

[5C + 1N] Annulation of 2,4-Pentadienenitriles with Hydroxylamine: A Synthetic Route to Multi-substituted 2-Aminopyridines

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130022, P. R. China

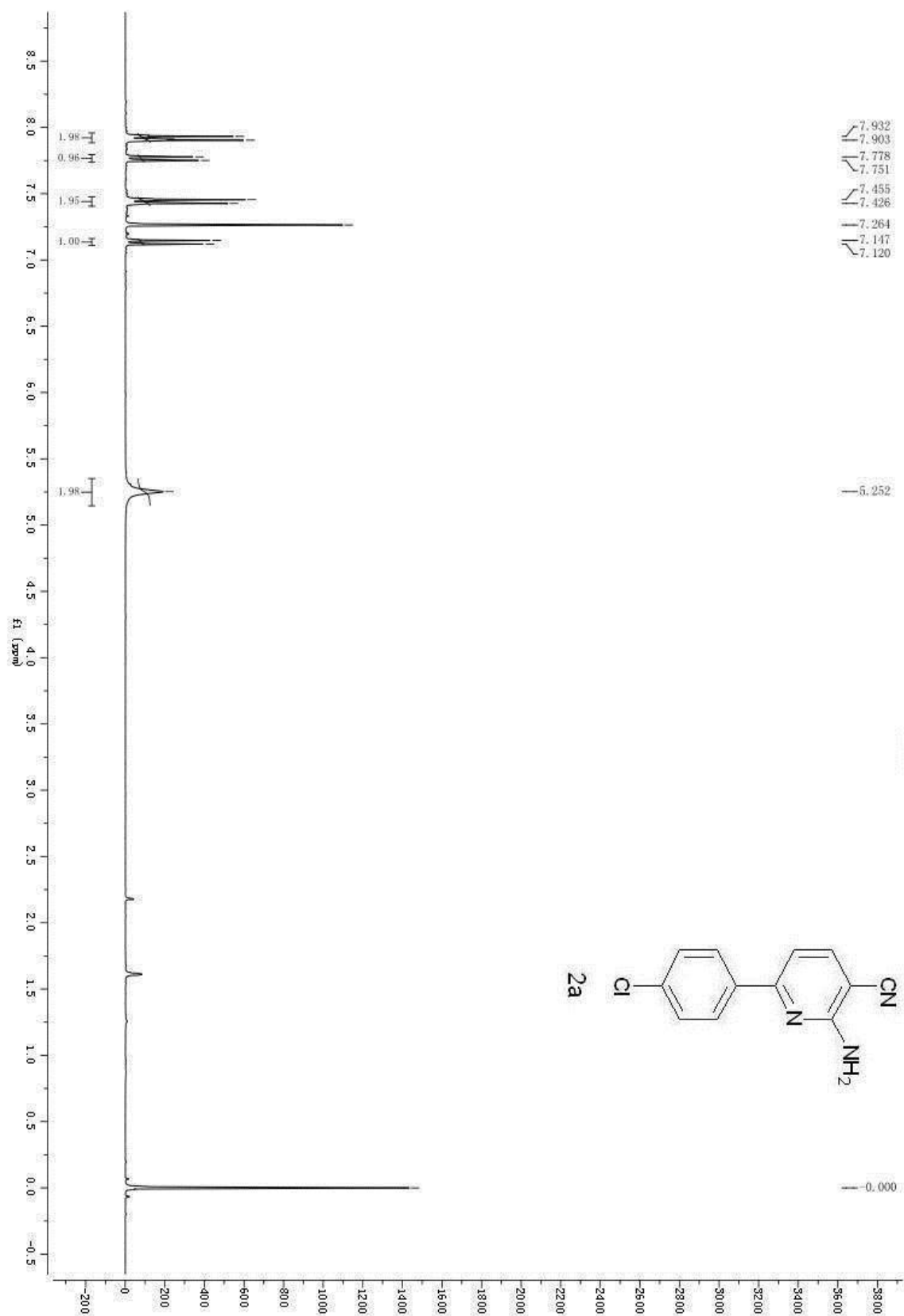
^b Changzhou Institute of Energy Storage Materials & Devices, Changzhou, 213000, P. R.
China

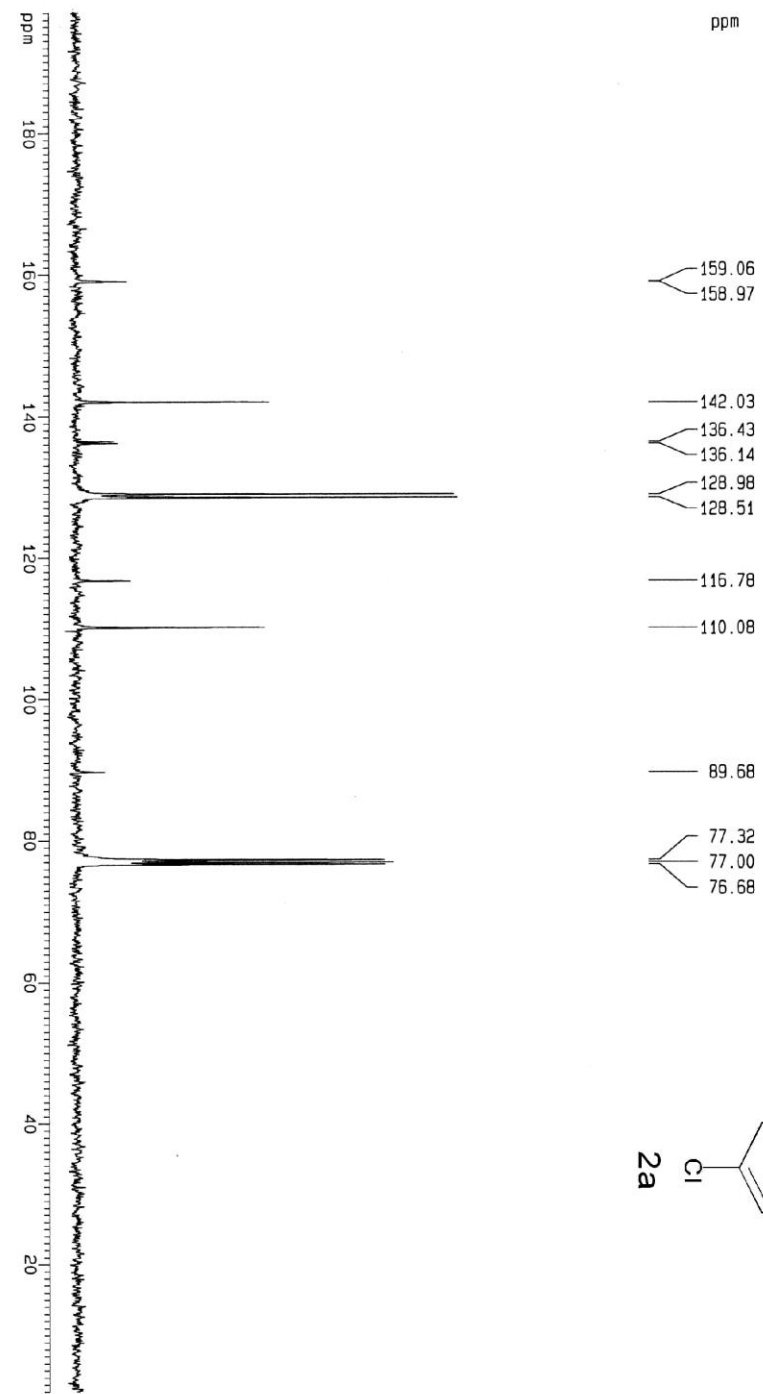
E-mail: dwdong@ciac.jl.cn

Supporting Information

Copies of NMR spectra for compounds **2a-u**S2-S43

Copies of NMR spectra for compounds 2a-u





xxxxB

Current Data Parameters:

NAME	XXB
EXPNO	8
PROCNO	1

F2 - Acquisition Parameters

Date_	20101221
Time	0.12
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg
TD	16384
SOLVENT	CDCl3
NS	1398
DS	0
SWH	25062.656 Hz
FIDRES	1.929703 Hz
AQ	0.3266108 sec
RG	8192
DM	19.950 usec
DE	6.00 usec
TE	300.0 K
D1	0.69999999 sec
d11	0.03000000 sec

----- CHANNEL f1 -----

NUC1	¹³ C
P1	2.80 usec
PL1	-3.00 dB
SFO1	100.5785254 MHz

----- CHANNEL f2 -----

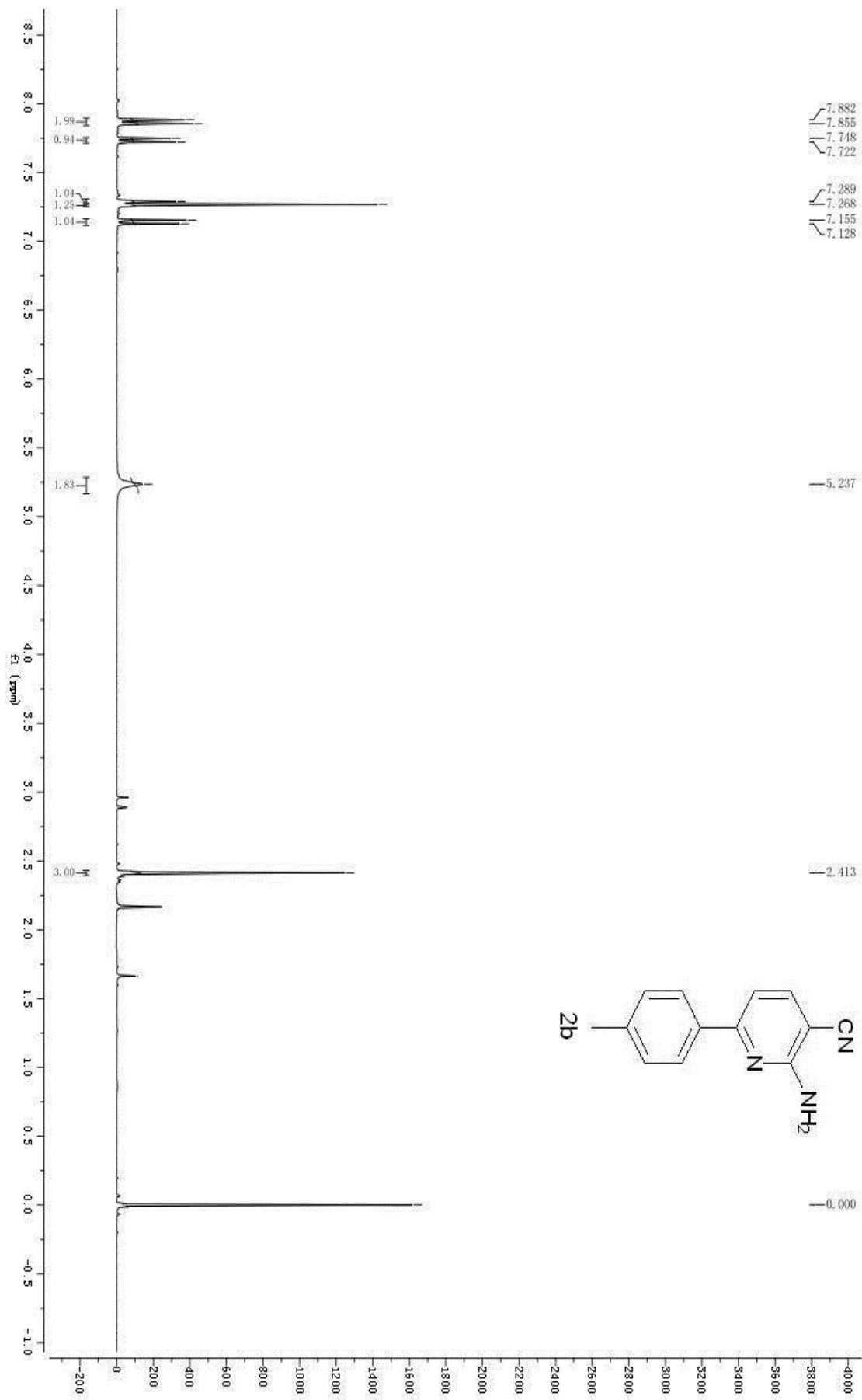
CPDPRG2	Waltz16
NUC2	¹ H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	19.50 dB
SFO2	399.9613998 MHz

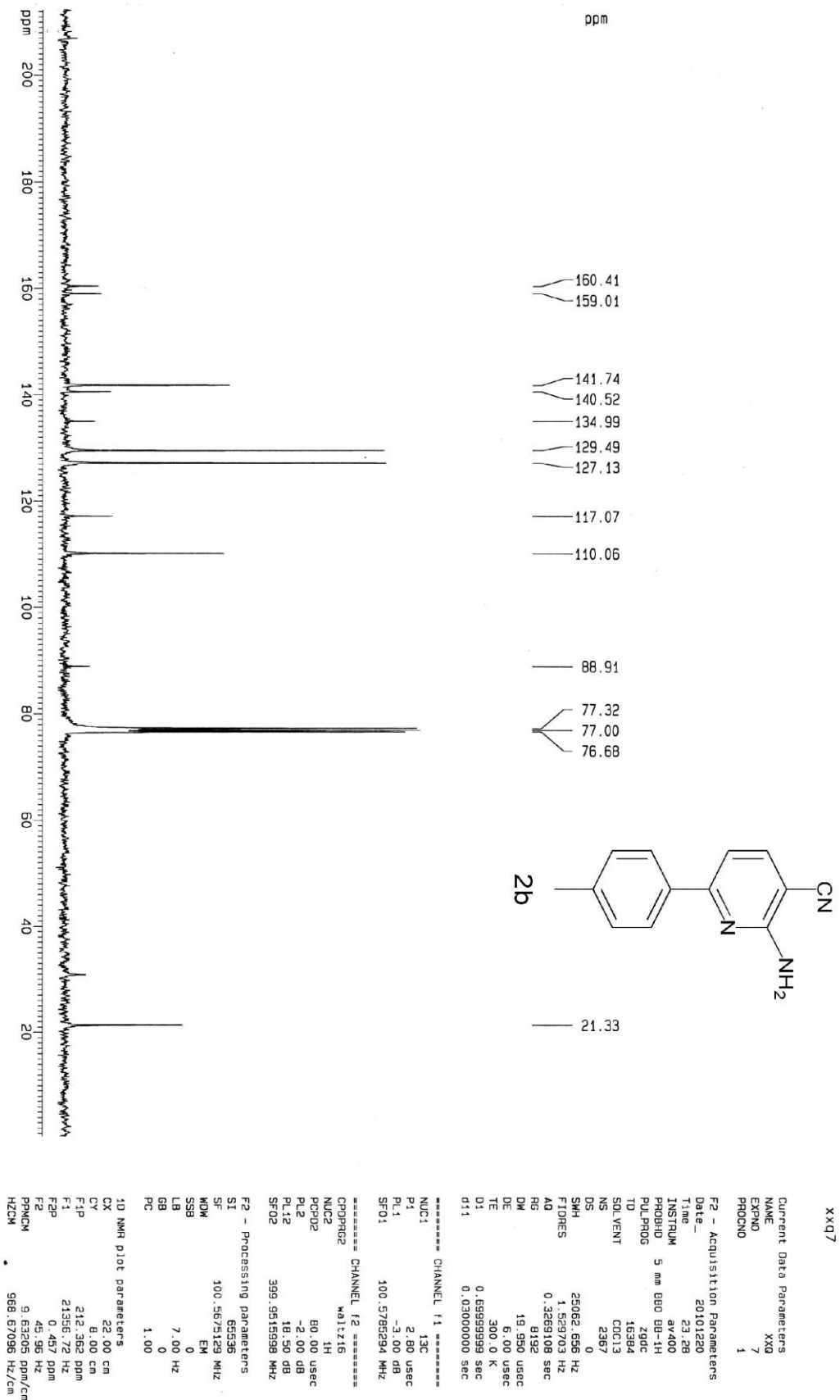
F2 - Processing parameters

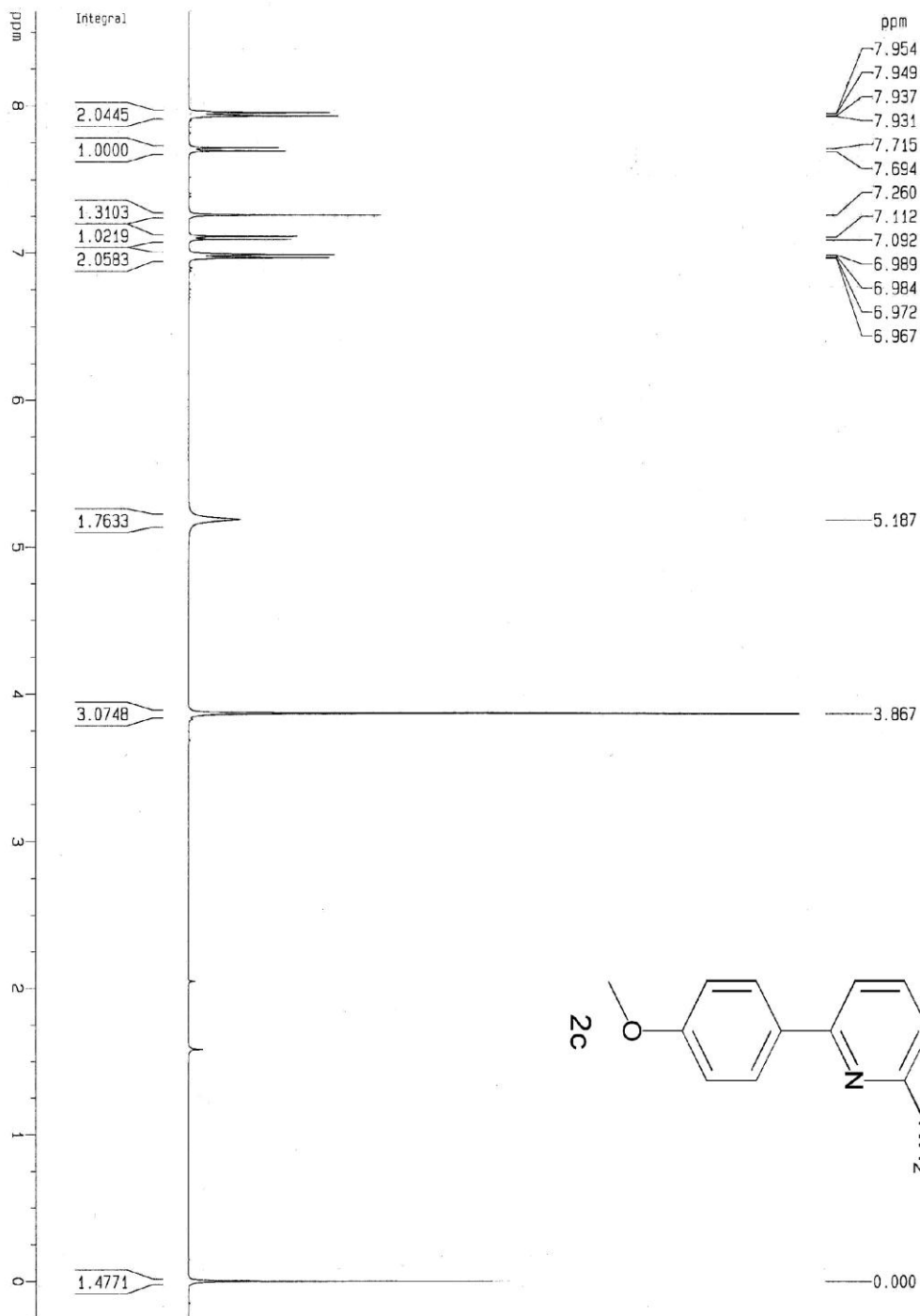
SF	65536
SF	100.5675122 MHz
WDW	EM
SSB	0
LB	7.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	7.00 cm
F1P	197.148 ppm
F1	19826.71 Hz
F2P	1.957 ppm
F2	196.80 Hz
PPMCK	8.87234 ppm/cm
HZCM	892.26880 Hz/cm







