SUPPLEMENTARY INFORMATION

Ionic liquids via efficient, solvent-free anion metathesis

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General Considerations. NMR spectra were recorded using a Varian Unity Plus 300 or 400 spectrometer. Chemical shifts are reported in delta (δ) units, expressed in parts per million (ppm) downfield from tetramethylsilane using residual protio solvent as an internal standard ($CDCl_3$, ^1H: 7.26 ppm, ^{13}C: 77.0 ppm; CD_2Cl_2 ^1H: 5.32 ppm, ^{13}C: 54.0 ppm). ^{31}P NMR spectra were recorded at 121 MHz and were externally referenced to H_3PO_4. ^{13}C and ^{31}P NMR spectra were routinely run with broadband decoupling. CH_2Cl_2 was distilled from CaH_2 under N_2 atmosphere prior to use. 1-Butyl-3-methylimidazolium ([BMIM]) halides were prepared via the reaction of 1-methylimidazole (1.0 equiv) and butyl halide (1.05 equiv) in a sealed vessel at 130 °C for 24 h, followed by removal of volatile components. Similarly, 1-(3-hydroxypropyl)-3-methylimidazolium bromide ([HPMIM]Br) was obtained from 1-methylimidazole and 3-bromo-1-propanol. 1-Butylpyridinium ([BPY]) halides were prepared in an analogous fashion from pyridine and the corresponding 1-haloalkane. All other reagents were purchased from commercial suppliers and used as received. All organic halide salts were stored in a desiccator prior to use. Iodide salts were protected from light using aluminum foil. Due to the hygroscopic nature of the salts discussed herein, reactions were routinely protected with a drying tube containing Drierite, unless otherwise noted. All reactions were conducted in a ventilated fume hood. Note: For solvent-free reactions involving solid azolium halides and trialkyloxonium salts, gradual liquefaction occurs over time. For comparative analyses, [BMIM][$MeSO_4$], [BPY][$MeSO_4$], [BMIM][OTs], [BMIM]BF_4, [BMIM]PF_6, [BPY]BF_4, trihexyl(tetradecyl)phosphonium tetrafluoroborate ([Hx_3PC_{14}]BF_4), and 1,3-dimesitylimidazolium tetrafluoroborate ([IMes]BF_4) were purchased from commercial suppliers. Caution: Dimethyl sulfate is toxic and should be handled with care.

Table 1. Summary of anion metathesis reactions reported in the accompanying manuscript.[a]

Entry	Trapping Reagent	Organic Halide Salt	Product
1	Me_2SO_4	[BMIM]Cl	[BMIM][$MeSO_4$]
2	Me_2SO_4	[BMIM]Br	[BMIM][$MeSO_4$]
3	Me_2SO_4	[BMIM]I	[BMIM][$MeSO_4$]
4	Me_2SO_4	[BPY]Cl	[BPY][$MeSO_4$]
5	Me_2SO_4	[BPY]I	[BPY][$MeSO_4$]
6	Me_2SO_4	[Hx_3PC_{14}]Cl	[Hx_3PC_{14}][$MeSO_4$]
7	Me_2SO_4	[Hx_3PC_{14}]Br	[Hx_3PC_{14}][$MeSO_4$]
8	MeOTs	[BMIM]Br	[BMIM][OTs]
9	MeOTs	[BMIM]I	[BMIM][OTs]
10	MeOTs	[BPY]Cl	[BPY][OTs]
11	MeOTs	[BPY]I	[BPY][OTs]
12	Me_3PO_4	[BPY]Cl	[BPY][Me_2PO_4]
13	$Me_3O{\cdot}BF_4$	[BMIM]Cl	[BMIM]BF_4
14	$Me_3O{\cdot}BF_4$	[BMIM]Br	[BMIM]BF_4
15	$Me_3O{\cdot}BF_4$	[BMIM]I	[BMIM]BF_4
16	$Et_3O{\cdot}BF_4$	[BMIM]Br	[BMIM]BF_4
17	$Et_3O{\cdot}PF_6$	[BMIM]Cl	[BMIM]PF_6
18	$Et_3O{\cdot}BF_4$	[BPY]Cl	[BPY]BF_4
19	$Et_3O{\cdot}BF_4$	[Hx_3PC_{14}]Cl	[Hx_3PC_{14}]BF_4
20	$Et_3O{\cdot}BF_4$	[Hx_3PC_{14}]Br	[Hx_3PC_{14}]BF_4
21[b]	$Et_3O{\cdot}BF_4$	[IMes]Cl	[IMes]BF_4
22[c]	$Et_3O{\cdot}BF_4$	[BMIM]Br	[BMIM]BF_4
23[b]	$Et_3O{\cdot}BF_4$	[HPMIM]Br	[HPMIM]BF_4

[a] Unless otherwise noted, all reactions were performed without the aid of solvent.
[b] Reaction was performed in dichloromethane.
[c] Reaction was performed in water.

Entry 1. A flask was charged with a magnetic stirbar and 1-butyl-3-methylimidazolium chloride (521 mg, 2.98 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.28 mL, 2.98 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 747 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

Entry 2. A flask was charged with a magnetic stirbar and 1-butyl-3-methylimidazolium bromide (639 mg, 2.90 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.28 mL, 2.90 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 727 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 3. A flask was charged with a magnetic stirbar and 1-butyl-3-methylimidazolium iodide (541 mg, 2.02 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.19 mL, 2.02 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 509 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 4. A flask was charged with a magnetic stirbar and 1-butylpyridinium chloride (603 mg, 3.51 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.33 mL, 3.51 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 868 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

Entry 5. A flask was charged with a magnetic stirbar and 1-butylpyridinium iodide (528 mg, 2.01 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.19 mL, 2.01 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 496 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 6. A flask was charged with a magnetic stirbar and trihexyl(tetradecyl)phosphonium chloride (141 mg, 0.27 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.03 mL, 0.27 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 162 mg (>99% yield) of the desired product as a clear viscous liquid. The product was analytically pure as determined by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy; spectra are provided below.

Entry 7. A flask was charged with a magnetic stirbar and trihexyl(tetradecyl)phosphonium bromide (152 mg, 0.27 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. Dimethyl sulfate (0.03 mL, 0.27 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 162 mg (>99% yield) of the desired product as a clear viscous liquid. The product was analytically pure as determined by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy.

Entry 8. A flask was charged with a magnetic stirbar and 1-butyl-3-methylimidazolium bromide (534 mg, 2.43 mmol), methyl tosylate (452 mg, 2.43 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. The mixture was then placed in an oil bath at 50 °C and vigorously stirred for 5 min, then placed under vacuum to provide 753 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

Entry 9. A flask was charged with a magnetic stirbar and 1-butyl-3-methylimidazolium iodide (548 mg, 2.04 mmol), methyl tosylate (381 mg, 2.04 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 635 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 10. A flask was charged with a magnetic stirbar and 1-butylpyridinium chloride (556 mg, 3.24 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. The flask was then placed in an oil bath at 120 °C and methyl tosylate (608 mg, 3.27 mmol) was injected via syringe through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 1.00 g (>99% yield) of the desired product as a beige solid. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy; spectra are provided below.

Entry 11. A flask was charged with a magnetic stirbar and 1-butylpyridinium iodide (699 mg, 2.66 mmol), methyl tosylate (495 mg, 2.66 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. The mixture was then vigorously stirred for 5 min, then placed under vacuum to provide 817 mg (>99% yield) of the desired product as a beige solid. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy.

Entry 12. A flask was charged with a magnetic stirbar and 1-butylpyridinium chloride (362 mg, 2.11 mmol), and then sealed with a rubber septum. A drying tube containing Drierite was fitted with a needle and inserted through the septum. The flask was then placed in an oil bath as 120 °C and trimethyl phosphate (0.25 mL, 2.12 mmol) was then injected via syringe through the septum. The mixture was then vigorously stirred for 90 min to provide 555 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy; spectra are provided below.

Entry 13. A flask was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium chloride (530 mg, 3.03 mmol), and trimethyloxonium tetrafluoroborate (448 mg, 3.03 mmol), and then fitted with a drying tube containing Drierite. The mixture was then vigorously stirred for 2 h to provide 685 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

Entry 14. A flask was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium bromide (1.0 g, 4.56 mmol), and trimethyloxonium tetrafluoroborate (675 mg, 4.56 mmol), and then fitted with a drying tube containing Drierite. The mixture was then vigorously stirred for 2 h to provide 1.02 g (99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 15. A flask was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium iodide (3.24 g, 12.2 mmol), and trimethyloxonium tetrafluoroborate (1.80 g, 12.2 mmol), and then fitted with a drying tube containing Drierite. After vigorously stirring for 2 h, the reaction mixture was placed under vacuum to provide 2.75 g (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 16. A flask was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium bromide (28.8 g, 132 mmol), and triethyloxonium tetrafluoroborate (25.0 g, 132 mmol), and then fitted with a drying tube containing Drierite. The mixture was then vigorously stirred for 2 h. After stirring was stopped, the top layer (determined by ^{1}H NMR spectroscopy to be EtBr and Et_2O) of the resulting biphasic mixture was decanted leaving 29.6 g (>99% yield) of the desired product. In a separate reaction (same scale), a flask was fitted with a distillation head in lieu of the drying tube. Upon completion, the biphasic mixture was placed in an oil bath and the EtBr and Et_2O were codistilled leaving the desired product in quantitative yield. (Note: The EtBr and Et_2O distillate was collected in 97% yield; a ^{1}H NMR spectrum is shown below). The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 17. A flask was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium chloride (185 mg, 1.06 mmol), and triethyloxonium hexafluorophosphate (263 mg, 1.06 mmol), and then fitted with a drying tube containing Drierite. After vigorously stirring the mixture for 2 h, the reaction was placed under vacuum to give 299 mg (99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 18. A flask was charged with a magnetic stirbar, 1-butylpyridinium chloride (621 mg, 3.62 mmol), and triethyloxonium tetrafluoroborate (688 mg, 3.62 mmol), and then fitted with a drying tube containing Drierite. After vigorously stirring the mixture for 2 h, the reaction was placed under vacuum to provide 807 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

Entry 19. A flask was charged with a magnetic stirbar, trihexyl(tetradecyl)phosphonium chloride (500 mg, 0.96 mmol), and triethyloxonium tetrafluoroborate (183 mg, 0.96 mmol), and then fitted with a drying tube containing Drierite. After vigorously stirring the mixture for 2 h, the reaction was placed under vacuum to provide 546 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

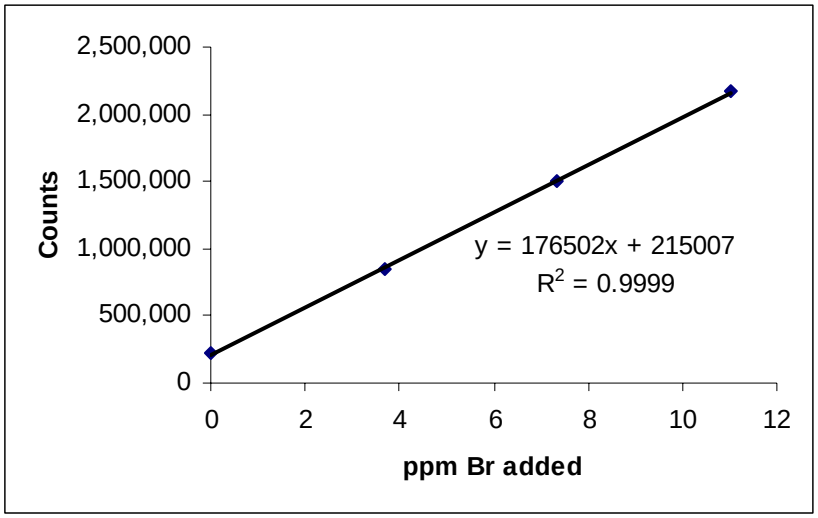
Entry 20. A flask was charged with a magnetic stirbar, trihexyl(tetradecyl)phosphonium bromide (330 mg, 0.59 mmol), and triethyloxonium tetrafluoroborate (111 mg, 0.59 mmol), and then fitted with a drying tube containing Drierite. After vigorously stirring the mixture for 2 h, the reaction was placed under vacuum to provide 336 mg (>99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample.

Entry 21. A vial was charged with 1,3-dimesitylimidazolium chloride (900 mg, 2.64 mmol), CD_2Cl_2 (9 mL), and triethyloxonium tetrafluoroborate (502 mg, 2.64 mmol). The reaction was monitored by ^{1}H NMR spectroscopy (a spectrum of an aliquot is shown below). The mixture was then concentrated under vacuum to give 1.03 g (99% yield) of the desired product. The product was analytically pure as determined by ^{1}H and ^{13}C NMR spectroscopy in comparison with an authentic sample; spectra are provided below.

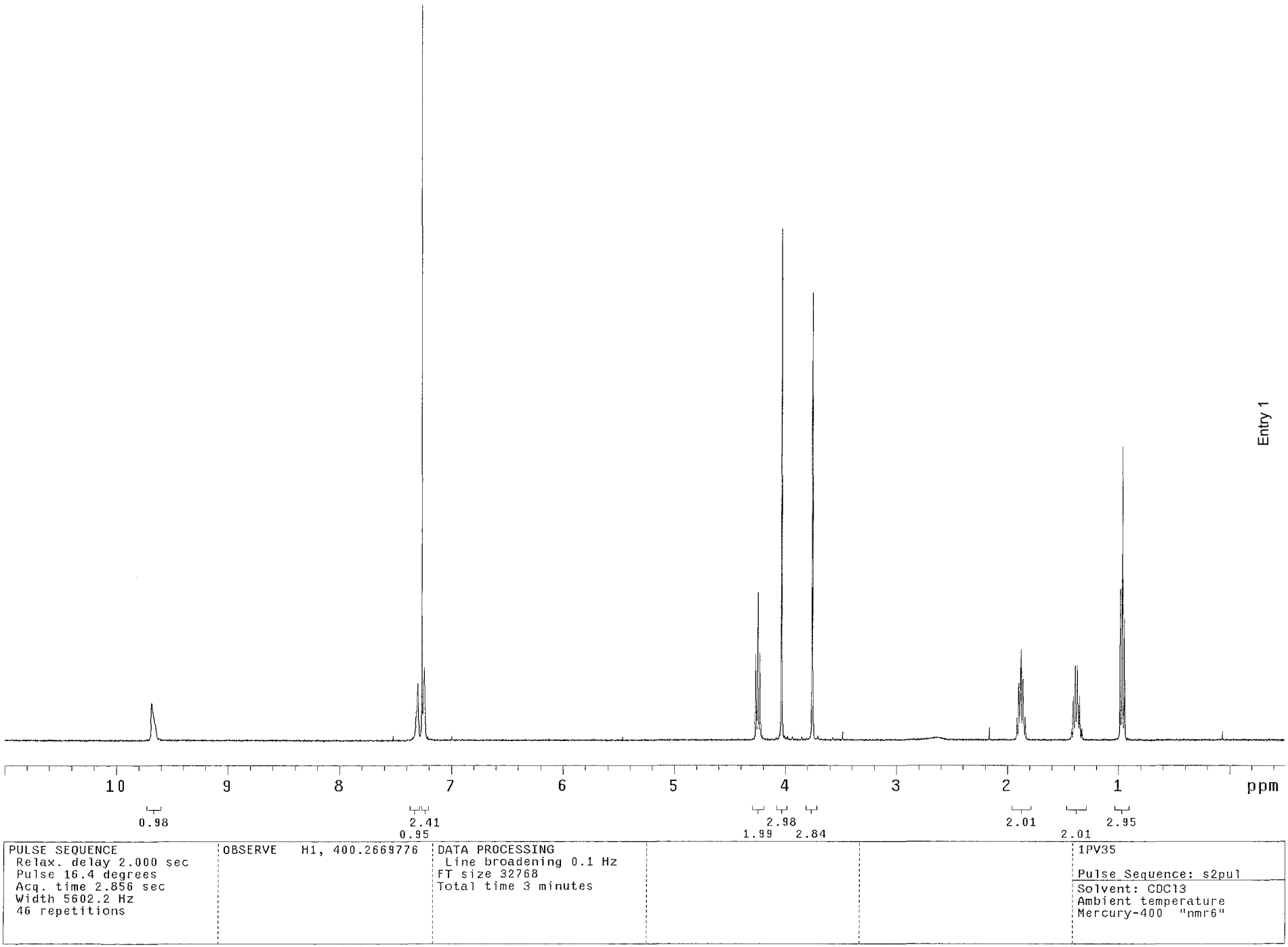
Entry 22. A vial was charged with a magnetic stirbar, 1-butyl-3-methylimidazolium bromide (1.08 g, 4.93 mmol), and distilled H_2O (2 mL). Triethyloxonium tetrafluoroborate (936 mg, 4.93 mmol) was added in one portion and the vial was capped and shaken. Upon standing, a biphasic mixture formed. The layers were separated, providing an aqueous solution of 1-butyl-3-methylimidazolium tetrafluoroborate and an organic layer comprised of EtBr and Et_2O in ca. 1:1 molar ratio, as determined by ^1H NMR spectroscopy (see NMR spectra below). The combined EtBr and Et_2O products (897 mg) were recovered in 99% yield. The aqueous layer was extracted with CD_2Cl_2 and found to contain 1-butyl-3-methylimidazolium tetrafluoroborate with trace amounts of diethyl ether. The solution was concentrated and corresponding NMR spectra (CD_2Cl_2) of the isolated product, [BMIM]BF_4, are shown below.

