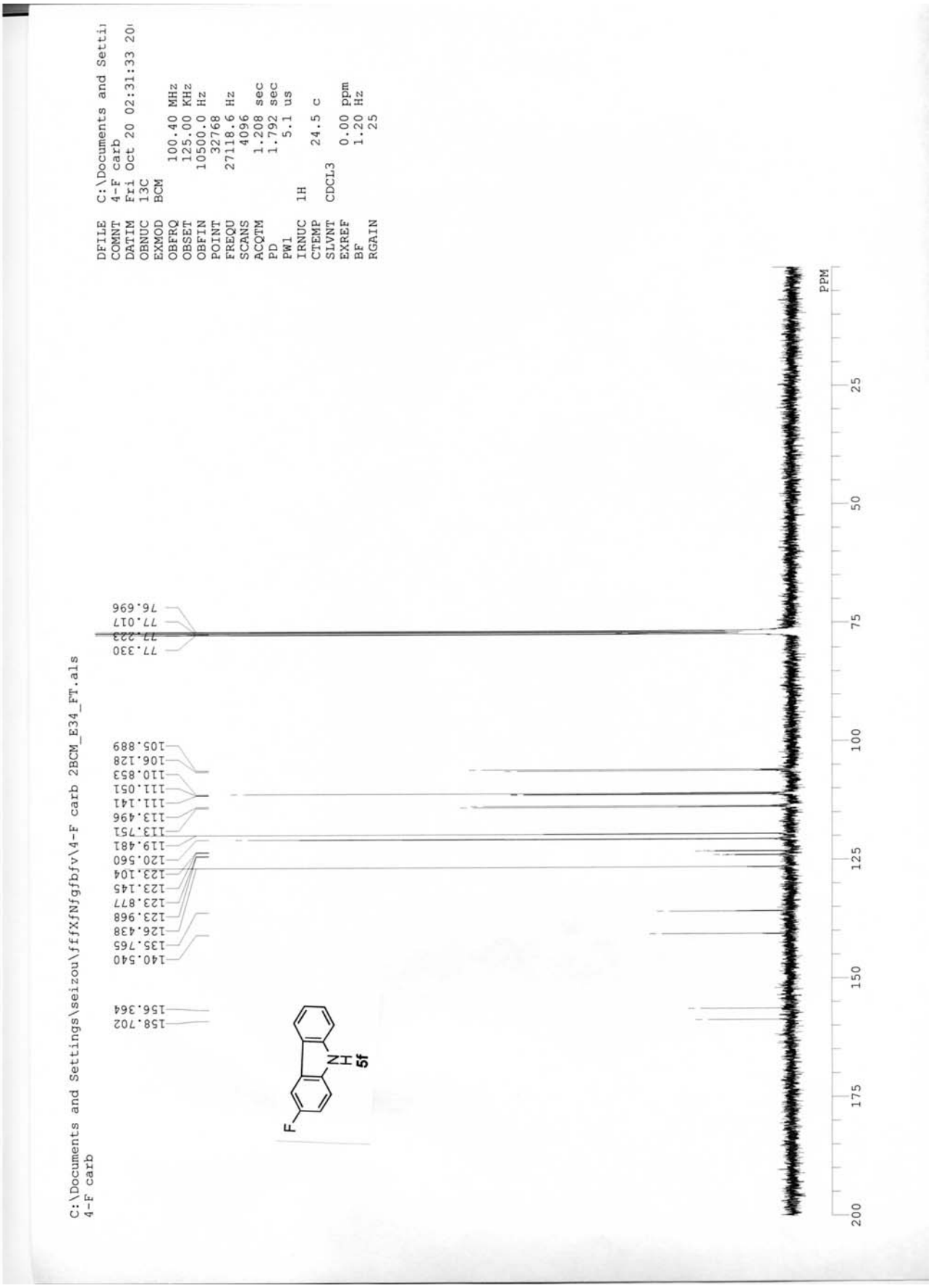

\\Nmr400mhz\seizo\watanabe\Tandem\4-CO2Me recry 1NON_E8_FT.als
4-CO2Me recry
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-CO2Me recry
DATIM Wed Oct 18 17:13:34 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 16
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18
3.976
3.00
8.813
8.385
8.137
8.116
8.099
7.437
7.415
7.394
2.98
7.284
7.247
1.94
1.18
0.90
0.92
MeO2C
NH
5d
PPM
0
2
4
6
8

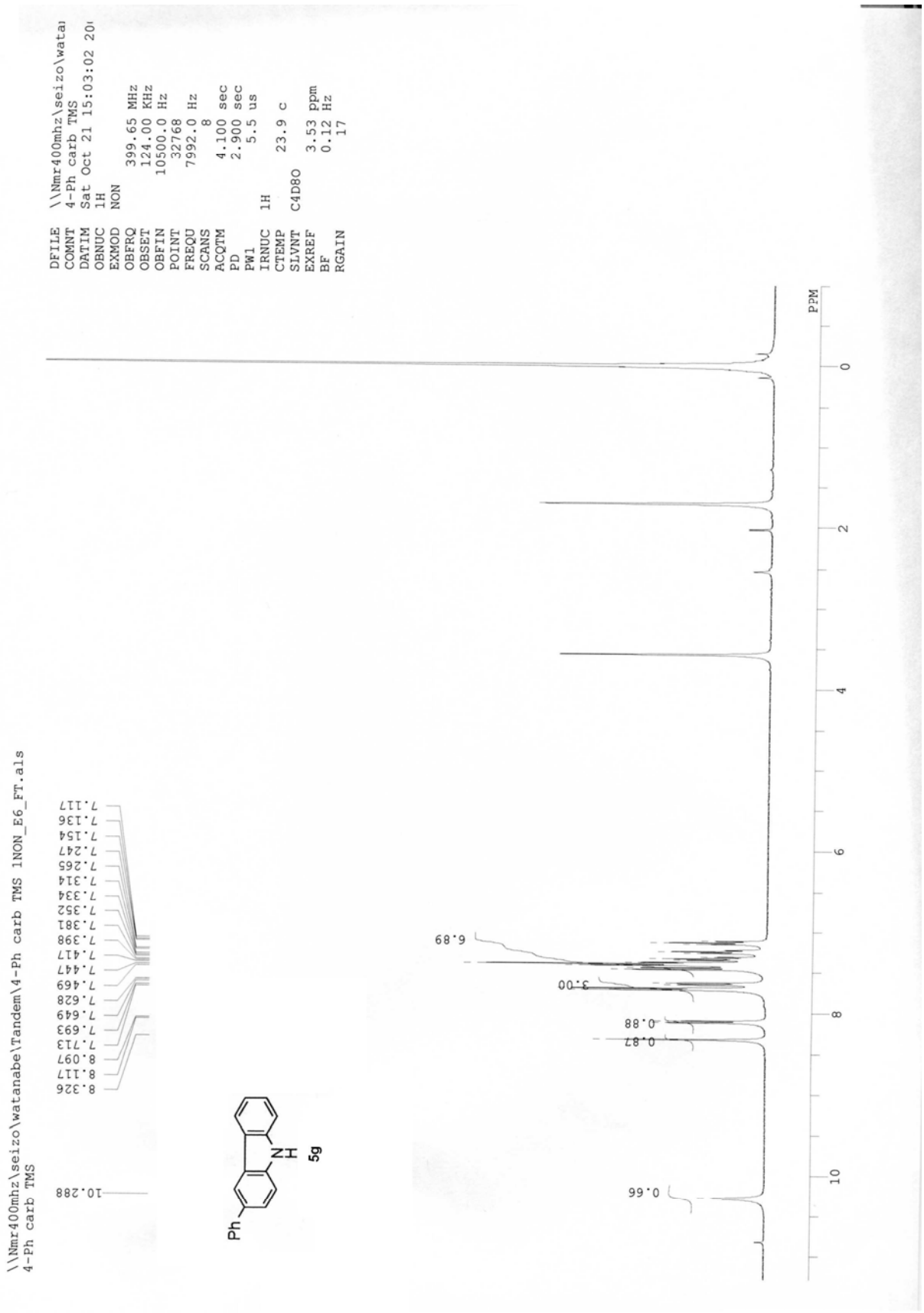

\\Nmr400mhz\seizo\watanabe\Tandem\4-CO2Me recry 1BCM_E9_FT.als
4-CO2Me recry
\\Nmr400mhz\seizo\wata
4-CO2Me recry
Wed Oct 18 18:05:15 20
13C
BCM
100.40 MHz
125.00 KHz
10500.0 Hz
32768
27118.6 Hz
1024
1.208 sec
1.792 sec
5.1 us
1H
24.1 c
CDCL3
77.00 ppm
1.20 Hz
25
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
51.956
76.679
77.000
77.313
110.137
110.894
120.320
120.609
121.358
122.881
123.111
123.301
126.553
127.434
139.923
142.285
167.930
MeO2C
NH
5d
PPM
25
50
75
100
125
150
175
200

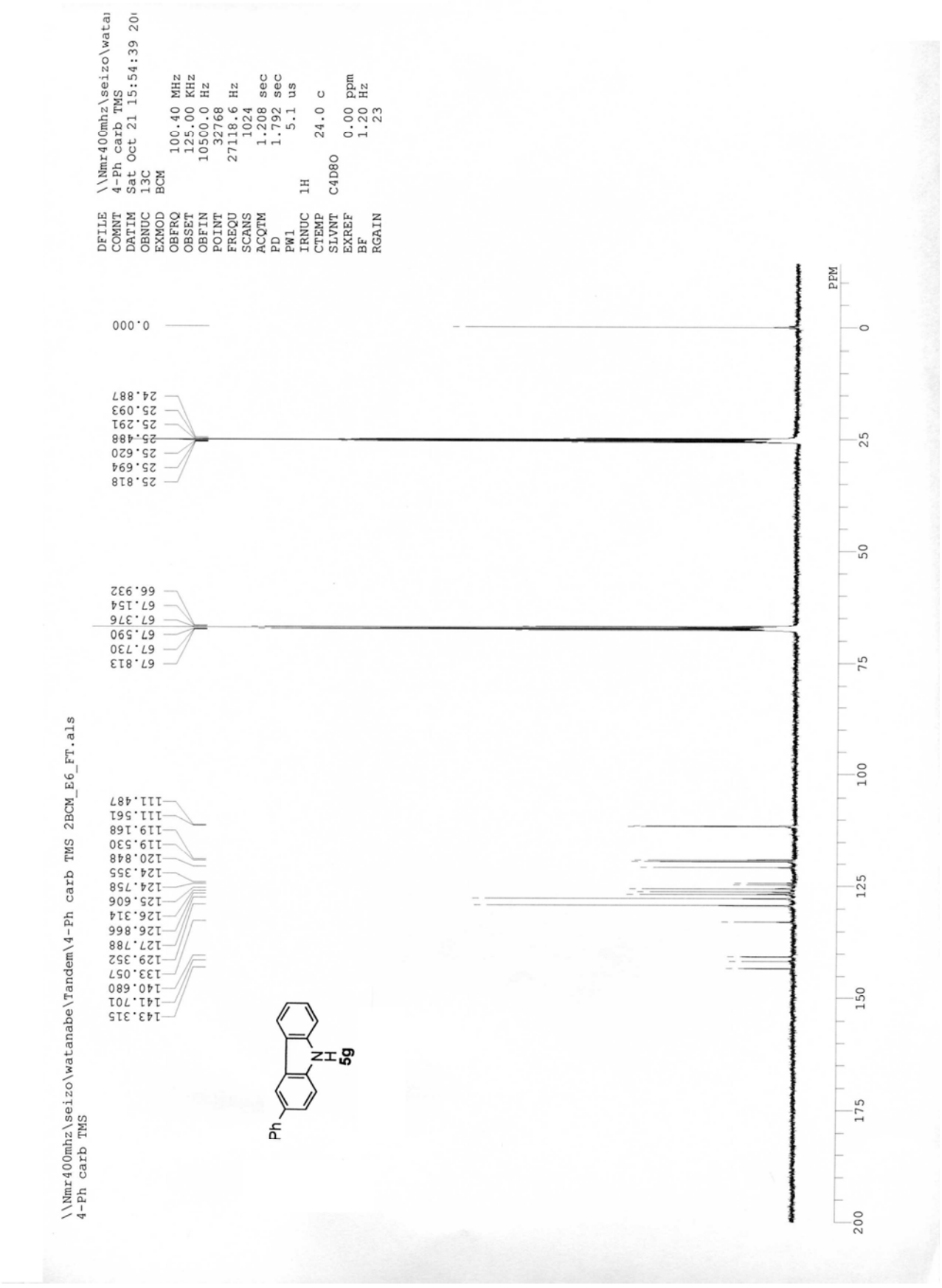

\\Nmr400mhz\seizo\watanabe\Tandem\4-CO2Bn 1NON_E6_FT.als
4-CO2Bn bunsyu
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-CO2Bn bunsyu
DATIM Wed Oct 18 14:35:47 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 32
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
5.432
2.00
1.61
0.74
0.73
8.837
8.345
8.176
8.172
8.154
8.151
8.110
8.091
7.514
7.497
7.444
7.429
7.407
7.386
7.366
7.348
7.293
7.273
7.259
7.241
BnO2C
NH
5e
PPM
0
2
4
6
8

