Stereoselective Synthesis of 1,2-Diamine Containing Indolines by a Conjugate Addition Nitro-Mannich Reaction

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Supplementary Information

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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/UV_{254} 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.

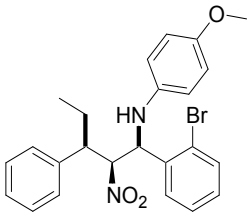
Toluene was obtained form a solvent tower, where degassed toluene was passed through two columns of activated alumina and a 7 micron filter under 4 bar pressure. Dichloromethane was distilled from calcium hydride powder or purchased as an analytical grade and stored over 4Å molecular sieves. Anhydrous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

Characterization:

Melting points are uncorrected. Infrared spectra were recorded as a thin film on sodium chloride discs or as dilute chloroform solutions. ^{1}H and ^{13}C NMR spectra were recorded as dilute solutions in deuterochloroform unless otherwise stated. All chemical shifts (δ) are reported in parts per million (ppm) relative to residual solvent peaks. All chemical shifts are reported relative to chloroform (δ_H = 7.27 ppm, δ_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 ^{1}H 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. For mass spectrometry, high resolution (HR) electrospray ionization (ES^+) mass data was obtained using a time-of-flight (TOF) analyzer, and HR chemical ionization (CI) or electron impact (EI) mass data were recorded using an ion trap.

Structure and X-ray structure:



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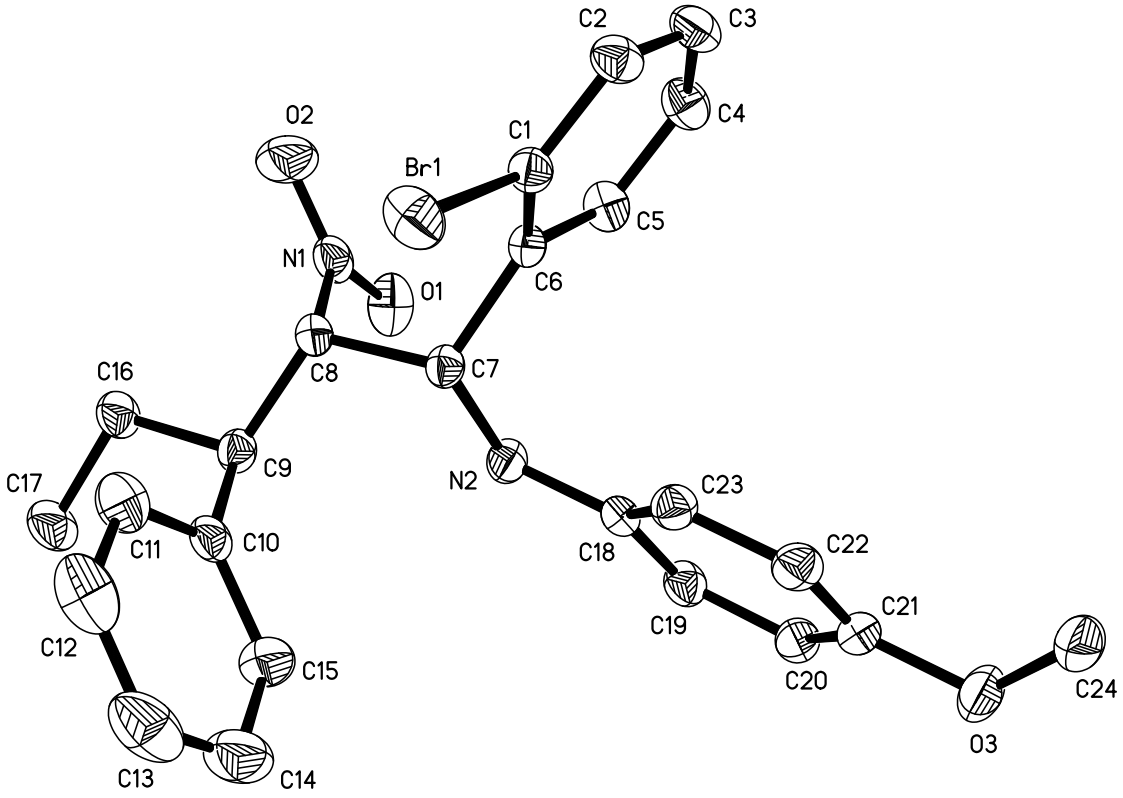
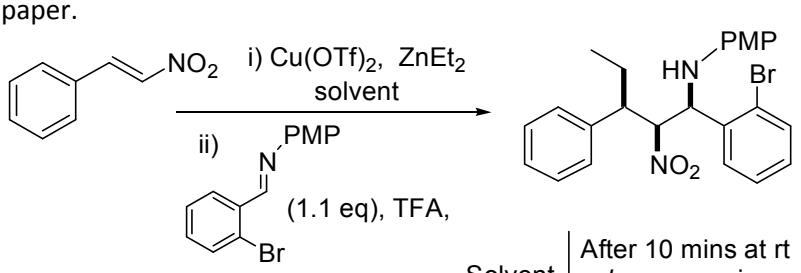


Crystallographic data (excluding structure factors) for this structure has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 1020679. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

Chemical formula	$C_{24}H_{25}BrN_2O_3$	
Formula weight	469.37	
Temperature	150(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	monoclinic, $P2_1/c$	
Unit cell parameters	a = 15.139(3) Å	α = 90°
	b = 10.0225(18) Å	β = 103.866(3)°
	c = 30.107(6) Å	γ = 90°
Cell volume	4435.1(14) Å^3	
Z	8	
Calculated density	1.406 g/cm^3	
Absorption coefficient μ	1.881 mm^{-1}	
F(000)	1936	
Crystal colour and size	yellow, 0.48 × 0.42 × 0.14 mm^3	
Data collection method	Bruker SMART APEX diffractometer ω/θ scans	
θ range for data collection	2.46 to 28.31°	
Index ranges	h −20 to 19, k −13 to 13, l −38 to 40	
Completeness to θ = 26.00°	99.6 %	
Reflections collected	36089	
Independent reflections	10436 (R_{int} = 0.0379)	
Reflections with $F^2>2\sigma$	8169	
Absorption correction	semi-empirical from equivalents	
Min. and max. transmission	0.4654 and 0.7787	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0640, 0.3959	
Data / restraints / parameters	10436 / 0 / 541	
Final R indices [$F^2>2\sigma$]	R1 = 0.0358, wR2 = 0.0953	
R indices (all data)	R1 = 0.0504, wR2 = 0.1023	
Goodness-of-fit on F^2	0.971	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.696 and −0.564 e Å^{-3}	

Solvent screen

Method used was 'General Procedure for the Synthesis of *syn, syn*-β-Nitroamines 3 (Table 1)' in main paper.



Solvent	After 10 mins at rt *dr* - conversion
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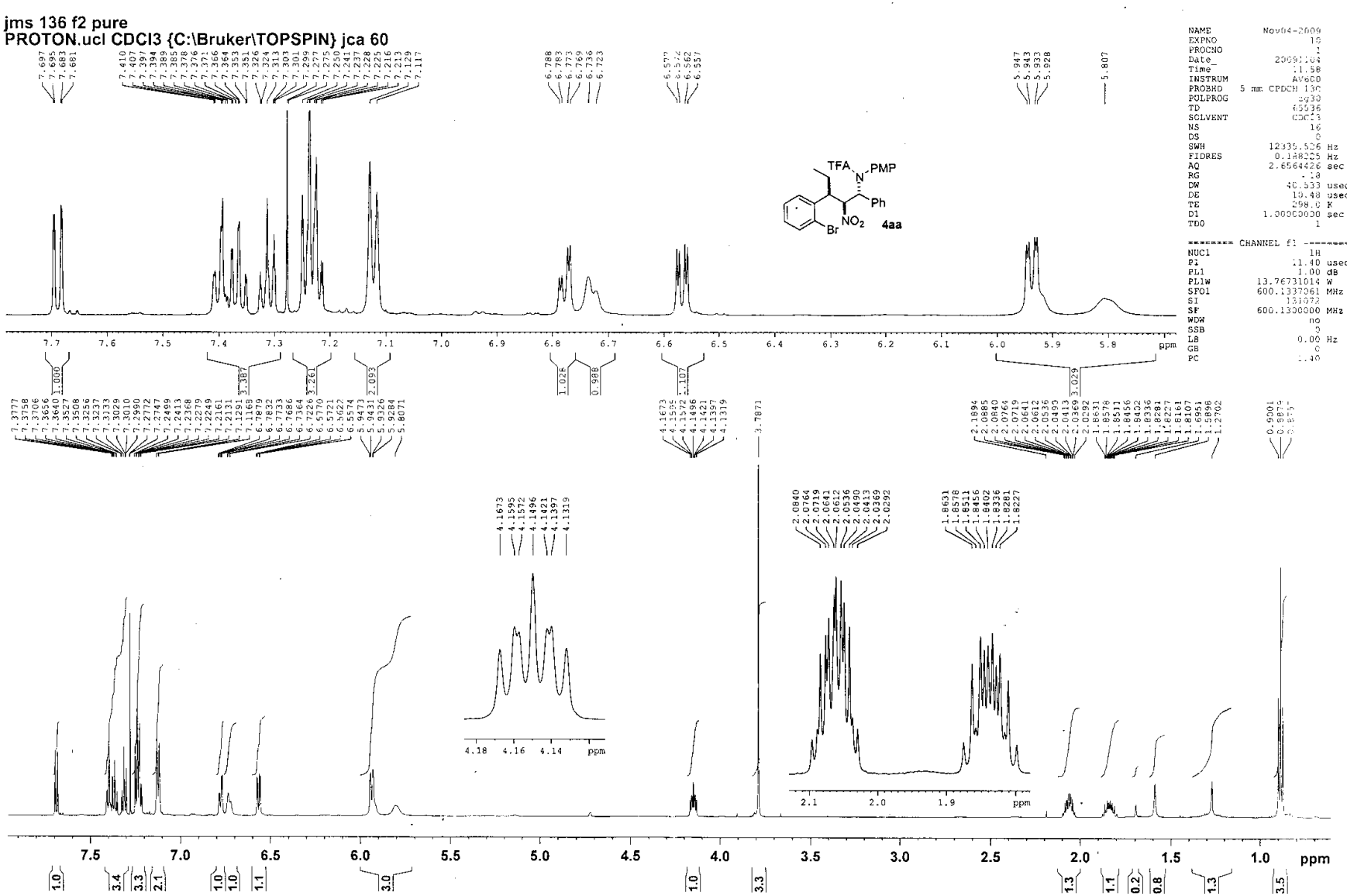
THF	50 : 50 - 100%
Toluene	80 : 20 - 80%
DME	65 : 35 - 100%
DCM	75 : 25 - 100%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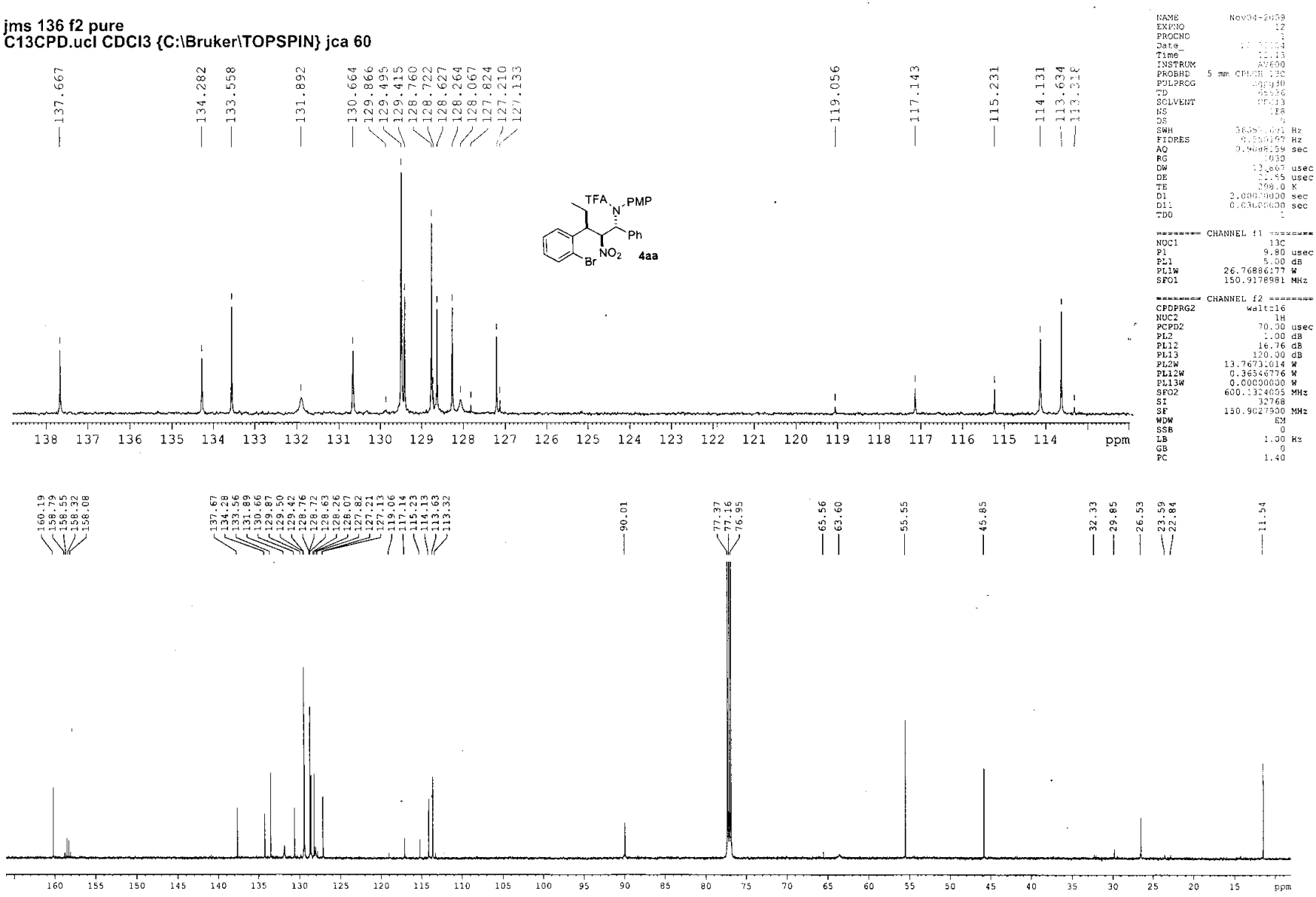
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115.231
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Ph
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Br
4aa
ppm
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158.32
158.08
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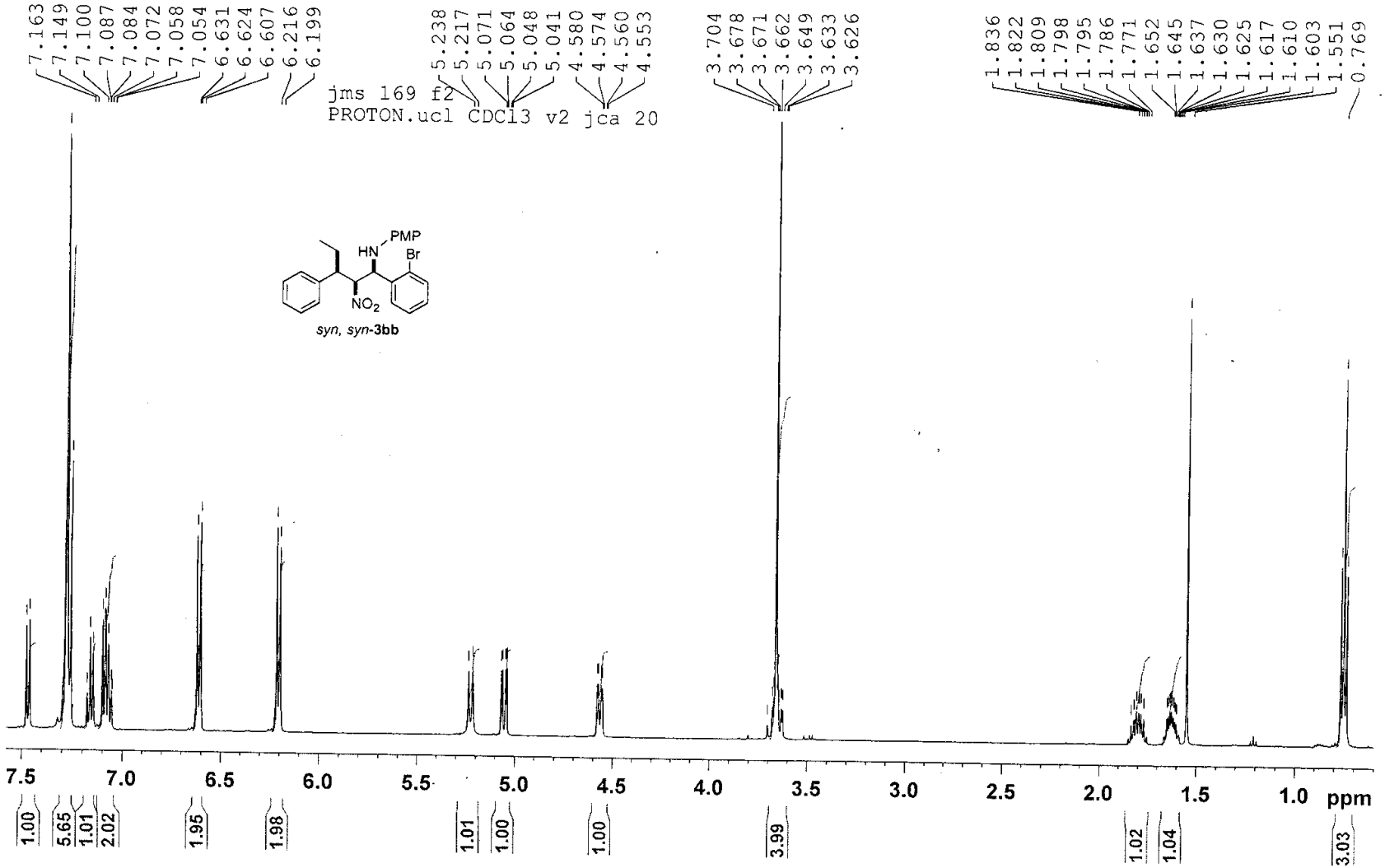
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3.03

