Supplementary data

Microwave-Assisted Synthesis of Allylic Amines: Considerable Rate Acceleration in the Hydrozirconation-Transmetalation-Aldimine Addition Sequence

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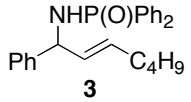
General:

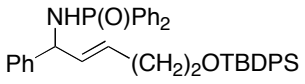
All moisture-sensitive reactions were performed under an atmosphere of N_2 and glassware was flame dried under vacuum prior to use. Toluene was purified by filtration through activated alumina. Me_2Zn was purchased from the Aldrich Chemical Company and Cp_2ZrHCl was prepared according to a modification of a literature protocol (Buchwald, S. L.; LaMaire, S. J.; Nielsen, R. B. *Org. Synth.* **1993**, *71*, 77). Unless otherwise stated, solvents or reagents were used as received. Analytical thin layer chromatography (TLC) was performed on pre-coated silica gel 60 F-254 plates (particle

size 0.040-0.055 mm, 230-400 mesh) and visualization was accomplished with a 254 nm UV light and/or by staining with Vaughn's reagent (4.8 g of $(NH_4)_6Mo_7O_{24}•4H_2O$ and 0.20 g of $Ce(SO_4)_2$ in 100 mL of 3.5 N H_2SO_4). NMR spectra were recorded in $CDCl_3$ at 300 MHz/75 MHz (^{1}H NMR/^{13}C NMR) at 21 °C unless stated otherwise. Chemical shifts (□) are reported as follows: chemical shift, multiplicity (s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, b=broad), coupling constants, and integration. Mass spectra were obtained on a double focusing instrument. Microwave reactions were run using a CEM Discover microwave reactor.



3

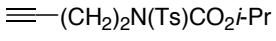
(*E*)-*N*-(1-Phenylhept-2-enyl)-*P*,*P*-diphenylphosphinamide (3).[1a] **General Protocol A.** To a suspension of Cp_2ZrHCl (0.13 g, 0.49 mmol) in dry toluene (2.0 mL) was added **1** (60 □L, 0.52 mmol). The reaction mixture was heated in the microwave reactor (60 °C, 150 W) for 5 min, cooled to –78 °C, treated with Me_2Zn (0.16 mL, 0.33 mmol, 2.0 M in toluene), and warmed to 0 °C. After addition of **2** (0.10 g, 0.33 mmol), the mixture was heated in the microwave reactor (100 °C, 150 W) for 5 min, cooled to 0 °C, quenched with MeOH (0.25-0.50 mL), diluted with EtOAc, filtered through SiO_2 and concentrated. The residue was purified by chromatography on deactivated SiO_2 (3:7, hexanes/EtOAc containing 1% v/v Et_3N) to afford **3** (93 mg, 73%) as a colorless solid: ^{1}H NMR □ 7.97-7.90 (m, 2 H), 7.87-7.80 (m, 2 H), 7.53-7.21 (m, 11 H), 5.66 (ddt, *J* = 15.3, 6.2, 1.3 Hz, 1 H), 5.51 (dtd, *J* = 15.3, 6.4, 0.9 Hz, 1 H), 4.81 (td, *J* = 9.4, 6.4 Hz, 1 H), 3.25 (dd, *J* = 9.2, 6.1 Hz, 1 H), 1.99 (q, *J* = 6.2 Hz, 2 H), 1.31-1.26 (m, 4 H), 0.88 (t, *J* = 6.9 Hz, 3 H).



10

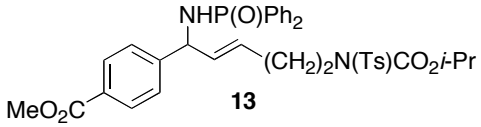
(*E*)-*N*-{5-[(tert-Butyldiphenylsilyl)oxy]-1-phenylpent-2-enyl}-*P*,*P*-diphenylphosphinamide (10).[1b] According to the General Protocol A, Cp_2ZrHCl (0.13 g, 0.49 mmol), **9** (0.16 g, 0.52 mmol), Me_2Zn (0.16 mL, 0.33 mmol, 2.0 M in toluene) and **2** (0.10 g, 0.33 mmol) afforded **10** (0.12 g, 62%) as a colorless foam: ^{1}H NMR □ 7.94-7.79 (m, 4 H), 7.63 (d, *J* = 6.4 Hz, 4 H), 7.45-7.23 (m, 17 H), 5.74 (dd, *J* = 15.4, 6.0 Hz, 1 H),

5.52 (dt, *J* = 15.5, 6.5 Hz, 1 H), 4.85-4.76 (m, 1 H), 3.63 (t, *J* = 6.5 Hz, 2 H), 3.25-3.20 (m, 1 H), 2.26 (q, *J* = 6.5 Hz, 2 H), 1.01 (s, 9 H).



11

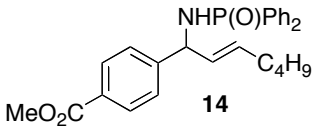
***O*-Isopropyl-*N*-but-3-ynyl-*N*-(4-methylphenyl)sulfonylcarbamate (11).** A solution of 4-methylbenzenesulfonyl isocyanate (2.6 g, 10 mmol) in dry THF (20 mL) was treated under nitrogen with Ph_3P (2.6 g, 10 mmol) and 3-butyn-1-ol (0.51 mL, 6.7 mmol), cooled to 0 °C, and treated dropwise with DIAD (1.6 mL, 10 mmol). The reaction mixture was warmed to r.t., stirred for 2 h, concentrated and purified by chromatography on SiO_2 (9:1, hexanes/EtOAc) to afford **11** (1.3 g, 65%) as a colorless solid: Mp 71.6-73.4 °C (hexanes/EtOAc); IR (KBr) 3308, 2988, 2934, 1731, 1597, 1357, 1170, 1089, 1102, 899, 811, 735 cm^{-1}; ^1H NMR □ 7.83 (d, *J* = 8.3 Hz, 2 H), 7.30 (d, *J* = 8.4 Hz, 2 H), 4.89 (septet, *J* = 6.3 Hz, 1 H), 4.01 (app t, *J* = 7.3 Hz, 2 H), 2.65 (td, *J* = 7.6, 2.7 Hz, 2 H), 2.43 (s, 3 H), 2.02 (t, *J* = 2.6 Hz, 1 H), 1.15 (d, *J* = 6.3 Hz, 6 H); ^{13}C NMR □ 151.93, 144.81, 137.21, 129.56, 129.30, 128.63, 80.60, 72.26, 70.79, 45.49, 21.85, 20.25; MS (EI) *m/z* (intensity) 309 (M^+, 27), 287 (12), 270 (34), 245 (28), 206 (37), 184 (100), 155 (74), 91 (46); HRMS (EI) *m/z* calculated for $C_{15}H_{19}NO_4S$ 309.1035, found 309.1041.