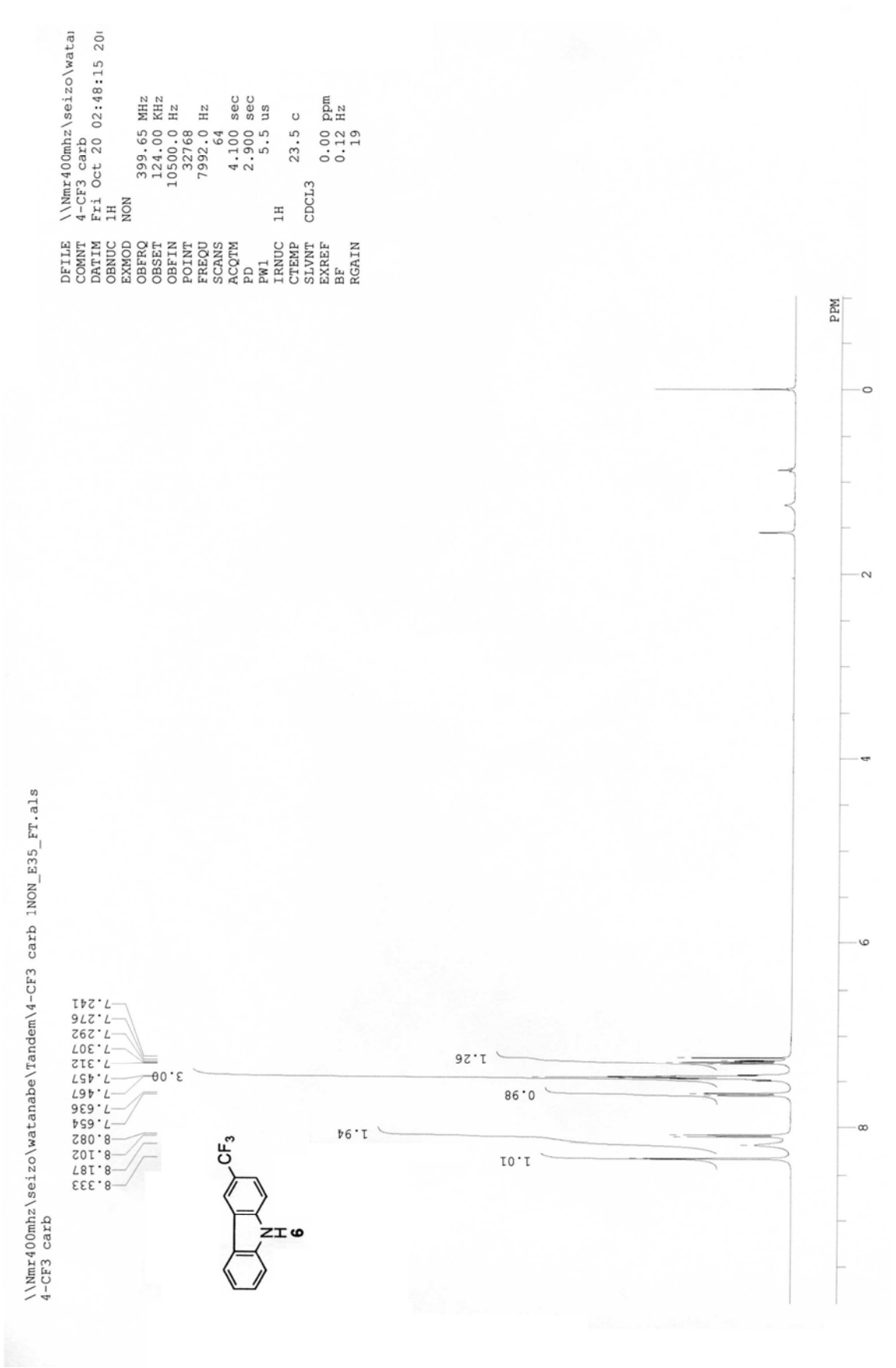

DFILE \\Nmr400mhz\seizo\wata
COMNT 4-CO2Bn bunsyu
DATIM Wed Oct 18 15:27:25 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 1024
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
\\Nmr400mhz\seizo\watanabe\Tandem\4-CO2Bn 2BCM_E6_FT.als
4-CO2Bn bunsyu
66.503
76.679
77.000
77.198
77.313
110.137
110.886
120.345
120.666
121.308
123.037
123.144
123.292
126.569
127.590
128.133
128.174
128.586
136.473
139.914
142.359
167.239
BnO2C
NH
5e
PPM
200
175
150
125
100
75
50
25

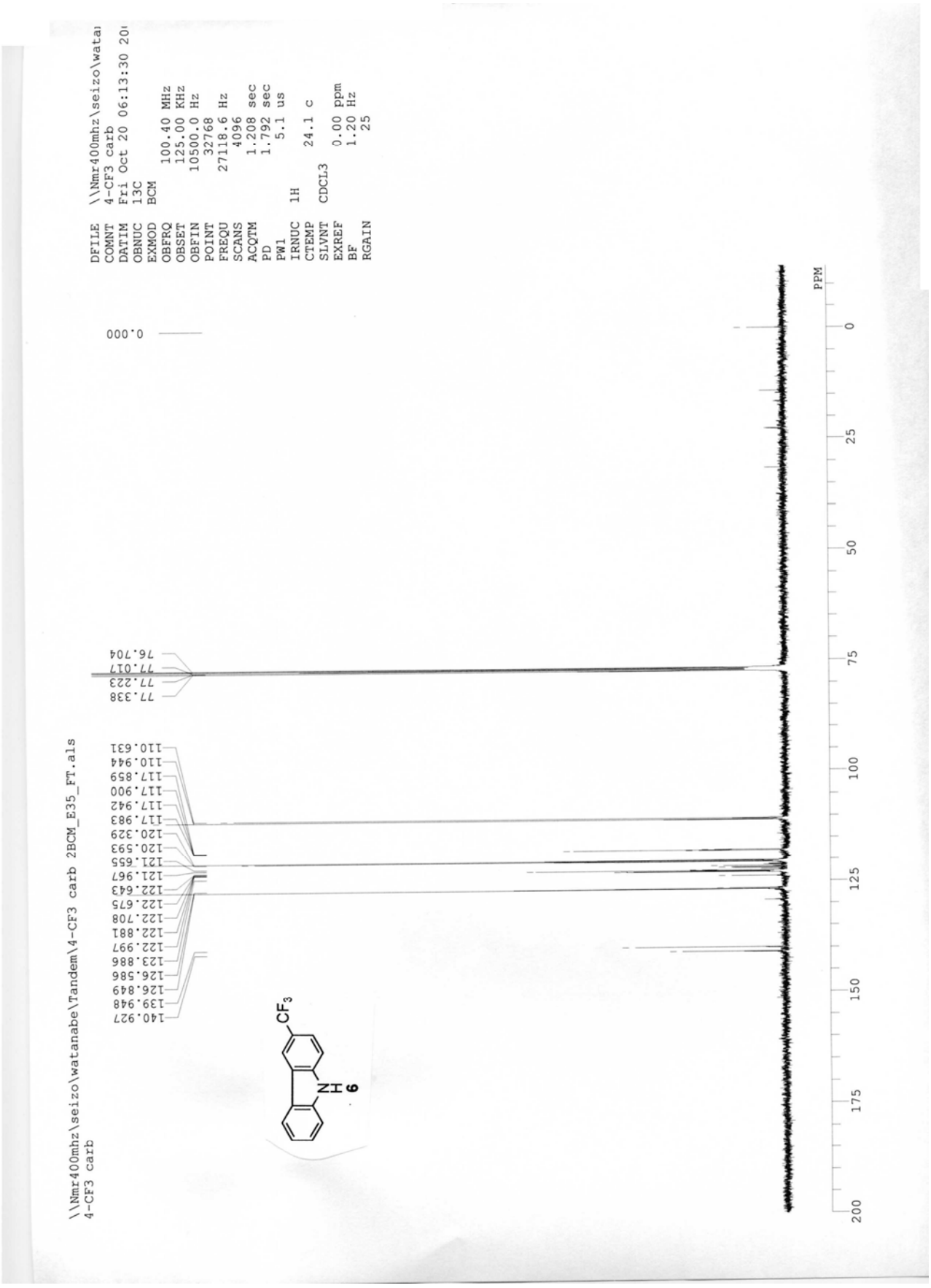

\\Nmr400mhz\seizo\watanabe\Tandem\p-F carb1NON_E5_FT.als
p-F carb
DFILE \\Nmr400mhz\seizo\wata
COMNT p-F carb
DATIM Fri Oct 20 11:44:01 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 16
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
7.124
7.131
7.146
7.153
7.169
7.176
7.207
7.214
7.228
7.250
7.317
7.328
7.339
7.350
7.418
7.424
7.432
7.703
7.710
7.726
7.732
8.005
8.025
2.00
1.97
1.36
0.94
0.97
0.92
5f
F
NH
PPM
0
2
4
6
8

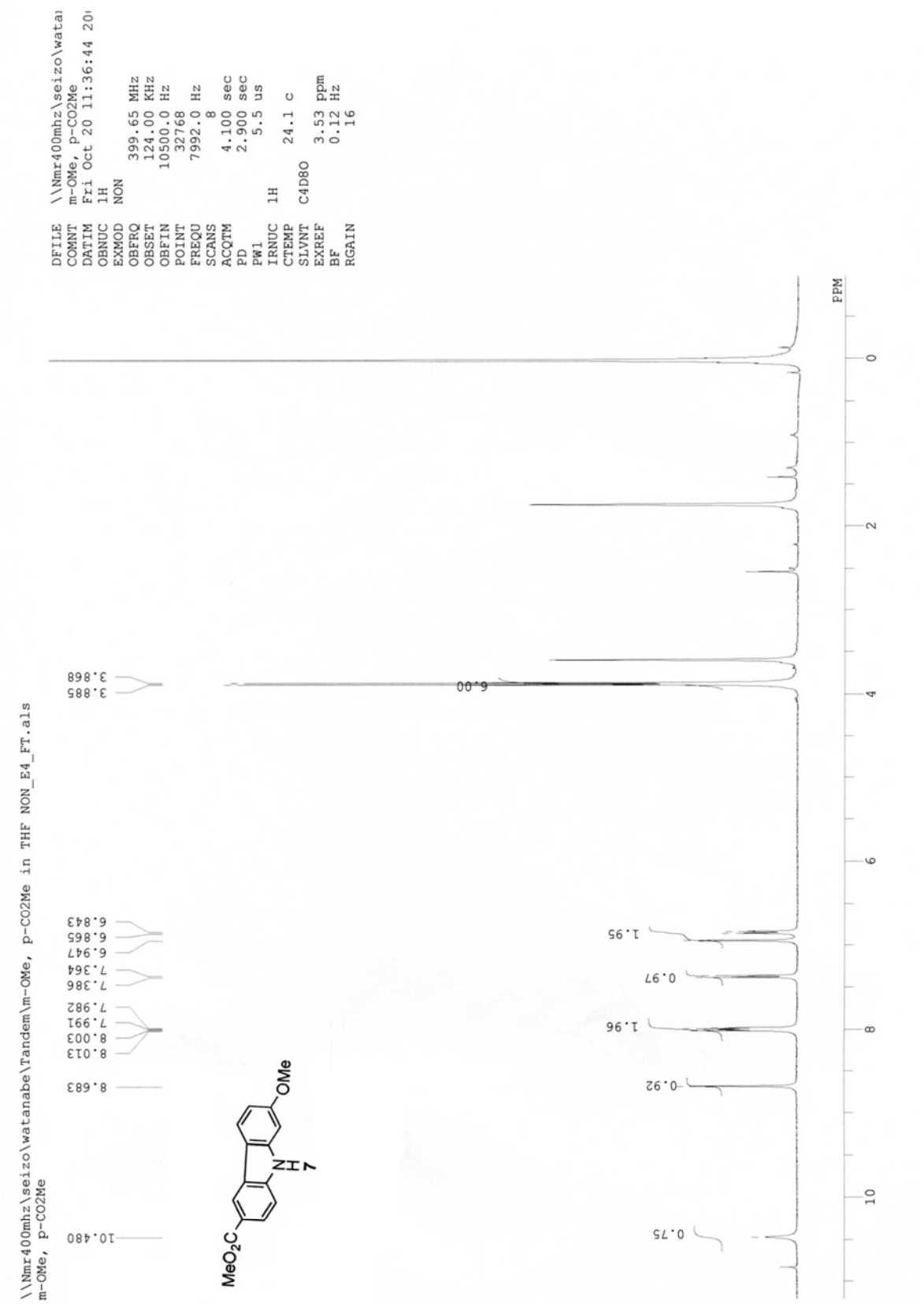

C:\Documents and Settings\seizou\fffXfNfgfbfv\4-F carb 2BCM_E34_FT.als
4-F carb
DFILE C:\Documents and Setti
COMNT 4-F carb
DATIM Fri Oct 20 02:31:33 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 4096
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 25
77.330
77.223
77.017
76.696
158.702
156.364
140.540
135.765
126.438
123.968
123.877
123.145
123.104
120.560
119.481
113.751
113.496
111.141
111.051
110.853
106.128
105.889
PPM
200
175
150
125
100
75
50
25
5f
F
N
H