Entry 23. A vial was charged with a magnetic stirbar, 1-(3-hydroxypropyl)-3-methylimidazolium bromide (1.14 g, 5.13 mmol), and CH_2Cl_2 (10 mL). The resulting suspension was vigorously stirred and triethyloxonium tetrafluoroborate (975 mg, 5.13 mmol) was added in one portion. The resulting mixture was stirred for ca. 10 min, then concentrated under vacuum to give 1.17 g (>99% yield) of the desired product. The ^1H and ^{13}C NMR were consistent with previously reported spectral data;[1] corresponding NMR spectra (D_2O) of the isolated product, [HPMIM]BF_4, are shown below.

Inductively-Coupled Mass Spectroscopy. Measurements performed on aqueous samples using standard additions of KBr. Mass spectrometry (ICP-MS) was performed on a GBC OptiMass 8000, assaying for total ^{81}Br presence. Experiments were conducted in triplicate. A representative example is as follows: A product sample solution was prepared with concentration of [BMIM]BF_4 = 1.11 wt % in 100 mL H_2O (MilliPore). From this solution, 10 mL solutions were prepared containing standard additions of 0, 11, 22, and 33 μL of 3336 ppm KBr. The solutions were analyzed in triplicate via ICP-MS to provide the graph shown below. Analysis of the data using a linear regression (R^2 = 0.9999) gave slope = 176502 counts/ppm, Y-int = 215007 counts, and X-int = -1.22 ppm. The concentration of Br in the initial [BMIM]BF_4 aqueous solution is the absolute value of the X-int (1.22 ppm). The background correction for the concentrations of Br in the H_2O was 0.27 ppm. This corresponds to an initial concentration of Br in [BMIM]BF_4 of 85 ± 8 ppm.



[1] A. Lesimple, O. Mamer, W. Miao, T. H. Chan. *J. Am. Soc. Mass Spectrom.*, 2006, **17**, 85.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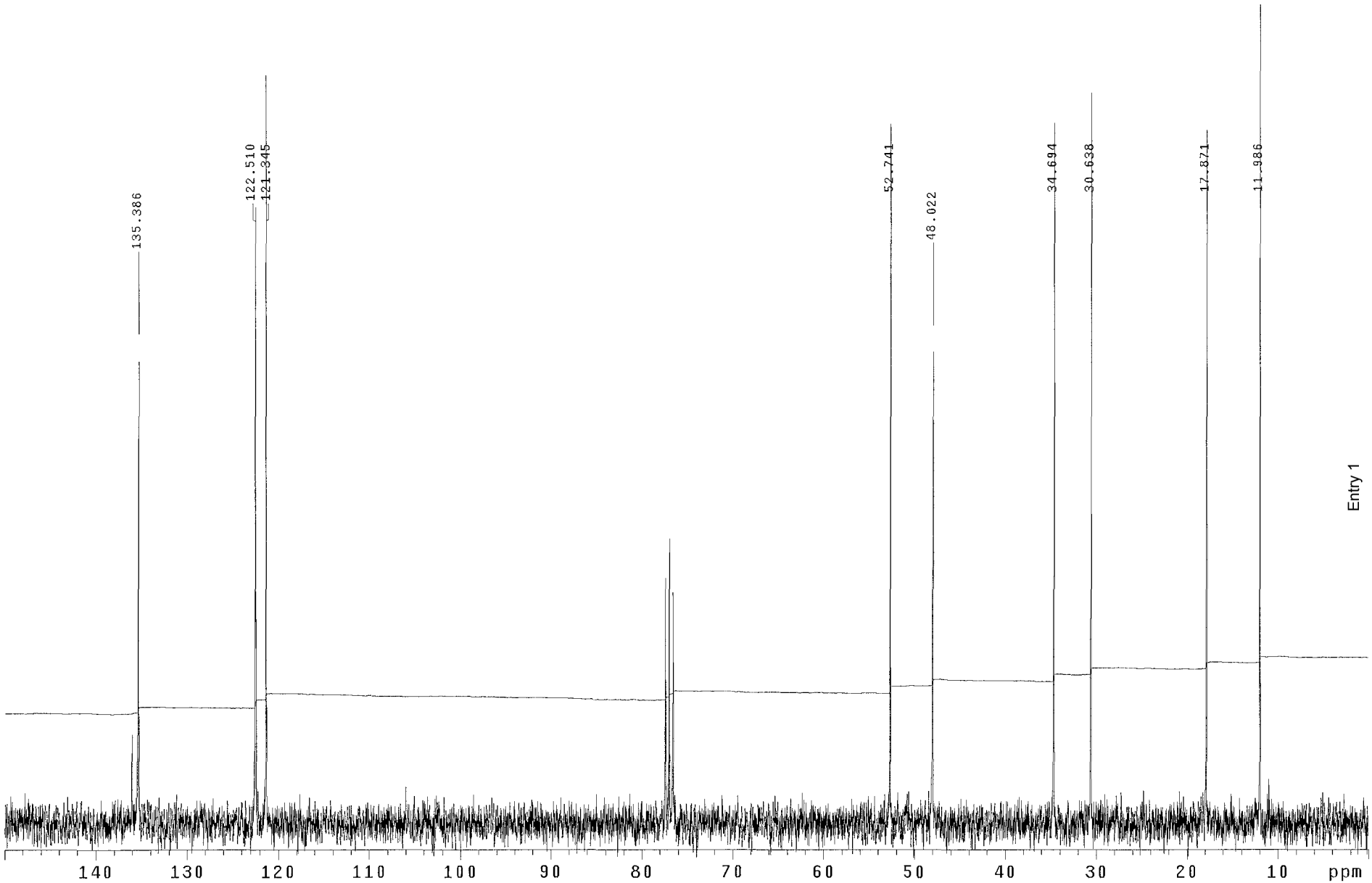

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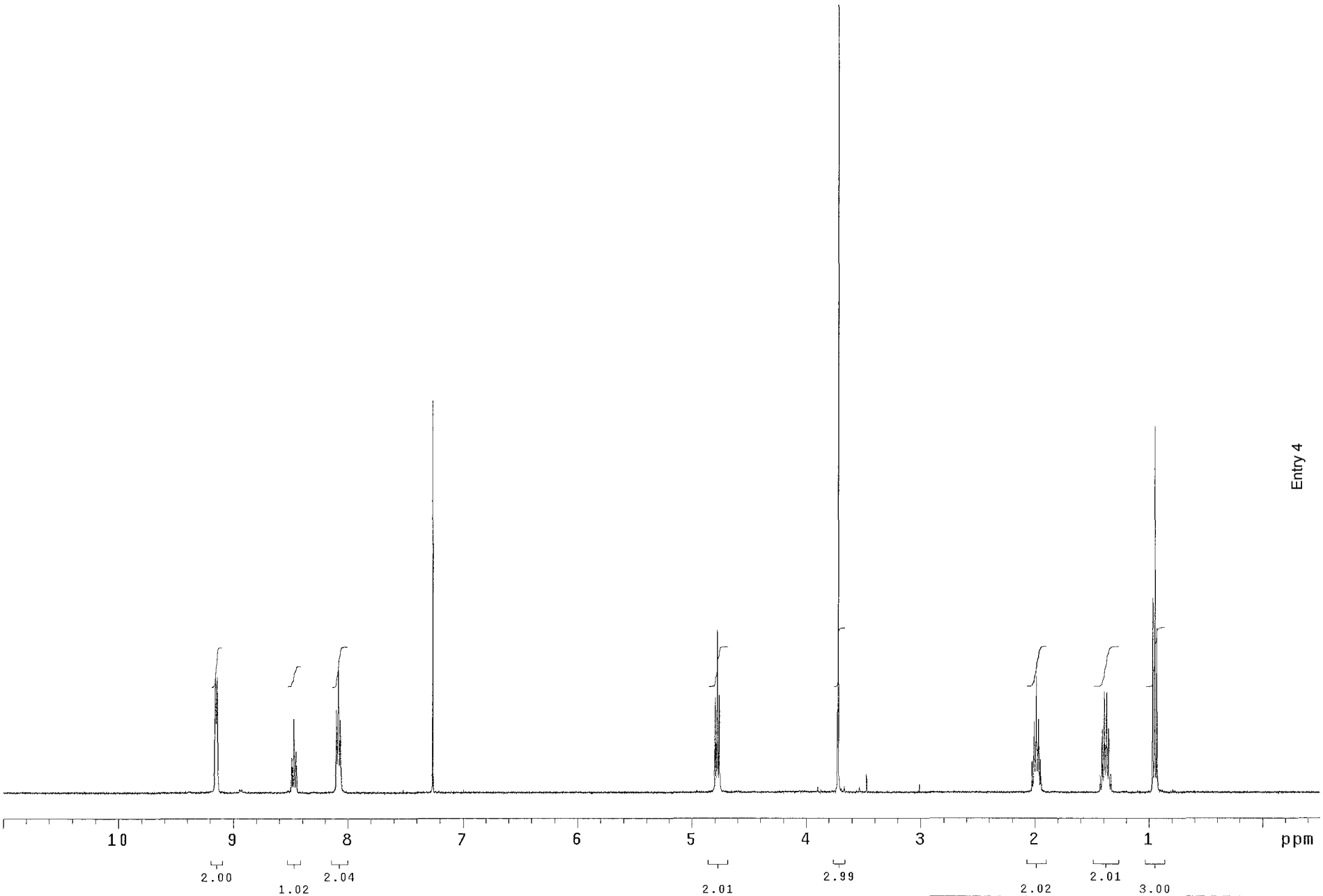

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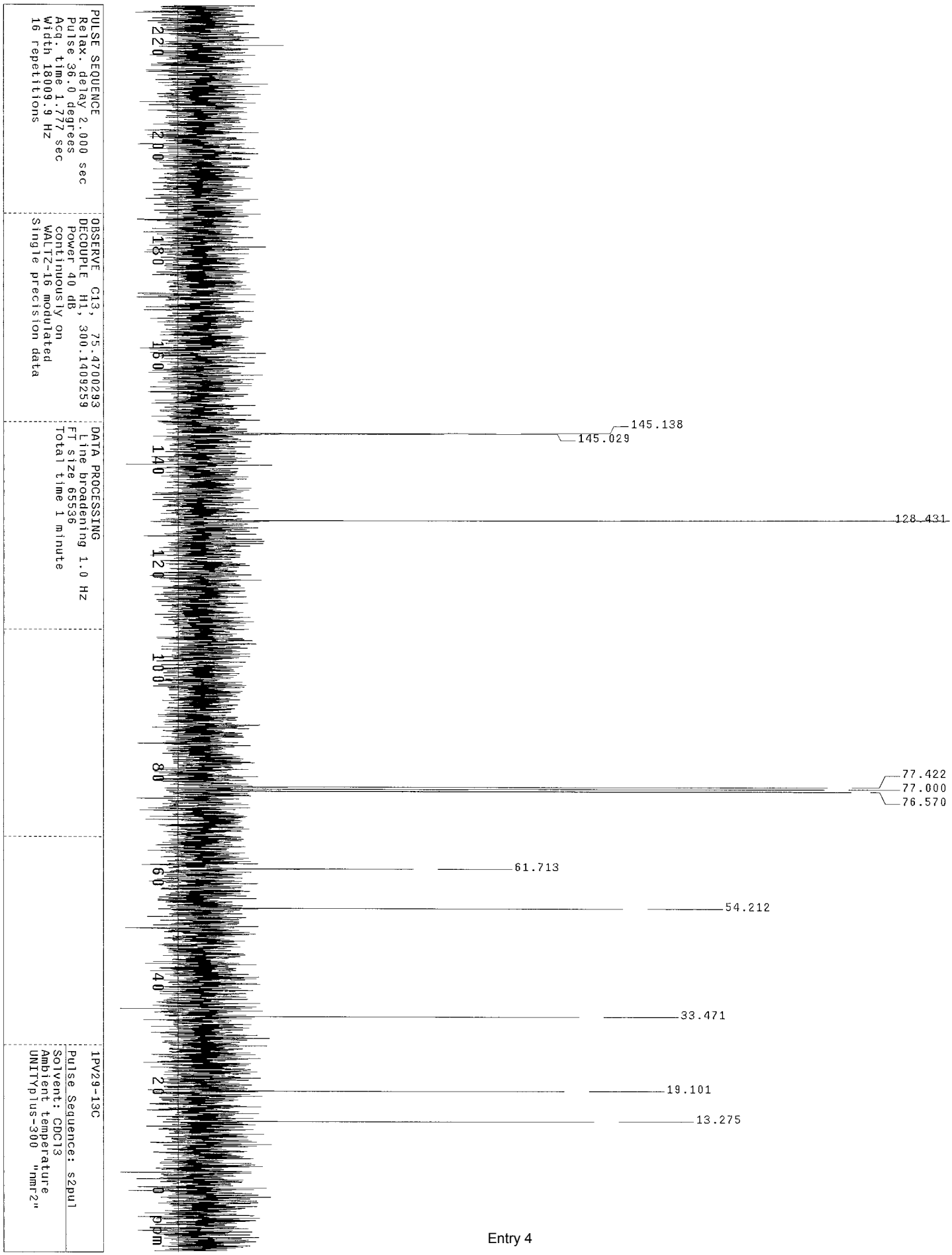
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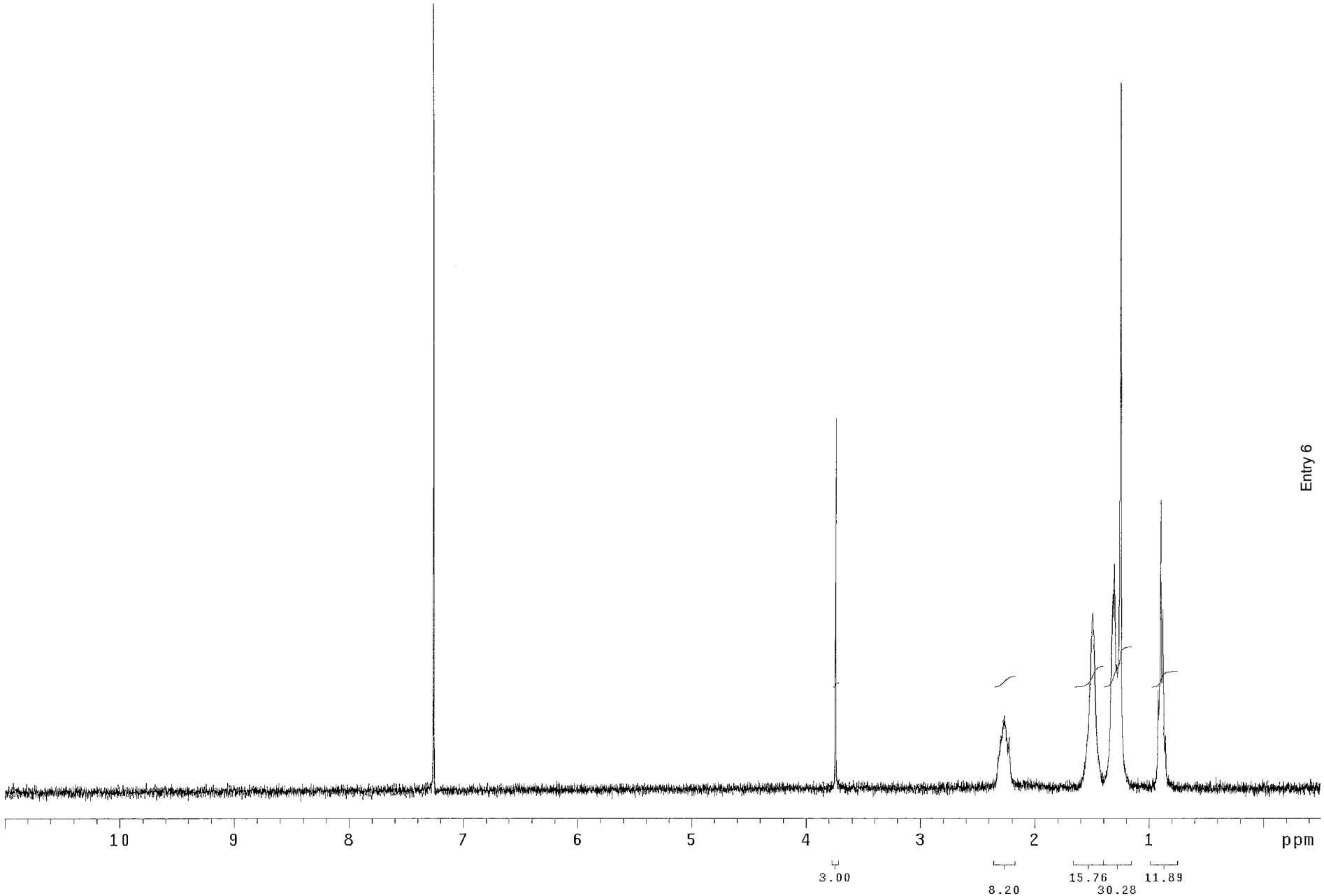

