

Supplementary information

Enantioselective Conjugate Addition Nitro-Mannich Reactions: Solvent Controlled Synthesis of Acyclic *anti*- and *syn*- β -nitroamines with 3 Contiguous Stereocenters

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	13C.....	S32
8	1H.....	S33
	13C.....	S34
9	1H.....	S35
	13C.....	S36
10	1H.....	S37
	13C.....	S38
11	1H.....	S39
	13C.....	S40
12	1H.....	S41
	13C.....	S42
13	1H.....	S43
	13C.....	S44
14	1H.....	S45
	13C.....	S46
	1H.....	S47
	13C.....	S48
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	13C.....	S50
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	13C.....	S52
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	13C.....	S54
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	13C.....	S60
21	1H.....	S61
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22	1H.....	S63
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General experimental

Unless otherwise stated, all reactions were carried out under an atmosphere of nitrogen. All glassware was flame dried and allowed to cool under a stream of nitrogen before use. Cooling to 0 °C was effected using an ice-water bath. Cooling to temperatures below 0 °C was effected using dry ice-acetone mixtures. Reactions were monitored by thin layer chromatography (TLC) using Polygram[®] SIL G/UV₂₅₄ 0.25 mm silica gel precoated plastic plates with fluorescent indicator. Sheets were visualised using ultra-violet light (254 nm) and/or anisaldehyde or KMnO_4 solutions, as appropriate. Flash column chromatography was performed using Fluorochem silica gel 60, 35-70 μ . The liquid phase was analytical grade 40-60 petroleum ether (petrol) and ethyl acetate (EtOAc) unless otherwise noted.

Purification of Solvents and Reagents

Commercial solvents and reagents were used as supplied or purified in accordance with standard procedures, as described below.

Tetrahydrofuran (THF) was pre-dried over sodium wire and distilled under an atmosphere of dry nitrogen from sodium benzophenone ketal or obtained from a solvent tower, where degassed THF was passed through two columns of activated alumina and a 7 micron filter under 4 bar pressure.

Diethyl ether (Et_2O) was pre-dried over sodium wire and distilled under an atmosphere of dry nitrogen from sodium benzophenone ketal or obtained from a solvent tower, where degassed Et_2O was passed through two columns of activated alumina and a 7 micron filter under 4 bar pressure.

Toluene was obtained from a solvent tower, where degassed toluene was passed through two columns of activated alumina and a 7 micron filter under 4 bar pressure. Dichloromethane was distilled from calcium hydride powder or purchased as an analytical grade and stored over 4 Å molecular sieves.

Characterisation

Melting points are uncorrected. Optical rotations were recorded at 25 °C and are reported in $\text{deg cm}^2 \text{g}^{-1}$. Infrared spectra were recorded as a thin film on sodium chloride discs or as dilute chloroform solutions. ^1H and ^{13}C NMR spectra were recorded as dilute solutions in deuteriochloroform unless otherwise stated. All chemical shifts (δ) are reported in parts per million (ppm) relative to chloroform ($\delta_{\text{H}} = 7.27$ ppm, $\delta_{\text{C}} = 77.1$ ppm). Coupling constants (J) are reported in Hertz and are recorded as observed in the spectrum without averaging. The multiplicity of an ^1H signal is designated by the following abbreviations: m = multiplet, s = singlet, d = doublet, t = triplet, q = quartet and quin = quintet, br = broad signal. ^{13}C multiplicities were assigned using a DEPT sequence. Where appropriate, HMQC and NOE experiments were carried out to aid assignment.

Nitroalkenes were prepared according to literature procedures¹ and their data was in accord with that published.²

Solvent studies

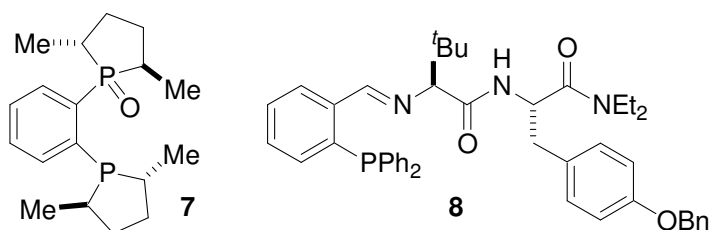
To a stirred mixture of nitroalkene (1.00 mmol) and Cu(Otf)₂ (0.05 mmol) in dry solvent (3 mL) at -78 °C was added Et₂Zn (1.10 mmol, 1M in hexane). The resulting orange solution was stirred for 10 min before being warmed to rt. After 1 h the brown suspension was cooled to -78 °C. A solution of imine (2.00 mmol) in dry solvent (2 mL) was added and the mixture stirred for 20 min. A solution of TFA (3.50 mmol) in dry solvent (0.6 mL) was added dropwise over 20 seconds and the reaction stirred for 1 h. The reaction was warmed to room temperature over 1 h to provide a suspension of white solid and vivid yellow supernatant. The reaction was quenched by the addition of Et₂O (5 mL) and saturated aq. NaHCO₃ (5 mL). The layers were separated and the aqueous phase extracted with Et₂O (2 x 5 mL). The organic layers were combined and the solvent removed *in vacuo* to provide crude β -nitroamine. Note: MgSO₄ was not used to dry the product β -nitroamines prior to solvent removal because the products were found to be mildly unstable towards this reagent.

Temperature studies

The reactions were carried out as above for the specified times recorded in Table 2.

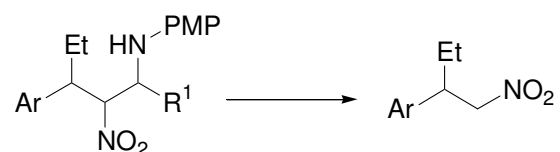
Chiral ligands

The Charetté ligand Bozphos (**7**) was used as the Cu(**7**)₂Otf complex and was prepared in two steps from commercially available Me-DuPHOS according to the literature.³ The Hoveyda ligand **8** was prepared according to the literature⁴ and was used as described.



Determination of *ee*.

Retention times were compared to racemic standards. If the products could not be separated by a chiral stationary phase, they were measured by degradation.



syn, anti-a. As is normal for the types of *anti*- β -nitroamines we generate, the NMR solutions gradually undergo retro addition if left in CDCl₃ at rt.⁵ Samples were left until complete degradation

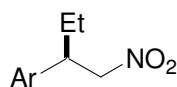
(1-2 days) by ^1H NMR and the crude nitroalkane was then used for HPLC analysis to measure the enantiomeric excess of the parent β -nitroamine.

Syn, syn-b. If the product could not be separated by a chiral stationary phase, they were measured by degradation. *B*-Nitroamine (*ca.* 20 mg) was dissolved in THF (3 mL) and TFA (0.2 mL) was added. The yellow solution was heated at reflux for 5 h before being cooled to rt. Et₂O (2 mL) was added and the organic phase was washed with saturated aq. NaHCO₃ (2 mL), dried (MgSO₄) and the solvent removed in vacuo. The crude nitroalkane was then used for HPLC analysis to measure the enantiomeric excess of the parent β -nitroamine.

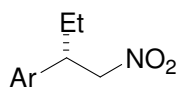
These processes were validated by subjecting entry 18, Table 4 (Ar=Ph, R=Et, R¹=*p*-Me-Ph) measured by chiral HPLC (OD-H column, 99.5 % hexane 0.5% IPA) to be 96% *ee* to the above conditions which showed the parent nitroalkane had 95% *ee*.

Assignment of absolute and relative stereochemistry

We have assumed that the initial conjugate addition with ligands **7** and **8** gives the opposite enantiomers as characterised by Charette³ and Hoveyda.⁴



using Charette ligand **7**



using Hoveyda ligand **8**

Entry 1/15. X-ray structures of each diastereoisomer confirmed relative stereochemistry.

Entry 2/17. The relative stereochemistry of these two diastereoisomers was assigned by analogy to the other compounds, **Entry 2** slowly degraded by retro addition in CDCl₃ indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. Whereas **Entry 17** was stable in CDCl₃, purification by chromatography and was unreactive towards trifluoroacetic anhydride, indicating *syn*- β -nitroamine stereochemistry.

Entry 3/18. **Entry 3** slowly degraded by retro addition in CDCl₃ indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. X-ray confirmed relative stereochemistry of **Entry 18**, which was stable in CDCl₃ and purification by chromatography.

Entry 4. The same diastereoisomer was isolated as the trifluoroacetamide from a THF or PhMe nitro Mannich reaction (both homogeneous) using the Charette ligand **5** and the stereochemistry was assigned by analogy to the other compounds. The parent β -nitroamine before treatment with trifluoroacetic anhydride was unstable in CDCl₃ and collectively indicated *anti*- β -nitroamine stereochemistry.⁵

Entry 5/19. X-ray confirmed relative stereochemistry of **Entry 5** which slowly degraded by retro addition in CDCl_3 and was reactive towards trifluoroacetic anhydride. **Entry 19** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

Entry 6/20. The relative stereochemistry of these two diastereoisomers was assigned by analogy to the other compounds. **Entry 6** slowly degraded by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. Whereas **Entry 20** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

Entry 7/21. X-ray confirmed relative stereochemistry of **Entry 7** which slowly degraded by retro addition in CDCl_3 and was reactive towards trifluoroacetic anhydride. **Entry 21** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

Entry 8/16. The relative stereochemistry of these two diastereoisomers was assigned by analogy to the other compounds (especially **Entry 1/15**). **Entry 8** slowly degraded by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. Whereas **Entry 16** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

Entry 9/22. X-ray structures of each diastereoisomer confirmed the relative stereochemistry.

Entry 10/23. X-ray confirmed relative stereochemistry of **Entry 10** which slowly degraded by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. **Entry 23** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

Entry 11/24. **Entry 11** slowly degrades by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. X-ray confirmed the relative stereochemistry of **Entry 24**, which was stable in CDCl_3 and purification by chromatography.

Entry 12/25. The relative stereochemistry of these two diastereoisomers was assigned by analogy to the other compounds. **Entry 12** slowly degraded by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was reactive towards trifluoroacetic anhydride. Whereas **Entry 25** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

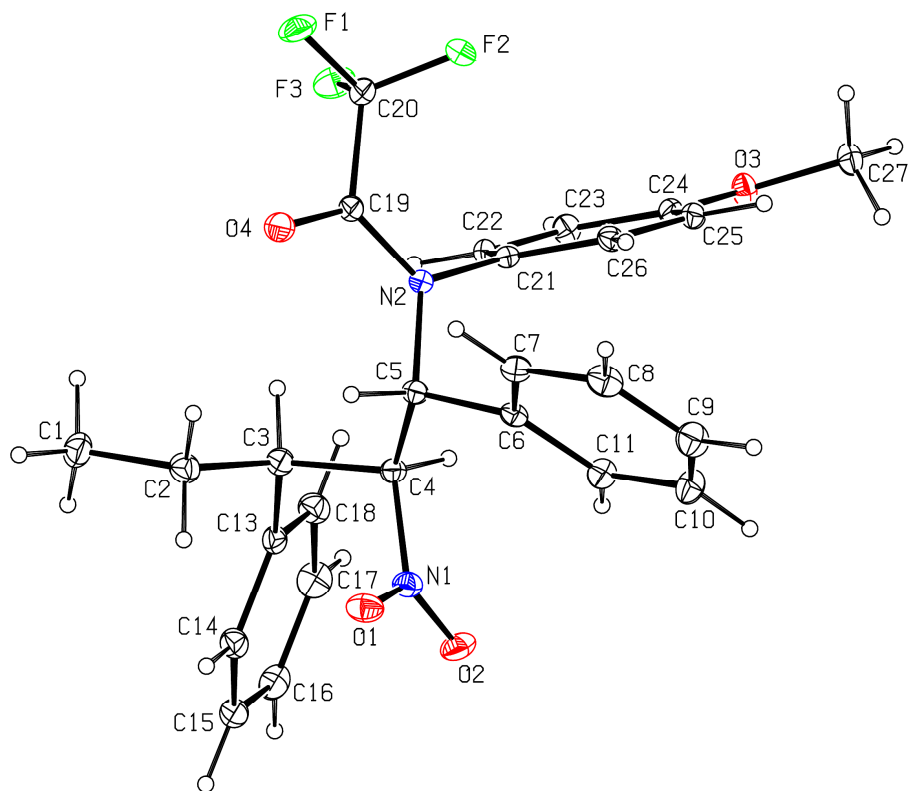
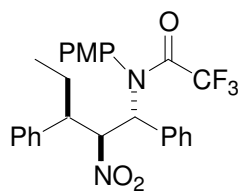
Entry 13. X-ray structure confirmed relative stereochemistry.

Entry 14. The relative stereochemistry of these two diastereoisomers was assigned by analogy to the other compounds. The *syn*, *anti*-**a** diastereoisomer slowly degraded by retro addition in CDCl_3 indicating *anti*- β -nitroamine stereochemistry⁵ and was unstable on chromatography. Whereas *syn*, *syn*-**b** was stable in CDCl_3 and purification by chromatography, indicating *syn*- β -nitroamine stereochemistry.

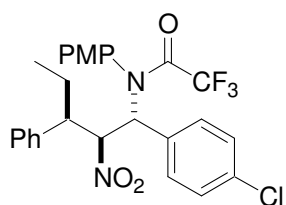
Entry 26. X-ray structure confirmed relative stereochemistry.

Structures and X-ray structures for compounds in Table 4

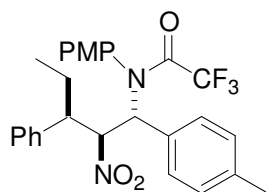
Entry 1 - Cu(7)₂OTf, THF



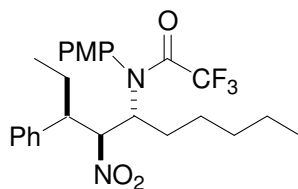
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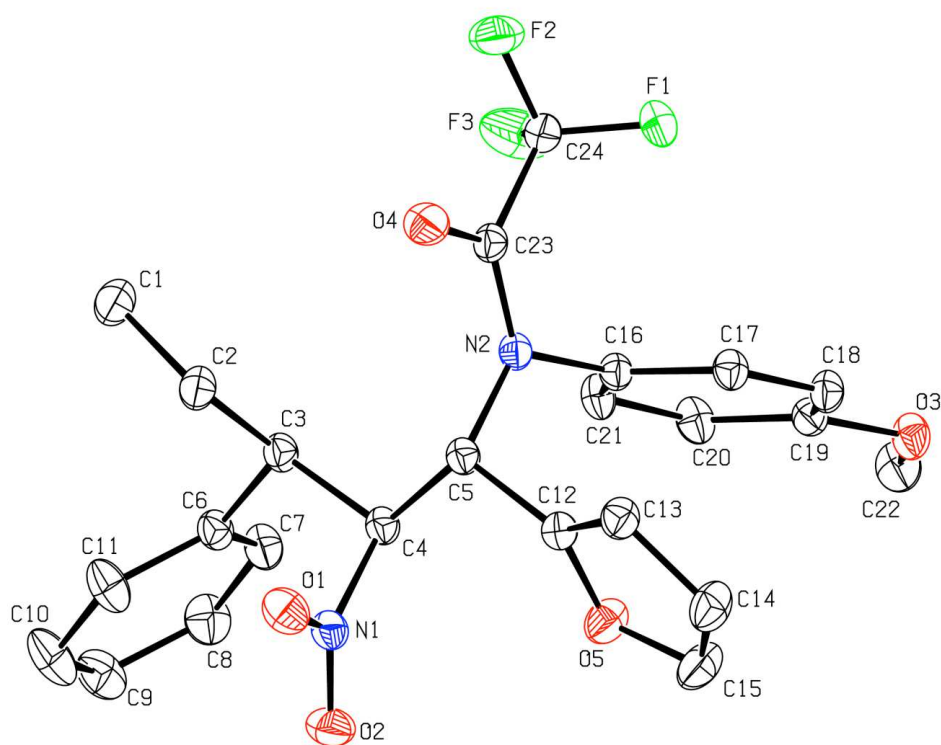
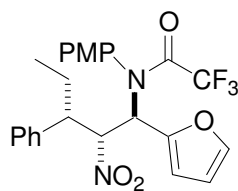
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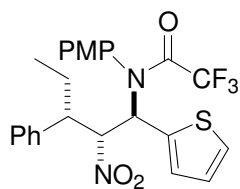
Entry 4 - Cu(7)₂OTf, THF or PhMe



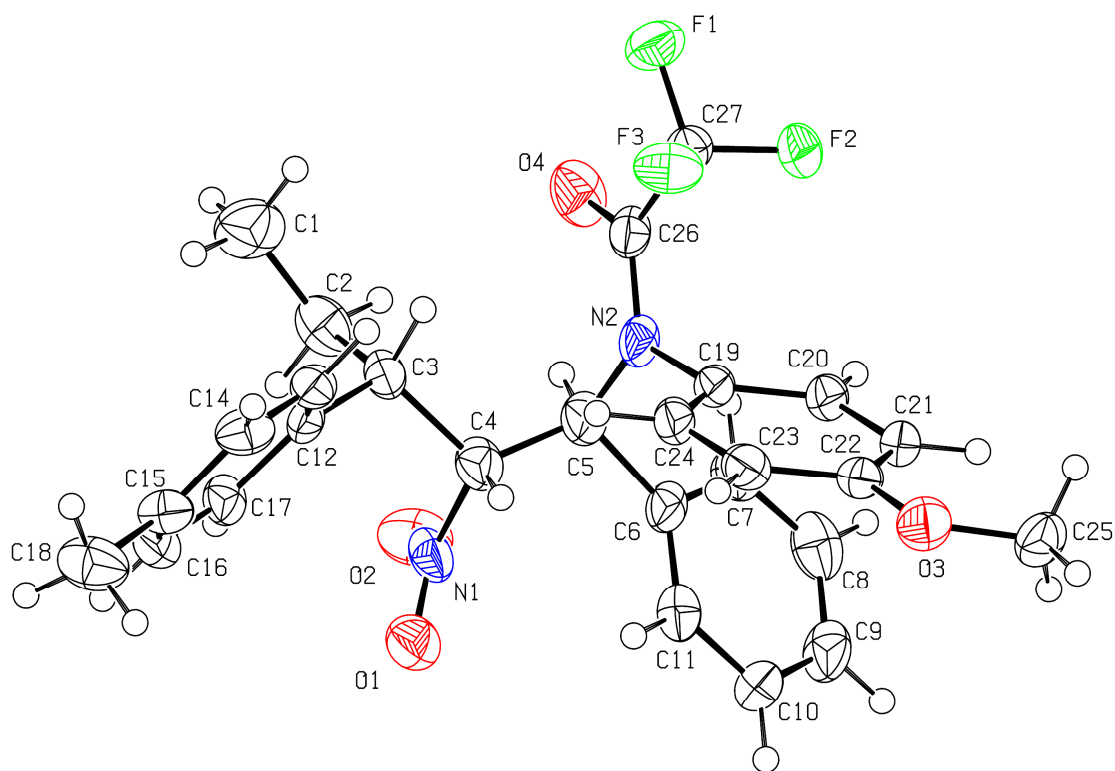
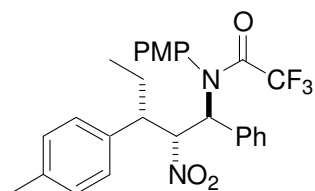
Entry 5 – Ligand 8, THF



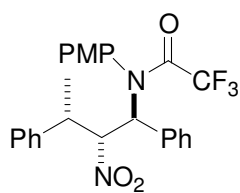
Entry 6 - Ligand 8, THF



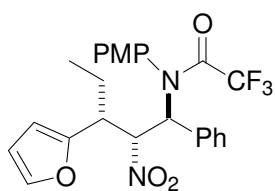
Entry 7 - Ligand 8, THF



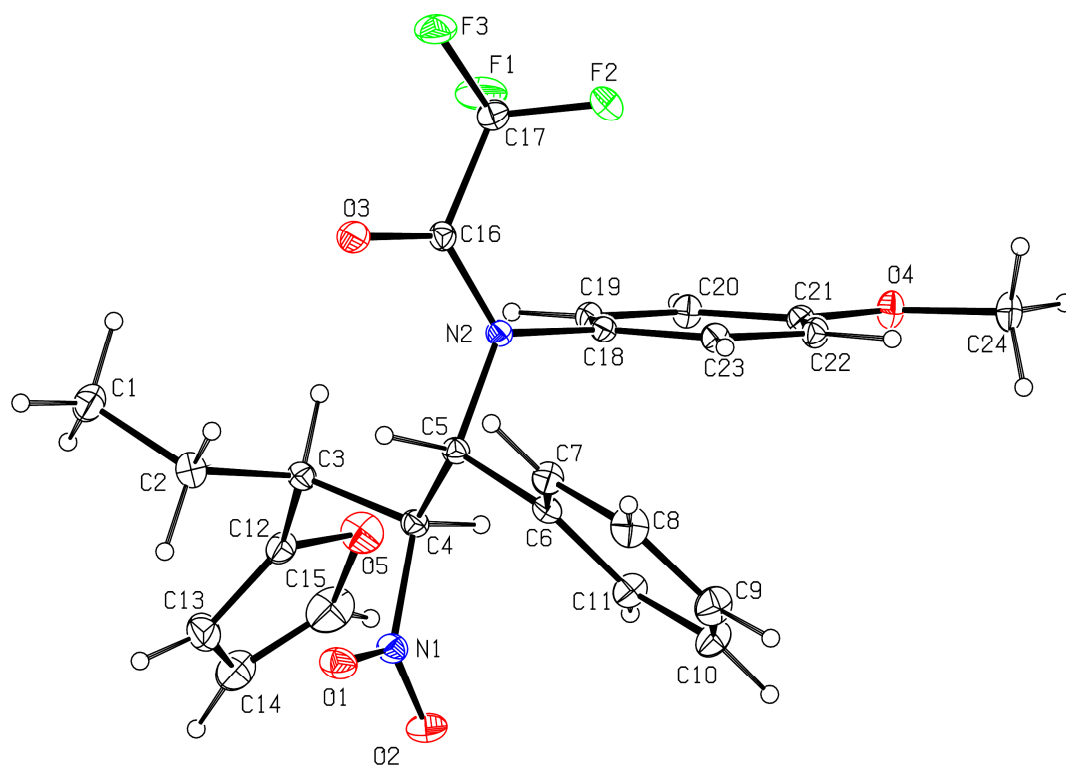
Entry 8 - Ligand 8, THF



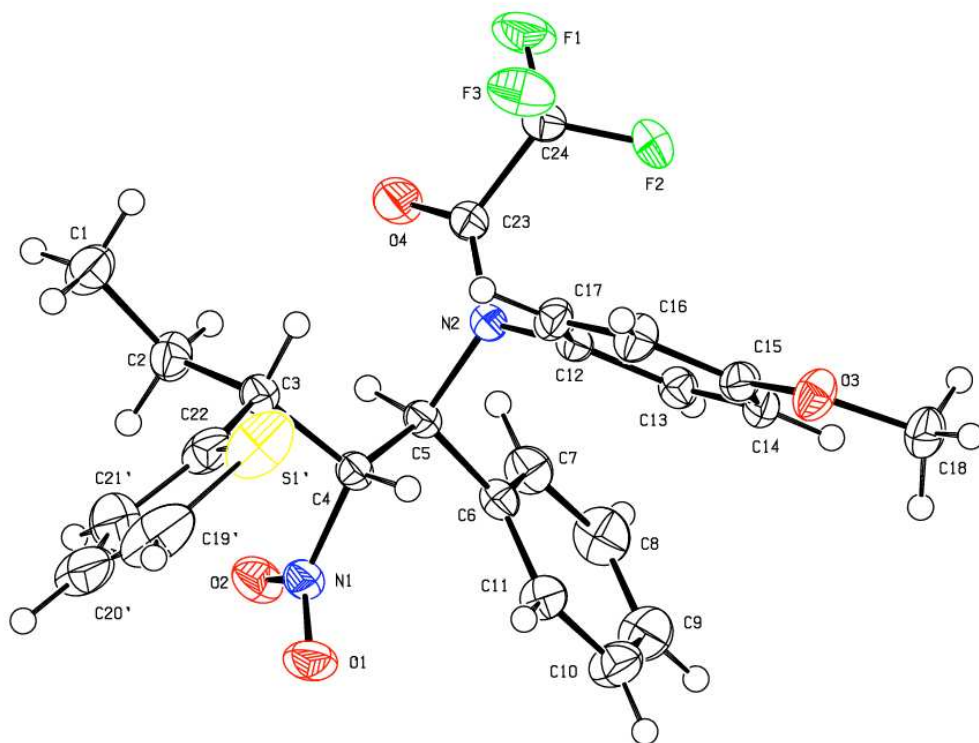
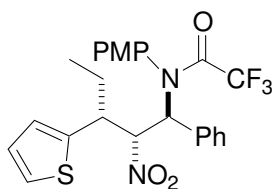
Entry 9 - Ligand 8, THF



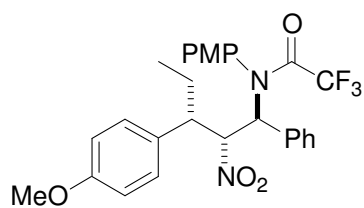
X-ray of enantiomer.



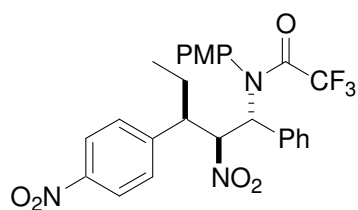
Entry 10 - Ligand 8, THF



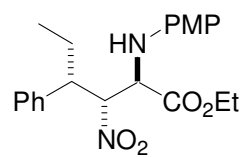
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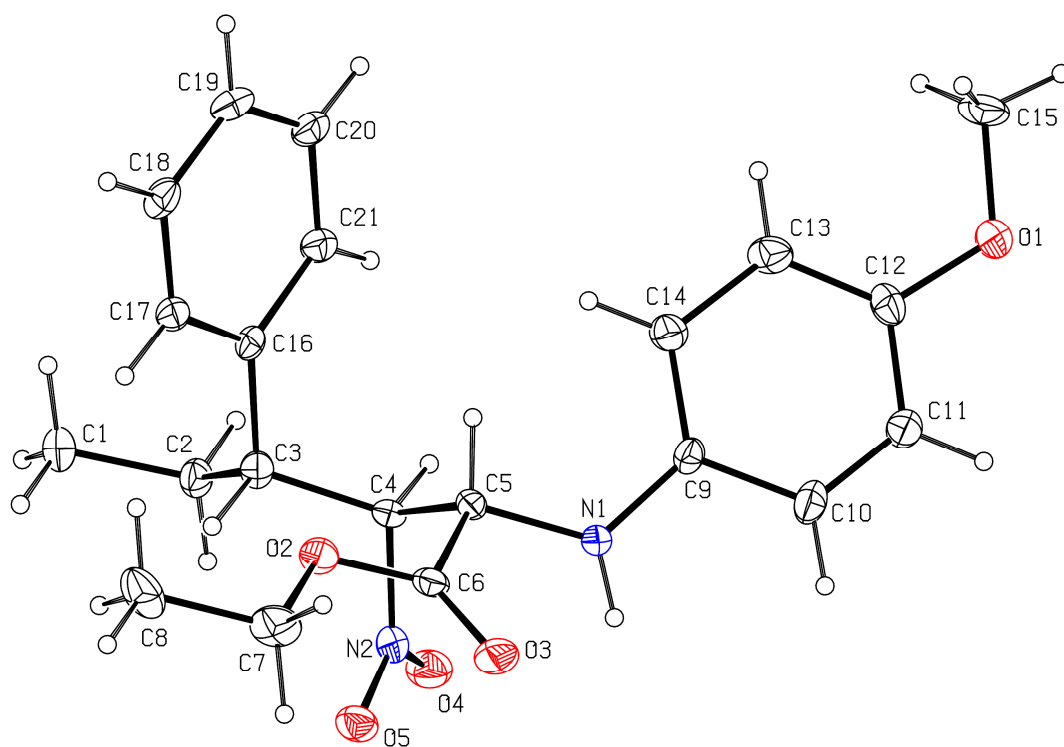
Entry 12 – no ligand, THF



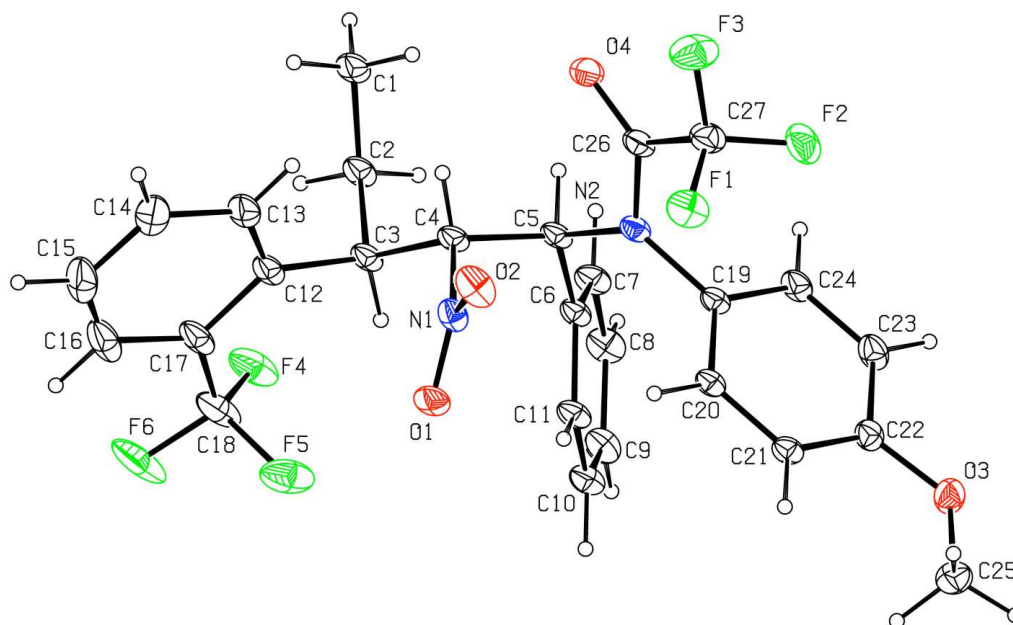
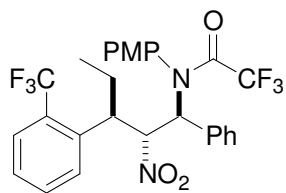
Entry 26 - Ligand 8, PhMe



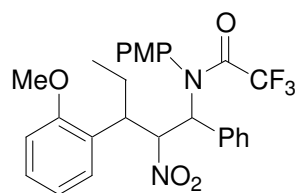
X-ray of enantiomer.



