Supporting Information

Heteroaryl Cross Coupling as an Entry Towards the Synthesis of Lavendamycin Analogues: A Model Study

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Table of Contents

Preparation of 2-Tri-*n*-butylstannylpyridine (11). A sample of 2-bromopyridine (**9a**) (2.1 mL, 22 mmol) was dissolved in dry THF (10 mL) and the solution was cooled to -78 ^{o}C under an argon atmosphere. To this mixture was added 9.7 mL of a 2.5 M *n*-butyllithium solution in hexane (24.2 mmol) dropwise. After stirring for 1 h at -78 ^{o}C, tri-*n*-butyltin chloride (6.0 mL, 22 mmol) was added dropwise and the solution was allowed to stir overnight. After removal of the solvent under reduced pressure, the residue was taken up in ethyl acetate (50 mL) and water (20 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solution was concentrated under reduced pressure to give 7.14 g (88% yield) of 2-tri-*n*-butyl-stannylpyridine (**11**)[1]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.86 (t, 9H, J = 7.2 Hz), 1.10 (t, 6H, J = 8.4 Hz), 1.30 (m, 6H), 1.54 (m, 6H), 7.10 (m, 1H), 7.40 (d, 1H, J = 1.2 Hz), 7.44 (m, 1H) and 8.66 (d, 1H, J = 1.2 Hz).

Preparation of 2-Tri-*n*-butylstannyl-5-methylpyridine (12). A sample of 2-bromo-5-methylpyridine (**10a**) (1.72 g, 10 mmol) was taken up in dry THF (10 mL) and the solution was cooled to -78 ^{o}C under an argon atmosphere. To this mixture was added 4.5 mL of a 2.5 M *n*-butyllithium solution in hexane (11 mmol) dropwise. After stirring the mixture for 1 h at -78 ^{o}C, tri-*n*-butyltin chloride (3 mL, 11 mmol) was added dropwise and the solution was stirred overnight at 25 ^{o}C. The solvent was removed under reduced pressure and the residue was taken up in ethyl acetate (50 mL) and water (20 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give 3.17 g (83%) of 2-tri-*n*-butylstannyl-5-methylpyridine (**12**)[1]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.89 (m, 9H), 1.10 (m, 6H), 1.30 (m, 6H), 1.45 (m, 6H), 2.27 (s, 3 H), 7.29(t, 2H, J = 1.2 Hz) and 8.58 (s, 1H).

Preparation of [2,2']-Bipyridinyl (13). To a solution of 2-bromopyridine (0.16 g, 1 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannylpyridine (**11**) (0.37 g, 1 mmol) and dichlorobis(triphenylphosphine)palladium(II) (70 mg, 0.1 mmol) under an argon atmosphere and the mixture was heated at 110 ^{o}C for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined

organic layers were washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.12 g (78%) of [2,2']bipyridinyl (**13**); mp 70-72 ^oC (lit.[2] mp 72-73 ^oC); ^1H-NMR (400 MHz, $CDCl_3$) δ 7.30 (m, 2H), 7.80 (m, 2H), 8.38 (m, 2H) and 8.67 (m, 2H).

Preparation of 5-Methyl-[2,2']bipyridinyl (14). To a solution of 2-bromopyridine (**9a**) (0.16 g, 1 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannyl-5-methylpyridine (**12**) (0.38 g, 1 mmol) and dichlorobis(triphenylphosphine)palladium(II) (70 mg, 0.1 mmol) under an argon atmosphere. The mixture was heated at 110 ^oC for 18 h, cooled to rt, water was added and the mixture was extracted with ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.11 g (66%) of 5-methyl-[2,2']bipyridinyl (**14**)[3] as a pale yellow oil; ^1H-NMR (400 MHz, $CDCl_3$) δ 2.37 (s, 3H), 7.28 (m, 1H), 7.60 (m, 1H), 7.78 (m, 1H), 8.27 (q,1H, J = 8.4 Hz) , 8.36 (m, 1H), 8.48 (m, 1H), and 8.65 (m, 1H).

5-Methyl-[2,2']bipyridinyl (**14**) could also be prepared by coupling 2-bromo-5-methylpyridine (**10a**) with 2-tri-*n*-butylstannylpyridine (**11**). To a solution of **10a** (0.17 g, 1 mmol) in 1 mL of dry degassed DMF was added a sample of **11** (0.37 g, 1 mmol) and dichlorobis(triphenylphosphine)palladium(II) (70 mg, 0.1 mmol) under an argon atmosphere. The mixture was heated at 110 ^oC for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.1 g (59%) of 5-methyl-[2,2']bipyridinyl (**14**).

Preparation of 5,5'-Dimethyl-[2,2']bipyridinyl (15). To a solution of 2-bromo-5-methylpyridine (**10a**) (0.17 g, 1 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannyl-5-methylpyridine (**12**) (0.38 g, 1 mmol) and dichlorobis(triphenyl-phosphine)palladium(II) (70 mg, 0.1 mmol) under an argon atmosphere. The mixture

was heated at 110 ^{o}C for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and the solution was concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.11 g (61%) of the titled compound **15** as a white solid, mp 113-114 ^{o}C (lit.[4] mp 114-115 ^{o}C); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.37 (s, 6H) , 7.61 (dd, 2H, J = 8.4 and 2.0 Hz), 8.24 (d, 2H, J = 8.0 Hz) and 8.48 (d, 2H, J = 1.6 Hz).

Preparation of 2-Bromo-8-acetoxyquinoline (17). A solution of 1.0 g (6.2 mmol) 2,8-dihydroxyquinoline (**16**)[5] in 5 mL of acetic anhydride was heated to reflux overnight and then the solution was concentrated under reduced pressure to afford 8-acetoxy-2-hydroxyquinoline[5] in 99% yield which was used directly in the next step without purification. To a suspension of 1.16 g (5.7 mmol) of 8-acetoxy-2-hydroxyquinoline in 5 mL of dry $CHCl_3$ was added 4.0 g (14.0 mmol) of $POBr_3$ and the reaction mixture was heated at reflux for 5 h under a nitrogen atmosphere. After pouring the reaction mixture over ice followed by extraction with $CHCl_3$, the organic extracts were dried over $MgSO_4/K_2CO_3$ (10:1), filtered and concentrated under reduced pressure to give 1.02 g (67%) of 2-bromo-8-acetoxyquinoline (**17**) whose spectral properties were identical to those recorded in the literature.[6]

Preparation of 2-(Pyridin-2-yl)quinoline (22). To a solution of 2-bromoquinoline (**20a**) (0.1 g, 0.5 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannyl-pyridine (**11**) (0.18 g, 0.5 mmol) and dichlorobis(triphenylphosphine)palladium(II) (35 mg, 0.0 5mmol) under an argon atmosphere. The mixture was heated at 110 ^{o}C for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and the solution was concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.07 g (71%) of the titled compound **22** as a white solid, mp 96-97 ^{o}C (lit.[7] mp 97-98 ^{o}C); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 1H), 7.55 （m, 1H), 7.73 (m,1H), 7.86 (m,

2H), 8.18 (d, 1H, J = 8.4 Hz), 8.29 (d, 1H, J = 8.8 Hz), 8.57 (d, 1H, J = 8.8Hz), 8.66 (d, 1H, J = 8.0 Hz) and 8.74 (m, 1H).

Preparation of 2-(5-Methylpyridin-2-yl)quinoline (23). To a solution of 2-bromoquinoline (**20a**) (0.1 g, 0.5 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannyl-5-methylpyridine (**12**) (0.19 g, 0.5 mmol) and dichlorobis(triphenylphosphine)palladium(II) (35 mg, 0.05 mmol) under an argon atmosphere. The mixture was heated at 110 ^{o}C for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.065 g (59%) of the titled compound **23** as a white solid, mp 112-113 ^{o}C (lit.[8] mp 112-114 ^{o}C); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H), 7.53 (m, 1H), 7.70 (m, 2H), 7.84 (d, 1H, J = 8.0 Hz), 8.17 (d, 1H, J = 8.8 Hz), 8.27 (d, 1H, J = 8.4 Hz) and 8.53 (m, 3H).

Preparation of 1-(Pyridin-2-yl)isoquinoline (24). To a solution of 1-bromoisoquinoline (**21a**) (0.1 g, 0.5 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannylpyridine (**11**) (0.18 g, 0.5 mmol) and dichlorobis(triphenylphosphine)palladium(II) (35 mg, 0.05 mmol) under an argon atmosphere. The mixture was heated at 110 ^{o}C for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.043 g (41%) of the titled compound **24** as a white solid; mp 71-72 ^{o}C (lit.[9] mp 71-72 ^{o}C); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.40 (m, 1H), 7.60 (m, 1H), 7.69 (m, 2H), 7.90 (m, 2H), 8.00 (dt, 1H, J = 8.0 and 1.2 Hz), 8.60 (dd, 1H, J = 8.4 and 1.0 Hz), 8.63 (d, 1H, J = 5.6 Hz) and 8.79 (m, 1H).

Preparation of 2-Tri-*n*-butylstannylquinoline (37). A sample of 2-bromoquinoline (**20a**) (1.04 g, 5 mmol) was taken up in dry THF (10 mL) and the solution was cooled to -78 ^{o}C under an argon atmosphere. To this mixture was added 2.2 mL of a 2.5 M *n*-butyllithium solution in hexane (5.5 mmol) dropwise. After stirring for 1 h at -78 ^{o}C, tri-*n*-butyltin chloride (1.76 mL, 6.5 mmol) was added dropwise to the above solution

which was then allowed to stir overnight. After removal of the solvent under reduced pressure, the residue was taken up in ethyl acetate (50 mL) and water (20 mL). The combined organic layer was washed with brine and dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give 1.64 g (78%) of 2-tri-*n*-butylstannyl-quinoline (**37**) which was used directly in the Stille coupling step without further purification.

Preparation of 2-(Isoquinolin-1-yl)quinoline (38). To a solution of 1-bromoiso-quinoline (**21a**) (0.1 g, 0.5 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butyl stannylquinoline (**37**) (0.21 g, 0.5 mmol) and dichlorobis(triphenylphosphine)-palladium(II) (35 mg, 0.05 mmol) under an argon atmosphere. The mixture was heated at 110 ^oC for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.062 g (49%) of the titled compound **38** as a light yellow solid, mp 128-129 ^oC (lit.[10] mp 128-129 ^oC); ^1H-NMR (400 MHz, $CDCl_3$) δ 7.61 (m, 2H), 7.75 (m, 3H), 7.93 (t, 2H, J = 6.8 Hz), 8.15 (d, 1H, J = 5.2 Hz), 8.25 (d, 1H, J = 8.4 Hz), 8.37 (d, 1H, J = 8.8 Hz), 8.69 (d, 1H, J = 5.6 Hz) and 8.82 (d, 1H, J = 8.4 Hz).

Preparation of [2,2']Biquinolinyl (39). To a solution of 2-bromoquinoline (**20a**) (0.1 g, 0.5 mmol) in 1 mL of dry degassed DMF was added 2-tri-*n*-butylstannylquinoline (**37**) (0.21 g, 0.5 mmol) and dichlorobis(triphenylphosphine)palladium(II) (35 mg, 0.05 mmol) under an argon atmosphere. The mixture was heated at 110 ^oC for 18 h, cooled to rt, water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with water, brine and dried over Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. The crude residue was subjected to flash silica gel chromatography to give 0.087 g (68%) of the titled compound **39** as a light yellow solid, mp 193-195 ^oC (lit.[11] mp 193-196 ^oC); ^1H-NMR (400 MHz, $CDCl_3$) δ 7.57 (m, 2H), 7.72 (m, 2H), 7.89 (d, 2H, J = 8.0 Hz), 8.24 (d, 2H, J = 8.4 Hz), 8.33 (d, 2H, J = 8.4 Hz) and 8.86 (d, 2H, J = 8.8 Hz).

References

1. (a) Zhang, H.; Tse, M. K.; Chan, K. S. *Synth. Commun.* **2001**, *31*, 1129. (b) Nicolaou, K. C.; Scarpelli, R.; Bollbuck, B.; Werschkun, B.; Pereira, M. M. A.; Wartmann, M.; Altmann, K-H.; Zaharevitz, D.; Gussio, R.; Giannakakou, P. *Chem. Biol.* **2000**, *7*, 593. (c) Bedel, S.; Ulrich, G.; Picard, C.; Tisnes, P. *Synthesis* **2002**, *11*, 1564.
2. Schwab, P. F. H.; Fleischer, F.; Michl, J. *J. Org. Chem.* **2002**, *67*, 443.
3. (a) Schubert, U. S.; Eschbaumer, C.; Heller, M. *Org. Lett.* **2000**, *2*, 3373 and references cited therein. (b) Potts, K. T.; Winslow, P. A. *J. Org. Chem.* **1985**, *50*, 5405.
4. Schubert, U. S.; Eschbaumer, C.; Hochswimmer, G. *Synthesis* **1999**, *5*, 779.
5. Sigouin, O.; Beauchamp, A. L.; *Can. J. Chem.* **2005**, *83*, 460.
6. Petitjean, A.; Kyritsakas, N.; Lehn, J.-M. *Chem. Eur. J.* **2005**, *11*, 6818.
7. Martinez, R.; Ramon, D. J.; Yus, M. *Tetrahedron* **2006**, *62*, 8988.
8. Haginiwa, J.; Higuchi, Y.; Kawashima, T.; Goto, T. *Yakugaku Zasshi* **1975**, *95*, 204.
9. Knapp, D. M.; Gillis, E. P.; Burke, M. D. *J. Am. Chem. Soc.* **2009**, *131*, 6961.
10. Hoste, J. *Anal. Chim. Acta* **1950**, *4*, 23.
11. Dumouchel, S.; Mongin, F.; Trécourt, F.; Quéguiner, G. *Tetrahedron* **2003**, *59*, 8629.

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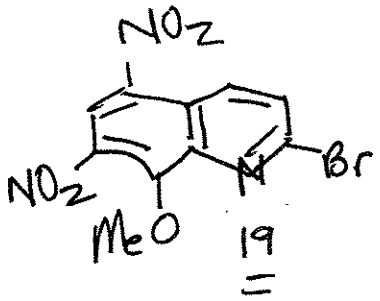

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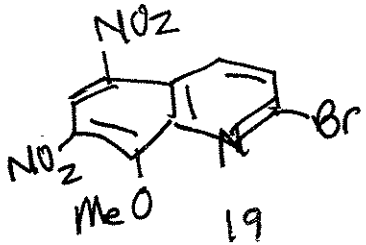

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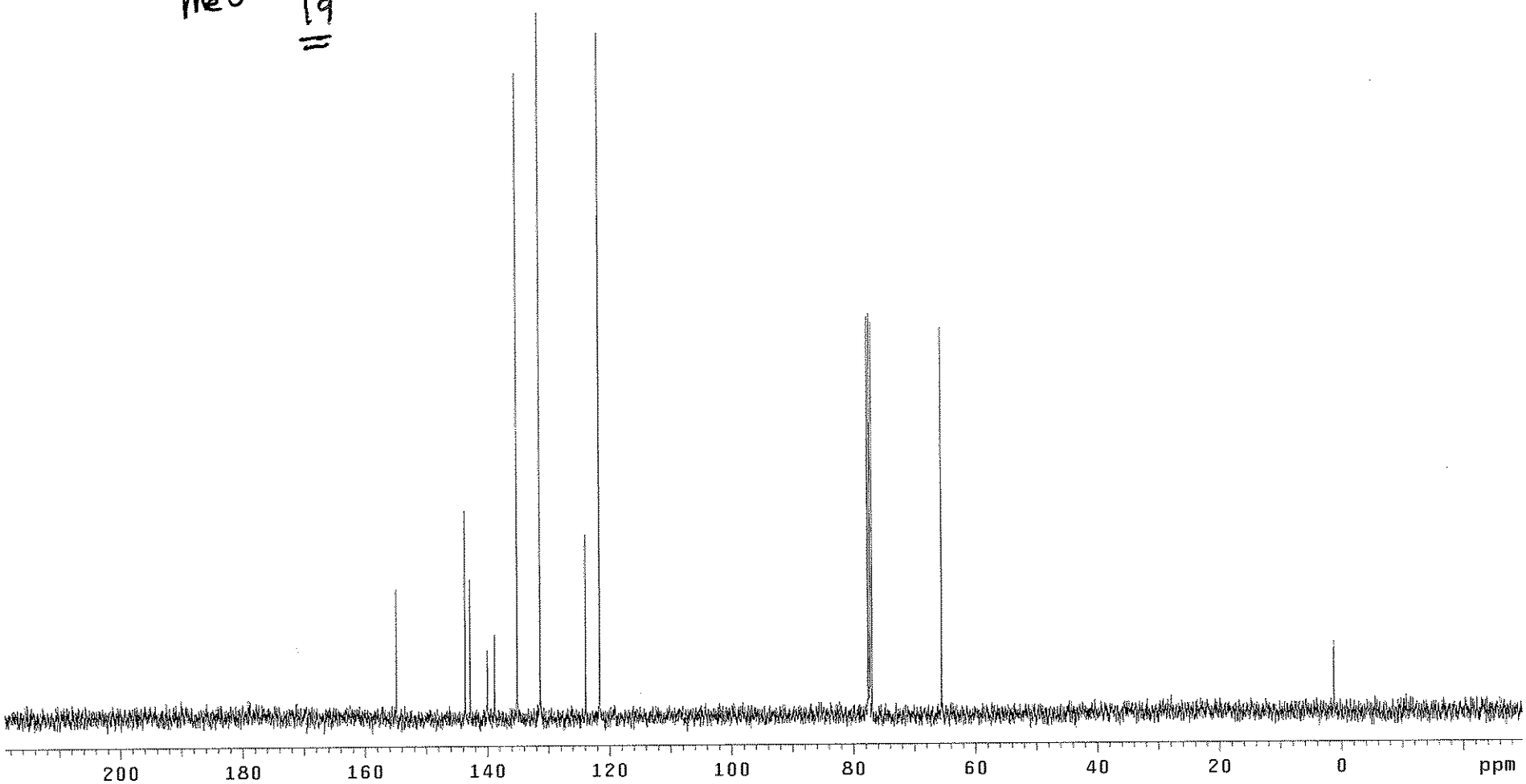