XXQ1-19H

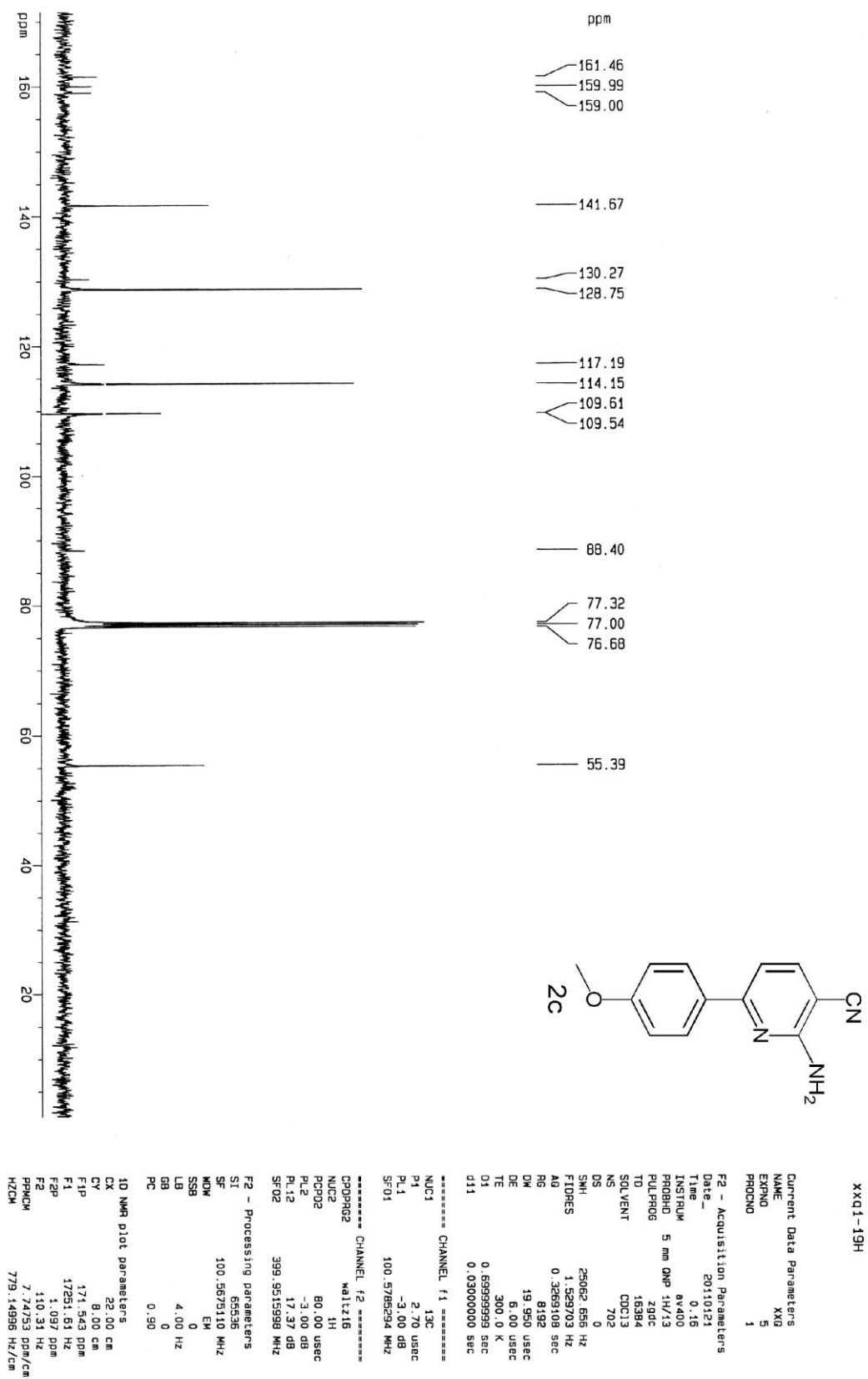
Current Data Parameters
 NAME XXX
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110119
 Time 22.05
 INSTRUM av400
 PROBNM 5 mm BBO BB-1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4401.409 Hz
 FIDRES 0.268841 Hz
 AQ 1.8612725 sec
 RG 128
 DW 113.600 usec
 DE 5.00 usec
 TE 300.0 K
 D1 10.00000000 sec

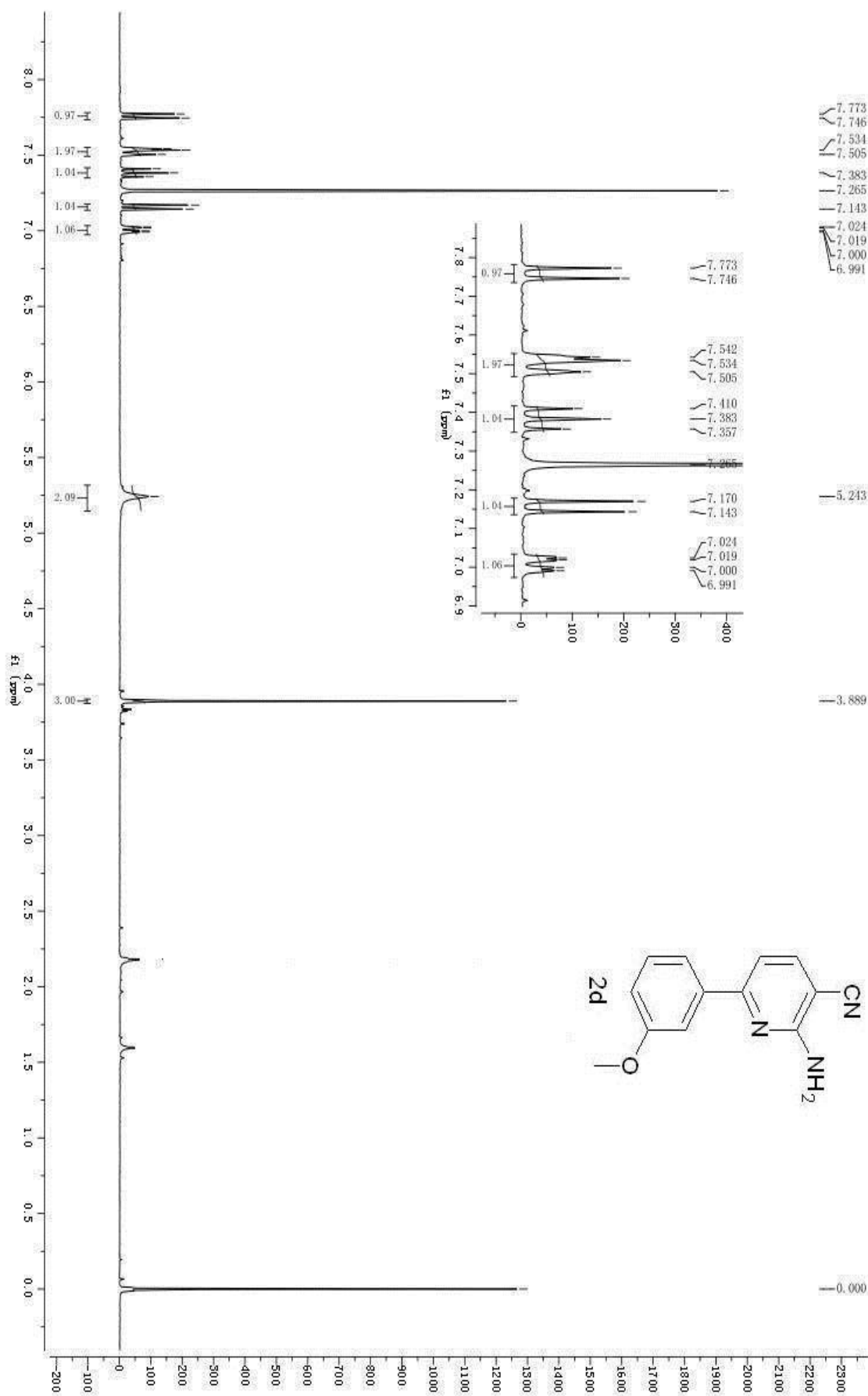
***** CHANNEL f1 *****
 NUC1 1H
 P1 8.50 usec
 PL1 -2.00 dB
 SFO1 399.9517683 MHz

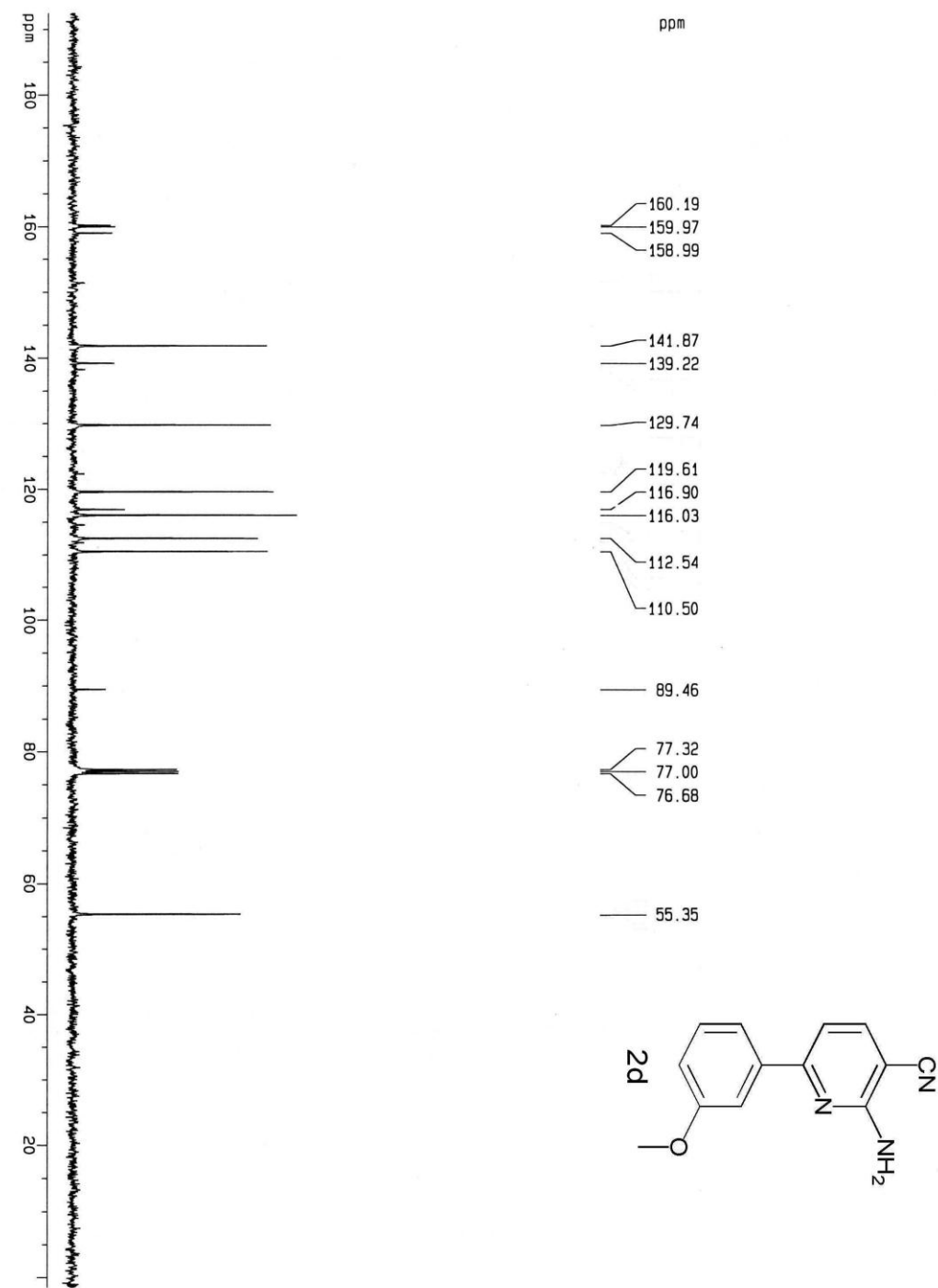
F2 - Processing parameters
 SI 65536
 SF 399.9500104 MHz
 KMDK EX
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 12.00 cm
 F1P 8.543 ppm
 F1 3456.91 Hz
 F2P -100.71 Hz
 F2 40433.000000000000 Hz/cm
 HZCM 161.70979 Hz/cm



XXQ1-19H





xxq1-20-1

Current Data Parameters
 NAME XXJ
 EXPNO 6
 PROCNO 1

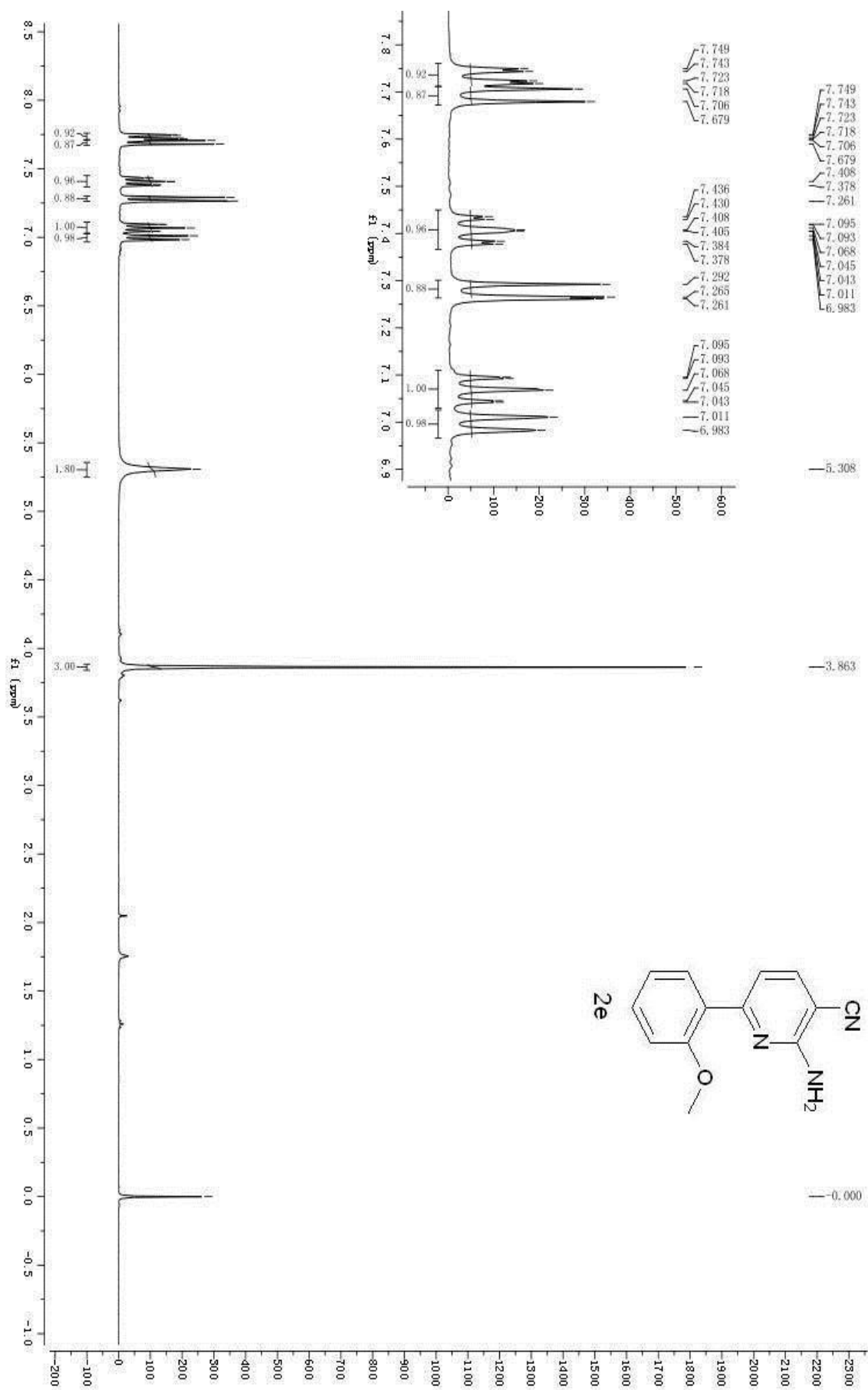
F2 - Acquisition Parameters
 Date_ 20110121
 Time 0.38
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 712
 DS 2
 SH 25062.658 Hz
 FIDRES 1.529703 Hz
 AQ 0.3289108 sec
 RG 8192
 DM 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

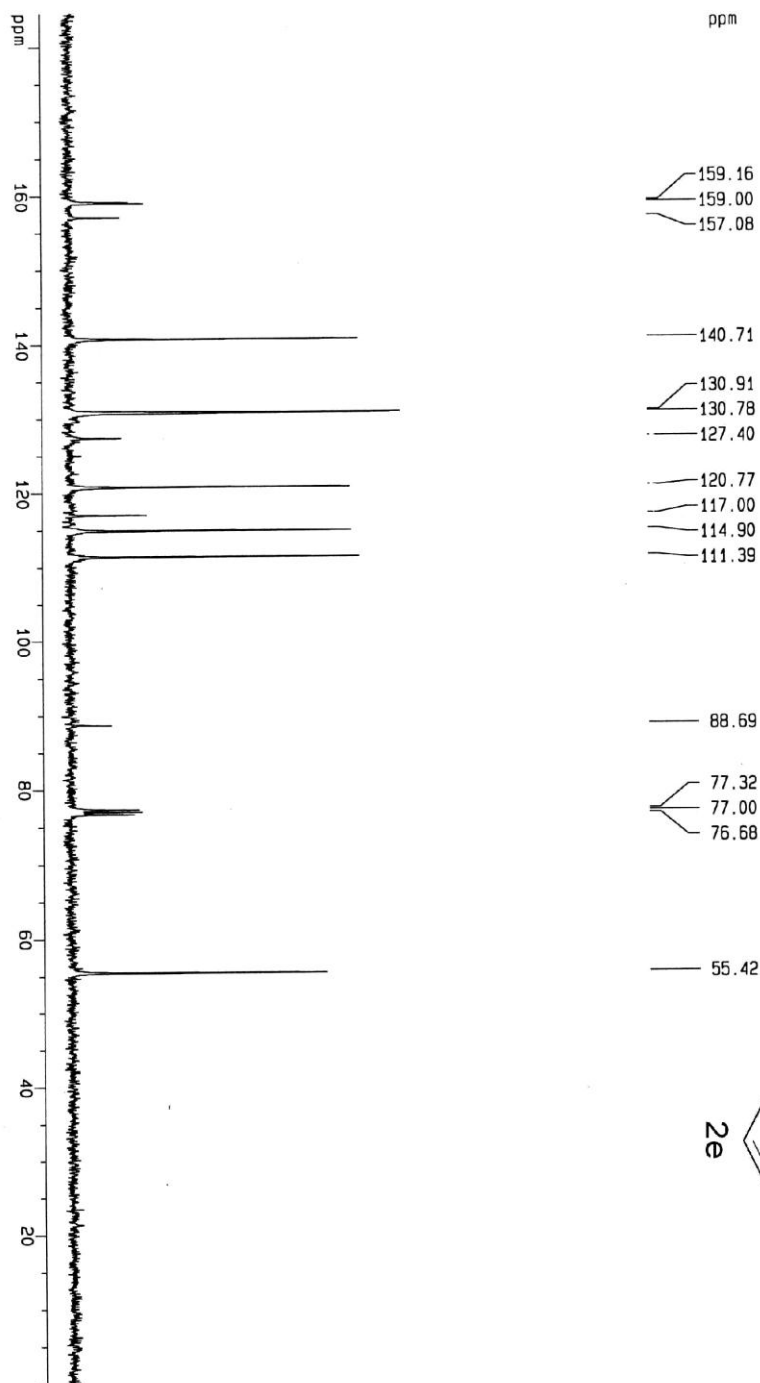
***** CHANNEL f1 *****
 NUC1 13C
 P1 2.70 usec
 PL1 -3.00 dB
 SF01 100.578294 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 17.37 dB
 SF02 399.9515998 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5675148 MHz
 MOW EM
 MSB 0
 LB 4.00 Hz
 BB 0
 PC 0.50