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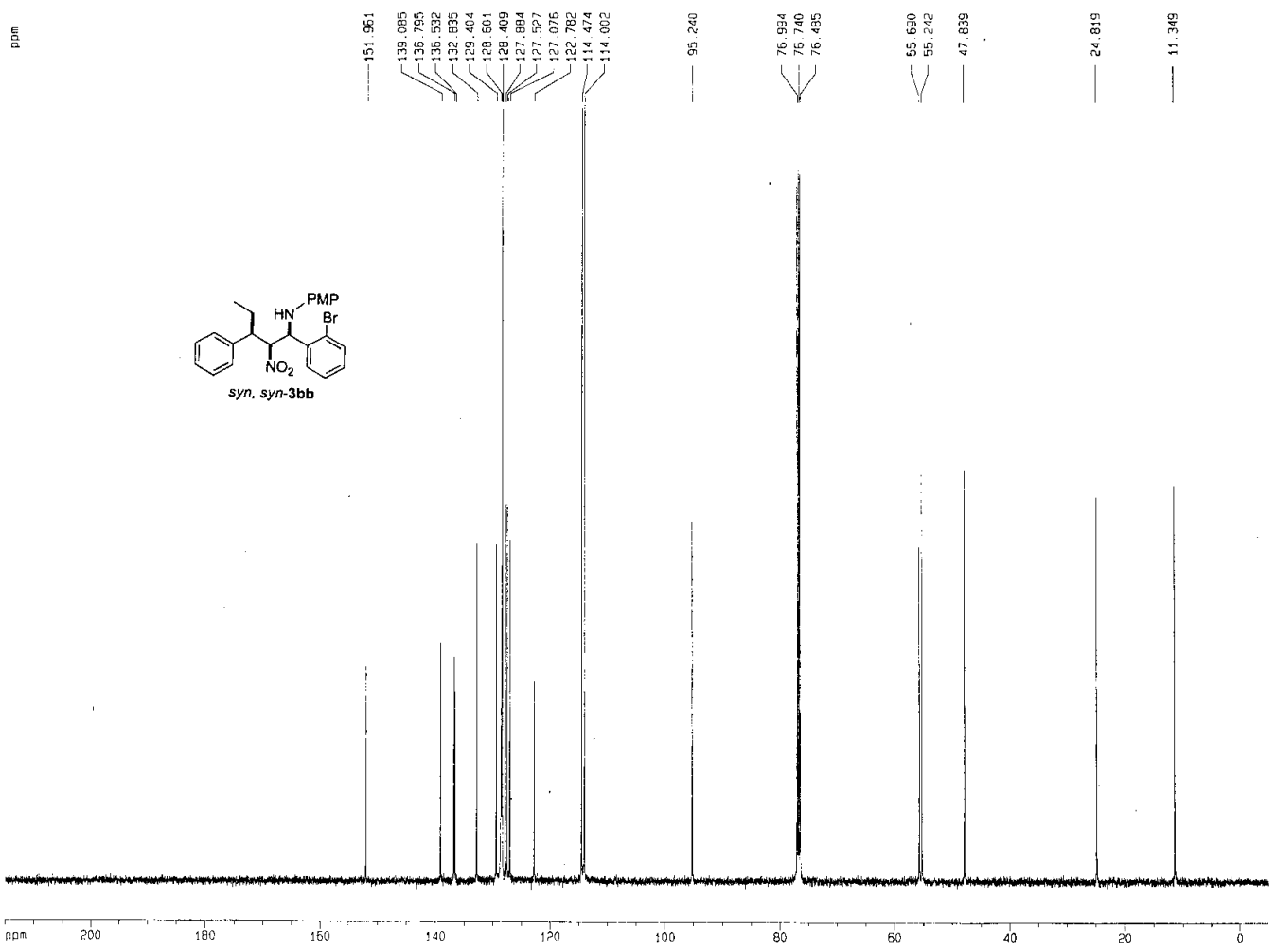


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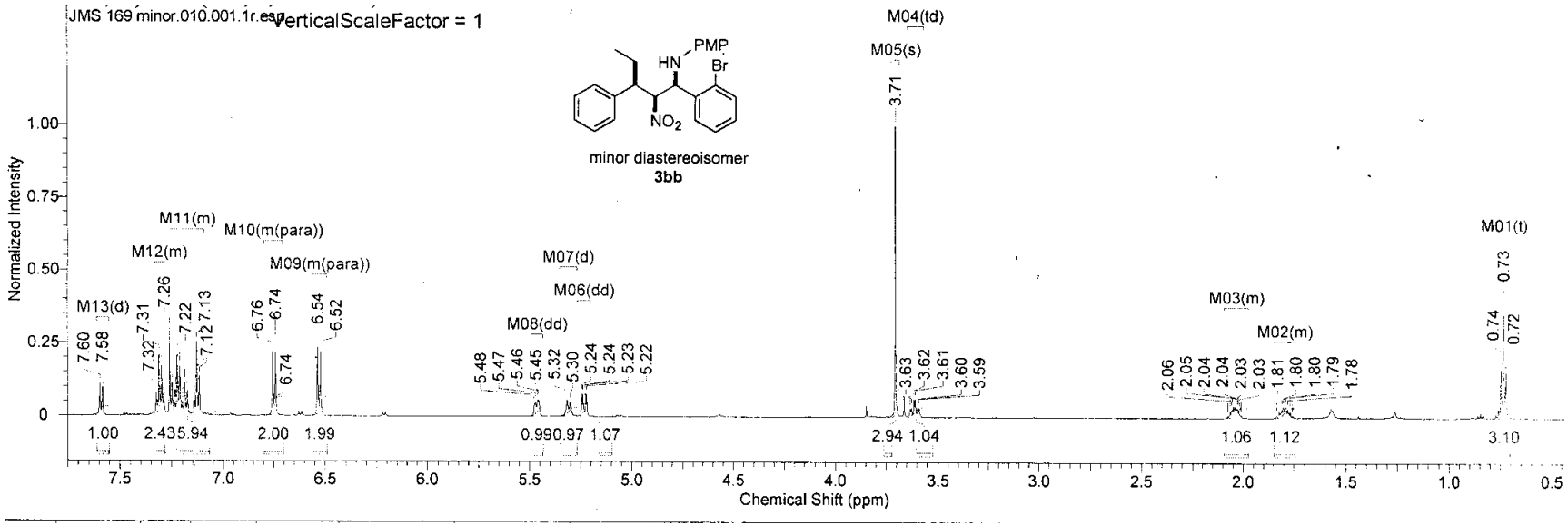
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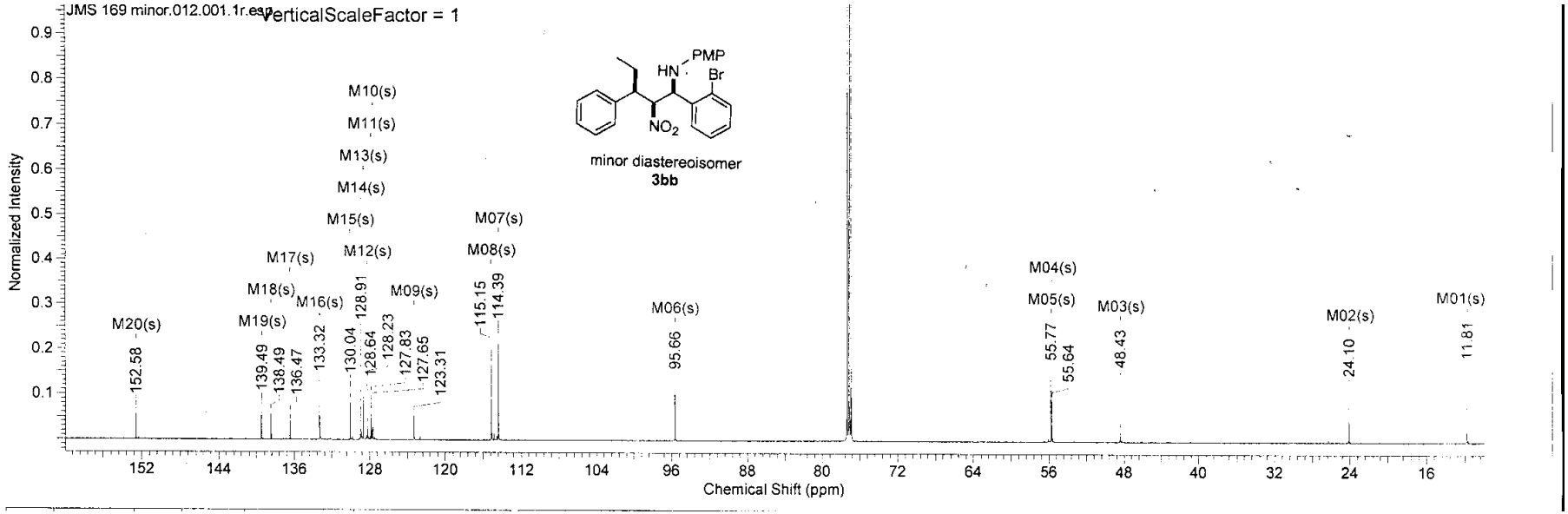
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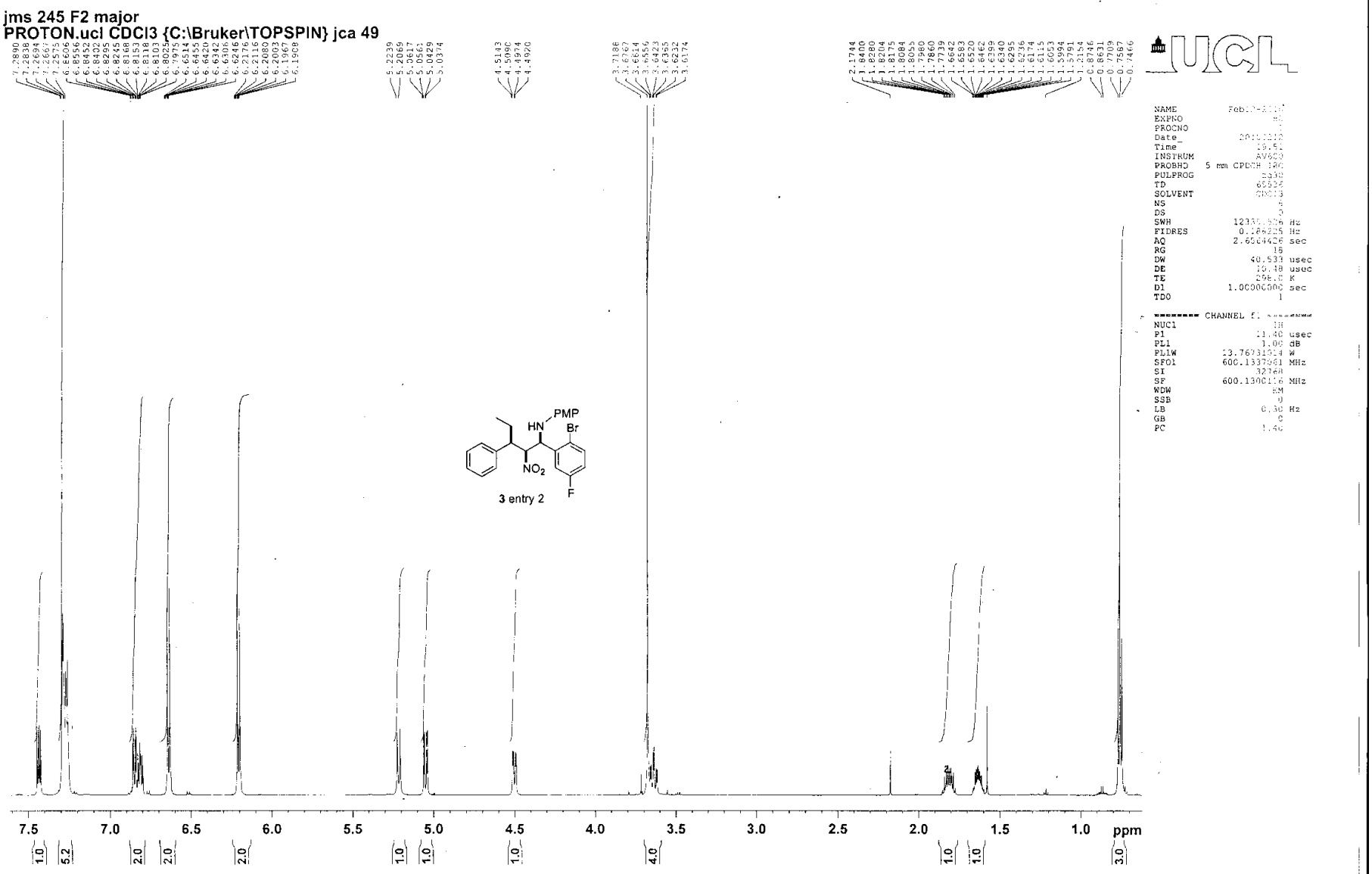
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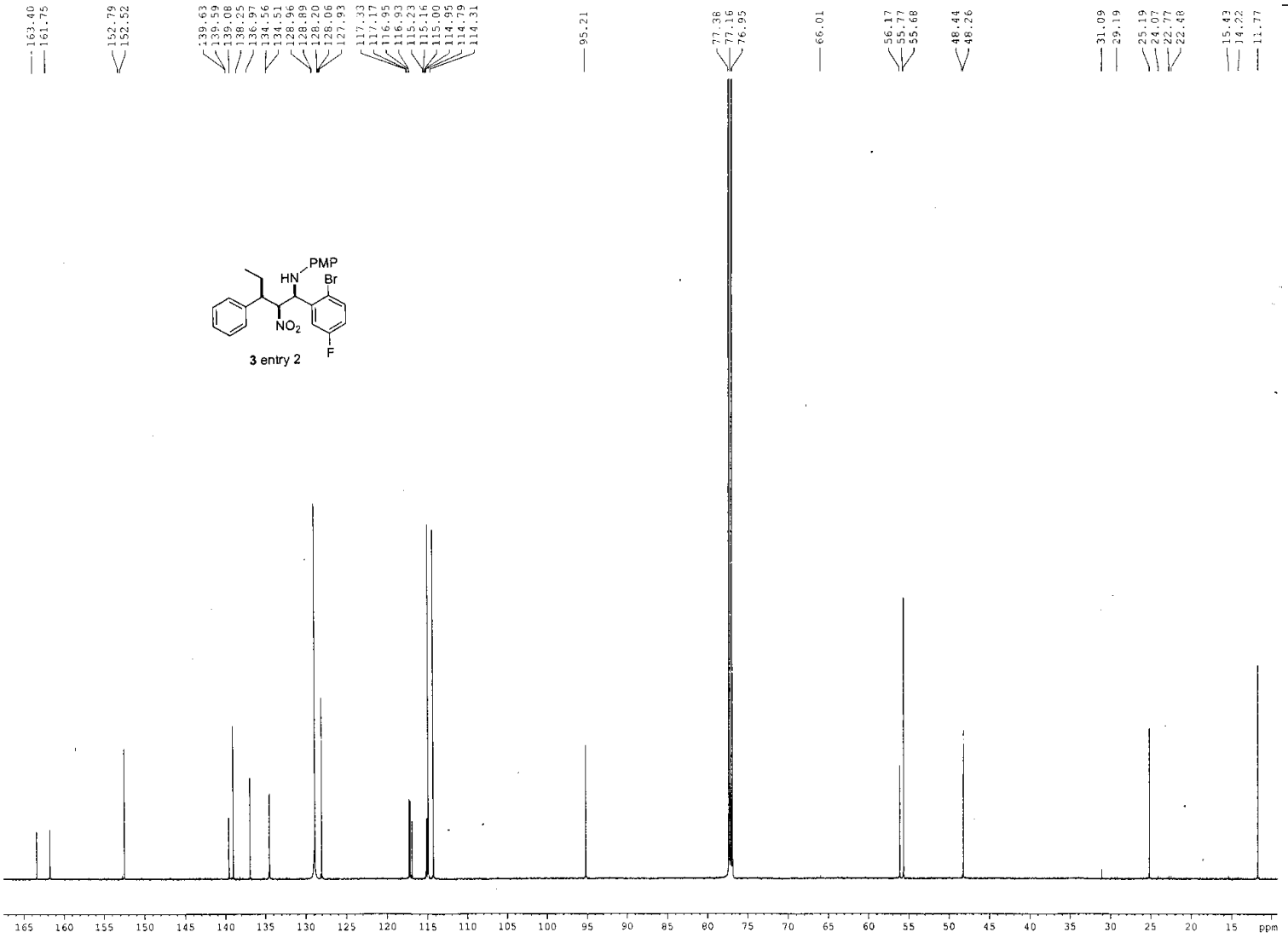