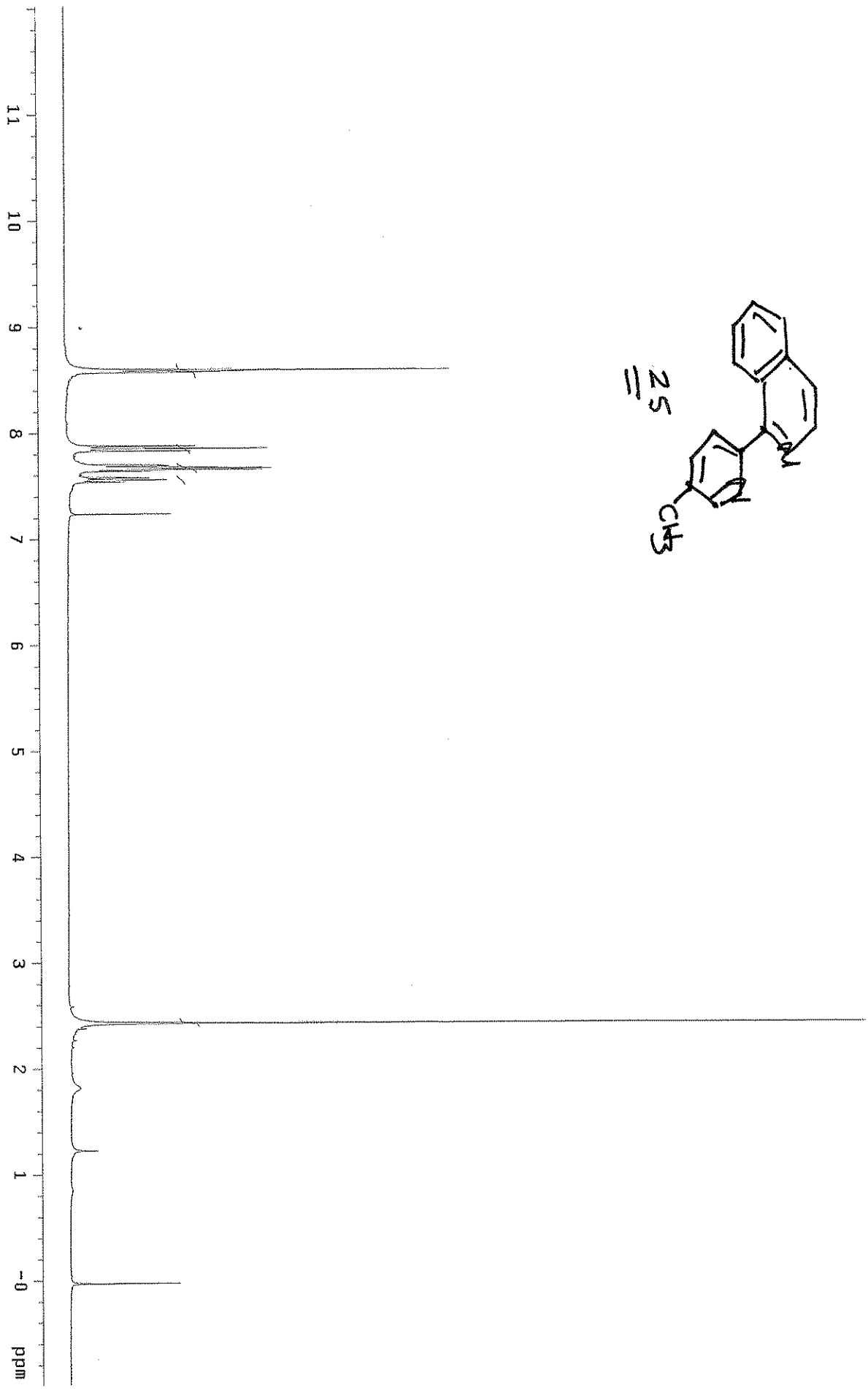
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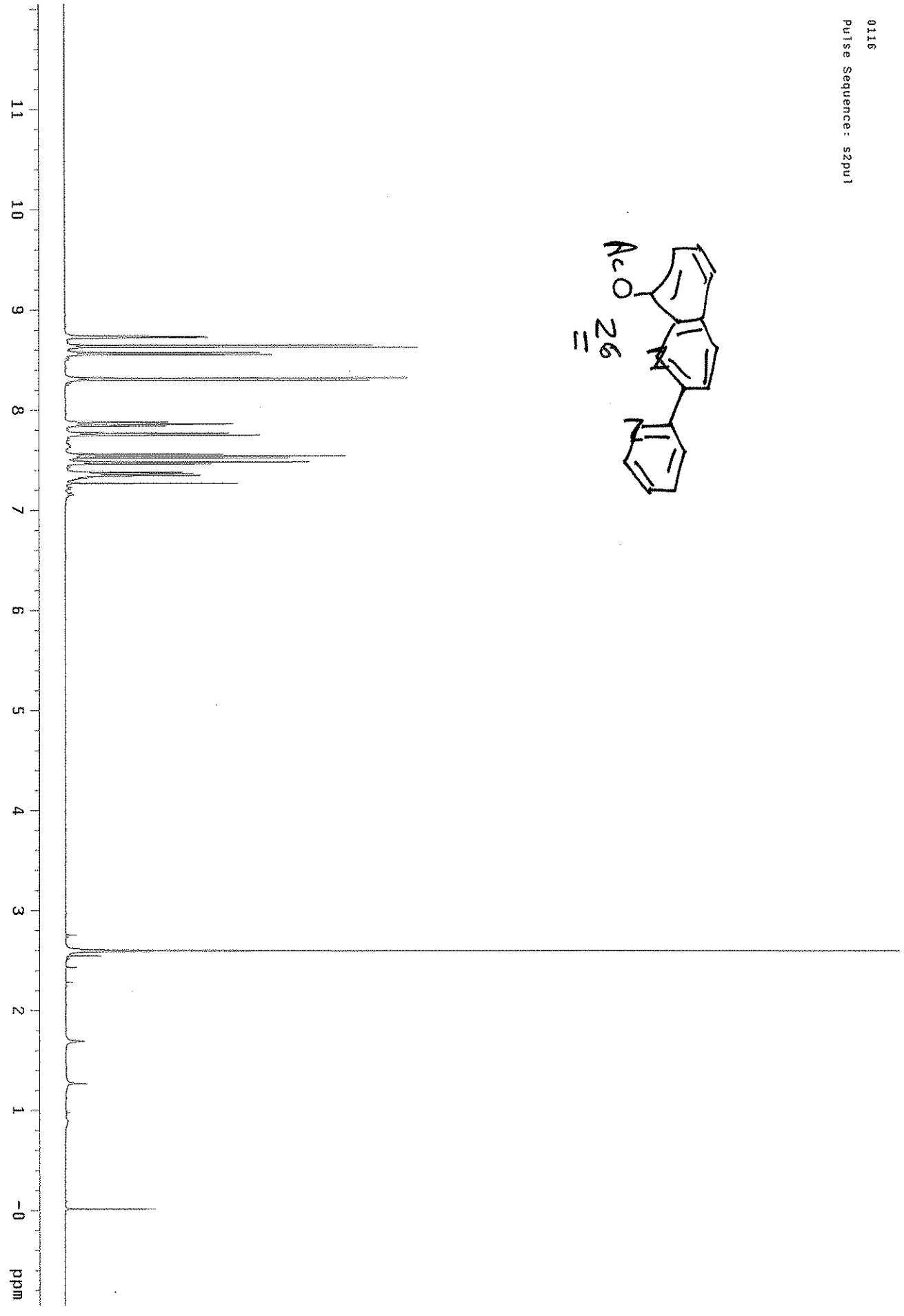
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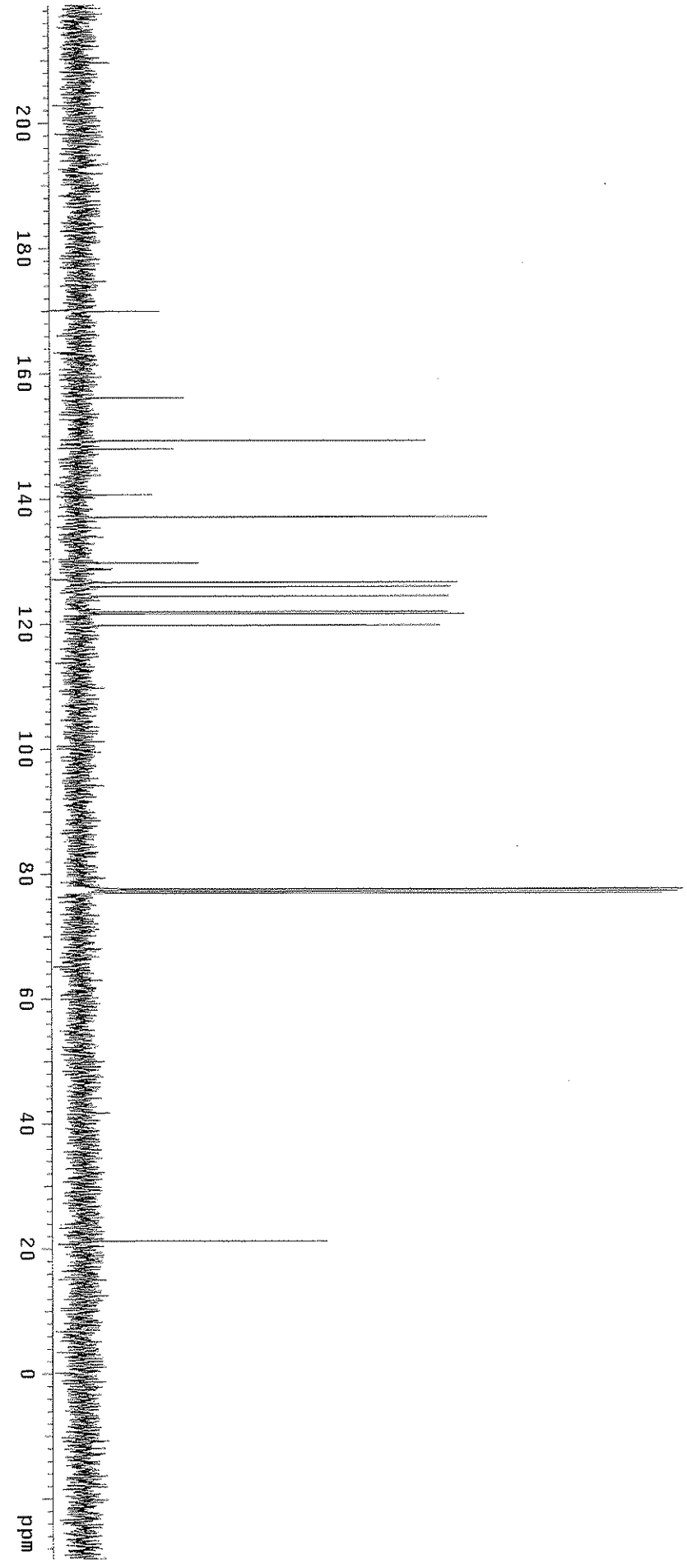

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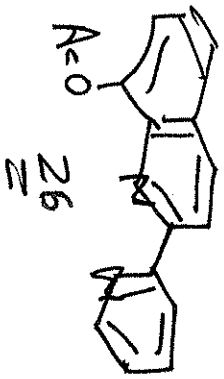

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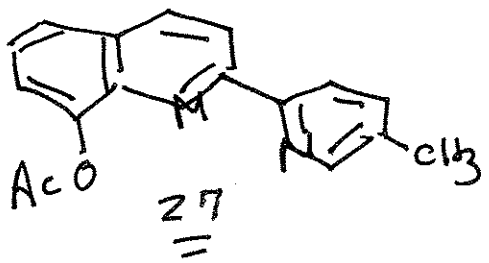

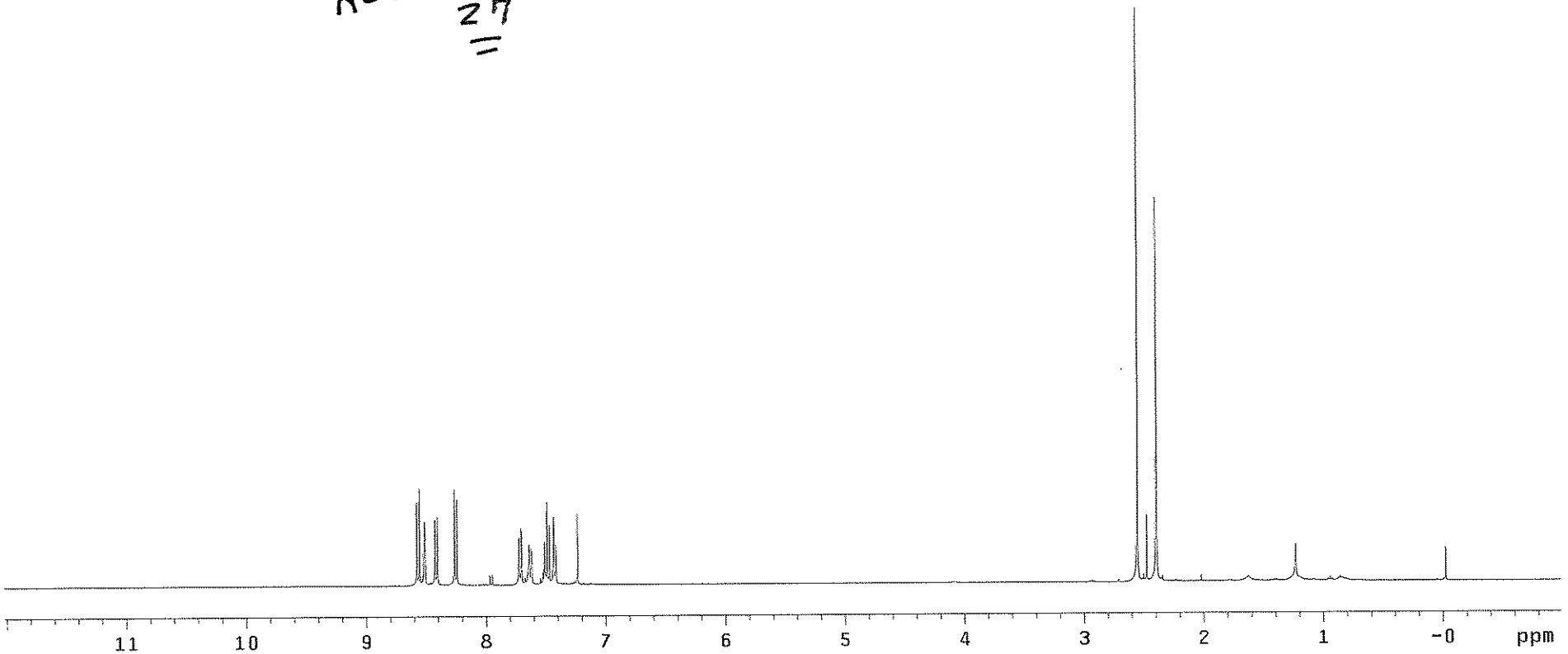

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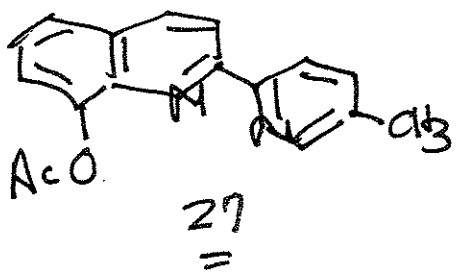

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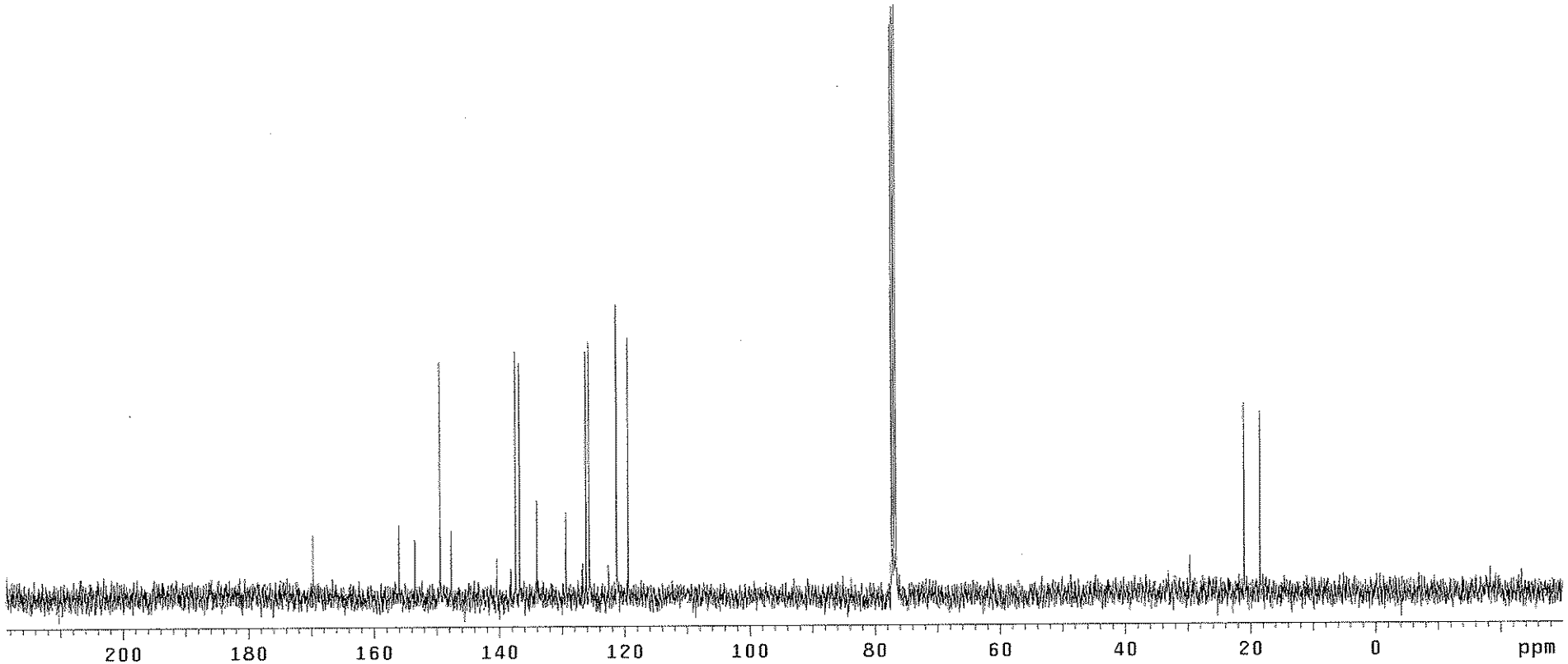