1D NMR plot parameters
 CX 22.00 cm
 CY 8.00 cm
 F1p 192.487 ppm
 F1 19357.98 Hz
 F2p -1.524 ppm
 F2 -153.23 Hz
 PPMCM 8.81869 ppm/cm
 HZCM 886.87329 Hz/cm





Current Data Parameters
 NAME XXQ
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110120
 Time 21.33
 INSTRUM 4000
 PROBNM 5 mm QNP 1H/13
 PULPROG zgpg
 TD 16384
 SOLVENT CDCl3
 NS 102
 DS 0
 SMH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 8192
 DM 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 D11 0.03000000 sec

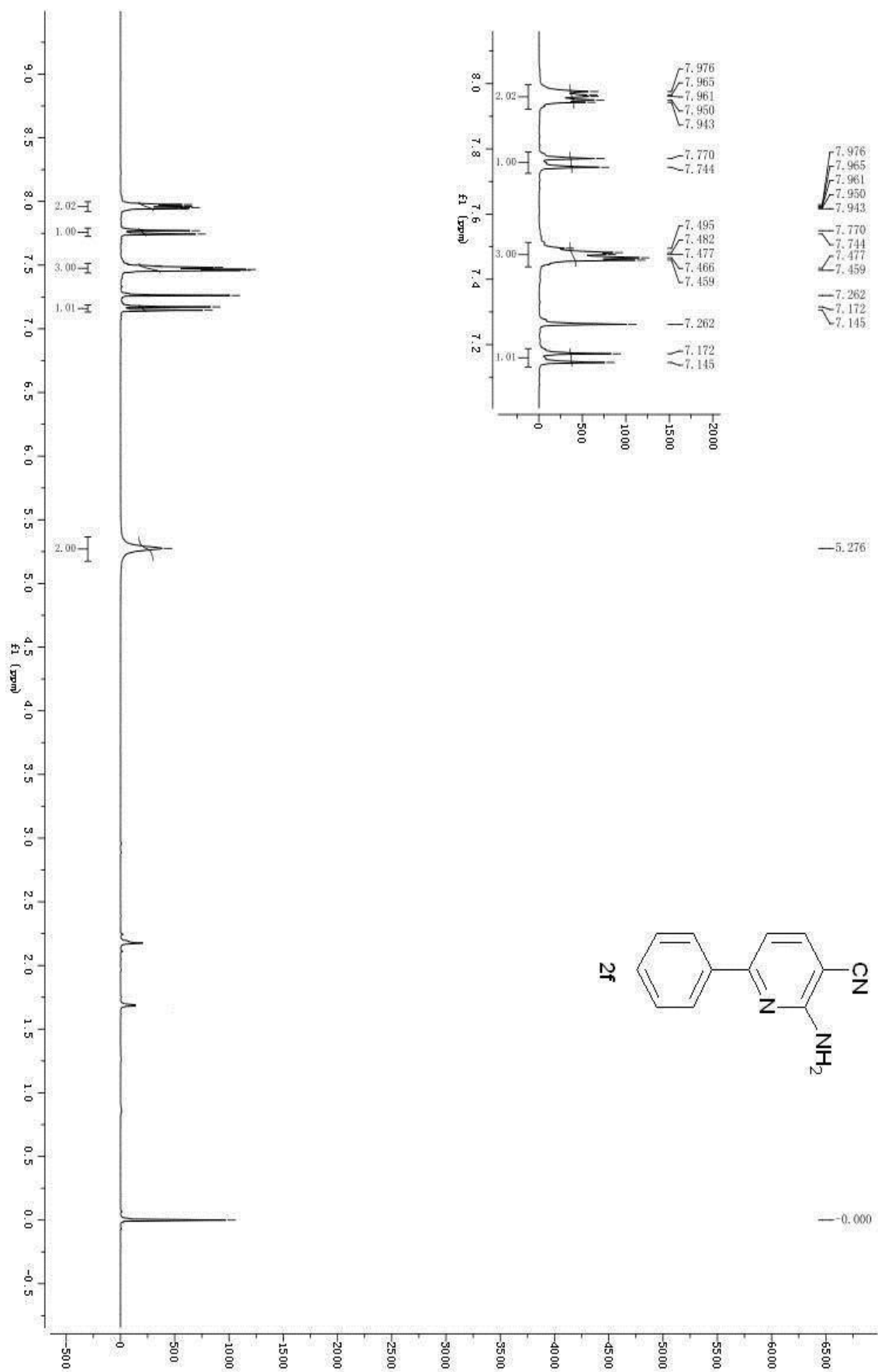
===== CHANNEL f1 =====
 NUC1 13C
 P1 2.70 usec
 PL1 -3.00 dB
 SFO1 100.5785294 MHz

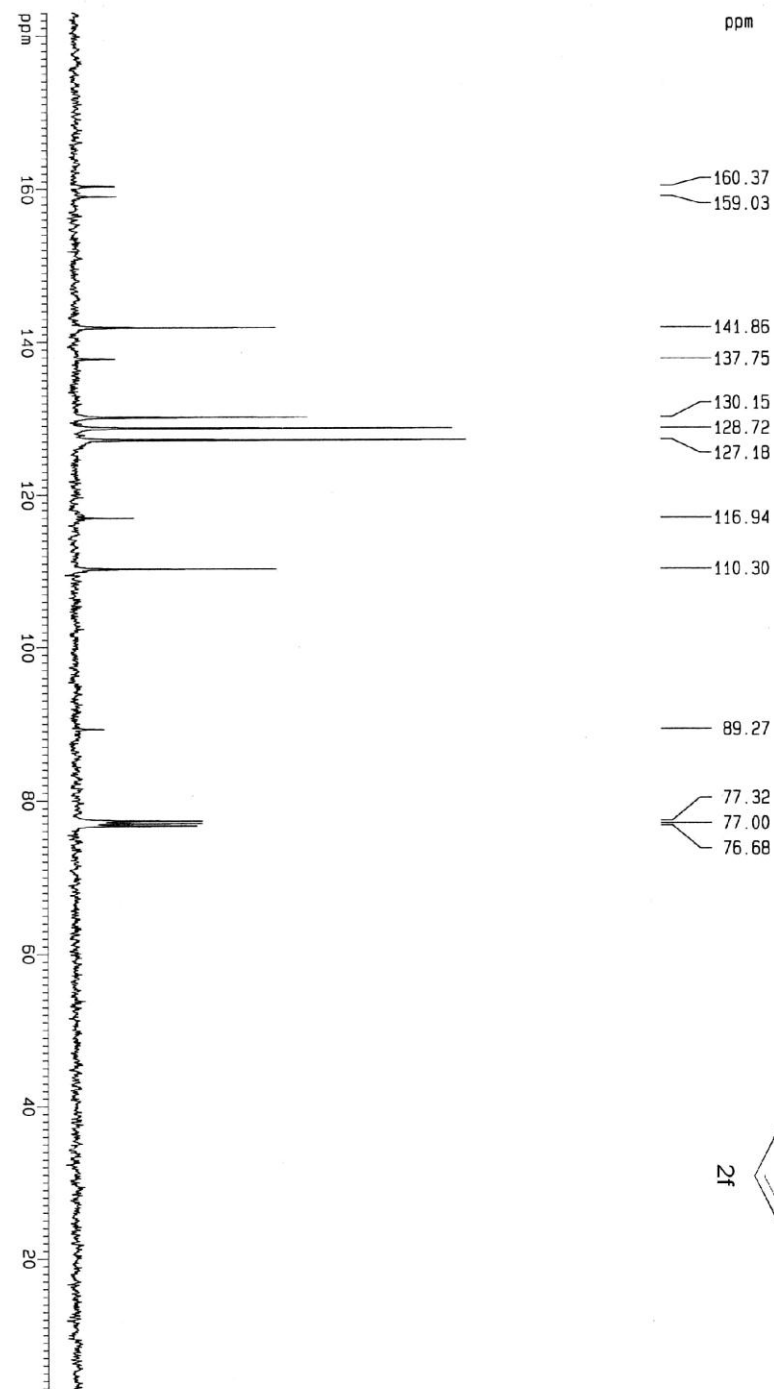
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 17.37 dB
 SFO2 399.9515998 MHz

F2 - Processing parameters
 S1 65536
 SF 100.5675275 MHz
 NQW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 0.90

1D NMR Plot parameters
 CX 22.00 cm
 CY 6.00 cm
 F1P 184.614 ppm
 F1 18565.21 Hz
 F2P -0.035 ppm
 F2 -3.53 Hz
 PPMCM 8.39316 ppm/cm
 HZCM 844.07916 Hz/cm

xxq1-20-2





xxxxb

Current Data Parameters

NAME	XXB
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20101220
Time	23.19
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg
TD	16384
SOLVENT	CDCl3
NS	168
DS	0
SMH	25062.656 Hz
FIDRES	1.529703 Hz
AQ	0.3269108 sec
RG	8192
DM	19.950 usec
DE	6.00 usec
TE	300.0 K
D1	0.69999999 sec
d11	0.03000000 sec

===== CHANNEL f1 =====

NUC1	¹³ C
P1	2.80 usec
PL1	-3.00 dB
SFO1	100.5795294 MHz

===== CHANNEL f2 =====

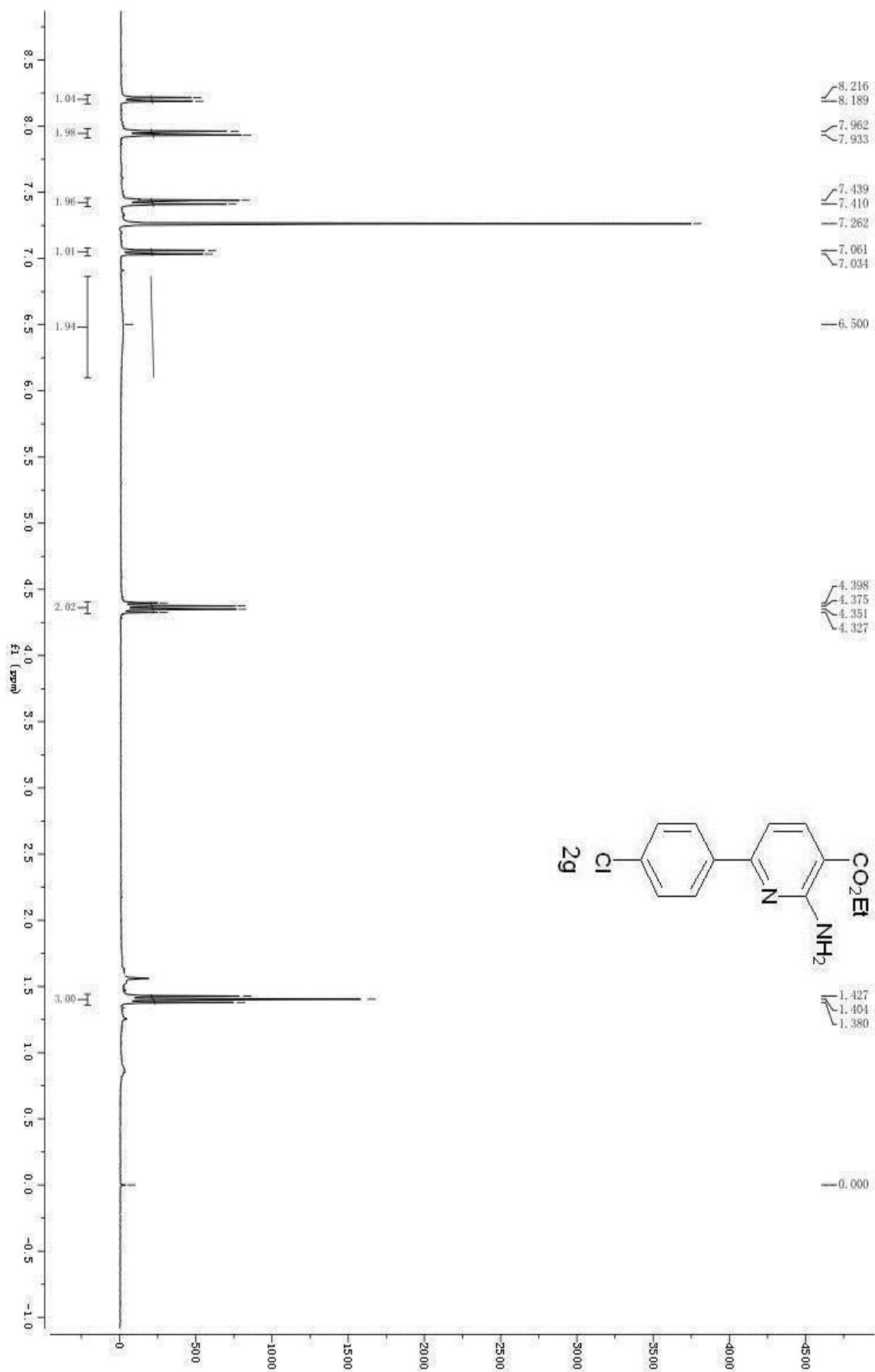
CPDPRG2	waltz16
NUC2	¹ H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	19.50 dB
SFO2	399.9515999 MHz

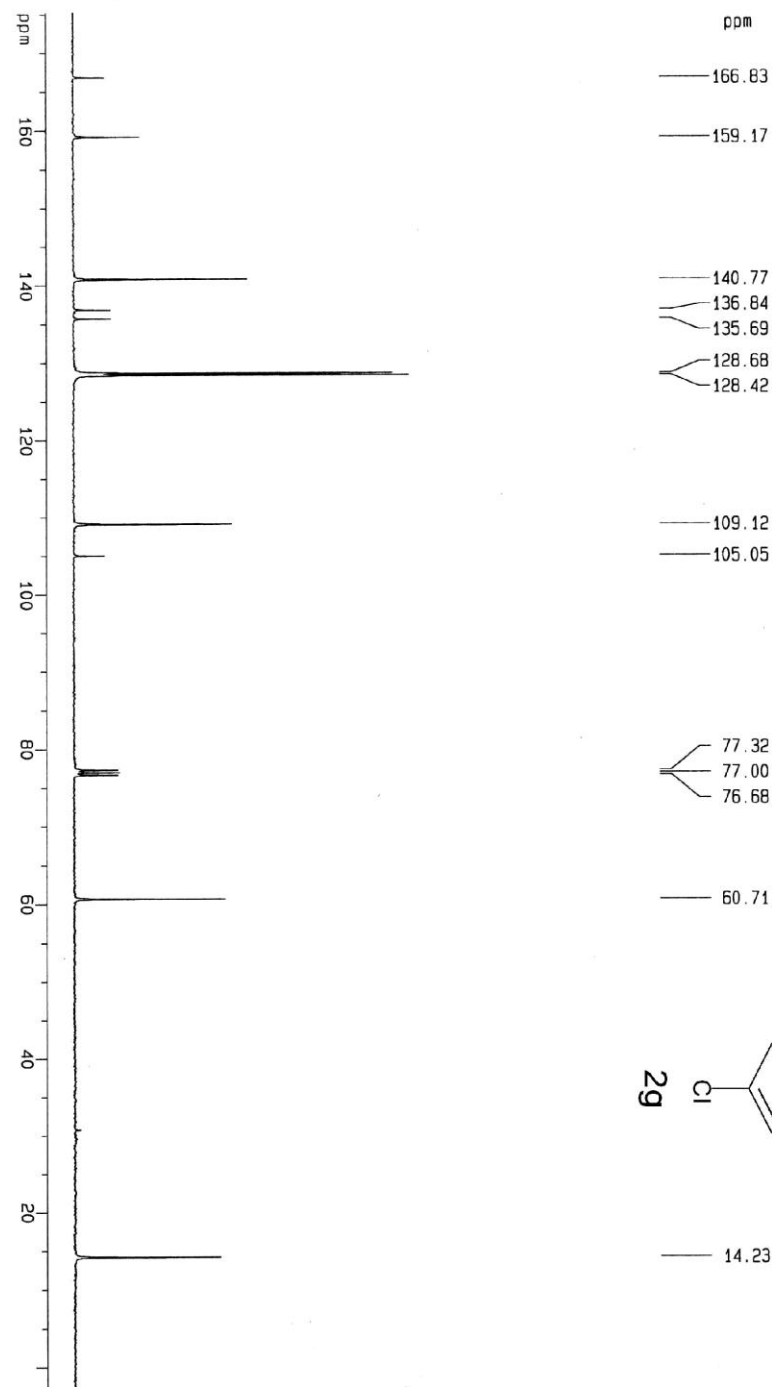
F2 - Processing parameters

SF	65536
SF	100.5675187 MHz
WDW	EM
SSB	0
LB	7.00 Hz
GB	0
PC	1.00

10 NMR plot parameters.

CX	22.00 cm
CY	7.00 cm
F1P	183.056 ppm
F1	18409.50 Hz
F2P	2.788 ppm
F2	280.34 Hz
PPMCM	8.19402 ppm/cm
HZCM	824.05267 Hz/cm





xxxx6

Current Data Parameters
 NAME: XXXD
 EXPNO: 5
 PROCNO: 1

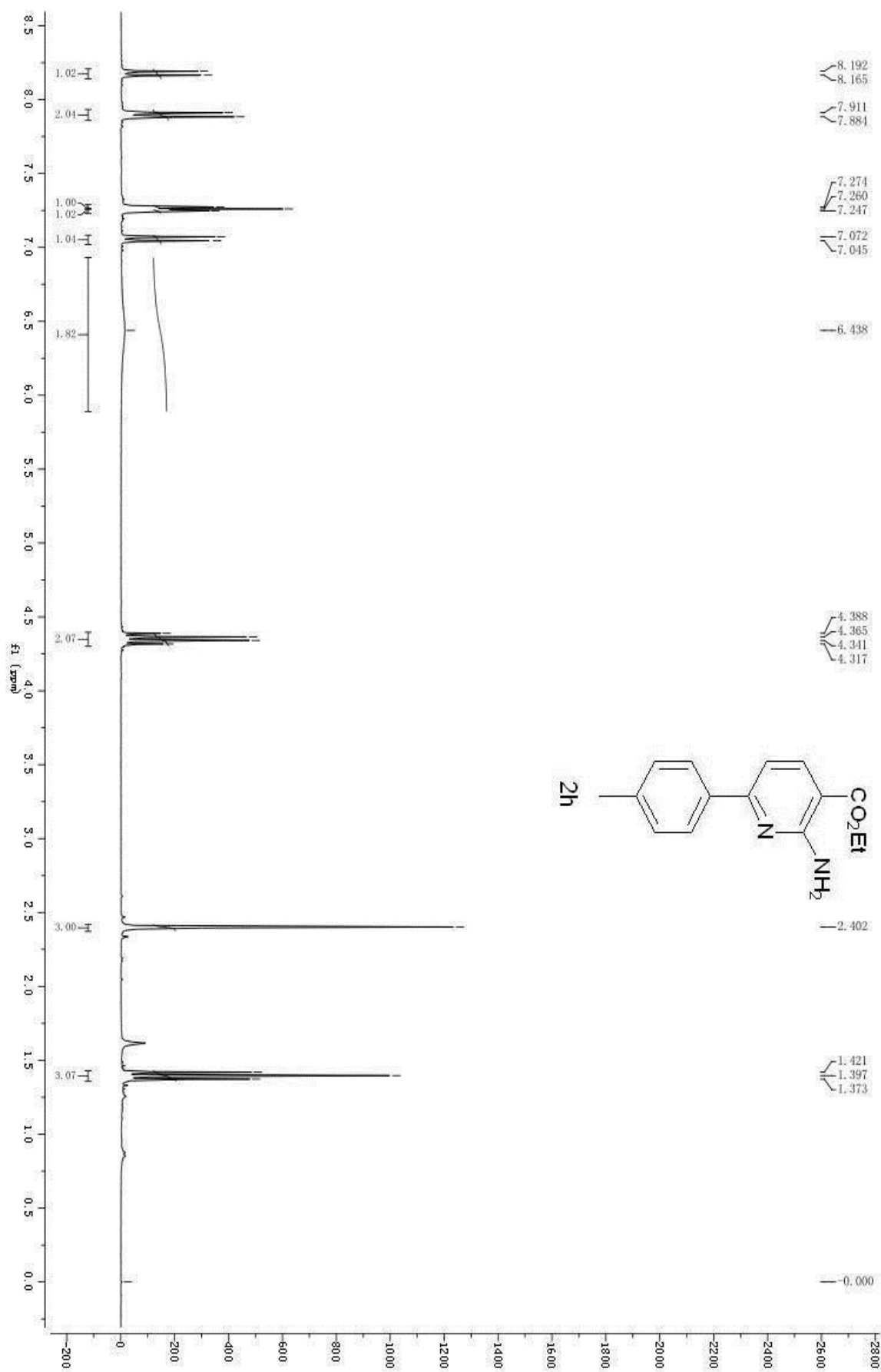
F2 - Acquisition Parameters
 Date_: 20101203
 Time: 15.38
 INSTRUM: AV400
 PROBHD: 5 mm QNP 1H/13
 PULPROG: zgpg
 TD: 16384
 SOLVENT: CDCl3
 NS: 1062
 DS: 0
 SWH: 25062.656 Hz
 FIDRES: 1.529703 Hz
 AQ: 0.3269108 sec
 RG: 8192
 DW: 19.950 usec
 DE: 6.00 usec
 TE: 313.0 K
 D1: 0.69999999 sec
 d11: 0.03000000 sec

