SUPPLEMENTARY MATERIAL

Tandem Zirconocene Homologation – Aldimine Allylation

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Experimental parts. ^{1}H and ^{13}C NMR spectra.

General. All moisture-sensitive reactions were performed using syringe-septum cap techniques under an N_2 atmosphere and all glassware was dried in an oven at 140 °C for 2 h prior to use. Reactions carried out at –78 °C employed a CO_2-acetone bath. THF was distilled over sodium / benzophenone ketyl, and CH_2Cl_2, toluene and Et_3N were distilled from CaH_2. Me_2Zn was purchased from Aldrich Company. Cp_2ZrHCl,[1] imines **2**, **14**, and **16**,[2] and alkyne **8**[3] were prepared according to literature procedures.

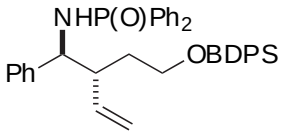
Reactions were monitored by TLC analysis (EM Science pre-coated silica gel 60 F_{254} plates, 250 μm layer thickness) and visualization was accomplished with a 254 nm UV light and by staining with a PMA solution (5 g of phosphomolybdic acid in 100 mL of 95% EtOH), *p*-anisaldehyde solution (2.5 mL of *p*-anisaldehyde, 2 mL of AcOH, and 3.5 mL of conc. H_2SO_4 in 100 mL of 95% EtOH), Vaughn's reagent (4.8 g of $(NH_4)_6Mo_7O_{24}$•4 H_2O and 0.2 g of $Ce(SO_4)_2$ in 100 mL of a 3.5 N H_2SO_4 solution) or a $KMnO_4$ solution (1.5 g of $KMnO_4$ and 1.5 g of K_2CO_3 in 100 mL of a 0.1% NaOH solution). Flash chromatography on SiO_2 or deactivated SiO_2 (1% Et_3N in mobile phase) was used to purify the crude reaction mixtures.

Melting points were determined using a Laboratory Devices Mel-Temp II. Infrared spectra were determined on a Nicolet Avatar 360 FT-IR spectrometer. ^1H and ^{13}C NMR spectra were obtained on a Bruker Avance 300 instrument in $CDCl_3$. Chemical shifts were reported in parts per million with the residual solvent peak used as an internal standard. ^1H NMR spectra were run at 300 MHz and are tabulated as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, m = multiplet), number of protons, and coupling constant(s). ^{13}C NMR spectra were run at 76 MHz using the proton-decoupled pulse sequence with a d_1 of 3 seconds, and are tabulated by chemical shift. Mass spectra were obtained on a Micromass Autospec double focusing instrument.

***N*-[(1*S**,2*R**)-2-Butyl-1-phenylbut-3-enyl]-*P*,*P*-diphenylphosphinamide (5a)** and ***N*-[(1*S**,2*S**)-2-butyl-1-phenylbut-3-enyl]-*P*,*P*-diphenylphosphinamide (5b). General Protocol A.** A suspension of 195 mg (0.756 mmol) of Cp_2ZrHCl in 2 mL of CH_2Cl_2 was treated at room temperature with 95.0 μL (0.827 mmol) of 1-hexyne. After 2 min, the yellow solution was