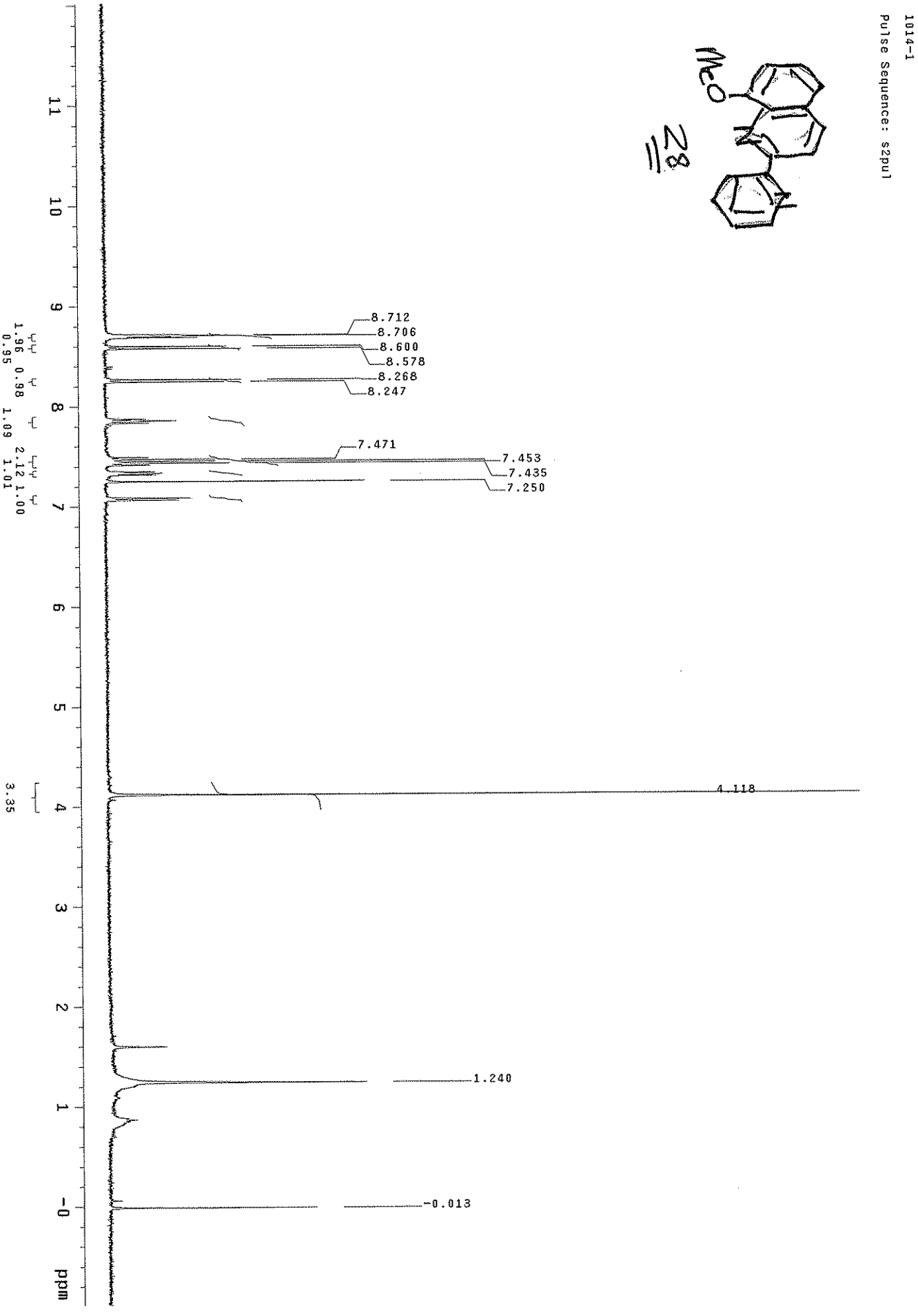
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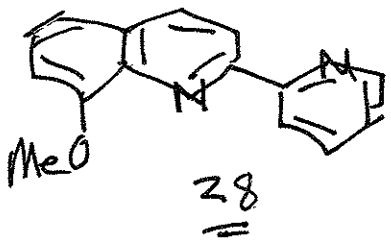