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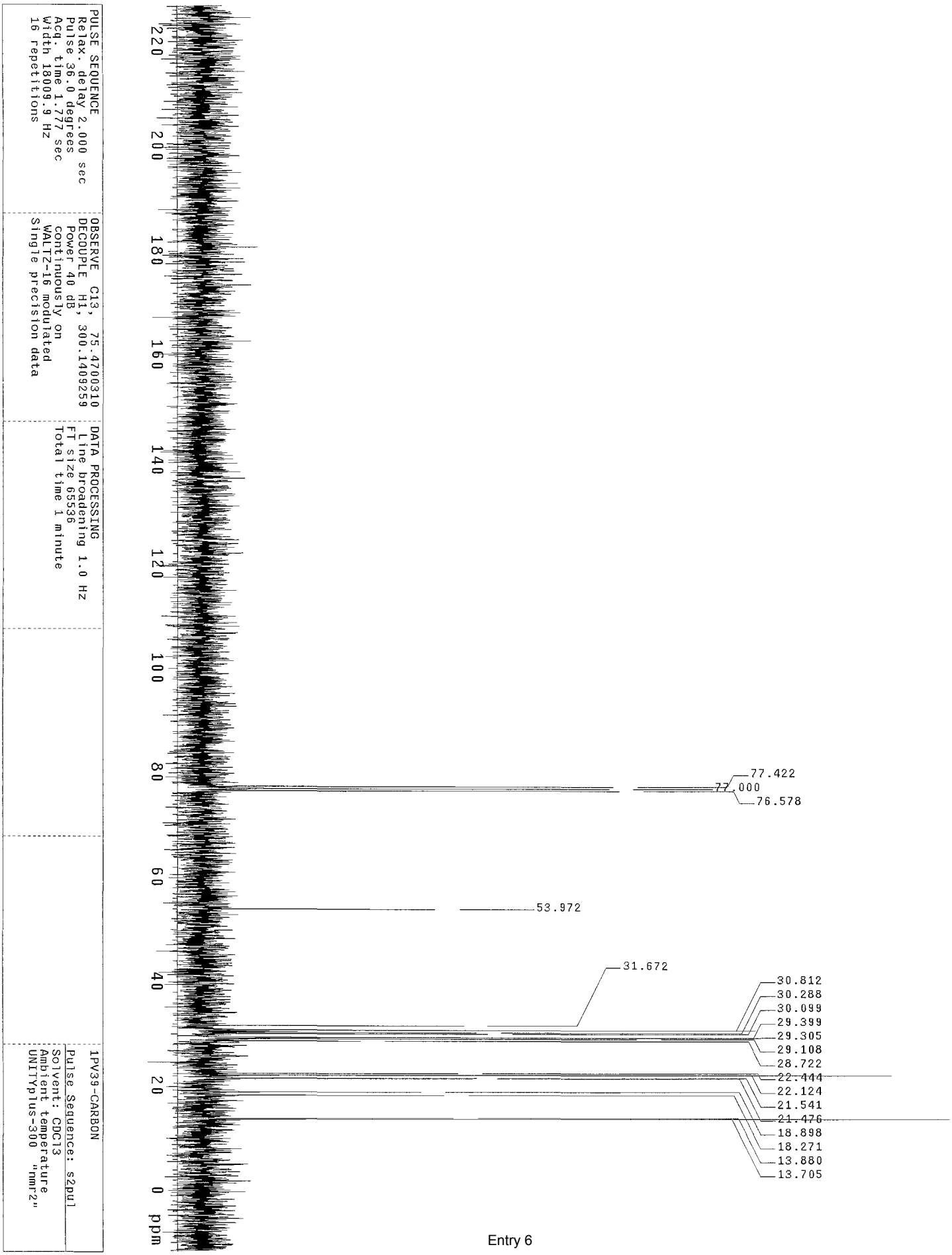
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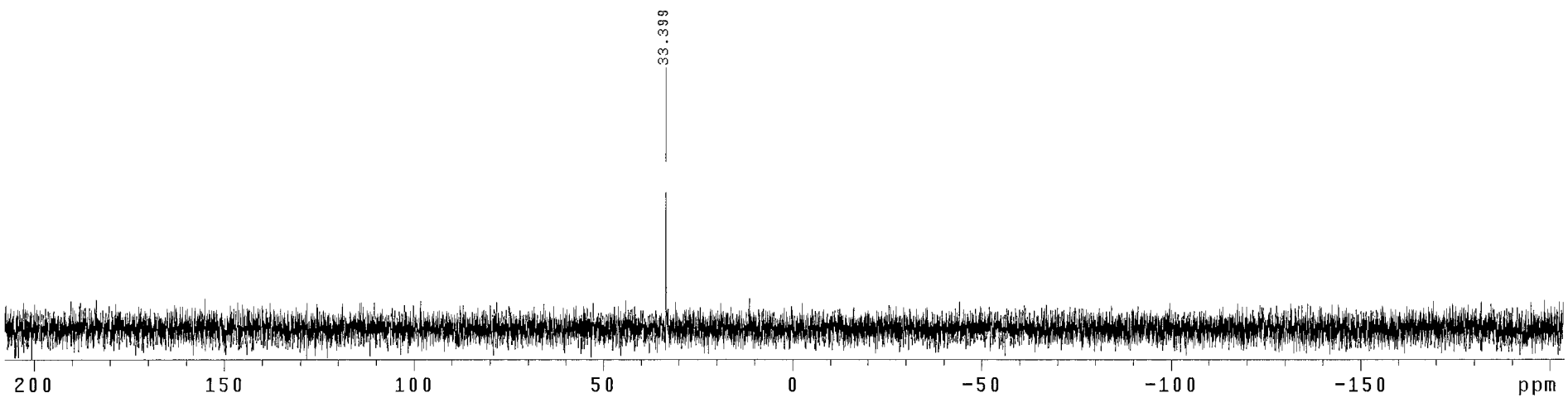
Entry 6

Entry 6



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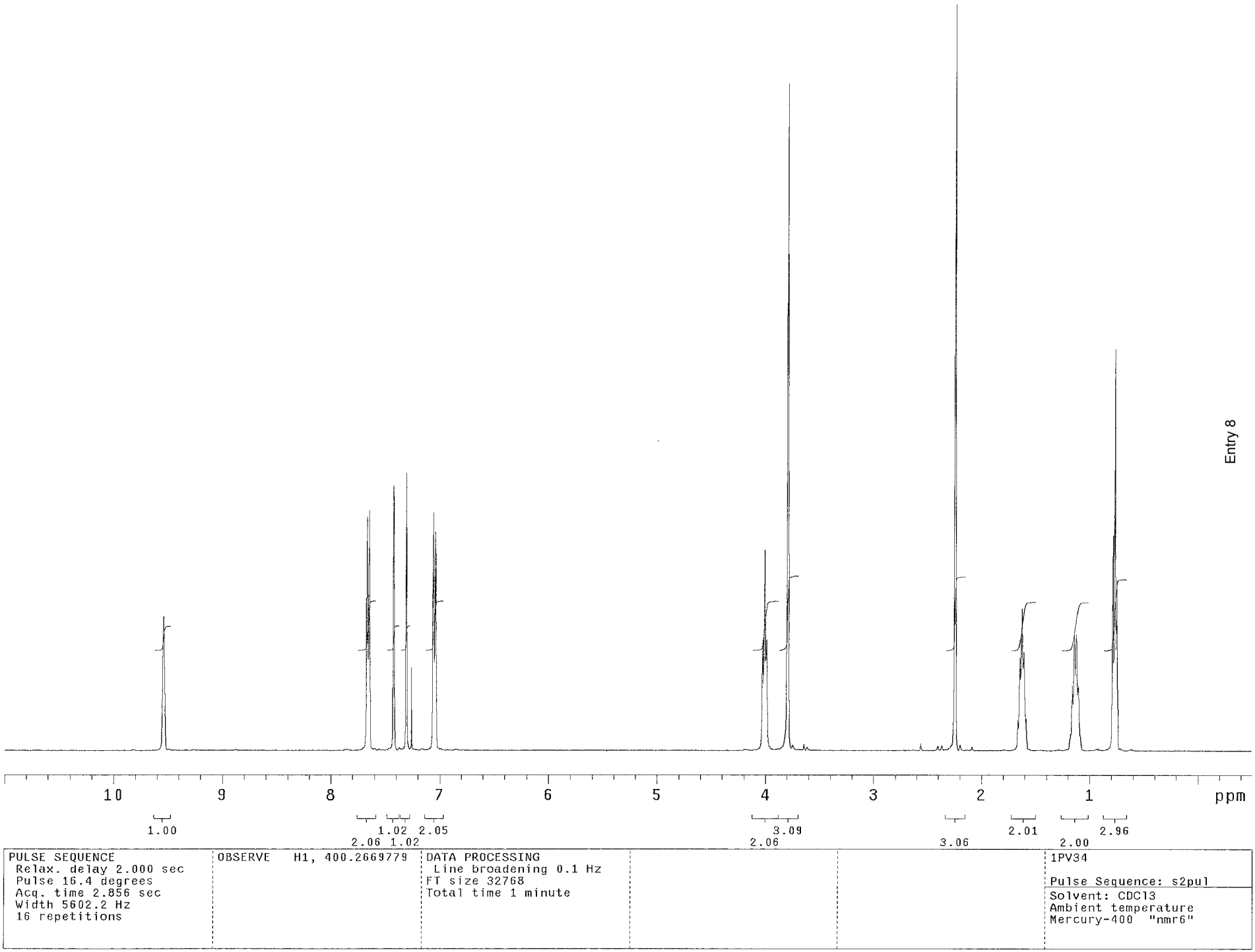
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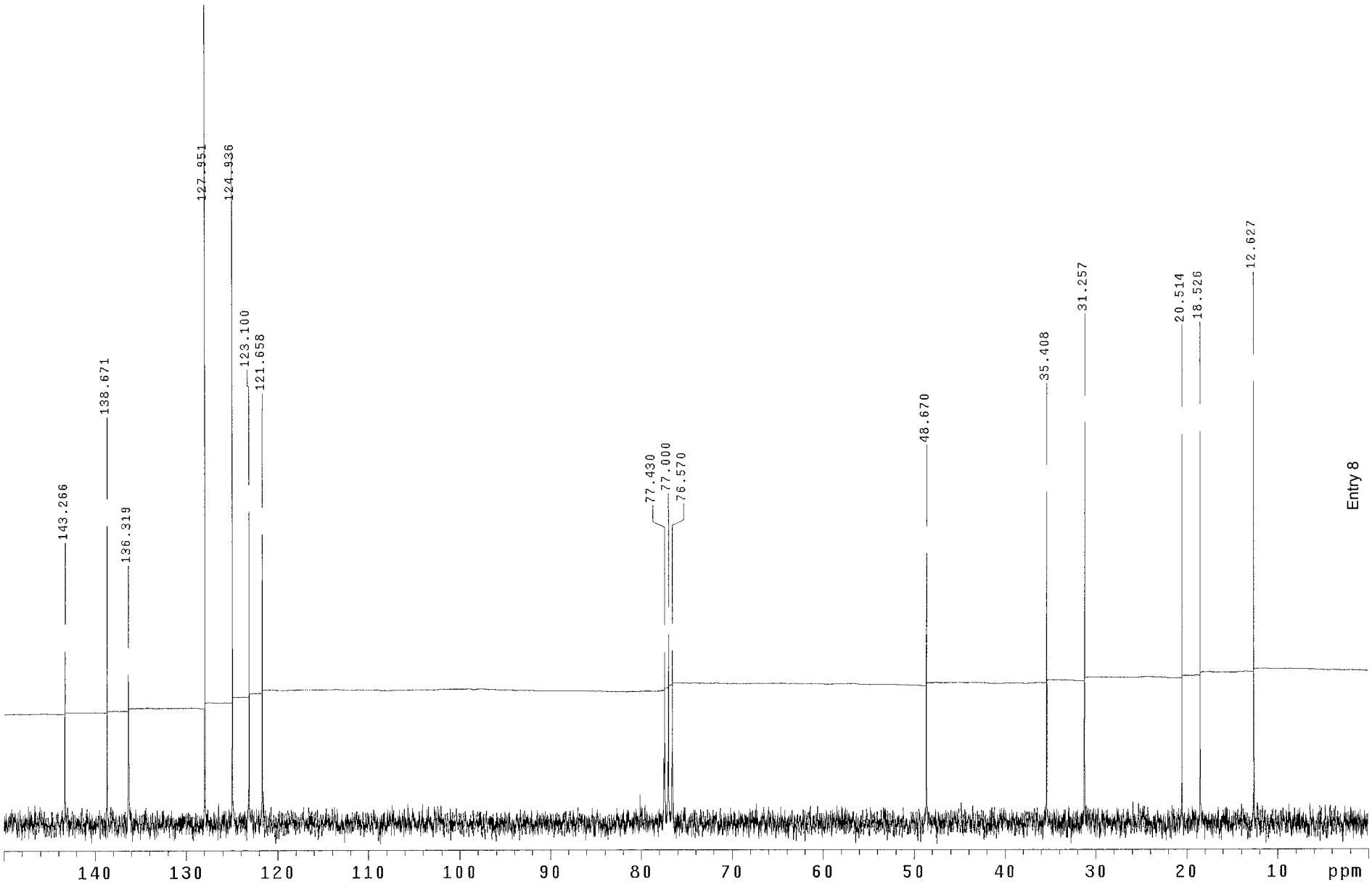

