Facile and efficient synthesis of 1-haloalkynes via DBU-mediated reaction of terminal alkynes and *N*-haloimides under mild conditions

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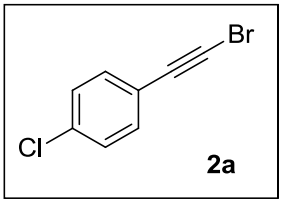
I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 ℃ on a Varian 500 MHz and 125 MHz, respectively, and TMS as internal standard.High resolution mass spectra (HRMS) were recorded on Bruck microTof by using ESI method.

II. Synthesis and analytical data of 2-4

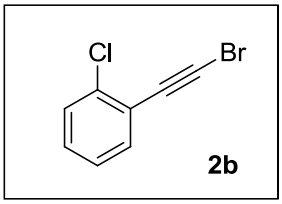
General procedure for the preparation of **2** (**2a** as anexample): To a solution of 1-chloro-4-ethynylbenzene**1a** (136.6 mg, 1.0 mmol) in MeCN (2.0 mL) was added NBS (195.8 mg, 1.1 mmol) and DBU (0.159 mL, 1.1 mmol). The mixture was stirred at room temperature for 1 min. The reaction mixture was poured into water and then extracted with CH_2Cl_2 (3 × 10 mL). The combined organic phase was washed with water (3 × 10 mL), filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography (silica gel, petroleum ether as eluent) to give **2a** (213 mg, 99%) as a white solid.

1-(bromoethynyl)-4-chlorobenzene (2a)



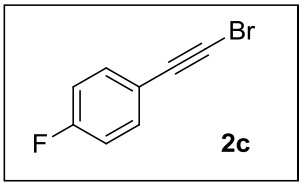
White solid. m.p. 76-78 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 51.0, 78.9, 121.1, 128.7, 133.2, 134.8; HRMS (ESI) m/z calcd for C_8H_4BrCl $[M+H]^+$: 214.9263; found: 214.9259.

1-(bromoethynyl)-2-chlorobenzene (2b)



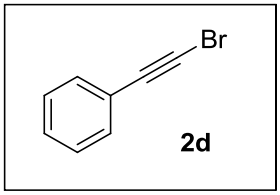
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$):δ = 7.17-7.23 (m, 1H), 7.24-7.26 (m, 1H), 7.36-7.38 (m, 1H), 7.45-7.47 (m, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 55.3, 76.9, 122.5, 126.4, 129.2, 129.6, 133.8, 136.3; HRMS (ESI) m/z calcd for C_8H_4BrCl $[M+H]^+$: 214.9263; found: 214.9268.

1-(bromoethynyl)-4-fluorobenzene (2c)



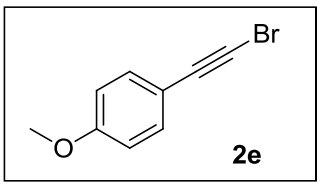
White solid. m.p. 39-41 ℃.^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.00 (t, *J* = 8.5 Hz, 2H), 7.41-7.44 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 49.4, 78.8, 115.6, 118.6, 133.8, 163.5; HRMS (ESI) m/z calcd for C_8H_4BrF $[M+H]^+$: 198.9559; found: 198.9564.

(bromoethynyl)benzene (2d)



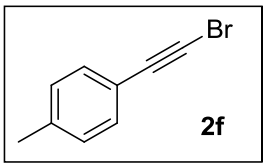
Colourless oil.^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29-7.35 (m, 3H), 7.44 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 49.7, 80.0, 122.6, 128.3, 128.7, 132.0; HRMS (ESI) m/z calcd for C_8H_5Br $[M+H]^+$: 180.9653; found: 180.9657.

1-(bromoethynyl)-4-methoxybenzene (2e)



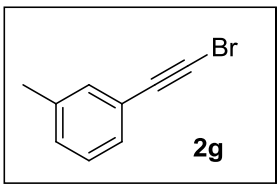
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.79 (s, 3H), 6.82 (d, *J* = 8.5 Hz, 2H), 7.38 (t, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 47.8, 55.2, 79.9, 113.9, 114.7, 133.4, 159.8; HRMS (ESI) m/z calcd for C_9H_7BrO $[M+H]^+$: 210.9759; found: 210.9752.

1-(bromoethynyl)-4-methylbenzene (2f)



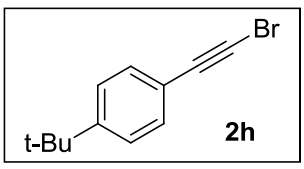
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.34 (s, 3H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.33 (t, *J* = 4.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 21.5, 48.7, 80.1, 119.6, 129.0, 131.8, 138.8; HRMS (ESI) m/z calcd for C_9H_7Br $[M+H]^+$: 194.9809; found: 194.9813.

1-(bromoethynyl)-3-methylbenzene (2g)



Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.30 (s, 3H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.16-7.25 (m, 1H), 7.29 (d, *J* = 11.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 21.1, 49.2, 80.2, 122.4, 128.2, 129.0, 129.6, 132.5, 138.0; HRMS (ESI) m/z calcd for C_9H_7Br $[M+H]^+$: 194.9809; found: 194.9805.

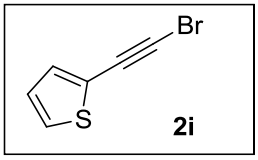
1-(bromoethynyl)-4-(tert-butyl)benzene (2h)



Colourless oil.^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.29 (s, 9H), 7.31 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 31.1, 34.7, 48.7, 80.1,

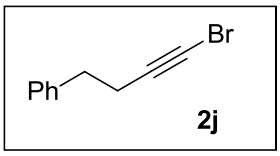
119.6, 125.3, 131.7, 131.8, 151.9; HRMS (ESI) m/z calcd for $C_{12}H_{13}Br$ $[M+H]^+$: 237.0279; found: 237.0284.

2-(bromoethynyl)thiophene (2i)



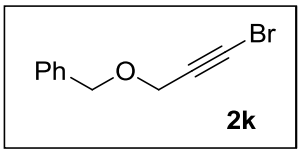
Yellow oil. m.p. 76-78 ℃.^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.95-6.97 (m, 1H), 7.23-7.25 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 54.1, 73.4, 122.7, 126.8, 127.3, 132.8; HRMS (ESI) m/z calcd for C_6H_3BrS $[M+H]^+$: 186.9217; found: 186.9215.

(4-bromobut-3-yn-1-yl)benzene(2j)



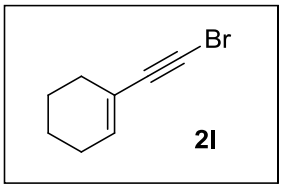
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.49 (t, *J* = 7.5 Hz, 2H), 2.83 (t, *J* = 7.5 Hz, 2H), 7.20-7.25 (m, 3H), 7.30 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 21.8, 34.6, 38.7, 79.6, 126.4, 128.3, 128.4, 140.2; HRMS (ESI) m/z calcd for $C_{10}H_9Br$ $[M+H]^+$: 208.9966; found: 208.9962.

(((3-bromoprop-2-yn-1-yl)oxy)methyl)benzene (2k)



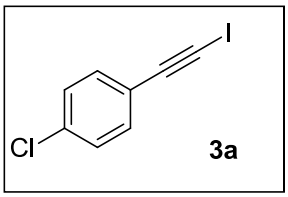
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 4.17 (s, 2H), 4.57 (s, 2H), 7.33 (m, 1H), 7.33-7.34 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 46.1, 57.9, 71.6, 76.1, 127.9, 128.0, 128.4, 137.1; HRMS (ESI) m/z calcd for $C_{10}H_9BrO$ $[M+H]^+$: 224.9915; found: 224.9913.

1-(bromoethynyl)cyclohex-1-ene (2l)



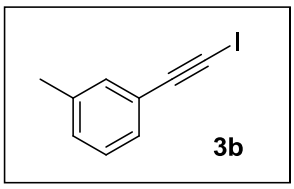
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.55-1.65 (m, 4H), 2.06-2.11 (m, 4H), 6.14-6.15 (m, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 21.3, 22.1, 25.6, 28.8, 46.2, 81.8, 120.4, 136.4; HRMS (ESI) m/z calcd for C_8H_9Br $[M+H]^+$: 184.9966; found: 184.9970.

1-chloro-4-(iodoethynyl)benzene (3a)



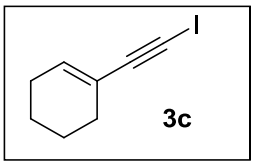
White solid. m.p. 81-83 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28 (d, *J* = 8.5 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 7.7, 92.9, 121.8, 128.6, 133.5, 134.9; HRMS (ESI) m/z calcd for C_8H_4BrI $[M+H]^+$: 262.9124; found: 262.9129.

1-(iodoethynyl)-3-methylbenzene (3b)



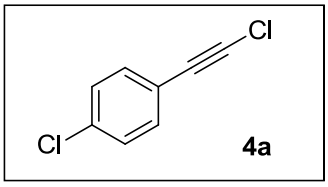
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.30 (s, 3H), 7.06 (s, 1H), 7.12 (d, *J* = 7.0 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 5.6, 21.2, 94.3, 123.1, 128.1, 129.3, 129.7, 132.8, 137.9; HRMS (ESI) m/z calcd for $C_9H_7I[M+H]^+$: 242.9671; found: 242.9679.

1-(iodoethynyl)cyclohex-1-ene (3c)



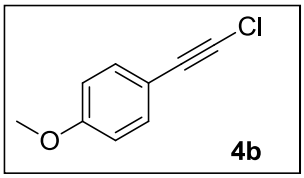
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.54-1.57 (m, 2H), 1.61-1.64 (m, 2H), 2.10- 2.11 (m, 4H), 6.13 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 2.0, 21.3, 22.1, 25.5, 28.9, 96.1, 121.2, 137.1; HRMS (ESI) m/z calcd for C_8H_9I $[M+H]^+$:232.9827; found: 232.9821.

1-chloro-4-(chloroethynyl)benzene (4a)



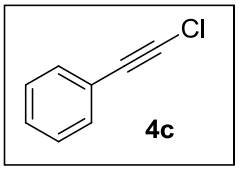
White solid. m.p.72-74 ℃.^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.27-7.30 (m, 2H), 7.35-7.37 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.3, 69.1, 120.6, 128.7, 133.2, 134.7; HRMS (ESI) m/z calcd for $C_8H_4Cl_2$ $[M+H]^+$: 170.9768; found: 170.9765.

1-(chloroethynyl)-4-methoxybenzene (4b)



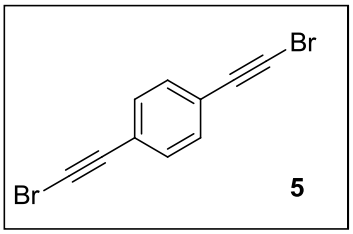
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.79 (s, 3H), 6.81-6.84 (m, 2H), 7.37 (d, *J* = 8.5 Hz, 1H), 7.42 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 55.2, 66.3, 69.2, 113.9, 114.1, 133.3, 159.7; HRMS (ESI) m/z calcd for C_9H_7ClO $[M+H]^+$: 167.0264; found: 167.0268.

(chloroethynyl)benzene(4c)



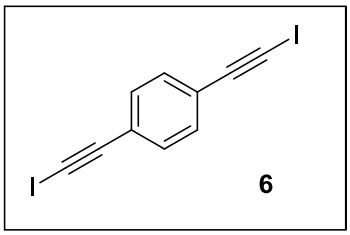
Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29-7.35 (m, 3H), 7.43-7.45 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.0, 69.3, 122.1, 128.3, 128.6, 131.9; HRMS (ESI) m/z calcd for C_8H_5Cl $[M+H]^+$: 137.0158; found: 137.0164.

1,4-bis(bromoethynyl)benzene (5)



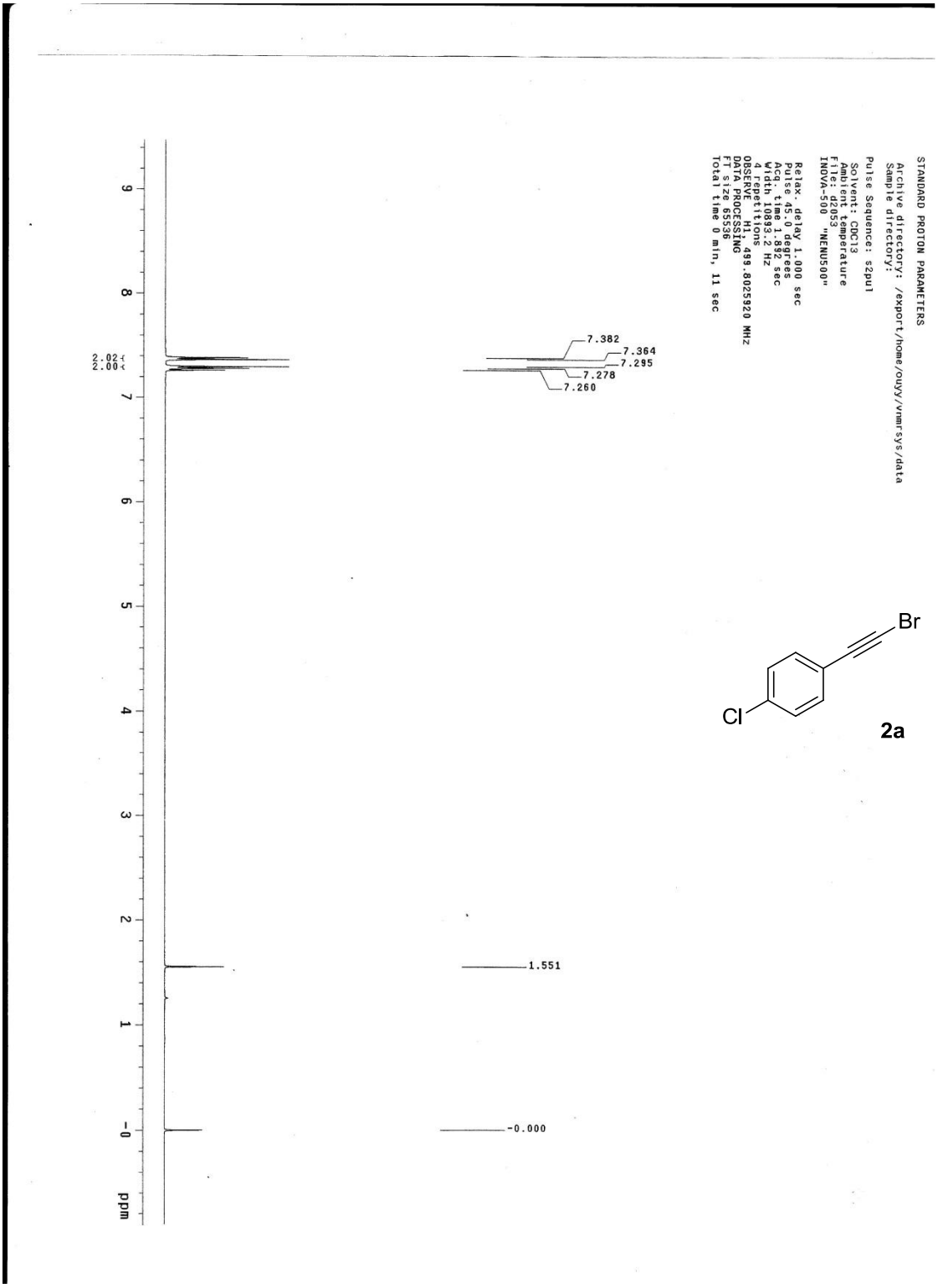
White solid. m.p.161-163 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.37 (s, 4H);^{13}C NMR ($CDCl_3$, 125 MHz): δ = 52.2, 79.5, 122.9, 131.9; HRMS (ESI) m/z calcd for $C_{10}H_4Br_2$ $[M+H]^+$: 282.8758; found: 282.8752.

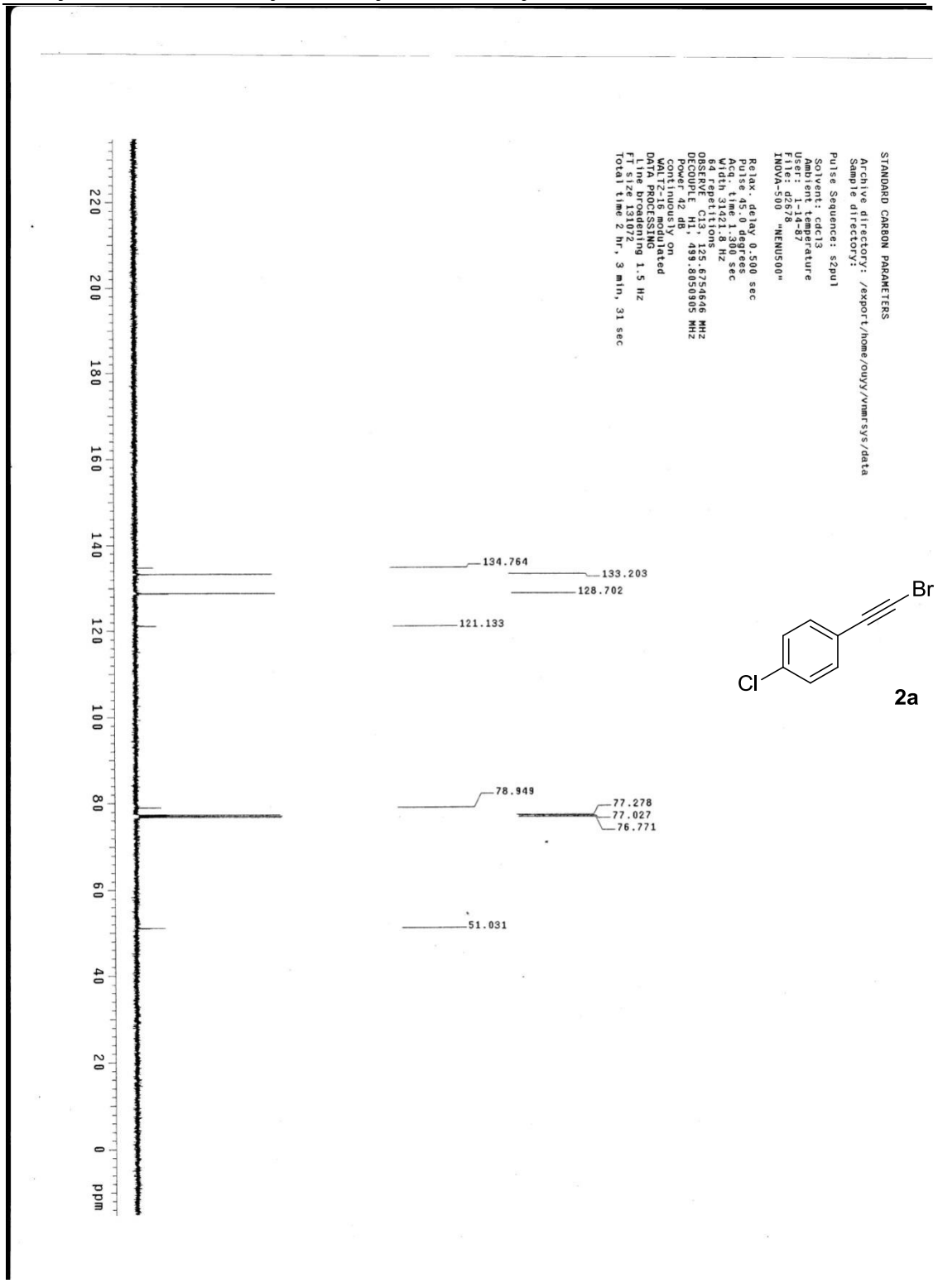
1,4-bis(iodoethynyl)benzene (6)



White solid. m.p. 182-184 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.37 (s, 4H);^{13}C NMR ($CDCl_3$, 125 MHz): δ = 9.13, 93.5, 123.7, 132.1; HRMS (ESI) m/z calcd for $C_{10}H_4I_2$ $[M+H]^+$: 378.8481; found: 378.8486.