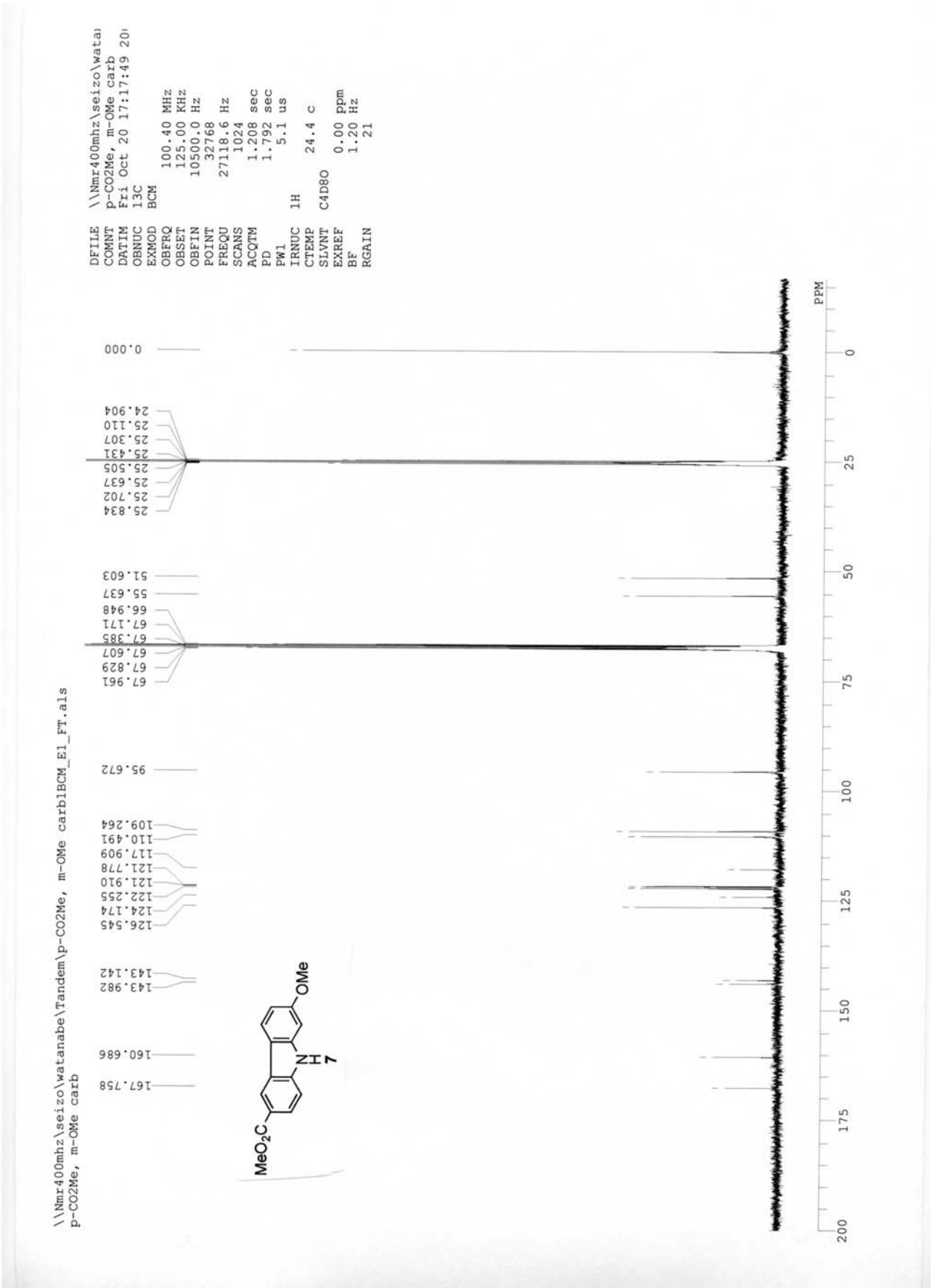

\\Nmr400mhz\seizo\watanabe\Tandem\4-Ph carb TMS 1NON_E6_FT.als
4-Ph carb TMS
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-Ph carb TMS
DATIM Sat Oct 21 15:03:02 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 8
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.9 c
SLVNT C4D8O
EXREF 3.53 ppm
BF 0.12 Hz
RGAIN 17
10.288
8.326
8.117
8.097
7.713
7.693
7.649
7.628
7.469
7.447
7.417
7.398
7.381
7.352
7.334
7.314
7.265
7.247
7.154
7.136
7.117
Ph
NH
5g
6.89
3.00
0.88
0.87
0.66
PPM
0
2
4
6
8
10

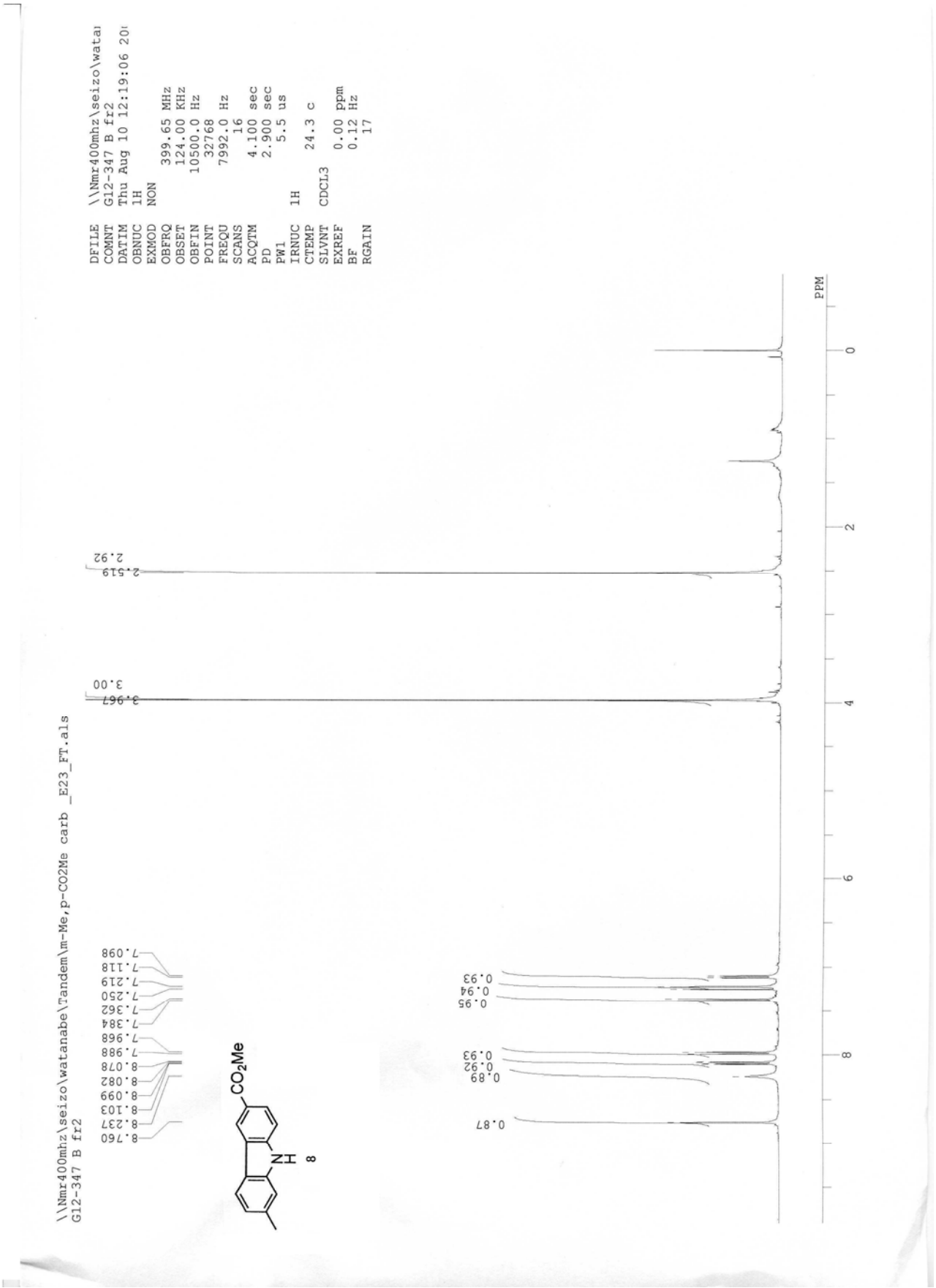

\\Nmr400mhz\seizo\watanabe\Tandem\4-Ph carb TMS 2BCM_E6_FT.als
4-Ph carb TMS
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-Ph carb TMS
DATIM Sat Oct 21 15:54:39 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 1024
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.0 c
SLVNT C4D8O
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 23
0.000
24.887
25.093
25.291
25.488
25.620
25.694
25.818
66.932
67.154
67.376
67.590
67.730
67.813
111.487
111.561
119.168
119.530
120.848
124.355
124.758
125.606
126.314
126.866
127.788
129.352
133.057
140.680
141.701
143.315
Ph
N
H
5g
PPM
0
25
50
75
100
125
150
175
200