cooled to –78 °C, treated with 560 μL (1.12 mmol) of Me_2Zn (2.0 M solution in toluene), warmed to room temperature over a period of 5 min, treated with 60.0 μL (0.745 mmol) of CH_2I_2, stirred 2 min and treated with a solution of 76.0 mg (0.249 mmol) of imine **2** in 1 mL of CH_2Cl_2. The reaction mixture was stirred at room temperature for 12 h, quenched with saturated NH_4Cl, diluted with EtOAc and saturated $NaHCO_3$, filtered through Celite, washed with H_2O and brine, dried ($MgSO_4$), filtered through a pad of Florisil and concentrated *in vacuo*. The residue was chromatographed on deactivated SiO_2 (1:9, hexanes/EtOAc containing 1% Et_3N) to yield 71 mg (71%) of an 85:15 mixture of diastereomers. **5a** (colorless solid): mp 158-159 °C (EtOAc/hexane); IR (KBr) 3221, 3059, 2951, 2914, 2858, 1436, 1185, 1123, 1108, 1068, 919, 723, 702, 692 cm^{-1}; ^1H NMR δ 7.90-7.83 (m, 2 H), 7.73-7.65 (m, 2 H), 7.54-7.38 (m, 4 H), 7.32-7.24 (m, 5 H), 7.07-7.04 (m, 2 H), 5.43 (dt, 1 H, *J* = 17.2, 9.7 Hz), 5.25 (dd, 1 H, *J* = 17.5, 2.2 Hz), 5.21 (dd, 1 H, *J* = 9.8, 2.4 Hz), 4.23 (td, 1 H, *J* = 11.2, 4.6 Hz), 3.75 (dd, 1 H, *J* = 10.8, 6.5 Hz), 2.59 (tt, 1 H, *J* = 10.0, 4.2 Hz), 1.47-1.38 (m, 1 H), 1.33-1.13 (m, 4 H), 1.02-0.86 (m, 1 H), 0.83 (t, 3 H, *J* = 6.7 Hz); ^{13}C NMR δ 140.89, 140.82, 137.80, 134.13, 132.68, 132.61, 132.55, 132.45, 131.76, 131.72, 131.66, 131.57, 131.53, 130.86, 128.51, 128.35, 128.16, 128.00, 127.71, 127.61, 126.85, 119.07, 57.72, 52.11, 52.07, 31.65, 29.57, 22.53, 13.98; EIMS *m/z* 403 (M^+, 1), 306 (100), 201 (98), 77 (22); HRMS (EI) *m/z* calcd for $C_{26}H_{30}NOP$ 403.2065, found 403.2068. **5b** (colorless solid): mp 160-161 °C (EtOAc/hexane); IR (KBr) 3181, 2953, 2928, 2852, 1435, 1180, 1126, 1109, 915, 745, 726, 696 cm^{-1}; ^1H NMR δ 7.87-7.80 (m, 2 H), 7.70-7.62 (m, 2 H), 7.54-7.34 (m, 4 H), 7.30-7.19 (m, 5 H), 7.13-7.09 (m, 2 H), 5.72 (ddd, 1 H, *J* = 17.2, 10.3, 8.8 Hz), 5.25 (dd, 1 H, *J* = 10.3, 1.9 Hz), 5.11 (ddd, 1 H, *J* = 17.2, 1.8, 0.7 Hz), 4.14 (dt, 1 H, *J* = 10.1, 7.4 Hz), 3.45 (t, 1 H, *J* = 6.1 Hz), 2.40-2.31 (m, 1 H), 1.35-1.09 (m, 6 H), 0.81 (t, 3 H, *J* = 6.6 Hz); ^{13}C NMR δ 142.78, 142.74, 139.25, 132.72, 132.57, 132.44, 132.34, 131.78, 131.73, 131.70, 131.66, 131.42, 131.39, 130.98, 128.48, 128.32, 128.04, 128.00, 127.87, 127.74, 127.65, 127.28, 126.86, 118.09, 58.13, 51.86, 51.79, 30.31, 29.39, 22.45, 13.97.

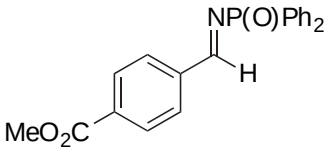
***N*-[(1*S**,2*R**)-2,3-Diethyl-1-phenylbut-3-enyl]-*P*,*P*-diphenylphosphinamide (7a)** and ***N*-[(1*S**,2*S**)-2,3-diethyl-1-phenylbut-3-enyl]-*P*,*P*-diphenylphosphinamide (7b).** According to the General Protocol A, 387 mg (1.50 mmol) of Cp_2ZrHCl, 190 μL (1.65 mmol) of 3-hexyne, 1.25 mL (2.50 mmol) of Me_2Zn (2.0 M solution in toluene), 120 μL (1.49 mmol) of CH_2I_2, and 153 mg (0.501 mmol) of imine **2** (24 h reaction time) afforded 99 mg (49%) of a 75:25 mixture of diastereomers. **7a** (colorless solid): mp 166-167 °C (EtOAc/hexane); IR (KBr) 3121, 2962, 2920, 2872, 2846, 1435, 1183, 1123, 1110, 1061, 897, 749, 726, 694 cm^{-1}; ^1H NMR δ 7.84-7.77 (m, 2 H), 7.67-7.61 (m, 2 H), 7.51-7.37 (m, 4 H), 7.29-7.17 (m, 5 H), 7.02-6.99 (m, 2 H), 4.79 (s, 1 H), 4.72 (s, 1 H), 4.12 (q, 1 H, *J* = 9.6 Hz), 3.39 (t, 1 H, *J* = 8.8 Hz), 2.34 (ddd, 1 H, *J* = 11.3, 7.9, 3.5 Hz), 2.00 (dqd, 1 H, *J* = 14.7, 7.4, 3.6 Hz), 1.65 (dq, 1 H, *J* = 14.8, 7.4 Hz), 1.57-1.42 (m, 2 H), 0.84 (t, 3 H, *J* = 7.3 Hz), 0.80 (t, 3 H, *J* = 7.3 Hz); ^{13}C NMR δ 149.63, 142.89, 142.84, 133.98, 132.74, 132.61, 132.29, 131.80, 131.77, 131.74, 131.68, 131.58, 131.54, 130.95, 128.49, 128.32, 128.15, 127.98, 127.78, 127.10, 126.68, 111.66, 58.61, 56.82, 56.76, 26.35, 23.12, 12.10, 11.54; EIMS *m/z* 404 ($[M+H]^+$, 1), 306 (100), 201 (93), 77 (86); HRMS (EI) *m/z* calcd for $C_{26}H_{30}NOP$ 403.2065, found 403.2052. **7b** (colorless solid): mp 164-165 °C (EtOAc/hexane); IR (KBr) 3197, 2961, 2932, 2873, 1436, 1185, 1124, 1109, 747, 725, 695 cm^{-1}; ^1H NMR δ 7.77-7.69 (m, 2 H), 7.60-7.53 (m, 2 H), 7.51-7.27 (m, 4 H), 7.20-7.04 (m, 7 H), 5.04 (s, 1 H), 4.96 (s, 1 H), 4.14 (td, 1 H, *J* = 9.2, 6.2 Hz), 3.60-3.50 (m, 1 H), 2.29 (ddd, 1 H, *J* = 10.7, 8.8, 4.2 Hz), 2.05 (dq, 1 H, *J* = 14.7, 7.4 Hz), 1.84 (dq, 1 H, *J* = 14.6, 7.3 Hz), 1.32 (ddq, 1 H, *J* = 14.0, 10.8, 7.3 Hz), 1.16 (dqd, 1 H, *J* = 14.2, 7.4, 4.4 Hz), 1.07 (t, 3 H, *J* = 7.3 Hz), 0.69 (t, 3 H, *J* = 7.4 Hz); ^{13}C NMR δ 150.73, 143.11, 134.08, 132.58, 132.45, 131.68, 131.65, 131.50, 131.37, 131.23, 130.79, 128.46, 128.29, 127.93, 127.89, 127.72, 127.46, 126.84, 112.49, 57.69, 57.60, 56.88, 24.31, 22.98, 12.09, 11.64; EIMS *m/z* 403 (M^+, 1), 306 (100), 201 (62), 91 (12), 77 (19); HRMS (EI) *m/z* calcd for $C_{26}H_{30}NOP$ 403.2065, found 403.2076.