III. Copies of ^{1}H and ^{13}C NMR spectra for compounds 2

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continuously on
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Line broadening 1.5 Hz
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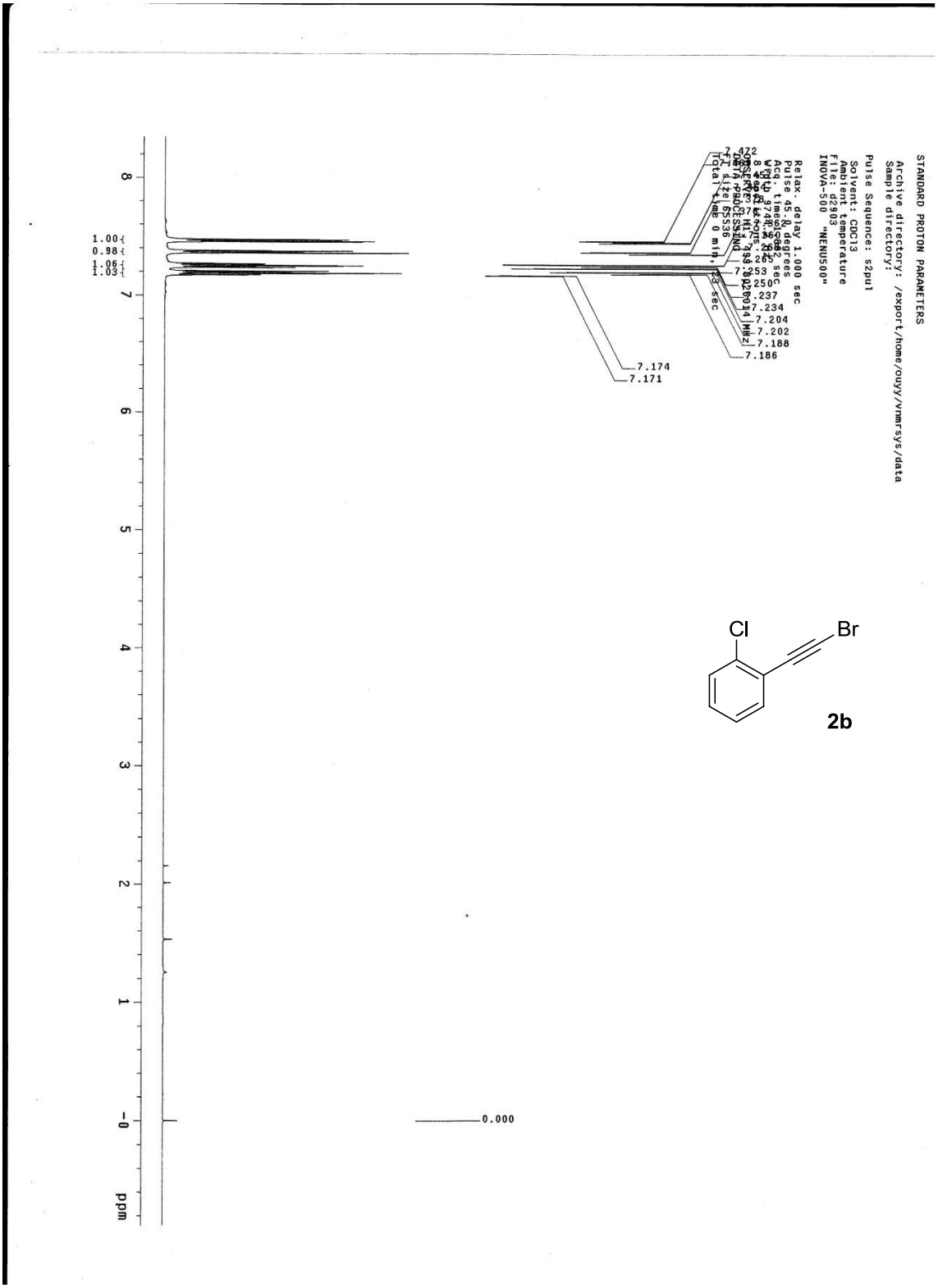
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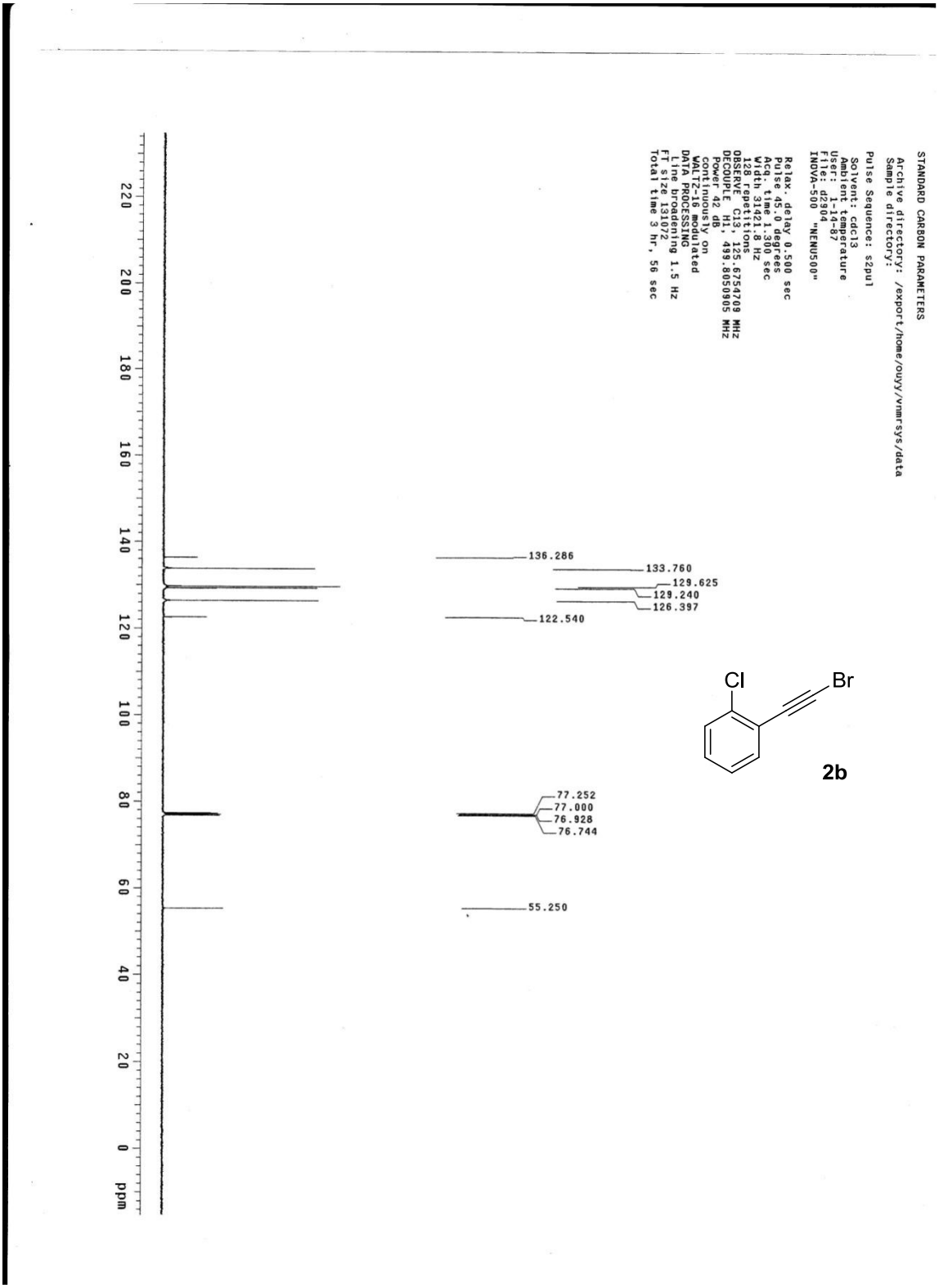
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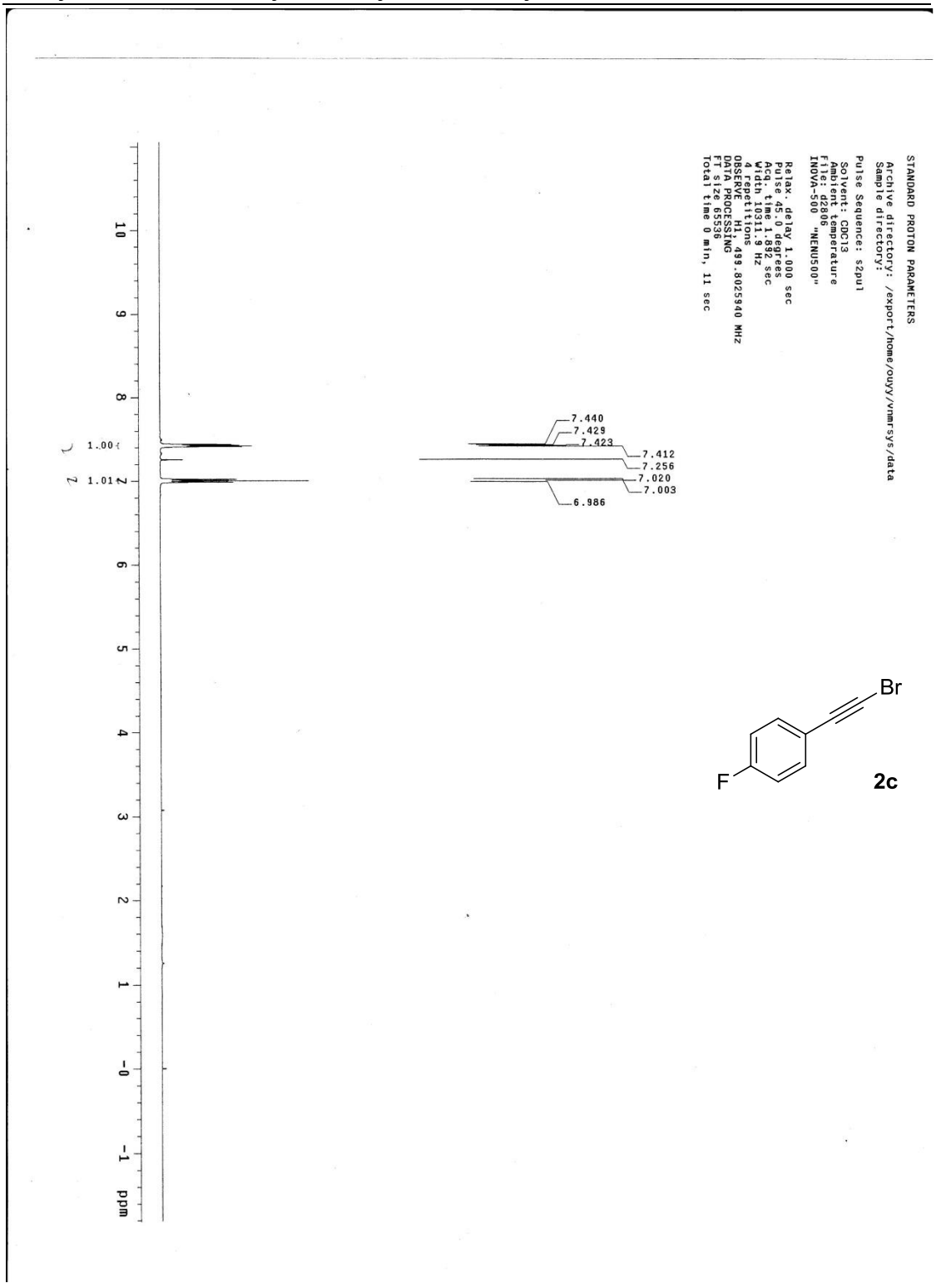
ppm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1 repetitions
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DATA PROCESSING
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Total time 0 min, 11 sec
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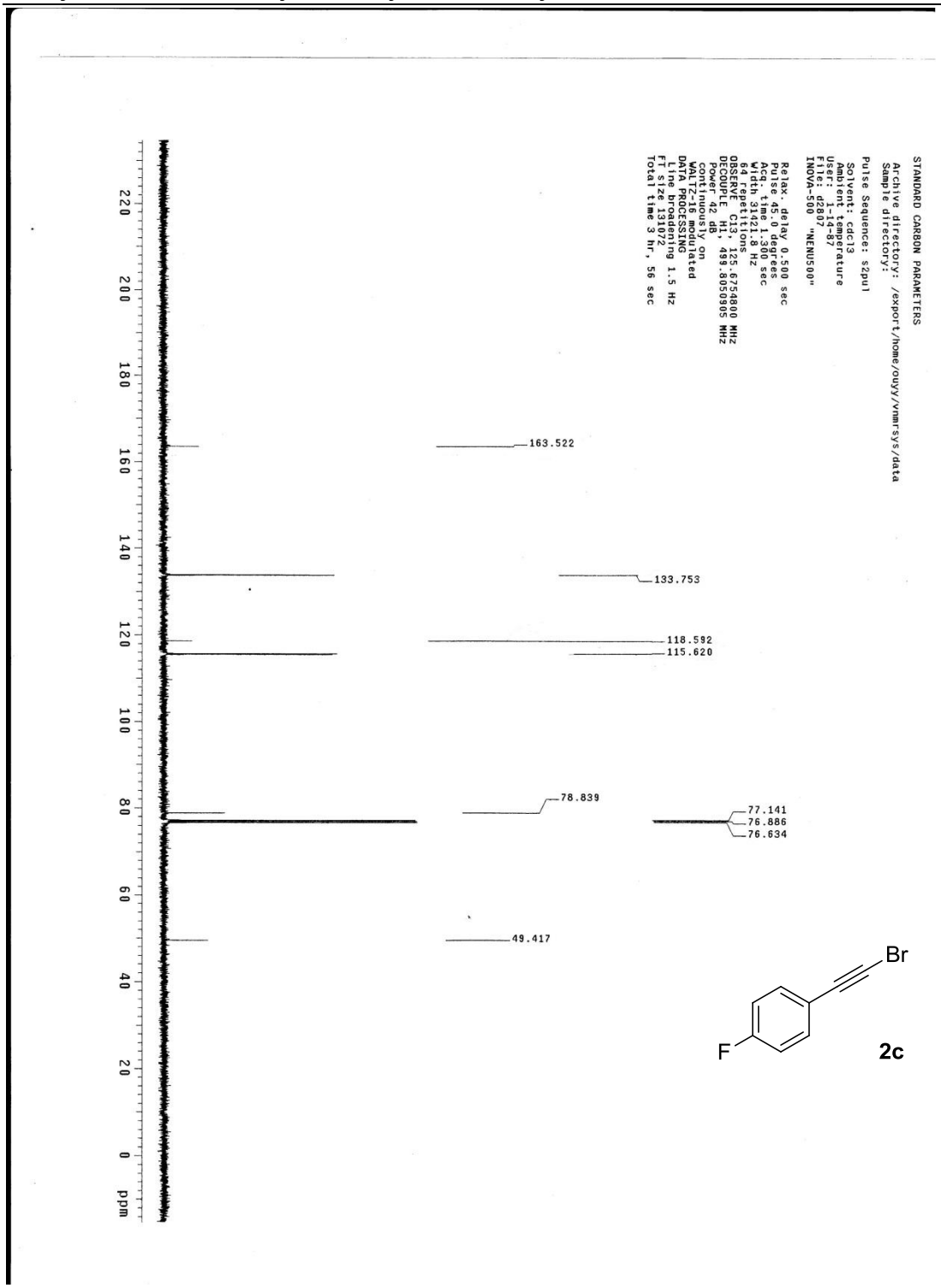
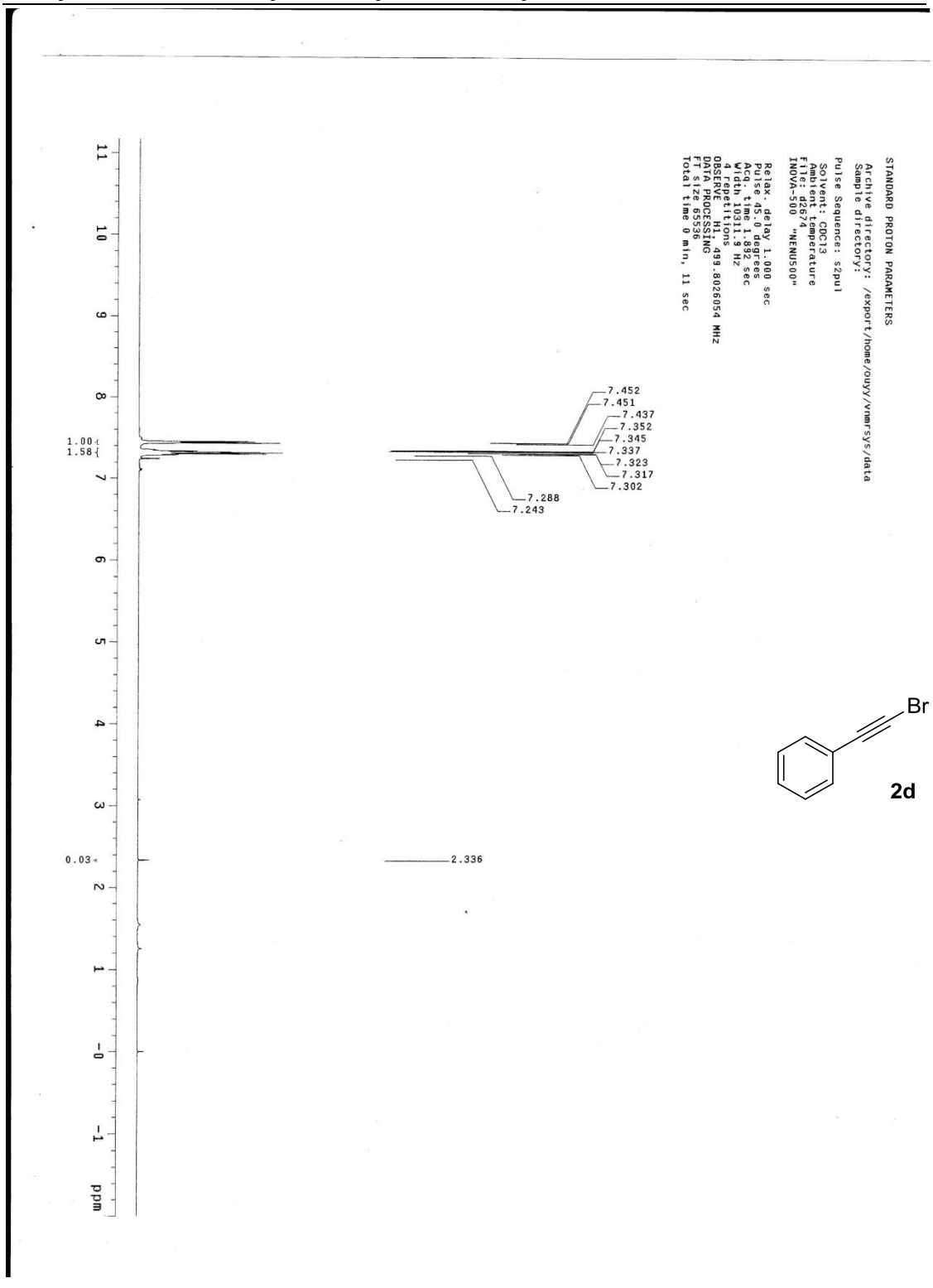
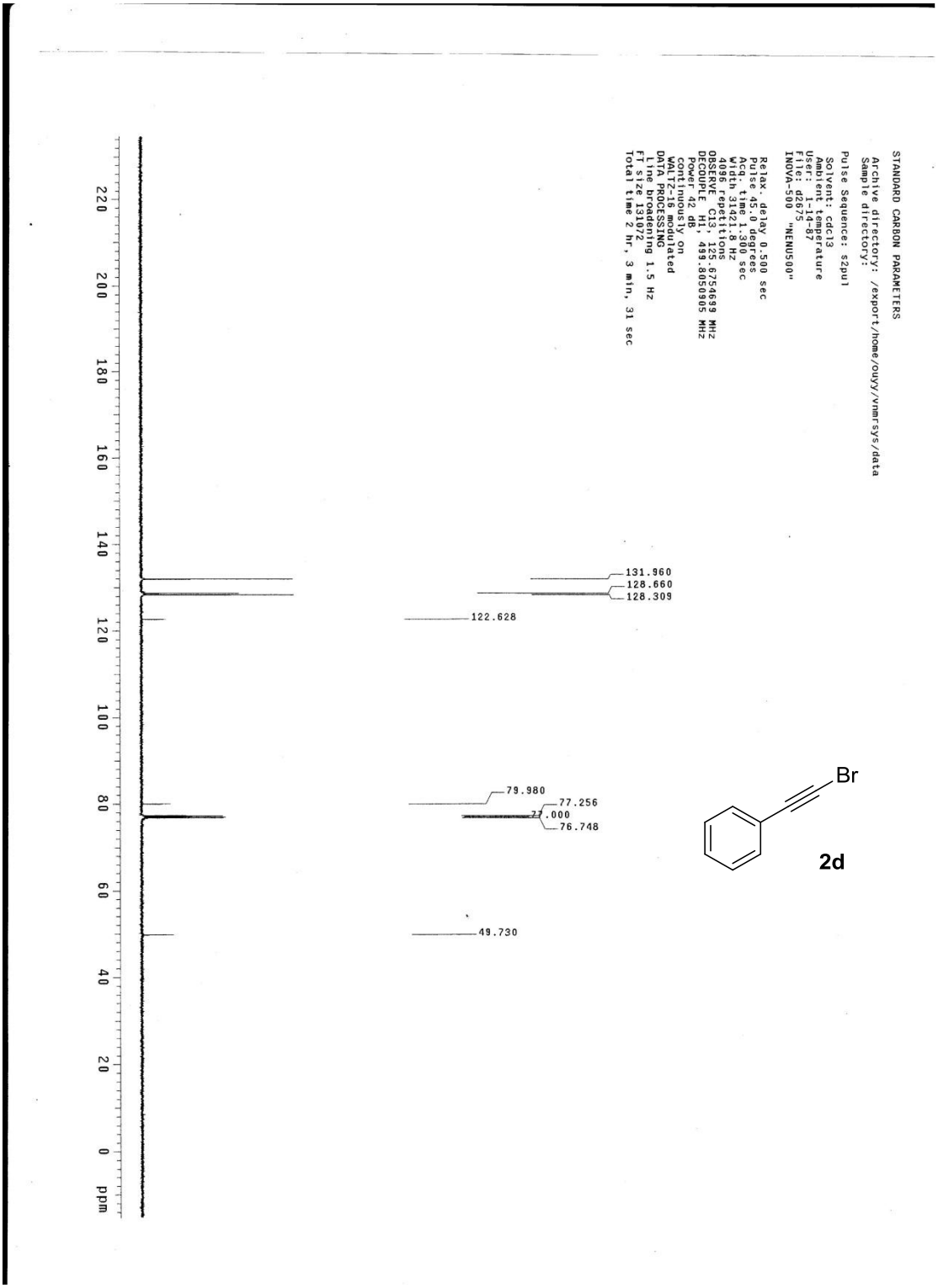
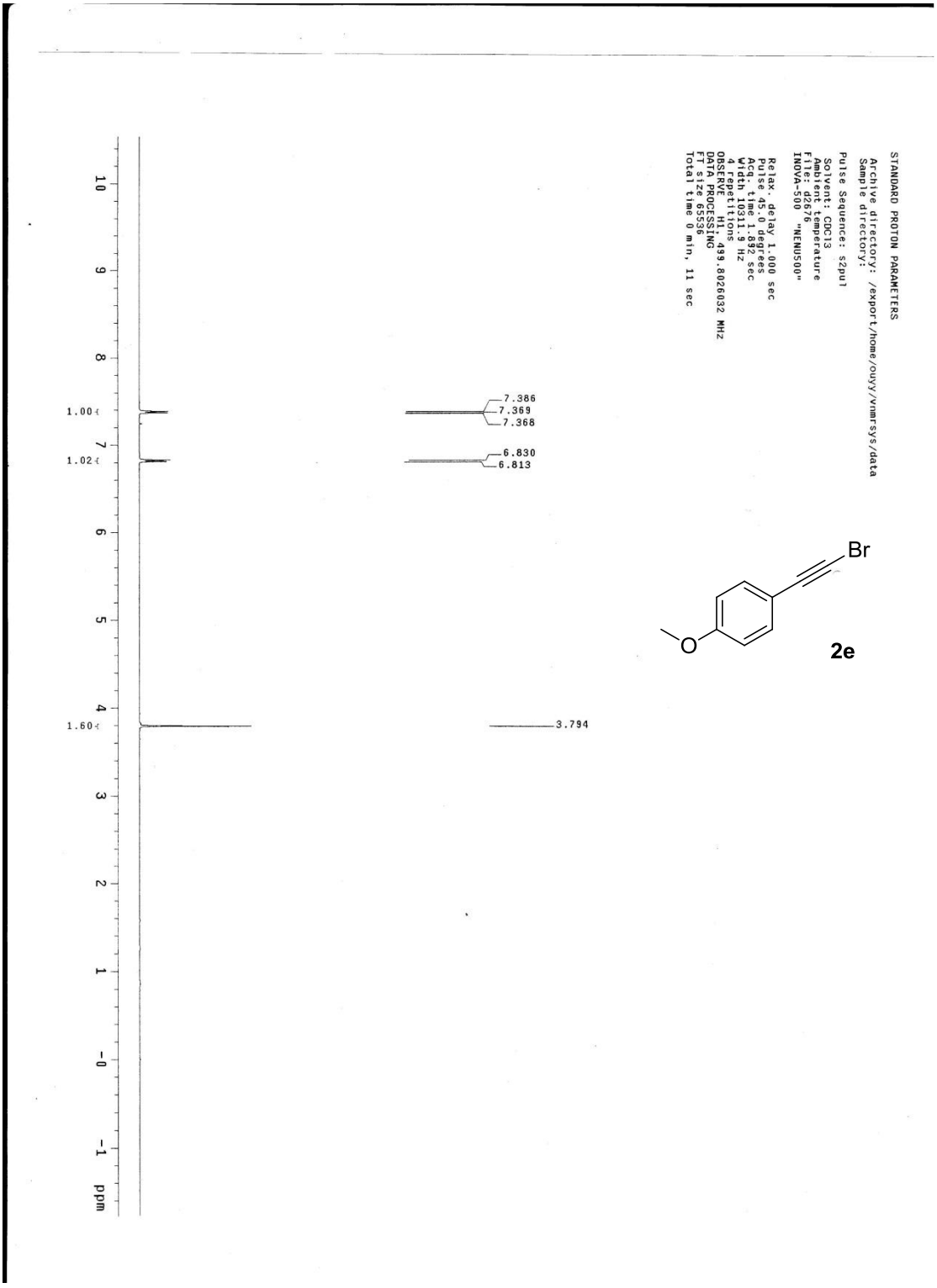
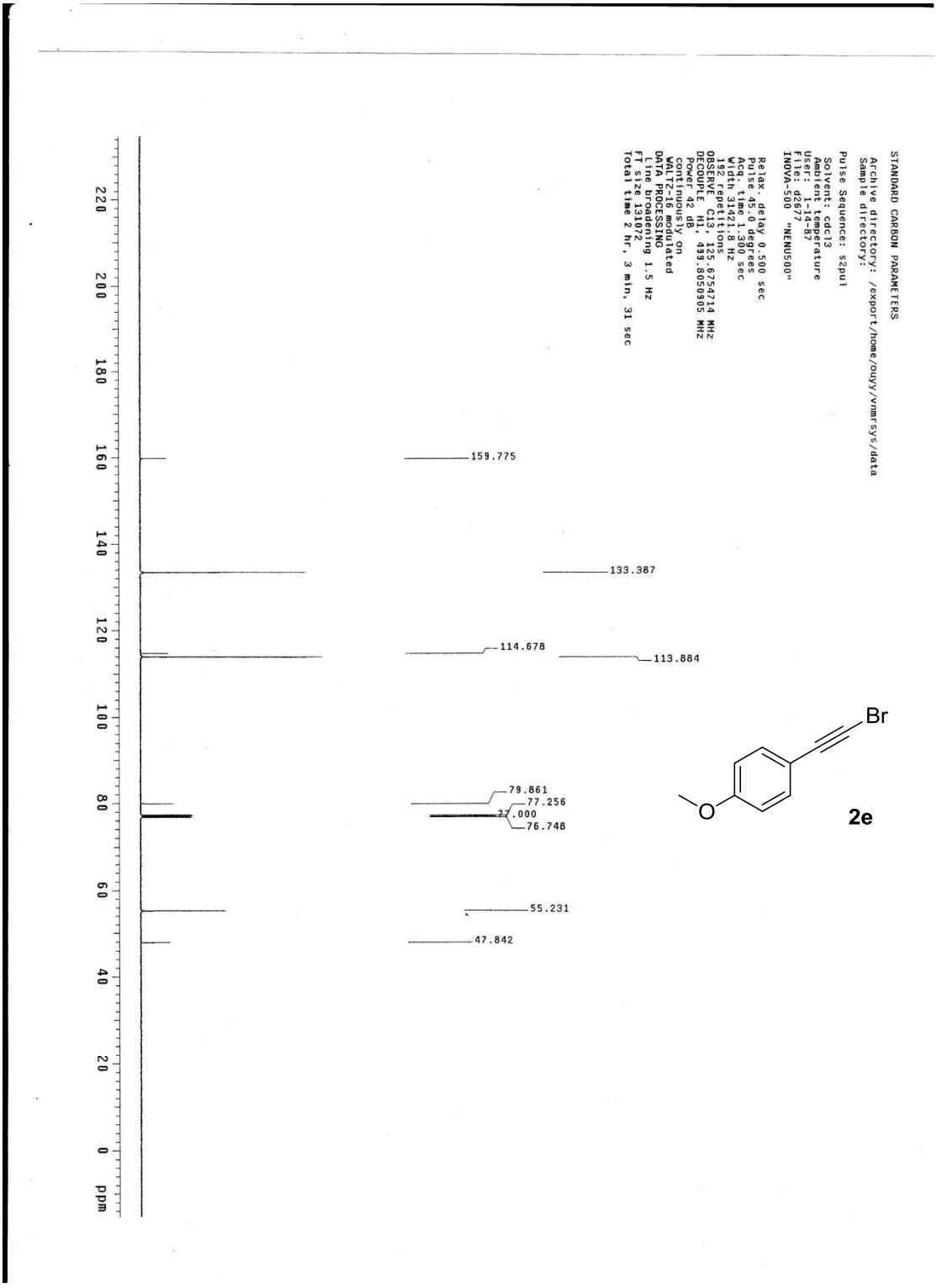
7.423
7.412
7.256
7.020
7.003
6.986
1.00
1.01
ppm
Br
F
2c