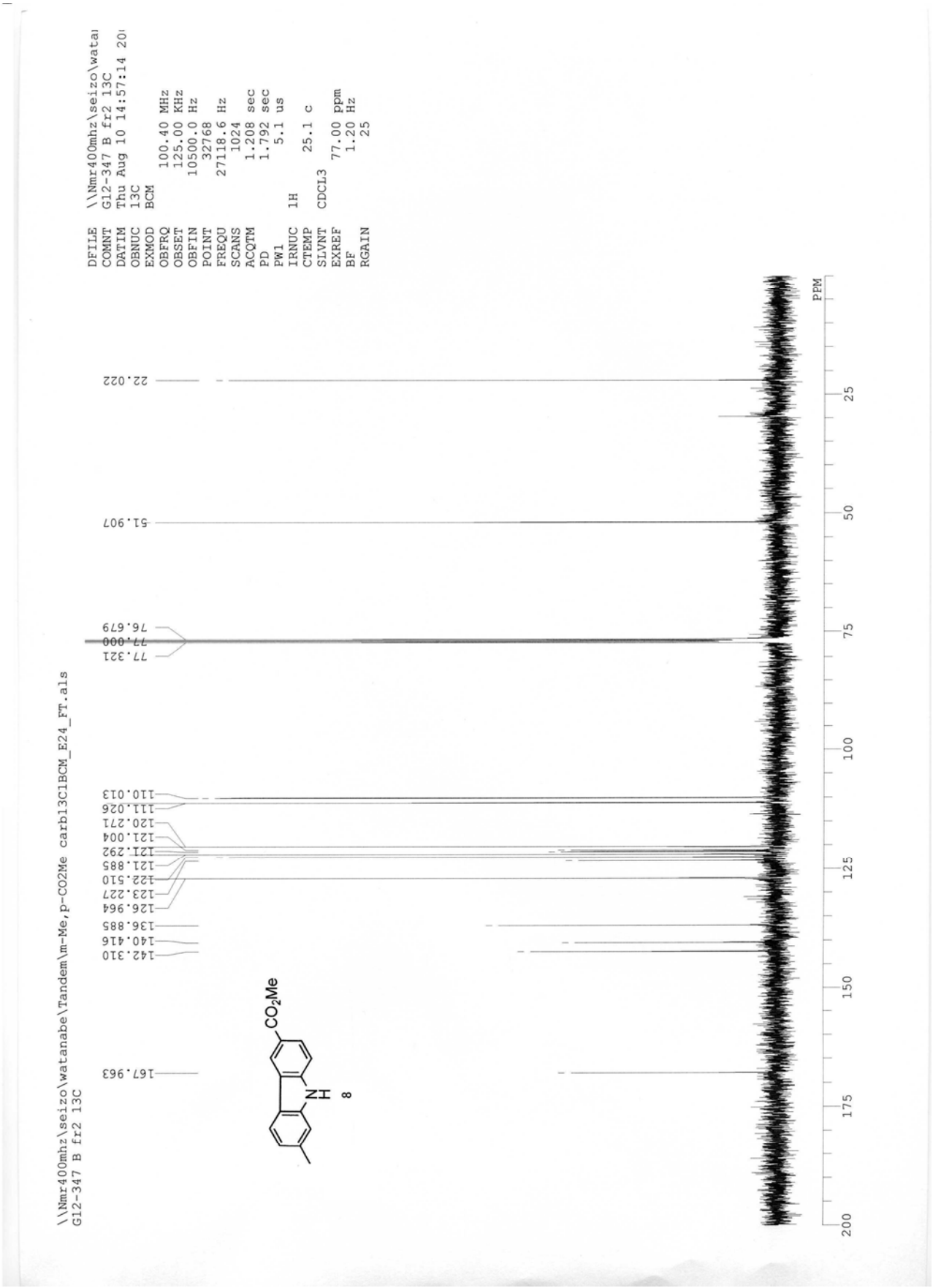

\\Nmr400mhz\seizo\watanabe\Tandem\4-CF3 carb 1NON_E35_FT.als
4-CF3 carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-CF3 carb
DATIM Fri Oct 20 02:48:15 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 64
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
7.241
7.276
7.292
7.307
7.312
7.457
7.467
7.636
7.654
8.082
8.102
8.187
8.333
3.00
1.26
0.98
1.94
1.01
CF3
NH
6
PPM
0
2
4
6
8

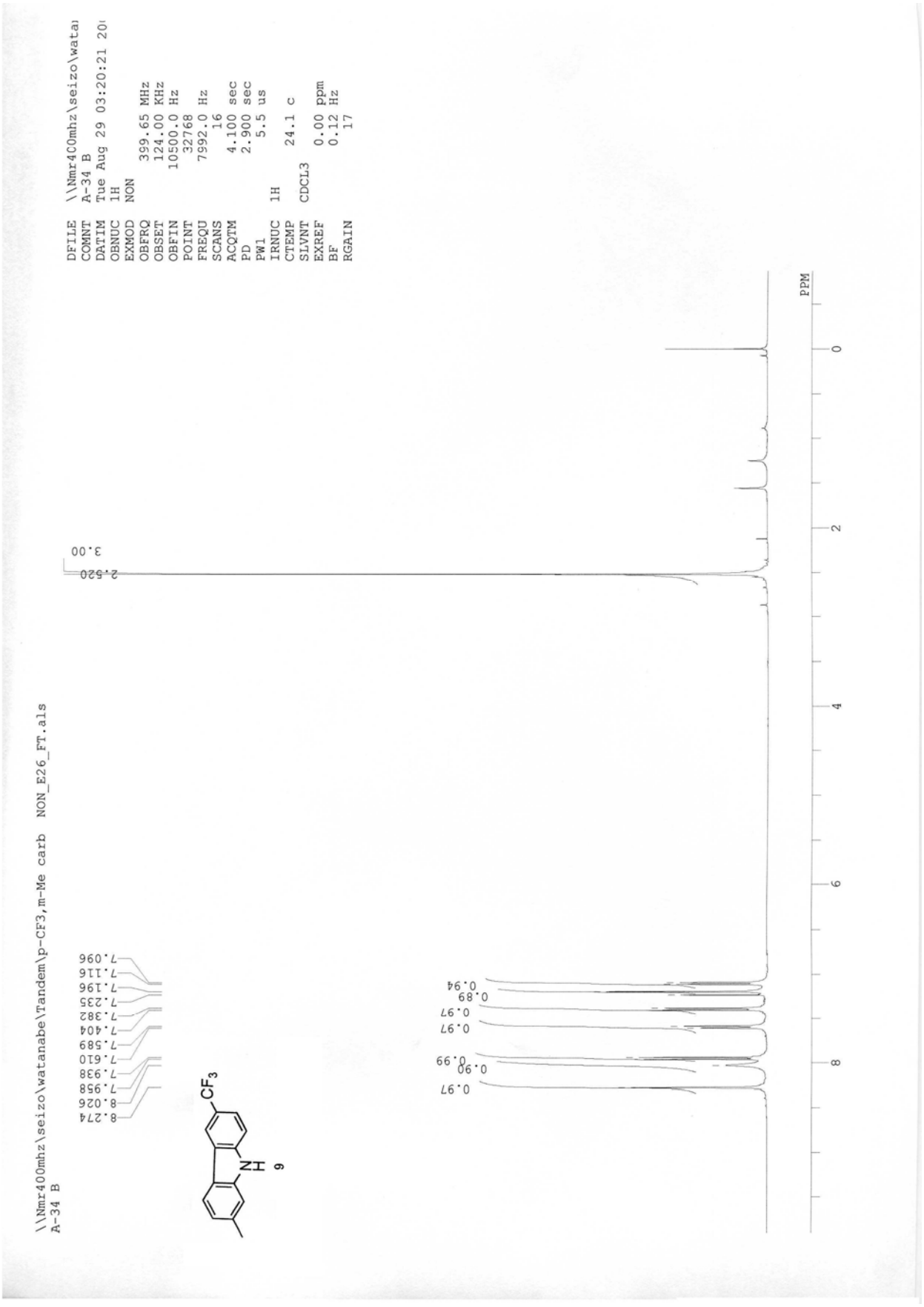

\\Nmr400mhz\seizo\watanabe\Tandem\4-CF3 carb 2BCM_E35_FT.als
4-CF3 carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-CF3 carb
DATIM Fri Oct 20 06:13:30 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 4096
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 25
0.000
140.927
139.948
126.849
126.586
123.886
122.997
122.881
122.708
122.675
122.643
121.967
121.655
120.593
120.329
117.983
117.942
117.900
117.859
110.944
110.631
77.338
77.223
77.017
76.704
CF3
NH
6
PPM
0
25
50
75
100
125
150
175
200

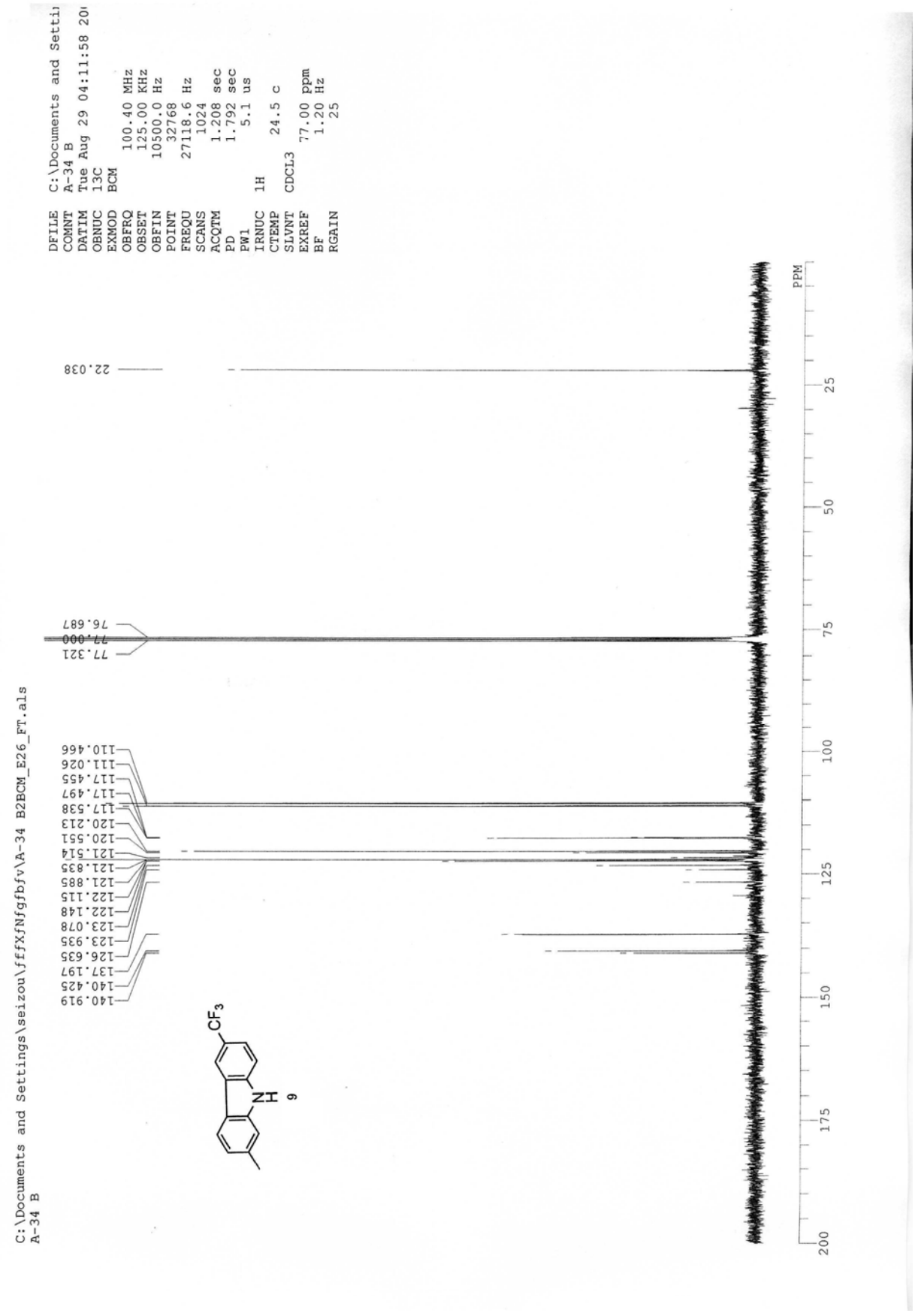

\\Nmr400mhz\seizo\watanabe\Tandem\m-OMe, p-CO2Me in THF NON_E4_FT.als
m-OMe, p-CO2Me
DFILE \\Nmr400mhz\seizo\wata
COMNT m-OMe, p-CO2Me
DATIM Fri Oct 20 11:36:44 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 8
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.1 c
SLVNT C4D8O
EXREF 3.53 ppm
BF 0.12 Hz
RGAIN 16
10.480
8.683
8.013
8.003
7.991
7.982
7.386
7.364
6.947
6.865
6.843
3.885
3.868
6.00
1.95
0.97
1.96
0.92
0.75
OMe
NH
7
MeO2C
PPM
0
2
4
6
8
10