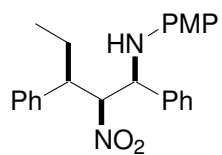
Entry 13 – no ligand, THF



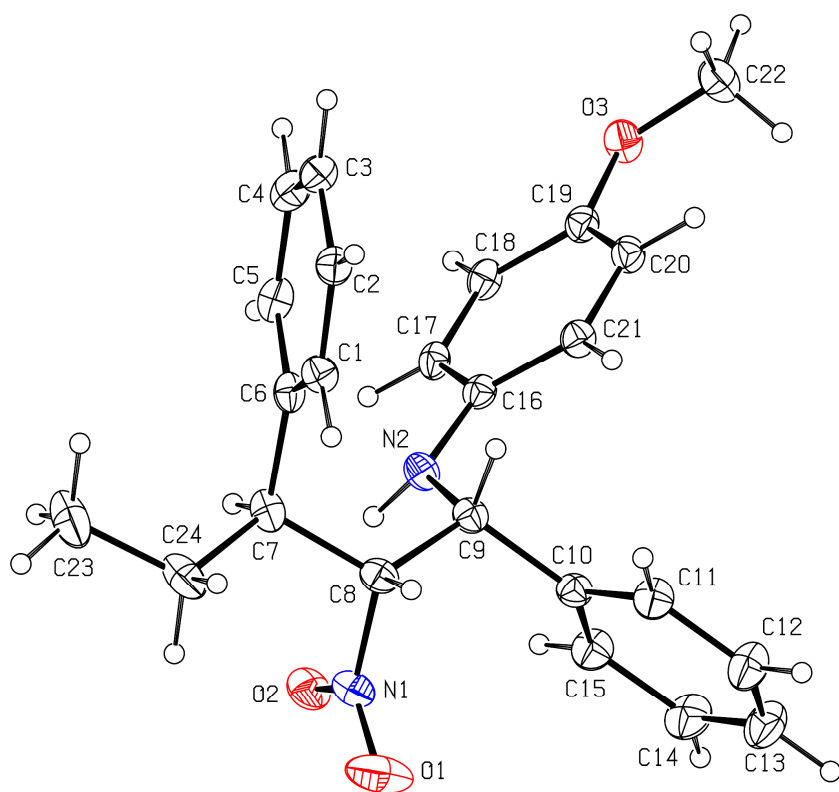
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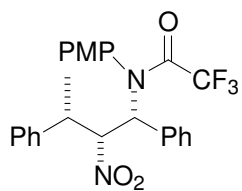
Entry 15 - Cu(7)₂OTf, Et₂O



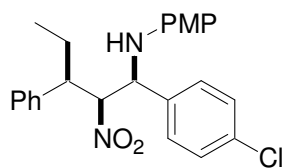
X-ray of enantiomer



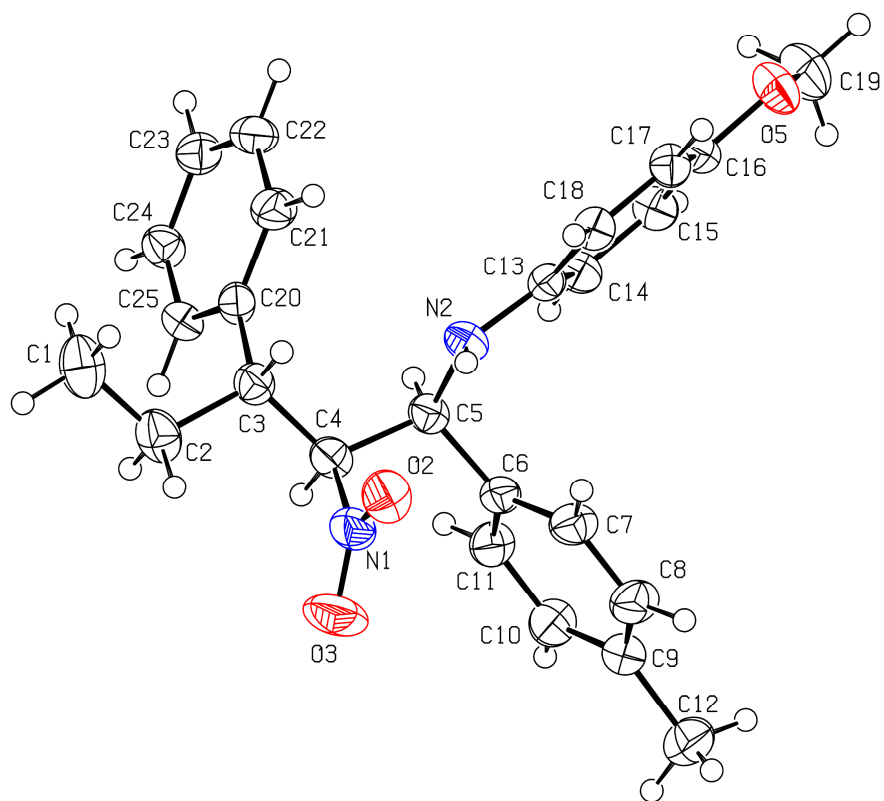
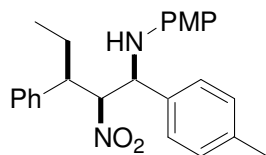
Entry 16 – Ligand 8, PhMe



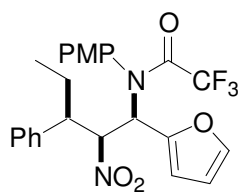
Entry 17 - Cu(7)₂OTf, Et₂O



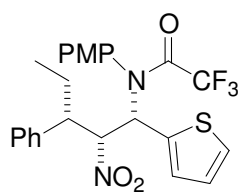
Entry 18 - Cu(7)₂OTf, Et₂O



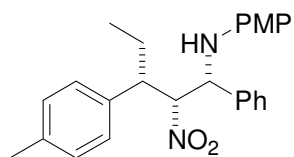
Entry 19 - Cu(7)₂OTf, Et₂O



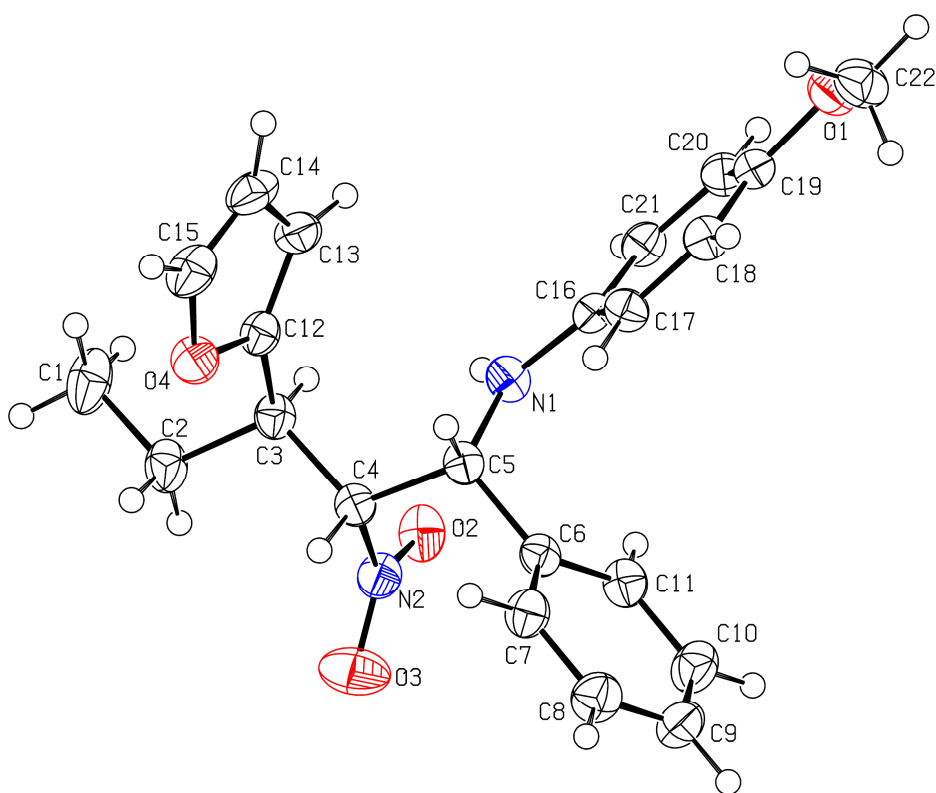
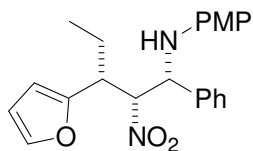
Entry 20 - Ligand **8**, PhMe



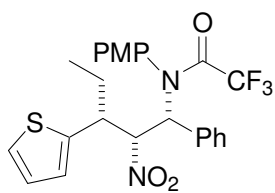
Entry 21 - Ligand 8, PhMe



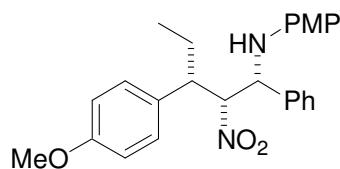
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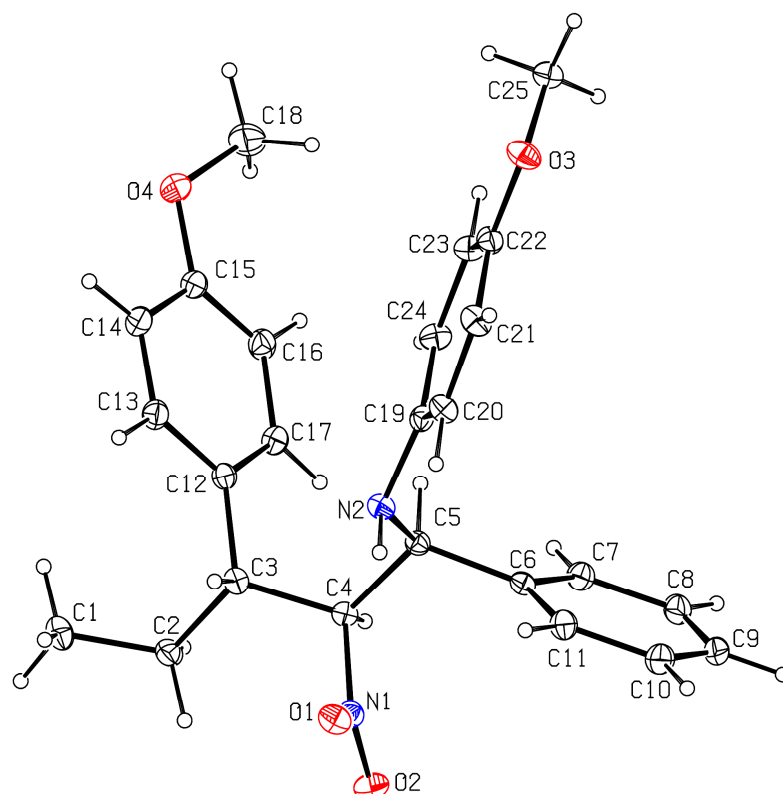
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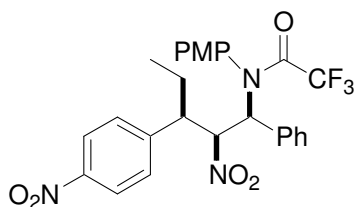
Entry 24 - Ligand 8, PhMe



X-ray of enantiomer.

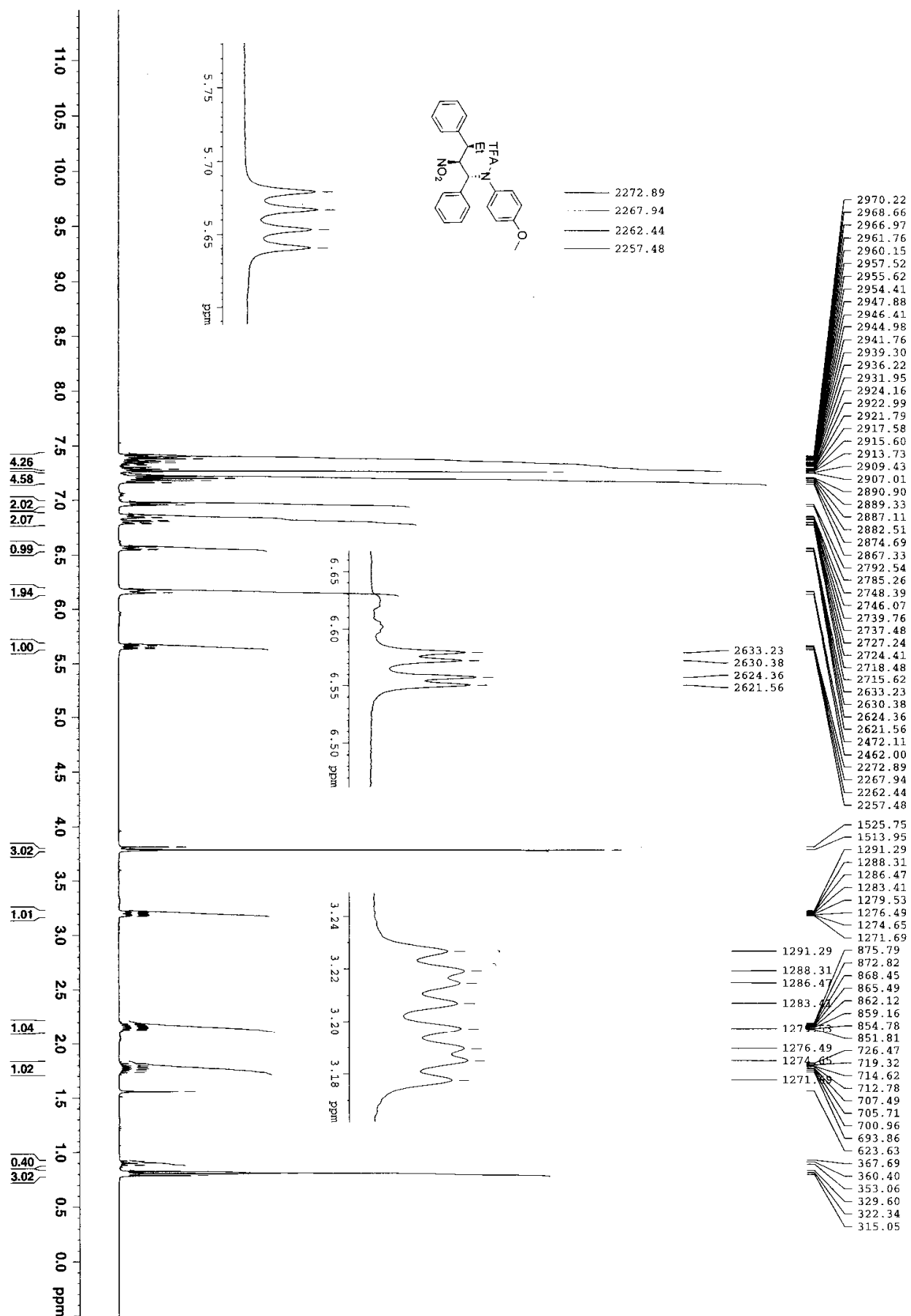


Entry 25 – no ligand, diethyl zinc addition in THF, nitro-Mannich in Et₂O

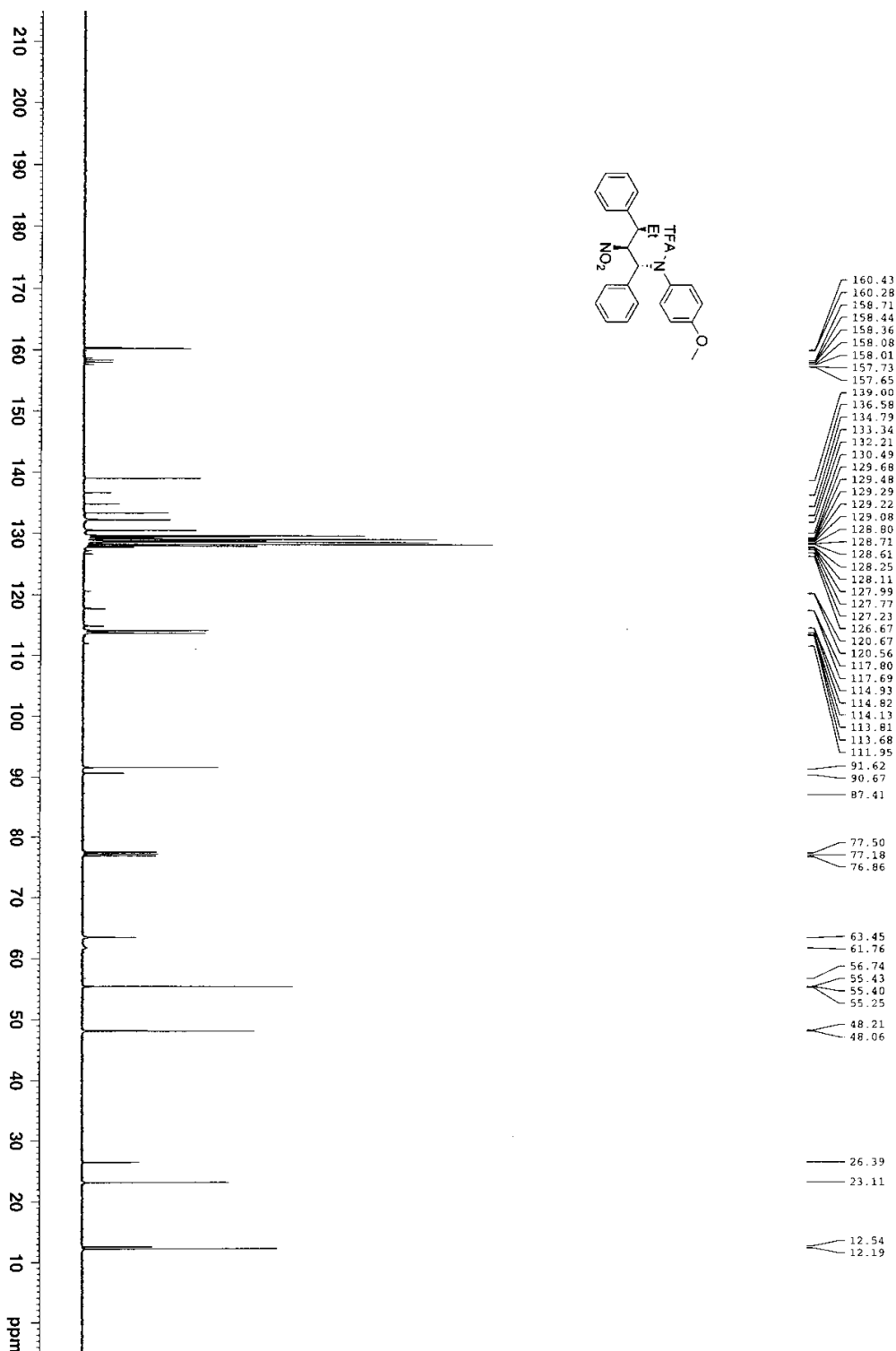
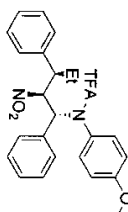


1. a) Fieser, L. F.; Gates, M. *J. Am. Chem. Soc.* **1946**, 68, 2249. b) Melton, J.; McMurry, J. E. *J. Org. Chem.* **1975**, 40, 2138.
2. a) Kumaran, G.; Kulkarni, G.H. *Synthesis*, **1995**, 1545. b) Enders, D.; Wiedemann, J. *Synthesis*, **1996**, 1443.
3. Côté, A.; Charette, A.B. *J. Org. Chem.* **2005**, 70, 10864.
4. Mampreian, D. M.; Hoveyda, A. H. *Org. Lett.* 2004, 6, 2829.
5. Adams, H.; Anderson, J. C.; Peace, S.; Pennell, A. M. K. *J. Org. Chem.* **1998**, 63, 9932.

¹H NMR Entry 1



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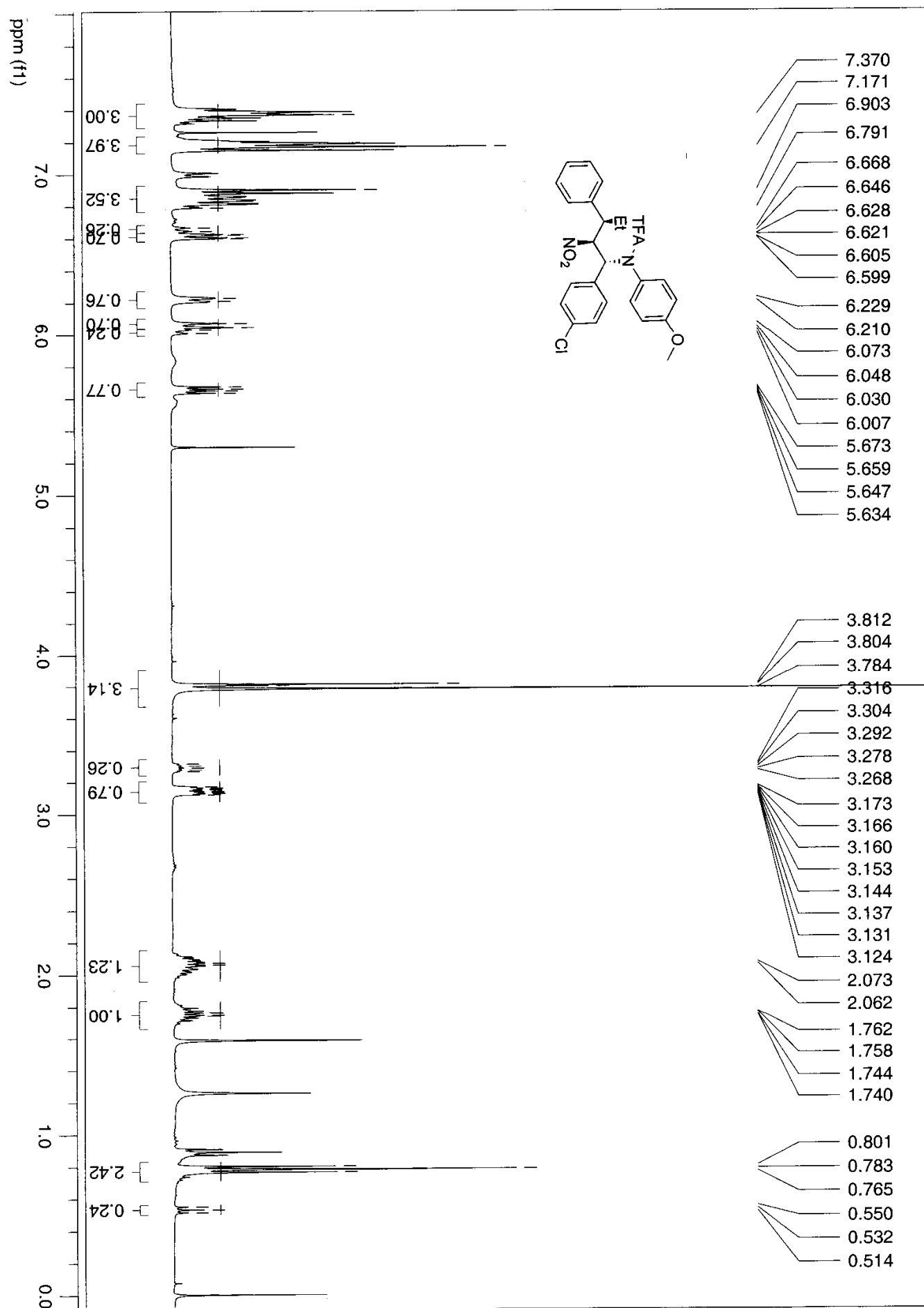
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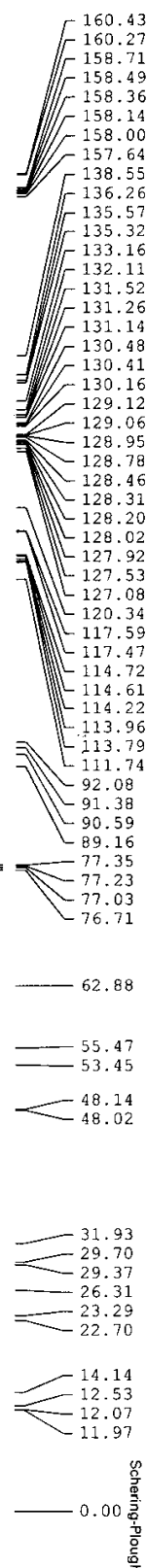
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NUC2       1H
PCPD2      100.00 usec
P2         -6.00 dB
PL2        11.00 dB
PL12       12.00 dB
SFO2       400.201608 MHz

F2 - Processing parameters
SI         32768
SF         100.630700 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

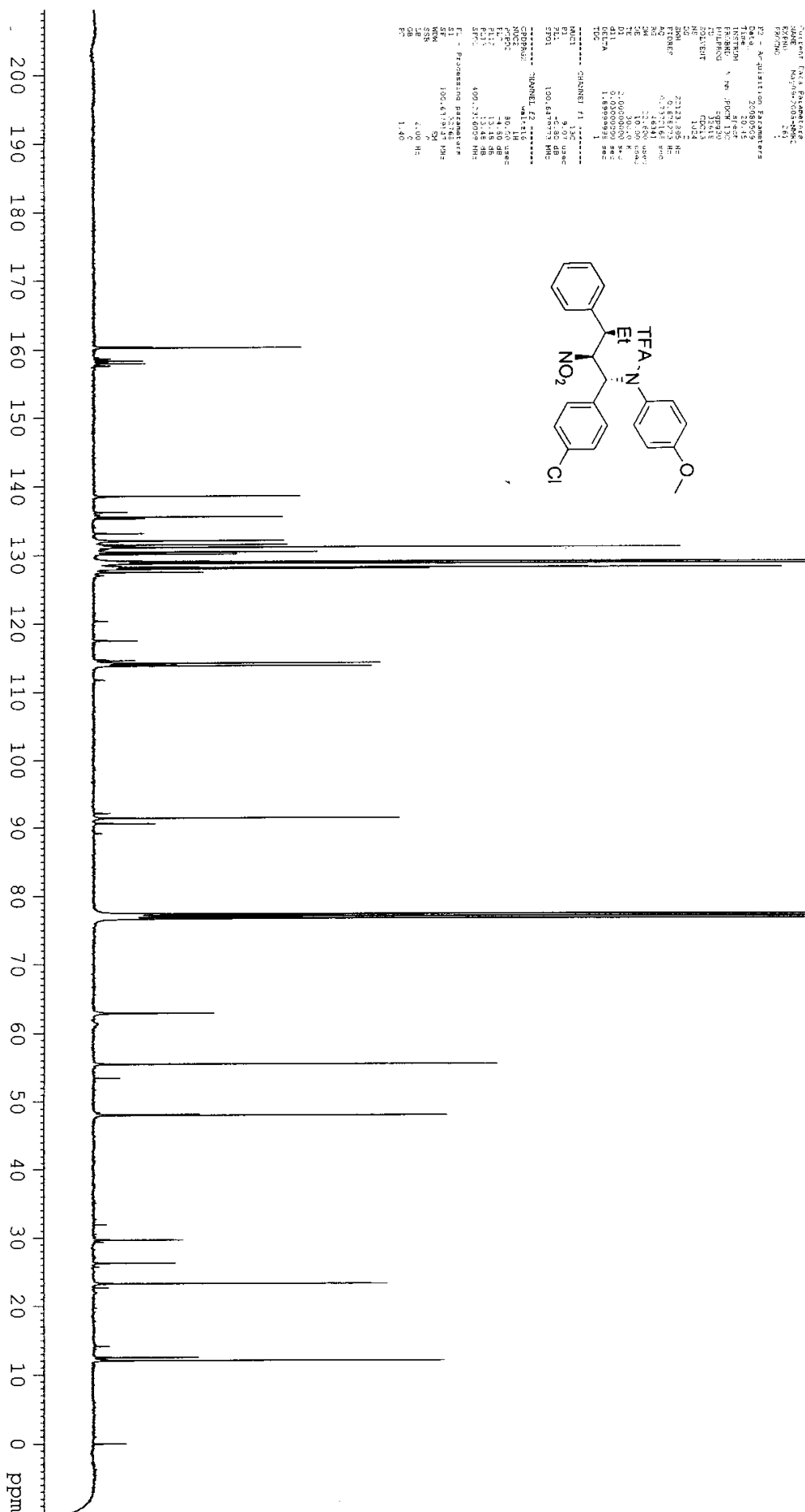
¹H NMR Entry 2



Sample Code mrm422

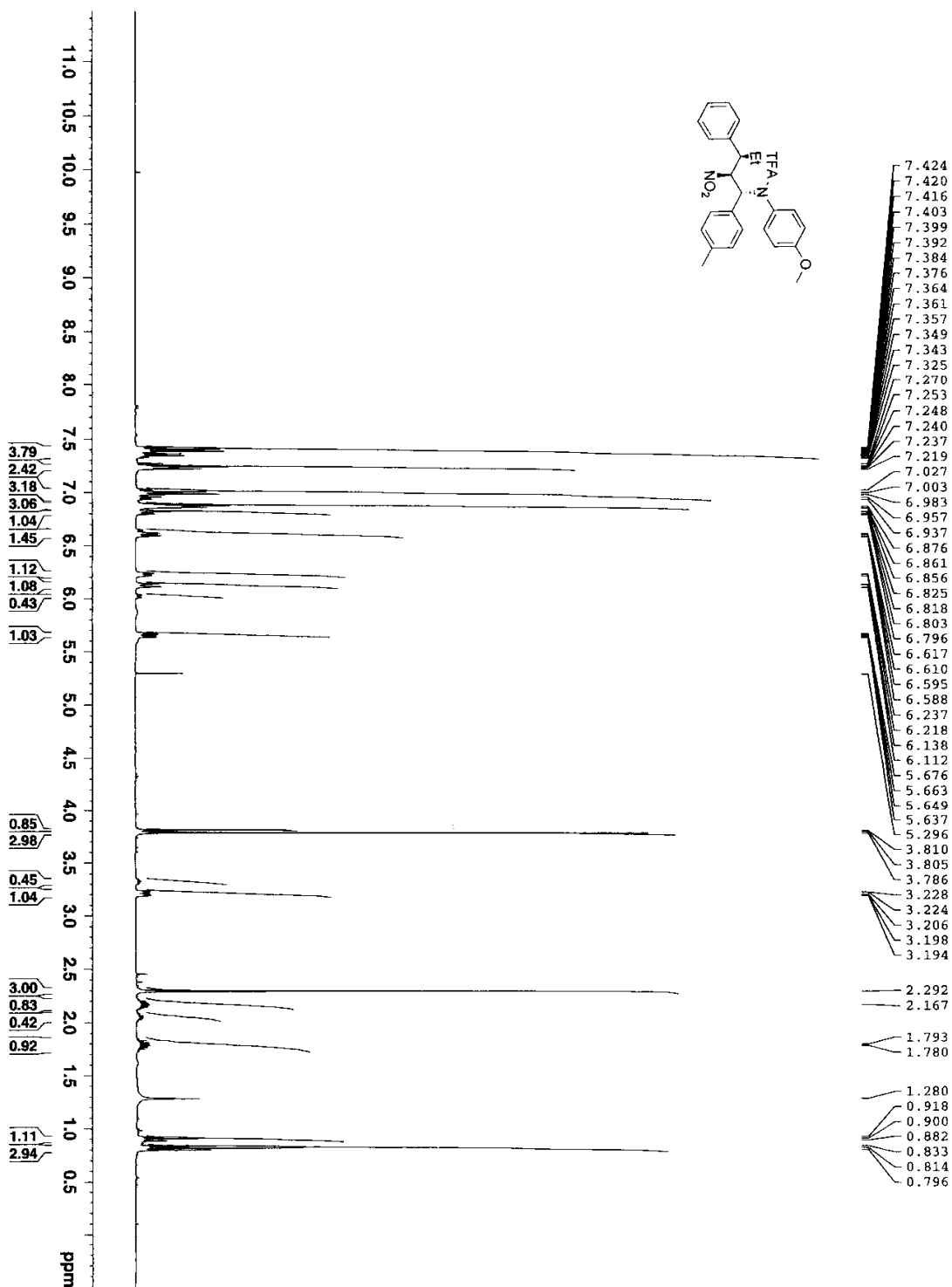


===== CHANNEL F1 =====
 Name: 13C NMR
 File: 13C NMR
 F1: 100.61773 MHz
 ===== CHANNEL F2 =====
 Name: 13C NMR
 File: 13C NMR
 F2: 100.61773 MHz
 ===== Processing parameters =====
 SI: 32768
 SF: 100.61773 MHz
 EQ: 1.00
 ZF: 4.00 MHz
 PC: 1.40



¹H NMR Entry 3

Slot No. 39 Sample ID mrm399 SupervisorID ander Lab Phone No. 1 UserID m_mil

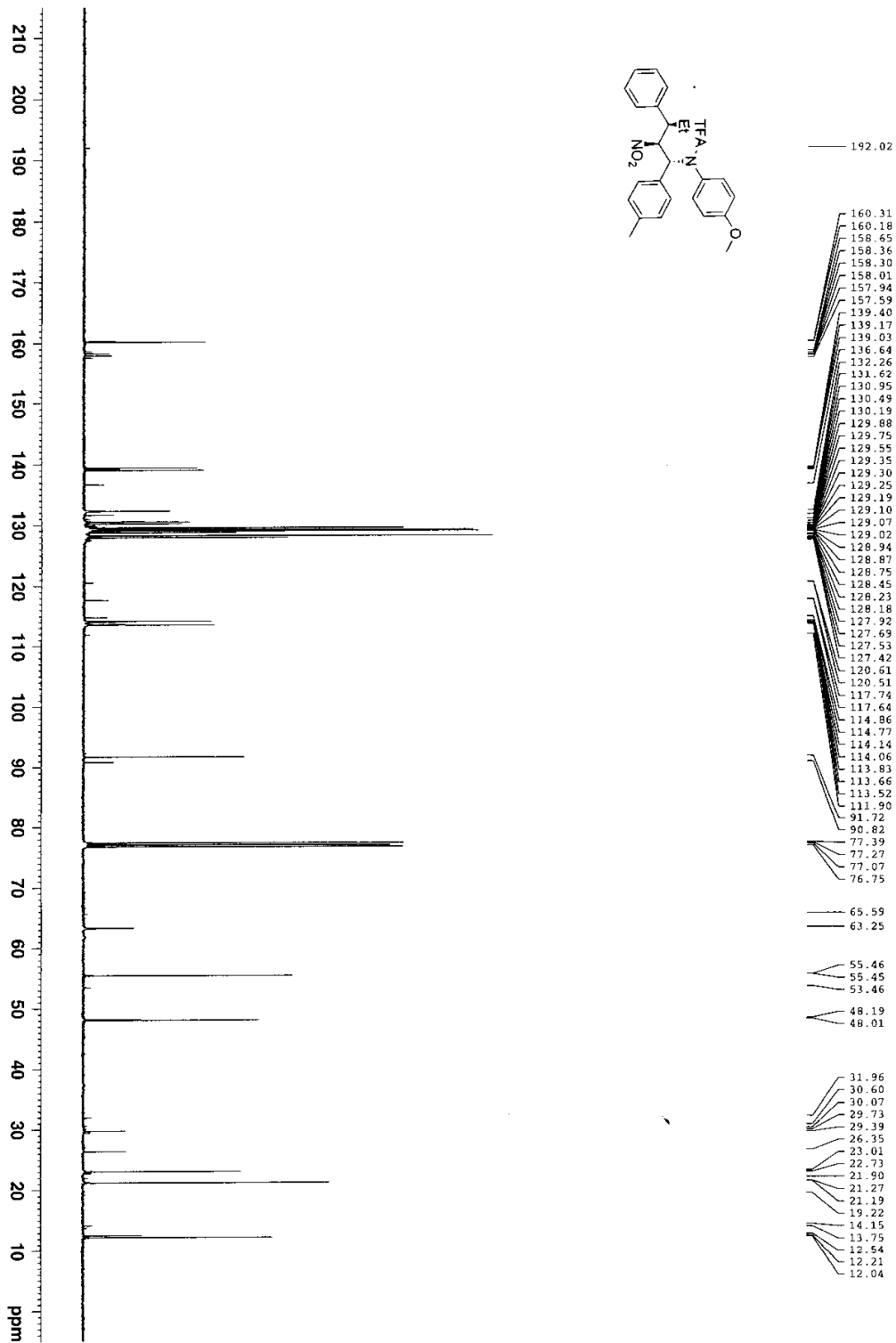


Current Data Parameters
NAME m_mil.mrm399
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080805
Time 10.04
INSTRUM dpx400
PROBHD 5 mm PABBO 2B/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DW 104.400 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz

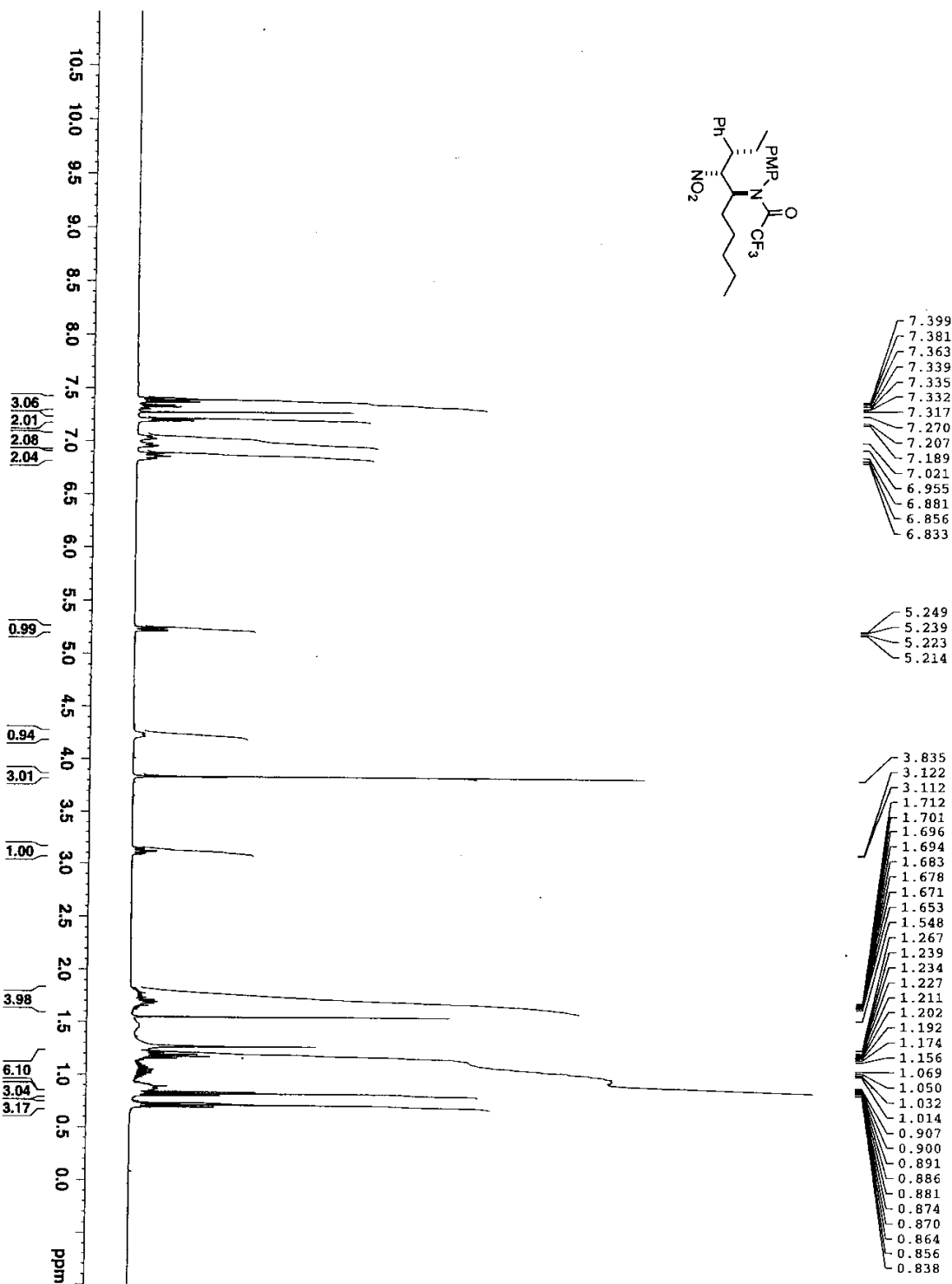
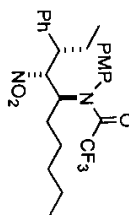
F2 - Processing parameters
SI 32768
SF 400.200087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters	
NAME	m_m1.mn399
EXFNO	4
EXPNO	1
P2 - Acquisition Parameters	
Date_	20080805
Time	23.03
INSTRUM	sp400
PROBHD	5 mm PABBO
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	4096
DS	25125.632
FIDRES	0.766773 Hz
AQ	0.653113 sec
RG	18390.4
DW	19.900 usec
DE	6.90 usec
TE	300.2 K
D1	1.00000000 sec
d11	0.03000000 sec
DELTA	0.89393938 sec
TD0	1
CHANNEL f1: =====	
NUC1	13C
P1	8.40 usec
PL1	-4.00 dB
SFO1	100.64143499 MHz
CHANNEL f2: =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-6.00 dB
PL12	1.00 dB
PL13	1.00 dB
SFO2	400.201608 MHz
P2 - Processing Parameters	
SI	32768
WDW	100.6307608 MHz
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