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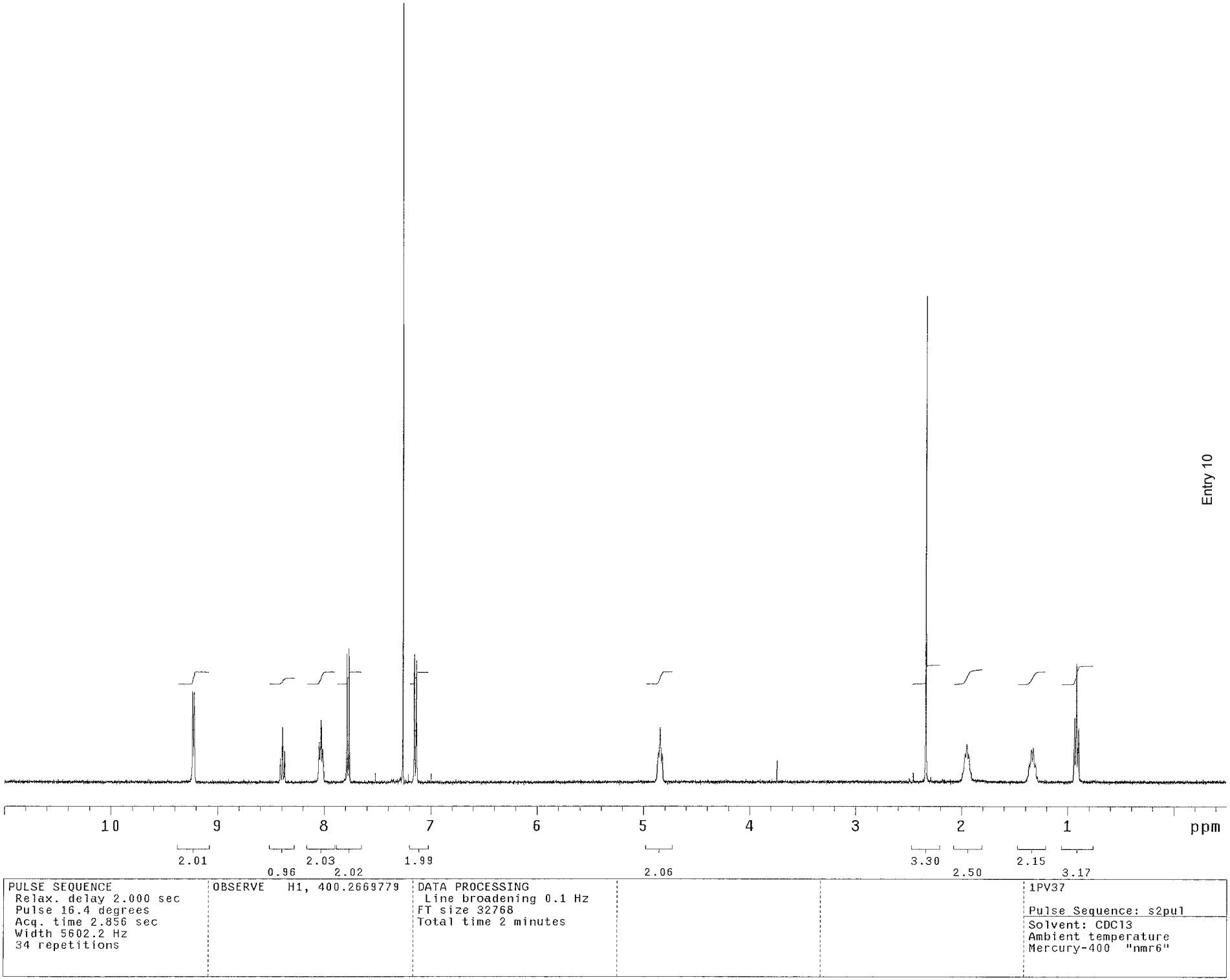
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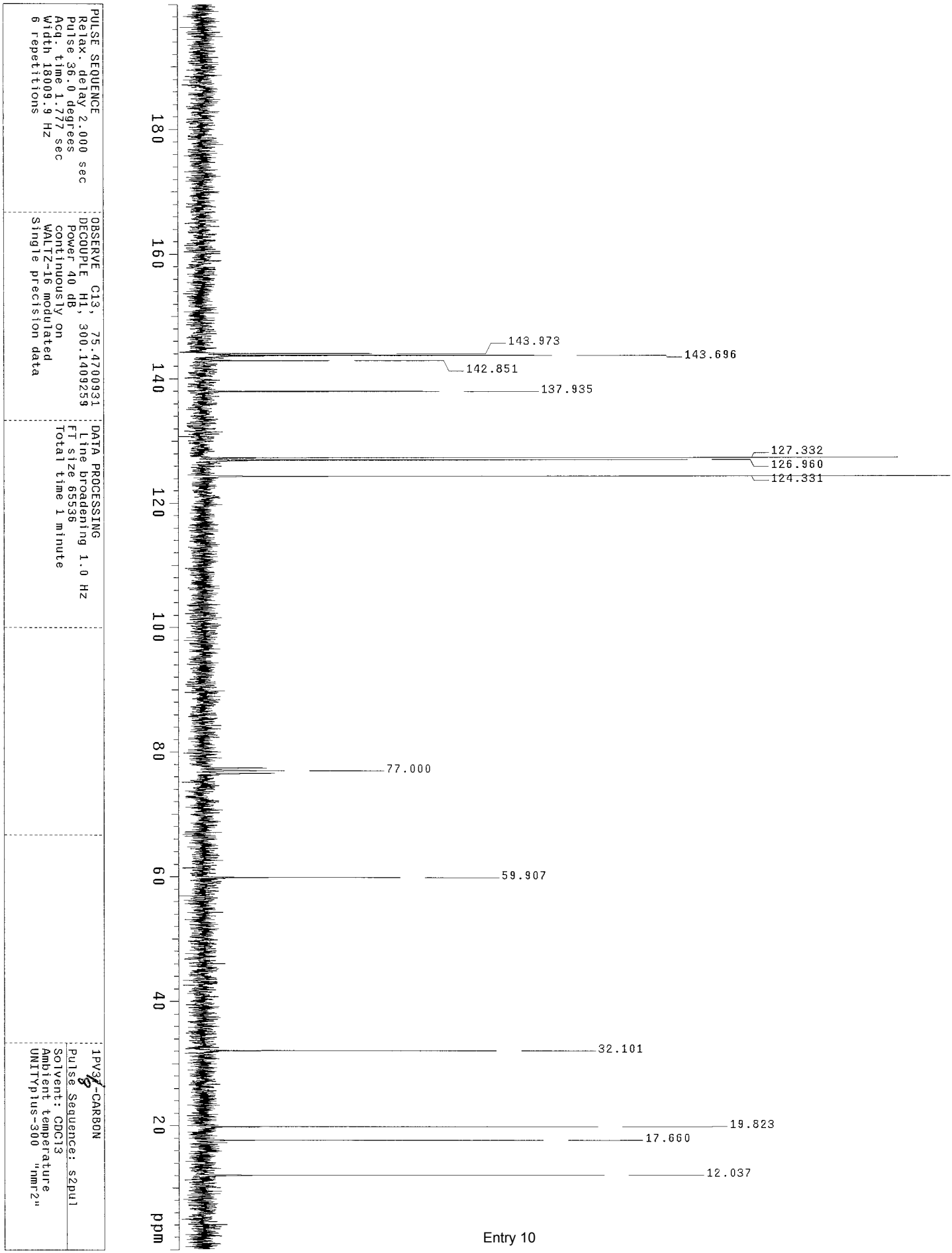
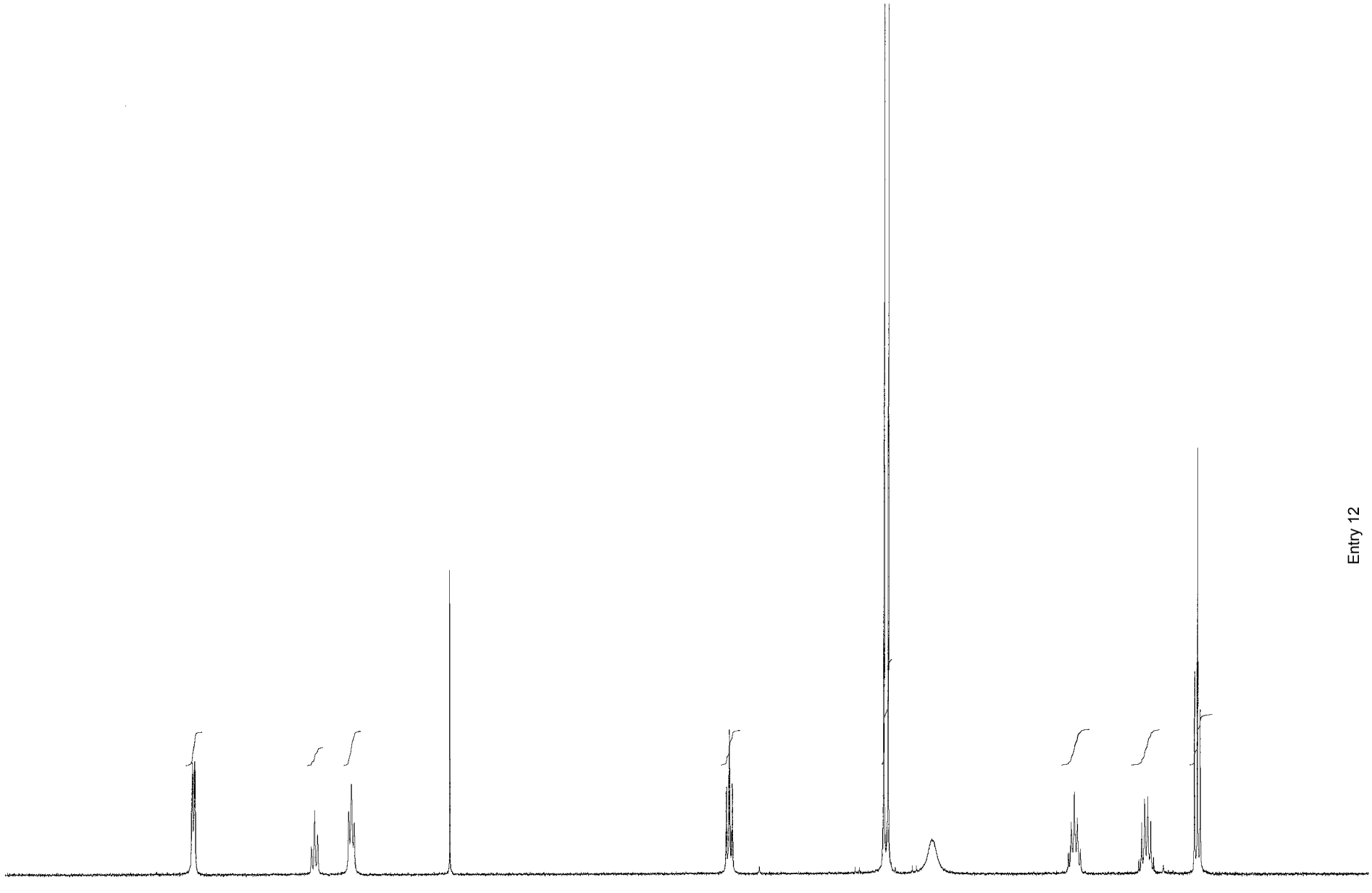
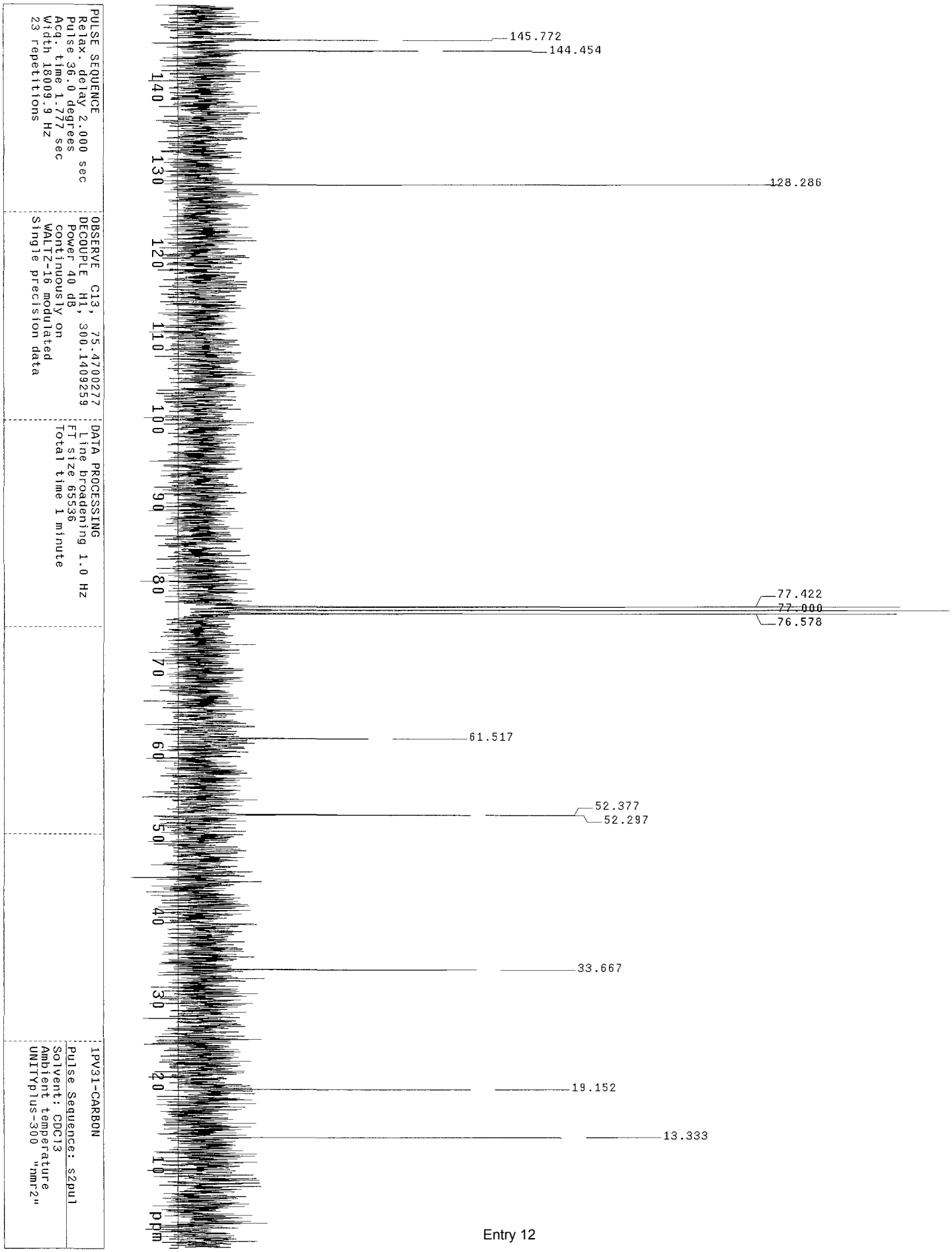
ppm
2.01
0.96
2.03
2.02
1.99
2.06
3.30
2.50
2.15
3.17
PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
34 repetitions
OBSERVE H1, 400.2669779
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 2 minutes
1PV37
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400 "nmr6"


PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
6 repetitions
OBSERVE C13, 75.4700931
DECOUPLE H1, 300.1409259
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute
1PV3/8-CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"
180
160
140
120
100
80
60
40
20
ppm
143.973
143.696
142.851
137.935
127.332
126.960
124.331
77.000
59.907
32.101
19.823
17.660
12.037

Entry 10

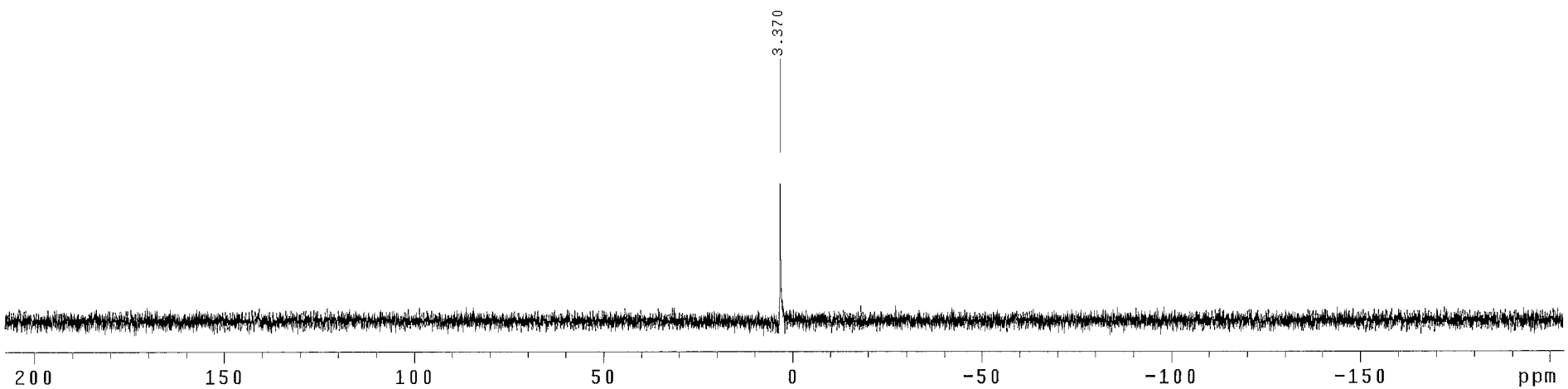


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 15.0 degrees Acq. time 3.813 sec Width 4196.4 Hz 43 repetitions	OBSERVE H1, 300.1390329	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 3 minutes			1PV31 Pulse Sequence: s2pul Solvent: CDCl3 Ambient temperature UNITYplus-300 "nmr2"

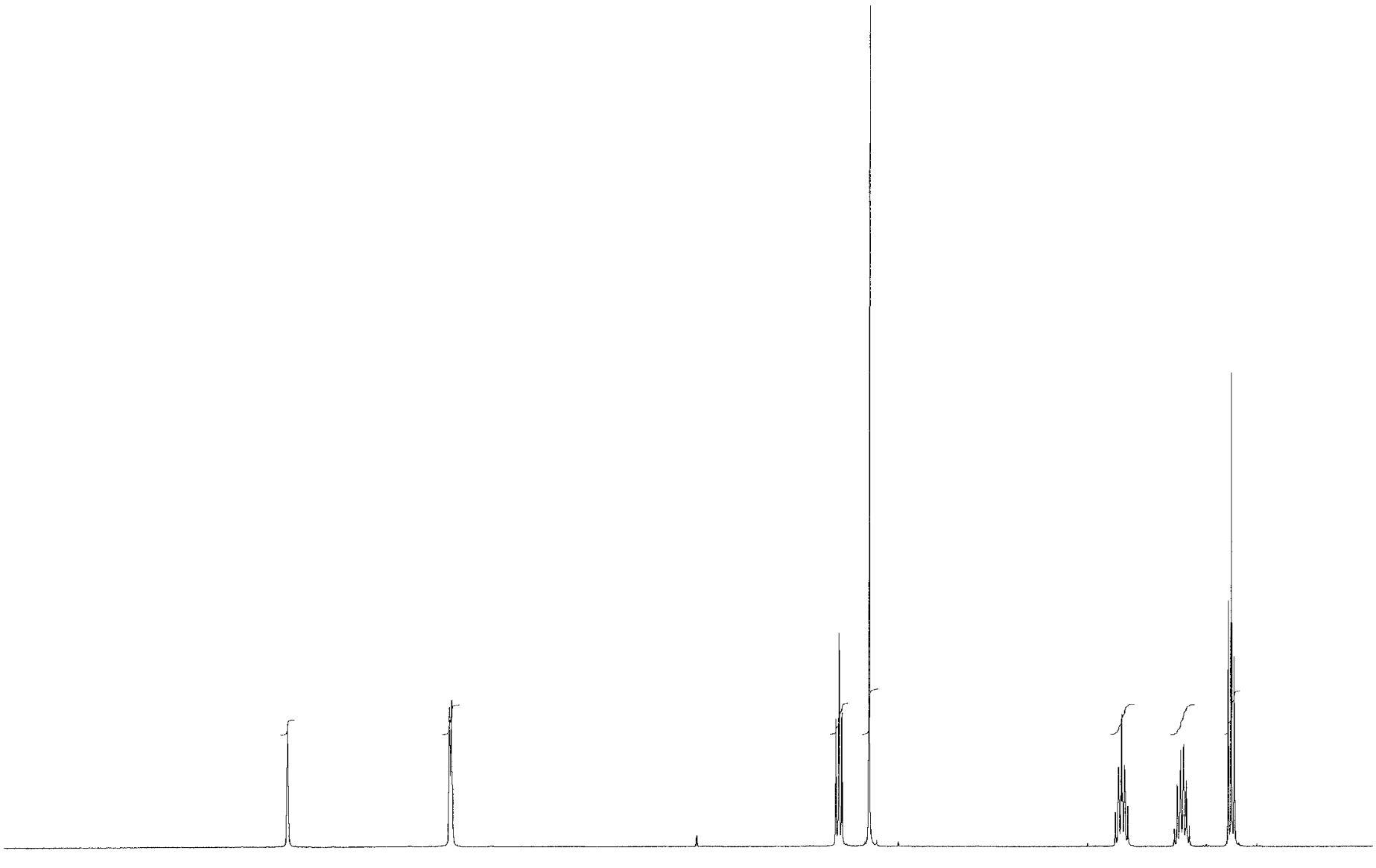

PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
23 repetitions
OBSERVE C13, 75.4700277
DECOUPLE H1, 300.1409259
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute
1PV31-CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"
145.772
144.454
128.286
77.422
77.000
76.578
61.517
52.377
52.297
33.667
19.152
13.333
ppm

Entry 12

Entry 12

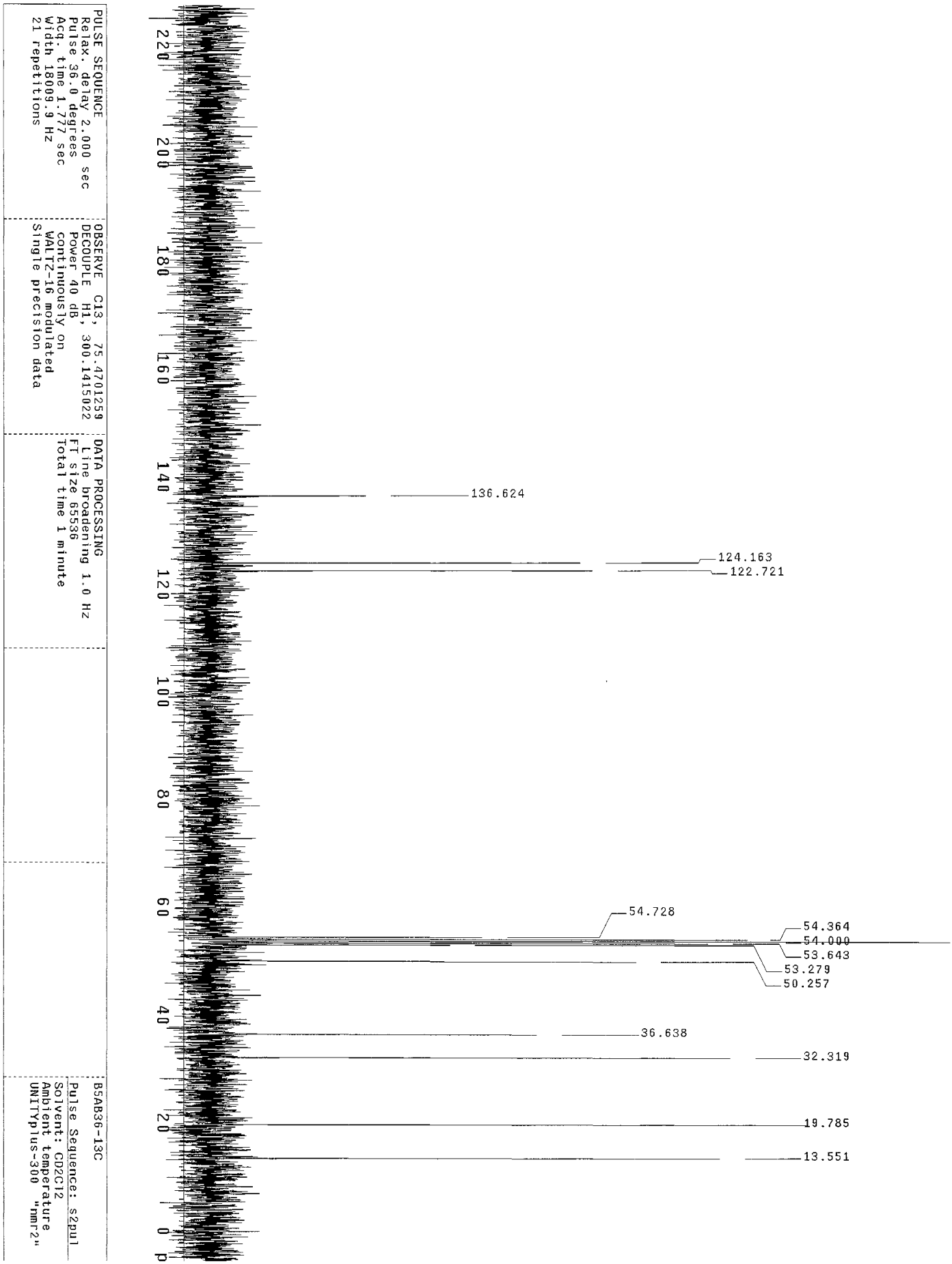


PULSE SEQUENCE Relax. delay 2.000 sec Pulse 12.9 degrees Acq. time 0.640 sec Width 50000.0 Hz 82 repetitions	OBSERVE P31, 121.4983688 DECOUPLE H1, 300.1405259 Power 40 dB continuously on WALTZ-16 modulated Single precision data	DATA PROCESSING Line broadening 2.0 Hz FT size 65536 Total time 3 minutes			1PV31-31P Pulse Sequence: s2pul Solvent: CDCl3 Ambient temperature File: 1PV31-31P UNITYplus-300 "nmr2"

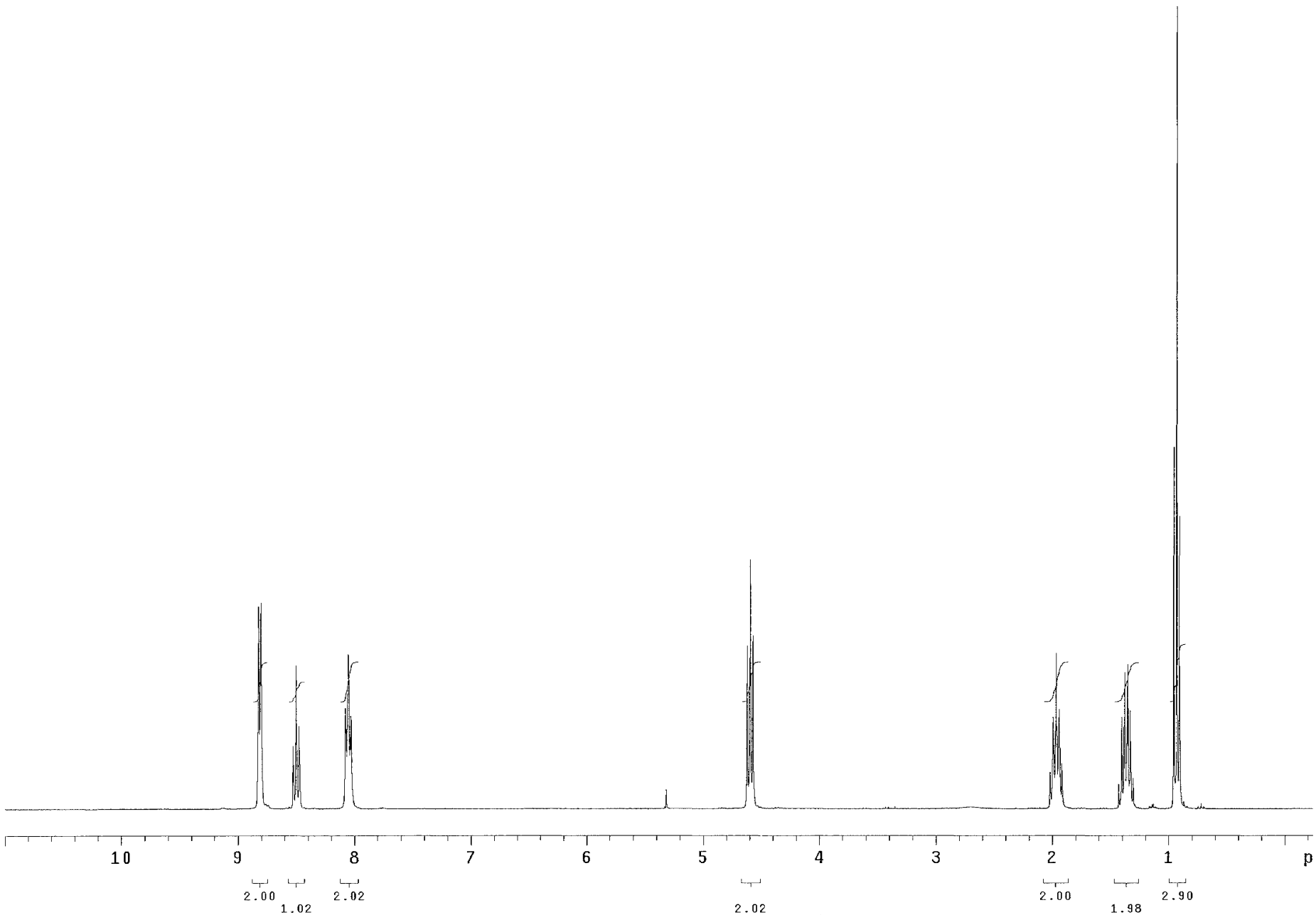


Entry 13

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 15.0 degrees Acq. time 3.813 sec Width 4196.4 Hz 5 repetitions	OBSERVE H1, 300.1396123	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B5AB36 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature UNITYplus-300 "nmr2"