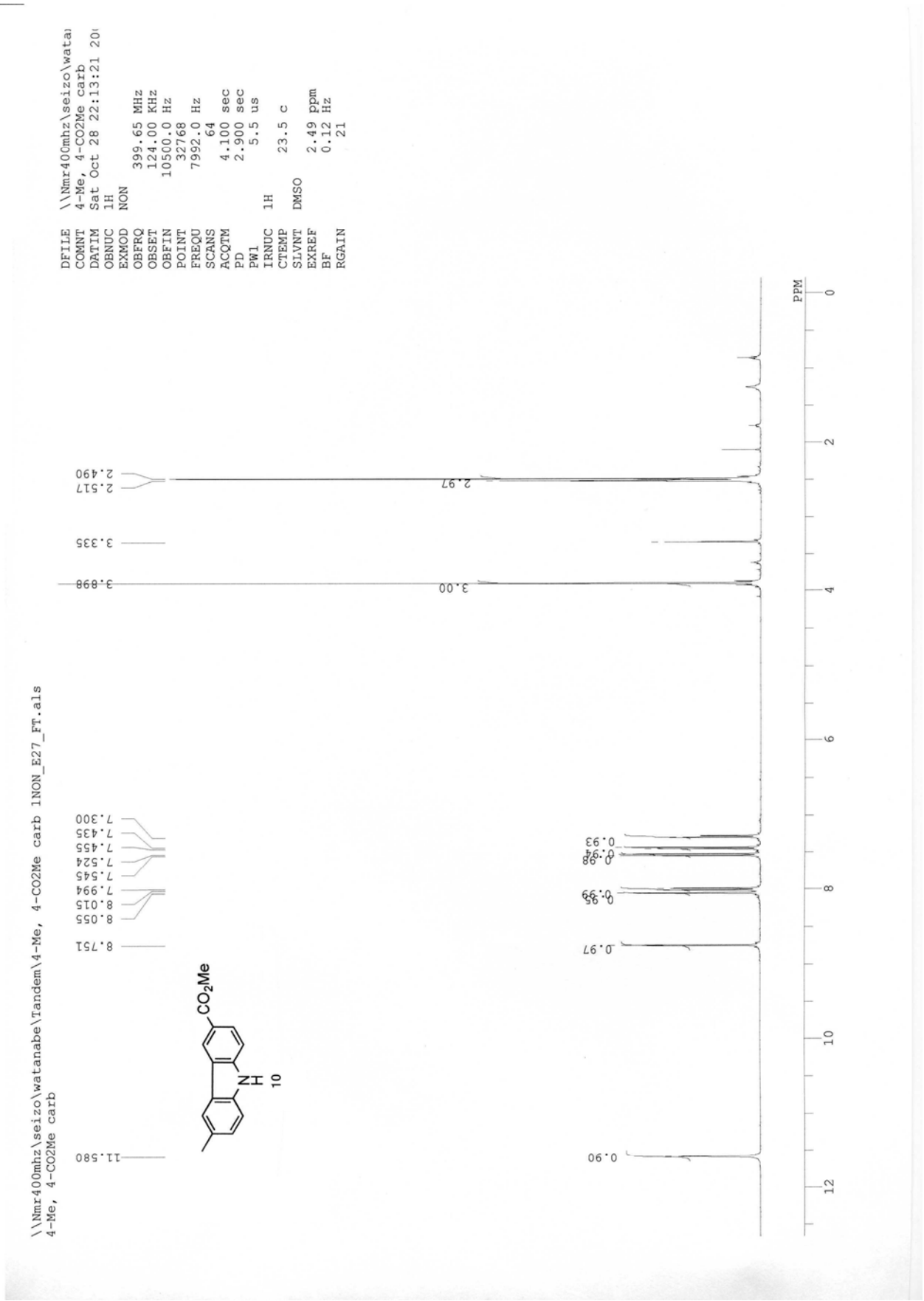

\\Nmr400mhz\seizo\watanabe\Tandem\p-CO2Me, m-OMe carb1BCM_E1_FT.als
p-CO2Me, m-OMe carb
DFILE \\Nmr400mhz\seizo\wata
COMNT p-CO2Me, m-OMe carb
DATIM Fri Oct 20 17:17:49 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 1024
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.4 c
SLVNT C4D8O
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 21
0.000
24.904
25.110
25.307
25.431
25.505
25.637
25.702
25.834
51.603
55.637
66.948
67.171
67.385
67.607
67.829
67.961
95.672
109.264
110.491
117.909
121.778
121.910
122.255
124.174
126.545
143.142
143.982
160.686
167.758
OMe
MeO2C
NH
7
PPM
0
25
50
75
100
125
150
175
200

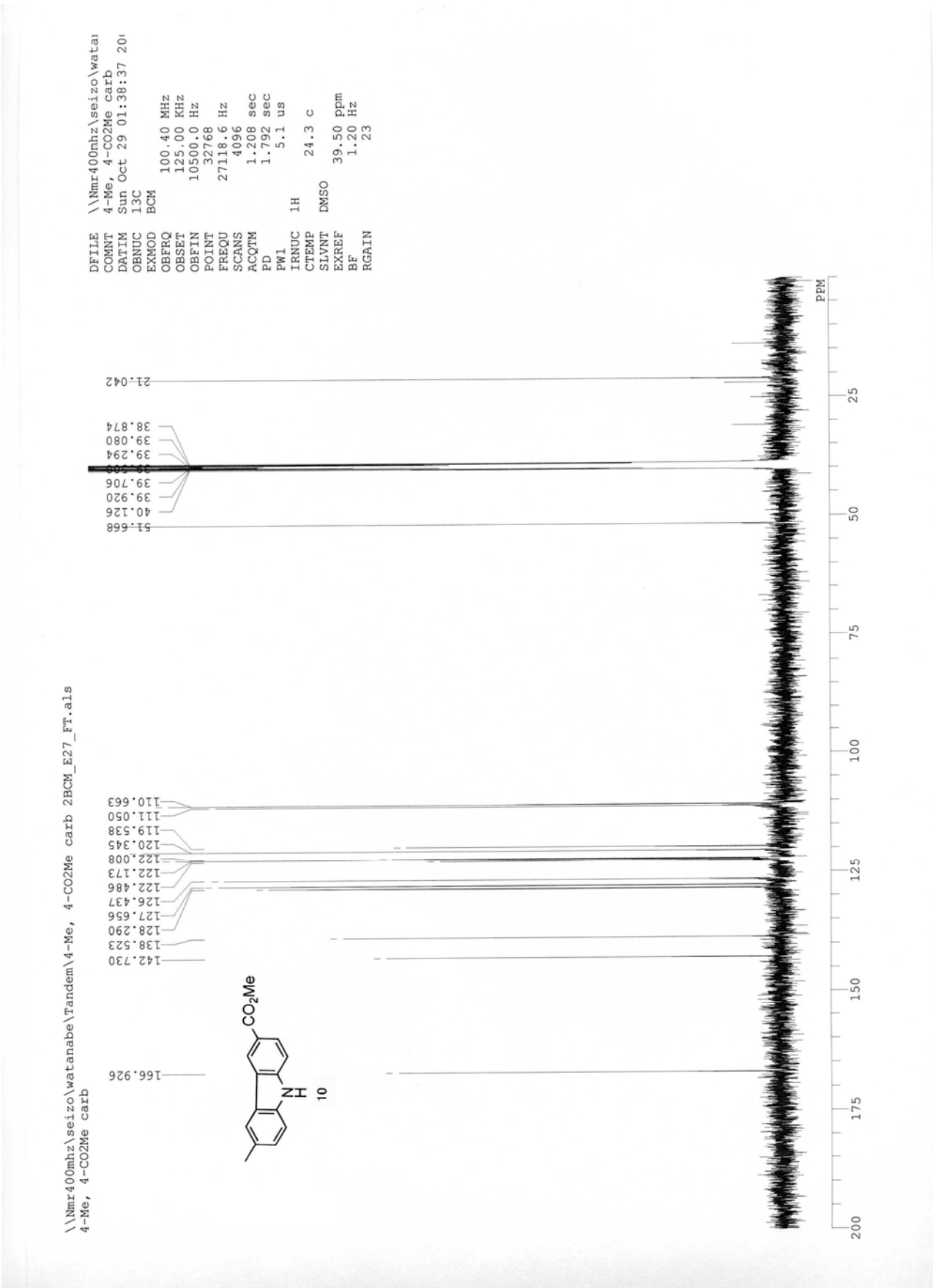

\\Nmr400mhz\seizo\watanabe\Tandem\m-Me,p-CO2Me carb _E23_FT.als
G12-347 B fr2
DFILE \\Nmr400mhz\seizo\wata
COMNT G12-347 B fr2
DATIM Thu Aug 10 12:19:06 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 16
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
CO2Me
NH
8
PPM

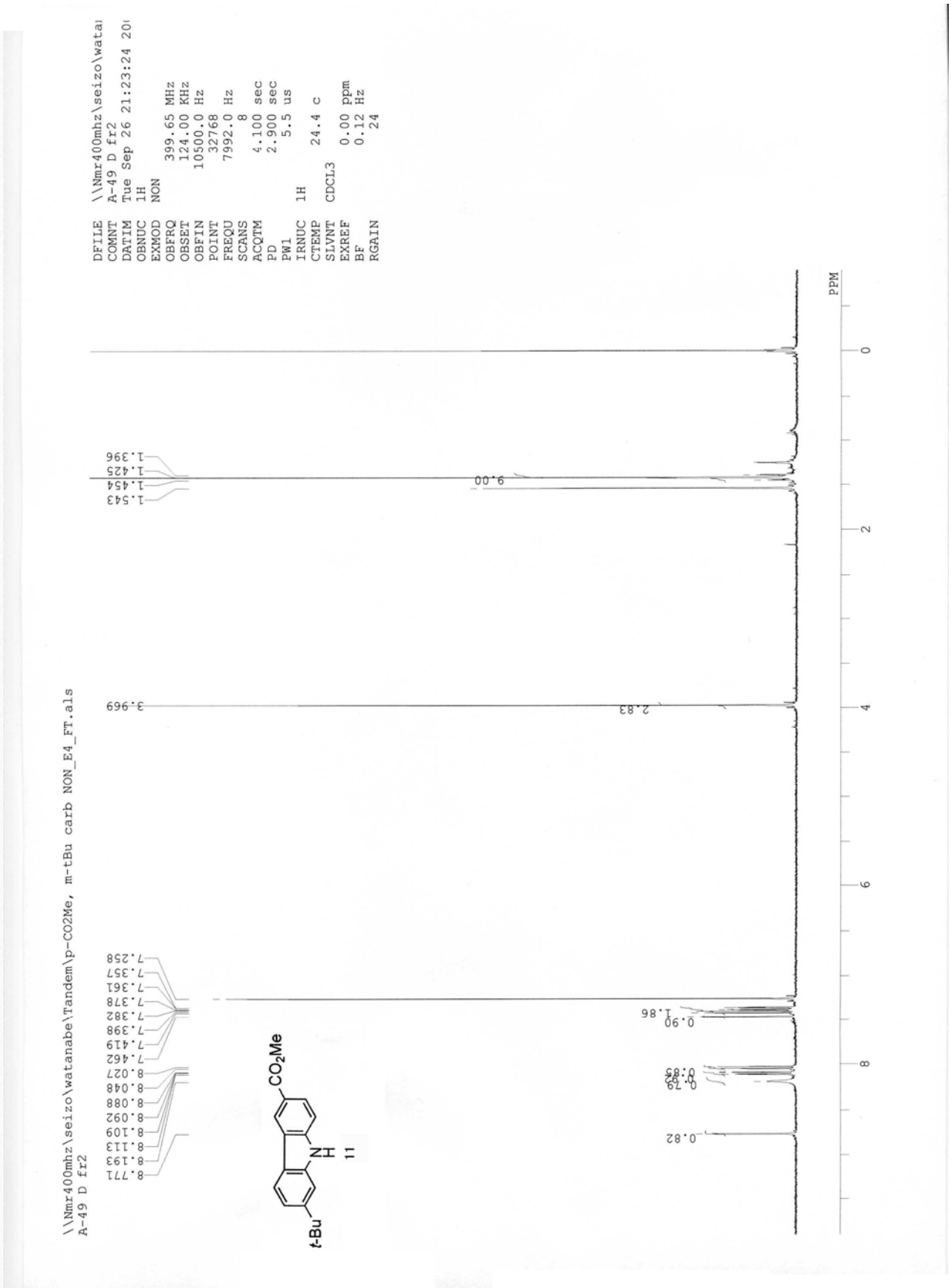

\\Nmr400mhz\seizo\watanabe\Tandem\m-Me,p-CO2Me carb13C1BCM_E24_FT.als
G12-347 B fr2 13C
DFILE \\Nmr400mhz\seizo\wata
COMNT G12-347 B fr2 13C
DATIM Thu Aug 10 14:57:14 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 1024
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 25.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
22.022
51.907
76.679
77.000
77.321
110.013
111.026
120.271
121.004
121.292
121.885
122.510
123.227
126.964
136.885
140.416
142.310
167.963
CO2Me
H
N
8
PPM
25
50
75
100
125
150
175
200