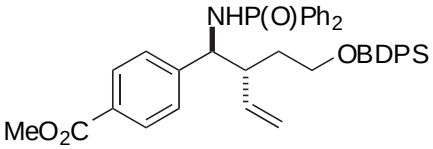
***N*-{(1*S**,2*R**)-2-[2-(*tert*-Butyldiphenylsilyl)oxyethyl]-1-phenylbut-3-enyl}-*P*,*P*-diphenylphosphinamide (9a).** According to the General Protocol A, 387 mg (1.50 mmol) of Cp_2ZrHCl, 510 mg (1.65 mmol) of alkyne **8**, 1.25 mL (2.50 mmol) of Me_2Zn (2.0 M solution in toluene), 120 μL (1.49 mmol) of CH_2I_2, and 153 mg (0.501 mmol) of imine **2** (12 h reaction time) afforded 227 mg (72%) of an 85:15 mixture of diastereomers. **9a** (colorless oil): IR (neat) 3196, 3070, 2930, 2857, 1438, 1428, 1191, 1110, 726, 700 cm^{-1}; ^1H NMR δ 7.96-7.88 (m, 2 H), 7.80-7.72 (m, 2 H), 7.72-7.67 (m, 4 H), 7.57-7.30 (m, 14 H), 7.12-7.09 (m, 2 H), 5.47 (dt, 1 H, *J* = 17.2, 9.7 Hz), 5.22 (dd, 1 H, *J* = 17.3, 2.2 Hz), 5.20 (dd, 1 H, *J* = 9.8, 2.2 Hz), 4.26 (td, 1 H, *J* = 10.8, 5.1 Hz), 3.75 (dd, 1 H, *J* = 10.7, 6.9 Hz), 3.70-3.63 (m, 2 H), 2.89-2.80 (m, 1 H), 1.92 (dtd, 1 H, *J* = 13.4, 7.9, 3.3 Hz), 1.34-1.15 (m, 1 H), 1.10 (s, 9 H); ^{13}C NMR δ 141.02, 140.96, 137.22, 135.49, 135.46, 133.94, 133.76, 133.71, 132.78, 132.54, 132.41, 132.24, 131.75, 131.62, 131.57, 131.05, 129.45, 128.50, 128.34, 128.19, 128.02, 127.80, 127.52, 126.89, 119.12, 61.73, 57.91, 48.28, 48.22, 34.54, 26.80, 19.08; EIMS *m/z* 628 ($[M-H]^+$, 1), 572 (48), 306 (100), 201 (75); HRMS (EI) *m/z* calcd for $C_{36}H_{35}NO_2PSi$ ($M-C_4H_9$) 572.2175, found 572.2174.