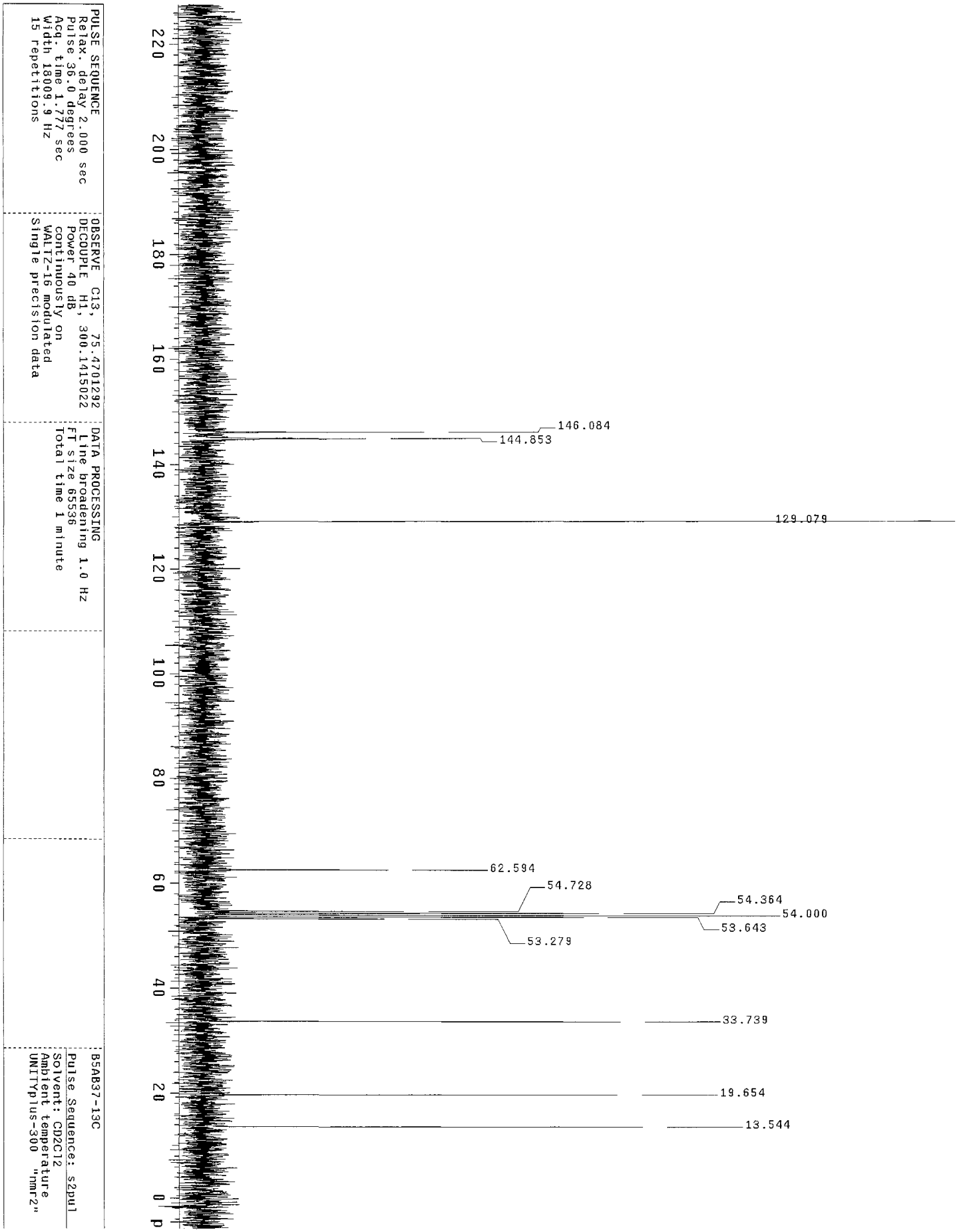

PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
width 18009.9 Hz
21 repetitions
OBSERVE C13, 75.4701259
DECOUPLE H1, 300.1415022
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute
B5AB36-13C
Pulse Sequence: s2pul
Solvent: CD2Cl2
Ambient temperature
UNITYplus-300 "nmr2"
220
200
180
160
140
120
100
80
60
40
20
0
p
136.624
124.163
122.721
54.728
54.364
54.000
53.643
53.279
50.257
36.638
32.319
19.785
13.551

Entry 13

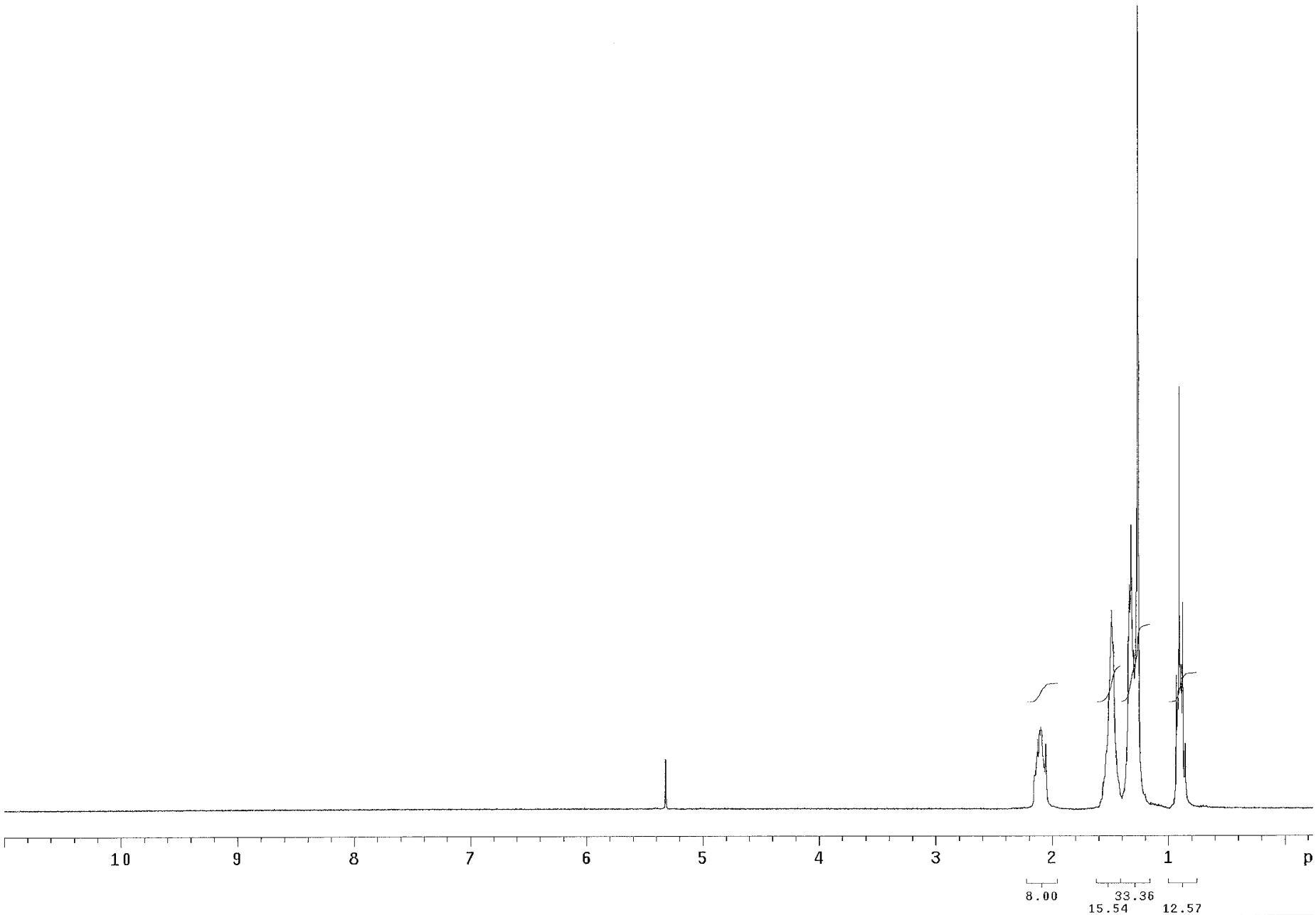


Entry 18

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 15.0 degrees Acq. time 3.813 sec Width 4196.4 Hz 13 repetitions	OBSERVE H1, 300.1396123	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B5AB42 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature UNITYplus-300 "nmr2"

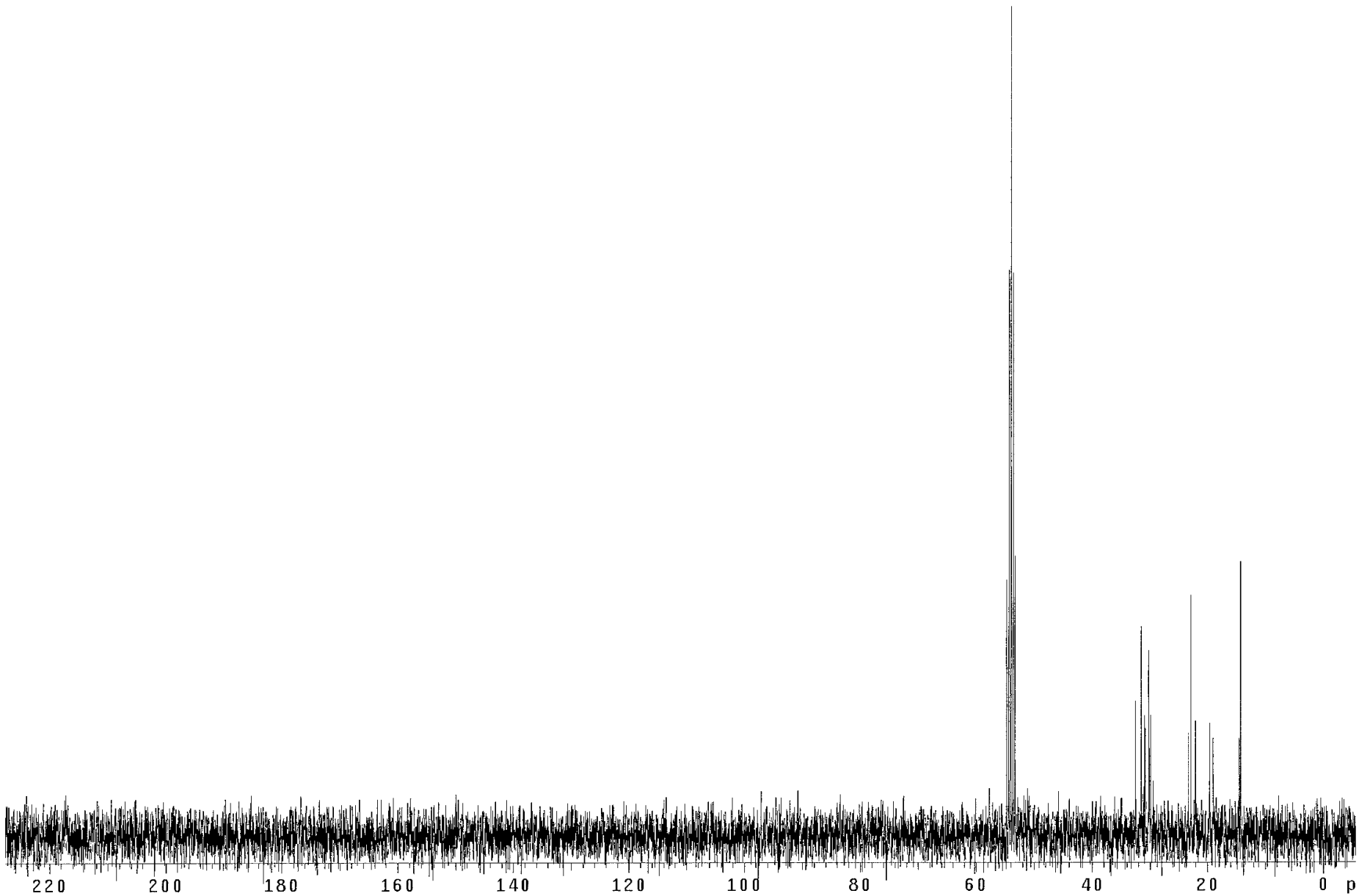

PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
15 repetitions
OBSERVE C13, 75.4701292
DECOUPLE H1, 300.1415022
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute
B5AB37-13C
Pulse Sequence: s2pul
Solvent: CD2Cl2
Ambient temperature
UNITYplus-300 "nmr2"
220
200
180
160
140
120
100
80
60
40
20
0
p
146.084
144.853
129.079
62.594
54.728
54.364
54.000
53.643
53.279
33.739
19.654
13.544

Entry 18



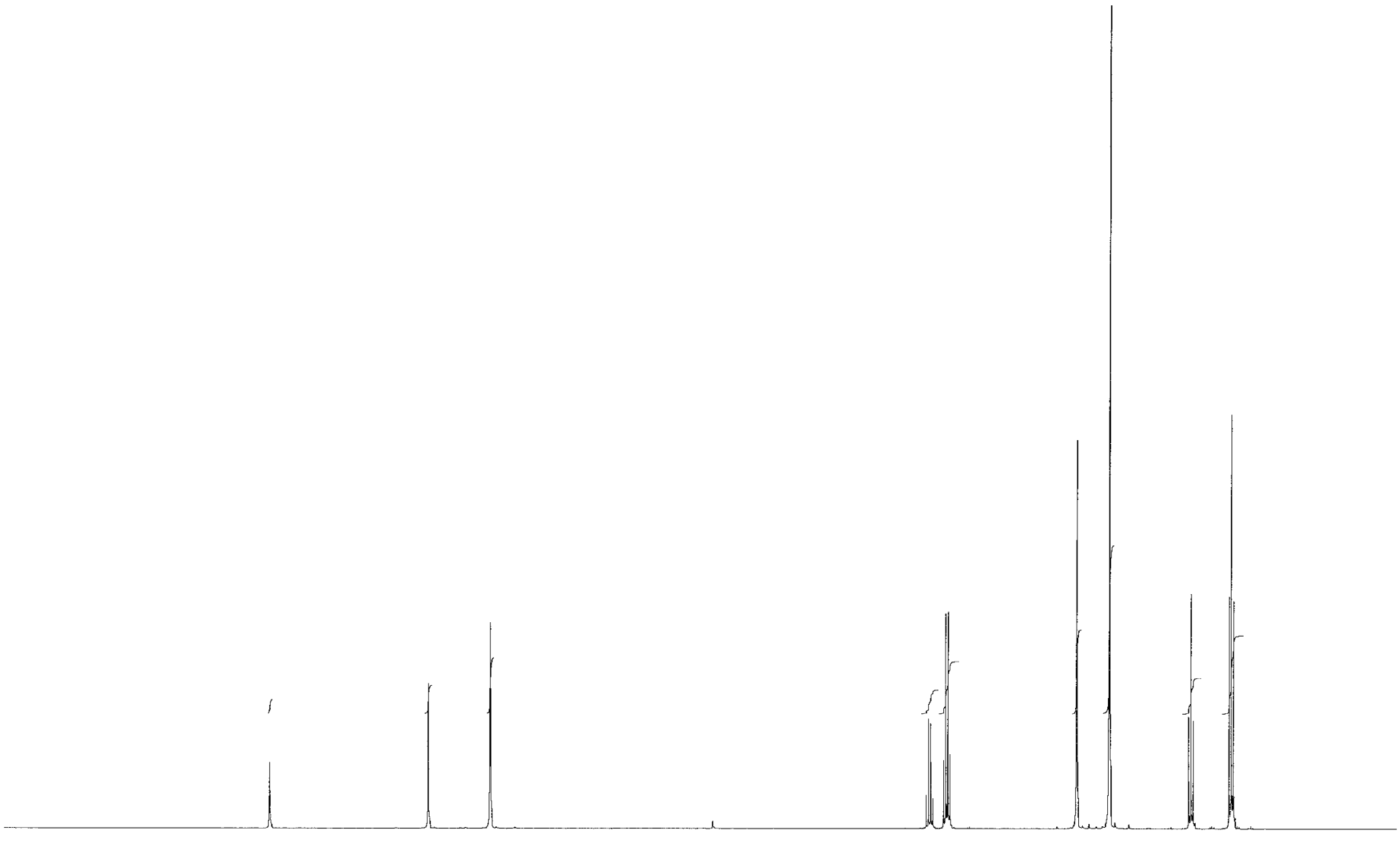
Entry 19

PULSE SEQUENCE Relax. delay 1.000 sec Pulse 15.0 degrees Acq. time 3.813 sec Width 4196.4 Hz 13 repetitions	OBSERVE H1, 300.1396125	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B6AB7 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature UNITYplus-300 "nmr2"