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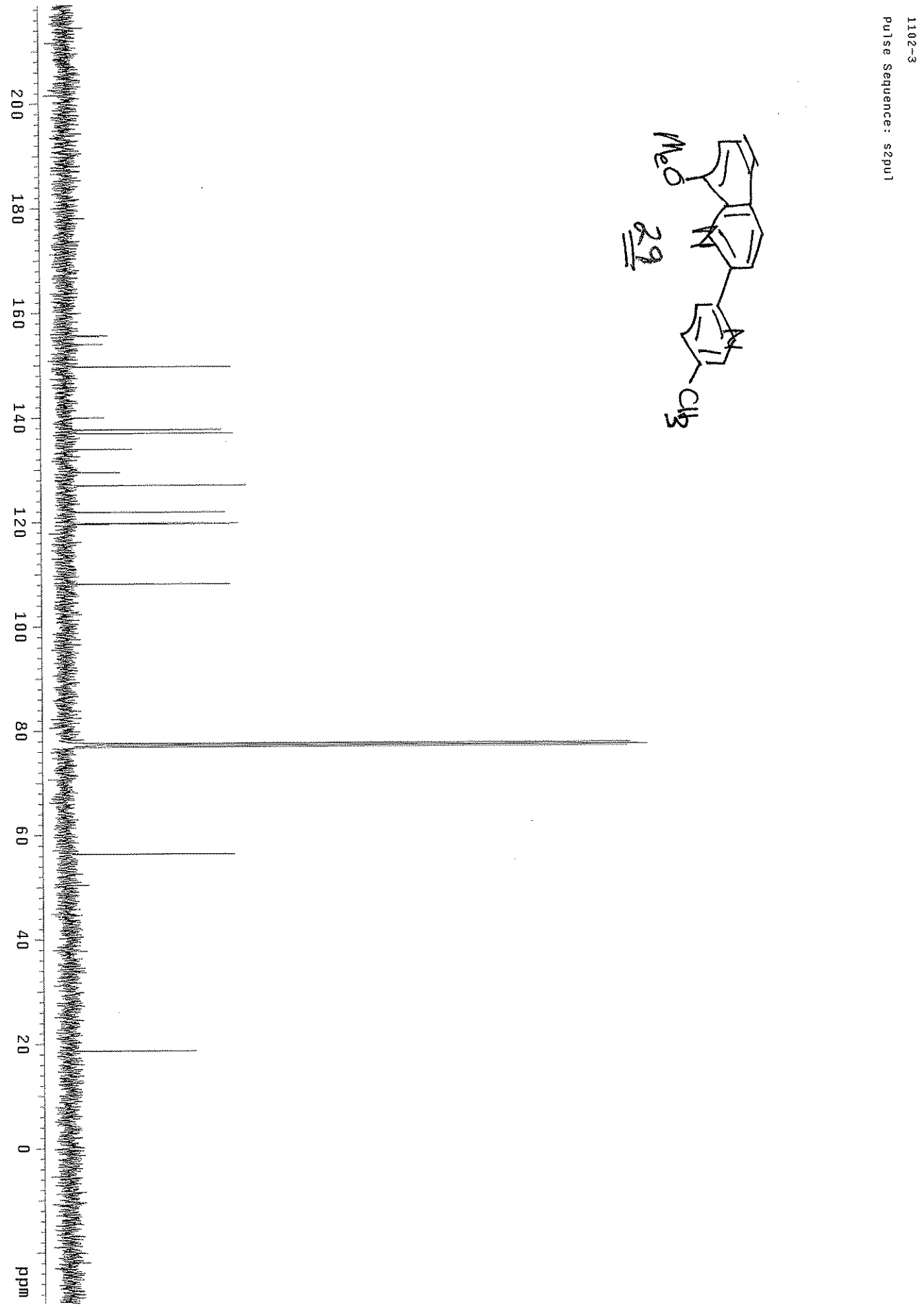
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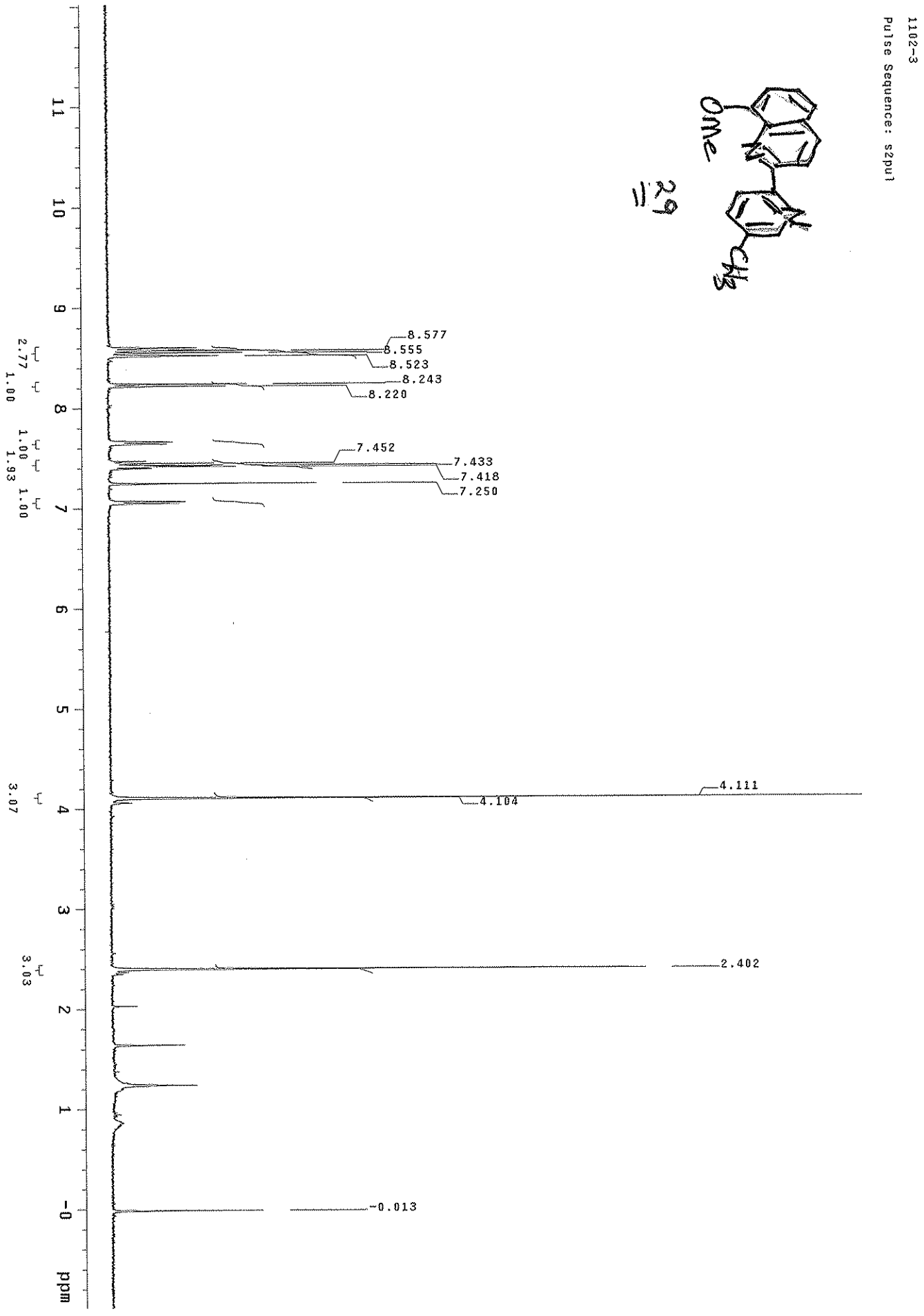
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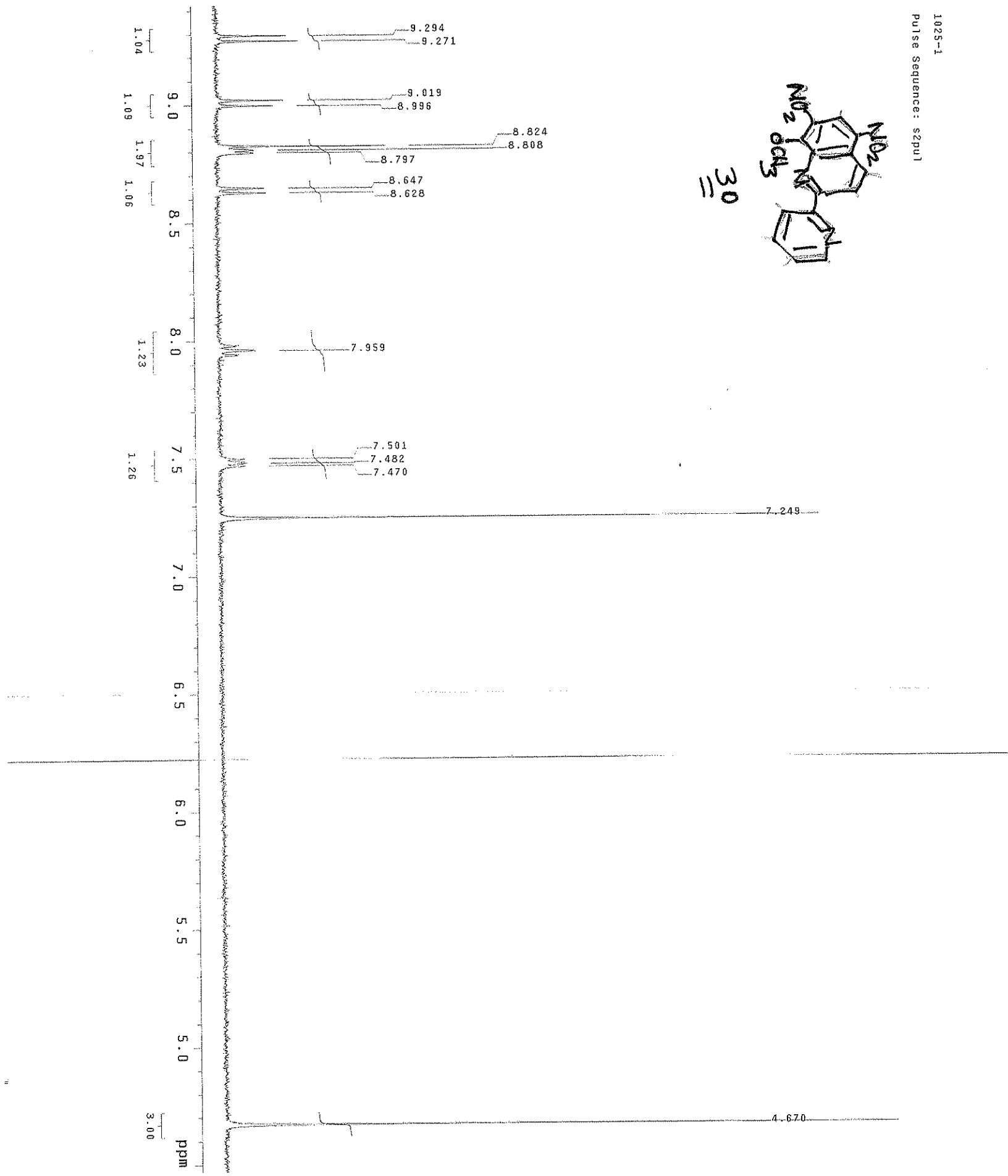
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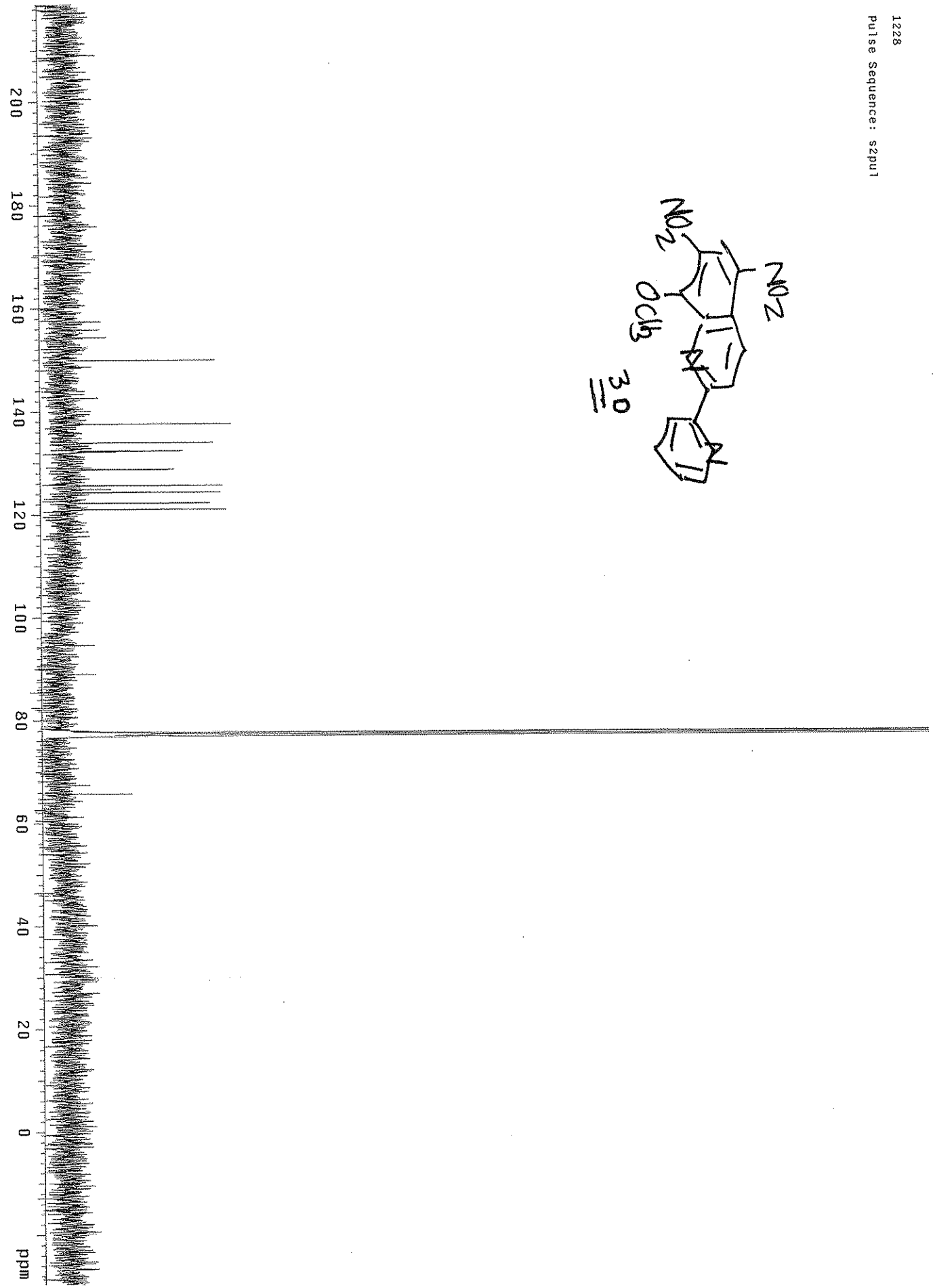
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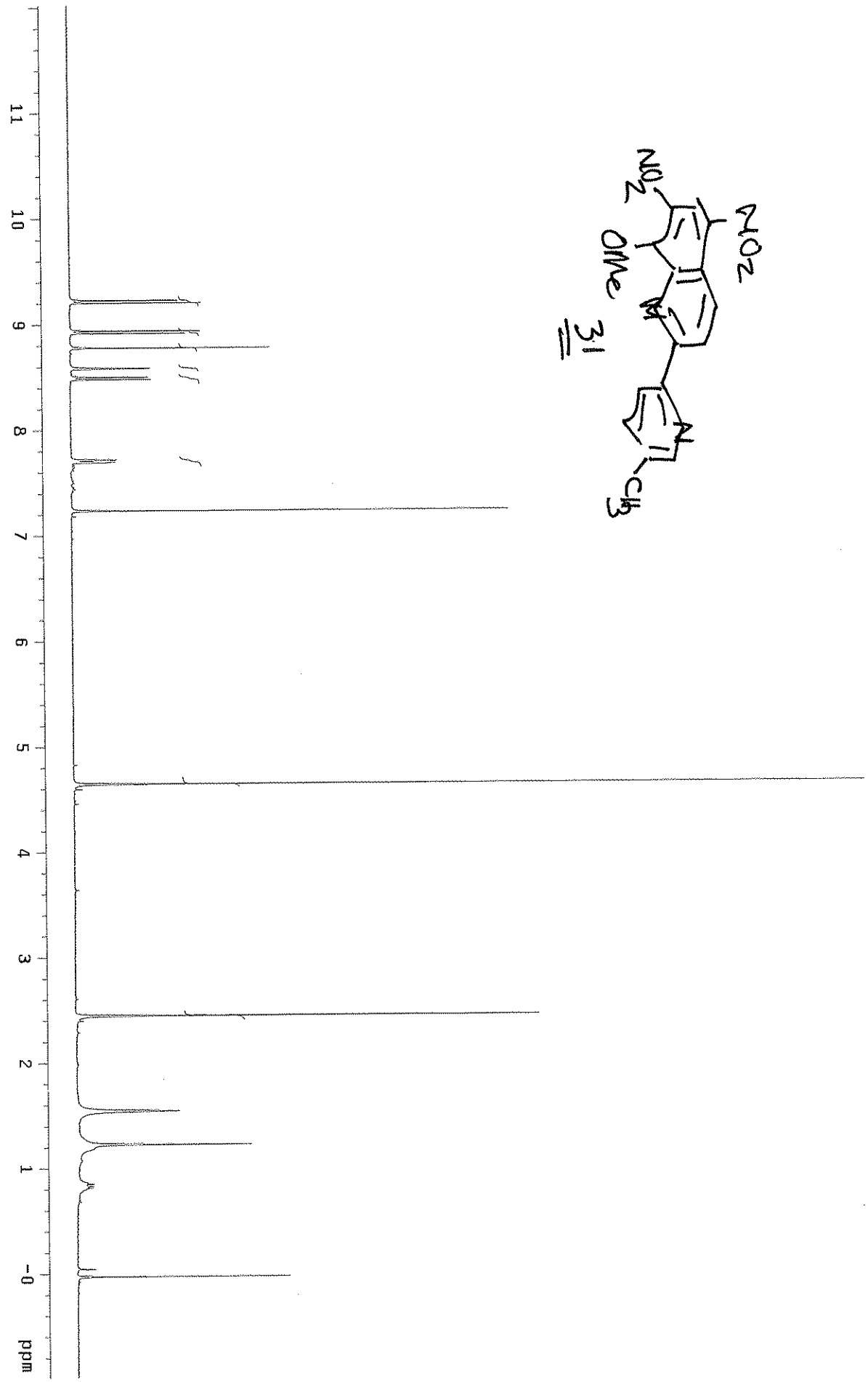
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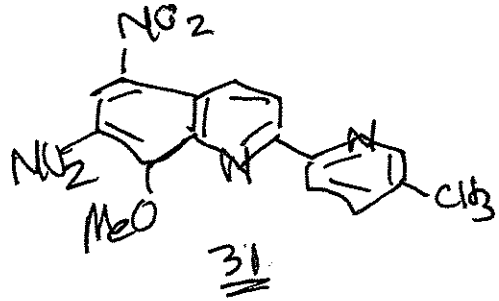
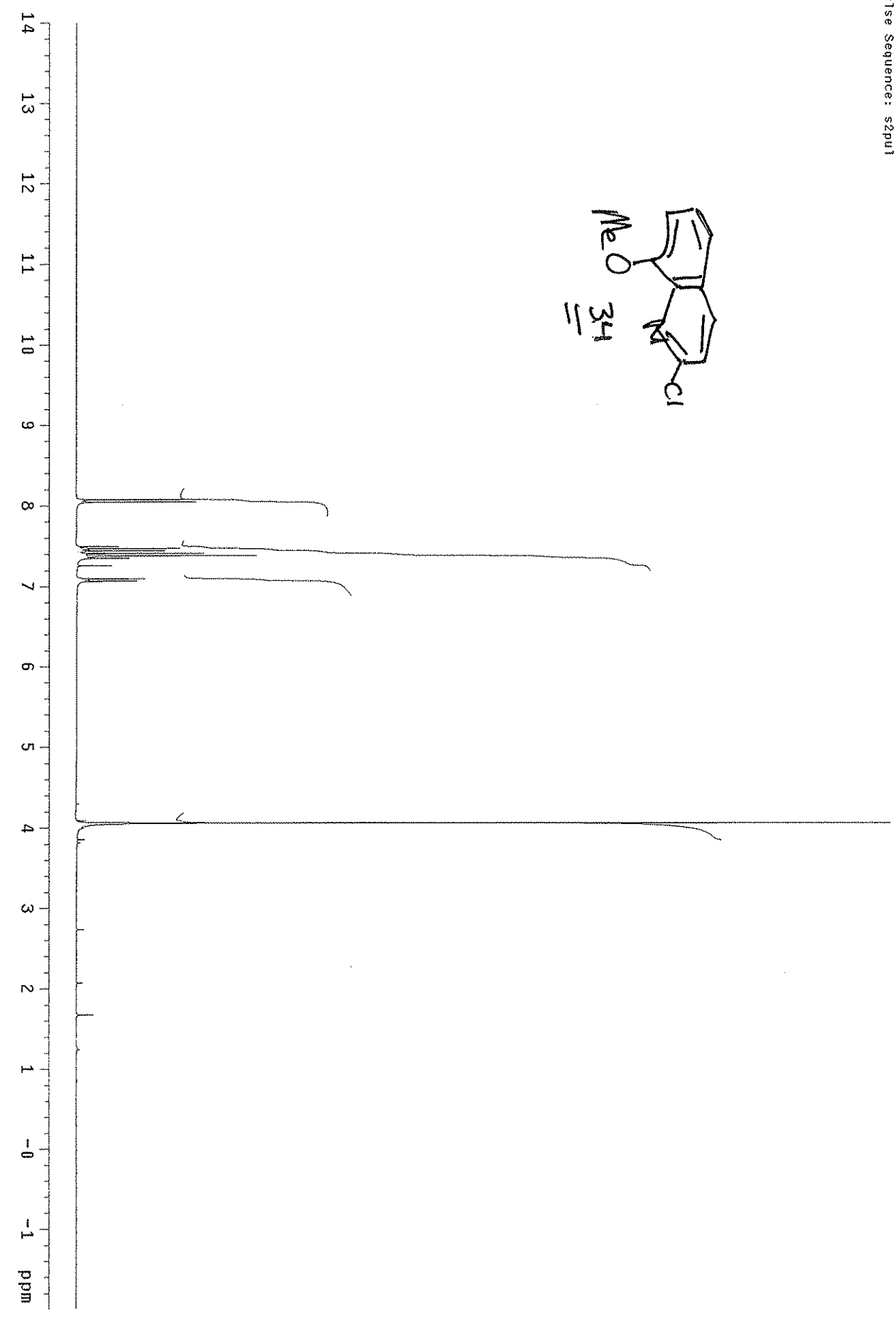
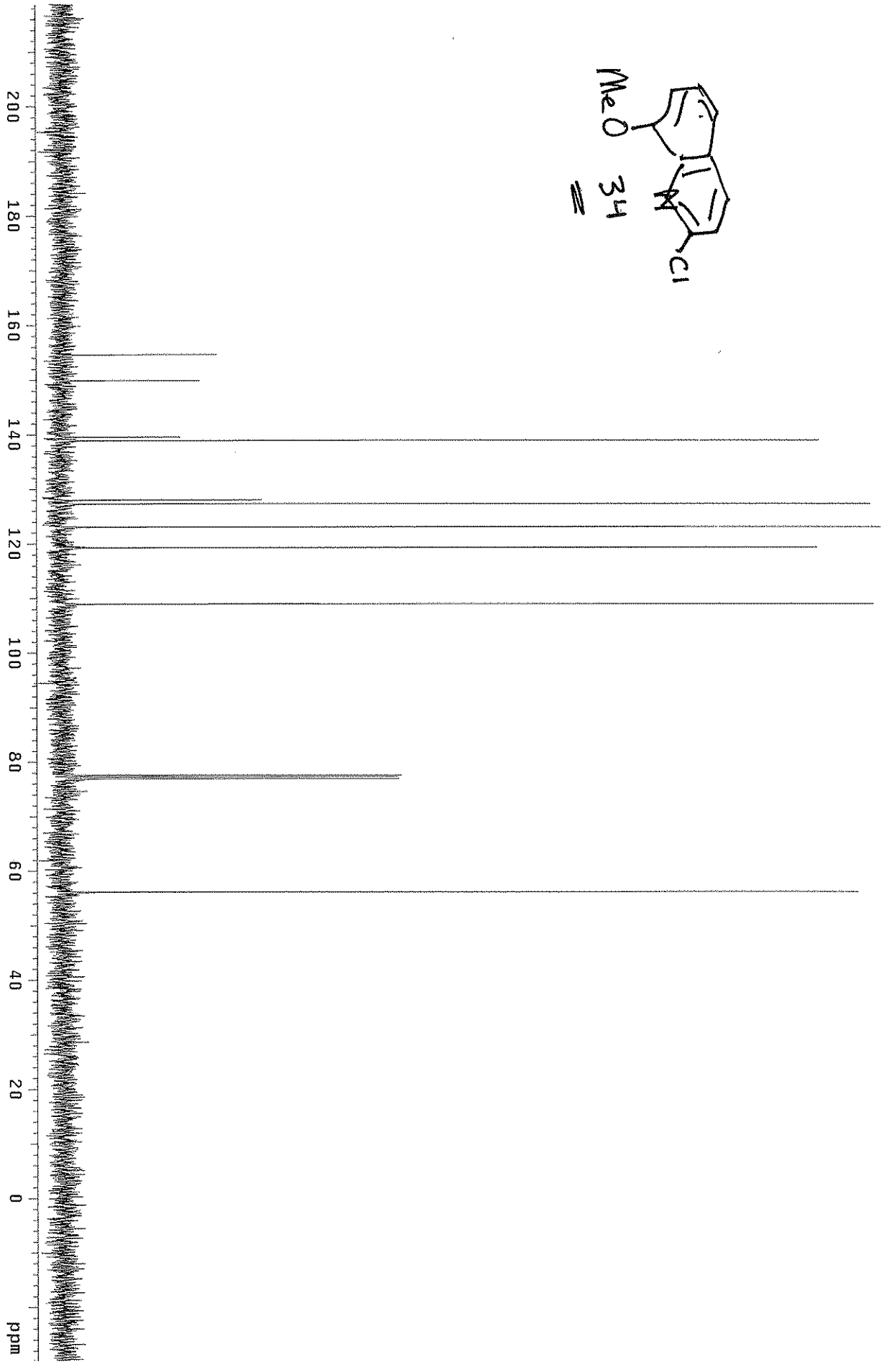
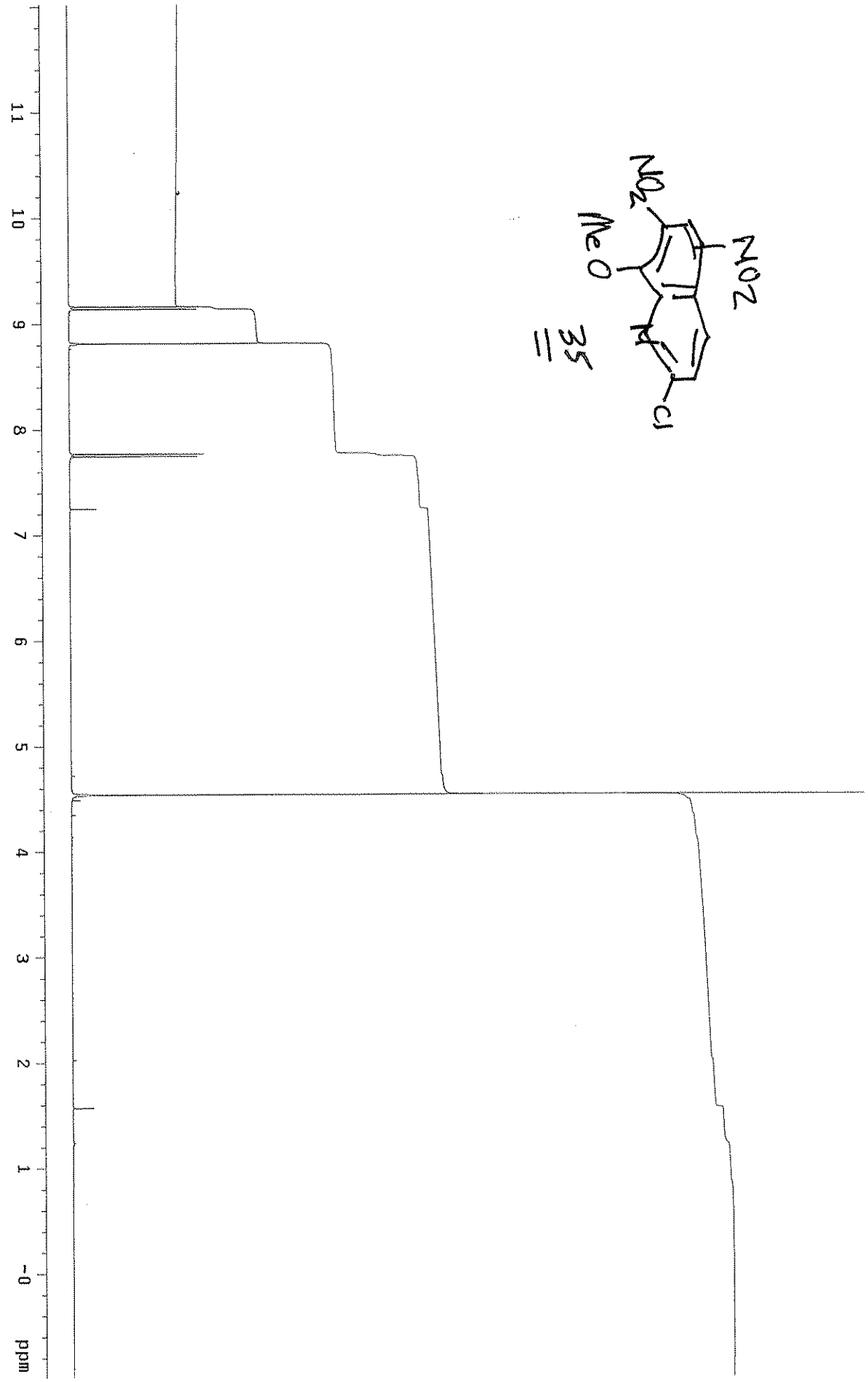


31

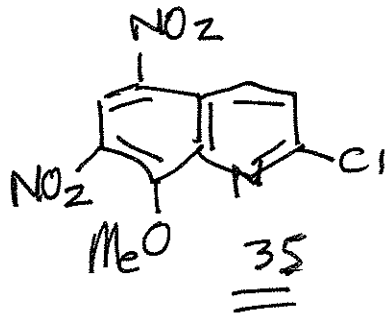

2-111
Automation directory:
Sample id : tmpstudy
Pulse Sequence: s2pul
MeO
34
Cl
14
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