(4*R)-*O*-Triisopropylsilyl-4-{(*S**)-[(diphenylphosphoryl)amino](phenyl) methyl}hex-5-enoate (11a) and (4*S**)-*O*-triisopropylsilyl-4-{(*S**)-[(diphenylphosphoryl)amino](phenyl)methyl}hex-5-enoate (11b).** A solution of 324 mg (3.30 mmol) of 4-pentynoic acid in 6 mL of CH_2Cl_2 was treated with 710 μL (3.32 mmol) of TIPS-Cl and 225 mg (3.30 mmol) of imidazole, stirred at room temperature for 1 h, washed with H_2O and brine, dried ($MgSO_4$) and concentrated *in vacuo*. The residue was dissolved in 1 mL of CH_2Cl_2 then transferred into a suspension of 780 mg (3.02 mmol) of Cp_2ZrHCl in 2 mL of CH_2Cl_2, strirred 5 min and concentrated *in vacuo*. A solution of the residue in 3 mL of toluene was cooled to –78 °C, treated with 1.50 mL (3.00 mmol) of Me_2Zn (2.0 M solution in toluene),

warmed to room temperature over a period of 5 min, treated with 400 μL (4.97 mmol) of CH_2I_2, stirred for 2 min and cannulated into a suspension of 305 mg (1.00 mmol) of imine **2** in 1 mL of toluene. The reaction mixture was stirred at room temperature for 6 h, quenched with saturated NH_4Cl, diluted with EtOAc and saturated $NaHCO_3$, filtered through Celite, washed with H_2O and brine, dried ($MgSO_4$), filtered through a pad of Florisil and concentrated *in vacuo*. The residue was chromatographed on deactivated SiO_2 (1:4, hexanes/EtOAc containing 1% Et_3N) to yield 278 mg (48%) of a 62:38 mixture of diastereomers. **11a** (colorless oil): IR (neat) 3187, 2945, 2867, 1716, 1456, 1438, 1189, 1124, 1110, 1070, 918, 884, 751, 726, 700 cm^{-1}; ^1H NMR δ 7.85-7.78 (m, 2 H), 7.69-7.62 (m, 2 H), 7.50-7.39 (m, 4 H), 7.36-7.22 (m, 5 H), 7.03-7.00 (m, 2 H), 5.39 (dt, 1 H, *J* = 17.3, 9.6 Hz), 5.24-5.17 (m, 2 H), 4.18 (td, 1 H, *J* = 11.0, 5.1 Hz), 3.76 (dd, 1 H, *J* = 10.8, 6.4 Hz), 2.63-2.52 (m, 1 H), 2.30-2.24 (m, 2 H), 1.93-1.87 (m, 1 H), 1.29-1.14 (m, 4 H), 1.02 (d, 18 H, *J* = 7.3 Hz); ^{13}C NMR δ 173.37, 140.50, 136.67, 133.92, 132.64, 132.51, 132.23, 131.72, 131.59, 131.46, 130.70, 128.49, 128.32, 128.17, 128.00, 127.85, 127.42, 126.99, 120.03, 57.97, 51.63, 33.56, 27.25, 17.67, 11.75; EIMS *m/z* 575 (M^+, 1), 532 (46), 306 (100), 201 (91); HRMS (EI) *m/z* calcd for $C_{34}H_{46}NO_3PSi$ 575.2985, found 575.2999. **11b** (colorless oil): IR (neat) 2924, 2863, 1712, 1439, 1416, 1185, 1161, 1124, 1109, 1069, 997, 920, 883, 753, 724, 698 cm^{-1}; ^1H NMR δ 7.92-7.85 (m, 2 H), 7.75-7.68 (m, 2 H), 7.57-7.42 (m, 4 H), 7.35-7.24 (m, 5 H), 7.18-7.15 (m, 2 H), 5.73 (ddd, 1 H, *J* = 17.1, 10.2, 9.0 Hz), 5.34 (dd, 1 H, *J* = 10.3, 1.7 Hz), 5.19 (dd, 1 H, *J* = 17.2, 1.1 Hz), 4.21 (dt, 1 H, *J* = 10.1, 7.9 Hz), 3.53 (dd, 1 H, *J* = 8.4, 6.0 Hz), 2.52-2.22 (m, 3 H), 1.79-1.75 (m, 1 H), 1.65-1.59 (m, 1 H), 1.37-1.25 (m, 3 H), 1.09 (d, 18 H, *J* = 7.3 Hz); ^{13}C NMR δ 173.33, 142.08, 142.04, 138.24, 133.93, 132.68, 132.55, 132.42, 132.24, 131.75, 131.62, 131.47, 130.95, 128.50, 128.33, 128.10, 127.91, 127.23, 127.03, 118.97, 58.30, 51.25, 51.17, 33.44, 25.98, 17.72, 11.81; EIMS *m/z* 575 (M^+, 1), 532 (12), 306 (100), 201 (78); HRMS (EI) *m/z* calcd for $C_{34}H_{46}NO_3PSi$ 575.2985, found 575.2995.

