Low-Valent Niobium-Catalyzed Reduction of α,α,α-Trifluorotoluenes

Supporting Information

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1. General Statement

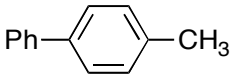
1,2-Dimethoxyethane (DME) was distilled, and stored over molecular sieves 4A under nitrogen atmosphere. $NbCl_5$ was used as purchased (Aldrich Co.) and handled under argon atmosphere. All the reactions were carried out under argon atmosphere.

Column chromatography and preparative thin layer chromatography (preparative TLC) were conducted on silica gel (PSQ 60B, Fuji Silysia Chemical Ltd. for column chromatography and Wakogel B-5F, Wako Pure Chemical Industries for preparative TLC, respectively).

NMR spectra were recorded on Unity Inova-400 instrument (Varian Ltd., 400 MHz for ^{1}H, 100 MHz for ^{13}C, 376 MHz for ^{19}F) and JNM-Al300 instrument (JEOL, 300 MHz for ^{1}H, 75 MHz for ^{13}C) using $CDCl_3$ as a solvent. Chemical shifts (δ) for ^{1}H were referenced to tetramethylsilane (δ = 0.00 ppm) as an internal standard. Chemical shifts (δ) for ^{13}C were referenced to a solvent signal ($CDCl_3$, δ = 77.00 ppm). Chemical shifts (δ) for ^{19}F were referenced to α,α,α-trifluorotoluene (δ = –63.9 ppm) or hexafluorobenzene (δ = –162.9 ppm) as an internal standard. IR spectra were recorded on FTIR-8600PC instrument (Shimadzu Co.) using $CHCl_3$ as a solvent.

2. Spectra Data of the Products

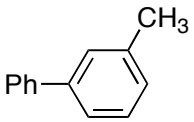
2-1. 4-Phenyltoluene **2a**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.58 (2H, d, *J* = 7.2 Hz), 7.49 (2H, d, *J* = 8.0 Hz), 7.42 (2H, t, *J* = 7.4 Hz), 7.32 (1H, t, *J* = 7.2 Hz), 7.25 (2H, d, *J* = 8.0 Hz), 2.39 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 141.1, 138.3, 137.0, 129.5, 128.7, 127.0, 126.9, 21.1.

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (Tao, B.; Boykin, D. W. *J. Org. Chem.* **2004**, *69*, 4330-4335.).

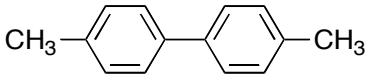
2-2. 3-Phenyltoluene **2b**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.59 (2H, d, *J* = 8.0 Hz), 7.46–7.37 (4H, m), 7.36–7.30 (2H, m), 7.17 (1H, d, *J* = 7.6 Hz), 2.42 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 141.3, 141.2, 138.3, 128.7, 128.6, 127.97, 127.96, 127.2, 127.1, 124.3, 21.5.

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (‘*The Aldrich Library of ^{13}C and ^{1}H FT NMR Spectra*’, Pouchert, C. P.; Behnke, J., Ed., Edition I, Vol. 2, 32A.).

2-3. 4,4’-Dimethylbiphenyl **2c**.

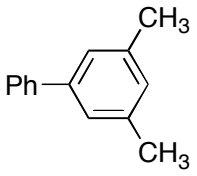


^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 (4H, d, *J* = 8.4 Hz), 7.24 (4H, d, *J* = 8.4 Hz), 2.39 (6H, s).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.2, 136.7, 129.4, 126.8, 21.1.

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (Xu, X.; Cheng, D.; Pei, W. *J. Org. Chem.* **2006**, *71*, 6637.).

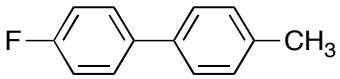
2-4. 3,5-Dimethylbiphenyl **2d**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.58 (2H, d, *J* = 7.4 Hz), 7.42 (2H, t, *J* = 7.6 Hz), 7.33 (1H, t, *J* = 7.4 Hz), 7.21 (2H, s), 7.00 (1H, s), 2.38 (6H, s). **^{13}C NMR** (75 MHz, $CDCl_3$): δ = 141.4, 141.2, 138.2, 128.9, 128.6, 127.2, 127.1, 125.1, 21.4.

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (Li, J.-H; Tang, B-X; Tao, L-M.; Xie, Y.-X.; Liang, Y.; Zhang, M.-B. *J. Org. Chem.* **2006**, 71, 7488.).

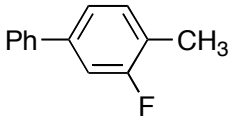
2-5. 4-Fluoro-4'-methylbiphenyl **2e**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54–7.51 (2H, dd, *J* = 8.8, 5.6 Hz), 7.44 (2H, d, *J* = 8.0 Hz), 7.24 (2H, d, *J* = 8.0 Hz), 7.11 (2H, t, *J* = 8.8 Hz), 2.39 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.3 (d, *J* = 244 Hz), 137.3, 137.2 (d, *J* = 3.0 Hz), 137.0, 129.5, 128.4 (d, *J* = 8.0 Hz), 126.8, 115.5 (d, *J* = 21 Hz), 21.1. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –117.5 (m).

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literatures (^{1}H NMR: Moore, L. R.; Shaughnessy, K. H. *Org. Lett.* **2004**, *6*, 225; ^{13}C NMR: Wang, Y.; Sauer, D. R. *Org. Lett.* **2004**, *6*, 2793.).

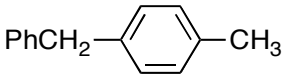
2-6. 3-Fluoro-4-phenyltoluene **2f**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56 (2H, d, *J* =7.4 Hz), 7.44 (2H, t, *J* = 7.4 Hz), 7.35 (1H, t, *J* = 7.2 Hz), 7.29–7.22 (3H, m), 2.31 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 161.6 (d, *J* = 243 Hz), 140.7 (d, *J* = 7.6 Hz), 139.9 (d, *J* = 2.3 Hz), 131.7 (d, *J* = 5.3 Hz), 128.8, 127.5, 126.9, 123.6 (d, *J* = 17 Hz), 122.3 (d, *J* = 3.1 Hz), 113.4 (d, *J* = 23 Hz), 14.3 (d, *J* = 3.4 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –118.7 (t, *J* = 9.2 Hz).

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (Lipshutz, B. H.; Siegmann, K.; Garcia, E.; Kayser, F. *J. Am. Chem. Soc.* **1993**, *115*, 9276.).

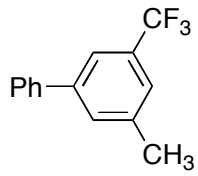
2-7. 4-Benzyltoluene **2h**.



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.29–7.26 (2H, m), 7.22–7.16 (3H, m), 7.09 (4H, s), 3.94 (2H, s), 2.31 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 141.4, 138.0, 135.5, 129.1, 128.84, 128.78, 128.4, 125.9, 41.5, 21.0.

The ^{1}H and ^{13}C NMR spectra were in complete agreement with those in the literature (Molander, G. A.; Elia, M. D. *J. Org. Chem.* **2006**, *71*, 9198.).

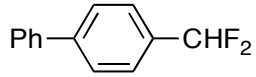
2-8. 3-Methyl-5-(trifluoromethyl)biphenyl **1d**.



1d

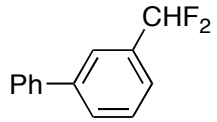
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63 (1H, s), 7.61–7.53 (3H, m), 7.50–7.43 (2H, m), 7.42–7.36 (2H, m), 2.48 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 142.0, 139.9, 139.2, 131.2, 131.1 (q, J = 31.7 Hz), 128.9, 127.9, 127.2, 124.5, (q, J = 3.7 Hz), 124.3 (q, J = 271 Hz), 121.2 (q, J = 3.8 Hz), 21.4. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –63.7 (s). **IR** ($CHCl_3$): ν~ = 3618, 3018, 2401, 1356, 1128, 872 cm^{-1}.

2-9. 4-Phenyl-α,α-difluorotoluene **3**.



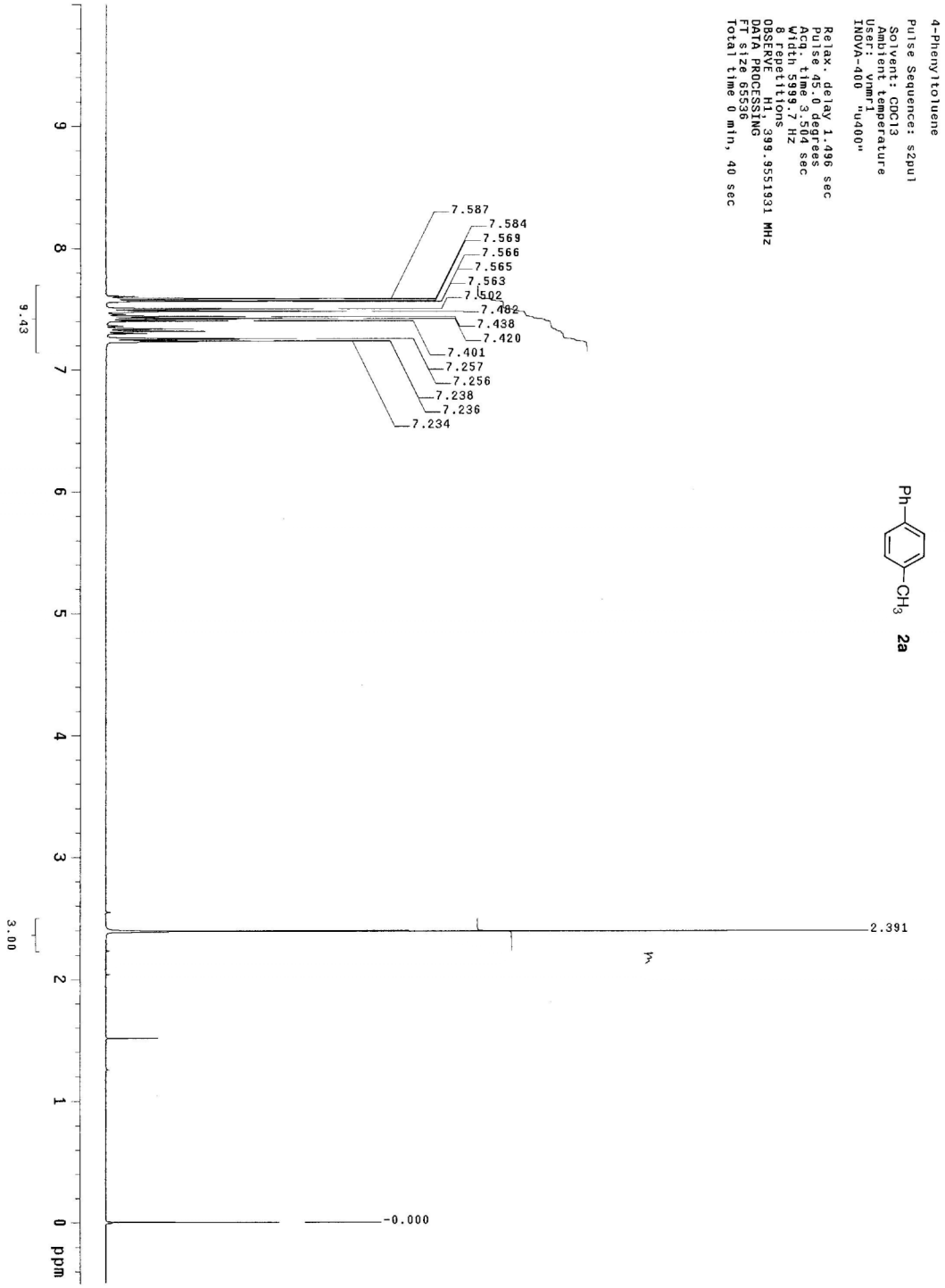
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.67 (2H, d, J = 7.6 Hz), 7.62–7.55 (4H, m), 7.46 (2H, t, J = 7.8 Hz), 7.38 (1H, t, J = 7.2 Hz), 6.69 (1H, t, J = 56 Hz). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 143.7, 140.1, 133.2 (t, J = 13 Hz), 128.9, 127.9, 127.4, 127.2, 126.0 (t, J = 5.9 Hz), 114.7 (t, J = 237 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –111.5 (d, J = 56 Hz). **IR** ($CHCl_3$): ν~ = 3020, 1377, 1219, 1076, 1034, 733 cm^{-1}.