13

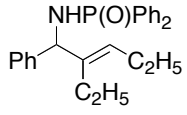
(*E*)-Methyl 4-{[1-diphenylphosphinoylamino-5-(isopropoxycarbonyltosylamino)]-pent-2-enyl}benzoate (13). General Protocol B. To a suspension of Cp_2ZrHCl (0.10 g, 0.40 mmol) in dry toluene (1.6 mL) was added **11** (0.14 g, 0.46 mmol). The reaction mixture was heated in the microwave reactor in the flash heat mode (80 °C, 75 W) for 40 sec, cooled to -78 °C, treated with Me_2Zn (0.19 mL, 0.38 mmol, 2.0 M in toluene), and warmed to 0 °C. After addition of **12** (94 mg, 0.26 mmol), the solution was heated in the microwave reactor in the flash heat mode (100 °C, 150 W) for 40 sec, cooled to r.t, quenched with 1.0 M NH_4Cl, and diluted with EtOAc and 1.0 M $NaHCO_3$. The aqueous layer was extracted with of EtOAc (2x) and the combined organic layers were washed with water (2x) and brine (2x), dried ($MgSO_4$) and purified by chromatography on deactivated SiO_2 (1:9, hexanes/EtOAc containing 1% v/v Et_3N) to afford **13** (0.14 g, 80%)

as a colorless solid: Mp 62.8-64.8 °C (hexanes/EtOAc); IR (KBr) 3167, 3057, 2982, 2952, 1723, 1609, 1437, 1362, 1280, 1186, 1167, 1107, 1122, 1087 cm^{-1}; ^1H NMR δ 7.97-7.89 (m, 4 H), 7.83-7.74 (m, 4 H), 7.51-7.26 (m, 10 H), 5.82 (dd, *J* = 15.4, 5.5 Hz, 1 H), 5.51 (td, *J* = 14.2, 6.4 Hz, 1 H), 4.85-4.79 (m, 2 H), 3.91 (s, 3 H), 3.84 (t, *J* = 7.0 Hz, 2 H), 3.63 (bt, *J* = 7.1 Hz, 1 H), 2.42 (s, 3 H), 1.27 (bs, 1 H), 1.09 (d, *J* = 6.2 Hz, 6 H), 0.97-0.86 (m, 1 H) ; ^{13}C NMR δ 167.18, 151.96, 147.85, 147.79, 144.67, 137.43, 135.00, 134.94, 132.60, 132.51, 132.38, 132.18, 132.10, 131.73, 130.11, 129.53, 129.39, 128.87, 128.76, 128.70, 128.58, 128.48, 127.538, 127.54, 72.05, 56.53, 52.70, 46.67, 33.09, 21.85; MS (EI) *m/z* (intensity) 674 (M^+, 45), 473 (48), 416 (28), 404 (52), 216 (87), 201 (100), 155 (40), 91 (65); HRMS (EI) *m/z* calculated for $C_{36}H_{39}N_2O_7PS$ 674.2216, found 674.2224.



14

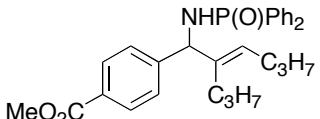
(*E*)-Methyl 4-{1-[(diphenylphosphinoyl)amino]hept-2-enyl}benzoate (14).[1b] According to the General Protocol B, Cp_2ZrHCl (0.10 g, 0.40 mmol), **1** (51 μL, 0.44 mmol), Me_2Zn (0.19 mL, 0.38 mmol, 2.0 M in toluene) and **12** (94 mg, 0.26 mmol) afforded **14** (0.11 g, 95%) as a colourless solid: ^1H NMR δ 7.99-7.81 (m, 4 H), 7.48-7.36 (m, 8 H), 5.65 (dd, *J* = 15.3, 6.1 Hz, 1 H), 5.54-5.44 (m, 1 H), 4.84 (td, *J* = 9.7, 6.1 Hz, 1 H), 3.92 (s, 3 H), 3.28 (td, *J* = 9.7, 6.1 Hz, 1 H), 1.98 (q, *J* = 6.6 Hz, 2 H), 1.31-1.24 (m, 4 H), 0.87 (t, *J* = 6.9 Hz, 3 H).



16

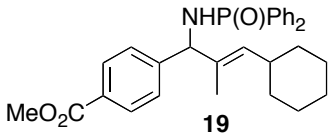
(*E*)-*N*-(2-Ethyl-1-phenylpent-2-enyl)-*P*,*P*-diphenylphosphinamide (16).[1b] **General Protocol C**: To a suspension of Cp_2ZrHCl (0.10 g, 0.40 mmol) in dry toluene (2.0 mL) was added **15** (50 μL, 0.44 mmol). The reaction mixture was heated in the microwave reactor in the flash heat mode (100 °C, 100 W) for 40 sec, cooled to -78 °C, treated with Me_2Zn (0.19 mL, 0.38 mmol, 2.0 M in toluene), and warmed to 0 °C. After addition of **2** (79 mg, 0.26 mmol), the solution was heated in the microwave reactor in the flash heat

mode (100 °C, 150 W) for 40 sec, cooled to r.t., quenched with 1.0 M NH_4Cl, vigorously stirred, and diluted with EtOAc and 1.0 M $NaHCO_3$. The aqueous layer was extracted with EtOAc (2x), and the combined organic layers were washed with water (2x) and brine (2x), dried ($MgSO_4$) and concentrated. The residue was purified by chromatography on deactivated SiO_2 (1:9, hexanes/EtOAc containing 1% v/v Et_3N) to afford **16** (75 mg, 77%) as a colorless solid: ^{1}H NMR 7.95-7.85 (m, 4 H), 7.45-7.29 (m, 11 H), 5.53 (t, *J* = 14.3 Hz, 1 H), 4.73 (t, *J* = 10.7 Hz, 1 H), 3.24 (td, *J* = 10.0, 6.1 Hz, 1 H), 2.16-2.04 (m, 3 H), 1.78-1.68 (m, 1 H), 1.63 (s, 1 H), 1.03 (t, *J* = 7.5 Hz, 3 H), 0.70 (t, *J* = 7.6 Hz, 3 H).