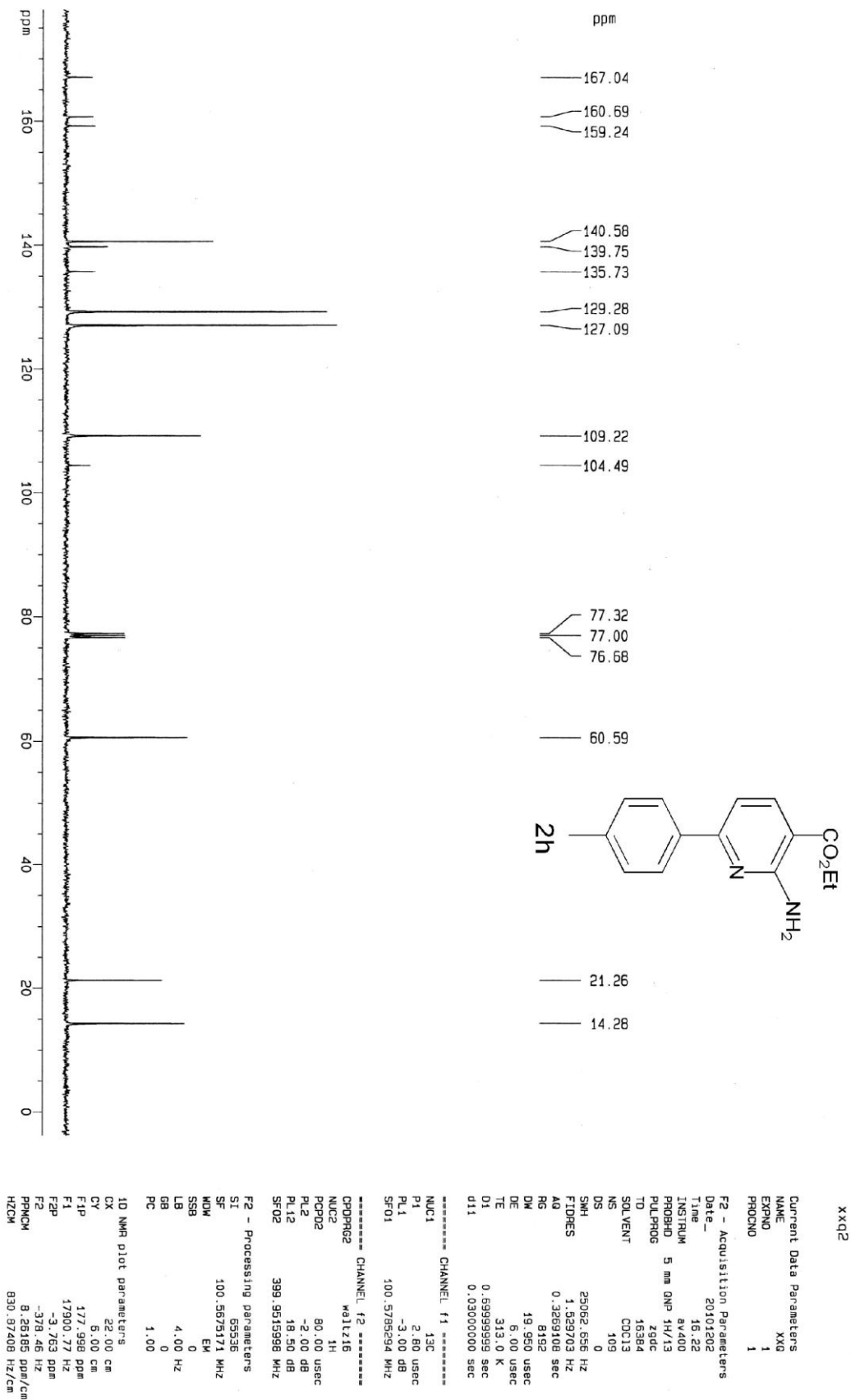
===== CHANNEL f1 =====
 NUC1: 13C
 P1: 2.80 usec
 PL1: -3.00 dB
 SFO1: 100.5785894 MHz

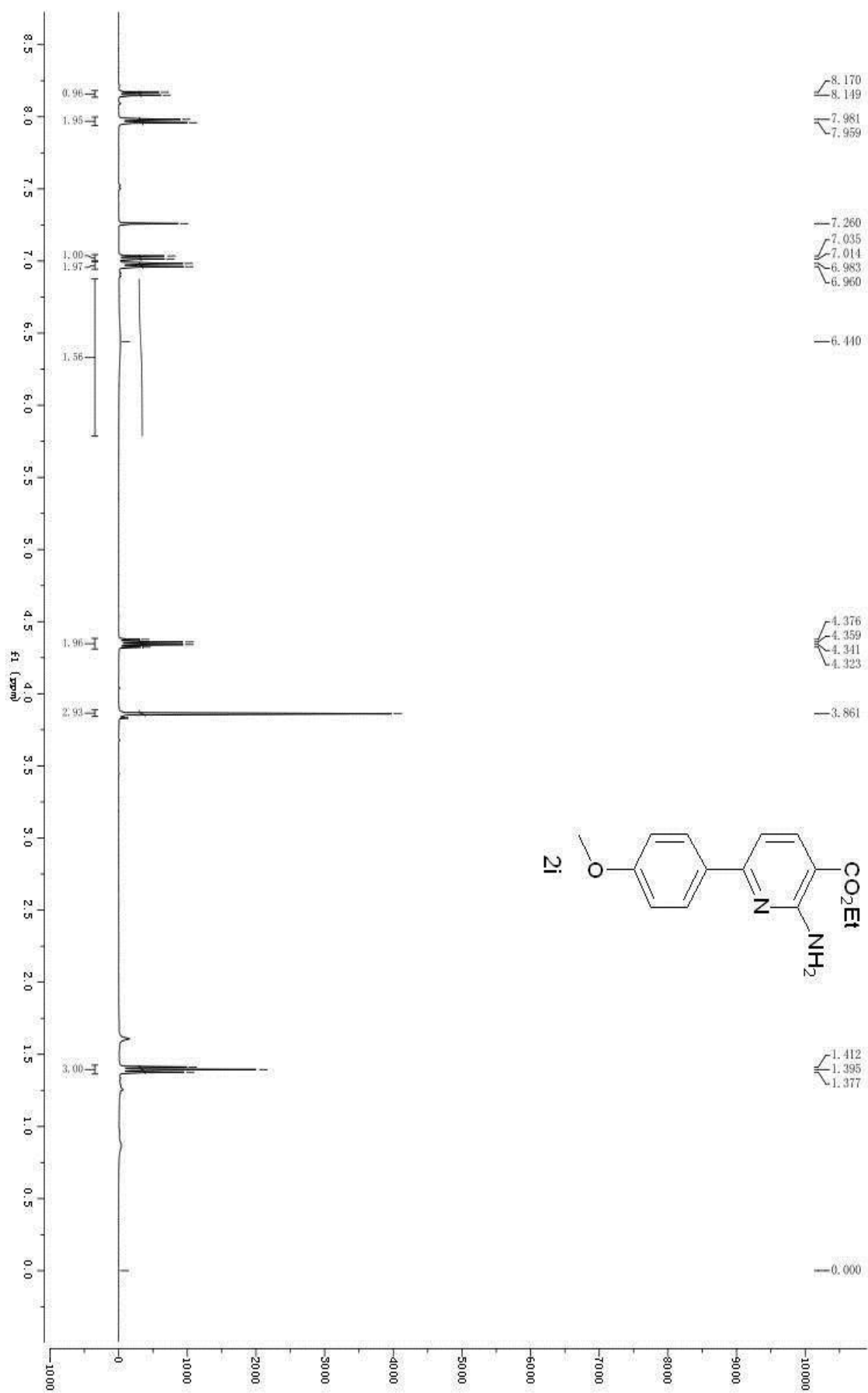
===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 P2: 80.00 usec
 PL2: -2.00 dB
 PL12: 18.50 dB
 SFO2: 399.9519998 MHz

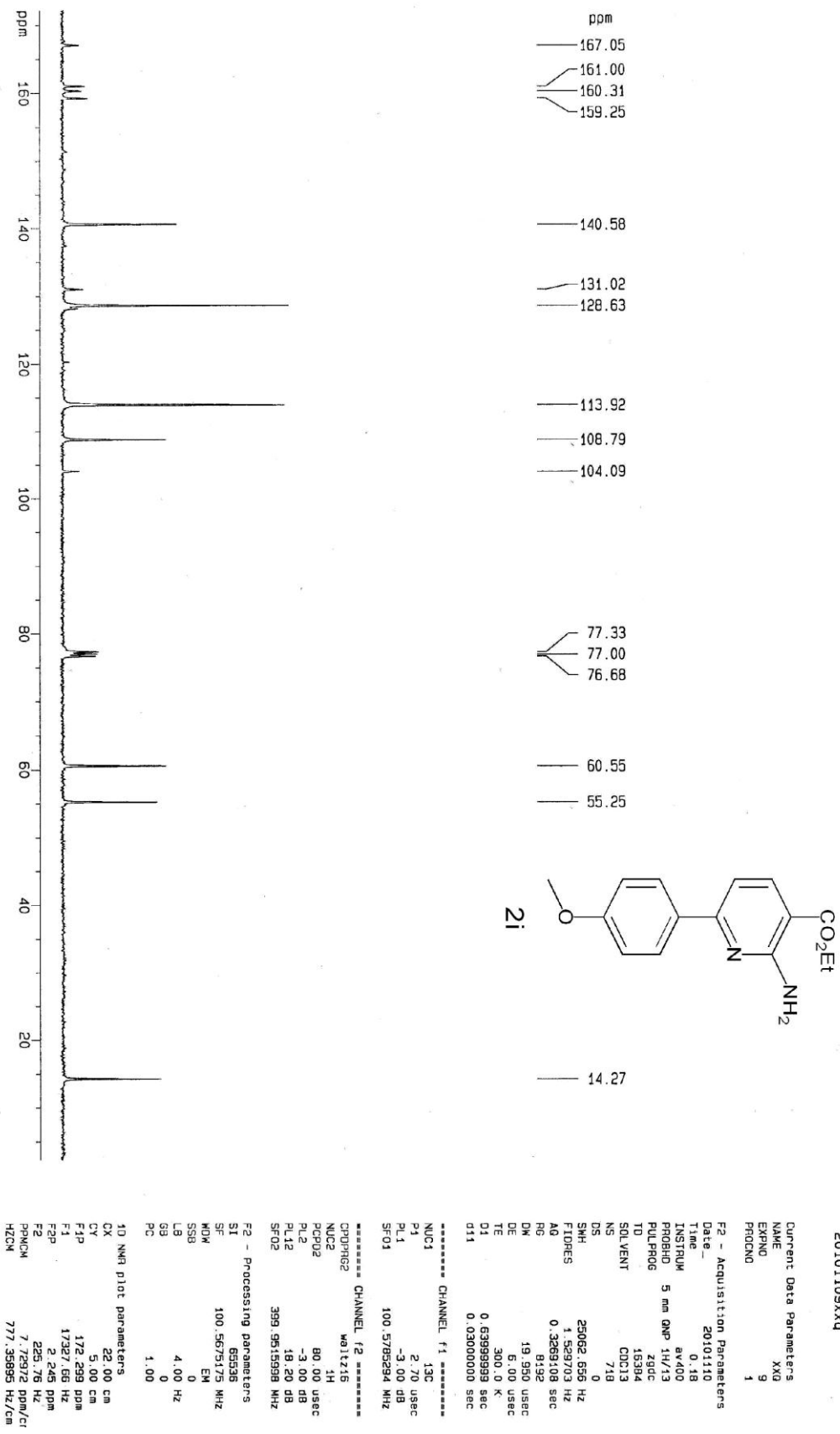
F2 - Processing parameters
 SI: 65536
 SF: 100.5675179 MHz
 KDN: EM
 SSB: 0
 LB: 4.00 Hz
 GB: 0
 PC: 1.00

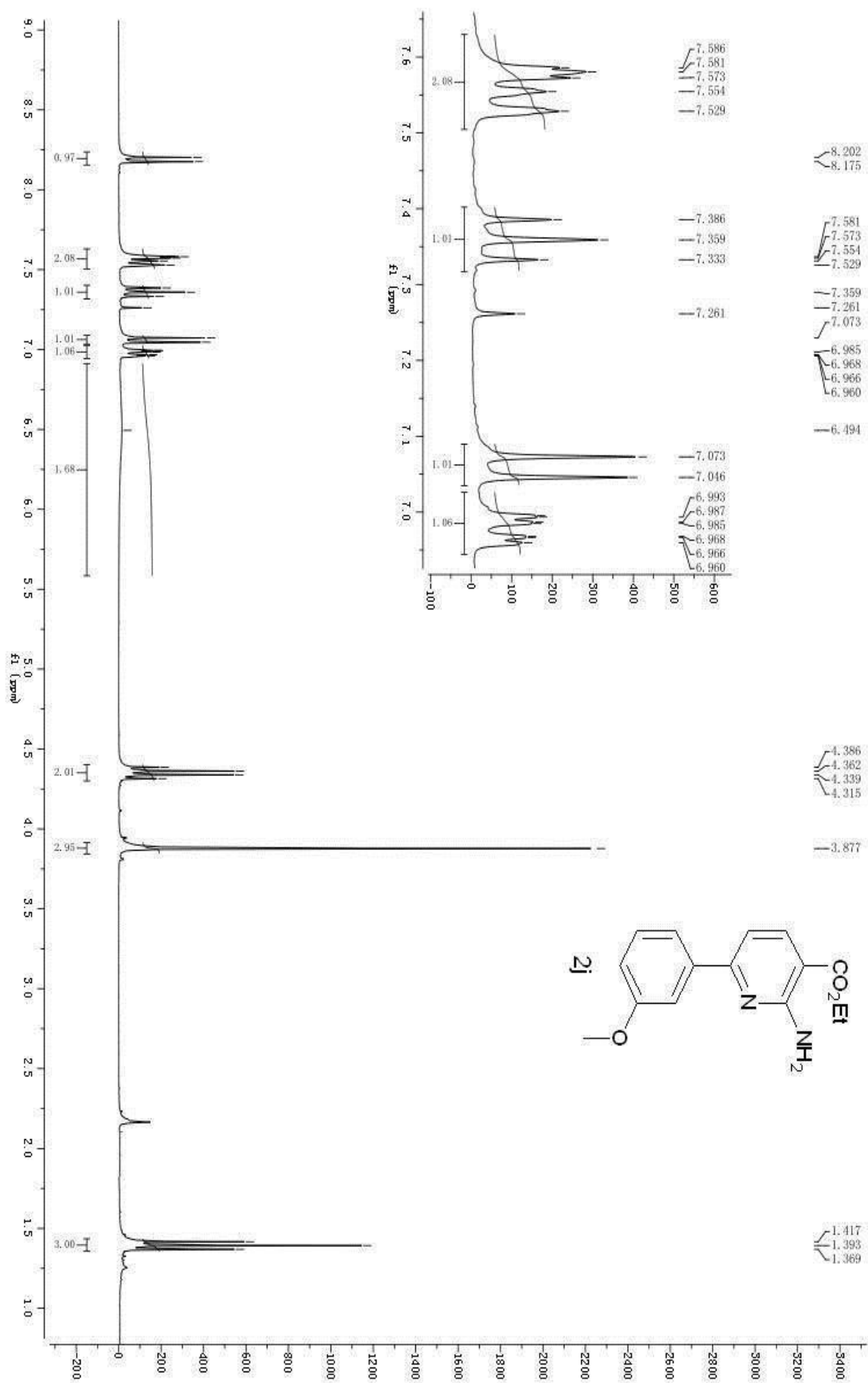
1D NMR plot parameters
 CX: 22.00 cm
 CY: 6.00 cm
 F1P: 179.304 ppm
 F1: 17629.87 Hz
 F2P: -2.875 ppm
 F2: -289.18 Hz
 PPMCM: 8.09906 ppm/cm
 HZCM: 814.50226 Hz/cm