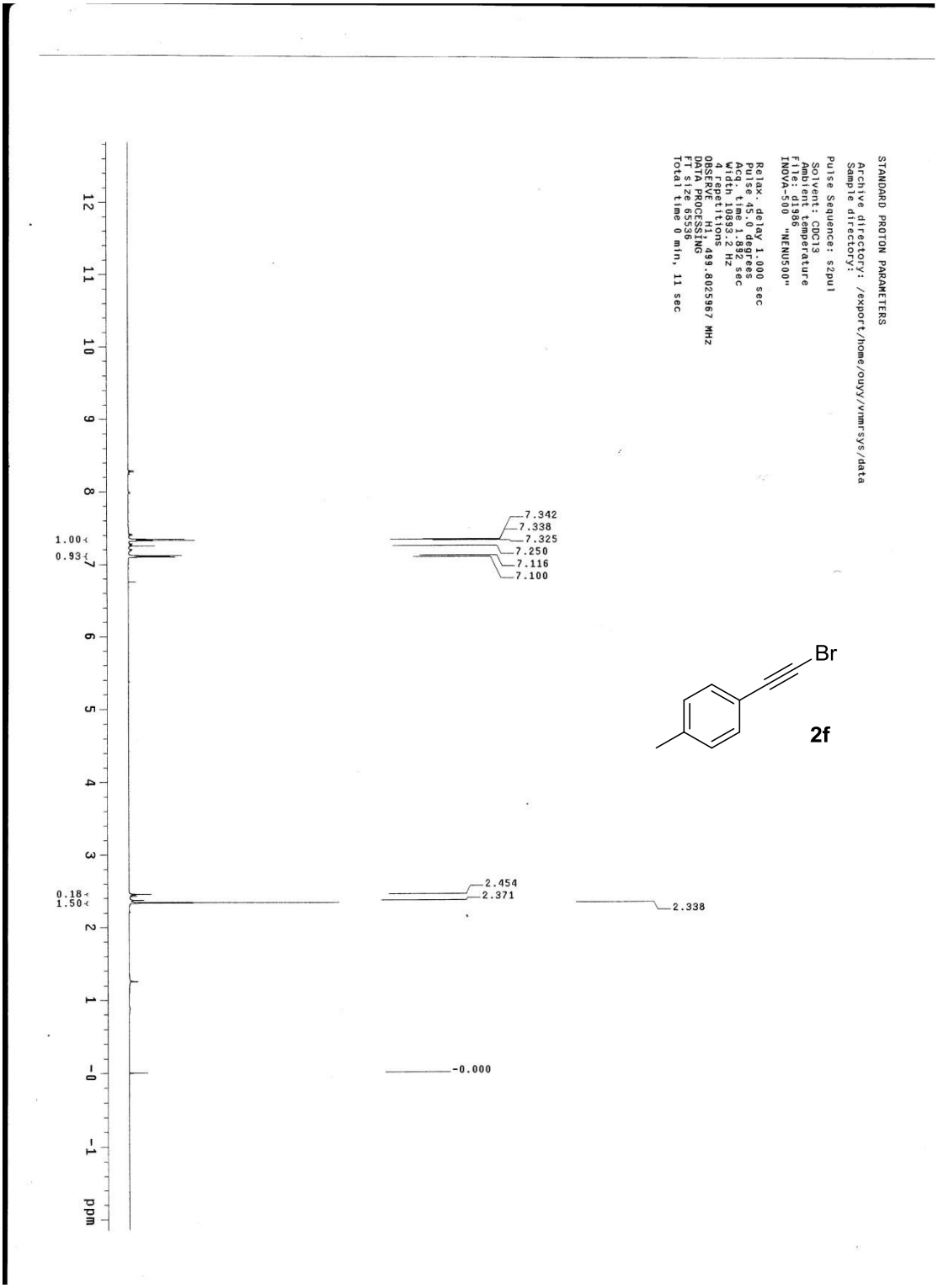

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: liuyi-87
File: d2a1-87
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754800 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
163.522
133.753
118.592
115.620
78.839
77.141
76.886
76.634
49.417
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Br
F
2c

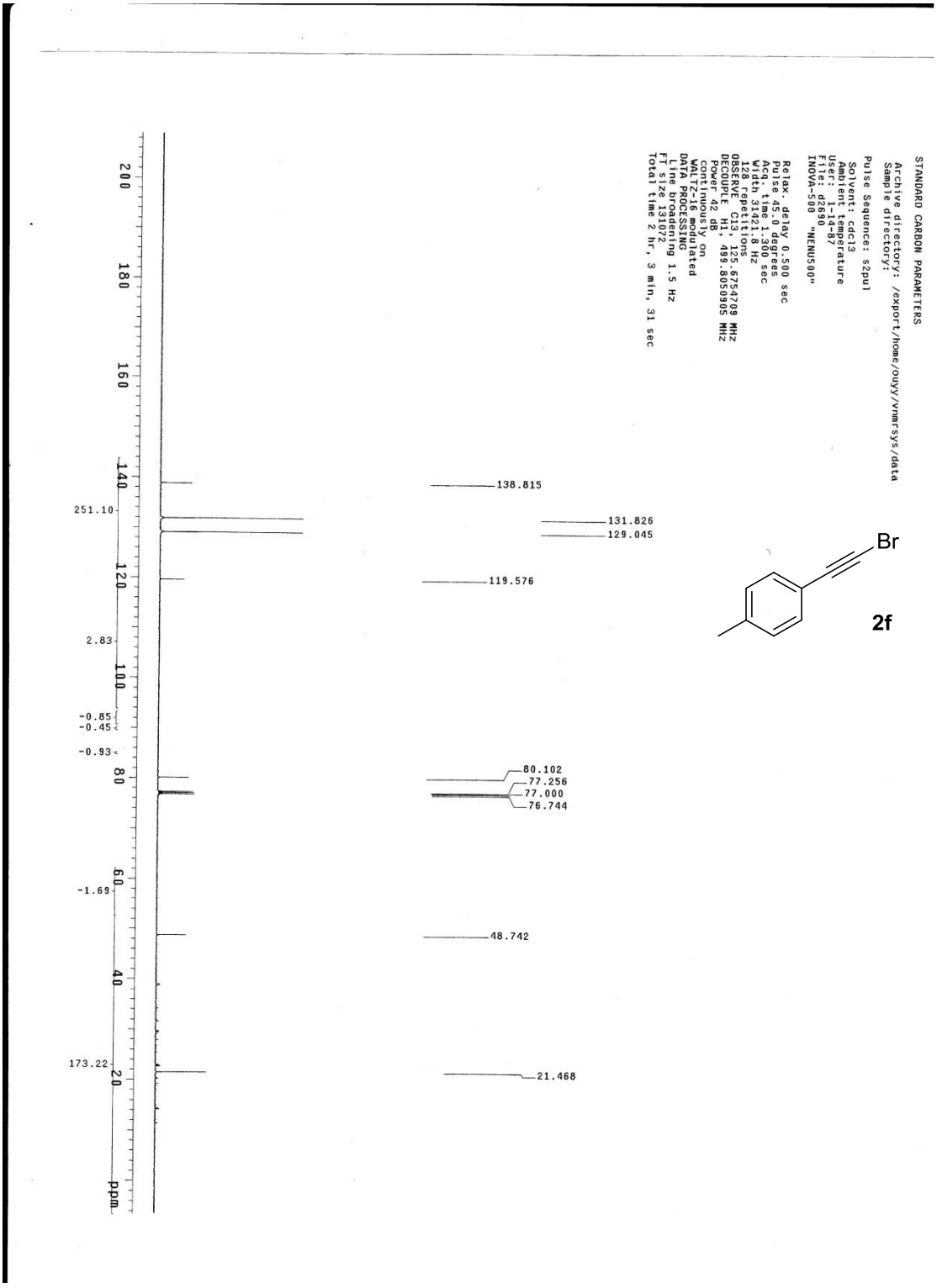

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: ouyy-026-74
INOVA-500 "NEMUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8026054 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.452
7.451
7.437
7.352
7.345
7.337
7.323
7.317
7.302
7.288
7.243
2.336
1.00
1.58
0.03
ppm
Br
2d

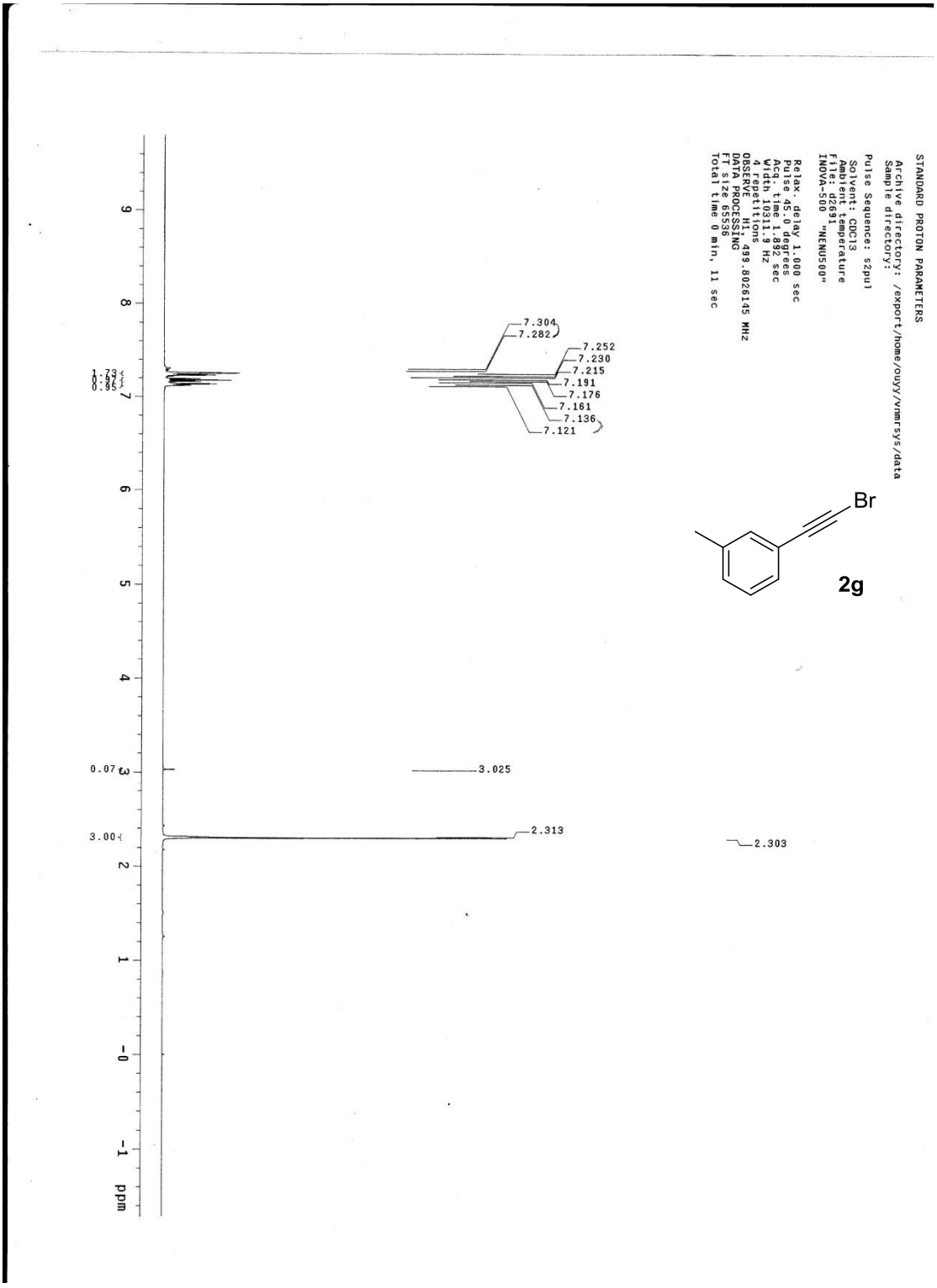

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2675 "MENUS00"
INOVA-500 "MENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 31021.8 Hz
4096 repetitions
OBSERVE C13, 125.6754699 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
131.960
128.660
128.309
122.628
79.980
77.256
77.000
76.748
49.730
Br
2d
220
200
180
160
140
120
100
80
60
40
20
0
ppm

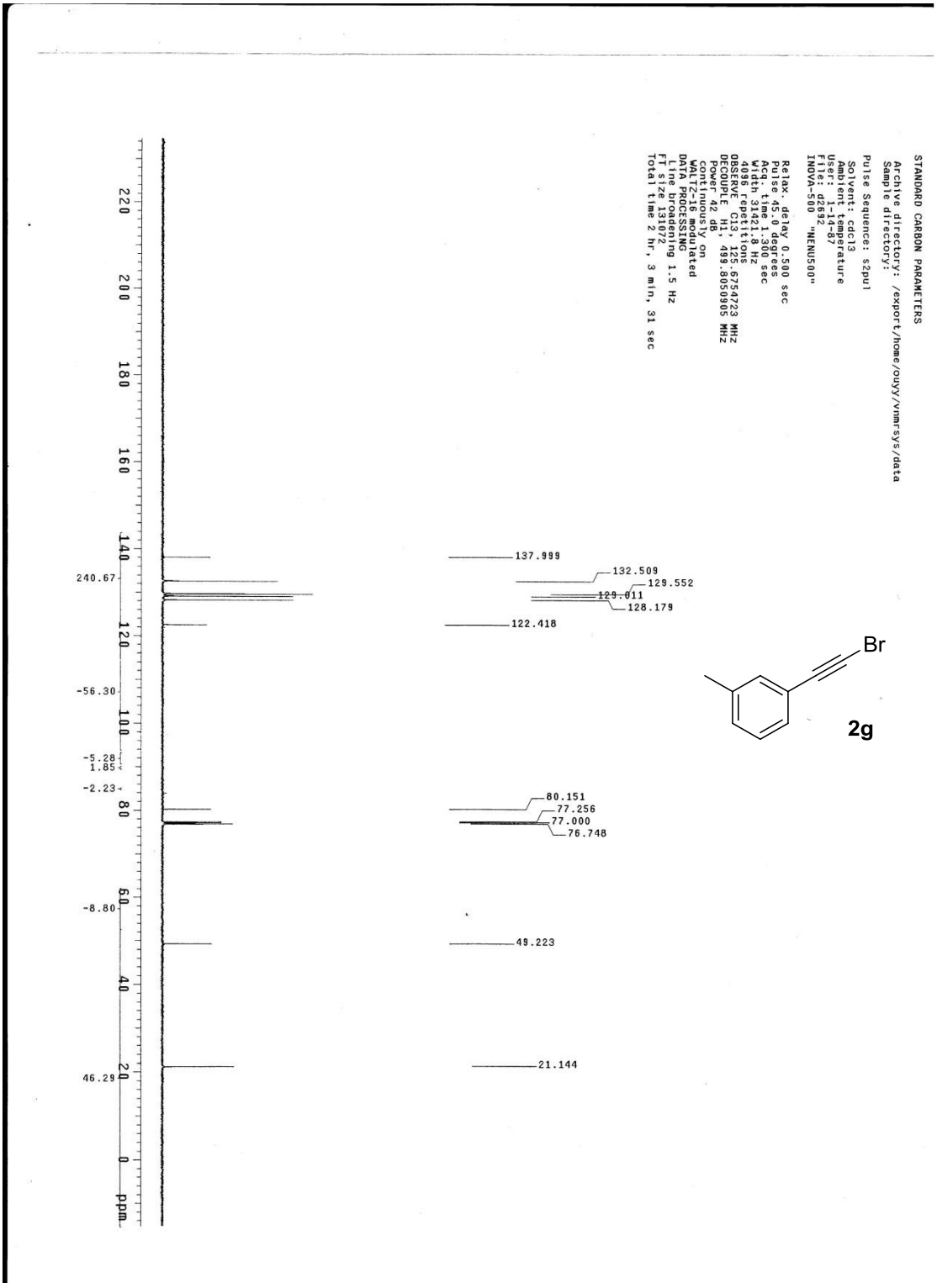

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2676
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
1 repetitions
OBSERVE H1, 499.8026032 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.386
7.369
7.368
6.830
6.813
3.794
1.00
1.02
1.60
Br
O
2e
ppm