¹H NMR Entry 4

UserID m_mil SampleID mnm606 SupervisorID ander Lab Phone No. 3 Slot Number 49



7.399
7.381
7.363
7.339
7.335
7.332
7.317
7.270
7.207
7.189
7.021
6.955
6.881
6.856
6.833

5.249
5.239
5.223
5.214

3.835
3.122
3.112
1.712
1.701
1.696
1.694
1.683
1.678
1.671
1.653
1.548
1.267
1.239
1.234
1.227
1.211
1.202
1.192
1.174
1.156
1.069
1.050
1.032
1.014
0.907
0.900
0.891
0.886
0.881
0.874
0.870
0.864
0.856
0.838

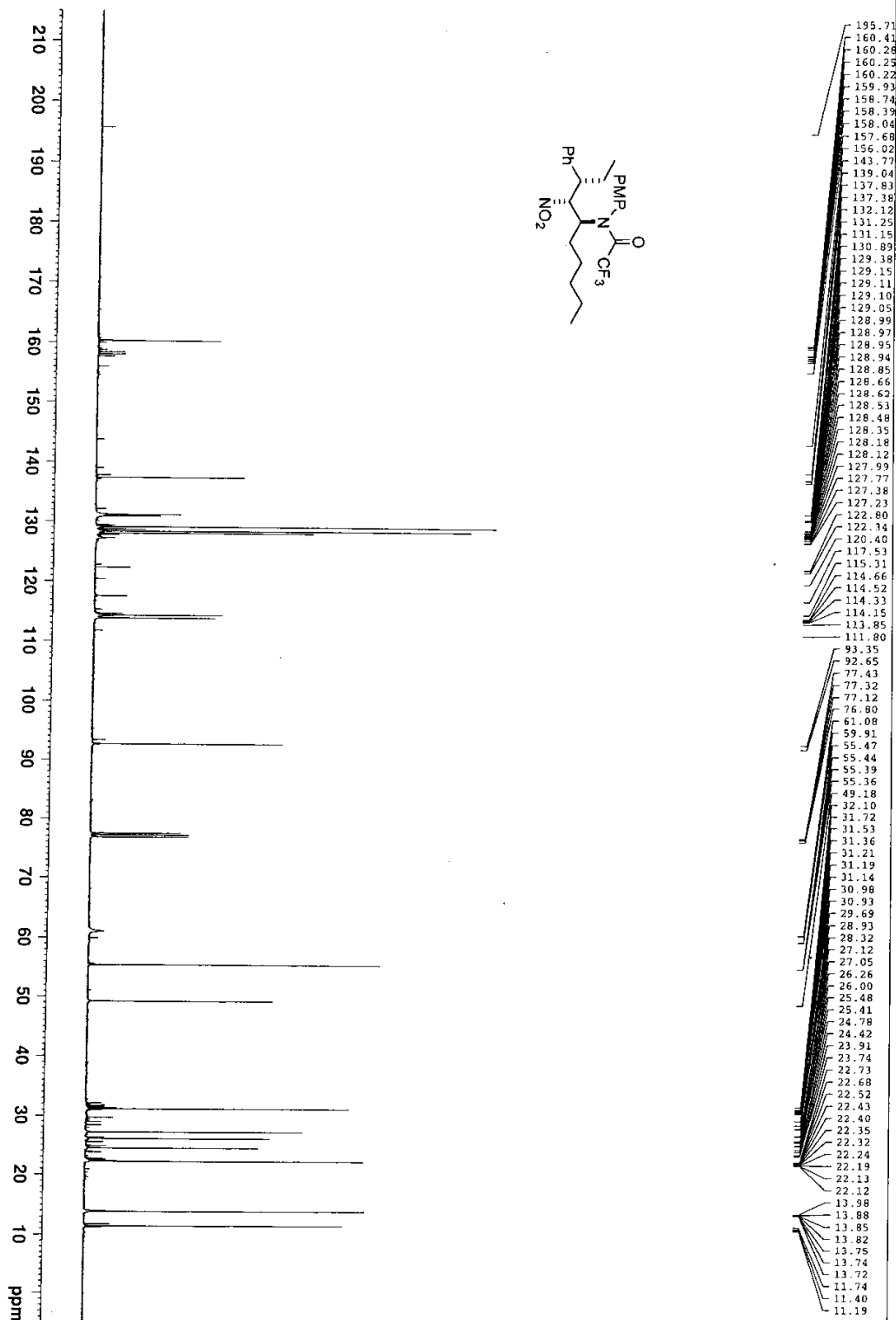
Current Data Parameters
NAME m_mil.mnm606
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081114
Time 14.36
INSTRUM av400
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.00 usec
PL1 -0.80 dB
PC1 11.52680206 W
SFO1 400.0724706 MHz

F2 - Processing parameters
SI 32768
SF 400.0700042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 12 Sample ID mrm600-1 SupervisorID ander Lab Phone No. 1 UserID m_mil



```

Current Data Parameters
NAME      m_mil.mrm600-1
EXPNO     2
PROCNO    1

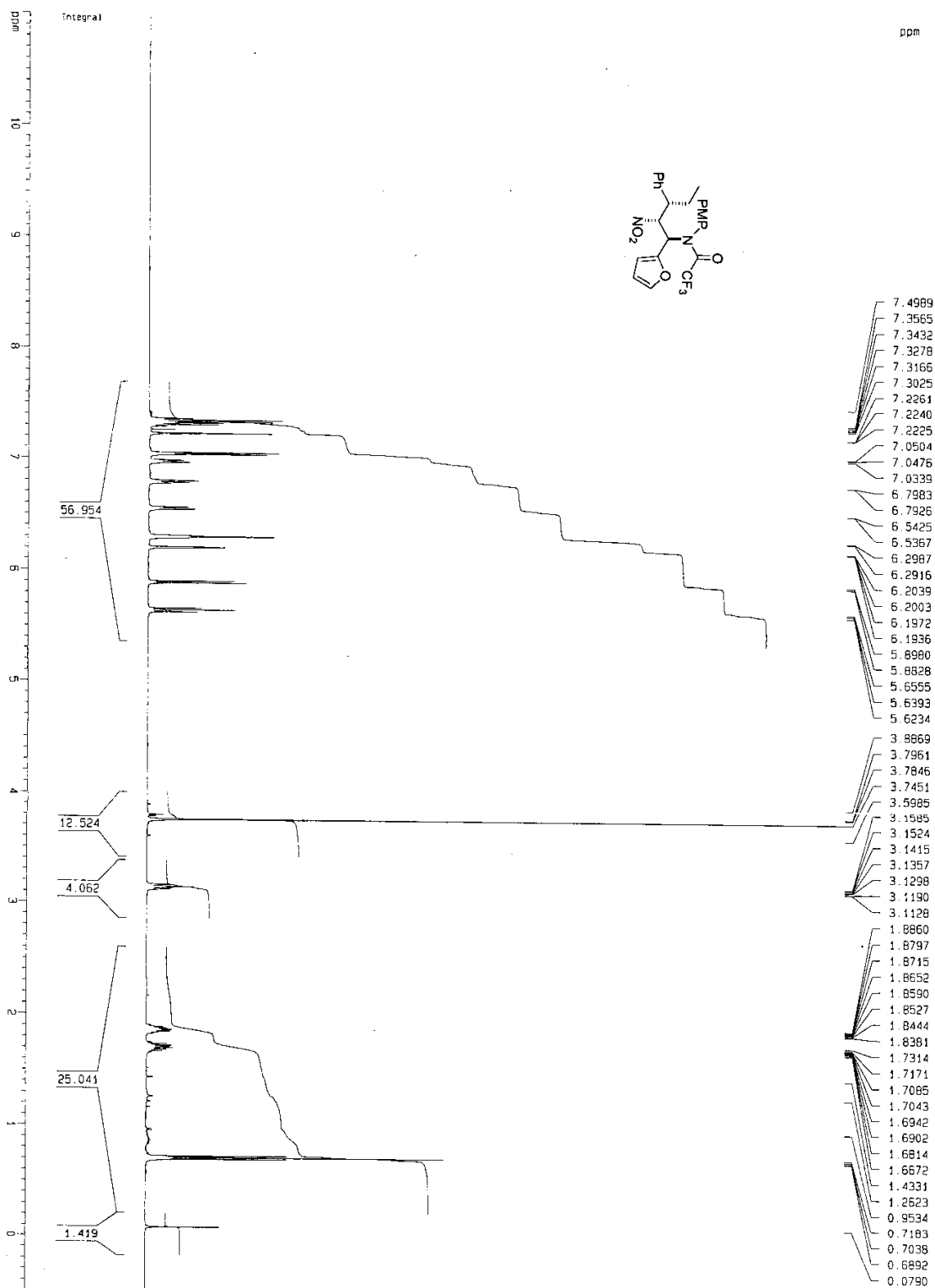
F2 - Acquisition Parameters
Date_     20081104
Time      0.17
INSTRUM   gp400
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2048
DS         2
SWH        25125.629 Hz
FIDRES     0.766773 Hz
AQ         0.145912 sec
RG         19.900 usec
DE         5.00 usec
TE         298.0 K
d11        1.0000000 sec
d12        0.1000000 sec
DELTA      0.8999998 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        -4.00 dB
SFO1       100.641390 MHz

===== CHANNEL f2 =====
NUC2       1H
P2         100.00 usec
PL2        -8.00 dB
SFO2       400.2016010 MHz

F2 - Processing parameters
SI         32768
SF         100.6303700 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

¹H NMR Entry 5



PROTON.uc1 CDCl3 v2 jca 32

Current Data Parameters
 NAME Jun30-2009
 EXPNO 101
 PROCNO 1

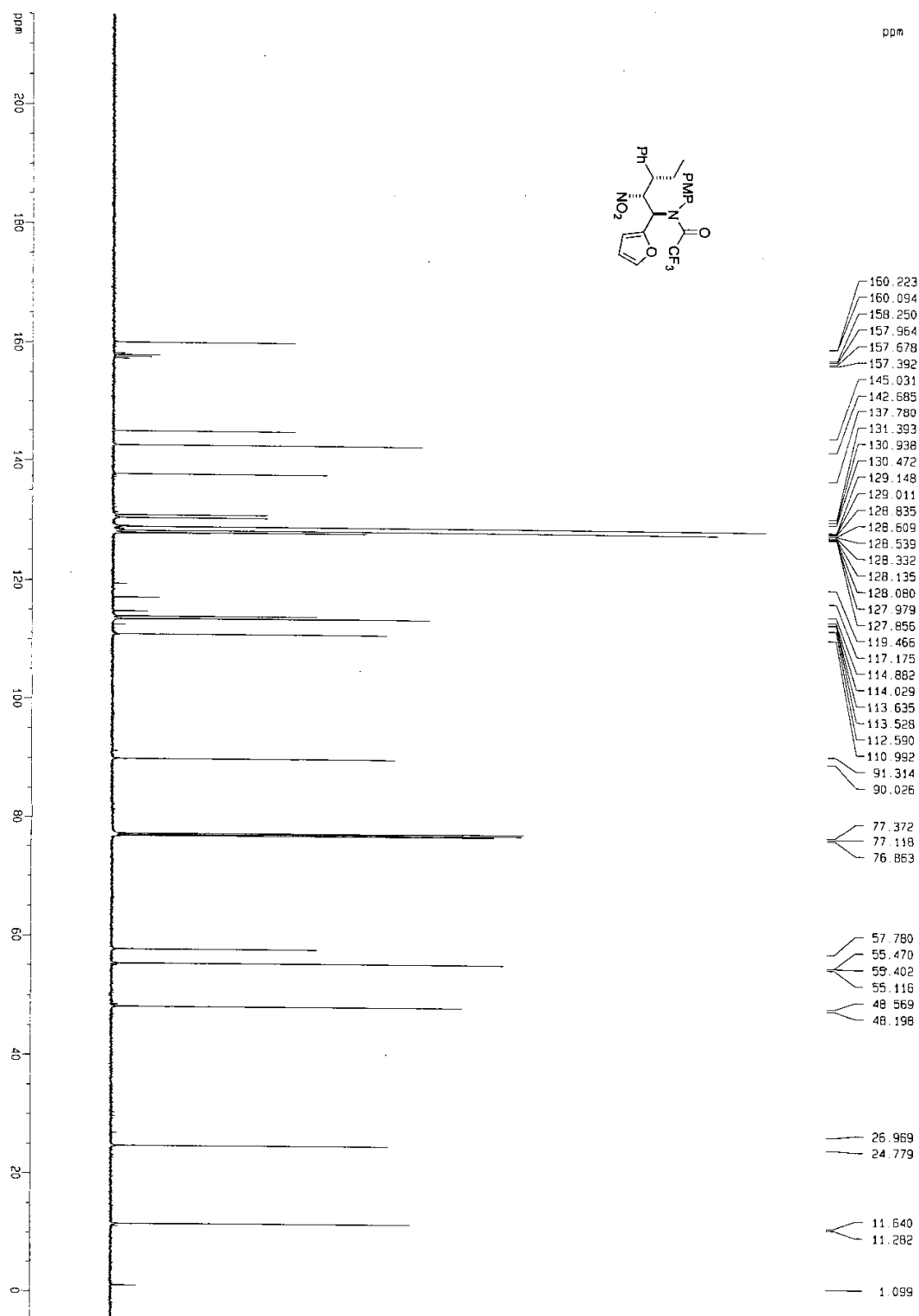
F2 - Acquisition Parameters
 Date_ 2009/07/01
 Time 0.13
 INSTRUM drx500
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.171923 sec
 RG 50.5
 OR 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.50 usec
 PL1 0.00 dB
 SFO1 500.130085 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300140 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 11.000 ppm
 F1 5501.43 Hz
 F2P -0.500 ppm
 F2 -250.06 Hz
 PPMCM 0.35937 ppm/cm
 HZCM 179.73421 Hz/cm

¹³C NMR Entry 5



mm687 furyl f13
c13.10ng CDCl3 v2 jca 32

Current Data Parameters
NAME Jun30-2009
EXPNO 103
PROCNO 1

F2 - Acquisition Parameters
Date_ 2009/07/01
Time 1.14
INSTRUM drx500
PROBHD 5 mm WJ11nv
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31446.541 Hz
FIDRES 0.479936 Hz
AQ 1.0420724 sec
RG 3649.1
DE 15.500 usec
TE 300.0 K
D1 2.00000000 sec
d12 0.0002000 sec

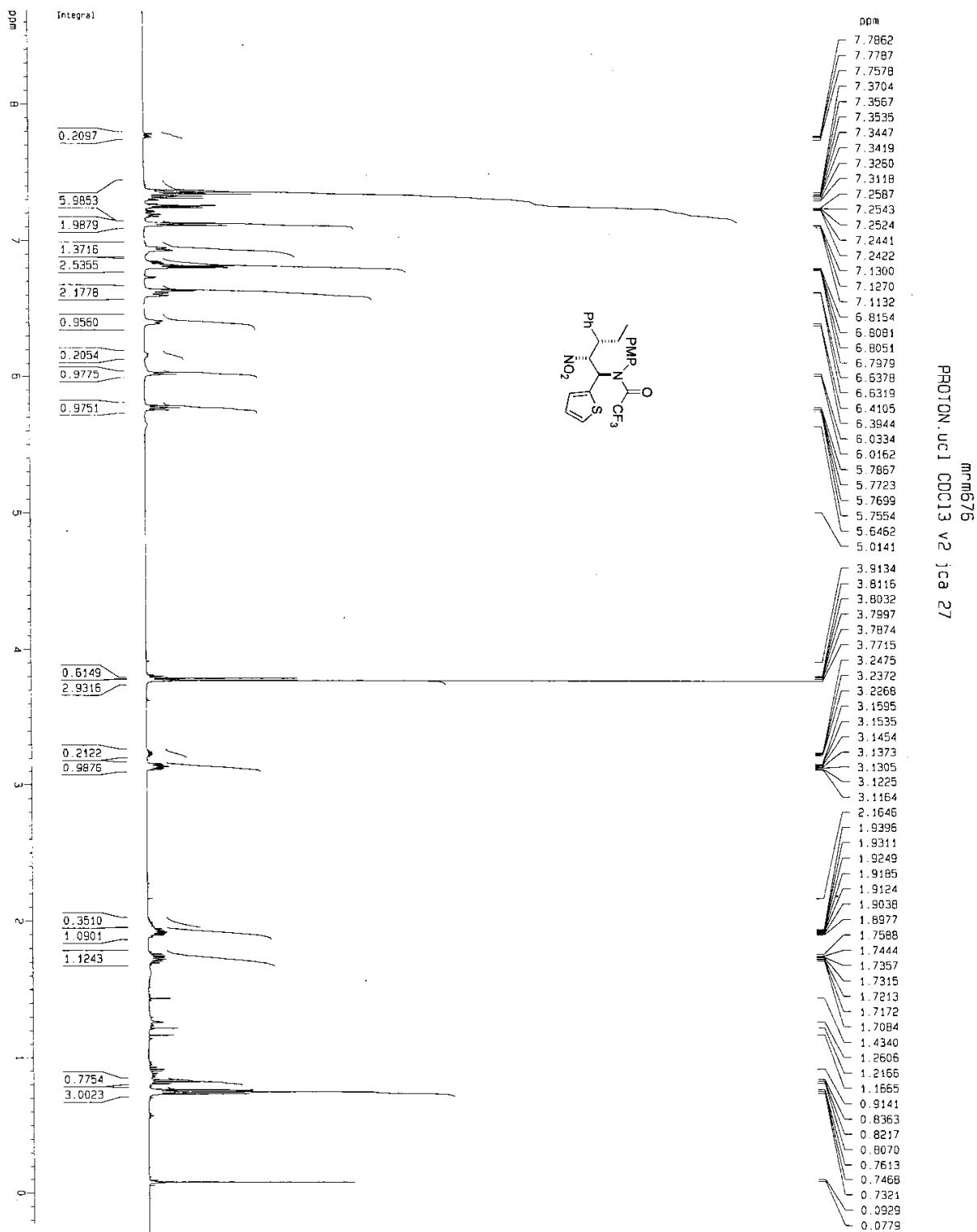
===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.7715719 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.132005 MHz

F2 - Processing parameters
SI 32768
SF 125.757814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
F1P 215.000 ppm
F1 27037.93 Hz
F2P -5.000 ppm
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 864.58478 Hz/cm

¹H NMR Entry 6



Current Data Parameters
 NAME: ju107-2009
 EXPNO: 22
 PROCNO: 1

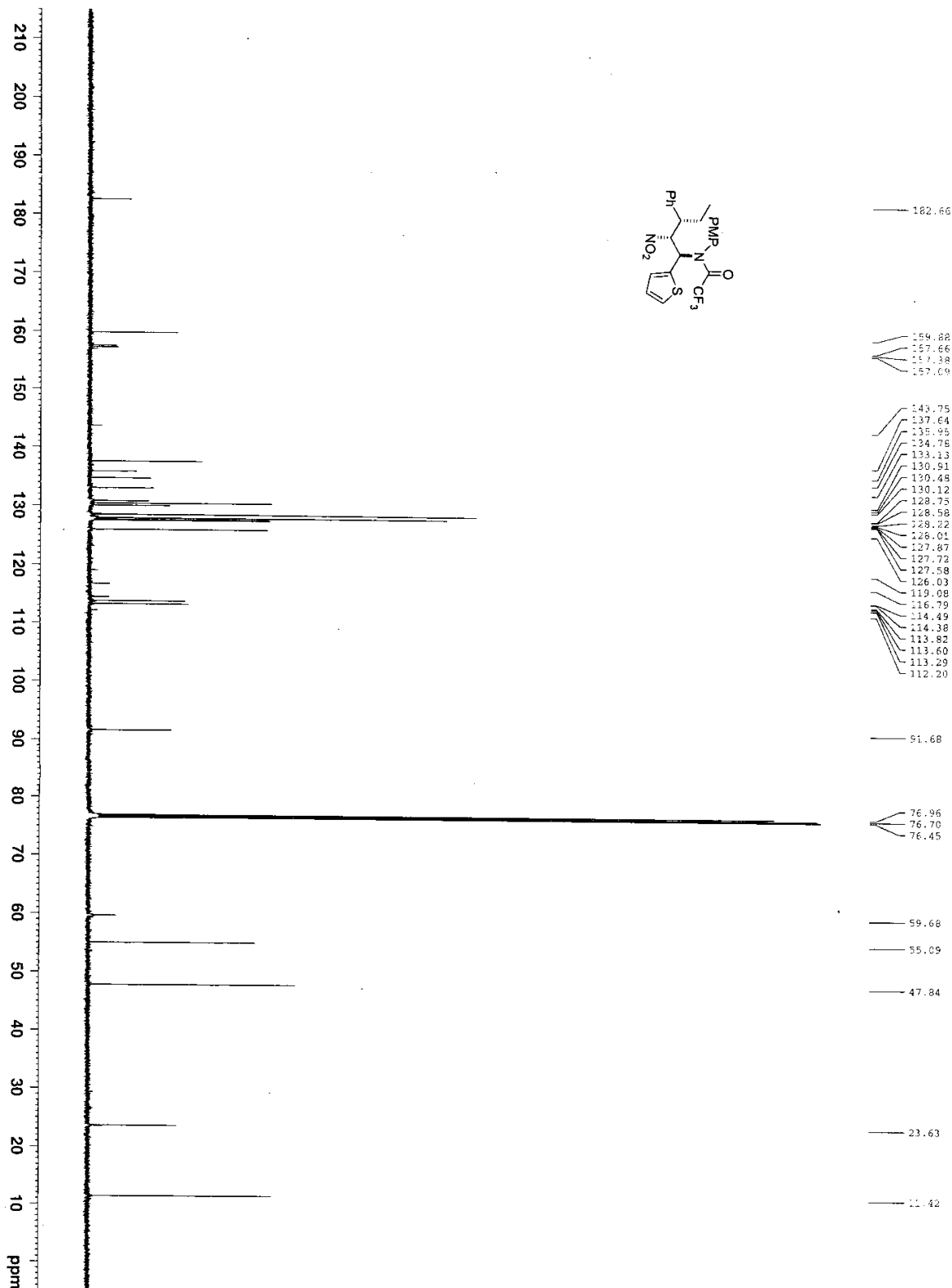
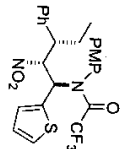
F2 - Acquisition Parameters
 Date_: 20090708
 Time: 3.24
 INSTRUM: drx500
 PROBHD: 5 mm Multinu
 PULPROG: zg30
 TO: 65536
 SOLVENT: CDCl3
 NS: 16
 DS: 0
 SWH: 10330.578 Hz
 FIDRES: 0.157632 Hz
 AQ: 3.1719923 sec
 RG: 90.5
 DW: 48.400 usec
 DE: 5.00 usec
 TE: 300.0 K
 D1: 1.00000000 sec

===== CHANNEL f1 =====
 NUC1: ¹H
 P1: 15.50 usec
 PL1: 0.00 dB
 SF01: 500.130085 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.1300140 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

10 NMR plot parameters
 CX: 32.00 cm
 F1P: 8.683 ppm
 F1: 4342.65 Hz
 F2P: -0.302 ppm
 F2: -151.14 Hz
 PPMCH: 0.28079 ppm/cm
 HZCM: 140.43115 Hz/cm

mrm11
C13CPD CDC13 v2 jca 51



182.66
159.88
157.66
157.38
157.00
143.75
137.64
135.95
134.78
133.13
130.91
130.48
130.12
128.75
128.58
128.22
128.01
127.87
127.72
127.58
126.03
119.08
116.79
114.49
114.38
113.82
113.60
113.29
112.20
91.68
76.96
76.70
76.45
59.68
55.09
47.84
23.63
11.42



Current Data Parameters
NAME Decol-2009
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091201
Time 14.03
INSTRUM 5 mm Multinuc1
PROBHD zgpg30
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 9195.2
DM 16.650 use
DE 6.00 use
TE 298.0 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
MCREST 0.0000000 sec
MCWRK 0.0150000 sec

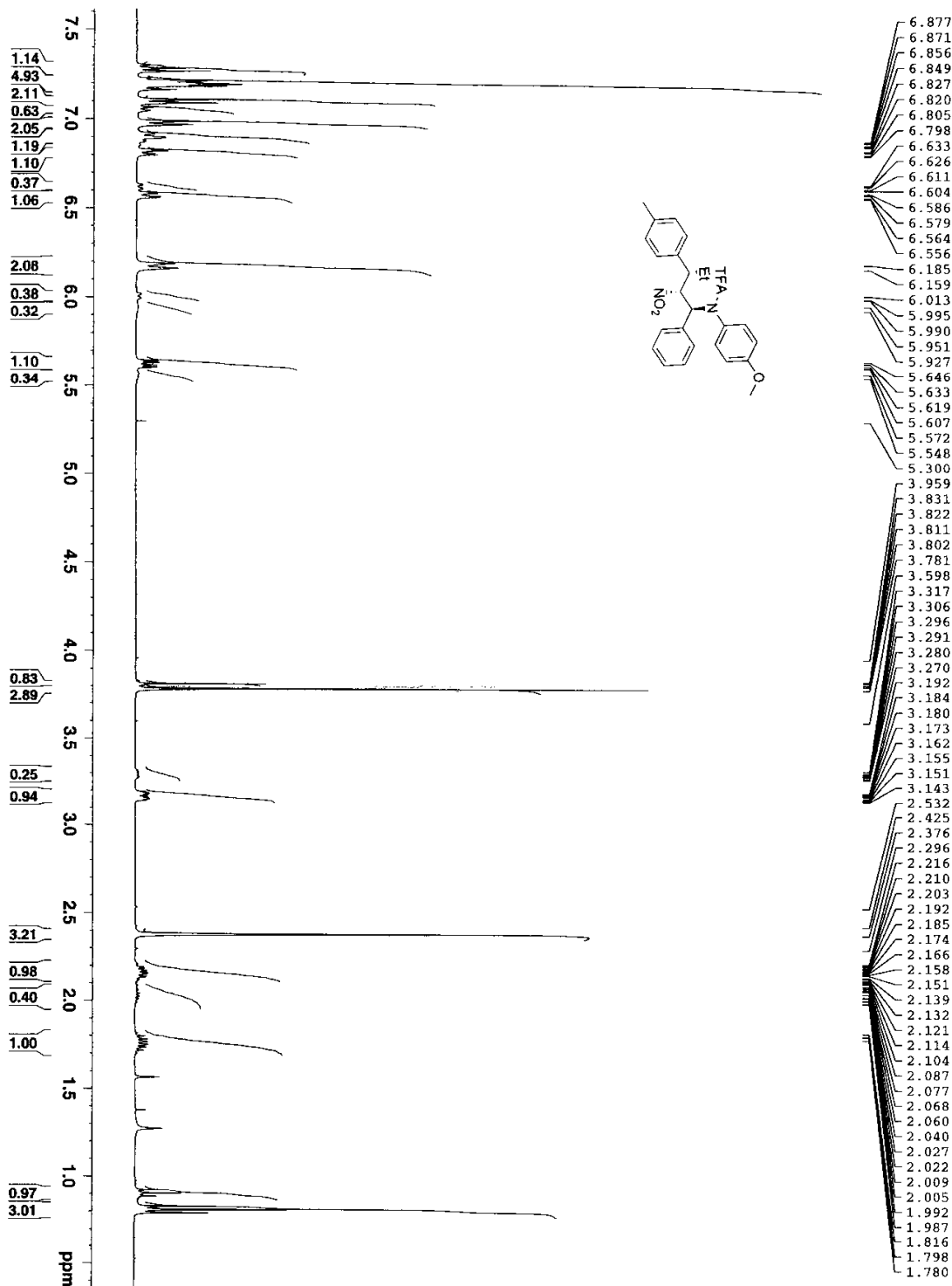
===== CHANNEL f1 =====
NUC1 13C
P1 5.50 use
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 use
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578314 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Entry 7

Slot No. 41 Sample ID mnm410 SupervisorID ander Lab Phone No. 5 UserID m.mil



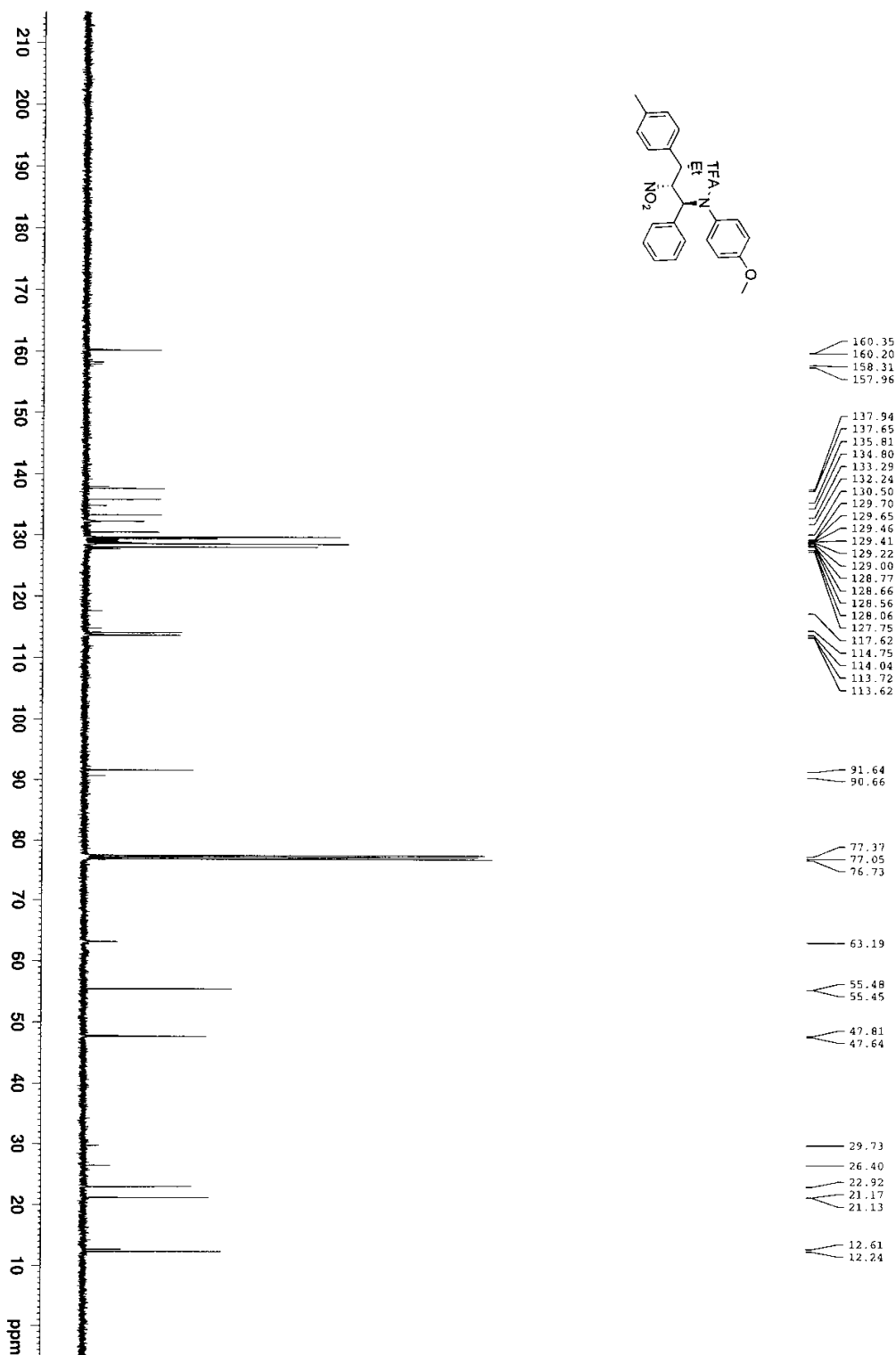
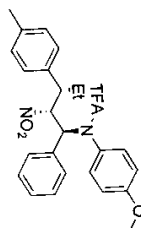
Current Data Parameters
NAME m.mil.mnm410
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080807
Time 13.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 203.2
DW 104.400 usec
DE 6.00 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz

F2 - Processing parameters
SI 32768
SF 400.2000103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 41 Sample ID mrm410 Supervisord ander Lab Phone No. 5 UserID m_mil