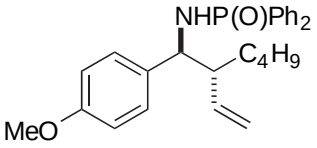


Methyl 4-[(diphenylphosphinylimino)methyl]benzoate (12).[2] A suspension of 1.50 g (6.91 mmol) of diphenylphosphinamide, 1.25 g (7.61 mmol) of methyl 4-formylbenzoate, and 3.00 mL (20.8 mmol) of Et_3N in 10 mL of CH_2Cl_2 was cooled to 0 °C, treated dropwise with a solution of 3.60 mL (3.60 mmol) of $TiCl_4$ (1.0 M solution in CH_2Cl_2) in 5 mL of CH_2Cl_2 over 20 min, and stirred at 0 °C for 5 min and at room temperature for 1 h. The reaction mixture was poured into 150 mL of Et_2O, stirred for 5 min, filtered through a pad of SiO_2, and concentrated *in vacuo*. The residue was precipitated from CH_2Cl_2 with hexanes to yield 1.19 g (47%) of **12** as a colorless solid: mp 143-145 °C (CH_2Cl_2/hexanes); IR (KBr) 3226, 3060, 3037, 1725, 1613, 1568, 1438, 1280, 1203, 1127, 1107, 861, 834, 766, 750, 729, 697 cm^{-1}; ^1H NMR δ 9.34 (d, 1 H, *J* = 31.7 Hz), 8.15-8.12 (m, 2 H), 8.06-8.03 (m, 2 H), 7.96-7.89 (m, 4 H), 7.48-7.36 (m, 6 H), 3.91 (s, 3 H); ^{13}C NMR δ 172.62, 172.52, 166.09, 139.22, 138.89, 134.15, 133.20, 131.91, 131.88, 131.76, 131.63, 131.53, 131.41, 129.95, 129.85, 128.55, 128.38, 128.26, 52.40; EIMS *m/z* 363 (M^+, 24), 201 (100); HRMS (EI) *m/z* calcd for $C_{21}H_{18}NO_3P$ 363.1024, found 363.1018.



Methyl 4-[(1*S,2*R**)-2-[2-(*tert*-butyldiphenylsilyl)oxyethyl]-1-(diphenylphosphoryl)aminobut-3-enyl]benzoate (13a).** According to the General Protocol A, 195 mg (0.756 mmol) of Cp_2ZrHCl, 255 mg (0.827 mmol) of alkyne 8, 500 µL (1.00 mmol) of Me_2Zn (2.0 M solution in toluene), 100 µL (1.24 mmol) of CH_2I_2, and 91.0 mg (0.250 mmol) of imine **12** (12 h reaction time) afforded 118 mg (69%) of an 85:15 mixture of diastereomers. **13a** (colorless oil): IR (neat) 3191, 3071, 2952, 2931, 2857, 1721, 1437, 1282, 1190, 1110, 911, 728, 702 cm^{-1}; ^1H NMR δ 8.00 (d, 2 H, *J* = 8.3 Hz), 7.94-7.87 (m, 2 H), 7.77-7.65 (m, 6 H), 7.62-7.31 (m, 12 H), 7.17 (d, 2 H, *J* = 8.3 Hz), 5.40 (dt, 1 H, *J* = 17.9, 9.3 Hz), 5.21-5.15 (m, 2 H), 4.31 (td, 1 H, *J* = 10.6, 5.4 Hz), 4.01 (s, 3 H), 3.76 (dd, 1 H, *J*

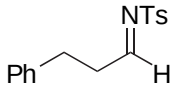
= 10.6, 6.9 Hz), 3.71-3.61 (m, 2 H), 2.89-2.79 (m, 1 H), 1.92 (dtd, 1 H, *J* = 13.3, 8.0, 3.2 Hz), 1.30-1.18 (m, 1 H), 1.09 (s, 9 H); ^{13}C NMR δ 167.06, 146.68, 146.62, 136.96, 135.66, 135.63, 133.84, 133.78, 133.75, 132.76, 132.61, 132.48, 132.05, 131.91, 131.79, 131.03, 129.69, 129.33, 128.96, 128.76, 128.60, 128.44, 128.27, 127.73, 119.70, 61.74, 57.87, 52.23, 48.34, 48.28, 34.62, 26.97, 19.24; EIMS *m/z* 688 ([M+H]$^+$, 1), 630 (80), 398 (19), 364 (100), 201 (99), 183 (33), 135 (46), 77 (70); HRMS (EI) *m/z* calcd for $C_{42}H_{45}NO_4PSi$ ([M-H]$^+$) 686.2856, found 686.2840.