2-111
Pulse Sequence: s2pul
MeO
34
Cl
200
180
160
140
120
100
80
60
40
20
0
ppm

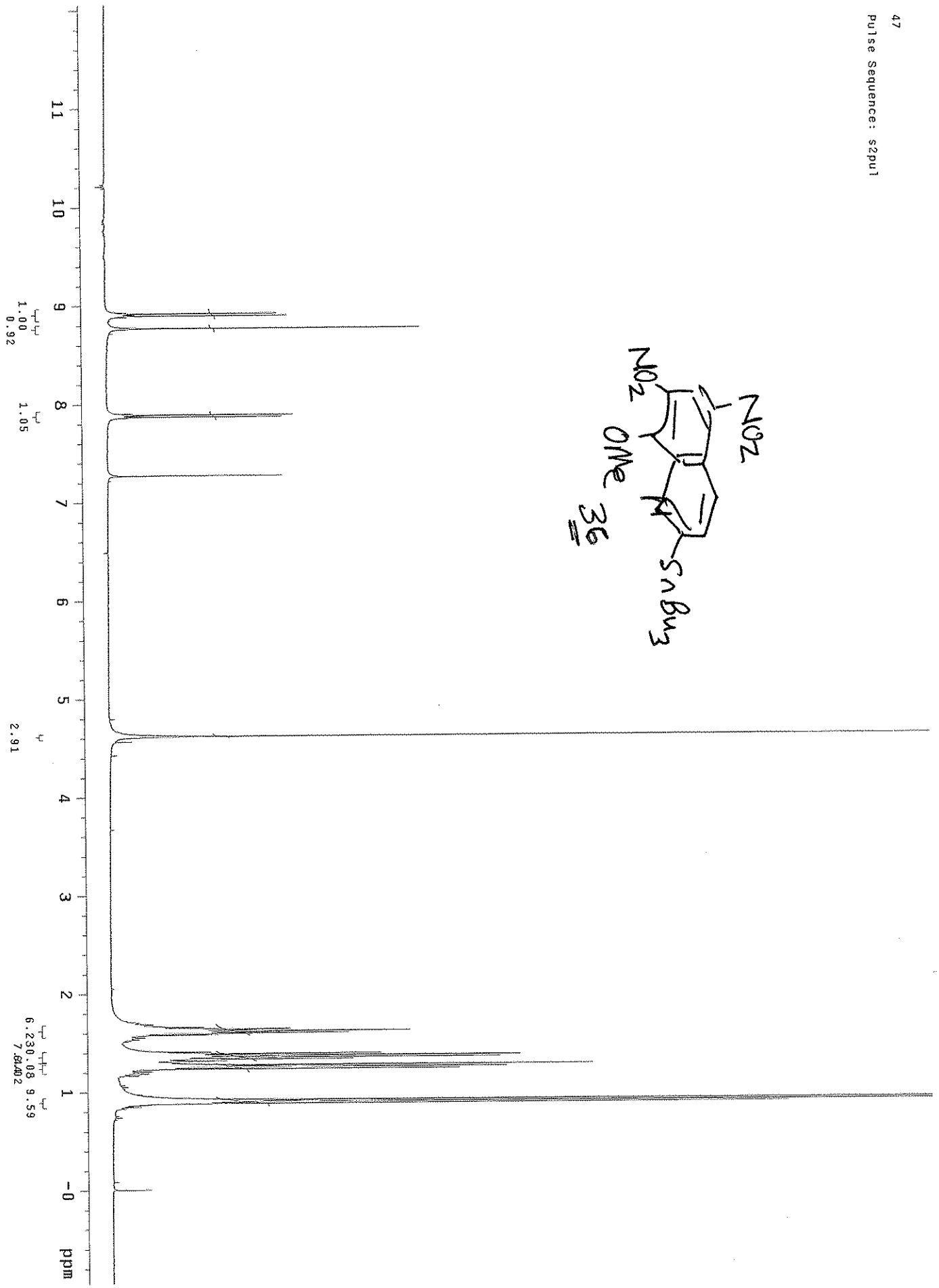

2-113fc
Pulse Sequence: s2pul
NO2
MeO
NO2
N
Cl
35
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

13C OBSERVE
Pulse Sequence: s2pul

INDEX	FREQUENCY	PPM	HEIGHT
1	19564.304	194.544	8.9
2	19459.922	193.414	-9.1
3	16768.046	166.739	9.0
4	15574.009	154.866	24.4
5	15351.224	152.650	22.9
6	14334.195	142.537	18.5
7	14101.491	140.223	11.4
8	13959.580	138.812	13.2
9	13665.076	135.883	103.5
10	12867.780	127.955	128.5
11	12435.180	123.654	31.2
12	12217.736	121.491	124.8
13	7874.952	78.307	-9.3
14	7798.656	77.549	150.0
15	7766.611	77.230	146.3
16	7735.330	76.919	138.0
17	6592.412	65.554	76.2
18	5306.820	52.770	-9.8
19	2010.058	19.988	9.3
20	1284.480	12.773	8.7
21	-1546.112	-15.374	9.5
22	-1733.038	-17.233	9.1
23	-1823.830	-18.136	9.0
24	-2193.104	-21.808	-9.3