```

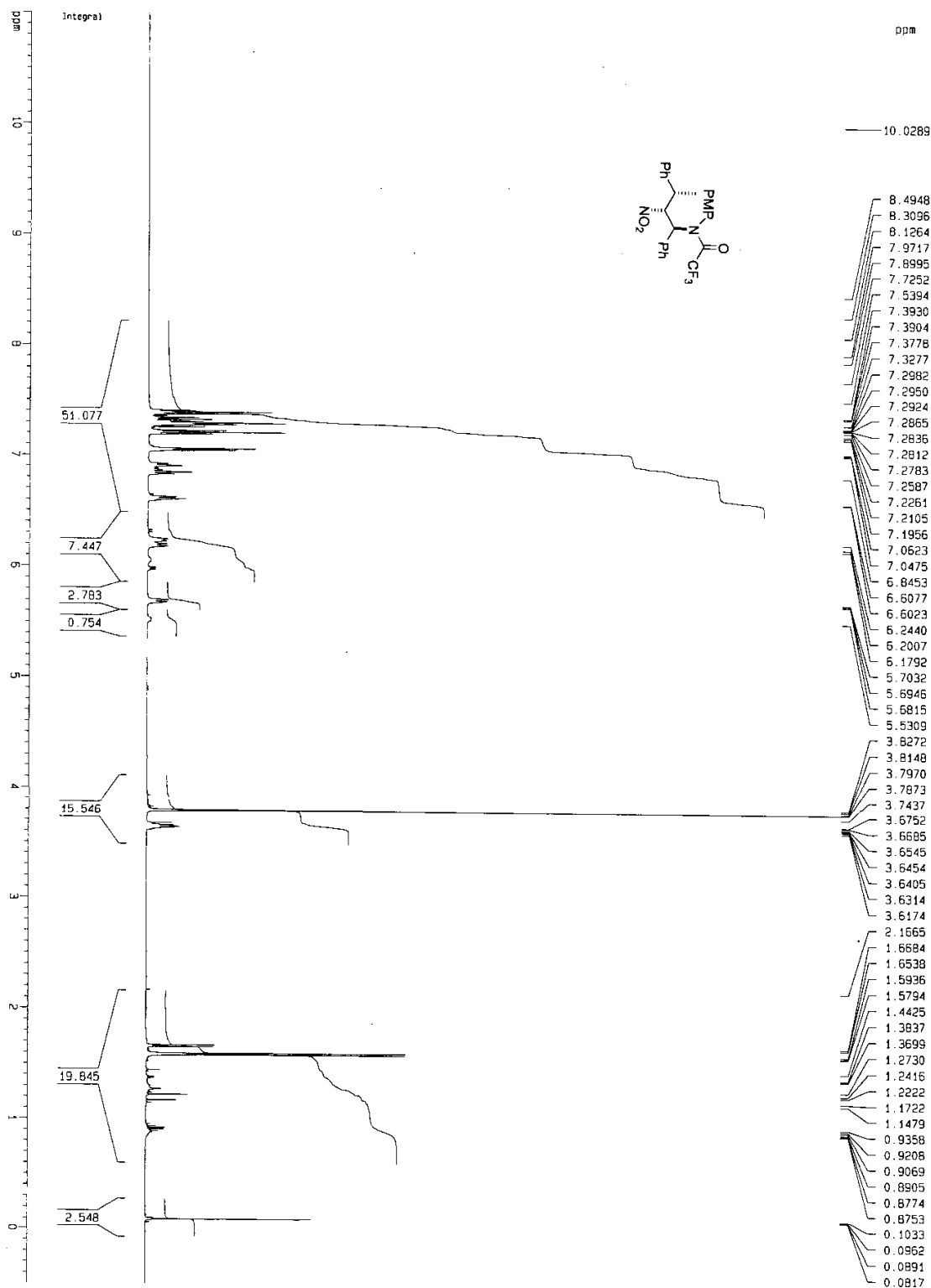
Current Data Parameters
=====
Date_ 20080807
Time_ 12:41
Instrum_ spect
PROBHD 5 mm PABO Bb/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 312
DS 4
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 1.0004
RG 19200
DE 19.400 usec
TE 297.5 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999996 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 0.00 dB
SFO1 100.624433 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2015008 MHz

F2 - Processing parameters
SI 32768
SF 100.6303700 MHz
WDW EM
SSB 0
GB 1.00 Hz
PC 1.40
  
```

¹H NMR Entry 8



nmr686
PROTON.uc1 CDCl3 v2 jca 28

Current Data Parameters
NAME J0107-2009
EXPNO 32
PROCNO 1

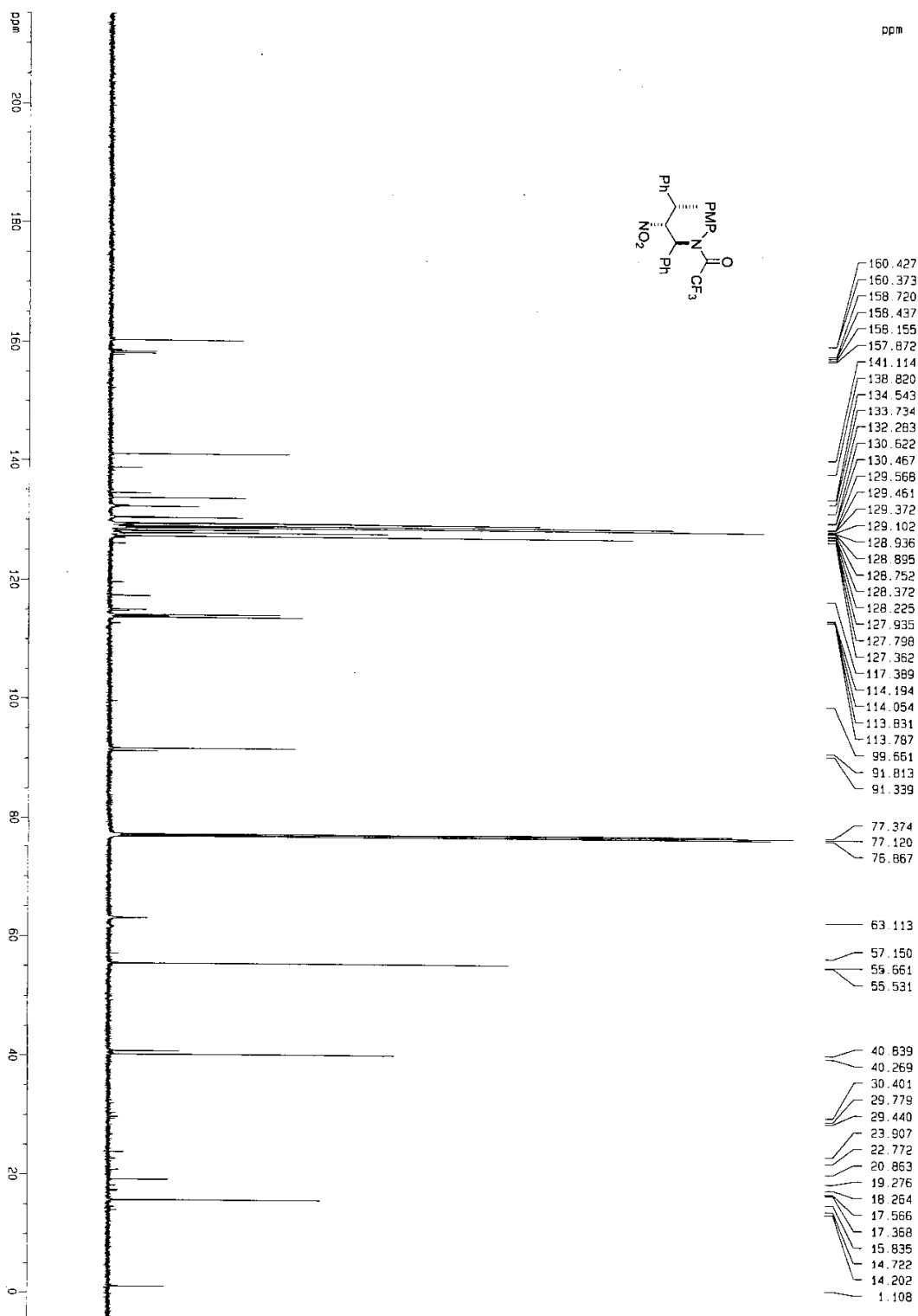
F2 - Acquisition Parameters
Date_ 20090708
Time 5:29
INSTRUM drx500
PROBHD 5 mm MULETHU
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 90.5
DM 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
SFO1 500.130085 MHz

F2 - Processing parameters
SI 65536
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 11.000 ppm
F1 5501.43 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCK 0.35938 ppm/cm
HZCM 179.73422 Hz/cm

¹³C NMR Entry 8



mrm686
C13.1ong CDCl3 v2 jca 28

Current Data Parameters
NAME Jul07-2009
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090708
Time 5.00
INSTRUM drx500
PROBHD 5 mm MUltinu
PULPROG zgpg30
TO 6536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 3649.1
DM 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.7715719 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

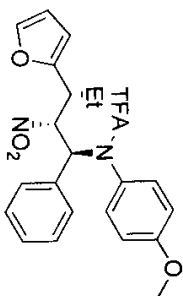
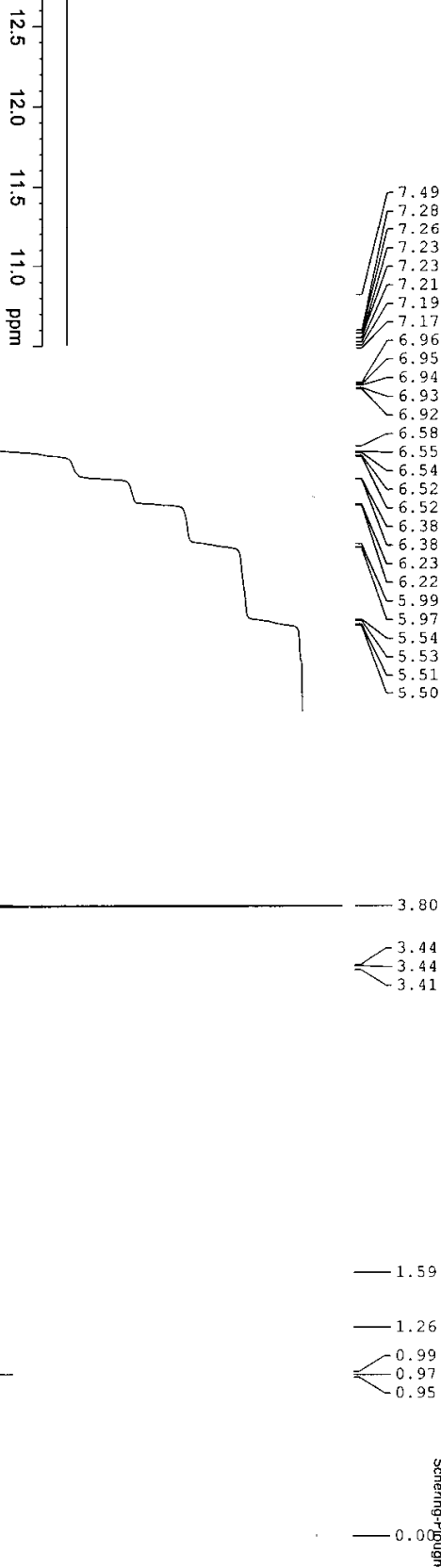
1D NMR plot parameters
CX 32.00 cm
FIP 215.000 DDM
F1 27037.93 Hz
F2 -5.000 DDM
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 864.58476 Hz/cm

¹H NMR Entry 9

Sample Code mrm 405 - data



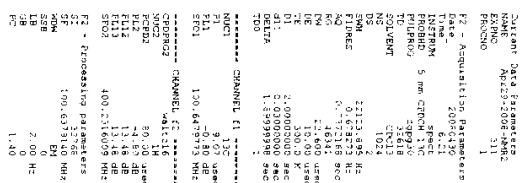
Current Data Parameters
 Name: mrm405
 EXNO: 20
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_ Time: 20060401 8:40
 INSTRUM: spect
 PROBD: 5 mm CPDCH 13C
 T2: 2.95
 T2RHO: 329.6
 T2RHO2: 329.6
 SOLVENT: CDCl3
 NS: 8
 DS: 8
 SWH: 9250.822 Hz
 FIDRES: 0.251795 Hz
 AQ: 1.9857908 sec
 RG: 812.7
 RM: 60.600 uSAC
 TD: 32768
 TE: 300.0 K
 D1: 1.00000000 sec
 TDO: 1
 CHANNEL: f1
 NUC1: 1H
 P1: 9.75 uSAC
 PL1: -8.80 dB
 SFO1: 400.238015 MHz
 F2 - Processing Parameters
 SI: 16384
 SF: 400.230085 MHz
 WDW: EM
 SSF: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00



Organon

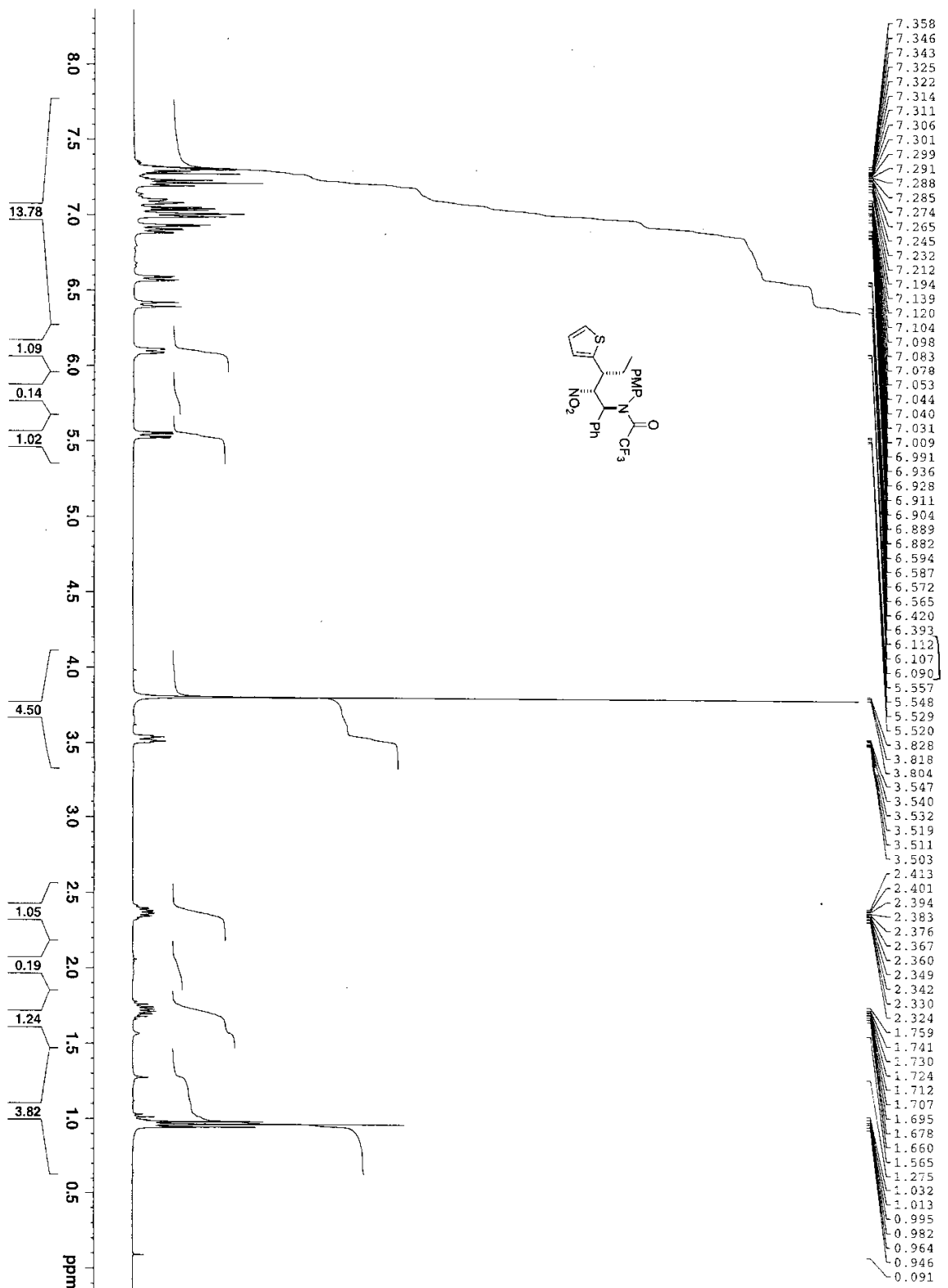
The dendrogram illustrates the hierarchical clustering of 20 Schering products. The y-axis lists the products, and the x-axis represents the distance between clusters. The products are clustered into several groups, with the most significant cluster containing 18 products at a distance of 100 or less. Two products, 160.31 and 158.65, are outliers at a distance of approximately 150. The remaining products are clustered at distances of 100 or less, with some products like 157.94 and 157.59 being very close to each other.

Schering	Distance
160.31	150.0
158.65	150.0
158.29	100.0
157.94	100.0
157.59	100.0
152.04	100.0
142.31	100.0
133.37	100.0
132.95	100.0
130.48	100.0
129.44	100.0
128.99	100.0
128.80	100.0
128.66	100.0
126.46	100.0
120.50	100.0
117.63	100.0
114.76	100.0
113.84	100.0
113.81	100.0
111.90	100.0
110.72	100.0
108.25	100.0
88.80	100.0
77.36	100.0
77.24	100.0
77.04	100.0
76.72	100.0
59.98	100.0
55.45	100.0
41.86	100.0
29.71	100.0
24.98	100.0
22.71	100.0
21.20	100.0
14.15	100.0
12.50	100.0
0.01	100.0



¹H NMR Entry 10

mrm674

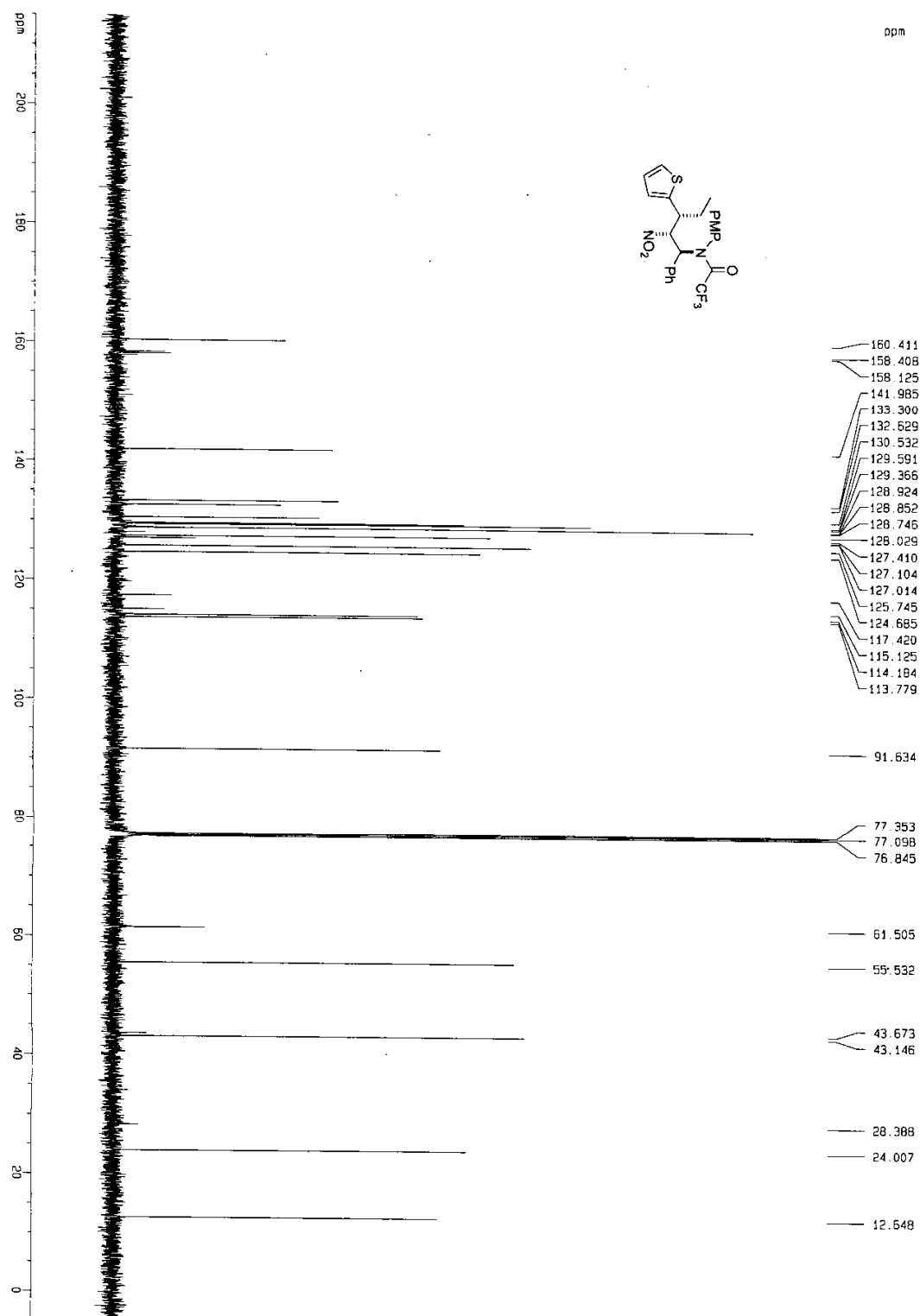


