Aminohydroxyacetone Synthons: Versatile Intermediates for the Organocatalytic Asymmetric Aldol Reaction

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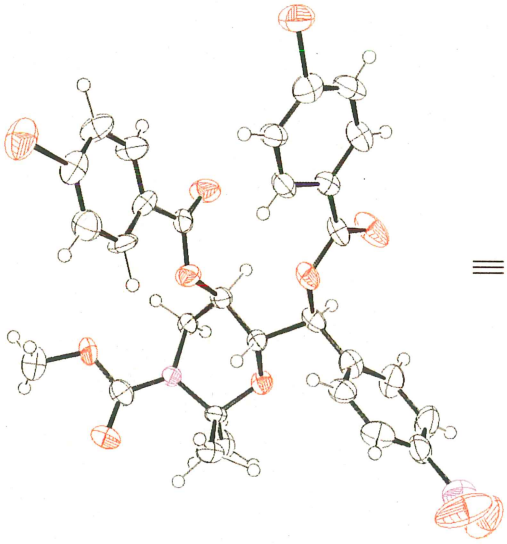
kotsuki@kochi-u.ac.jp

X-Ray Crystal & ^{1}H & ^{13}C NMR Data
(52 Pages)

X-Ray Crystal Data for Compound 7.



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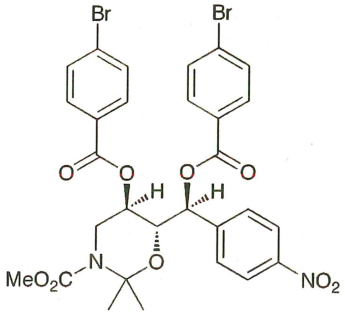
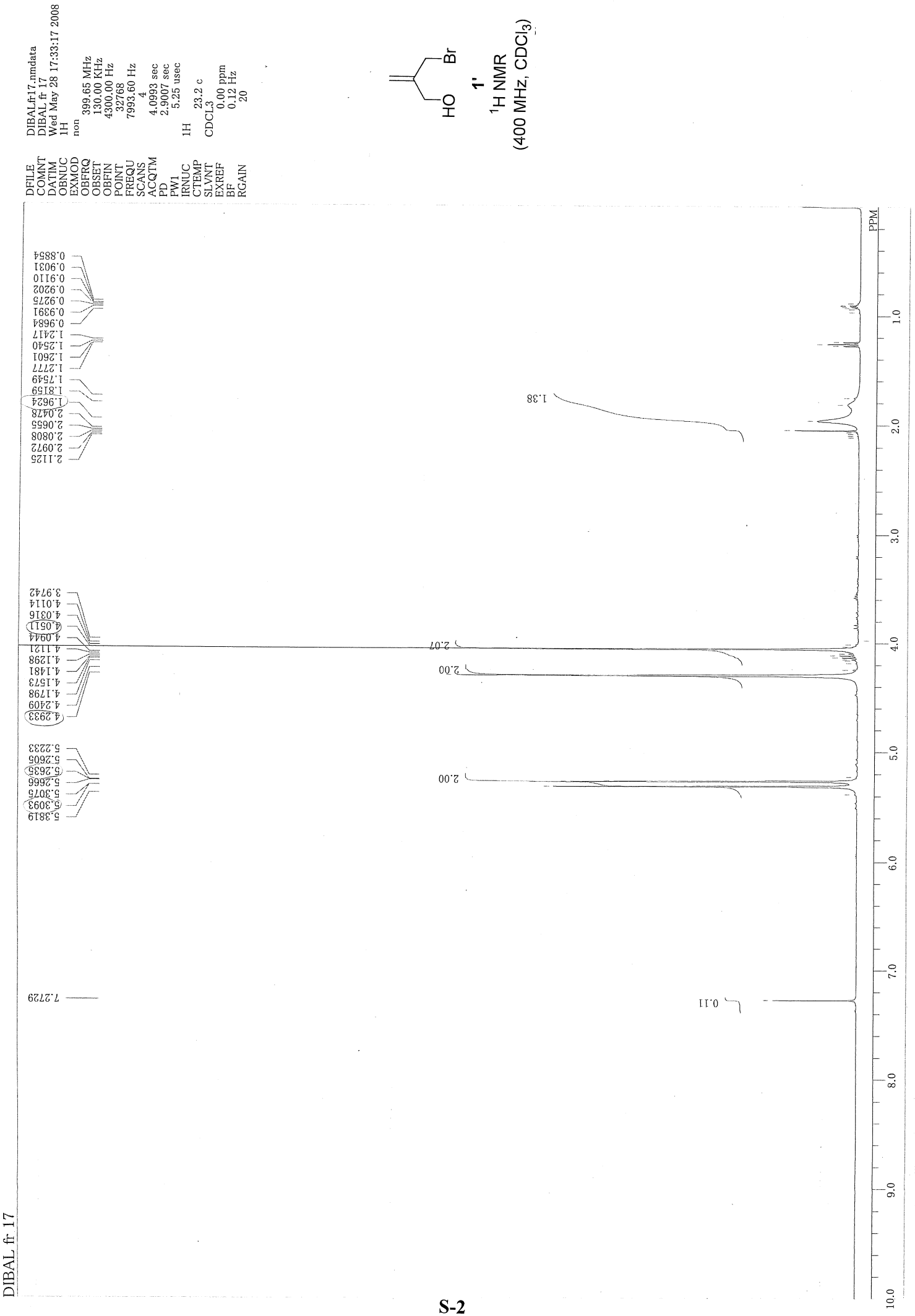


7

Datablock:

Empirical formula	$C_{29}H_{26}Br_2N_2O_9$
Formula weight	706.34
Temperature	173 K
Radiation	CuKα (λ = 1.54187Å)
Crystal system	Orthorhombic
Space group	$P2_12_12_1$ (#19)
Lattice parameters	a = 6.64749(14) Å
	b = 13.2825(3) Å
	c = 33.3205(7) Å
	V = 2942.05(11) Å^3
Z value	4
Density (calculated)	1.595 g / cm^3
$F_{(000)}$	1424.000
μ (CuKα)	39.886 cm^{-1}
Crystal dimensions	0.30 × 0.30 × 0.30 mm
Reflections collected / unique	28423 / 5336 (R_{int} = 0.071)
Refinement method	Full-matrix least-squares on F
Residuals: R [I > 2.00σ(I)]	0.0592
Residuals: Rw [I > 2.00σ(I)]	0.0723
Goodness-of-fit indicator	5.036
Max shift / error in final cycle	0.051
Maximum peak in final diff. map	7.18 e$^-$/Å^3
Minimum peak in final diff. map	–5.06 e$^-$/Å^3
CCDC deposition number	844613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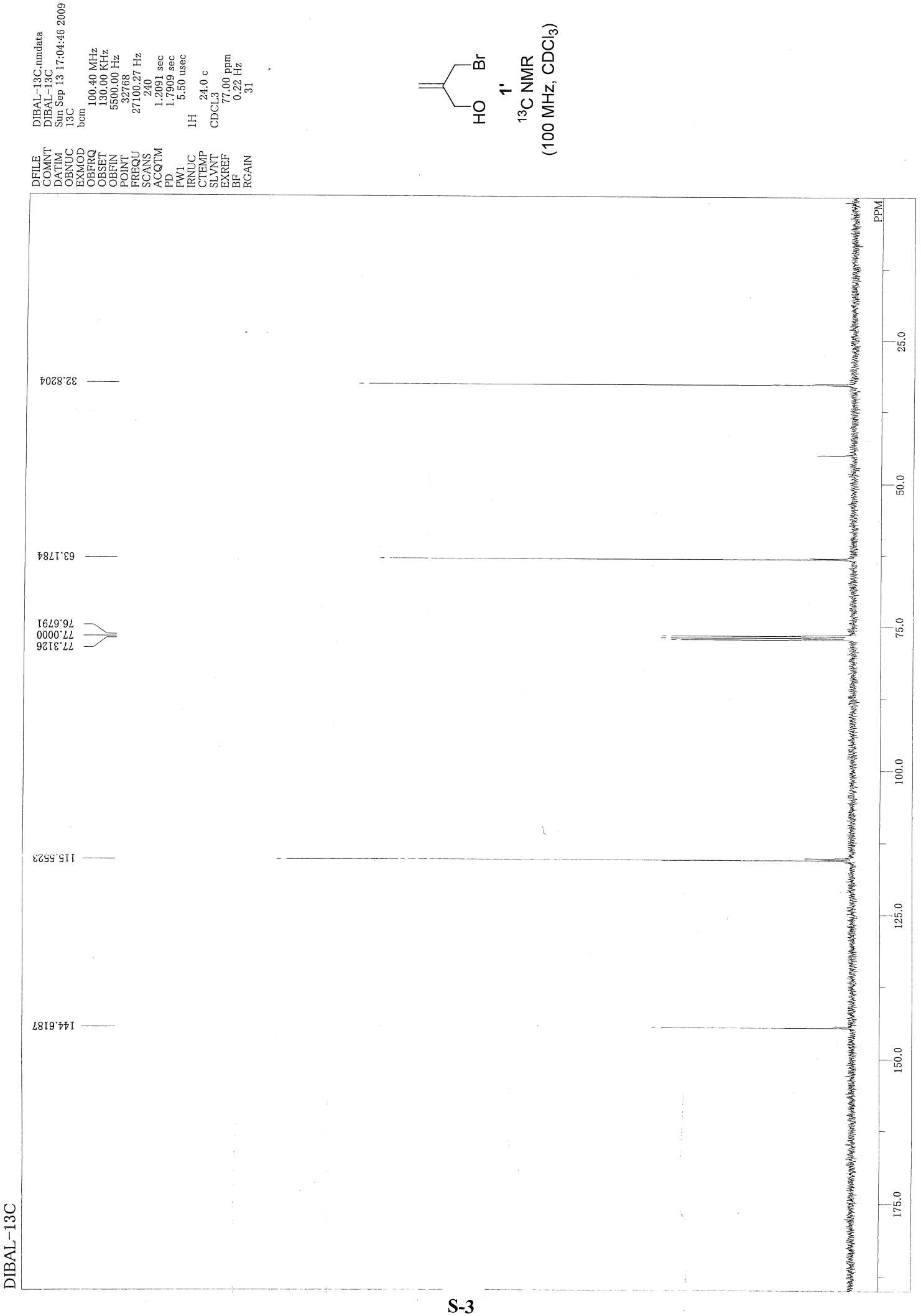
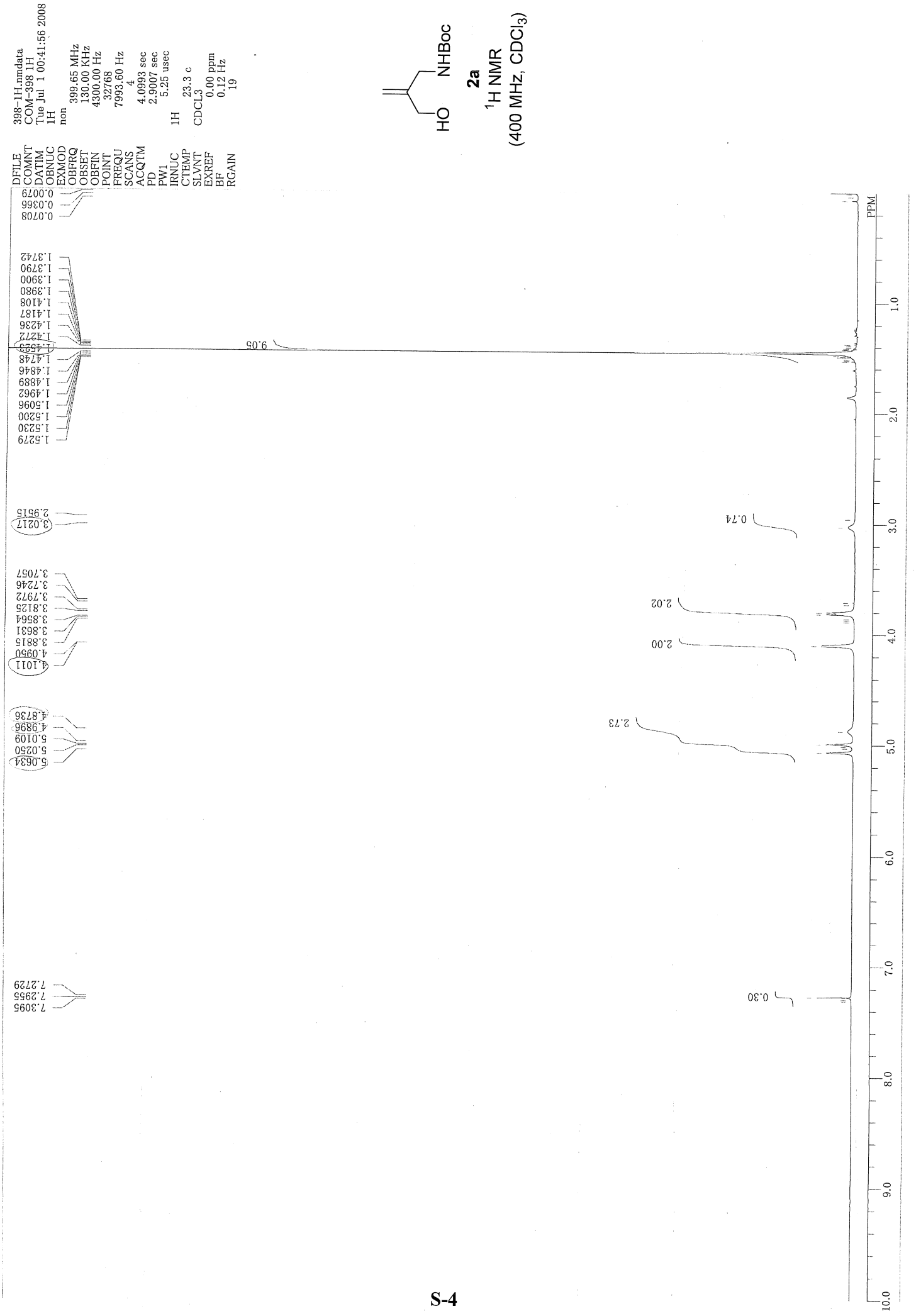
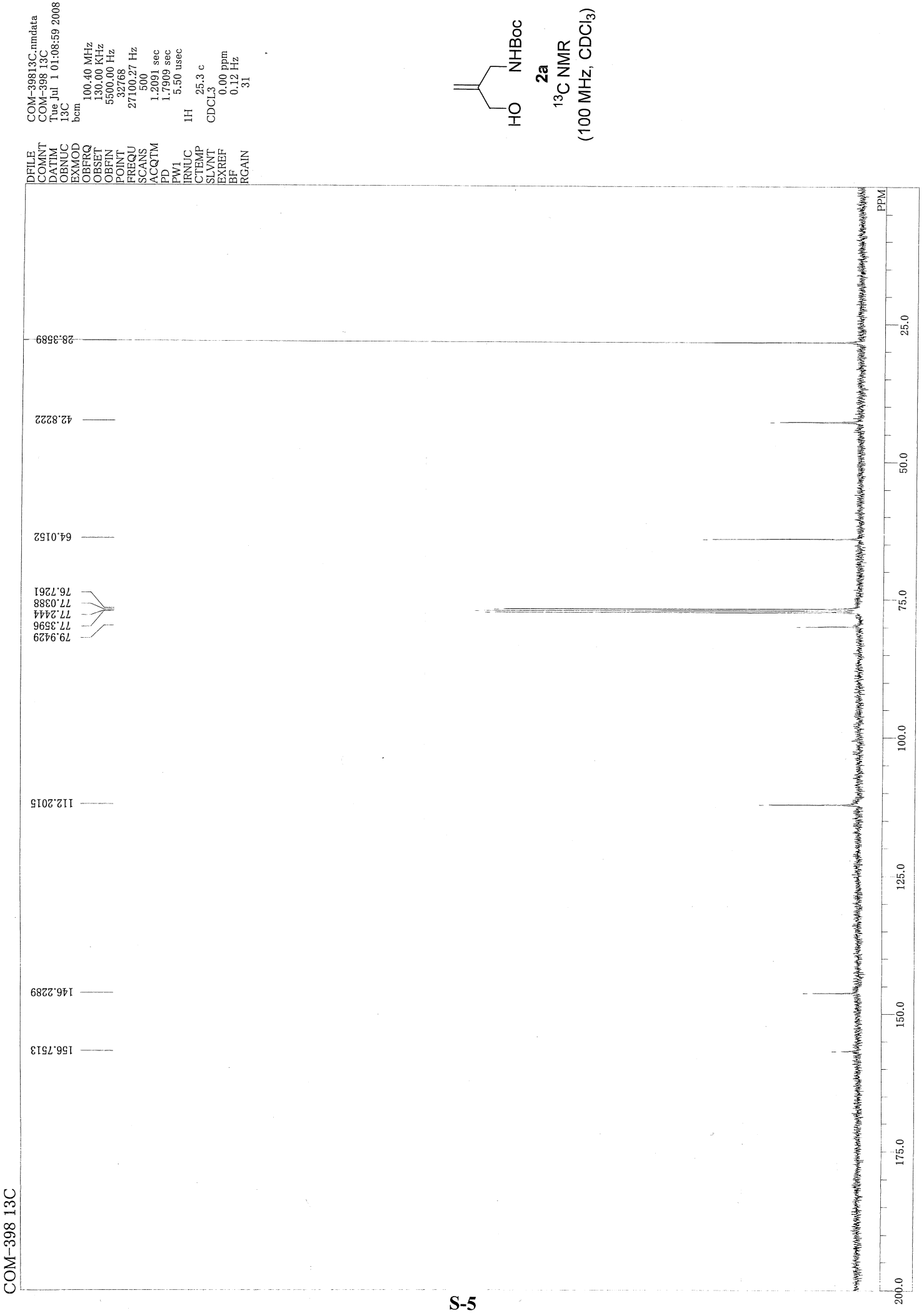
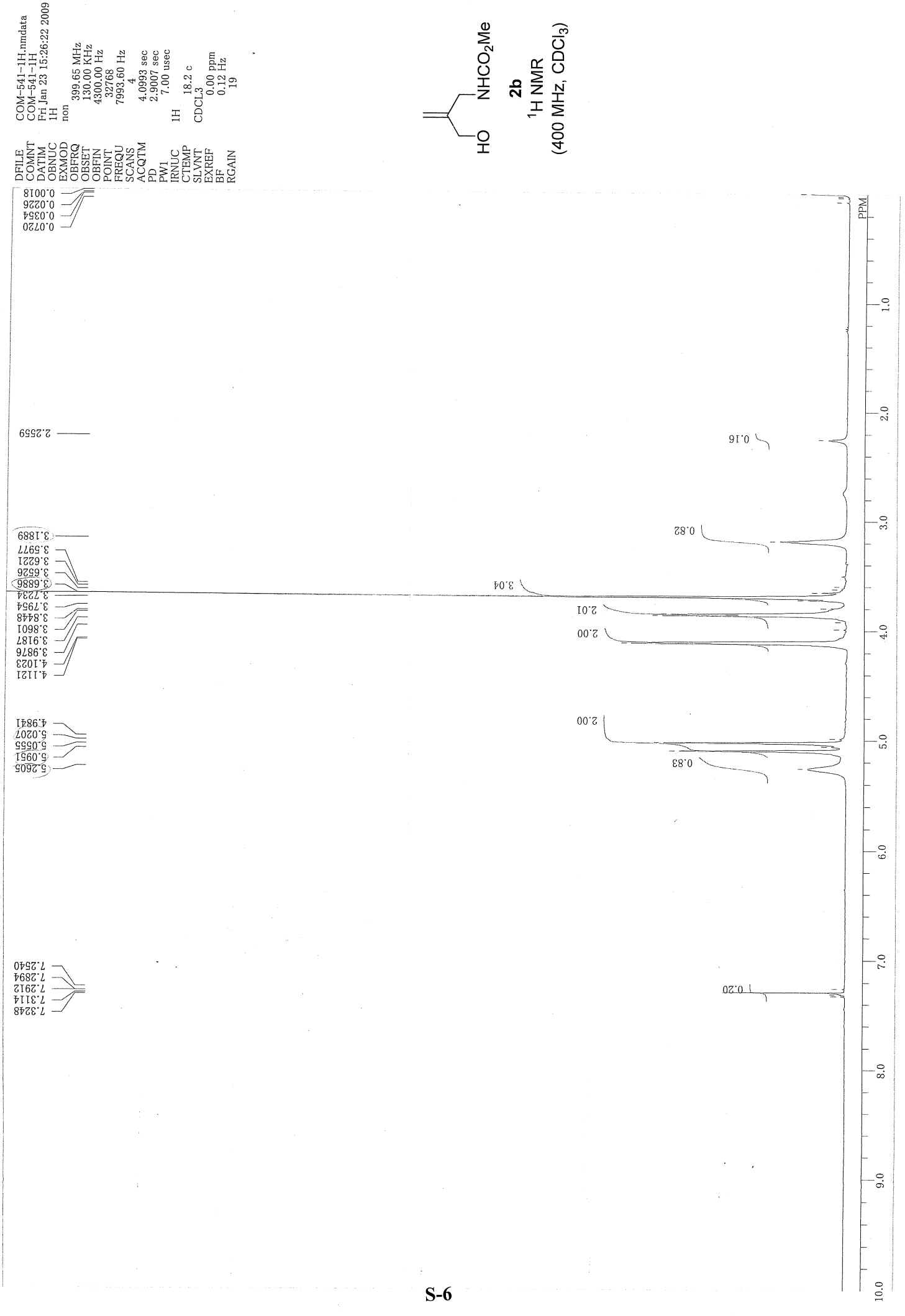
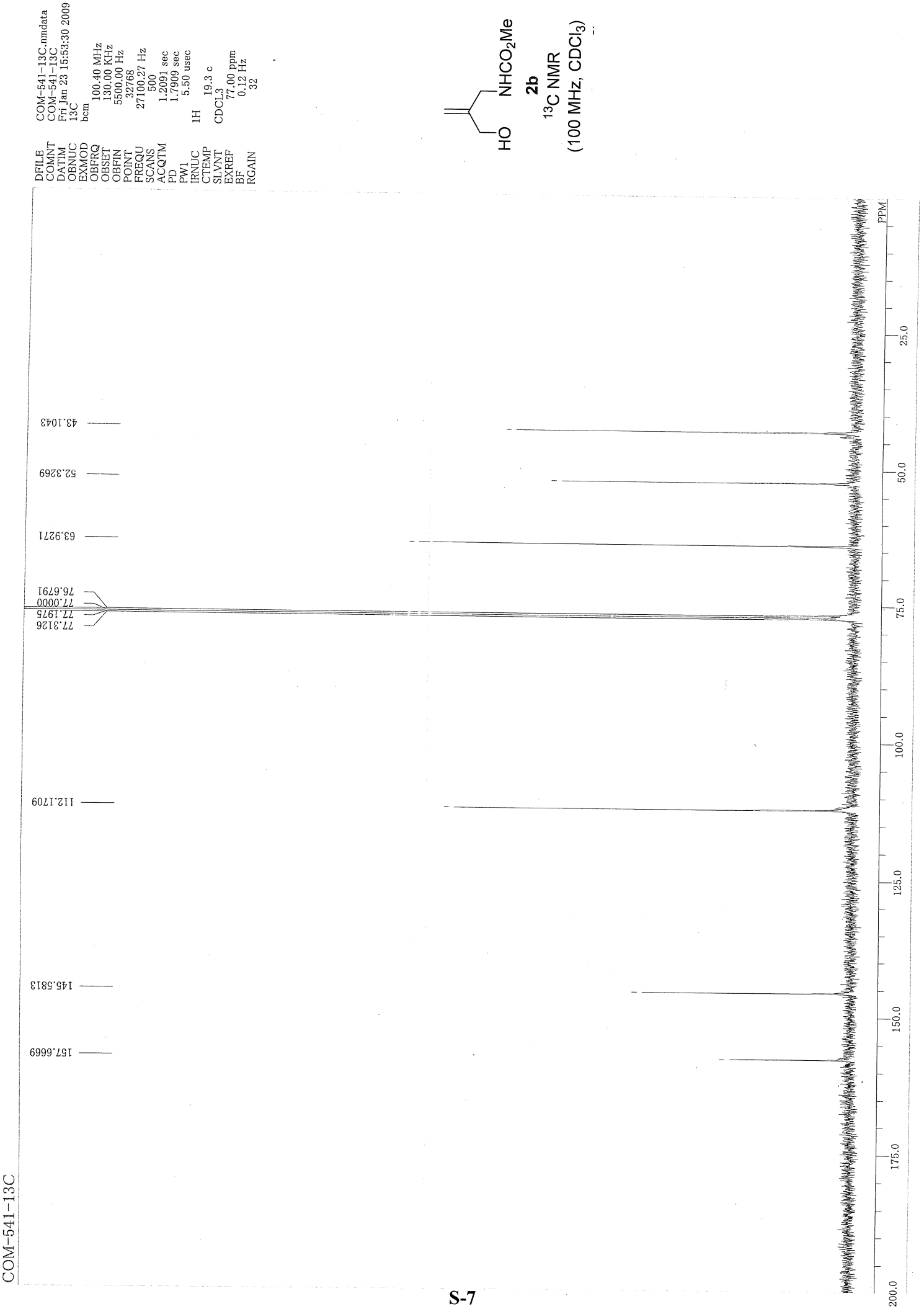
OBFIN 4300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 4
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.25 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20
HO
Br
1'
1H NMR
(400 MHz, CDCl3)
PPM
1.38
2.07
2.00
2.00
0.11

