SUPPORTING INFORMATION

Fluoride Catalytic Effect in Silver Oxide-mediated Cross-coupling of Functional Trimethysilylpyridines: Access to Arylpyridines and Bihetaryl Compounds.

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General Methods. All solvents were distilled before use. 4-chloro-2-trimethysilylpyridine **1**[1] and 3-chloro-2-trimethysilylpyridine **2,**[1] 2-chloro-2-trimethysilylpyridine **2**[2] and 4-methyl-2-trimethysilylpyridine **2**[3] were prepared by lithiation-silylation of 4-chloropyridine and 4-methylpyridine respectively using the BuLi-LiDMAE reagent. Ag_2O was dried overnight under vacuum before use. All other reagents and catalysts were commercially available and used as such. ^{1}H and ^{13}C NMR spectra were performed on Brucker spectrometers at 200 or 400 MHz (^{1}H) and 50 or 100 MHz (^{13}C) in $CDCl_3$ using TMS as reference. GC experiments were performed on a Shimadzu chromatograph fitted with a 15m capillary column. GC-MS spectra with electronic impact were performed on a Shimadzu QP 2010 apparatus. High resolution mass spectra were performed on a Brucker microTOF-Q. Column Chromatography was performed on silica gel (70–230 mesh).

General procedure for cross-coupling reactions

To a suspension of the aryl halide (0.5 mmol) and Ag_2O (1 mmol) in degassed DMF (5 mL) under argon were added the trimethylsilylpyridine (1 mmol), $PdCl_2(PPh_3)_2$ (0.025 mmol) and TBAF (0.05 mmol, 0.05 mL of a 1M solution in THF). The resulting suspension was stirred at 90°C for 3h. After cooling, the reaction medium was diluted with EtOAc (5 mL), filtered, and concentrated. Column chromatography using Cyclohexane-AcOEt as eluent afforded products.

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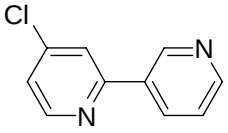
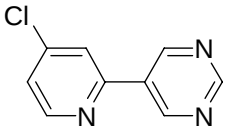
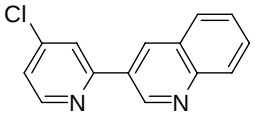
[1] Choppin, S. Gros, Ph. Fort, *Eur. J. Org. Chem.,* **2001**, 603-606.
[2] Choppin, S. Gros, Ph. Fort, Org. Lett., **2000**, 803-805.
[3] Kaminiski, T. ; Gros P. ; Fort, Y., *Eur. J. Org. Chem.,* **2003**, 3855-3860.



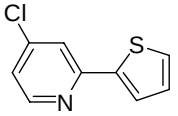
1a,[4] Yield, 70%. ^{1}H NMR (400MHz, $CDCl_3$) : δ = 7.29 (dd, J = 5.3, 1.9 Hz, 1H), 7.40 (ddd, J = 8.8, 5.0, 0.9 Hz, 1H), 7.74 (dd, J = 1.9, 0.6 Hz, 1H), 8.28 (dt, J = 7.9, 1.9 Hz, 1H), 8.59 (s, 1H), 8.61 (d, J = 0.6 Hz, 1H), 8.67 (dd, J = 5.0, 1.9 Hz, 1H), 9,13 (d, J = 5.0, 1.9 Hz, 1H) . ^{13}C NMR (100 MHz, $CDCl_3$): δ = 120.8, 123.0, 123.6, 133.6, 134.3, 145.0, 148.2, 150.5, 150.8, 156.3. FTIR (KBr): 3043, 2923, 1724, 1572, 1542, 1413, 1102, 1023, 804. MS (EI) ; m/z (%): 190[M+] (100), 164 (22).



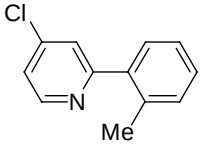
1b, Yield, 85%. 1H NMR (400MHz, CDCl3) : δ = 7.36 (dd, J = 5.3, 1.9 Hz, 1H), 7.76 (d, J = 1.9 Hz, 1H), 8.64 (d, J = 5.4 Hz, 1H), 8.29 (d, J = 8.2 Hz, 1H), 9.30 (s, 2H). 13C NMR (100 MHz, CDCl3): δ = 120.8, 123.8, 131.3, 145.3, 151.2, 153.5, 155.1, 159.1. FTIR (KBr): 3045, 2923, 1564, 1376, 1185, 1056, 821. MS (EI) ; m/z (%): 191 [M+] (85), 113 (100). HRMS(ESI (M+H+)): calcd. for C9H6ClN3 192.0323, found 192.0330.



1c, Yield, 62%. ^{1}H NMR (400MHz, $CDCl_3$) : δ = 7.30 (dd, J = 5.3, 1.9 Hz, 1H), 7.57 (td, J = 7.6, 1.2 Hz, 1H), 7.74 (td, J = 8.2, 1.6 Hz, 1H), 7.86 (dd, J = 1.9, 0.6 Hz, 1H), 7.90 (dd, J = 8.2, 1.6 Hz, 1H), 8.14 (d, J = 8.5 Hz, 1H), 8.64 (d, J = 5.7 Hz, 1H), 8.71 (d, J = 2.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 121.1, 123.0, 127.3, 127.7, 128.6, 129.4, 130.4, 130.7, 134.2, 145.1, 148.5, 149.0, 151.0, 156.4. FTIR (KBr): 3039, 1573, 1329, 1121, 966, 753. MS (EI) ; m/z (%): 240 [M+] (100), 205 (20). HRMS(ESI ($M+H^+$)): calcd. for $C_{14}H_9ClN_2$ 241.0527 ; found 241.0530.



1e,[5] Yield, 52%. ^{1}H NMR (400MHz, $CDCl_3$) : δ = 7.10-7.16 (m, 2H), 7.43 (dd, J = 5.0, 0.9 Hz, 1H), 7.59 (dd, J = 3.5, 1.0 Hz, 1H), 7.65 (dd, J = 1.9, 0.6 Hz, 1H), 8.45 (dd, J = 5.3, 0.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 118.5, 118.9, 122.0, 124.4, 125.0, 125.3, 128.1, 128.4, 150.3. FTIR (KBr): 3073, 2924, 1568, 1538, 1383, 1089, 819, 699. MS (EI) ; m/z (%): 195 [M+] (100), 160 (25).

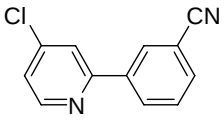


1f, Yield, 25%. ^{1}H NMR (250 MHz, CD_3OD) δppm: 2.30 (s, 3 H), 7.27 - 7.37 (m, 4 H), 7.48 (dd, *J*=5.48, 1.98 Hz, 1H), 7.57 (d, *J*=1.98 Hz, 1 H), 8.55 (d, *J*=5.48 Hz, 1 H). ^{13}C NMR (62.5 MHz, CD_3OD) δppm: 23.3, 122.0, 124.4, 126.0, 129.0, 129.5, 130.9, 135.8,

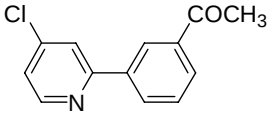
[4] Cruskie, M.P., Zoltewicz, J. A. ; Abboud, K.A. *J. Org.Chem.* **1995**, 60, 7491-7495.

[5] Pierrat, P.; Gros, P.; Fort, Y.. Org. Lett. **2005**, *7*, 697-700.

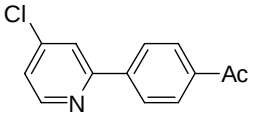
139.1, 144.1, 150.0, 161.45 ppm. FTIR (KBr): 3050, 1567, 1540, 1149, 1353, 1114, 1087, 824, 723. LCMS (ESI): 206 (60), 204 ($M+H^+$) (100), 157 (15). HRMS (ESI ($M+H^+$)): calcd. for $C_{12}H_{10}ClN$ 204.0502, found 204.0497.



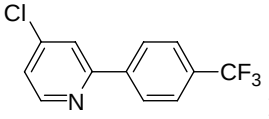
1g, Yield, 80%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: 7.34 (dd, *J*=5.33, 1.98 Hz, 1 H), 7.56 - 7.66 (m, 1 H), 7.68 - 7.78 (m, 2 H), 8.22 (, dt, *J*=7.92, 1.60 Hz, 1 H), 8.33 (t, *J*=1.45 Hz, 1 H), 8.57 - 8.68 (m, 1 H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 113.2, 118.5, 121.0, 123.4, 129.7, 130.8, 131.0, 132.9, 139.2, 145.3, 150.8, 156.4 ppm. FTIR (KBr): 3050, 2227, 1573, 1549, 1458, 1367, 1058, 910, 873. LCMS (ESI): 217 (40), 215 ($M+H^+$) (100). HRMS(ESI ($M+H^+$)): calcd. for $C_{12}H_7ClN_2$ 215.0376, found 215.0371.



1h, Yield, 59%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: 2.70 (s, 3 H), 7.31 (dd, *J*=5.33, 1.98 Hz, 1 H), 7.61 (t, *J*=7.77 Hz, 1 H), 7.82 (d, *J*=1.37 Hz, 1 H), 8.05 (dt, *J*=7.77, 1.45 Hz, 1 H), 8.21 (ddd, *J*=7.80, 1.87, 1.14 Hz, 1 H), 8.57 (t, *J*=1.52 Hz, 1 H), 8.62 (d, *J*=5.33 Hz, 1 H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 26.8, 121.1, 122.9, 126.8, 129.2, 131.5, 137.7, 138.5, 145.1, 150.5, 157.8, 197.9. FTIR (KBr): 3100, 1674, 1571, 1554, 1458, 1360, 1300, 1245, 802. LCMS (ESI): 234 (60), 232 ($M+H^+$) (100). HRMS(ESI ($M+H^+$)): calcd. for $C_{13}H_{10}ClNO$ 232.0529, found 232.0530.

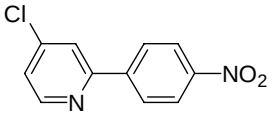


1i,[3] Yield, 60%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: 2.67 (s, 3 H), 7.32 (dd, *J*=5.33, 1.83 Hz, 1 H), 7.80 (d, *J*=1.37 Hz, 1 H), 8.09 (brs, 4 H,), 8.63 (d, *J*=5.33Hz, 1 H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 26.8, 121.4, 123.1, 127.2, 128.9, 137.6, 142.2, 145.0, 150.7, 157.6, 197.7. FTIR (KBr): 3060, 1672, 1567, 1548, 1355, 1266, 967, 822. LCMS (ESI): 234 (60), 232 ($M+H^+$) (100), 157 (14).

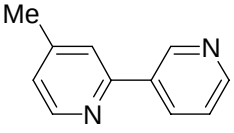


1j,[6] Yield, 27%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: 7.29 - 7.36 (m, 1 H), 7.67 - 7.84 (m, 3 H), 8.11 (d, *J*=8.07 Hz, 2 H), 8.63 (dd, *J*=5.25, 0.53 Hz, 1 H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 121.2, 123.1, 125.8, 127.3, 131.1, 131.7, 141.4, 145.1, 150.7, 157.3 ppm. NMR (62.5 MHz, $CDCl_3$) δ_C = 121.2, 123.1, 124.5, 124.7 (d, $^1J_{C\text{-}F}$, 272 Hz), 125.8, 126.5 (d, $^3J_{C\text{-}F}$, 5 Hz), 127.3, 131.5 (q, $^2J_{C\text{-}F}$, 33 Hz), 141.4, 145.1, 150.7, 157.3 ppm. NMR (235 MHz, $CDCl_3$) δ_F = -62.65. FTIR (KBr): 2985, 1674, 1549, 1329, 1167, 1104, 1245, 849, 827. LCMS (ESI): 260 (50), 258 ($M+H^+$) (100).HRMS(ESI ($M+H^+$)): calcd. for $C_{11}H_7ClF_3N$ 258.0219, found 258.0215.

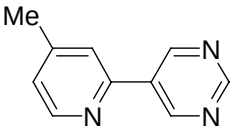
[6] Chen, N.; Nixey, T.; Norman, M.H. PCT Int Appl. (2005), WO 2005033105 A2 20050414.



1k, Yield, 86%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: 7.37 (dd, *J*=5.25, 1.90 Hz, 1 H), 7.78 - 7.87 (m, 1 H), 8.14 - 8.24 (m, 2 H), 8.30 - 8.43 (m, 2 H), 8.61 - 8.71 (m, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 121.6, 123.8, 124.1, 127.8, 143.9, 145.2, 148.5, 150.9, 156.3. LCMS (ESI): 237 (55), 235 ($M+H^+$) (100). HRMS(ESI ($M+H^+$)): calcd. for $C_{11}H_7ClN_2O_2$ 235.0274, found 235.0266.

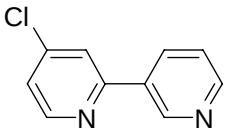


4a,[7] Yield, 43%. ^{1}H NMR (250 MHz, $CDCl_3$) δppm: δ = 9.18 (m, 1H), 8.65 (m, 1H), 8.58 (d, *J* = 5.0, Hz, 1H), 8.31 (ddd, *J* = 8.2, 4.1, 1.9 Hz, 1H), 7.57 (m, 1H), 7.40 (dd, J = 4.9, 3.7 Hz, 1H), 7.12 (d, J = 5.1 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δppm: 21.2, 121.6, 123.5, 123.8, 134.4, 148.1, 148.2, 149.3, 149.7, 150.0, 154.7. FTIR (KBr): 2918, 1602, 1545, 1379, 1207, 988, 829. MS (EI) ; m/z (%): 170 [M+] (100), 144 (29).



4b,[8] Yield, 46%. ^{1}H NMR (250 MHz, $CDCl_3$) δ ppm: 9.31 (s, 2H), 9.25 (s, 1H), 8.60 (d, J = 5.0, Hz, 1H), 7.57 (m, 1H), 7.17 (d, J = 5.0, Hz, 1H), 2.46 (s, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ ppm: 21.2, 121.5, 124.5, 132.5, 148.4, 150.1, 151.8, 155.0, 158.5. FTIR (KBr): 2924, 2854, 1725, 1605, 1464, 14136, 1190, 1023, 869, 807.MS (EI) ; m/z (%): 171 [M+] (97), 93 (100).