```

NAME mrm674
EXPNO 1
PROCNO 1
Date_ 20090612
Time 15.45
INSTRUM av400
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 823.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 45.2
DM 60.800 use
DE 16.52 use
TE 298.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 use
PL1 0.00 dB
PL1W 13.8153753 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WPM 0
SRB 0
GB 0
PC 1.40
  
```

¹³C NMR Entry 10



mrm674
C13CPD.uc1 CDC13 v3 jca 1

Current Data Parameters
NAME Jun12-2009
EXPNO 73
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090612
Time 20.08
INSTRUM drx500
PROBHD 5 mm WALTIN
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 31446.541 Hz
FIDRES 0.479636 Hz
AQ 1.0420724 sec
RG 3649.1
DM 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

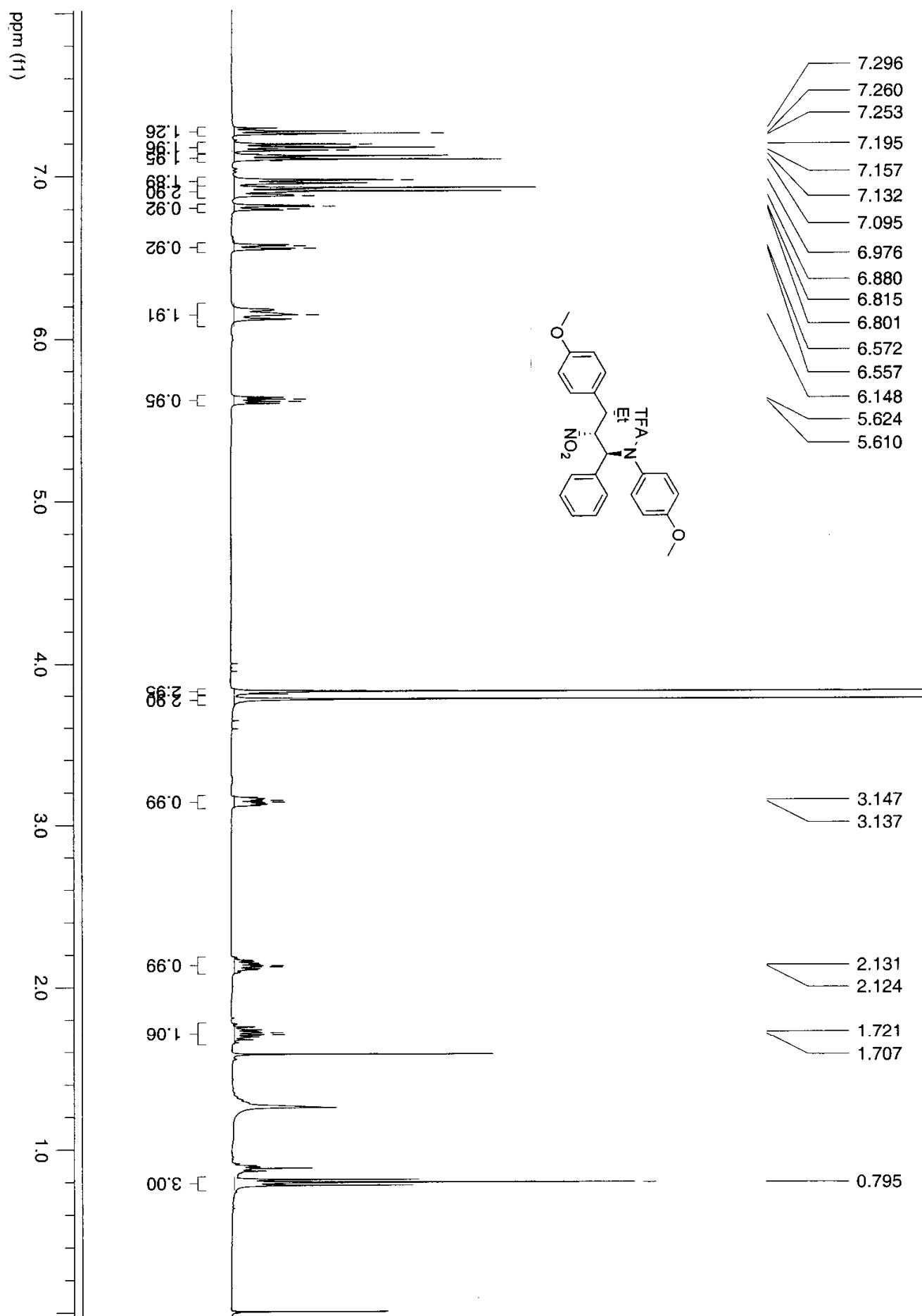
===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.7715719 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

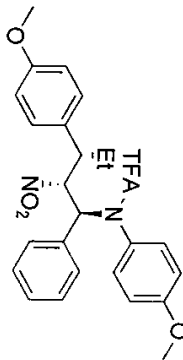
F2 - Processing Parameters
SI 32768
SF 125.7577614 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
F1P 215.000 ppm
F1 27037.53 Hz
F2P -5.000 ppm
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 854.58478 Hz/cm

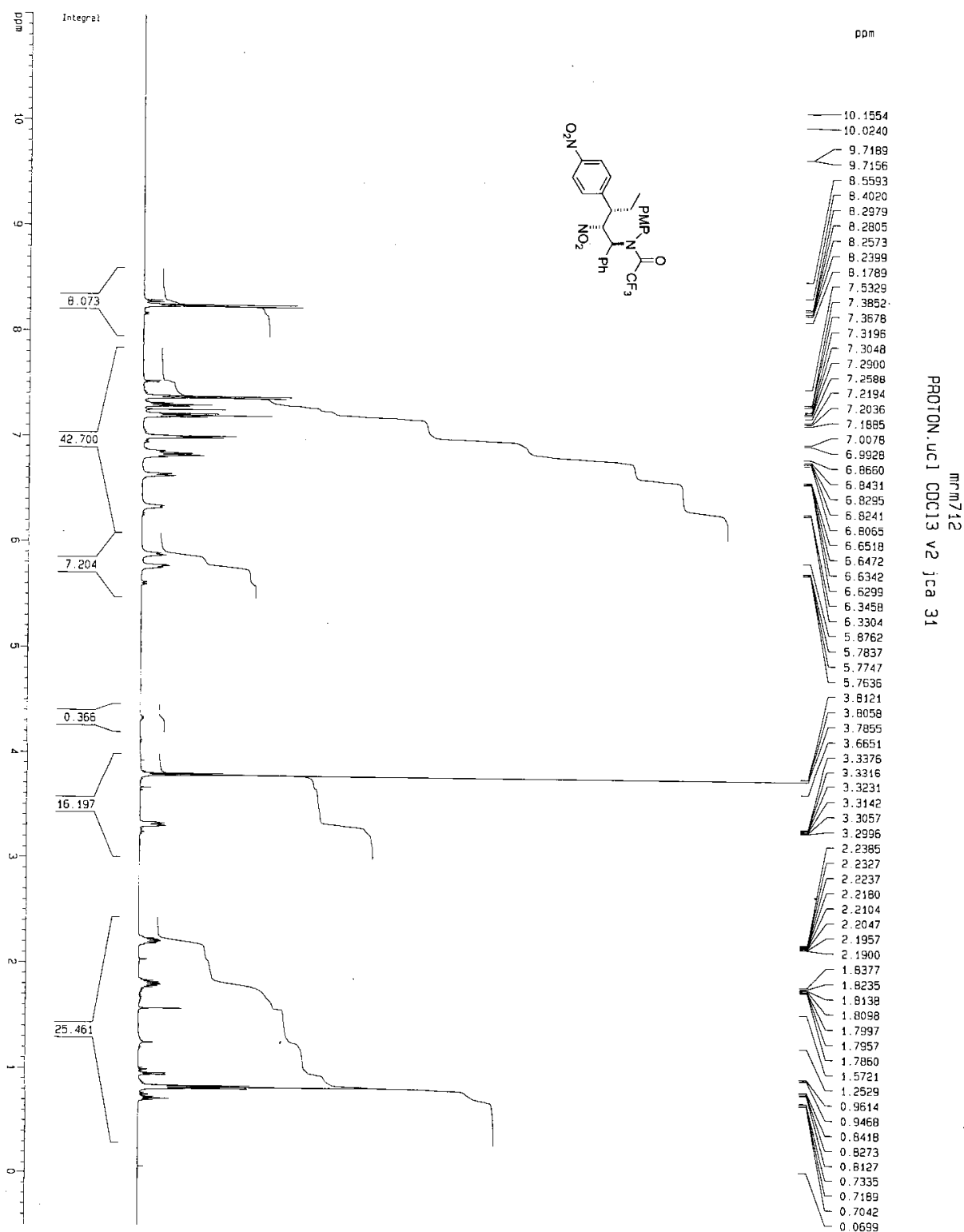
¹H NMR Entry 11



Schering-Plough

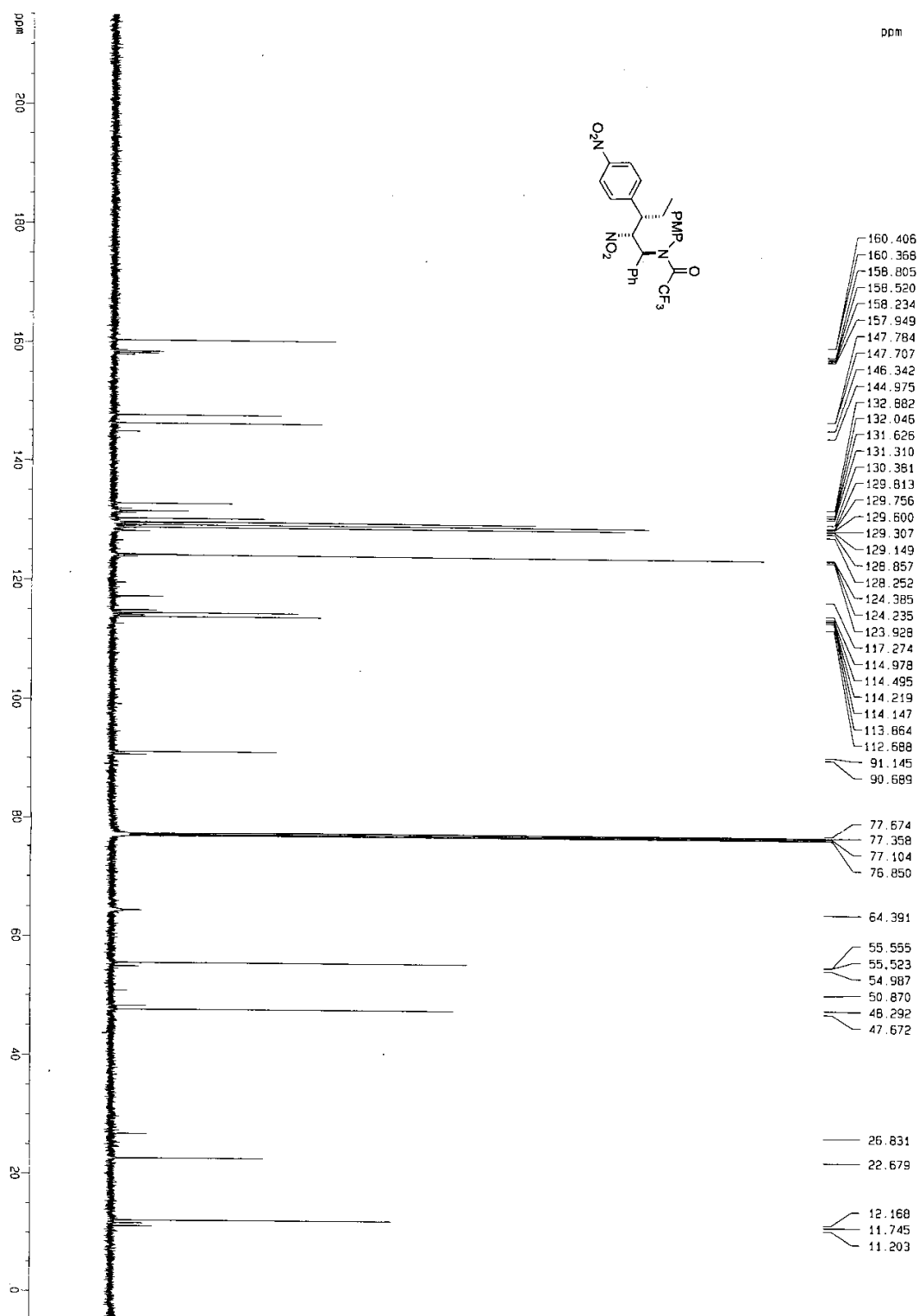


¹H NMR Entry 12



Current Data Parameters	
NAME	Jul30-2009
EXPNO	10
PROCNO	1
F2 - Acquisition Parameters	
Date_	20090730
Time	14.38
INSTRUM	0r5000
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65556
SOLVENT	CDCl3
NS	16
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719993 sec
RG	181
DM	48.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
***** CHANNEL f1 *****	
NUC1	1H
P1	15.50 usec
PL1	0.00 dB
PO1	500.1300885 MHz
F2 - Processing parameters	
SF	65536
SI	500.1300140 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00
10 NMR plot parameters	
CX	32.00 cm
F1P	11.000 ppm
F1	5501.43 Hz
F2P	-0.500 ppm
F2	-250.07 Hz
PPMCM	0.35038 ppm/cm
HZCM	179.73422 Hz/cm

¹³C NMR Entry 12



mm712
C13-10mg CDC13 v2 jca 31

Current Data Parameters
NAME JUL30-2009
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090731
Time 8.03
INSTRUM grx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 3649.1
DM 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

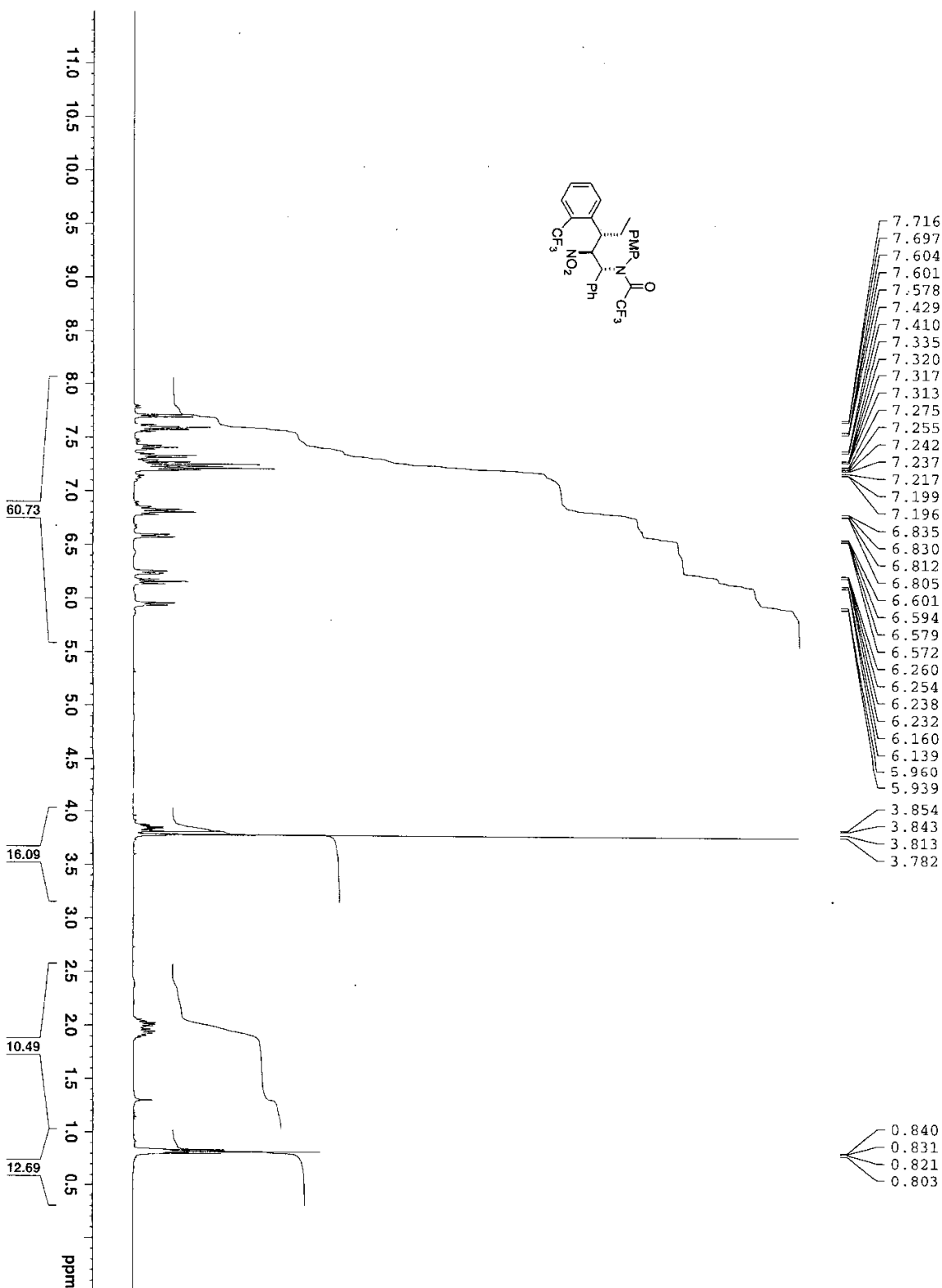
===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SF01 125.7715719 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.1320095 MHz

F2 - Processing parameters
SI 32768
SF 125.7577814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
F1P 215.000 ppm
F1 2703.93 Hz
F2P -5.000 ppm
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 864.56478 Hz/cm

Slot No. 47 Sample ID mmm407 SupervisorID ander Lab Phone No. 1 UserID m_mil

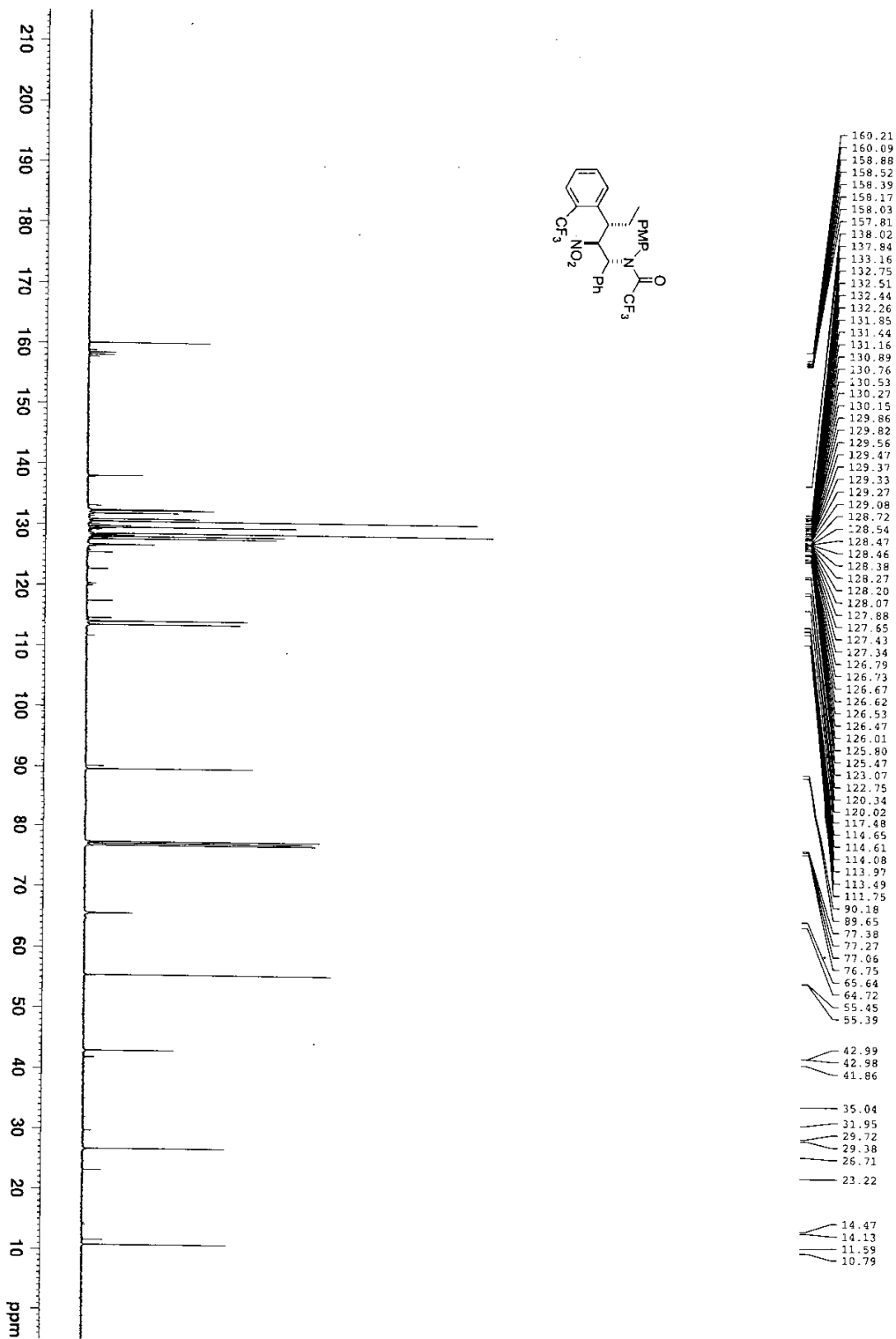


Current Data Parameters
NAME m_mil.mmm407
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080805
Time 10.20
INSTRUM dpx400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DE 104.400 use
TE 296.7 K
D1 1.0000000 sec
TD0 1

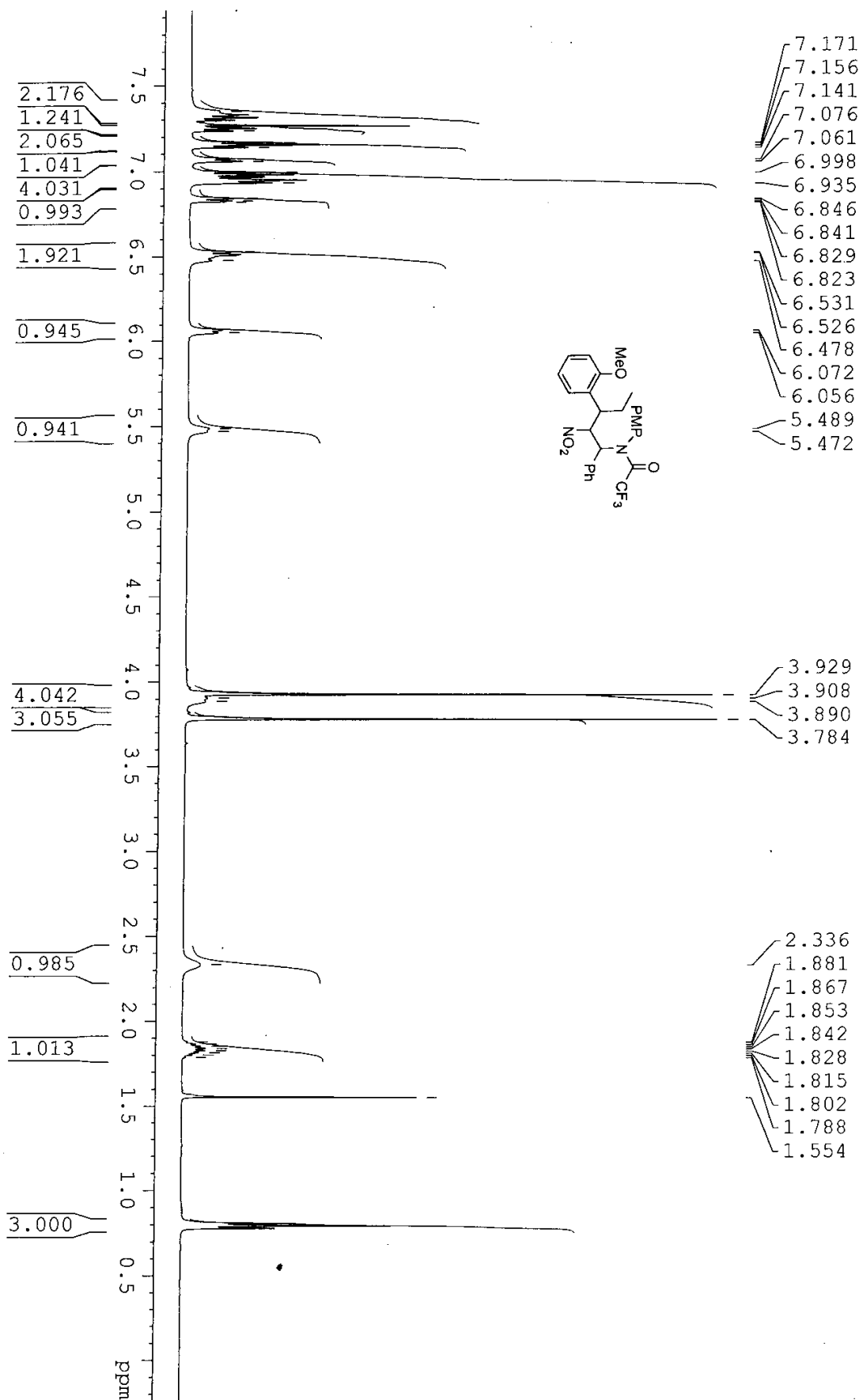
===== CHANNEL f1 =====
NUC1 1H
P1 13.40 use
PL1 6.00 dB
SFO1 400.2022011 MHz

F2 - Processing parameters
SI 32768
SF 400.2000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

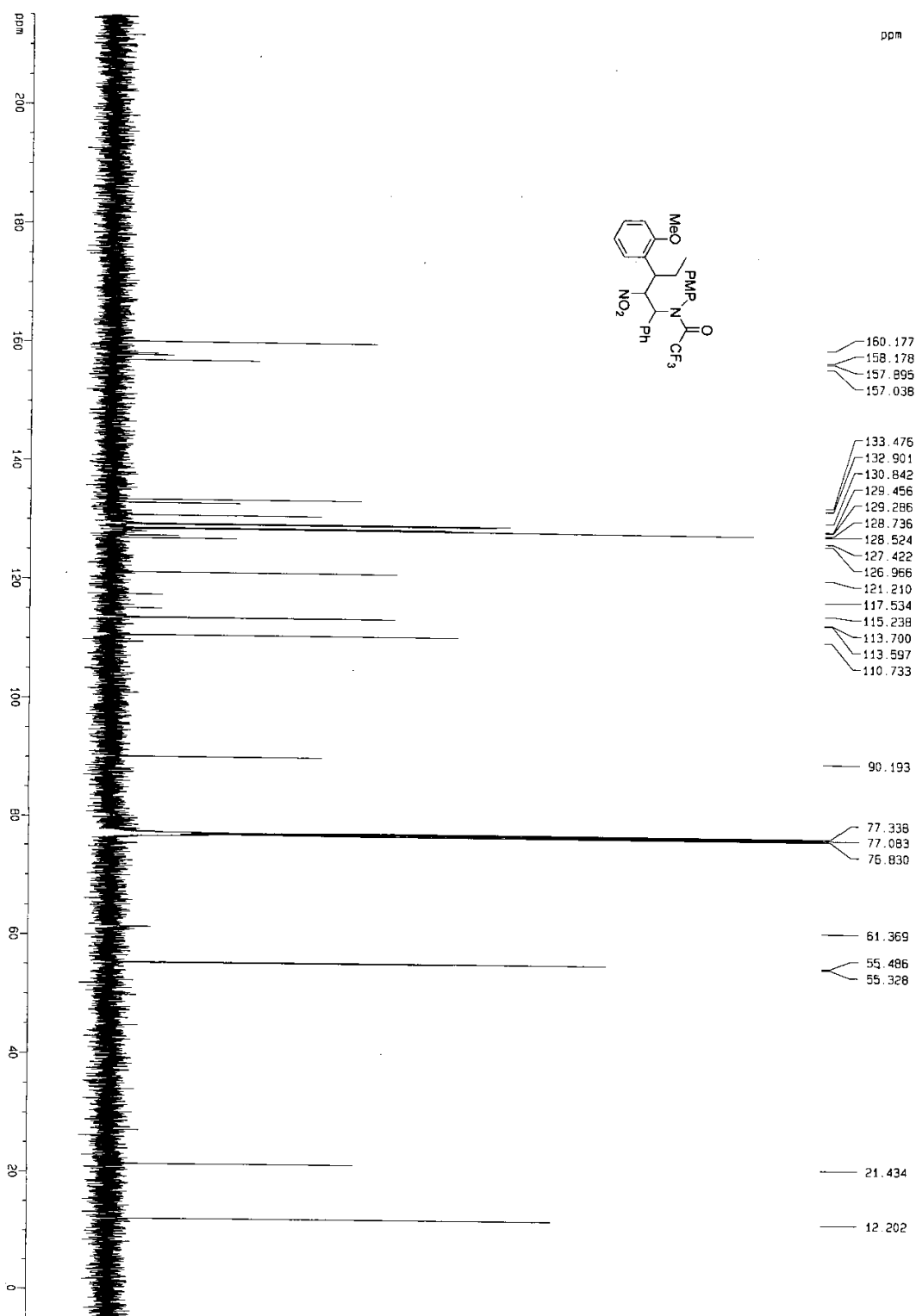
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Current Data Parameters	
NAME	m_mill.mn0407
EXNO	1
PRNO	1
File - Acquisition Parameters	
Date -	20080806
Time	5, 10
INSTRUM	dp4-400
PULPROG	5 mm PABBO H2O
TD	32768
SOLVENT	CDCl3
NS	4096
DS	2
SWH	253.6428 Hz
FIDRES	0.746600 Hz
AQ	0.6521137 sec
RG	16384
DM	19.90 sec
DE	6.90 usec
TE	300.2 K
DETEM	1.00000000 sec
d11	0.03000000 sec
d12	0.03000000 sec
DELTA	0.89393999 sec
TD0	1
NAME	CHANNEL F1
NUC1	13C
P1	8.40 usec
P11	-4.00 usec
SP01	100.634393 Mhz
NAME	CHANNEL F2
CPDPRG2	mz13c13
NUC2	1H
PCPD2	100.00 usec
P2	-5.00 usec
P12	1.00 usec
P111	1.00 usec
SP02	400.261608 Mhz
P2 - Processing parameters	
SF	327.68 Mhz
MDW	100.6301900 Mhz
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

¹H NMR Entry 14



¹³C NMR Entry 14



mm706-1-0-OMe
C13.10mg CDCl3 v2 jca 2

Current Data Parameters
NAME Jul29-2009
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090723
Time 17.38
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 4597.6
DM 15.500 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

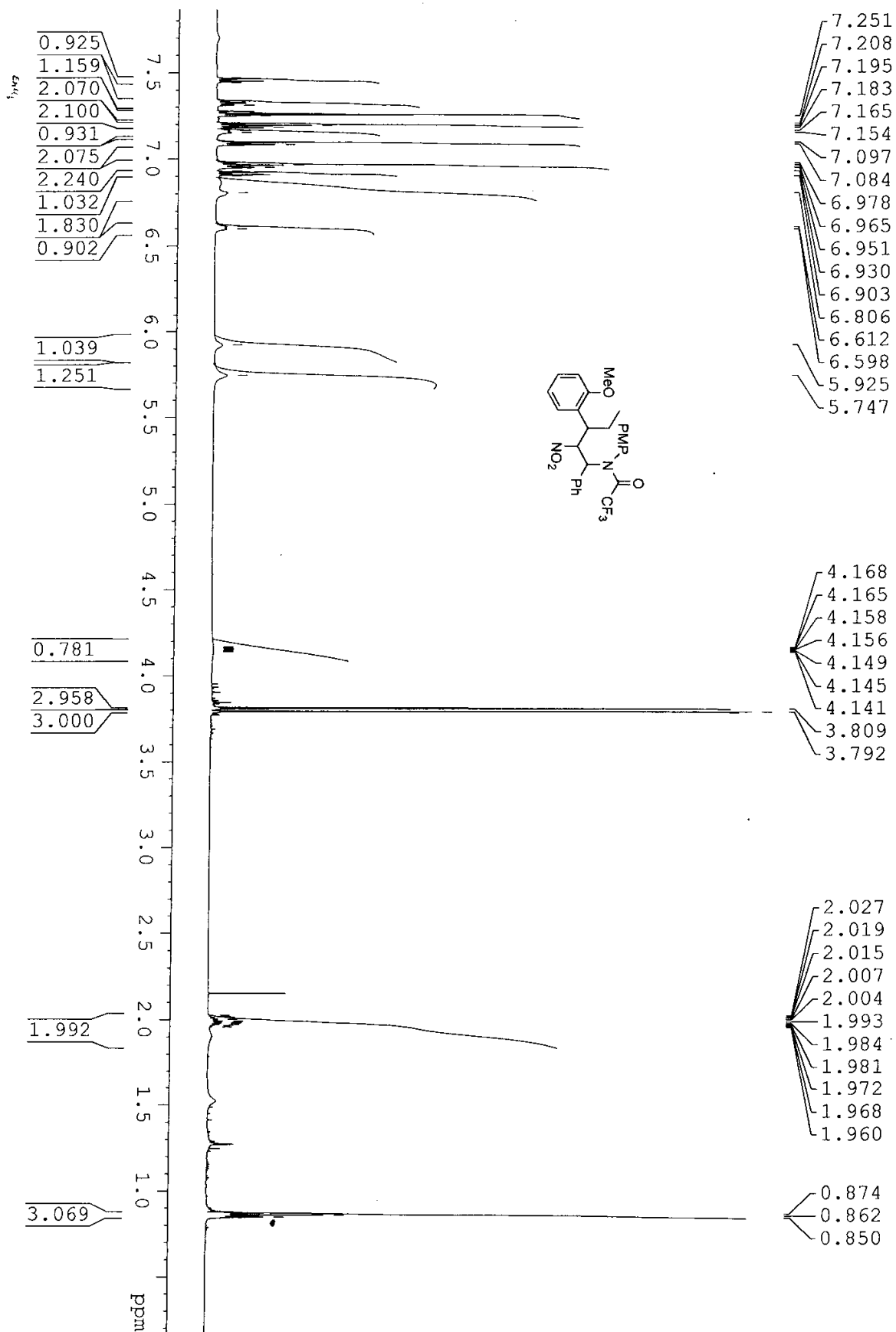
===== CHANNEL f1 =====
NUC1 ¹³C
P1 5.50 usec
PL1 0.00 dB
SF01 125.7715719 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.1320005 MHz

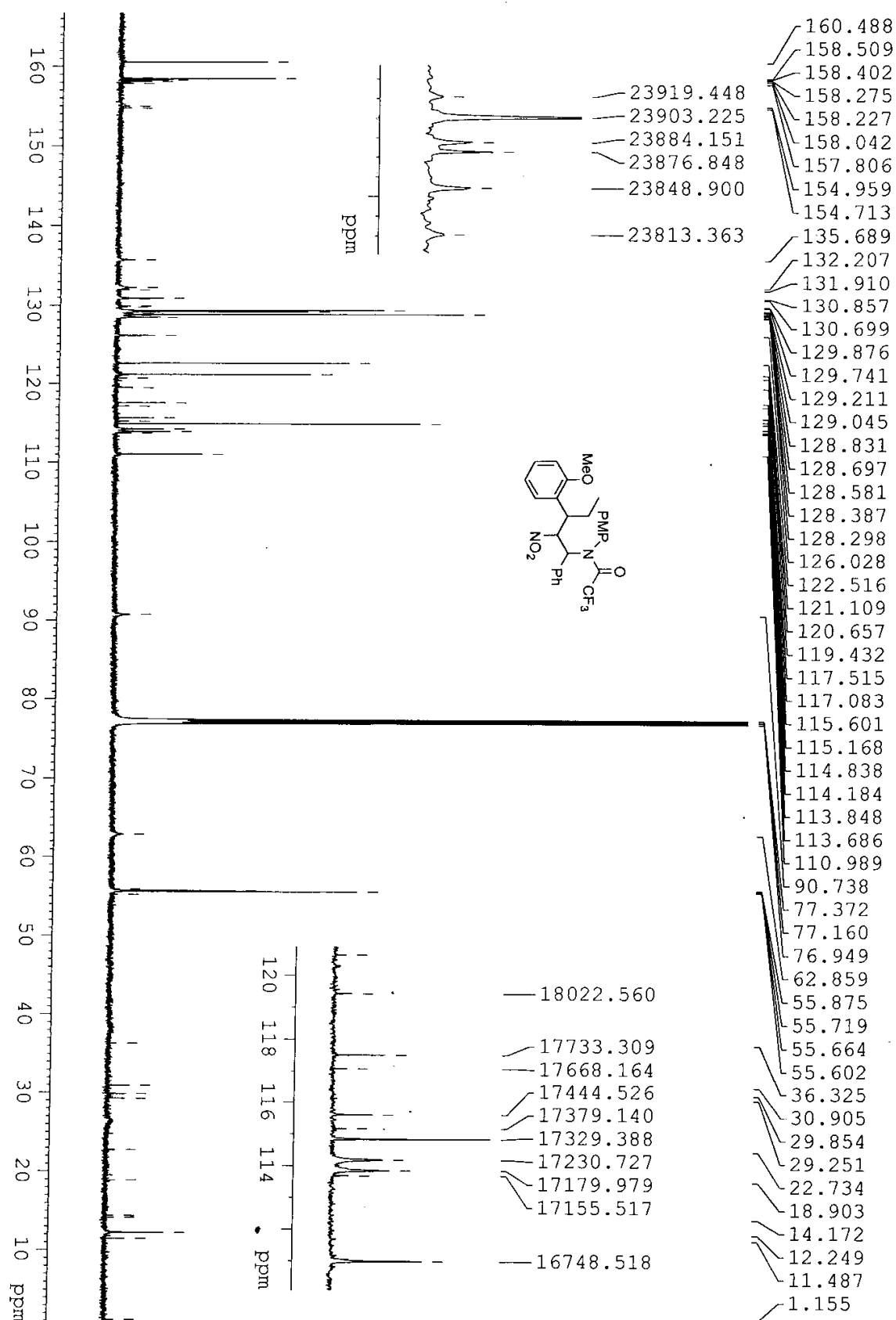
F2 - Processing parameters
SI 32768
SF 125.757814 MHz
MDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
F1P 215.000 ppm
F1 27037.93 Hz
F2P -5.000 ppm
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 664.58478 Hz/cm

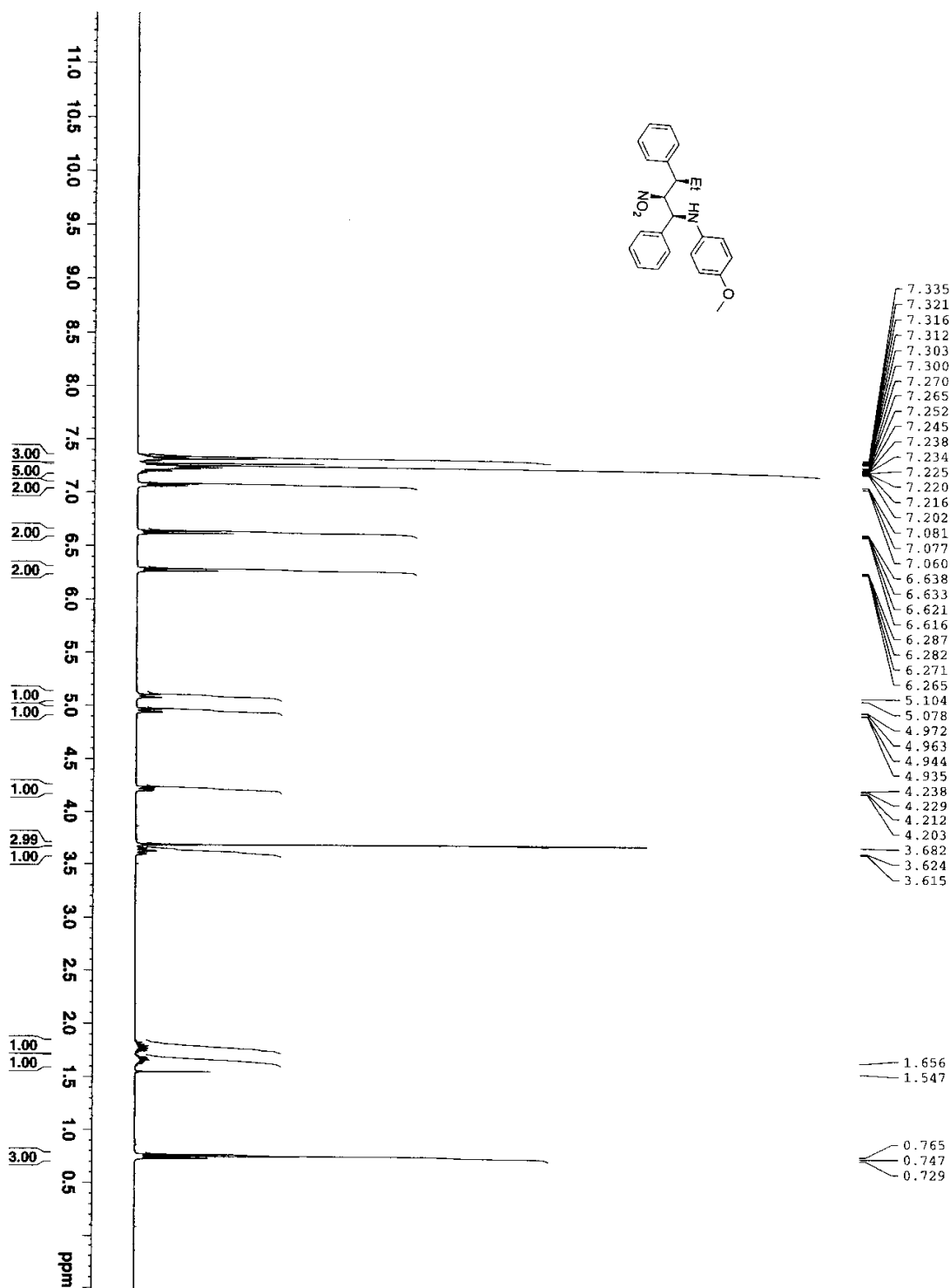
¹H NMR Entry 14



¹³C NMR Entry 14



Slot No. 5 Sample ID 533-3c0 SupervisorID ander Lab Phone No. 13540 UserID g_ste



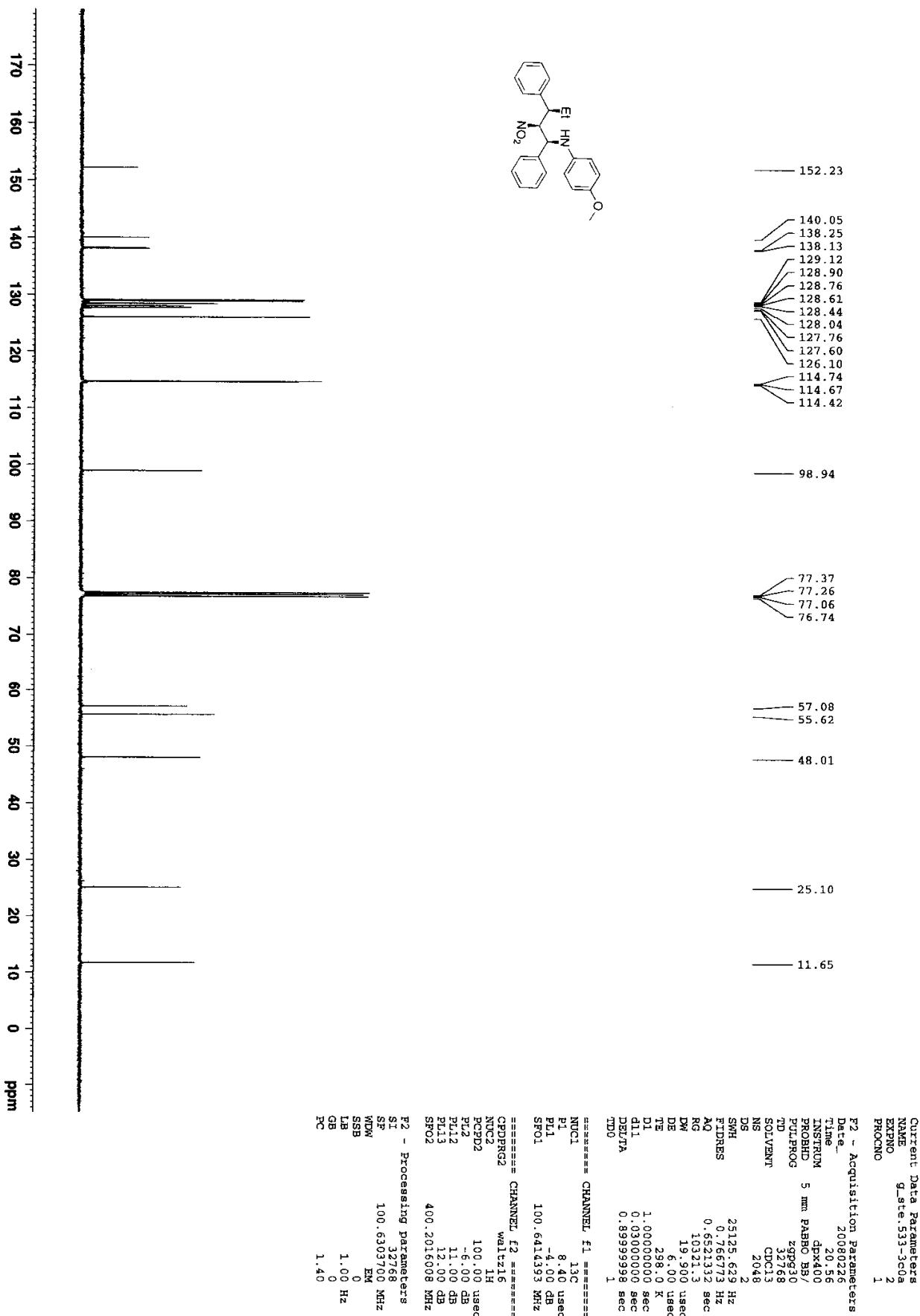
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7.245
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7.234
7.225
7.220
7.216
7.202
7.081
7.077
7.060
6.638
6.633
6.621
6.616
6.287
6.282
6.271
6.265
5.104
5.078
4.972
4.963
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4.229
4.212
4.203
3.682
3.624
3.615

1.656
1.547

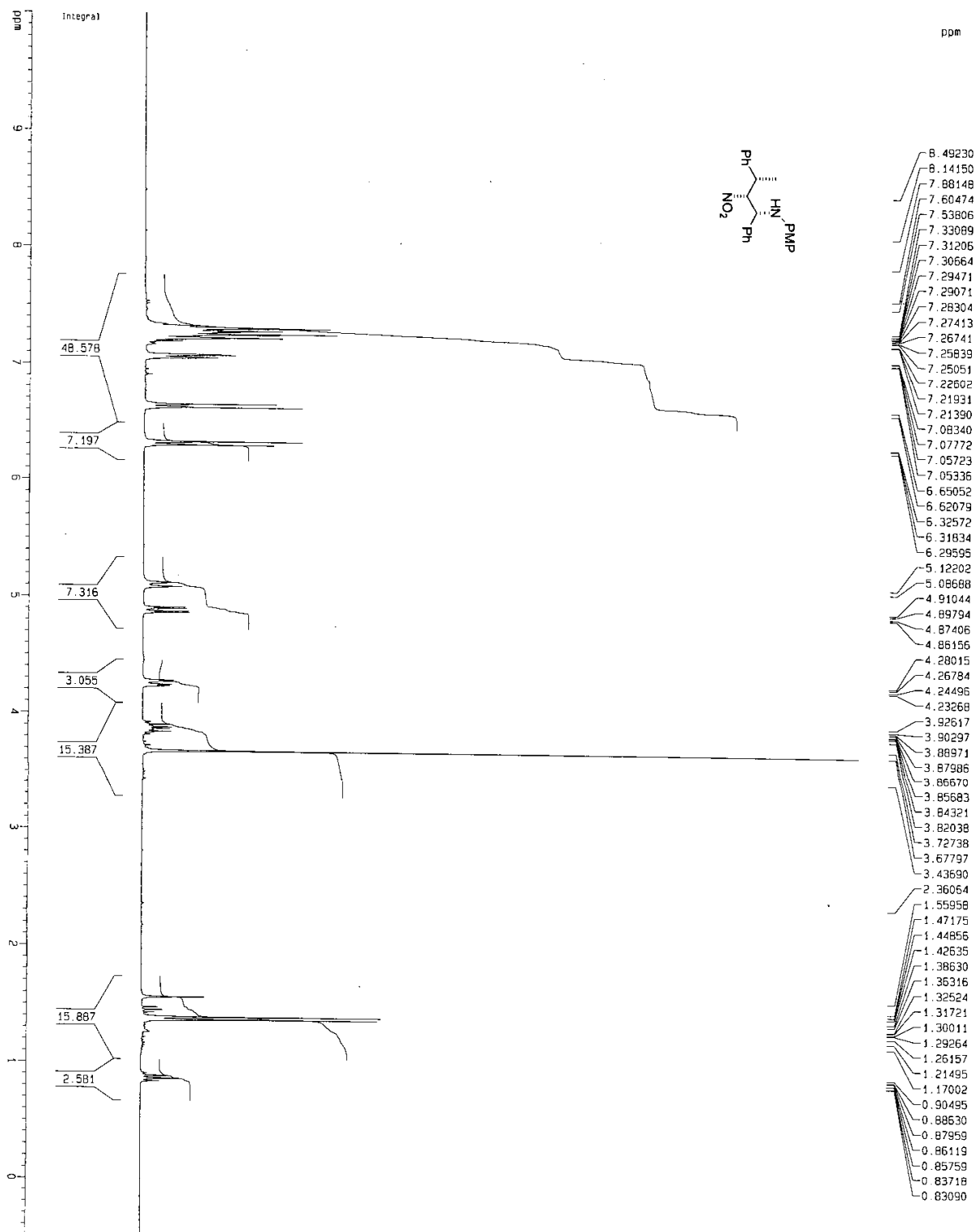
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0.747
0.729

Current Data Parameters
NAME g_ste:533-3c0
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080226
Time 15.30
INSTRUM dpx400
PROBHD 5 mm PABO BBI
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210251 sec
RG 456.1
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz
F2 - Processing parameters
SI 32768
SF 400.200091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

SLOT No. 53 Sample ID 533-3c0a SupervisorID ander Lab Phone No. 13540 UserID g_ste



¹H NMR Entry 16



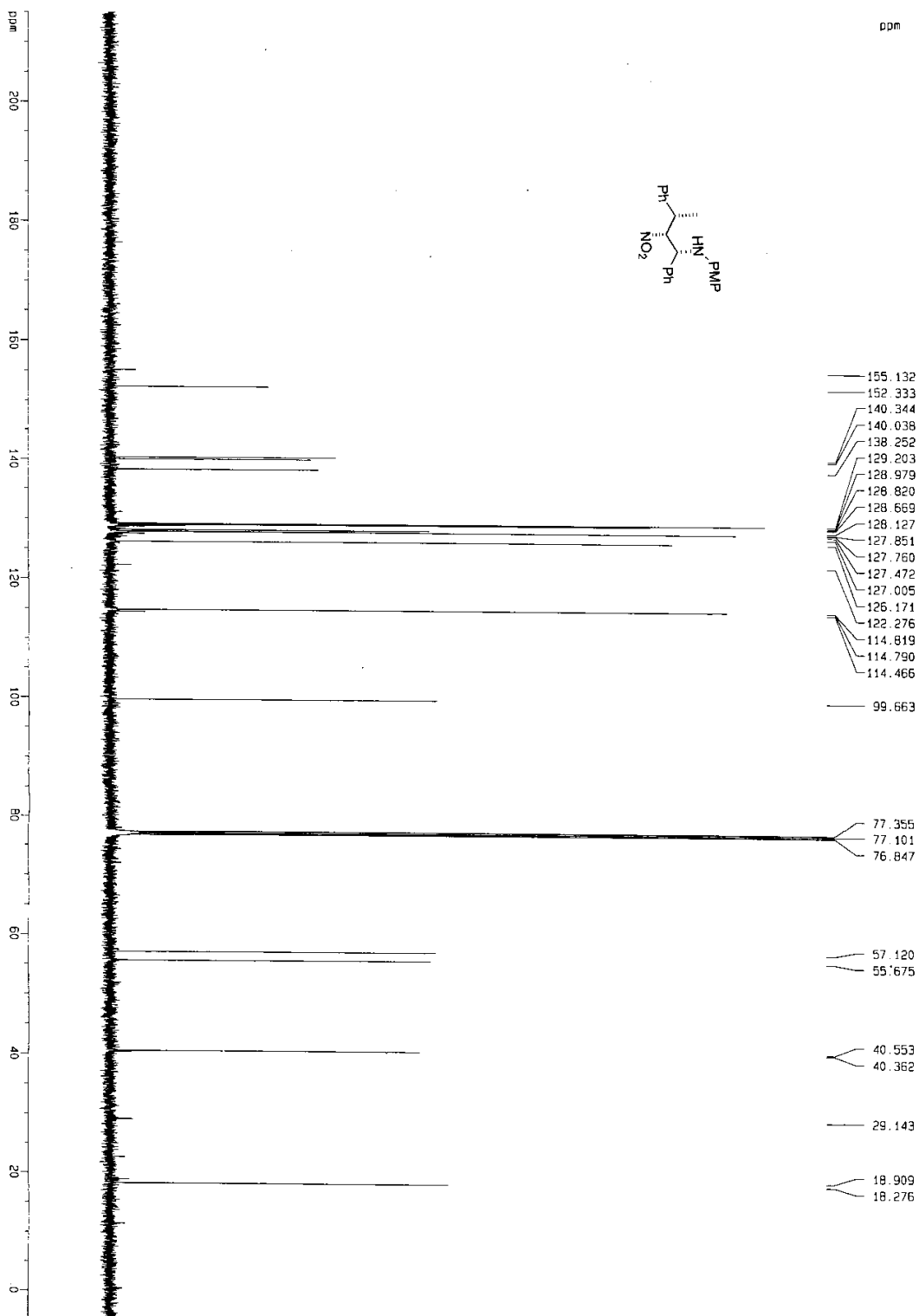
Current Data Parameters
 NAME: Sep09-2009
 EXPNO: 50
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20090909
 Time: 13.28
 INSTRUM: amx300
 PROBRD: 5 mm QNP 1H
 PULPROG: zg30
 TD: 53506
 SOLVENT: CDCl3
 RO: 20
 NS: 16
 DS: 20
 SMH: 8771.930 Hz
 FIDRES: 0.163843 Hz
 AQ: 3.0498919 sec
 RG: 2048
 DW: 57.000 usec
 DE: 81.43 usec
 TE: 300.0 K
 HL1: 1 dB
 D1: 0.12000000 sec
 P1: 5.80 usec
 SFO1: 299.8758200 MHz
 NUCLEUS: 1H

F2 - Processing parameters
 SI: 32768
 SF: 299.8727940 MHz
 WDW: EM
 SSF: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00
 SR: 2794.00 Hz

1D NMR plot parameters
 CX: 32.00 cm
 F1P: 10.000 ppm
 F1: 2998.73 Hz
 F2P: -0.500 ppm
 F2: -149.94 Hz
 PPMCM: 0.32613 ppm/cm
 HZCM: 98.39577 Hz/cm

¹³C NMR Entry 16



mm746
C13.10ng CDCl3 v2 jca 35

Current Data Parameters
NAME Sep09-2009
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090912
Time 8.13
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420724 sec
RG 6502
DM 15.900 usec
DE 5.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.60 usec
PL1 0.00 dB
SF01 125.7715719 MHz

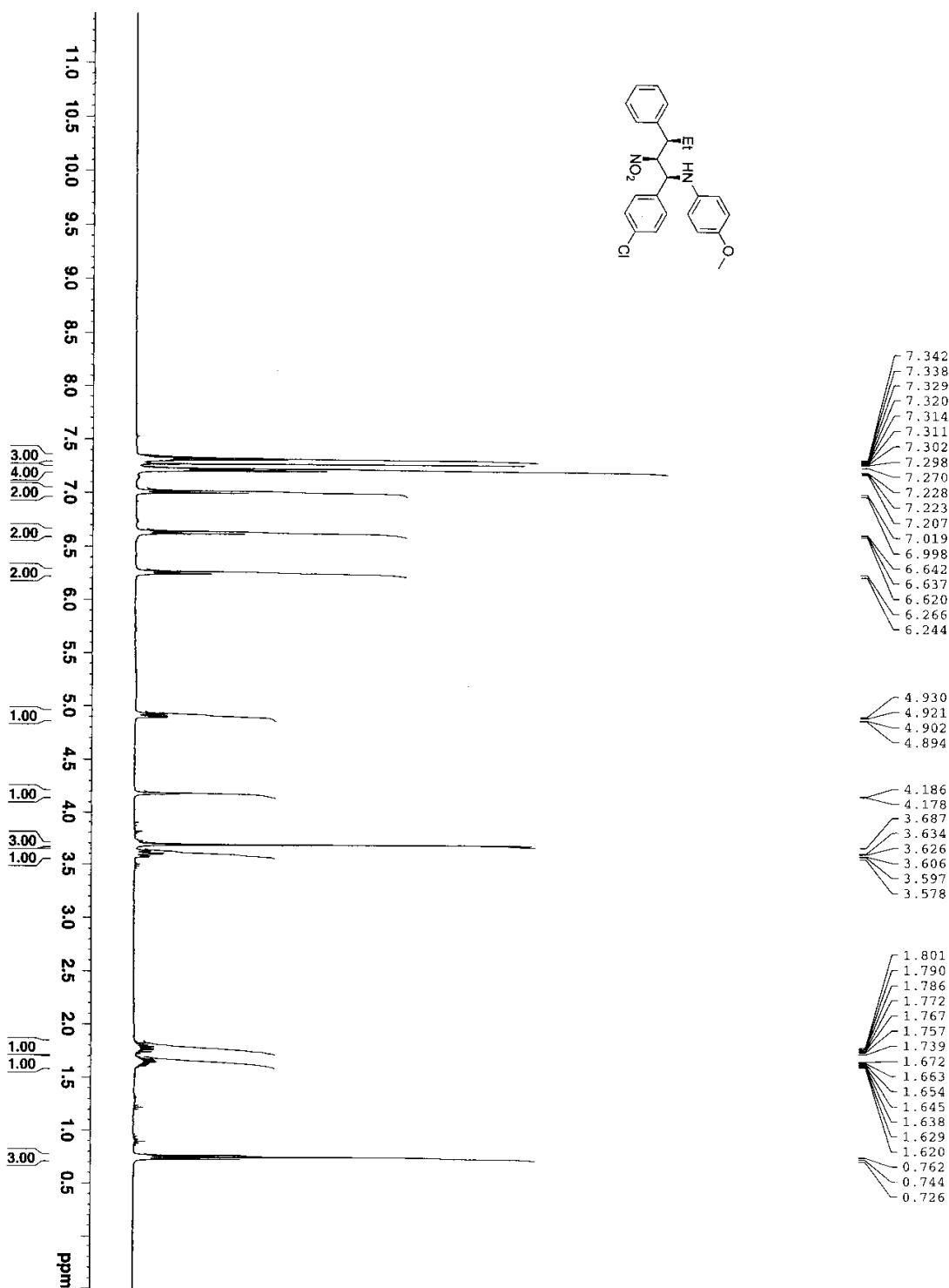
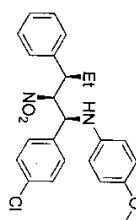
===== CHANNEL f2 =====
COPRPG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.132005 MHz

F2 - Processing Parameters
SI 32768
SF 125.757814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
F1P 215.000 ppm
F1 27037.93 Hz
F2P -5.000 ppm
F2 -628.79 Hz
PPMCM 6.87500 ppm/cm
HZCM 864.58478 Hz/cm

¹H NMR Entry 17

Slot No. 34 Sample ID 574-cryst3 SupervisorID ander Lab Phone No. 13540 UserID g_ste



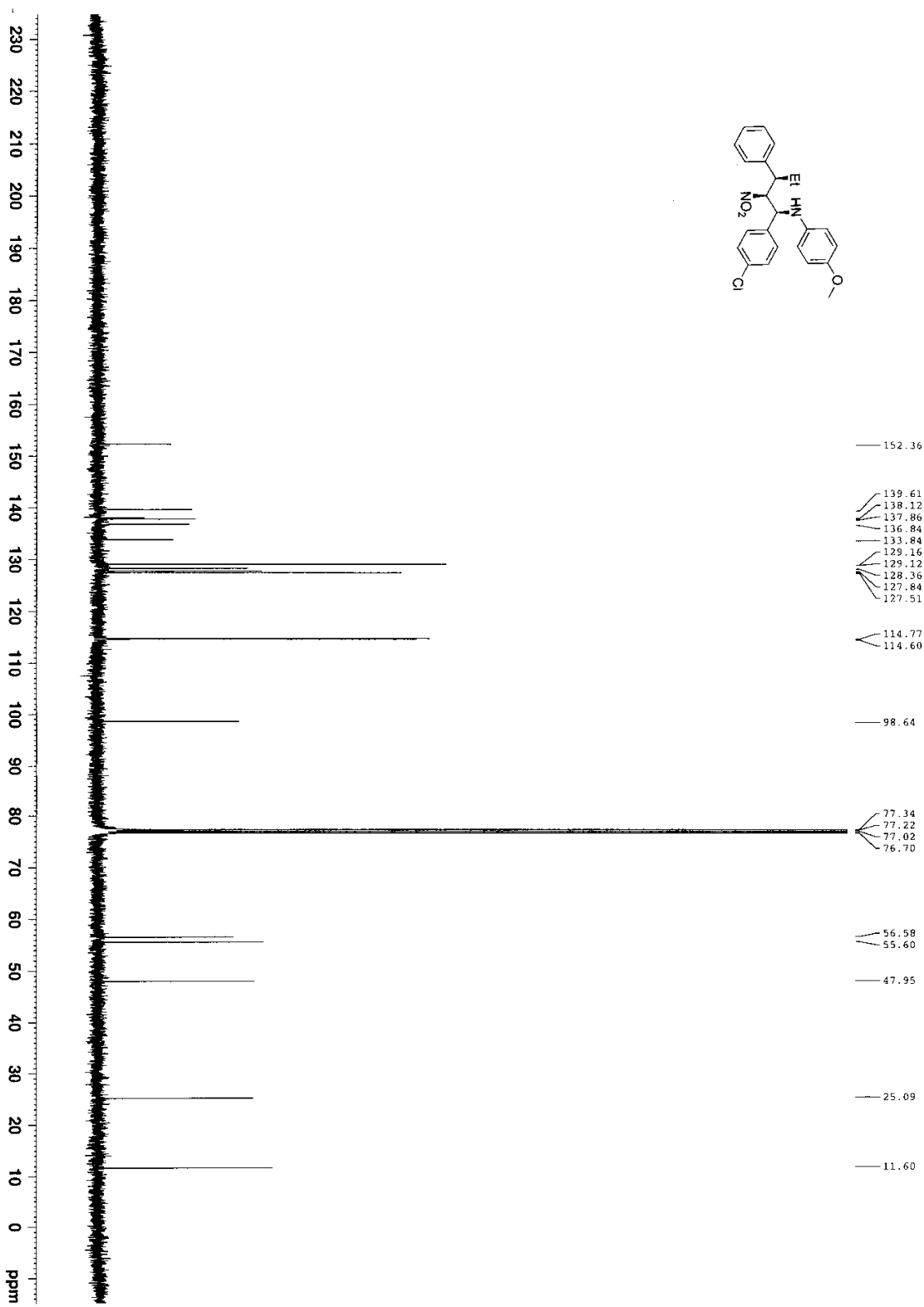
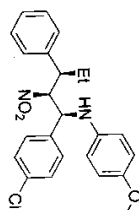
Current Data Parameters
NAME g_ste:574-cryst3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080430
Time 13.19
INSTRUM dpx400
PROBHD 5 mm PABO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210251 sec
RG 512
DM 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz

F2 - Processing parameters
SI 32768
SF 400.200090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

UserID g_ste SampleID 574-cryst1 SupervisorID ander Lab Phone No. 13540 Slot Number 54