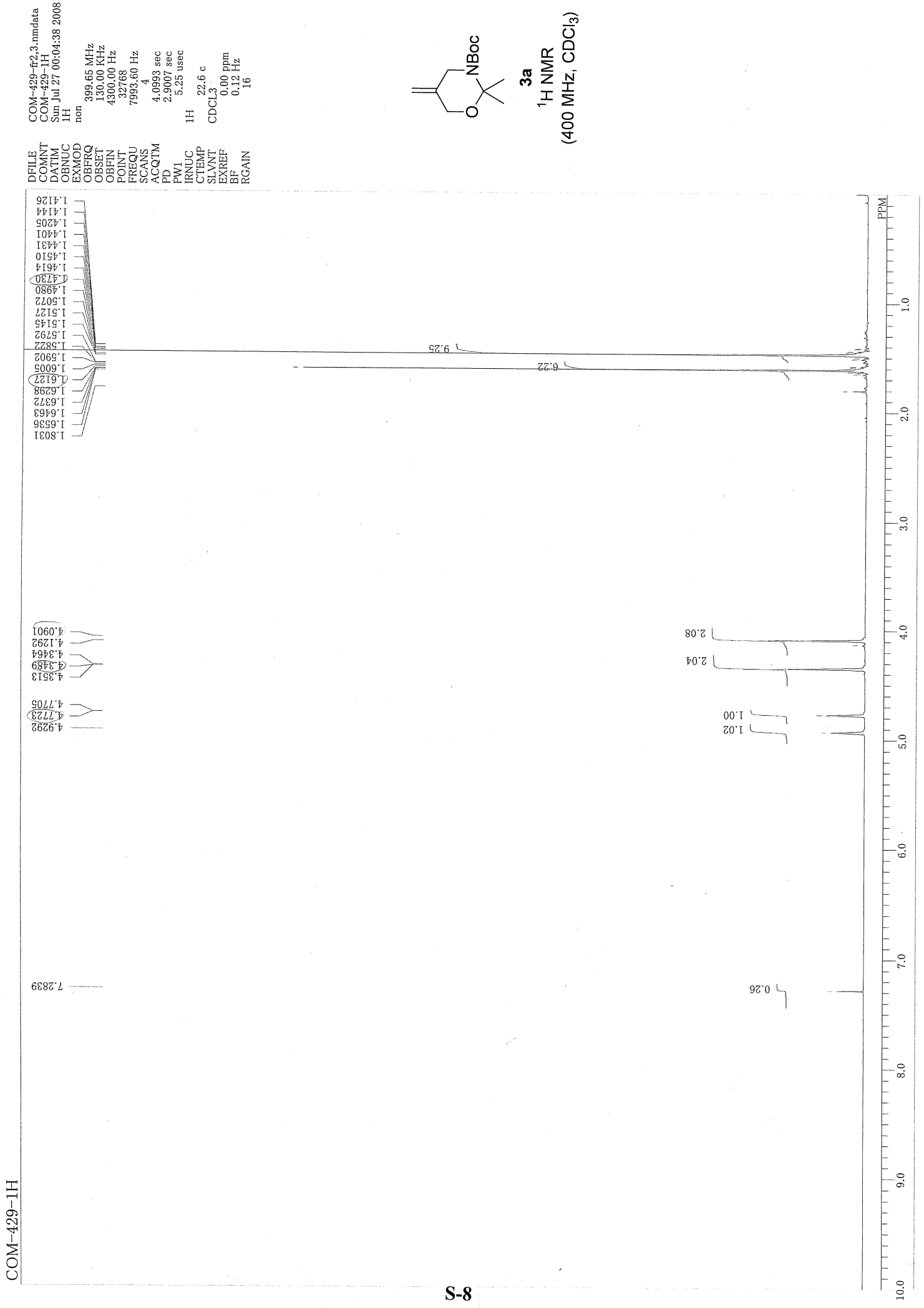








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130.00 KHz
4300.00 Hz
32768
7993.60 Hz
4
4.0993 sec
2.9007 sec
5.25 usec
1H
22.6 c
CDCL3
0.00 ppm
0.12 Hz
16
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
NBoc
O
3a
1H NMR
(400 MHz, CDCl3)
PPM
9.25
6.22
2.08
2.04
1.00
1.02
0.26
7.2839
COM-429-1H

COM-429-13C

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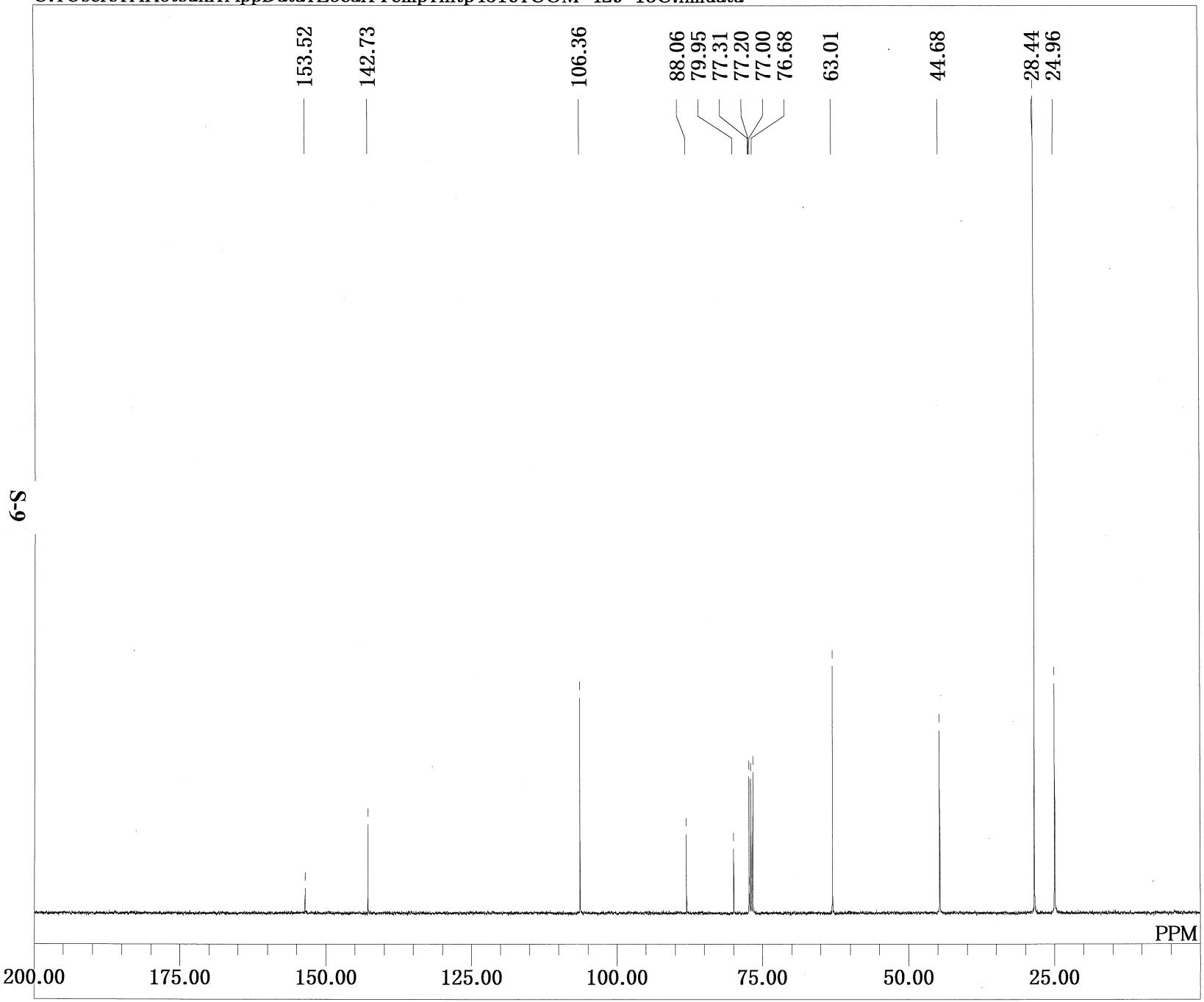

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POINT	32768
FREQU	27100.27 Hz
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PD	1.7909 sec
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CTEMP	24.5 c
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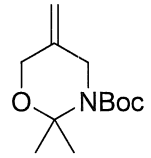
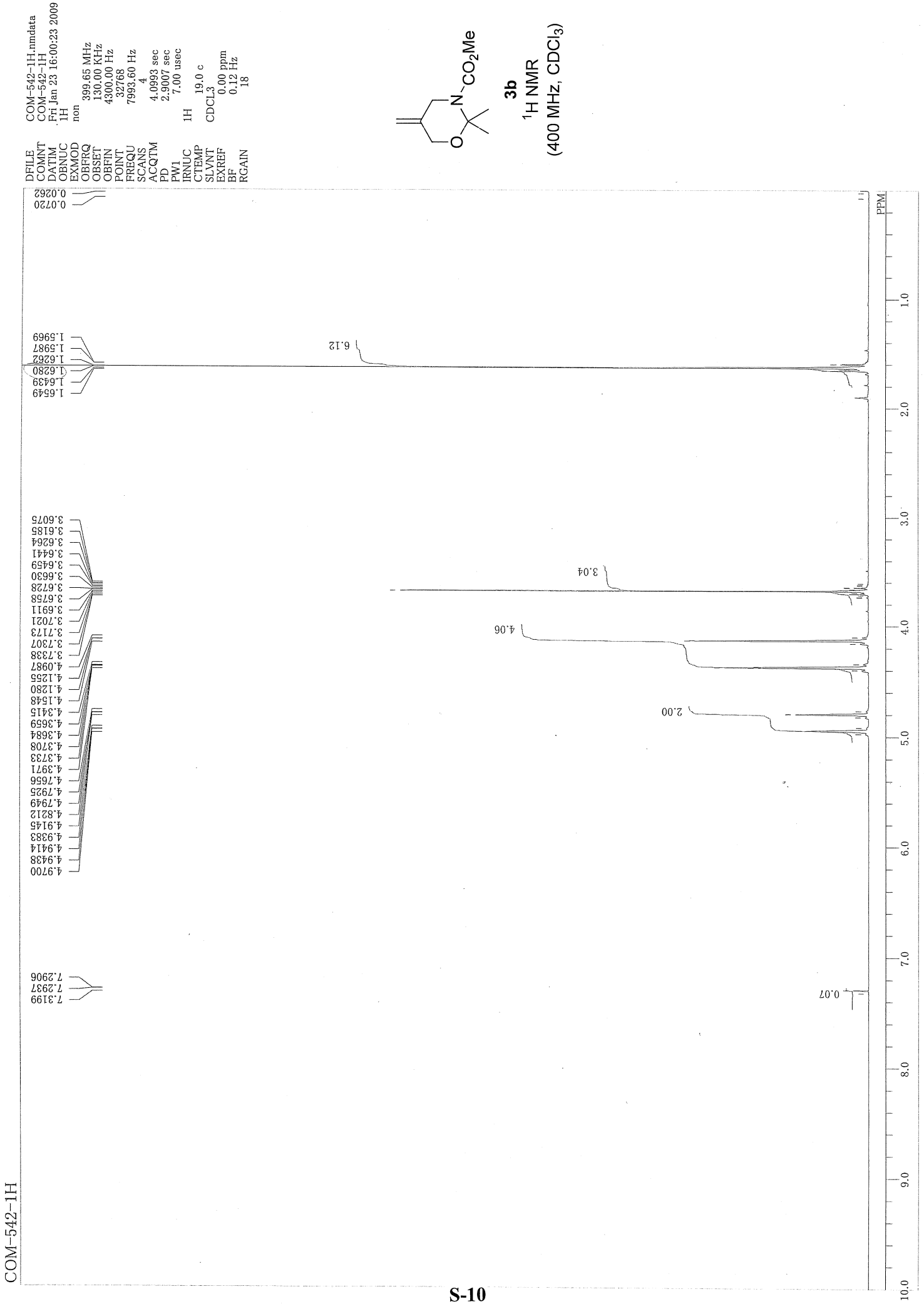
RGAIN	32