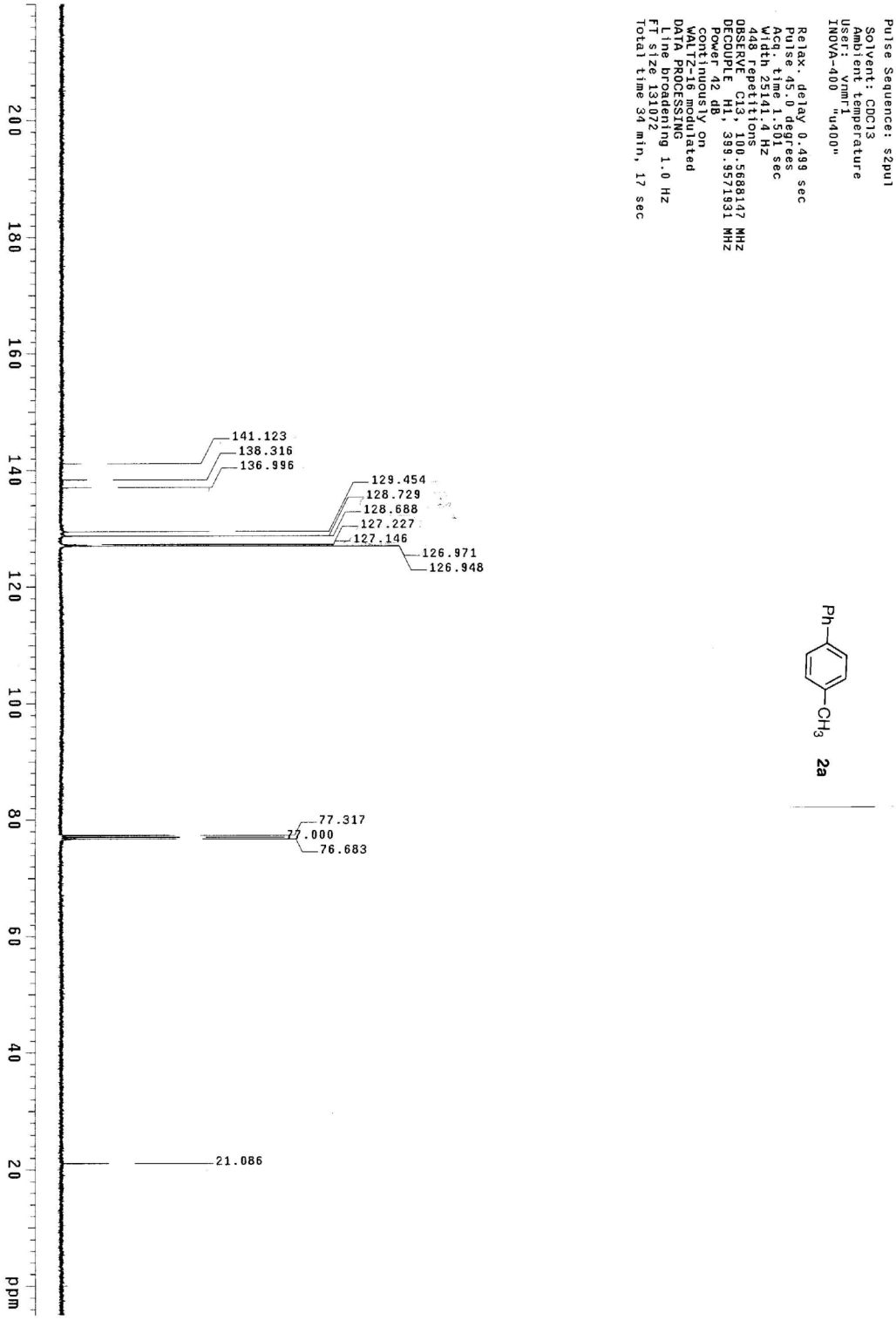
2-10. 3-Phenyl-α,α-difluorotoluene **4**.

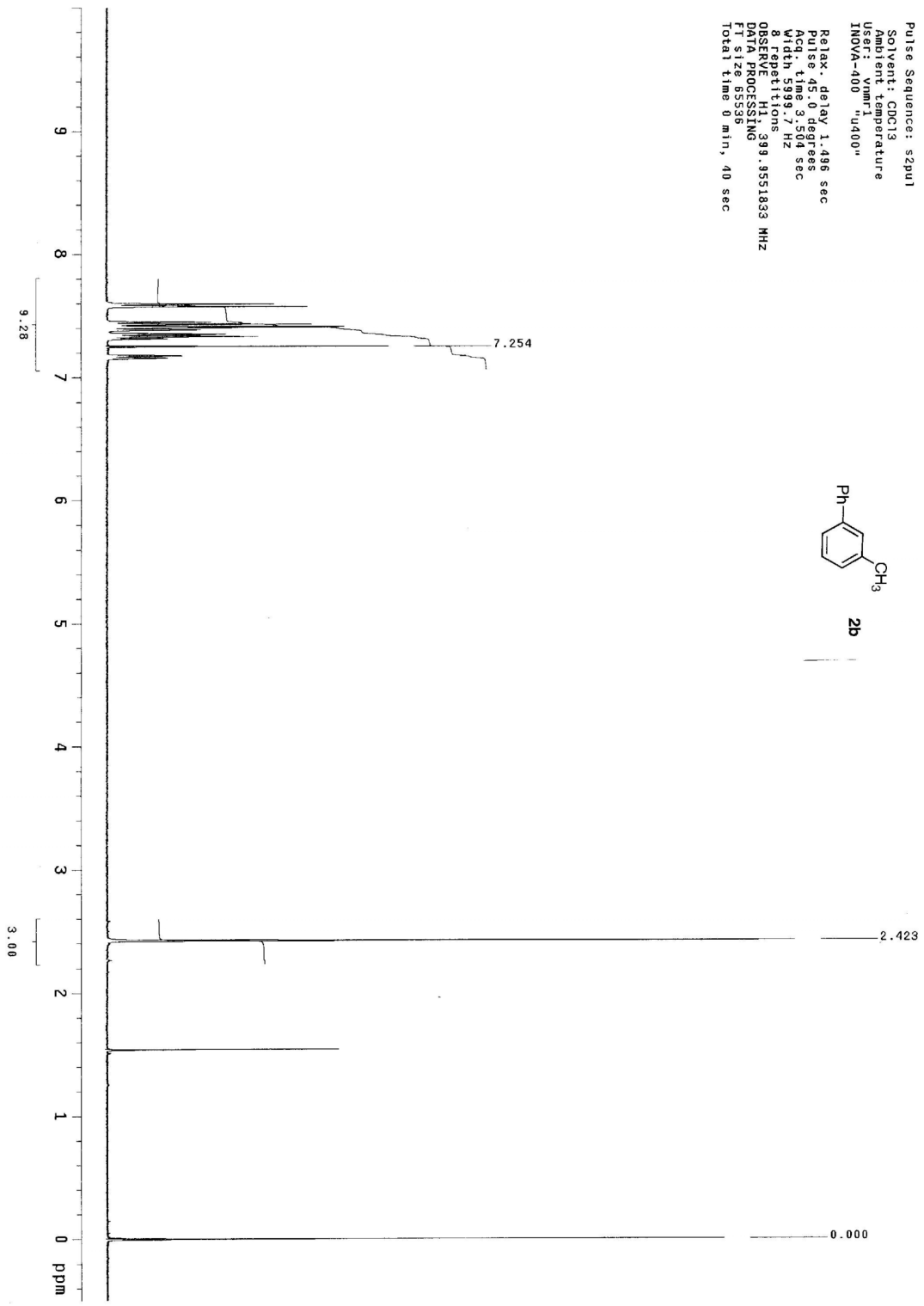


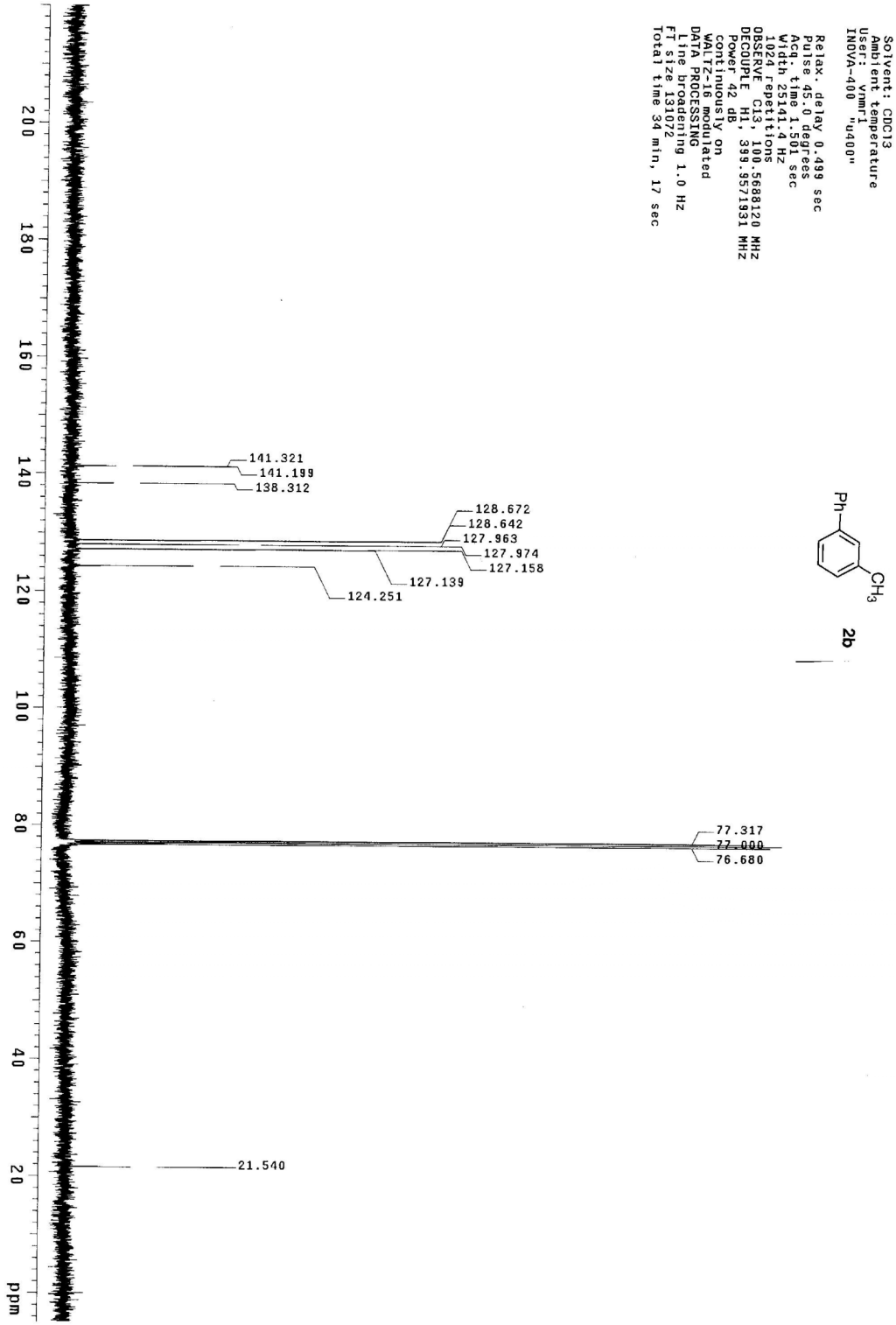
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.73–7.67 (2H, m), 7.59 (2H, d, J = 8.4 Hz), 7.55–7.43 (4H, m), 7.38 (1H, t, J = 7.2 Hz), 6.70 (1H, t, J = 56 Hz). **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 141.8, 140.2, 134.9 (t), 129.4 (t, J = 2.0 Hz), 129.2, 128.9, 127.8, 127.2, 124.32 (t, J = 6.1 Hz), 124.28 (t, J = 6.1 Hz), 114.7 (t, J = 238 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$): δ = –111.8 (d, J = 56 Hz). **IR** ($CHCl_3$): ν~ = 3067, 1369, 1219, 1065, 1034, 702 cm^{-1}.

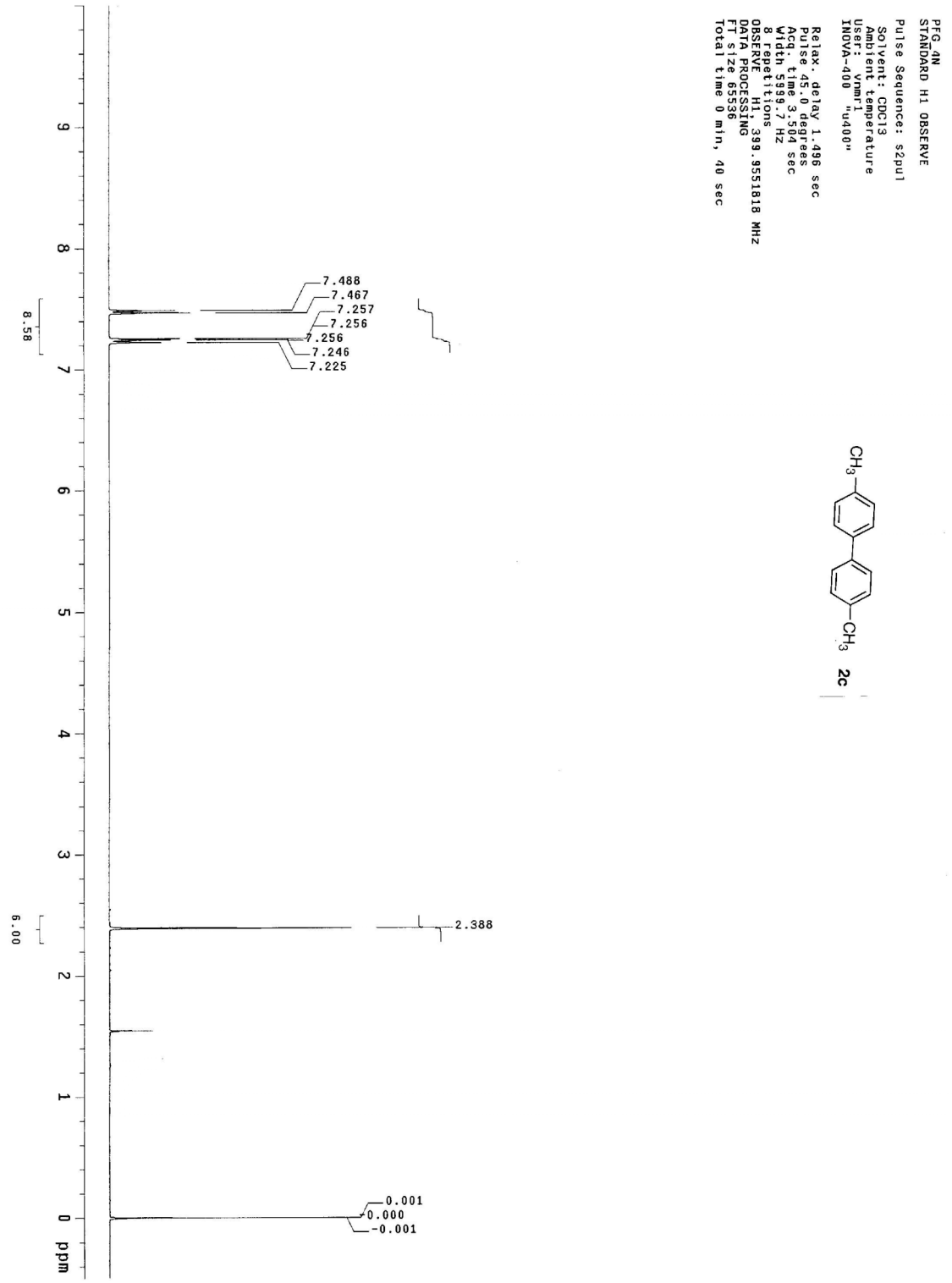
3. ^{1}H, ^{13}C , and ^{19}F NMR Spectra of **2a-h**, **1d**, **3**, and **4**