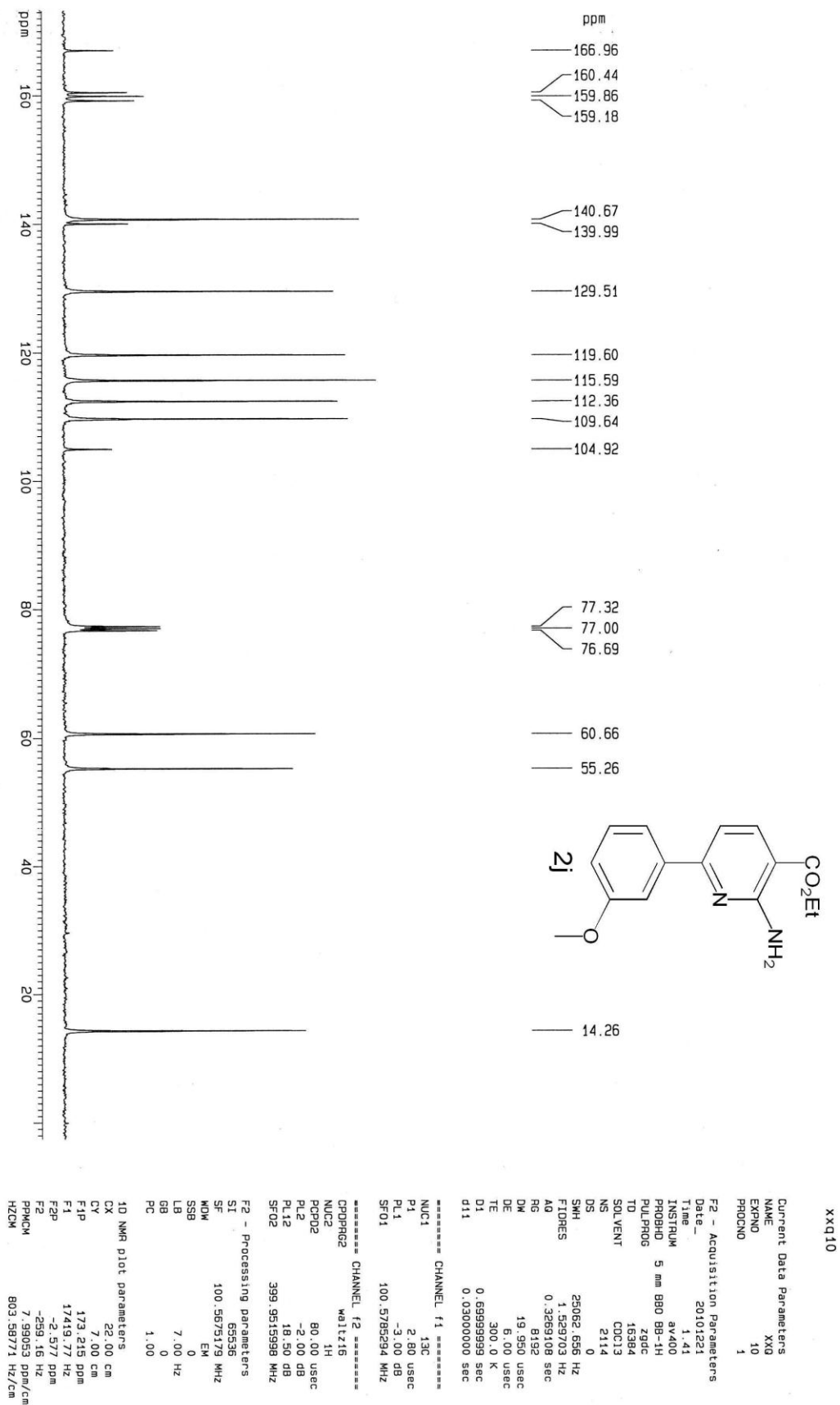


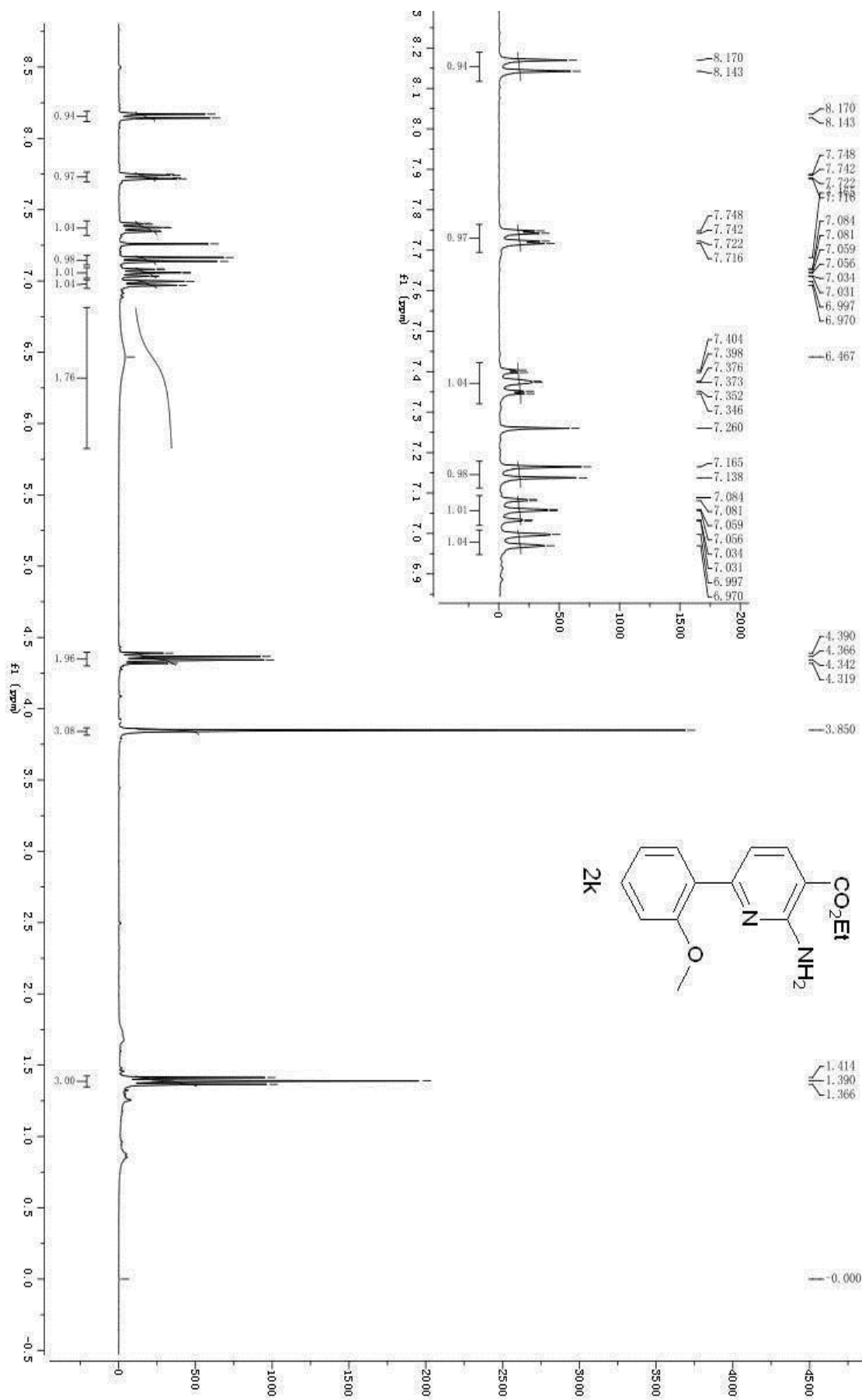


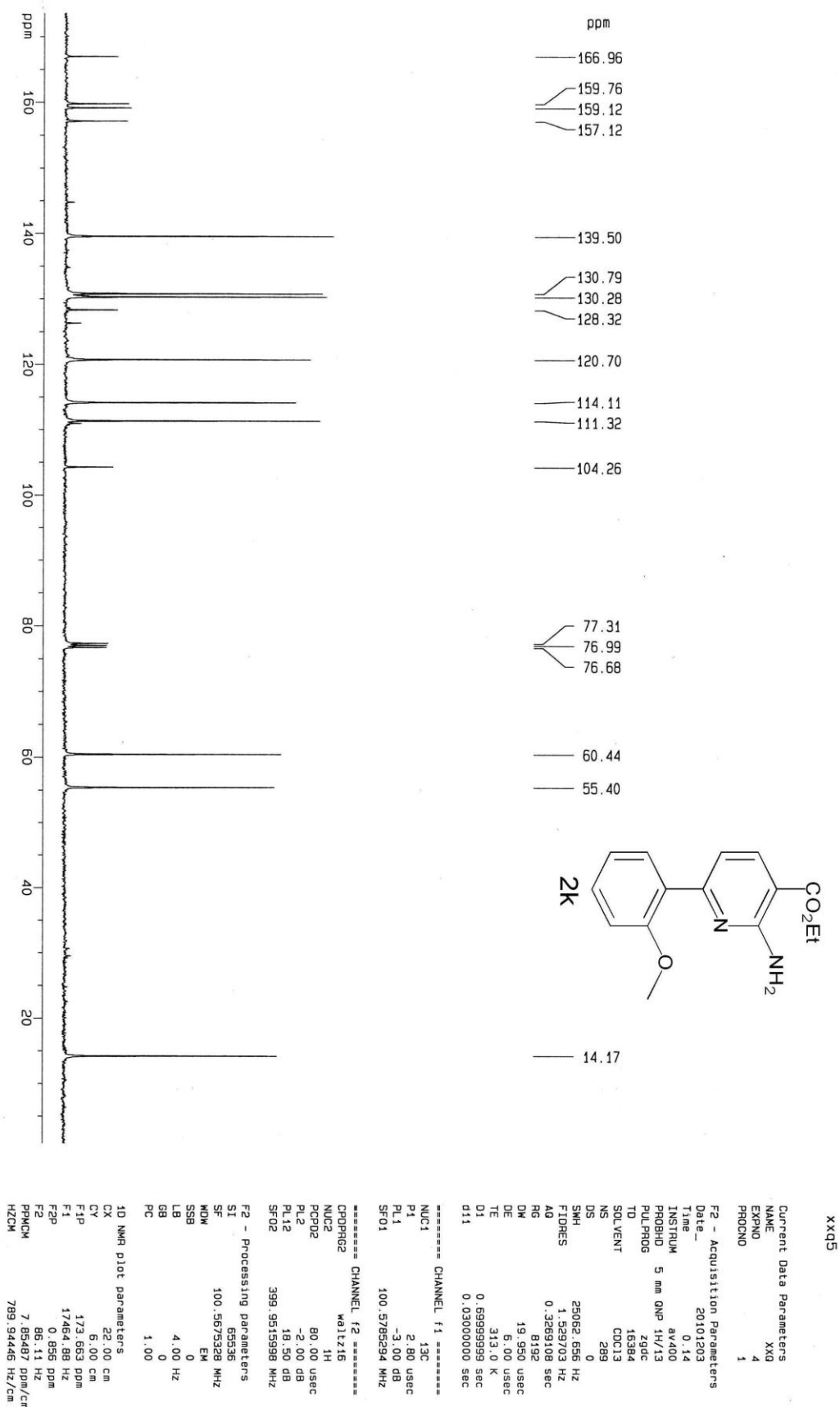


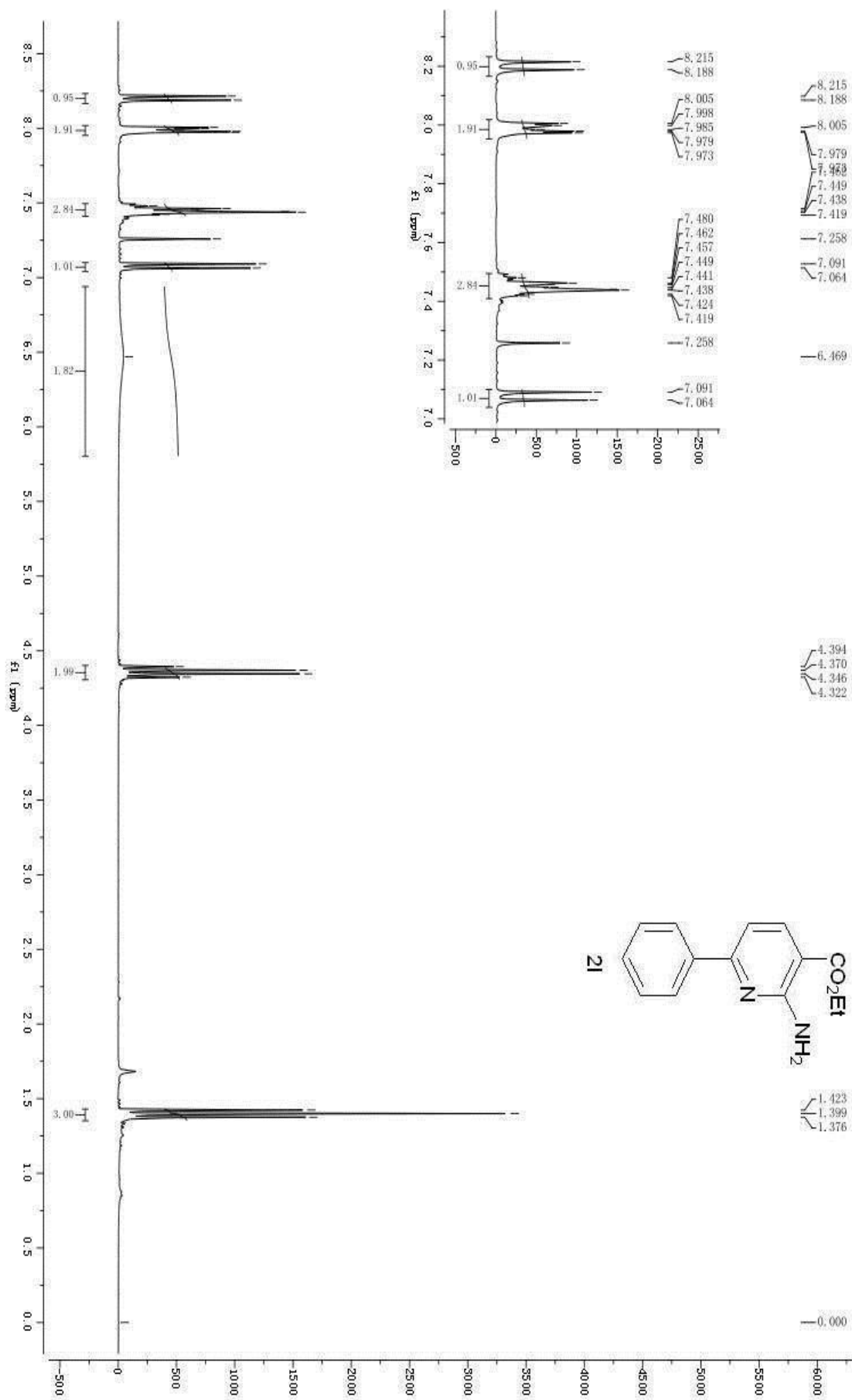


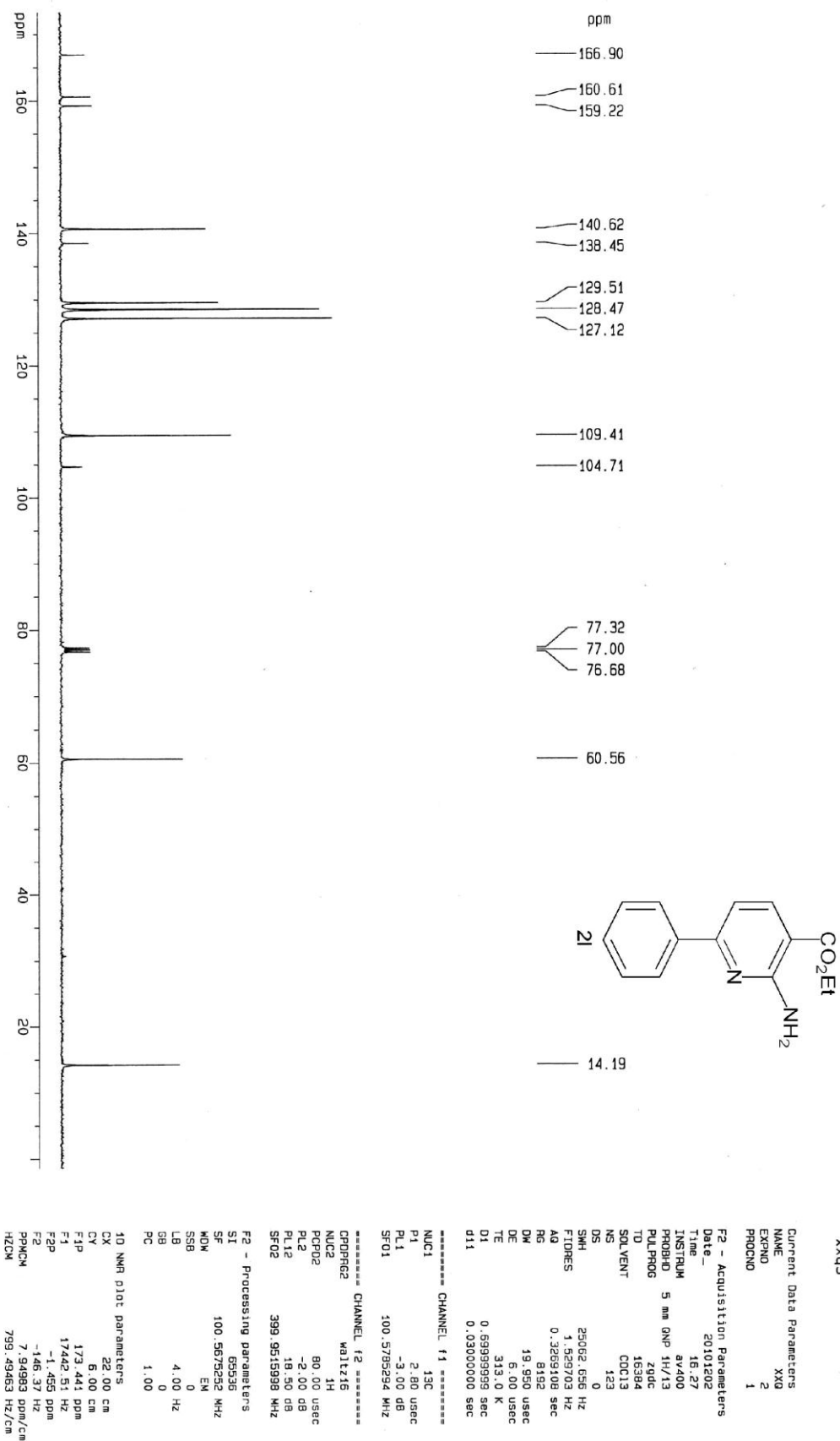


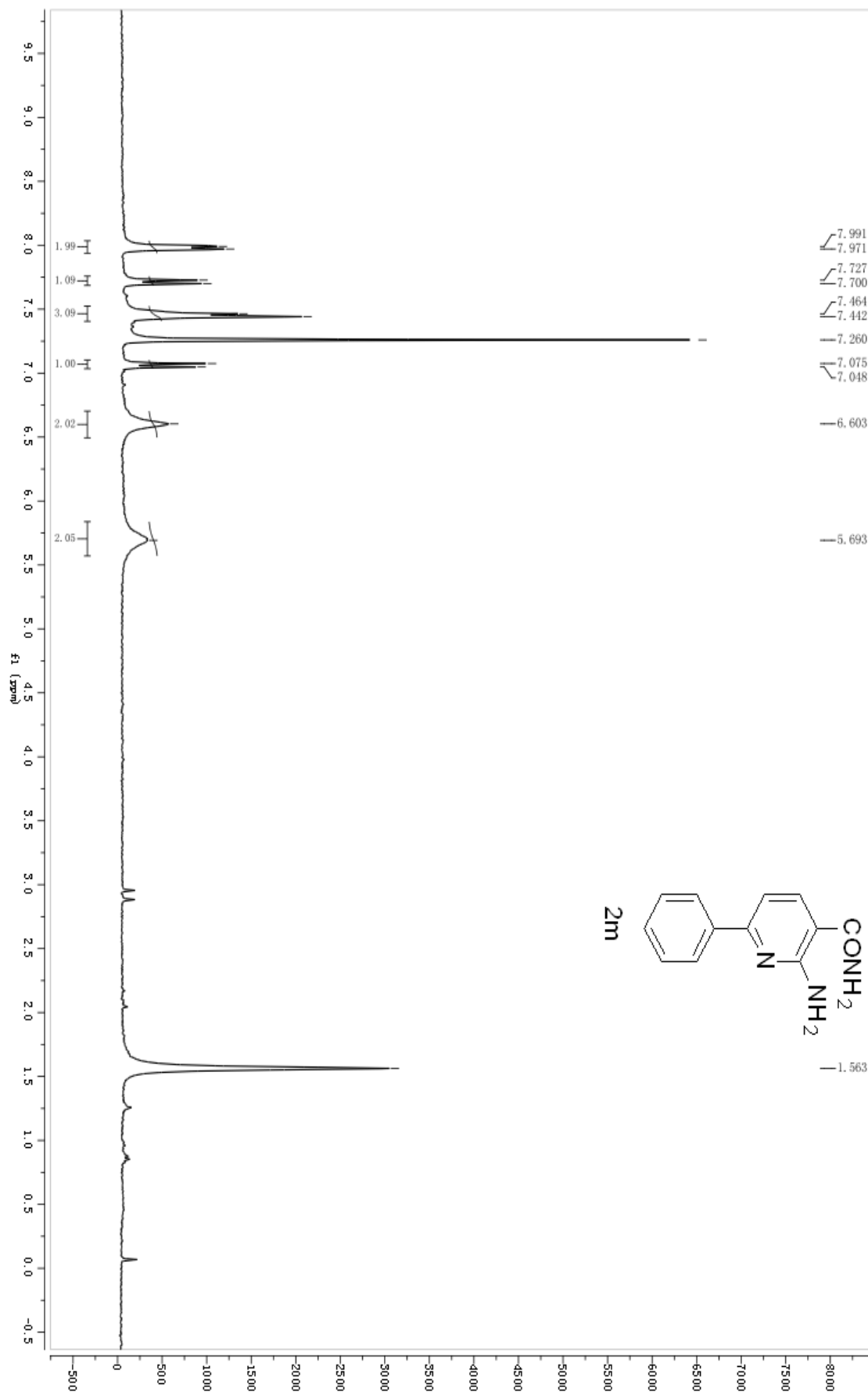


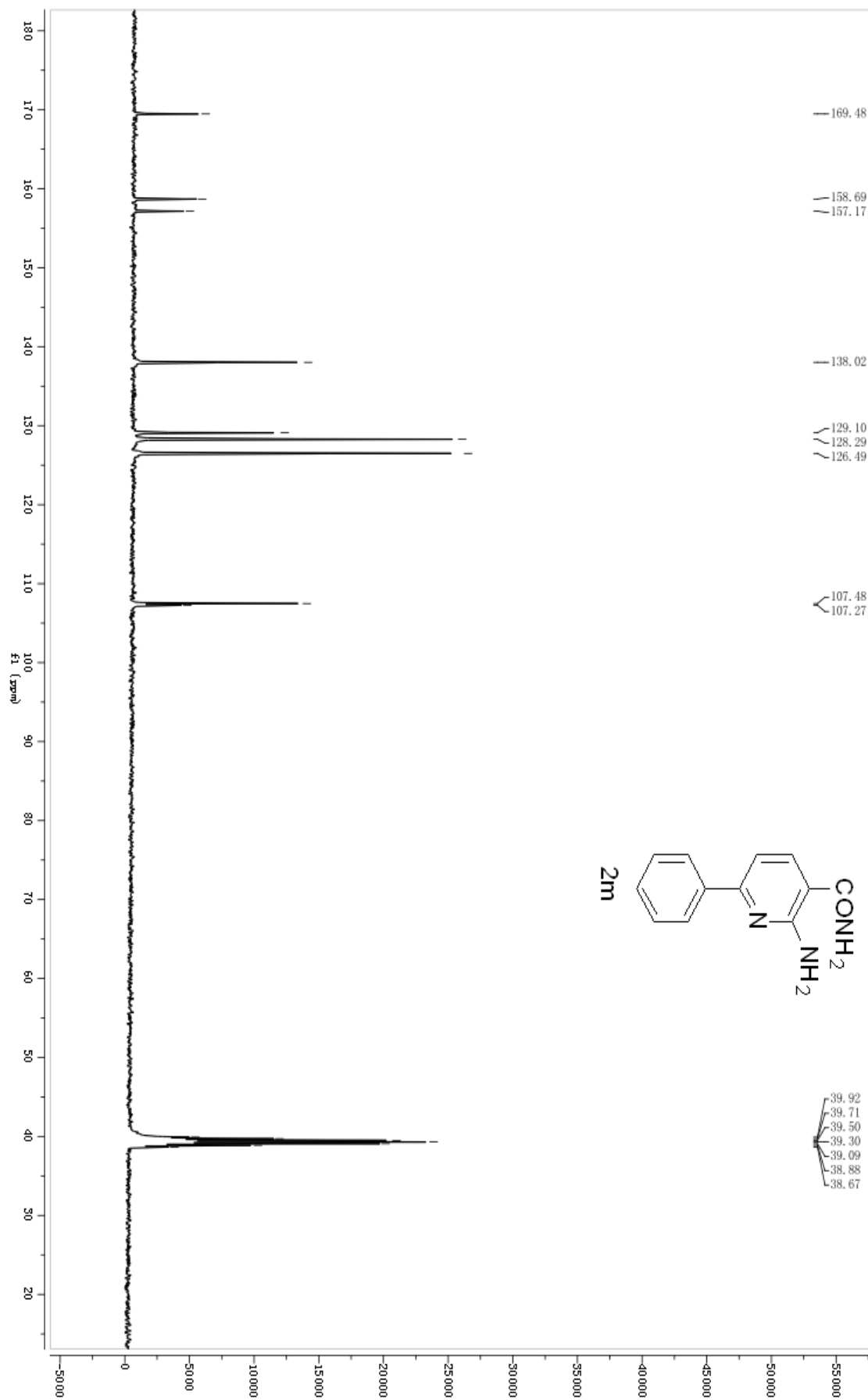


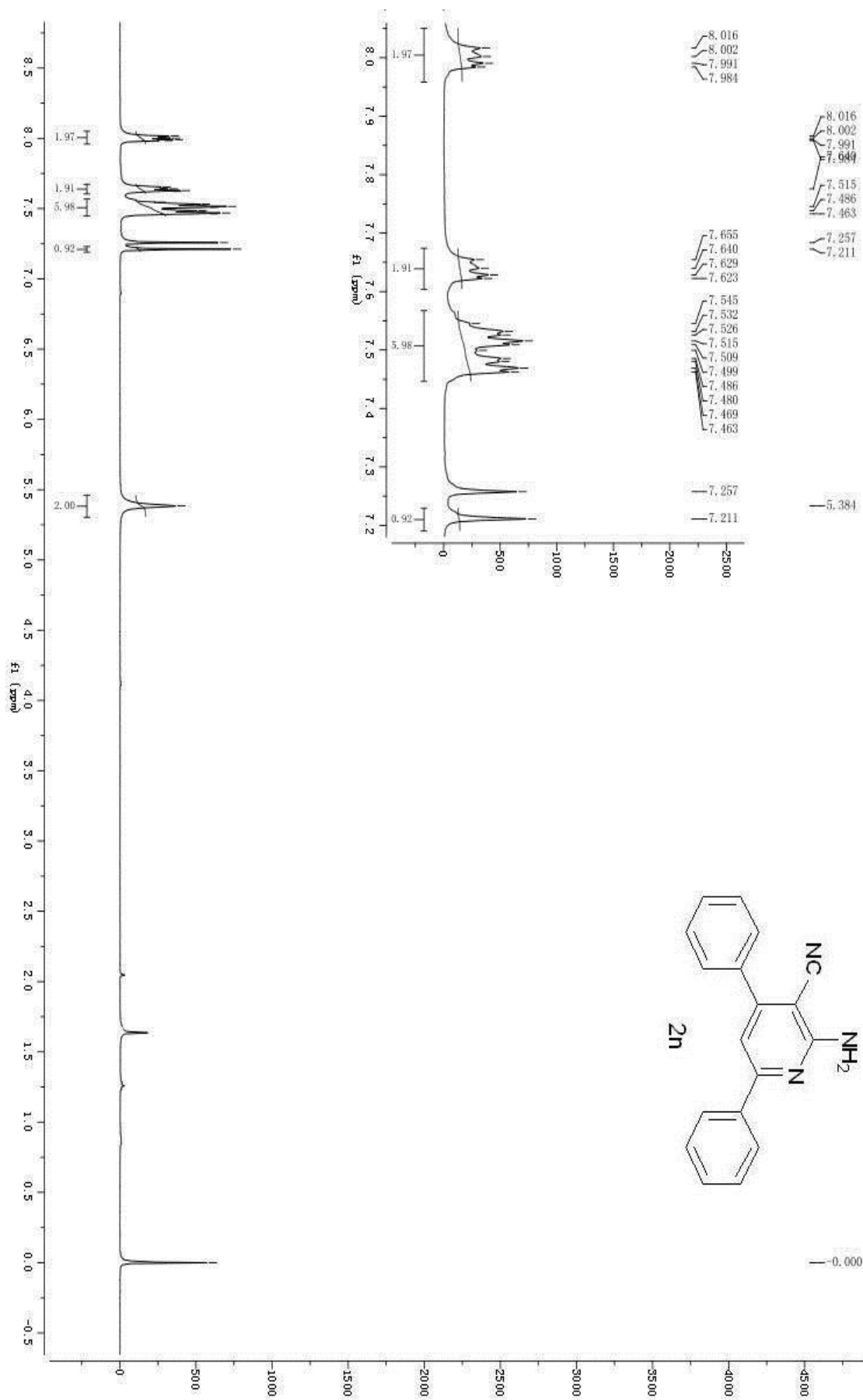


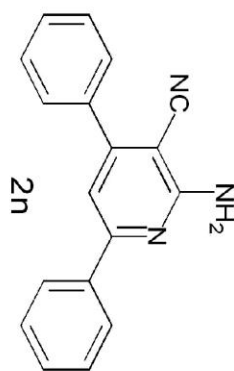
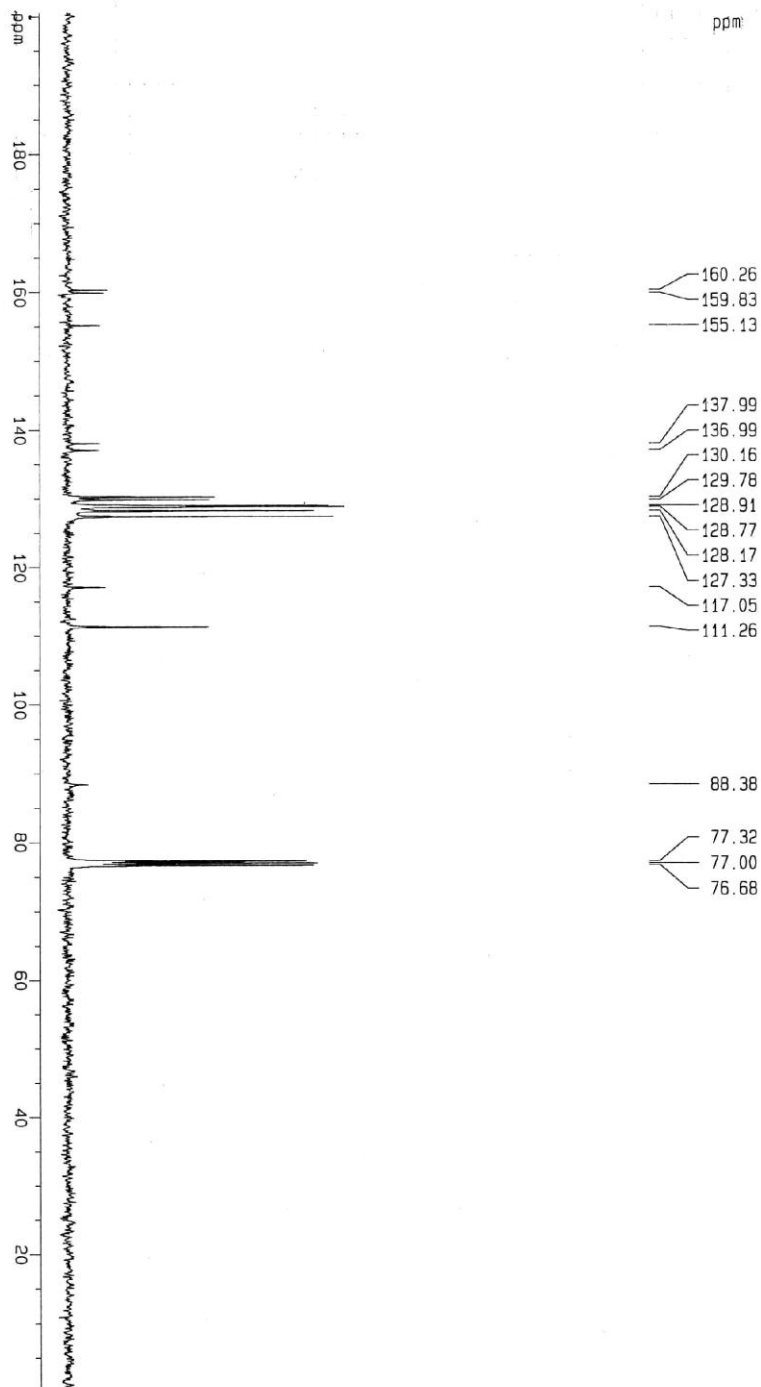












XXQ1-15

Current Data Parameters

NAME	XXQ
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20110116
Time	14.48
INSTRUM	AV400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg
TD	16384
SOLVENT	CDCl3
NS	628
DS	0
SWH	25062.656 Hz
FTIDRES	1.529703 Hz
AD	0.3269108 sec
RG	8192
DM	19.950 usec
DE	5.00 usec
TE	303.0 K
D1	0.69999999 sec
D11	0.03000000 sec

===== CHANNEL f1 =====

NUC1	13C
P1	2.70 usec
PL1	-3.00 dB
SFO1	100.5785294 MHz

===== CHANNEL f2 =====