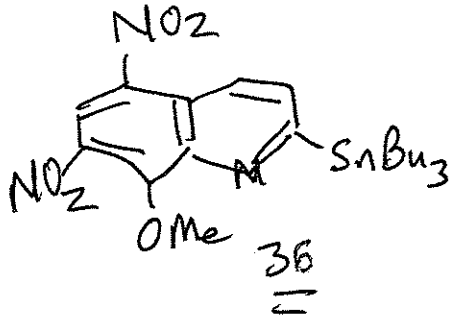


35

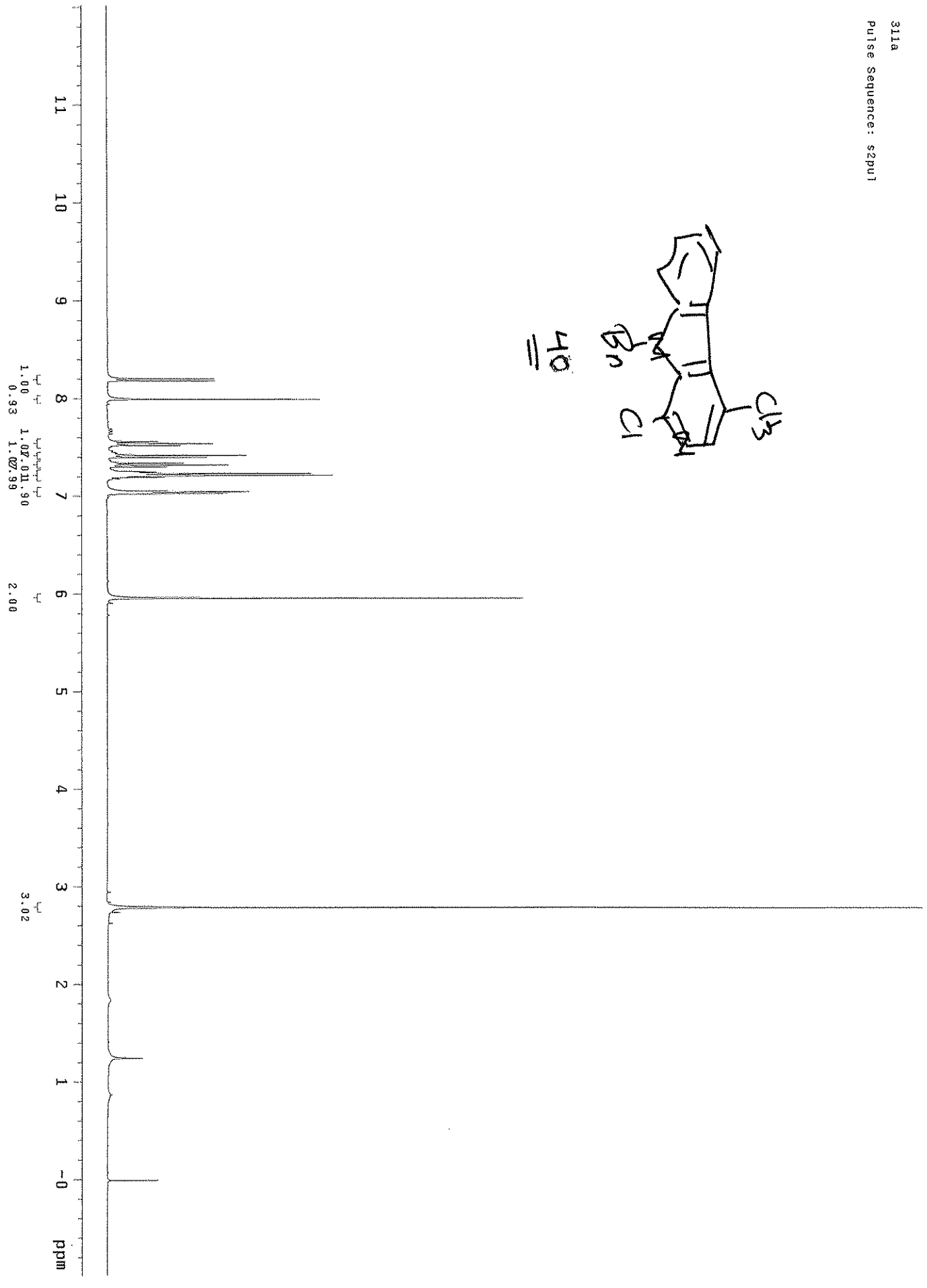

47
Pulse Sequence: s2pul
NO2
OMe
NO2
SnBu3
36
ppm
11
10
9
8
7
6
5
4
3
2
1
0
1.00
0.92
1.05
2.91
6.23
0.08
9.59
7.84
0.02

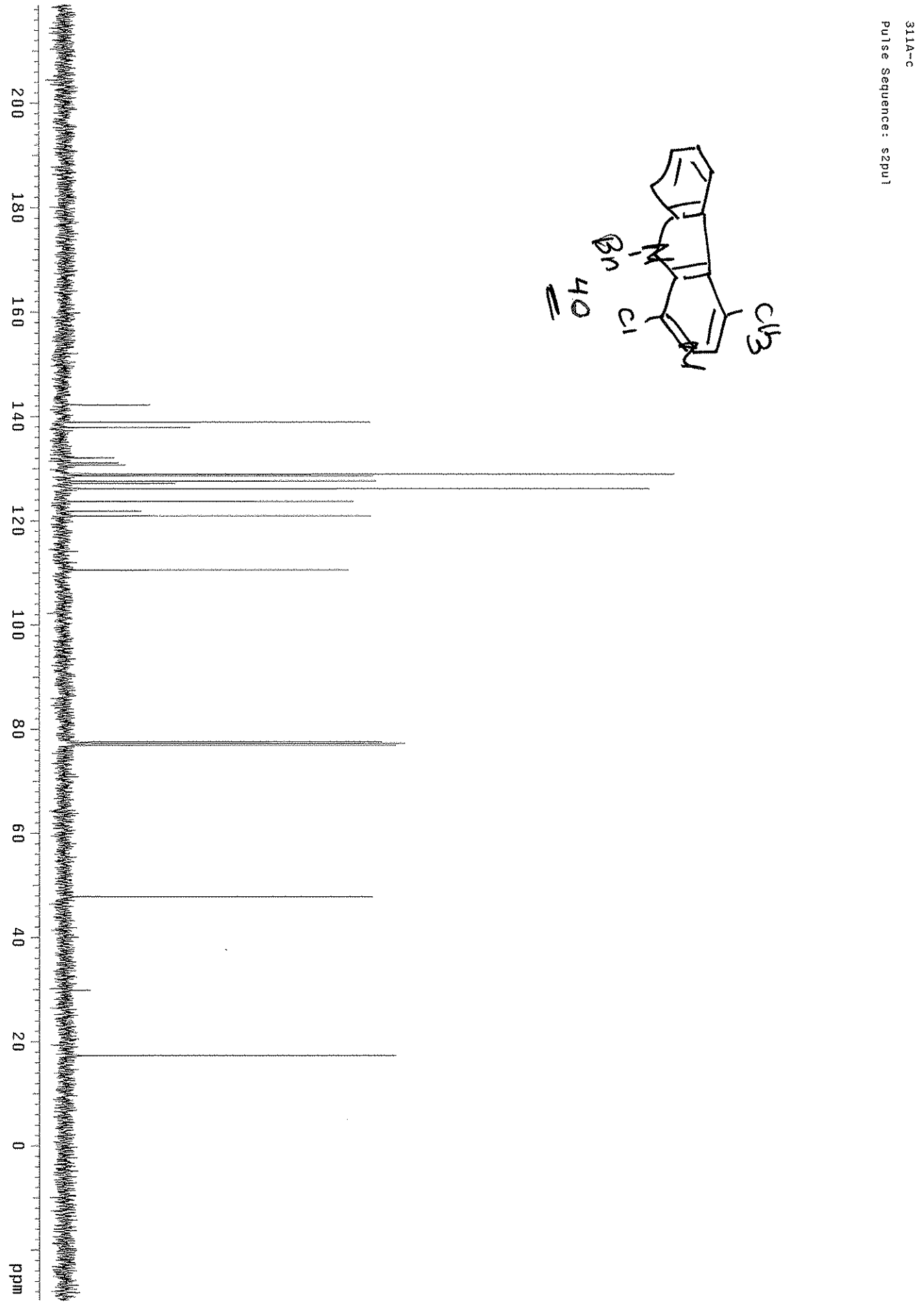
47-c13

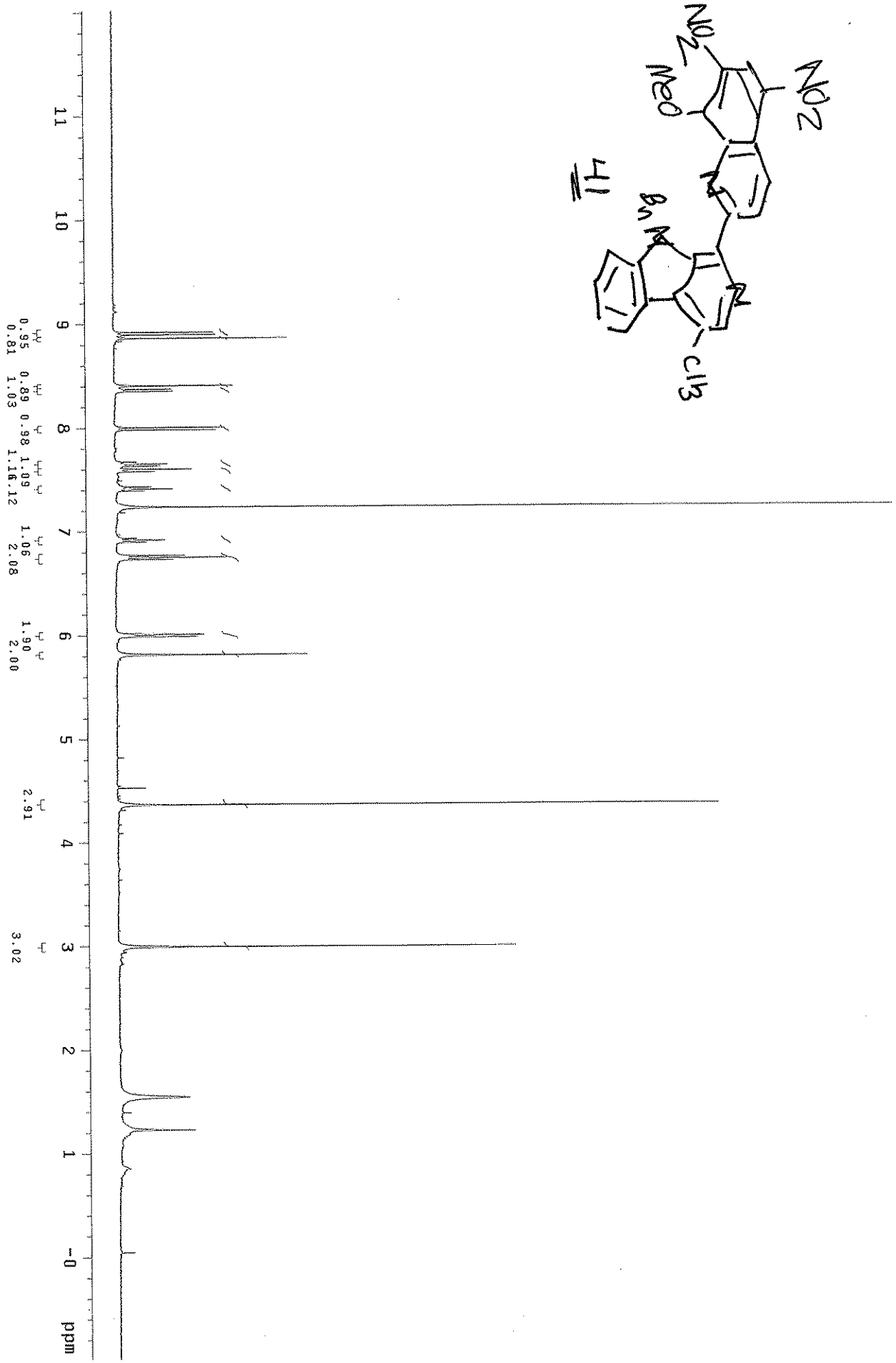
Pulse Sequence: s2pul

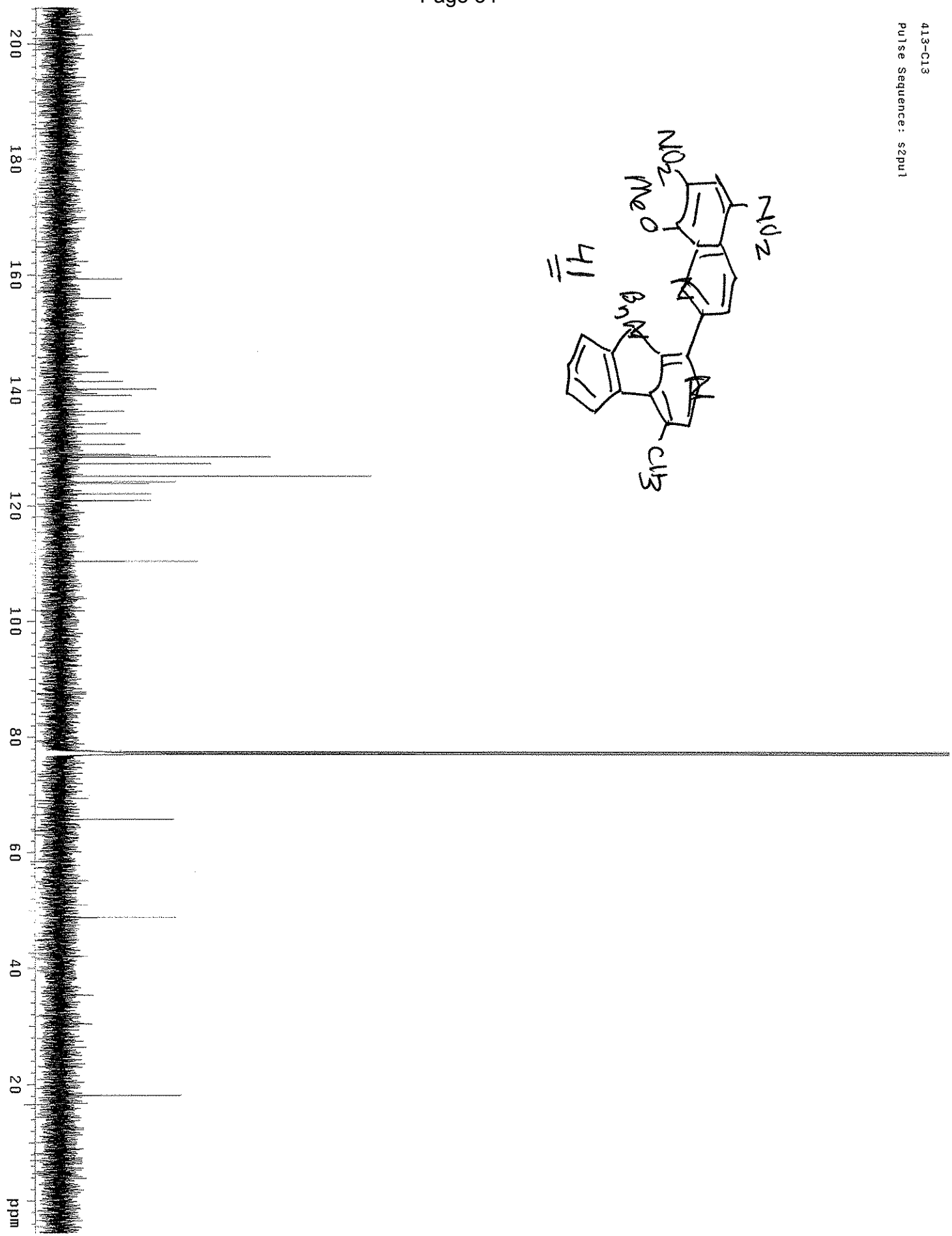


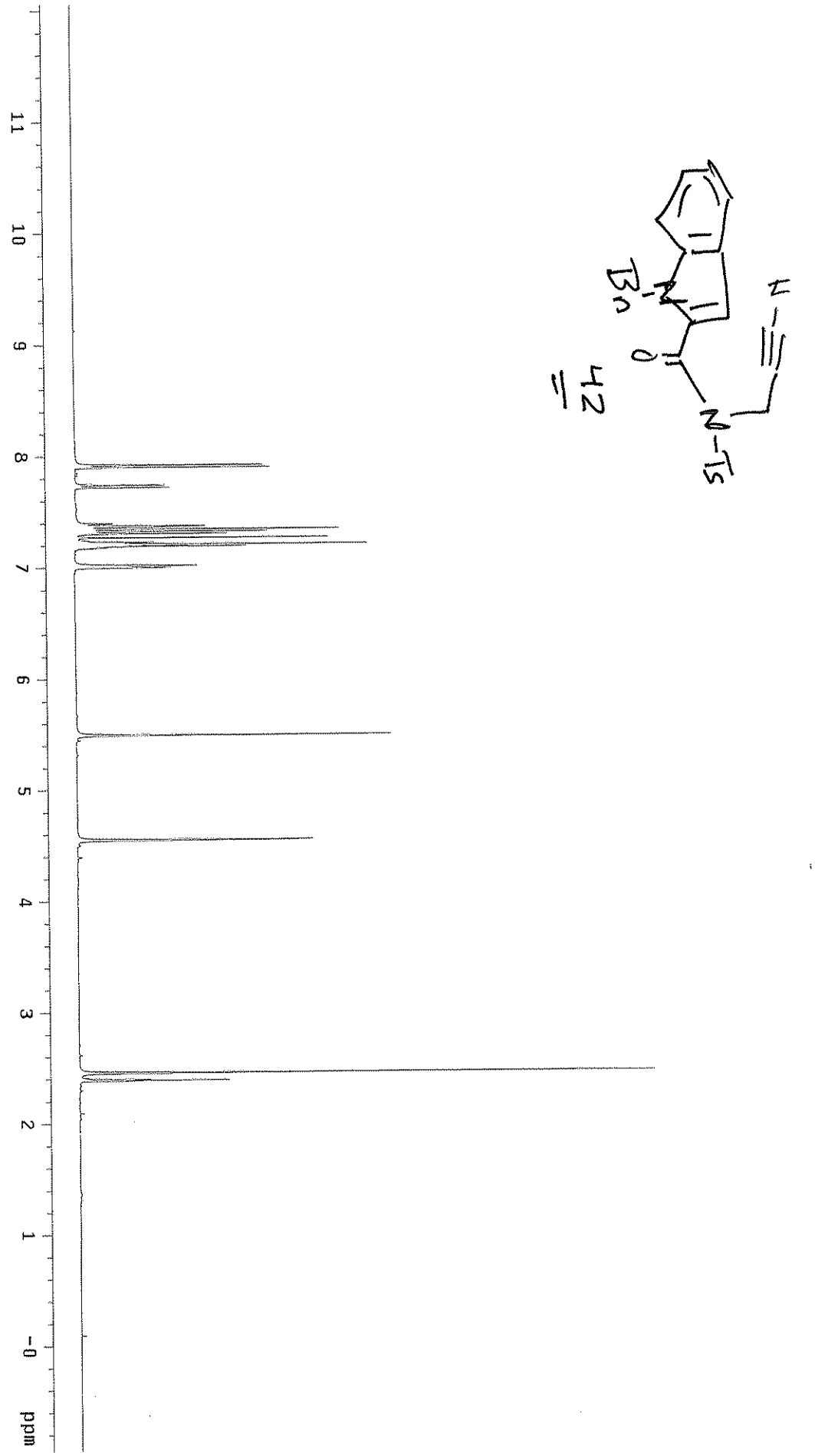
36

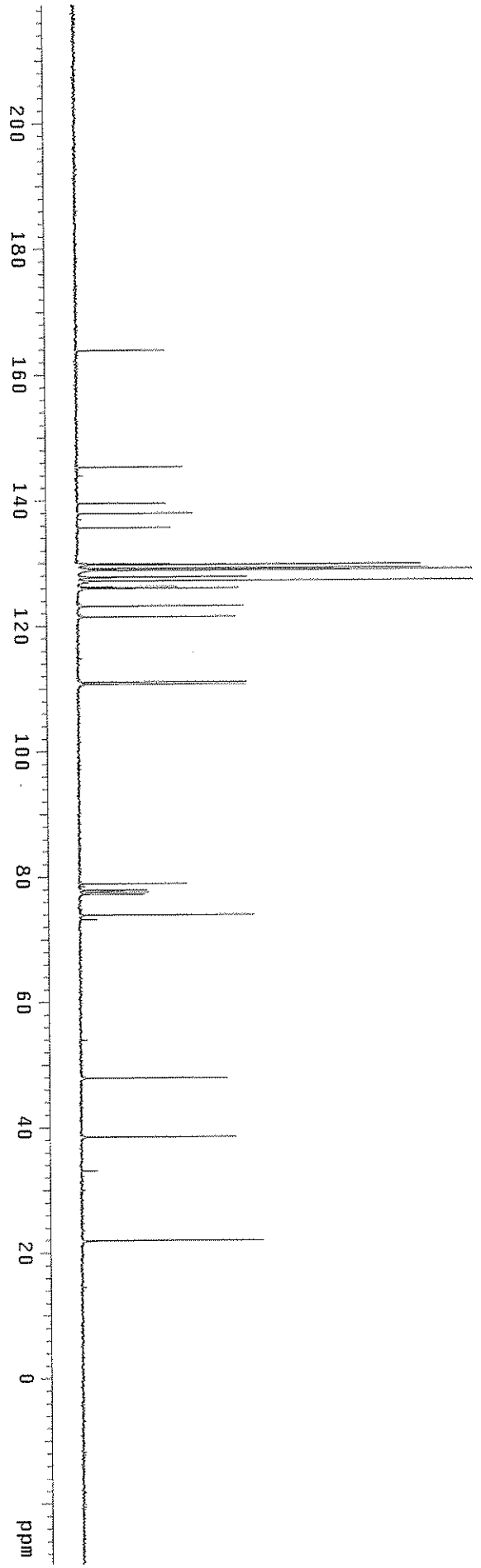

311a
Pulse Sequence: s2pul
40
Bn
Cl
CH3
ppm
11
10
9
8
7
6
5
4
3
2
1
0
1.00
0.93
1.07
1.11
1.90
1.07
1.07
1.99
2.00
3.02

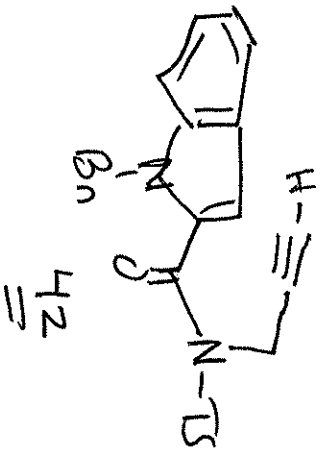

311A-c
Pulse Sequence: s2pul
Bn
N
HO
Cl
CH3
N
200
180
160
140
120
100
80
60
40
20
0
ppm


413-h
Pulse Sequence: s2pul
NO2
MeO
NO2
H
Bn
Cl3
ppm
0
1
2
3
4
5
6
7
8
9
10
11
0.81
0.95
1.03
0.89
0.98
1.09
1.16
1.12
1.06
2.08
1.90
2.00
2.91
3.02

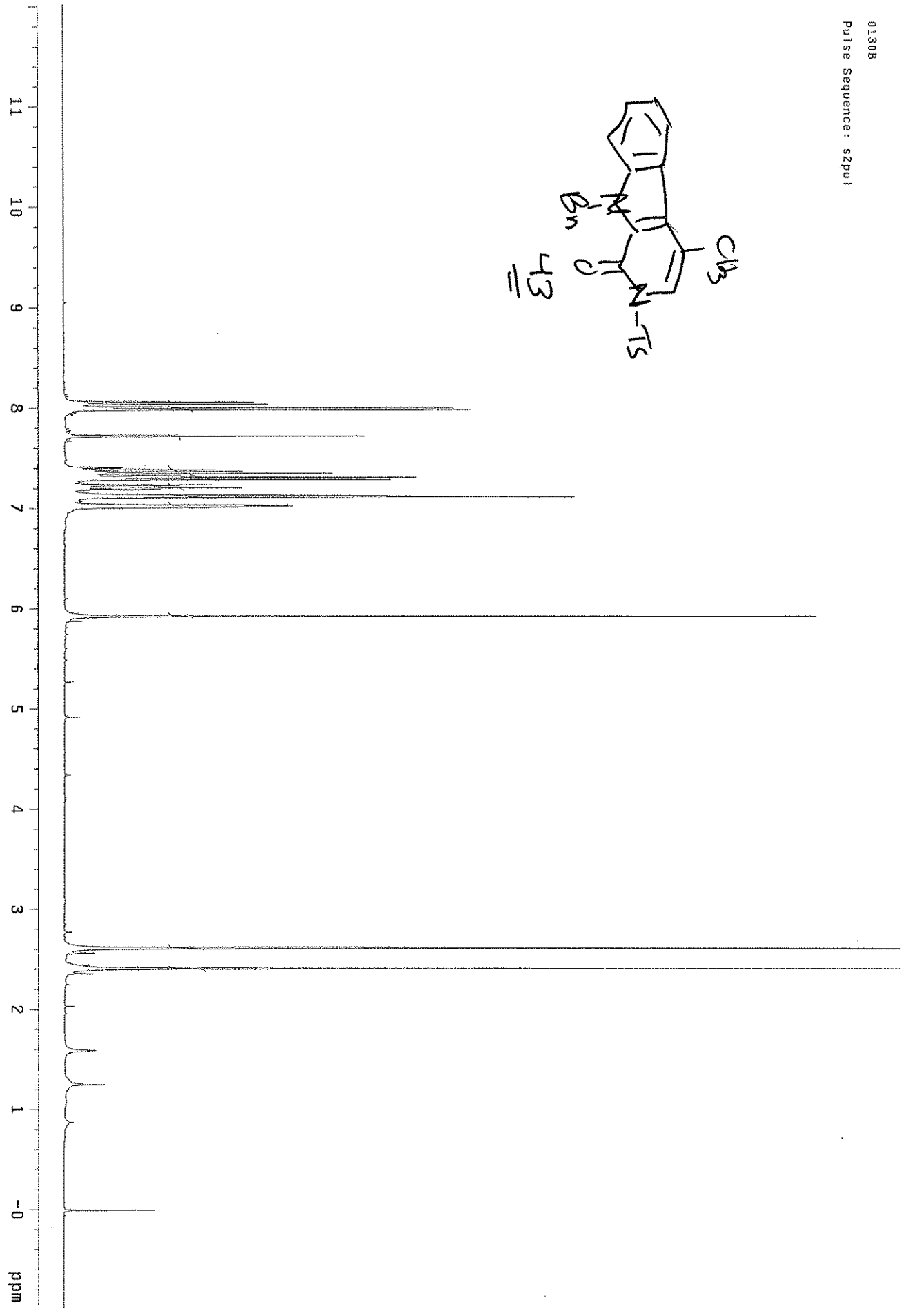

413-C13
Pulse Sequence: s2pul
NO2
MeO
NO2
H
BnN
CH3
200
180
160
140
120
100
80
60
40
20
ppm

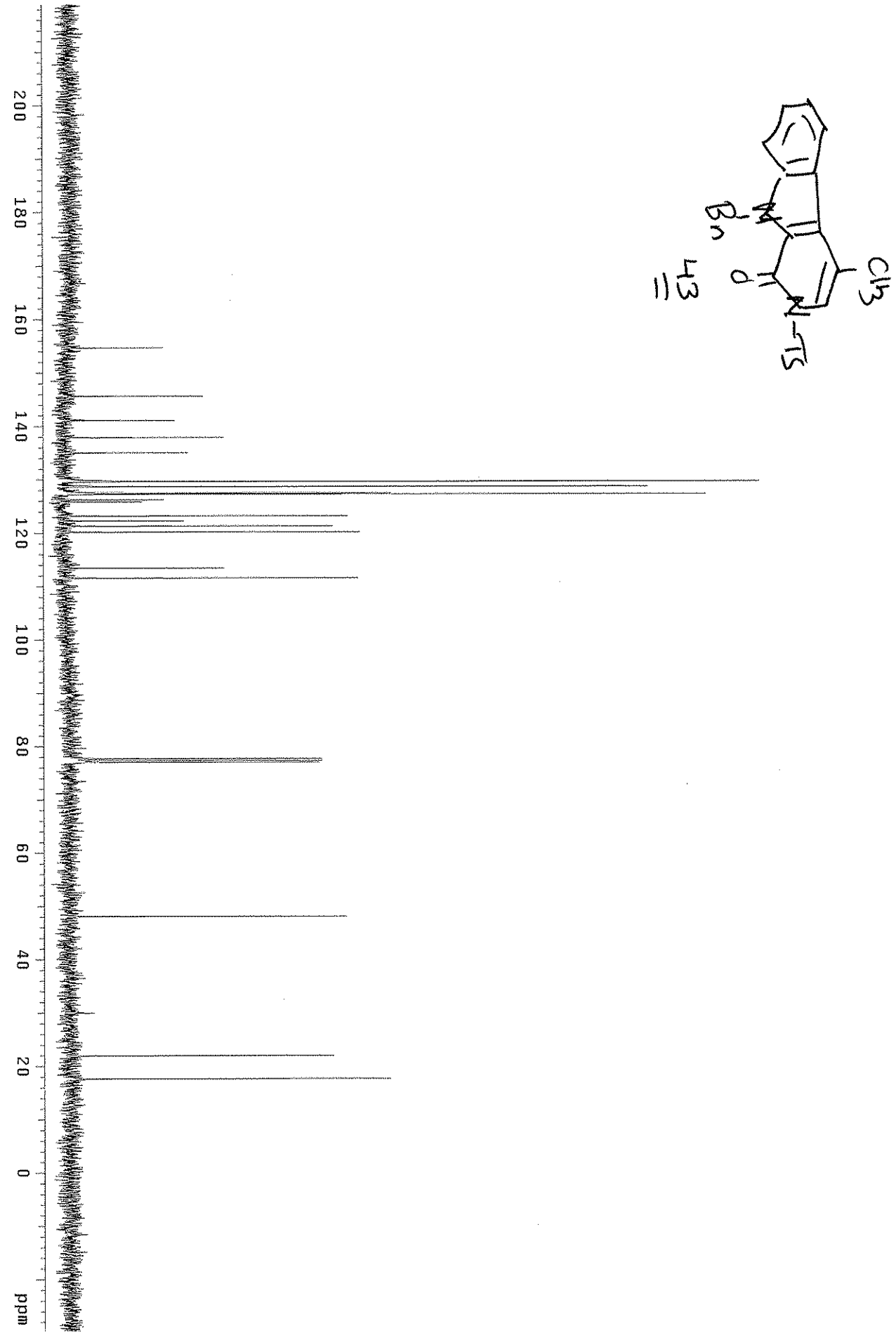

47-1-h
Pulse Sequence: s2pul
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