[7] Fleckenstein, C.A.; Plenio, H., *Chem. Eur. J.*, **2008**, *14*, 4267-4279.
[8] Ackermann, L.; Potukuchi, H. K. *Synlett* **2009**, *17*, 2852-2856.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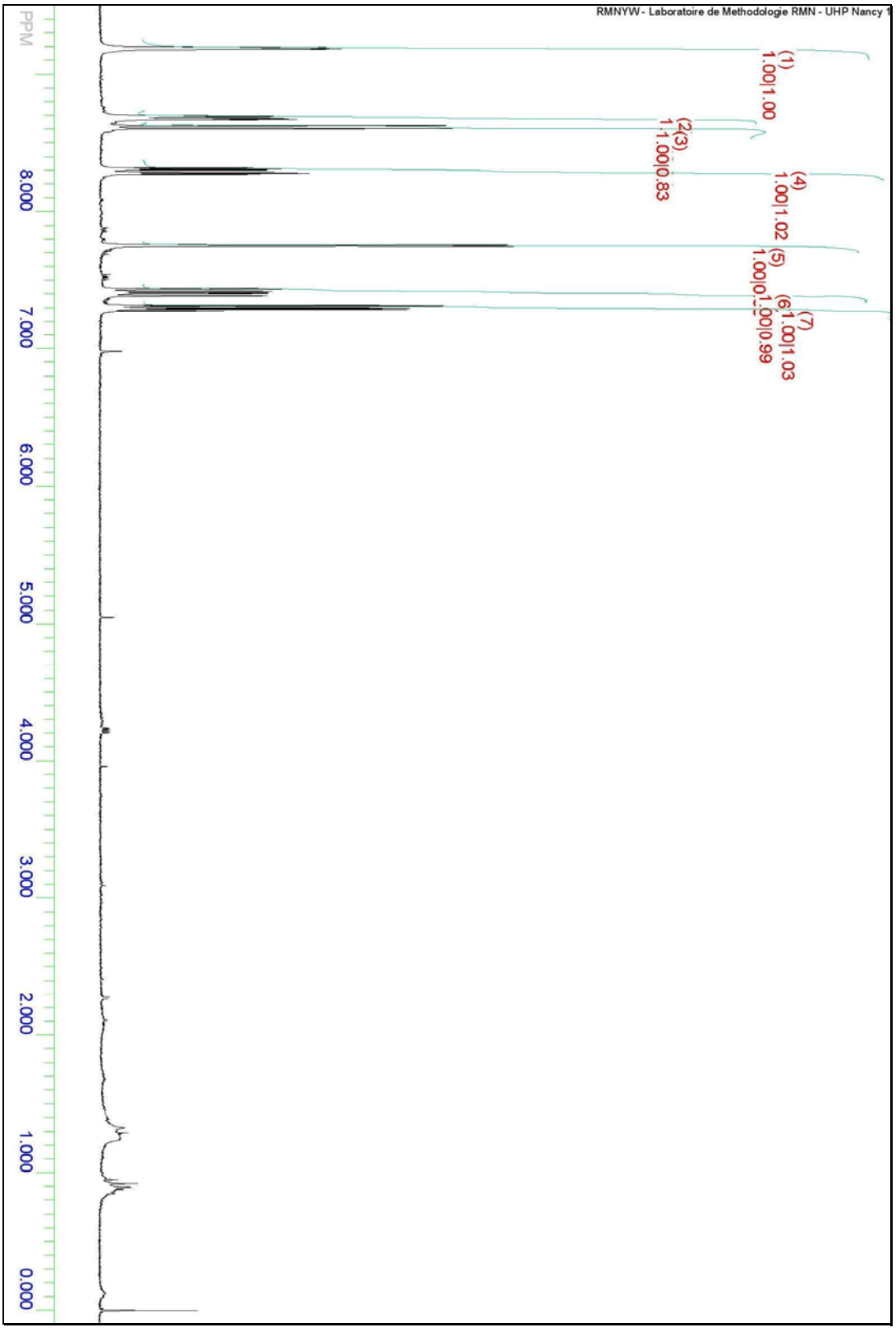
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5.000
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1.000
0.000
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(4) 1.00|1.02
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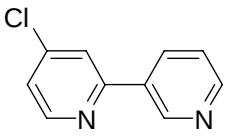
(7)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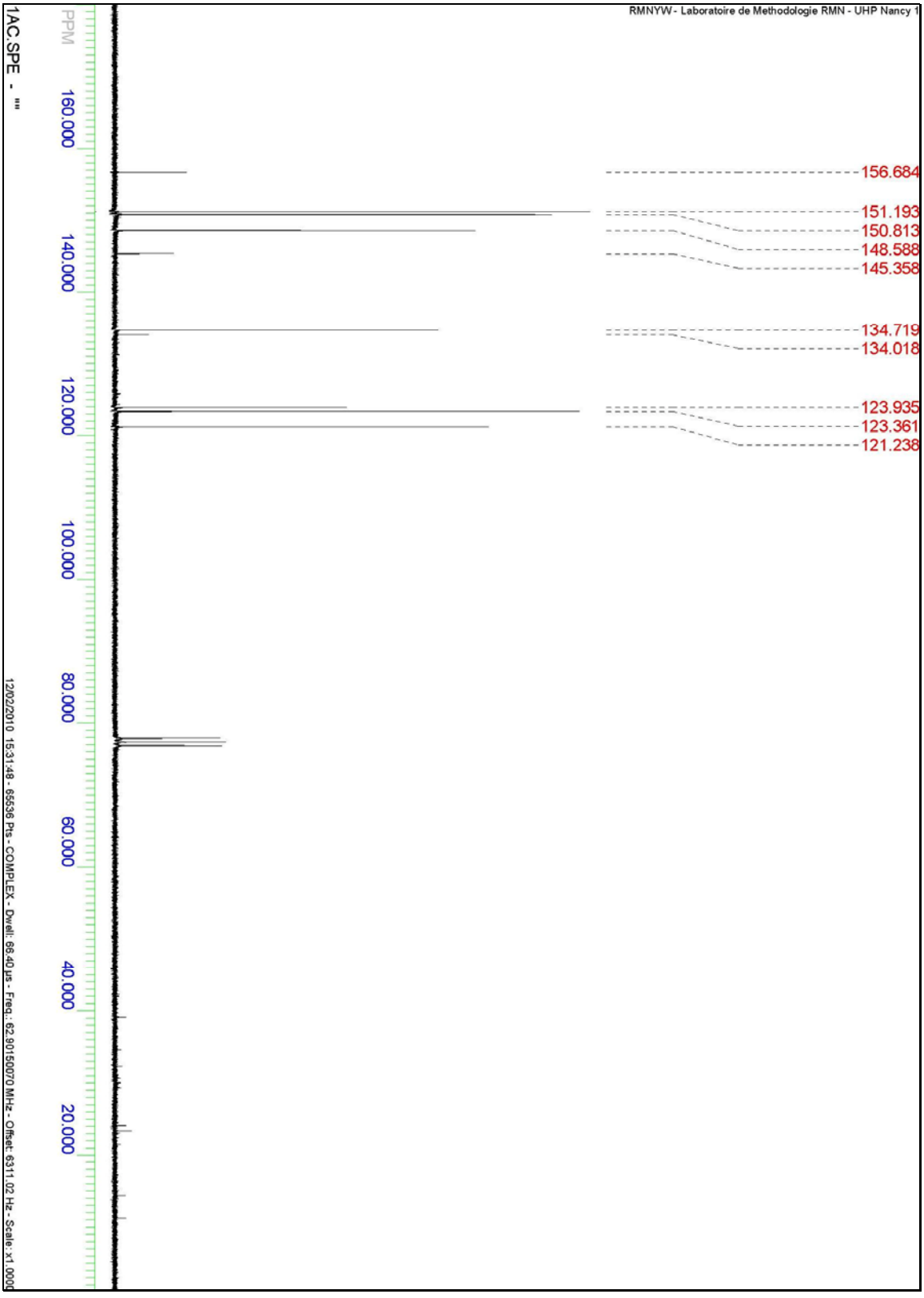
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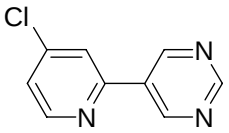
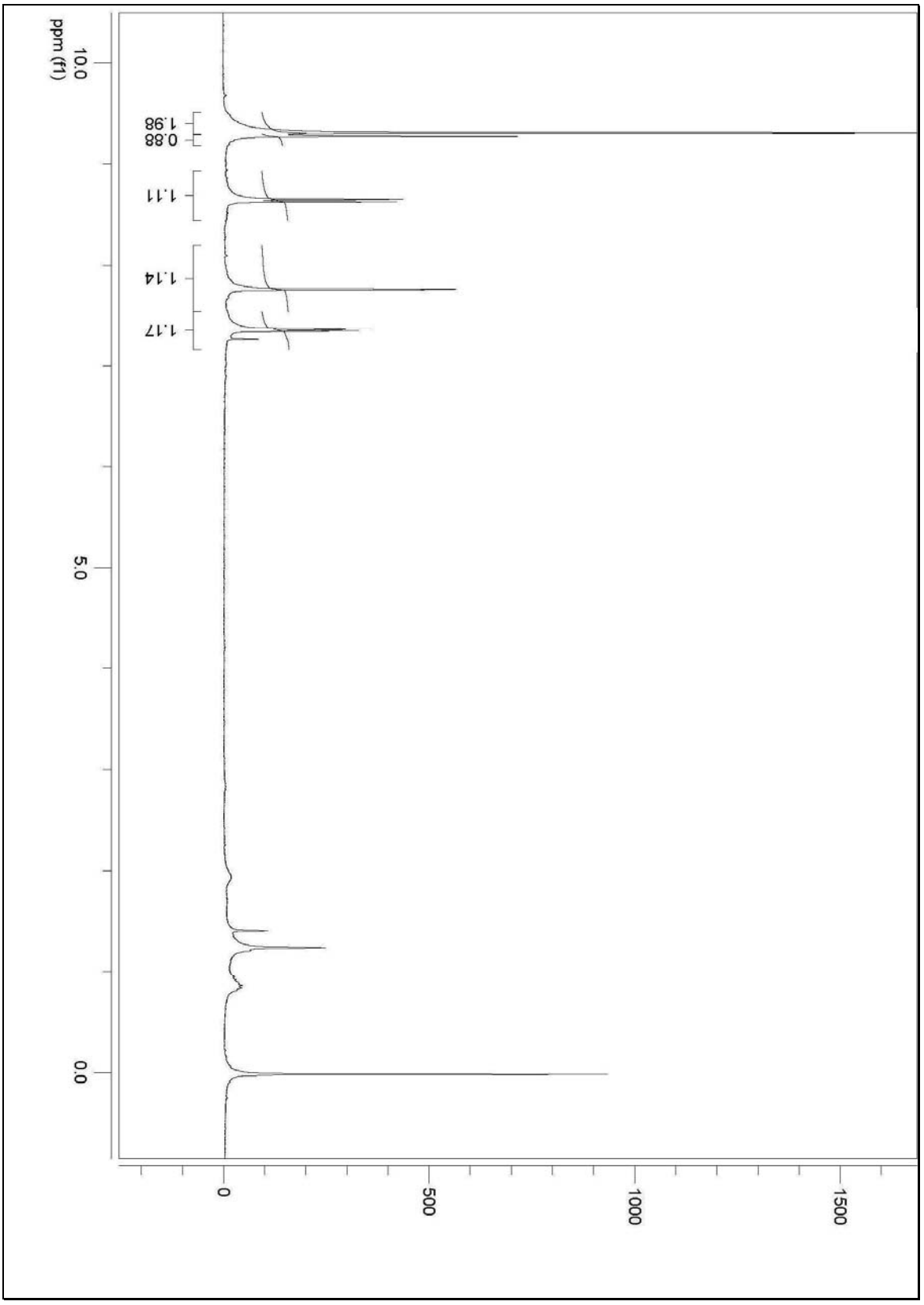
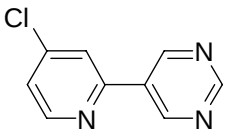


1b

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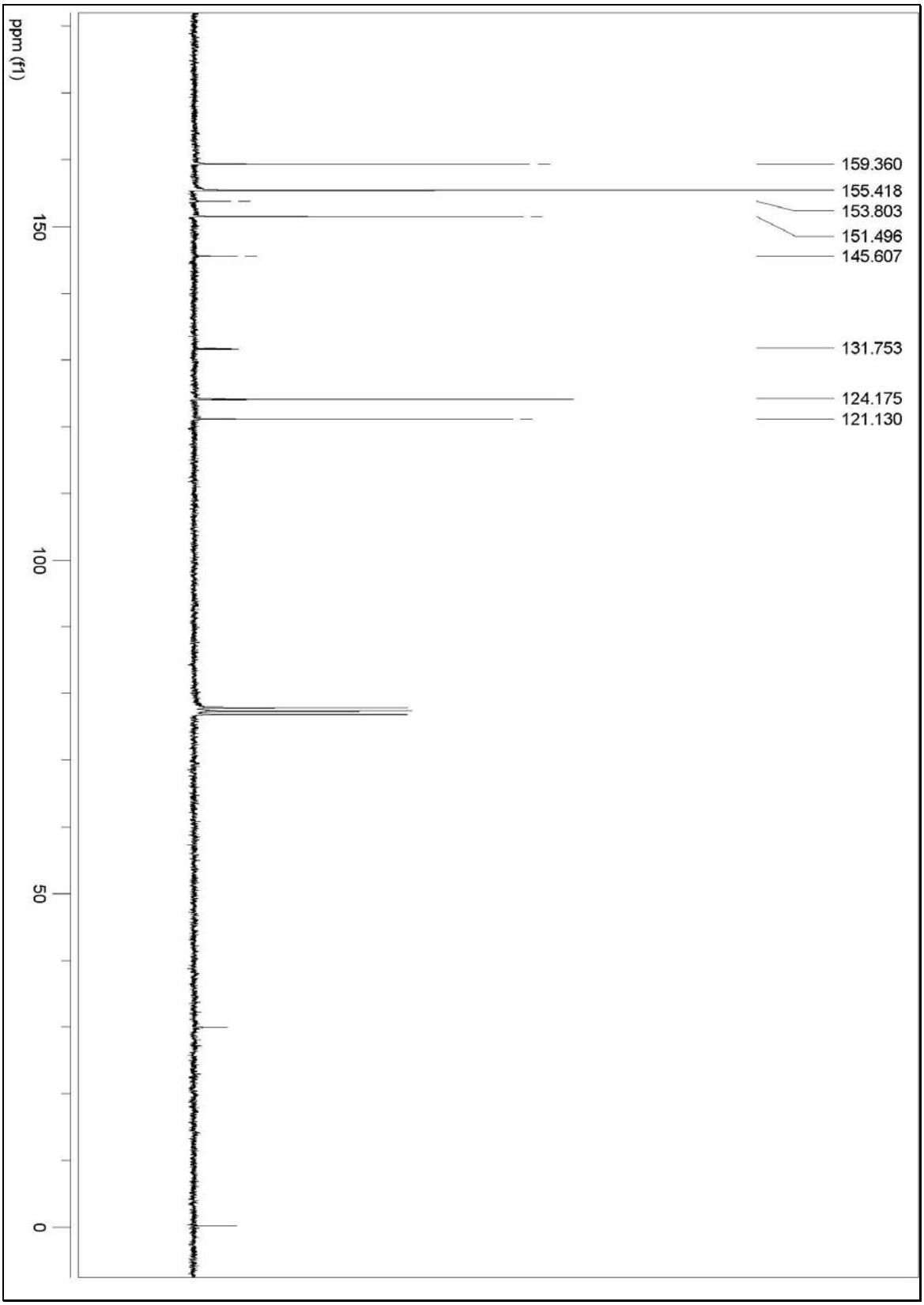
1b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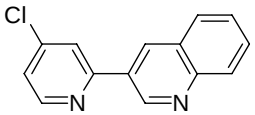