***N*-[(1*S**,2*R**)-2-Butyl-1-(4-methoxyphenyl)but-3-enyl]-*P*,*P*-diphenylphosphinamide (15a).** According to the General Protocol A, 390 mg (1.51 mmol) of Cp_2ZrHCl, 190 μL (1.65 mmol) of 1-hexyne, 1.00 mL (2.00 mmol) of Me_2Zn (2.0 M solution in toluene), 200 μL (2.48 mmol) of CH_2I_2, and 168 mg (0.501 mmol) of imine **14** (12 h reaction time) afforded 169 mg (79%) of an 83:17 mixture of diastereomers. **15a** (colorless solid): mp 155-156 °C (EtOAc/hexane); IR (KBr) 3218, 2956, 2916, 2857, 1516, 1436, 1252, 1183, 1123, 1108, 1072, 909, 825, 748, 724, 694 cm^{-1}; ^{1}H NMR δ 7.94-7.86 (m, 2 H), 7.79-7.72 (m, 2 H), 7.56-7.44 (m, 4 H), 7.38-7.31 (m, 2 H), 7.03 (d, 2 H, *J* = 8.7 Hz), 6.87 (d, 2 H, *J* = 8.7 Hz), 5.48 (dt, 1 H, *J* = 17.3, 9.7 Hz), 5.29 (dd, 1 H, *J* = 17.2, 2.5 Hz), 5.25 (dd, 1 H, *J* = 9.7, 2.5 Hz), 4.23 (td, 1 H, *J* = 11.1, 4.5 Hz), 3.88 (s, 3 H), 3.74 (dd, 1 H, *J* = 10.8, 6.4 Hz), 2.61 (tt, 1 H, *J* = 9.9, 4.2 Hz), 1.51-1.40 (m, 1 H), 1.32-1.18 (m, 4 H), 1.07-0.96 (m, 1 H), 0.88 (t, 3 H, *J* = 6.7 Hz); ^{13}C NMR δ 158.38, 137.92, 134.19, 133.09, 133.02, 132.77, 132.65, 132.52, 131.68, 131.65, 131.52, 131.49, 131.02, 128.59, 128.47, 128.31, 128.15, 127.98, 118.94, 113.05, 57.17, 55.14, 52.19, 52.15, 31.69, 29.53, 22.53, 13.96.

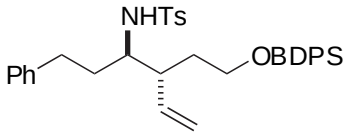
***N*-{(1*S**,2*R**)-2-[2-(*tert*-Butyldiphenylsilyl)oxyethyl]-1-phenylbut-3-enyl}-*S*-(4-methylphenyl)sulfonamide (17a)** and ***N*-{(1*S**,2*S**)-2-[2-(*tert*-**

butyldiphenylsilyl)oxyethyl]-1-phenylbut-3-enyl}-*S*-(4-methylphenyl)sulfonamide (17b). According to the General Protocol A, 390 mg (1.51 mmol) of Cp_2ZrHCl, 510 mg (1.65 mmol) of alkyne **8**, 750 μL (1.50 mmol) of Me_2Zn (2.0 M solution in toluene), 200 μL (2.48 mmol) of CH_2I_2, and 130 mg (0.501 mmol) of imine **16** (2 h reaction time) afforded 238 mg (81%) of a 60:40 (inseparable) mixture of diastereomers: IR (neat) 3280, 3070, 2930, 2857, 1428, 1326, 1162, 1111, 1093, 736, 702, 667 cm^{-1}; ^1H NMR δ 7.72-7.35 (m, 12 H), 7.25-7.11 (m, 6 H), 7.01-6.98 (m, 1 H), 5.56-5.07 (m, 4 H), 4.46 (dd, 0.6 H, *J* = 8.6, 5.5 Hz), 4.22 (dd, 0.4 H, *J* = 8.3, 5.3 Hz), 3.70-3.55 (m, 2 H), 2.75-2.64 (m, 1 H), 2.41 (s, 1.2 H), 2.39 (s, 1.8 H), 1.90-1.80 (m, 0.6 H), 1.57-1.47 (m, 0.4 H), 1.44-1.19 (m, 1 H), 1.11 (s, 5.4 H), 1.07 (s, 3.6 H); ^{13}C NMR δ 142.74, 139.30, 138.03, 137.71, 137.61, 137.40, 136.52, 135.49, 135.42, 133.62, 129.57, 129.50, 129.14, 129.02, 128.07, 127.80, 127.61, 127.58, 127.54, 127.23, 127.08, 127.04, 126.88, 119.67, 119.18, 61.19, 60.78, 60.71, 60.57, 46.77, 46.33, 33.90, 33.25, 26.81, 26.75, 21.38, 19.10; EIMS *m/z* 526 ($[M-C_4H_9]^+$, 42), 352 (83), 260 (96), 199 (68), 155 (68), 135 (46), 91 (100); HRMS (EI) *m/z* calcd for $C_{31}H_{32}NO_3SSi$ (M-C_4H_9) 526.1872, found 526.1869.