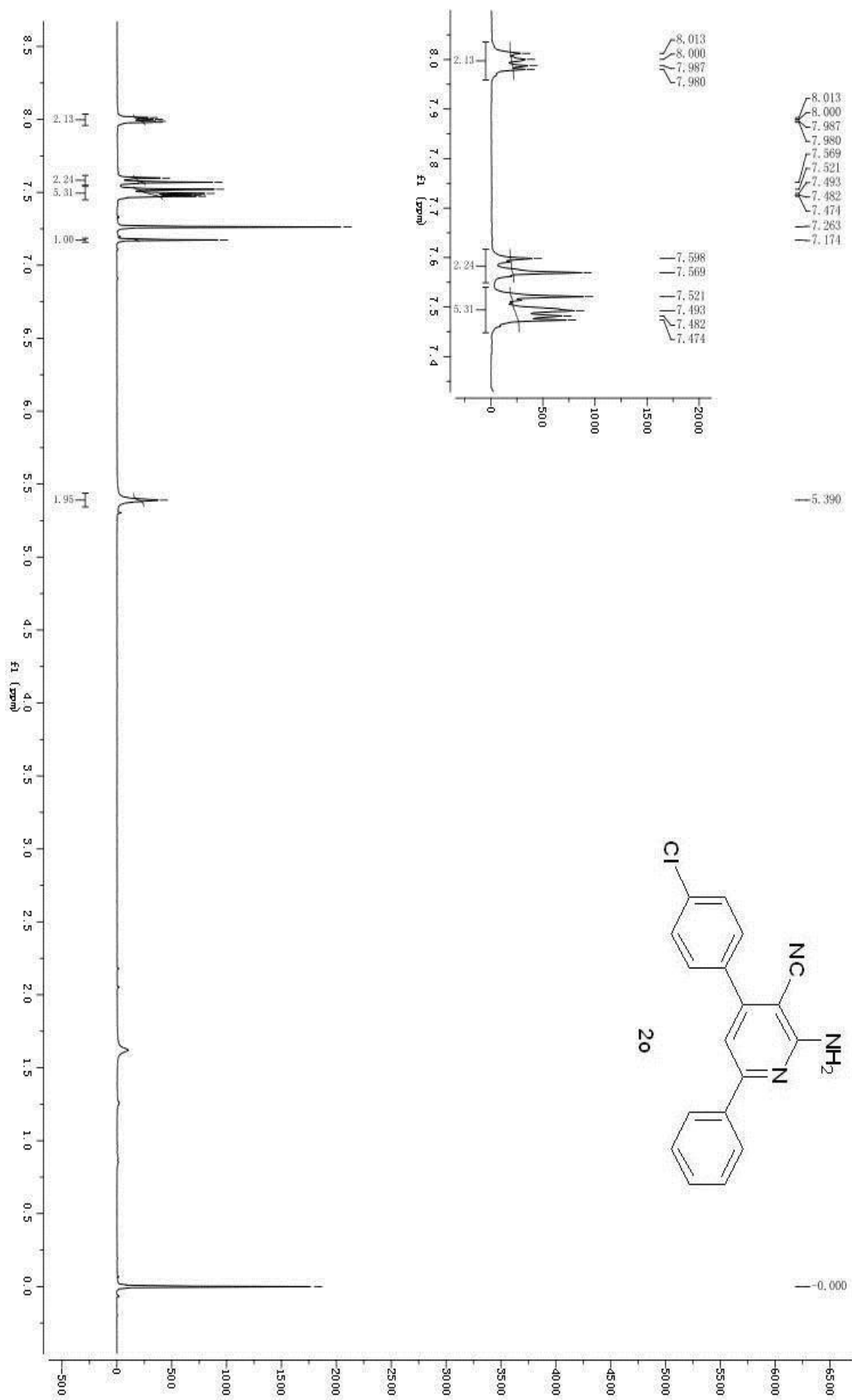
CPDPRG2	WALTZ16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.00 dB
PL12	18.20 dB
SFO2	399.9515999 MHz

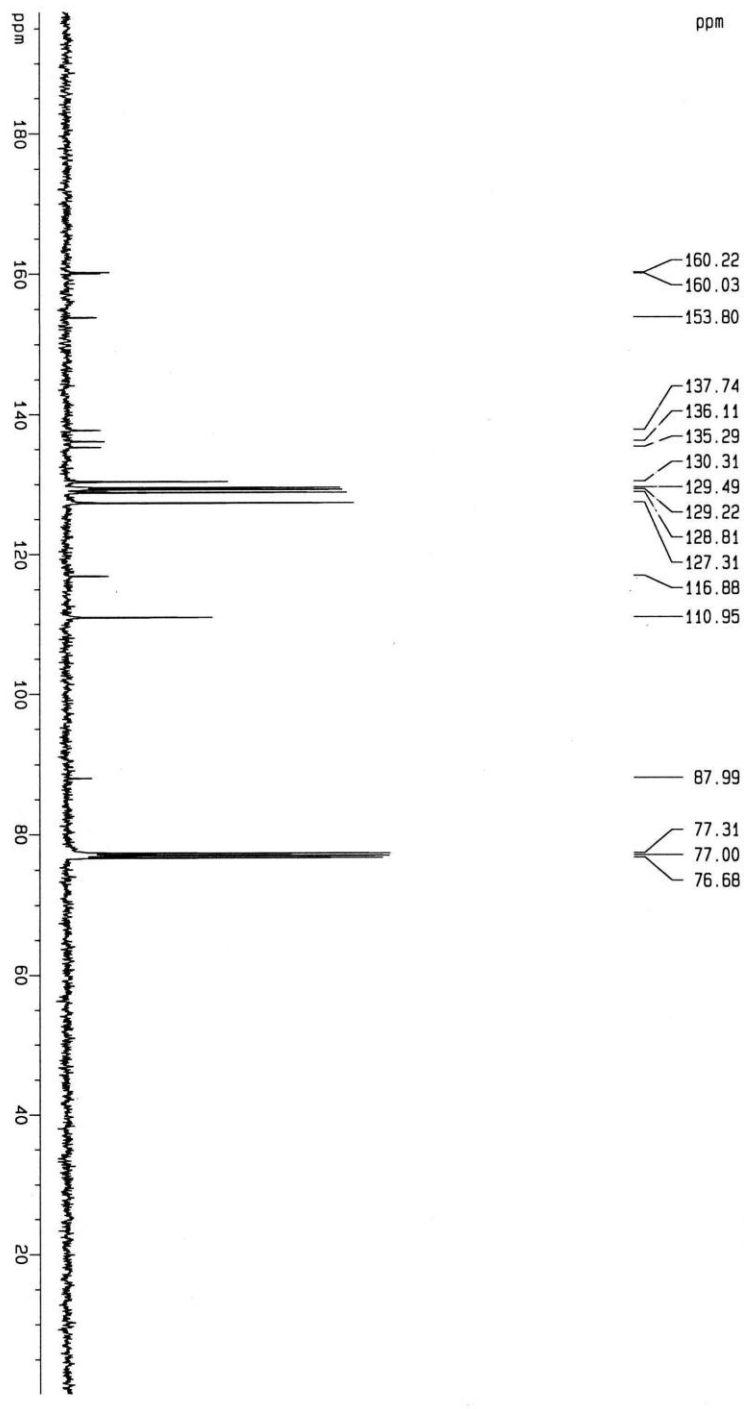
F2 - Processing parameters

SI	65536
SF	100.5675087 MHz
WDW	EM
SSB	0
LB	7.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	5.00 cm
F1P	200.619 ppm
F2P	20.7572 Hz
F2	0.797 ppm
PPMCM	9.08261 ppm/cm
HZCM	913.43530 Hz/cm





xxq1-19C

Current Data Parameters
 NAME: XXQ
 EXPNO: 3
 PROCNO: 1

F2 - Acquisition Parameters

Date_: 20110120
 Time: 21:27
 INSTRUM: av400
 PROBR0: 5 mm QNP 1H/13
 PULPROG: zgpg
 TD: 16384
 SOLVENT: CDCl3
 NS: 463
 DS: 0
 SMH: 25062.656 Hz
 FIDRES: 1.529703 Hz
 AQ: 0.3269108 sec
 RG: 8192
 DM: 19.950 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 0.6999999 sec
 d11: 0.0300000 sec

----- CHANNEL f1 -----
 NUC1: 13C
 P1: 2.70 usec
 PL1: -3.00 dB
 SF01: 100.5785294 MHz