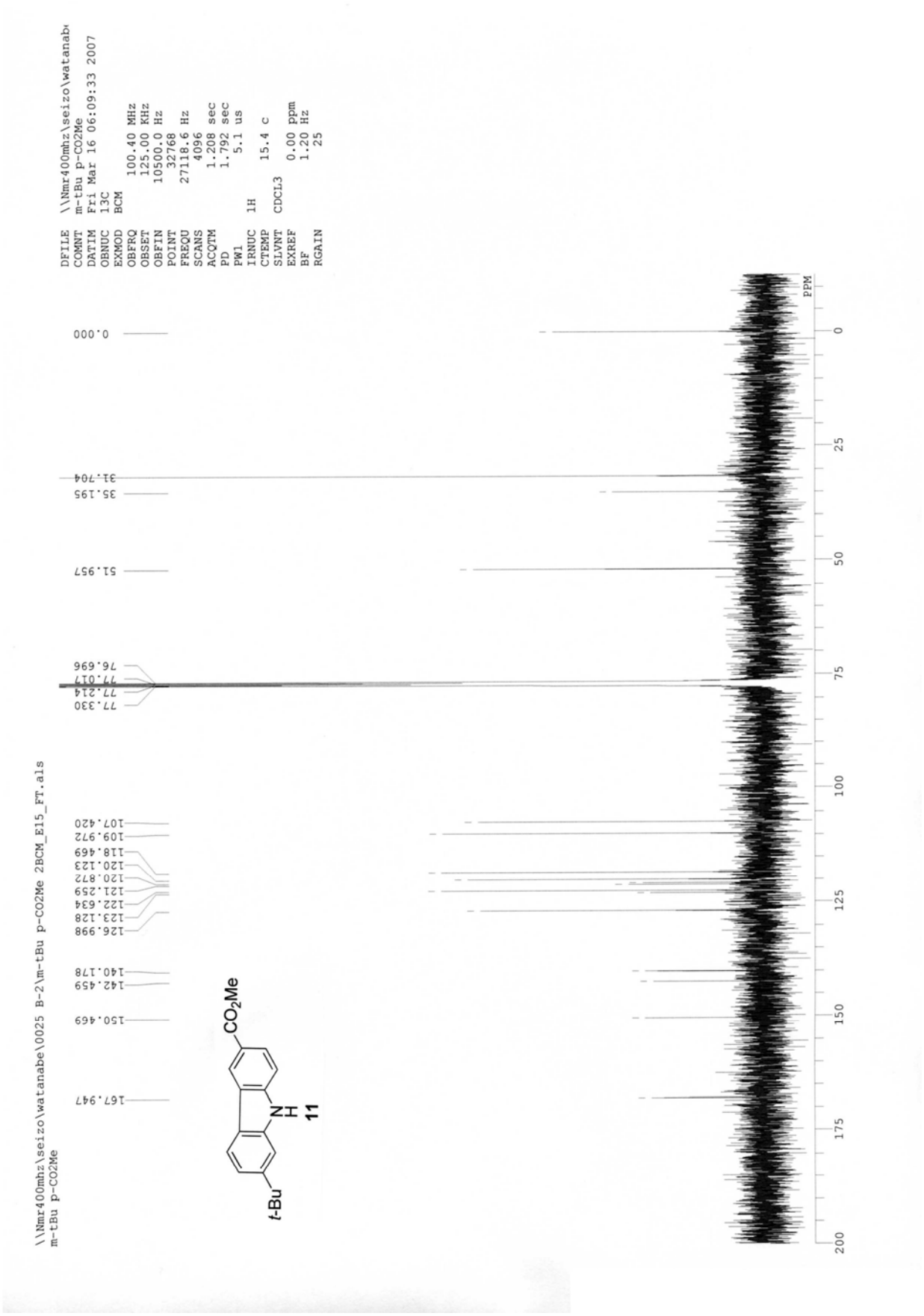

\\Nmr400mhz\seizo\watanabe\Tandem\p-CF3,m-Me carb NON_E26_FT.als
A-34 B
DFILE \\Nmr400mhz\seizo\wata
COMNT A-34 B
DATIM Tue Aug 29 03:20:21 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 16
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
2.520
3.00
8.274
8.026
7.958
7.938
7.610
7.589
7.404
7.382
7.235
7.196
7.116
7.096
0.97
0.90
0.99
0.97
0.97
0.89
0.94
CF3
NH
9
PPM
0
2
4
6
8

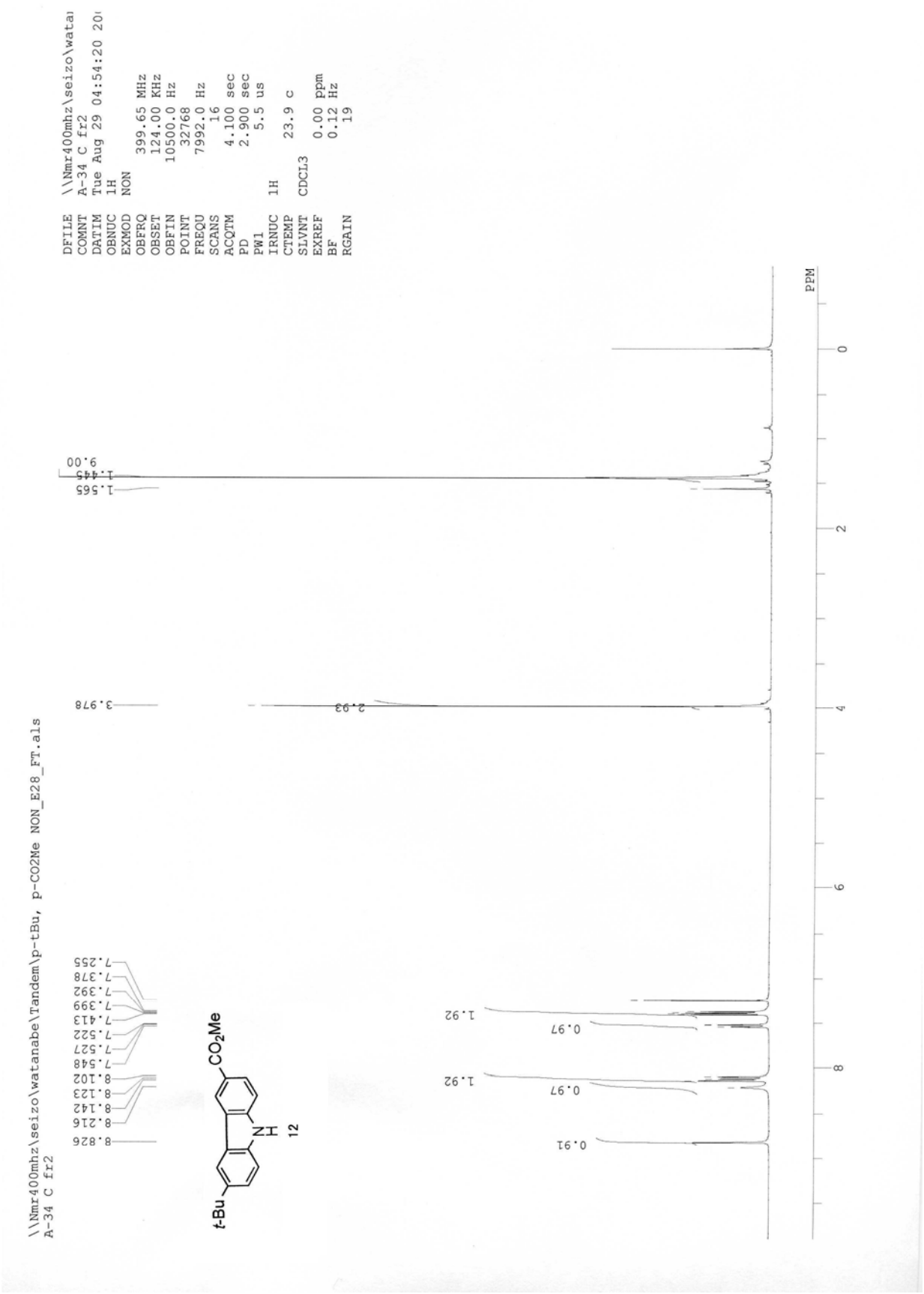

DFILE C:\Documents and Setti
COMNT A-34 B
DATIM Tue Aug 29 04:11:58 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 1024
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
22.038
76.687
77.000
77.321
110.466
111.026
117.455
117.497
117.538
120.213
120.551
121.514
121.835
121.885
122.115
122.148
123.078
123.935
126.635
137.197
140.425
140.919
CF3
N
H
9
PPM
200
175
150
125
100
75
50
25
C:\Documents and Settings\seizou\ffXfNfgfbfv\A-34 B2BCM_E26_FT.als
A-34 B

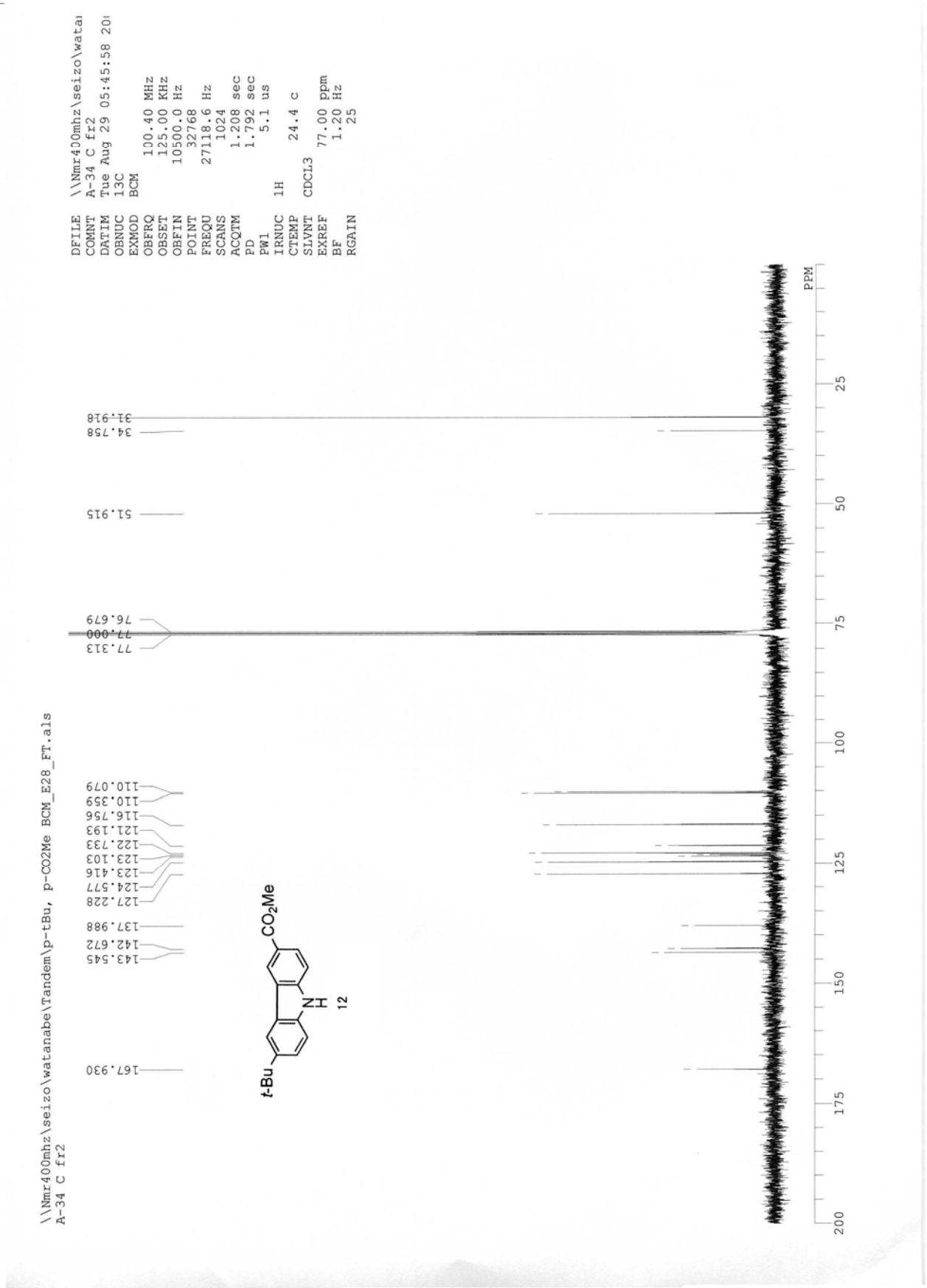

DFILE \\Nmr400mhz\seizo\wata
COMNT 4-Me, 4-CO2Me carb
DATIM Sat Oct 28 22:13:21 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 64
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.5 c
SLVNT DMSO
EXREF 2.49 ppm
BF 0.12 Hz
RGAIN 21
\\Nmr400mhz\seizo\watanabe\Tandem\4-Me, 4-CO2Me carb 1NON_E27_FT.als
4-Me, 4-CO2Me carb
PPM
CO2Me
NH
10