```

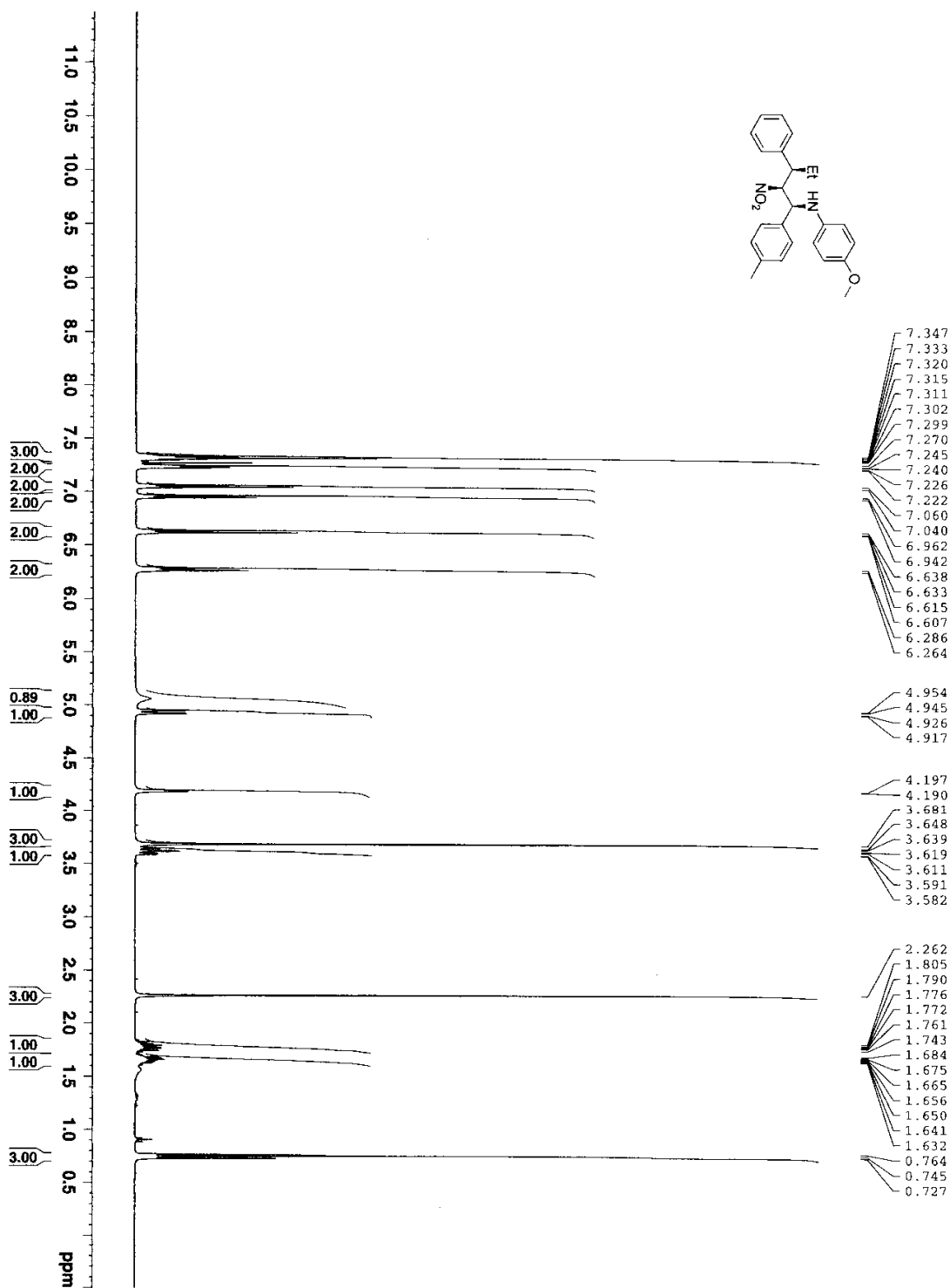
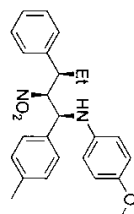
NAME g_ste.574-cryst1
EXPNO 2
PROCNO 1
Date_ 20080331
Time 20.40
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 35.9
DE 19.900 use
WDW 6.00 use
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 use
PL1 -3.00 dB
PL1W 68.66265106 W
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 17.00 dB
PL13 20.00 dB
PL12W 20.24796486 W
PL12W 0.20247965 W
PL13W 0.10148021 W
SFO2 400.1315005 MHz
SI 100.6127690 MHz
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

¹H NMR Entry 18

Slot No. 11 Sample ID 571-cryst1 SupervisorID ander Lab Phone No. 13540 UserID g_ste



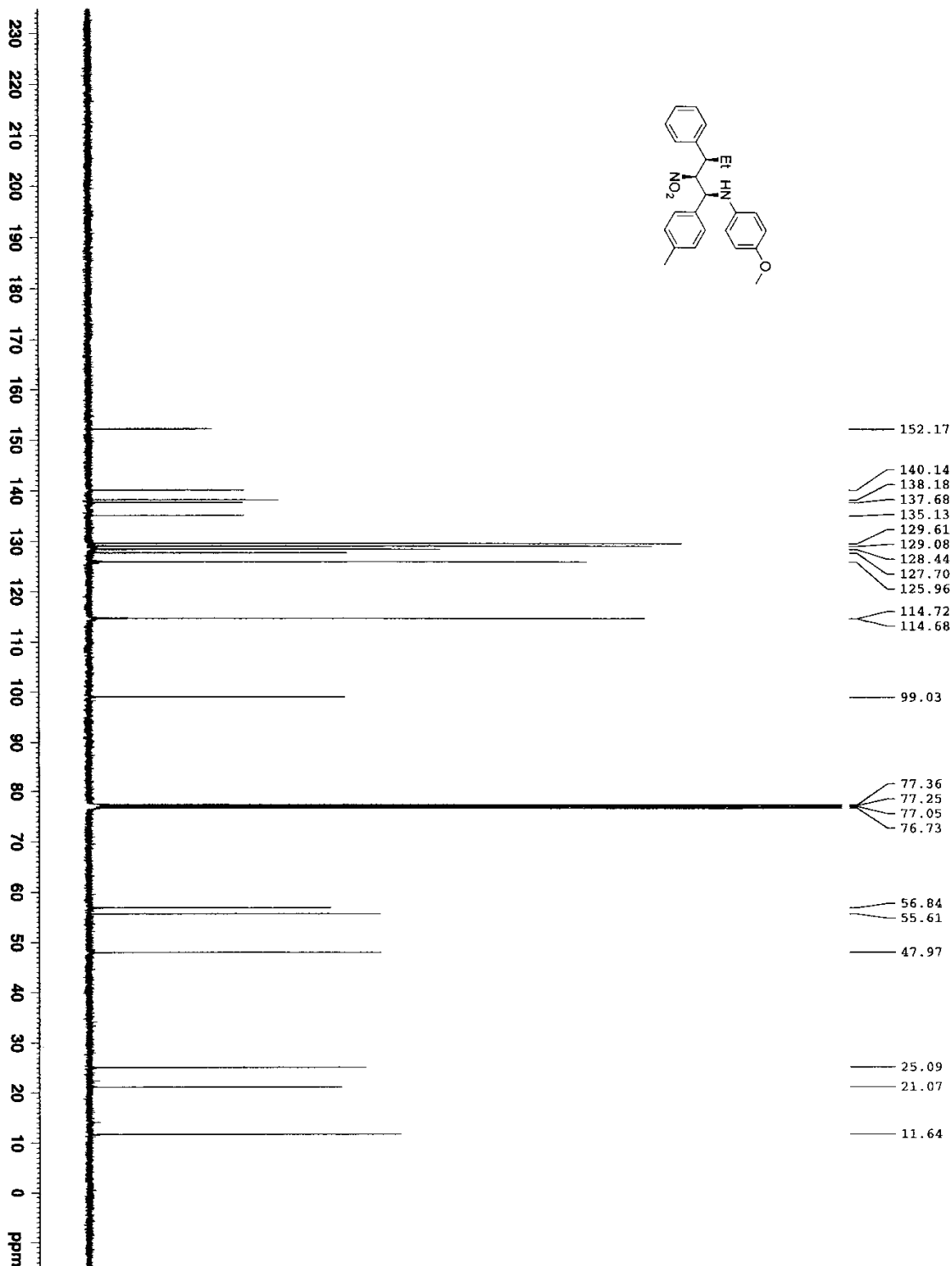
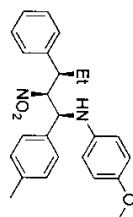
Current Data Parameters
NAME g_ste.571-cryst1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080329
Time 18.36
INSTRUM spect
PROBHD 5 mm PABO 297
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 287.4
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz

F2 - Processing parameters
SI 32768
SF 400.2000990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 11 Sample ID 571-cryst1 SupervisorID ander Lab Phone No. 13540 UserID g_ste



Current Data Parameters
NAME g_ste.571-cryst1
EXPNO 2
PROCNO 1

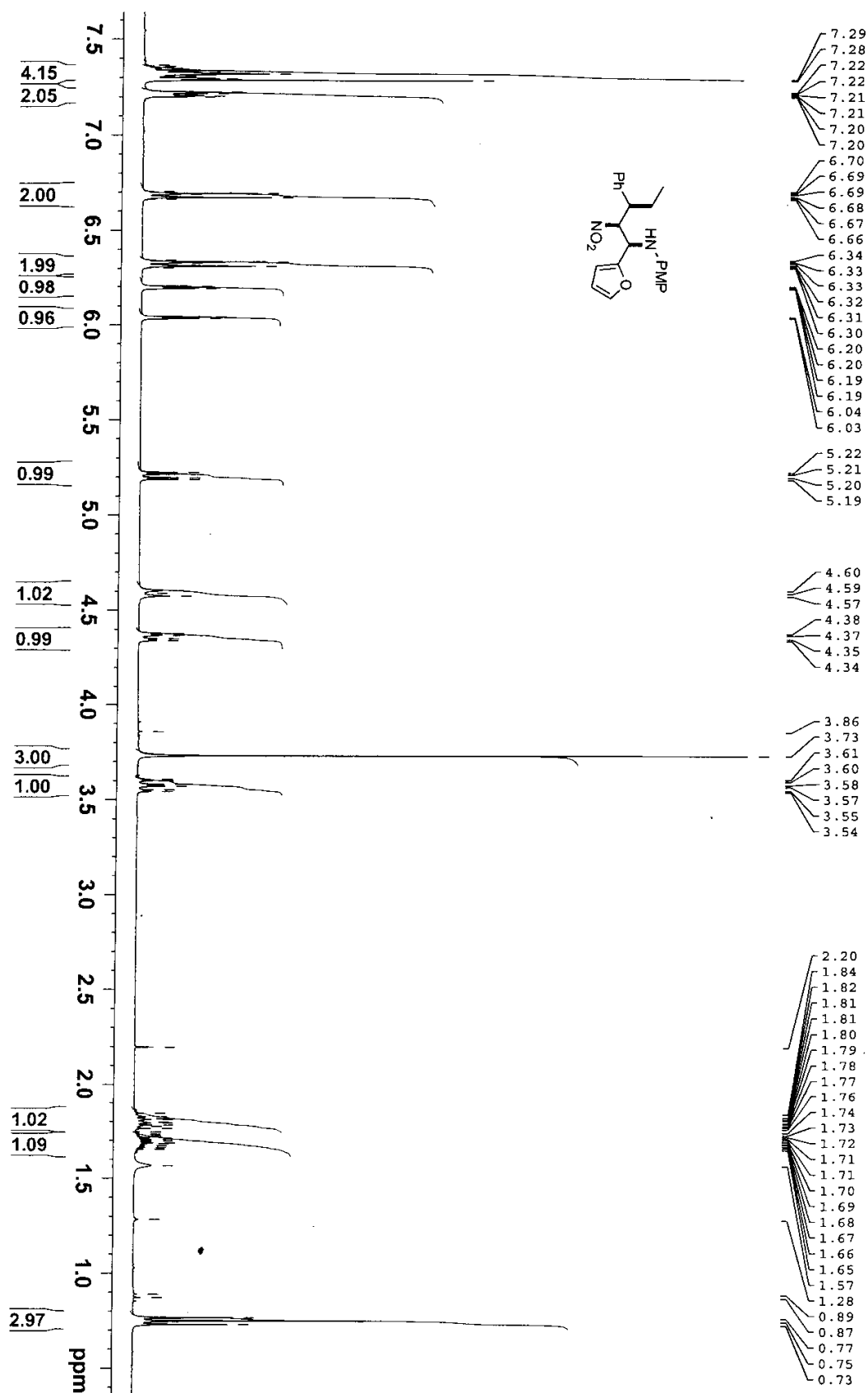
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Date_ 20080329
Time 20:47
INSTRUM dpx400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 9195.2
DW 19.900 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL -4.00 dB
SFO1 100.6414393 MHz

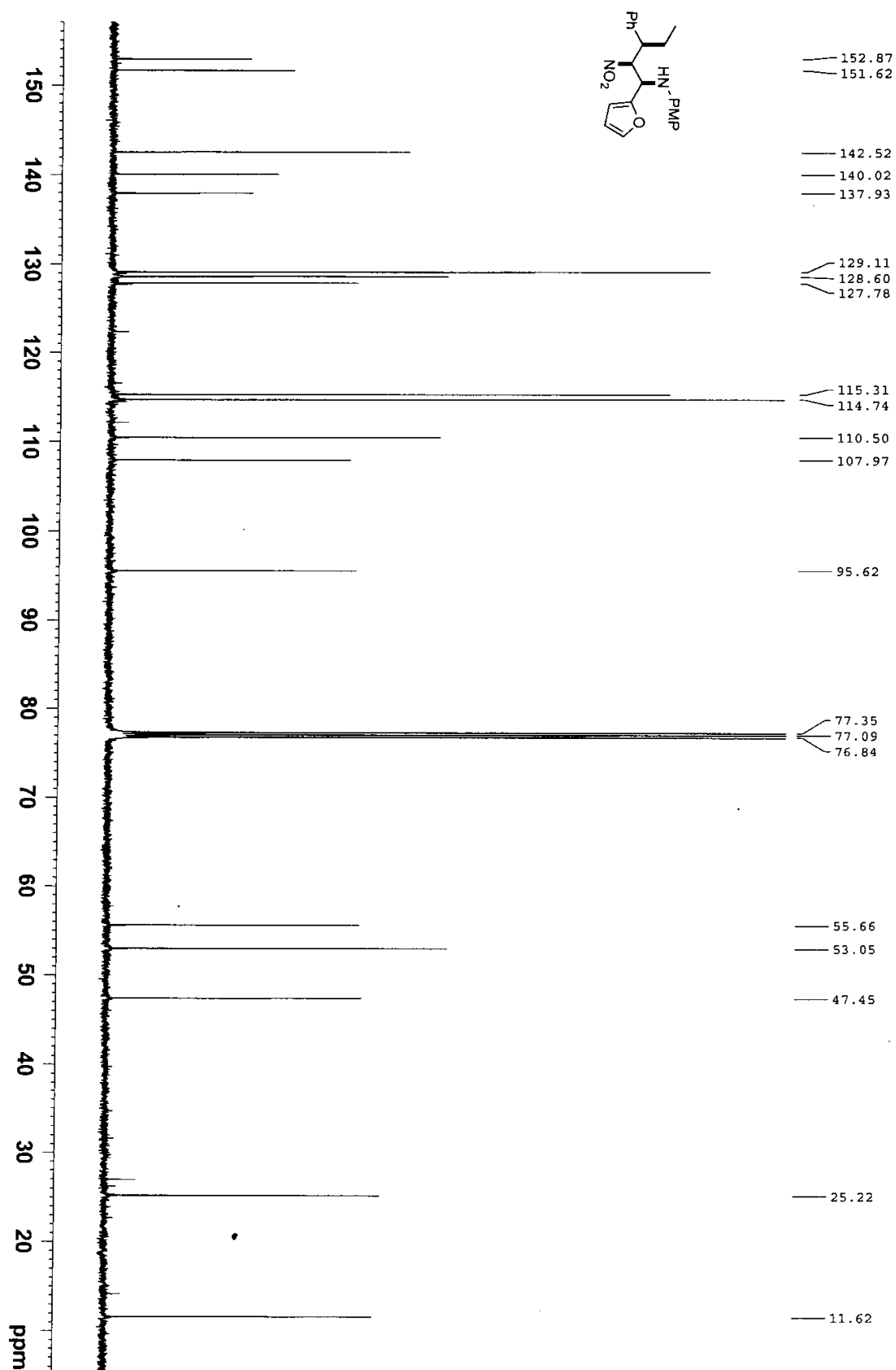
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2016008 MHz

F2 - Processing parameters
SI 32768
SF 100.630700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Entry 19



¹³C NMR Entry 19

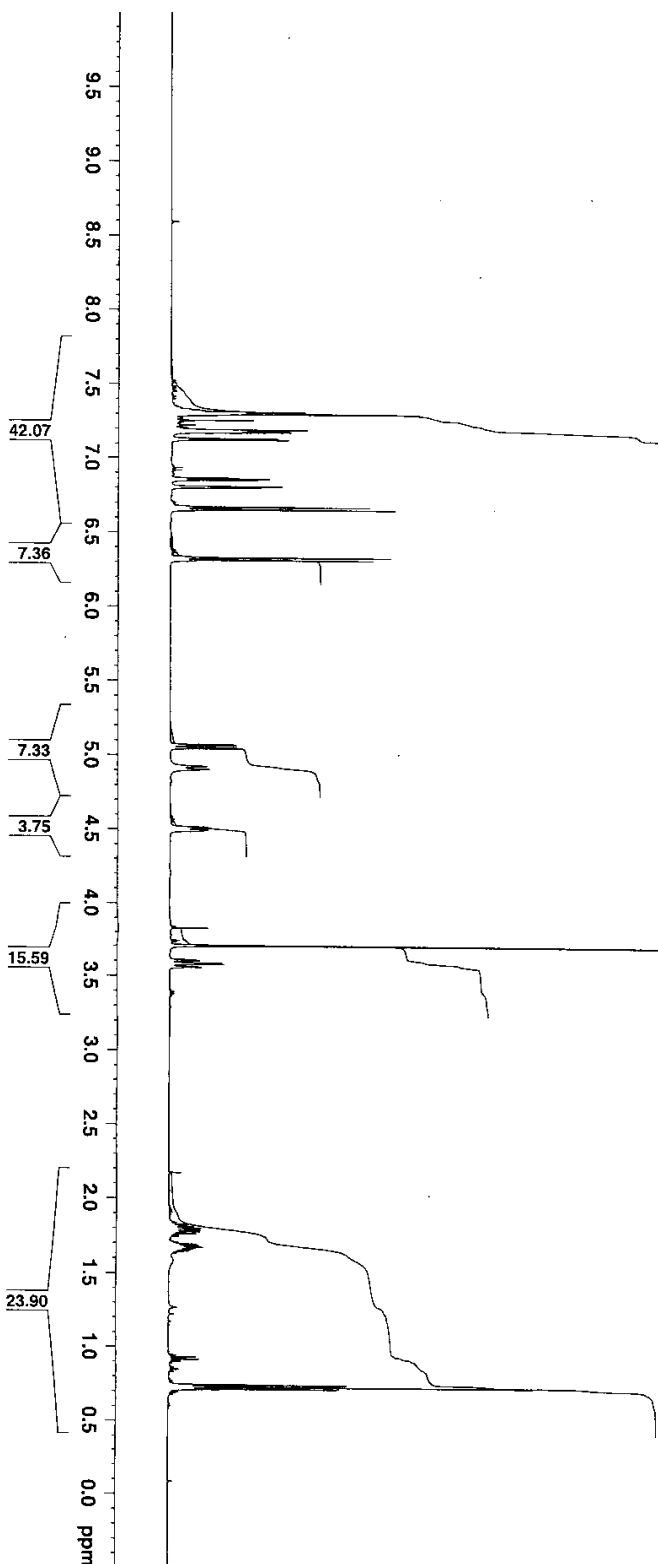
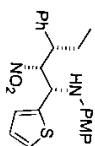