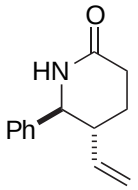


***N*-(3-Phenylpropylidene)-*S*-(4-methylphenyl)sulfonamide (18).**[4] A solution of 1.85 g (11.3 mmol) of sodium benzenesulfinate, 1.71 g (10.0 mmol) of *p*-toluenesulfonamide, and 1.32 mL (10.0 mmol) of hydrocinnamaldehyde in 30 mL of 50% formic acid was stirred at room temperature for 12 h, diluted with 20 mL of H_2O, filtered and rinsed with H_2O and pentane. The filter cake was dissolved in 75 mL of CH_2Cl_2 and stirred with 75 mL of saturated $NaHCO_3$ for 2 h. The organic layer was separated and the aqueous layer extracted with CH_2Cl_2. The combined organic layers were dried ($MgSO_4$), and concentrated *in vacuo*. The residue was precipitated from CH_2Cl_2 with hexanes to yield 2.03 g (71%) of **18** as a colorless solid: mp 61-63 °C (CH_2Cl_2/hexanes); IR (KBr) 3271, 3030, 2938, 1634, 1613, 1494, 1453, 1318, 1159, 1091, 784, 705, 682 cm^{-1}; ^1H NMR δ 8.67 (t, 1 H, *J* = 4.0 Hz), 7.81 (d, 2 H, *J* = 8.3 Hz), 7.38-7.15 (m, 7 H), 3.02-2.96 (m, 2 H), 2.91-2.85 (m, 2 H), 2.49 (s, 3 H); ^{13}C NMR δ 177.36, 144.69, 139.53, 134.36, 129.75, 128.58, 128.25, 128.09, 126.41, 37.30, 30.53, 21.61; EIMS *m/z* 287 (M^+, 2), 171

(12), 155 (25), 132 (100), 105 (16), 91 (20); HRMS (EI) *m/z* calcd for $C_{16}H_{17}NO_2S$ 287.0980, found 287.0979.

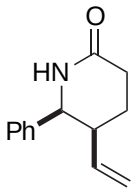


***N*-{(1*S**,2*R**)-2-[2-(*tert*-Butyldiphenylsilyl)oxyethyl]-1-phenethylbut-3-enyl}-*S*-(4-methylphenyl)sulfonamide (19a).** According to the General Protocol A, 195 mg (0.756 mmol) of Cp_2ZrHCl, 255 mg (0.827 mmol) of alkyne **8**, 500 μL (1.00 mmol) of Me_2Zn (2.0 M solution in toluene), 100 μL (1.24 mmol) of CH_2I_2, and 72.0 mg (0.251 mmol) of imine **18** (4 h reaction time) afforded 134 mg (87%) of **19a** as a colorless oil: IR (neat) 3280, 2930, 2857, 1428, 1327, 1160, 1111, 1094, 912, 816, 737, 702, 665 cm^{-1}; ^1H NMR δ 7.84 (d, 2 H, *J* = 8.3 Hz), 7.80-7.70 (m, 5 H), 7.54-7.45 (m, 6 H), 7.39-7.25 (m, 4 H), 7.20-7.15 (m, 2 H), 5.50 (dt, 1 H, *J* = 17.0, 9.9 Hz), 5.15 (dd, 1 H, *J* = 10.1, 1.9 Hz), 4.95 (dd, 1 H, *J* = 17.0, 1.8 Hz), 4.72 (d, 1 H, *J* = 9.7 Hz), 3.67-3.38 (m, 3 H), 2.84-2.71 (m, 1 H), 2.60-2.49 (m, 1 H), 2.44 (s, 3 H), 2.28-2.20 (m, 1 H), 1.92-1.81 (m, 1 H), 1.73-1.64 (m, 1 H), 1.61-1.32 (m, 2 H), 1.13 (s, 9 H); ^{13}C NMR δ 143.17, 141.63, 138.51, 136.86, 135.48, 135.46, 133.64, 129.60, 128.35, 128.31, 127.60, 126.93, 125.84, 119.03, 61.65, 56.96, 44.89, 33.95, 32.88, 32.01, 26.80, 21.45, 19.08; EIMS *m/z* 554 ($[M-C_4H_9]^+$, 66), 352 (46), 288 (70), 199 (67), 155 (31), 135 (37), 117 (69), 91 (100); HRMS (EI) *m/z* calcd for $C_{33}H_{36}NO_3SSi$ ($M-C_4H_9$) 554.2185, found 554.2188.

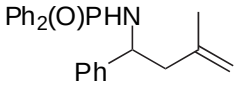


(5*R,6*S**)-6-Phenyl-5-vinylpiperidin-2-one (20a). General Protocol B.** A solution of 92.0 mg (0.160 mmol) of **11a** in 5 mL of HCl-MeOH (1 N solution) was stirred at room temperature for 12 h and concentrated *in vacuo*. The residue was dissolved in 3 mL of CH_2Cl_2, treated with 220 μL (1.58 mmol) of Et_3N and 75.0 μL (0.494 mmol) of diethyl cyanophosphonate, stirred for 16 h, filtered through a pad of Florisil and concentrated *in vacuo*. The residue was purified by preparative