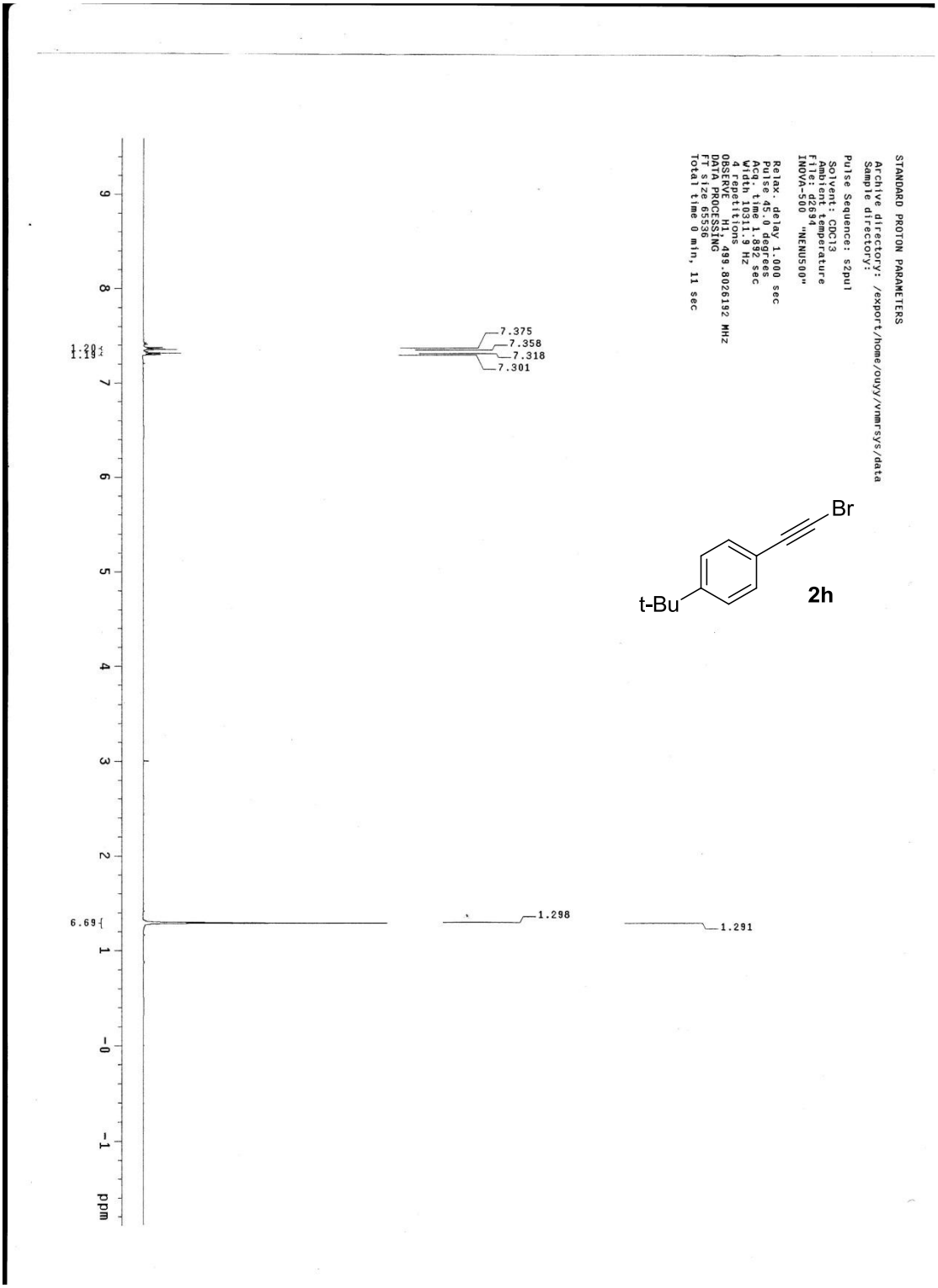

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2677 "NENUS00"
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31152.3 Hz
192 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
159.775
133.387
114.678
113.884
79.861
77.256
77.000
76.748
55.231
47.842
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Br
O
2e

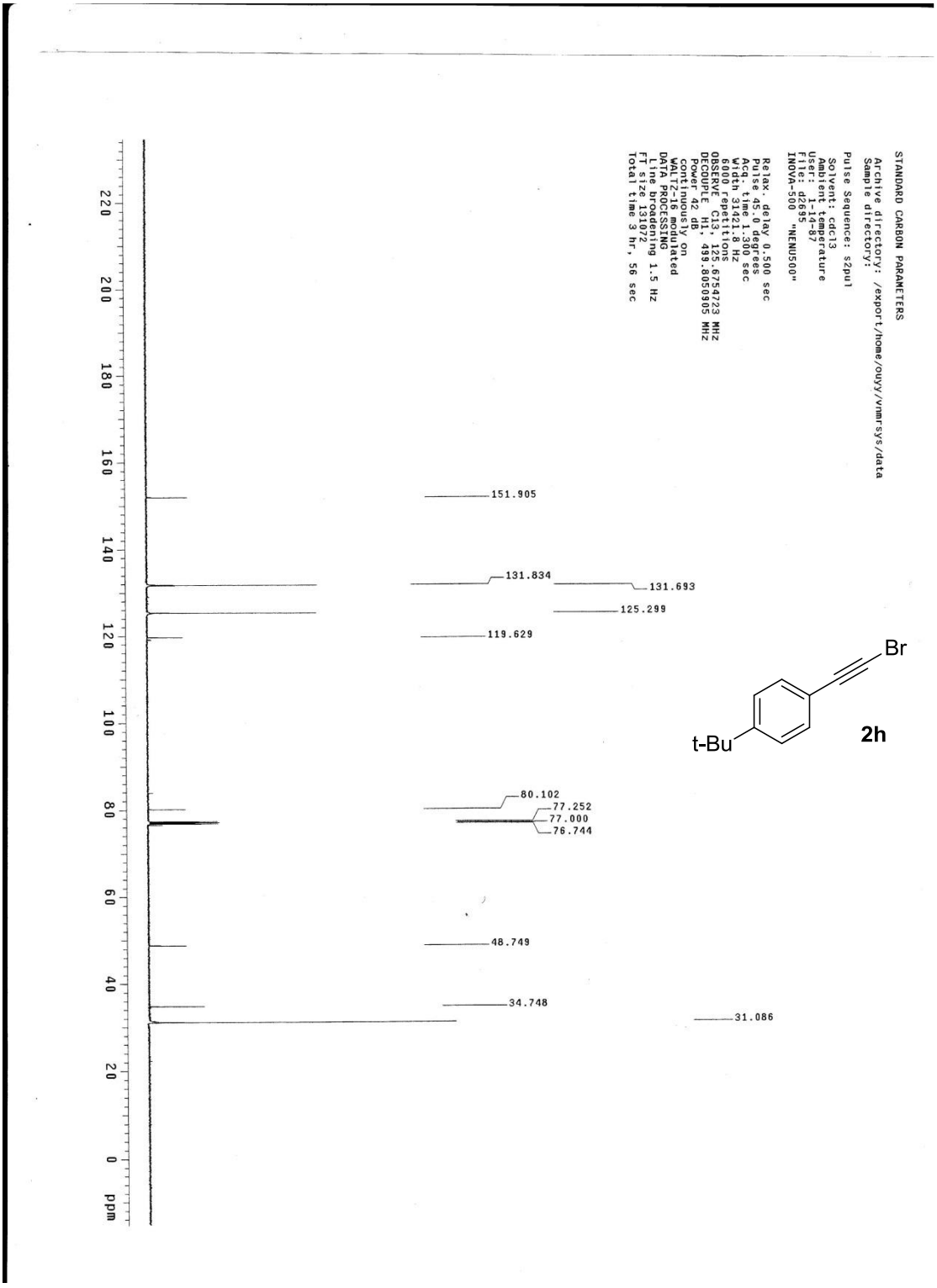

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d1986
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10893.2 Hz
4 repetitions
OBSERVE H1 499.8025867 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.342
7.338
7.325
7.250
7.116
7.100
2.454
2.371
2.338
-0.000
1.00
0.93
0.18
1.50
Br
2f
ppm

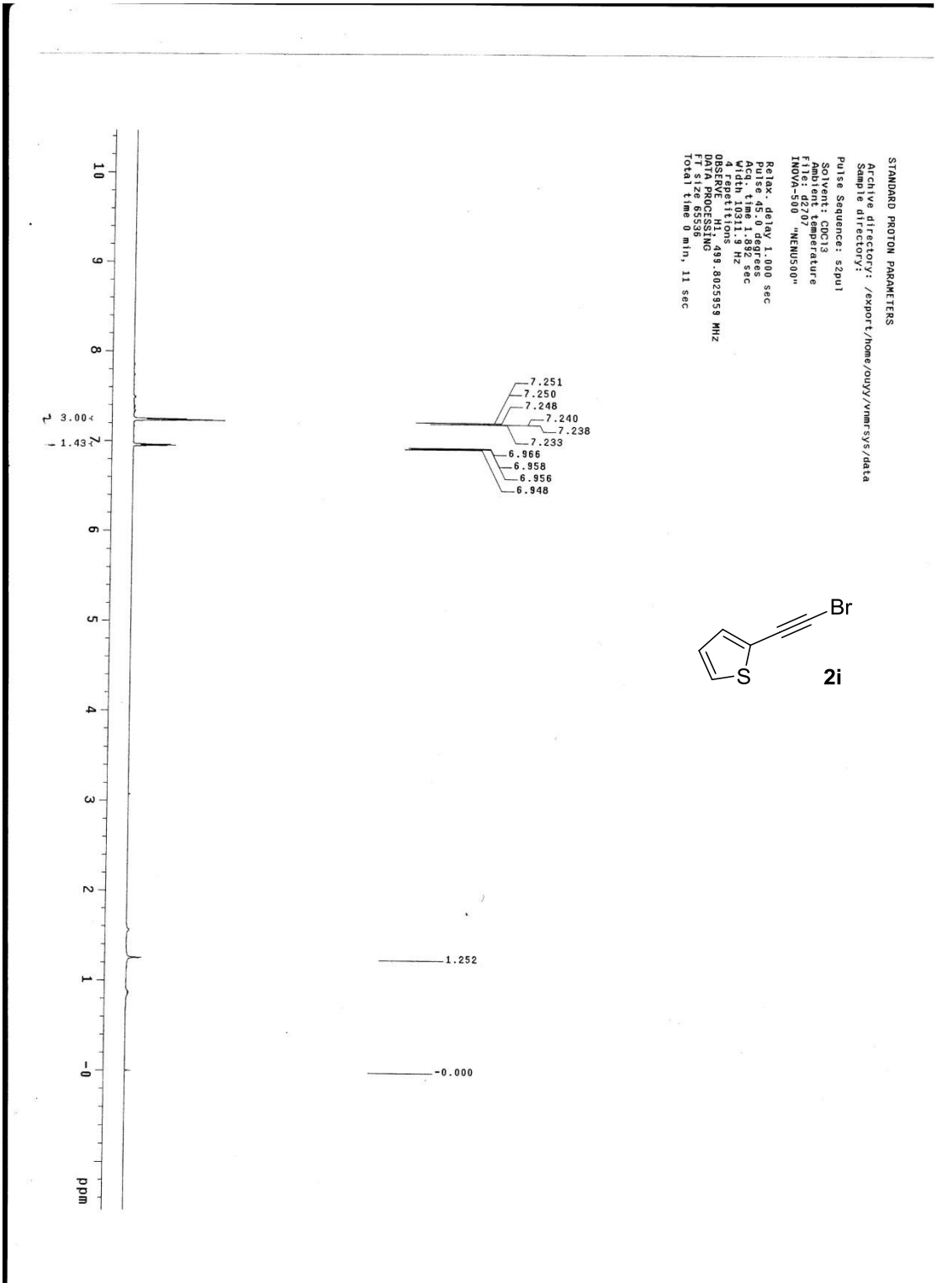

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2690
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 32421.8 Hz
128 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
138.815
131.826
129.045
119.576
80.102
77.256
77.000
76.744
48.742
21.468
251.10
2.83
-0.85
-0.45
-0.93
-1.69
173.22
Br
2f
ppm

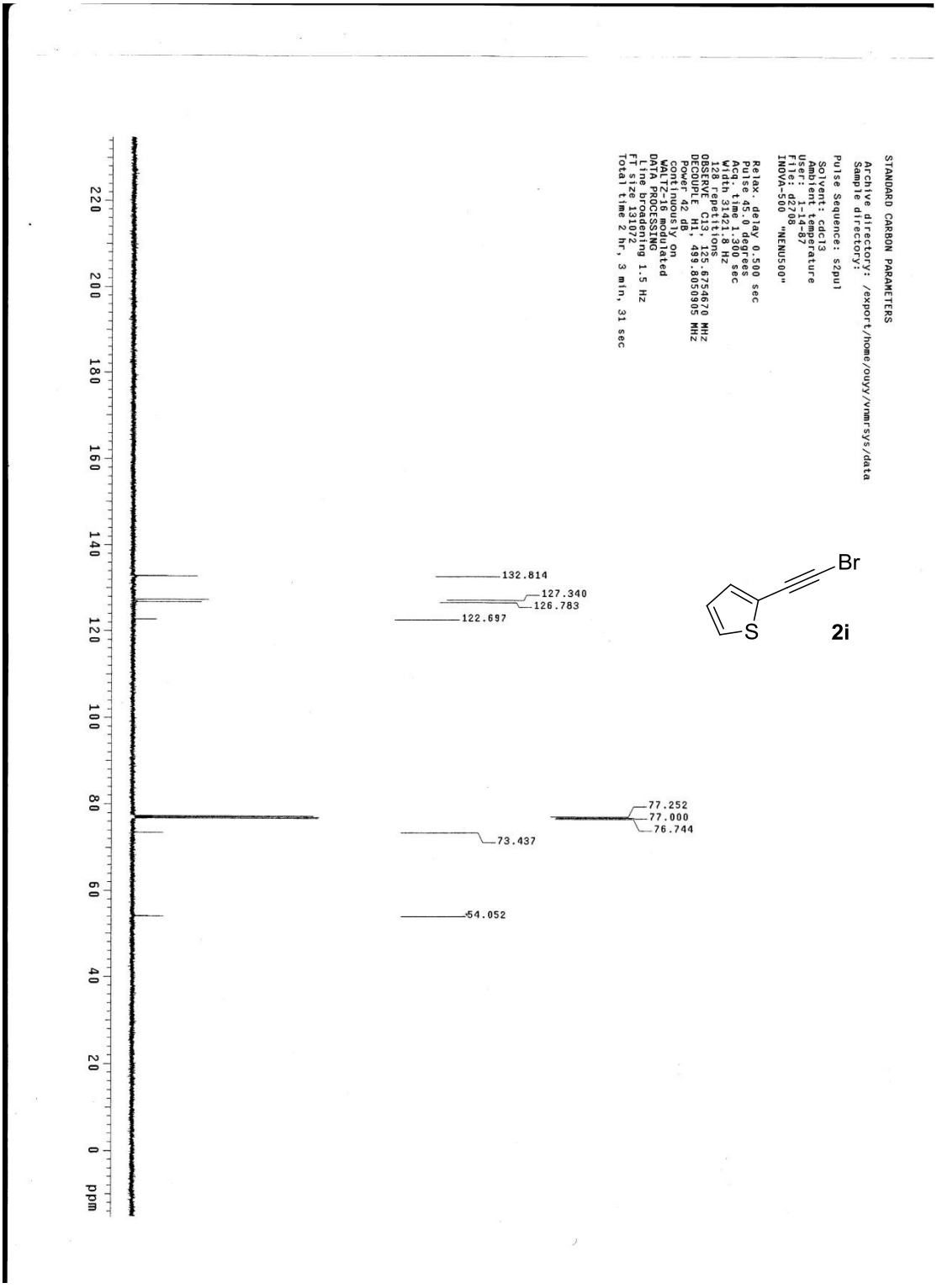

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2691
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11993.1 Hz
4 repetitions
OBSERVE H1, 499.8026145 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.304
7.282
7.252
7.230
7.215
7.191
7.176
7.161
7.136
7.121
1.73
0.97
0.95
3.025
2.313
2.303
0.07
3.00
Br
2g
ppm

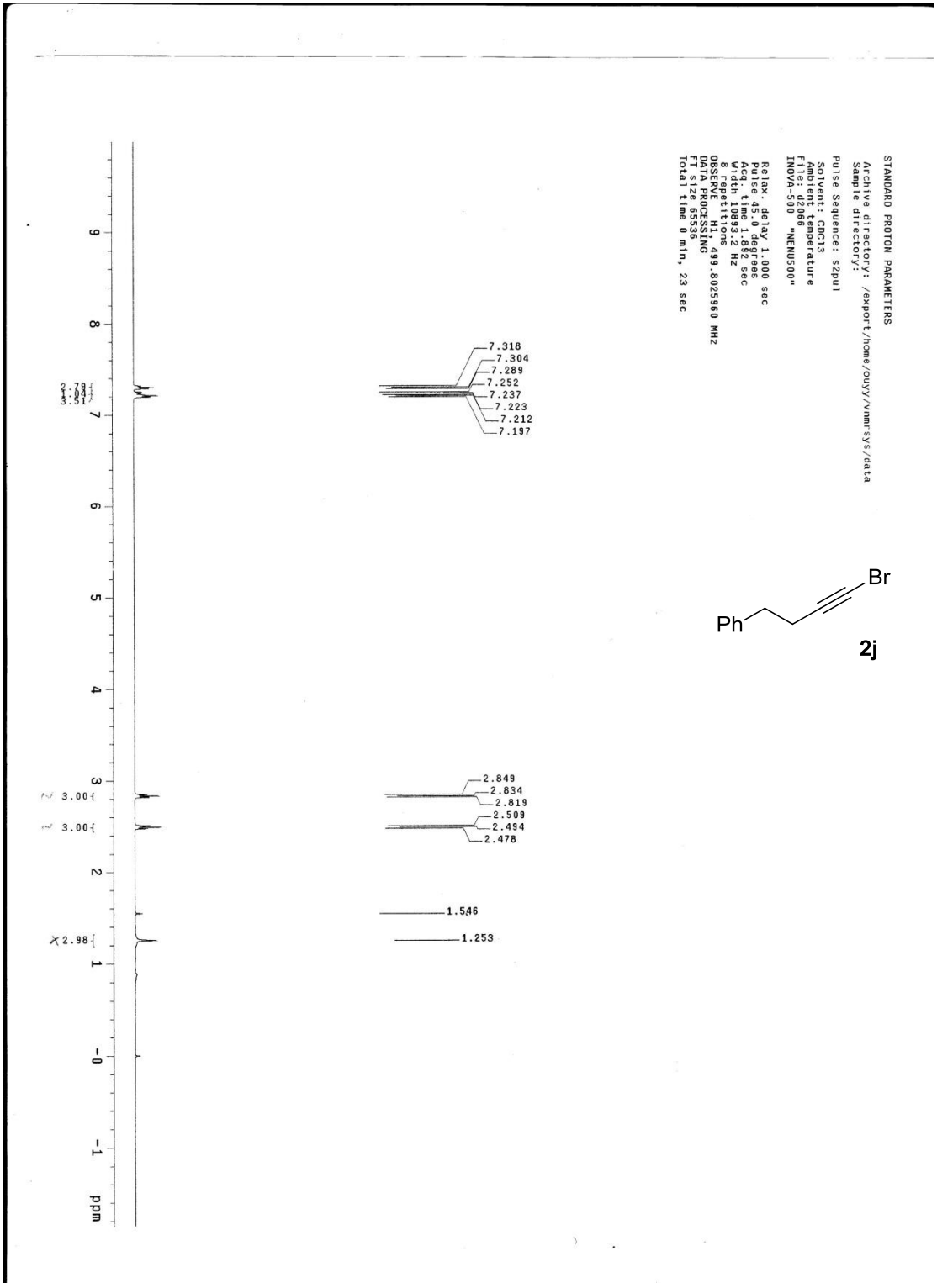

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2692
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 32051.3 Hz
4096 repetitions
OBSERVE C13, 125.6754723 MHz
DECOUPLE H1, 499.8059905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
137.999
132.509
129.552
129.011
128.179
122.418
80.151
77.256
77.000
76.748
49.223
21.144
240.67
-56.30
-5.28
1.85
-2.23
-8.80
46.29
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Br
2g