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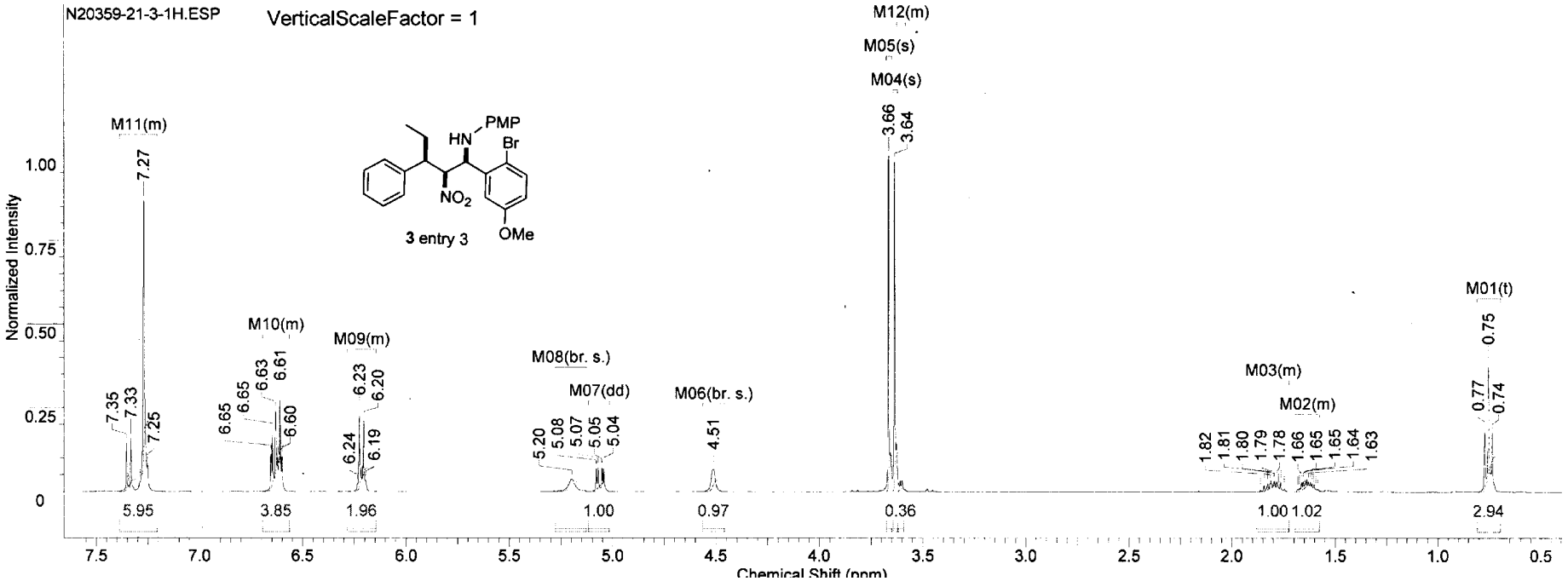
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M02(m)
M01(t)
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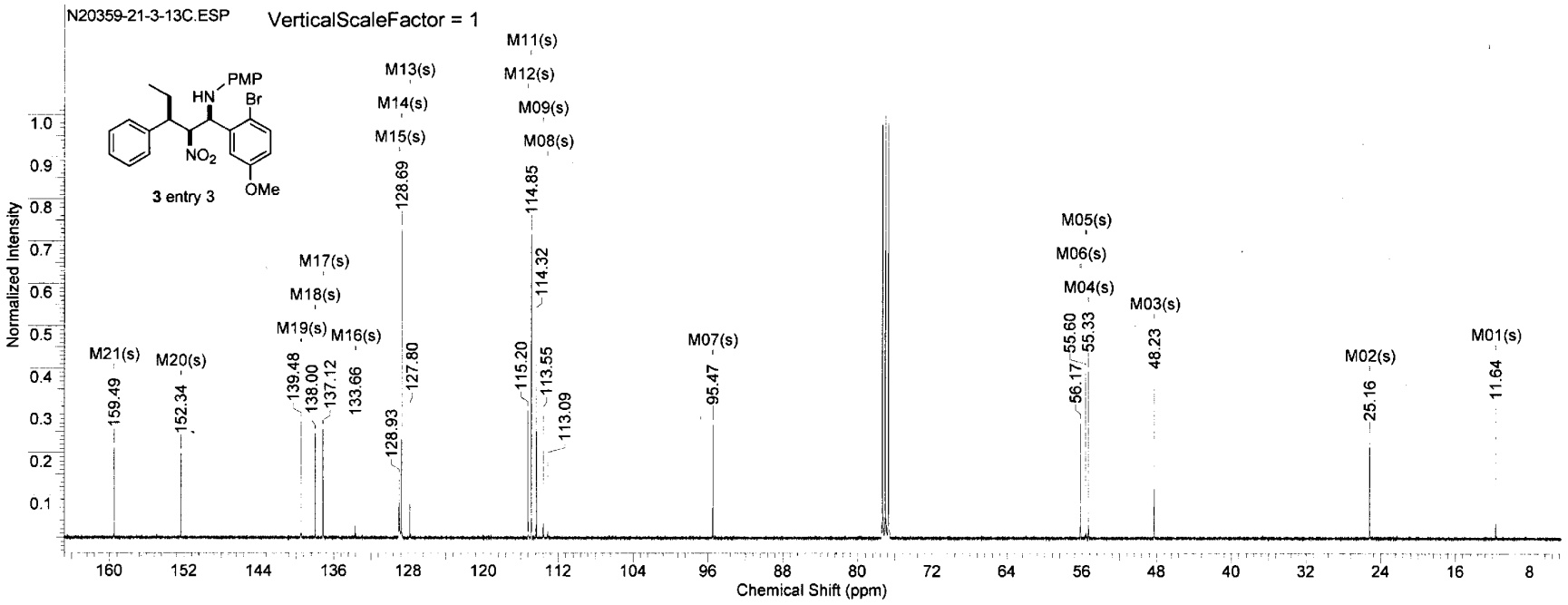
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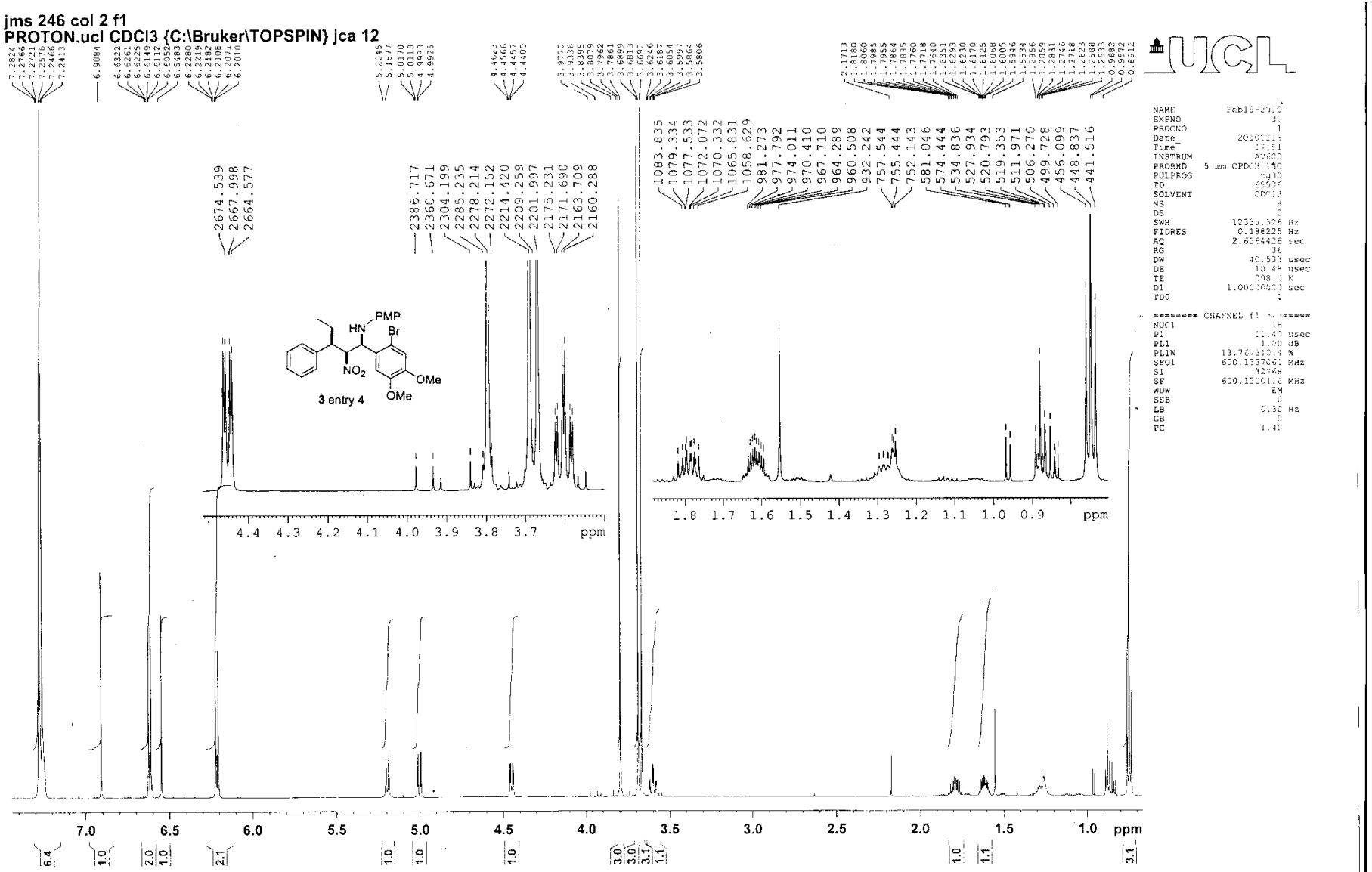
Chemical Shift (ppm)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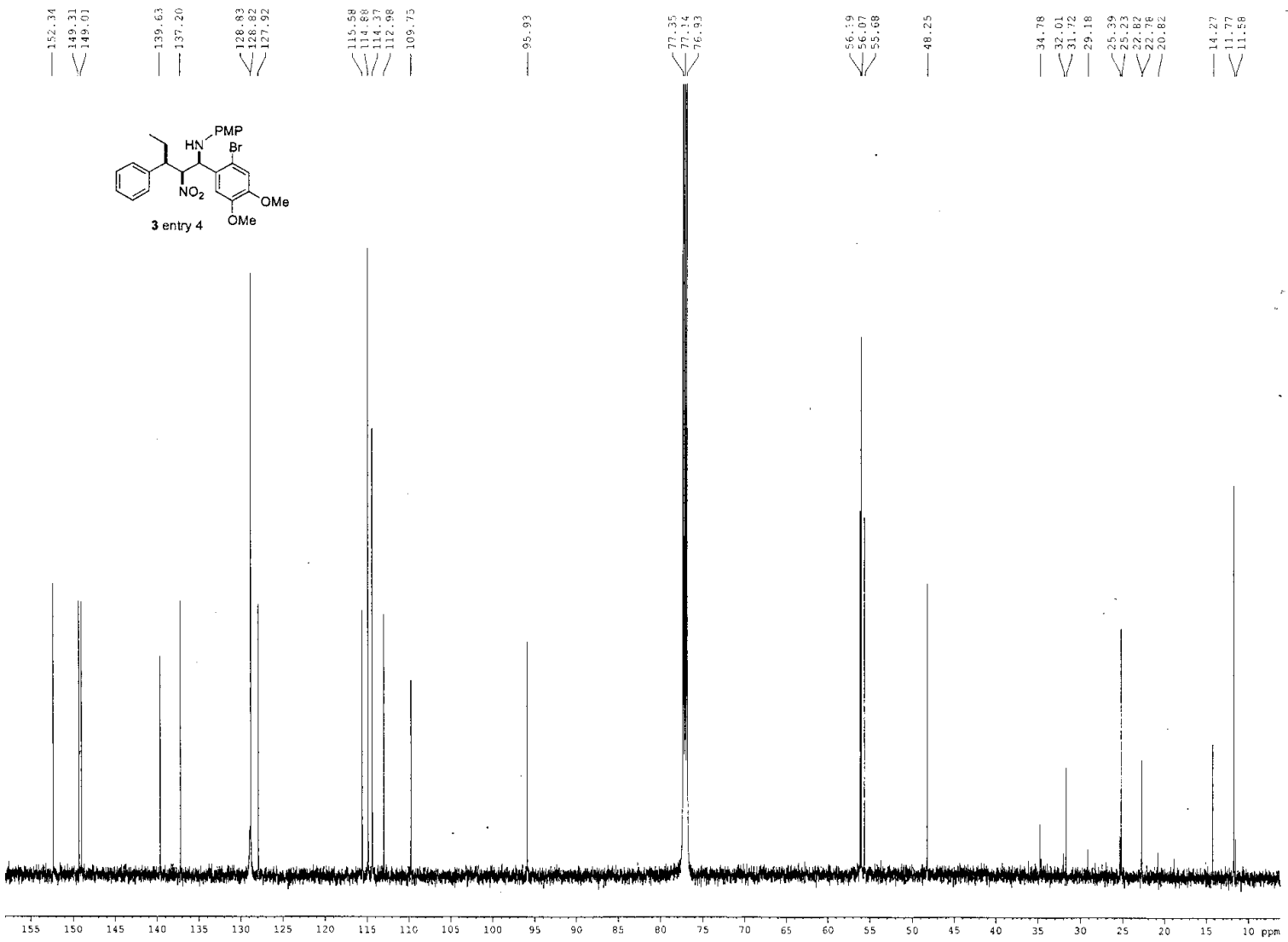