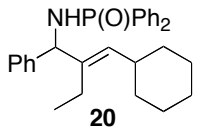
18

(*E*)-Methyl 4-(1-diphenylphosphinoylamino-2-propylhex-2-enyl)benzoate (18). According to the General Protocol C, Cp_2ZrHCl (0.21 g, 0.80 mmol), **17** (0.13 mL, 0.87 mmol), Me_2Zn (0.40 mL, 0.80 mmol, 2.0 M in toluene) and **12** (0.19 g, 0.52 mmol) afforded **18** (0.18 g, 74%) as a colorless solid: Mp 91.9-92.3 °C (hexanes/EtOAc); IR (KBr) 3181, 2957, 2930, 2869, 1722, 1610, 1436, 1281, 1185, 1107, 1123, 1019 cm-1; ^{1}H NMR 7.97-7.77 (m, 6 H), 7.50-7.35 (m, 8 H), 5.50 (t, *J* = 7.2 Hz, 1 H), 4.76 (t, *J* = 10.6 Hz, 1 H), 3.90 (s, 3 H), 3.28 (dd, *J* = 10.3, 6.3 Hz, 1 H), 2.14-2.02 (m, 3 H), 1.74-1.71 (dq, *J* = 14.5, 7.3 Hz, 1 H), 1.45-1.37 (m, 2 H), 1.17-1.09 (m, 2 H), 0.93 (t, *J* = 7.3 Hz, 3 H), 0.74 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR 167.21, 148.28, 140.24, 140.18, 133.71, 133.61, 132.72, 132.59, 132.41, 132.28, 132.16, 131.89, 130.02, 129.29, 128.77, 128.61, 128.78, 59.51, 52.29, 31.40, 30.15, 23.23, 22.34, 14.46, 14.23; MS (EI) *m/z* (intensity) 475 (M^+, 59), 446 (16), 432 (14), 364 (63), 274 (45), 218 (53), 201 (100), 77 (48); HRMS (EI) *m/z* calculated for $C_{29}H_{34}NO_3P$ 475.2276, found 475.2283.



19

(*E*)-Methyl 4-(1-diphenylphosphinoylamino-3-cyclohexyl-2-methylallyl)benzoate (19). General Protocol D. To a suspension of Cp_2ZrHCl (0.14 g, 0.55 mmol) in dry toluene (1.5 mL) was added a freshly prepared solution of **5** (0.25 mL, 0.28 mmol, 1.1 M in toluene). The reaction mixture was heated in the microwave reactor (60 °C, 150 W) for 30 min, treated with **5** (0.25 mL, 0.28 mmol, 1.1 M in toluene), heated in the microwave reactor (60 °C) for 15 min, cooled to –78 °C, treated with Me_2Zn (0.14 mL, 0.28 mmol, 2.0 M in toluene), and warmed to 0 °C. After addition of **12** (0.10 g, 0.28 mmol), the solution was heated in the microwave reactor (100 °C, 150 W) for 5 min, cooled to 0 °C, quenched with MeOH (0.25-0.50 mL), diluted with EtOAc, filtered through SiO_2 and concentrated. The residue was purified by chromatography on deactivated SiO_2 (3:7, hexanes/EtOAc containing 1% v/v Et_3N) to afford **19** (81 mg, 60%) as a colorless foam: Mp 147.0-148.6 °C (hexanes/EtOAc); IR (KBr) 3165, 3057, 2926, 2849, 1721, 1609, 1438, 1273, 1199, 1183, 1108 cm^{-1}; ^1H NMR 7.96-7.87 (m, 4 H), 7.85-7.77 (m, 2 H), 7.50-7.31 (m, 8 H), 5.24 (d, J = 9.1 Hz, 1 H), 4.71 (t, J = 10.7 Hz, 1 H), 3.88 (s, 3 H), 3.43 (dd, J = 10.5, 6.8 Hz, 1 H), 2.27-2.14 (m, 1 H), 1.73-1.62 (m, 5 H), 1.51 (d, J = 1.0 Hz, 3 H), 1.35-1.19 (m, 3 H), 1.15-0.98 (m, 2 H); ^{13}C NMR 166.83, 147.46, 147.40, 134.47, 133.38, 132.98, 132.61, 132.55, 132.47, 132.34, 131.94, 131.82, 131.68, 131.25, 129.61, 128.76, 128.42, 128.38, 128.26, 128.21, 127.02, 61.23, 51.95, 36.75, 32.83, 32.74, 25.91, 25.81, 13.30; MS (EI) *m/z* (intensity) 487 (M^+, 27), 404 (50), 364 (22), 286 (100), 218 (81), 201 (100); HRMS (EI) *m/z* calculated for $C_{30}H_{34}NO_3P$ 487.2276, found 487.2266.



20

(*E*)-*N*-(3-Cyclohexyl-2-ethyl-1-phenylallyl)-*P*,*P*-diphenylphosphinamide (20). According to the General Protocol D, Cp_2ZrHCl (0.17 g, 0.66 mmol), **6** (89 mg, 0.66 mmol), Me_2Zn (0.16 mL, 0.33 mmol, 2.0 M in toluene) and **2** (0.10 g, 0.33 mmol) afforded **20** (91 mg, 63%) as a colorless foam: Mp 135.5-137.2 °C (hexanes/EtOAc); IR

(KBr) 3207, 3056, 2962, 2923, 2849, 1491, 1447, 1437, 1185, 1123, 1108 cm^{-1}; ^{1}H NMR δ 7.99-7.92 (m, 2 H), 7.89-7.82 (m, 2 H), 7.51-7.33 (m, 8 H), 7.31-7.18 (m, 3 H), 5.44 (d, *J* = 9.5 Hz, 1 H), 4.72 (t, *J* = 11.0 Hz, 1 H), 3.23 (dd, *J* = 10.2, 6.1 Hz, 1 H), 2.34-2.23 (m, 1 H), 2.17-2.05 (m, 1 H), 1.74-1.62 (m, 6 H), 1.37-1.07 (m, 5 H), 0.68 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR δ 142.57, 142.52, 139.39, 139.33, 133.70, 133.19, 132.82, 132.66, 132.53, 131.99, 131.86, 131.73, 131.69, 131.65, 131.45, 128.37, 128.30, 128.22, 128.13, 127.79, 127.53, 127.04, 58.74, 36.77, 33.44, 26.01, 25.95, 22.16, 13.92; MS (EI) *m/z* (intensity) 443 (M^{+}, 23), 360 (28), 306 (28), 242 (94), 218 (78), 201 (100); HRMS (EI) *m/z* calculated for $C_{29}H_{34}NOP$ 443.2378, found 443.2387.