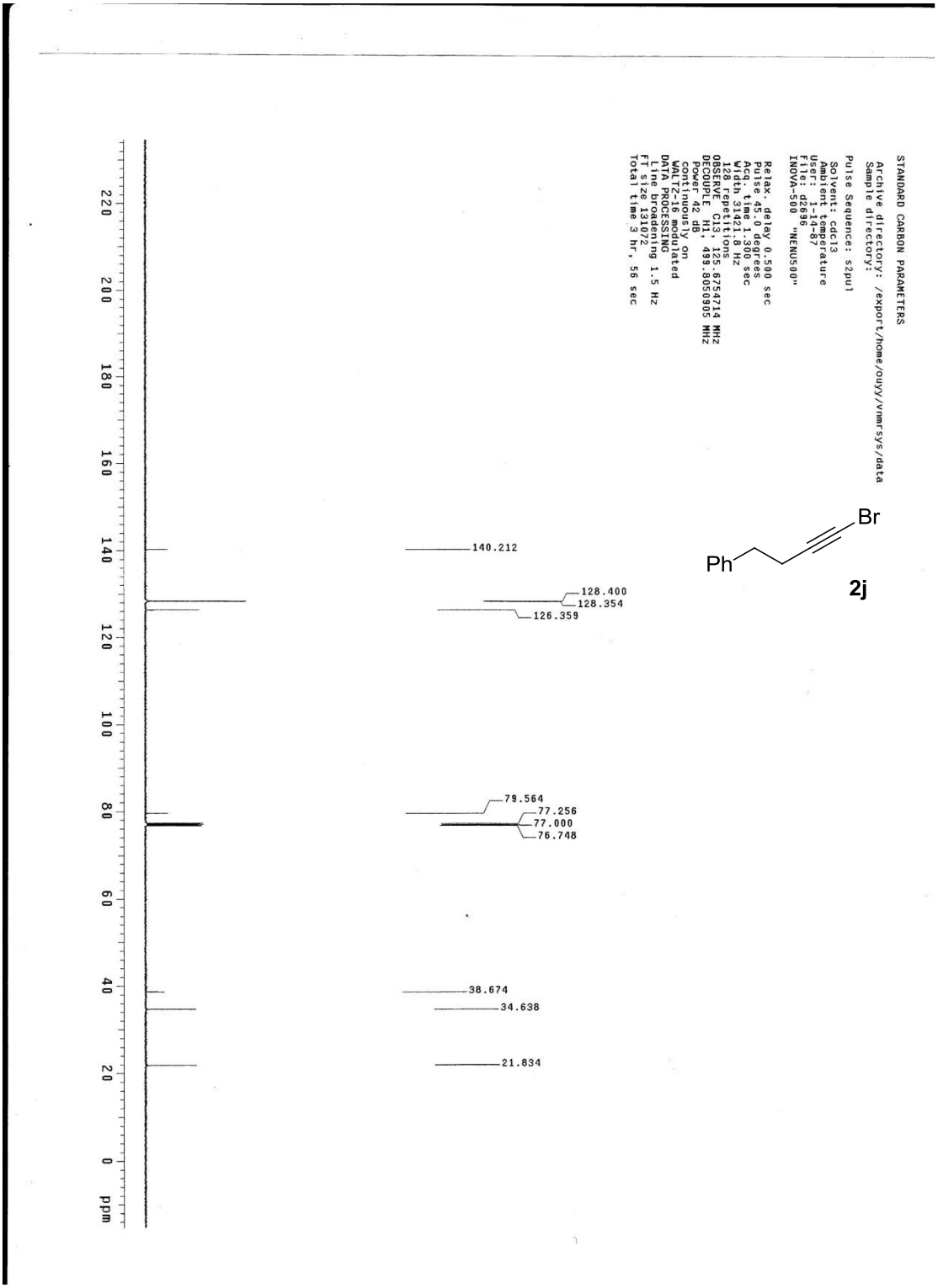

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2694
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.897 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8026192 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.375
7.358
7.318
7.301
1.298
1.291
6.69
1.20
1.19
Br
t-Bu
2h
ppm

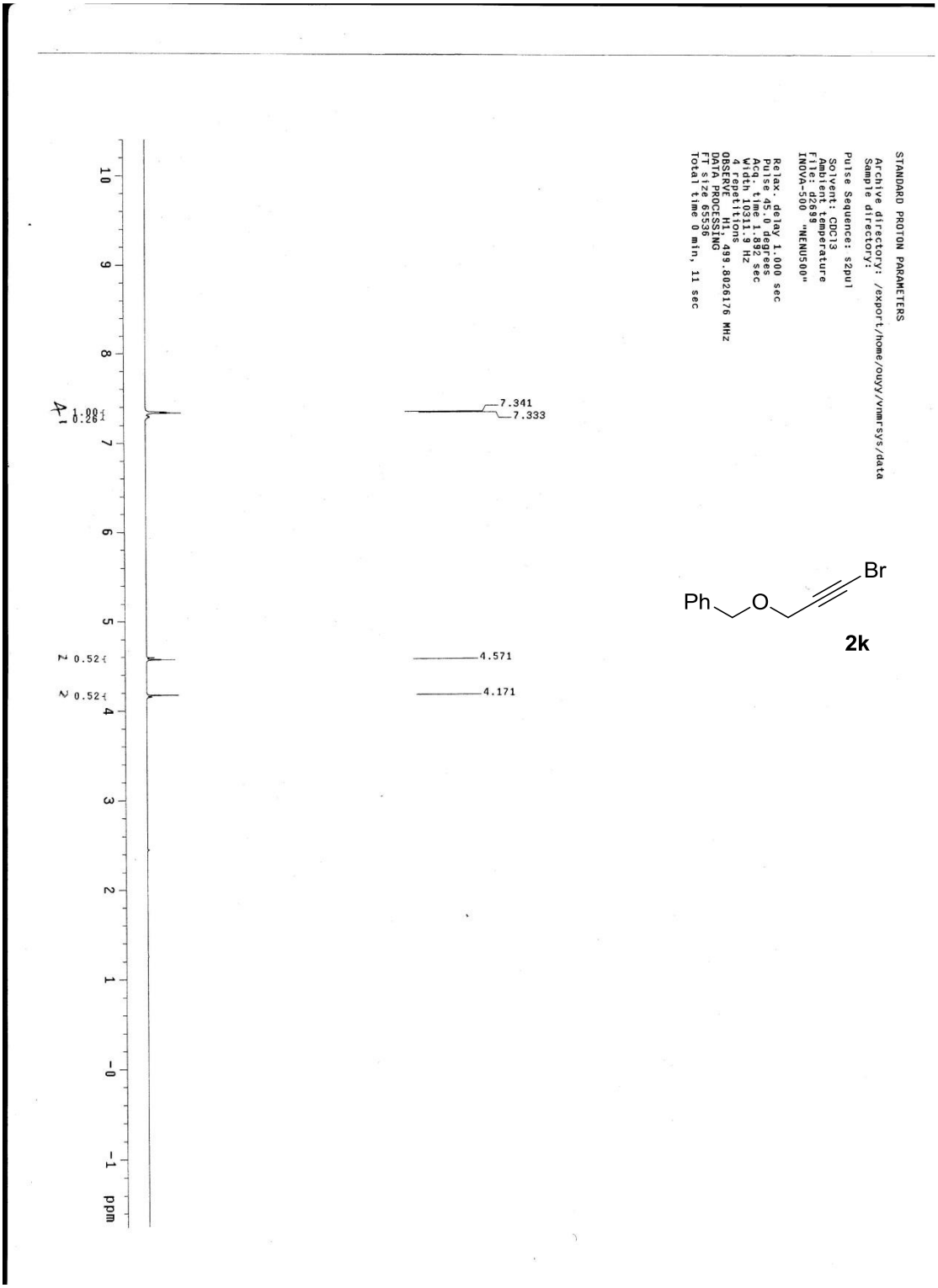

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy
File: d2s5-07
INOVA-500 "NEMU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754723 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
Continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
151.905
131.834
131.693
125.299
119.629
80.102
77.252
77.000
76.744
48.749
34.748
31.086
Br
t-Bu
2h
220
200
180
160
140
120
100
80
60
40
20
0
ppm

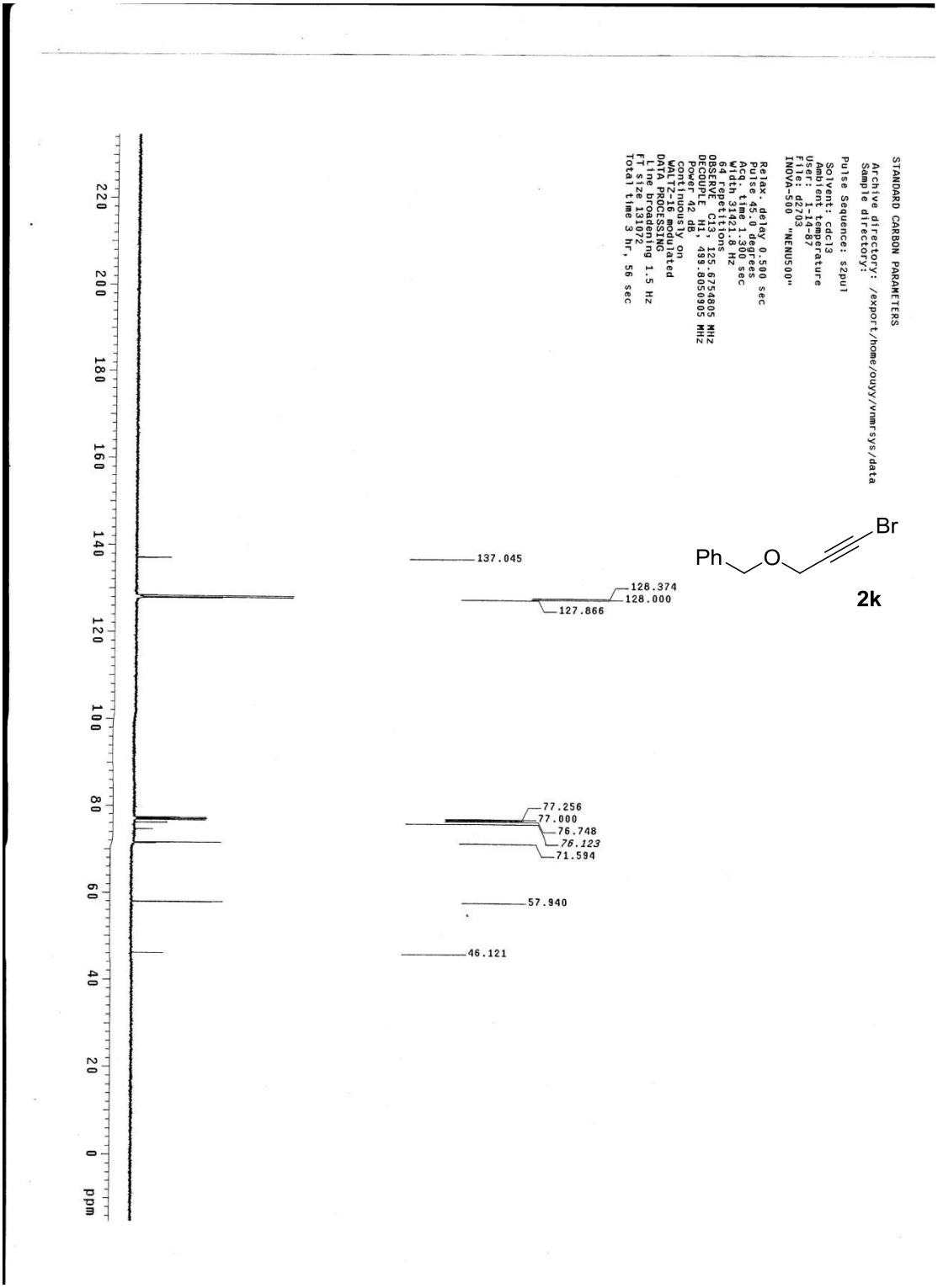

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2707
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
8 repetitions
OBSERVE H1, 499.8025959 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.251
7.250
7.248
7.240
7.238
7.233
6.966
6.958
6.956
6.948
3.00
1.43
1.252
-0.000
Br
S
2i
ppm

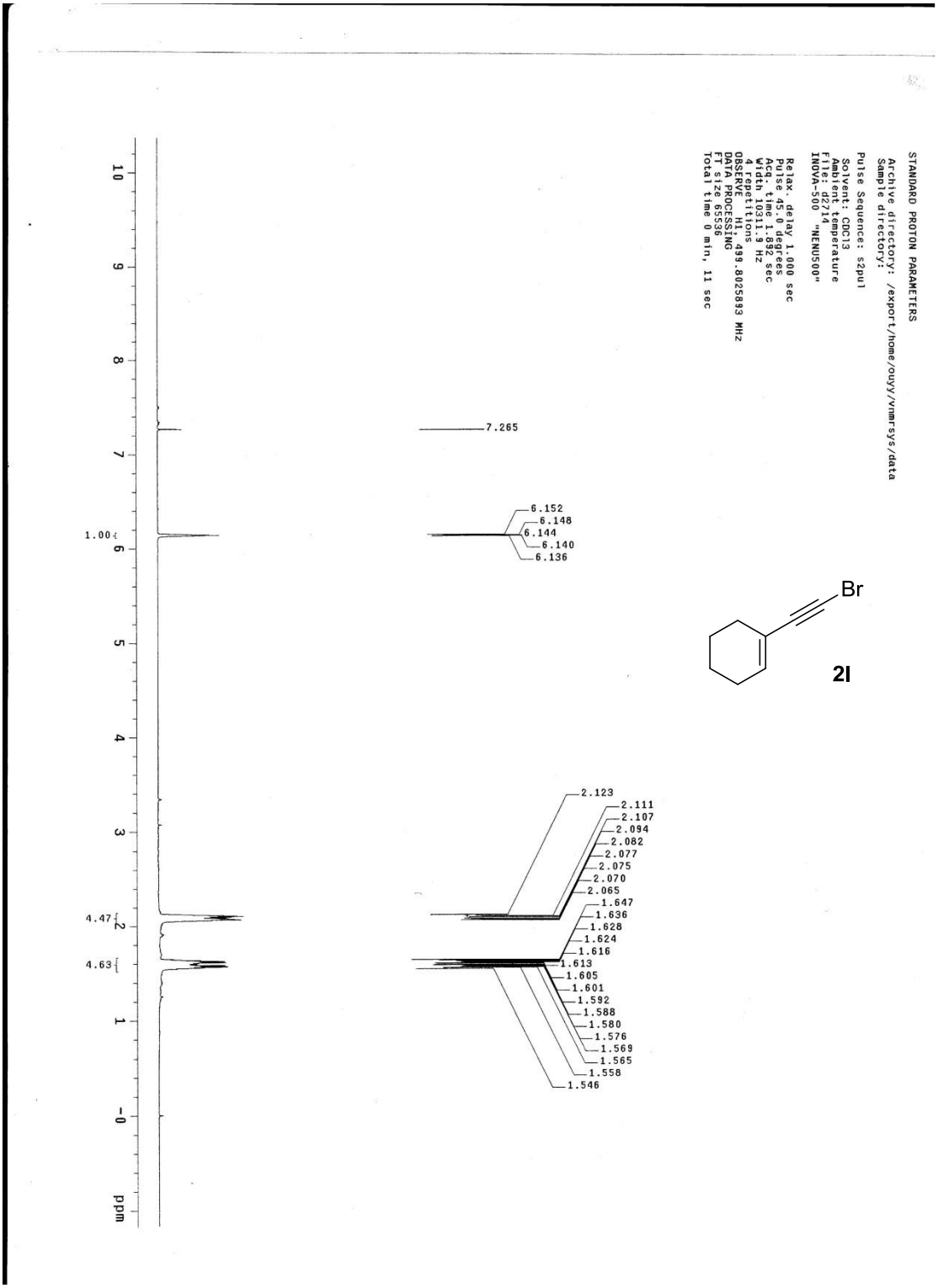

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy-67
File: d2708
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
132.814
127.340
126.783
122.697
77.252
77.000
76.744
73.437
54.052
Br
S
2i
220
200
180
160
140
120
100
80
60
40
20
0
ppm

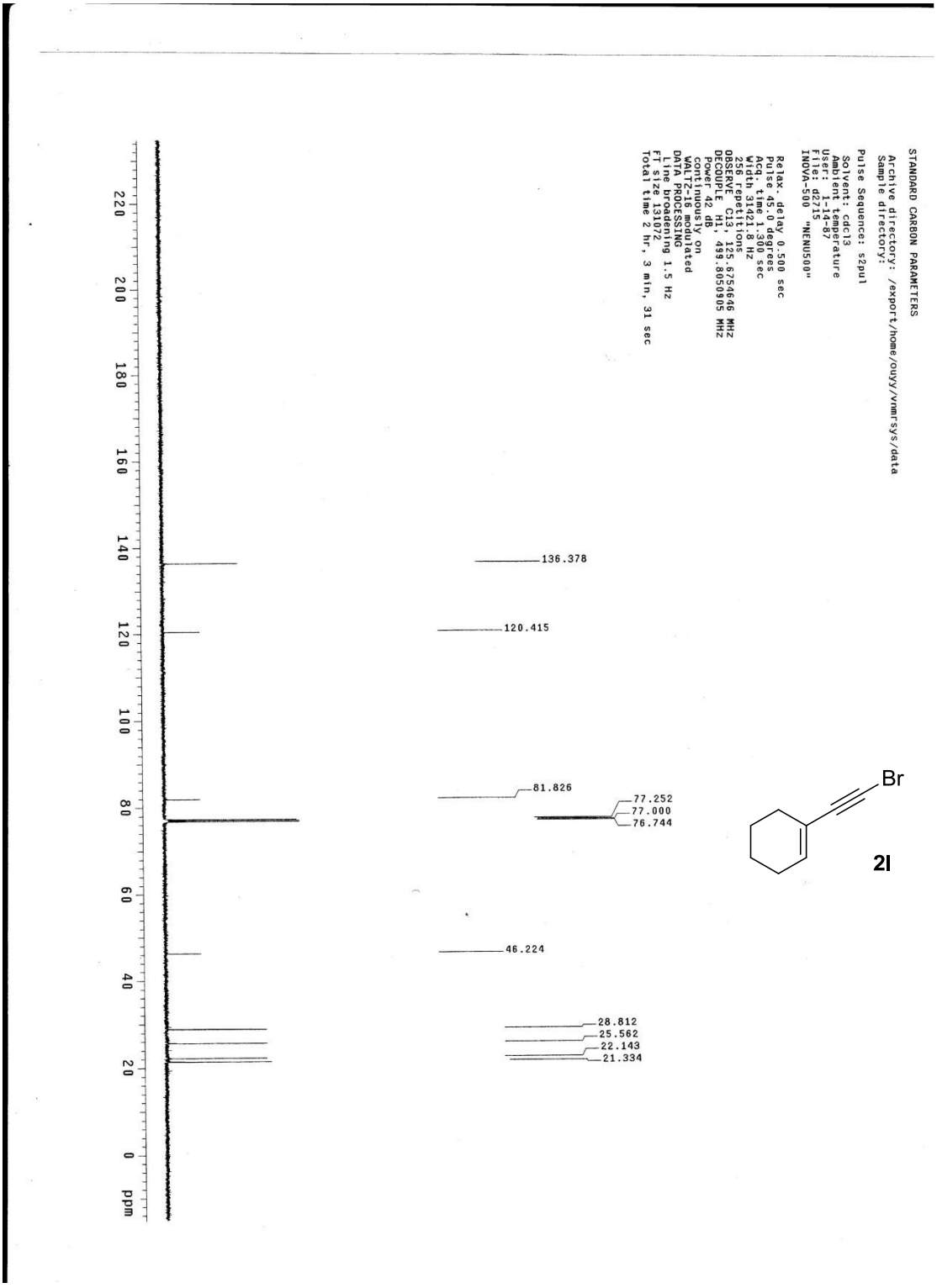

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10893.2 Hz
8 repetitions
OBSERVE H1, 499.8025960 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.318
7.304
7.289
7.252
7.237
7.223
7.212
7.197
2.849
2.834
2.819
2.509
2.494
2.478
1.546
1.253
2.79
1.00
3.51
3.00
3.00
2.98
Br
Ph
2j
ppm

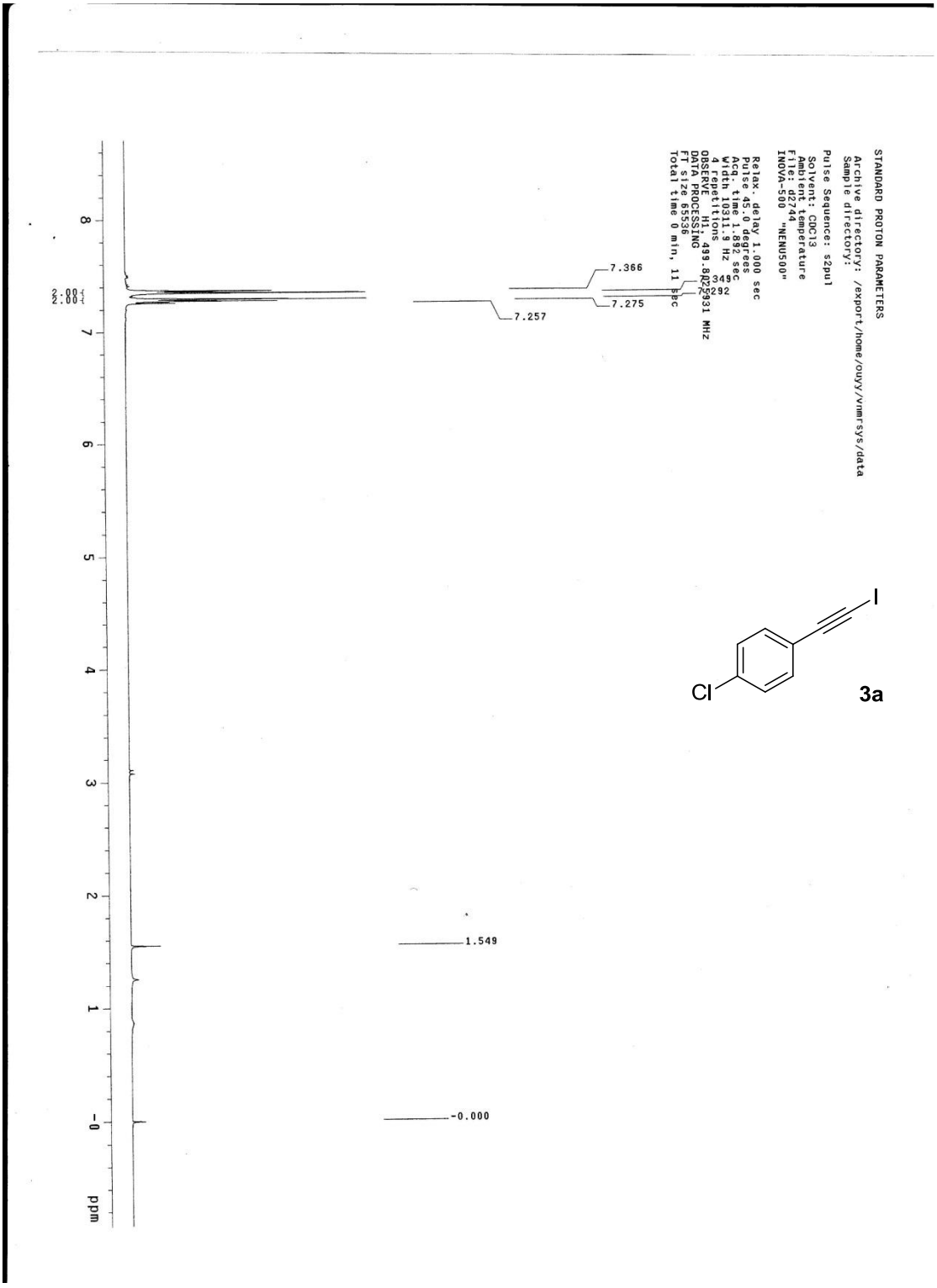

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy
File: d269-67
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8059905 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Br
Ph
2j
140.212
128.400
128.354
126.359
79.564
77.256
77.000
76.748
38.674
34.638
21.834
220
200
180
160
140
120
100
80
60
40
20
0
ppm

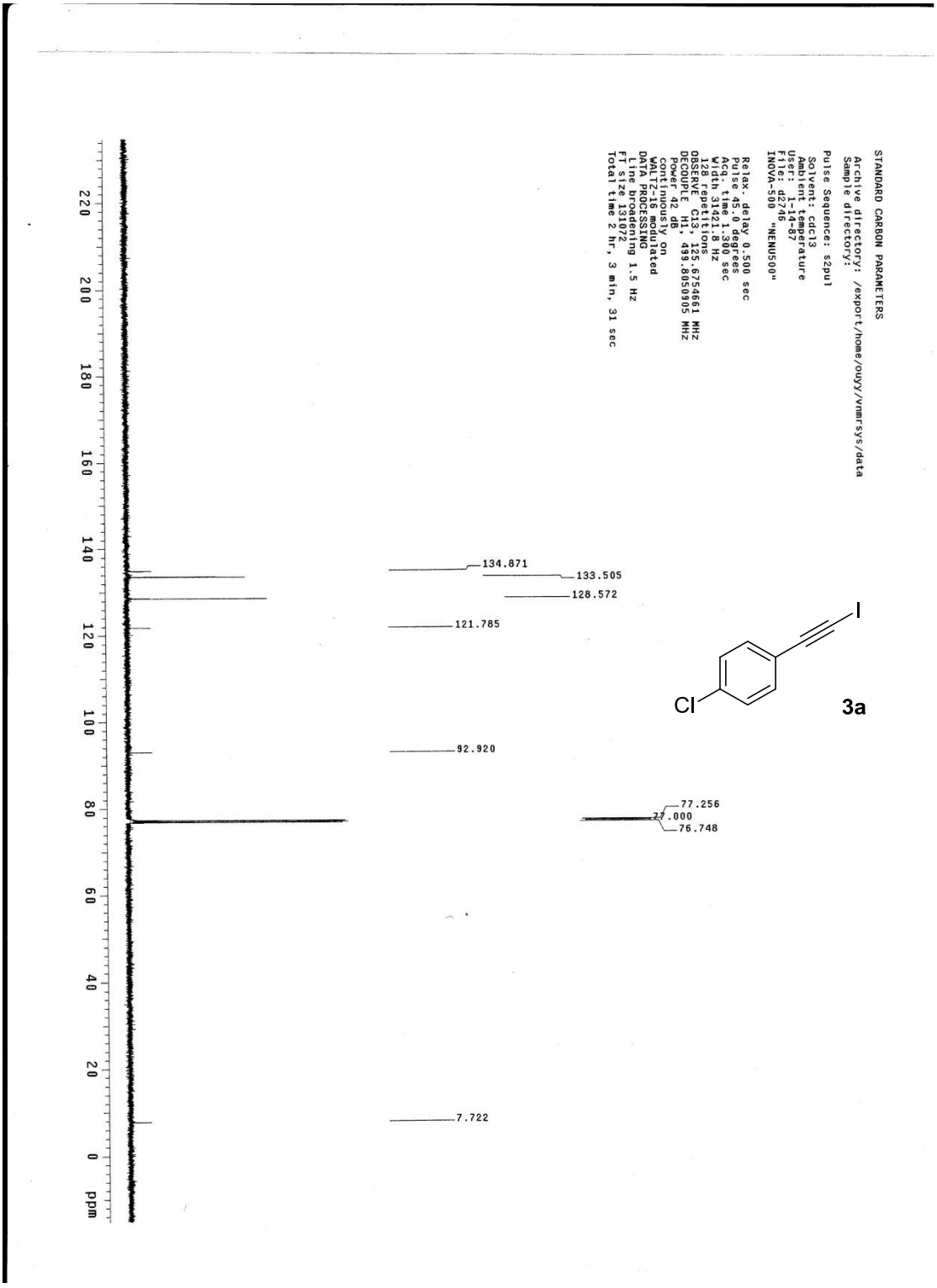

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2699
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.897 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8026176 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
Ph
O
Br
2k
7.341
7.333
4.571
4.171
1.00
0.28
0.52
0.52
ppm

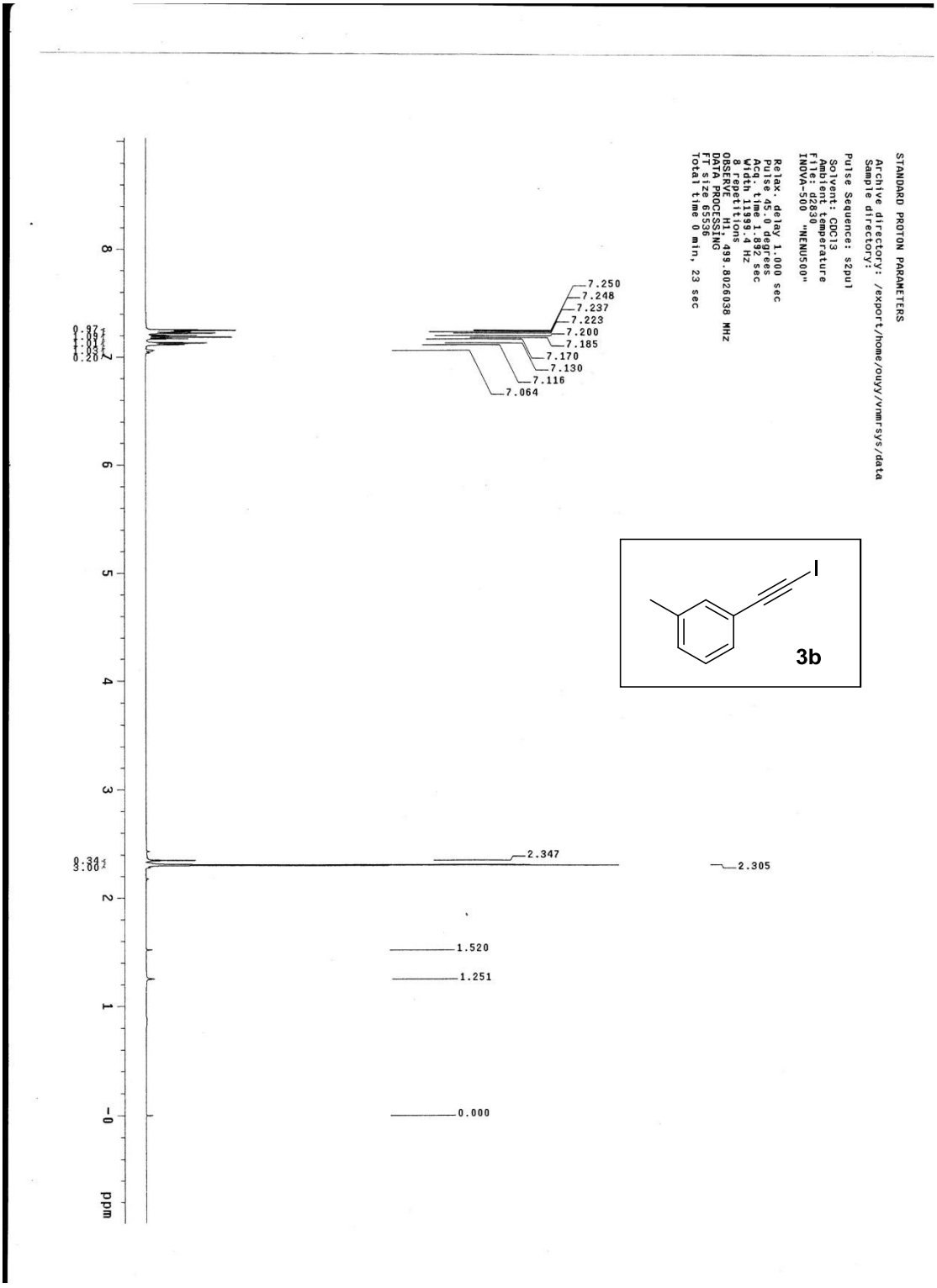

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2703
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 31621.8 Hz
64 repetitions
OBSERVE C13, 125.6754805 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
137.045
128.374
128.000
127.866
77.256
77.000
76.748
76.123
71.594
57.940
46.121
Ph
O
Br
2k
220
200
180
160
140
120
100
80
60
40
20
0
ppm

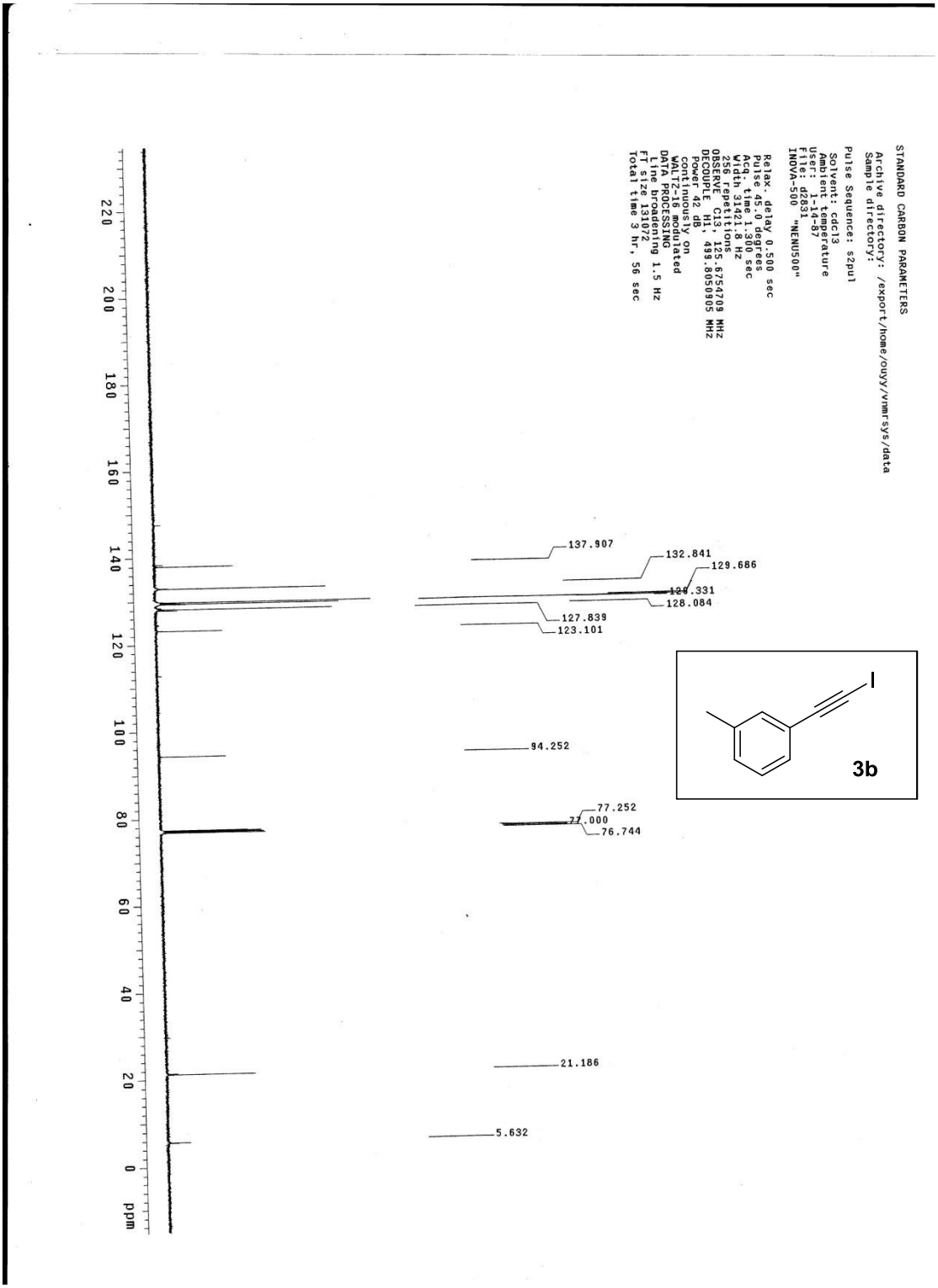

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0214
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8025893 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.265
6.152
6.148
6.144
6.140
6.136
2.123
2.111
2.107
2.094
2.082
2.077
2.075
2.070
2.065
1.647
1.636
1.628
1.624
1.616
1.613
1.605
1.601
1.592
1.588
1.580
1.576
1.569
1.565
1.558
1.546
1.00
4.47
4.63
Br
2l
ppm

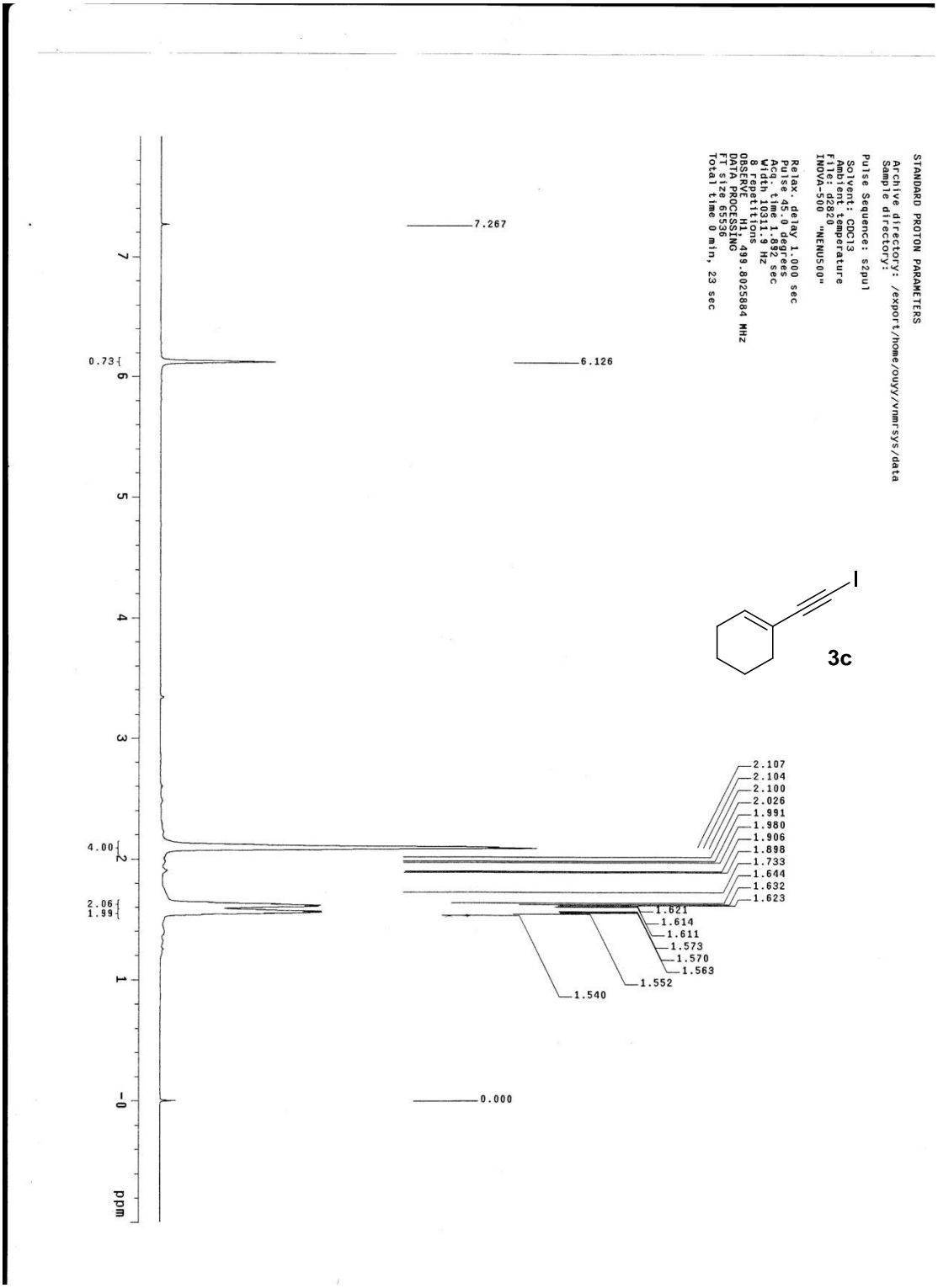

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ll-ouyy-07
File: d2715
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.675606 MHz
DECOUPLE H1, 499.805905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
136.378
120.415
81.826
77.252
77.000
76.744
46.224
28.812
25.562
22.143
21.334
Br
2l
220
200
180
160
140
120
100
80
60
40
20
0
ppm

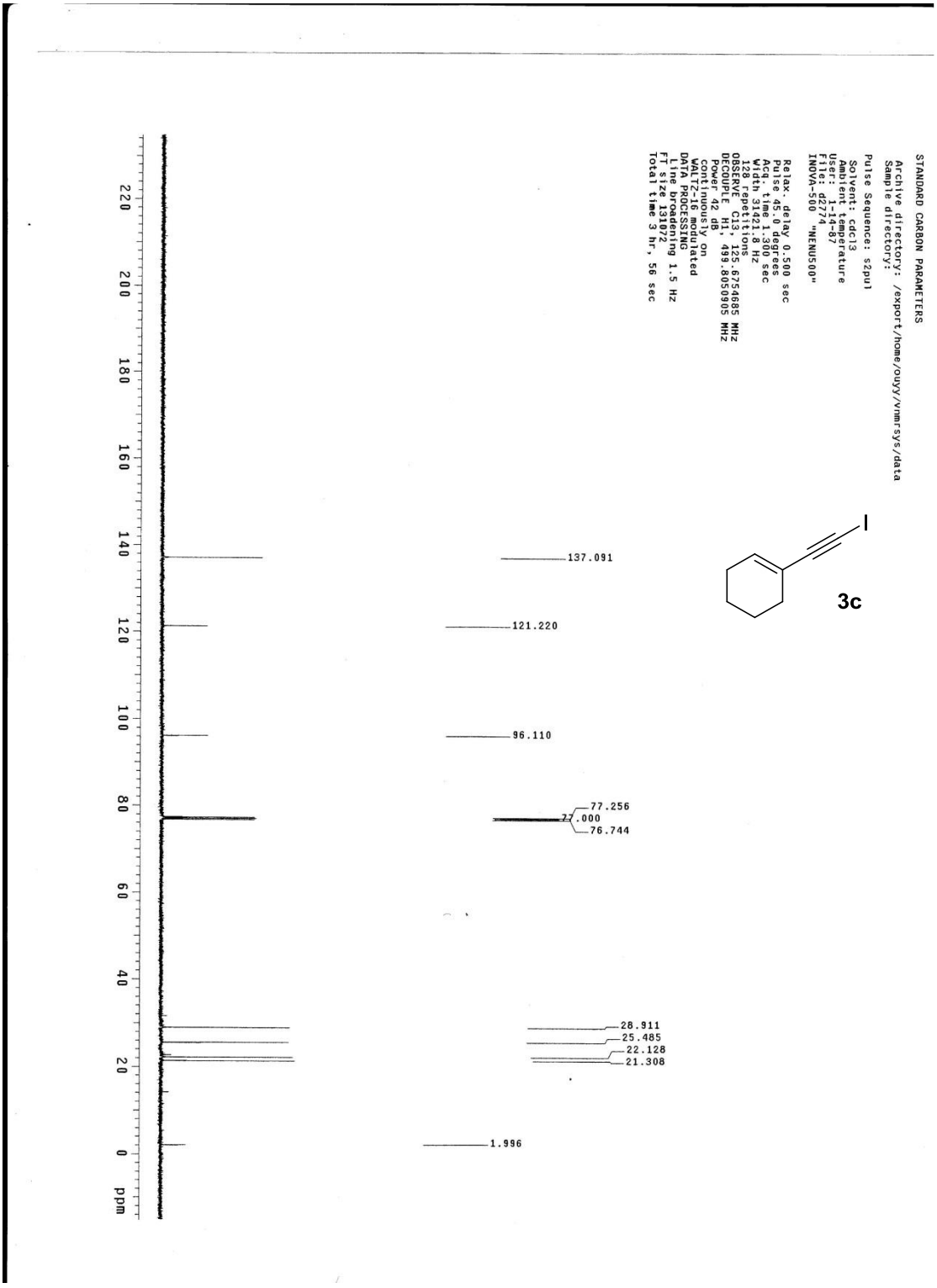

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2744
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8822931 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.366
7.349
7.292
7.275
7.257
1.549
-0.000
2.00
2.00
8
7
6
5
4
3
2
1
-0
ppm
I
Cl
3a

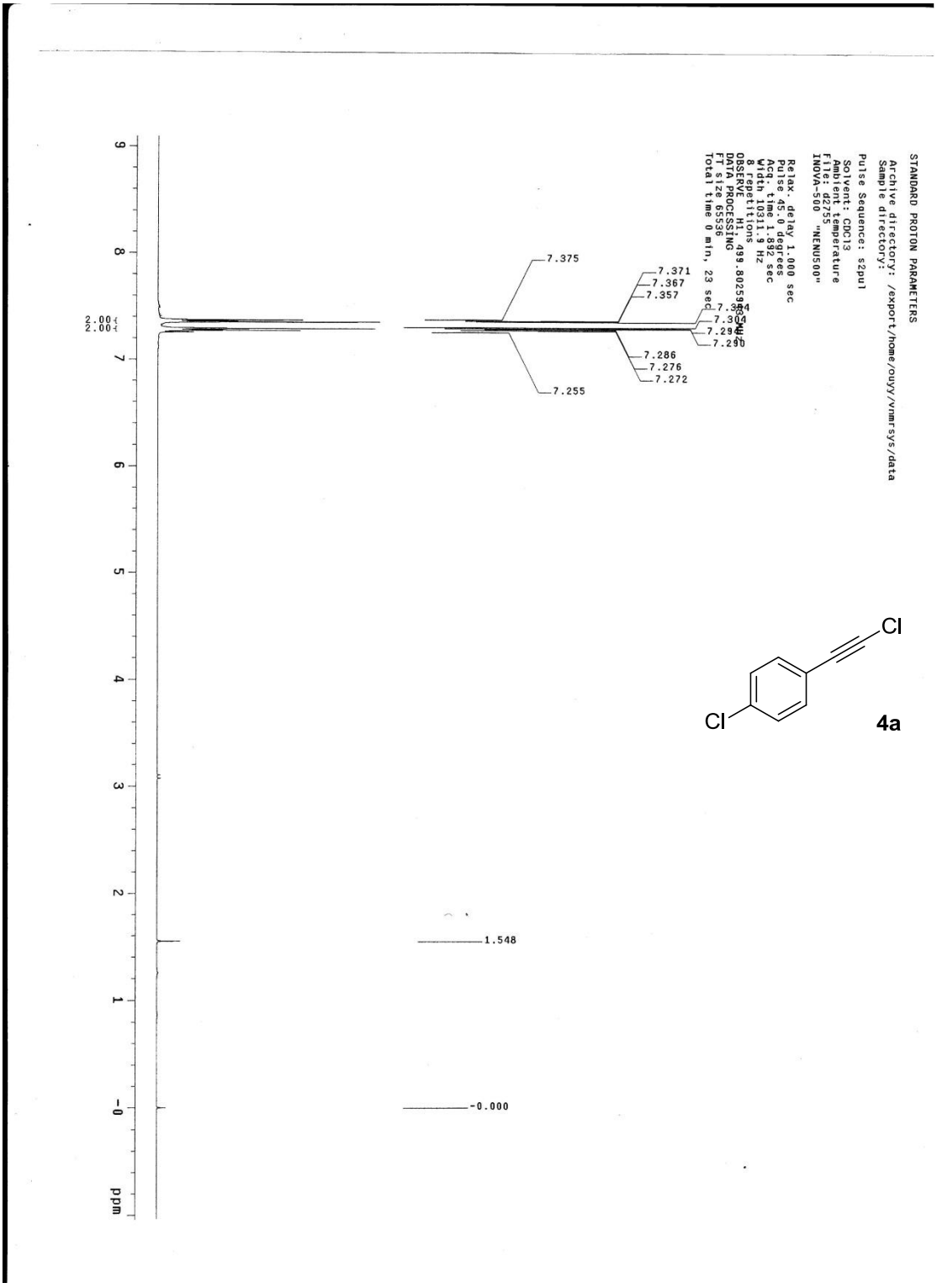

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ll-14-87
File: d2746
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754661 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
134.871
133.505
128.572
121.785
92.920
77.256
77.000
76.748
7.722
220
200
180
160
140
120
100
80
60
40
20
0
ppm
I
Cl
3a

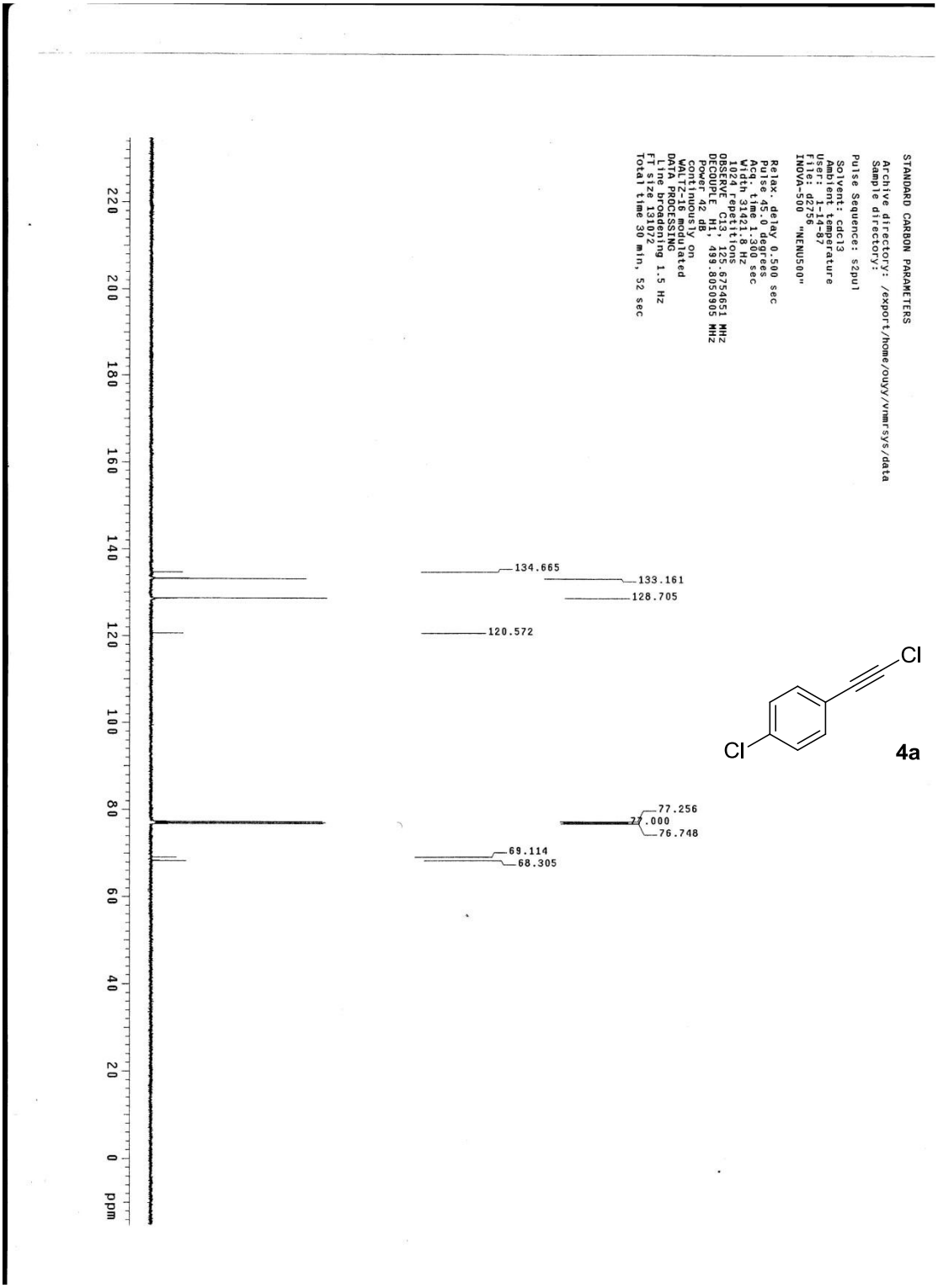

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11999.4 Hz
8 repetitions
OBSERVE H1, 499.8026038 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.250
7.248
7.237
7.223
7.200
7.185
7.170
7.130
7.116
7.064
2.347
2.305
1.520
1.251
0.000
3b
ppm

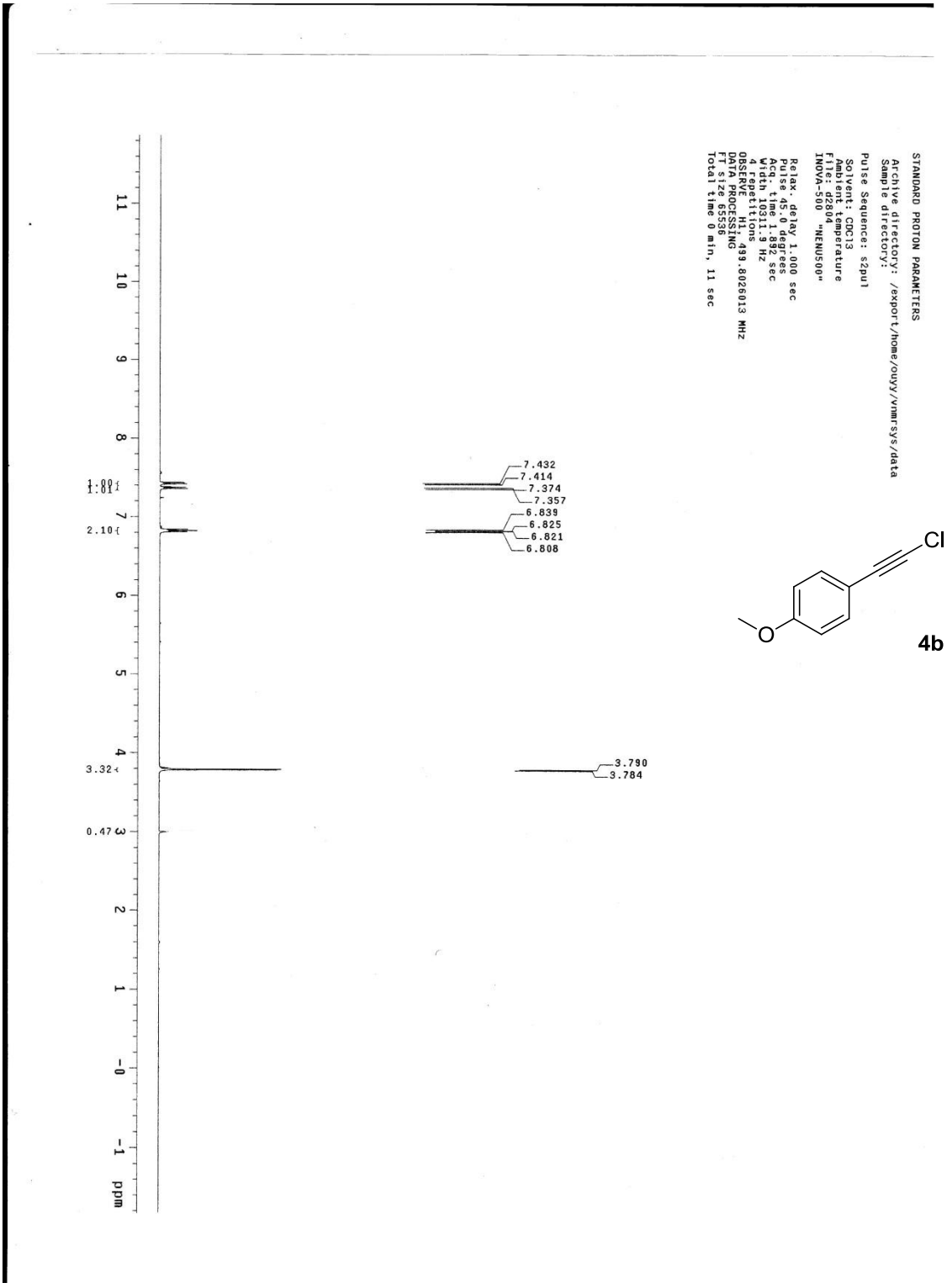

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy
File: d2831
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
137.907
132.841
129.686
128.331
128.084
127.839
123.101
94.252
77.252
77.000
76.744
21.186
5.632
I
3b
220
200
180
160
140
120
100
80
60
40
20
0
ppm

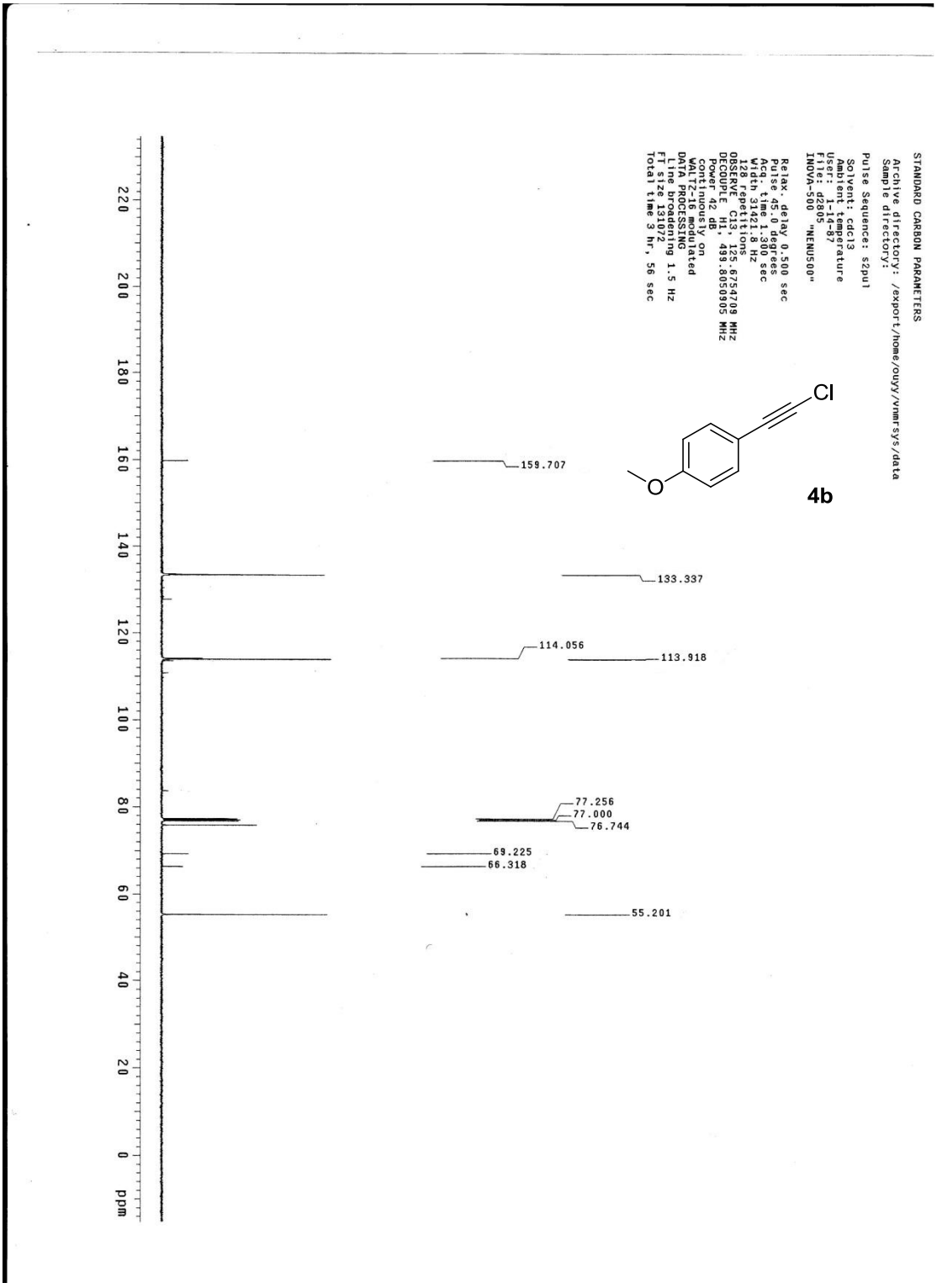

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2820
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
8 repetitions
OBSERVE H1, 499.8025884 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.267
6.126
0.000
0.73
4.00
2.06
1.99
2.107
2.104
2.100
2.026
1.991
1.980
1.906
1.898
1.733
1.644
1.632
1.623
1.621
1.614
1.611
1.573
1.570
1.563
1.552
1.540
I
3c
ppm

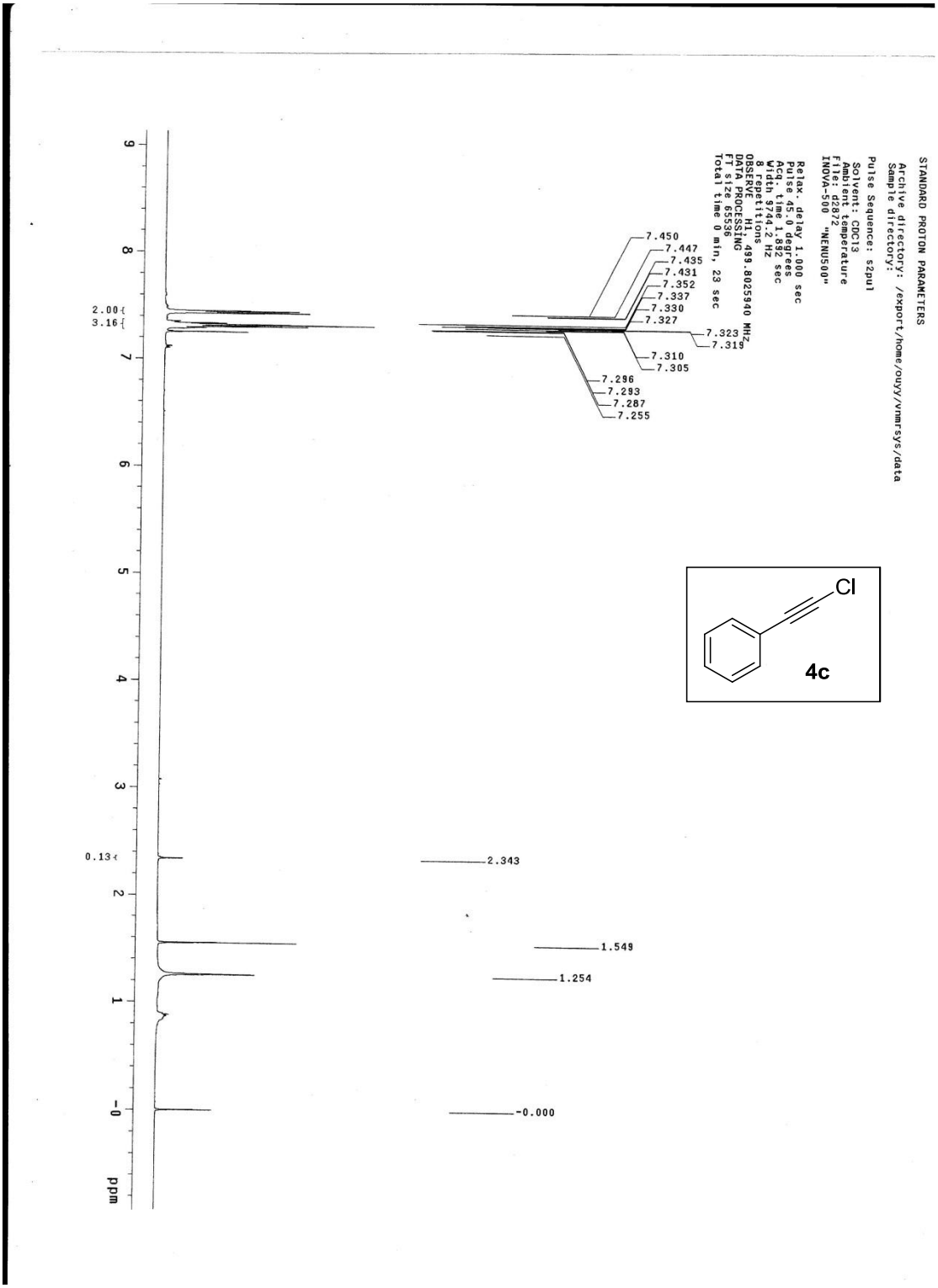

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-07
File: d2774
INOVA-500 "MENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754685 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
137.091
121.220
96.110
77.256
77.000
76.744
28.911
25.485
22.128
21.308
1.996
ppm
3c