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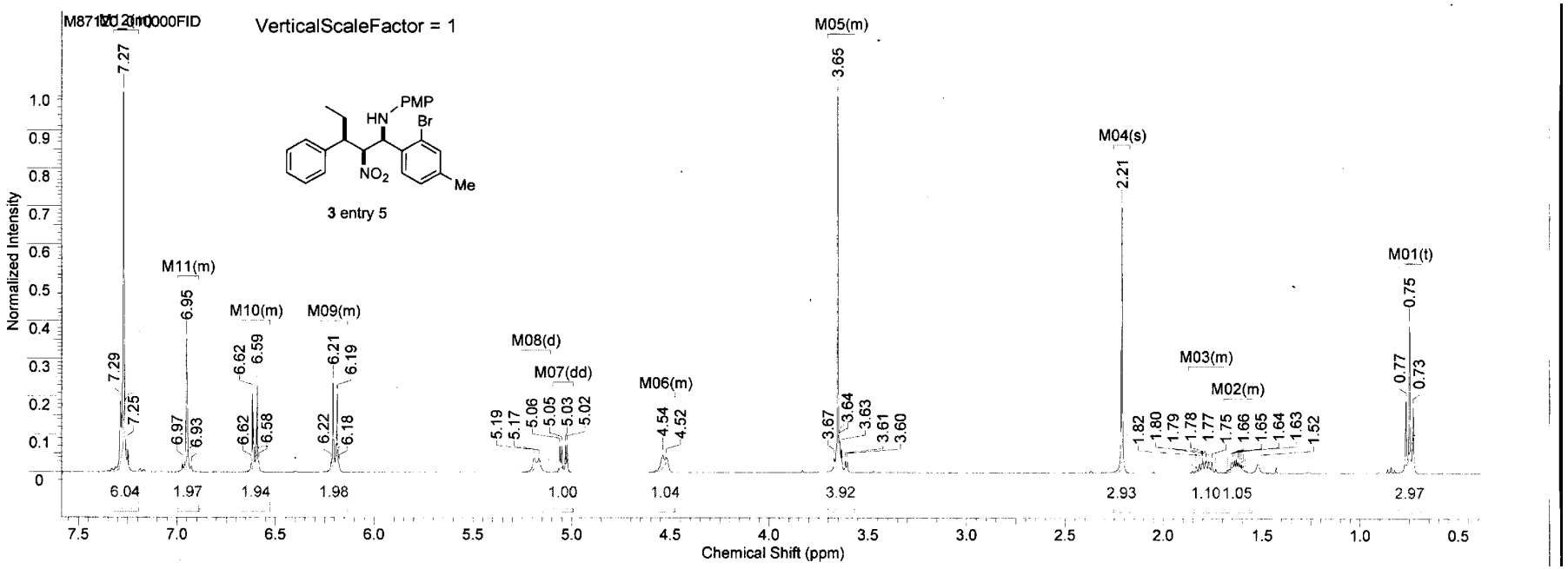
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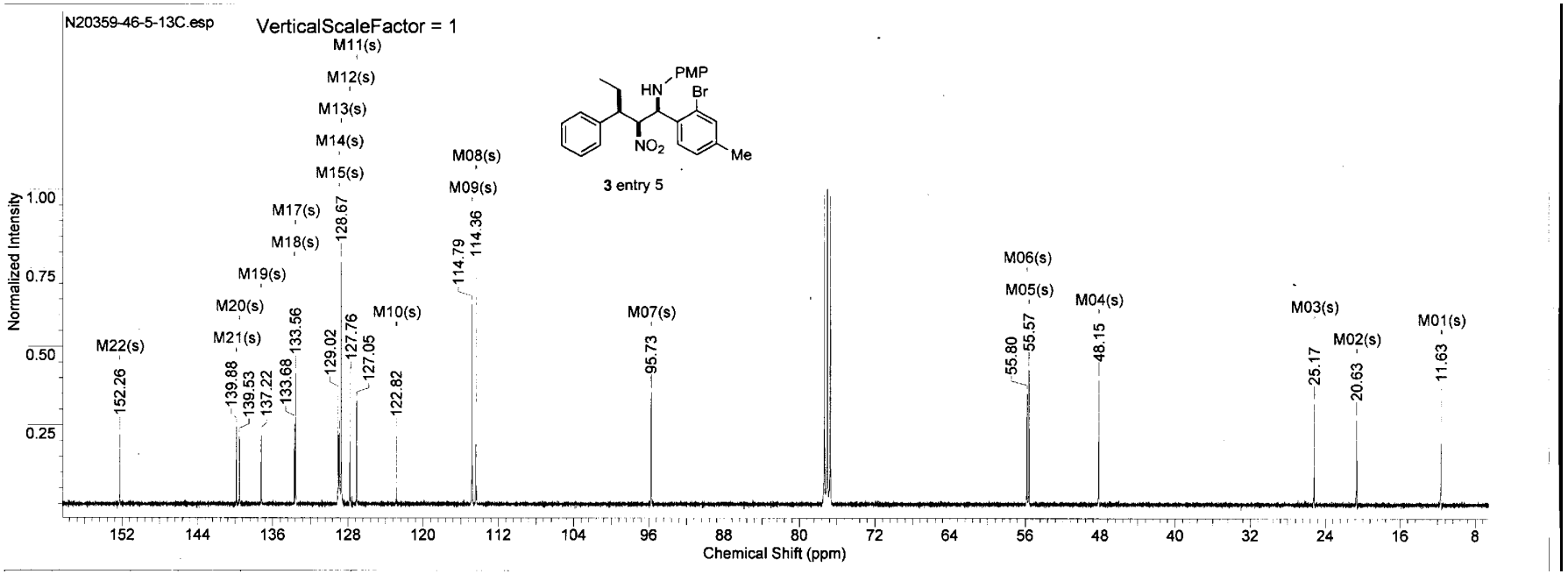
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Br
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Me
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144
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128
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96
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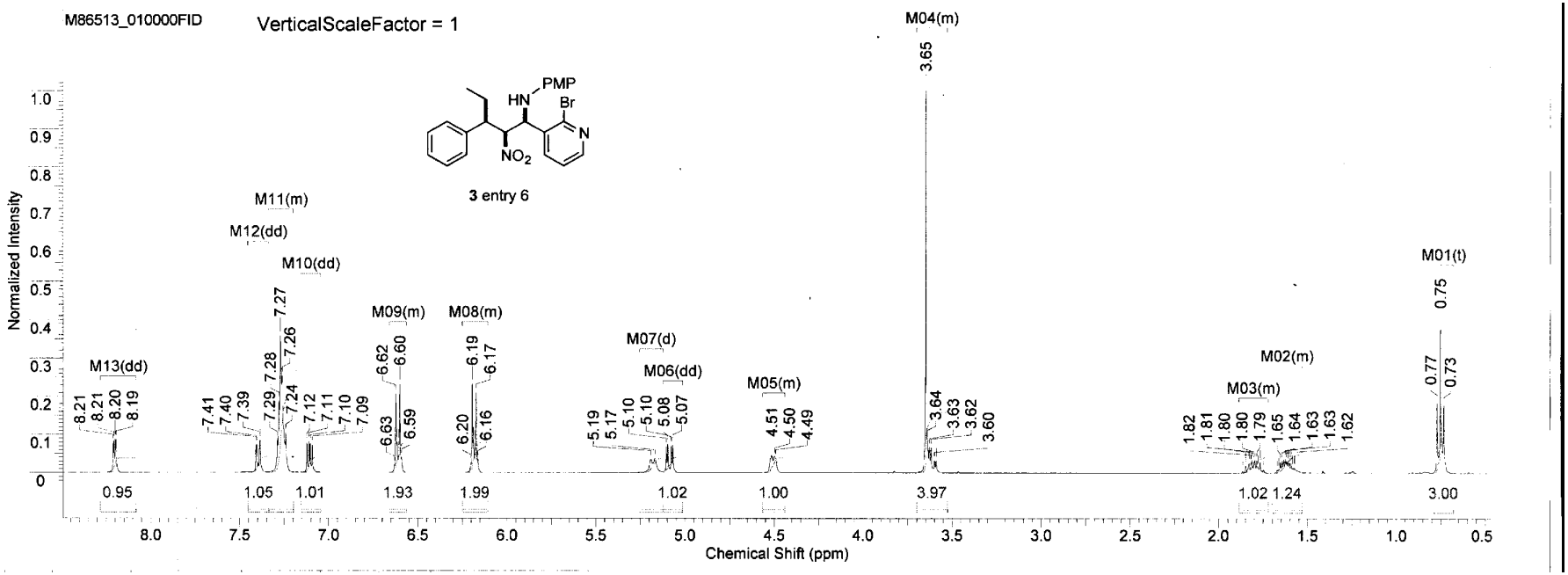
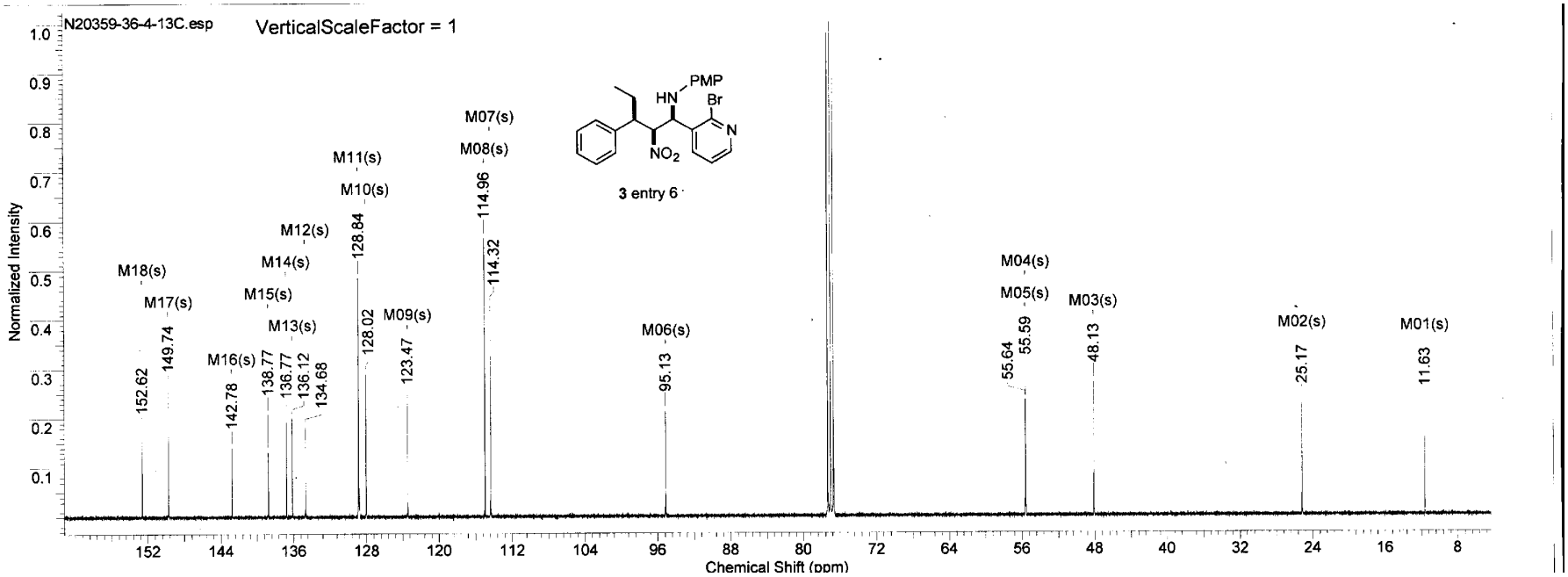
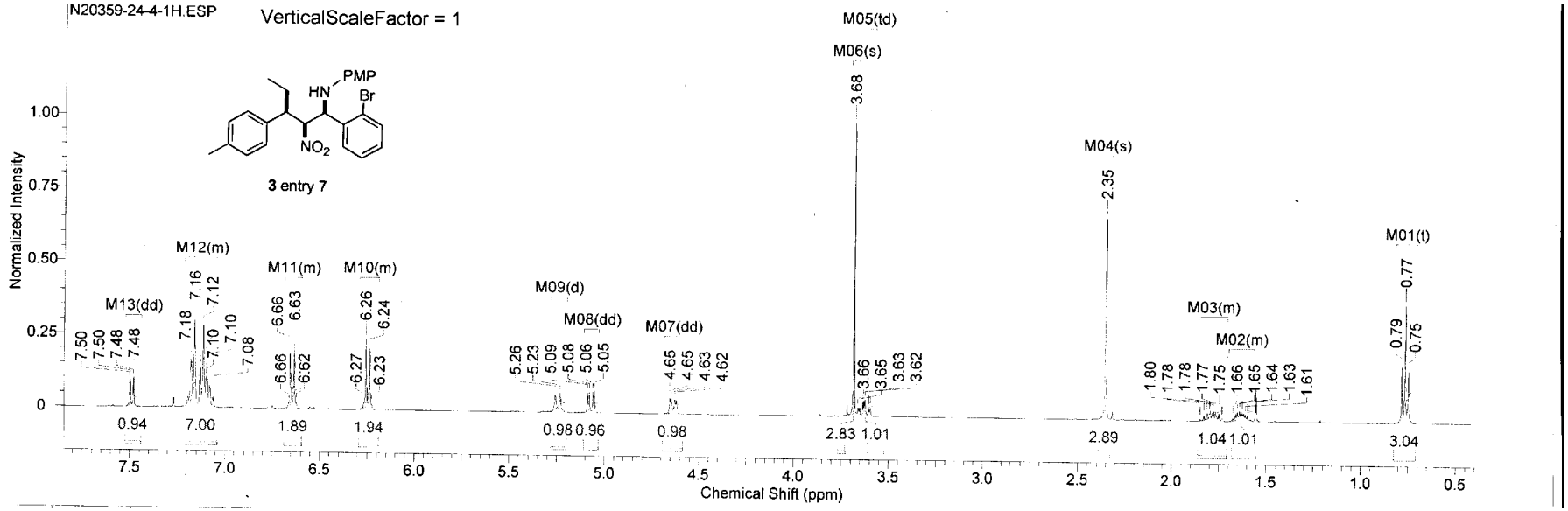
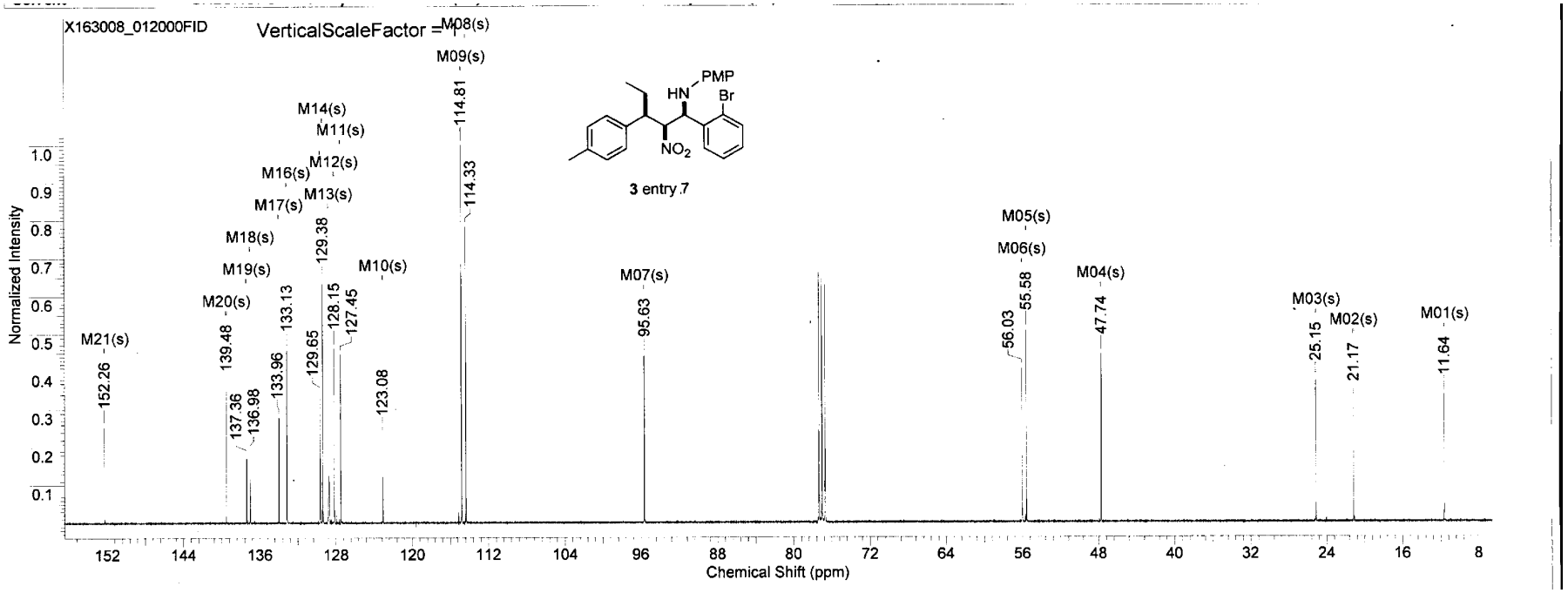
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)

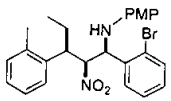

M86513_010000FID
VerticalScaleFactor = 1
PMP
HN
Br
N
NO2
3 entry 6
M04(m)
3.65
M11(m)
M12(dd)
M10(dd)
M09(m)
M08(m)
M07(d)
M06(dd)
M05(m)
M02(m)
M03(m)
M01(t)
M13(dd)
8.21
8.21
8.20
8.19
7.41
7.40
7.39
7.29
7.28
7.27
7.26
7.24
7.12
7.11
7.10
7.09
6.63
6.62
6.60
6.59
6.20
6.19
6.17
6.16
5.19
5.17
5.10
5.10
5.08
5.07
4.51
4.50
4.49
3.64
3.63
3.62
3.60
1.82
1.81
1.80
1.80
1.79
1.65
1.64
1.63
1.63
1.62
0.77
0.75
0.73
0.95
1.05
1.01
1.93
1.99
1.02
1.00
3.97
1.02
1.24
3.00
Normalized Intensity
Chemical Shift (ppm)


N20359-36-4-13C.esp
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
PMP
HN
Br
N
NO2
3 entry 6
M18(s)
M17(s)
M16(s)
M15(s)
M14(s)
M13(s)
M12(s)
M11(s)
M10(s)
M09(s)
M08(s)
M07(s)
M06(s)
M05(s)
M04(s)
M03(s)
M02(s)
M01(s)
152.62
149.74
142.78
138.77
136.77
136.12
134.68
128.84
128.02
123.47
114.96
114.32
95.13
55.64
55.59
48.13
25.17
11.63

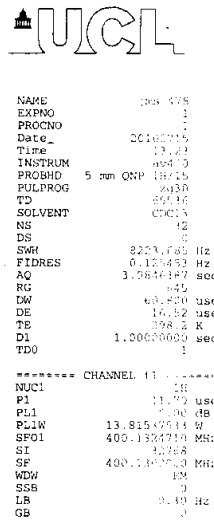

N20359-24-4-1H.ESP
VerticalScaleFactor = 1
PMP
HN
Br
NO2
3 entry 7
Normalized Intensity
Chemical Shift (ppm)
M13(dd)
M12(m)
M11(m)
M10(m)
M09(d)
M08(dd)
M07(dd)
M06(s)
M05(td)
M04(s)
M03(m)
M02(m)
M01(t)
0.94
7.00
1.89
1.94
0.98
0.96
0.98
2.83
1.01
2.89
1.04
1.01
3.04


X163008_012000FID
VerticalScaleFactor =
M08(s)
M09(s)
M14(s)
M11(s)
M12(s)
M16(s)
M13(s)
M17(s)
M18(s)
M19(s)
M20(s)
M21(s)
M10(s)
M07(s)
M05(s)
M06(s)
M04(s)
M03(s)
M02(s)
M01(s)
114.81
114.33
129.38
133.13
128.15
127.45
129.65
133.96
136.98
137.36
139.48
123.08
152.26
95.63
56.03
55.58
47.74
25.15
21.17
11.64
PMP
HN
Br
NO2
3 entry 7
Normalized Intensity
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)