3a

^{13}C NMR

(100 MHz, $CDCl_3$)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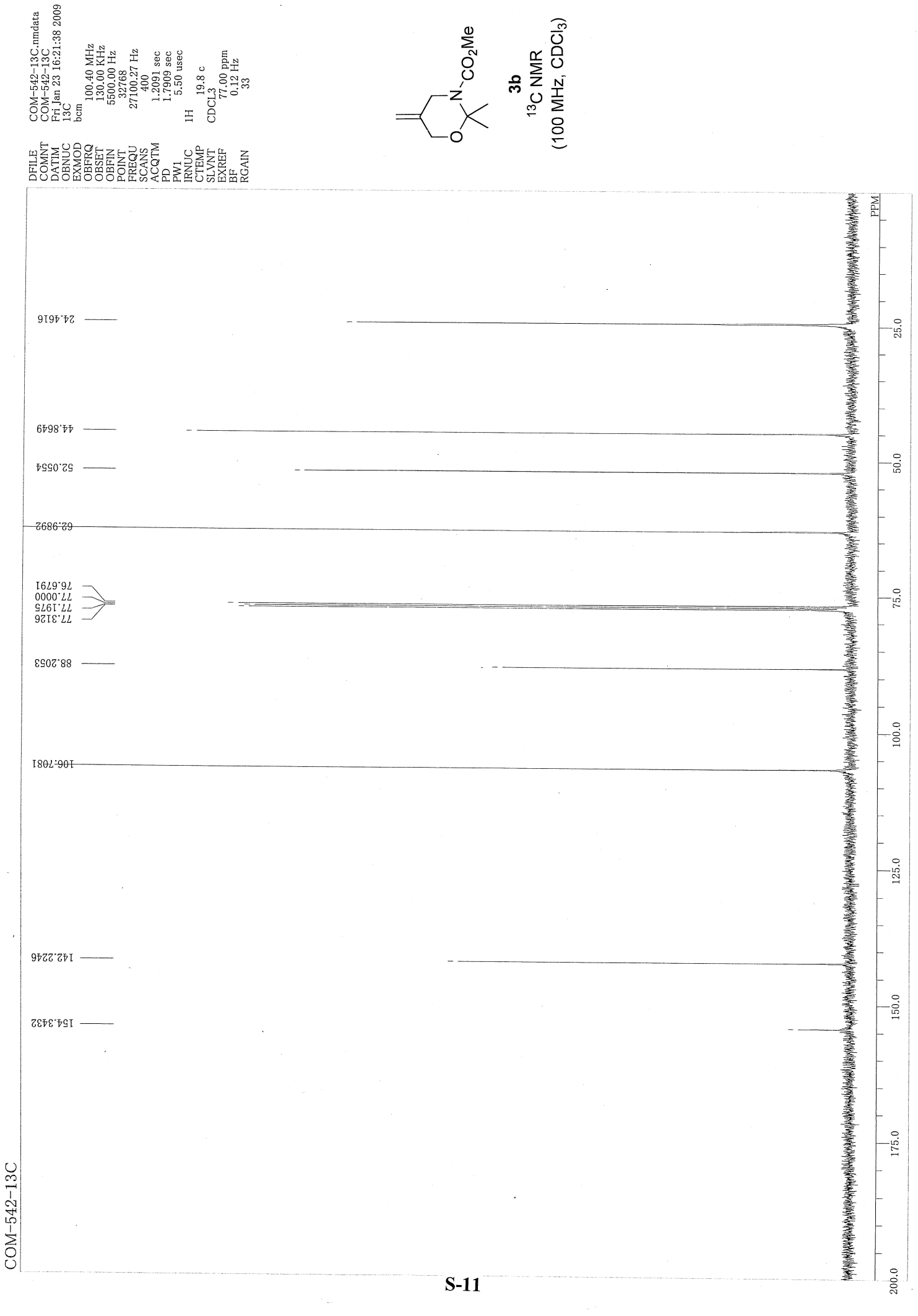
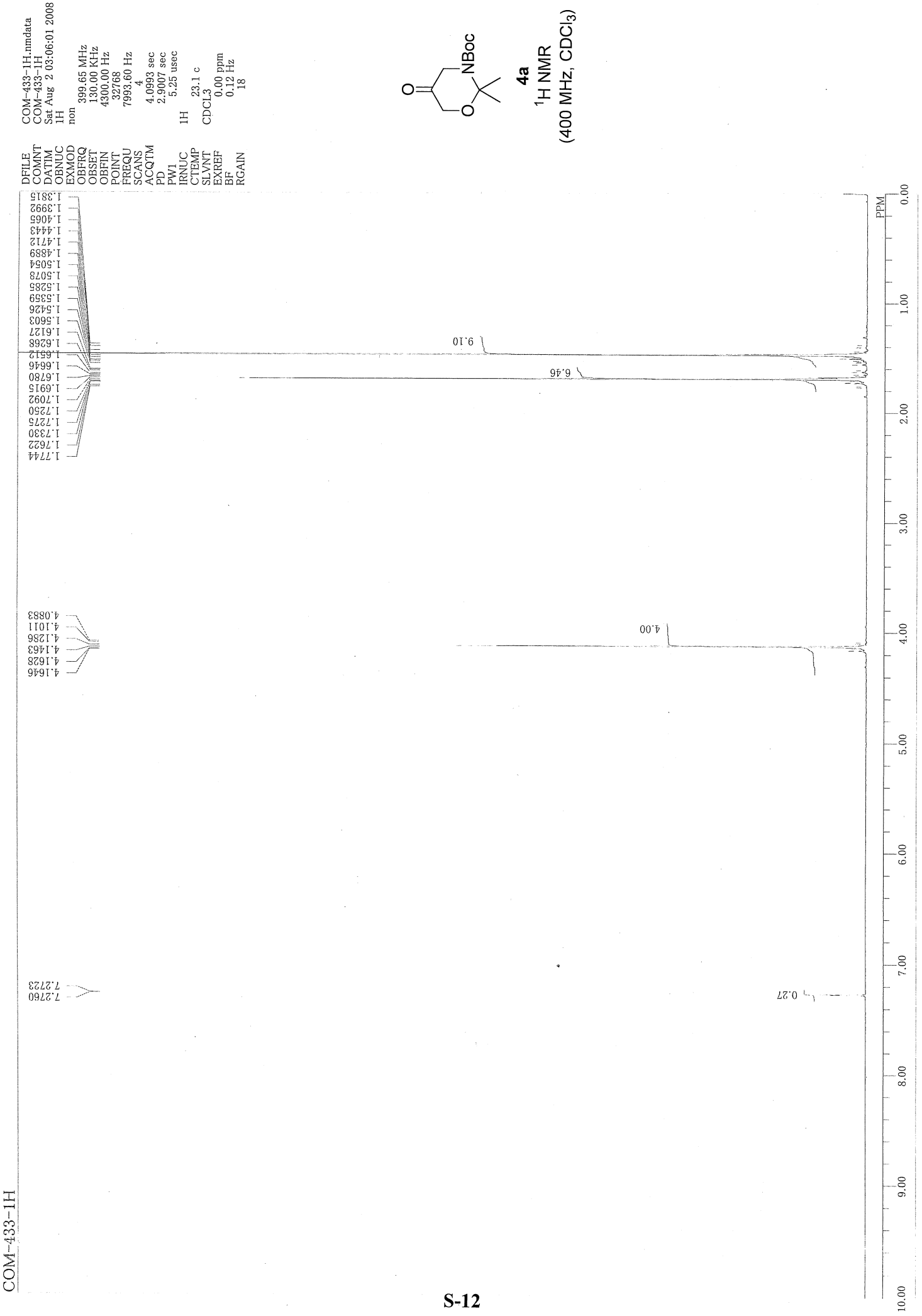
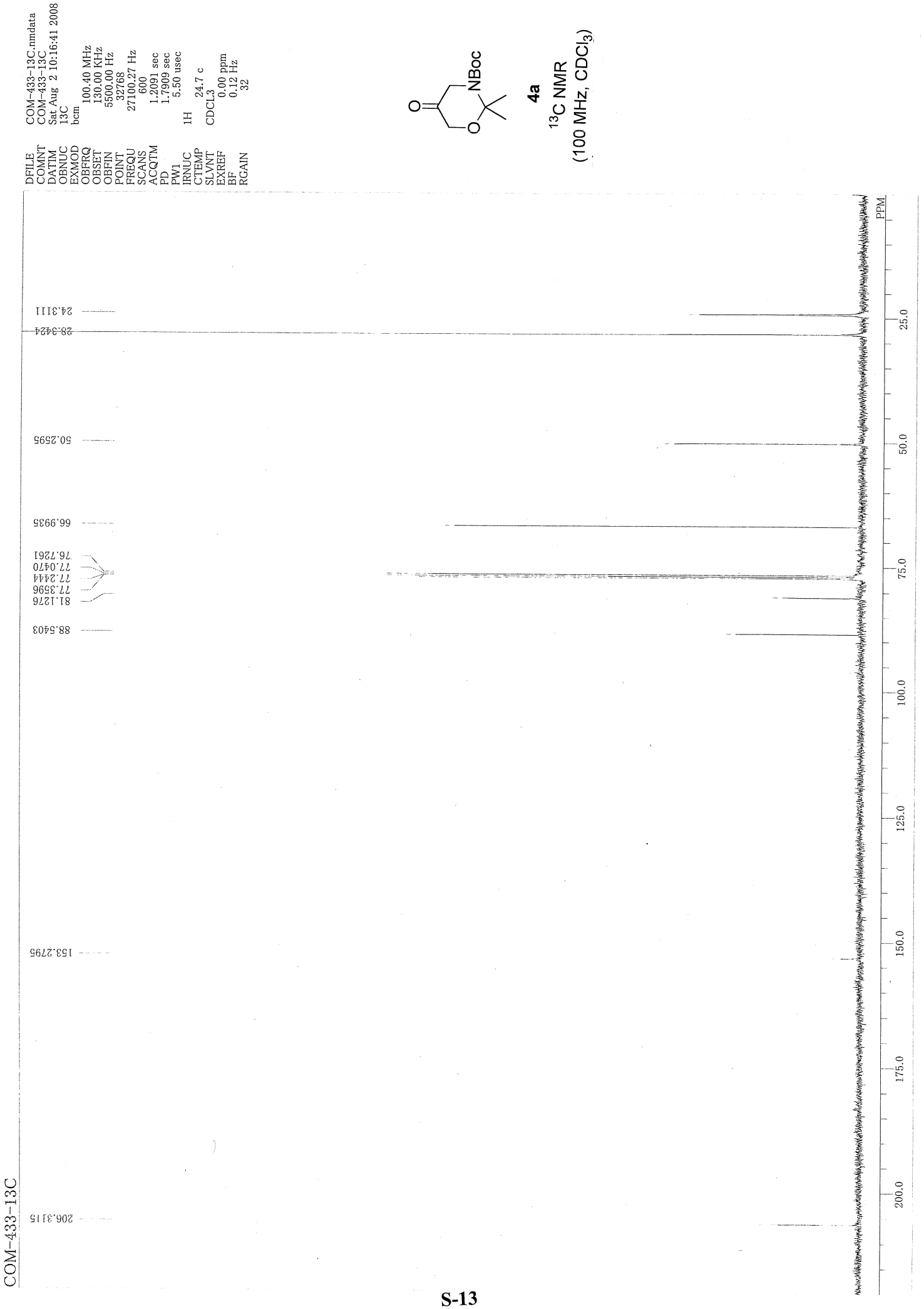
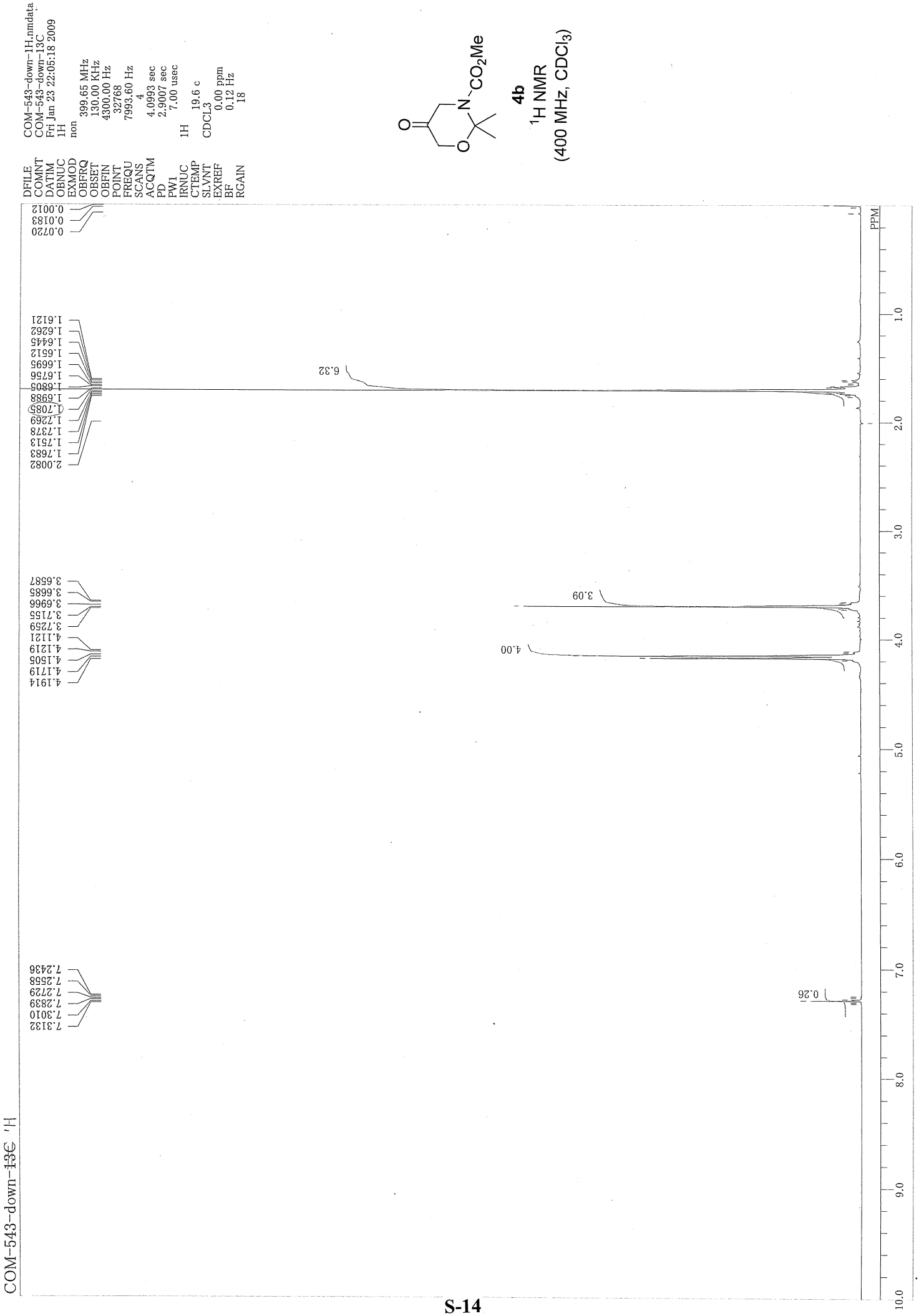
OBSET 130.00 KHz
OBFIN 4300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 4
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 7.00 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18
3b
^{1}H NMR
(400 MHz, $CDCl_3$)
CO_2Me
N
O
PPM
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
6.12
3.04
4.06
2.00
0.07
0.0262
0.0720
1.5969
1.5987
1.6262
1.6280
1.6439
1.6549
3.6075
3.6185
3.6264
3.6441
3.6459
3.6630
3.6728
3.6758
3.6911
3.7021
3.7173
3.7307
3.7338
4.0987
4.1255
4.1280
4.1548
4.3415
4.3659
4.3684
4.3708
4.3733
4.3971
4.7656
4.7925
4.7949
4.8212
4.9145
4.9383
4.9414
4.9438
4.9700
7.2906
7.2937
7.3199