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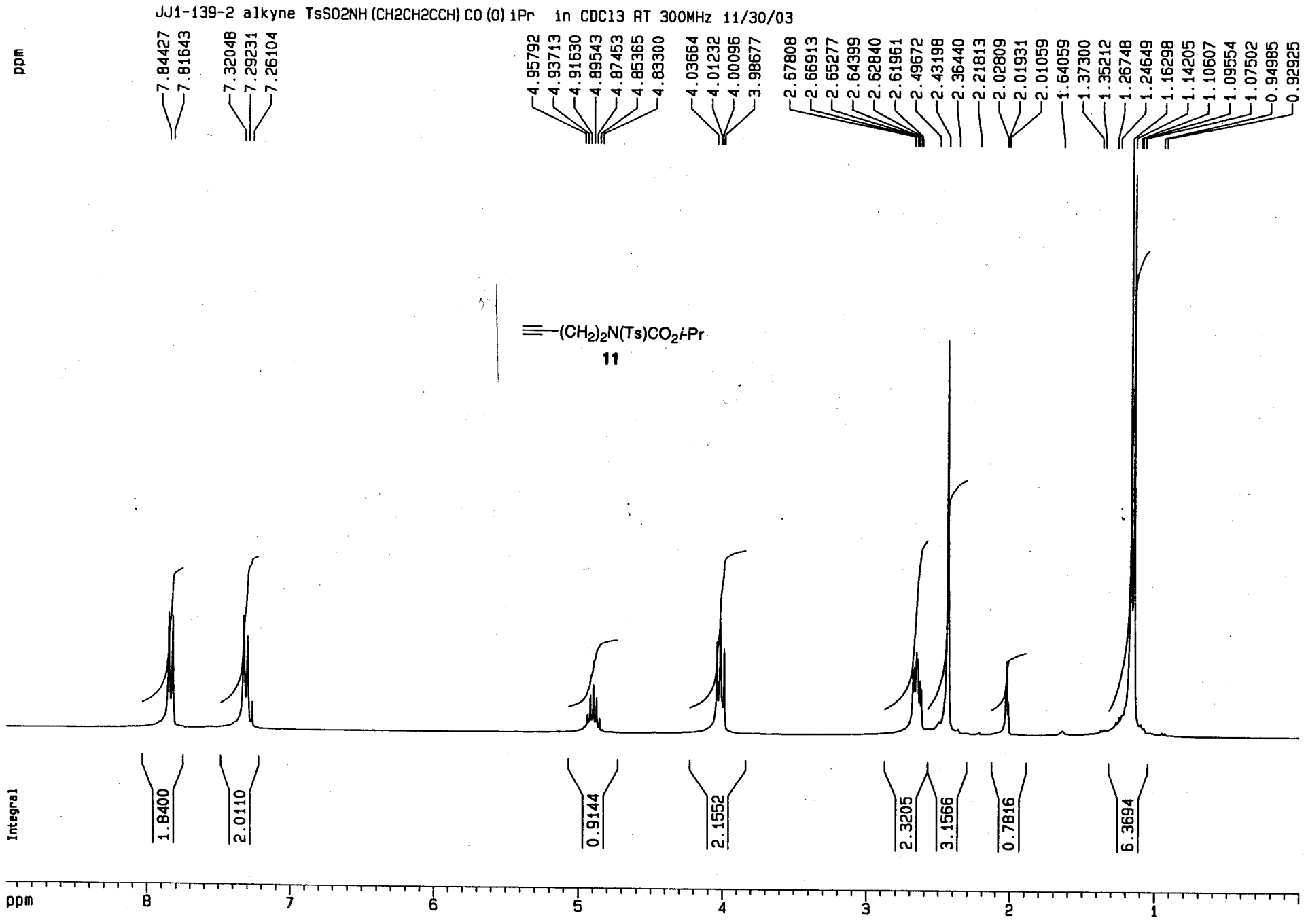
1 For full characterization data, see (a) P. Wipf, C. Kendall, and C. R. J. Stephenson, *J. Am. Chem. Soc.*, 2001, **123**, 5122. (b) P. Wipf, C. Kendall, and C. R. J. Stephenson, *J. Am. Chem. Soc.* 2003, **125**, 14694.