¹H NMR Entry 20

thio imine greg
PROTON.uci CDC13 v2 jca 1



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0.01
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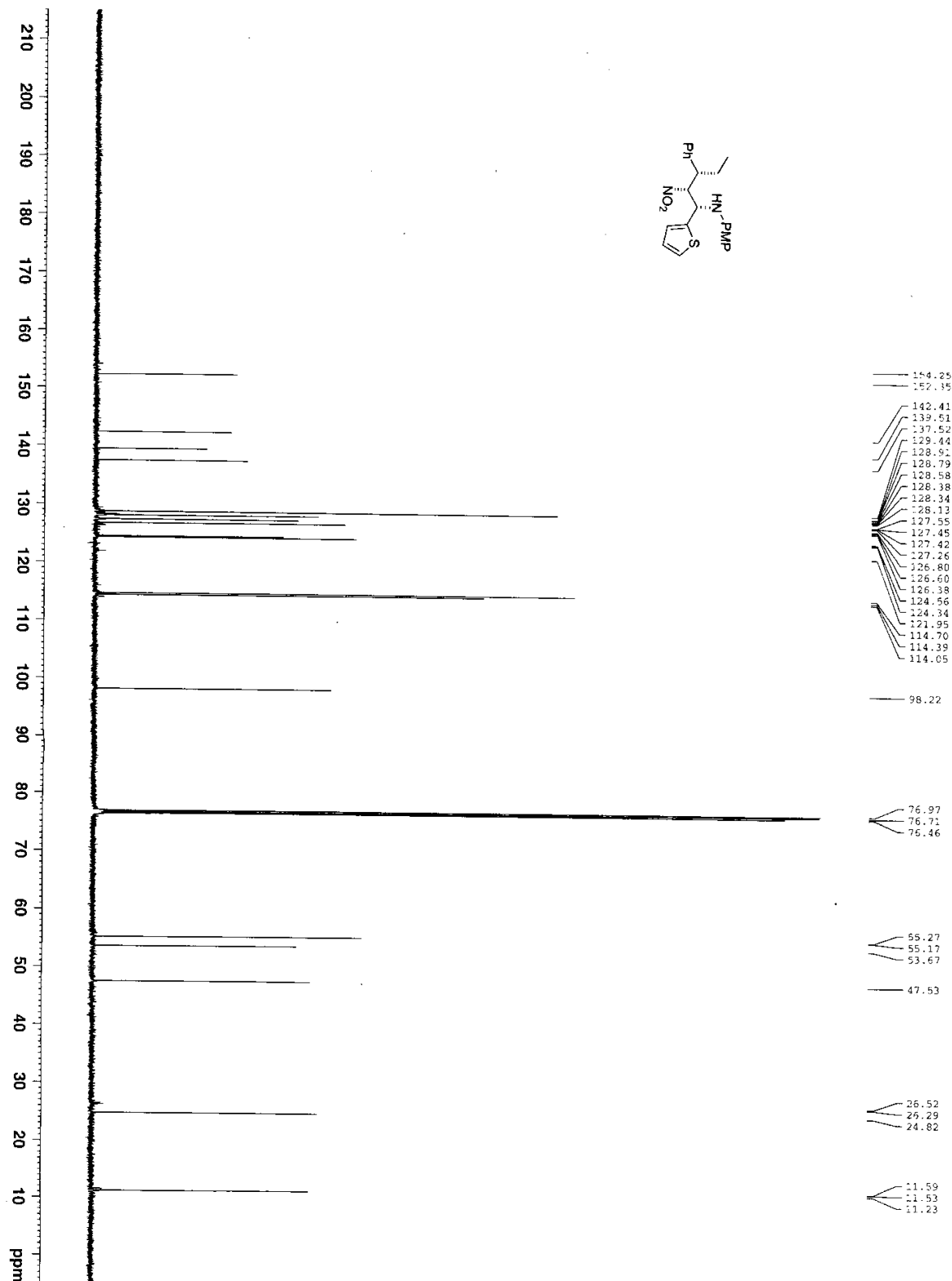
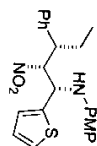


F2 - Acquisition Parameters
Date_ 20091202
Time 16.11
INSTRUM dtx500
PROBHD 5 mm MUltiNuc1
PULPROG zg30
TD 82642
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.125604 Hz
AQ 3.999712 sec
RG 128
DW 48.400 use
DE 6.00 use
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 14.80 use
PL 0.00 dB
SFO1 500.1330885 MHz

F2 - Processing Parameters
SI 131072
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

mrm 10 thio imine greg
C13CPD CDCl3 v2 jca 50



154.25
152.95
142.41
139.51
137.52
129.44
128.91
128.79
128.58
128.38
128.34
128.13
127.55
127.45
127.42
127.26
126.80
126.60
126.38
124.56
124.34
121.95
114.70
114.39
114.05
98.22
76.97
76.71
75.46
55.27
55.17
53.67
47.53
26.52
26.29
24.82
11.59
11.53
11.23



Current Data Parameters
NAME Dec01-2009
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091201
Time 17.32
INSTRUM dirx500
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4096
DW 16.650 use
DE 28.0 K
TE 2.00000000 sec
D1 0.03000000 sec
d11 1.89999998 sec
DELTA 0.00000000 sec
MCREST 0.01500000 sec
MCWRK

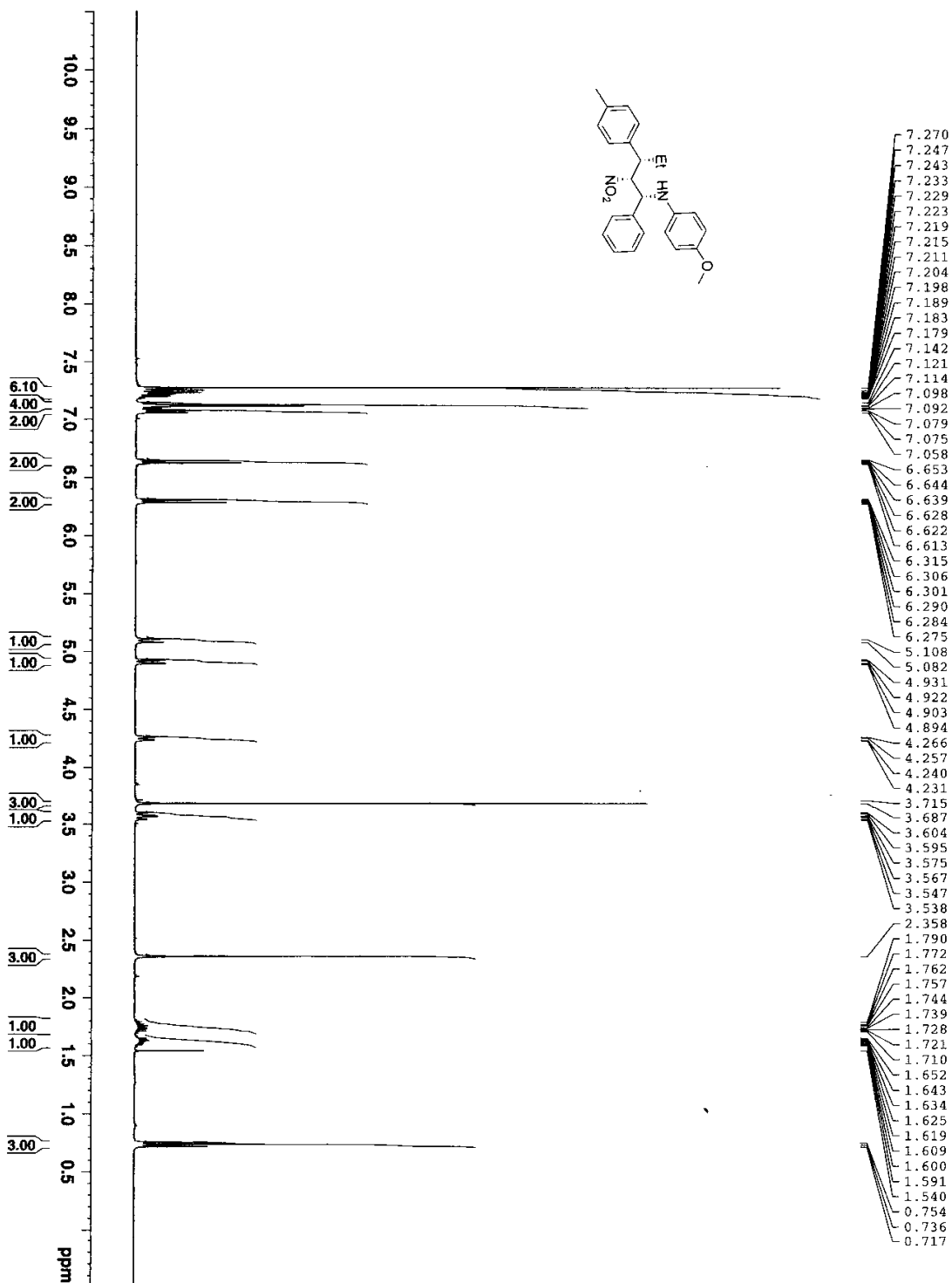
===== CHANNEL f1 =====
NUC1 13C
P1 5.50 use
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 use
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578314 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Entry 21

Slot No. 6 Sample ID B SupervisorID ander Lab Phone No. 13540 UserID g_ste



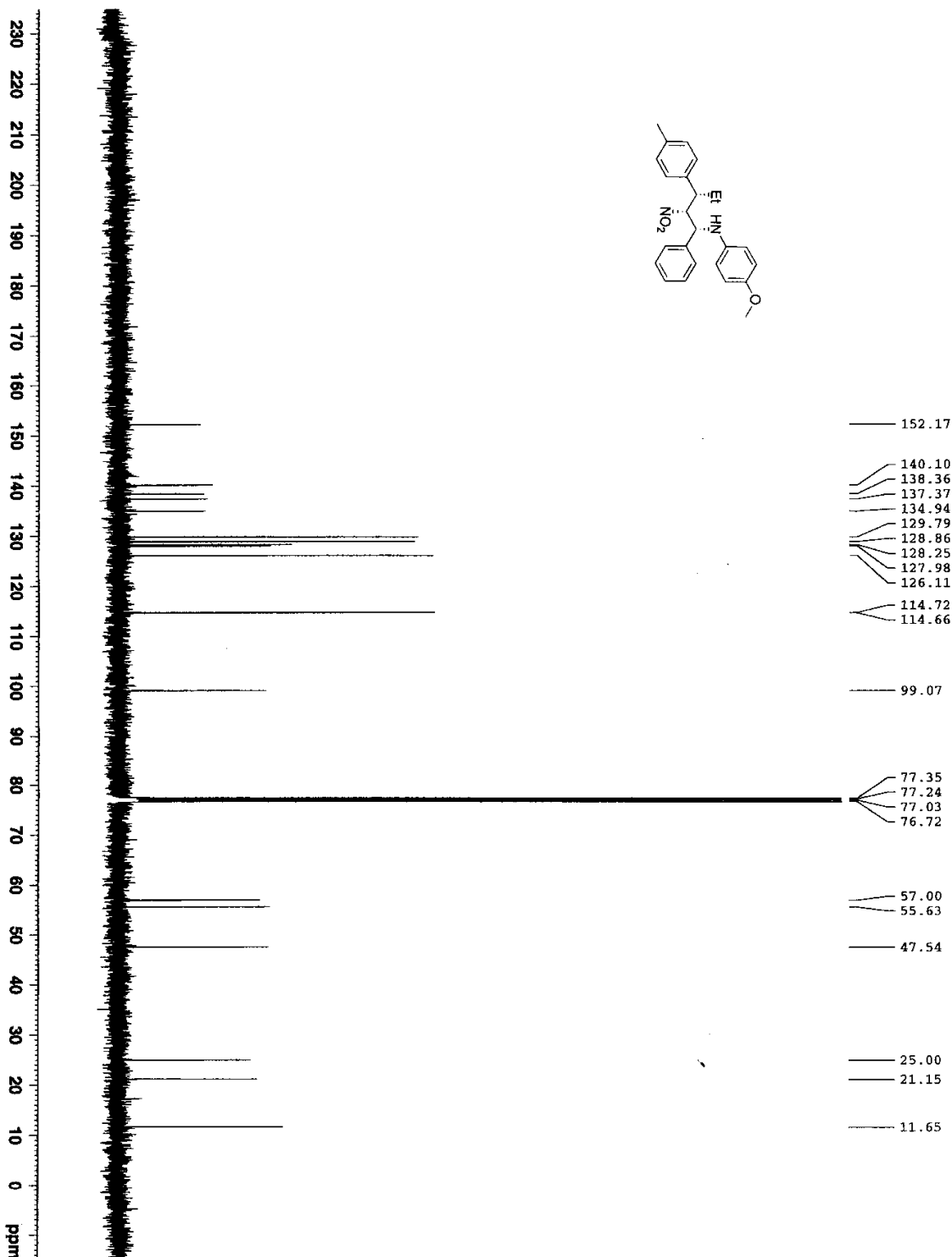
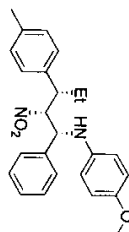
Current Data Parameters
NAME g_ste.B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080317
Time 14.10
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 724.1
DM 104.400 usec
DE 6.00 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz

F2 - Processing parameters
SI 32768
SF 400.200093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 40 Sample ID 462-cryst1 SupervisorID ander Lab Phone No. 13540 UserID g_ste



152.17
140.10
138.36
137.37
134.94
129.79
128.86
128.25
127.98
126.11
114.72
114.66
99.07
77.35
77.24
77.03
76.72
57.00
55.63
47.54
25.00
21.15
11.65

Current Data Parameters
NAME g_ste.462-cryst1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20080315
Time 11:11
INSTRUM dpx400
PROBHD 5 mm PABO BBO
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 16384
DM 19.900 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d1.1 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

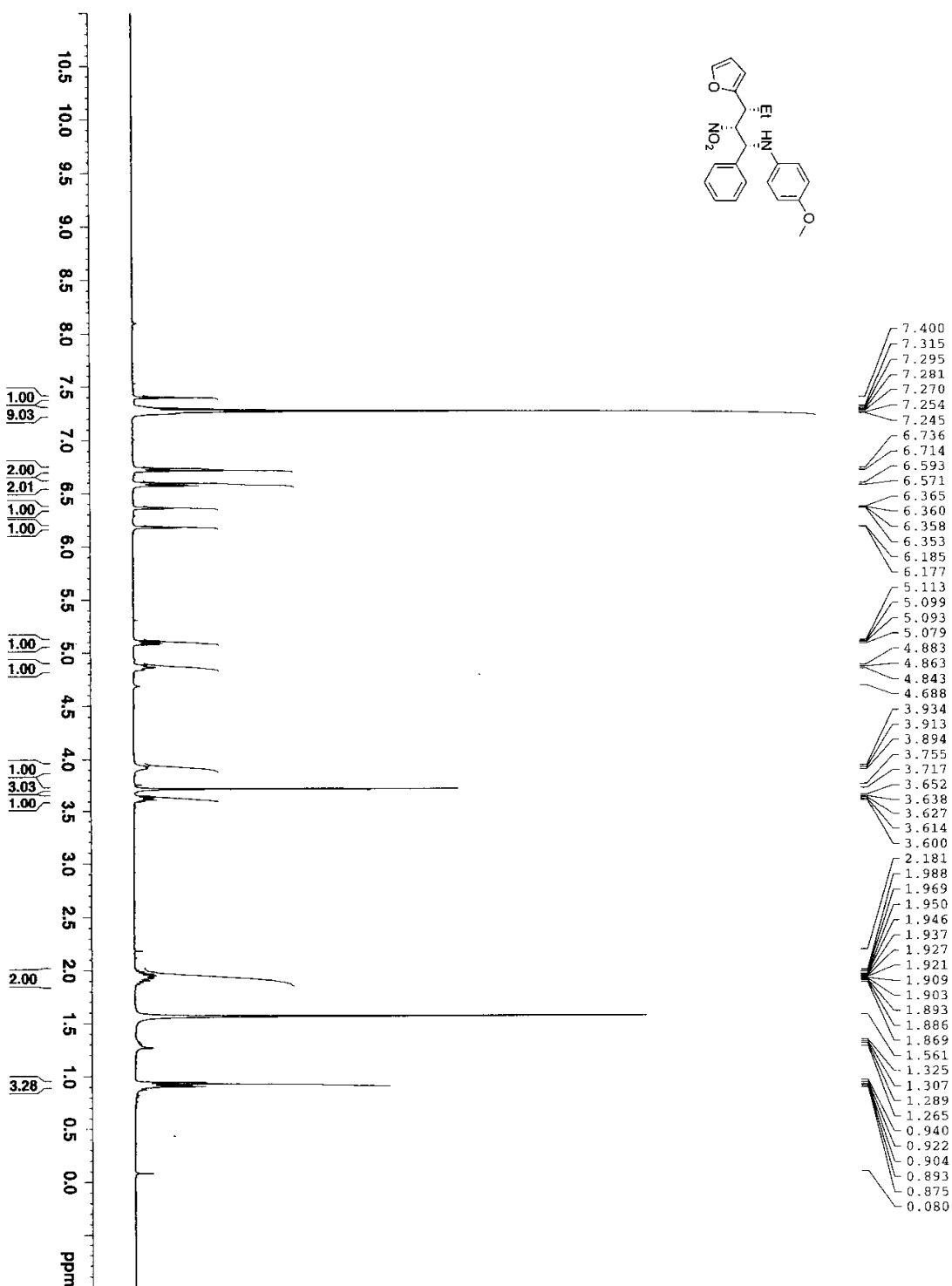
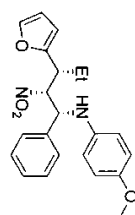
===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 -4.00 dB
SFO1 100.614393 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2016008 MHz

F2 - Processing Parameters
SI 32768
SF 100.6301700 MHz
WPM 2M
SGB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Entry 22

UserID g_ste SampleID 702-1c2a SupervisorID ander Lab Phone No. 13540 Slot Number 4



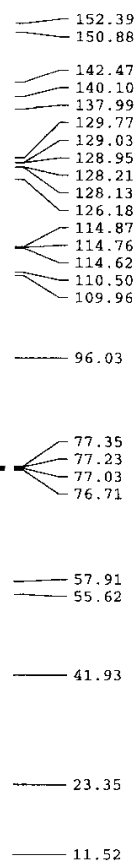
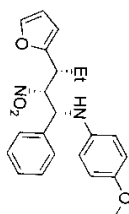
Current Data Parameters
NAME g_ste,701-1c2a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080811
Time 18.25
INSTRUM av3400
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 512
RG 60.800 usec
DE 6.50 usec
TE 296.5 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.00 usec
PL1 -0.90 dB
PL1W 11.52680206 W
SFO1 400.0724706 MHz

F2 - Processing parameters
SI 65536
SF 400.070040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 22 Sample ID 566-cryst1 SupervisorID ander Lab Phone No. 13540 UserID g_ste



Current Data Parameters
NAME g_ste.566-cryst1
EXPNO 4
PROCNO 1

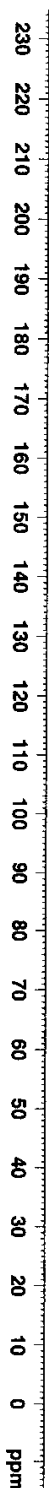
F2 - Acquisition Parameters
Date_ 20080320
Time 3.58

INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 20642.5
DM 19.900 usec
DE 6.00 usec
TE 297.9 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

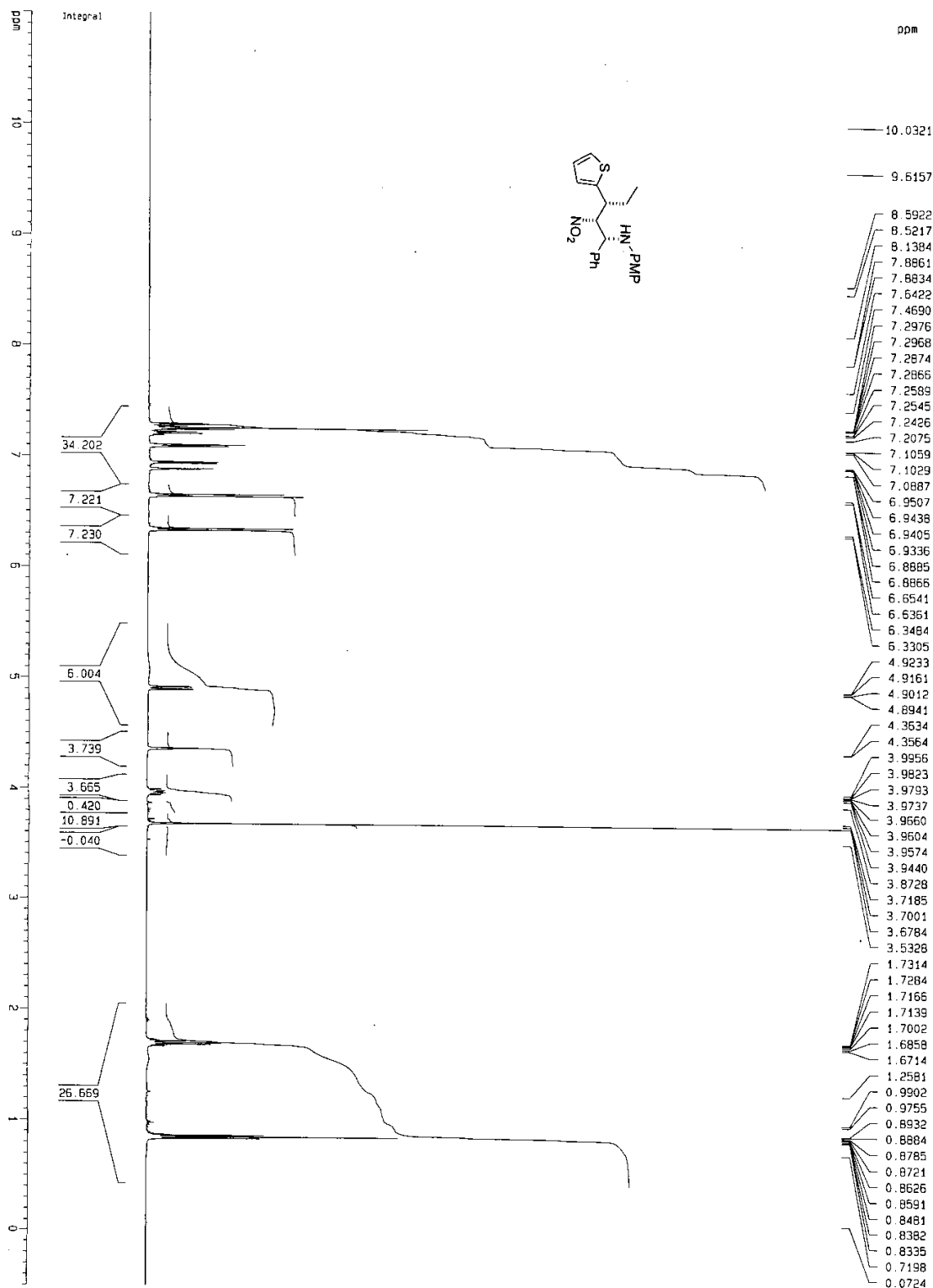
===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 -4.00 dB
SFO1 100.6414393 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2016008 MHz

F2 - Processing parameters
SI 32768
SF 100.6303700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



¹H NMR Entry 23



Current Data Parameters
 NAME Sep17-2009
 EXPNO 10
 PROCNO 1

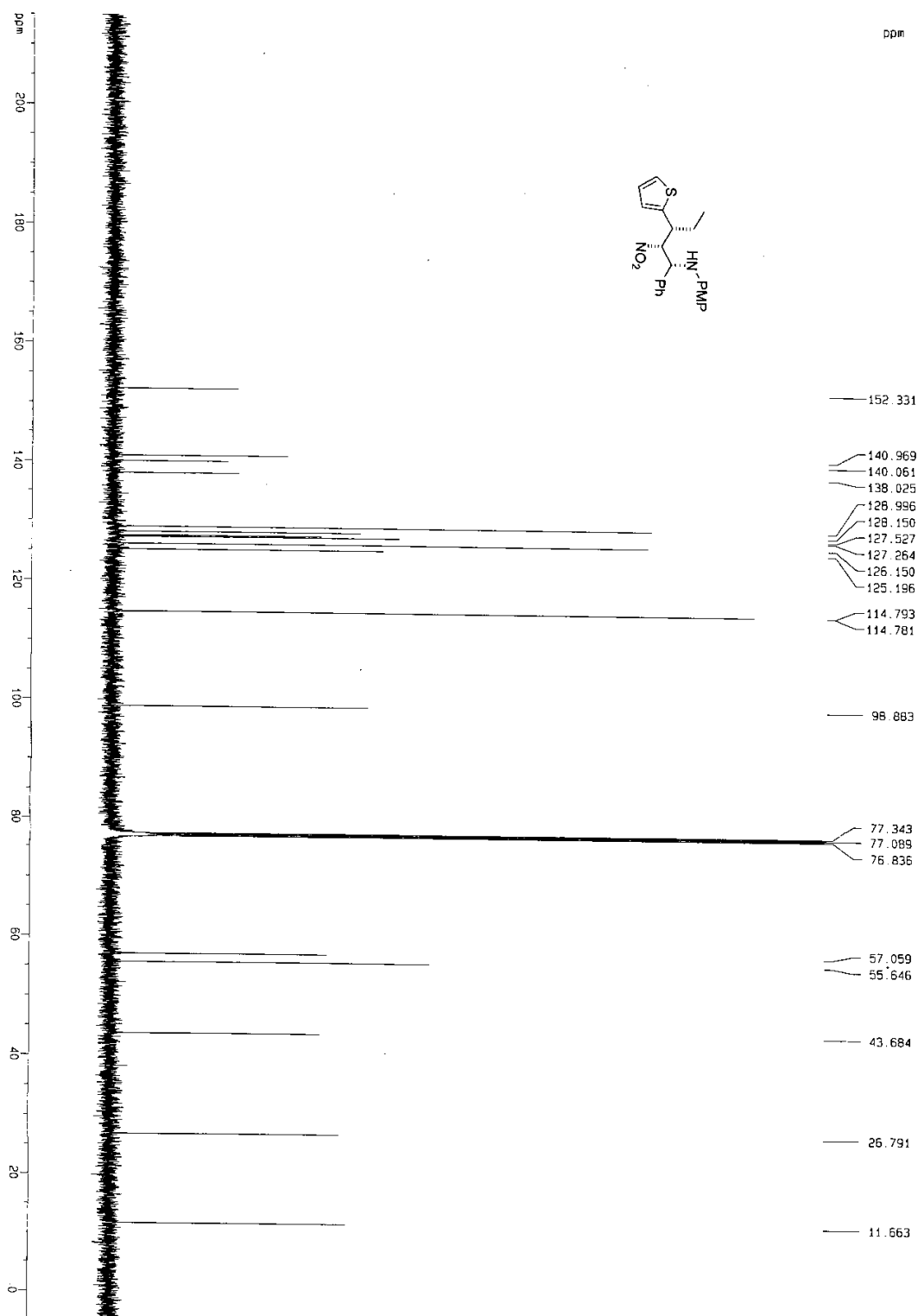
F2 - Acquisition Parameters
 Date_ 20090917
 Time 17.23
 INSTRUM grx500
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDC13
 NS 16
 DS 0
 SMH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 456.1
 DN 48.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 500.130085 MHz

F2 - Processing parameters
 SI 65536
 SF 500.130140 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 11.000 DPM
 F1 5501.43 Hz
 F2P -0.500 DPM
 F2 -250.07 Hz
 PPMCM 0.35938 DPM/cm
 HZCM 179.73422 Hz/cm

¹³C NMR Entry 23



13. 10mg CDCl3 v2 jca 23

mrm753-1

Current Data Parameters

NAME	Sep17-2009
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20090917
Time	18.16
INSTRUM	gpcx500
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	31446.541 Hz
FIDRES	0.479836 Hz
AQ	1.0420724 sec
RG	3649.1
DW	15.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00020000 sec

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

NUC1	¹³ C
P1	5.60 usec
PL1	0.00 dB
SFO1	125.7715719 MHz

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

CPDPRG2	waltz16
NUC2	¹ H
PCPD2	95.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	19.00 dB
SFO2	500.1320095 MHz

F2 - Processing Parameters

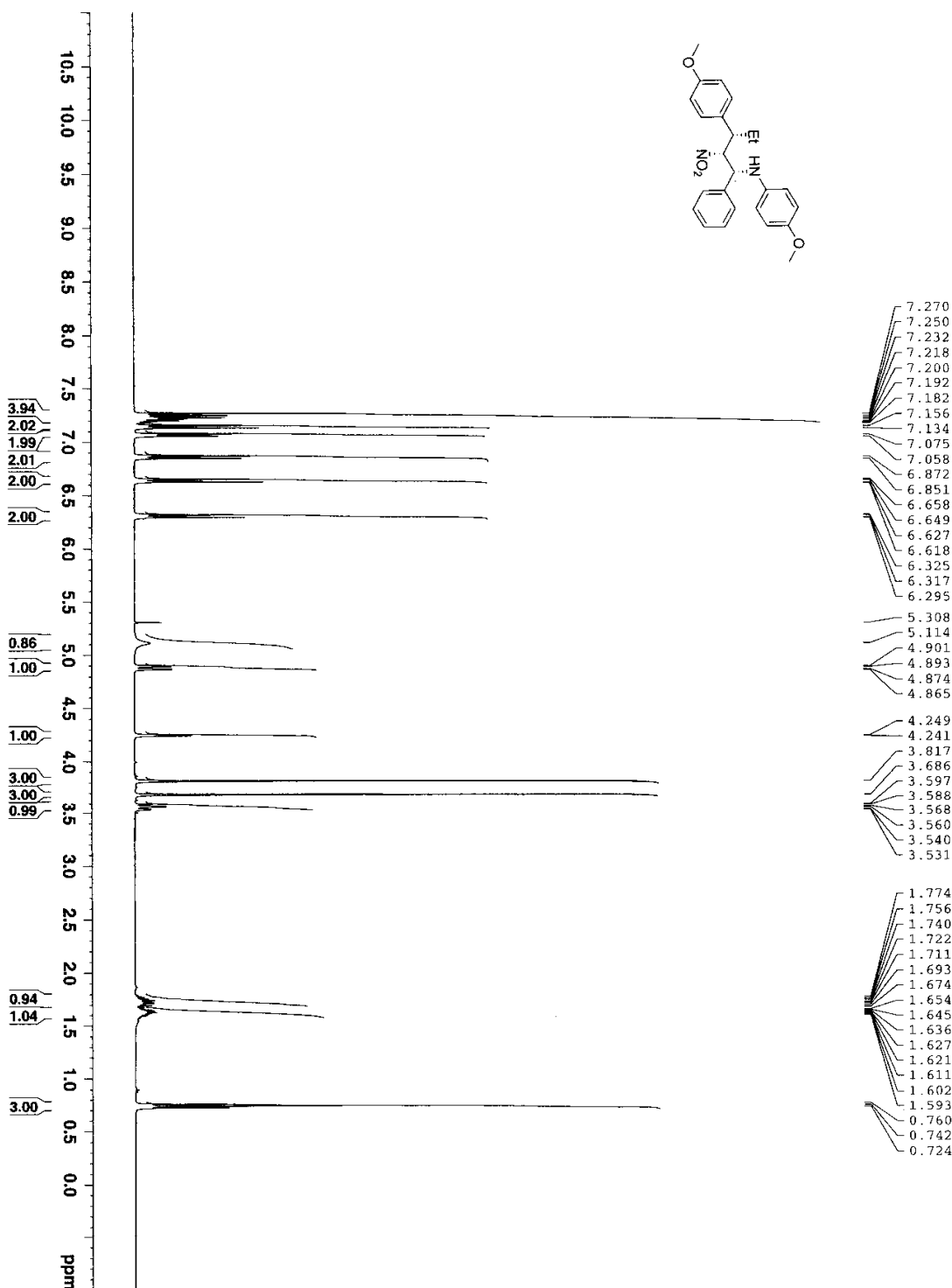
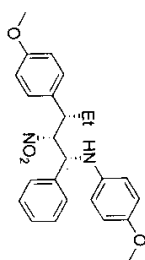
SF	32768
SF	125.7577814 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot Parameters

CX	32.00 cm
F1P	215.000 DPM
F1	27037.93 Hz
F2P	-5.000 DPM
F2	-628.79 Hz
PPMCM	6.87500 DPM/cm
MZCM	864.58478 Hz/cm

¹H NMR Entry 24

UserID g_ste SampleID 674-c4 SupervisorID ander Lab Phone No. 13540 Slot Number 44