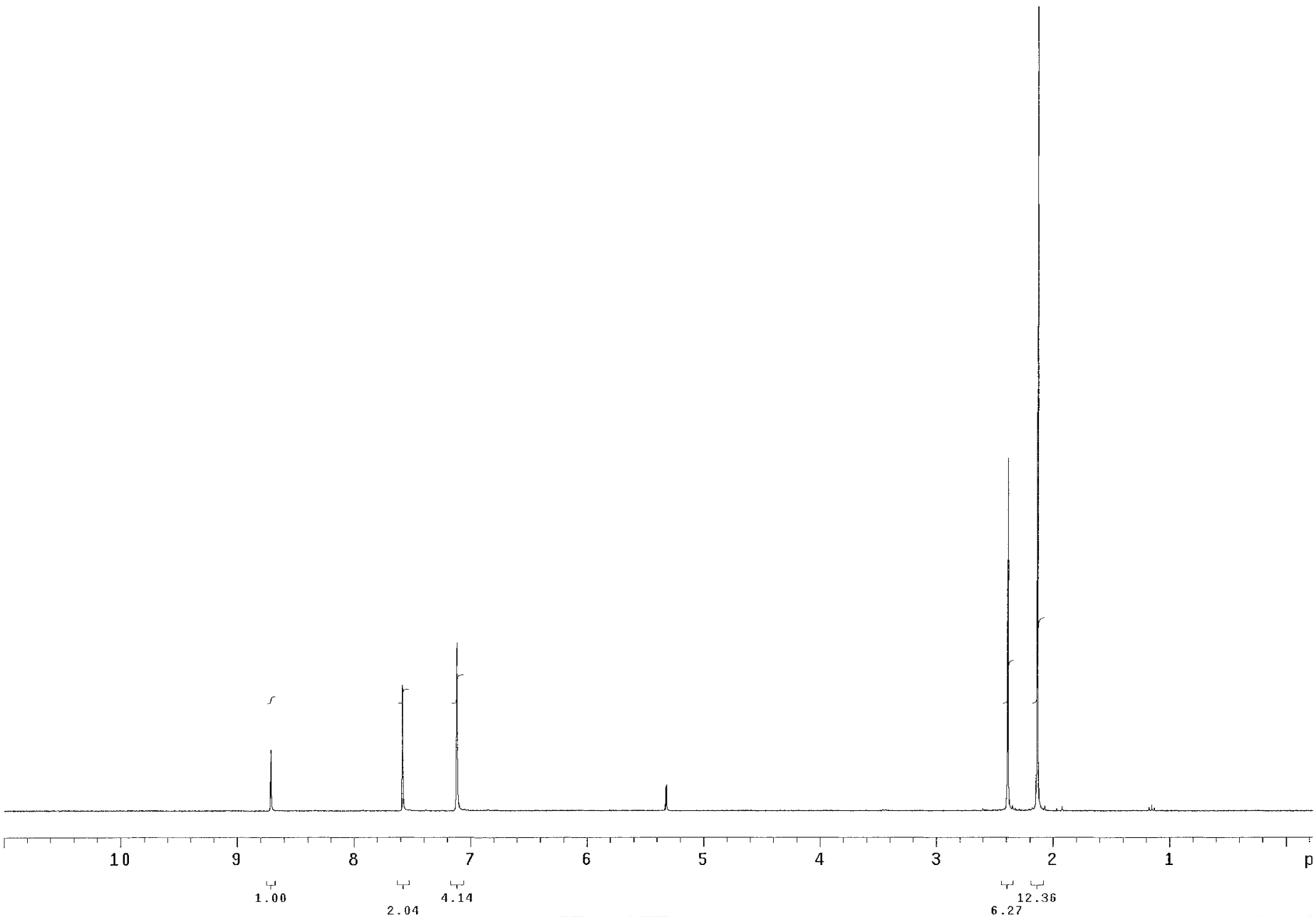
Entry 19

PULSE SEQUENCE Relax. delay 2.000 sec Pulse 36.0 degrees Acq. time 1.777 sec Width 18009.9 Hz 33 repetitions	OBSERVE C13, 75.4701193 DECOUPLE H1, 300.1415022 Power 40 dB continuously on WALTZ-16 modulated Single precision data	DATA PROCESSING Line broadening 1.0 Hz FT size 65536 Total time 2 minutes			B6AB7-13C <u>Pulse Sequence: s2pul</u> Solvent: CD2Cl2 Ambient temperature UNITYplus-300 "nmr2"



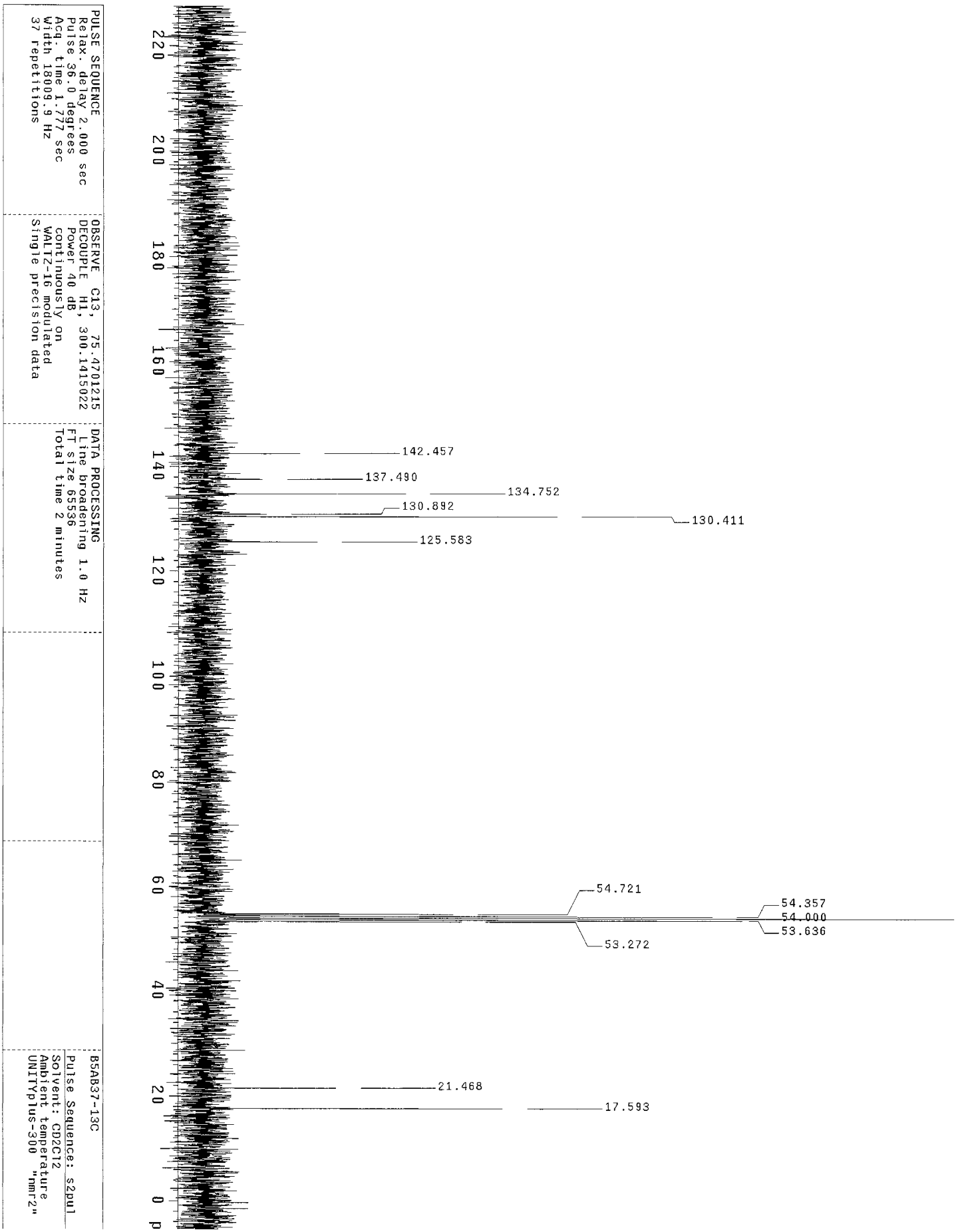
Entry 21 - Reaction Aliquot

PULSE SEQUENCE Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 30 repetitions	OBSERVE H1, 400.2677522	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 2 minutes			B5AB278 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature Mercury-400 "nmr6"

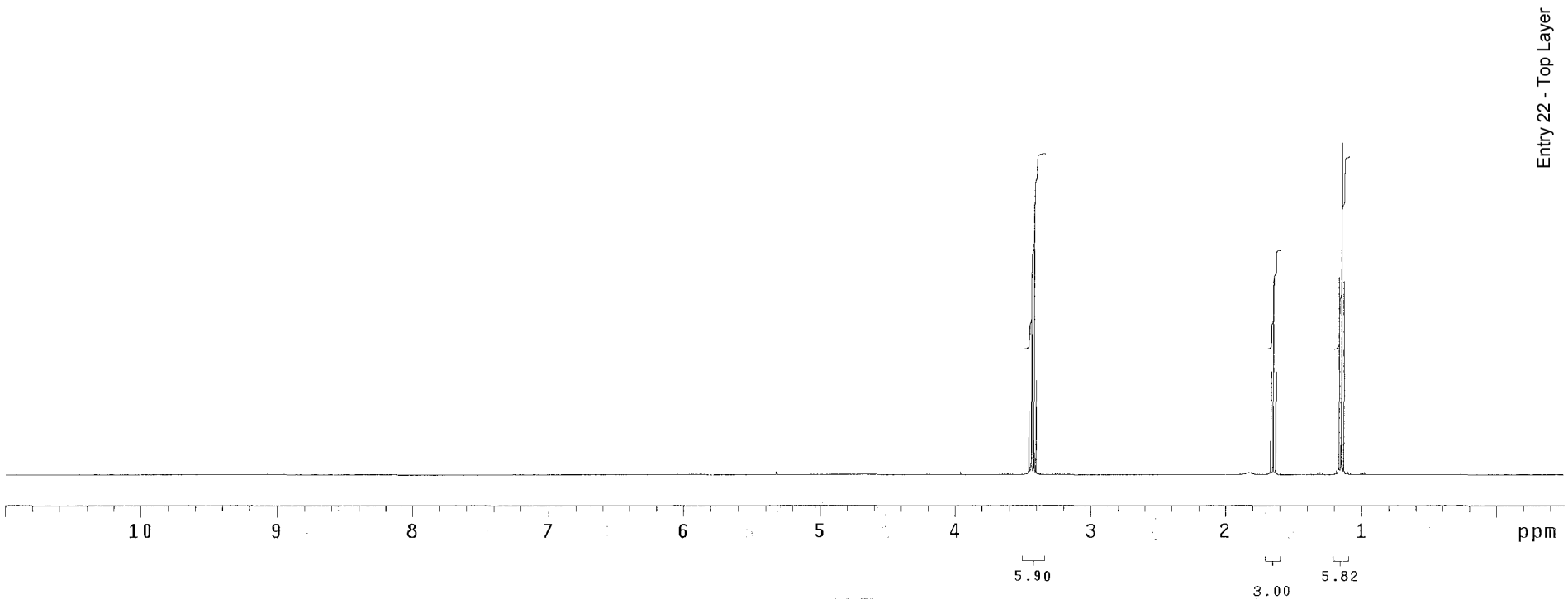


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 15.0 degrees Acq. time 3.813 sec Width 4196.4 Hz 8 repetitions	OBSERVE H1, 300.1396123	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B5AB46 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature UNITYplus-300 "nmr2"