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11
Integral
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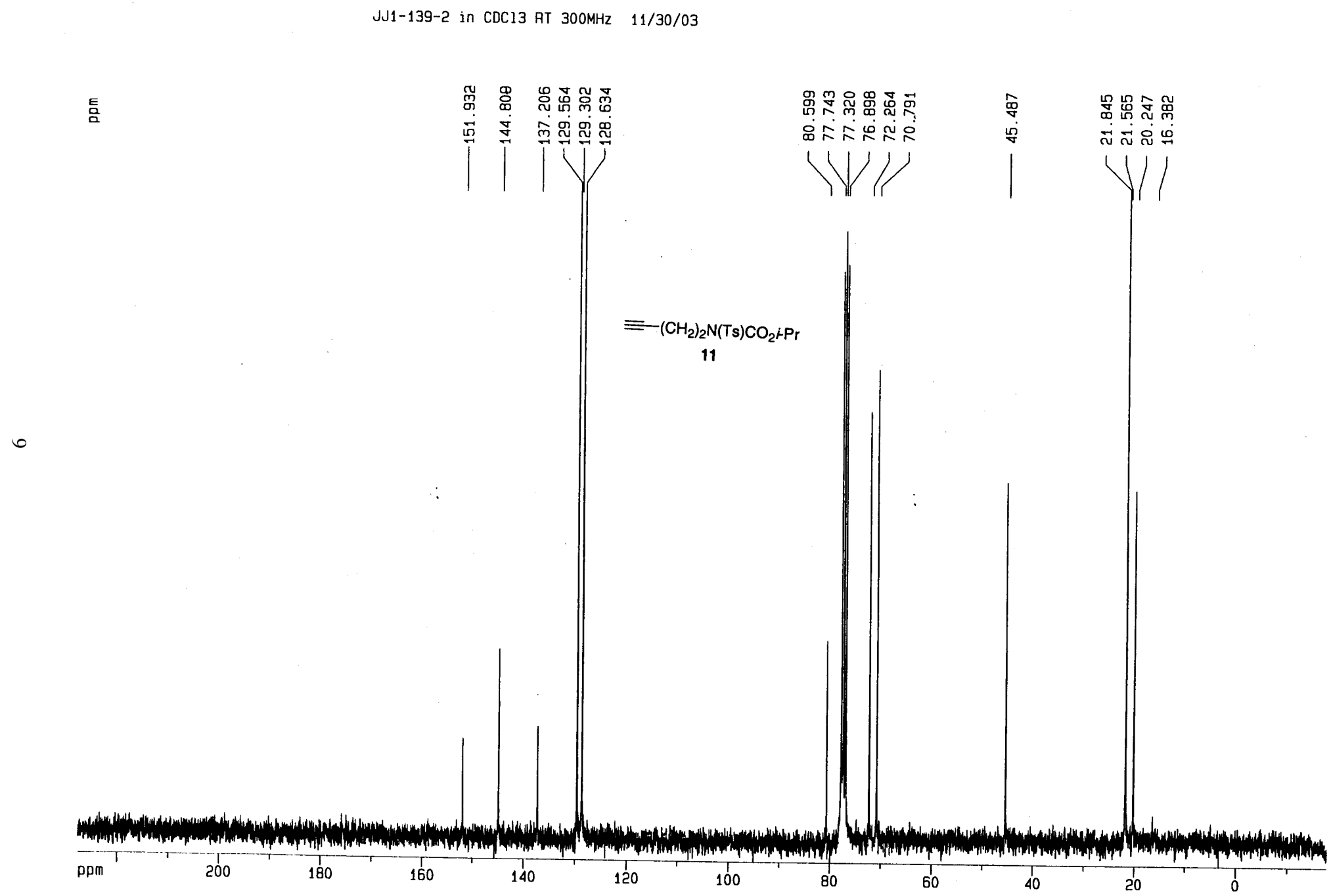
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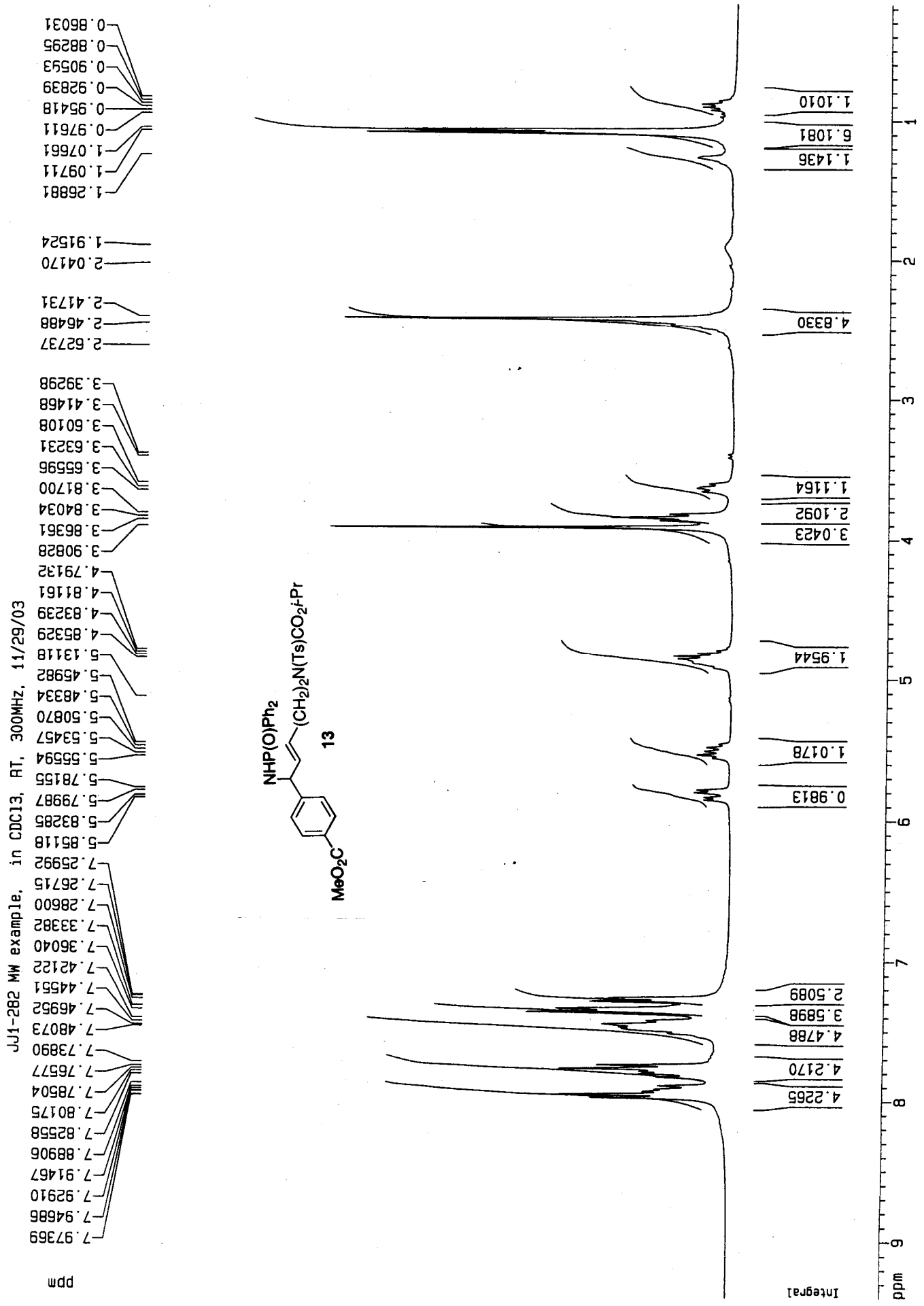
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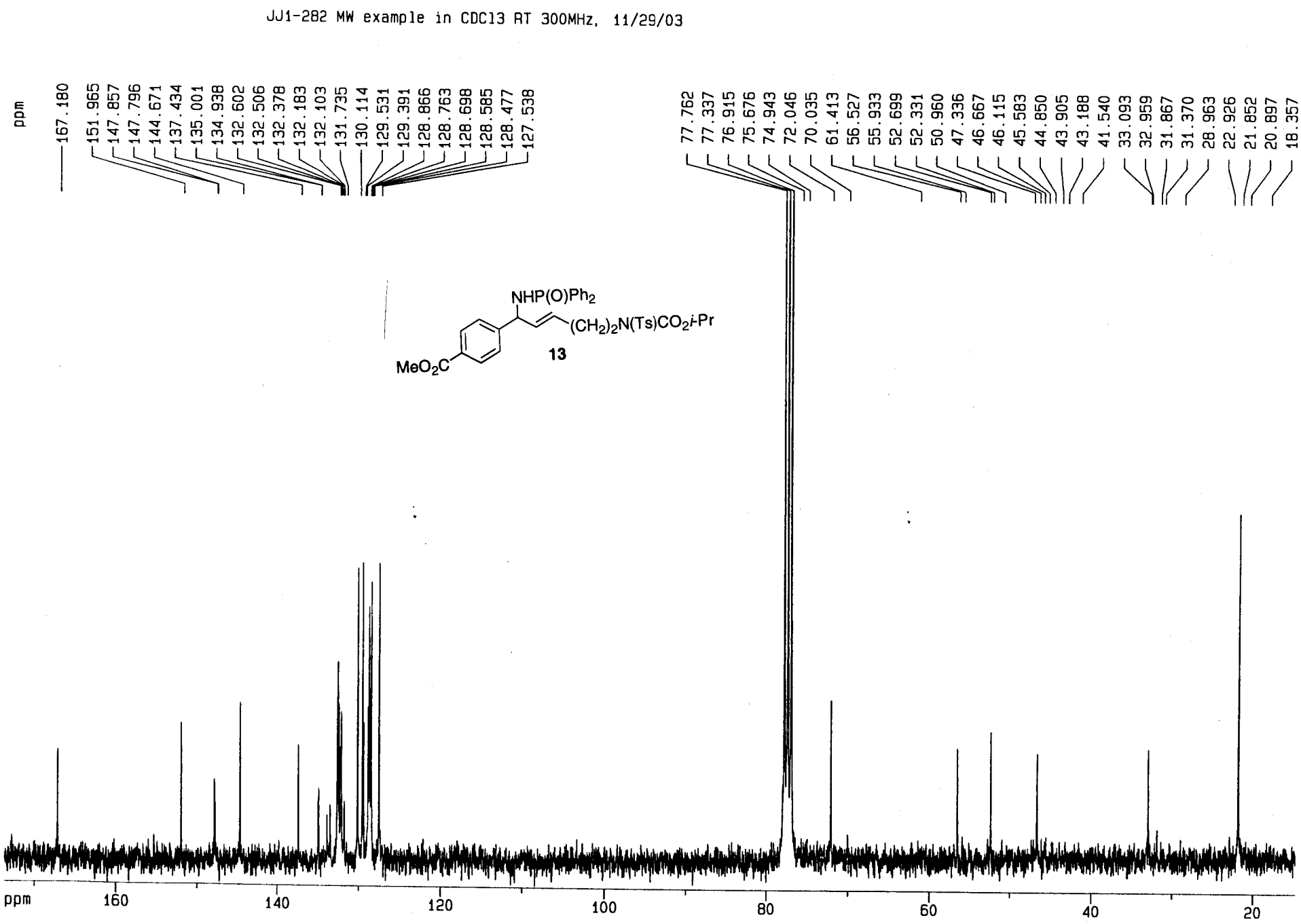