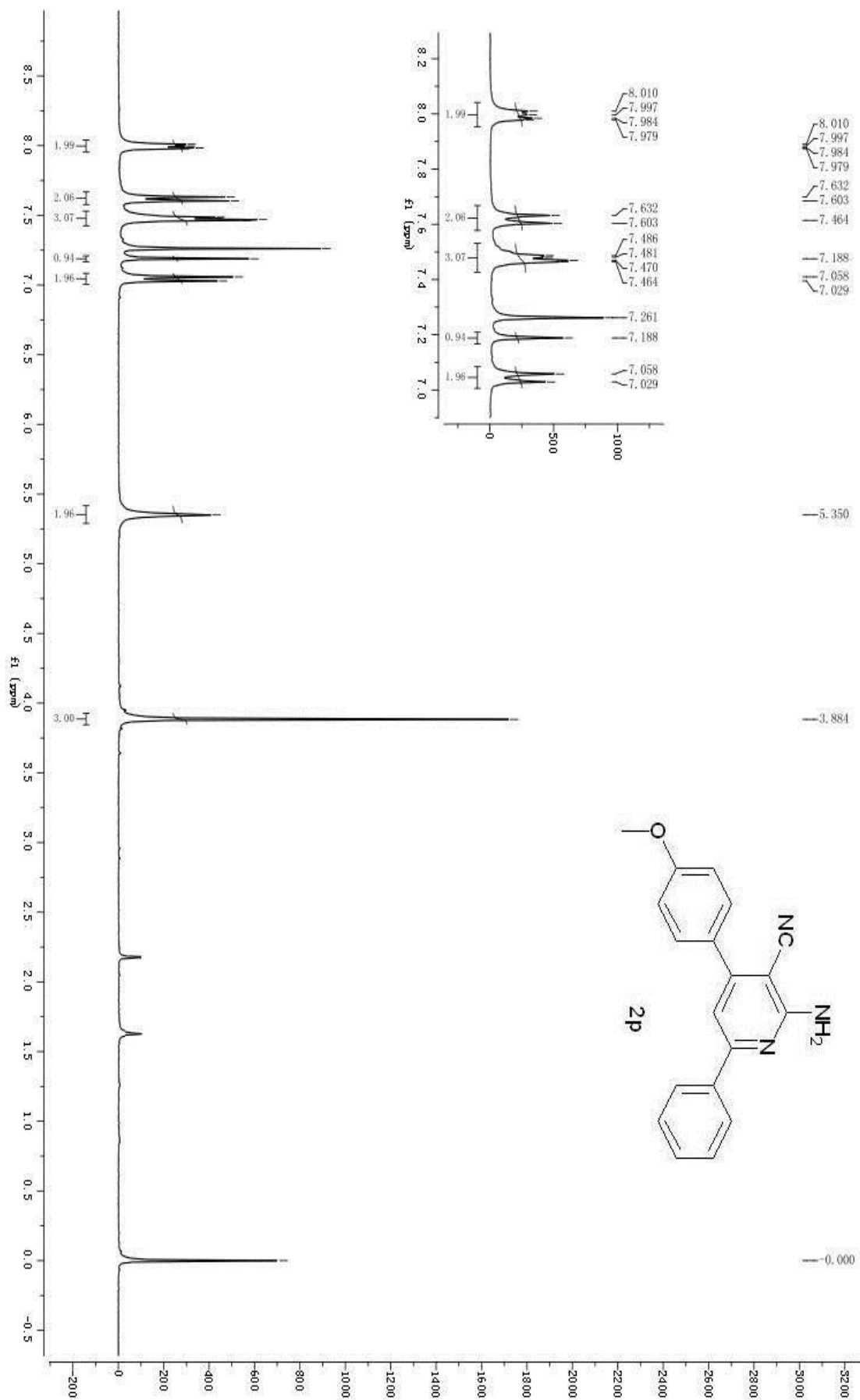
----- CHANNEL f2 -----
 CPDPRG2: waltz16
 NUC2: 1H
 PCPD2: 80.00 usec
 PL2: -3.00 dB
 PL12: 17.37 dB
 SF02: 399.9515998 MHz

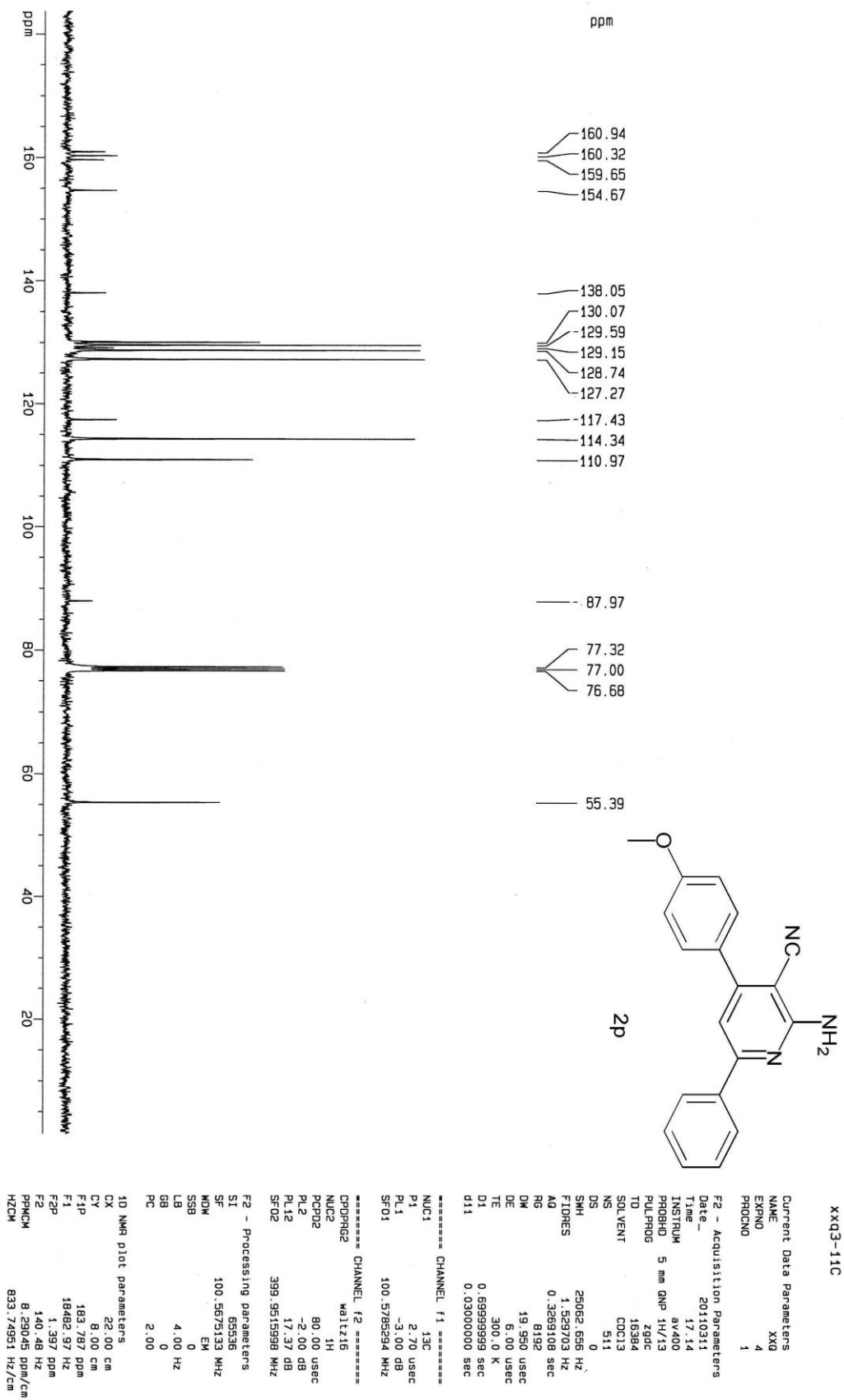
F2 - Processing parameters

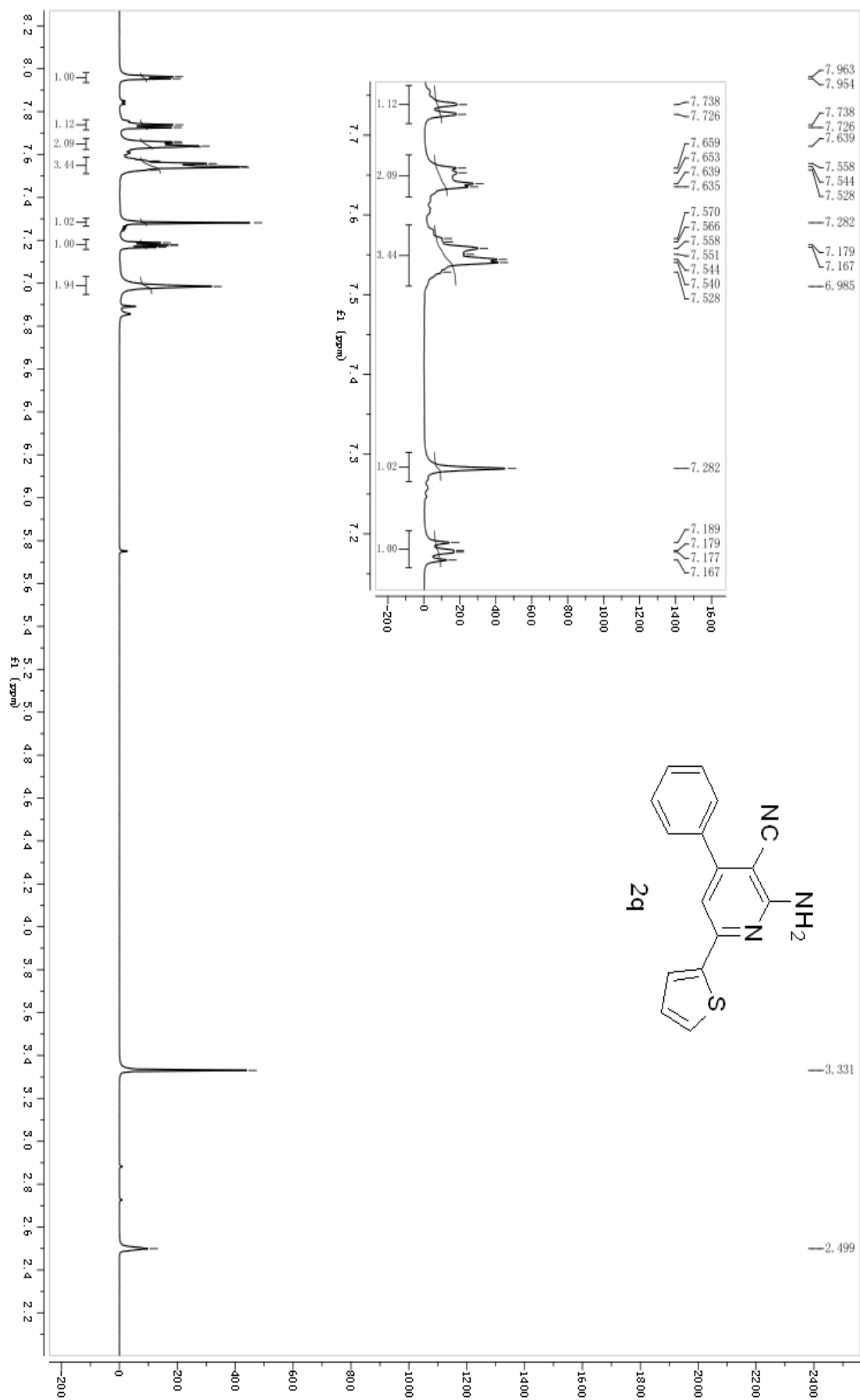
SI: 65536
 SF: 100.5675122 MHz
 MDW: EM
 SSB: 0
 LB: 4.00 Hz
 GB: 0
 PC: 0.90

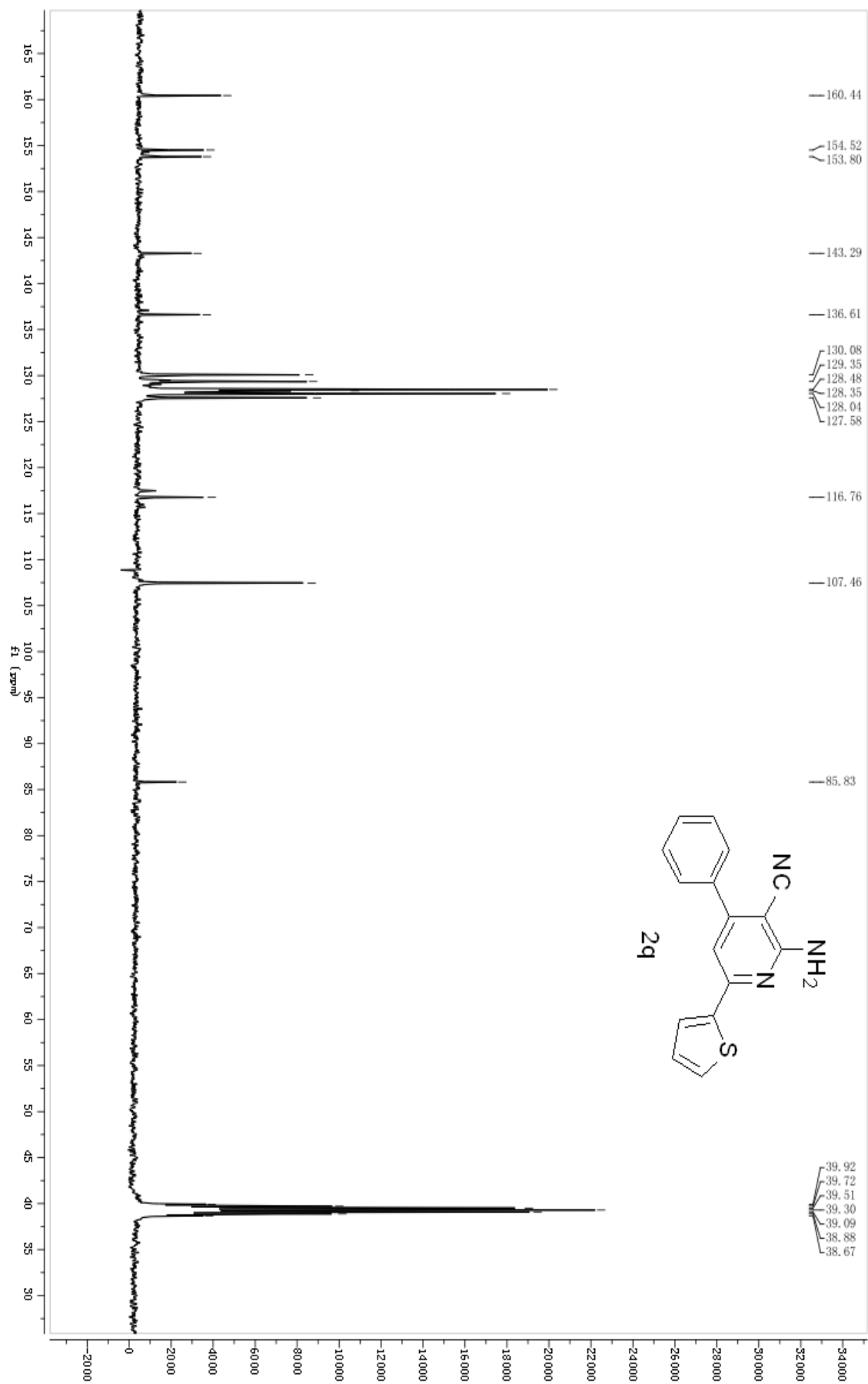
1D NMR plot parameters

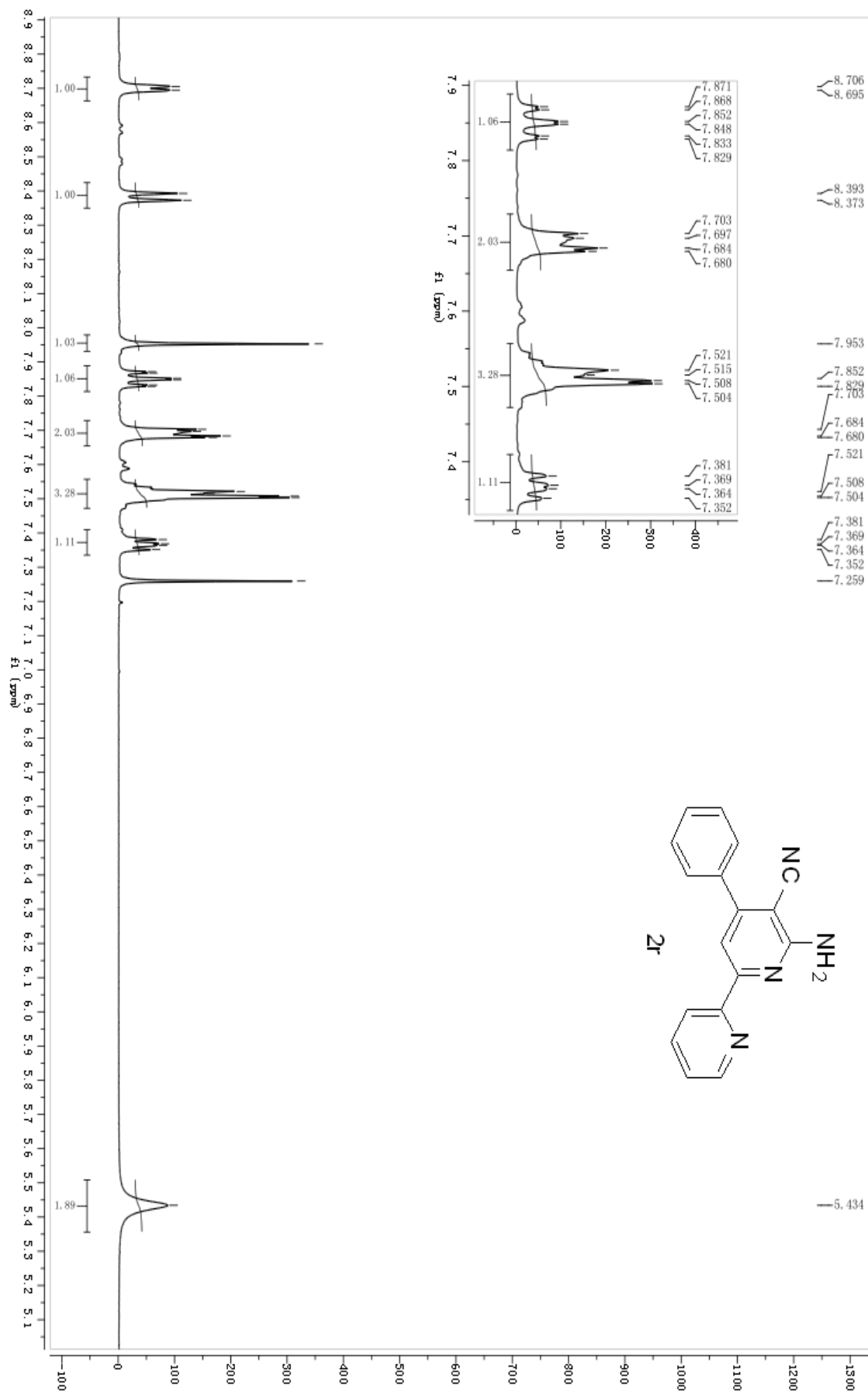
CX: 22.00 cm
 CY: 6.00 cm
 F1P: 197.352 ppm
 F1: 19847.24 Hz
 F2P: 0.113 ppm
 F2: 11.38 Hz
 PPMCM: 8.96542 ppm/cm
 HZCM: 901.63000 Hz/cm