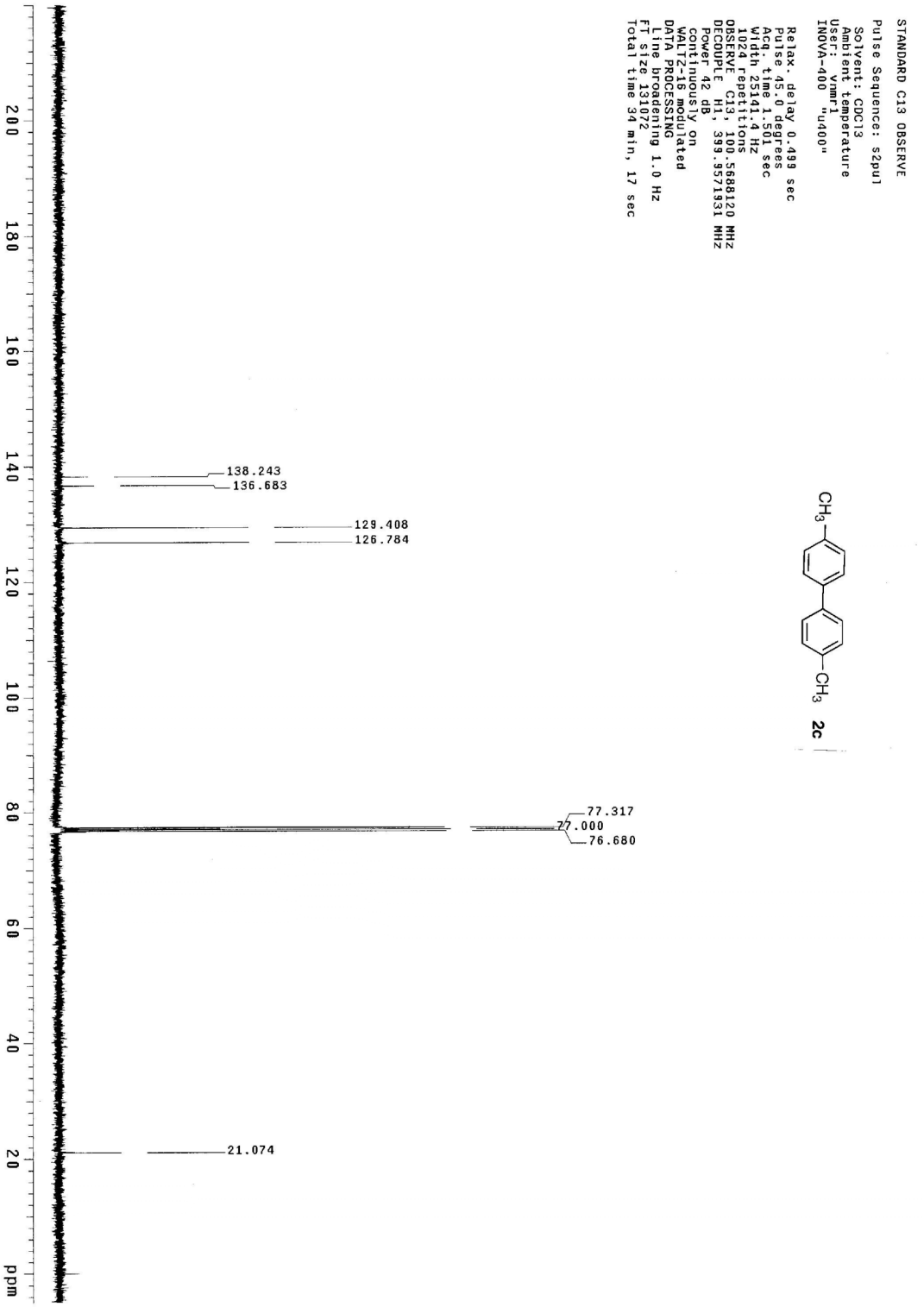


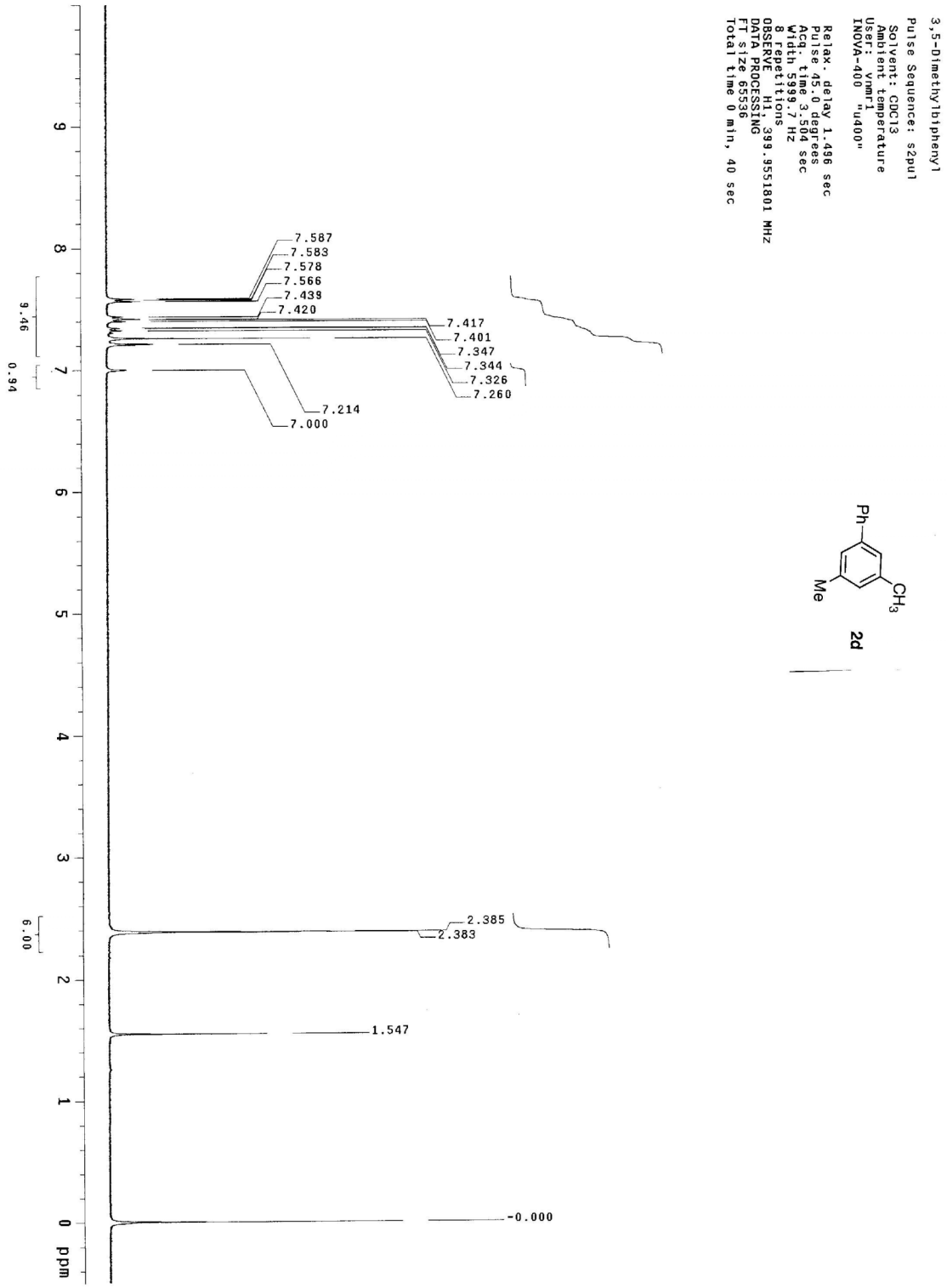

STANDARD C13 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
448 repetitions
OBSERVE C13, 100.5688147 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
141.123
138.316
136.996
129.454
128.729
128.688
127.227
127.146
126.971
126.948
77.317
77.000
76.683
21.086
200
180
160
140
120
100
80
60
40
20
ppm
Ph
CH3
2a


PFG_4N
STANDARD H1 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 HZ
8 repetitions
OBSERVE H1, 399.9551833 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
Ph
CH3
2b
7.254
2.423
0.000
9.28
3.00
ppm


STANDARD C13 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.581 sec
Width 25141.4 Hz
1024 repetitions
OBSERVE C13, 100.5688120 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
Ph
CH3
2b
141.321
141.199
138.312
128.672
128.642
127.963
127.974
127.158
127.139
124.251
77.317
77.000
76.680
21.540
200
180
160
140
120
100
80
60
40
20
ppm


PFG_4N
STANDARD H1 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551818 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
CH3
CH3
2c
7.488
7.467
7.257
7.256
7.256
7.246
7.225
8.58
2.388
6.00
0.001
0.000
-0.001
9
8
7
6
5
4
3
2
1
0
ppm

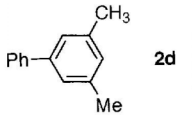

STANDARD C13 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
1024 repetitions
OBSERVE C13, 100.5688120 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
CH3
CH3
2c
138.243
136.683
129.408
126.784
77.317
77.000
76.680
21.074
200
180
160
140
120
100
80
60
40
20
ppm


3,5-Dimethylbiphenyl
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 HZ
8 repetitions
OBSERVE H1, 399.9551801 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.587
7.583
7.578
7.566
7.439
7.420
7.417
7.401
7.347
7.344
7.326
7.260
7.214
7.000
2.385
2.383
1.547
-0.000
9.46
0.94
6.00
ppm
Ph
Me
CH3
2d