JJ1-282 MW example in CDCl3 RT 300MHz, 11/29/03
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45.583
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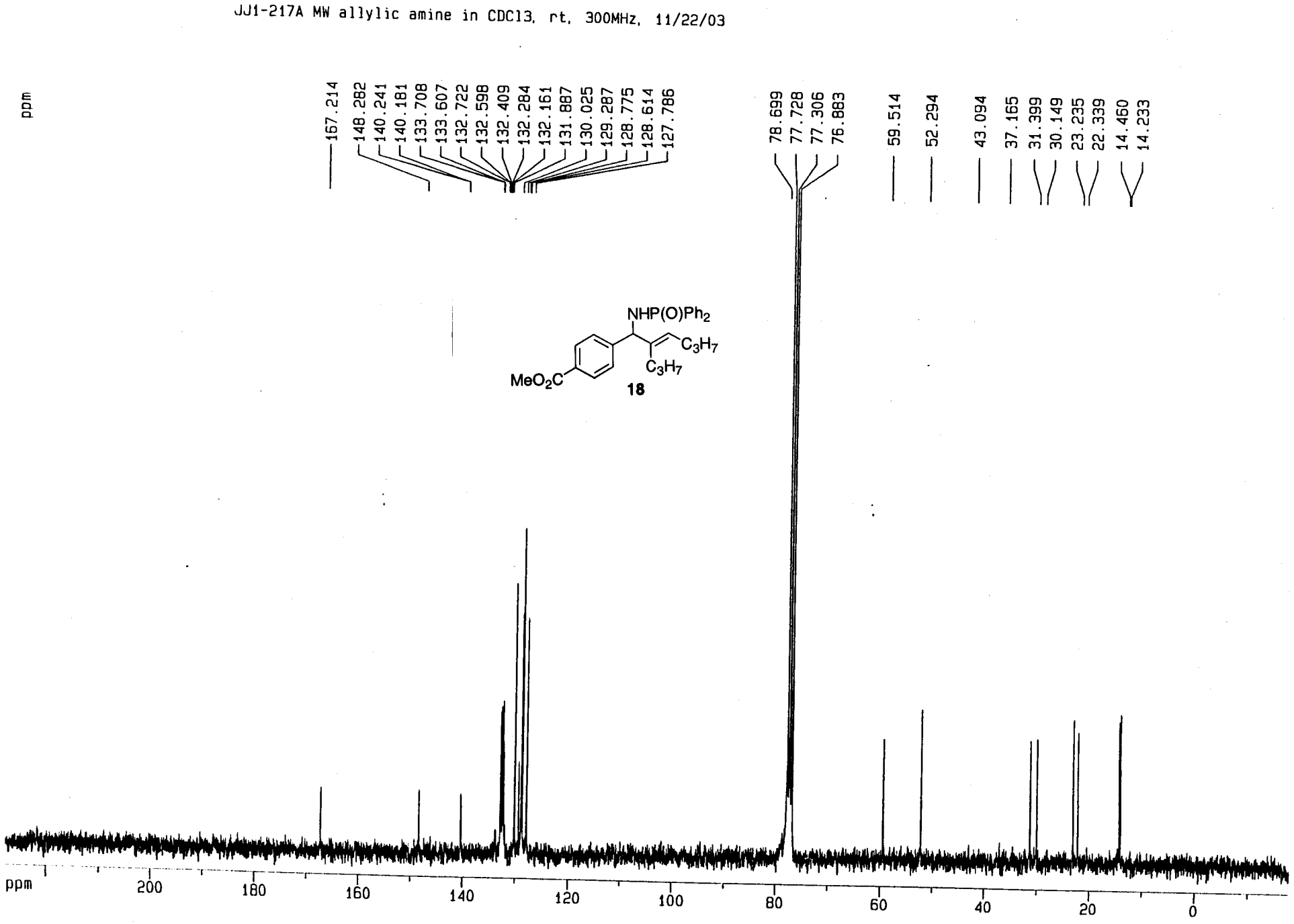
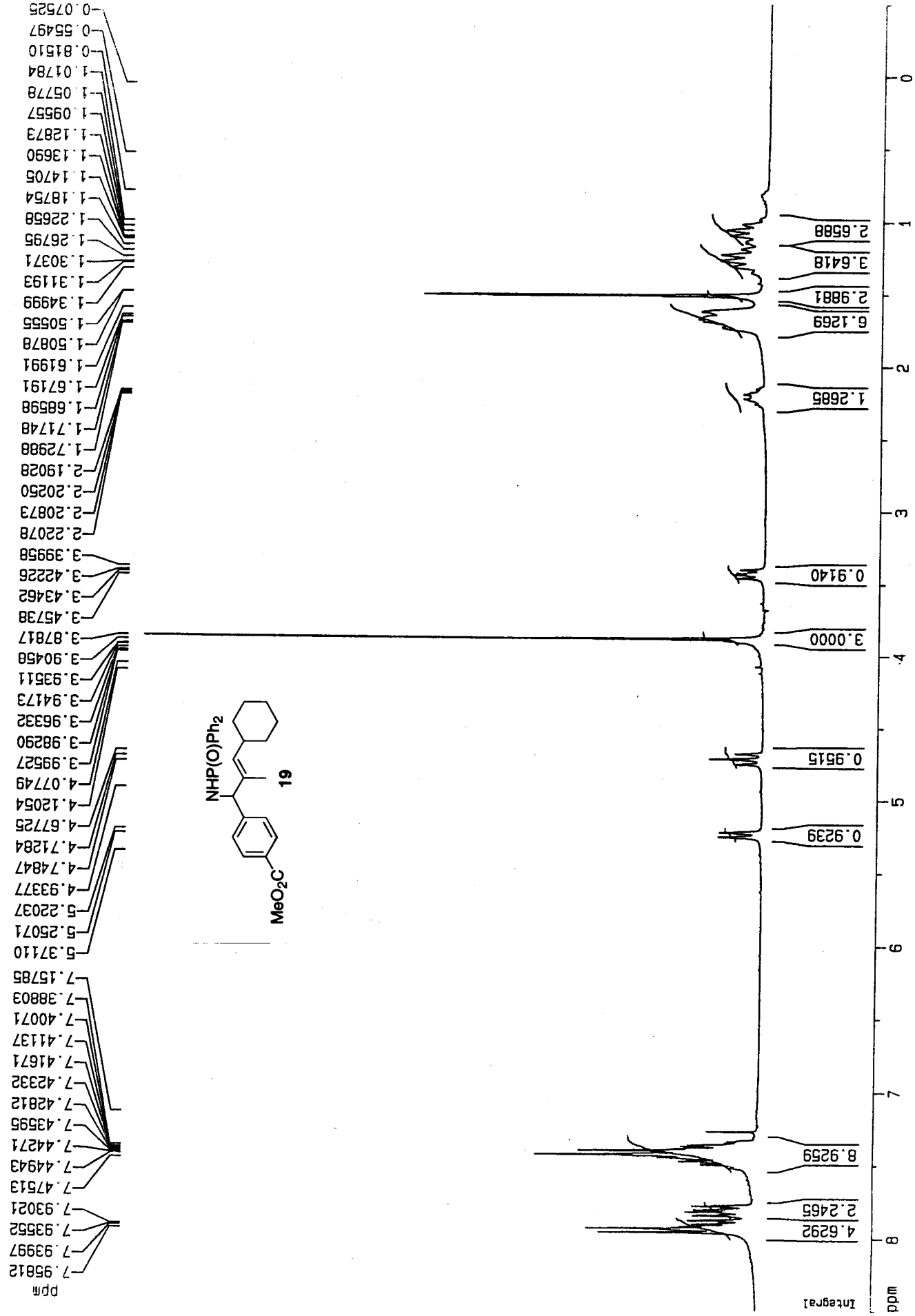
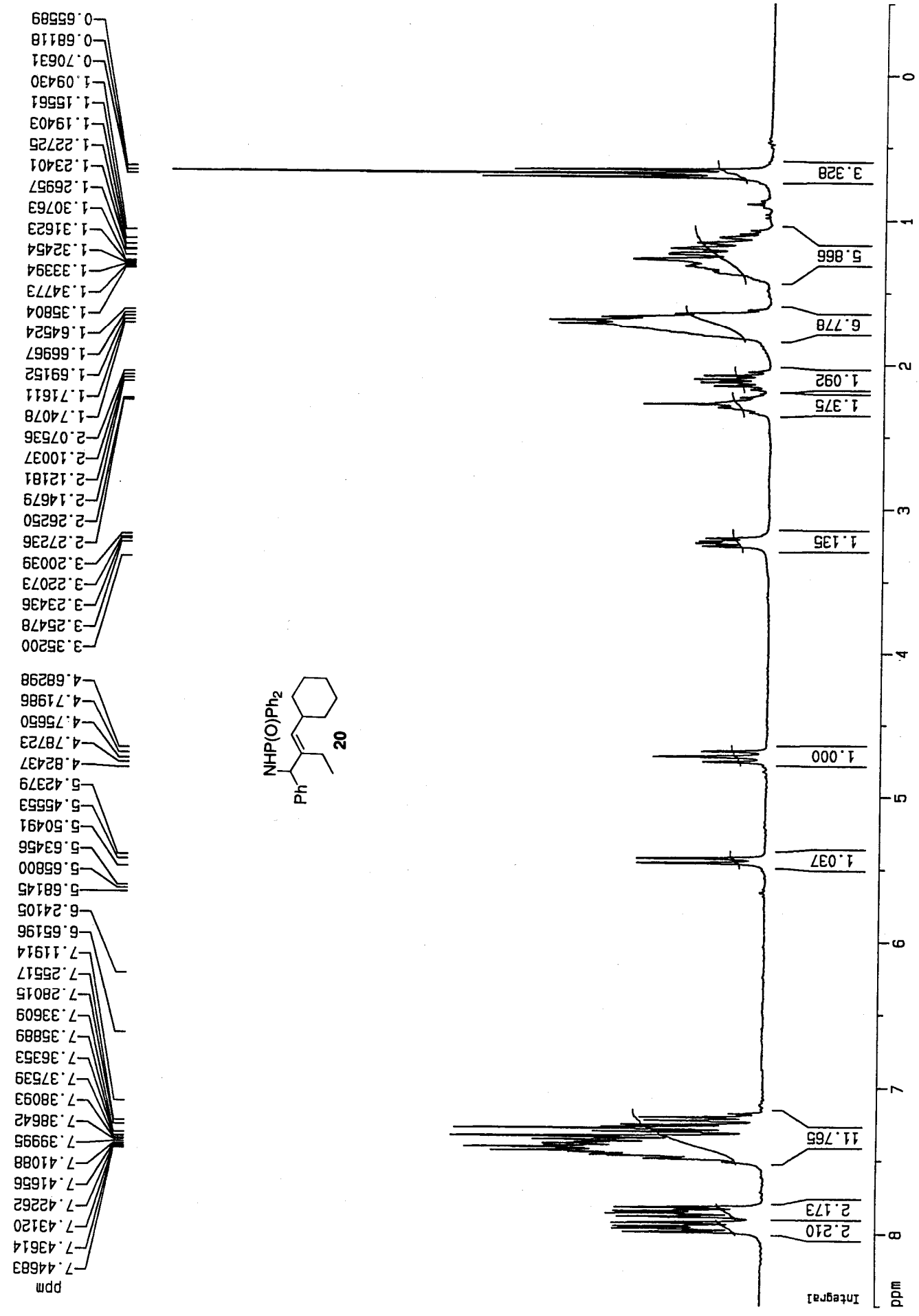
120
100
80
60
40
20