COM-542-1H





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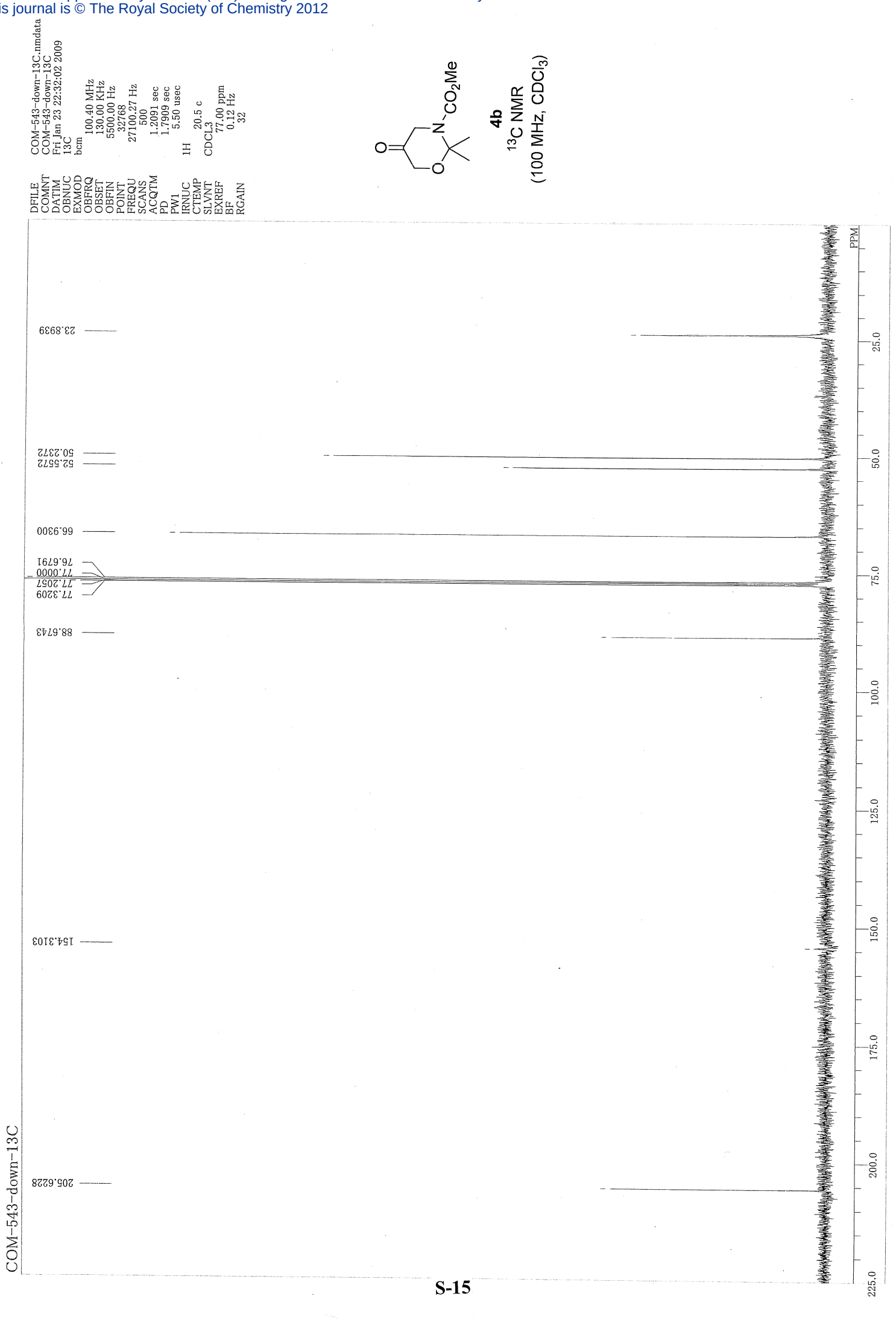
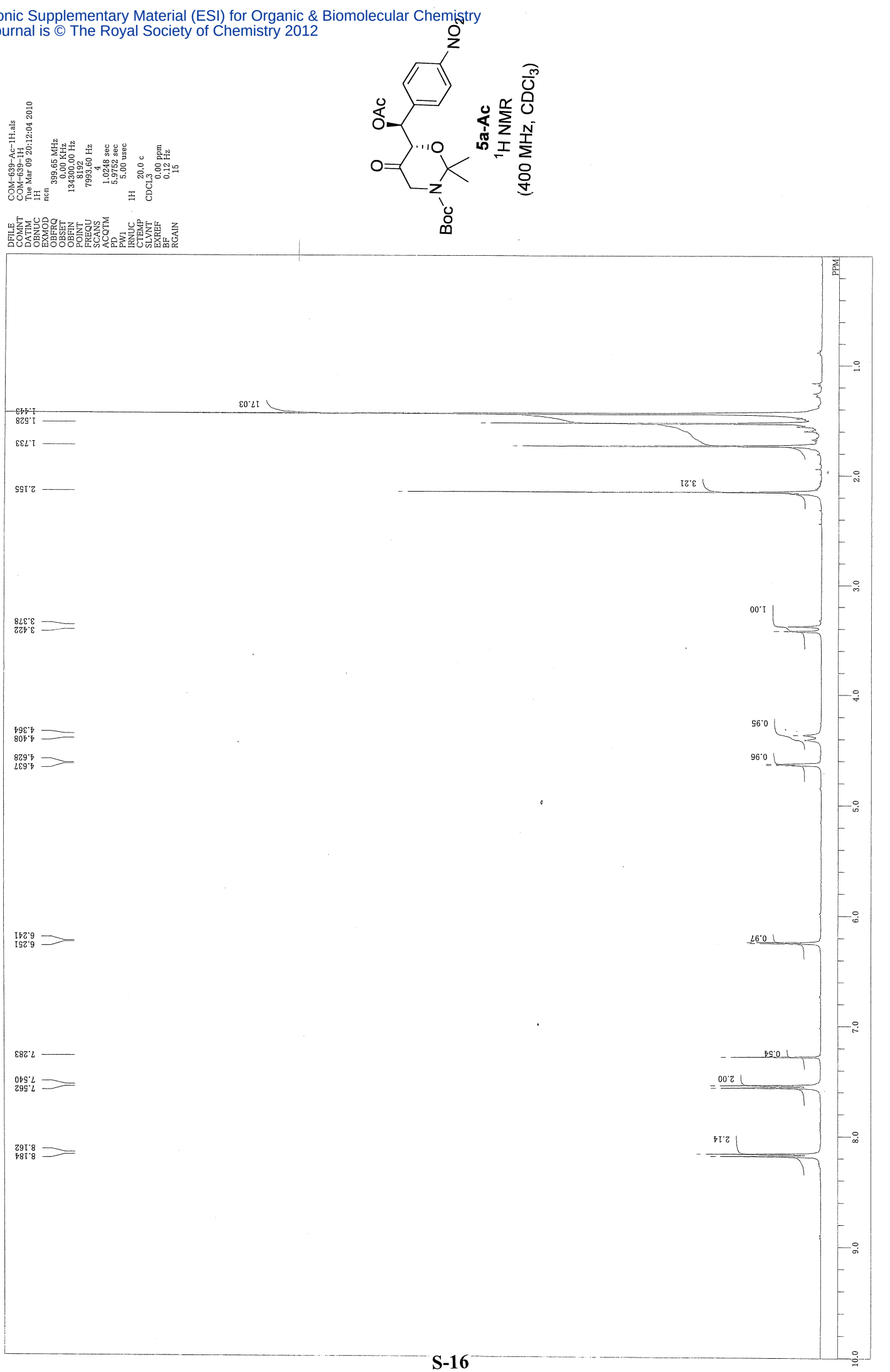
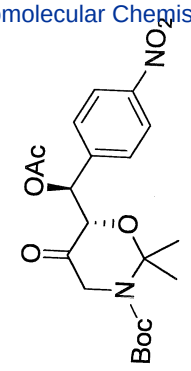
non
399.65 MHz
130.00 KHz
4300.00 Hz
32768
7993.60 Hz
4
4.0993 sec
2.9007 sec
7.00 usec
1H
19.6 c
CDCL3
0.00 ppm
0.12 Hz
18
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
4b
1H NMR
(400 MHz, CDCl3)
N
CO2Me
O
O
0.0012
0.0183
0.0720
1.6121
1.6262
1.6445
1.6512
1.6695
1.6756
1.6805
1.6988
1.7085
1.7269
1.7378
1.7513
1.7683
2.0082
3.6587
3.6685
3.6966
3.7155
3.7259
4.1121
4.1219
4.1505
4.1719
4.1914
7.2436
7.2558
7.2729
7.2839
7.3010
7.3132
6.32
3.09
4.00
0.26
PPM
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
COM-543-down-13C 1H







5a-Ac
^{13}C NMR
(100 MHz, $CDCl_3$)

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OBNUC 13C
EXMOD bcm
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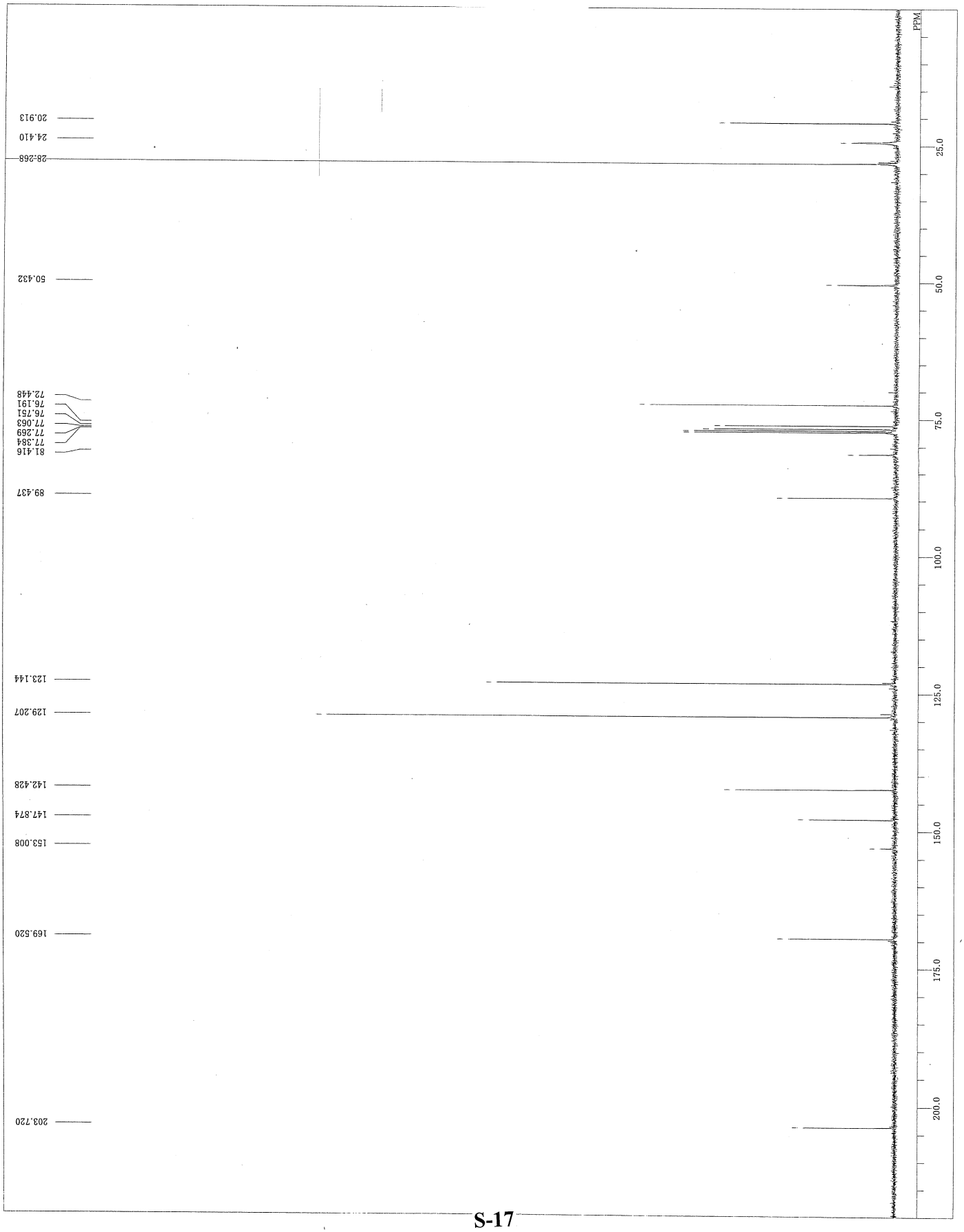
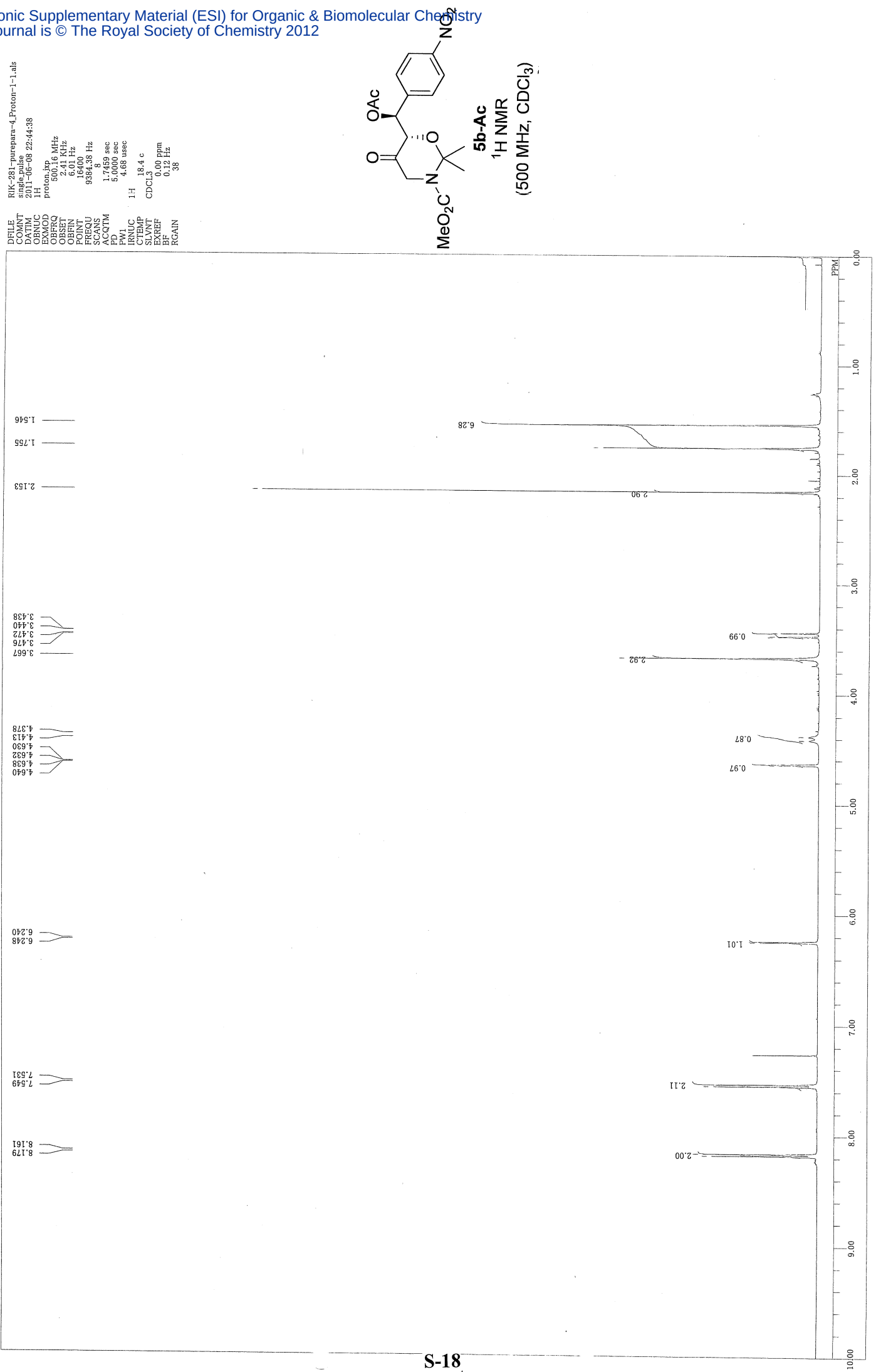
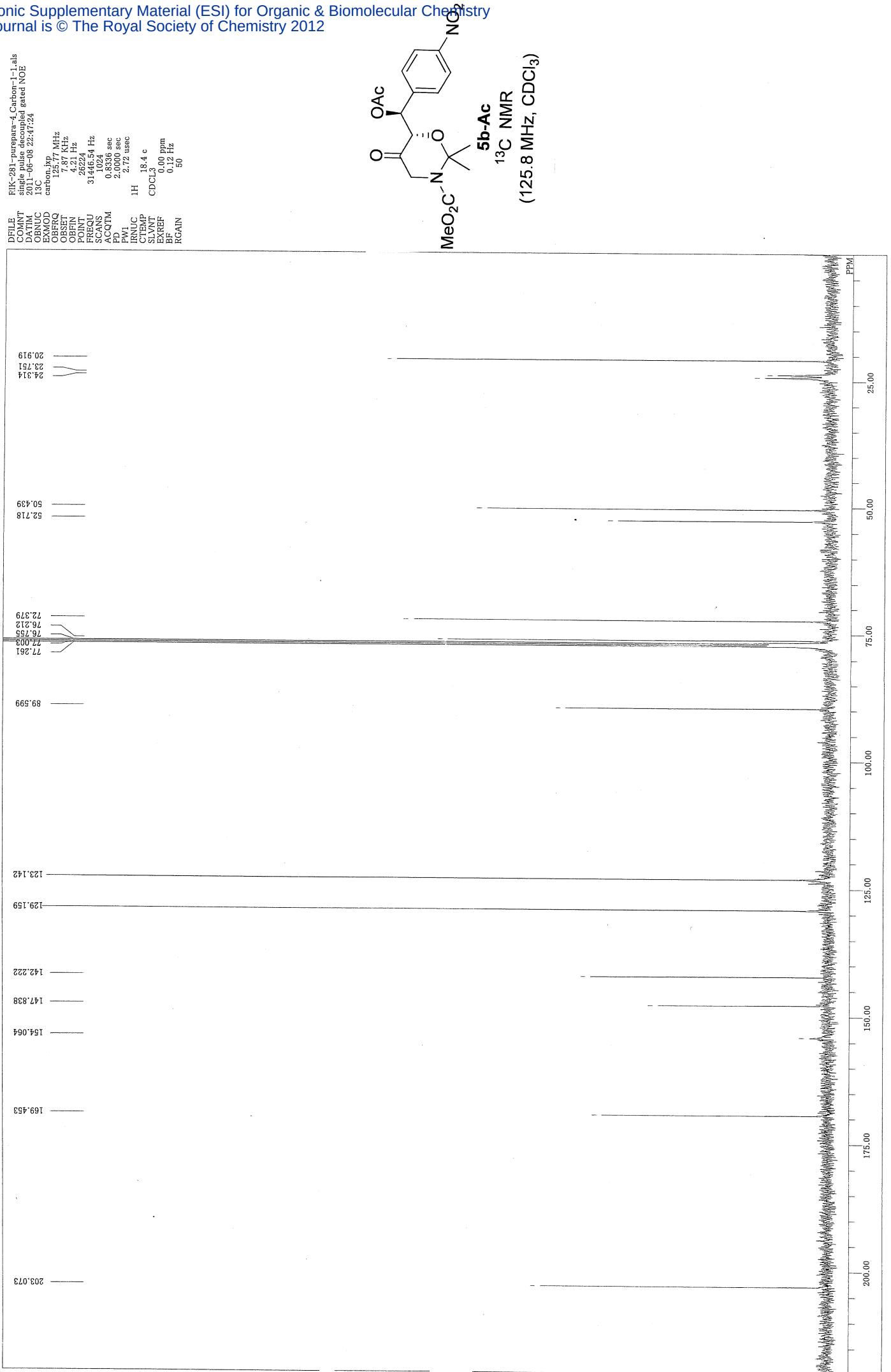
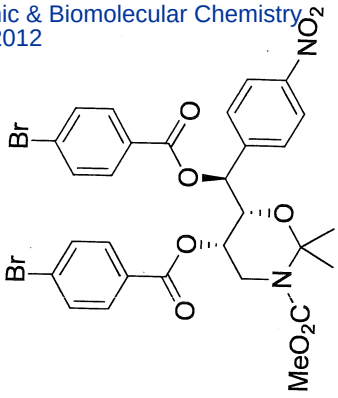
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1000
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28