\\Nmr400mhz\seizo\watanabe\Tandem\4-Me, 4-CO2Me carb 2BCM_E27_FT.als
4-Me, 4-CO2Me carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 4-Me, 4-CO2Me carb
DATIM Sun Oct 29 01:38:37 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 4096
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.3 c
SLVNT DMSO
EXREF 39.50 ppm
BF 1.20 Hz
RGAIN 23
21.042
38.874
39.080
39.294
39.706
39.920
40.126
51.668
110.663
111.050
119.538
120.345
122.008
122.173
122.486
126.437
127.656
128.290
138.523
142.730
166.926
CO2Me
NH
10
PPM
25
50
75
100
125
150
175
200


\\Nmr400mhz\seizo\watanabe\Tandem\p-CO2Me, m-tBu carb NON_E4_FT.als
A-49 D fr2
DFILE \\Nmr400mhz\seizo\wata
COMNT A-49 D fr2
DATIM Tue Sep 26 21:23:24 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 8
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 24
PPM
1.396
1.425
1.454
1.543
9.00
3.969
2.83
7.258
7.357
7.361
7.378
7.382
7.398
7.419
7.462
8.027
8.048
8.088
8.092
8.109
8.113
8.193
8.771
0.90
1.86
0.82
CO2Me
t-Bu
11


\\Nmr400mhz\seizo\watanabe\0025 B-2\m-tBu p-CO2Me 2BCM_E15_FT.als
m-tBu p-CO2Me
DFILE \\Nmr400mhz\seizo\watanab
COMNT m-tBu p-CO2Me
DATIM Fri Mar 16 06:09:33 2007
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 4096
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 15.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 25
0.000
31.704
35.195
51.957
76.696
77.017
77.214
77.330
107.420
109.972
118.469
120.123
120.872
121.259
122.634
123.128
126.998
140.178
142.459
150.469
167.947
CO2Me
t-Bu
H
N
11
PPM
0
25
50
75
100
125
150
175
200


DFILE \\Nmr400mhz\seizo\wata
COMNT A-34 C fr2
DATIM Tue Aug 29 04:54:20 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 16
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
\\Nmr400mhz\seizo\watanabe\Tandem\p-tBu, p-CO2Me NON_E28_FT.als
A-34 C fr2
PPM
t-Bu
CO2Me
12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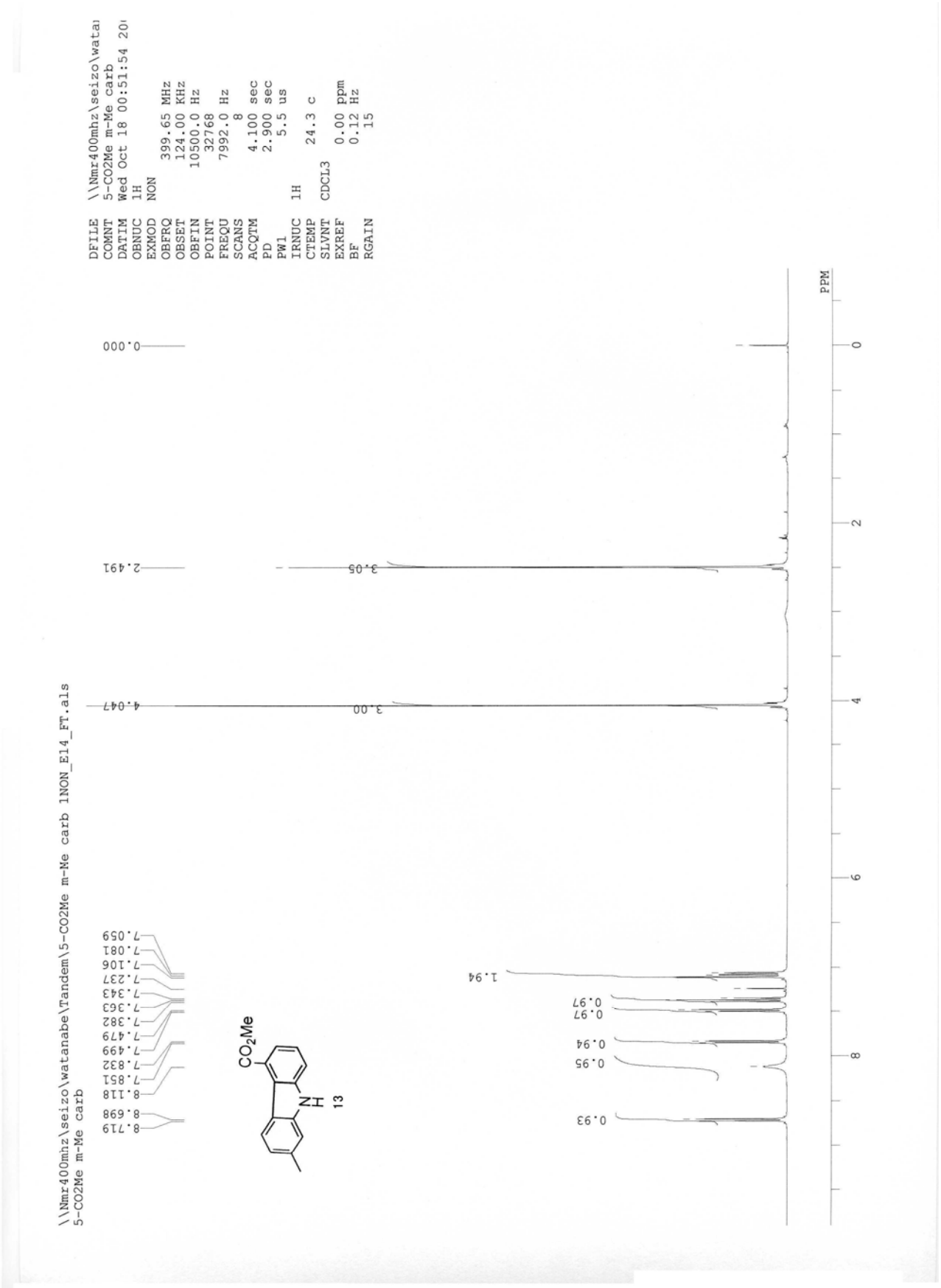
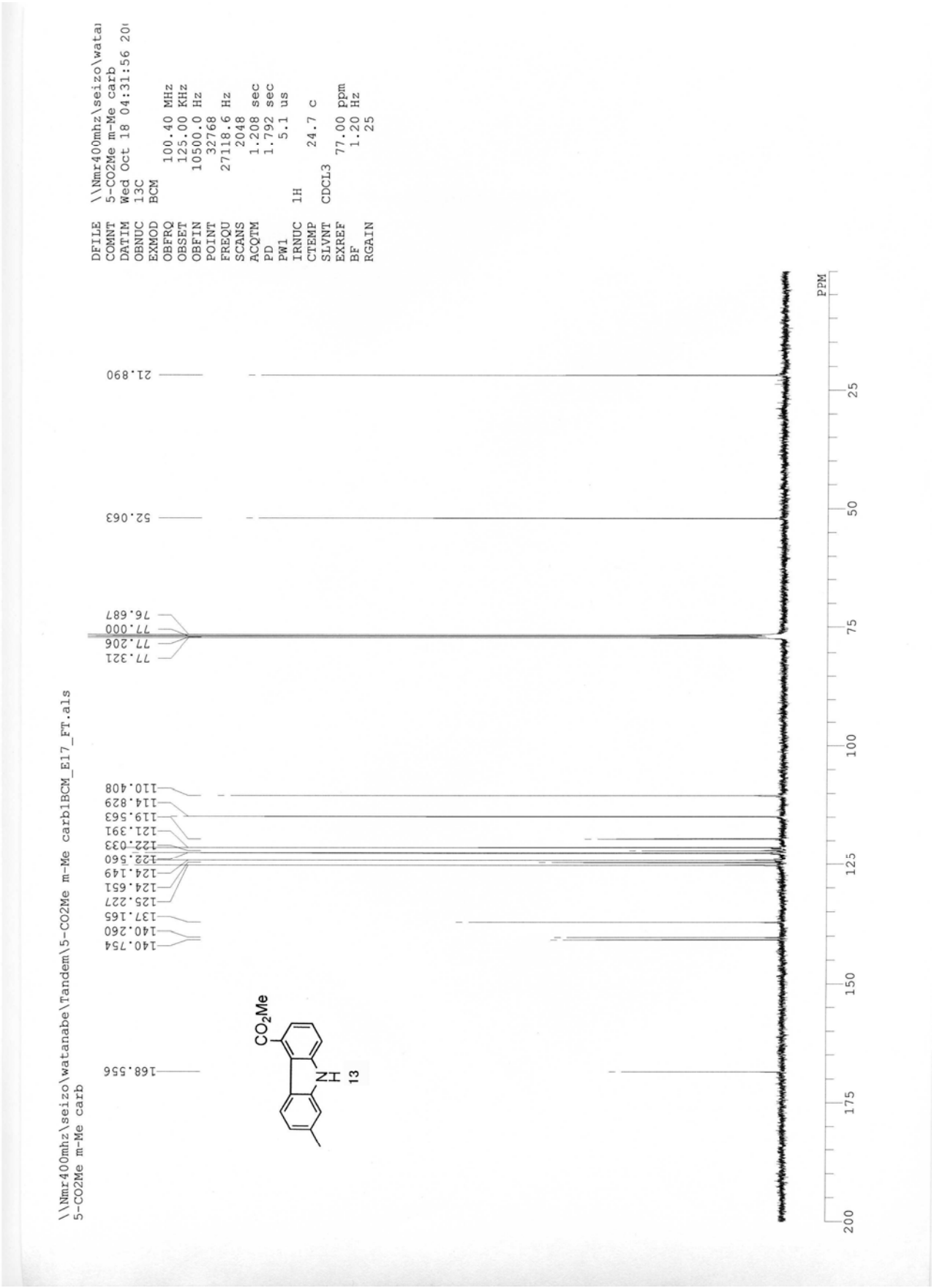
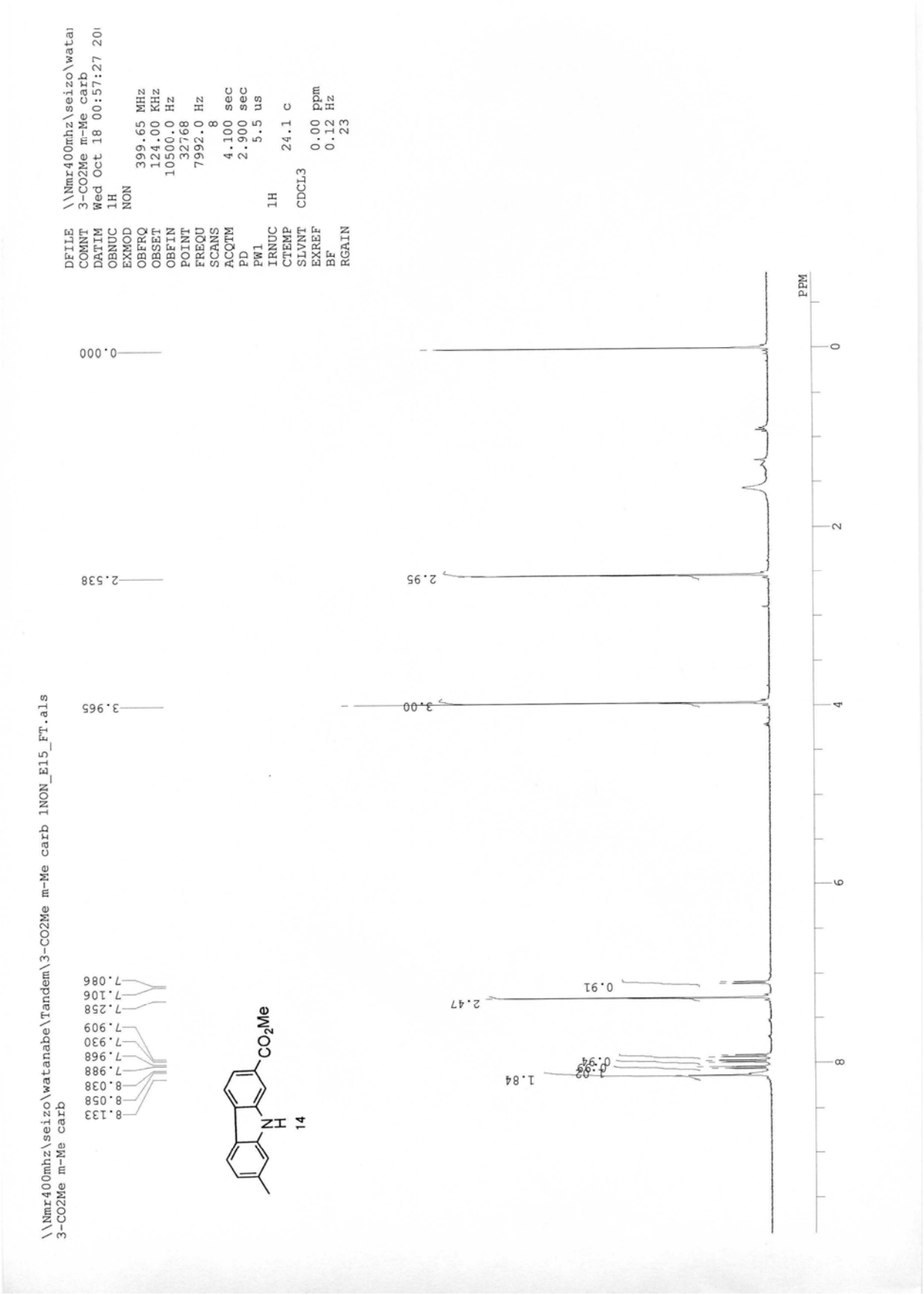
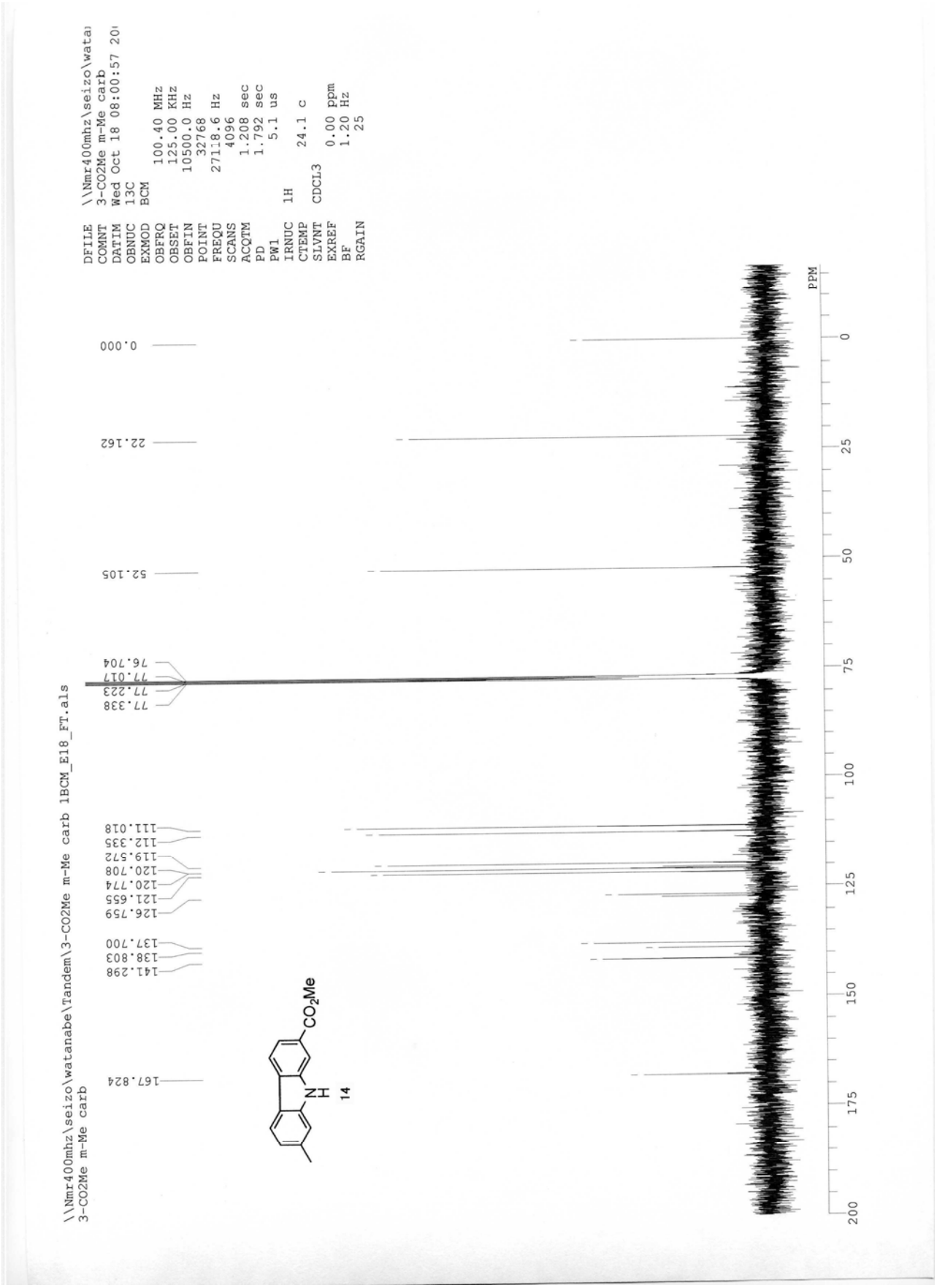
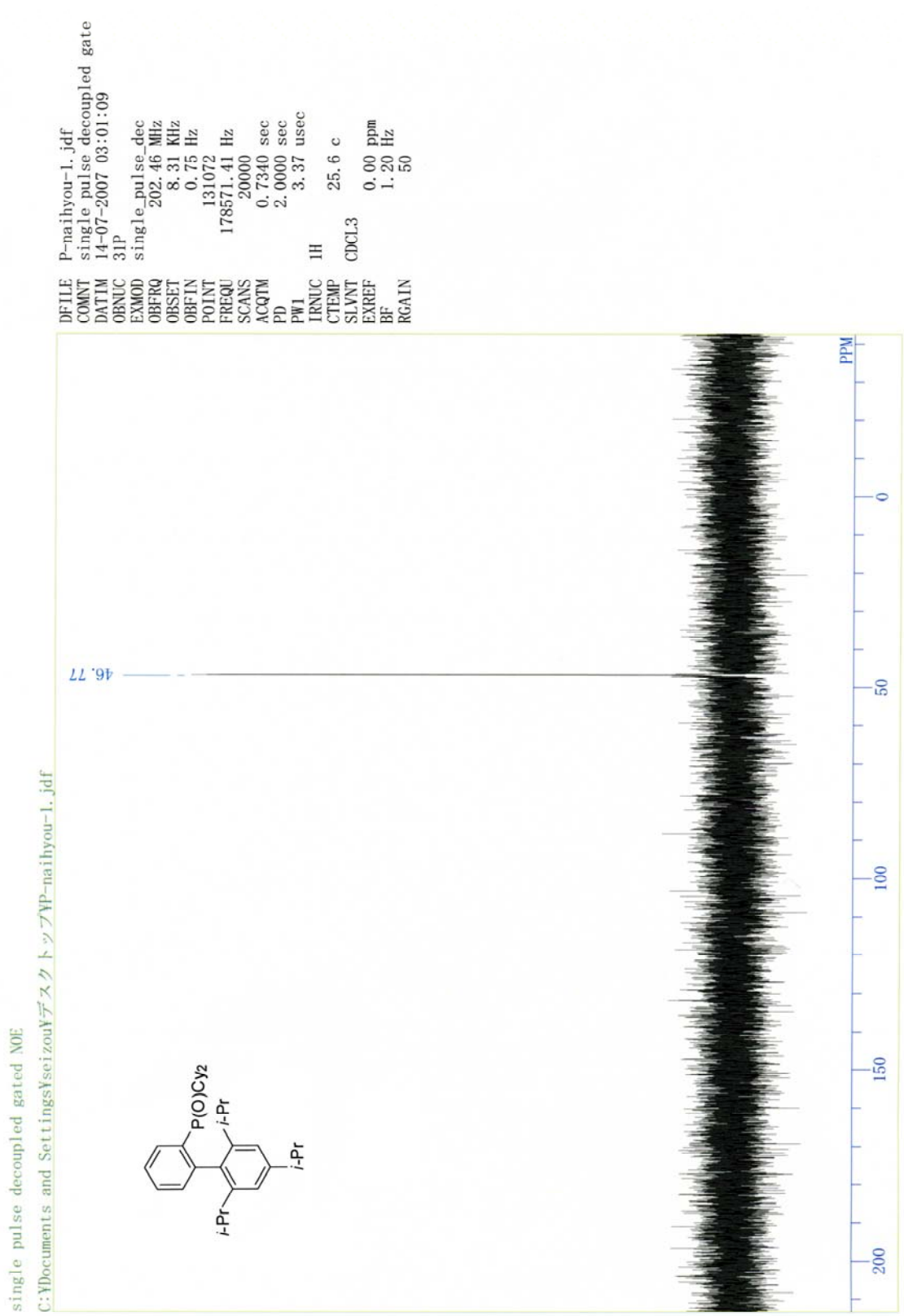
A-34 C fr2
\\Nmr400mhz\seizo\wata
A-34 C fr2
Tue Aug 29 05:45:58 20
13C
BCM
100.40 MHz
125.00 KHz
10500.0 Hz
32768
27118.6 Hz
1024
1.208 sec
1.792 sec
5.1 us
1H
24.4 c
CDCL3
77.00 ppm
1.20 Hz
25
31.918
34.758
51.915
76.679
77.000
77.313
110.079
110.359
116.756
121.193
122.733
123.103
123.416
124.577
127.228
137.988
142.672
143.545
167.930
CO2Me
t-Bu
NH
12
PPM
25
50
75
100
125
150
175
200