JJ1-217A MW example
ppm
7.96653
7.94455
7.93925
7.92333
7.90624
7.88323
7.81320
7.50423
7.48433
7.47982
7.45997
7.43536
7.38100
7.36922
7.35347
5.53167
5.50795
5.48403
5.28774
4.80012
4.76483
4.72903
4.14530
3.96547
3.90286
3.65545
3.30673
3.28614
3.27257
3.25206
2.09589
2.07245
2.04845
2.02415
1.73616
1.72514
1.70583
1.45206
1.42774
1.40334
1.37864
1.17185
1.14876
1.12592
1.10274
1.09457
1.07166
0.95721
0.93287
0.90840
0.76008
0.75582
0.71155
NHP(O)Ph2
C3H7
C3H7
MeO2C
18
Integral
6.7407
8.1854
0.4887
0.8898
0.9482
3.0000
0.8883
3.0369
1.4319
2.3563
2.5642
3.4395
3.2361
ppm


JJ1-217A MW allylic amine in CDCl3, rt, 300MHz, 11/22/03
ppm
167.214
148.282
140.241
140.181
133.708
133.607
132.722
132.598
132.409
132.284
132.161
131.887
130.025
129.287
128.775
128.614
127.786
78.699
77.728
77.306
76.883
59.514
52.294
43.094
37.165
31.399
30.149
23.235
22.339
14.460
14.233
NHP(O)Ph2
C3H7
C3H7
MeO2C
18
ppm
200
180
160
140
120
100
80
60
40
20
0