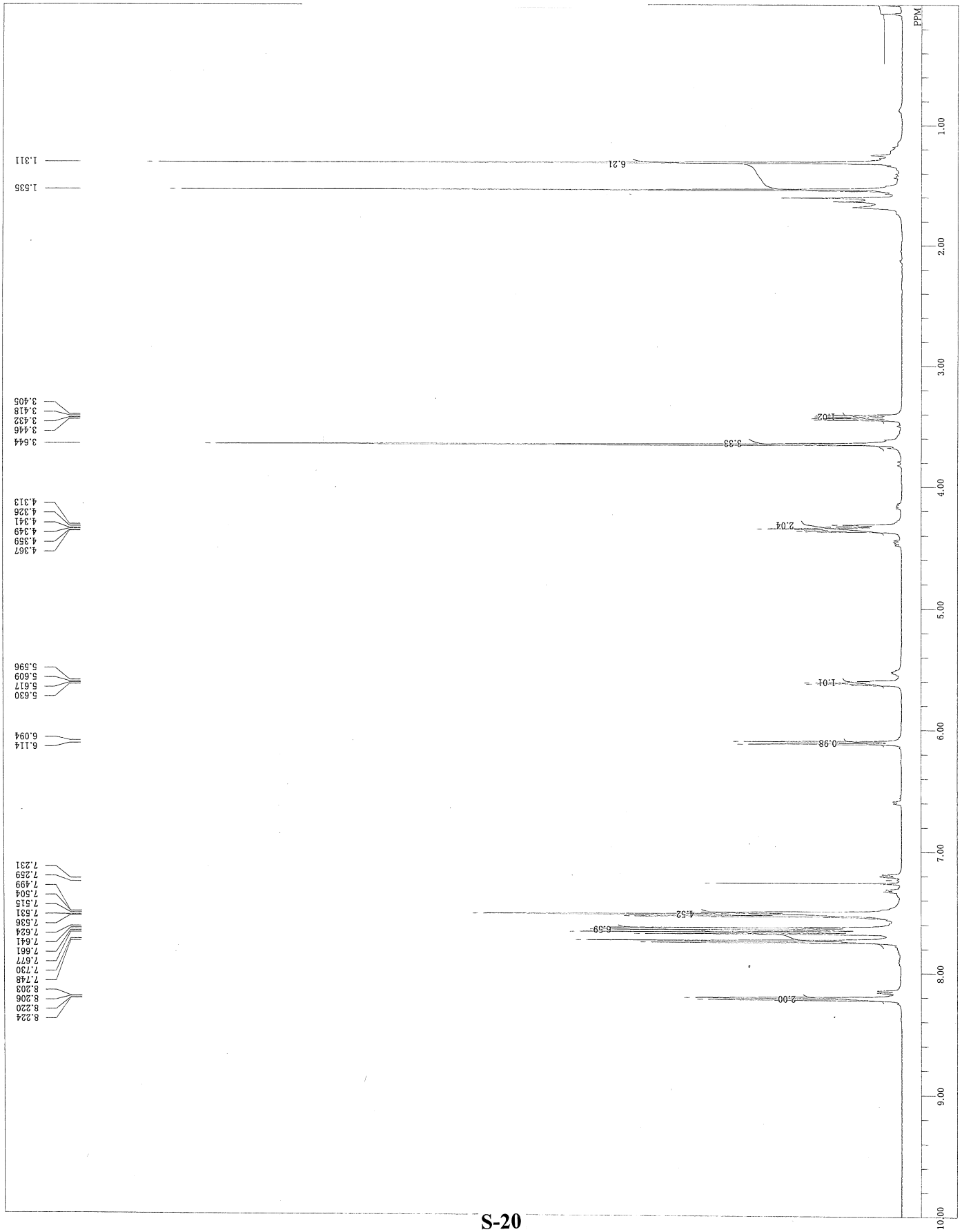


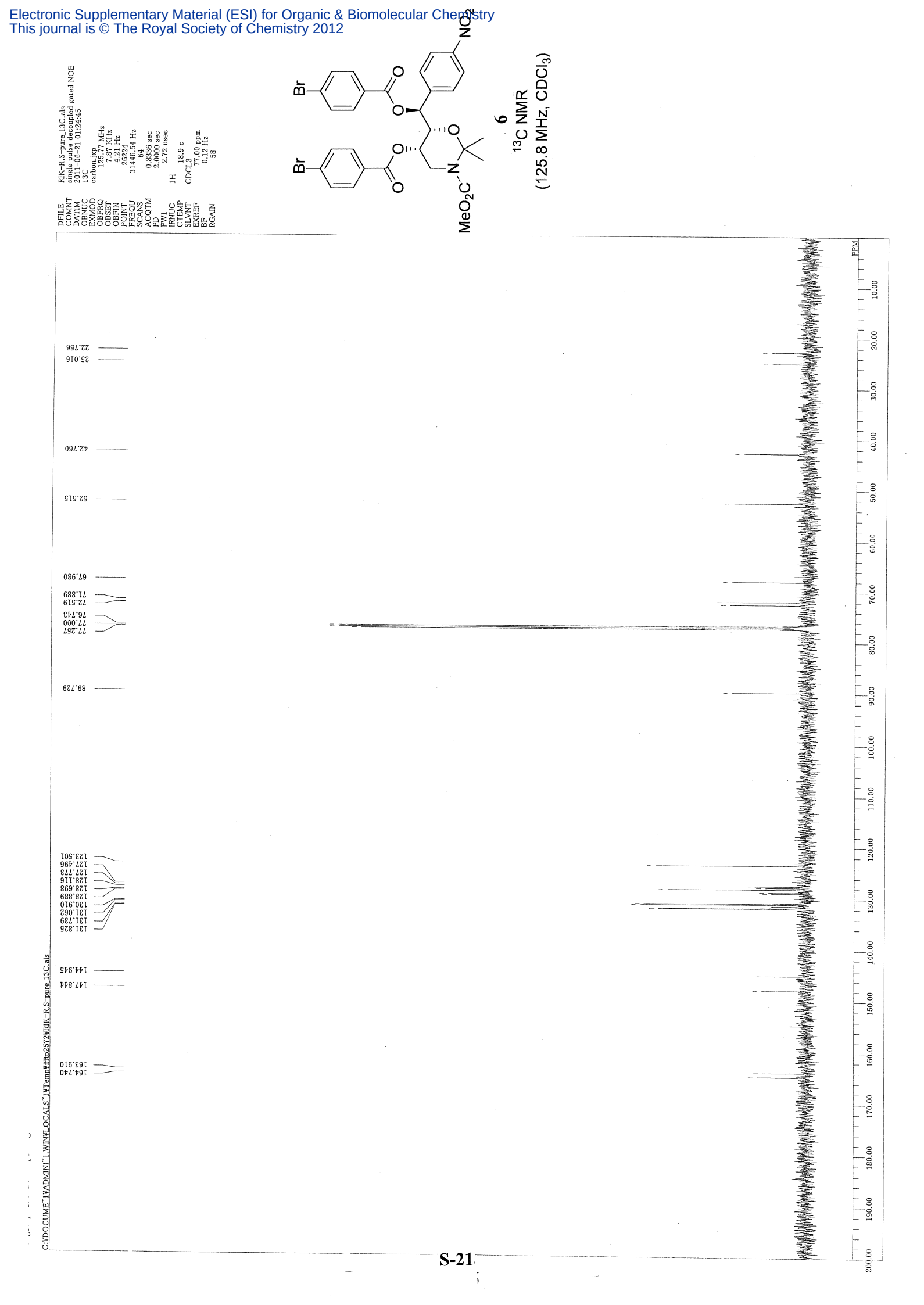


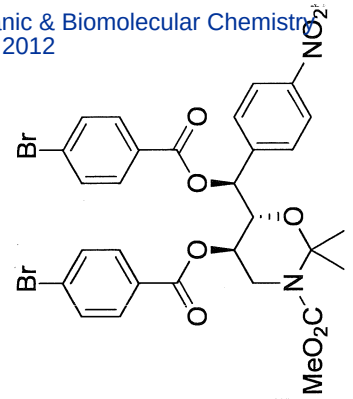


6

^{1}H NMR
(500 MHz, $CDCl_3$)

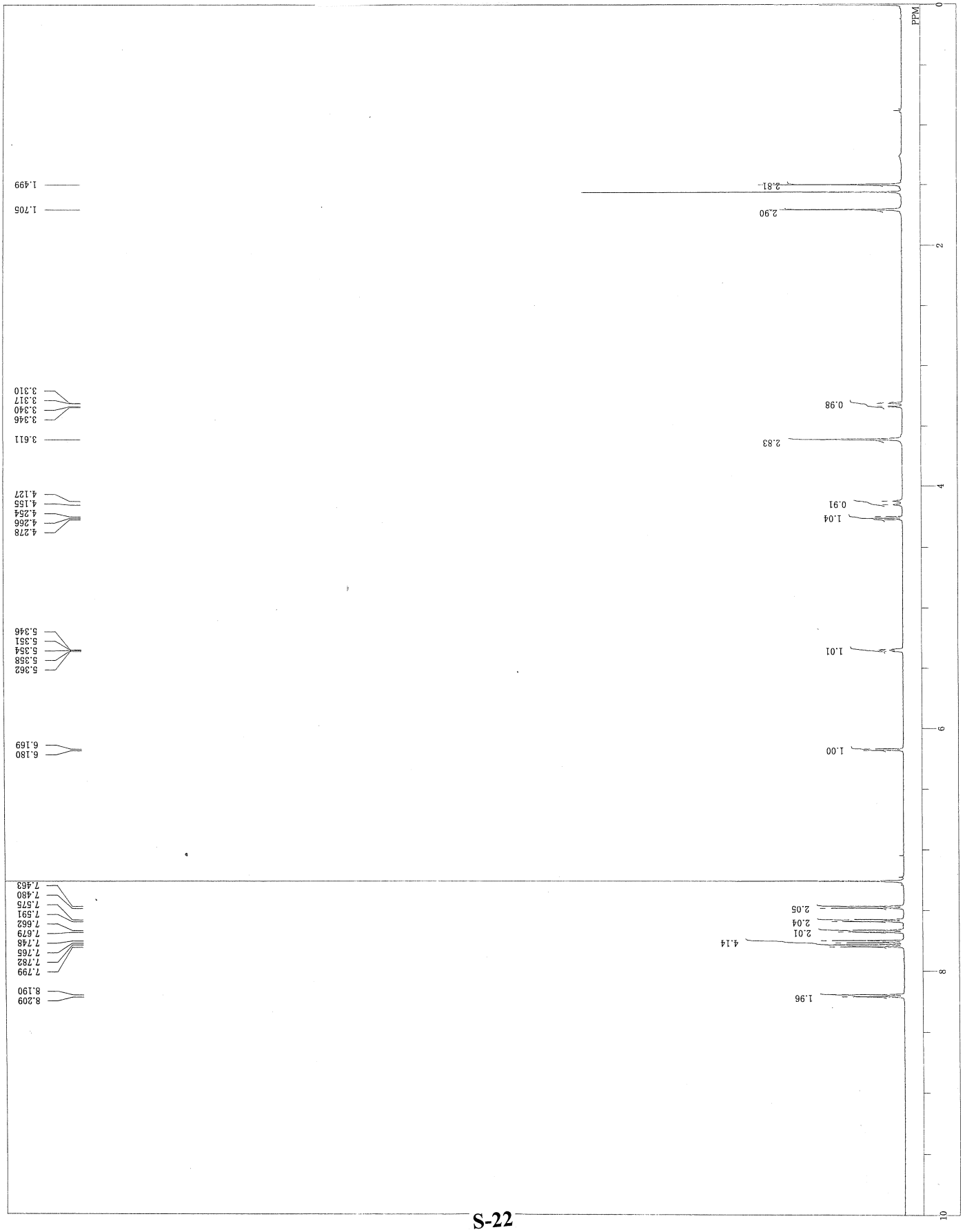


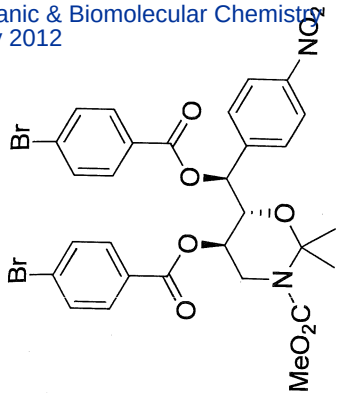




7

^{1}H NMR
(500 MHz, $CDCl_3$)





7

^{13}C NMR

(125.8 MHz, $CDCl_3$)

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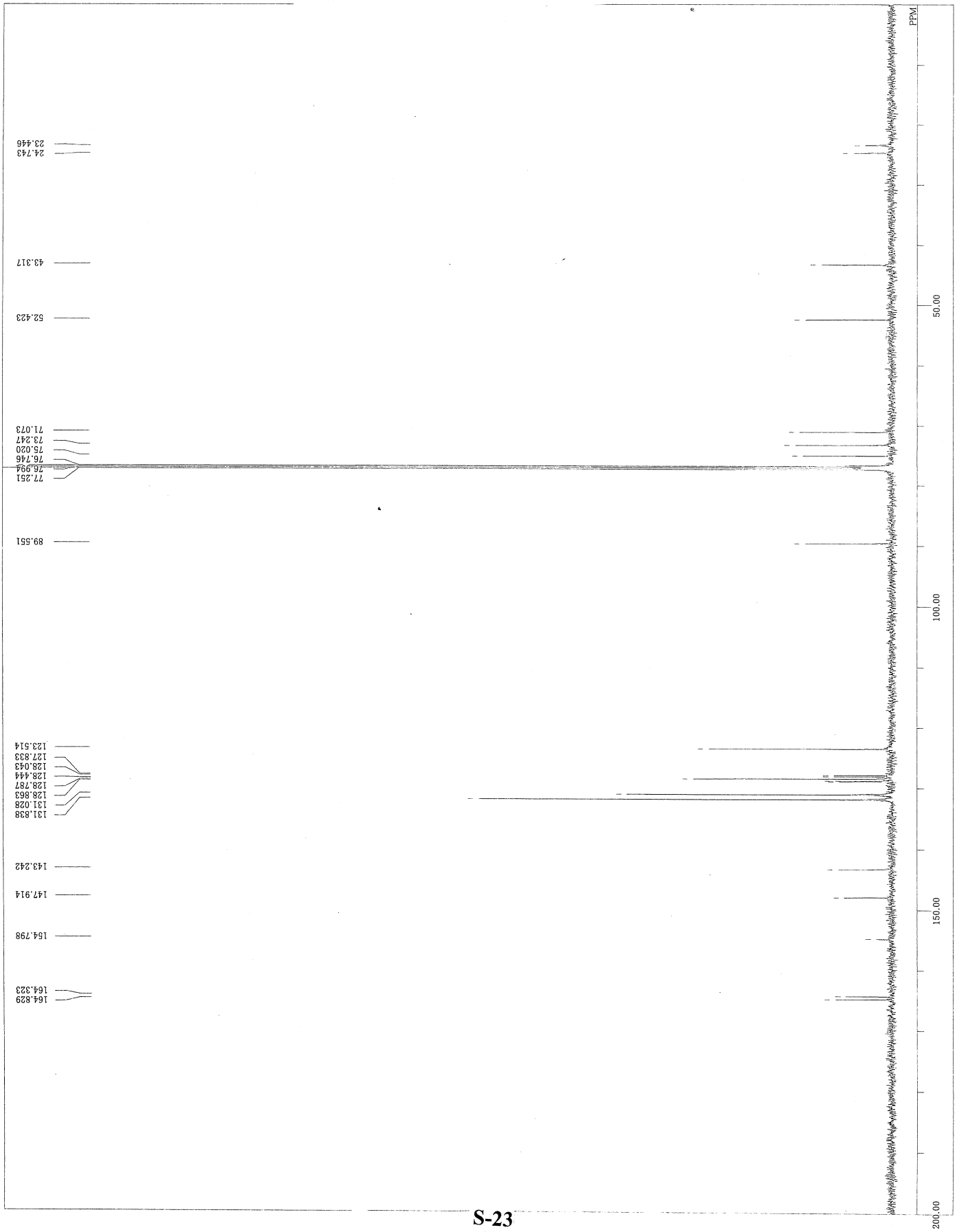
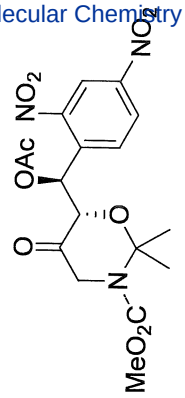
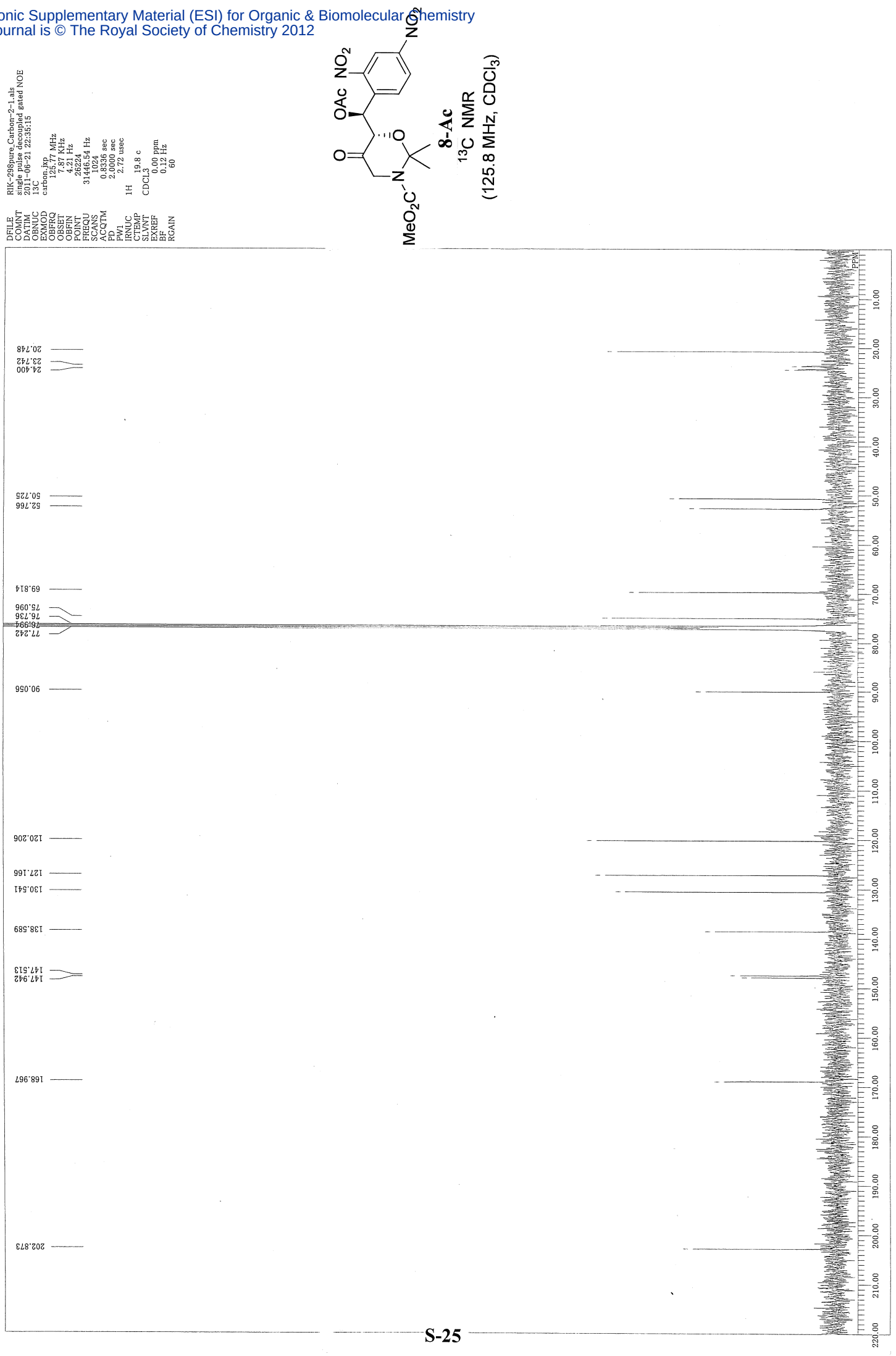
single pulse decoupled gated NOE
2011-06-16 00:35:60
13C
carbon.jxp
125.77 MHz
7.87 KHz
4.21 Hz
32780
39308.18 Hz
512
0.8336 sec
2.0000 sec
2.72 usec
1H 18.9 c
CDCl3
0.00 ppm
0.12 Hz
50