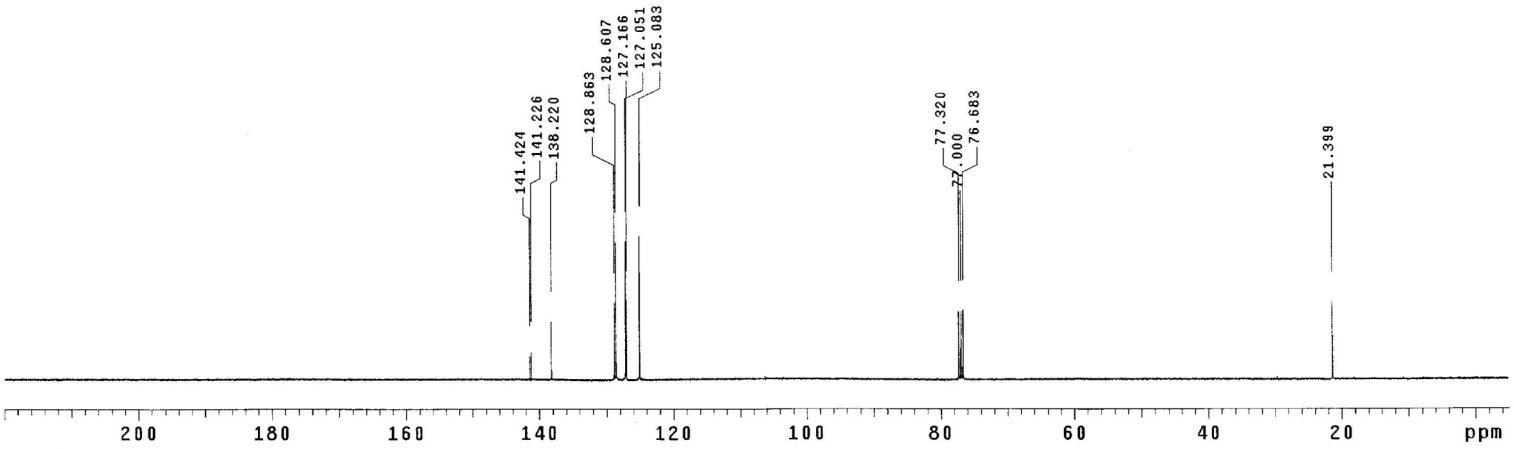
3-5
~~4,4'~~-Dimethylbiphenyl

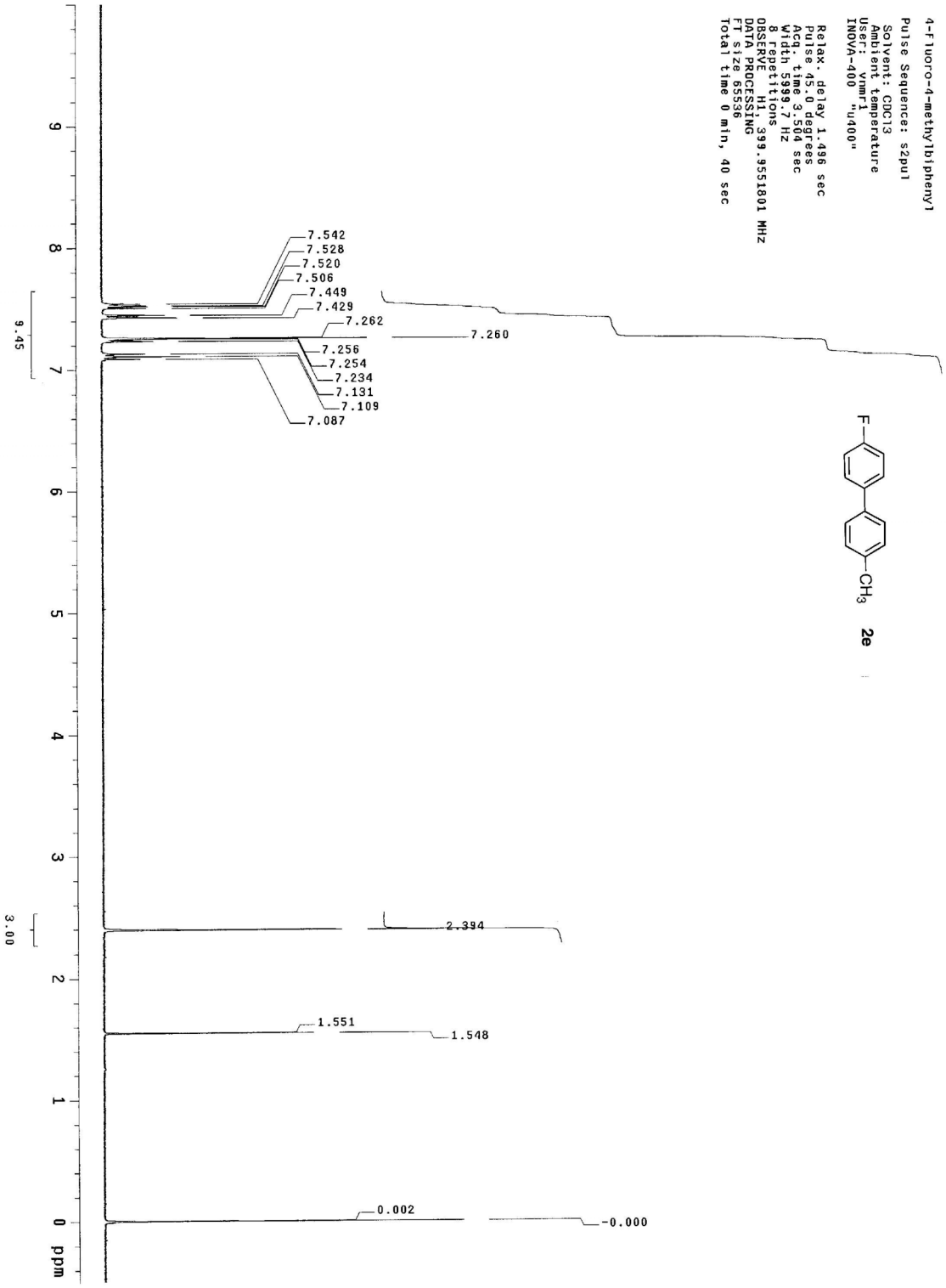
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"

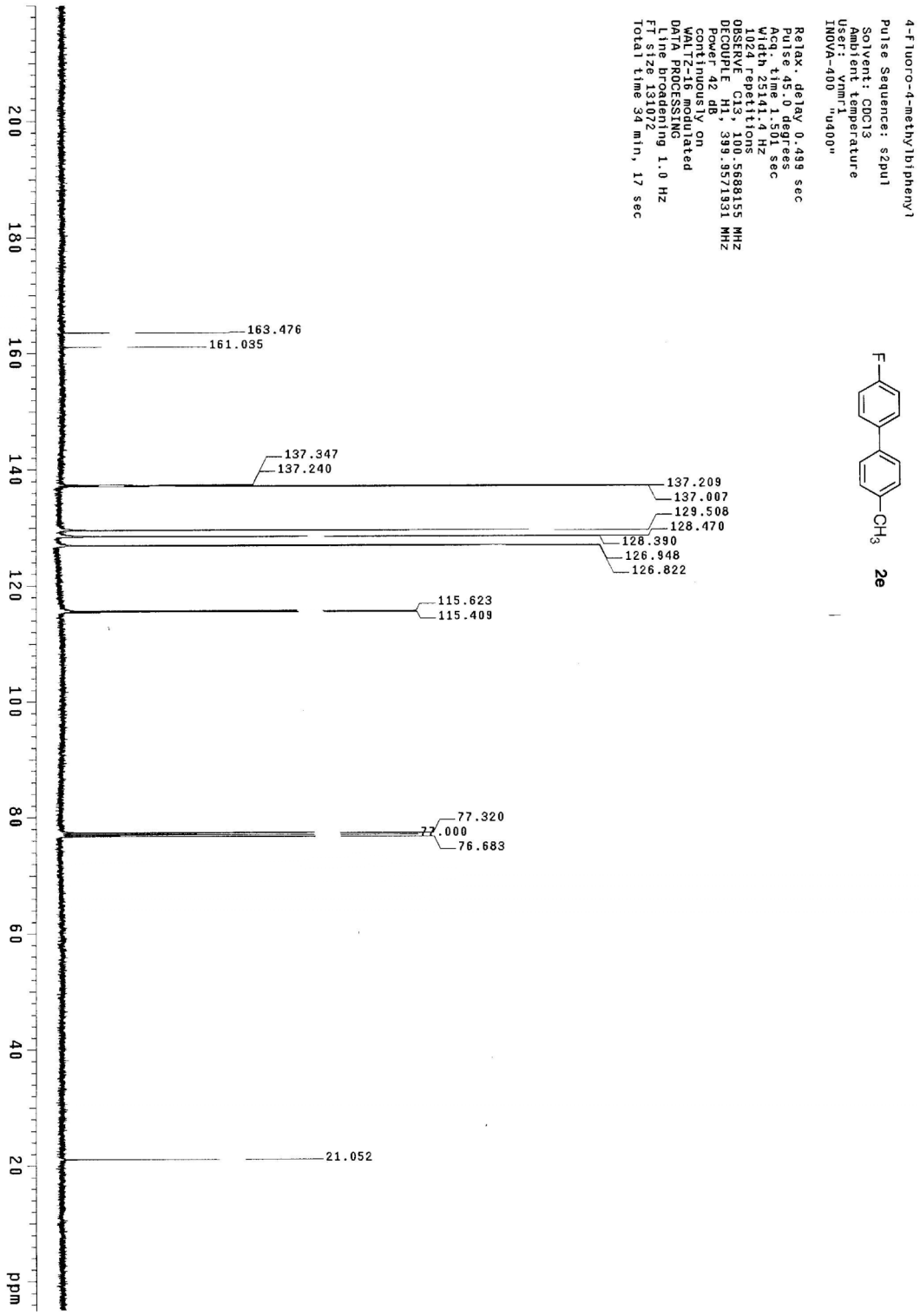
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
1024 repetitions
OBSERVE C13, 100.5688158 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec



2d



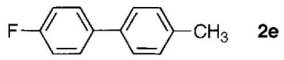

4-fluoro-4-methylbiphenyl
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551801 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.542
7.528
7.520
7.506
7.449
7.429
7.262
7.260
7.256
7.254
7.234
7.131
7.109
7.087
2.394
1.551
1.548
0.002
-0.000
9.45
3.00
F
CH3
2e
ppm


4-Fluoro-4-methylbiphenyl
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
1024 repetitions
OBSERVE C13, 100.5688155 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
F
CH3
2e
163.476
161.035
137.347
137.240
137.209
137.007
129.508
128.470
128.390
126.948
126.822
115.623
115.409
77.320
77.000
76.683
21.052
200
180
160
140
120
100
80
60
40
20
ppm

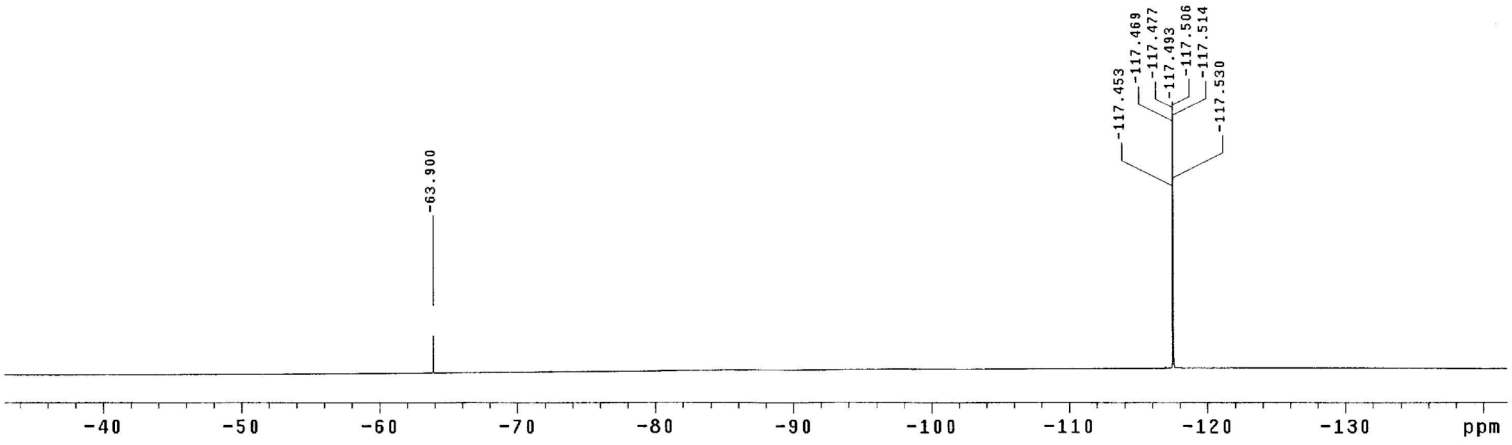
STANDARD F19 OBSERVE

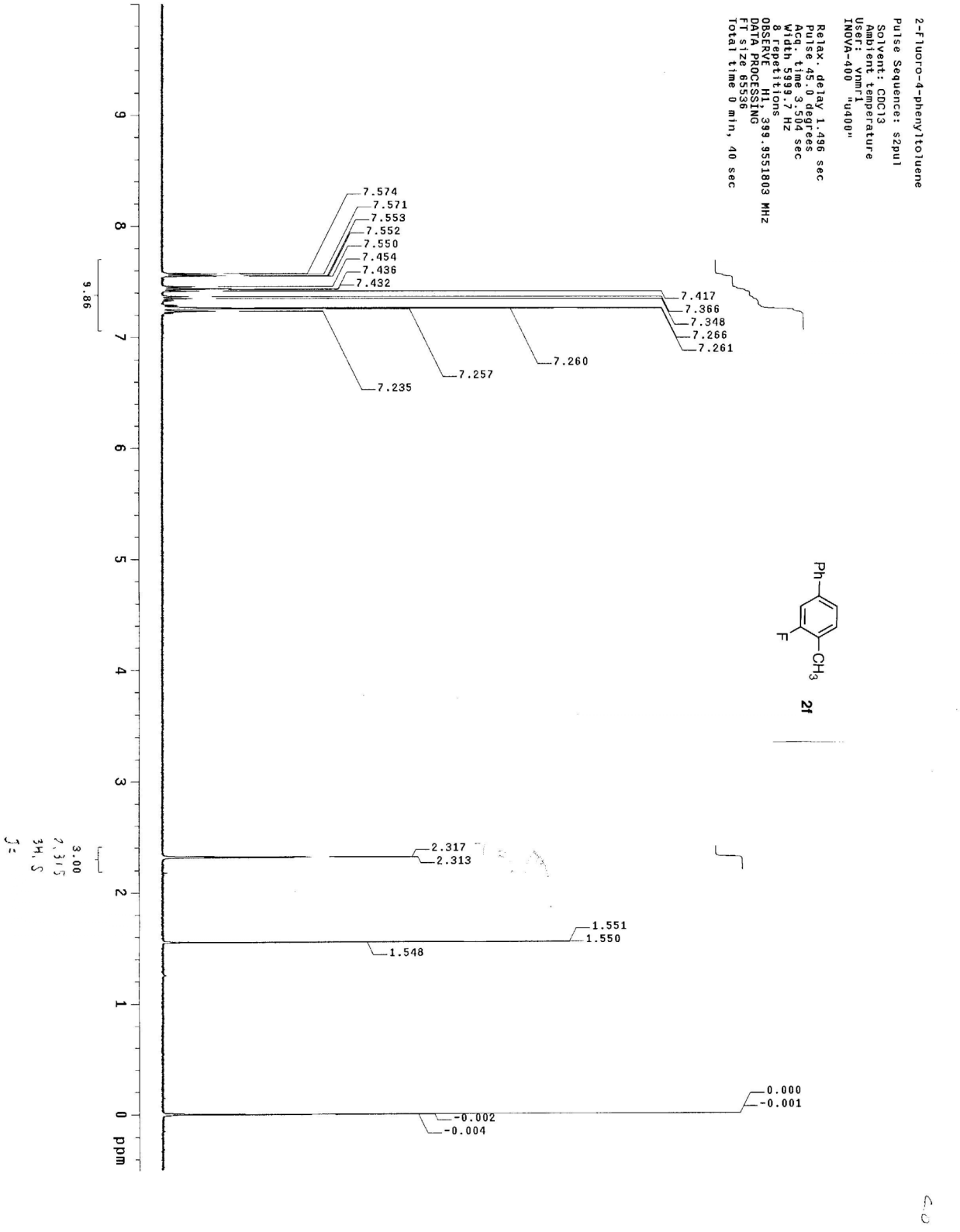
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"