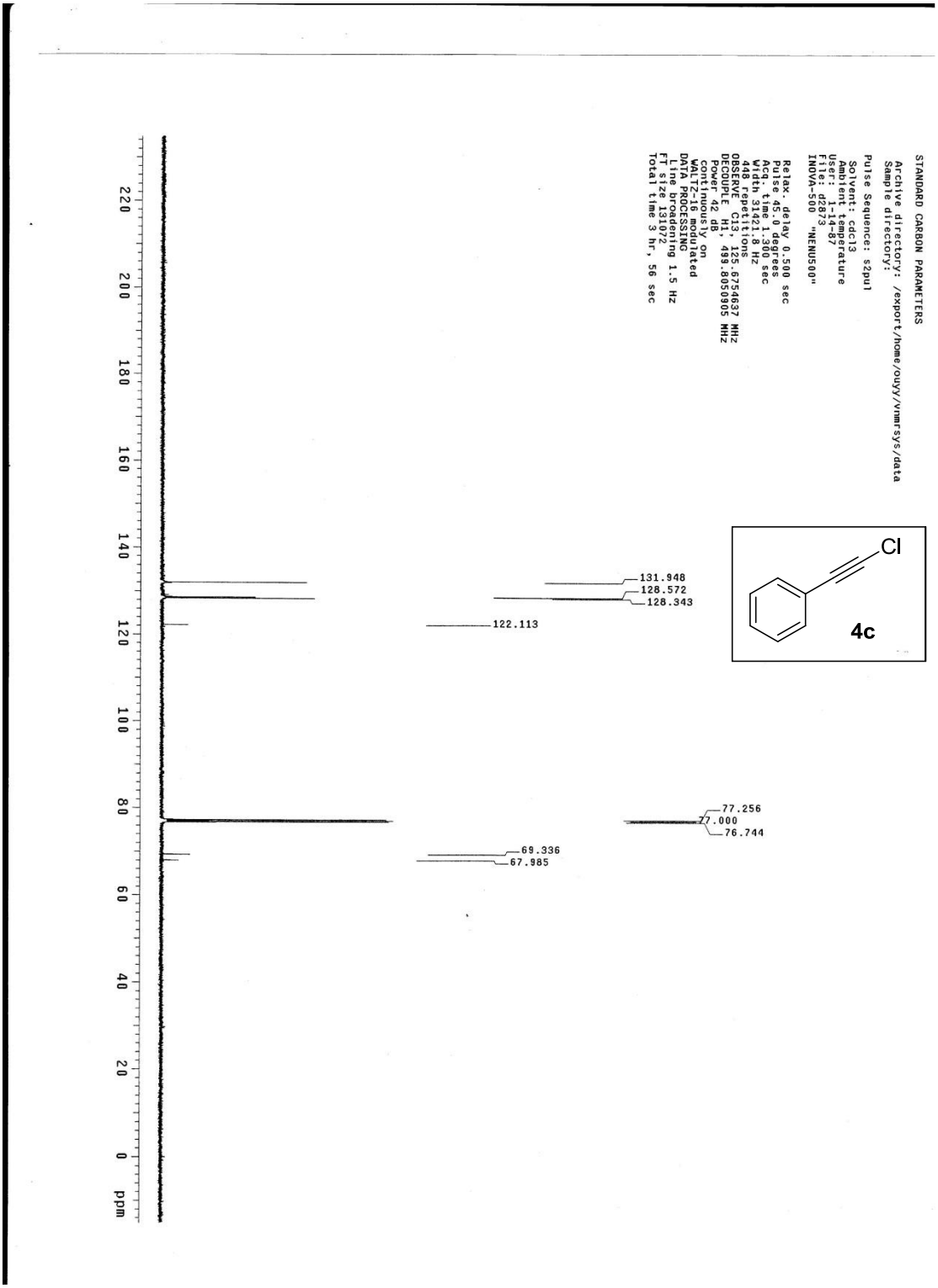

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d0275
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
8 repetitions
OBSERVE H1, 499.8025994 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.375
7.371
7.367
7.357
7.294
7.290
7.286
7.276
7.272
7.255
2.00
2.00
1.548
-0.000
ppm
Cl
Cl
4a

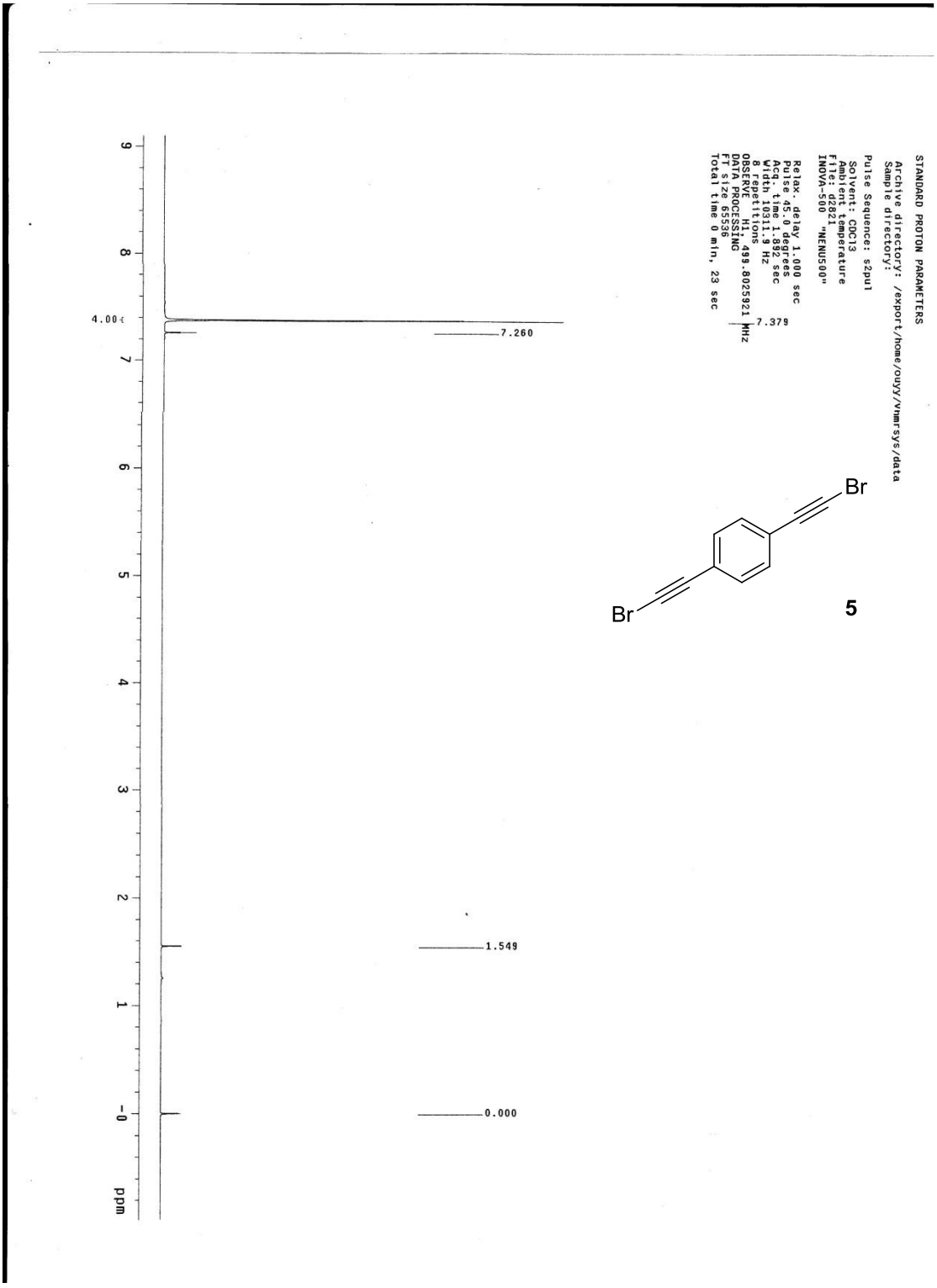

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-07
File: d2756
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 32154.3 Hz
1024 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 30 min, 52 sec
134.665
133.161
128.705
120.572
77.256
77.000
76.748
69.114
68.305
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Cl
Cl
4a

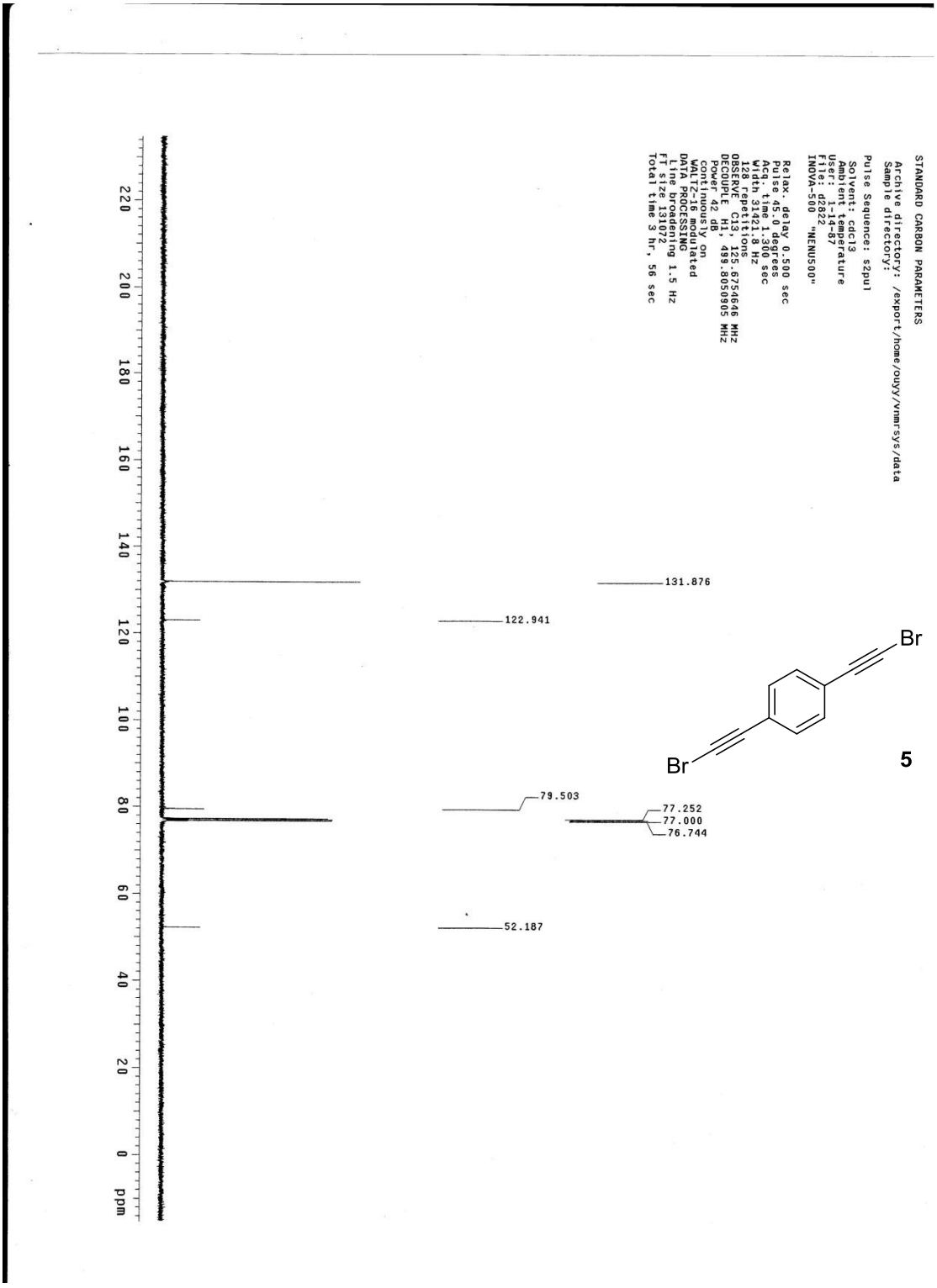

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2804
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
width 10311.9 Hz
1 repetitions
OBSERVE H1, 499.8026013 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.432
7.414
7.374
7.357
6.839
6.825
6.821
6.808
3.790
3.784
1.00
1.01
2.10
3.32
0.47
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
Cl
O
4b

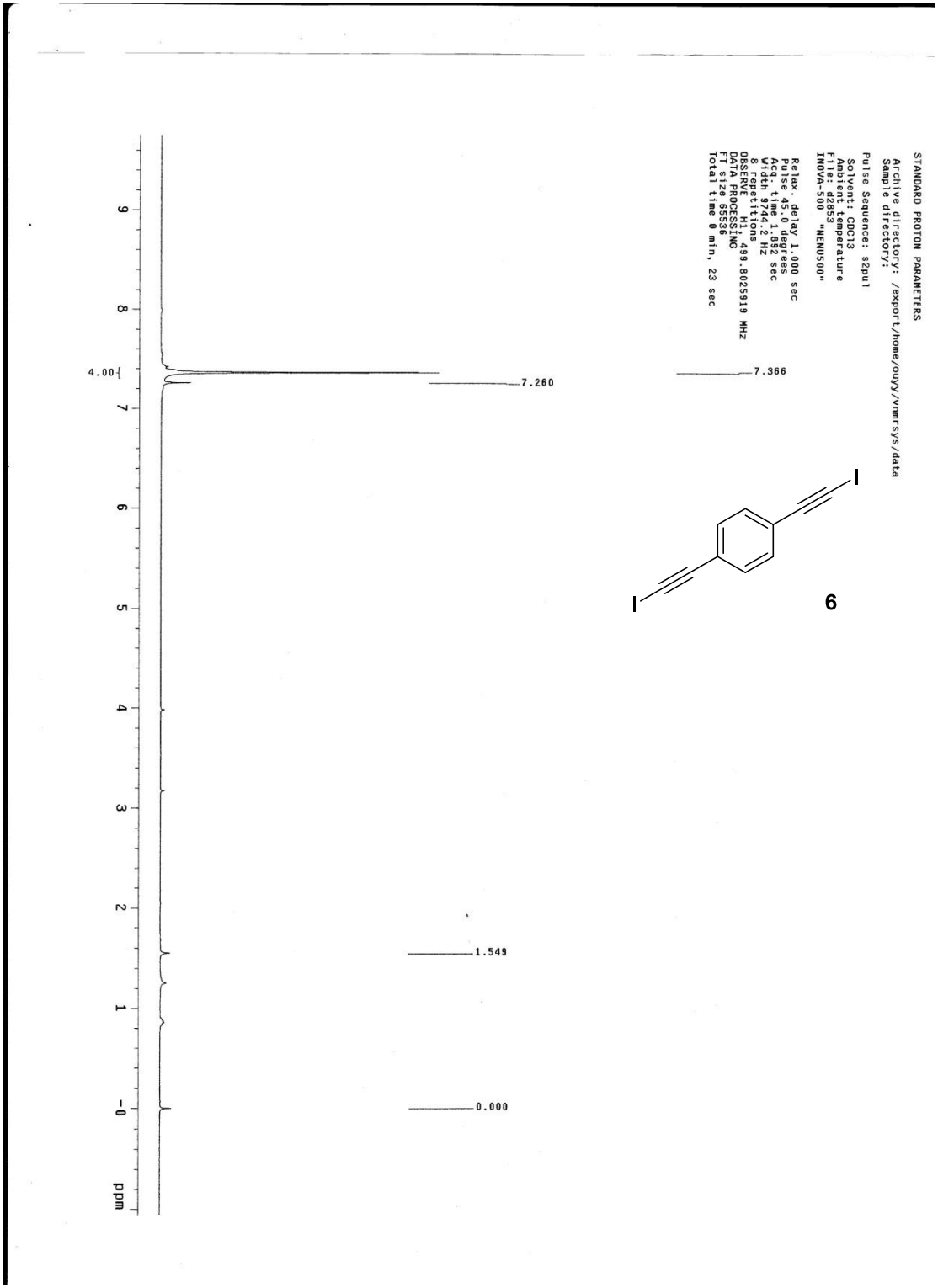

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2805
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31347.8 Hz
128 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
O
4b
159.707
133.337
114.056
113.918
77.256
77.000
76.744
69.225
66.318
55.201
220
200
180
160
140
120
100
80
60
40
20
0
ppm

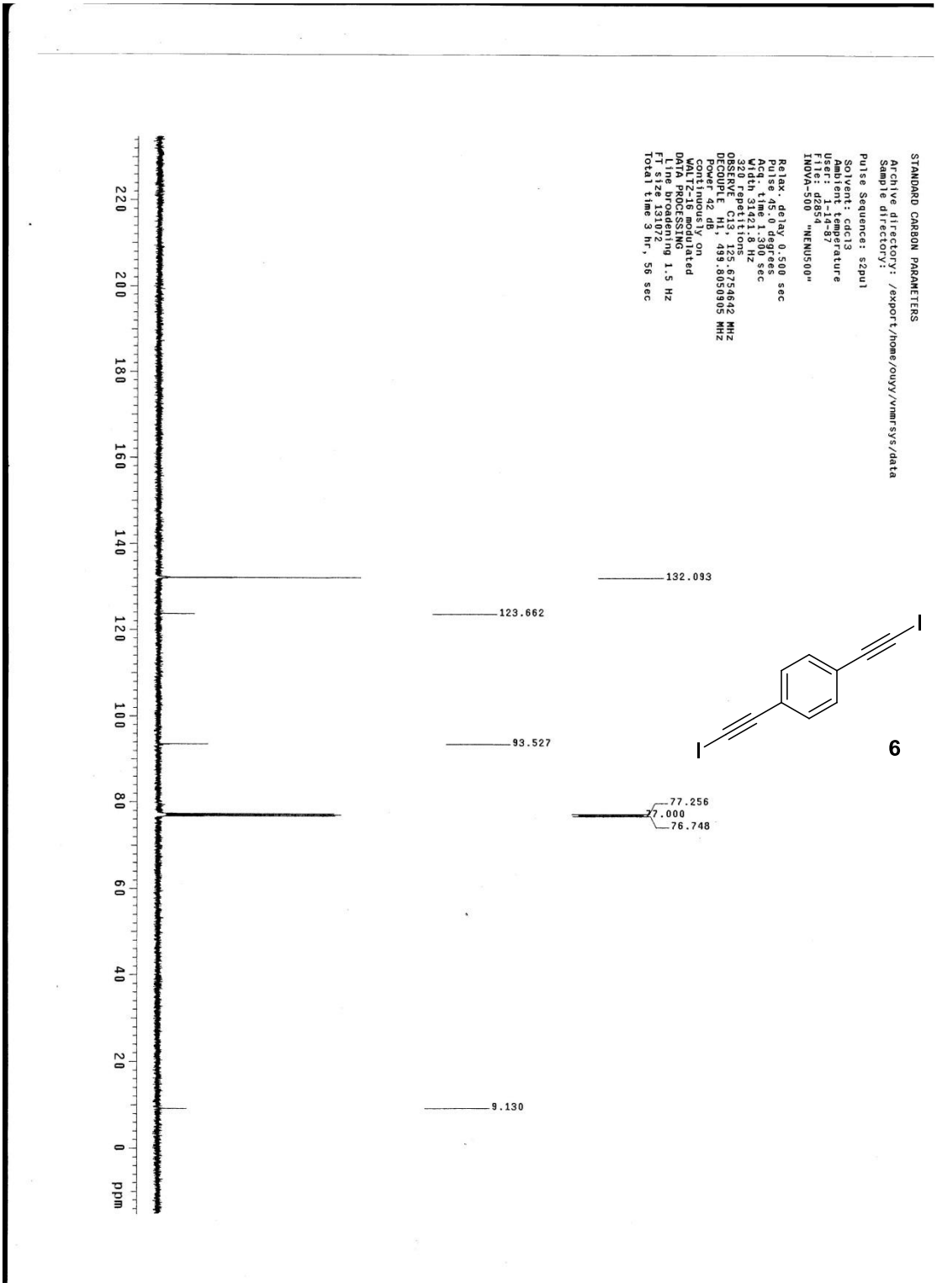

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2872
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.492 sec
Width 9744.2 Hz
8 repetitions
OBSERVE H1, 499.8025940 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.450
7.447
7.435
7.431
7.352
7.337
7.330
7.327
7.323
7.319
7.310
7.305
7.296
7.293
7.287
7.255
2.343
1.549
1.254
-0.000
2.00
3.16
0.13
Cl
4c
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2873
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 32102.1 Hz
448 repetitions
OBSERVE C13, 125.6754637 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
4c
131.948
128.572
128.343
122.113
77.256
77.000
76.744
69.336
67.985
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2021
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8119.5 Hz
8 repetitions
OBSERVE H1, 499.8025921 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.379
7.260
1.549
0.000
4.00
ppm
Br
Br
5


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2822
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
131.876
122.941
79.503
77.252
77.000
76.744
52.187
Br
Br
5
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2853
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9744.2 Hz
8 repetitions
OBSERVE H1, 499.8025919 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.366
7.260
1.549
0.000
4.00
9
8
7
6
5
4
3
2
1
-0
ppm
I
I
6


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2854
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
32 repetitions
OBSERVE C13, 125.6756642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
132.093
123.662
93.527
77.256
77.000
76.748
9.130
220
200
180
160
140
120
100
80
60
40
20
0
ppm
6