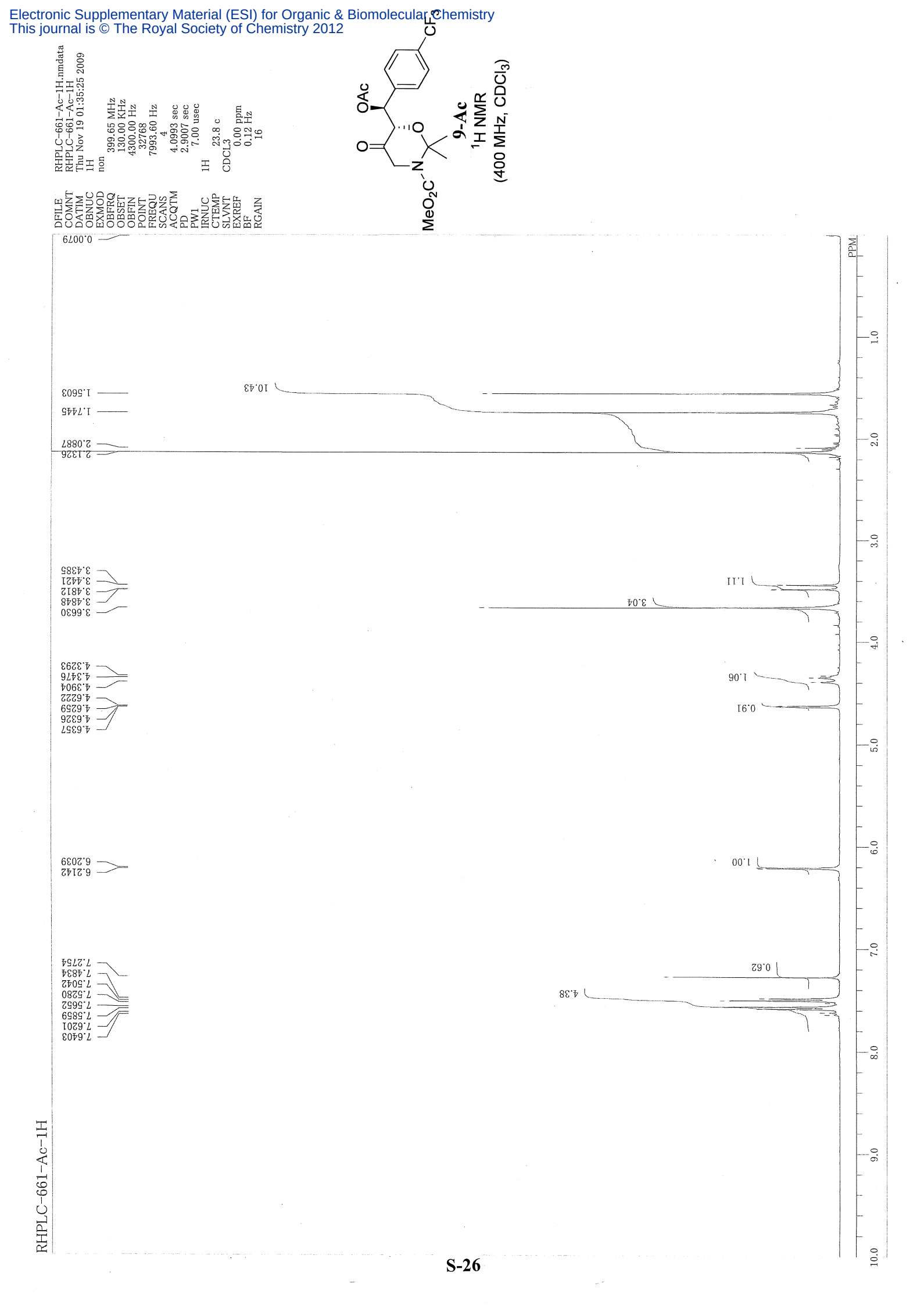
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

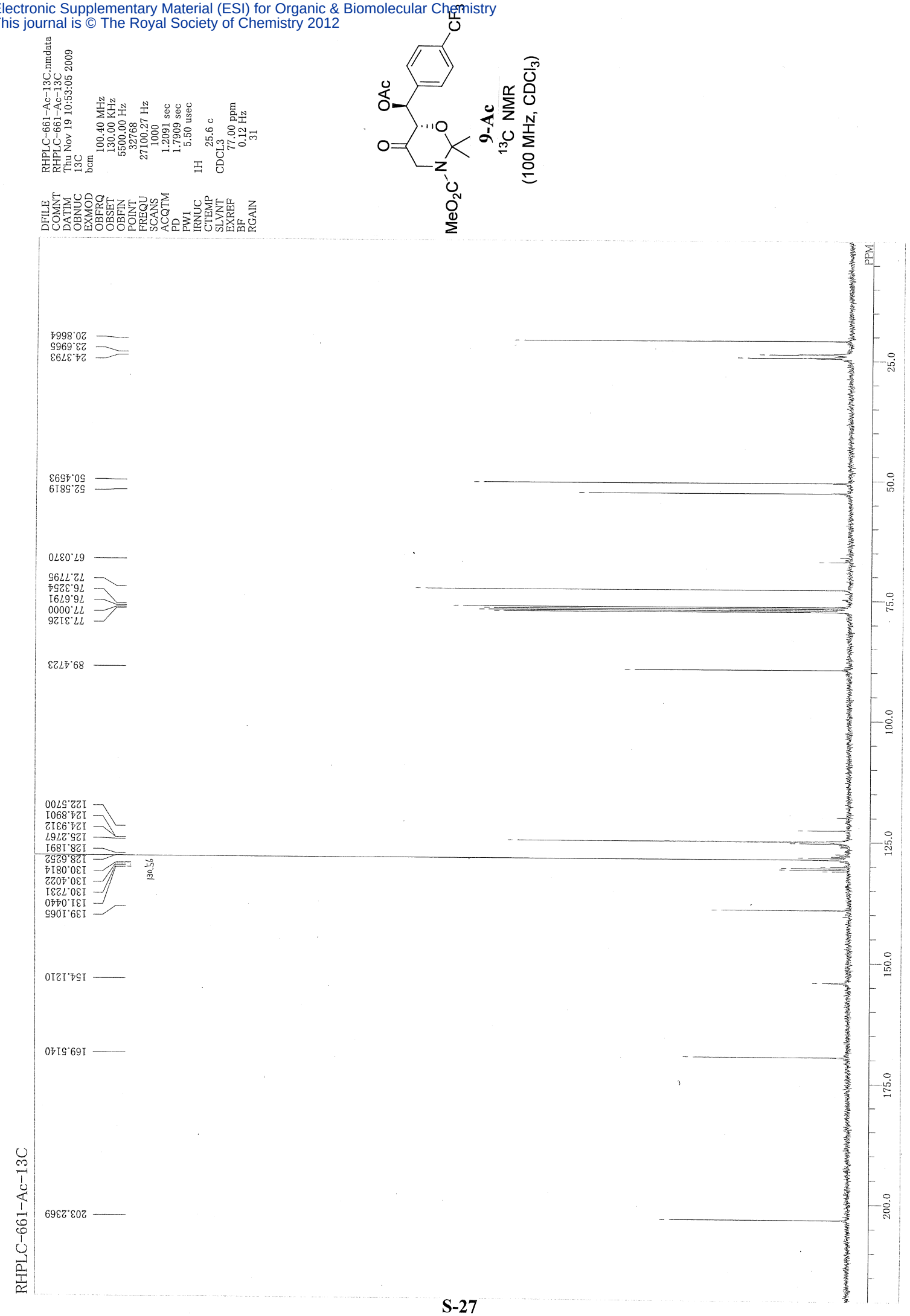


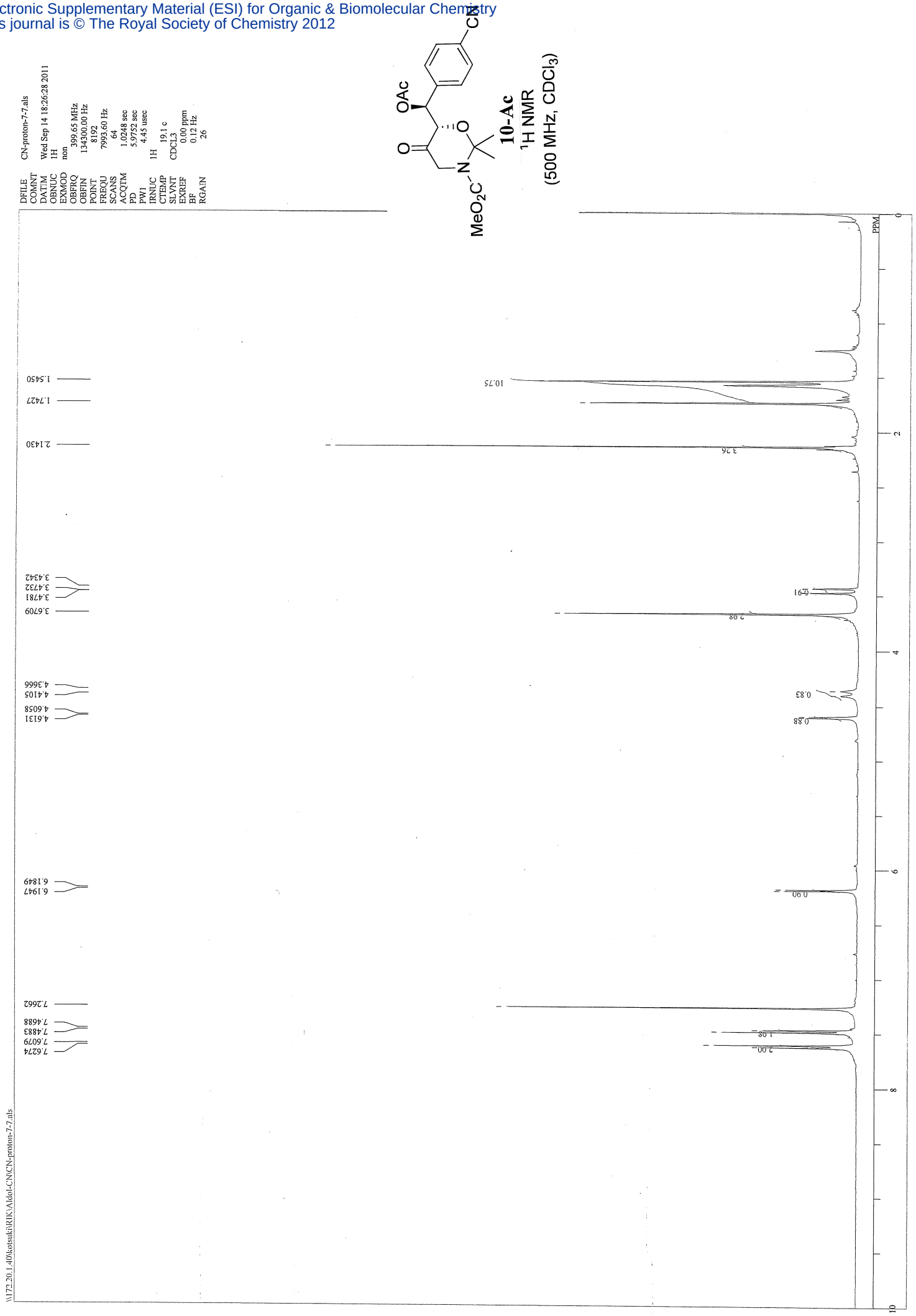


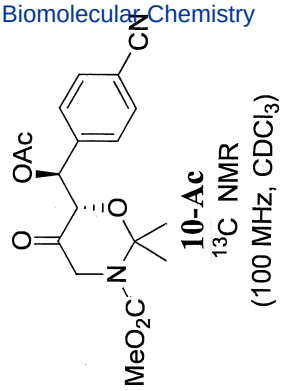
8-Ac
^{1}H NMR
(500 MHz, $CDCl_3$)