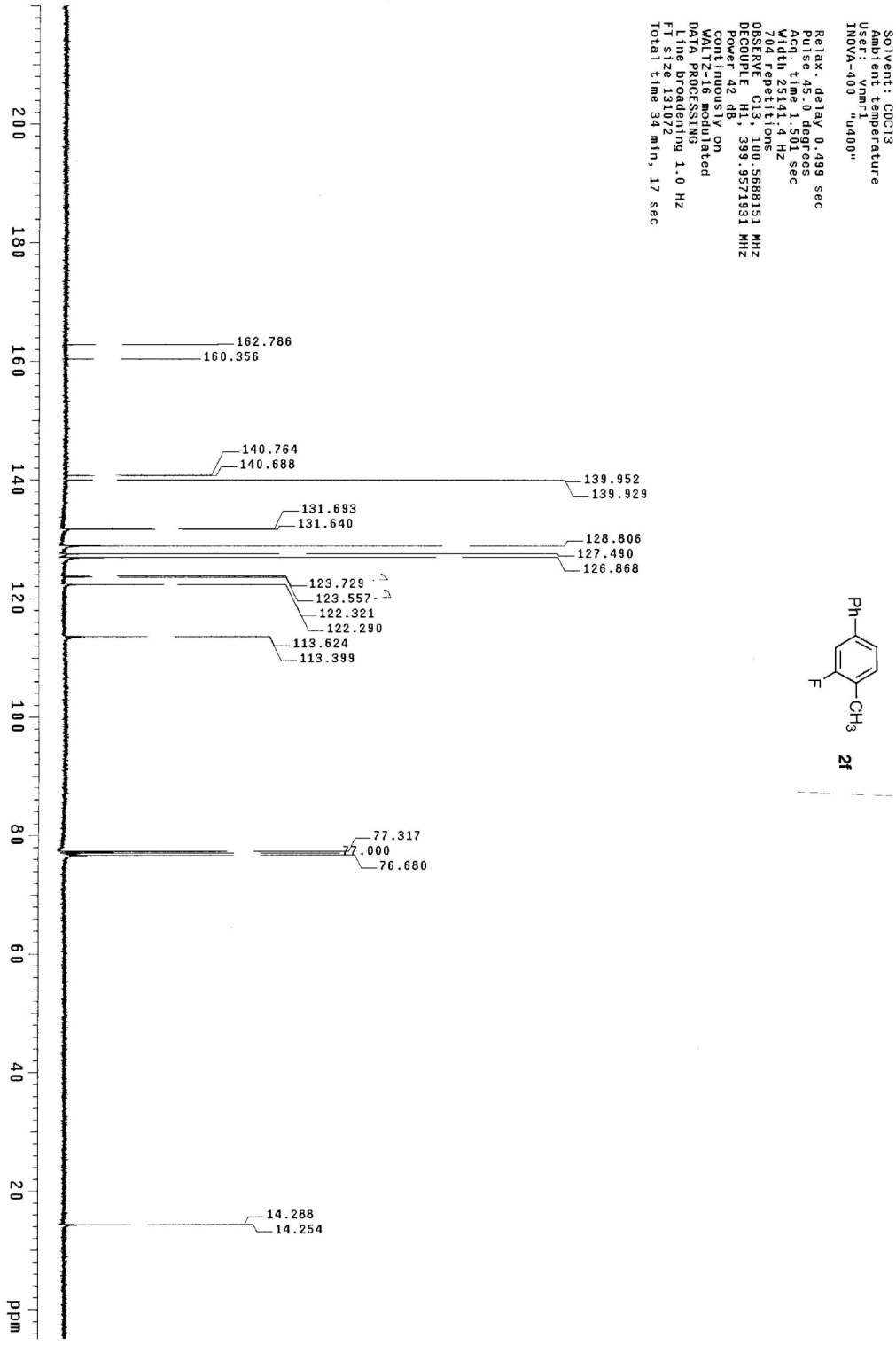
Relax. delay 9.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 100.0 kHz
16 repetitions
OBSERVE F19, 376.3343341 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec

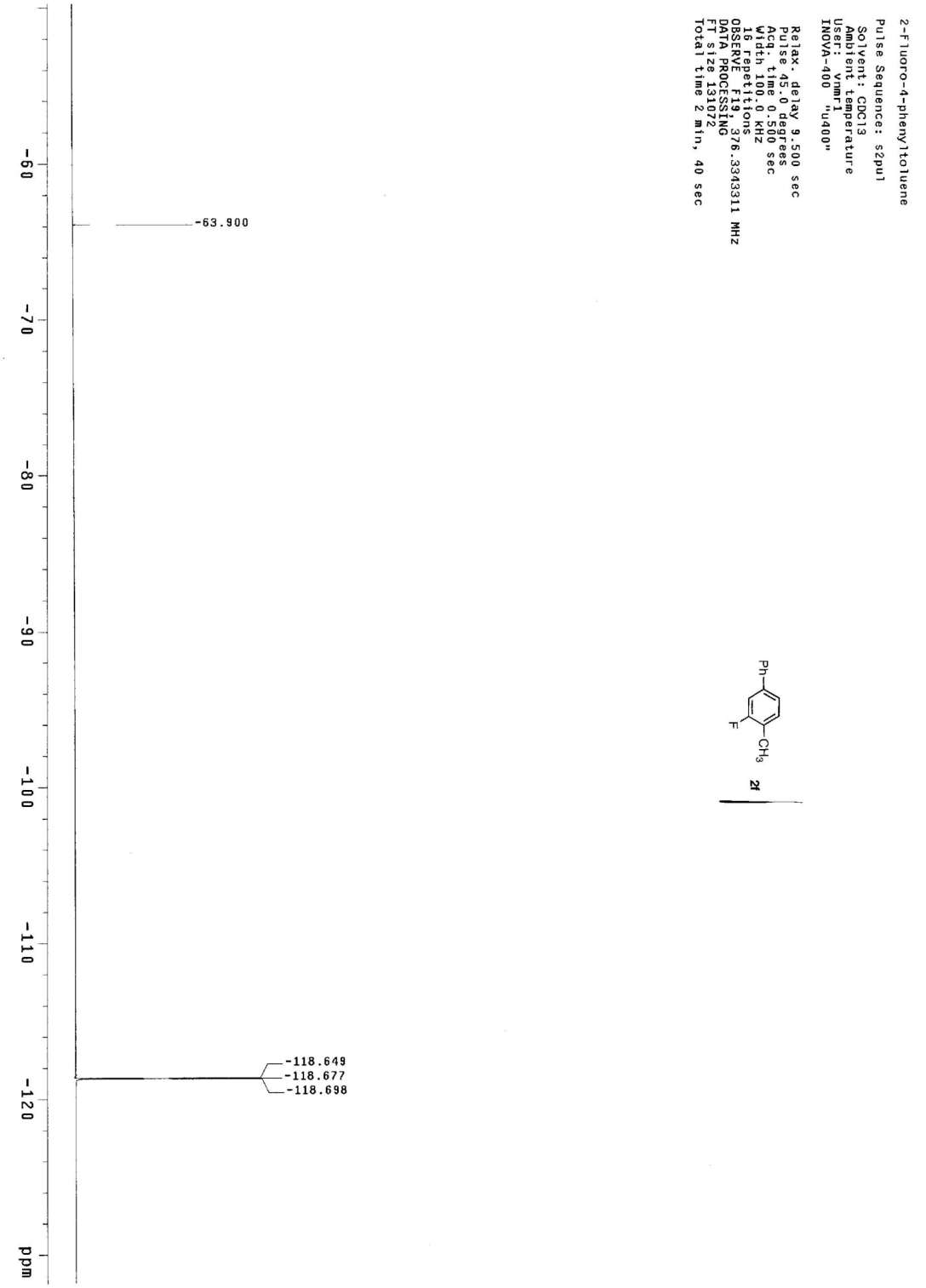


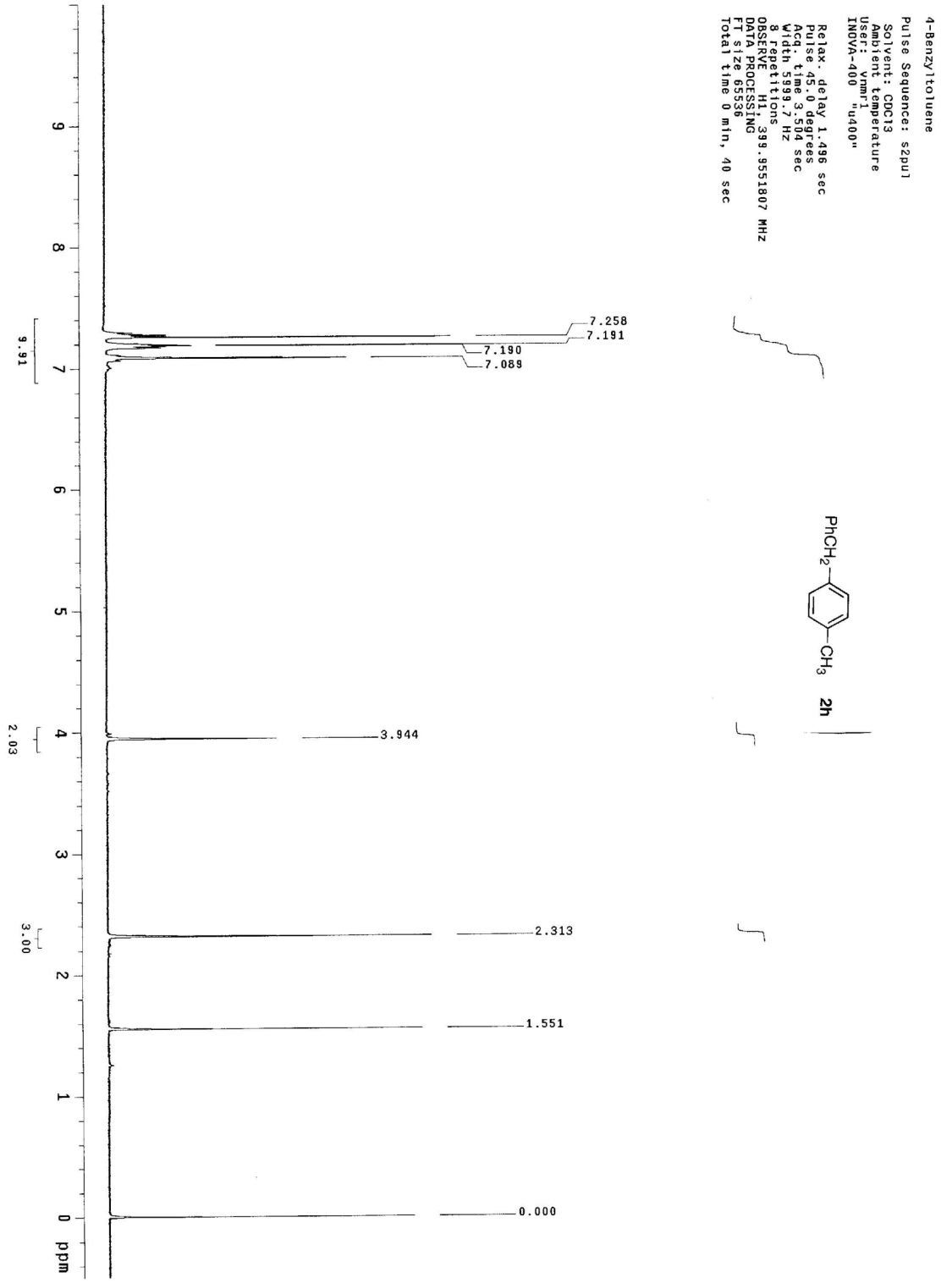
2e

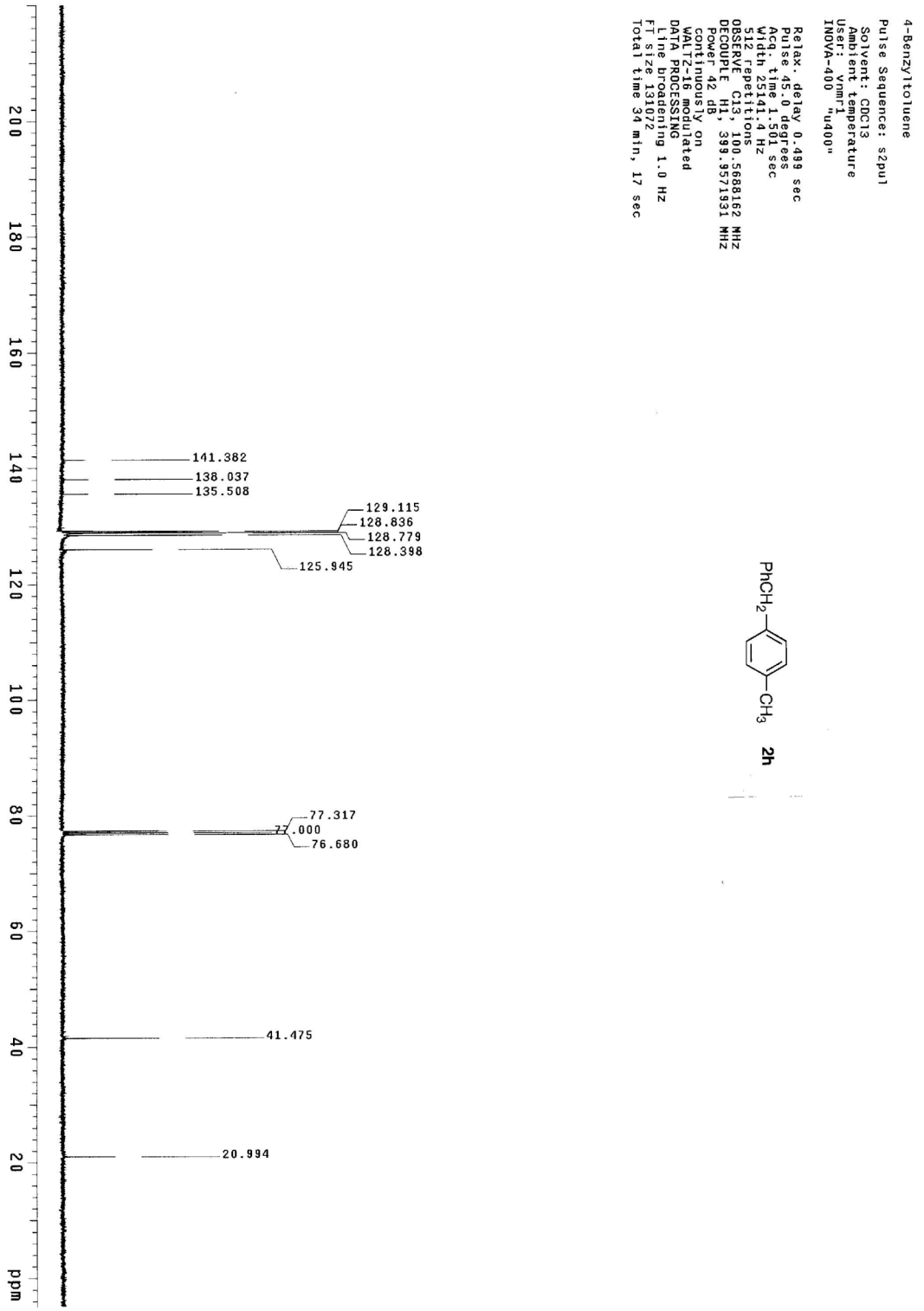


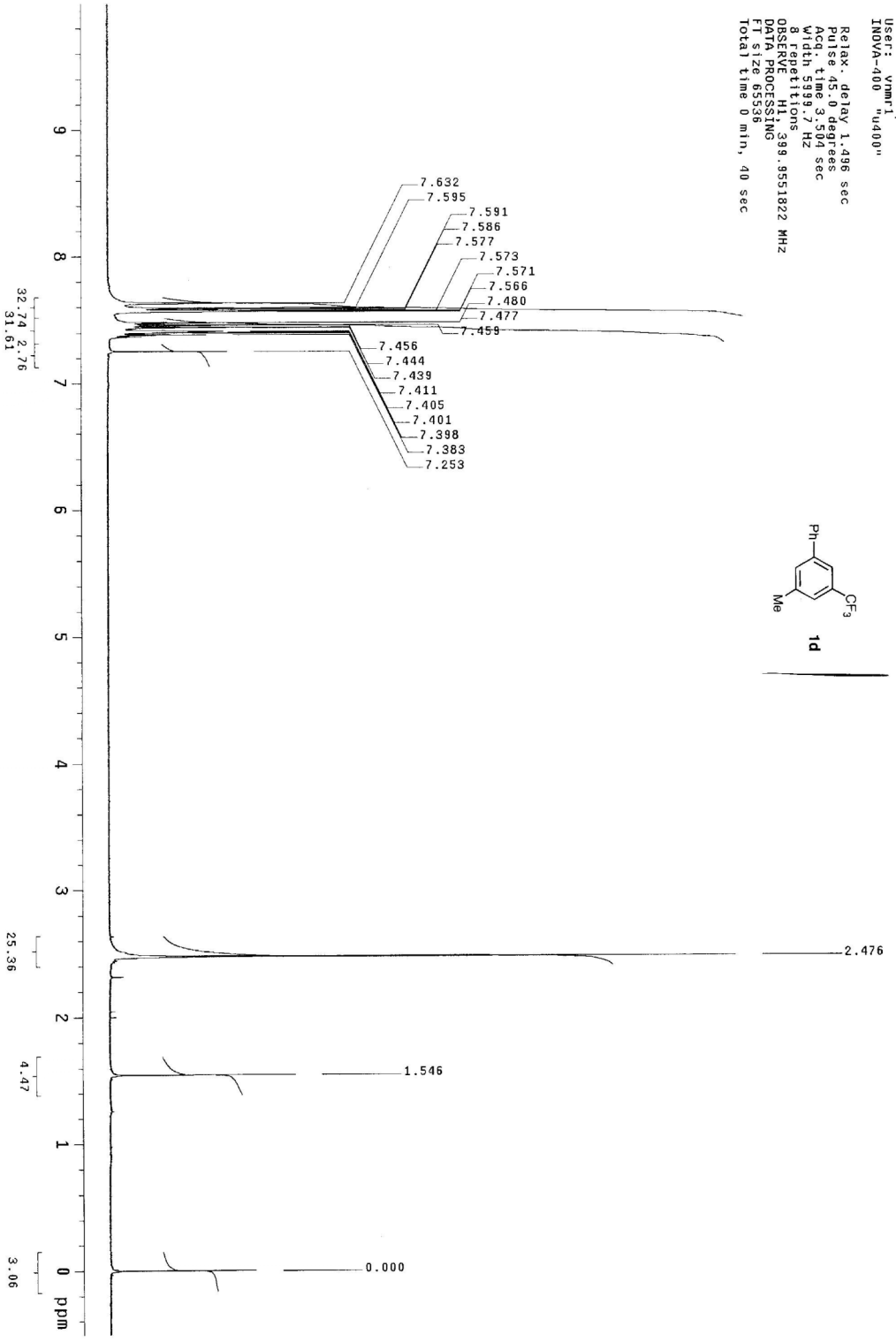

2-Fluoro-4-phenyltoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551803 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.574
7.571
7.553
7.552
7.550
7.454
7.436
7.432
7.417
7.366
7.348
7.266
7.261
7.260
7.257
7.235
2.317
2.313
1.551
1.550
1.548
0.000
-0.001
-0.002
-0.004
9.86
3.00
Ph
F
CH3
2f
ppm

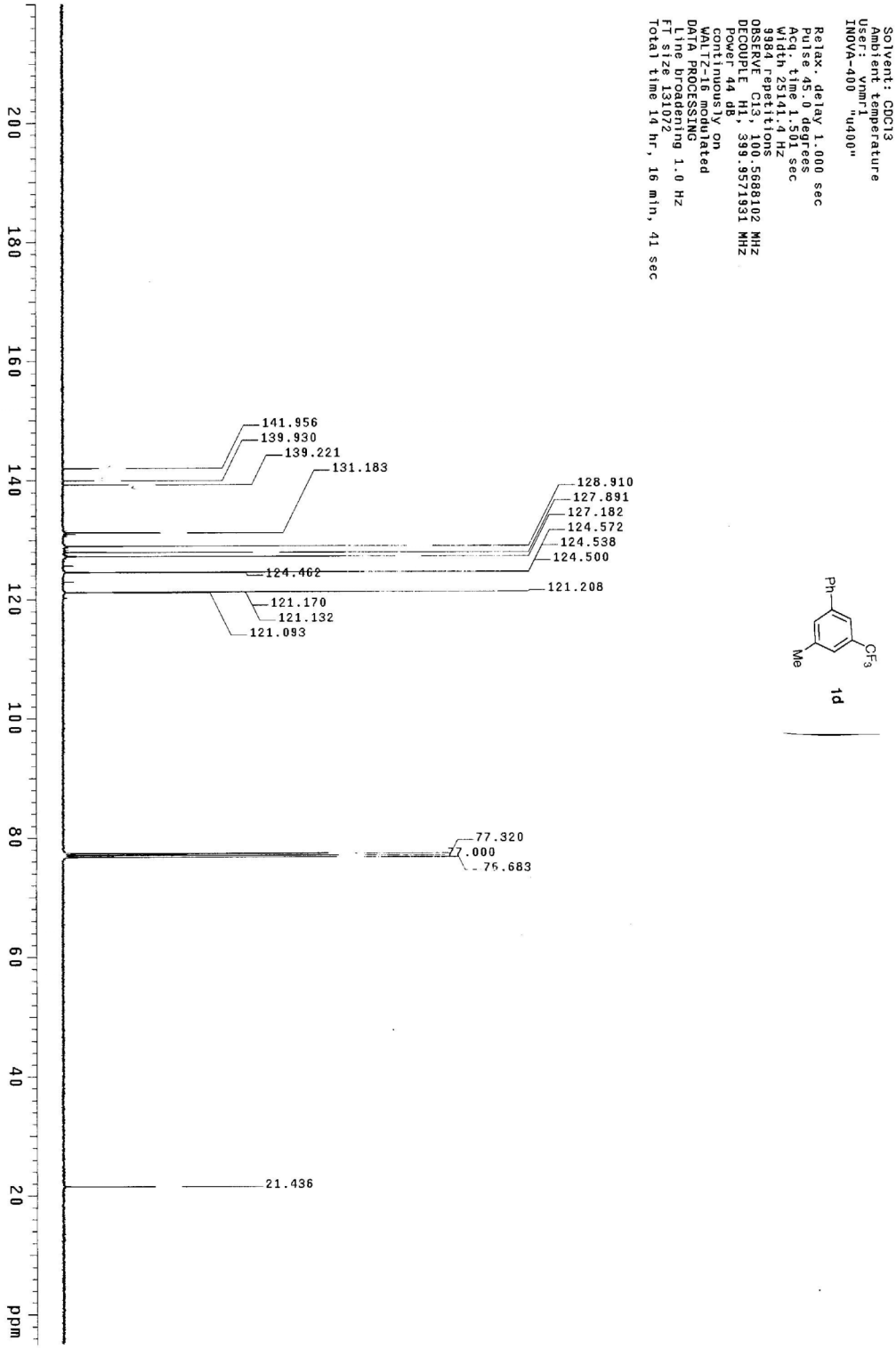

2-fluoro-4-phenyltoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
704 repetitions
OBSERVE C13, 100.5688151 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
162.786
160.356
140.764
140.688
139.952
139.929
131.693
131.640
128.806
127.490
126.868
123.729
123.557
122.321
122.290
113.624
113.399
77.317
77.000
76.680
14.288
14.254
200
180
160
140
120
100
80
60
40
20
ppm
Ph
F
CH3
2f


2-Fluoro-4-phenyltoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 9.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 100.0 kHz
16 repetitions
OBSERVE F19, 376.3343311 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec
Ph
F
CH3
2f
-63.900
-118.649
-118.677
-118.698
-60
-70
-80
-90
-100
-110
-120
ppm


4-Benzyltoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551807 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
PhCH2
CH3
2h
7.258
7.191
7.190
7.089
3.944
2.313
1.551
0.000
9.91
2.03
3.00
ppm


4-Benzyltoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
512 repetitions
OBSERVE C13, 100.5688162 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 34 min, 17 sec
PhCH2
CH3
2h
141.382
138.037
135.508
129.115
128.836
128.779
128.398
125.945
77.317
77.000
76.680
41.475
20.994
200
180
160
140
120
100
80
60
40
20
ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1 399.9551822 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.632
7.595
7.591
7.586
7.577
7.573
7.571
7.566
7.480
7.477
7.459
7.456
7.444
7.439
7.411
7.405
7.401
7.398
7.383
7.253
Ph
Me
CF3
1d
2.476
1.546
0.000
32.74
2.76
31.61
25.36
4.47
3.06
ppm

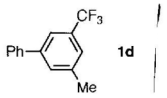

STANDARD C13 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
3984 repetitions
OBSERVE C13, 100.5688102 MHz
DECOUPLE H1, 399.9571931 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 14 hr, 16 min, 41 sec
141.956
139.930
139.221
131.183
128.910
127.891
127.182
124.572
124.538
124.500
124.462
121.208
121.170
121.132
121.093
77.320
77.000
76.683
21.436
200
180
160
140
120
100
80
60
40
20
ppm
Ph
CF3
Me
1d