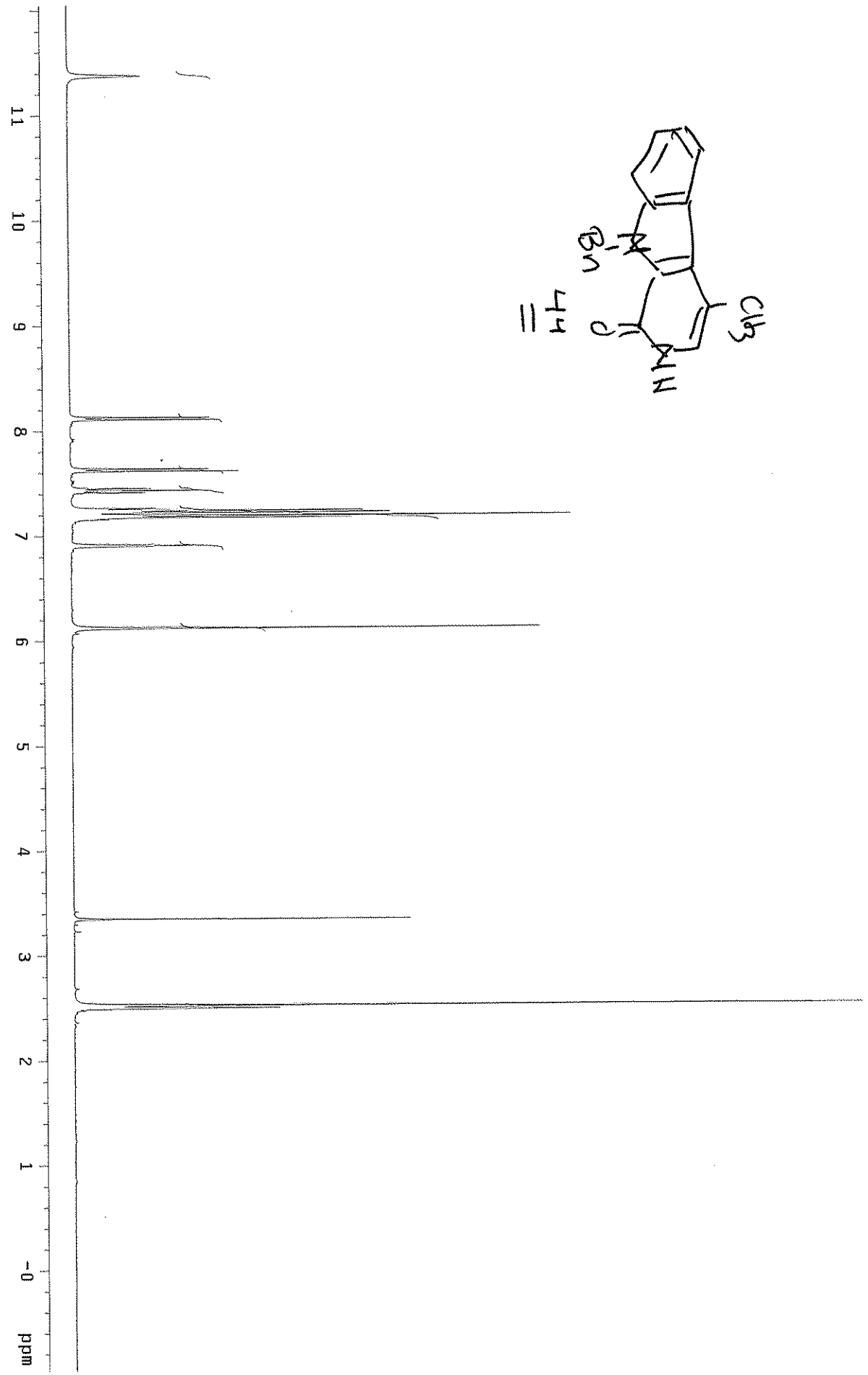


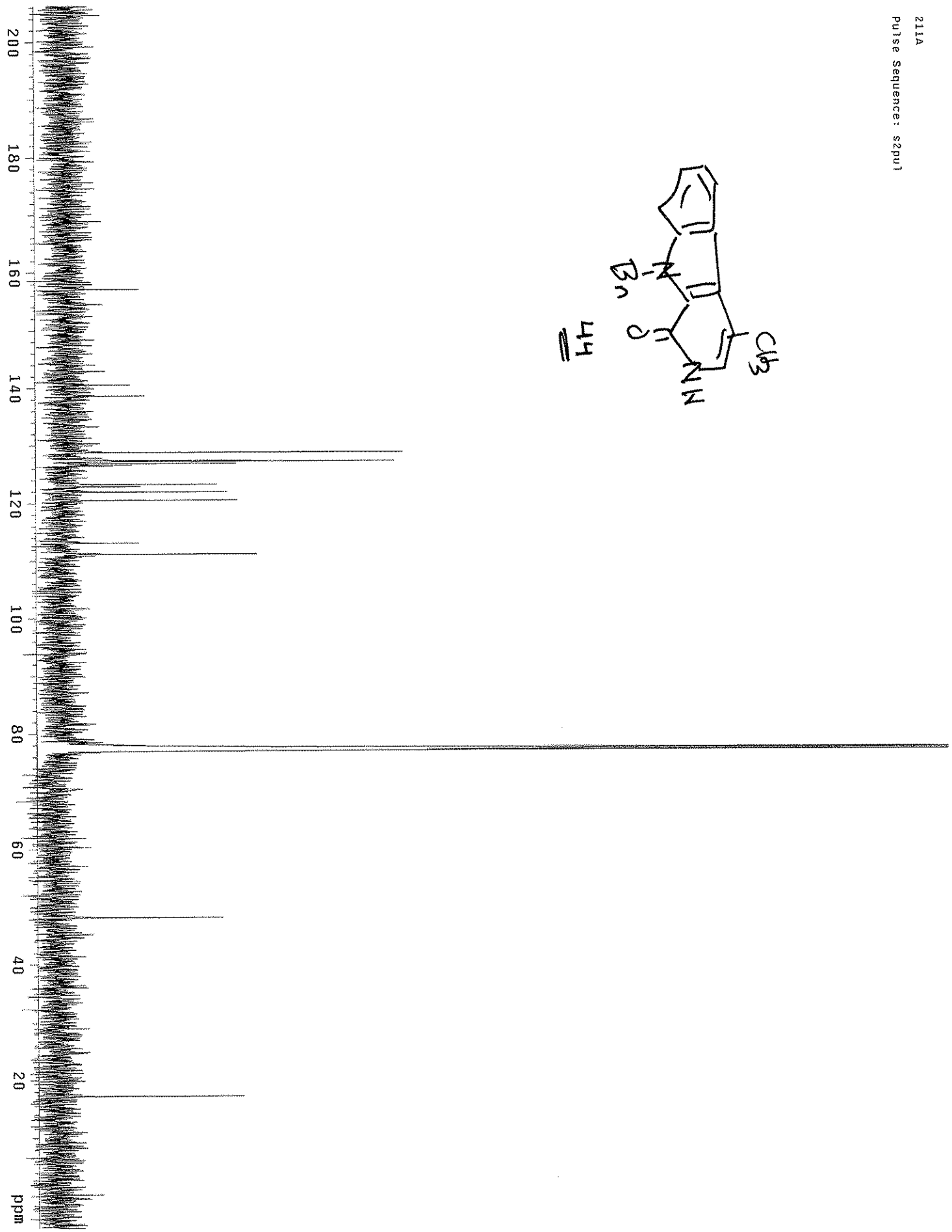


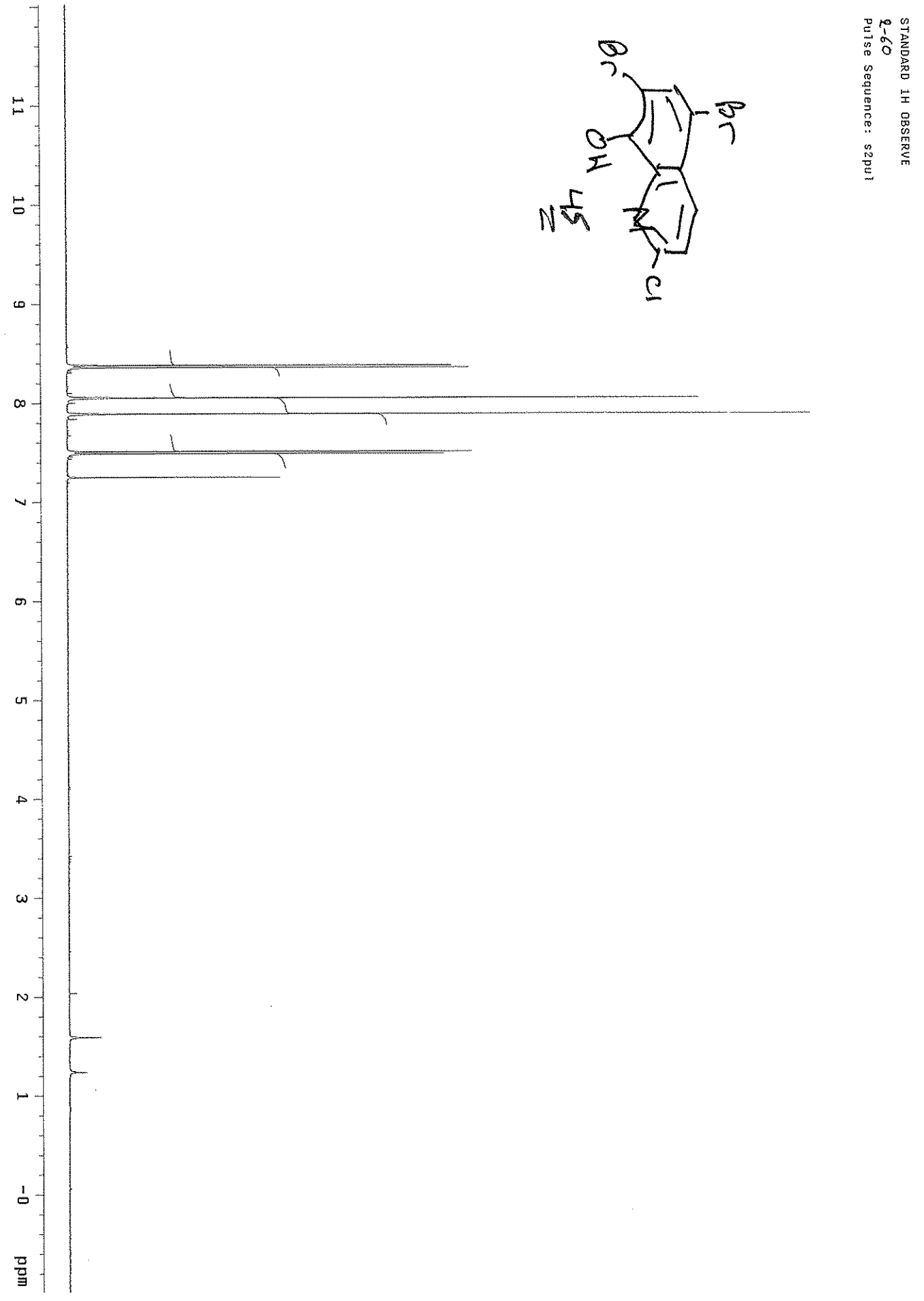
0130-c13
Pulse Sequence: s2pul


0130B
Pulse Sequence: s2pul
11
10
9
8
7
6
5
4
3
2
1
0
ppm


01308-c13
Pulse Sequence: s2pul
200
180
160
140
120
100
80
60
40
20
0
ppm
Bn
N
O
N
Ts
CH3
43


211A-DMSO-h
Pulse Sequence: s2pul
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

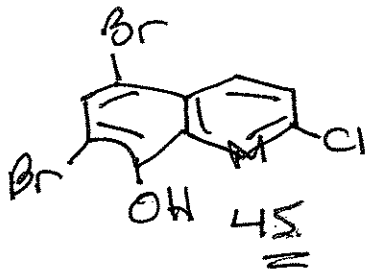

211A
Pulse Sequence: s2pul
200
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
2-60
Pulse Sequence: s2pul
Br
OH
N
Cl
ppm

2-60

Automation directory: /home/nmruser/Padwa_A/vnmrsys/data/auto_2006.09.06
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

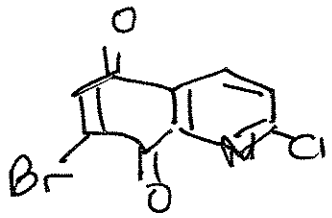


45

2-61b fc in dmso-d6

Automation directory: /home/nmruser/Padwa_A/vnmrsys/data/auto_2006.09.06
File : exp
Sample id : tmpstudy