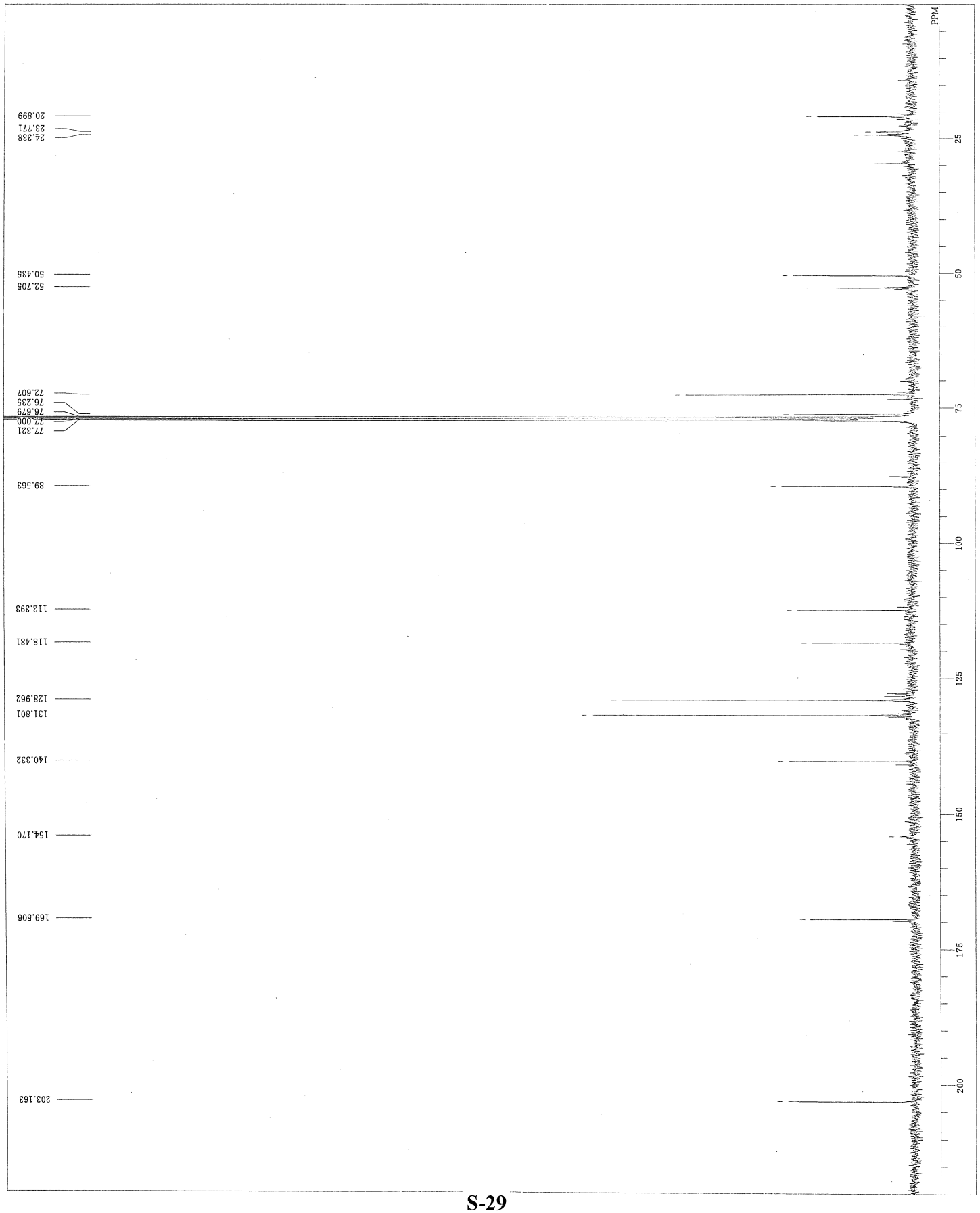


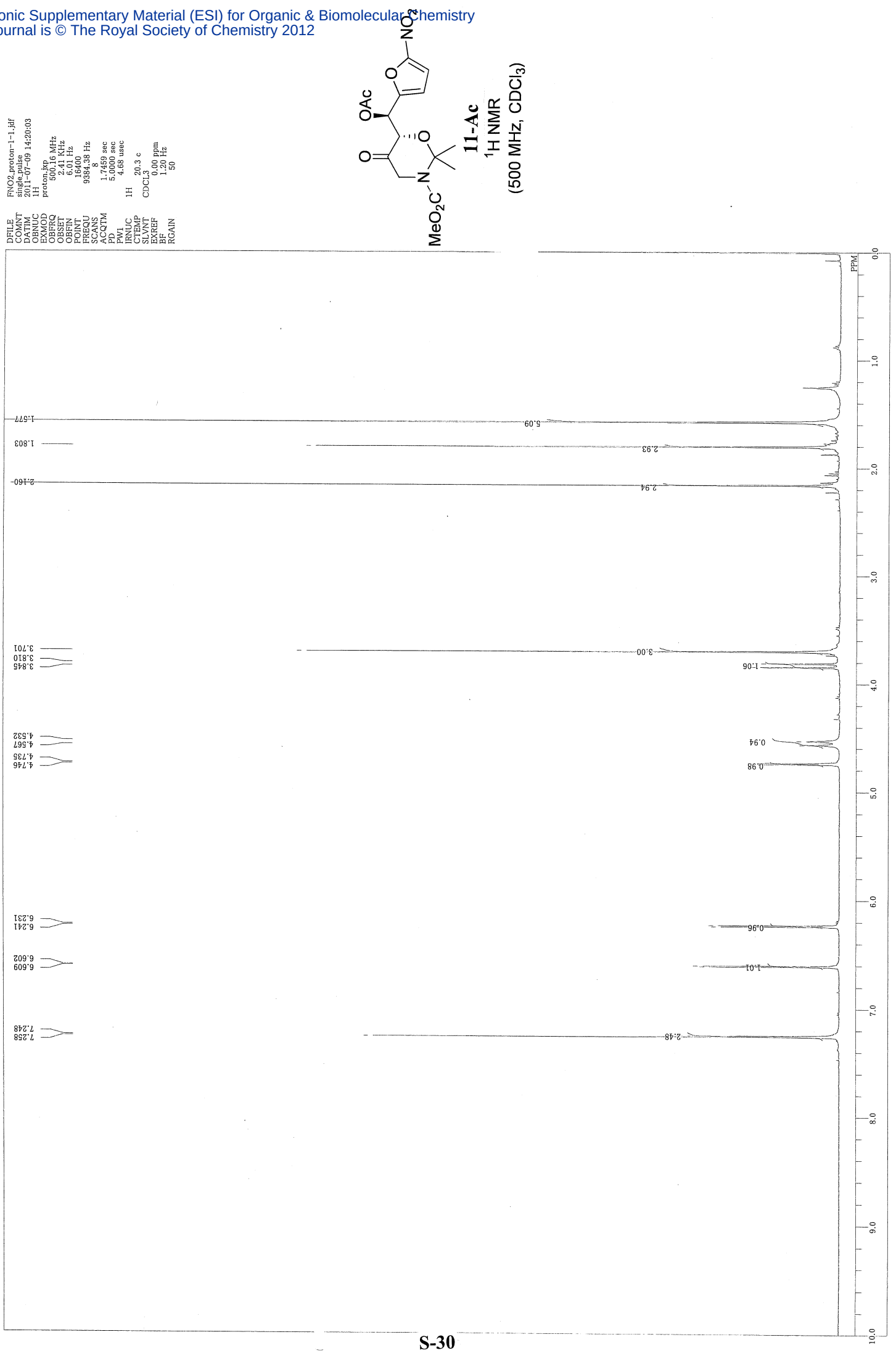


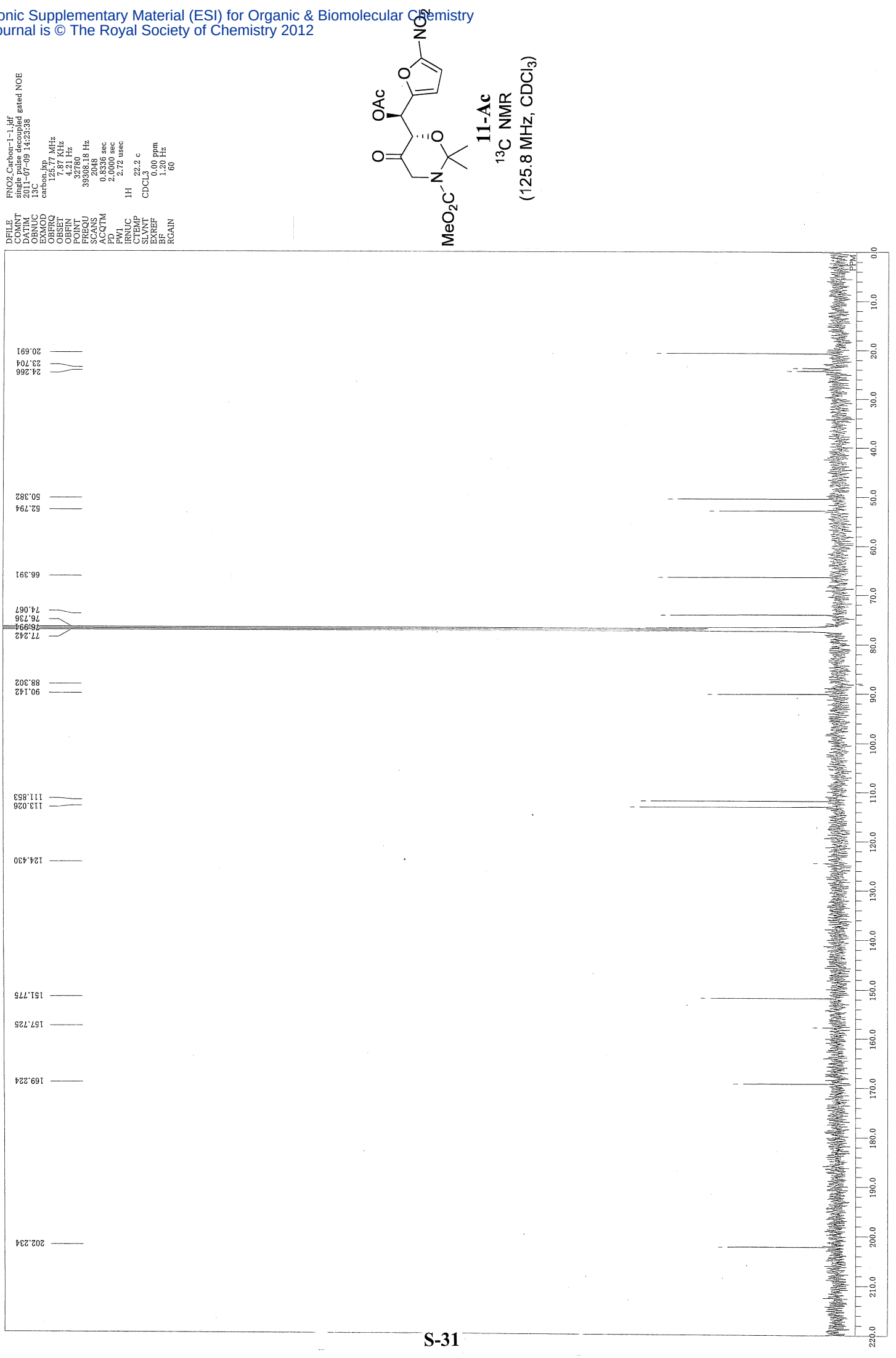


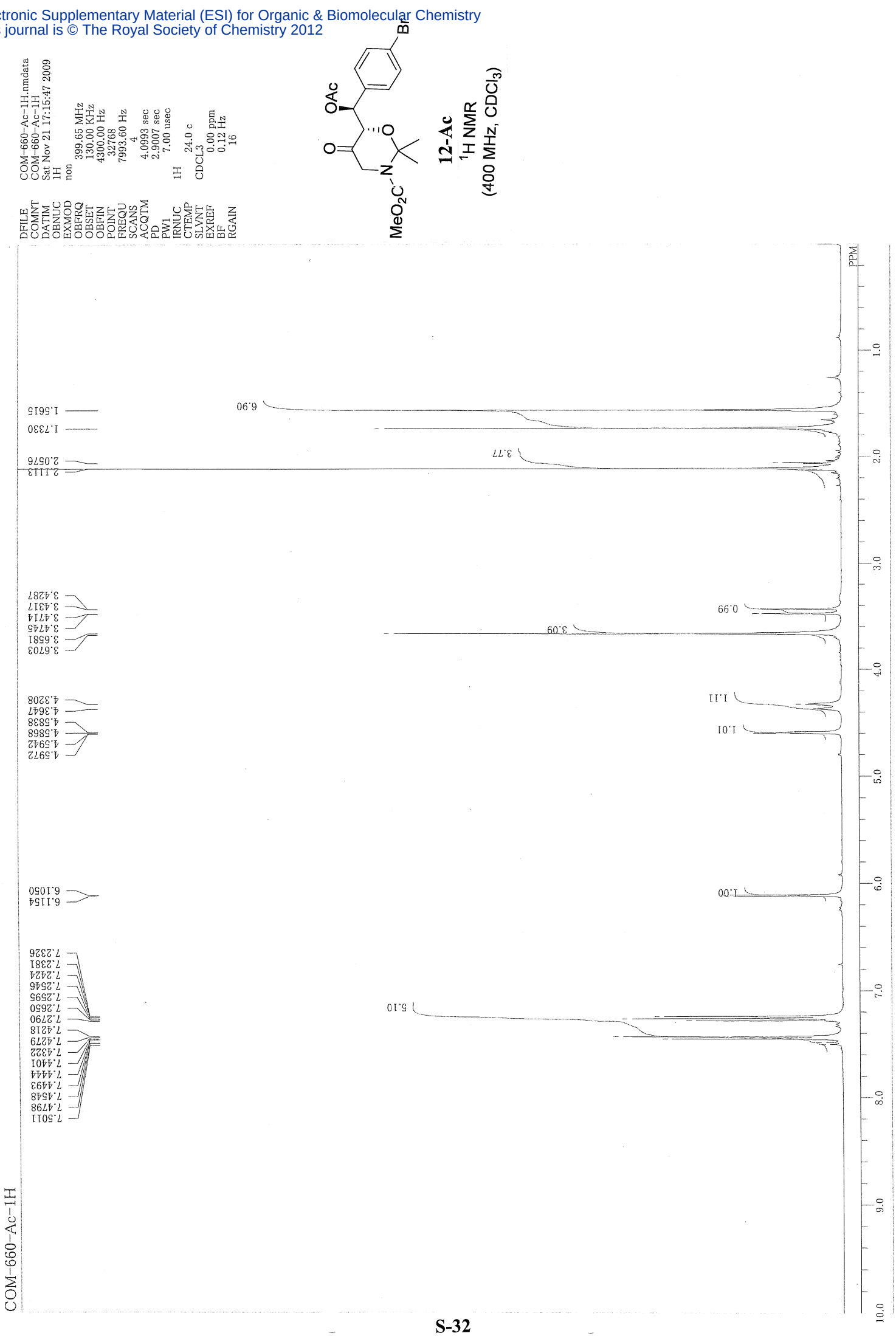


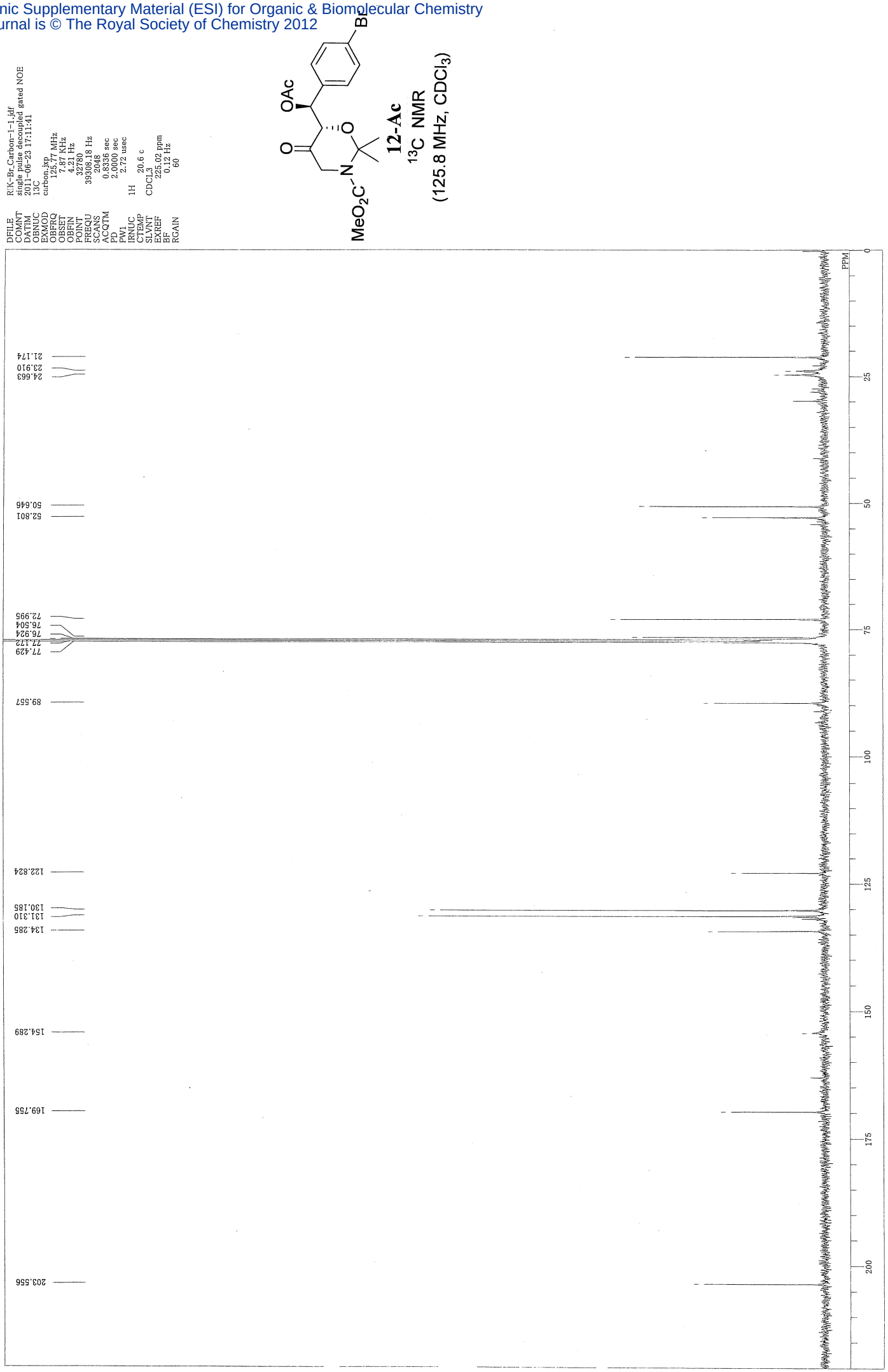
10-Ac
^{13}C NMR
(100 MHz, $CDCl_3$)

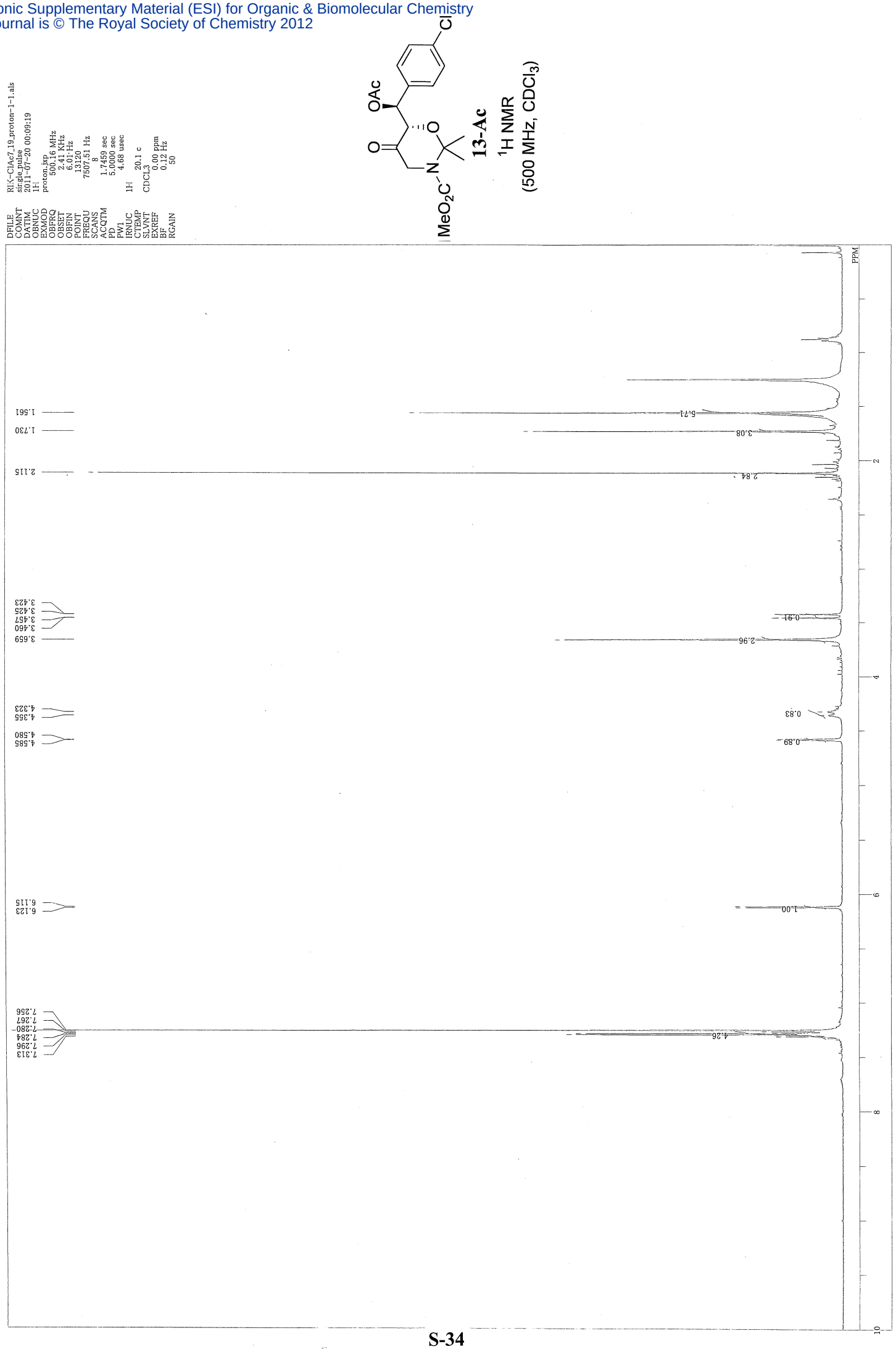


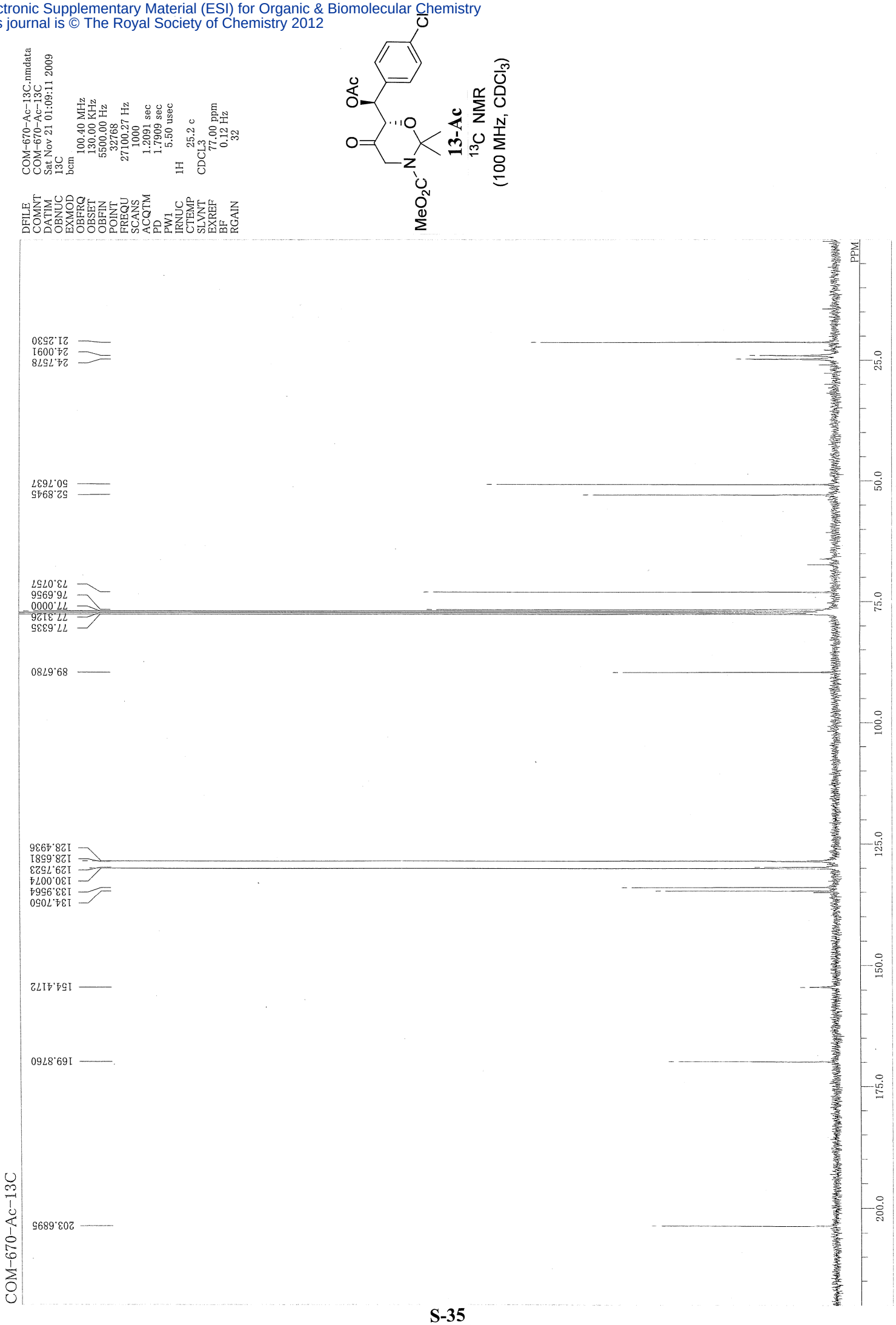


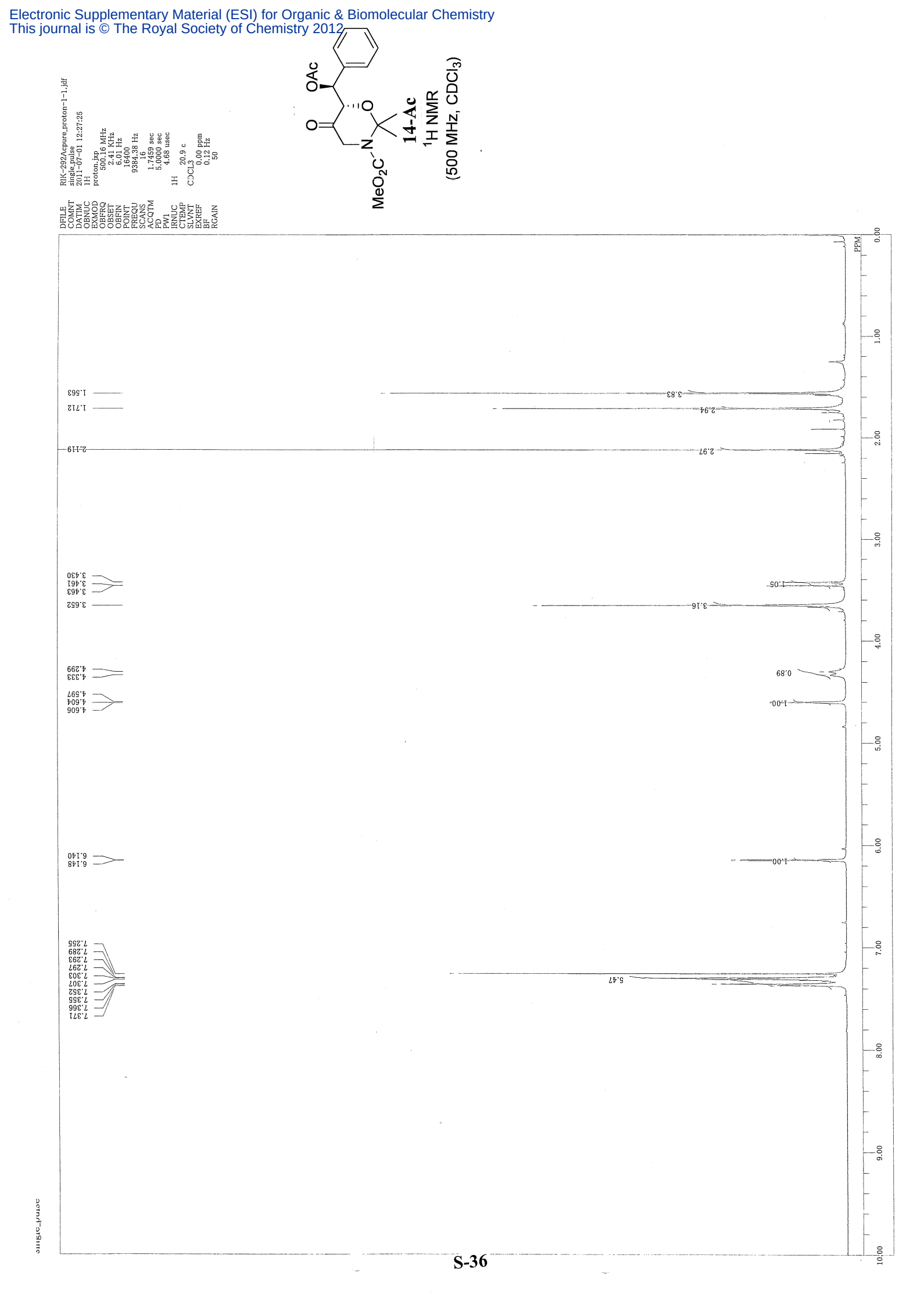


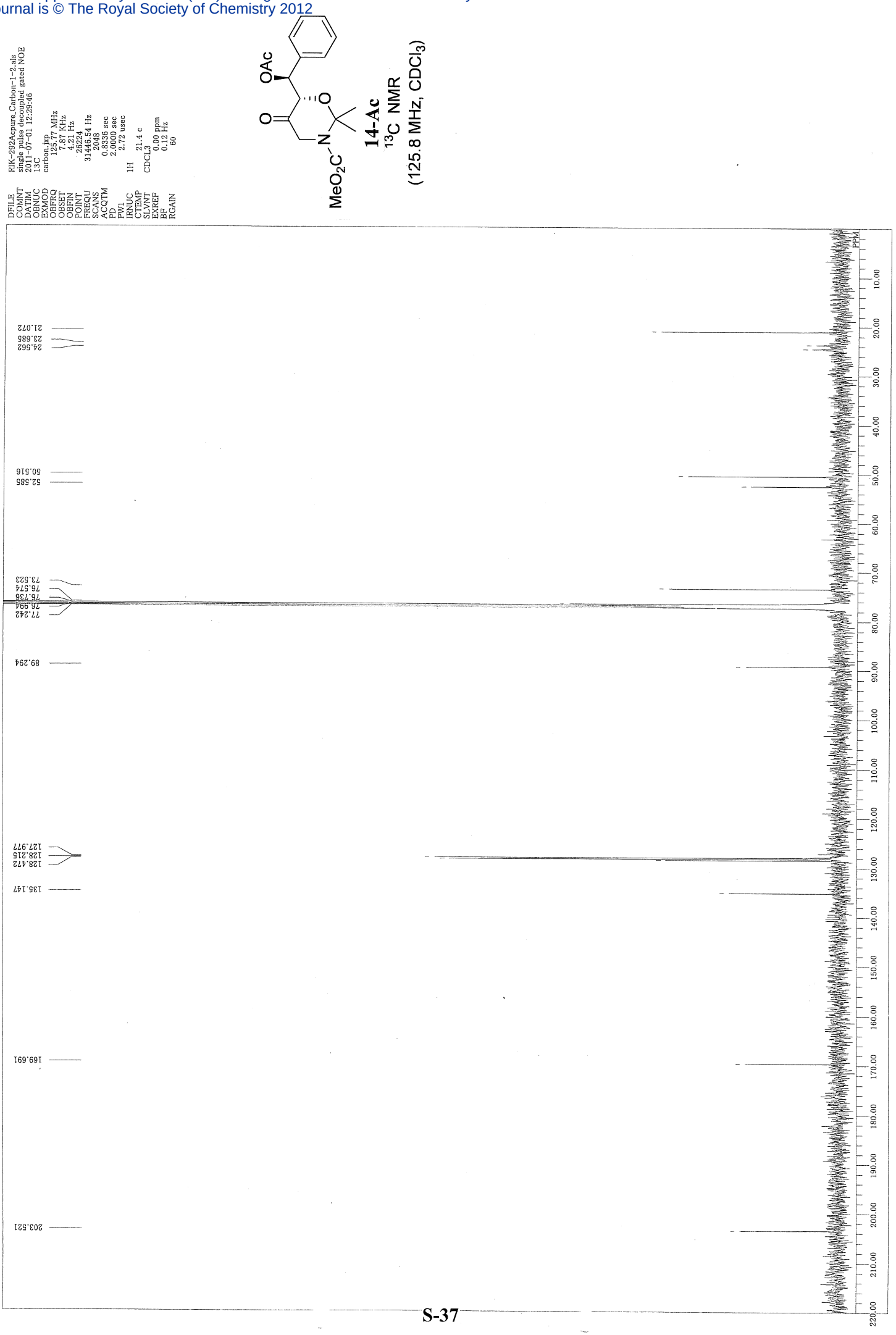


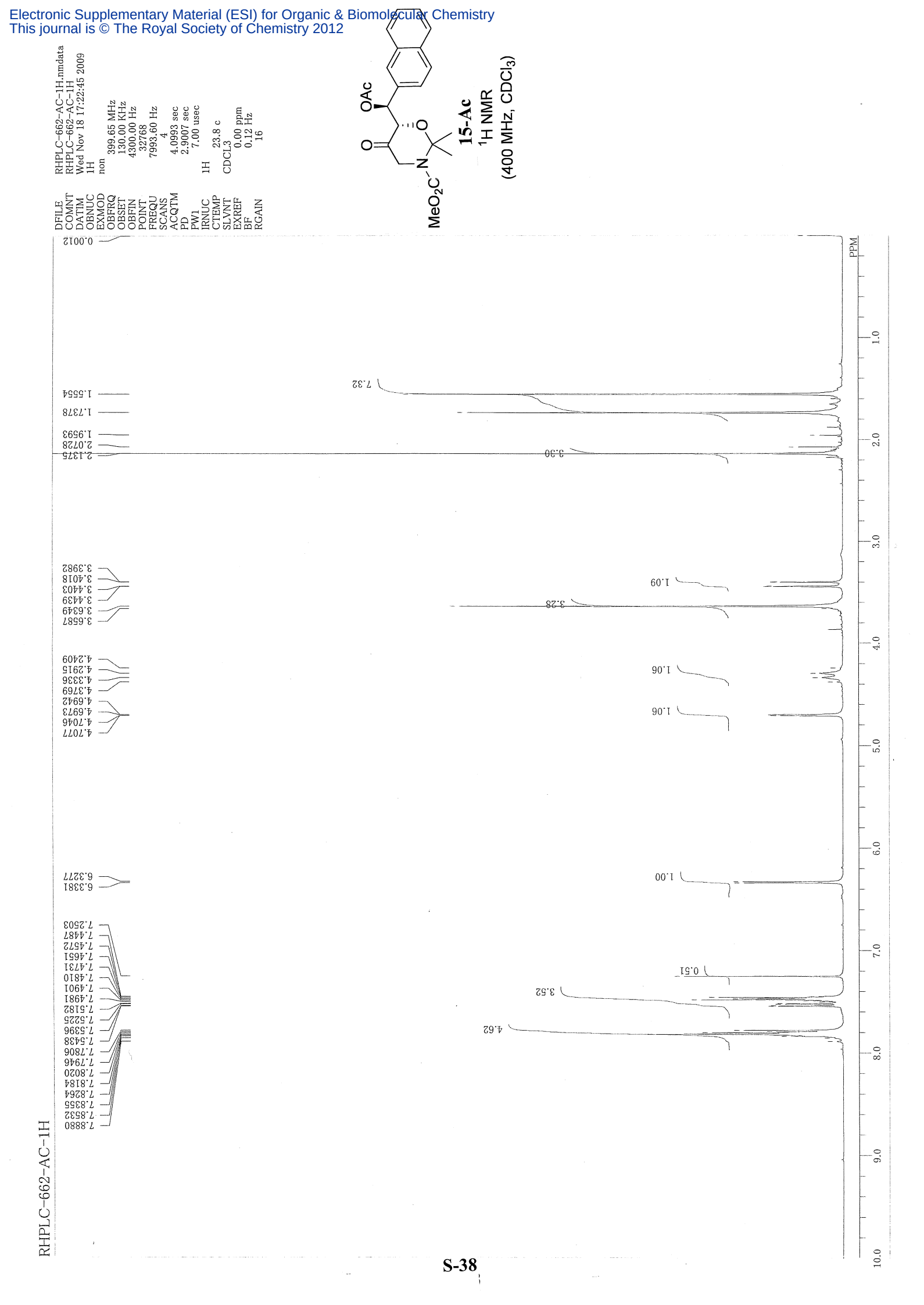


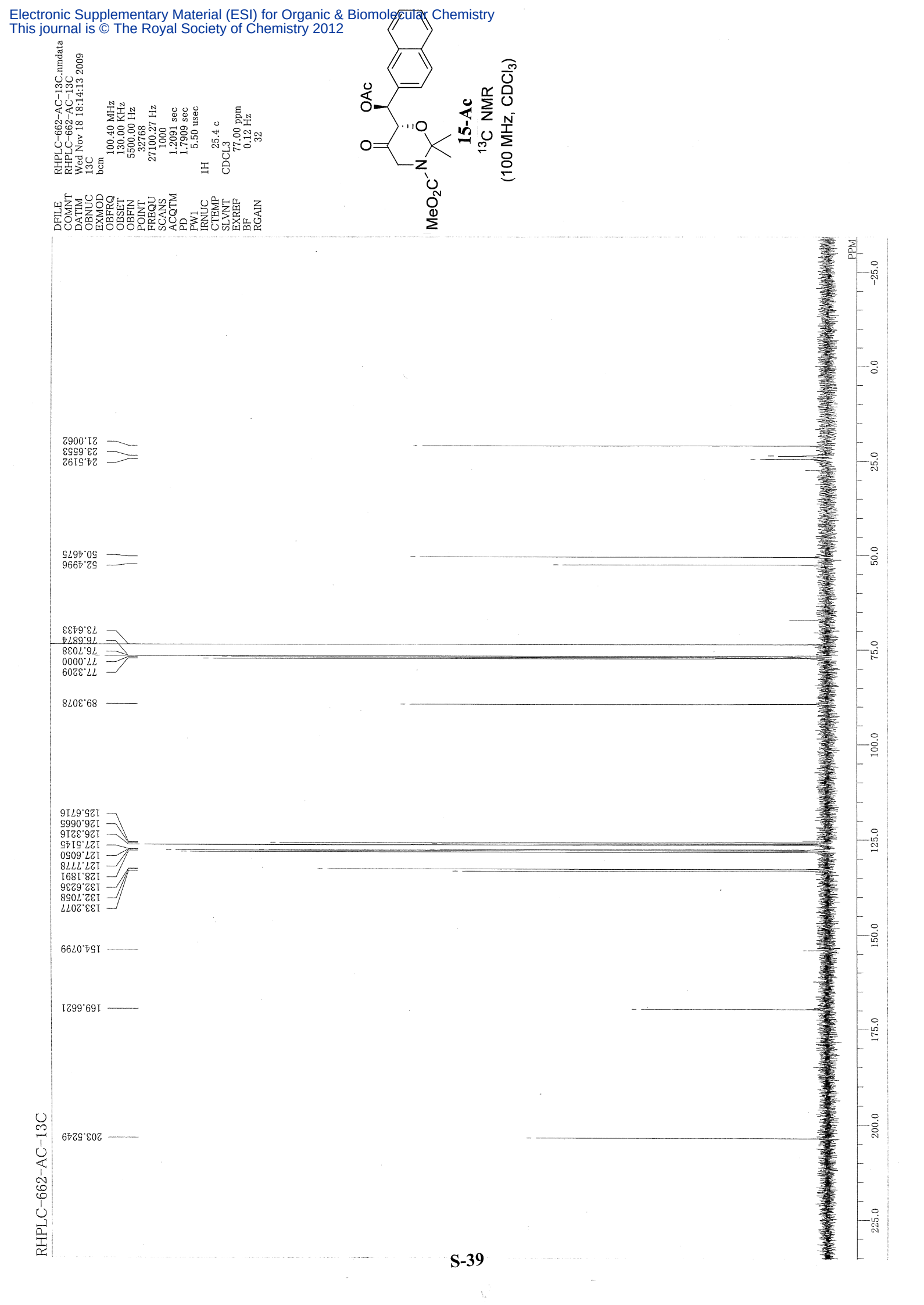


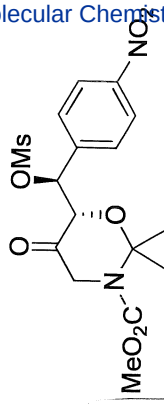






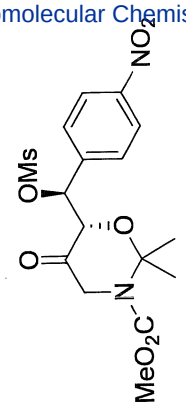






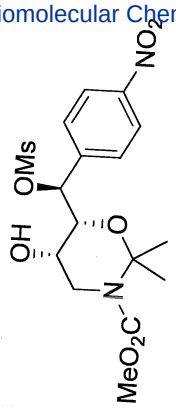
16

^{1}H NMR
(500 MHz, $CDCl_3$)



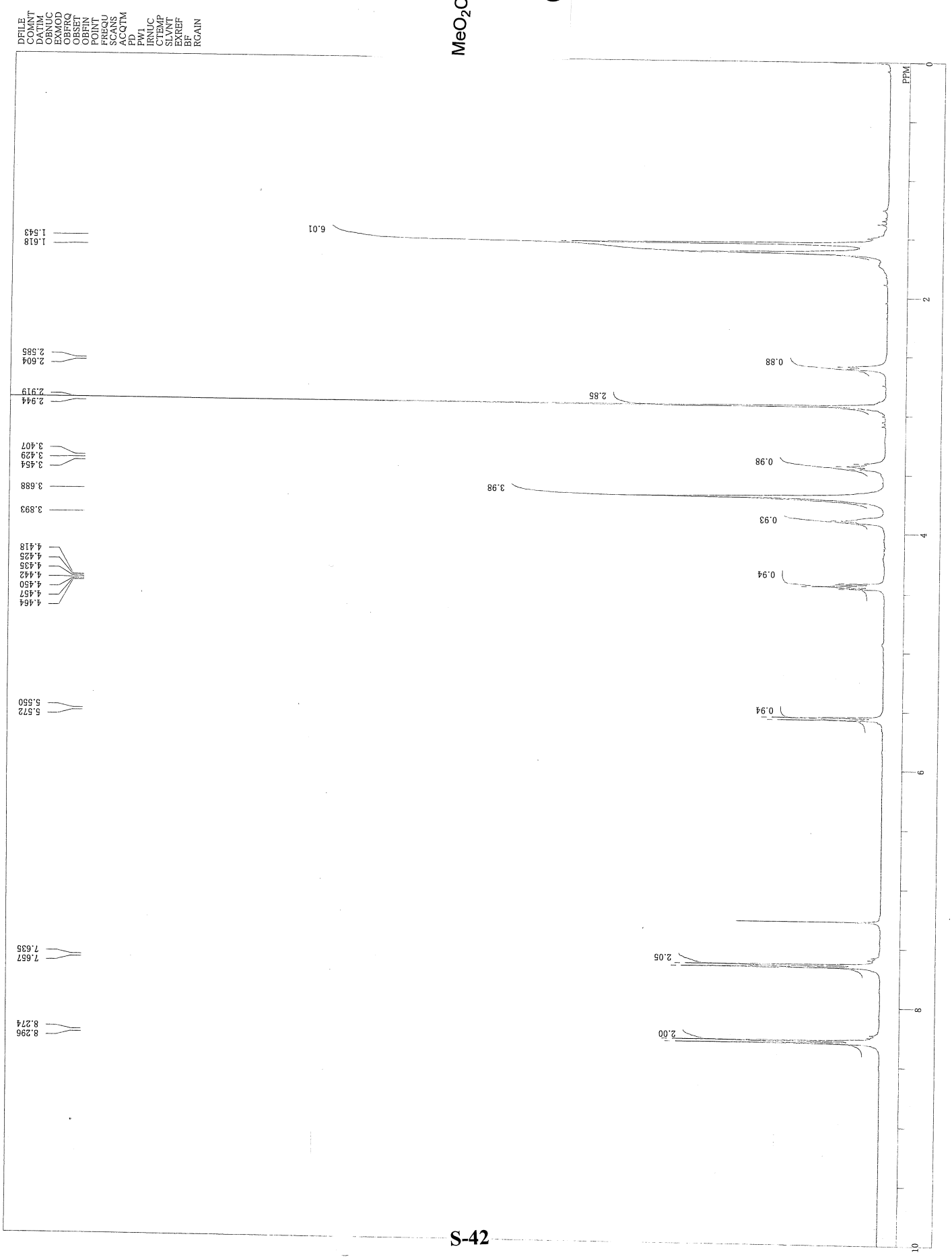
16

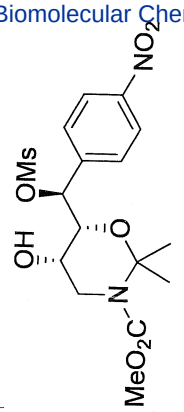
^{13}C NMR
(100 MHz, $CDCl_3$)



17

^{1}H NMR
(400 MHz, $CDCl_3$)





17

^{13}C NMR

(100 MHz, $CDCl_3$)