DFILE \\Nmr400mhz\seizo\wata
COMNT 5-CO2Me m-Me carb
DATIM Wed Oct 18 00:51:54 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 8
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15
0.000
2.491
4.047
3.05
3.00
1.94
0.97
0.97
0.94
0.95
0.93
7.059
7.081
7.106
7.237
7.343
7.363
7.382
7.479
7.499
7.832
7.851
8.118
8.698
8.719
CO2Me
NH
13
PPM
0
2
4
6
8
\\Nmr400mhz\seizo\watanabe\Tandem\5-CO2Me m-Me carb 1NON_E14_FT.als
5-CO2Me m-Me carb


\\Nmr400mhz\seizo\watanabe\Tandem\5-CO2Me m-Me carb1BCM_E17_FT.als
5-CO2Me m-Me carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 5-CO2Me m-Me carb
DATIM Wed Oct 18 04:31:56 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 2048
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
21.890
52.063
76.687
77.000
77.206
77.321
110.408
114.829
119.563
121.391
122.033
122.560
124.149
124.651
125.227
137.165
140.260
140.754
168.556
CO2Me
NH
13
PPM
200
175
150
125
100
75
50
25


\\Nmr400mhz\seizo\watanabe\Tandem\3-CO2Me m-Me carb 1NON_E15_FT.als
3-CO2Me m-Me carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 3-CO2Me m-Me carb
DATIM Wed Oct 18 00:57:27 20
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 7992.0 Hz
SCANS 8
ACQTM 4.100 sec
PD 2.900 sec
PW1 5.5 us
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23
0.000
2.538
3.965
7.086
7.106
7.258
7.909
7.930
7.968
7.988
8.038
8.058
8.133
CO2Me
N
H
14
0.91
2.47
1.84
2.95
3.00
PPM
0
2
4
6
8


\\Nmr400mhz\seizo\watanabe\Tandem\3-CO2Me m-Me carb 1BCM_E18_FT.als
3-CO2Me m-Me carb
DFILE \\Nmr400mhz\seizo\wata
COMNT 3-CO2Me m-Me carb
DATIM Wed Oct 18 08:00:57 20
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 27118.6 Hz
SCANS 4096
ACQTM 1.208 sec
PD 1.792 sec
PW1 5.1 us
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 25
0.000
22.162
52.105
76.704
77.017
77.223
77.338
111.018
112.335
119.572
120.708
120.774
121.655
126.759
137.700
138.803
141.298
167.824
CO2Me
NH
14
PPM
0
25
50
75
100
125
150
175
200


single pulse decoupled gated NOE
C:¥Documents and Settings¥seizou¥デスクトップ¥P-naihyou-1.jdf
46.77
P(O)Cy2
i-Pr
i-Pr
i-Pr
PPM
200
150
100
50
0
DFILE P-naihyou-1.jdf
COMNT single pulse decoupled gate
DATIM 14-07-2007 03:01:09
OBNUC 31P
EXMOD single_pulse_dec
OBFRQ 202.46 MHz
OBSET 8.31 KHz
OBFIN 0.75 Hz
POINT 131072
FREQU 178571.41 Hz
SCANS 20000
ACQTM 0.7340 sec
PD 2.0000 sec
PW1 3.37 usec
IRNUC 1H
CTEMP 25.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 50