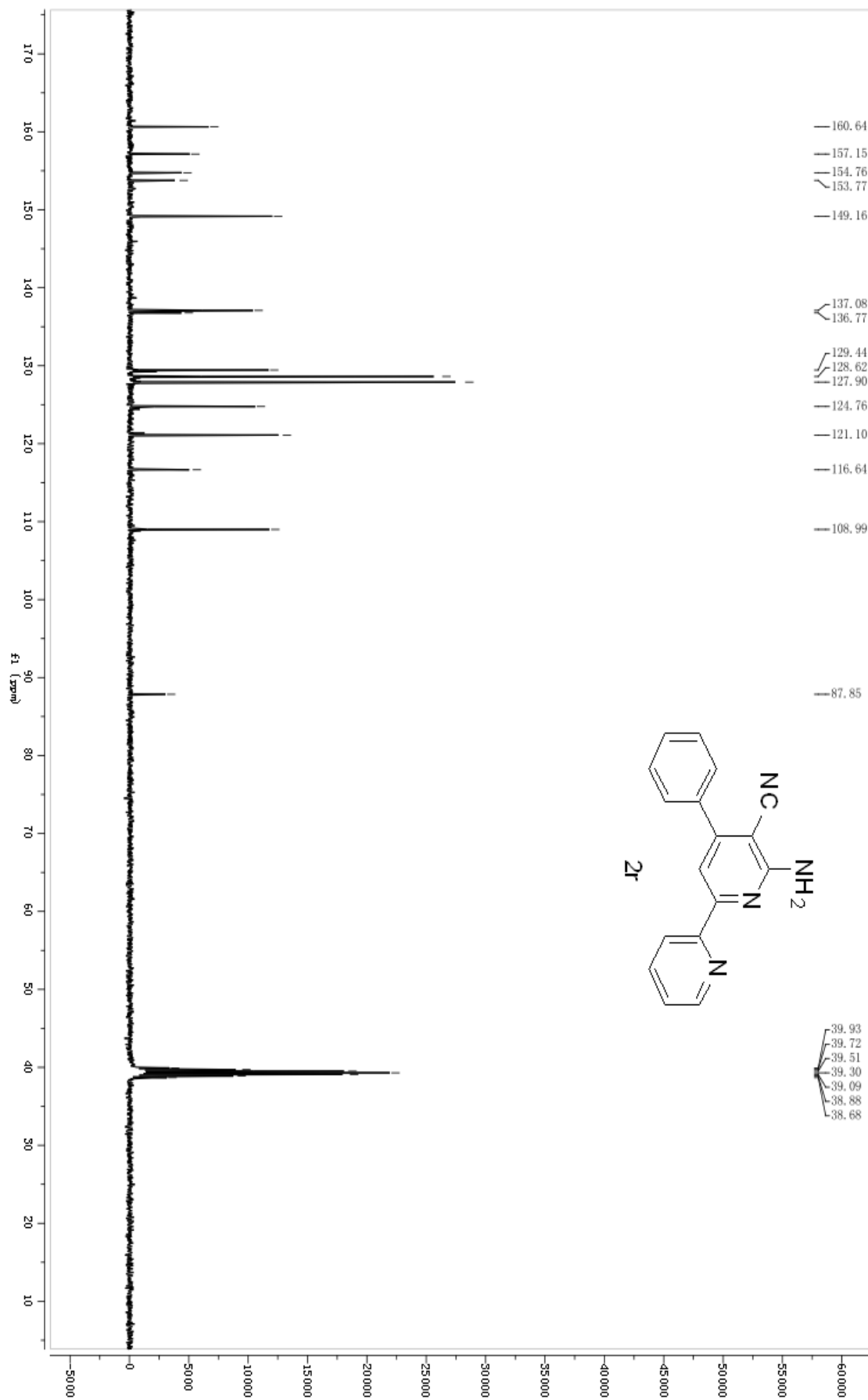


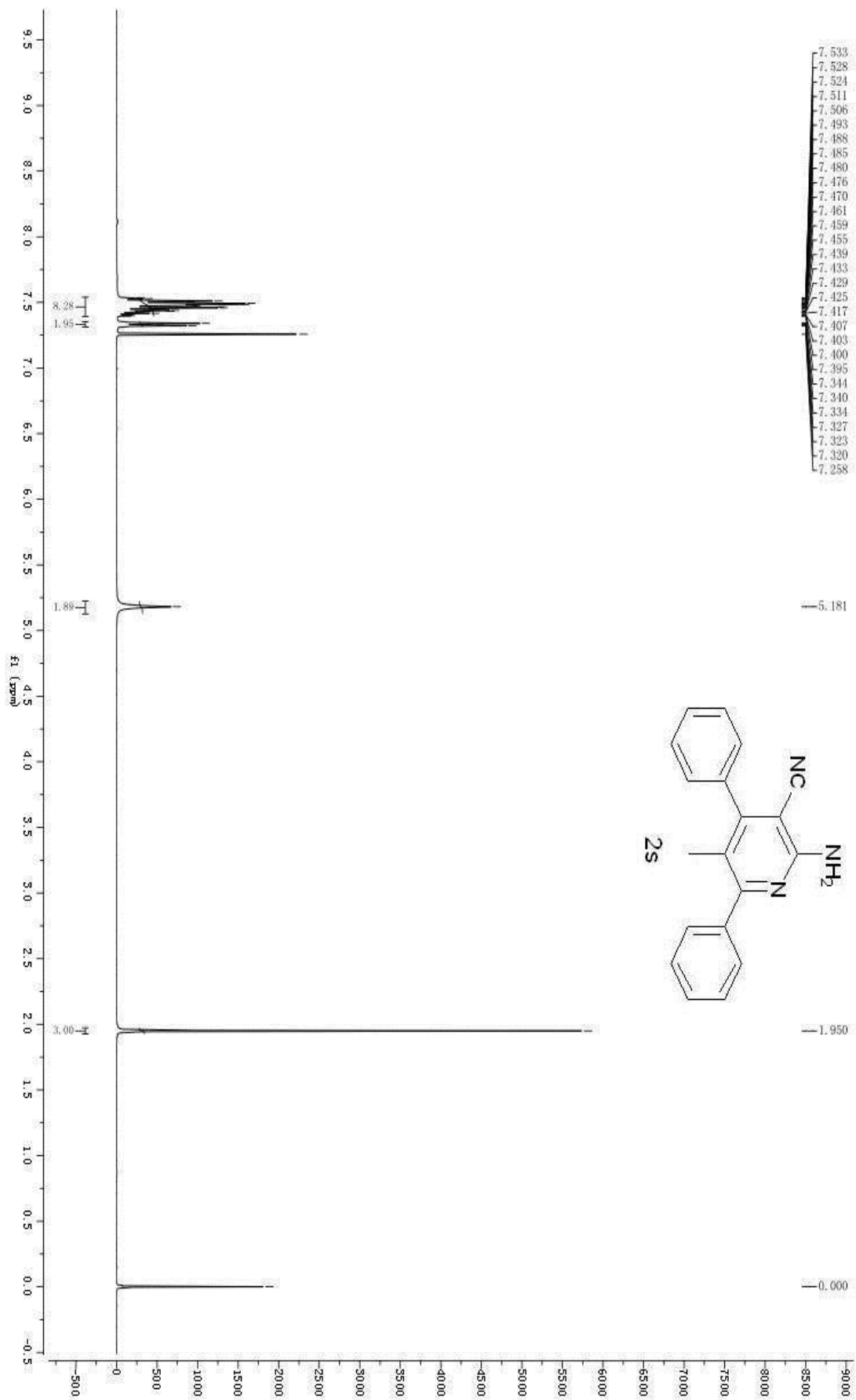


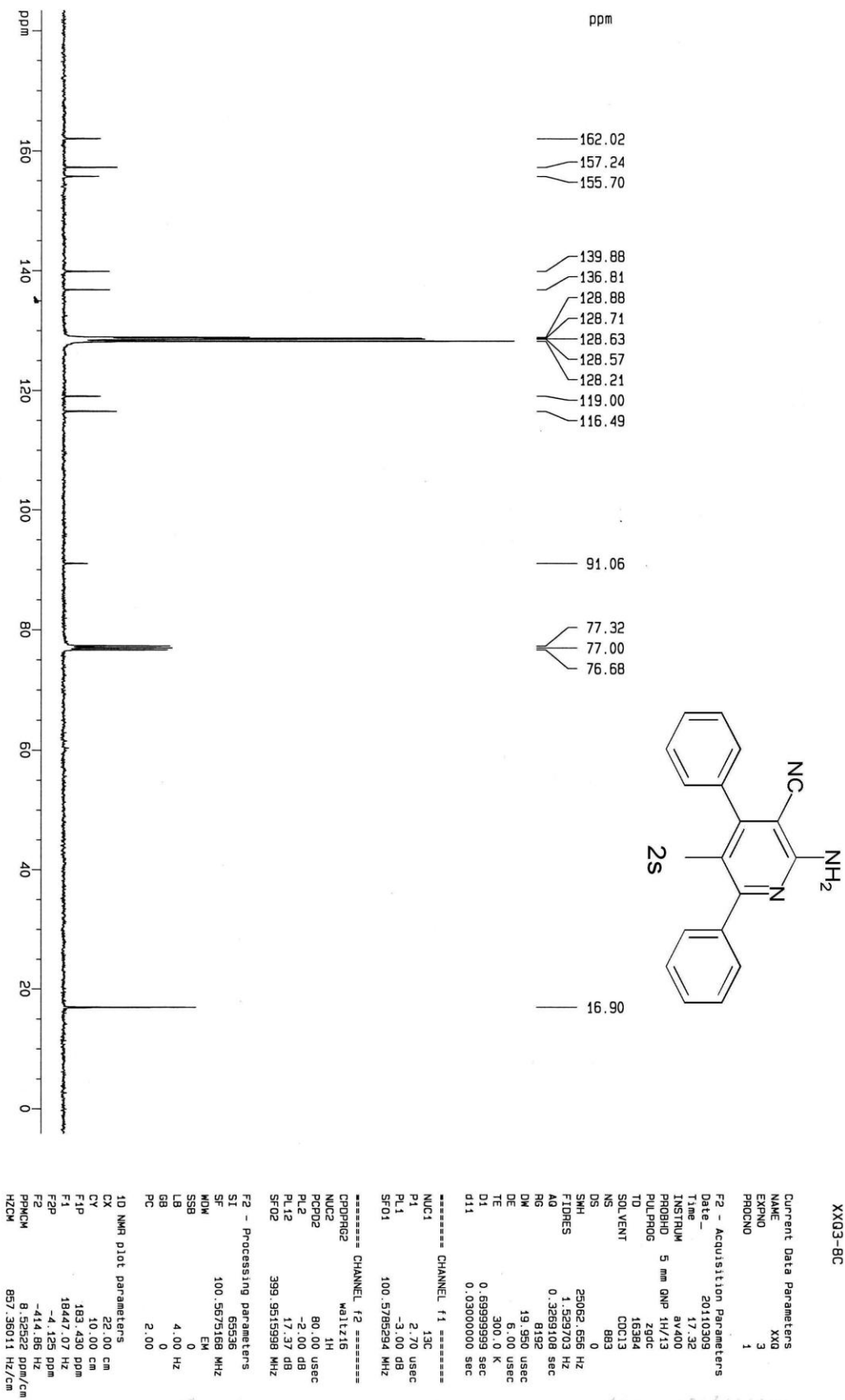


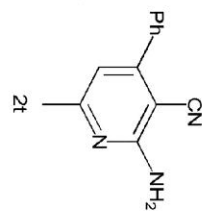
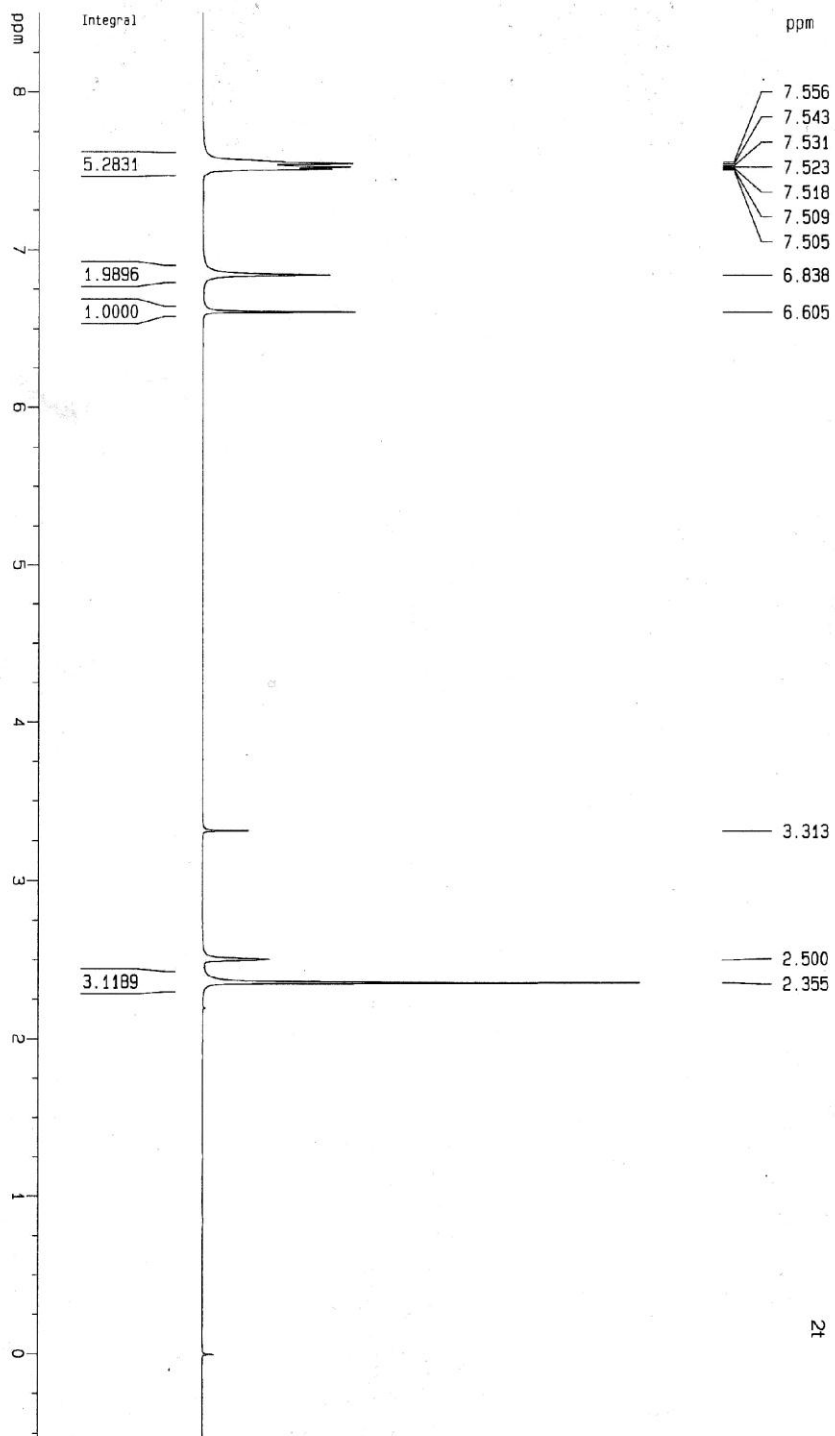












xxd-11-26-1

Current Data Parameters
 NAME XXX
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20121126
 Time 18.03
 INSTRUM av400
 PROBNM 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5307.855 Hz
 FIDRES 0.323965 Hz
 AQ 1.5434228 sec
 RG 16
 DE 94.200 usec
 TE 313.0 K
 D1 10.00000000 sec

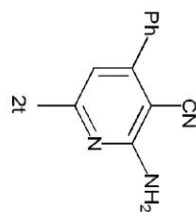
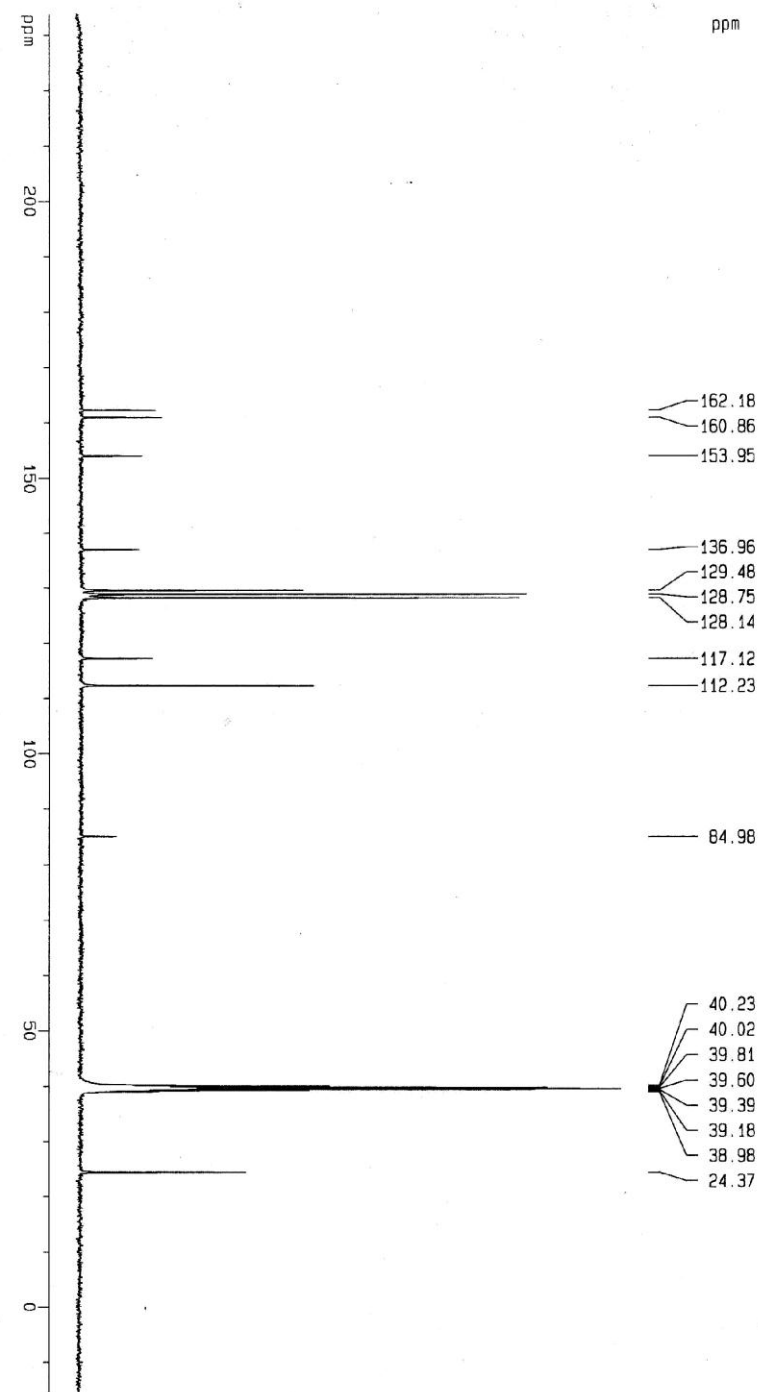
CHANNEL f1
 NUC1 1H
 P1 8.60 usec
 PL1 -3.00 dB
 SFO1 399.9518697 MHz

F2 - Processing parameters

SF 399.9500033 MHz
 MDW 65536
 SSB EM
 LB 0
 GB 0.50 Hz
 PC 1.00

1D NMR plot parameters

CX 23.00 cm
 CY 8.00 cm
 F1P 8.499 ppm
 F1 3399.37 Hz
 F2P -0.540 ppm
 F2 -215.91 Hz
 PPMICM 0.39301 ppm/C
 HZCM 157.18611 Hz/cm



xxq-11-26-1

Current Data Parameters
 NAME XXQ
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121126
 Time 18.04
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg
 TD 16384
 SOLVENT D2O
 NS 7045
 DS 0
 SFO 25062.536 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 8192
 DW 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.50000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.90 usec
 PL1 -3.00 dB
 SFO1 100.5785294 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 17.50 dB
 SFO2 399.9515998 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5675505 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 10.00 cm
 F1P 233.776 ppm
 F1 23510.28 Hz
 F2P -15.436 ppm
 F2 -1552.38 Hz
 PPM1CM 11.32783 ppm/cm
 HZ1CM 1139.21167 Hz/cm