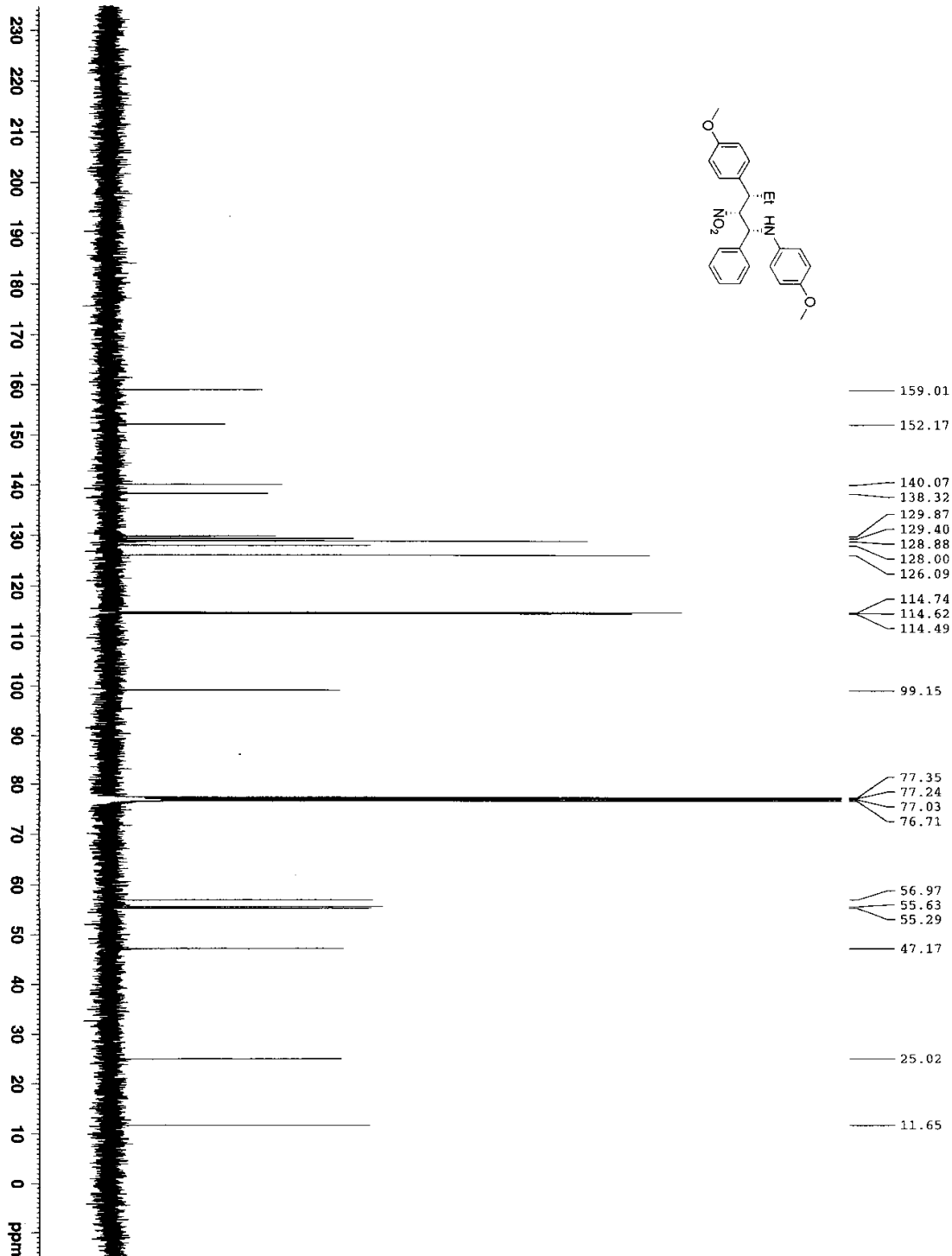
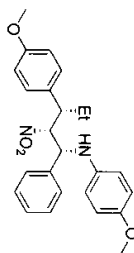
Current Data Parameters
NAME g_ste.674-c4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080723
Time 18.23
INSTRUM aw3400
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.00 usec
PL1 -0.90 dB
PL1W 11.52680206 W
SFO1 400.0724706 MHz

F2 - Processing Parameters
SI 65536
SF 400.070042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 54 Sample ID 674-cryst1 SupervisorID ander Lab Phone No. 13540 UserID g_ste



Current Data Parameters
NAME g_ste.674-cryst1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080711
Time 19.38
INSTRUM dpx400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 4096
DE 19.900 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 -4.00 dB
SFO1 100.641393 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2016008 MHz

F2 - Processing Parameters
SI 32768
SF 100.6303700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR Entry 25

mrm768
PROTON.ucl CDC13 v2 jca 38



Current Data Parameters
NAME OCT09-2009
EXPNO 20
PROCNO 1

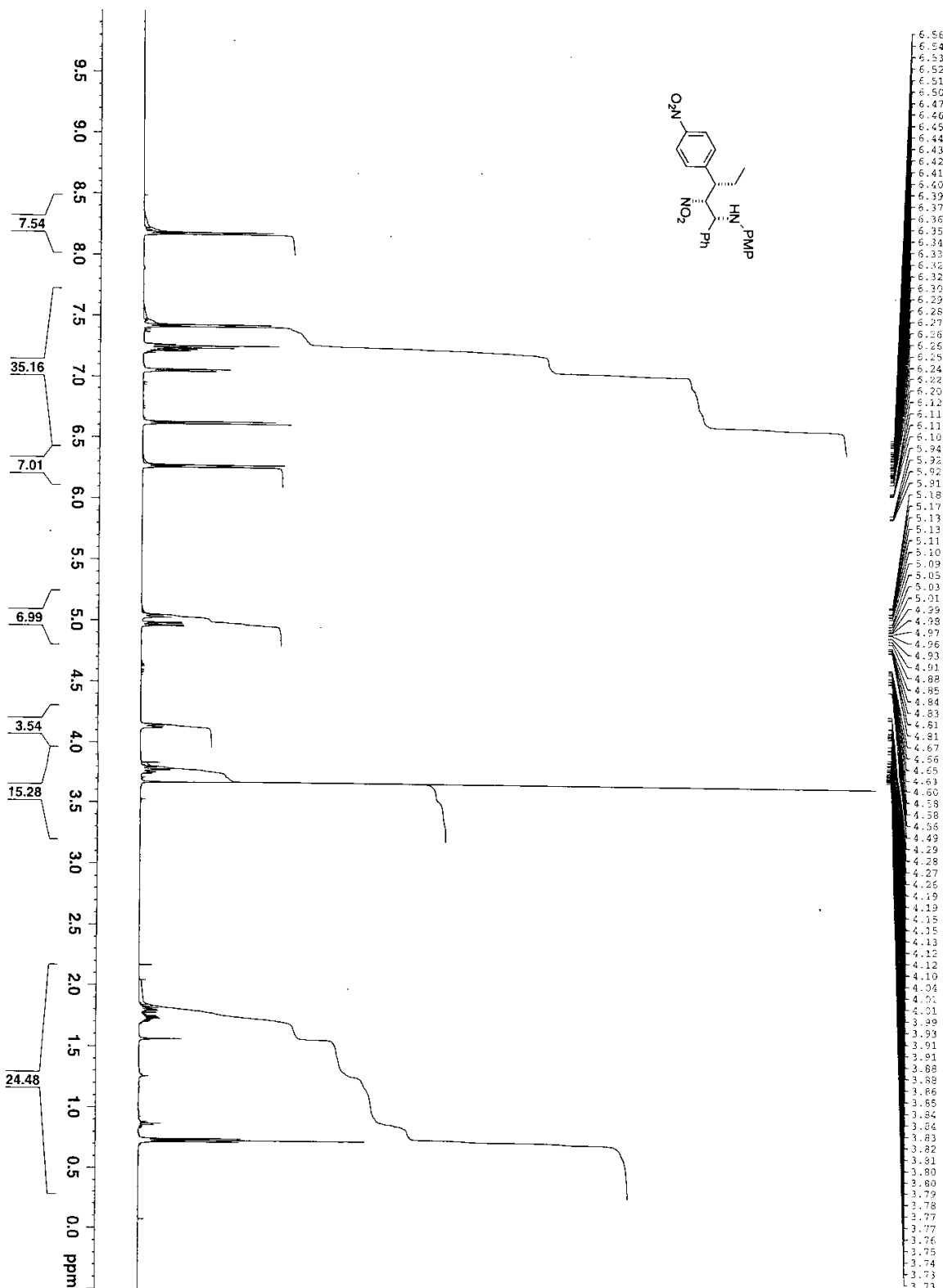
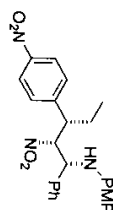
F2 - Acquisition Parameters
Date_ 20091009
Time 13.30

INSTRUM dpx500
PROBHD 5 mm Multinucl
PULPROG zg30
TD 62642
SOLVENT CDCl3
NS 16
DS 2

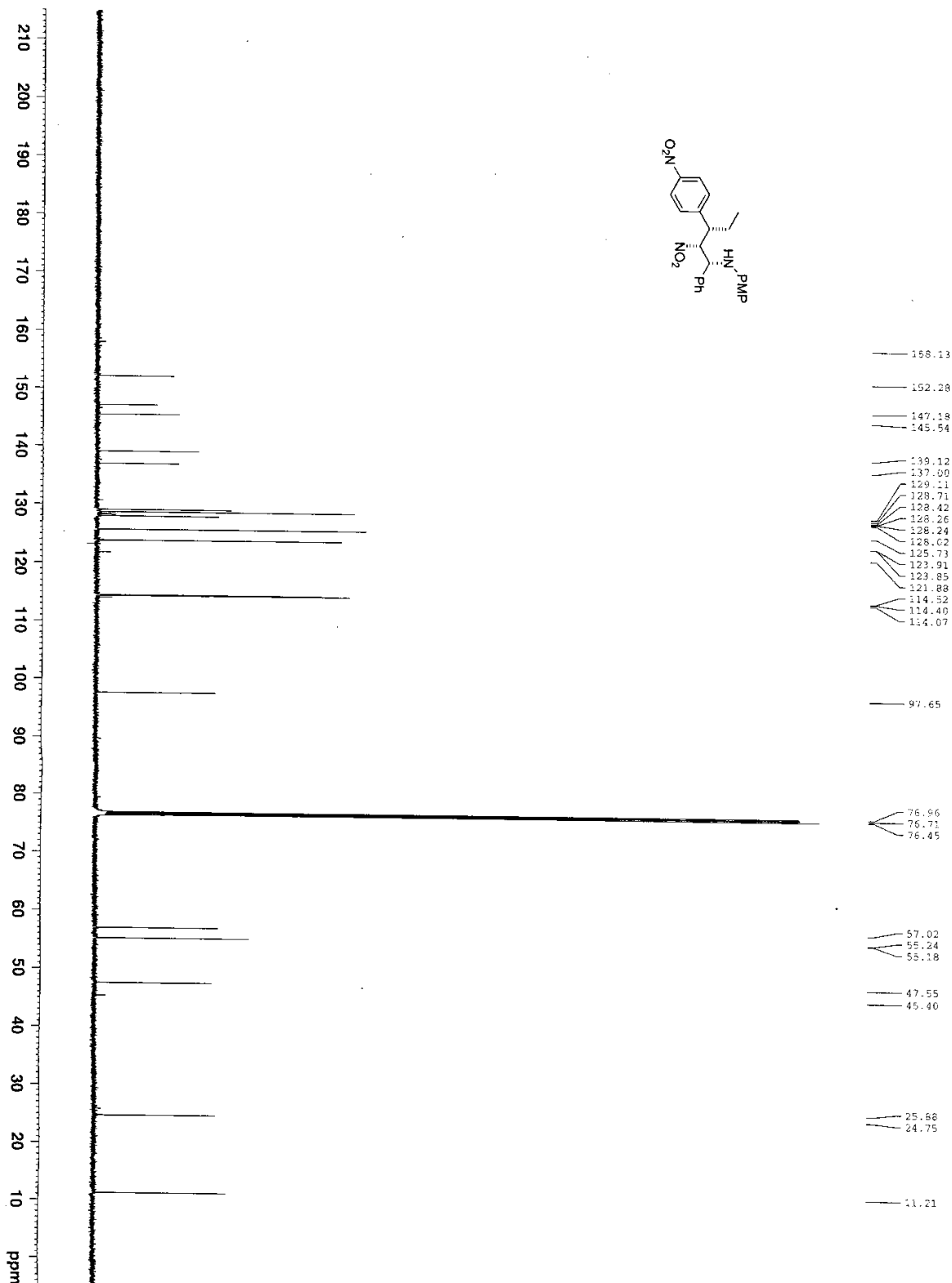
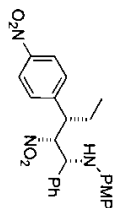
SWH 10330.578 Hz
FIDRES 0.125004 Hz
AQ 3.9999712 sec
RG 228.1
DE 48.400 use
TE 298.0 K
D1 1.00000000 sec
MCPRST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 14.80 use
PL1 0.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 131072
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



mrm768
C13CPD CDC13 v2 | ca 38



Current Data Parameters
NAME Oct09-2009
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091010
Time 16.06
INSTRUM drx500
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
FIDRES 0.456222 Hz
AQ 1.0918410 sec
RG 11385.2
DE 16.650 use
DM 6.00 use
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

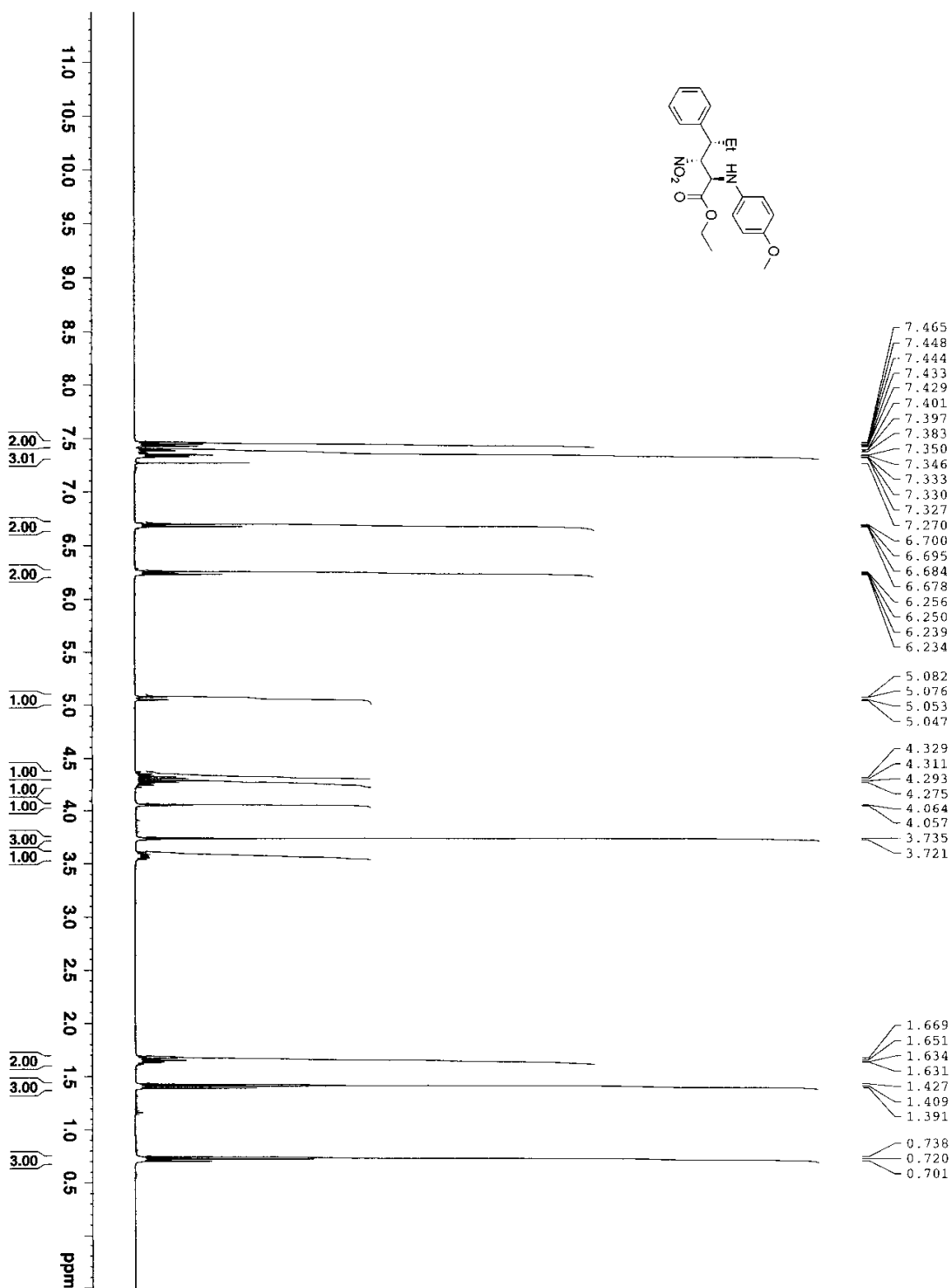
===== CHANNEL f1 =====
NUC1 ¹³C
P1 5.10 use
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 95.00 use
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578314 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

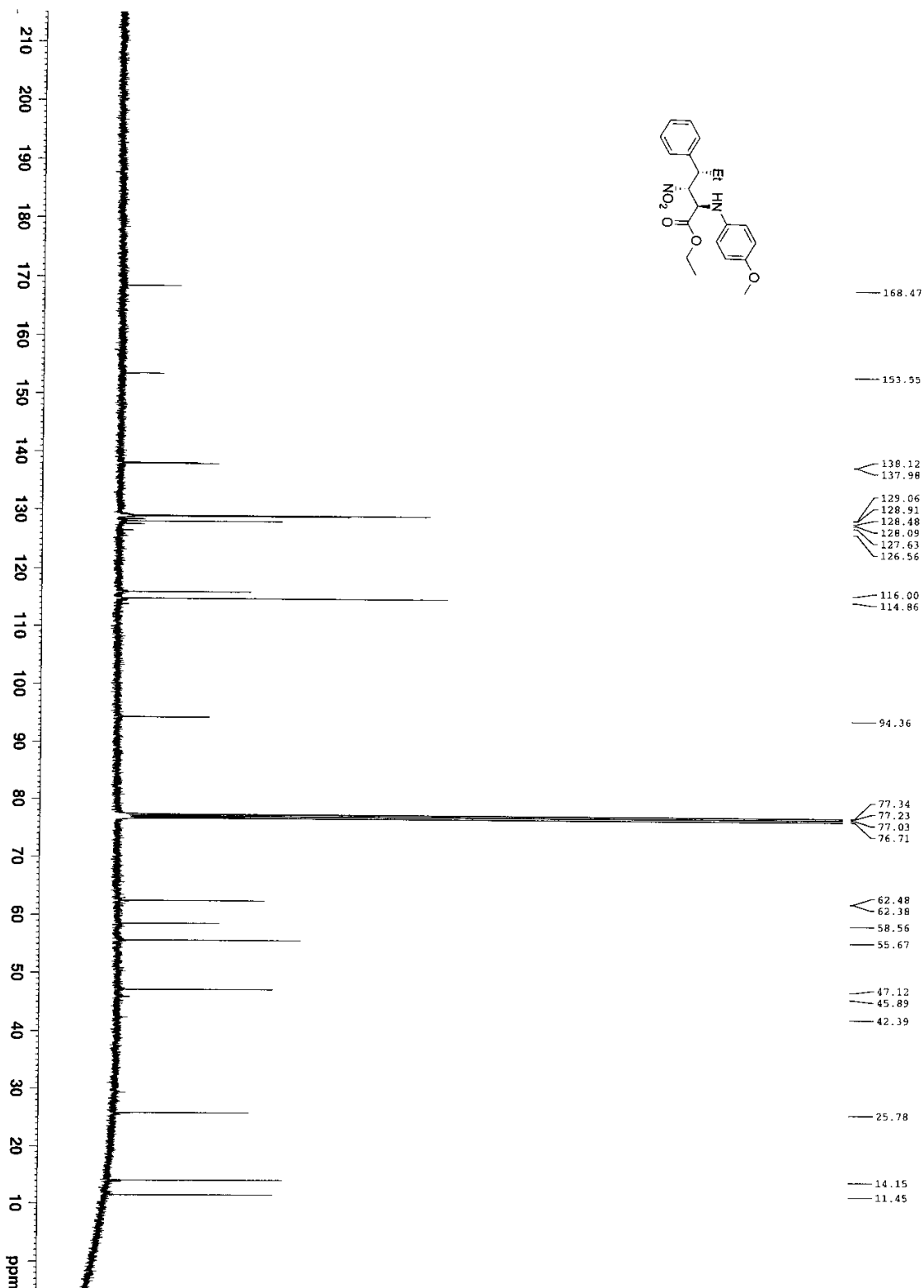
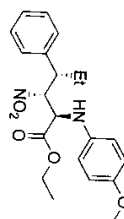
¹H NMR Entry 26

Slot No. 38 Sample ID 580-cryst3 SupervisorID ander Lab Phone No. 13540 UserID g_ste



Current Data Parameters
NAME g_ste:580-cryst3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080430
Time 13.33
INSTRUM dpx400
PROBHD 5 mm PABBO BH/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210251 sec
RG 287.4
DM 104.400 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 13.40 usec
PL1 -6.00 dB
SFO1 400.2022011 MHz
F2 - Processing parameters
SI 32768
SF 400.2000090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

UserID g_ste SampleID 580-cryst3 SupervisorID ander Lab Phone No. 13540 Slot Number 8



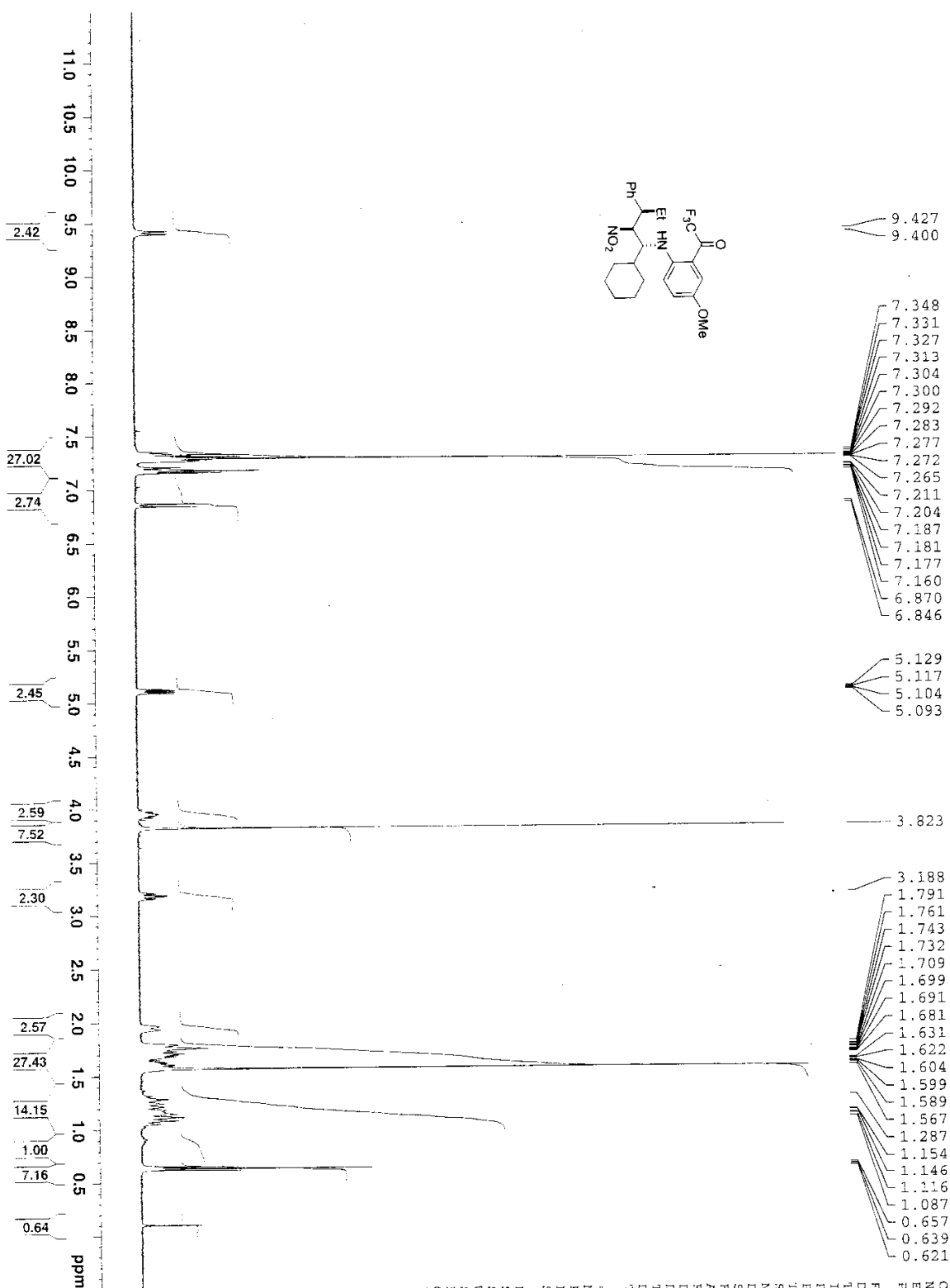
```

NAME g_ste.580-cryst3
EXPNO 1
PROCNO 1
Date_ 20080502
Time 4.18
INSTRUM av400
PROBHD 5 mm PABBO BP
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 114
DE 19.900 use
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 use
PL1 -2.20 dB
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 -2.10 dB
PL12 15.40 dB
PL13 120.00 dB
PL14 120.00 dB
PL15 16.45816231 W
PL16 0.29267216 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127630 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

Slot No. 37 Sample ID mrm488-proton SupervisorID ander Lab Phone No. 1 UserID m_mil

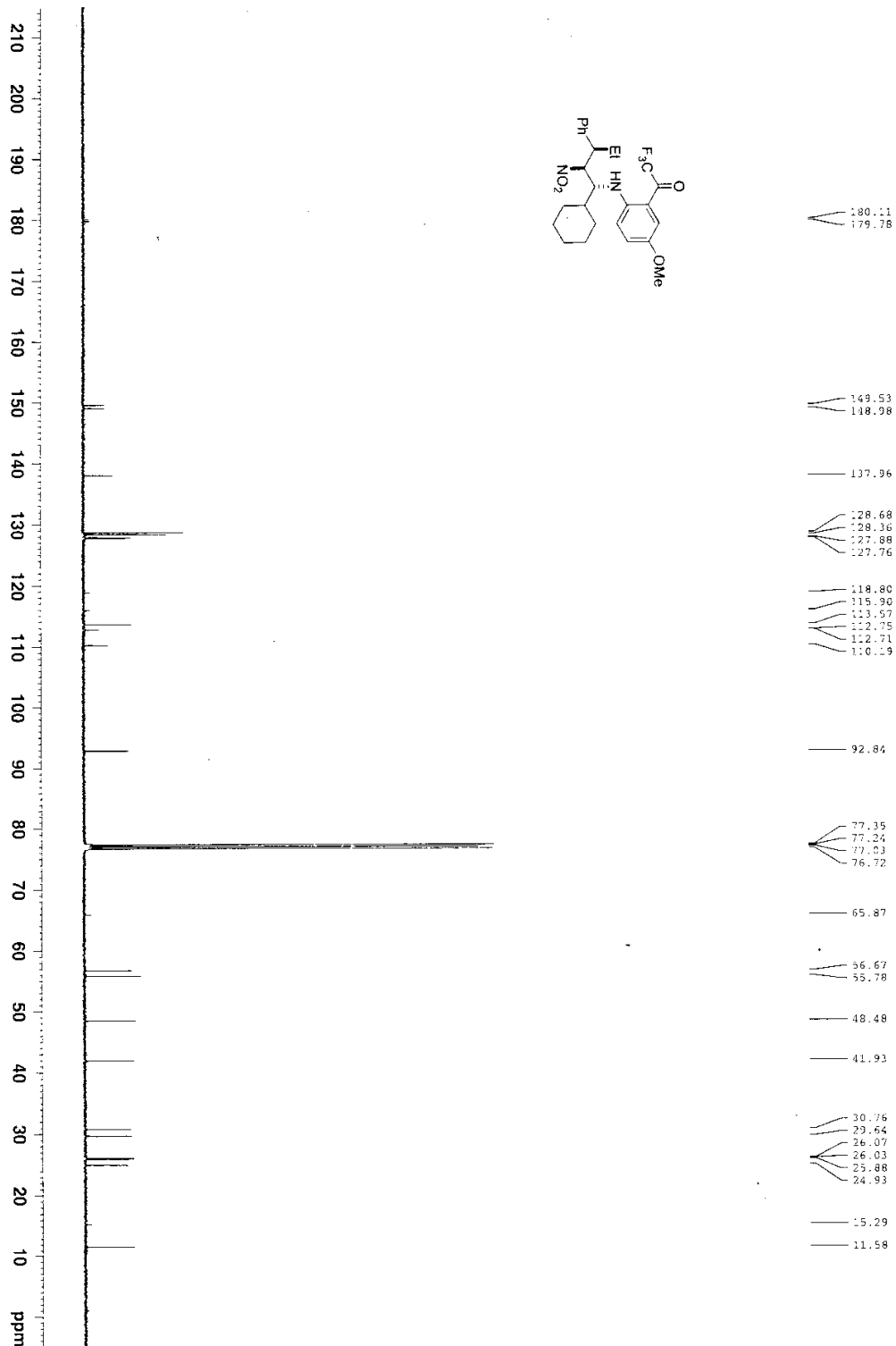
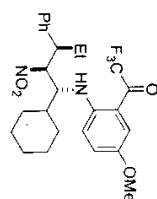


Current Data Parameters:
 Name: m_mil.mrm488-proton
 ExpNO: 1
 F2 - Acquisition Parameters
 Date_: 20080716
 Time: 15.16
 INSTRUM: gpc400
 PROBHD: 5 mm PABO BB/
 PULPROG: zg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 4789.272 Hz
 FIDRES: 0.146157 Hz
 AQ: 3.4210291 sec
 RG: 912.3
 DW: 104.400 usec
 DE: 6.00 usec
 TE: 298.1 K
 D1: 1.0000000 sec
 TDO: 1

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 13.40 usec
 PL1: -6.00 dB
 SFO1: 400.2022011 MHz

F2 Processing parameters
 SI: 32768
 SF: 400.200000 MHz
 WDW: EM
 SSF: 0
 L/R: 0.30 Hz
 GB: 0
 PC: 1.00

Slot No. 28 Sample ID mrm488 SupervisorID ander Lab Phone No. 1 UserID m_mil



```

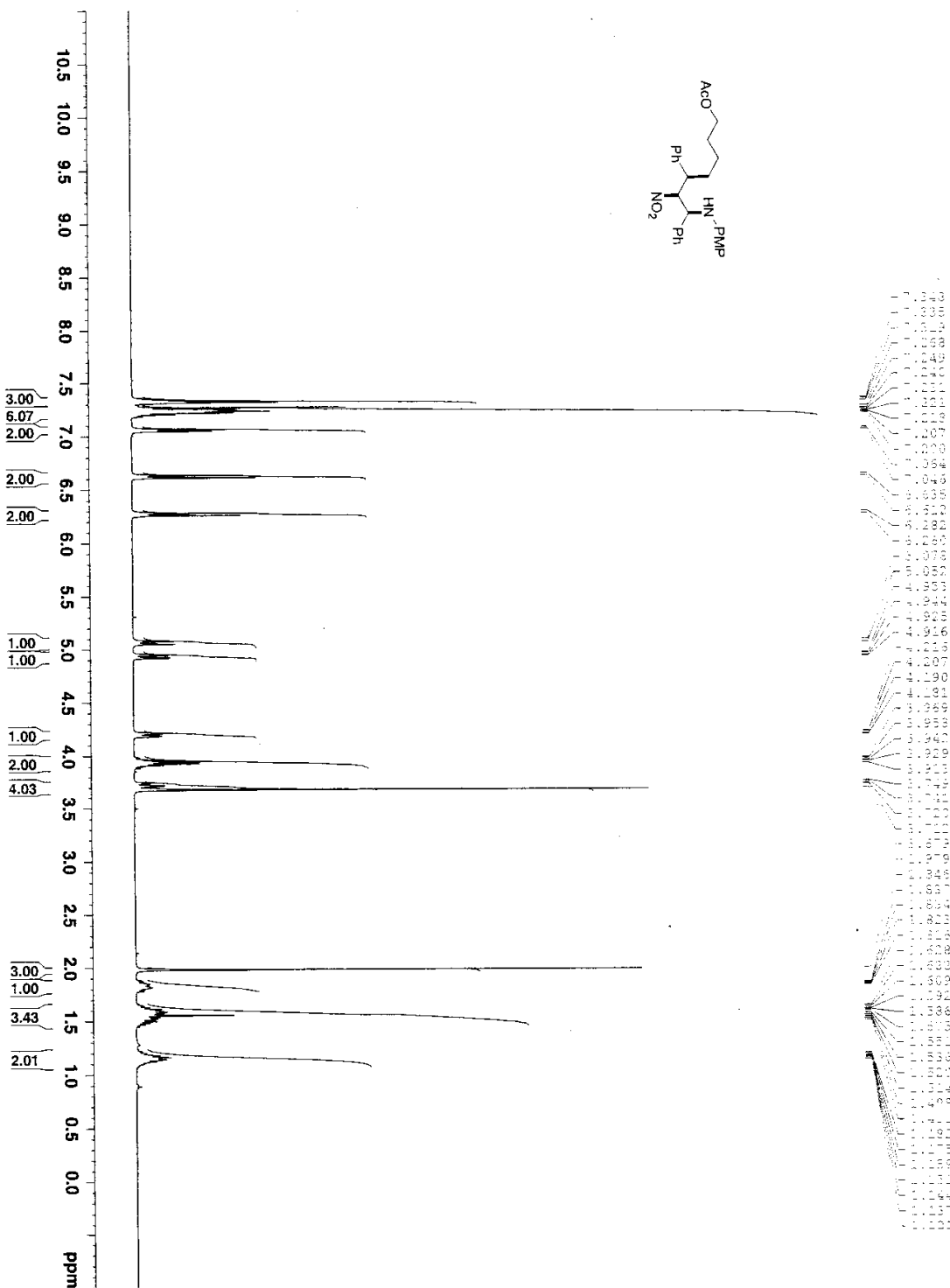
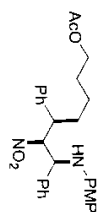
Current Data Parameters
NAME m_mil.mrm488
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080716
Time 2.53
INSTRUM spect
PROBHD 5 mm BBOH BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.65113 sec
AQ 18390.4
RG 19.900 usec
DE 6.00 usec
TE 300.2 K
d1 1.0000000 sec
d11 0.0300000 sec
DELTA 0.8999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 135
PL1 -4.00 dB
SFO1 100.614393 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.7610008 MHz

F2 - Processing parameters
SI 32768
SF 100.610100 MHz
SD 5534
LB 1.00 Hz
GB 0
PC 1.40
  
```

UserID g_ste SampleID 662-2c2a SupervisorID ander Lab Phone No. 13540 Slot Number 35



Current Data Parameters
NAME g_ste.662-2c2a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080708
Time 15.52
INSTRUM av3400
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DM 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.00 usec
PL1 -0.90 dB
PL1W 11.52680206 W
SFO1 400.0724706 MHz

F2 - Processing parameters
SI 65536
SF 400.0700050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Slot No. 20 Sample ID 662-2c2 SupervisorID ander Lab Phone No. 13540 UserID g_ste

Current Data Parameters
NAME g_ste-662-2c2
EXNO 4
PROCNO 1

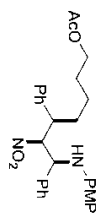
F2 - Acquisition Parameters
Date_ 20080709
Time 7.35

INSTRUM dpx400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 9195.2
DW 19.900 usec
TE 6.00 usec
DE 298.0 K
D1 1.0000000 sec
d11 0.0300000 sec
DELTA 0.8999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 -4.00 dB
SFO1 100.6414393 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
PL13 12.00 dB
SFO2 400.2016008 MHz

F2 - Processing parameters
SI 32768
SF 100.6303700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



171.09
152.27
139.94
138.10
138.02
129.22
128.91
128.31
128.07
127.89
126.07
114.73
114.71
98.86
77.35
77.24
77.03
76.72
64.04
57.00
55.61
46.19
31.53
28.15
23.46
20.93

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm