Electronic Supplementary Material (ESI) for RSC Advances.

Direct α-C-H amination of β-dicarbonyl compounds using DBU-activated *N*-haloimides as nitrogen sources

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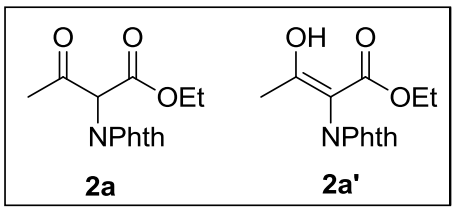
I. General

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 BruckmicroTof by using ESI method.

II. Synthesis and analytical data of 2-4 and 6

General procedure for the preparation of **2** (**2a** and **2a'** as an example): To a solution of ethyl 3-oxobutanoate **1a** (0.128 mL, 1.0 mmol) in MeCN (2.0 mL) was added NBP (339.0 mg, 1.5 mmol) and DBU (0.261 mL, 1.8 mmol). The mixture was stirred at room temperature for 20 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 : ethyl acetate = 80 : 3 as eluent) to give **2a** and **2a'** (245.0 mg, 89%) as colorless oil.

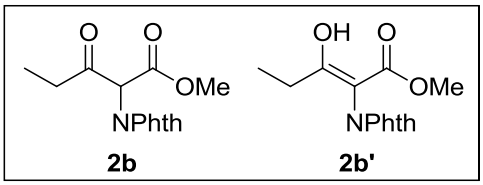
Ethyl 2-(1, 3-dioxoisoindolin-2-yl)-3-oxobutanoate (2a) and ethyl 2-(1, 3-dioxoisoindolin-2-yl)-3-hydroxybut-2-enoate (2a')



Colorless oil. mixture ketone/enol form 1/4.3. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7.0 Hz, 3H, enol form), 1.31 (t, *J* = 7.0 Hz, 3H, ketone form), 1.97 (s, 1H, enol form), 2.48 (s, 3H, ketone form), 4.17-4.22 (m, 2H, enol form), 4.31 (t, *J* = 7.5 Hz, 3H, ketone form), 7.78-7.79 (m, 2H, ketone form), 7.80-7.82 (m, 2H, enol form), 7.90-7.93 (m, 2H, ketone form), 7.94 (t, *J* = 2.8 Hz, enol form), 12.78 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.8, 13.9, 17.9, 28.3, 60.6, 61.2, 62.5, 96.1, 123.6, 123.6, 131.5, 131.7, 134.2, 164.7, 166.6, 167.2, 169.0, 176,7 196.3; HRMS

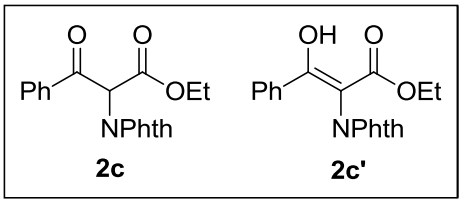
(ESI) m/z calcd for $C_{14}H_{13}NO_5$ $[M+H]^+$: 276.0872; found: 276.0881.

Ethyl 2-(1, 3-dioxoisoindolin-2-yl)-3-oxopentanoate (2b) and ethyl 2-(1, 3-dioxoisoindolin-2-yl)-3-hydroxypent-2-enoate (2b')



Colorless oil. mixture ketone/enol form 1/3. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 6.0 Hz, 6H, ketone form and enol form), 2.22-2.26 (m, 2H, enol form), 2.67-2.73 (m, 1H, ketone form), 2.86-2.92 (m, 1H, ketone form), 3.71 (s, 3H, enol form), 3.84 (s, 3H, ketone form), 5.48 (s, 1H, ketone form), 7.77-7.82 (m, 4H, ketone form and enol form), 7.90-7.92 (m, 2H, ketone form); 7.94 (t, *J* = 2.8 Hz, 2H, enol form), 12.81 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 7.3, 10.0, 24.5, 33.9, 52.0, 52.9, 59.7, 94.8, 123.5, 131.4, 131.5, 134.2, 134.2, 165.5, 166.5, 167.3, 169.4, 180.7, 199.2; HRMS (ESI) m/z calcd for $C_{14}H_{13}NO_5$ $[M+H]^+$: 276.0872; found: 276.0878.

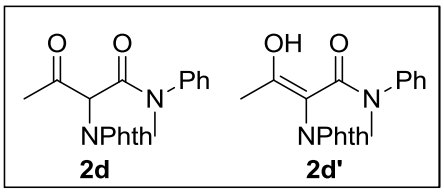
Ethyl 2-(1,3-dioxoisoindolin-2-yl)-3-oxo-3-phenylpropanoate (2c) and ethyl 2-(1,3-dioxoisoindolin-2-yl)-3-hydroxy-3-phenylacrylate (2b')



Colorless oil. mixture ketone/enol form 1.3/1. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.21 (t, *J* = 13.0 Hz, 3H, enol form), 1.28 (t, *J* = 7.0 Hz, 3H, ketone form), 4.23-4.30 (m, 2H, enol form), 4.31-4.35 (m, 2H, ketone form), 6.32 (s, 1H, ketone form), 7.28 (t, *J* = 7.3 Hz, 2H, ketone form), 7.35 (t, *J* = 7.5 Hz, 1H, enol form), 7.45-7.51 (m, 3H, ketone form), 7.58 (t, *J* = 7.5 Hz, 1H, ketone form), 7.73-7.77 (m, 5H, enol form), 7.85 (t, *J* = 2.5 Hz, 3H, enol form), 7.86-7.91 (m, 3H, ketone form), 13.19 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.6, 13.7, 57.0, 61.5, 62.3, 95.6, 123.4,

123.4, 126.8, 128.0, 128.1, 128.5, 130.7, 131.2, 131.3, 132.1, 133.5, 134.1, 134.2, 134.6, 165.2, 166.4, 167.4, 169.7, 174.4, 189.2; HRMS (ESI) m/z calcd for $C_{19}H_{15}NO_5$ $[M+H]^+$: 338.1028; found: 338.1022.

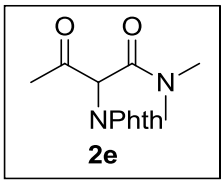
2-(1, 3-Dioxoisoindolin-2-yl)-*N*-methyl-3-oxo-*N*-phenylbutanamide (2d) and 2-(1, 3-dioxoisoindolin-2-yl)-3-hydroxy-*N*-methyl-*N*-phenylbut-2-enamide (2d')



2d **2d'**

Colorless oil. mixture ketone/enol form 1/1.7. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.67 (s, 3H, enol form), 2.21 (s, 3H, ketone form), 3.26 (s, 3H, enol form), 3.34 (s, 3H, ketone form), 5.45 (s, 1H, ketone form), 6.61 (d, *J* = 7.5 Hz, 1H, enol form), 6.88 (t, *J* = 7.8 Hz, 2H, enol form), 6.97 (d, *J* = 7.5 Hz, 2H, enol form), 7.25 (t, *J* = 10.0 Hz, 2H, ketone form), 7.32 (t, *J* = 7.0 Hz, 3H, ketone form), 7.55-7.61 (m, 4H, enol form), 7.73-7.75 (m, 2H, ketone form), 7.81-7.83 (m, 2H, ketone form), 15.80 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 18.3, 27.6, 37.6, 39.7, 59.9, 95.4, 122.5, 123.2, 125.5, 126.5, 128.1, 128.9, 129.5, 130.9, 131.1, 133.5, 134.1, 141.7, 142.2, 164.0, 166.4, 166.6, 168.7, 178.0, 197.6; HRMS (ESI) m/z calcd for $C_{19}H_{16}N_2O_4$ $[M+H]^+$: 337.1188; found: 337.1181.

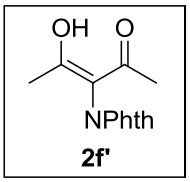
2-(1, 3-Dioxoisoindolin-2-yl)-*N*, *N*-dimethyl-3-oxobutanamide (2e)



2e

White solid. m.p. 130-132 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.35 (s, 3H), 2.98 (s, 3H), 3.03 (s, 3H), 5.62(s, 1H), 7.77-7.79 (m, 2H), 7.90-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 28.0, 36.0, 37.2, 60.4, 123.8, 131.7, 134.5, 164.0, 167.3, 197.8; HRMS (ESI) m/z calcd for $C_{14}H_{14}N_2O_4$ $[M+H]^+$: 275.1023; found: 275.1029.

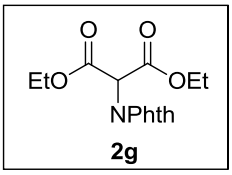
2-(2-Hydroxy-4-oxopent-2-en-3-yl)isoindoline-1, 3-dione (2f')



2f'

White solid. m.p. 114-116 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.00 (s, 6H), 7.84-7.85 (m, 2H), 7.96-7.98 (m, 2H), 16.23 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 21.8, 106.4, 124.0, 131.5, 134.7, 167.4, 191.1; HRMS (ESI) m/z calcd for $C_{13}H_{11}NO_4$ $[M+H]^+$: 246.0766, found 246.0761.

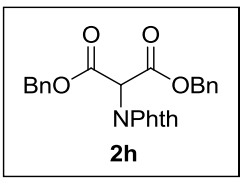
Diethyl 2-(1, 3-dioxoisoindolin-2-yl)malonate (2g)



2g

White solid. m.p. 52-54 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.32 (t, *J* = 7.5 Hz, 6H), 4.27-4.38 (m, 4H), 5.49 (s, 1H), 7.77-7.80 (m, 2H), 7.90-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.9, 54.4, 62.8, 123.8, 131.7, 134.4, 164. 3, 166.5; HRMS (ESI) m/z calcd for $C_{15}H_{15}NO_6$ $[M+H]^+$: 306.0978; found: 306.0969.

Dibenzyl 2-(1,3-dioxo-2, 3-dihydro-1*H*-inden-2-yl)malonate (2h)

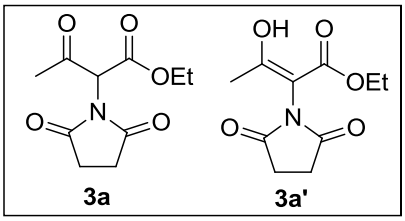


2h

White solid. m.p. 101-103 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.21-5.28 (m, 4H), 5.60 (s, 1H), 7.31 (s, 10H), 7.75 (t, *J* = 4.2 Hz, 2H), 7.88 (t, *J* = 3.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 54.4, 68.4, 123.5, 123.8, 128.3, 128.4, 128.5, 131.6, 134.2, 134.4, 134.5, 164.1, 166.4; HRMS (ESI) m/z calcd for $C_{25}H_{19}NO_6$ $[M+H]^+$: 430.1291; found: 430.1298.

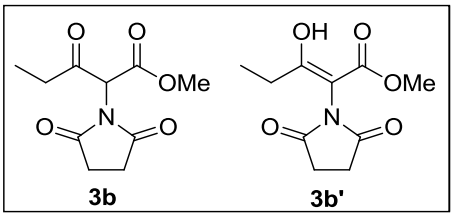
Ethyl 2-(2,5-dioxopyrrolidin-1-yl)-3-oxobutanoate (3a) and ethyl 2-(2,5-

dioxopyrrolidin-1-yl)-3-oxobutanoatec (3a')



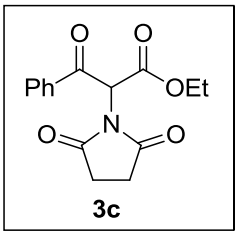
Yellow oil. mixture ketone/enol form 1.2/1.7. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.23 (t, *J* = 7.0 Hz, 3H, enol form), 1.31 (t, *J* = 7.2 Hz, 3H, ketone form), 1.90 (s, 3H, enol form), 2.44 (s, 1H, ketone form), 2.79-2.89 (m, 8H, ketone form and enol form), 4.18-4.23 (m, 2H, enol form), 4.28-4.32 (m, 2H, ketone form), 5.28 (s, 1H, ketone form), 12.66 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.8, 13.9, 17.7, 28.0, 18.1, 28.5, 61.0, 61.3, 62.6, 96.8, 164.3, 168.3, 175.5, 175.8, 176.0, 195.6; HRMS (ESI) m/z calcd for $C_{10}H_{13}NO_5$ $[M+H]^+$: 228.0872; found: 228.0880.

Methyl 2-(2,5-dioxopyrrolidin-1-yl)-3-oxopentanoate (3b) and methyl 2-(2,5-dioxopyrrolidin-1-yl)-3-hydroxypent-2-enoate (3b')



Yellow oil. mixture ketone/enol form 1.49/1.56. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.11-1.14 (m, 6H, ketone form and enol form), 2.12 (d, *J* = 7.5 Hz, 2H, ketone form), 2.16 (t, *J* = 7 Hz, 2H, enol form), 2.79-2.91 (m, 8H, ketone form and enol form), 3.73 (s, 3H, ketone form), 3.82 (s, 3H, enol form), 5.30 (s, 1H, ketone form), 12.70 (s, 1H, enol form); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 7.2, 9.8, 24.2, 27.9, 33.9, 52.0, 52.8, 60.0, 95.4, 164.9, 168.8, 175.5, 176.1, 179.7, 198.6; HRMS (ESI) m/z calcd for $C_{10}H_{13}NO_5$ $[M+H]^+$: 228.0872; found: 228.0891.

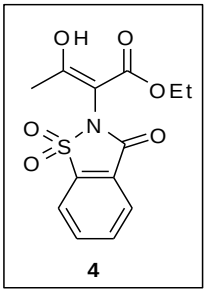
Ethyl 2-(2,5-dioxopyrrolidin-1-yl)-3-oxo-3-phenylpropanoate (3c)



3c

White solid. m.p. 80-82 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.20 (t, *J* = 6.8 Hz, 3H), 2.79 (s, 4H), 4.22-4.26 (m, 2H), 6.15 (s, 1H), 7.45 (t, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 6.8 Hz, 1H), 8.84 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.8, 28.2, 57.7, 62.6, 128.2, 128.7, 133.8, 135.0, 164.9, 175.7, 188.9; HRMS (ESI) m/z calcd for $C_{15}H_{15}NO_5$ $[M+H]^+$: 290.1028; found: 290.1020.

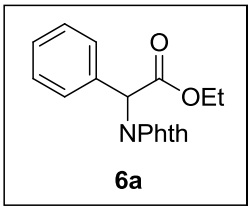
Ethyl 2-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)-3-hydroxybut-2-enoate (4)



4

White solid. m.p. 91-93 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.19 (t, *J* = 7.0 Hz, 3H), 2.17 (s, 3H), 4.18-4.23 (m, 1H), 4.24-4.29 (m, 1H), 7.90 (t, *J* = 7.5 Hz, 1H), 7.94 (t, *J* = 7.5 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 8.14 (d, *J* = 7.5 Hz, 1H), 13.22 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 13.9, 18.6, 61.7, 93.0, 121.4, 125.6, 126.8, 134.4, 135.2, 137.8, 158.6, 169.3, 181.6; HRMS (ESI) m/z calcd for $C_{13}H_{13}NO_6S$ $[M+H]^+$: 312.0542; found: 312.0549.

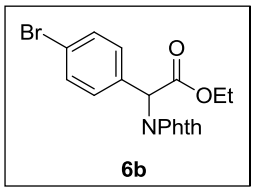
Ethyl 2-(1,3-dioxoisoindolin-2-yl)-2-phenylacetate (6a)



6a

White solid. m.p. 76-78 ℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.26 (t, *J* = 7.3, 3H), 4.28-4.32 (m, 2H), 6.01 (s, 1H), 7.33-7.38 (m, 3H), 7.55 (d, *J* = 7.0 Hz, 2H), 7.71-7.73 (m, 2H), 7.84-7.86 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 14.1, 55.8, 62.2, 123.5, 128.5, 128.5, 129.7, 131.7, 134.2, 134.5, 167.1, 167.9; HRMS (ESI) m/z calcd for $C_{18}H_{15}NO_4$ $[M+H]^+$: 310.1079; found: 310.1071.

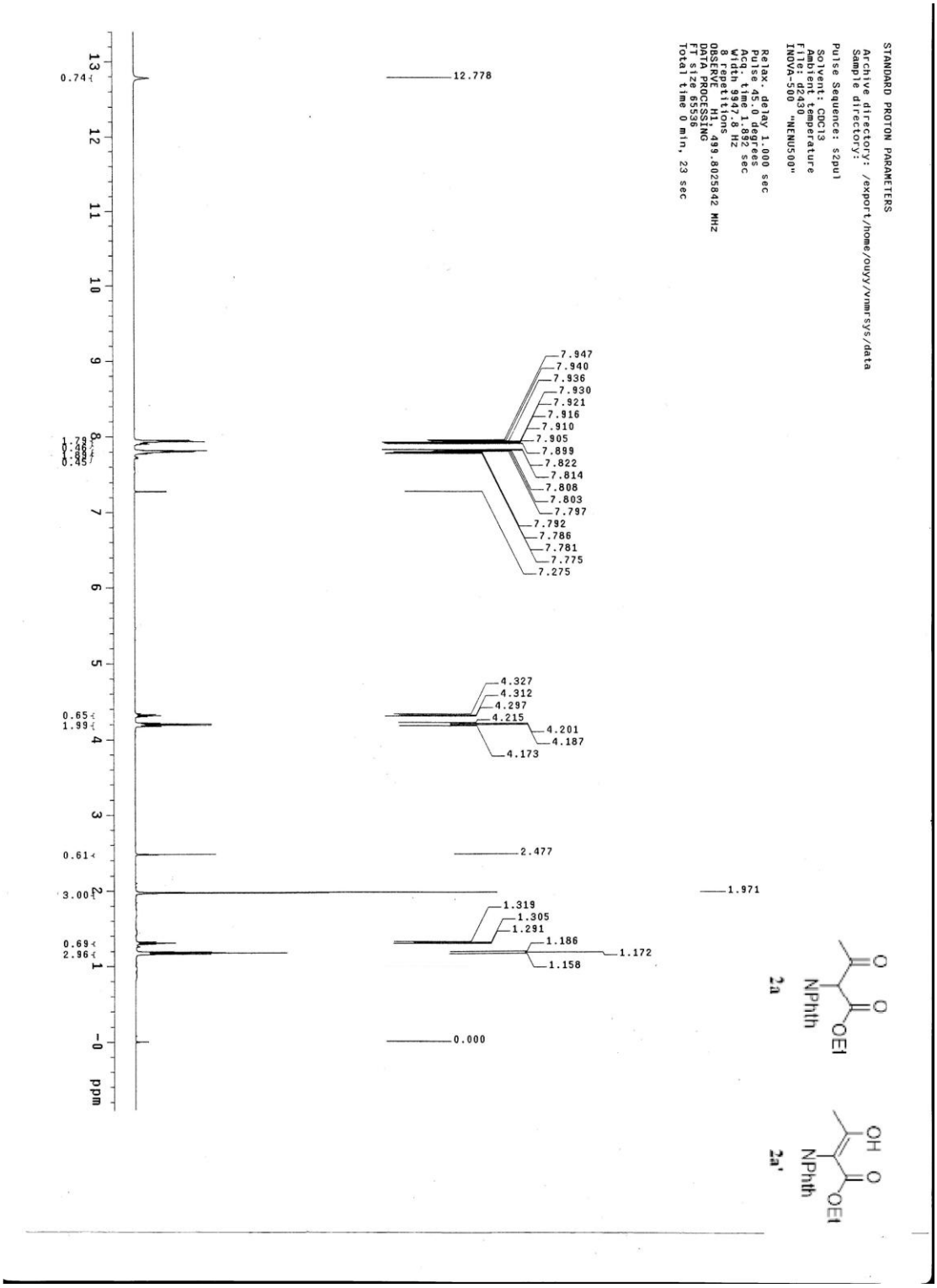
Ethyl 2-(4-bromophenyl)-2-(1,3-dioxoisoindolin-2-yl)acetate (6b)

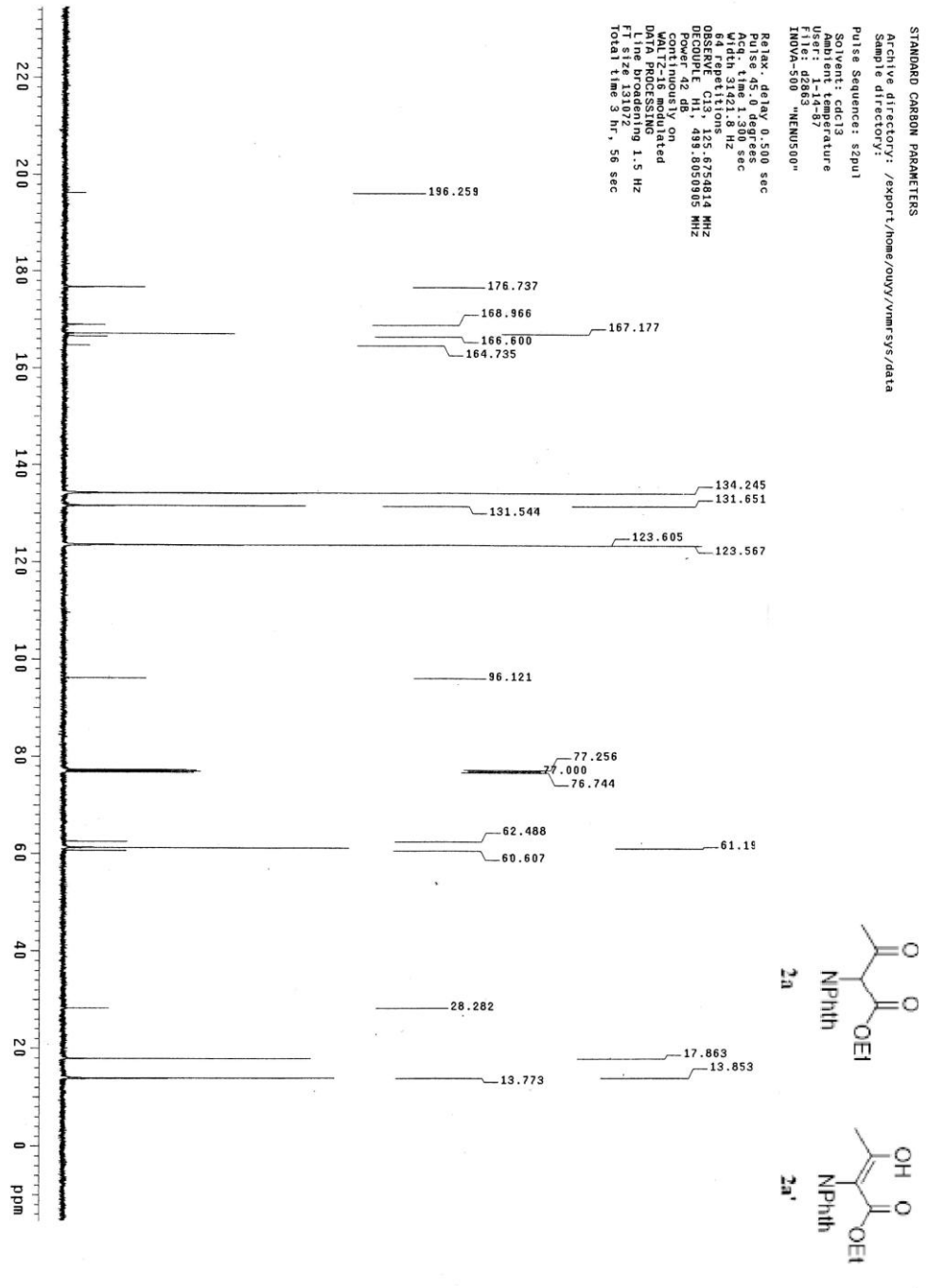


6b

Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.25 (t, *J* = 7.0 Hz, 3H), 4.27-4.31 (m, 2H), 5.95 (s, 1H), 7.3 (d, *J* = 9.0 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.72-7.75 (m, 2H), 7.85-7.86 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 14.0, 55.1, 62.4, 122.8, 123.6, 131.4, 131.6, 133.5, 134.3, 167.0, 167.5; HRMS (ESI) m/z calcd for $C_{18}H_{14}BrNO_4$ $[M+H]^+$: 388.0184; found: 388.0178.

III. Copies of ^{1}H and ^{13}C NMR spectra for compounds 2-4 and 6

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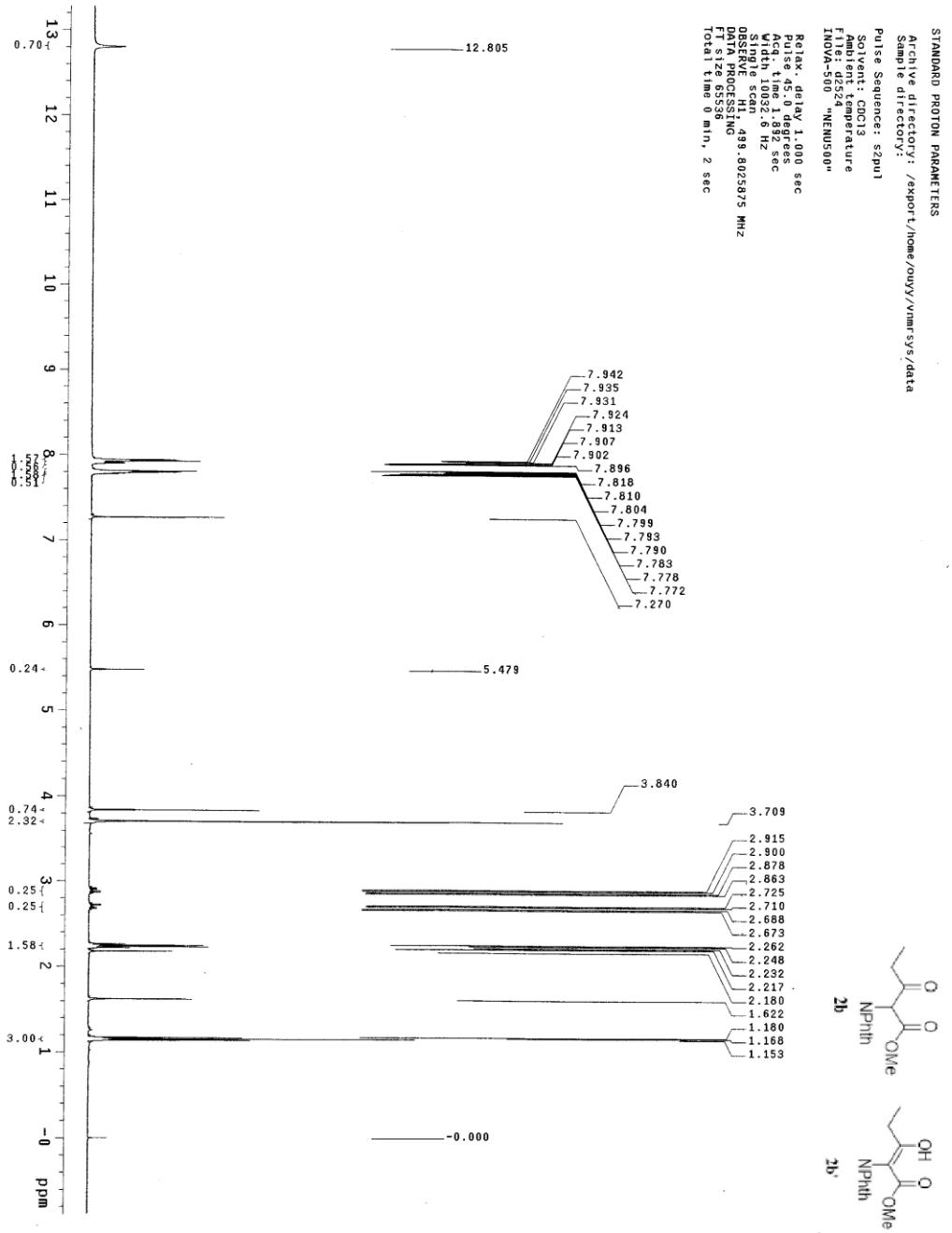
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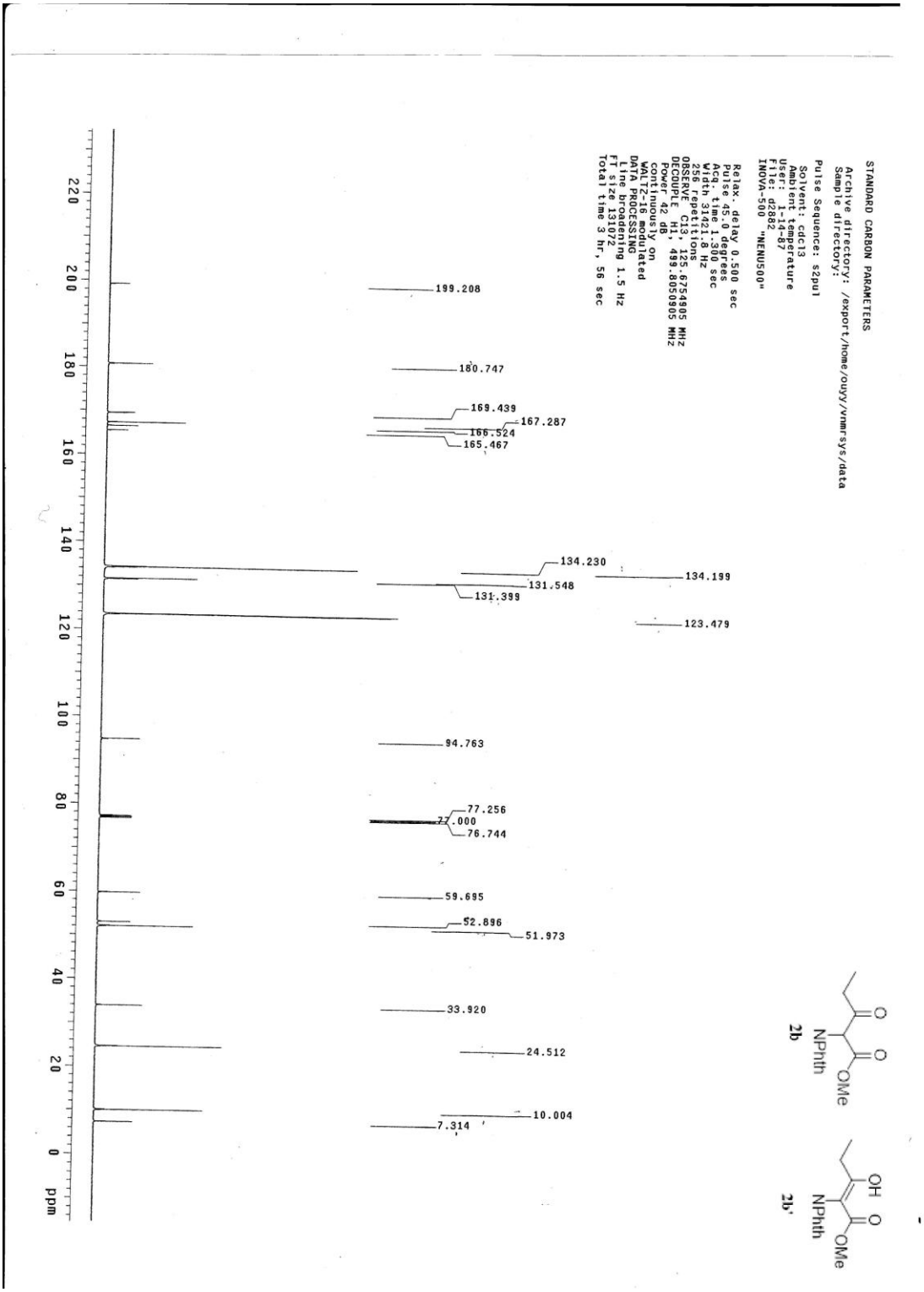
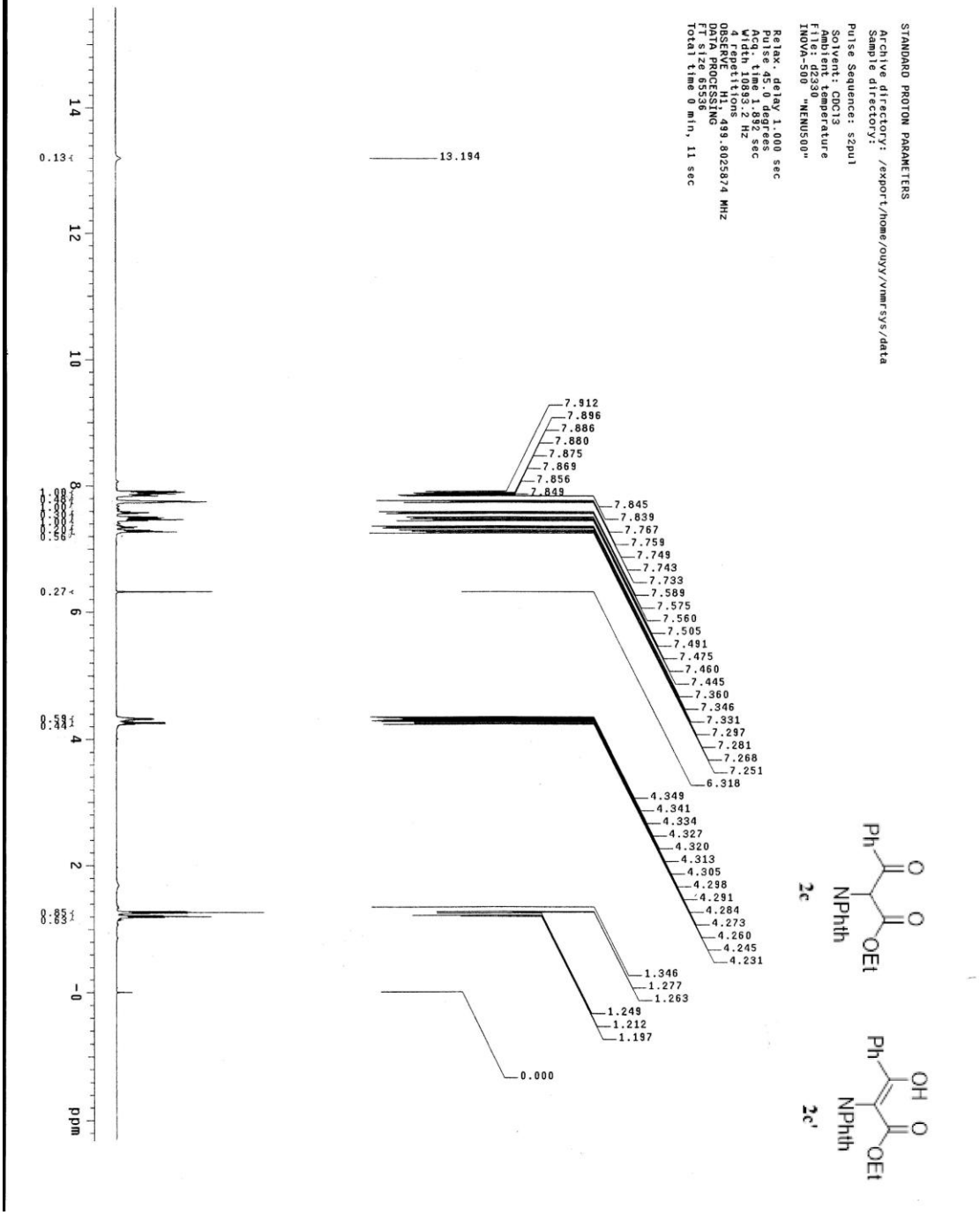
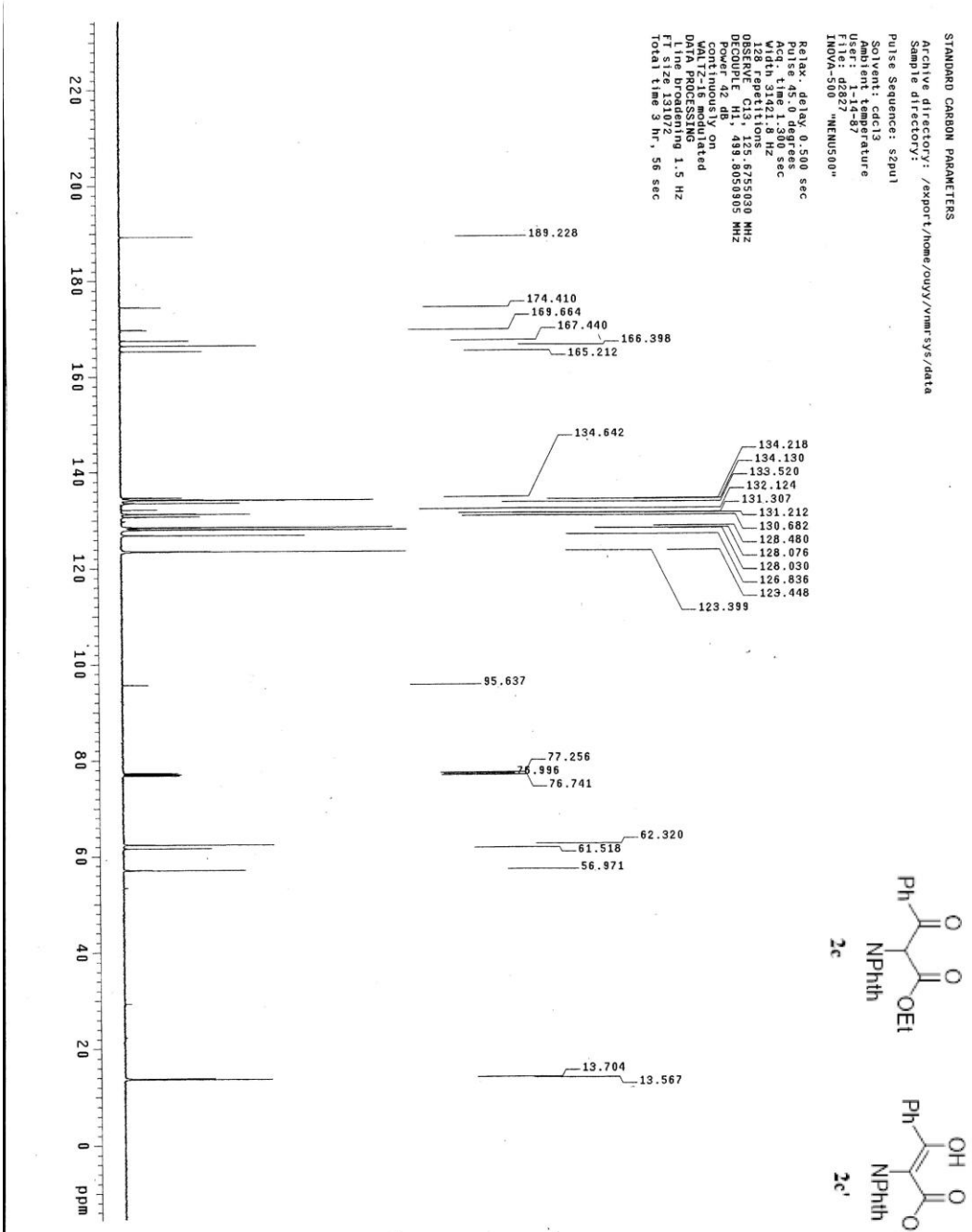
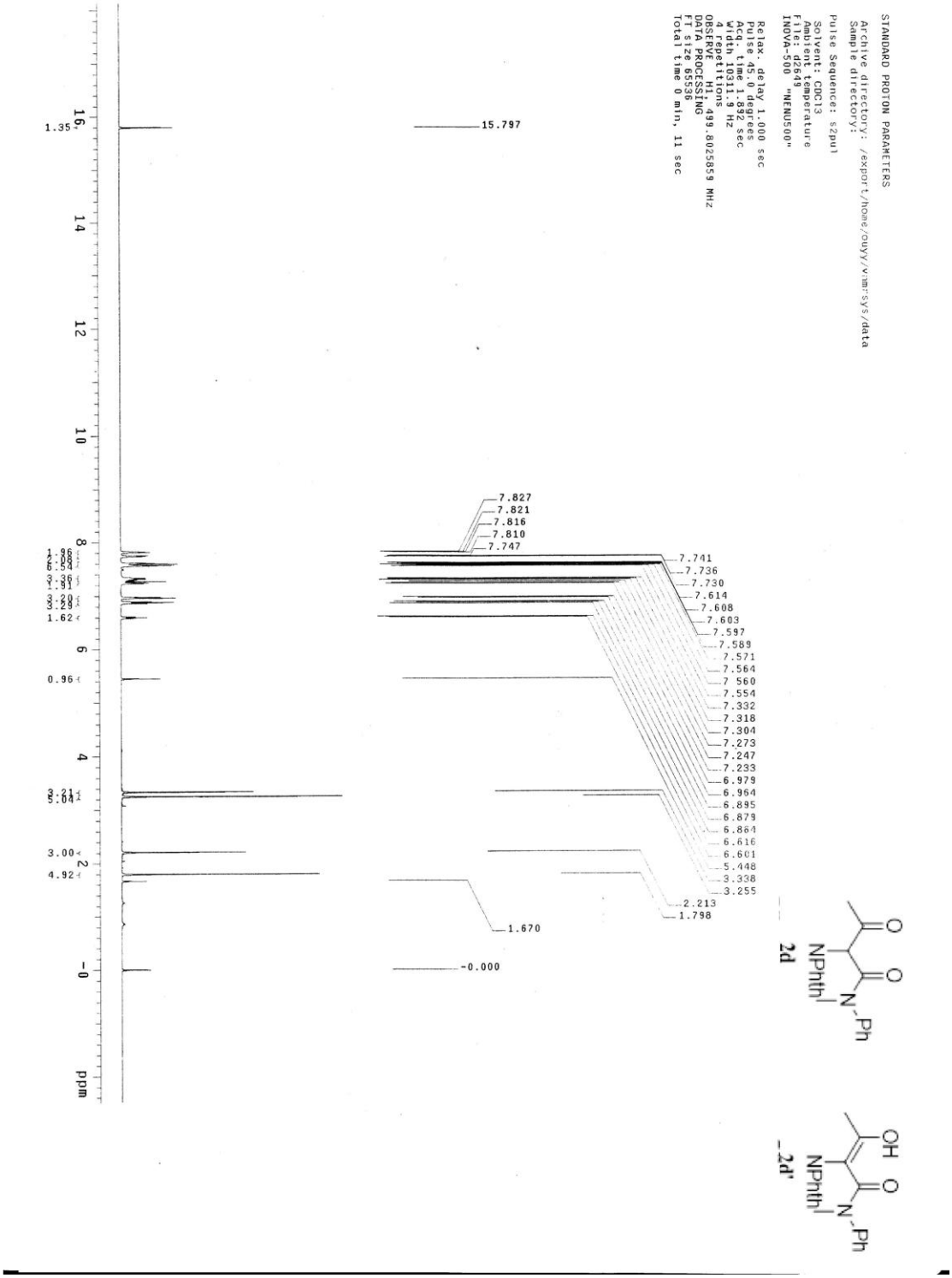
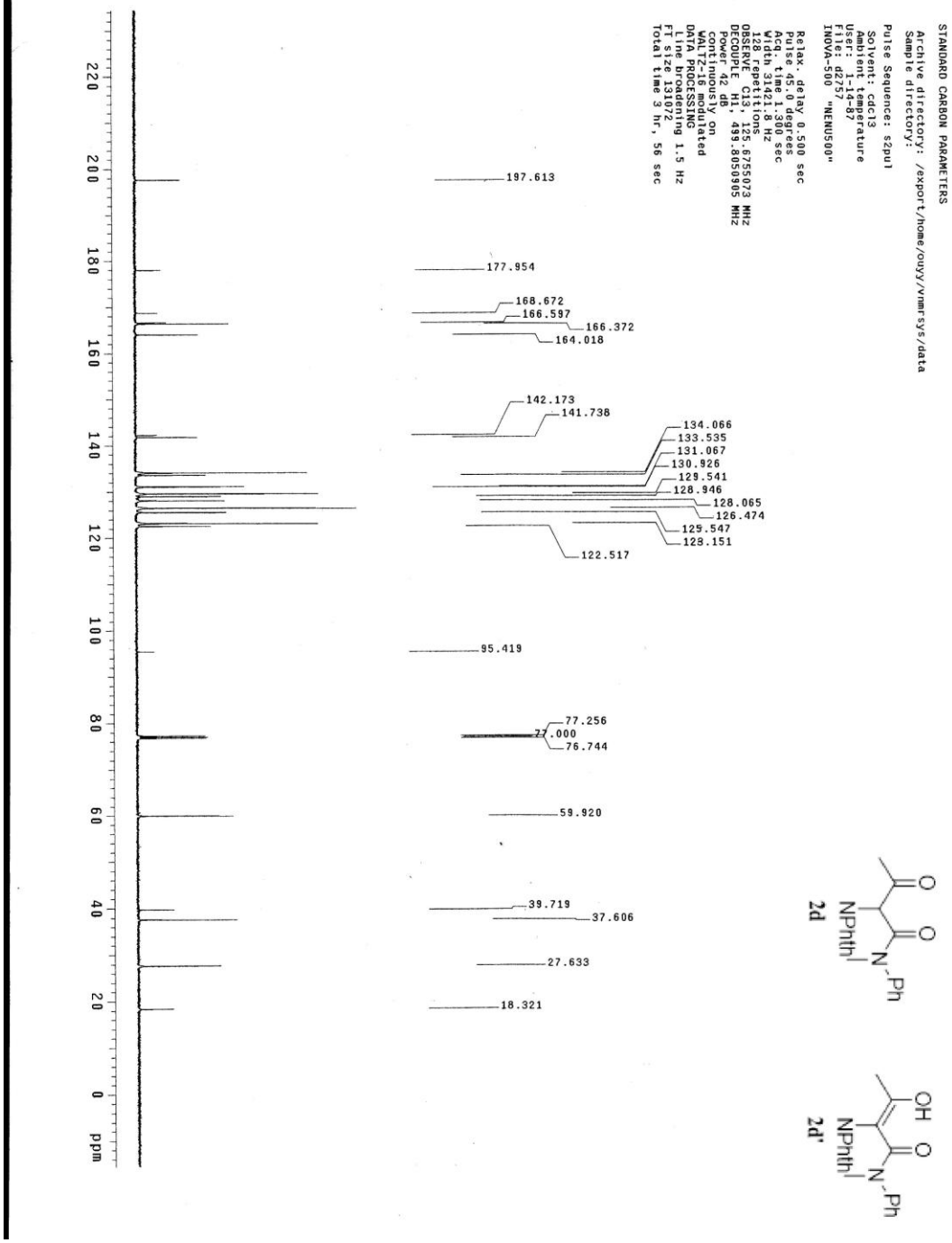
2.32
0.25
0.25
1.58
3.00
ppm
2b
2b'

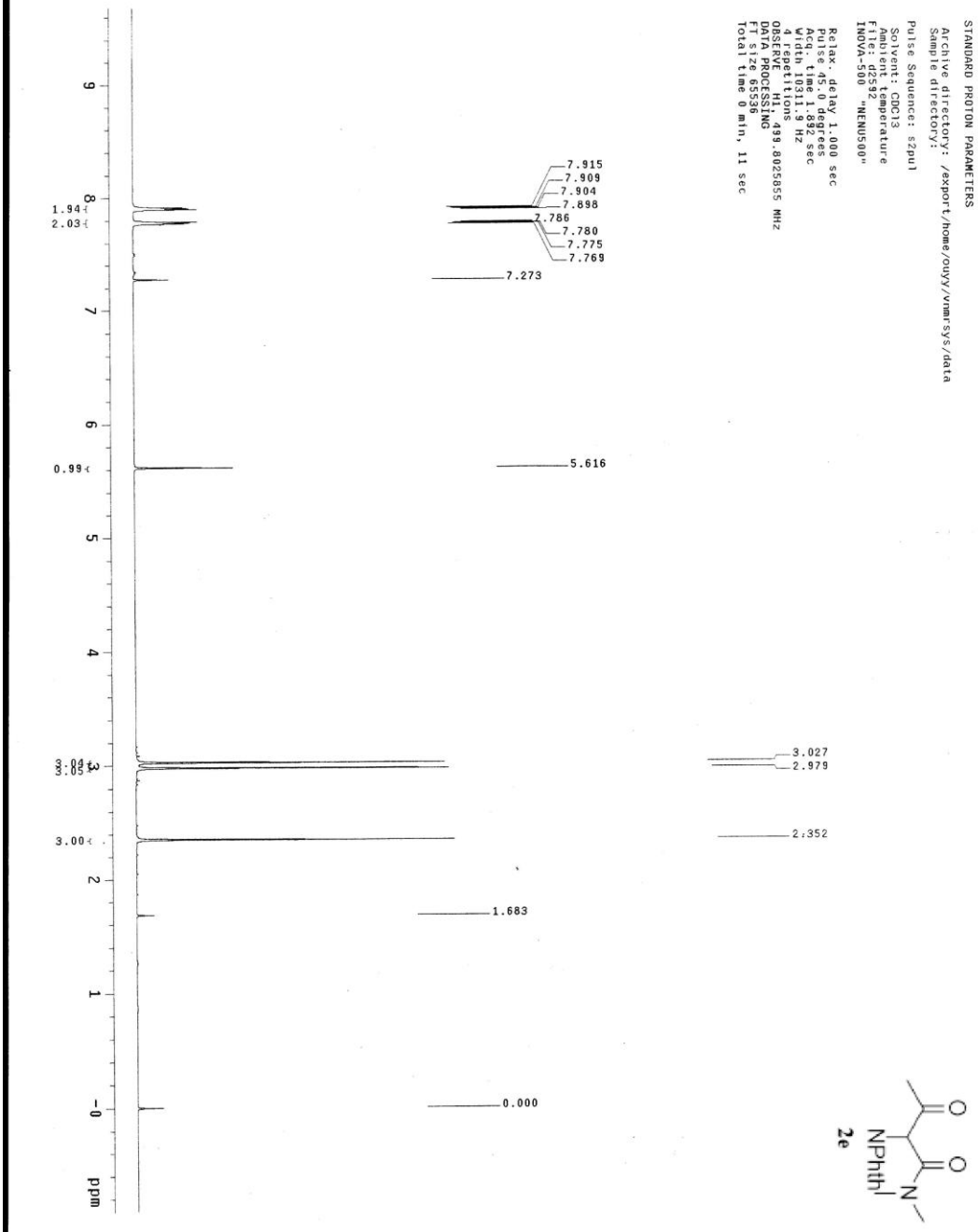

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Temp: 1.5-87
User: d2882
File: d2882
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754905 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
199.208
180.747
169.439
167.287
166.524
165.467
134.230
134.199
131.548
131.399
123.479
94.763
77.256
77.000
76.744
59.695
52.896
51.973
33.920
24.512
10.004
7.314
220
200
180
160
140
120
100
80
60
40
20
0
ppm
O
O
OMe
NPhth
2b
OH
O
OMe
NPhth
2b'

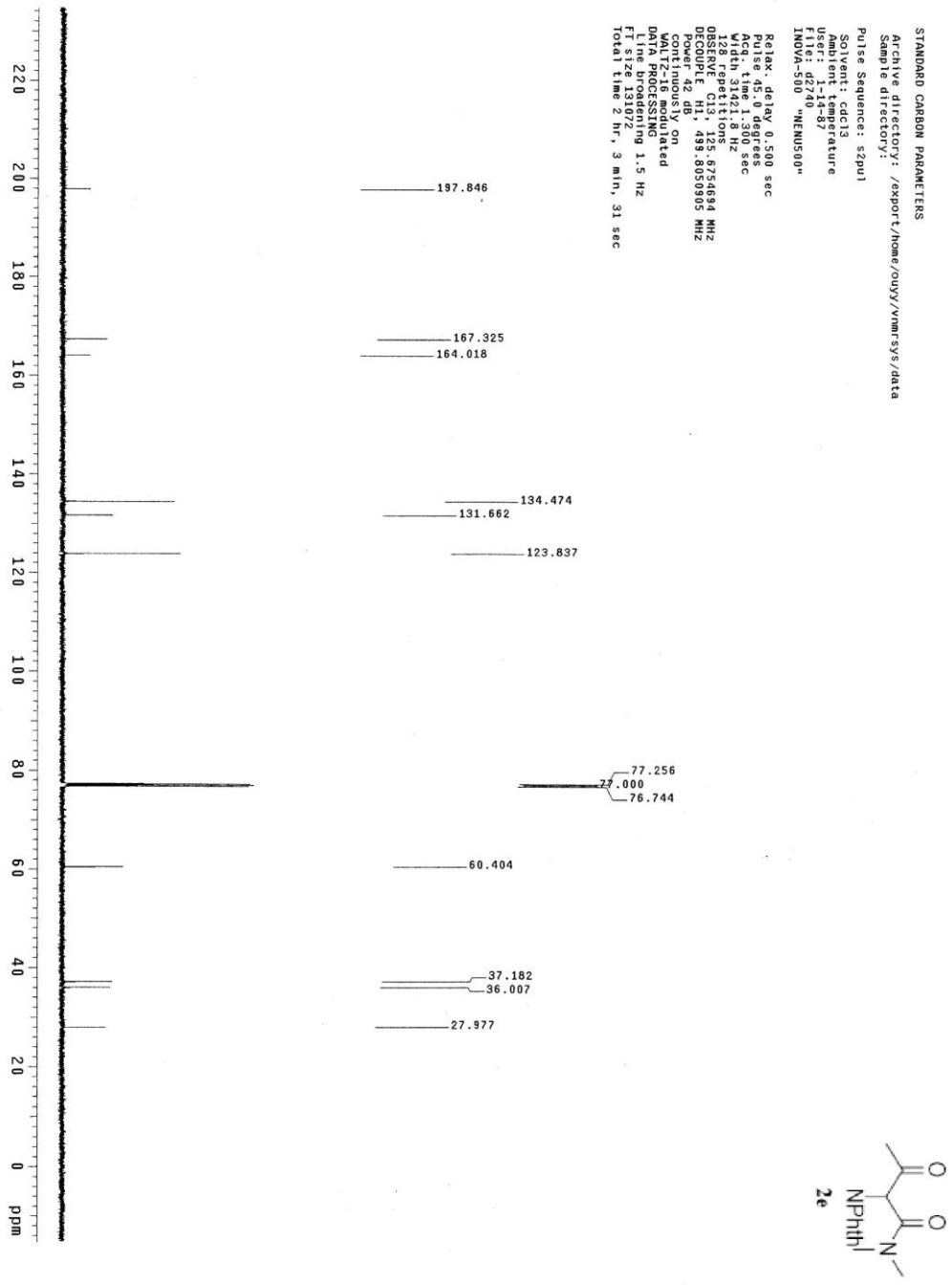

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2330
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 90 degrees
Acq. time 1.892 sec
Width 10893.2 Hz
4 repetitions
OBSERVE H1, 499.8025874 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
13.194
7.912
7.896
7.886
7.880
7.875
7.869
7.856
7.849
7.845
7.839
7.767
7.759
7.749
7.743
7.733
7.589
7.575
7.560
7.505
7.491
7.475
7.460
7.445
7.360
7.346
7.331
7.297
7.281
7.268
7.251
6.318
4.349
4.341
4.334
4.327
4.320
4.313
4.305
4.298
4.291
4.284
4.273
4.260
4.245
4.231
1.346
1.277
1.263
1.249
1.212
1.197
0.000
ppm
Ph
O
O
NPhth
OEt
2c
Ph
OH
O
NPhth
OEt
2c'

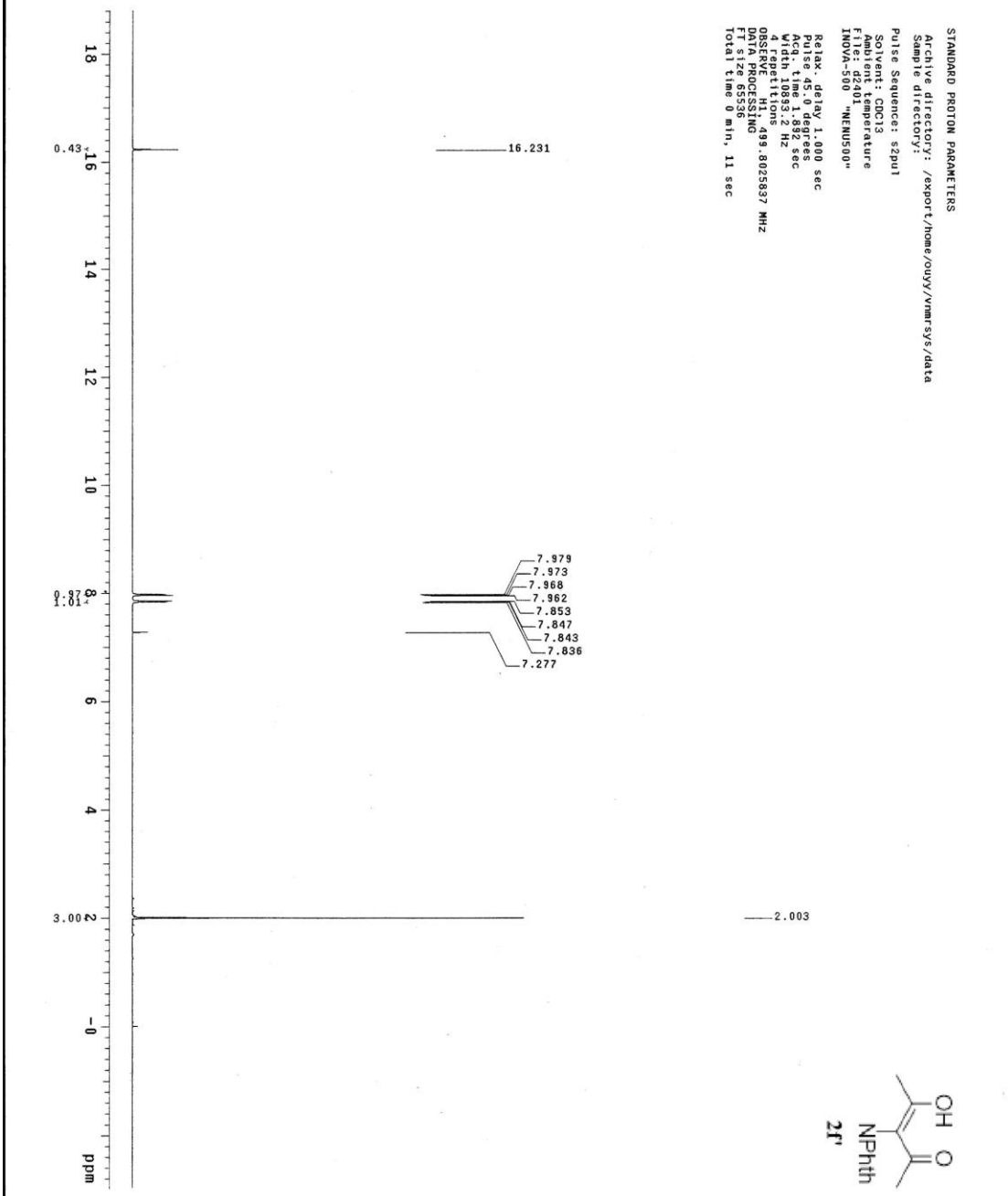

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2827 "MENU500"
INOVA-500 "MENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6755030 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
189.228
174.410
169.664
167.440
166.398
165.212
134.642
134.218
134.130
133.520
132.124
131.307
131.212
130.682
128.480
128.076
128.030
126.836
123.448
123.399
95.637
77.256
76.996
76.741
62.320
61.518
56.971
13.704
13.567
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Ph
O
O
OEt
NPhth
2c
Ph
OH
O
OEt
NPhth
2c'

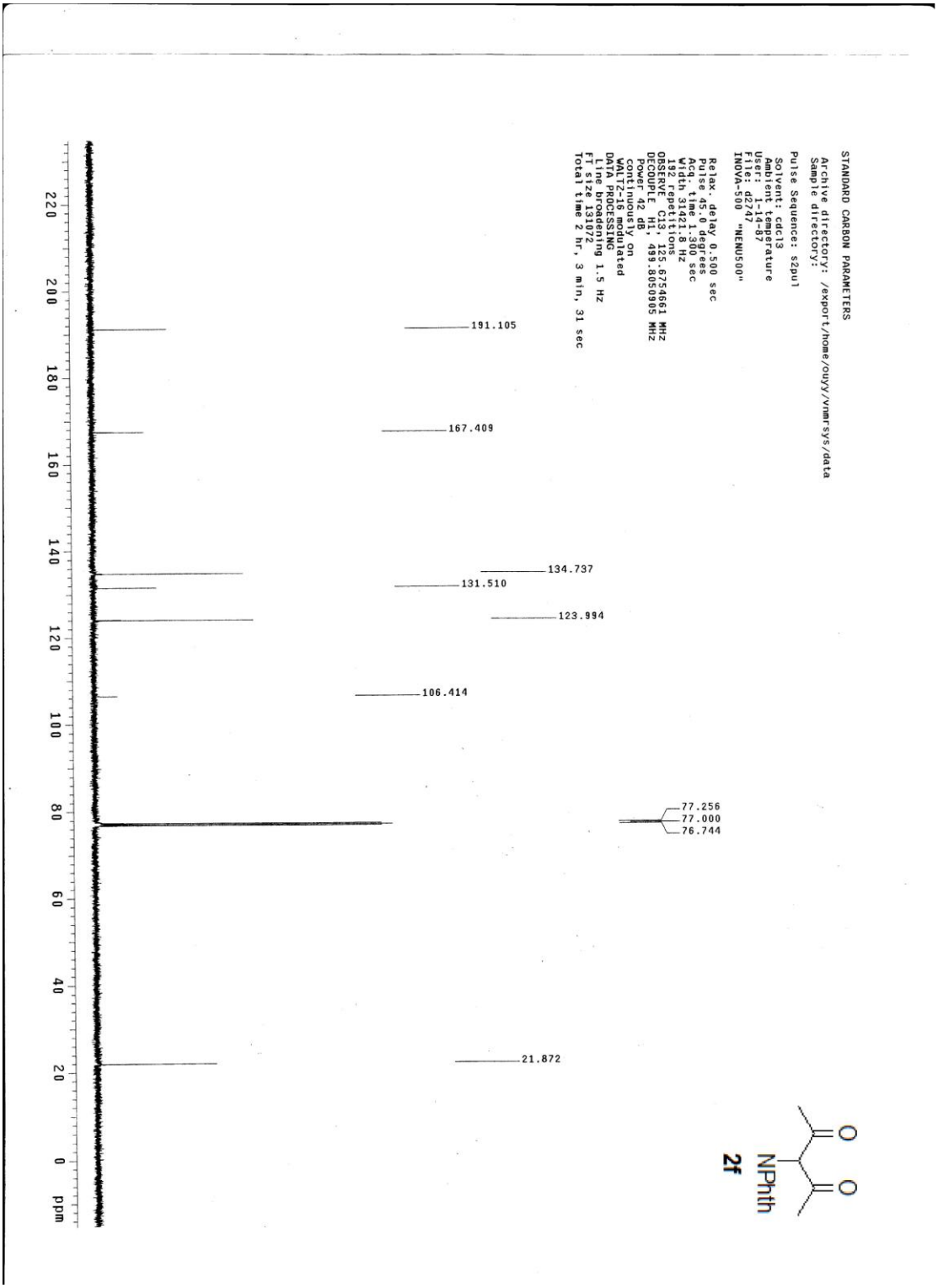

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2649
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1, 499.8025859 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
15.797
7.827
7.821
7.816
7.810
7.747
7.741
7.736
7.730
7.614
7.608
7.603
7.597
7.589
7.571
7.564
7 560
7.554
7.332
7.318
7.304
7.273
7.247
7.233
6.979
6.964
6.895
6.879
6.864
6.616
6.601
5.448
3.338
3.255
2.213
1.798
1.670
-0.000
1.35
1.96
1.91
3.20
3.29
1.62
0.96
3.21
5.04
3.00
4.92
ppm
2d
2d'
NPhth
N-Ph
OH

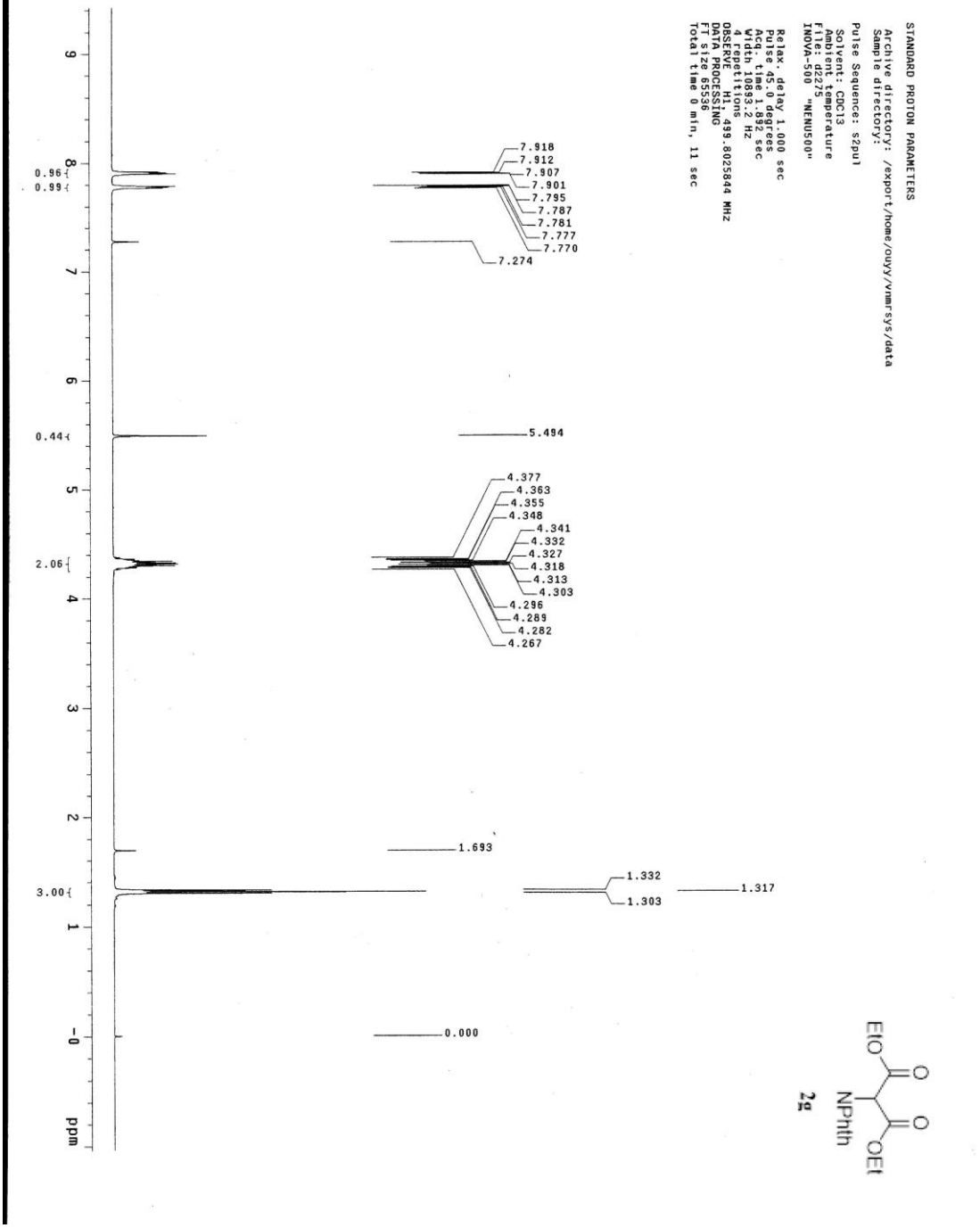

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy-14-87
File: d2757
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6755073 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 3 hr, 56 sec
197.613
177.954
168.672
166.597
166.372
164.018
142.173
141.738
134.066
133.535
131.067
130.926
129.541
128.946
128.065
126.474
125.547
128.151
122.517
95.419
77.256
77.000
76.744
59.920
39.719
37.606
27.633
18.321
220
200
180
160
140
120
100
80
60
40
20
0
ppm
O
O
N
Ph
NPhth
2d
OH
O
N
Ph
NPhth
2d'

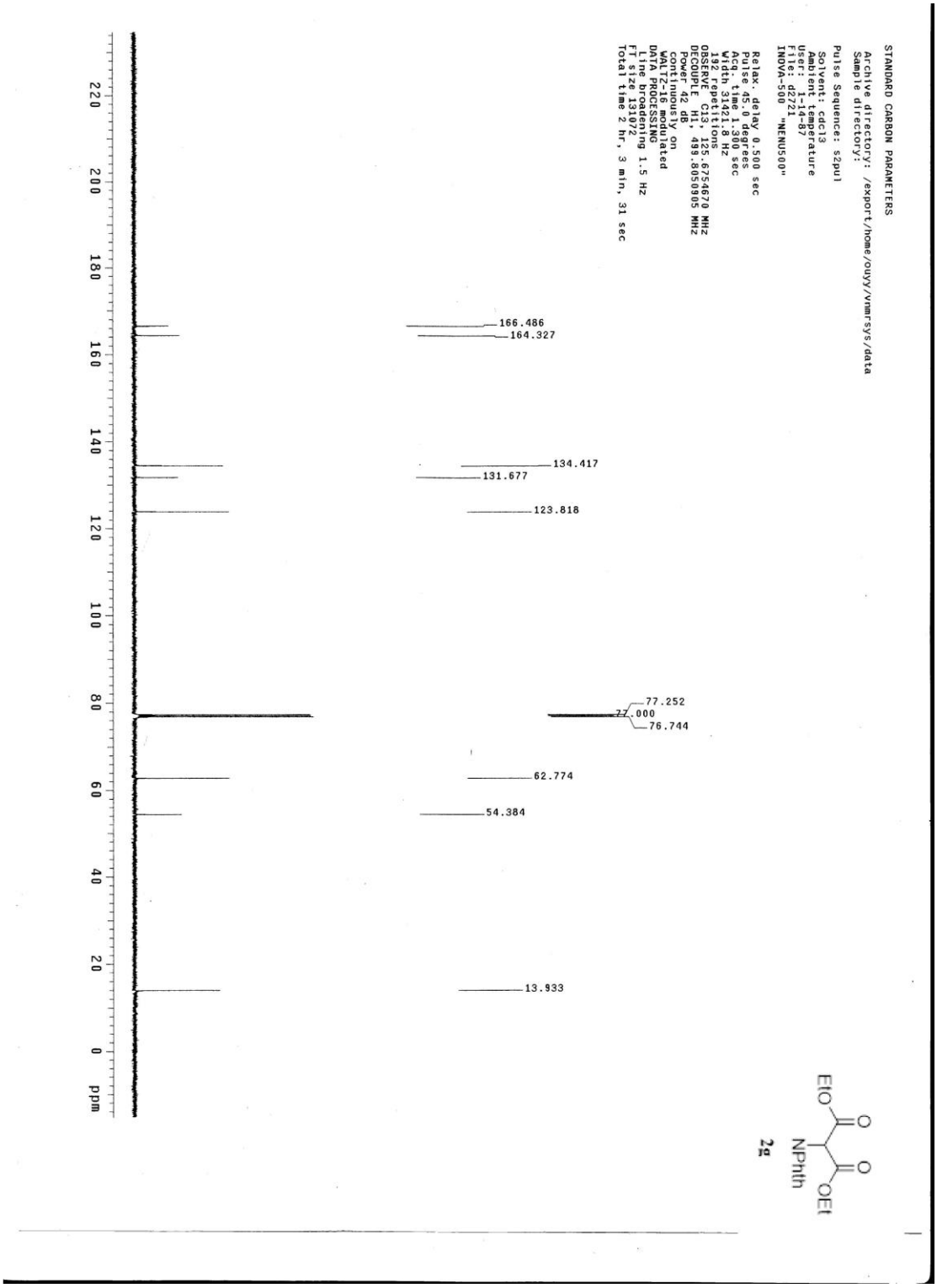

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2592.fid
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 10331.9 Hz
4 repetitions
OBSERVE H1, 499.8025855 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.915
7.909
7.904
7.898
7.786
7.780
7.775
7.769
7.273
5.616
3.027
2.979
2.352
1.683
0.000
1.94
2.03
0.99
3.04
3.05
3.00
ppm
NPhth
2e


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2740
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 32318.0 Hz
128 repetitions
OBSERVE C13, 125.6754694 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
197.846
167.325
164.018
134.474
131.662
123.837
77.256
77.000
76.744
60.404
37.182
36.007
27.977
220
200
180
160
140
120
100
80
60
40
20
0
ppm
O
O
N
NPhth
2e


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: s2401
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, 499.8025837 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
16.231
7.979
7.973
7.968
7.962
7.853
7.847
7.843
7.836
7.277
2.003
0.43
0.97
1.01
3.00
ppm
OH O
NPhth
2f'


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: i-14-87
File: d2747
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1 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
191.105
167.409
134.737
131.510
123.994
106.414
77.256
77.000
76.744
21.872
220
200
180
160
140
120
100
80
60
40
20
0
ppm
O
O
NPhth
2f


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2275
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10693.2 Hz
4 repetitions
OBSERVE H1, 499.8025844 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.918
7.912
7.907
7.901
7.795
7.787
7.781
7.777
7.770
7.274
5.494
4.377
4.363
4.355
4.348
4.341
4.332
4.327
4.318
4.313
4.303
4.296
4.289
4.282
4.267
1.693
1.332
1.303
1.317
0.000
0.96
0.99
0.44
2.06
3.00
ppm
EtO
O
O
OEt
NPhth
2g


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy-07
File: d2721-07
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 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
166.486
164.327
134.417
131.677
123.818
77.252
77.000
76.744
62.774
54.384
13.933
220
200
180
160
140
120
100
80
60
40
20
0
ppm
EtO
O
O
OEt
NPhth
2g

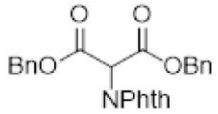
STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

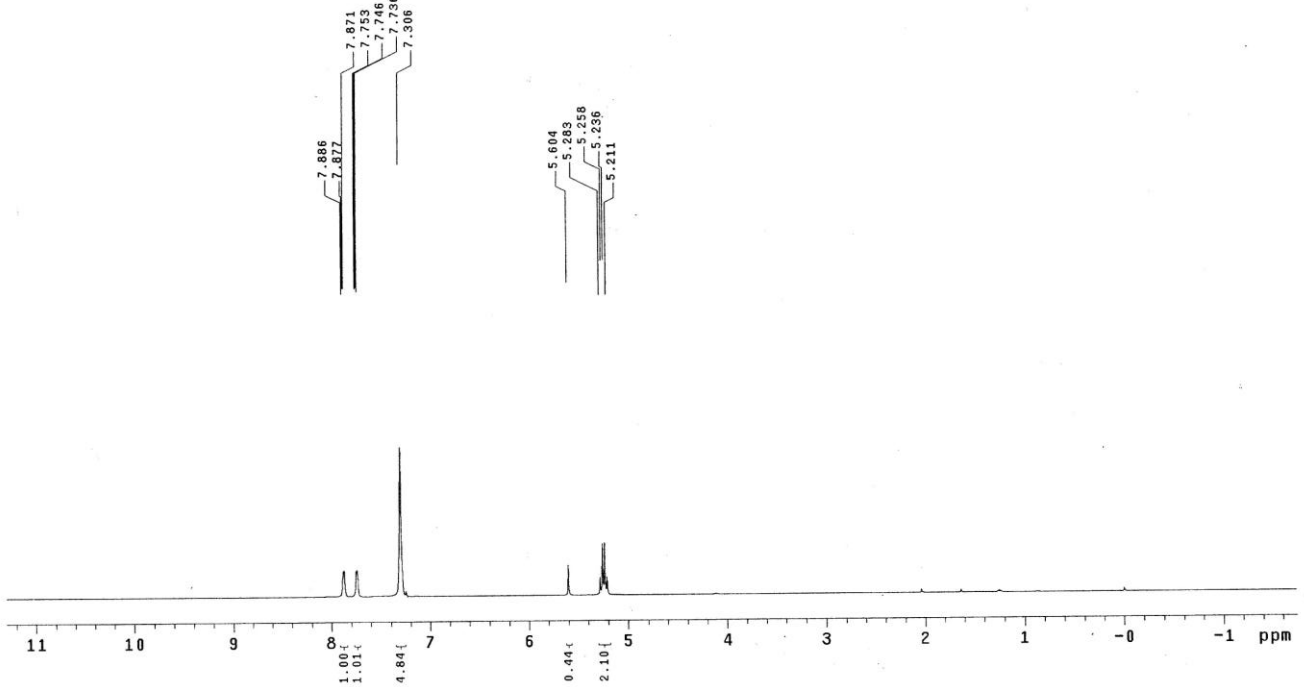
Pulse Sequence: s2pul

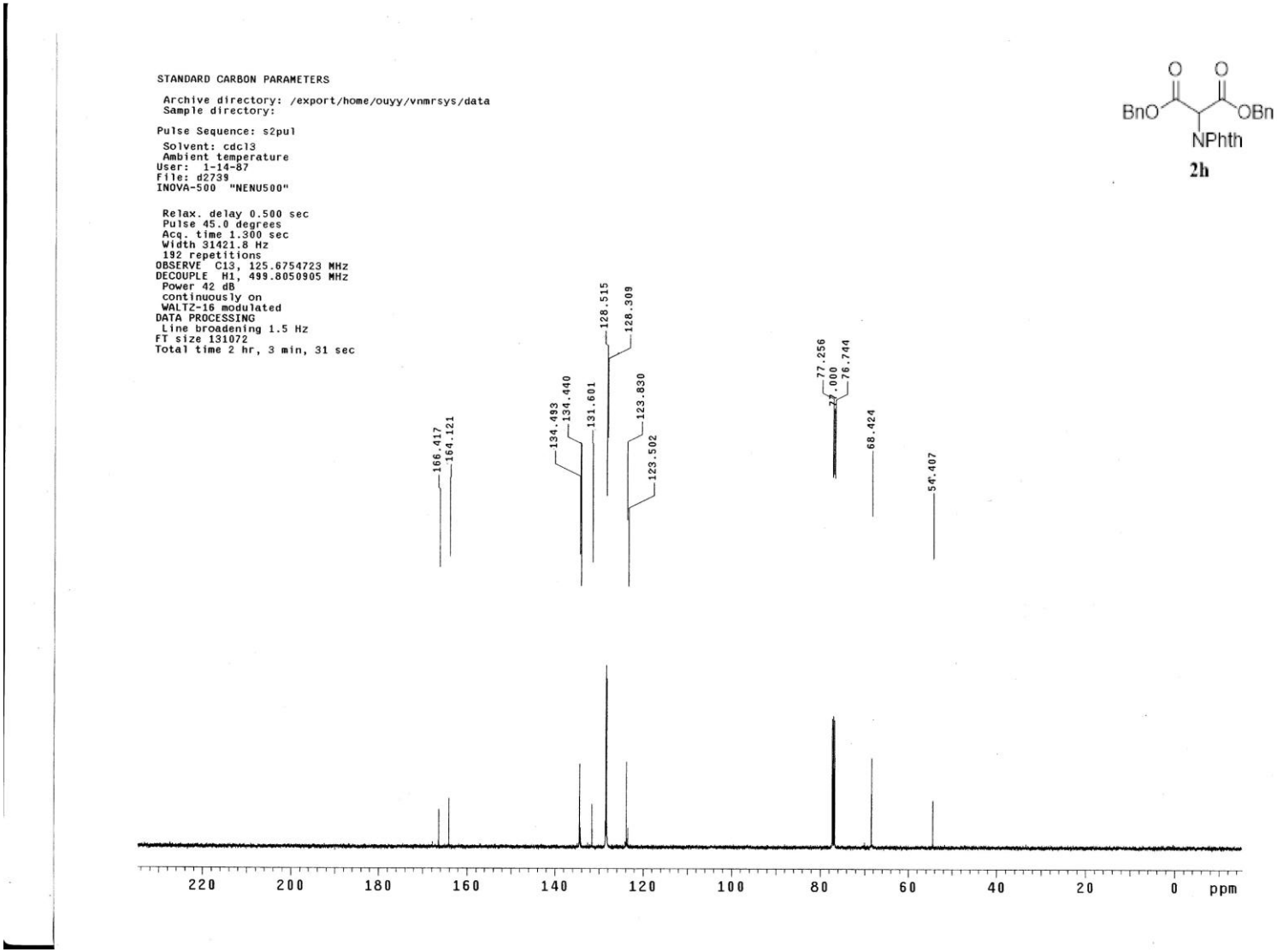
Solvent: CDCl3
Ambient temperature
File: d2890
INOVA-500 "NENU500"

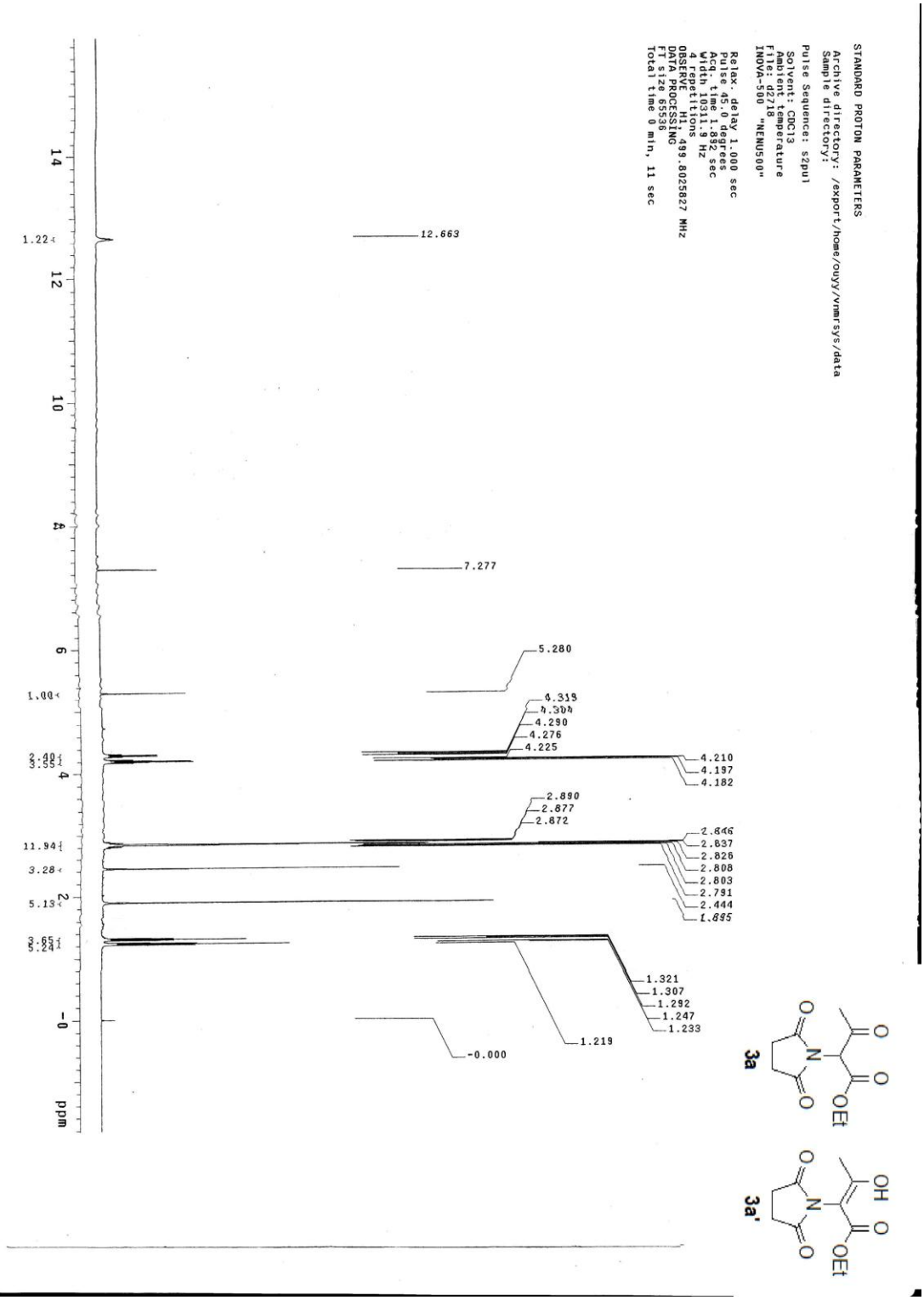
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9744.2 Hz
4 repetitions
OBSERVE H1, 499.8025984 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec

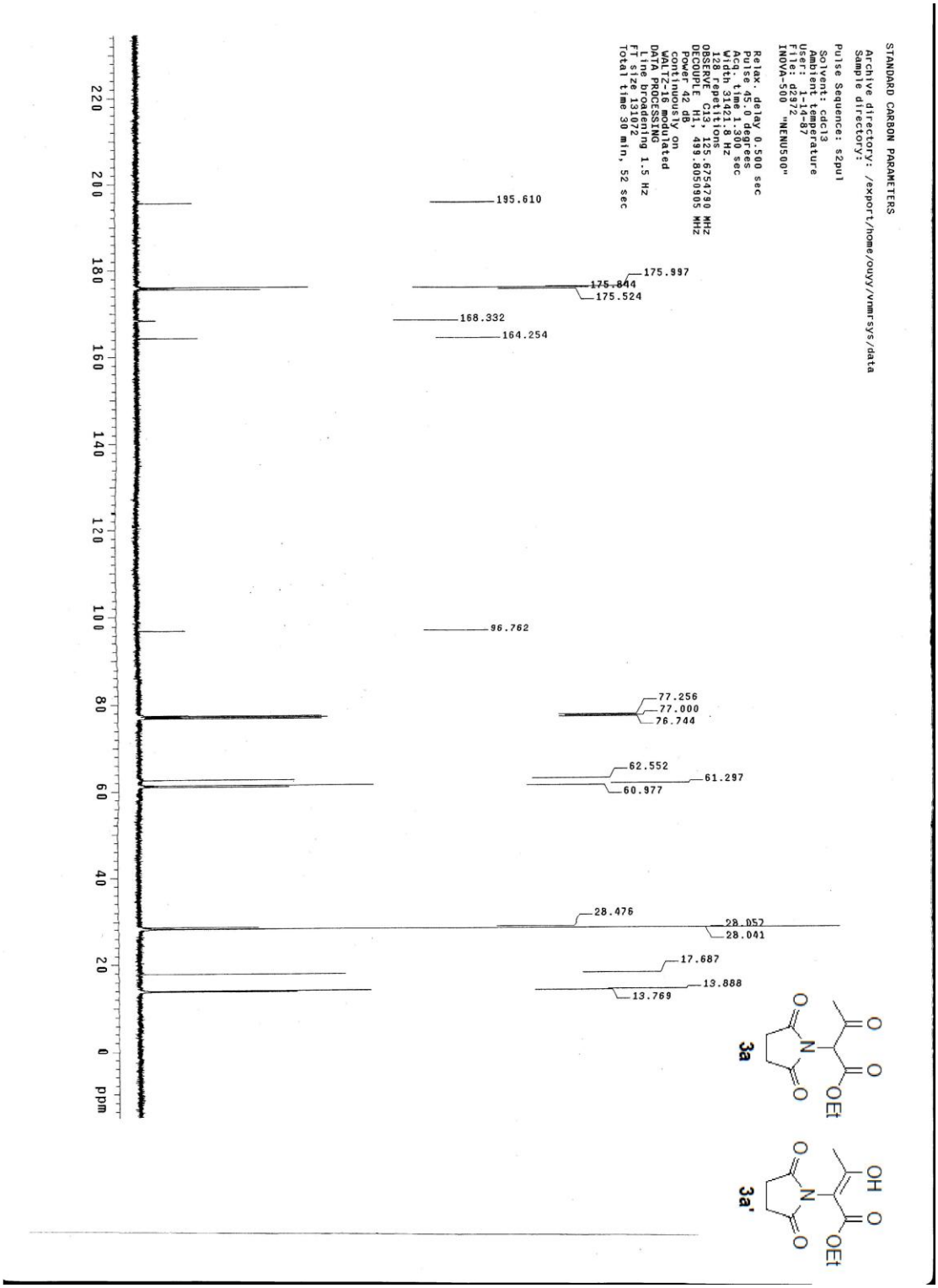


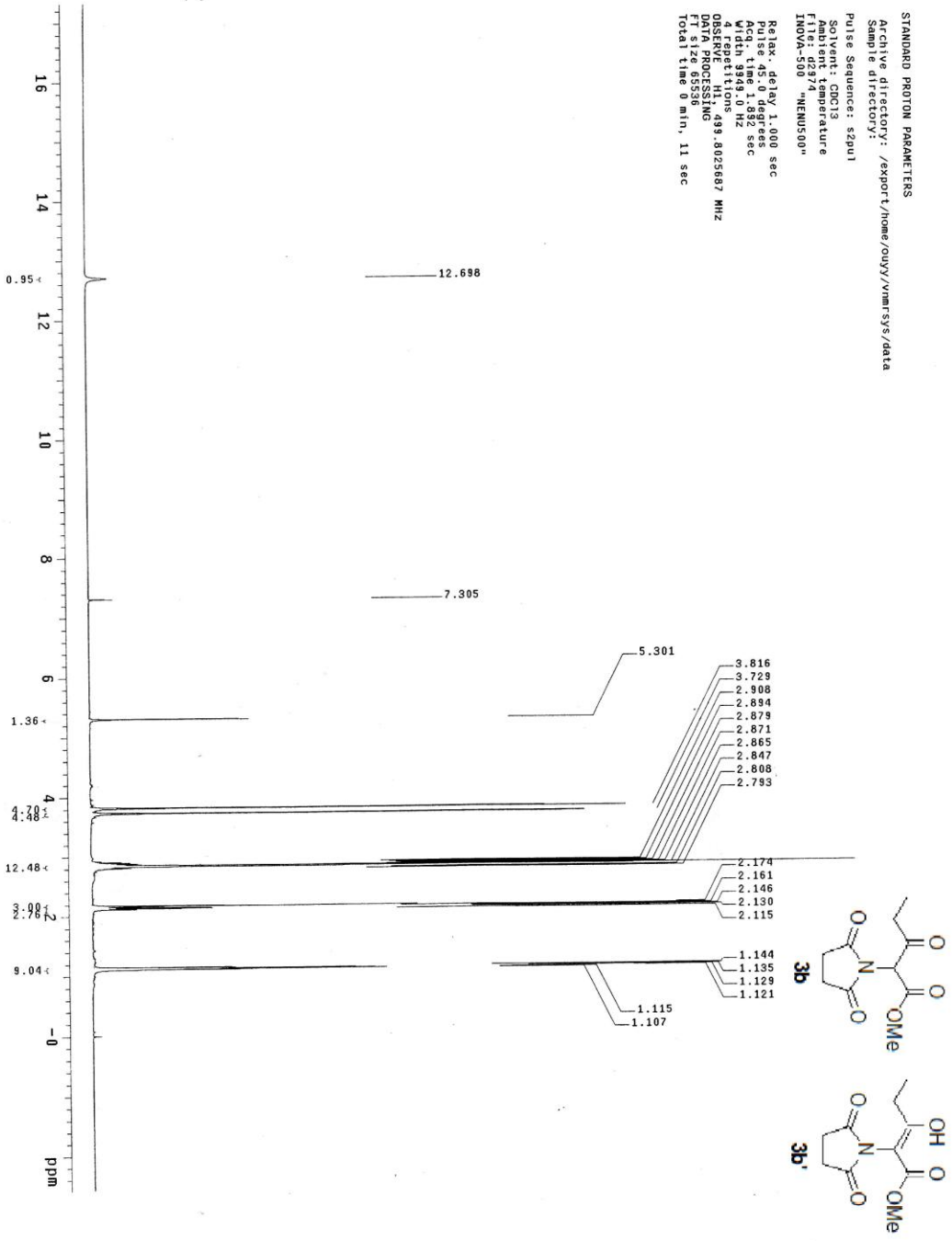
2h

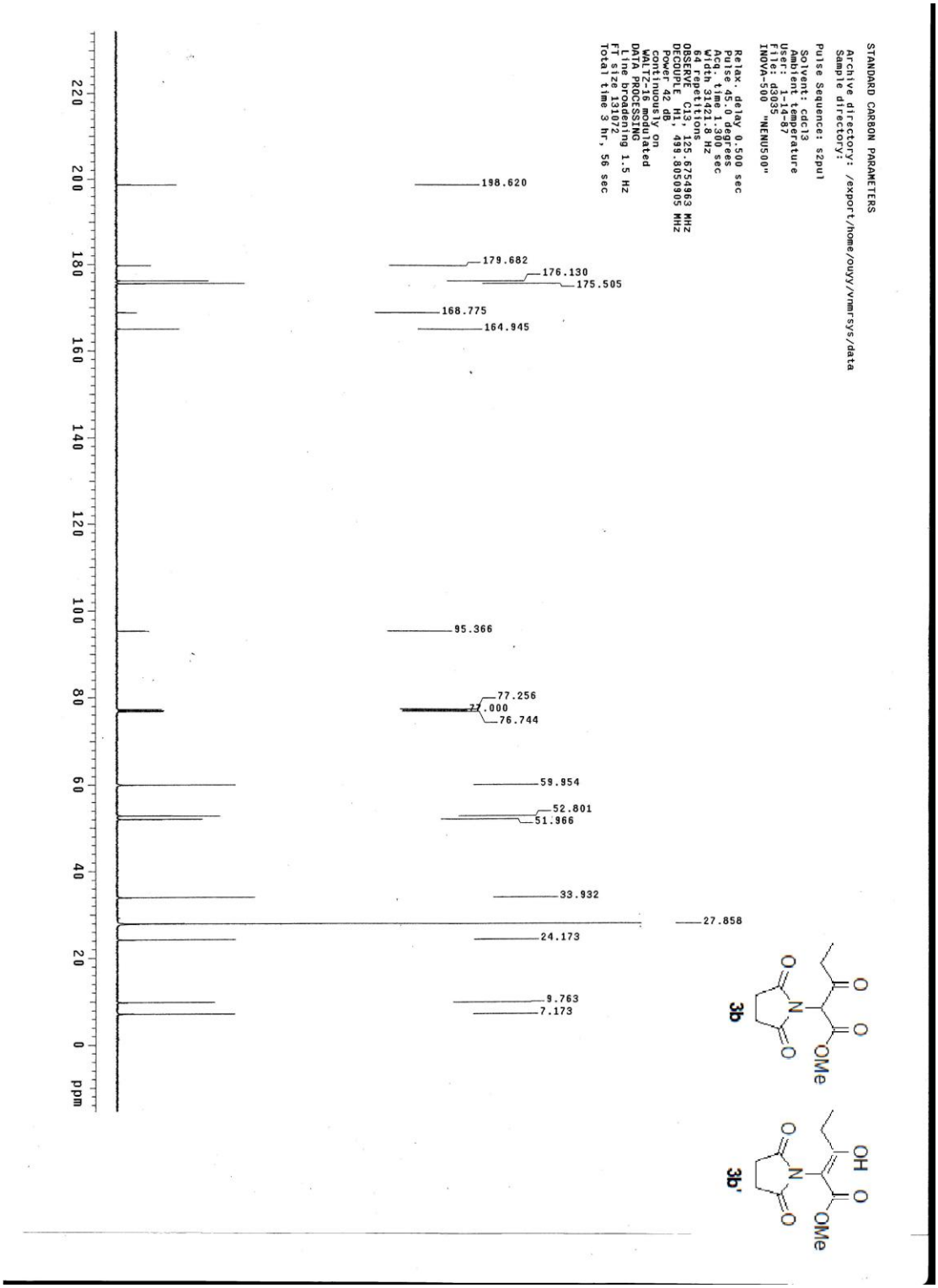


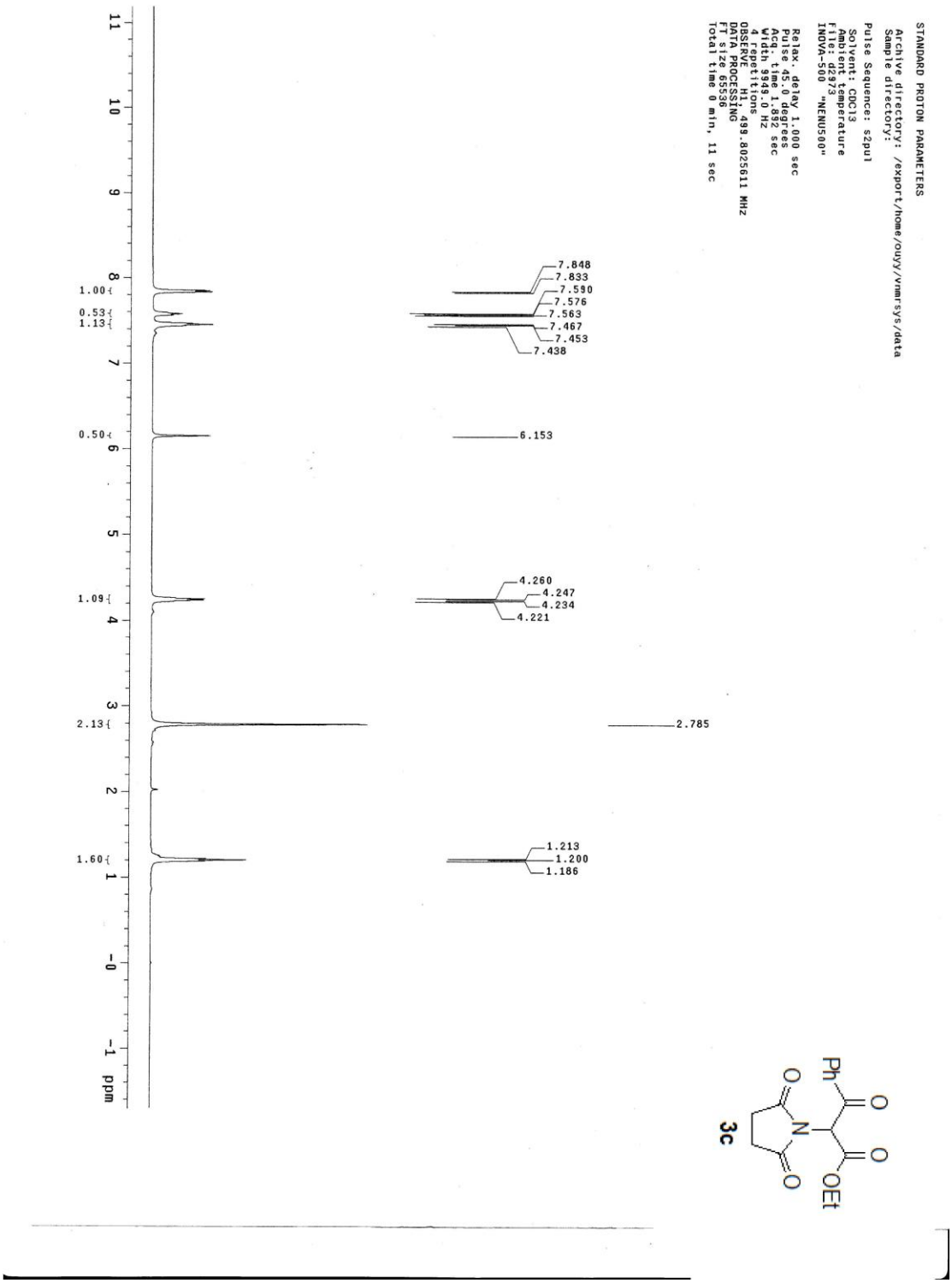

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2739
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE 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 2 hr, 3 min, 31 sec
BnO
OBn
NPhth
2h
166.417
164.121
134.493
134.440
131.601
128.515
128.309
123.830
123.502
77.256
77.000
76.744
68.424
54.407
220
200
180
160
140
120
100
80
60
40
20
0
ppm

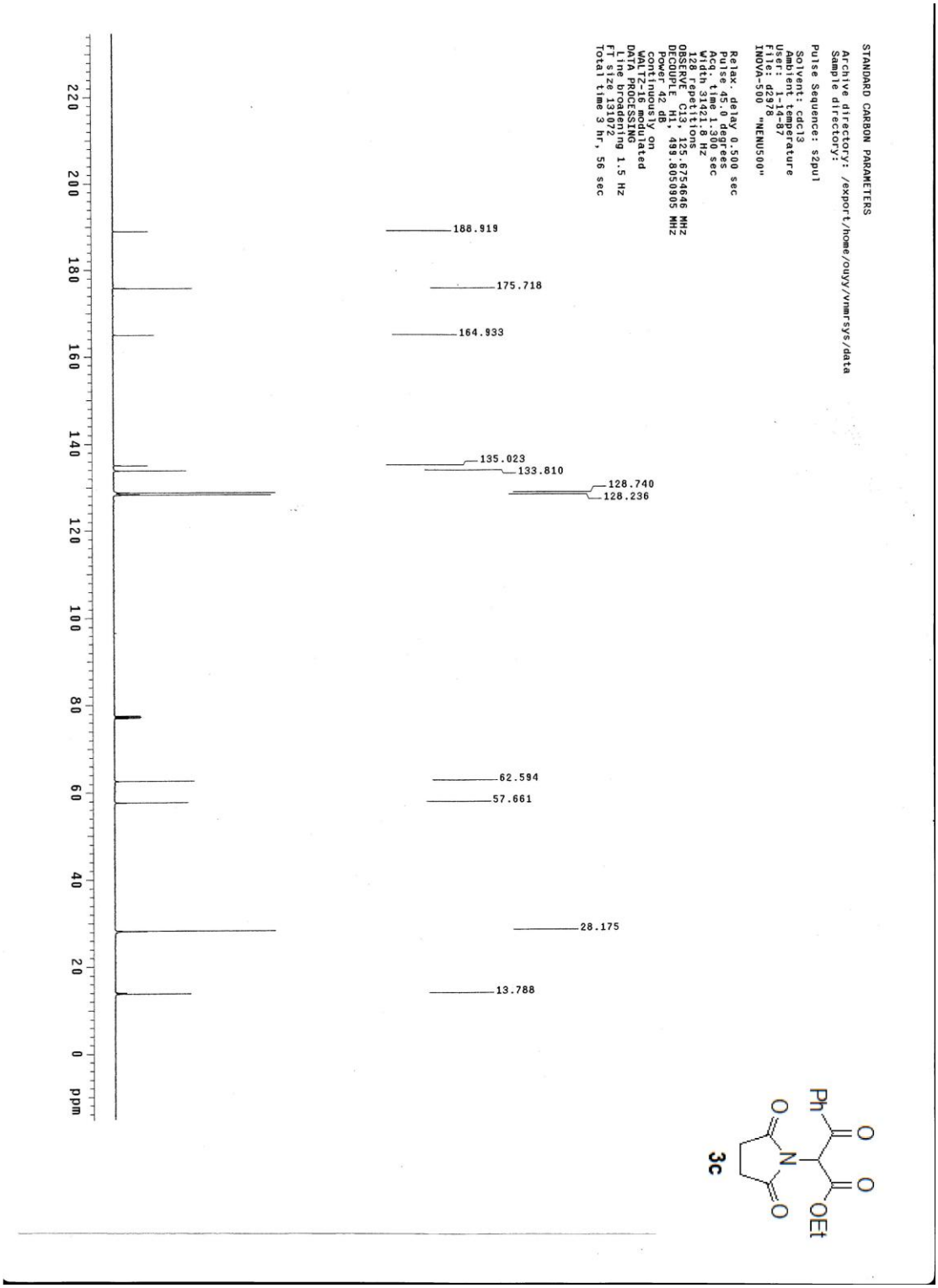

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2718
INOVA-500 "NEMU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
4 repetitions
OBSERVE H1
499.8025827 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
12.663
7.277
5.280
4.318
4.304
4.290
4.276
4.225
4.210
4.197
4.182
2.890
2.877
2.872
2.846
2.837
2.826
2.808
2.803
2.791
2.444
1.895
1.321
1.307
1.292
1.247
1.233
1.219
-0.000
ppm
3a
3a'
OEt
OH

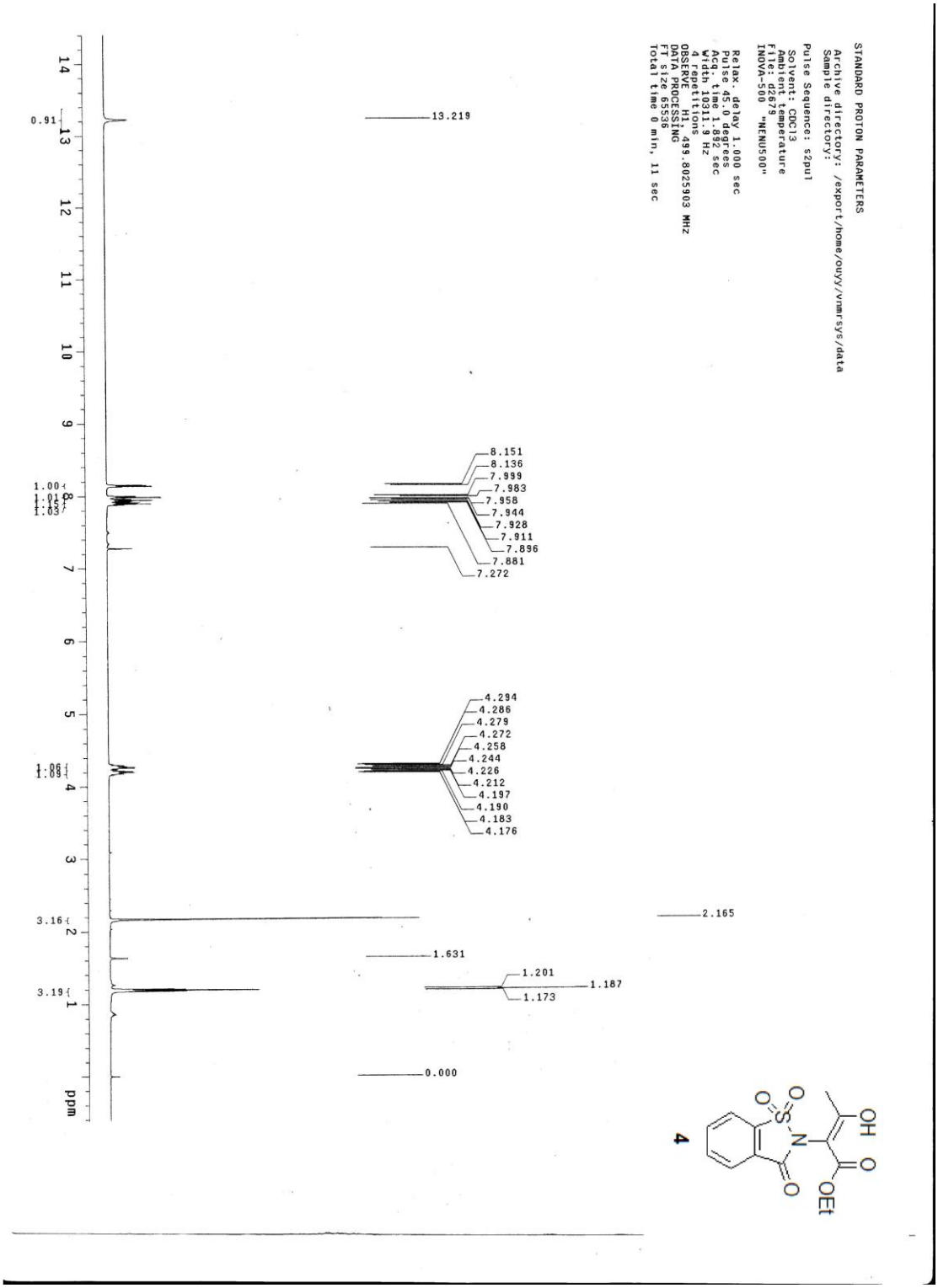

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: liuyan-87
File: d2972
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754790 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
195.610
175.997
175.844
175.524
168.332
164.254
96.762
77.256
77.000
76.744
62.552
61.297
60.977
28.476
28.052
28.041
17.687
13.888
13.769
3a
3a'
O
N
OEt
OH
220
200
180
160
140
120
100
80
60
40
20
0
ppm

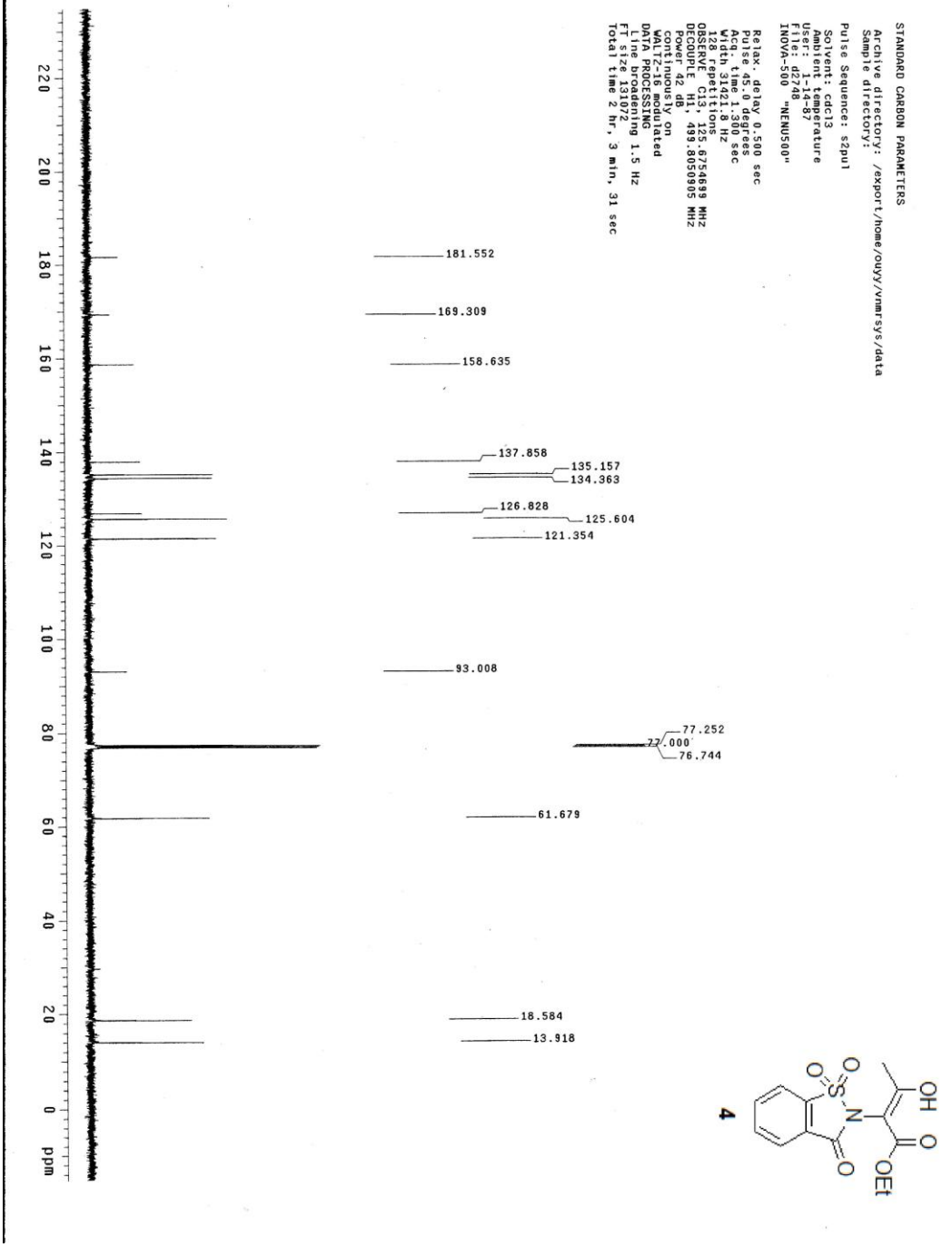

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2374
INOVA-500 "NEMUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9949.0 Hz
4 repetitions
OBSERVE H1, 499.8025687 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
12.698
7.305
5.301
3.816
3.729
2.908
2.894
2.879
2.871
2.865
2.847
2.808
2.793
2.174
2.161
2.146
2.130
2.115
1.144
1.135
1.129
1.121
1.115
1.107
0.95
1.36
4.70
4.48
12.48
3.00
2.76
9.04
ppm
3b
3b'
OMe
OH