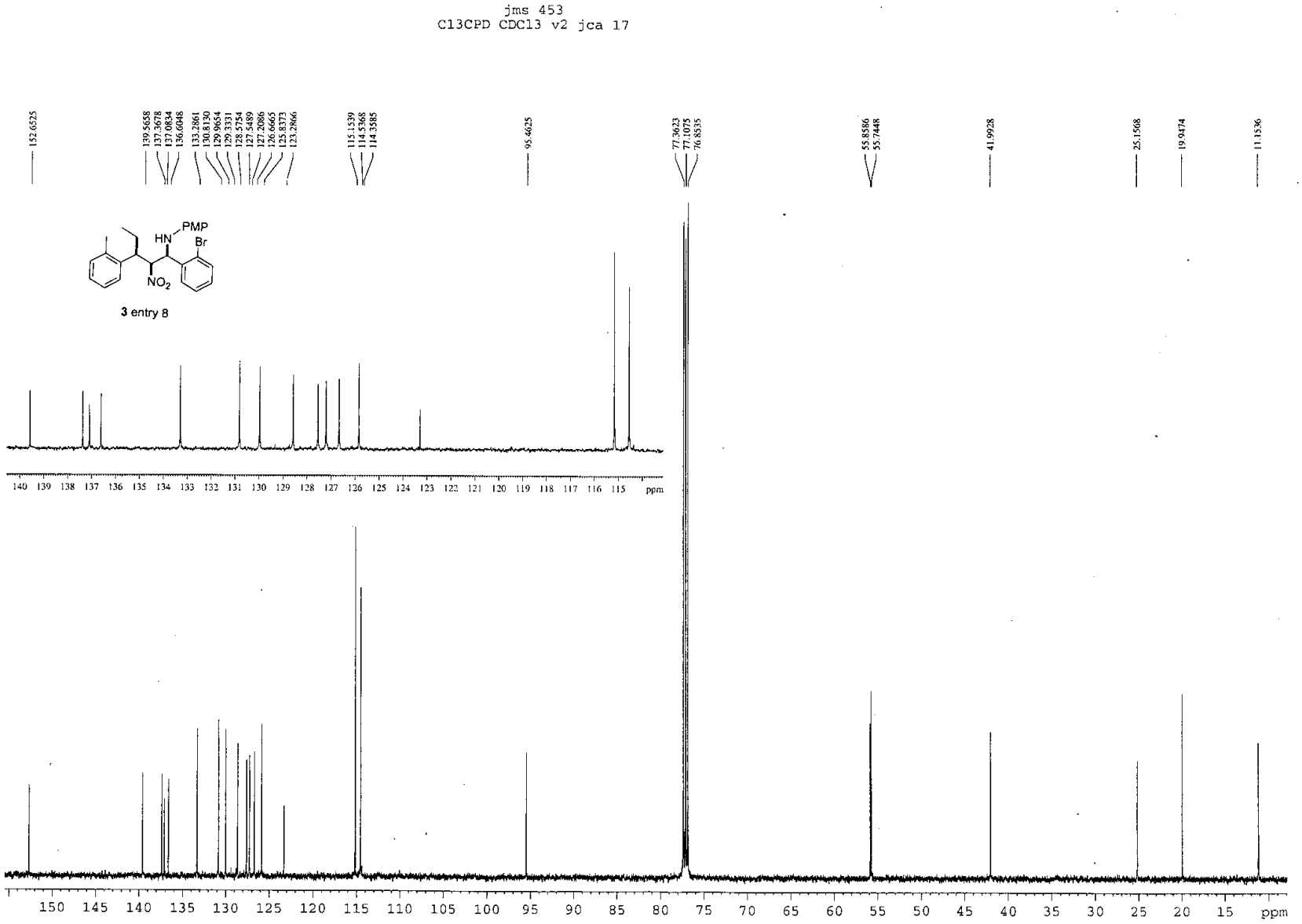
jms 478

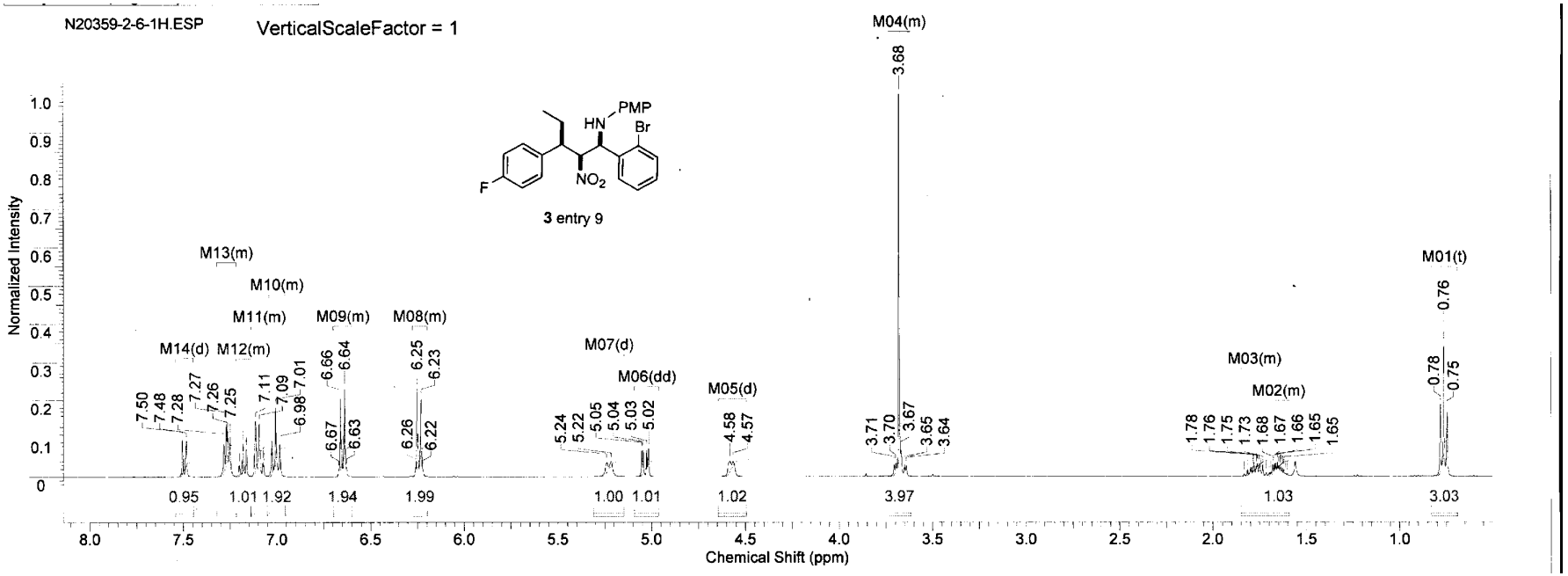


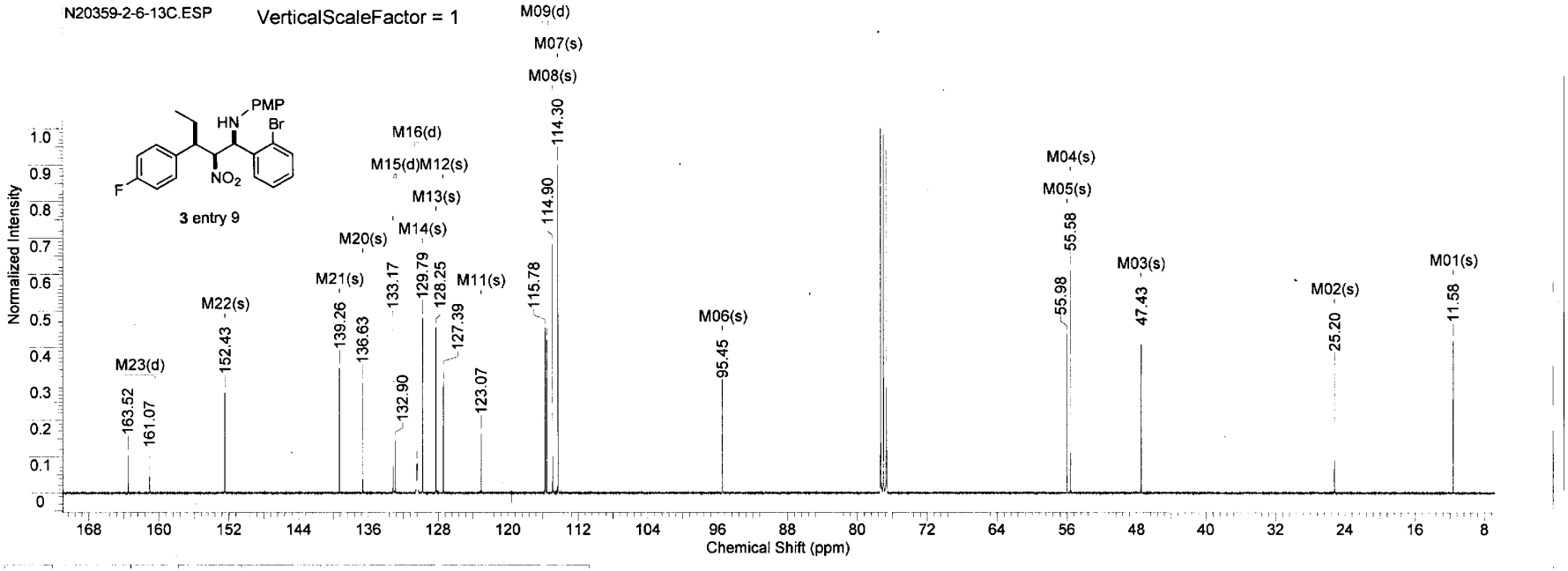
3 entry 8

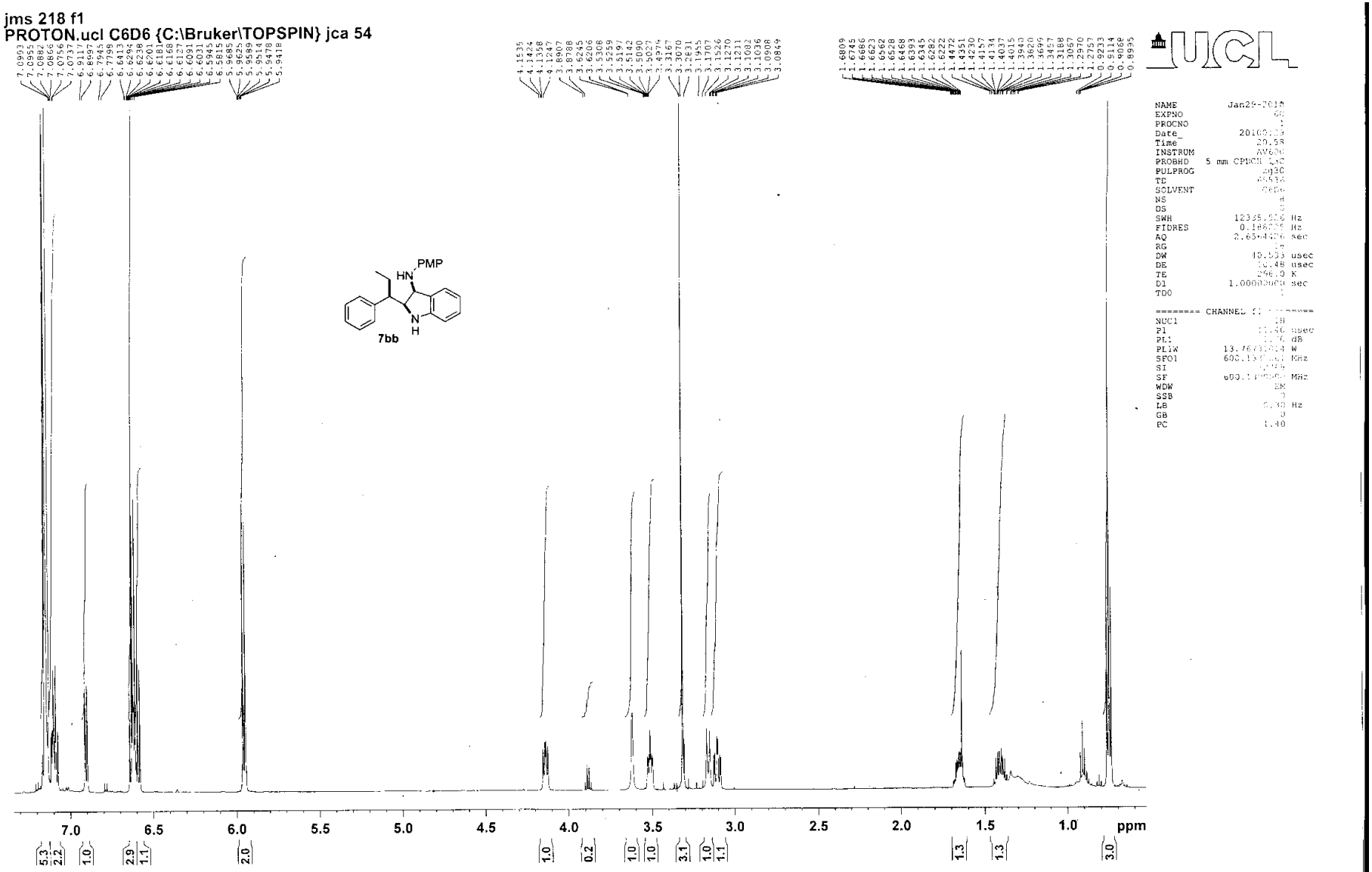


NAME	jms 478
EXPNO	1
PROCNO	1
Date_	20160705
Time	12.23
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125453 Hz
AQ	3.9846127 sec
RG	145
DW	60.800 use
DE	14.62 use
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	11.70 use
PL1	0.00 dB
PL1W	13.81547618 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

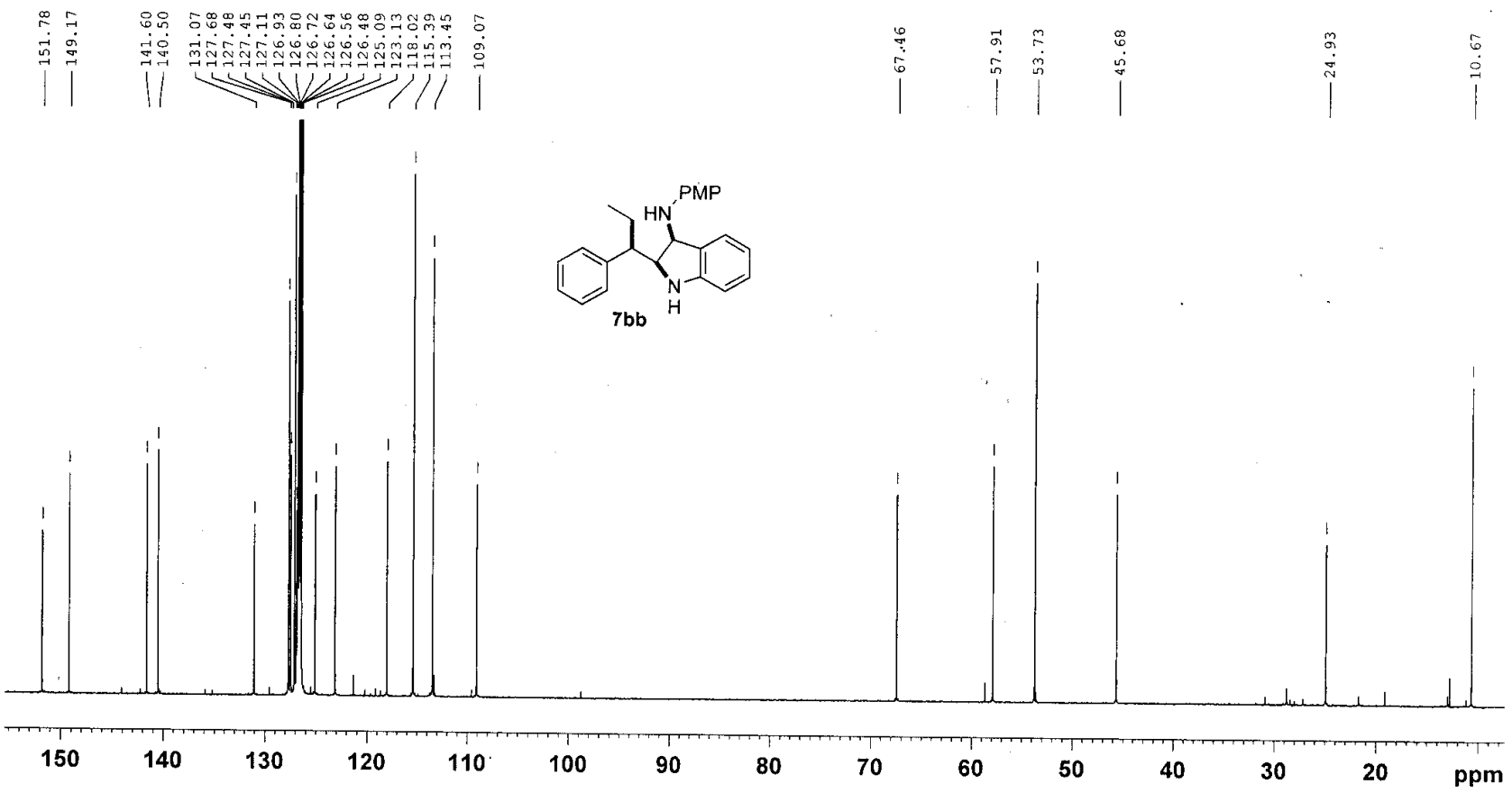

jms 453
C13CPD CDCl3 v2 jca 17
152.6525
139.5658
137.3678
137.0834
136.6048
133.2861
130.8130
129.9654
129.3331
128.5754
127.5489
127.2086
126.6665
125.8373
123.2866
115.1539
114.5368
114.3585
95.4625
77.3623
77.1075
76.8535
55.8586
55.7448
41.9928
25.1568
19.9474
11.1536
HN
PMP
Br
NO2
3 entry 8
ppm

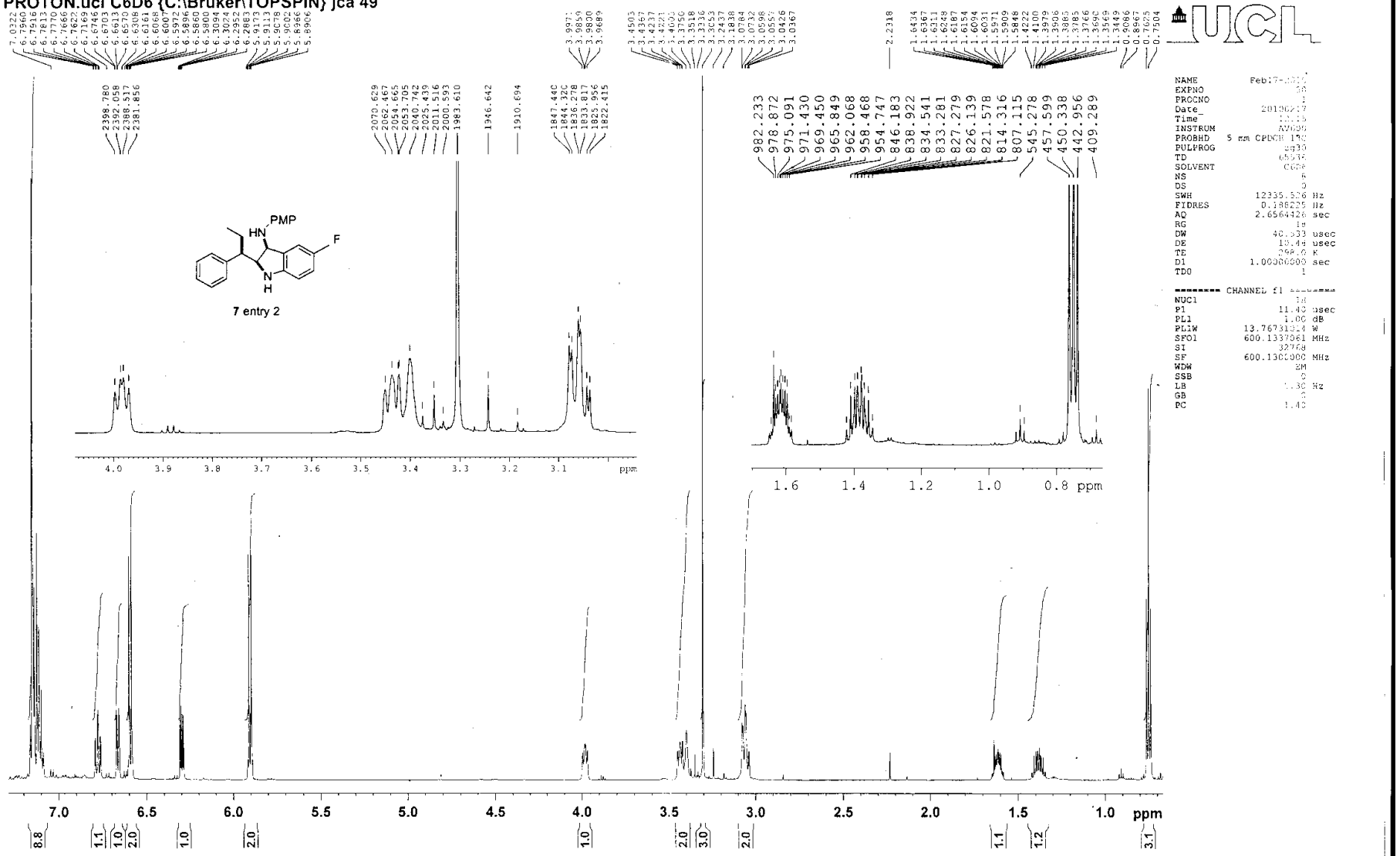

N20359-2-6-1H.ESP
VerticalScaleFactor = 1
3 entry 9
Normalized Intensity
Chemical Shift (ppm)
M01(t)
M02(m)
M03(m)
M04(m)
M05(d)
M06(dd)
M07(d)
M08(m)
M09(m)
M10(m)
M11(m)
M12(m)
M13(m)
M14(d)
0.95
1.01
1.92
1.94
1.99
1.00
1.01
1.02
3.97
1.03
3.03