3-trifluoromethyl-5-methylbiphenyl

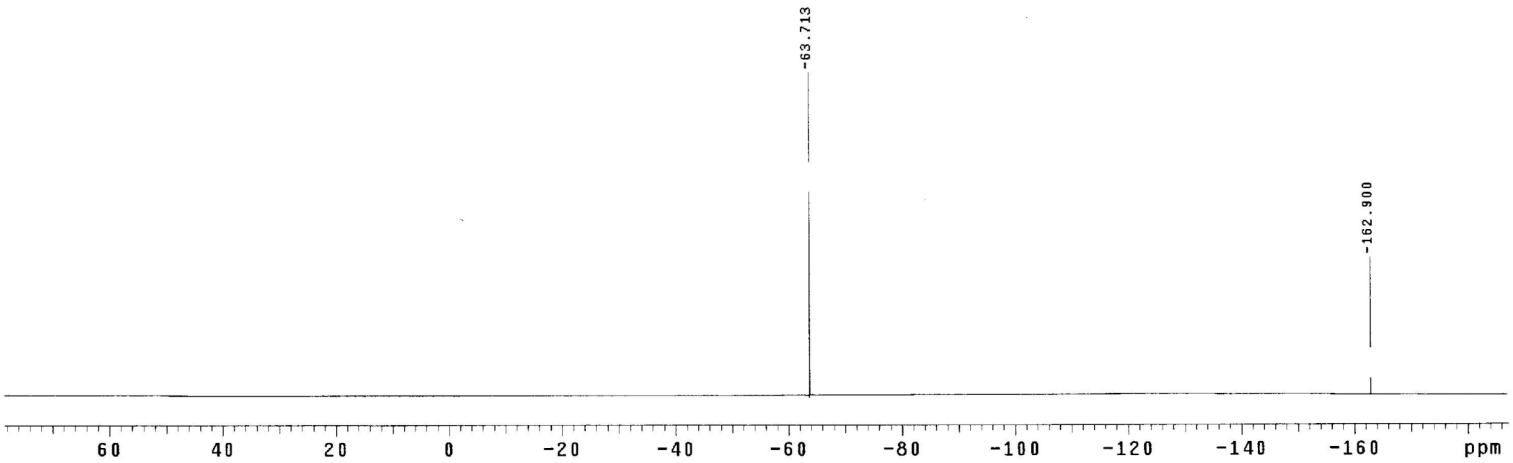
Pulse Sequence: s2pul

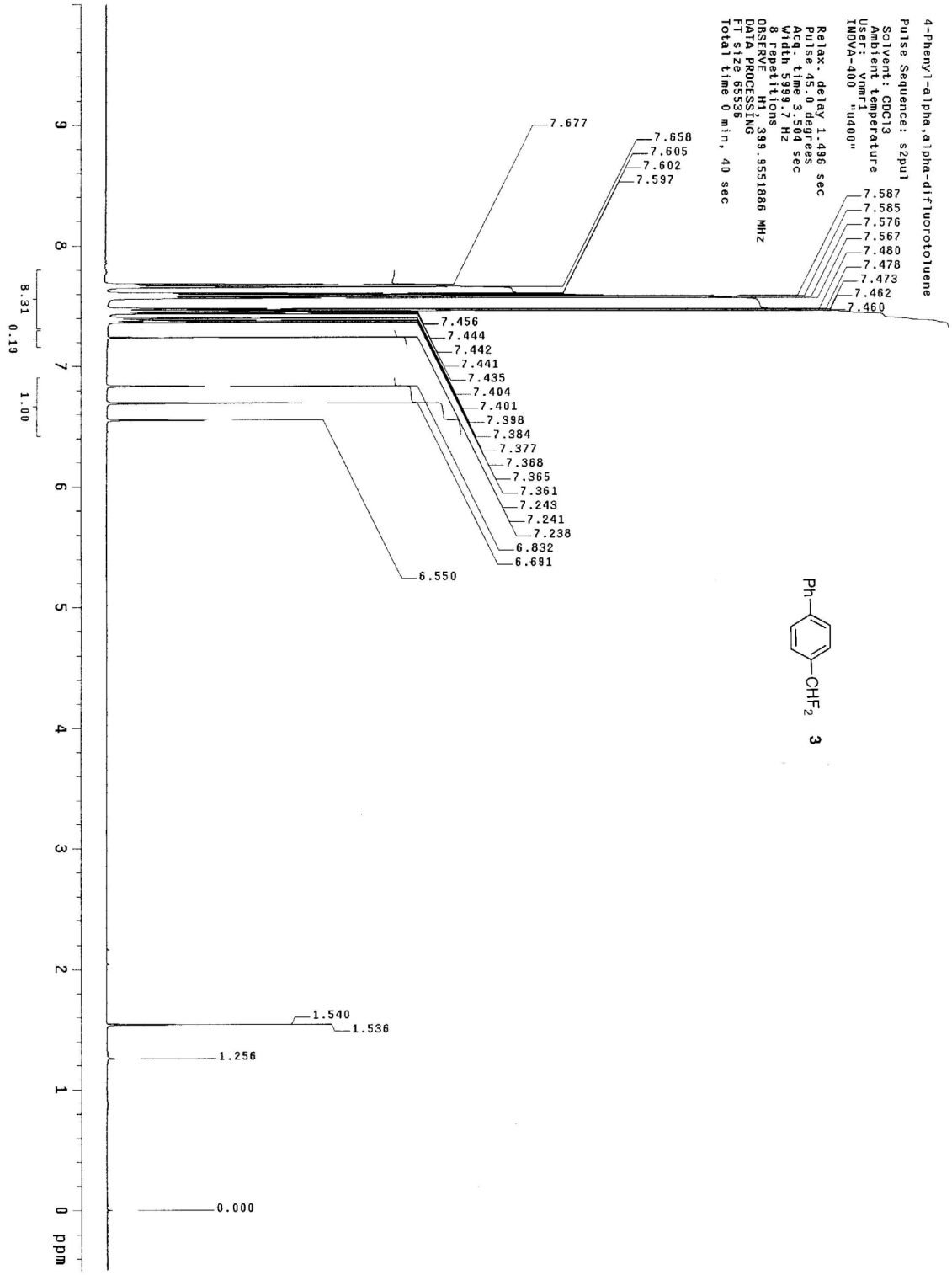
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"

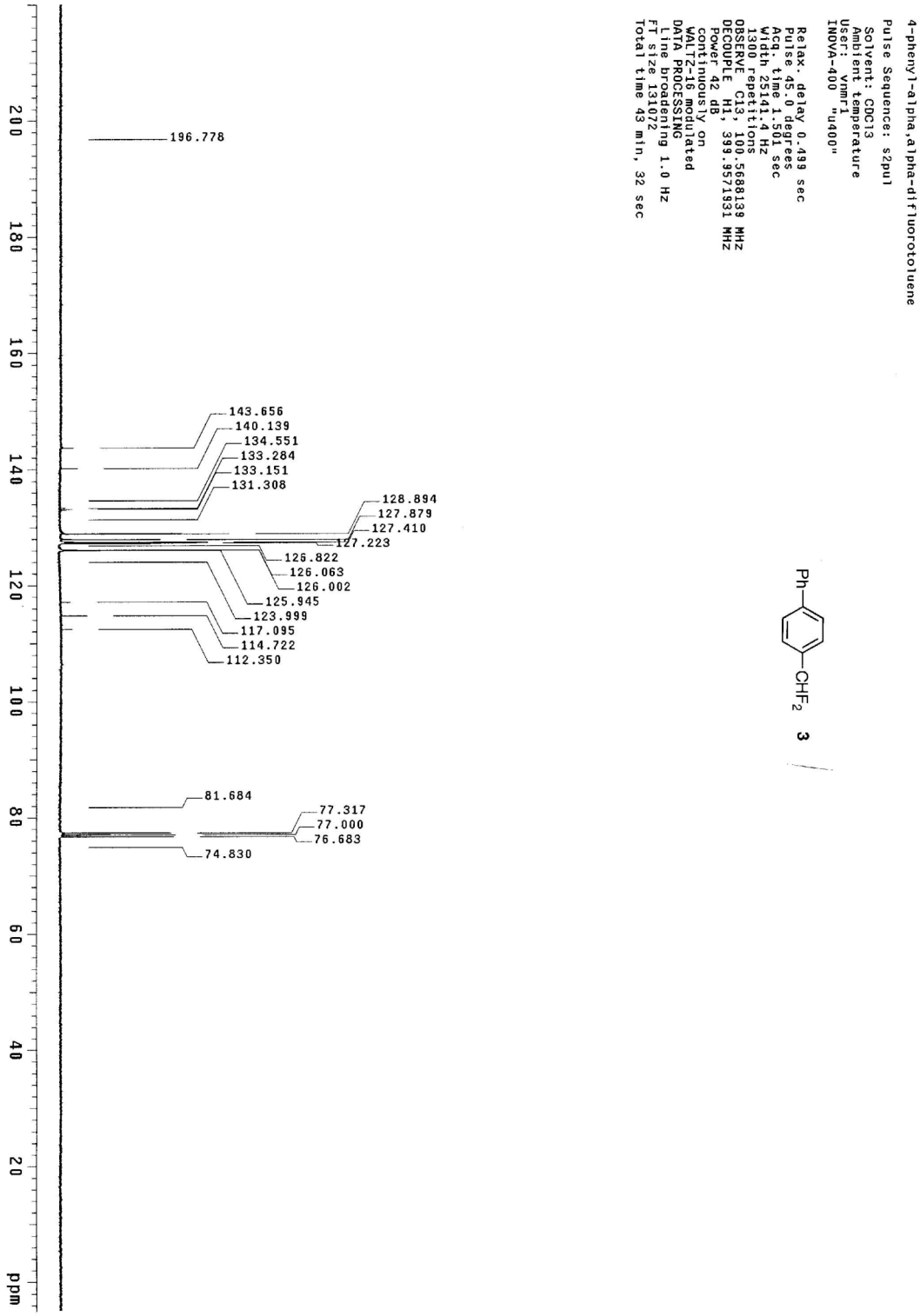
Relax. delay 9.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 100.0 kHz
54 repetitions
OBSERVE F19, 376.3343216 MHz
DATA PROCESSING
FT size 131072
Total time 9 min, 0 sec