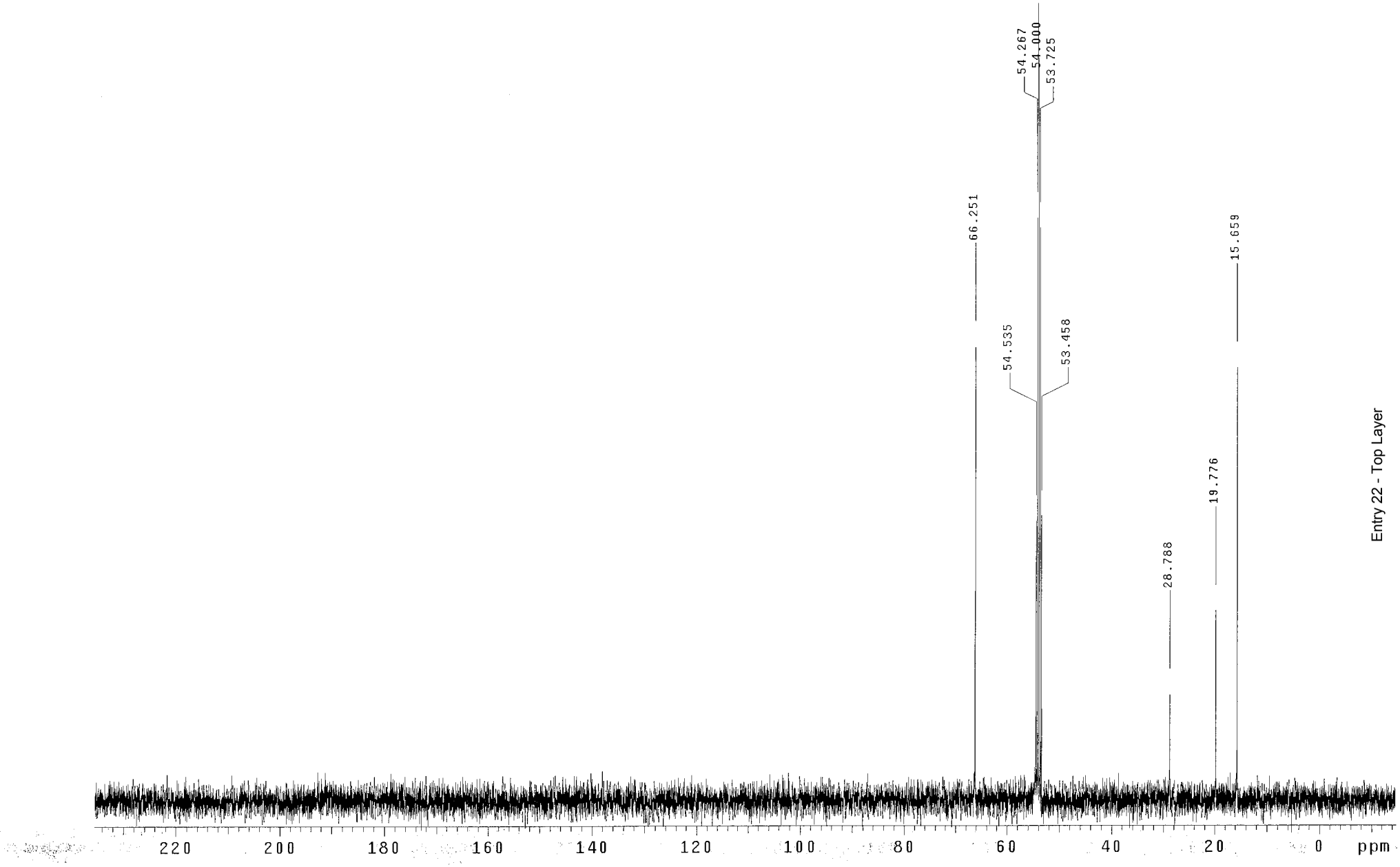
Entry 21 - Isolated Product



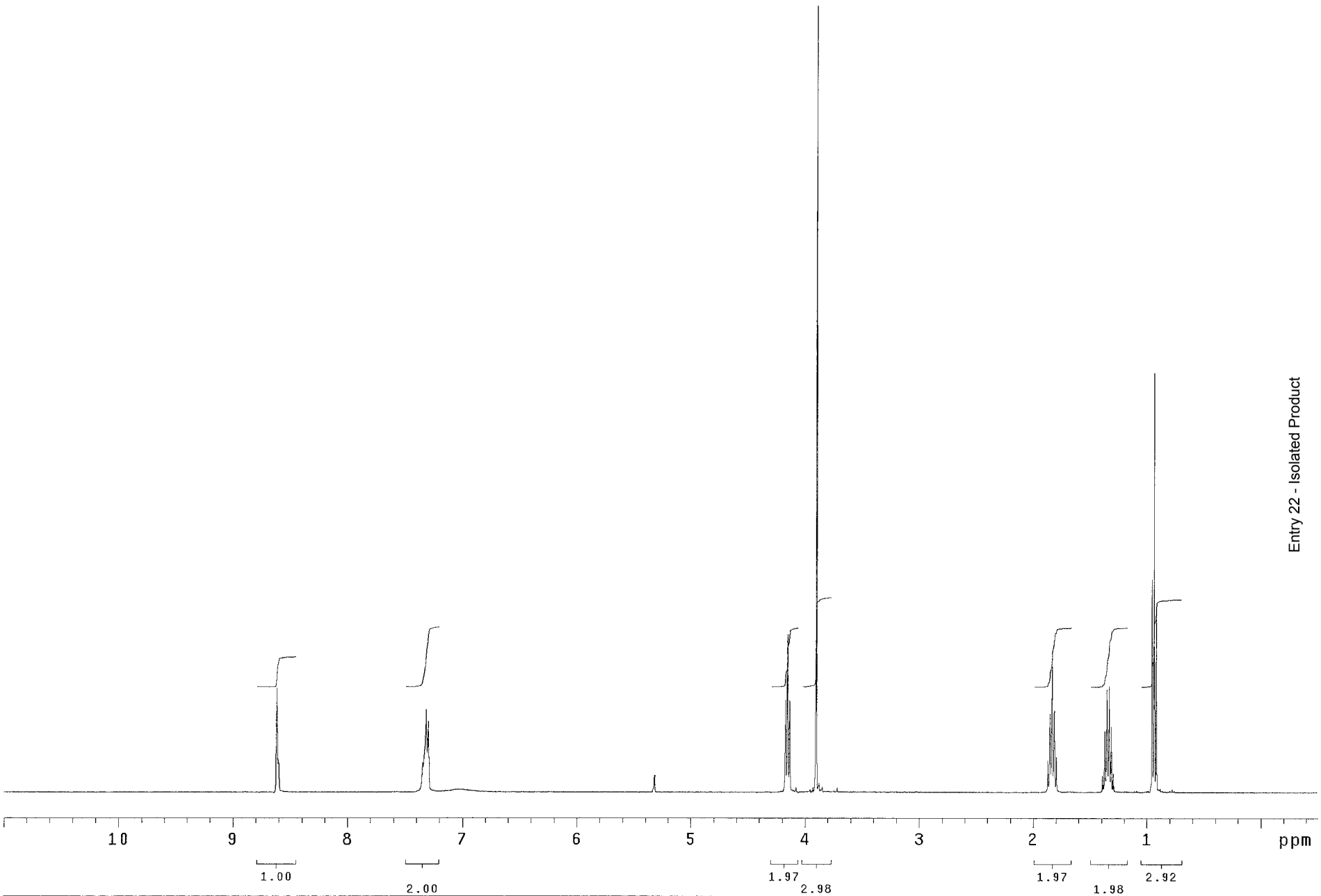
Entry 21 - Isolated Product



PULSE SEQUENCE Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 13 repetitions	OBSERVE H1, 400.2677522	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B5AB201-toplayer Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature Mercury-400 "nmr6"



PULSE SEQUENCE Relax. delay 2.000 sec Pulse 22.5 degrees Acq. time 1.280 sec Width 25188.9 Hz 32 repetitions	OBSERVE C13, 100.6473420 DECOUPLE H1, 400.2697641 Power 38 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 1.0 Hz FT size 65536 Total time 1 minutes			B5AB201-TL-13C Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature Mercury-400 "nmr6"



PULSE SEQUENCE Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 7 repetitions	OBSERVE H1, 400.2677522	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 1 minute			B5AB201 Pulse Sequence: s2pul Solvent: CD2Cl2 Ambient temperature Mercury-400 "nmr6"

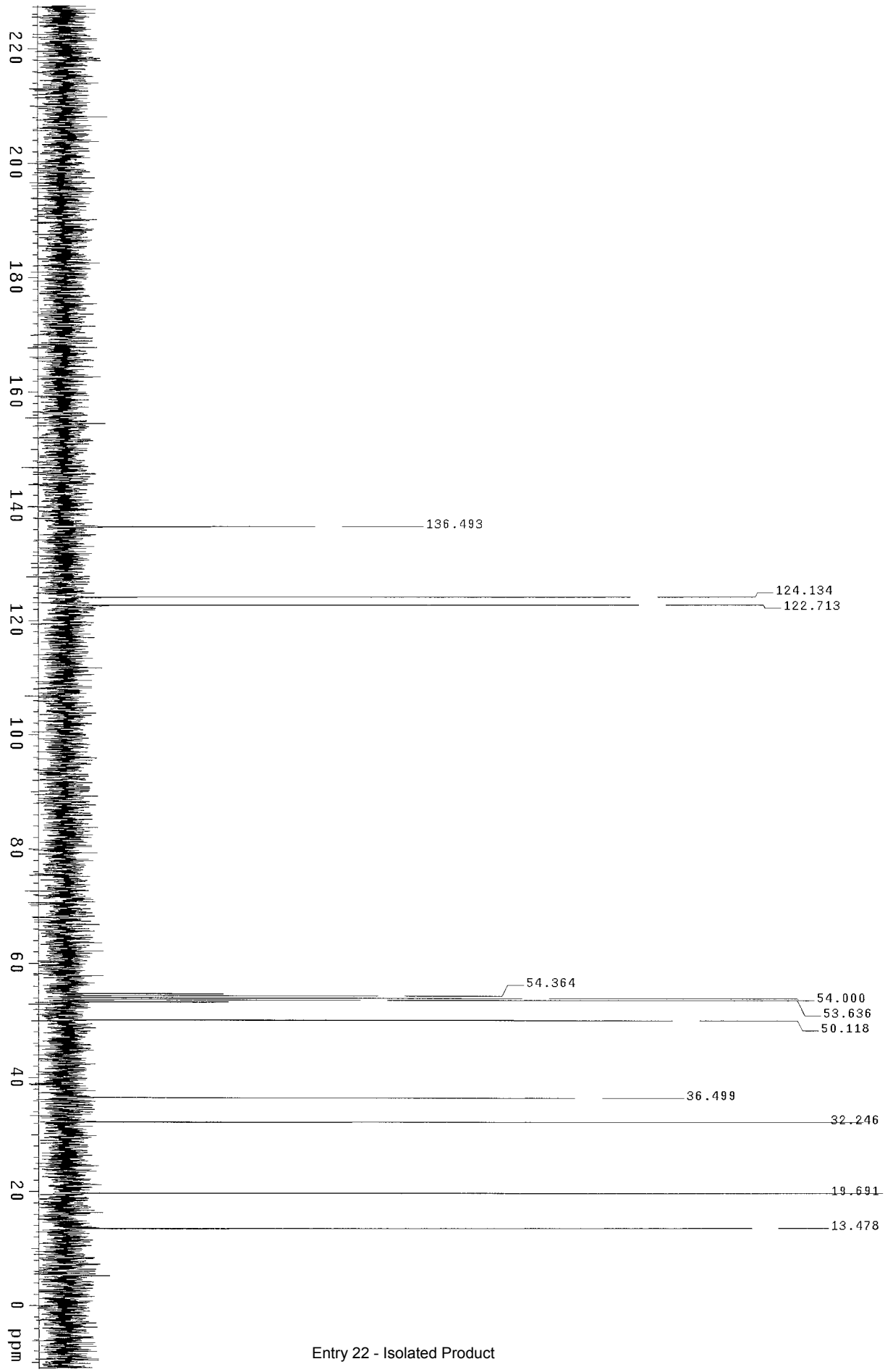
PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
22 repetitions

OBSERVE C13, 75.4701309
DECOUPLE H1, 300.1415022
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data

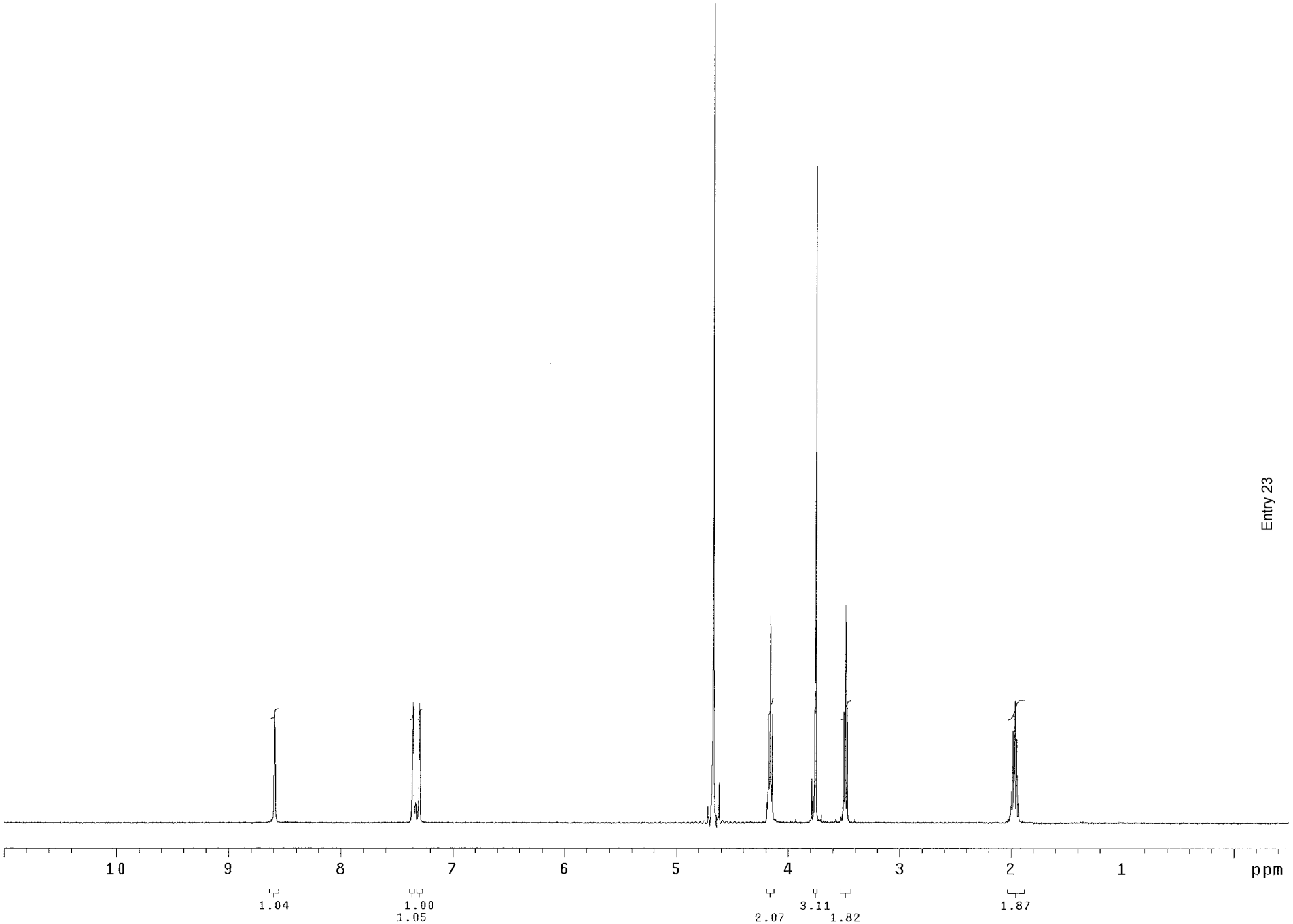
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 minute

B5AB201-13C

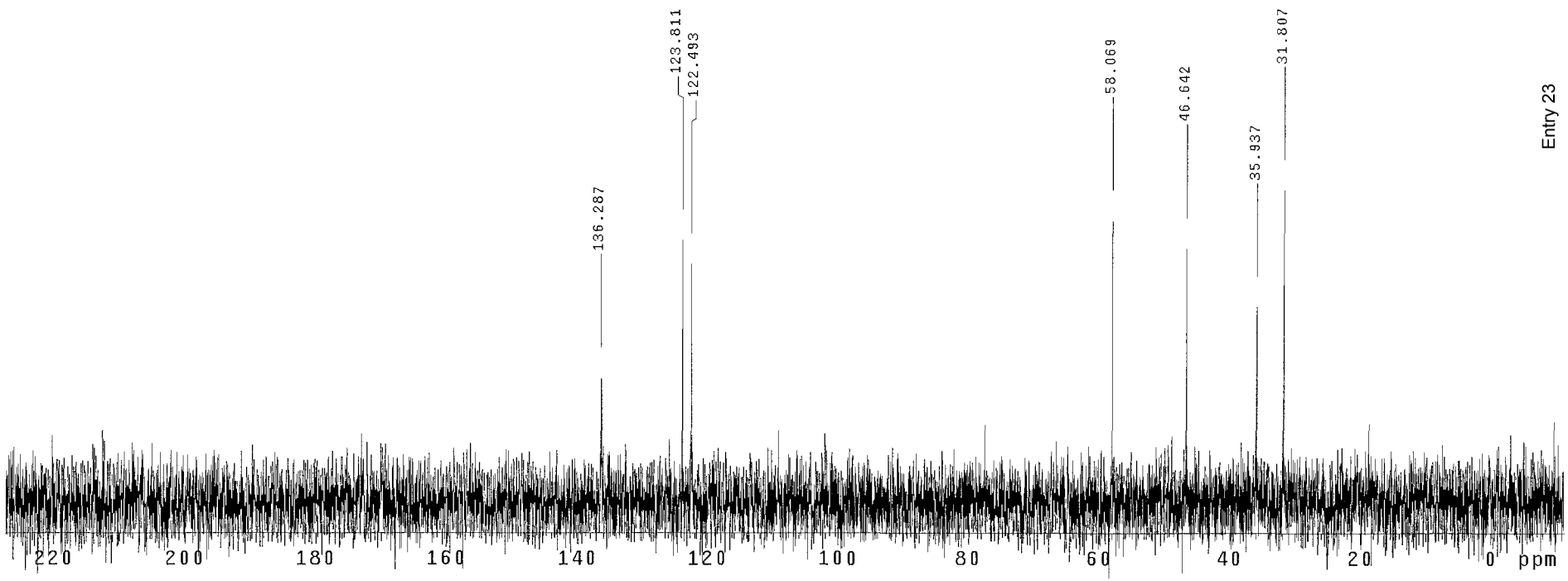
Pulse Sequence: s2pul
Solvent: CD2Cl2
Ambient temperature
UNITYplus-300 "nmr2"



Entry 22 - Isolated Product



PULSE SEQUENCE Relax. delay 2.000 sec Pulse 16.4 degrees Acq. time 2.856 sec Width 5602.2 Hz 30 repetitions	OBSERVE H1, 400.2679973	DATA PROCESSING Line broadening 0.1 Hz FT size 32768 Total time 2 minutes			B5AB295-D2O Pulse Sequence: s2pul Solvent: D2O Ambient temperature Mercury-400 "nmr6"



PULSE SEQUENCE Relax. delay 2.000 sec Pulse 36.0 degrees Acq. time 1.777 sec Width 18009.9 Hz 24 repetitions	OBSERVE C13, 75.4701938 DECOUPLE H1, 300.1416973 Power 40 dB continuously on WALTZ-16 modulated Single precision data	DATA PROCESSING Line broadening 1.0 Hz FT size 65536 Total time 1 minutes			13C OBSERVE Pulse Sequence: s2pul Solvent: D2O Ambient temperature UNITYplus-300 "nmr2"