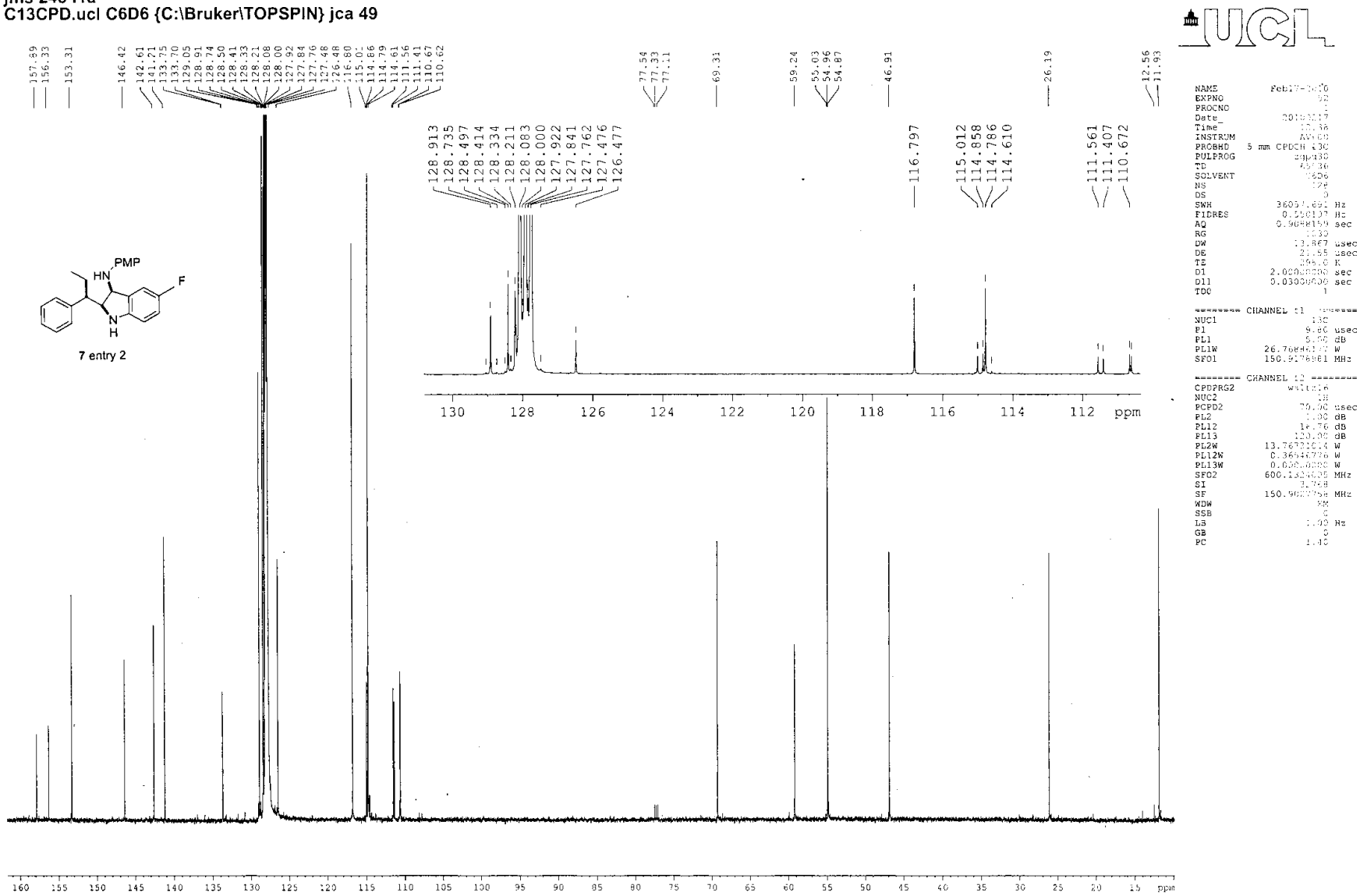

N20359-2-6-13C.ESP
VerticalScaleFactor = 1
PMP
HN
Br
F
NO2
3 entry 9
M09(d)
M07(s)
M08(s)
M16(d)
M15(d)M12(s)
M13(s)
M14(s)
M20(s)
M21(s)
M22(s)
M23(d)
M11(s)
M06(s)
M04(s)
M05(s)
M03(s)
M02(s)
M01(s)
114.30
114.90
133.17
129.79
128.25
127.39
115.78
139.26
136.63
152.43
163.52
161.07
132.90
123.07
95.45
55.58
55.98
47.43
25.20
11.58
Normalized Intensity
Chemical Shift (ppm)

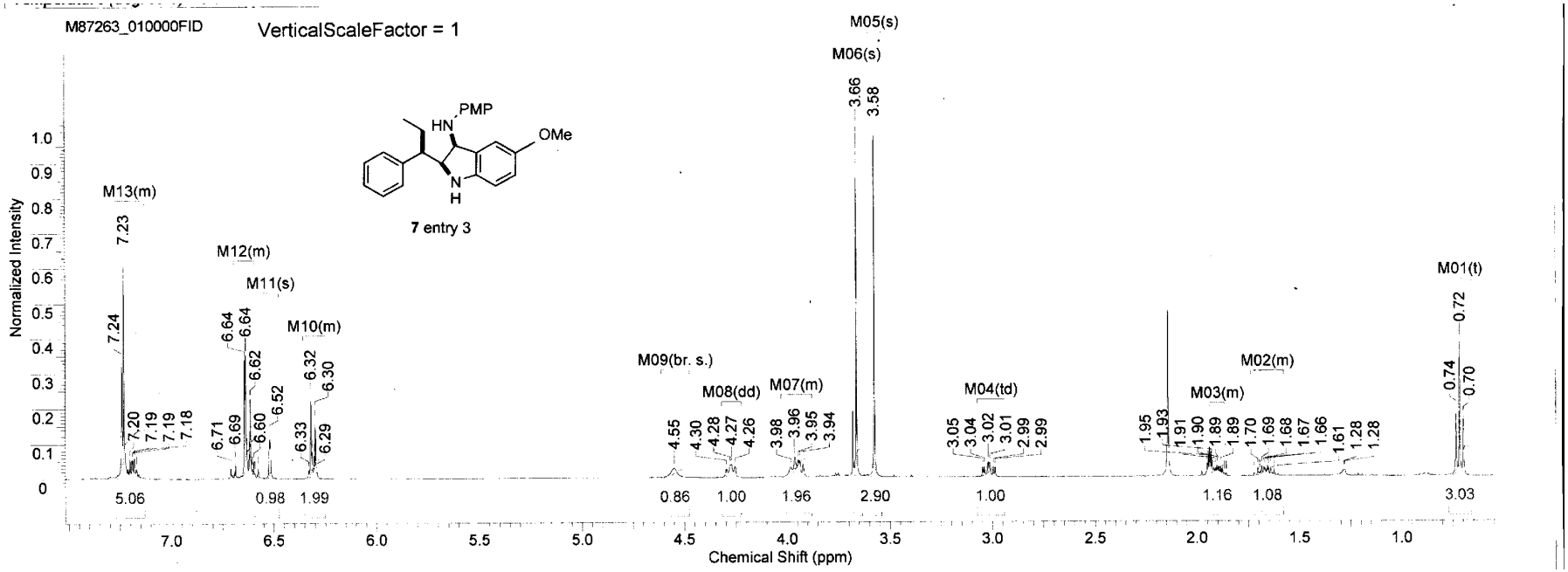

jms 218 f1
PROTON.ucl C6D6 {C:\Bruker\TOPSPIN} jca 54
UCL
HN
PMP
N
H
7bb
7.0993
7.0955
7.0882
7.0866
7.0756
7.0737
6.9117
6.8967
6.8947
6.7798
6.6413
6.6294
6.6238
6.6201
6.6181
6.6168
6.6127
6.6091
6.6031
6.5945
6.5815
5.9685
5.9625
5.9499
5.9478
5.9418
4.1535
4.1424
4.1358
4.1247
3.8907
3.8788
3.6245
3.6206
3.5306
3.5259
3.5147
3.5042
3.5090
3.5027
3.4974
3.3167
3.3070
3.2831
3.1955
3.1707
3.1526
3.1270
3.1211
3.1092
3.1036
3.0908
3.0844
1.6809
1.6745
1.6686
1.6623
1.6562
1.6528
1.6468
1.6393
1.6345
1.6282
1.6222
1.4472
1.4351
1.4230
1.4157
1.4134
1.4037
1.4015
1.3940
1.3820
1.3694
1.3457
1.3188
1.3067
1.2970
1.2757
0.9233
0.9114
0.9068
0.8995
NAME Jan29-2016
EXPNO 60
PROCNO 1
Date_ 20160129
Time 20.58
INSTRUM AV600
PROBHD 5 mm CPDCH 13C
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 2
SWH 12335.526 Hz
FIDRES 0.188279 Hz
AQ 2.6542476 sec
RG 57
DW 40.533 usec
DE 12.48 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.40 usec
PL1 -1.70 dB
PL1W 13.76733208 W
SFO1 600.1324005 MHz
SI 32768
SF 600.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
5.3
2.2
1.0
2.9
1.1
2.0
1.0
0.2
1.0
1.0
3.1
1.0
1.1
1.3
1.3
3.0

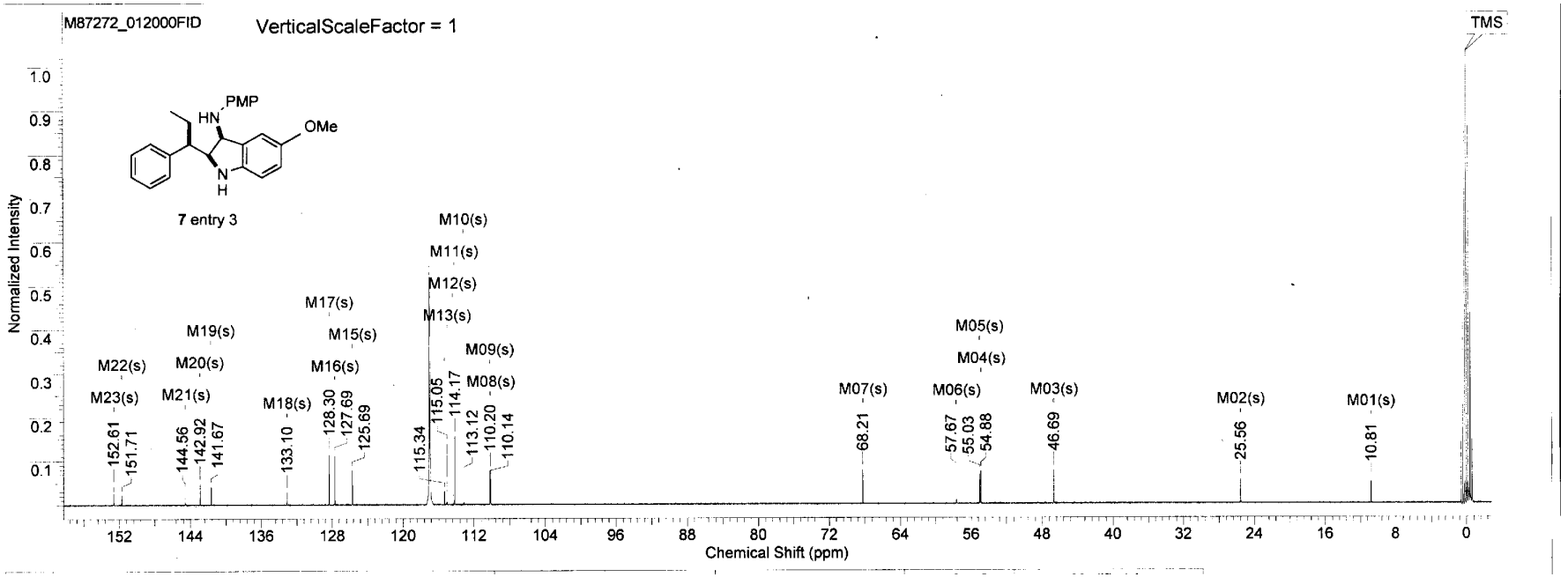
jms 218 f1
C13CPD.ucl C6D6 {C:\Bruker\TOPSPIN} jca 54

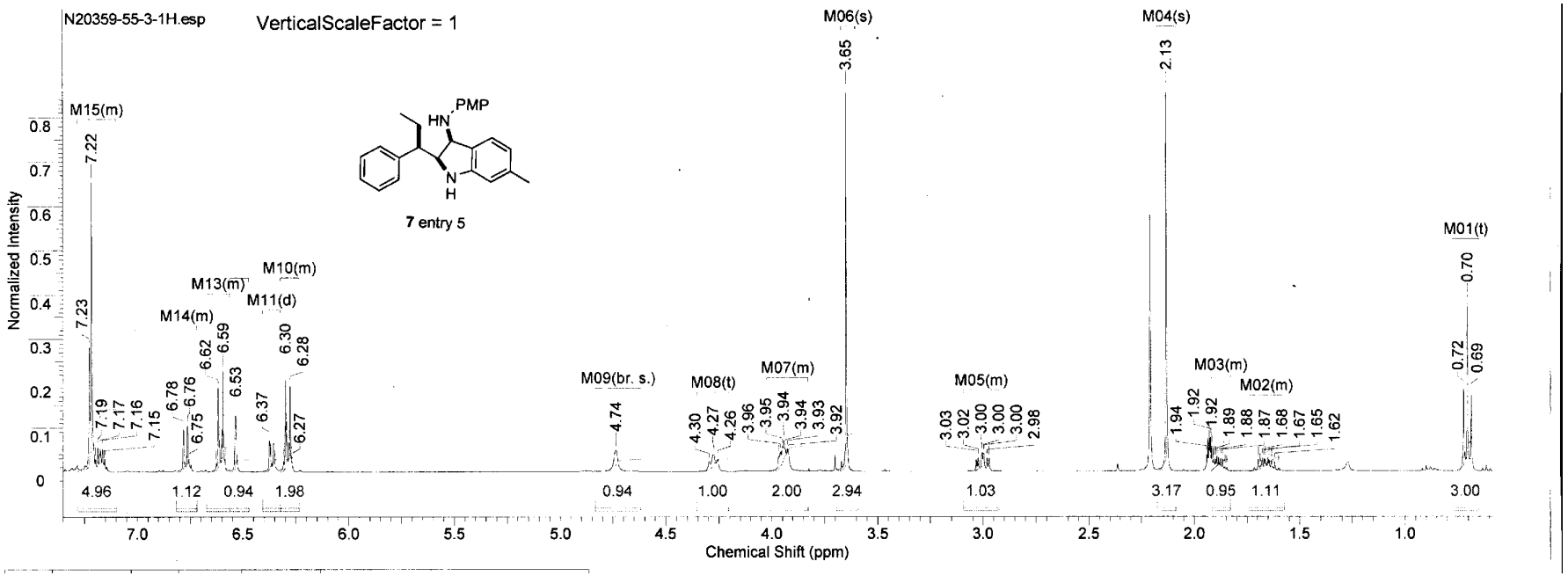


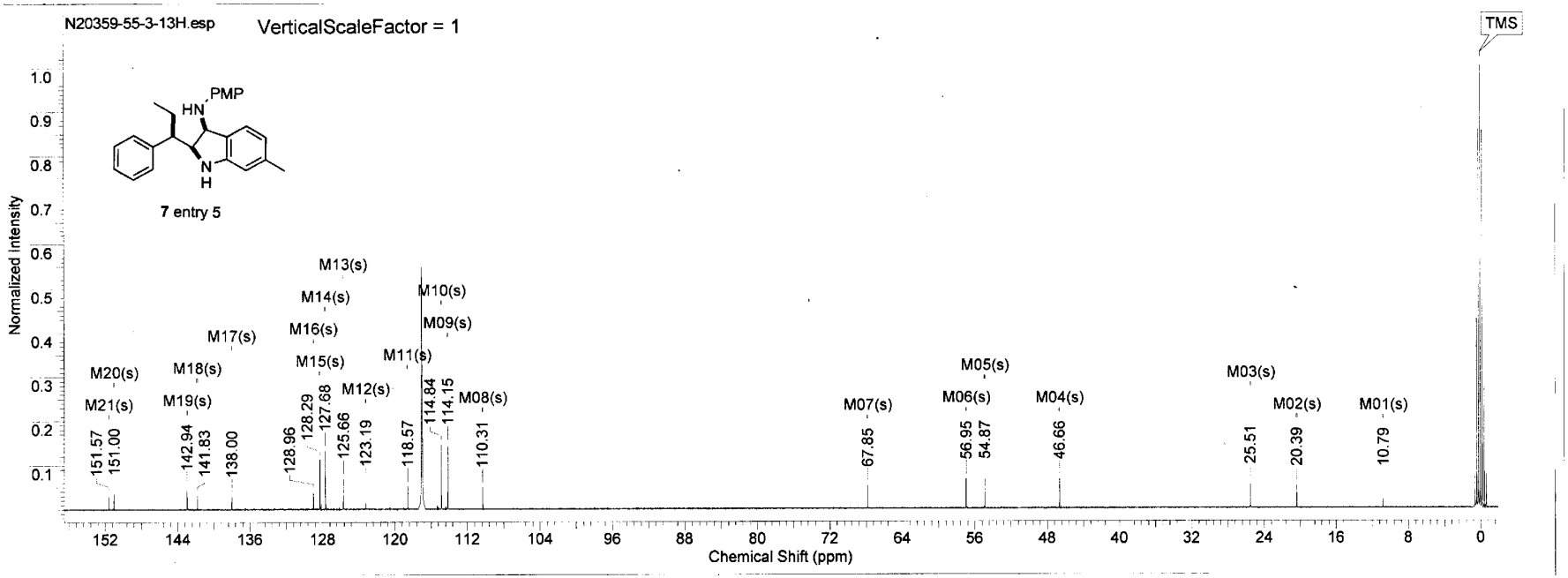

jms 248 f1a
PROTON.ucl C6D6 {C:\Bruker\TOPSPIN} jca 49
UCL
PMP
HN
F
N
H
7 entry 2
ppm

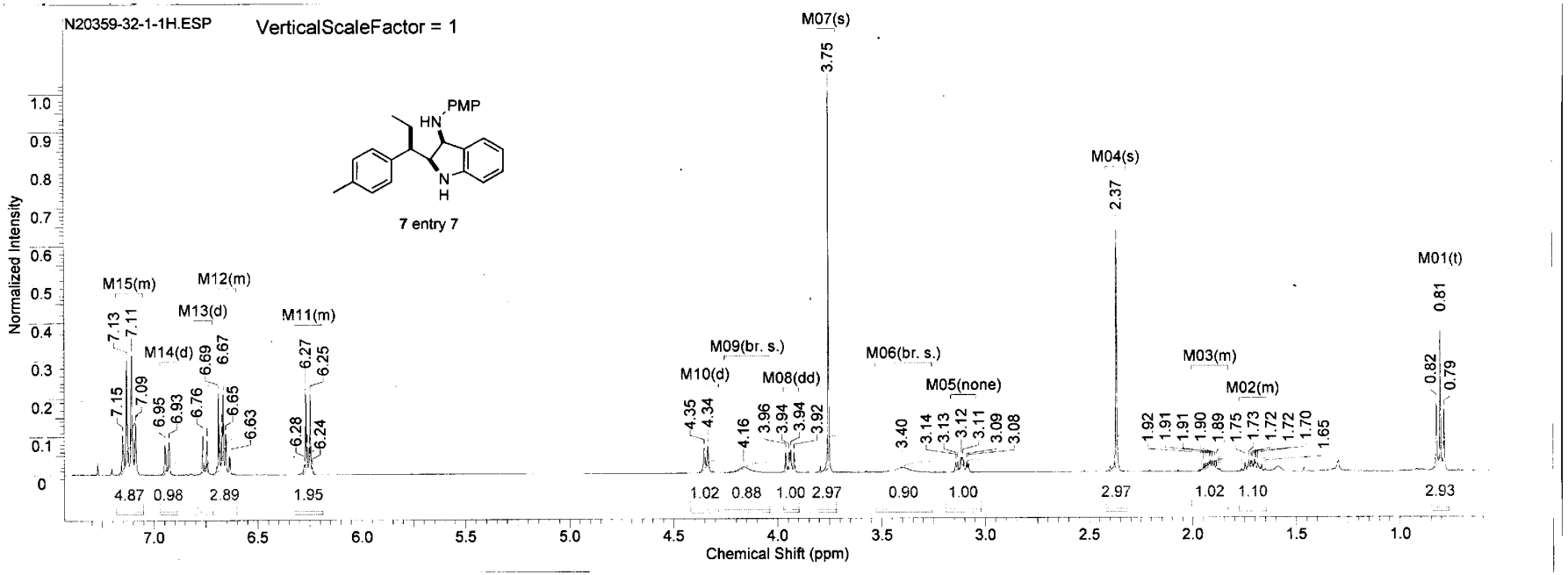

jms 248 f1a
C13CPD.ucl C6D6 {C:\Bruker\TOPSPIN} jca 49
157.89
156.33
153.31
146.42
142.61
141.71
133.75
133.70
129.05
128.91
128.74
128.50
128.41
128.33
128.21
128.08
128.00
127.92
127.84
127.76
127.48
126.48
116.80
115.02
114.86
114.79
114.61
111.56
111.41
110.67
110.62
77.54
77.33
77.11
69.31
59.24
55.03
54.96
54.87
46.91
26.19
12.56
11.93
128.913
128.735
128.497
128.414
128.334
128.211
128.083
128.000
127.922
127.841
127.762
127.476
126.477
116.797
115.012
114.858
114.786
114.610
111.561
111.407
110.672
PMP
HN
F
N
H
7 entry 2
130
128
126
124
122
120
118
116
114
112
ppm
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
ppm
UCL
NAME
EXPNO
PROCNO
Date_
Time
INSTRUM
PROBHD 5 mm CPDCH 13C
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
TD0
CHANNEL f1
NUC1
P1
PL1
PL1W
SFO1
CHANNEL f2
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
PL2W
PL12W
PL13W
SFO2
SI
SF
WDW
SSB
LB
GB
PC