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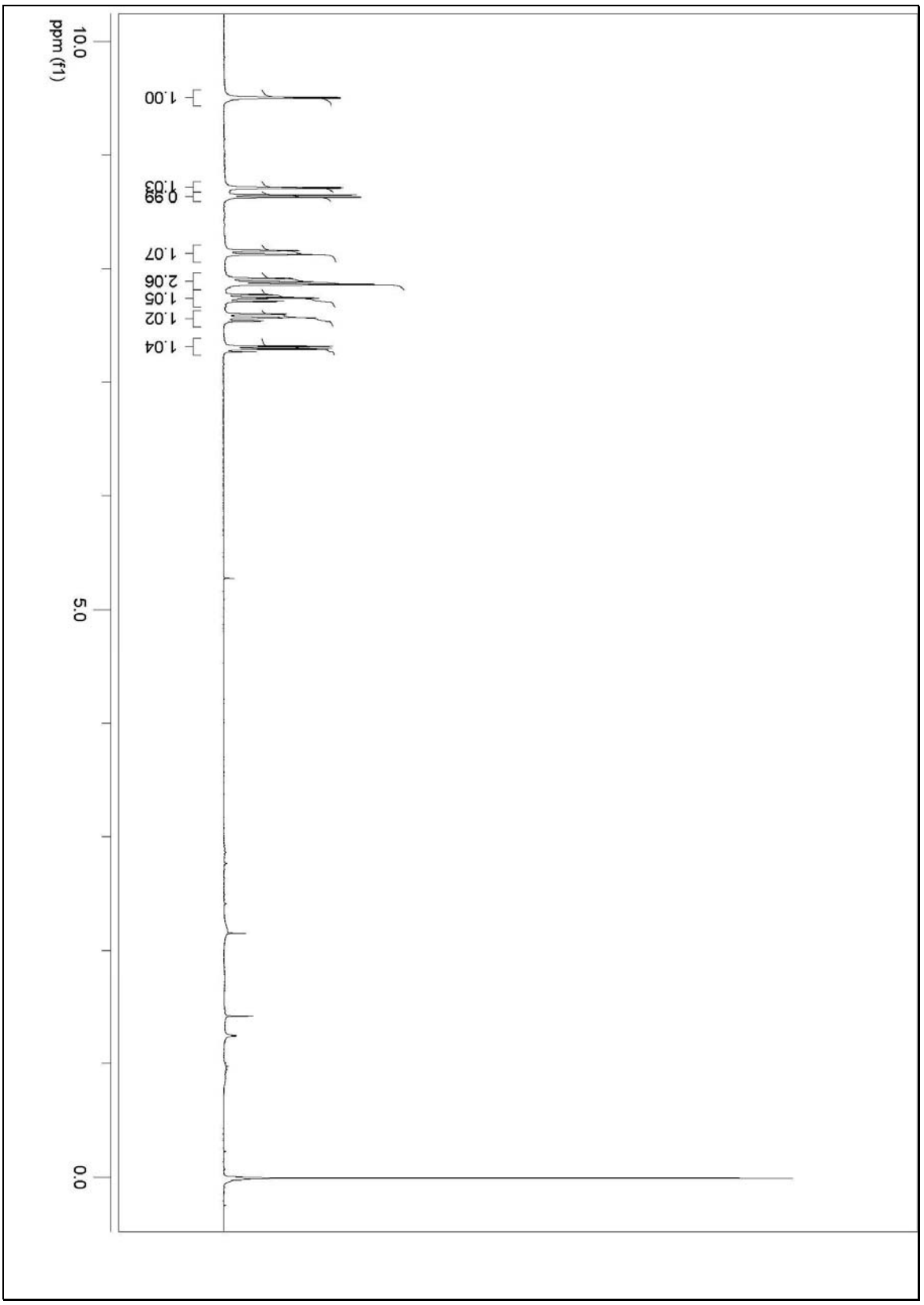

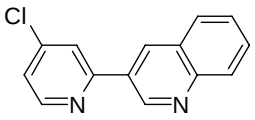
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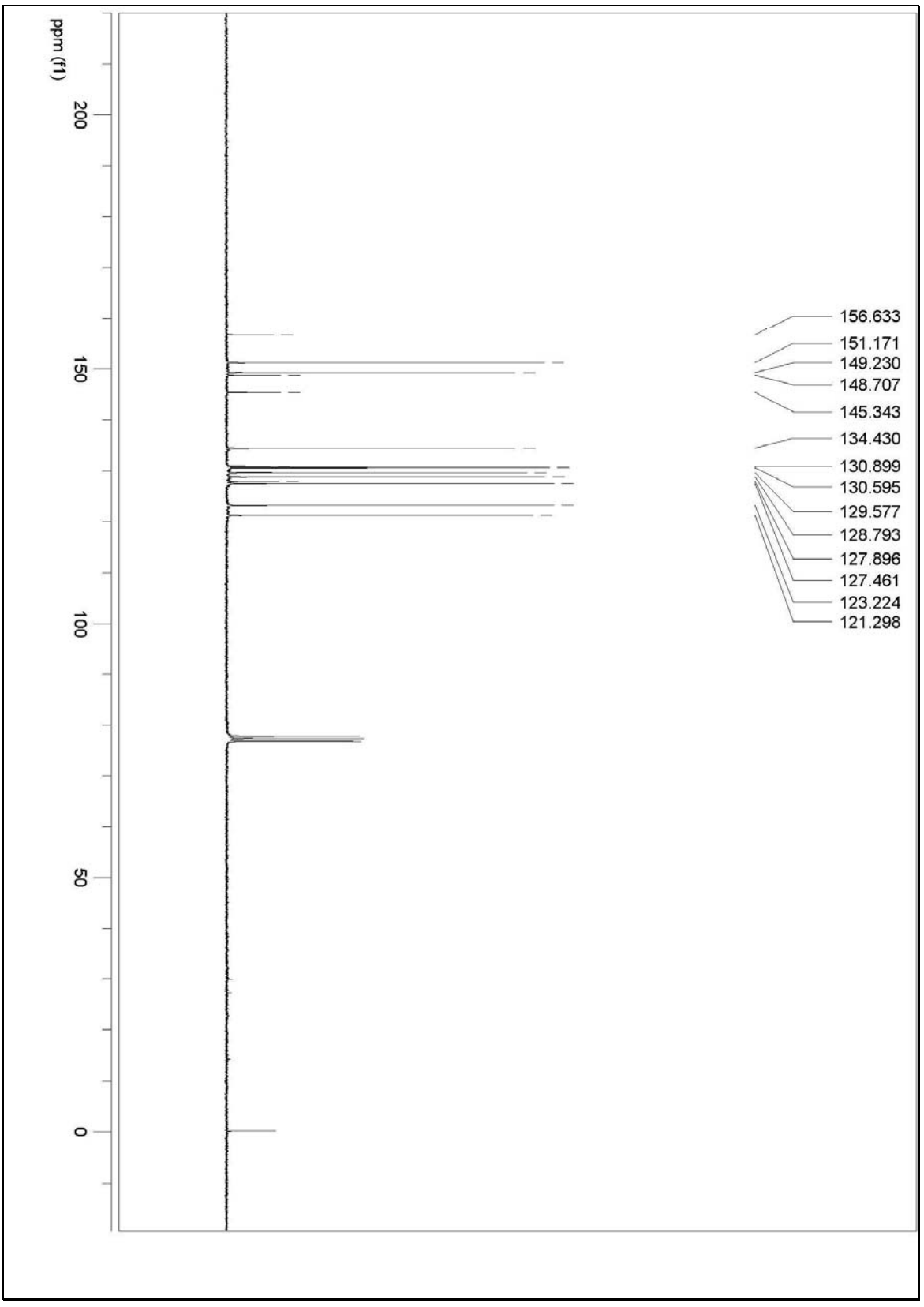
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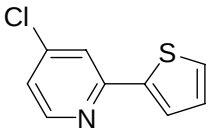
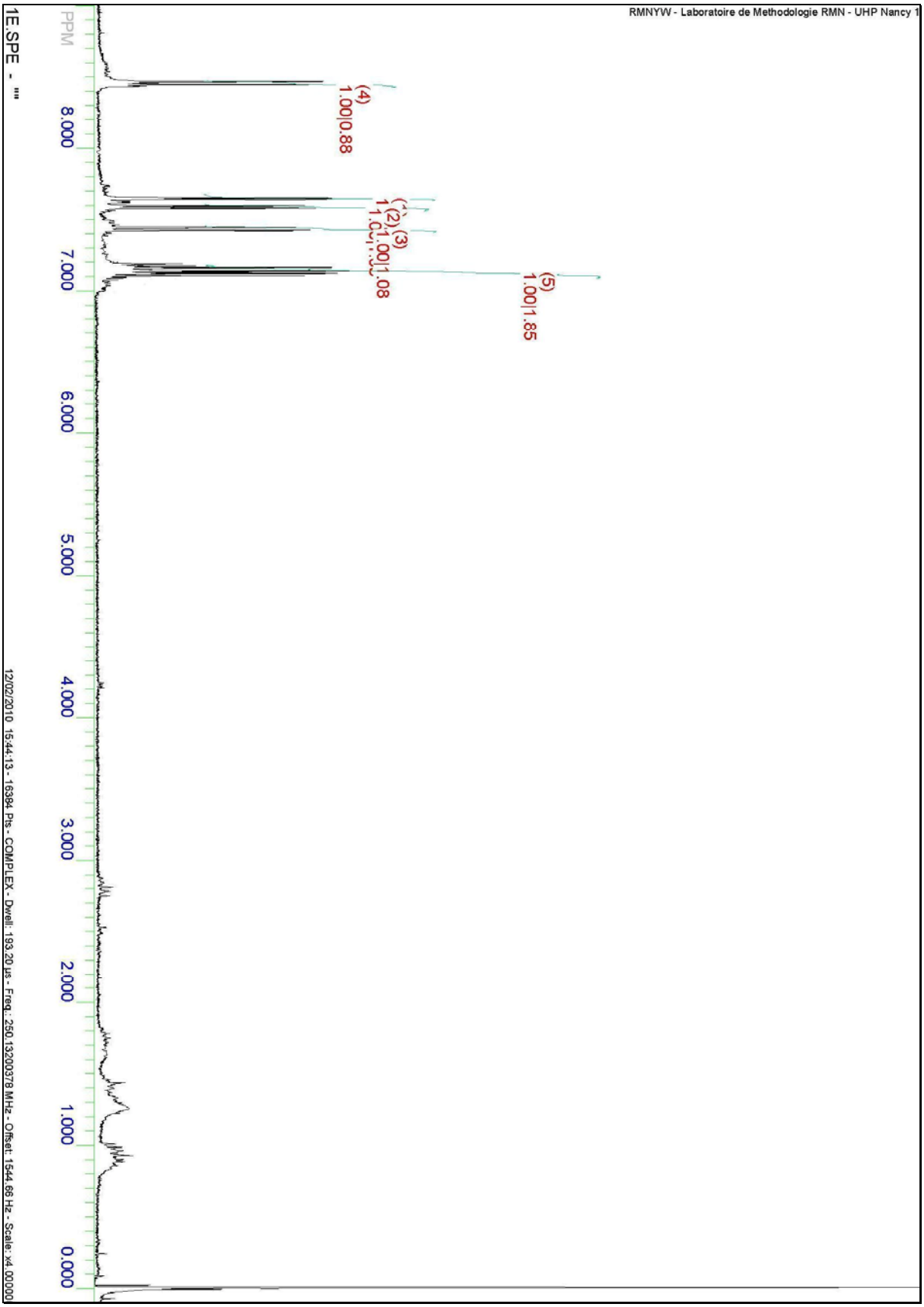

Cl
S
N

1e


RMNYW - Laboratoire de Methodologie RMN - UHP Nancy 1
1E.SPE - ""
PPM
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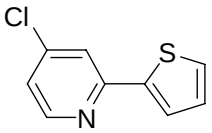
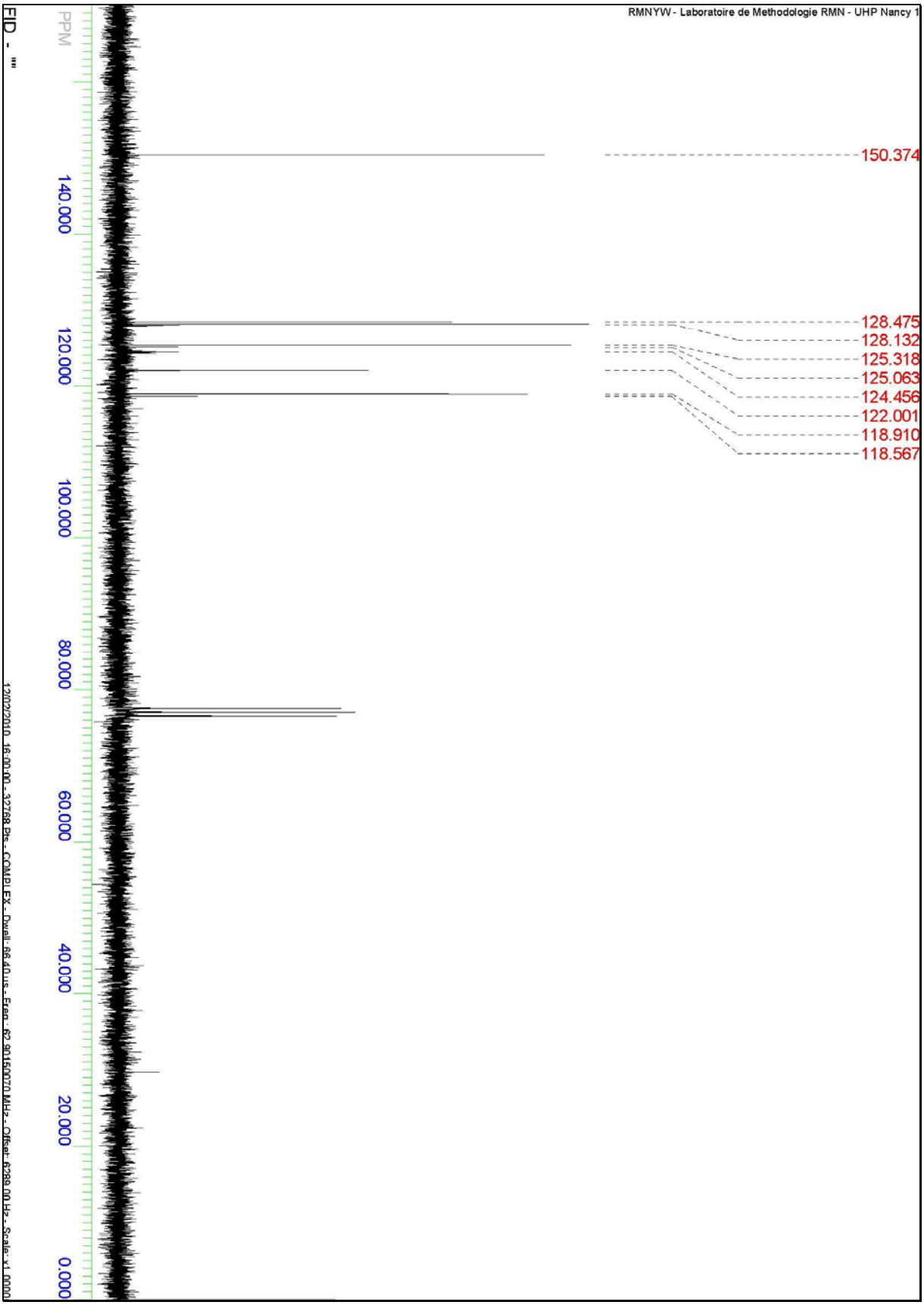
7.000
6.000
5.000
4.000
3.000
2.000
1.000
0.000
(4)
1.00|0.88
(2)(3)
1.00|1.08
(5)
1.00|1.85
12/02/2010 15:44:13 - 16384 Pts - COMPLEX - Dwell: 193.20 µs - Freq.: 250.1320378 MHz - Offset: 1544.66 Hz - Scale: x4.00000