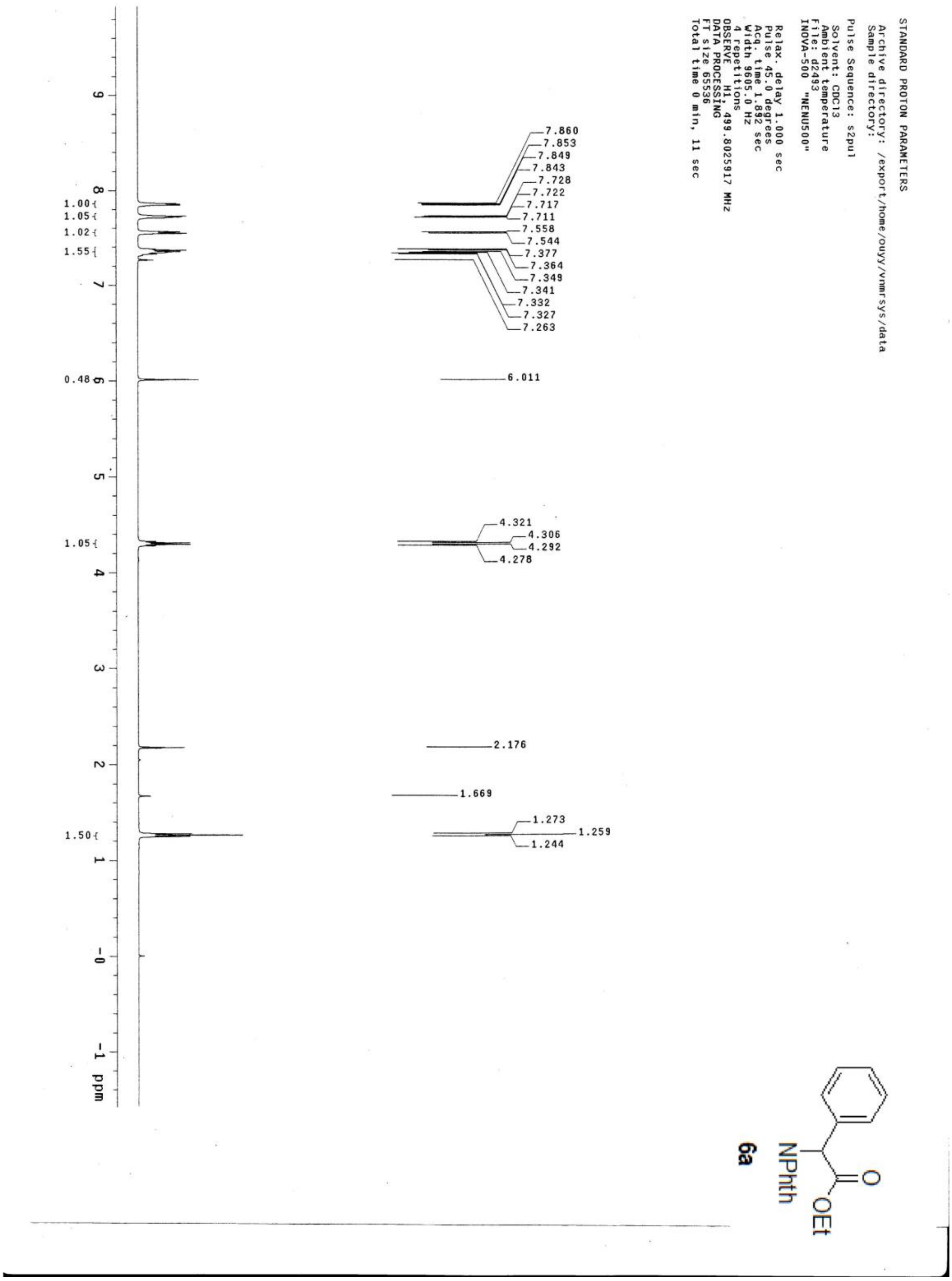

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d3035
INOVA-500 "MENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754963 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
198.620
179.682
176.130
175.505
168.775
164.945
95.366
77.256
77.000
76.744
59.954
52.801
51.966
33.932
27.858
24.173
9.763
7.173
220
200
180
160
140
120
100
80
60
40
20
0
ppm
3b
3b'
OMe
OH


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2973
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
width 9949.0 Hz
4 repetitions
OBSERVE H1, 499.8025611 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.848
7.833
7.590
7.576
7.563
7.467
7.453
7.438
6.153
4.260
4.247
4.234
4.221
2.785
1.213
1.200
1.186
1.00
0.53
1.13
0.50
1.09
2.13
1.60
ppm
Ph
O
OEt
N
3c

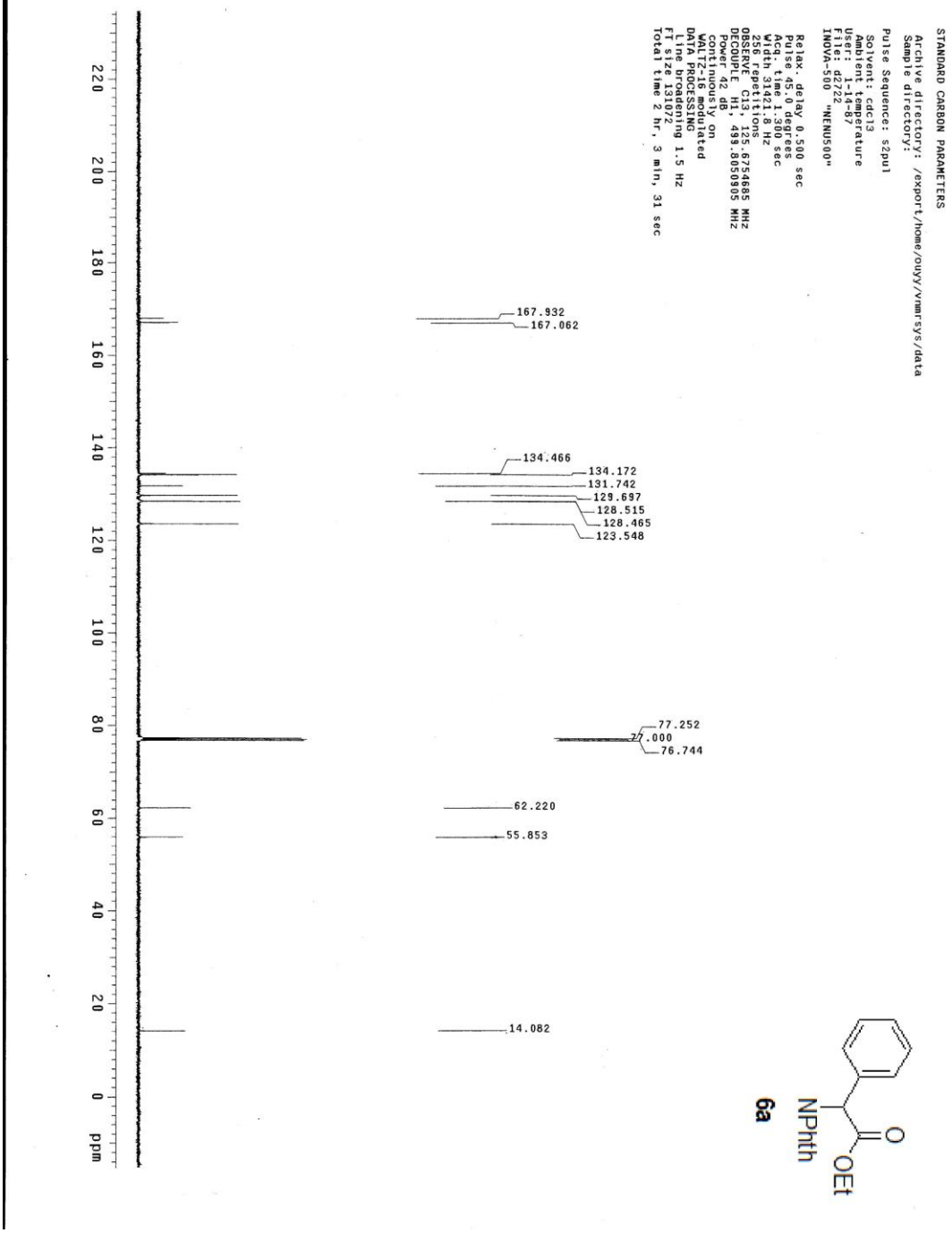

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: ouyy-07
File: d2978
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754646 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
188.919
175.718
164.933
135.023
133.810
128.740
128.236
62.594
57.661
28.175
13.788
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Ph
O
O
OEt
N
O
O
3c

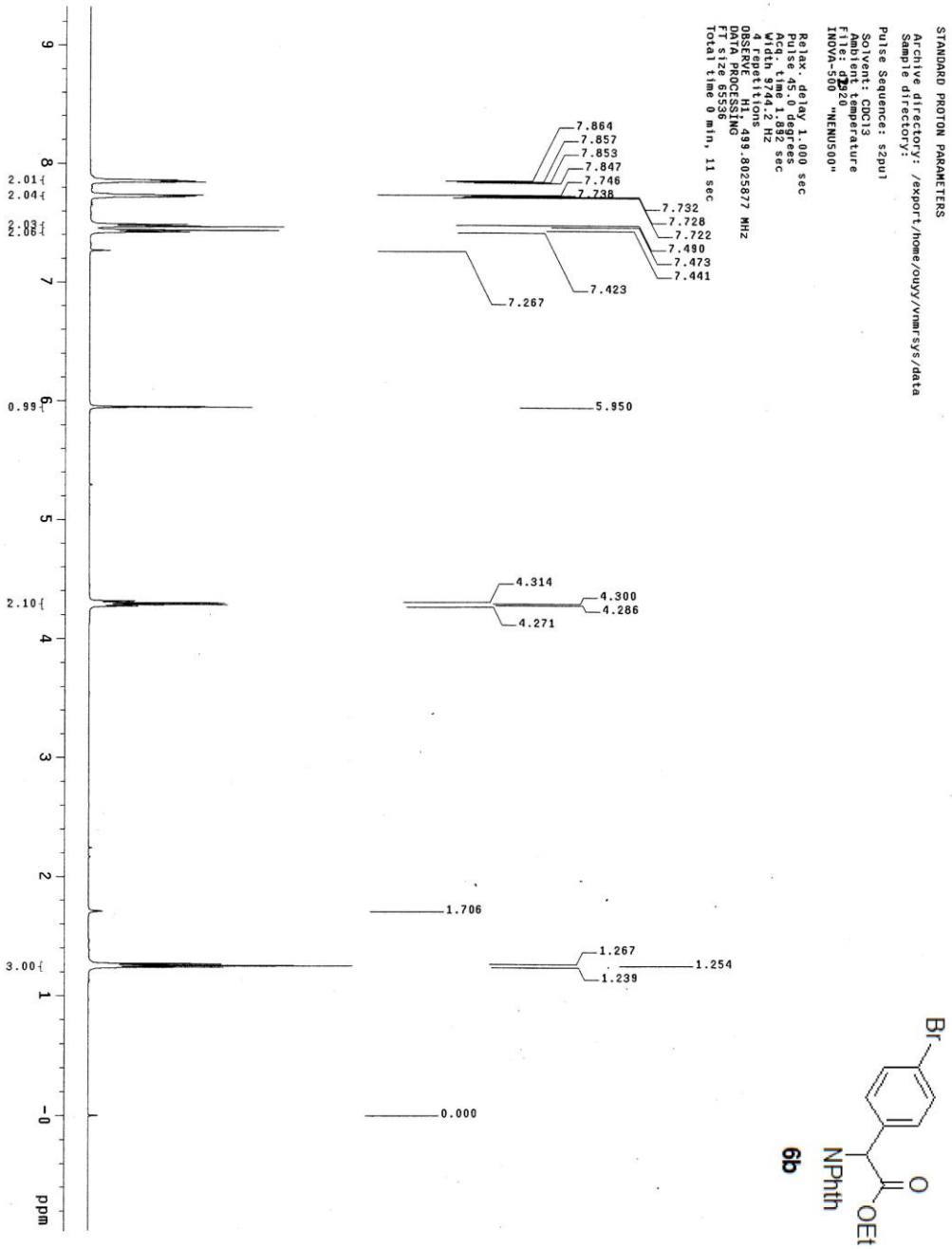

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2679
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10311.9 Hz
1 repetitions
OBSERVE H1, 499.8025903 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
13.219
8.151
8.136
7.999
7.983
7.958
7.944
7.928
7.911
7.896
7.881
7.272
4.294
4.286
4.279
4.272
4.258
4.244
4.226
4.212
4.197
4.190
4.183
4.176
2.165
1.631
1.201
1.187
1.173
0.000
0.91
1.00
1.01
1.15
1.03
1.06
1.08
3.16
3.19
ppm
OH
O
OEt
4

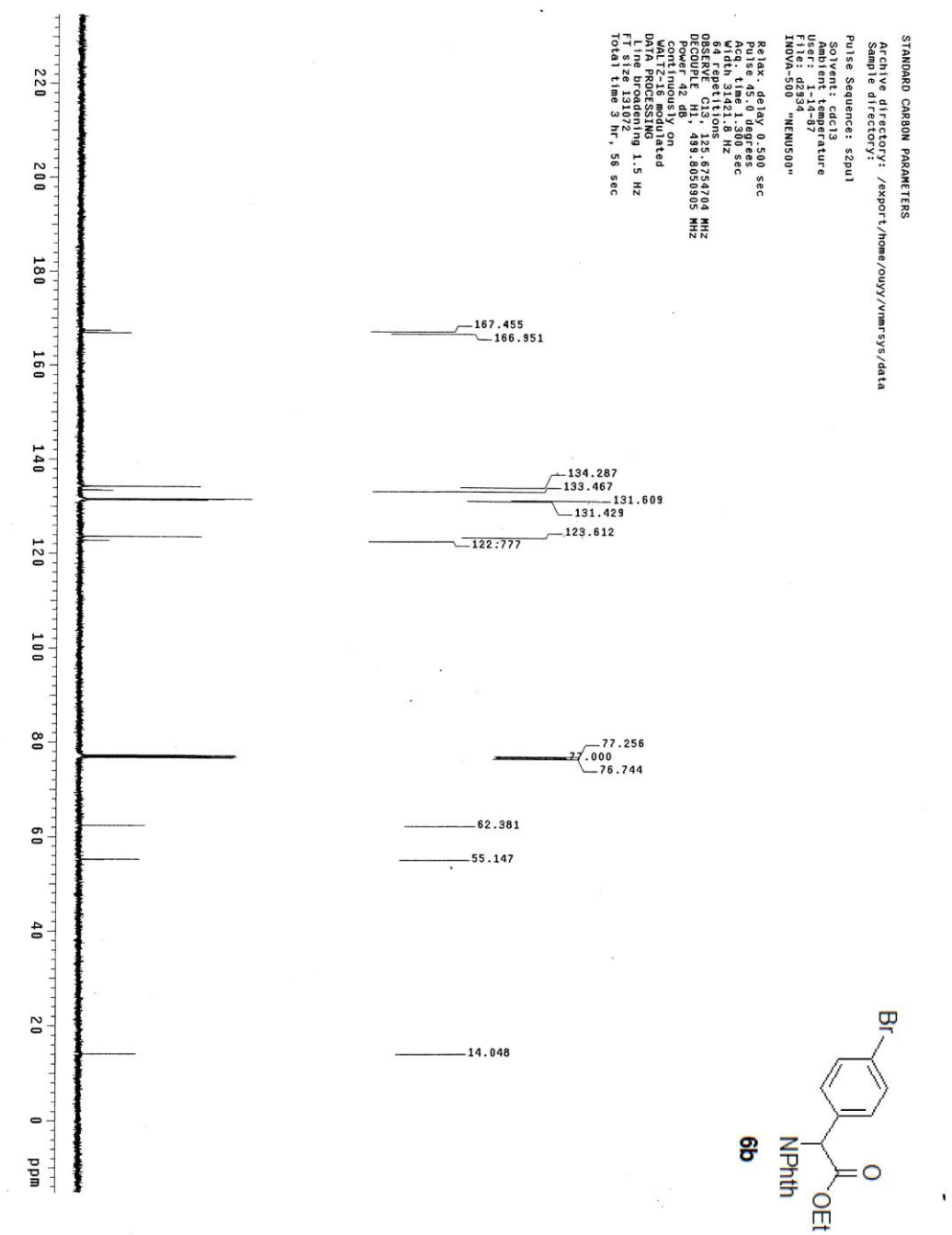

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2748
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1 repetitions
OBSERVE C13, 125.6754699 MHz
DECOUPLE H1, 499.8050995 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
181.552
169.309
158.635
137.858
135.157
134.363
126.828
125.604
121.354
93.008
77.252
77.000
76.744
61.679
18.584
13.918
220
200
180
160
140
120
100
80
60
40
20
0
ppm
4
OH
O
OEt



STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d2493
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9605.0 Hz
4 repetitions
OBSERVE H1, 499.8025917 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.860
7.853
7.849
7.843
7.728
7.722
7.717
7.711
7.558
7.544
7.377
7.364
7.349
7.341
7.332
7.327
7.263
6.011
4.321
4.306
4.292
4.278
2.176
1.669
1.273
1.259
1.244
1.00
1.05
1.02
1.55
0.48
1.05
1.50
9
8
7
6
5
4
3
2
1
0
-1 ppm
O
OEt
NPhth
6a


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2722 "MENU500"
INOVA-500 "MENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.301 sec
Width 3421.8 Hz
256 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 2 hr, 3 min, 31 sec
167.932
167.062
134.466
134.172
131.742
129.697
128.515
128.465
123.548
77.252
77.000
76.744
62.220
55.853
14.082
220
200
180
160
140
120
100
80
60
40
20
0
ppm
O
OEt
NPhth
6a


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dft20
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9744.2 Hz
4 repetitions
OBSERVE H1, 499.8025877 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 11 sec
7.864
7.857
7.853
7.847
7.746
7.738
7.732
7.728
7.722
7.490
7.473
7.441
7.423
7.267
5.950
4.314
4.300
4.286
4.271
1.706
1.267
1.254
1.239
0.000
2.01
2.04
2.03
2.06
0.99
2.10
3.00
ppm
Br
O
OEt
NPhth
6b


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d2934 "NENUS00"
INOVA-500
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31446.1 Hz
64 repetitions
OBSERVE C13, 125.675404 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
167.455
166.951
134.287
133.467
131.609
131.429
123.612
122.777
77.256
77.000
76.744
62.381
55.147
14.048
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Br
O
OEt
NPhth
6b