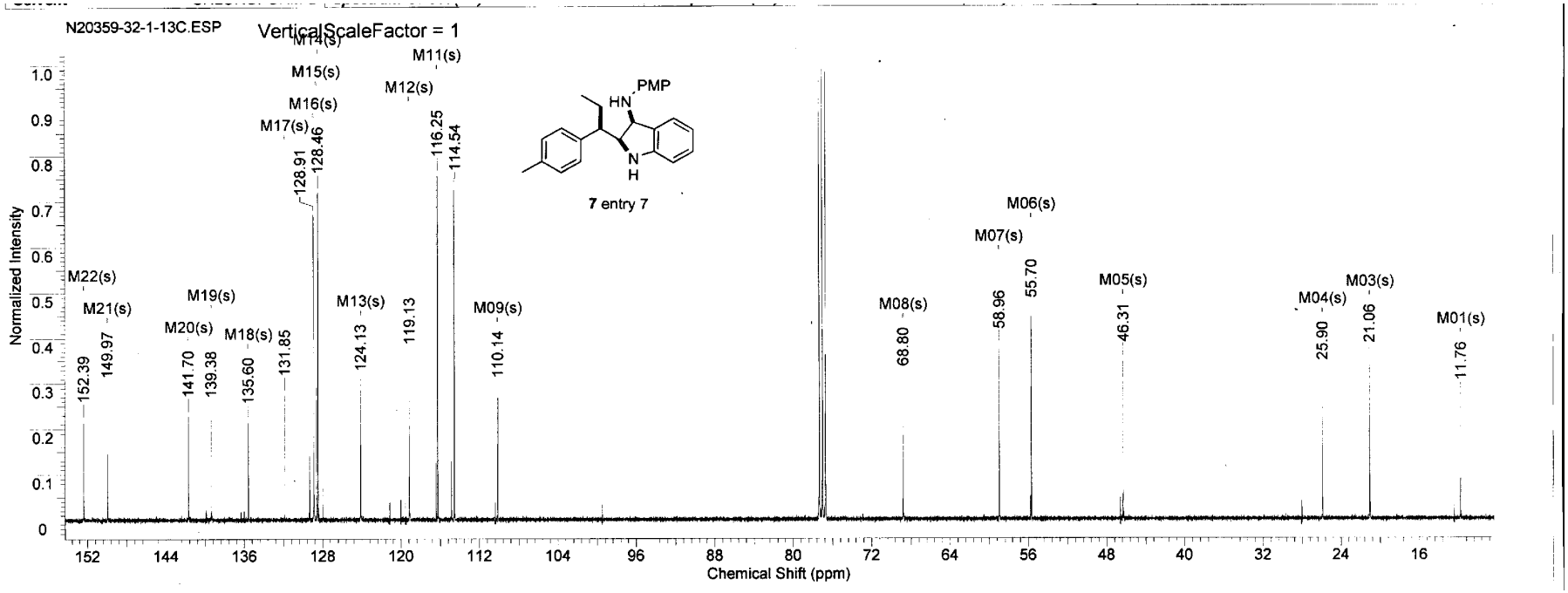

M87263_010000FID
VerticalScaleFactor = 1
PMP
HN
OMe
N
H
7 entry 3
M13(m)
M12(m)
M11(s)
M10(m)
M09(br. s.)
M08(dd)
M07(m)
M06(s)
M05(s)
M04(td)
M03(m)
M02(m)
M01(t)
Normalized Intensity
Chemical Shift (ppm)
5.06
0.98
1.99
0.86
1.00
1.96
2.90
1.00
1.16
1.08
3.03

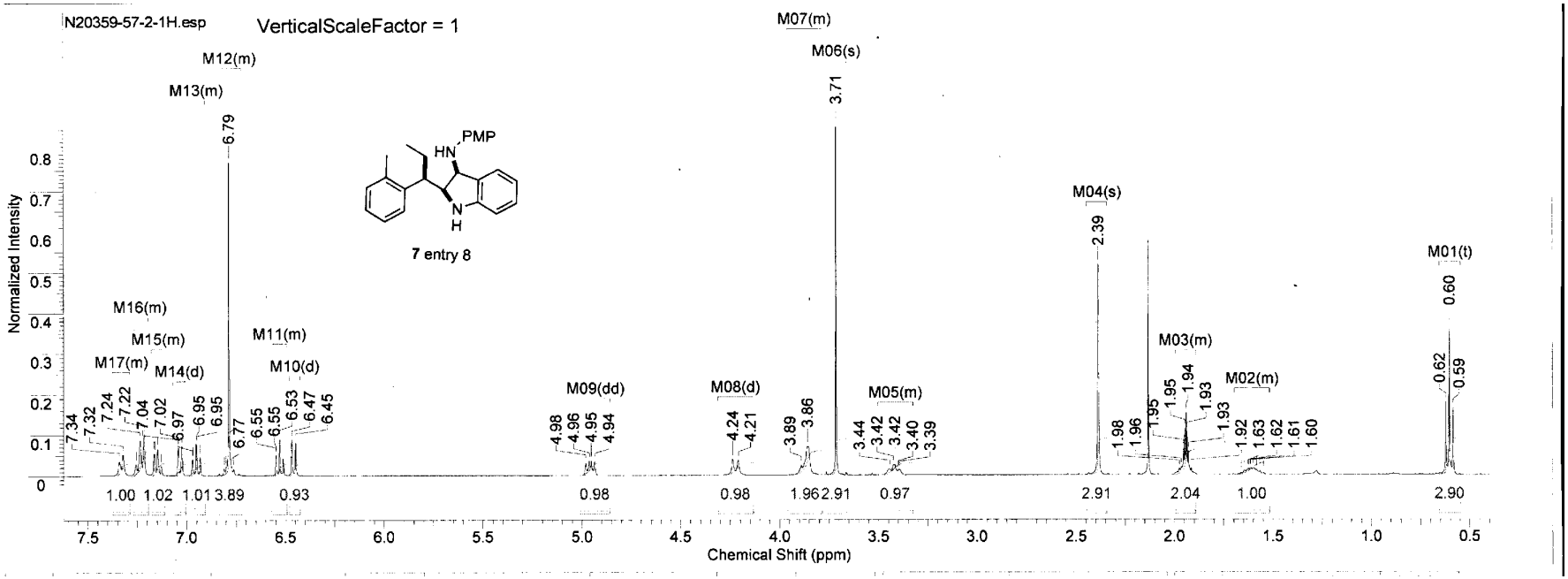

M87272_012000FID
VerticalScaleFactor = 1
TMS
PMP
HN
OMe
N
H
7 entry 3
Normalized Intensity
Chemical Shift (ppm)
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M06(s)
M07(s)
M08(s)
M09(s)
M10(s)
M11(s)
M12(s)
M13(s)
M15(s)
M16(s)
M17(s)
M18(s)
M19(s)
M20(s)
M21(s)
M22(s)
M23(s)
10.81
25.56
46.69
54.88
55.03
57.67
68.21
110.14
110.20
113.12
114.17
115.05
115.34
125.69
127.69
128.30
133.10
141.67
142.92
144.56
151.71
152.61

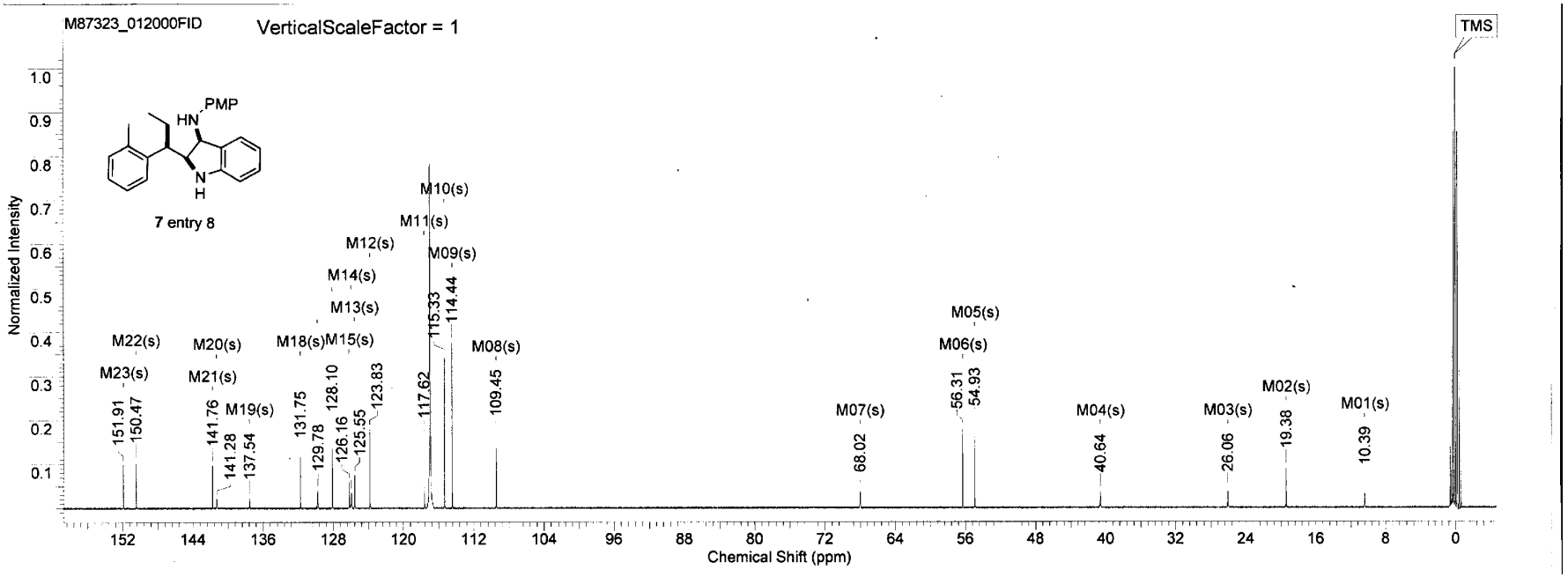

N20359-55-3-1H.esp
VerticalScaleFactor = 1
PMP
HN
N
H
7 entry 5
Normalized Intensity
Chemical Shift (ppm)
M15(m)
M14(m)
M13(m)
M11(d)
M10(m)
M09(br. s.)
M08(t)
M07(m)
M06(s)
M05(m)
M04(s)
M03(m)
M02(m)
M01(t)
4.96
1.12
0.94
1.98
0.94
1.00
2.00
2.94
1.03
3.17
0.95
1.11
3.00

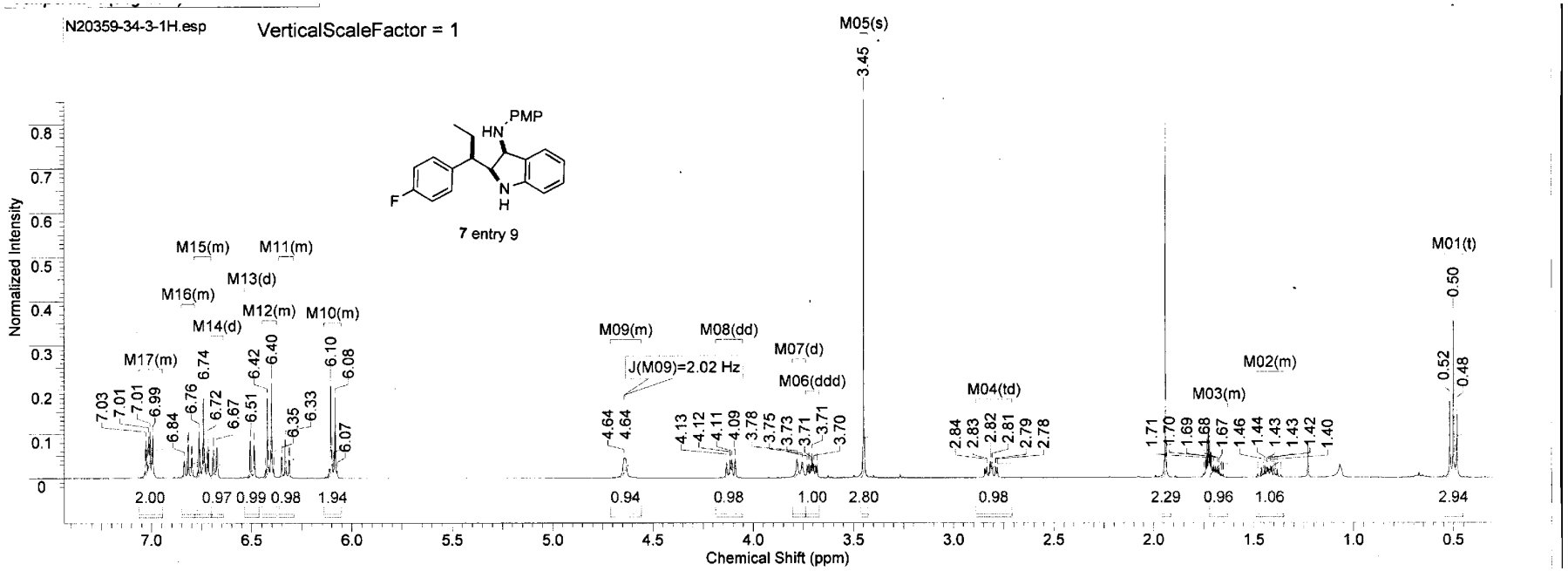

N20359-55-3-13H.esp
VerticalScaleFactor = 1
TMS
PMP
HN
N
H
7 entry 5
Normalized Intensity
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
M13(s)
M14(s)
M10(s)
M09(s)
M16(s)
M17(s)
M15(s)
M11(s)
M20(s)
M18(s)
M12(s)
M08(s)
M21(s)
M19(s)
M07(s)
M05(s)
M06(s)
M04(s)
M03(s)
M02(s)
M01(s)
151.57
151.00
142.94
141.83
138.00
128.96
128.29
127.68
125.66
123.19
118.57
114.84
114.15
110.31
67.85
56.95
54.87
46.66
25.51
20.39
10.79
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

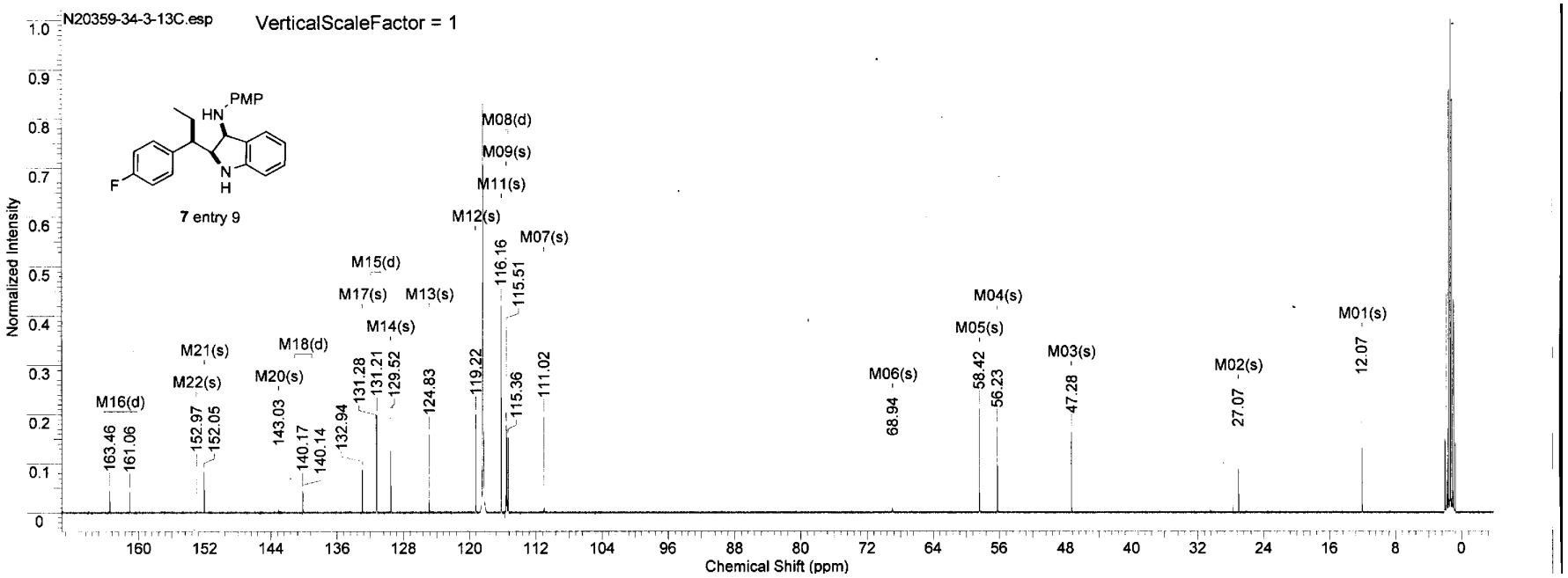

N20359-32-1-1H.ESP
VerticalScaleFactor = 1
PMP
HN
H
N
7 entry 7
Normalized Intensity
Chemical Shift (ppm)
M15(m)
M12(m)
M13(d)
M11(m)
M14(d)
M09(br. s.)
M10(d)
M08(dd)
M07(s)
M06(br. s.)
M05(none)
M04(s)
M03(m)
M02(m)
M01(t)
7.13
7.11
7.15
7.09
6.95
6.93
6.76
6.69
6.67
6.65
6.63
6.27
6.25
6.28
6.24
4.35
4.34
4.16
3.96
3.94
3.94
3.92
3.75
3.40
3.14
3.13
3.12
3.11
3.09
3.08
2.37
1.92
1.91
1.91
1.90
1.89
1.75
1.73
1.72
1.72
1.70
1.65
0.82
0.81
0.79
4.87
0.98
2.89
1.95
1.02
0.88
1.00
2.97
0.90
1.00
2.97
1.02
1.10
2.93