Pulse Sequence: s2pul

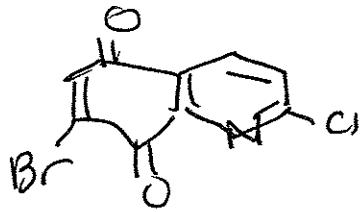


46

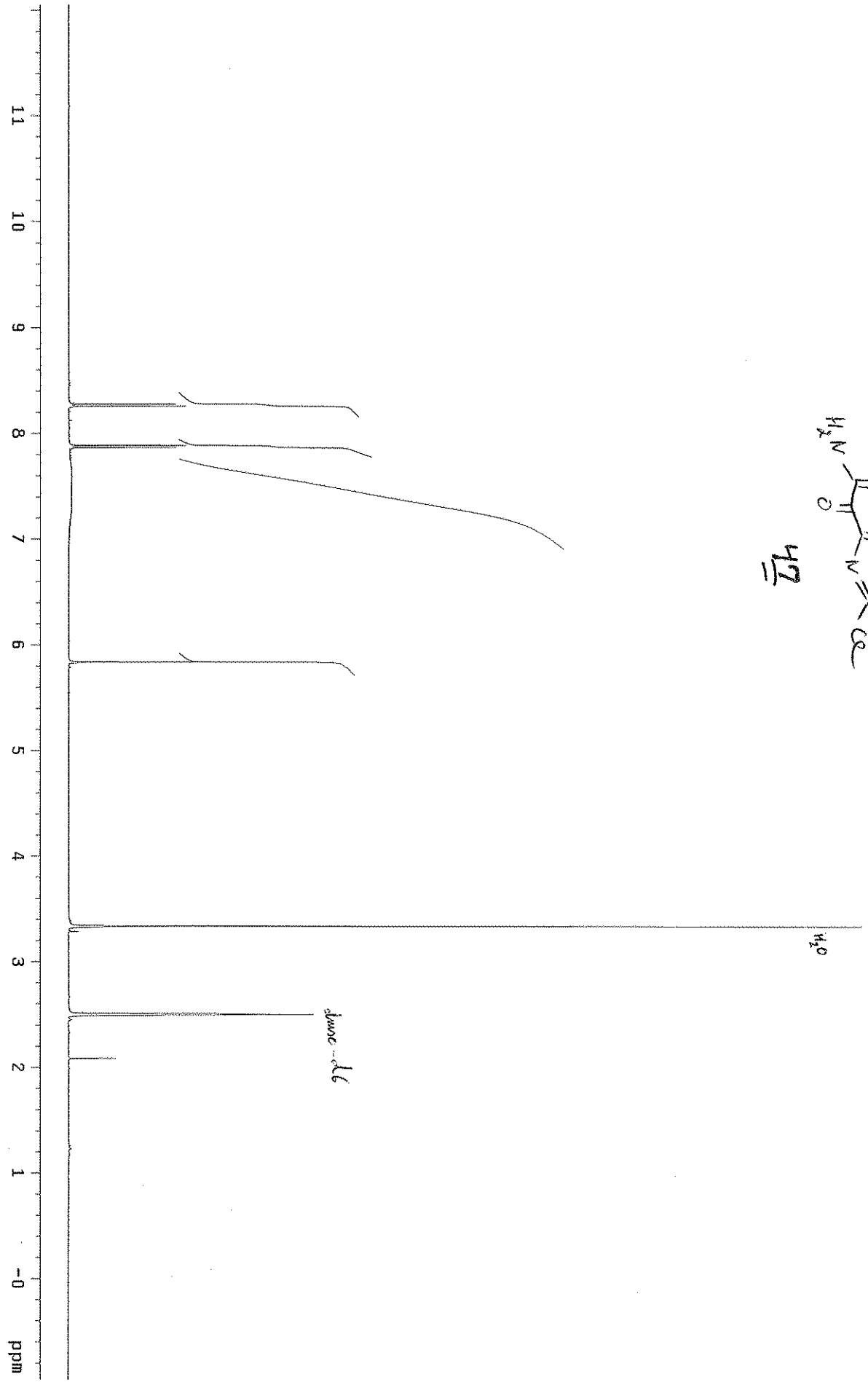
2-61b fc in dmso-d6

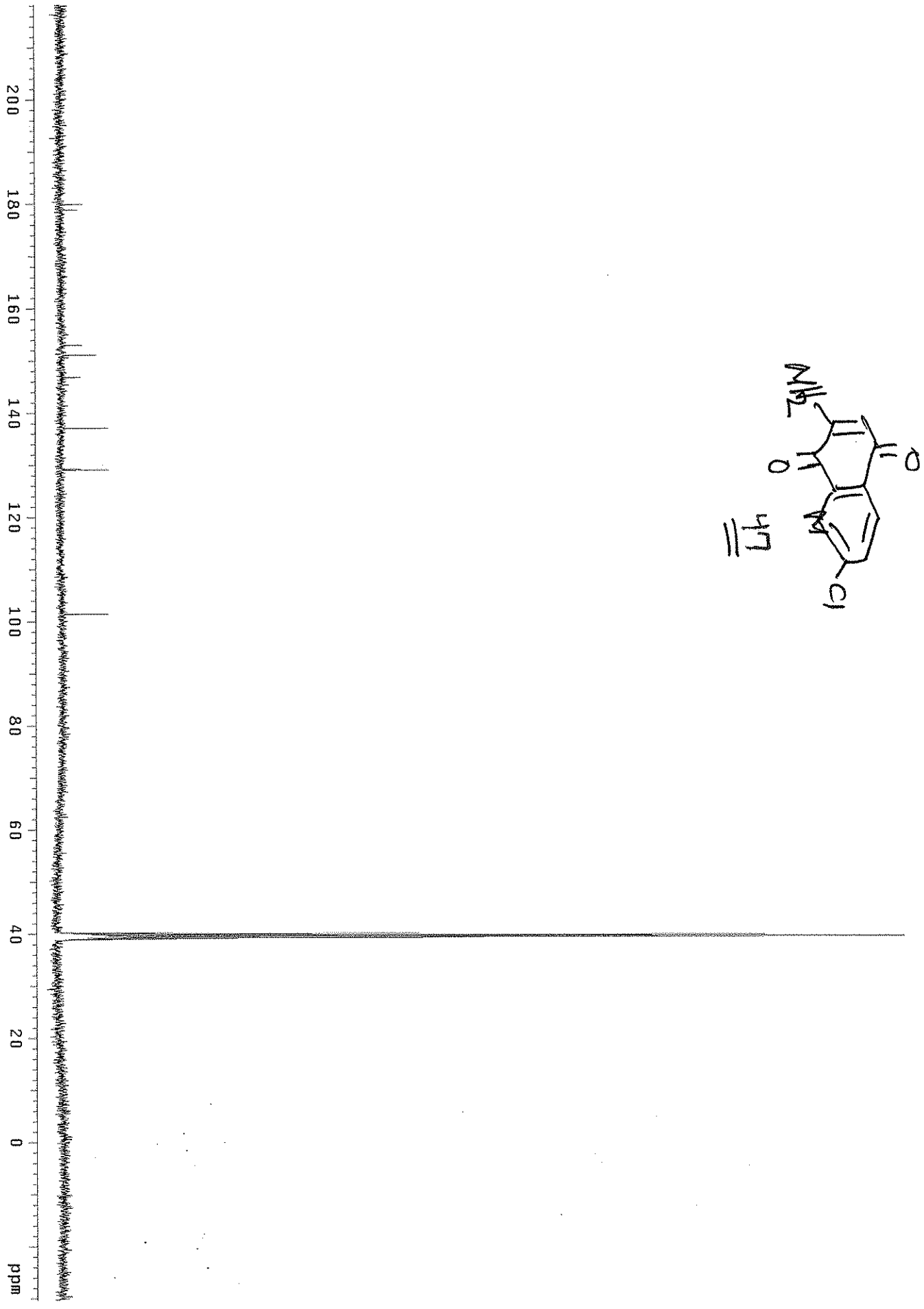
Automation directory: /home/nmruser/Padwa_A/vnmrsys/data/auto_2006.09.06
File : exp
Sample id : tmpstudy

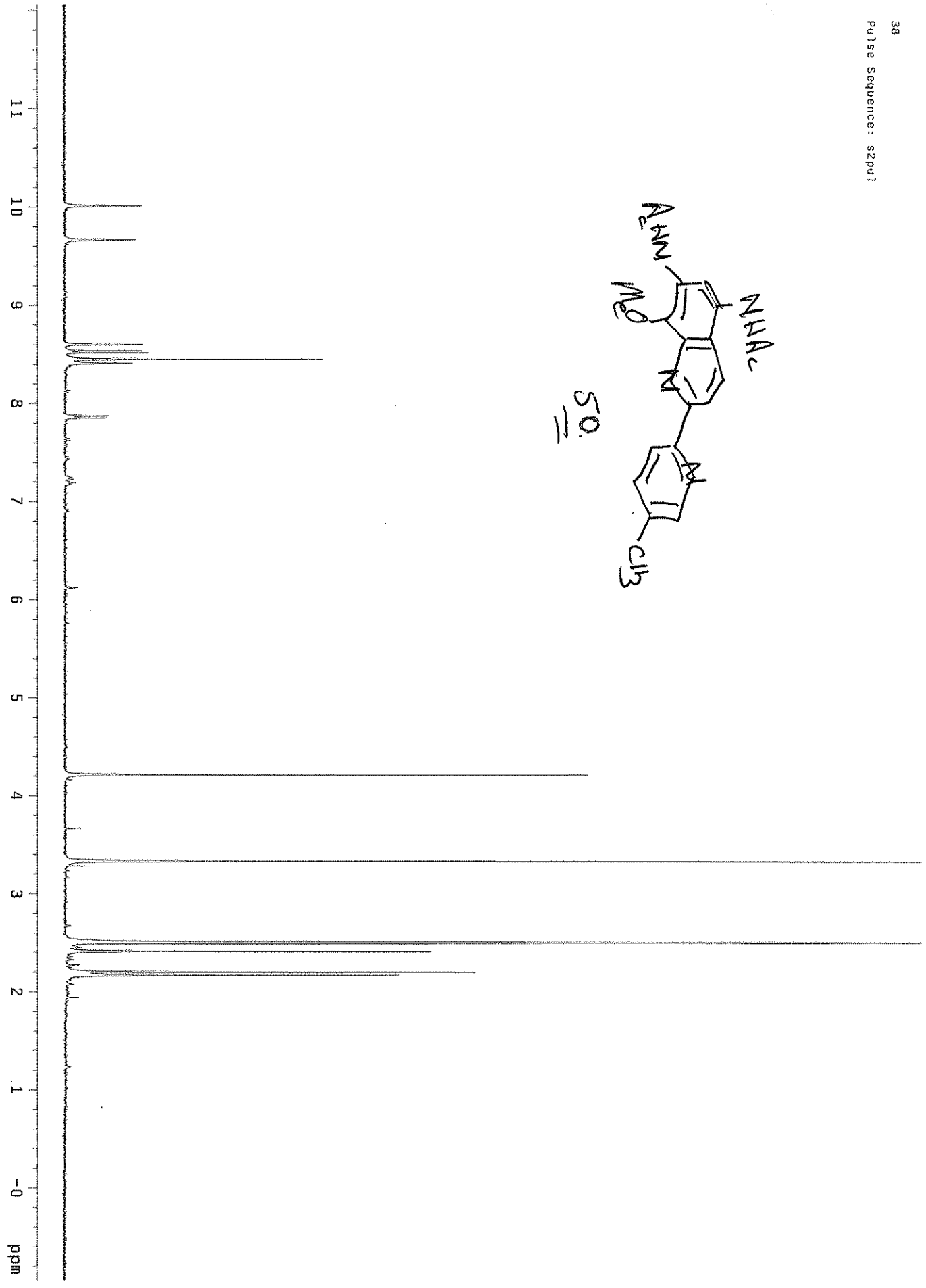
Pulse Sequence: s2pul

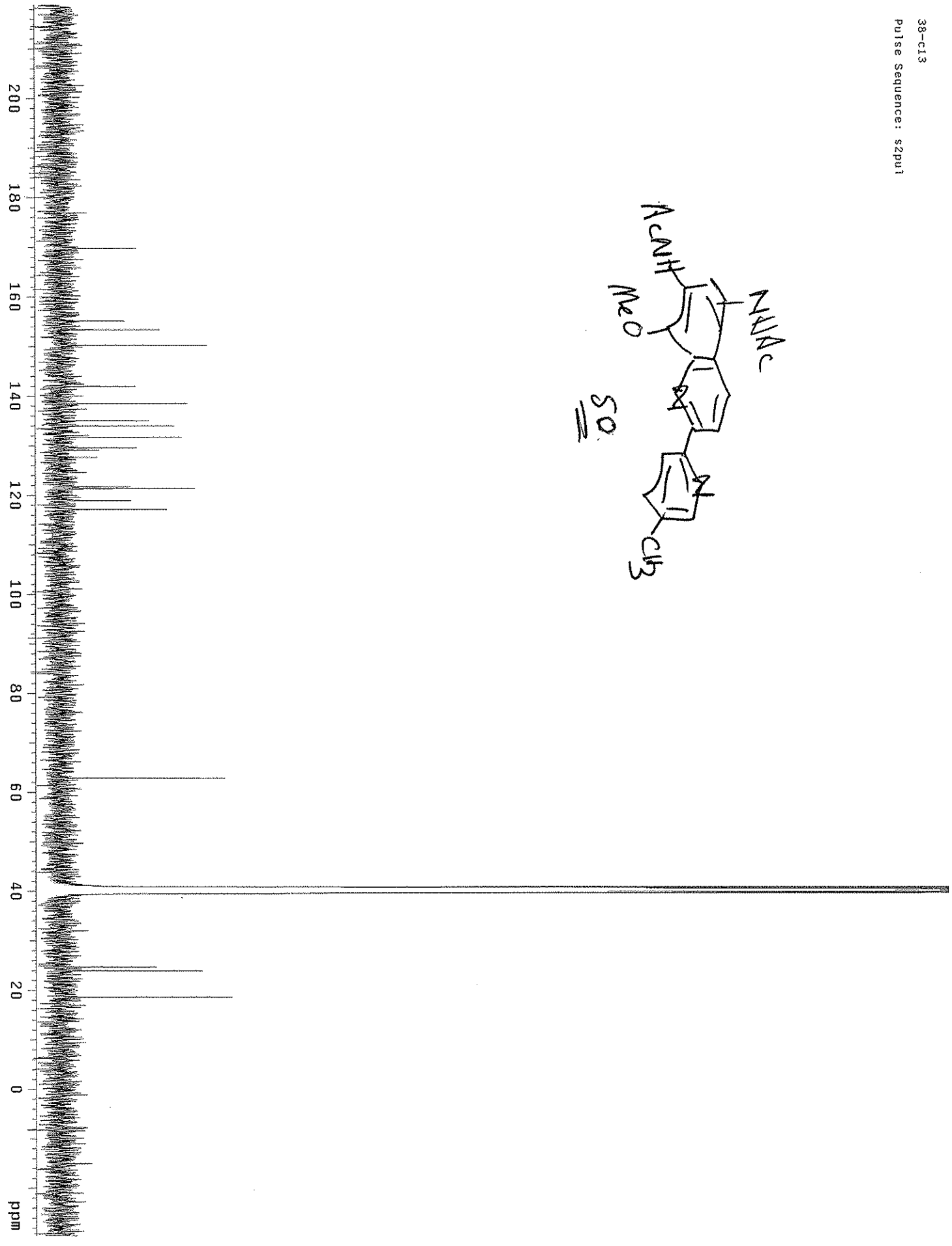


46


2-95fc2
Pulse Sequence: s2pul
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

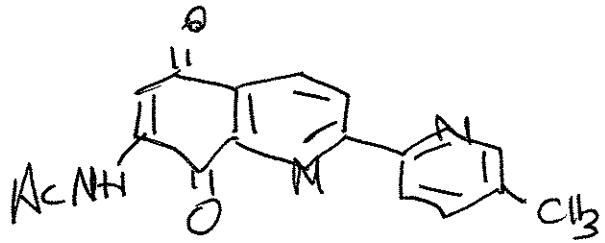

2-95fc2
Pulse Sequence: s2pul
200
180
160
140
120
100
80
60
40
20
0
ppm


38
Pulse Sequence: s2pul
11.0
5.0
NHAc
CH3
ppm

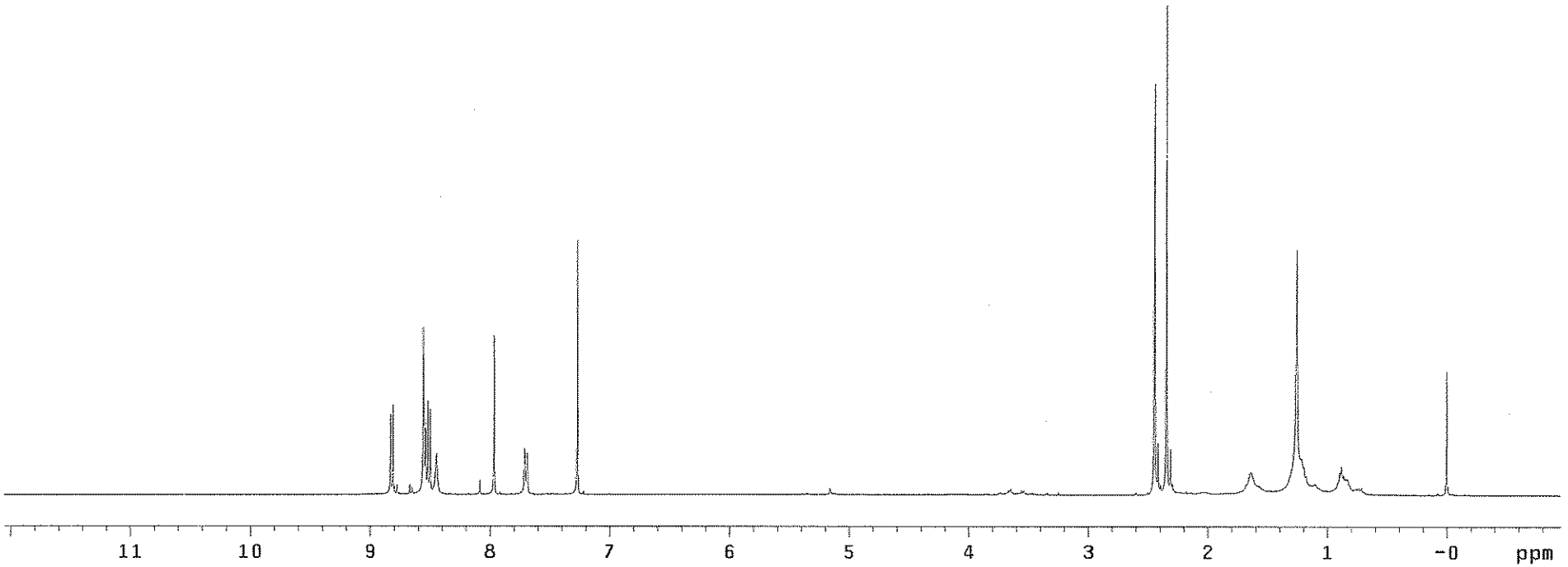

38-c13
Pulse Sequence: s2pul
AcNH
MeO
NHAc
SO
CH3
200
180
160
140
120
100
80
60
40
20
0
ppm

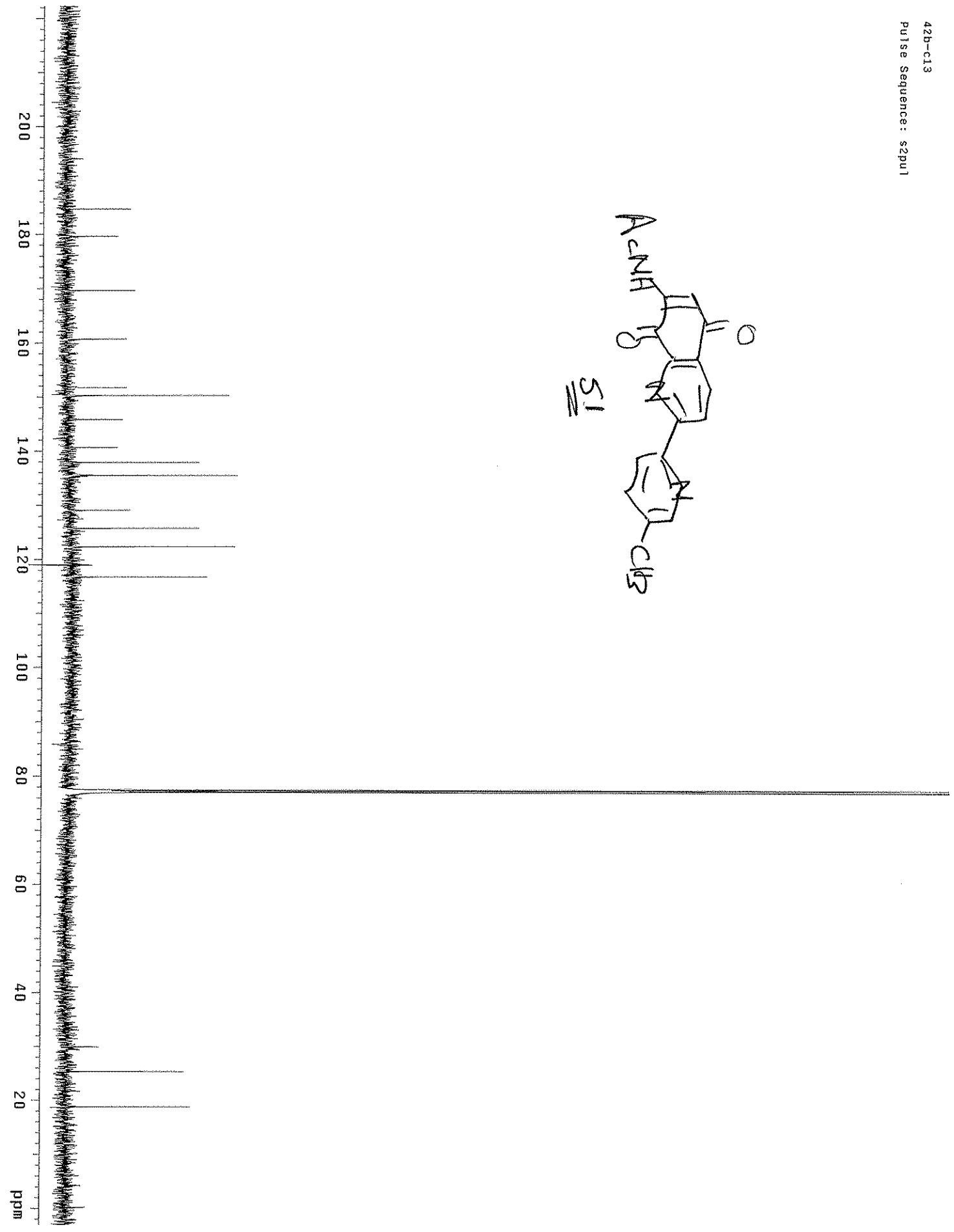
41h

Pulse Sequence: s2pul



S1




42b-c13
Pulse Sequence: s2pul
200
180
160
140
120
100
80
60
40
20
ppm
AcNH
O
N
S
Cl