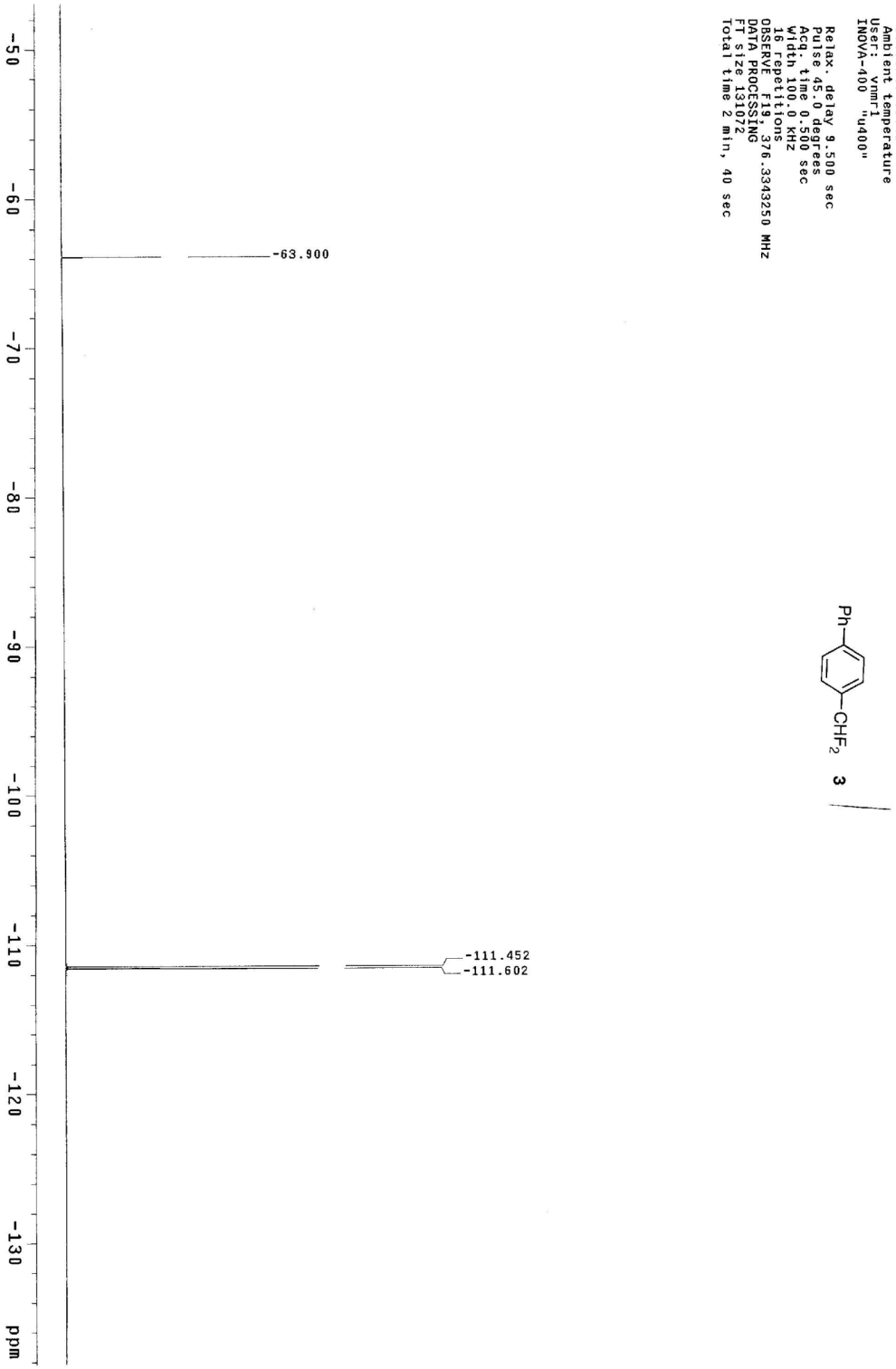


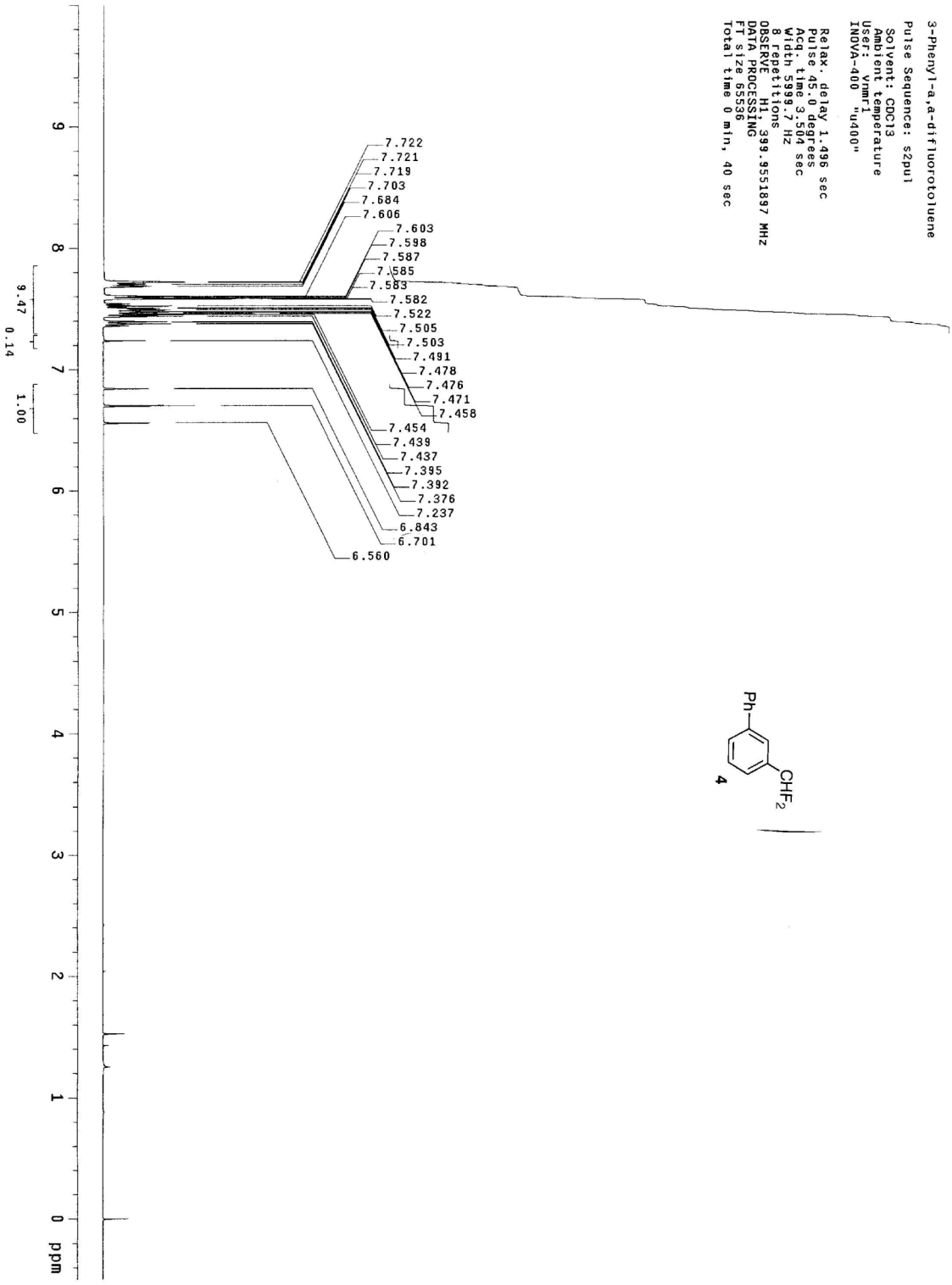
1d

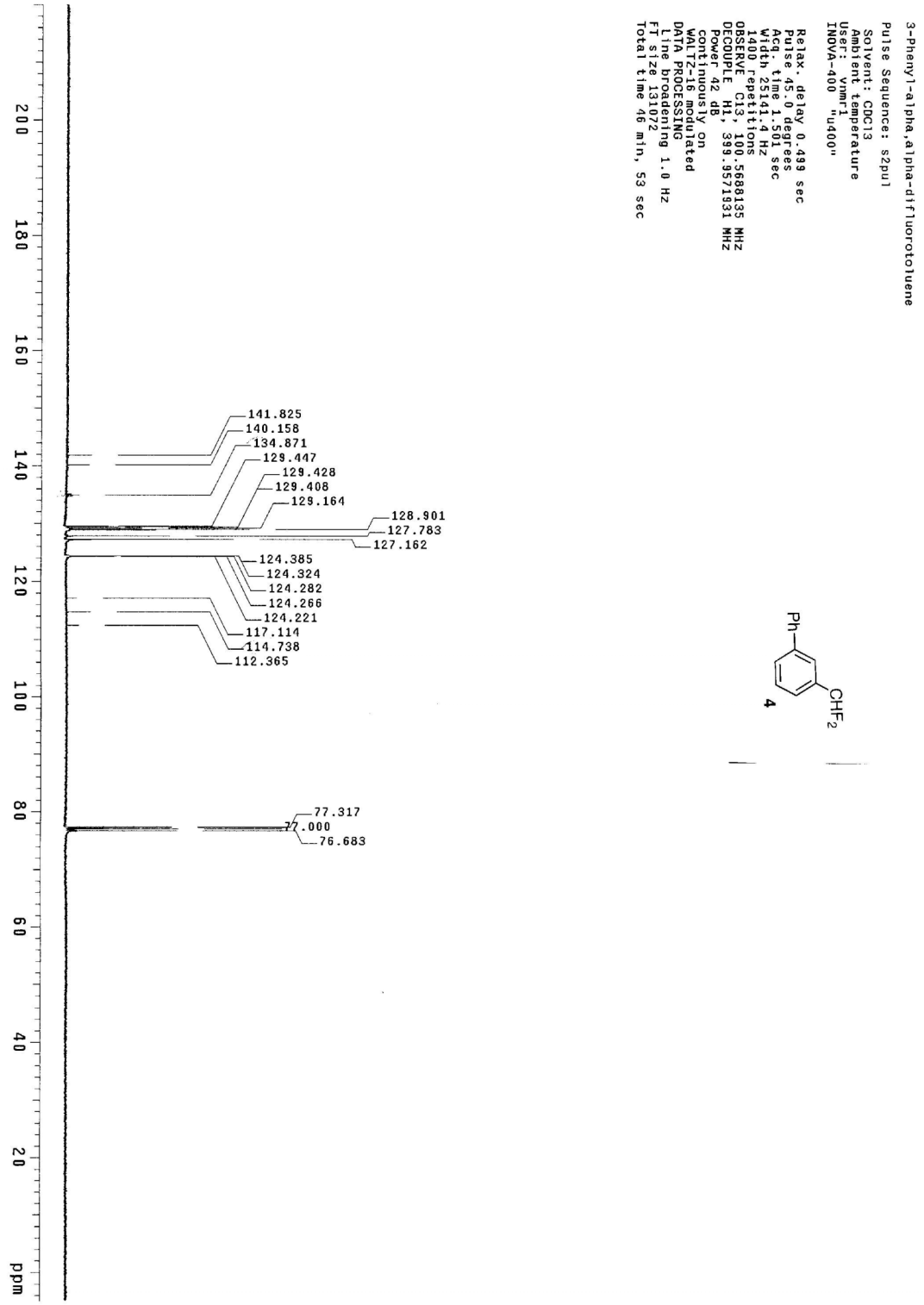


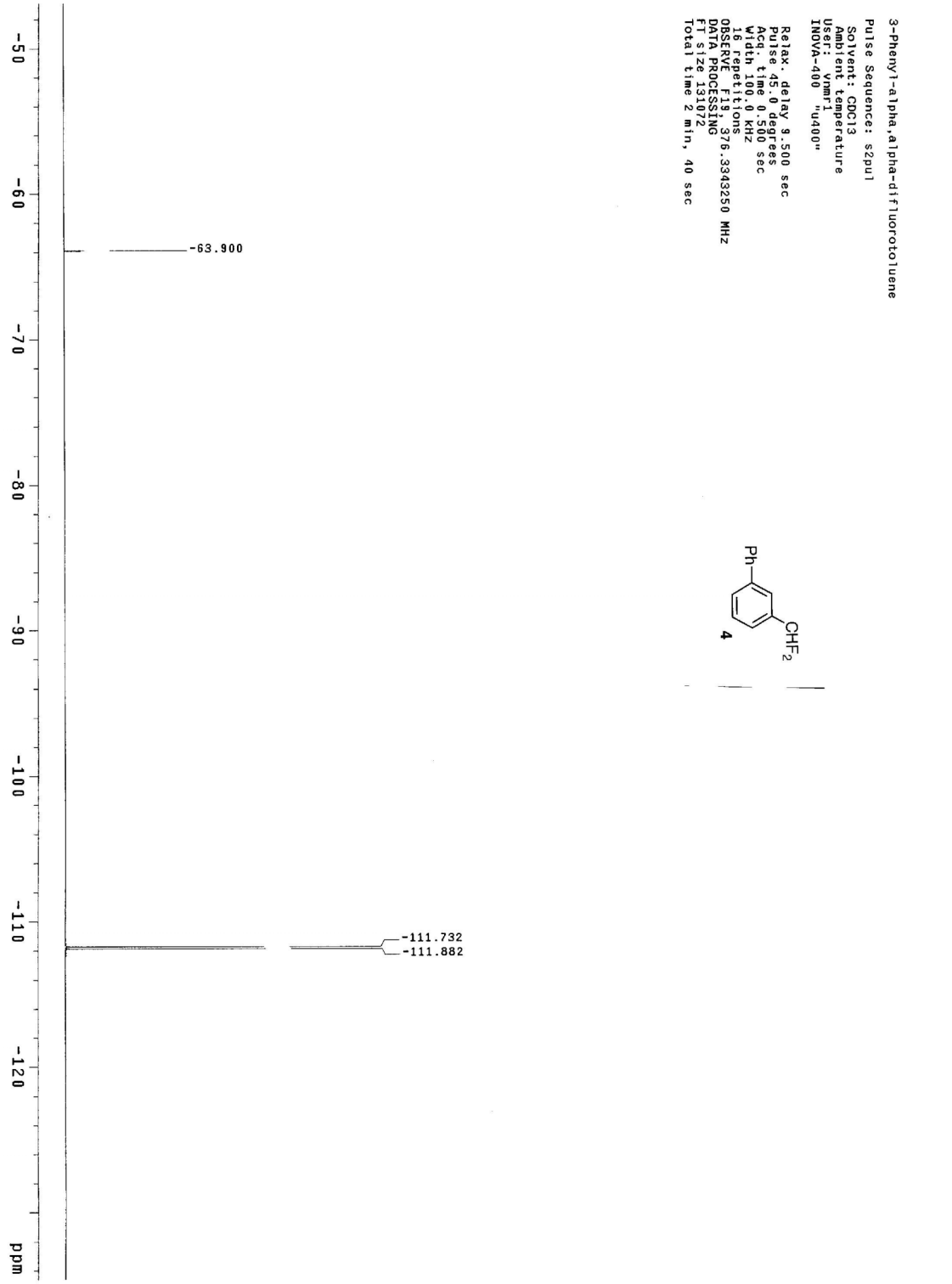

4-Phenyl-alpha,alpha-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551886 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.677
7.658
7.605
7.602
7.597
7.587
7.585
7.576
7.567
7.480
7.478
7.473
7.462
7.460
7.456
7.444
7.442
7.441
7.435
7.404
7.401
7.398
7.384
7.377
7.368
7.365
7.361
7.243
7.241
7.238
6.832
6.691
6.550
1.540
1.536
1.256
0.000
8.31
0.19
1.00
Ph
CHF2
3
ppm


4-phenyl-alpha,alpha-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
Width 25141.4 Hz
1300 repetitions
OBSERVE C13, 100.5688139 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 43 min, 32 sec
196.778
143.656
140.139
134.551
133.284
133.151
131.308
128.894
127.879
127.410
127.223
126.822
126.063
126.002
125.945
123.999
117.095
114.722
112.350
81.684
77.317
77.000
76.683
74.830
200
180
160
140
120
100
80
60
40
20
ppm
Ph
CHF2
3


4-Phenyl-alpha,alpha-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 9.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 100.0 kHz
16 repetitions
OBSERVE F19, 376.3343250 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec
Ph
CHF2
3
-63.900
-111.452
-111.602
-50
-60
-70
-80
-90
-100
-110
-120
-130
ppm


3-Phenyl-a,a-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 1.496 sec
Pulse 45.0 degrees
Acq. time 3.504 sec
Width 5999.7 Hz
8 repetitions
OBSERVE H1, 399.9551897 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
7.722
7.721
7.719
7.703
7.684
7.606
7.603
7.598
7.587
7.585
7.583
7.582
7.522
7.505
7.503
7.491
7.478
7.476
7.471
7.458
7.454
7.439
7.437
7.395
7.392
7.376
7.237
6.843
6.701
6.560
9.47
0.14
1.00
Ph
CHF2
4
9
8
7
6
5
4
3
2
1
0
ppm


3-Phenyl-alpha,alpha-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 0.499 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
width 25141.4 Hz
1400 repetitions
OBSERVE C13, 100.5688135 MHz
DECOUPLE H1, 399.9571931 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 46 min, 53 sec
141.825
140.158
134.871
129.447
129.428
129.408
129.164
128.901
127.783
127.162
124.385
124.324
124.282
124.266
124.221
117.114
114.738
112.365
77.317
77.000
76.683
200
180
160
140
120
100
80
60
40
20
ppm
Ph
CHF2
4


3-Phenyl-alpha,alpha-difluorotoluene
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: vnmr1
INOVA-400 "u400"
Relax. delay 9.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 100.0 kHz
16 repetitions
OBSERVE F19, 376.3343250 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec
Ph
CHF2
4
-63.900
-111.732
-111.882
-50
-60
-70
-80
-90
-100
-110
-120
ppm