NHP(O)Ph2
MeO2C
19
ppm
Integral
8.9259
4.6292
2.2465
0.9515
0.9239
3.0000
0.9140
6.1269
2.9881
3.6418
2.6588
1.2685

13.304
25.807
25.915
32.744
32.832
36.753
51.951
61.227
76.579
77.003
77.427
NHP(O)Ph2
19
MeO2C
127.024
128.212
128.257
128.380
128.423
128.760
129.609
131.682
131.817
131.945
132.343
132.471
132.550
132.614
132.977
133.383
134.471
147.402
147.455
166.827
ppm
0
20
40
60
80
100
120
140
160


NHP(O)Ph2
Ph
20
Integral
ppm
3.328
5.866
6.778
1.092
1.375
1.135
1.000
1.037
11.765
2.173
2.210
0.55589
0.68118
0.70631
1.09430
1.15561
1.19403
1.22725
1.23401
1.26957
1.30763
1.31623
1.32454
1.33394
1.34773
1.35804
1.64524
1.66967
1.69152
1.71611
1.74078
2.07536
2.10037
2.12181
2.14679
2.26250
2.27236
3.20039
3.22073
3.23436
3.25478
3.35200
4.68298
4.71986
4.75650
4.78723
4.82437
5.42379
5.45553
5.50491
5.63456
5.65800
5.68145
6.24105
6.51196
7.11914
7.25517
7.28015
7.33609
7.35889
7.36353
7.37539
7.38093
7.38642
7.39955
7.41088
7.41656
7.42262
7.43120
7.43614
7.44683

13.922
22.159
25.950
26.009
33.441
36.768
58.736
76.578
77.002
77.425
127.045
127.533
128.127
128.221
128.295
128.374
131.648
131.686
131.726
131.865
131.992
132.532
132.660
132.820
139.392
ppm
NHP(O)Ph2
Ph
20
0
20
40
60
80
100
120
140
ppm