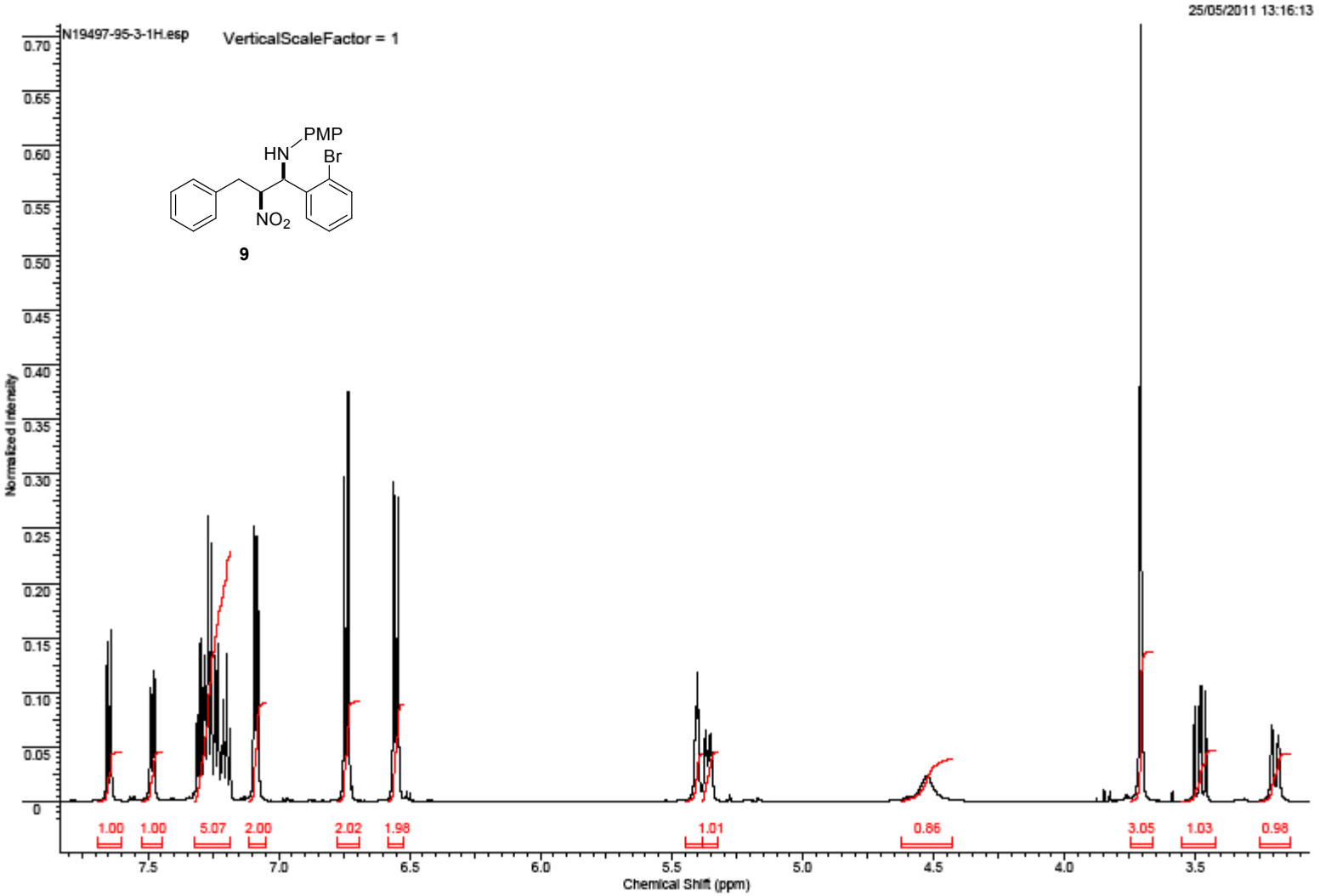

N20359-32-1-13C.ESP
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
PMP
HN
N
H
7 entry 7
M22(s)
M21(s)
M19(s)
M20(s)
M18(s)
M17(s)
M16(s)
M15(s)
M14(s)
M13(s)
M12(s)
M11(s)
M09(s)
M08(s)
M07(s)
M06(s)
M05(s)
M04(s)
M03(s)
M01(s)
152.39
149.97
141.70
139.38
135.60
131.85
128.91
128.46
124.13
119.13
116.25
114.54
110.14
68.80
58.96
55.70
46.31
25.90
21.06
11.76

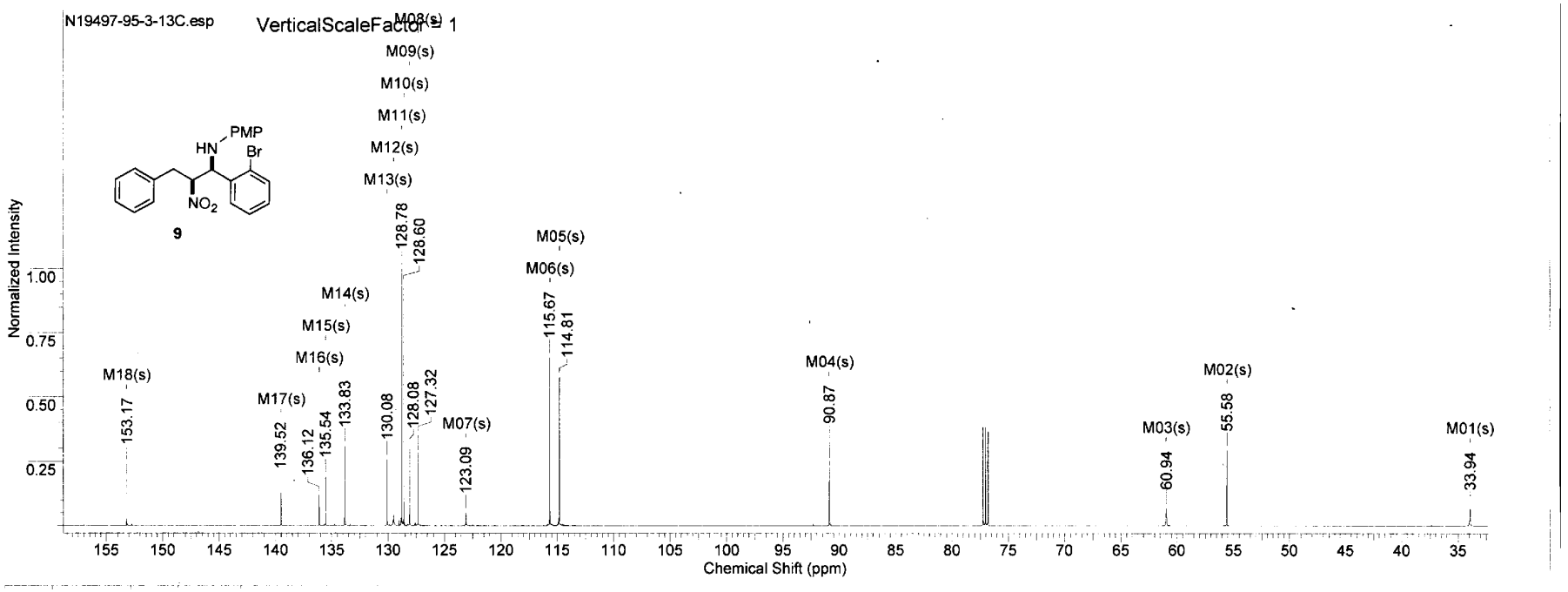

N20359-57-2-1H.esp
VerticalScaleFactor = 1
M12(m)
M13(m)
6.79
M07(m)
M06(s)
3.71
PMP
HN
N
H
7 entry 8
M04(s)
2.39
M01(t)
0.60
M16(m)
M15(m)
M17(m)
M14(d)
M11(m)
M10(d)
M09(dd)
M08(d)
M05(m)
M03(m)
M02(m)
0.62
0.59
Normalized Intensity
Chemical Shift (ppm)
1.00 1.02 1.01 3.89 0.93 0.98 0.98 1.96 2.91 0.97 2.91 2.04 1.00 2.90

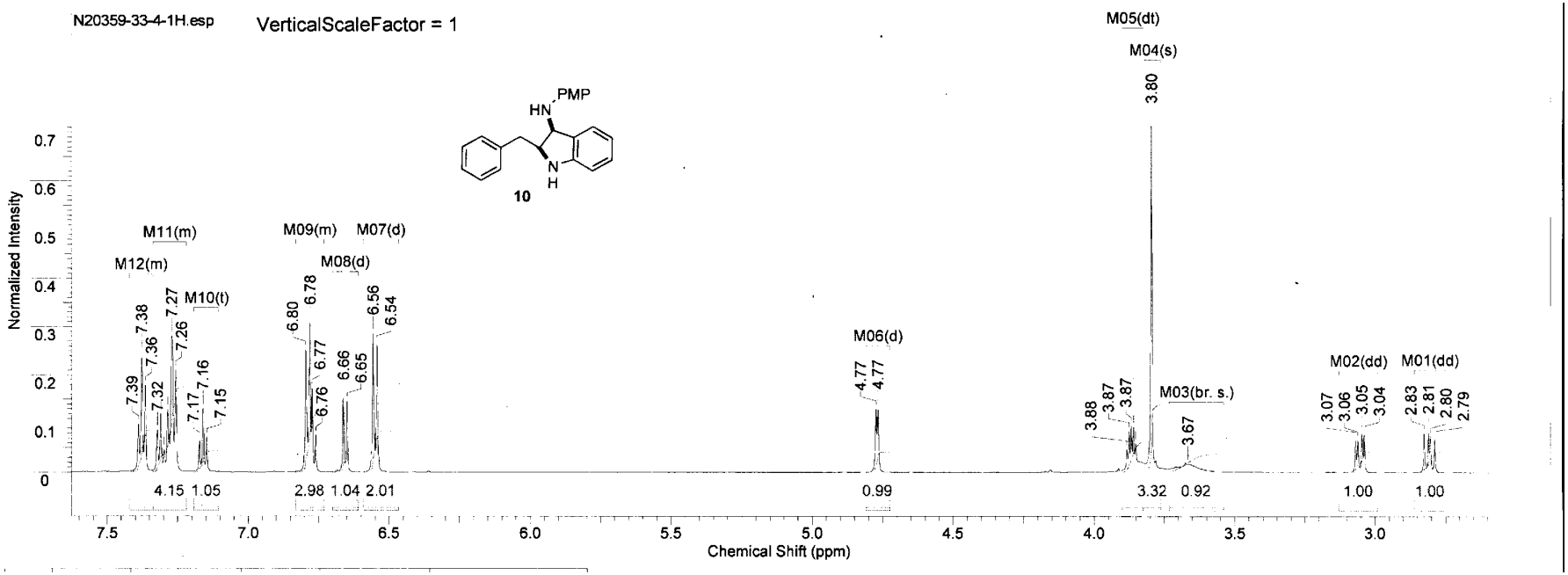

M87323_012000FID
VerticalScaleFactor = 1
TMS
PMP
HN
N
H
7 entry 8
Normalized Intensity
Chemical Shift (ppm)
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M06(s)
M07(s)
M08(s)
M09(s)
M10(s)
M11(s)
M12(s)
M13(s)
M14(s)
M15(s)
M18(s)
M20(s)
M22(s)
M23(s)
M19(s)
M21(s)
151.91
150.47
141.76
141.28
137.54
131.75
129.78
128.10
126.16
125.55
123.83
117.62
115.33
114.44
109.45
68.02
56.31
54.93
40.64
26.06
19.38
10.39

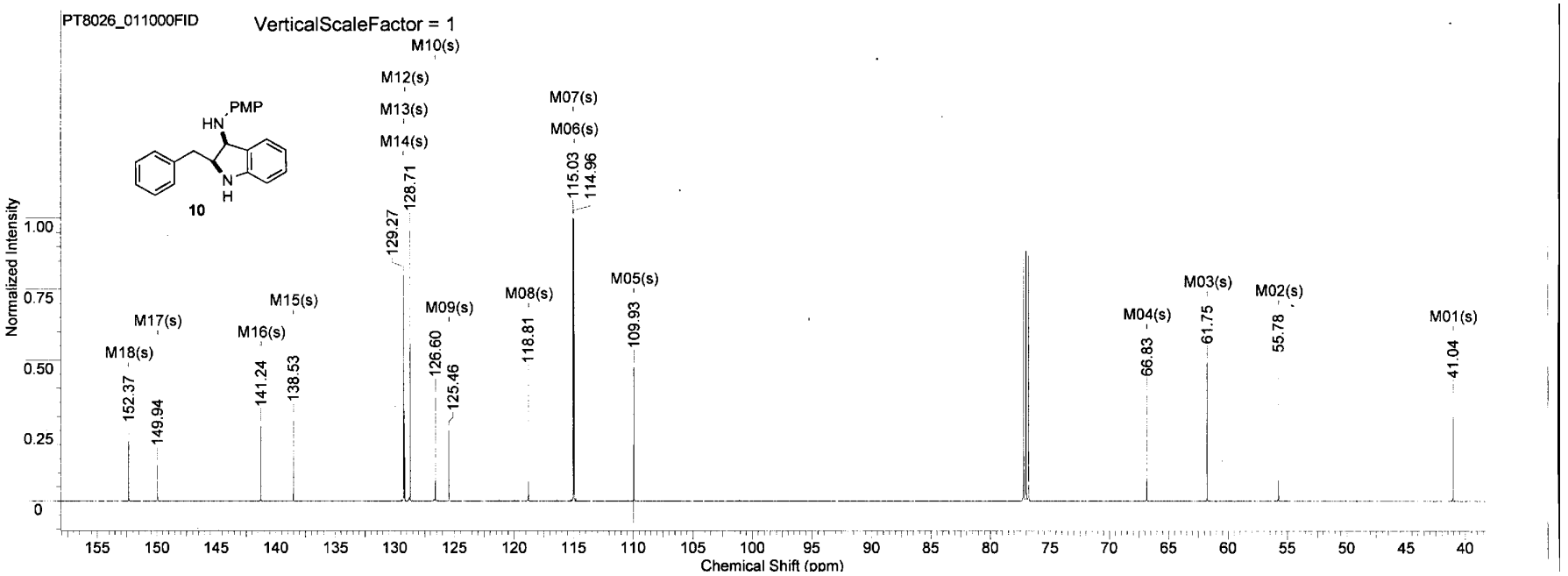

N20359-34-3-1H.esp
VerticalScaleFactor = 1
PMP
HN
N
H
F
7 entry 9
Normalized Intensity
Chemical Shift (ppm)
M01(t)
M02(m)
M03(m)
M04(td)
M05(s)
M06(ddd)
M07(d)
M08(dd)
M09(m)
M10(m)
M11(m)
M12(m)
M13(d)
M14(d)
M15(m)
M16(m)
M17(m)
J(M09)=2.02 Hz
3.45
0.50
0.52
0.48
2.00
0.97
0.99
0.98
1.94
0.94
0.98
1.00
2.80
0.98
2.29
0.96
1.06
2.94


N20359-34-3-13C.esp
VerticalScaleFactor = 1
PMP
HN
N
H
F
7 entry 9
Normalized Intensity
Chemical Shift (ppm)
M08(d)
M09(s)
M11(s)
M12(s)
M07(s)
M15(d)
M17(s)
M13(s)
M14(s)
M21(s)
M22(s)
M18(d)
M20(s)
M16(d)
M04(s)
M05(s)
M01(s)
M03(s)
M06(s)
M02(s)
163.46
161.06
152.97
152.05
143.03
140.17
140.14
132.94
131.28
131.21
129.52
124.83
119.22
116.16
115.51
115.36
111.02
68.94
58.42
56.23
47.28
27.07
12.07


25/05/2011 13:16:13
N19497-95-3-1H.esp
VerticalScaleFactor = 1
PMP
HN
Br
NO2
9
Normalized Intensity
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
1.00
1.00
5.07
2.00
2.02
1.98
1.01
0.86
3.05
1.03
0.98
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
Chemical Shift (ppm)


N19497-95-3-13C.esp
VerticalScaleFactor = 1
M08(s)
M09(s)
M10(s)
M11(s)
M12(s)
M13(s)
M14(s)
M15(s)
M16(s)
M17(s)
M18(s)
M05(s)
M06(s)
M07(s)
M04(s)
M03(s)
M02(s)
M01(s)
128.78
128.60
115.67
114.81
153.17
139.52
136.12
135.54
133.83
130.08
128.08
127.32
123.09
90.87
60.94
55.58
33.94
PMP
HN
Br
NO2
9
Normalized Intensity
1.00
0.75
0.50
0.25
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35
Chemical Shift (ppm)


N20359-33-4-1H.esp
VerticalScaleFactor = 1
PMP
HN
N
H
10
M05(dt)
M04(s)
3.80
M11(m)
M12(m)
M10(t)
M09(m)
M07(d)
M08(d)
M06(d)
M02(dd)
M01(dd)
M03(br. s.)
7.39
7.38
7.36
7.32
7.27
7.26
7.17
7.16
7.15
6.80
6.78
6.77
6.76
6.66
6.65
6.56
6.54
4.77
4.77
3.88
3.87
3.87
3.67
3.07
3.06
3.05
3.04
2.83
2.81
2.80
2.79
4.15 1.05
2.98 1.04 2.01
0.99
3.32 0.92
1.00
1.00
Normalized Intensity
Chemical Shift (ppm)


PT8026_011000FID
VerticalScaleFactor = 1
PMP
HN
N
H
10
M10(s)
M12(s)
M13(s)
M14(s)
M07(s)
M06(s)
M17(s)
M18(s)
M15(s)
M16(s)
M09(s)
M08(s)
M05(s)
M04(s)
M03(s)
M02(s)
M01(s)
152.37
149.94
141.24
138.53
129.27
128.71
126.60
125.46
118.81
115.03
114.96
109.93
66.83
61.75
55.78
41.04
Normalized Intensity
1.00
0.75
0.50
0.25
0
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40
Chemical Shift (ppm)