Cl
S
N

1e


RMNYW - Laboratoire de Methodologie RMN - UHP Nancy 1
FID - ""
PPM
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128.475
128.132
125.318
125.063
124.456
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118.910
118.567
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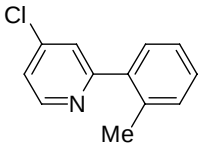
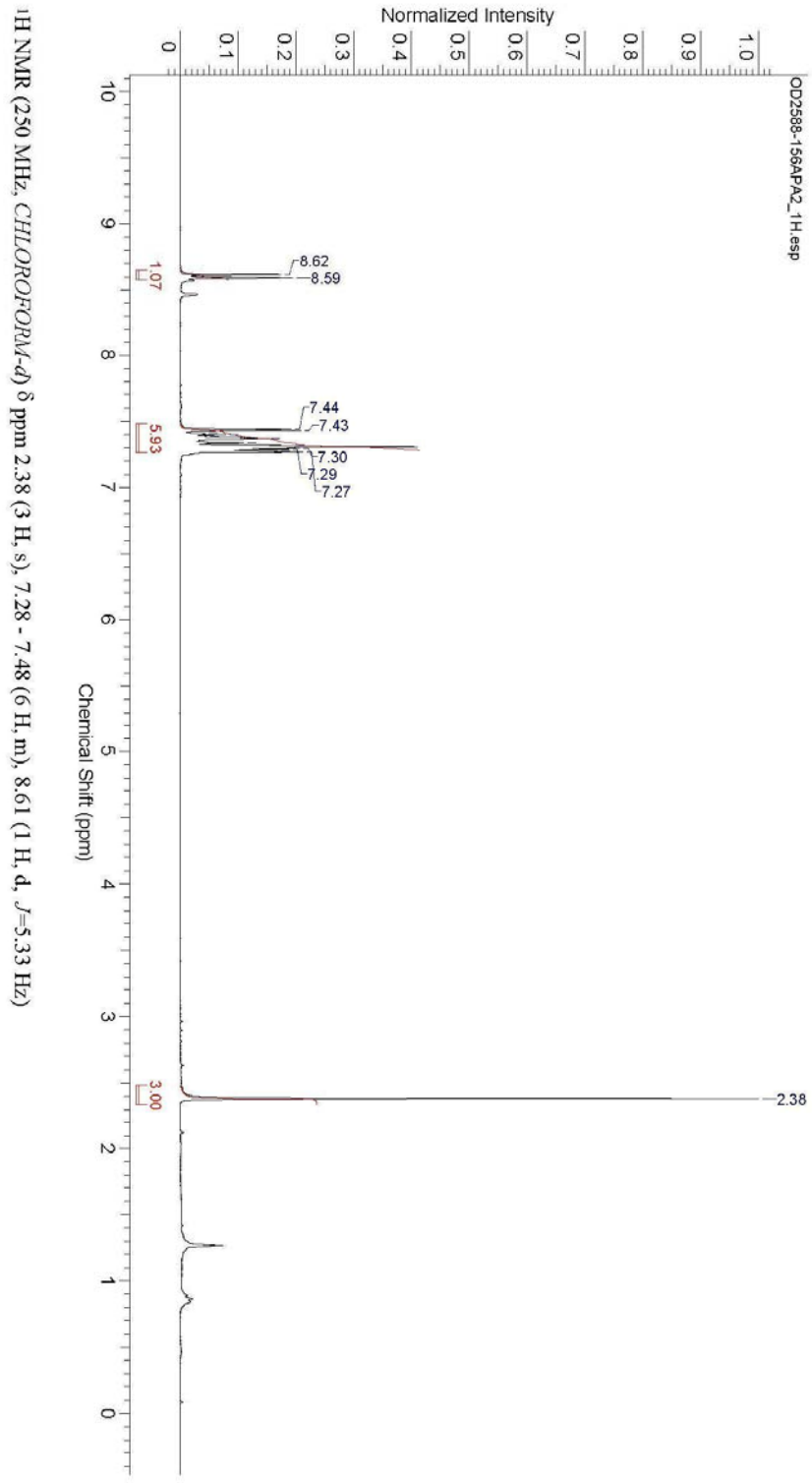
100.000
80.000
60.000
40.000
20.000
0.000

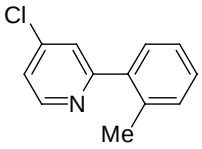


1f

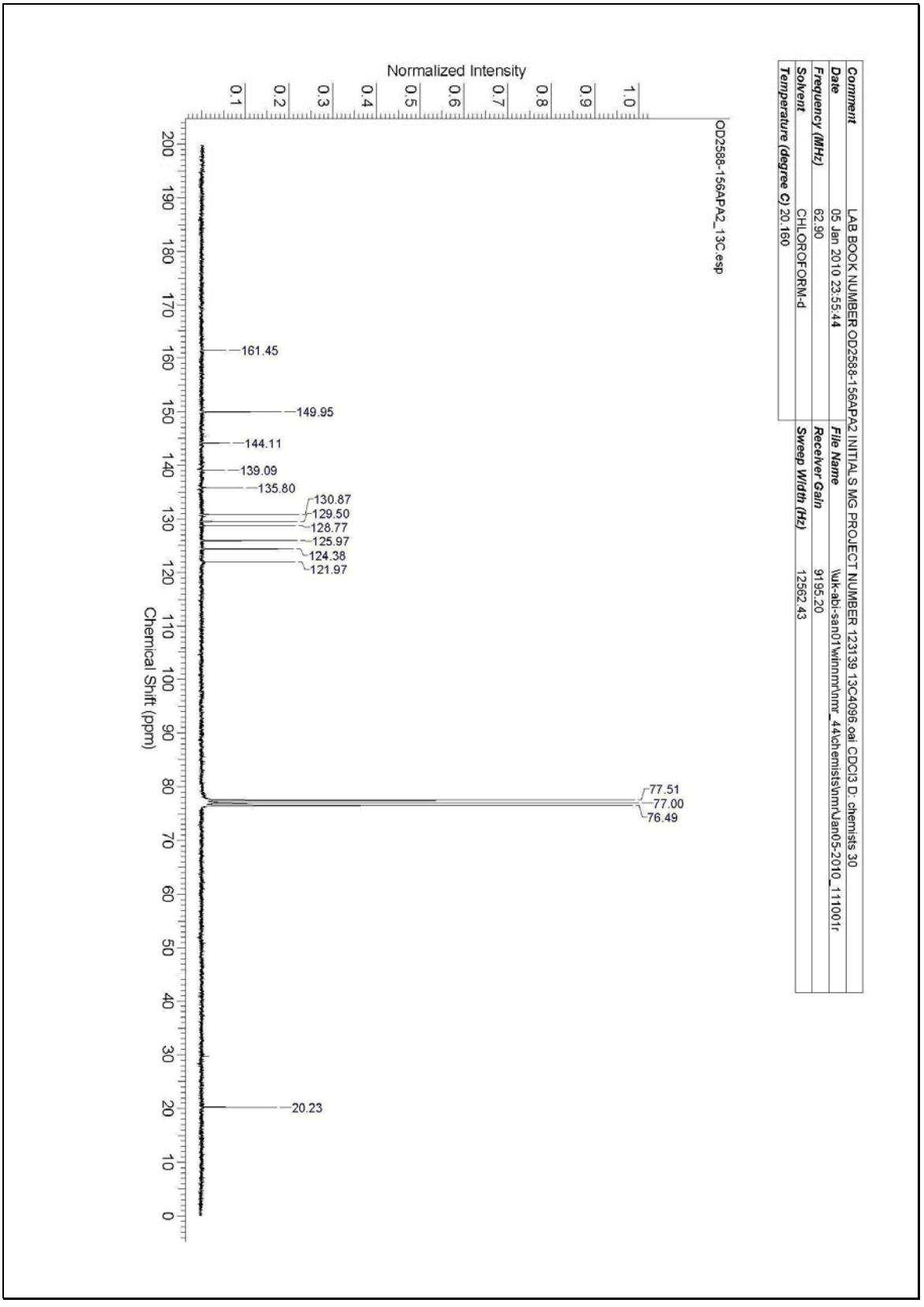
Comment	LAB BOOK NUMBER OD2588-156APA2 INITIALS MG PROJECT NUMBER 123139 proton.oal CDCl3 D: chemists 30
Date	05 Jan 2010 17:21:04
File Name	\\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Jan05-2010_110001r
Frequency (MHz)	250.13
Receiver Gain	1290.20
Solvent	CHLOROFORM-d
Sweep Width (Hz)	4999.87
Temperature (degree C)	22.160

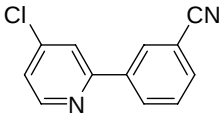


^{1}H NMR (250 MHz, *CHLOROFORM-d*) δ ppm 2.38 (3 H, s), 7.28 - 7.48 (6 H, m), 8.61 (1 H, d, *J*=5.33 Hz)

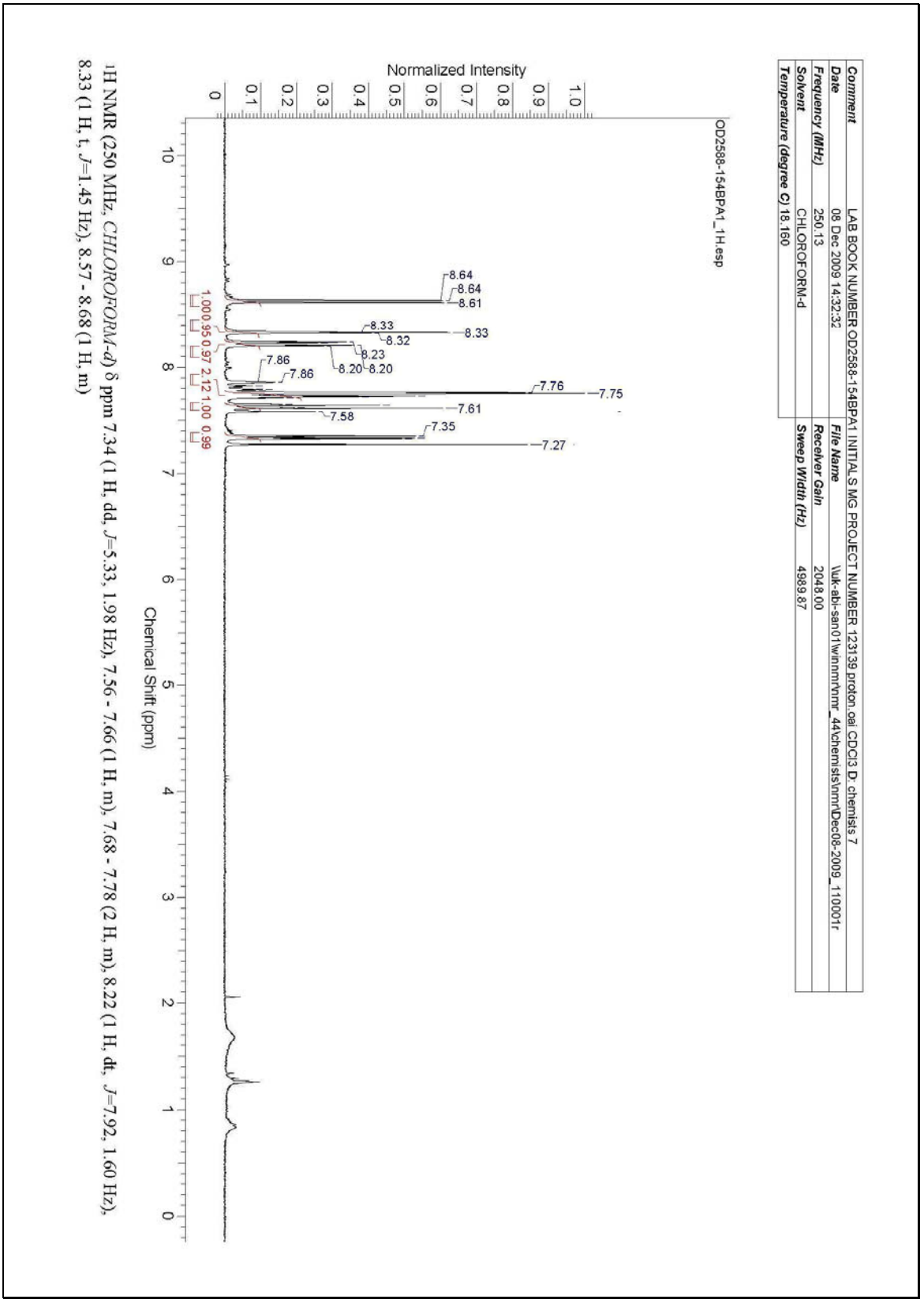

Cl
N
Me

1f

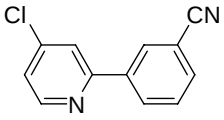

Comment LAB BOOK NUMBER OD2588-156APA2 INITIALS MG PROJECT NUMBER 123139 13C4096.oai CDCl3 D: chemists 30
Date 05 Jan 2010 23:55:44
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Jan05-2010_111001r
Frequency (MHz) 62.90
Receiver Gain 9195.20
Solvent CHLOROFORM-d
Sweep Width (Hz) 12562.43
Temperature (degree C) 20.160
OD2588-156APA2_13C.esp
Normalized Intensity
Chemical Shift (ppm)
161.45
149.95
144.11
139.09
135.80
130.87
129.50
128.77
125.97
124.38
121.97
77.51
77.00
76.49
20.23



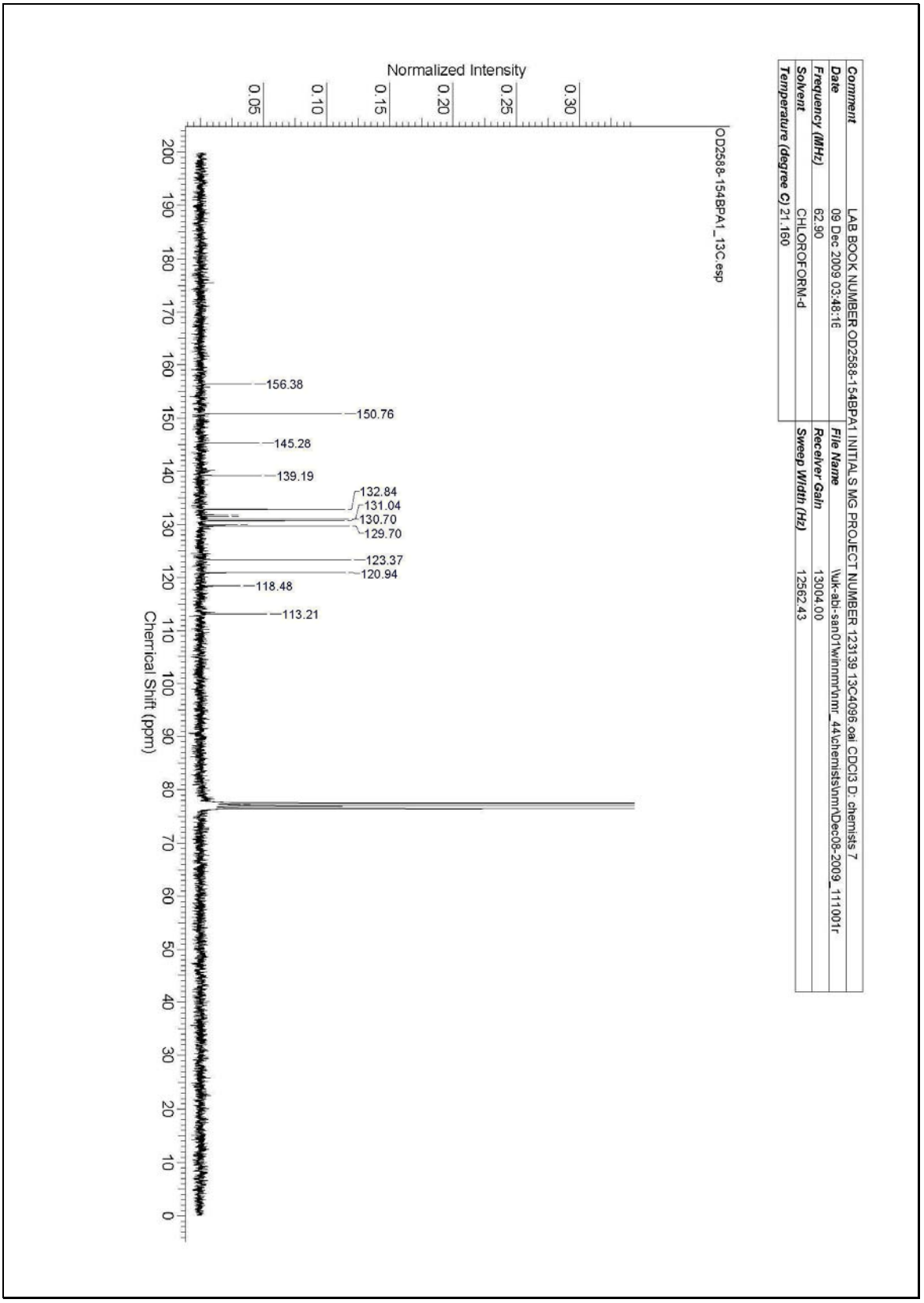
1g

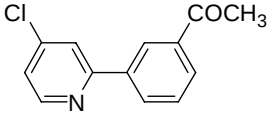


^{1}H NMR (250 MHz, *CHLOROFORM-d*) δ ppm 7.34 (1 H, dd, *J*=5.33, 1.98 Hz), 7.56 - 7.66 (1 H, m), 7.68 - 7.78 (2 H, m), 8.22 (1 H, dt, *J*=7.92, 1.60 Hz), 8.33 (1 H, t, *J*=1.45 Hz), 8.57 - 8.68 (1 H, m)

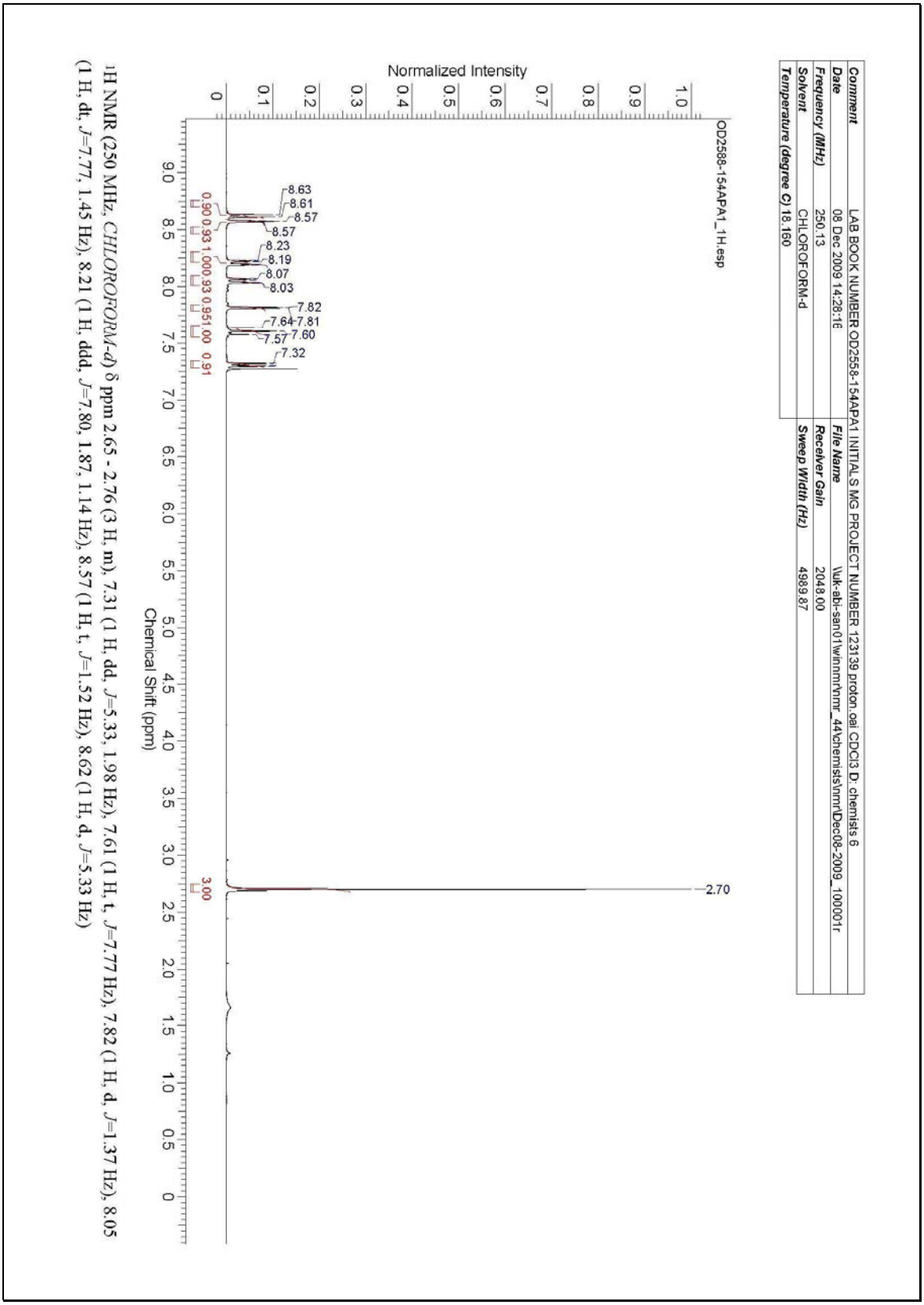

Cl
CN
N

1g

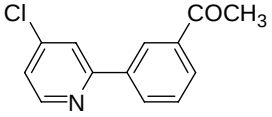

Comment LAB BOOK NUMBER OD2588-154BPA1 INITIALS MG PROJECT NUMBER 123139 13C4096.oai CDCl3 D: chemists 7
Date 09 Dec 2009 03:48:16
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Dec08-2009_111001r
Frequency (MHz) 62.90
Receiver Gain 13004.00
Solvent CHLOROFORM-d
Sweep Width (Hz) 12562.43
Temperature (degree C) 21.160
OD2588-154BPA1_13C.esp
Normalized Intensity
0.05
0.10
0.15
0.20
0.25
0.30
156.38
150.76
145.28
139.19
132.84
131.04
130.70
129.70
123.37
120.94
118.48
113.21
Chemical Shift (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



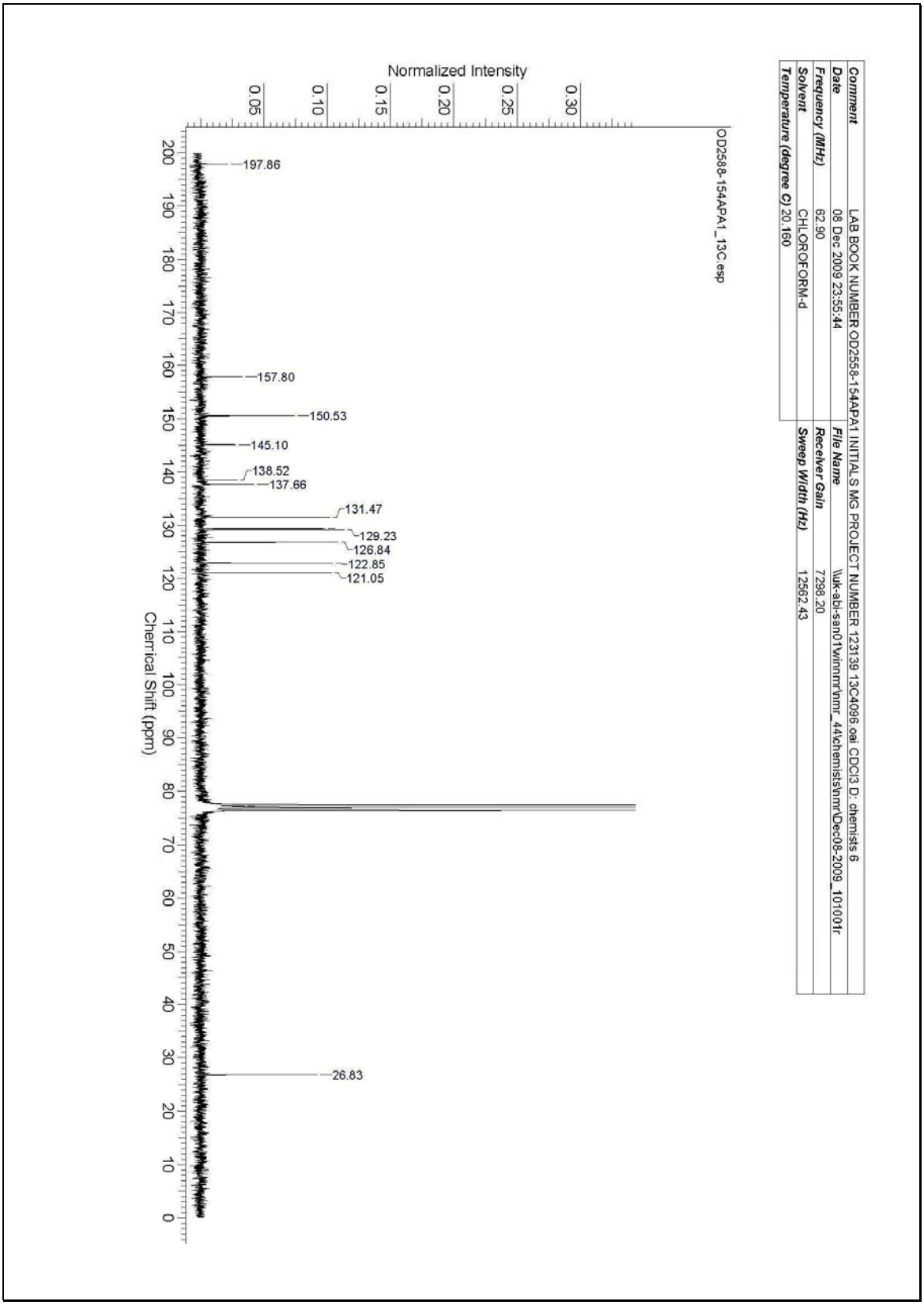
1h

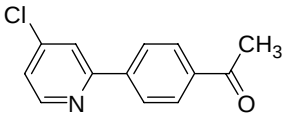


^{1}H NMR (250 MHz, *CHLOROFORM-d*) δ ppm 2.65 - 2.76 (3 H, m), 7.31 (1 H, dd, *J*=5.33, 1.98 Hz), 7.61 (1 H, t, *J*=7.77 Hz), 7.82 (1 H, d, *J*=1.37 Hz), 8.05 (1 H, dt, *J*=7.77, 1.45 Hz), 8.21 (1 H, ddd, *J*=7.80, 1.87, 1.14 Hz), 8.57 (1 H, t, *J*=1.52 Hz), 8.62 (1 H, d, *J*=5.33 Hz)


Cl
COCH3
N

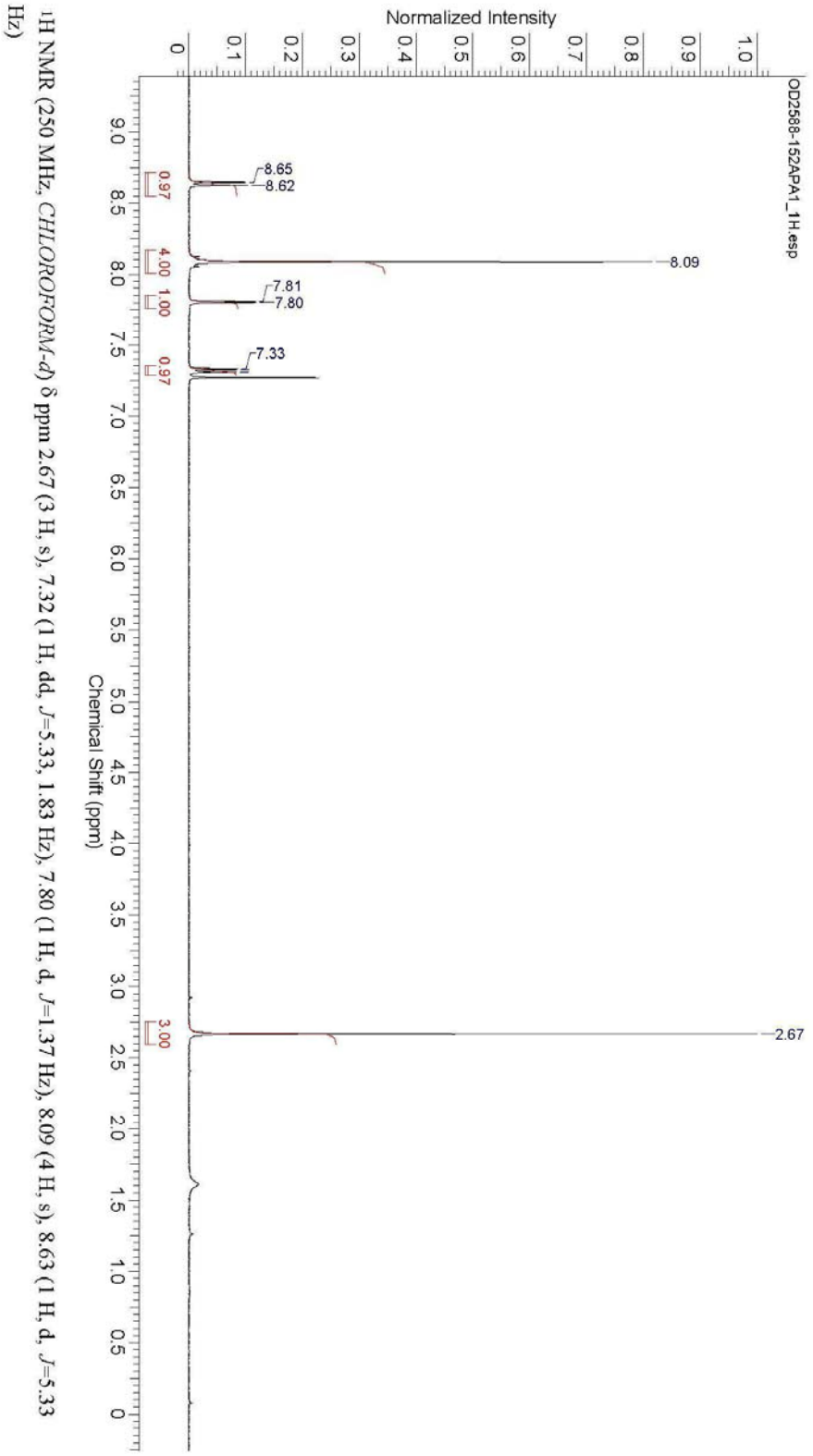
1h


Comment LAB BOOK NUMBER OD2558-154APA1 INITIALS MG PROJECT NUMBER 123139 13C4096.oai CDCl3 D: chemists 6
Date 08 Dec 2009 23:55:44
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Dec08-2009_101001r
Frequency (MHz) 62.90
Receiver Gain 7298.20
Solvent CHLOROFORM-d
Sweep Width (Hz) 12562.43
Temperature (degree C) 20.160
OD2558-154APA1_13C.esp
Normalized Intensity
Chemical Shift (ppm)
197.86
157.80
150.53
145.10
138.52
137.66
131.47
129.23
126.84
122.85
121.05
26.83

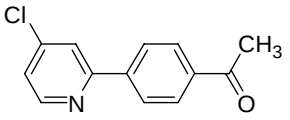


1i

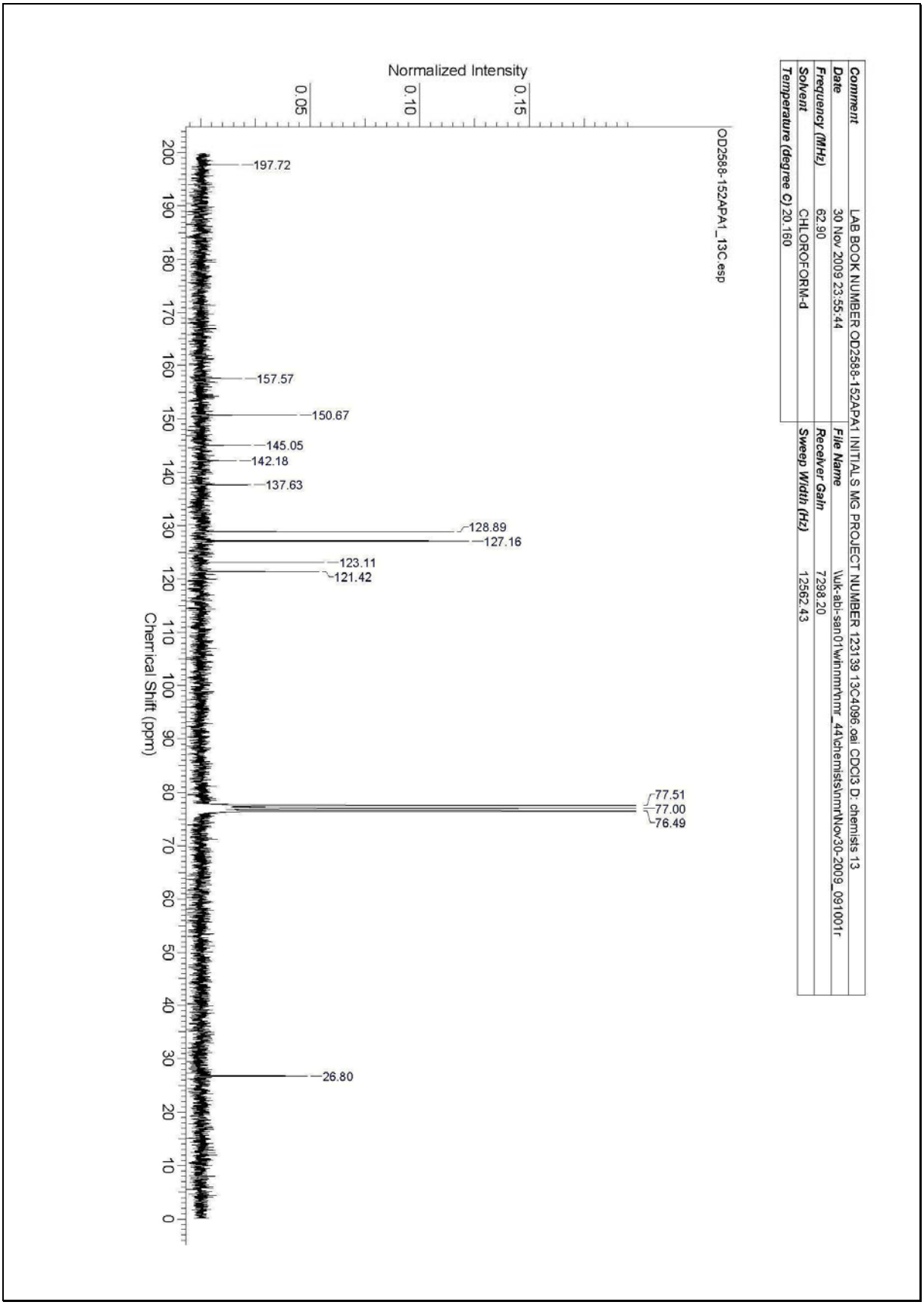
Comment	LAB BOOK NUMBER OD2588-152APA1 INITIALS MG PROJECT NUMBER 123139 proton.oai CDCl3 D: chemists 13
Date	30 Nov 2009 14:19:44
File Name	\\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Nov30-2009_090001r
Frequency (MHz)	250.13
Receiver Gain	3251.00
Solvent	CHLOROFORM-d
Sweep Width (Hz)	4989.87
Temperature (degree C)	21.160

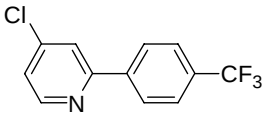


^{1}H NMR (250 MHz, *CHLOROFORM-d*) δ ppm 2.67 (3 H, s), 7.32 (1 H, dd, *J*=5.33, 1.83 Hz), 7.80 (1 H, d, *J*=1.37 Hz), 8.09 (4 H, s), 8.63 (1 H, d, *J*=5.33 Hz)

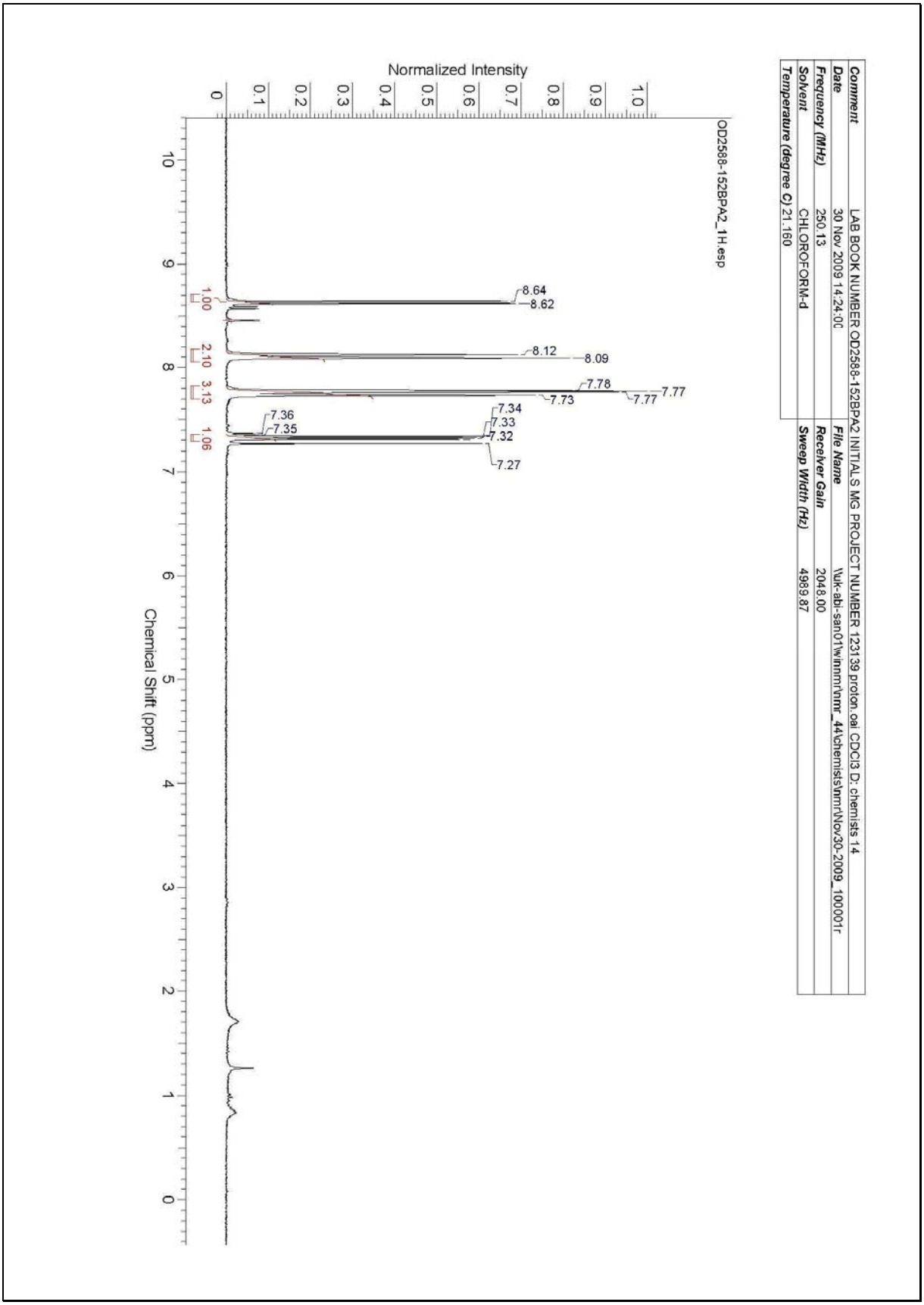

Cl
CH3
N
O

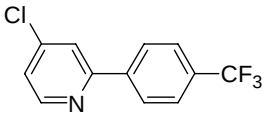
1i


Comment LAB BOOK NUMBER OD2588-152APA1 INITIALS MG PROJECT NUMBER 123139 13C4096.oel CDCl3 D: chemists 13
Date 30 Nov 2009 23:55:44
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmrNov30-2009_091001r
Frequency (MHz) 62.90
Receiver Gain 7298.20
Solvent CHLOROFORM-d
Sweep Width (Hz) 12562.43
Temperature (degree C) 20.160
OD2588-152APA1_13C.esp
Normalized Intensity
0.05
0.10
0.15
197.72
157.57
150.67
145.05
142.18
137.63
128.89
127.16
123.11
121.42
77.51
77.00
76.49
26.80
Chemical Shift (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

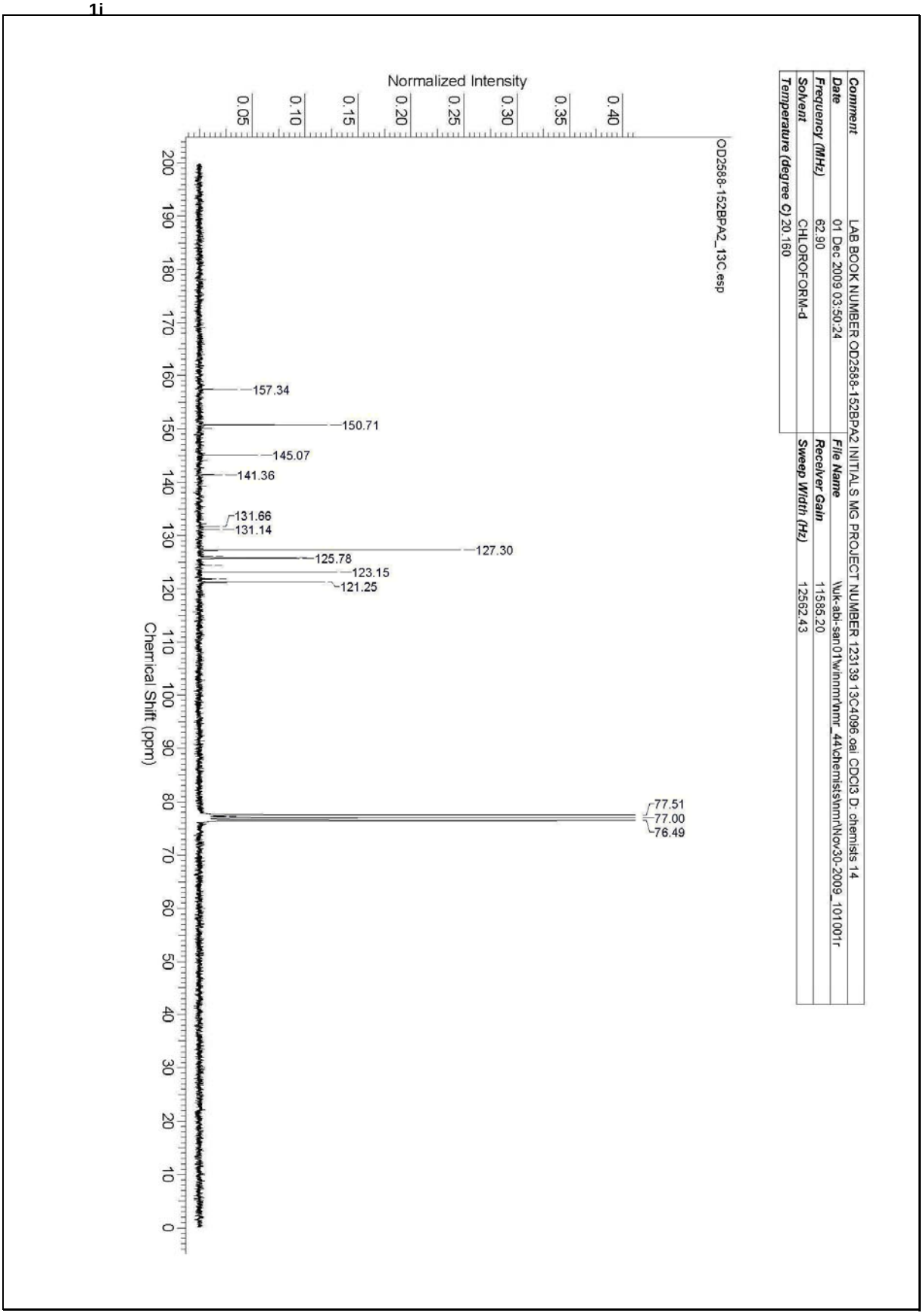

Cl
CF3
N

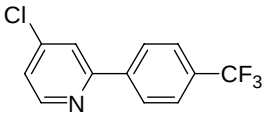
1j


Comment LAB BOOK NUMBER OD2588-152BPA2 INITIALS MG PROJECT NUMBER 123139 proton.oai CDCl3 D: chemists 14
Date 30 Nov 2009 14:24:00
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Nov30-2009_100001r
Frequency (MHz) 250.13
Receiver Gain 2048.00
Solvent CHLOROFORM-d
Sweep Width (Hz) 4999.87
Temperature (degree C) 21.160
Normalized Intensity
OD2588-152BPA2_1H.esp
8.64
8.62
8.12
8.09
7.78
7.77
7.73
7.77
7.36
7.35
7.34
7.33
7.32
7.27
1.00
2.10
3.13
1.06
Chemical Shift (ppm)

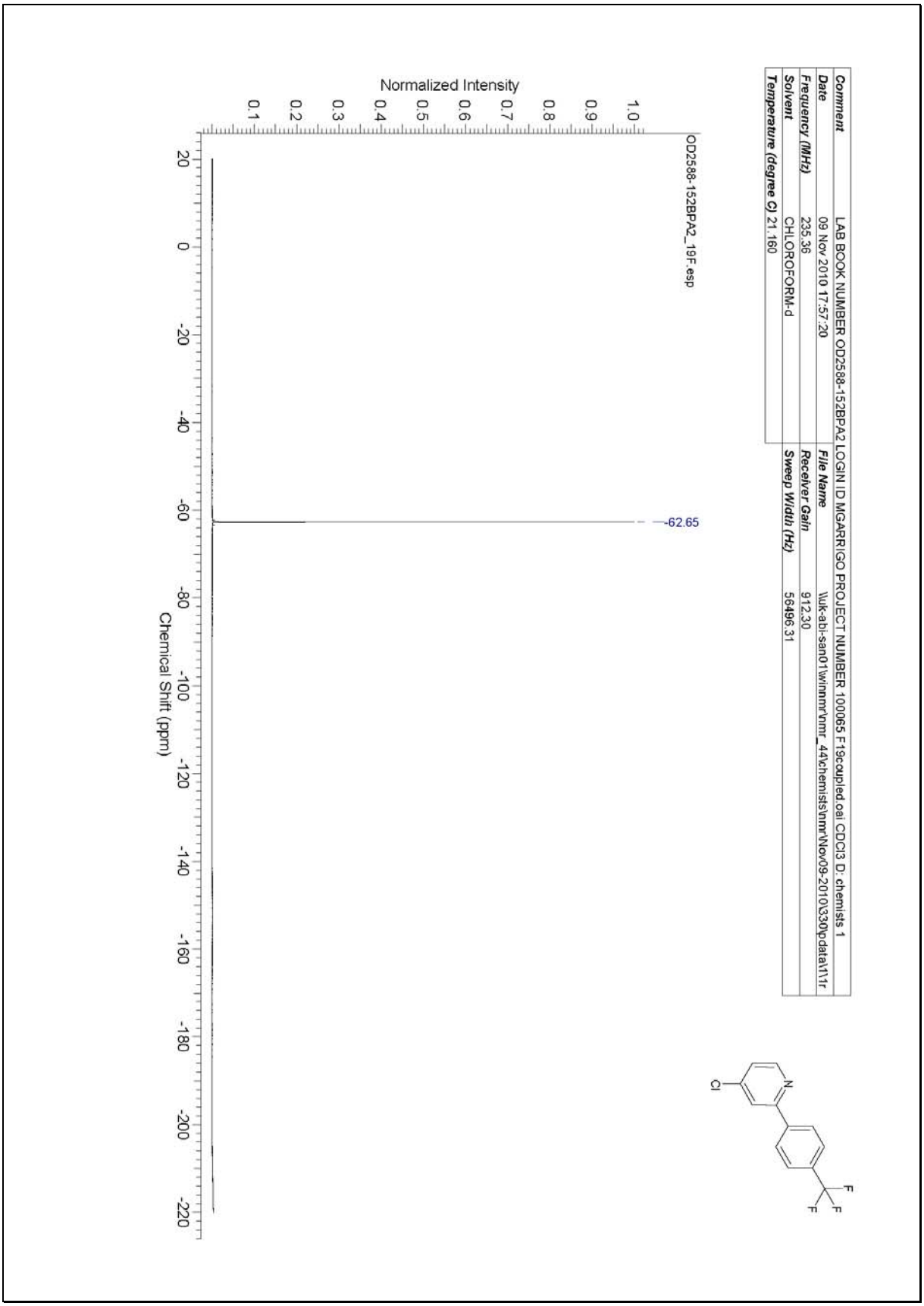

Cl
CF3
N

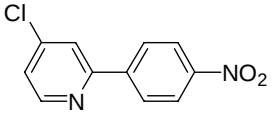
1i


Comment LAB BOOK NUMBER OD2588-152BPA2 INITIALS MG PROJECT NUMBER 123139 13C4096.oai CDCl3 D:\chemists 14
Date 01 Dec 2009 03:50:24
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Nov30-2009_101001r
Frequency (MHz) 62.90
Receiver Gain 11585.20
Solvent CHLOROFORM-d
Sweep Width (Hz) 12562.43
Temperature (degree C) 20.160
OD2588-152BPA2_13C.esp
Normalized Intensity
Chemical Shift (ppm)
157.34
150.71
145.07
141.36
131.66
131.14
127.30
125.78
123.15
121.25
77.51
77.00
76.49


Cl
N
CF3

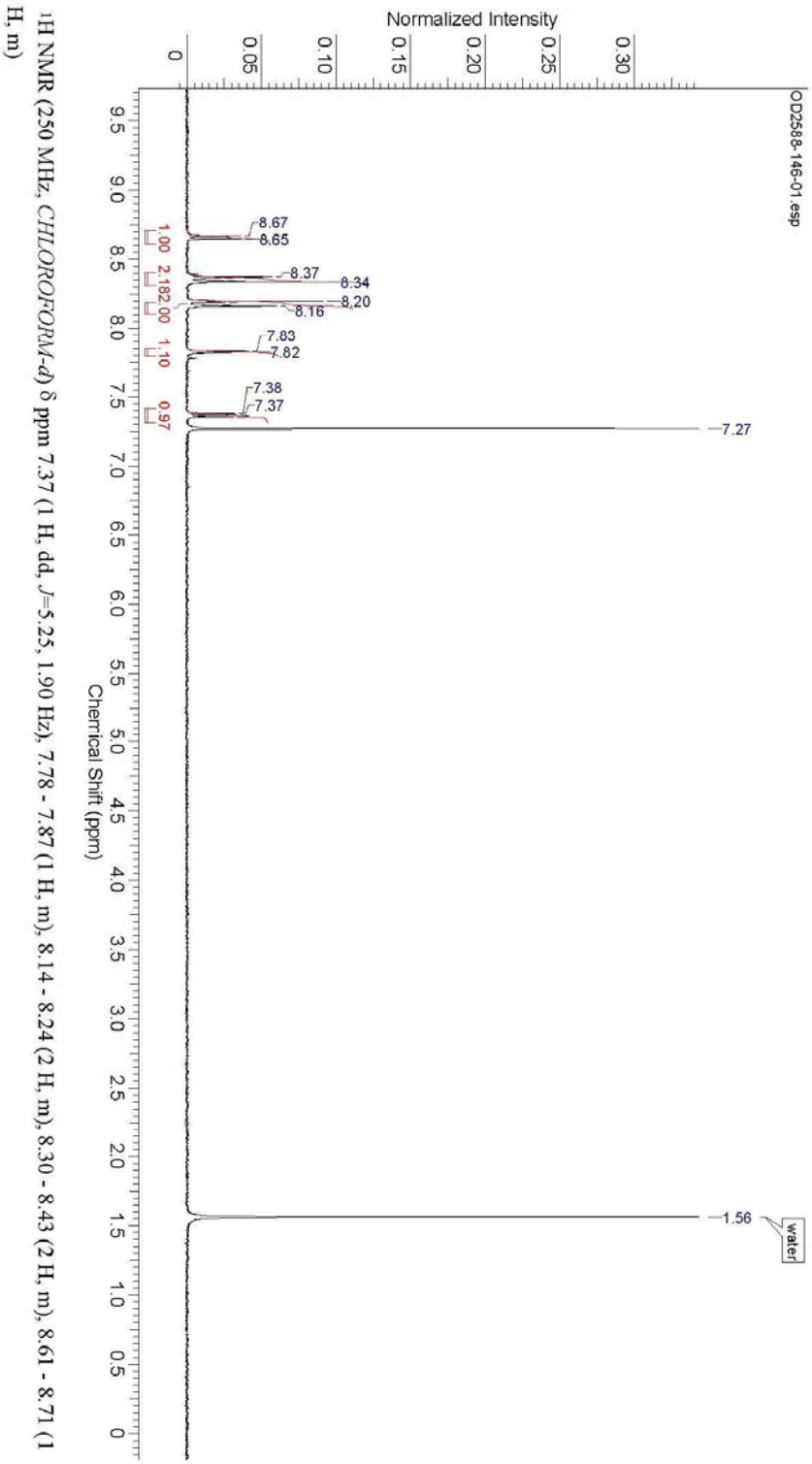
1j


Comment LAB BOOK NUMBER OD2588-152BPA2 LOGIN ID MGARRIGO PROJECT NUMBER 100065 F19cupled.oai CDCl3 D: chemists 1
Date 09 Nov 2010 17:57:20
File Name \\uk-abi-san01\winnmr\nmr_44\chemists\nmr\Nov09-20100330\pdata\11\r
Frequency (MHz) 235.36
Receiver Gain 912.30
Solvent CHLOROFORM-d
Sweep Width (Hz) 56496.31
Temperature (degree C) 21.160
Normalized Intensity
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
OD2588-152BPA2_19F.esp
-62.65
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
Chemical Shift (ppm)
Cl
N
F
F
F

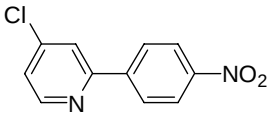


1k

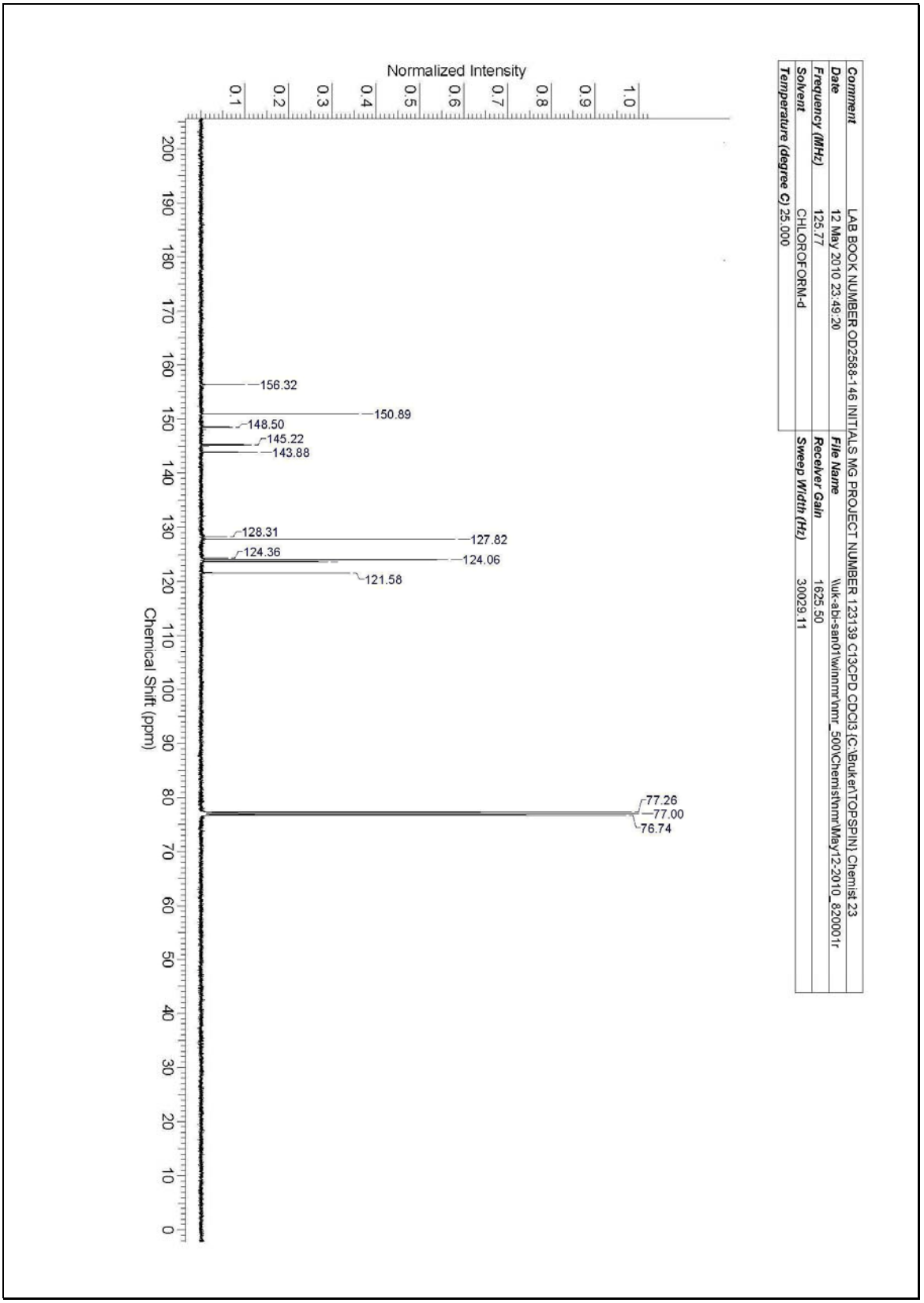
Comment	LAB BOOK NUMBER OD2588-146-01 INITIALS SN PROJECT NUMBER 120008 proton.oai CDCl3 D: chemists 37
Date	29 Jun 2009 09:38:08
File Name	\\uk-abi-san01\winnmr\Archive\NMR_250_2009\2009\Chemist09\June09\Jun29-2009_210001r
Frequency (MHz)	250.13
Receiver Gain	4597.60
Solvent	CHLOROFORM-d
Sweep Width (Hz)	4989.87
Temperature (degree C)	25.160

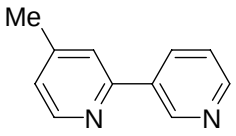


^{1}H NMR (250 MHz, *CHLOROFORM-d*) δ ppm 7.37 (1 H, dd, *J*=5.25, 1.90 Hz), 7.78 - 7.87 (1 H, m), 8.14 - 8.24 (2 H, m), 8.30 - 8.43 (2 H, m), 8.61 - 8.71 (1 H, m)

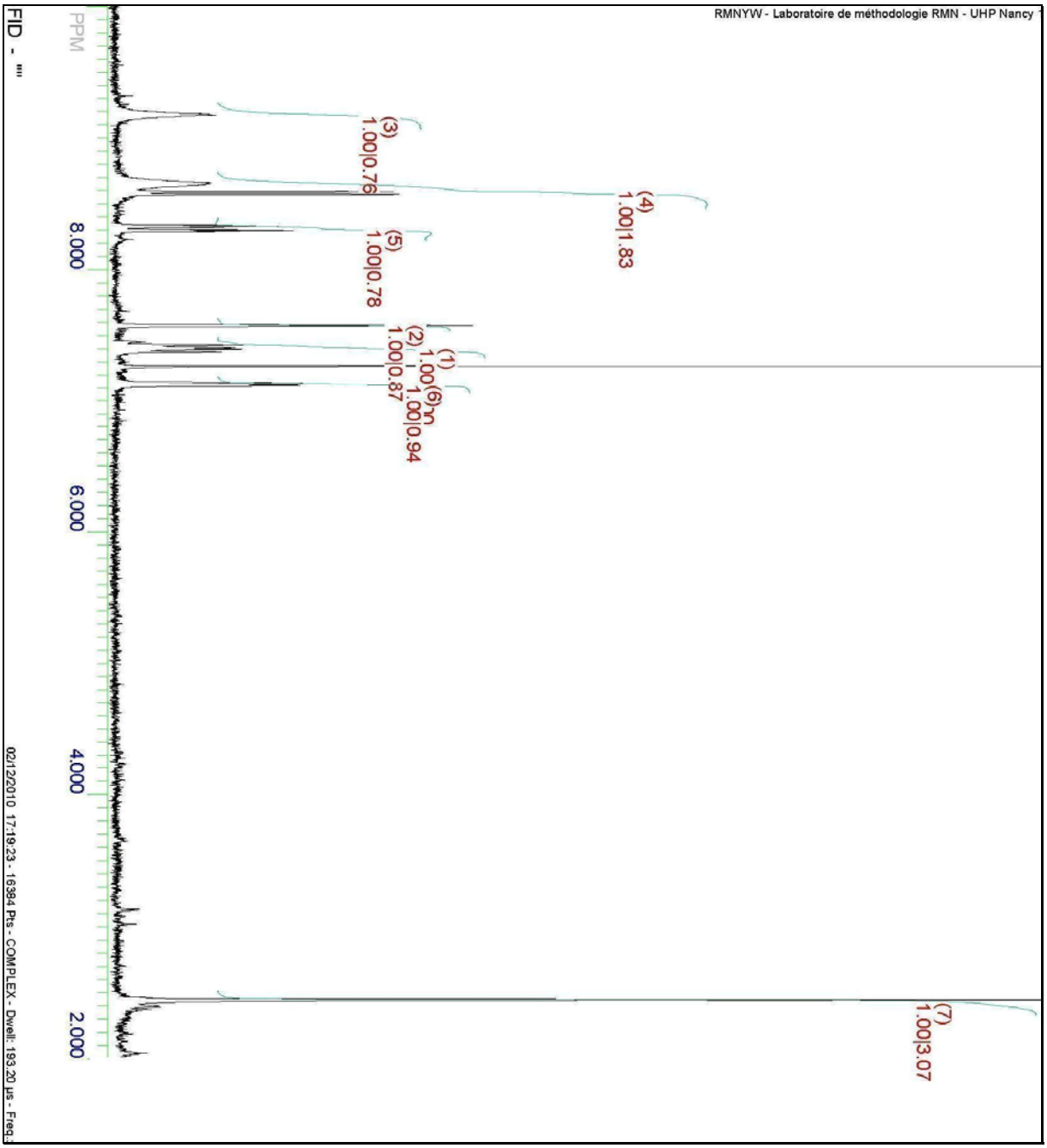

Cl
N
NO2

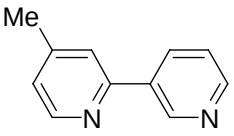
1k


Comment LAB BOOK NUMBER OD2588-146 INITIALS MG PROJECT NUMBER 123139 C13CPD CDCl3 {C:\Bruker\TOPSPIN} Chemist 23
Date 12 May 2010 23:49:20
File Name \\uk-abi-san01\winnmr\nmr_500\Chemist\nmr\May12-2010_820001r
Frequency (MHz) 125.77
Receiver Gain 1625.50
Solvent CHLOROFORM-d
Sweep Width (Hz) 30029.11
Temperature (degree C) 25.000
Normalized Intensity
Chemical Shift (ppm)
156.32
150.89
148.50
145.22
143.88
128.31
127.82
124.36
124.06
121.58
77.26
77.00
76.74

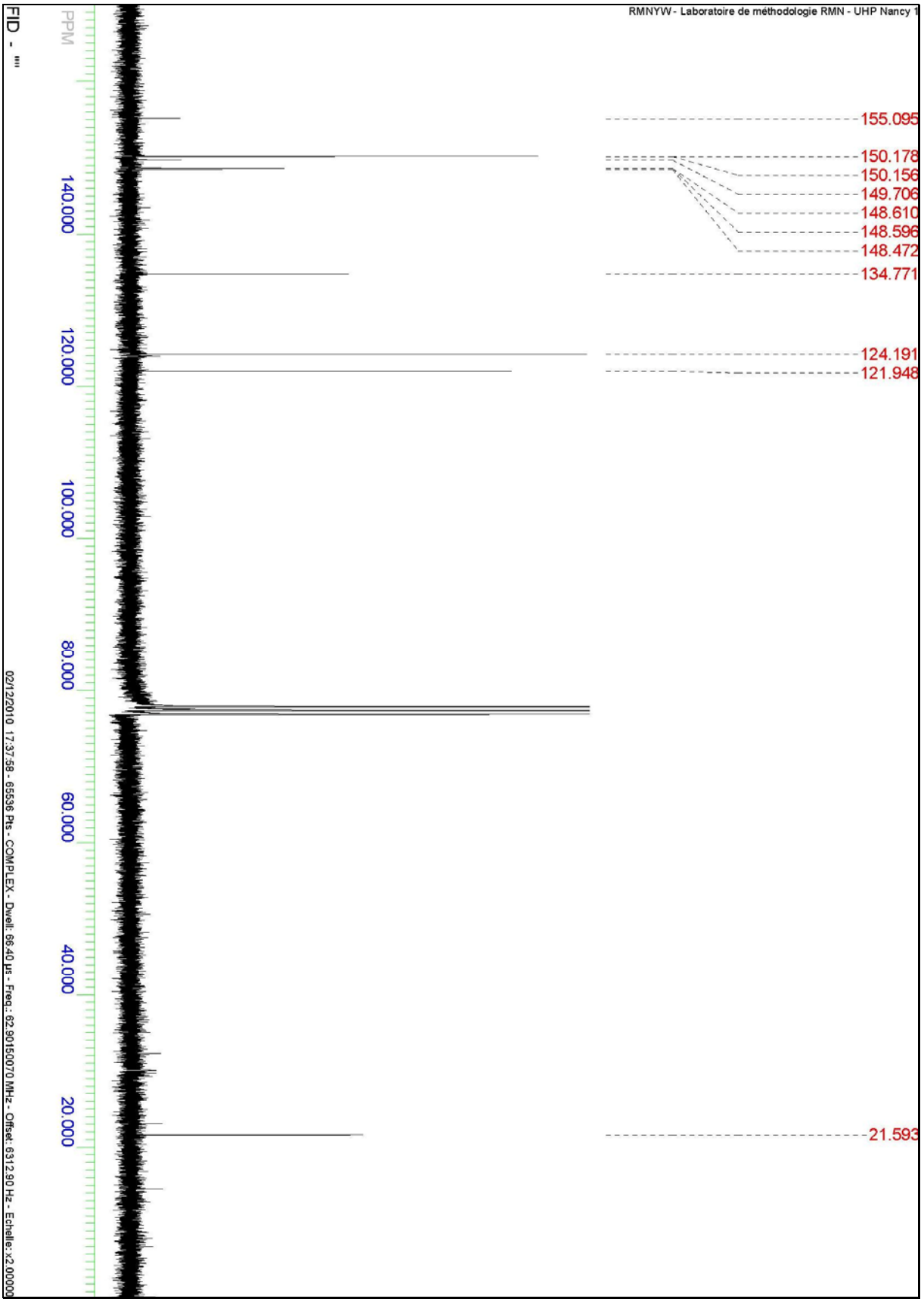

Me
N
N

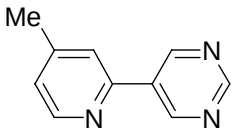
4a


RMNYW - Laboratoire de méthodologie RMN - UHP Nancy
FID - "
PPM
8.000
6.000
4.000
2.000
(3) 1.00|0.76
(4) 1.00|1.83
(5) 1.00|0.78
(2) 1.00|0.87
(1)
(6) 1.00|0.94
(7) 1.00|3.07
02/12/2010 17:19:23 - 16384 Pts - COMPLEX - Dwell: 193.20 µs - Freq

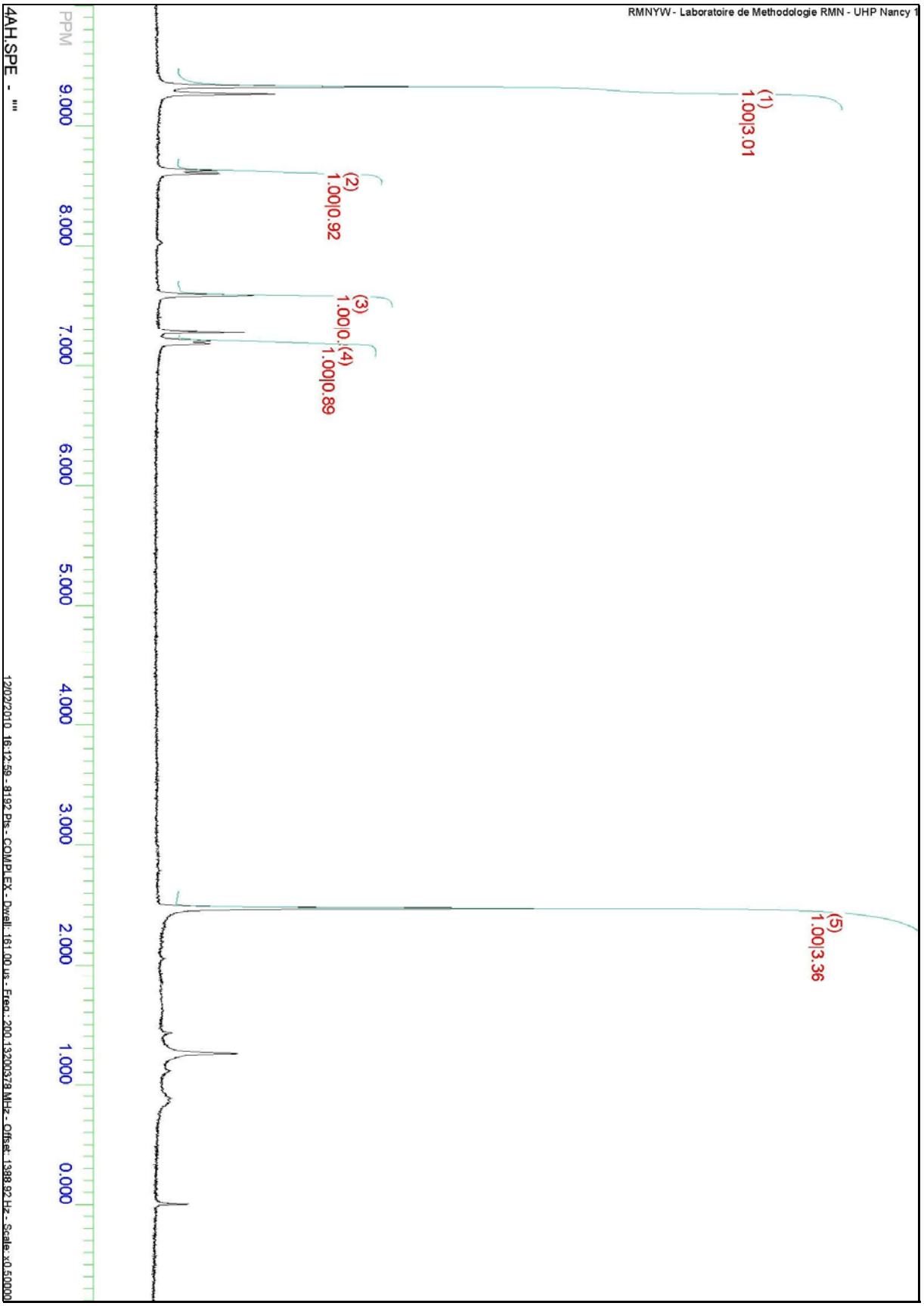

Me
N
N

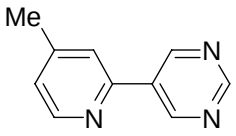
4a


FID - ""
PPM
RMNYW - Laboratoire de méthodologie RMN - UHP Nancy 1
155.095
150.178
150.156
149.706
148.610
148.596
148.472
134.771
124.191
121.948
21.593
140.000
120.000
100.000
80.000
60.000
40.000
20.000
02/12/2010 17:37:58 - 65536 Pts - COMPLEX - Dwell: 66.40 µs - Freq.: 62.90150070 MHz - Offset: 6312.90 Hz - Echelle: x2.00000

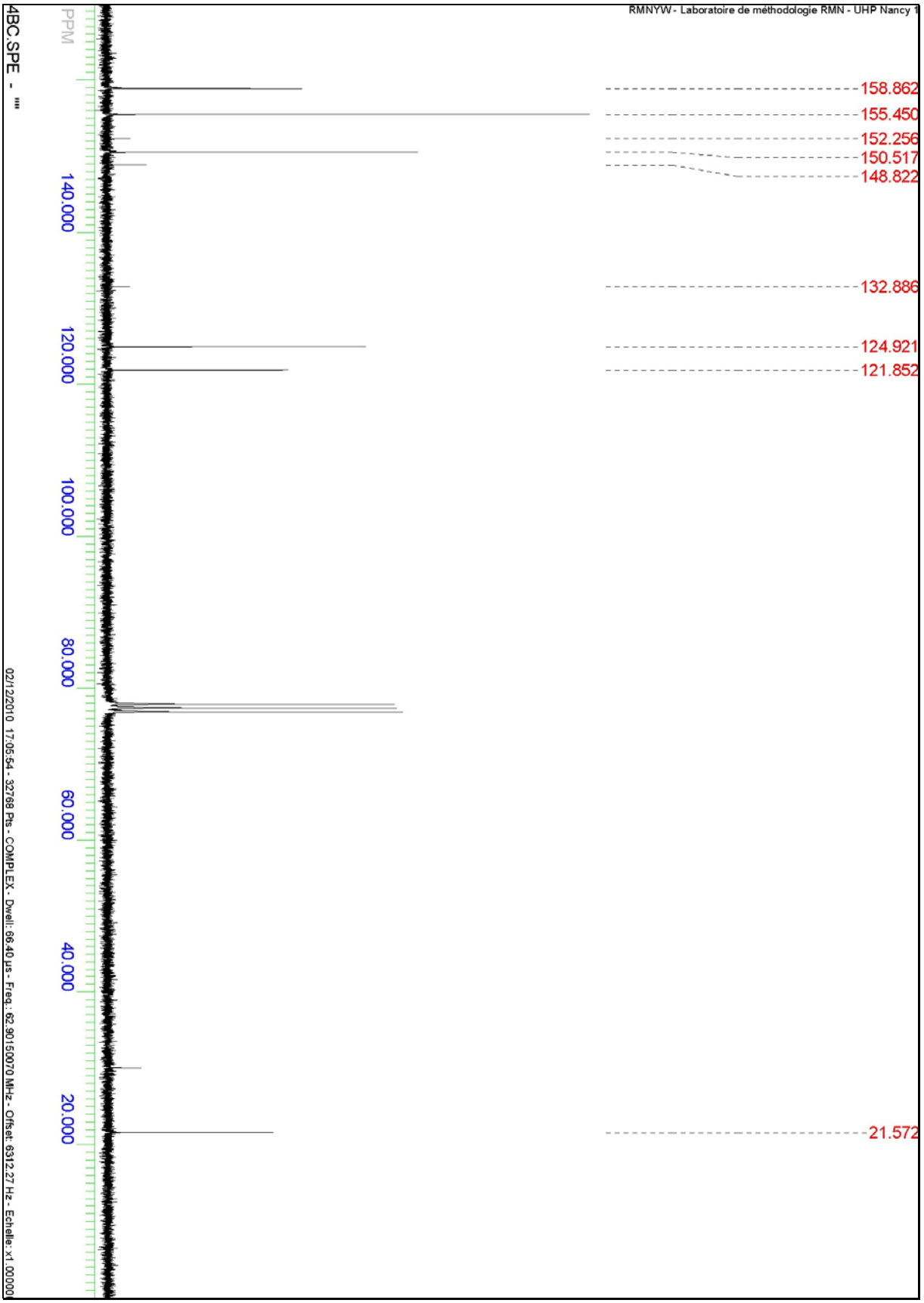

Me
N
N
N

4b


RMNYW - Laboratoire de Methodologie RMN - UHP Nancy 1
4AH.SPE - ""
PPM
9.000
8.000
7.000
6.000
5.000
4.000
3.000
2.000
1.000
0.000
(1)
1.00|3.01
(2)
1.00|0.92
(3)
1.00|0.
(4)
1.00|0.89
(5)
1.00|3.36
12/02/2010 16:12:58 - 8192 Pts - COMPLEX - Dwell: 161.00 us - Freq: 200.13200378 MHz - Offset: 1388.92 Hz - Scale: x0.50000


Me
N
N
N

4b


4BC.SPE - ""
PPM
RMNYW - Laboratoire de méthodologie RMN - UHP Nancy 1
158.862
155.450
152.256
150.517
148.822
132.886
124.921
121.852
21.572
140.000
120.000
100.000
80.000
60.000
40.000
20.000
02/12/2010 17:05:54 - 32768 Pts - COMPLEX - Dwell: 86.40 µs - Freq: 62.90150070 MHz - Offset: 6312.27 Hz - Echelle: x1.00000