TLC (EtOAc) to yield 18 mg (56%) of **20a** as a colorless solid: mp 131-132 °C (Et_2O/hexane); IR (KBr) 3171, 3070, 2883, 1647, 1400, 1004, 917, 760, 702 cm^{-1}; ^1H NMR δ 7.38-7.22 (m, 5 H), 5.72 (bs, 1 H), 5.62 (ddd, 1 H, *J* = 17.3, 10.4, 7.1 Hz), 4.99 (d, 1 H, *J* = 10.4 Hz), 4.91 (d, 1 H, *J* = 17.2 Hz), 4.22 (d, 1 H, *J* = 9.3 Hz), 2.57-2.38 (m, 3 H), 2.03-1.95 (m, 1 H), 1.90-1.79 (m, 1 H); ^{13}C NMR δ 171.61, 140.73, 137.32, 128.61, 128.22, 127.21, 116.86, 62.69, 45.40, 30.55, 26.14; EIMS *m/z* 201 (M^+, 26), 173 (9), 147 (12), 118 (13), 106 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{15}NO$ 201.1154, found 201.1158.



(5*S,6*S**)-6-Phenyl-5-vinylpiperidin-2-one (20b).** According to the General Protocol B, 25.0 mg (0.0434 mmol) of **11b**, 60.0 μL (0.430 mmol) of Et_3N and 20.0 μL (0.132 mmol) of diethyl cyanophosphonate afforded 6 mg (69%) of **20b** as a colorless solid: mp 108-109 °C (Et_2O/hexane); IR (thin film) 3200, 3061, 2933, 1660, 1404, 1353, 998, 915, 763, 700 cm^{-1}; ^1H NMR δ 7.37-7.26 (m, 3 H), 7.19-7.16 (m, 2 H), 5.89 (bs, 1 H), 5.46 (ddd, 1 H, *J* = 17.3, 10.1, 8.3 Hz), 5.01 (d, 1 H, *J* = 16.9 Hz), 4.99 (d, 1 H, *J* = 11.1 Hz), 4.68 (dd, 1 H, *J* = 4.9, 2.5 Hz), 2.82 (tt, 1 H, *J* = 8.7, 4.4 Hz), 2.67-2.44 (m, 2 H), 1.94-1.77 (m, 2 H); ^{13}C NMR δ 172.03, 138.90, 136.63, 128.23, 127.87, 127.52, 117.12, 60.38, 42.65, 29.66, 23.33; EIMS *m/z* 201 (M^+, 6), 173 (2), 147 (3), 119 (4), 106 (37), 84 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{15}NO$ 201.1154, found 201.1156.



***N*-(3-Methyl-1-phenylbut-3-enyl)-*P*,*P*-diphenylphosphinamide (24).** A solution of 584 mg (2.00 mmol) of Cp_2ZrCl_2 in 6 mL of THF was cooled to –78 °C, treated with 2.50 mL (4.00 mmol) of *n*-BuLi (1.6 M solution in hexanes), stirred at –78 °C for 1 h, treated with 200 μL (2.25 mmol) of 2-bromopropene, warmed to room temperature over 5 min, stirred for 1 h and concentrated *in vacuo*. The residue was suspended in 2 mL of toluene, cooled to –78 °C, treated with 1.00 mL (2.00 mmol) of Me_2Zn (2.0 M solution in toluene), warmed to room temperature over 5

min, treated with 200 μL (2.48 mmol) of CH_2I_2, stirred for 2 min, and treated with a solution of 153 mg (0.501 mmol) of imine **2** in 0.5 mL of CH_2Cl_2 and 1.5 mL of toluene. The reaction mixture was stirred at room temperature for 4 h, quenched with saturated NH_4Cl, diluted with EtOAc and saturated $NaHCO_3$, filtered through Celite, washed with H_2O and brine, dried ($MgSO_4$), filtered through a pad of Florisil and concentrated *in vacuo*. The residue was chromatographed on deactivated SiO_2 (1:9, hexanes/EtOAc containing 1% Et_3N) to yield 136 mg (75%) of **24** as a colorless solid: mp 165-166 °C (Et_2O/hexane); IR (thin film) 3182, 3058, 2910, 1437, 1186, 1123, 1109, 724, 696 cm^{-1}; ^1H NMR δ 7.87-7.79 (m, 2 H), 7.76-7.67 (m, 2 H), 7.51-7.33 (m, 4 H), 7.31-7.14 (m, 7 H), 4.77 (s, 1 H), 4.68 (s, 1 H), 4.36 (qn, 1 H, *J* = 6.3 Hz), 3.28 (dd, 1 H, *J* = 7.9, 6.0 Hz), 2.61 (dd, 1 H, *J* = 13.6, 7.3 Hz), 2.52 (dd, 1 H, *J* = 13.7, 7.0 Hz), 1.57 (s, 3 H); ^{13}C NMR δ 143.49, 142.00, 133.88, 132.86, 132.51, 132.38, 132.19, 131.83, 131.71, 131.62, 131.13, 128.52, 128.35, 128.30, 128.26, 128.09, 127.06, 126.56, 114.40, 53.39, 48.20, 48.13, 22.26; EIMS *m/z* 361 (M^+, 1), 306 (74), 201 (100); HRMS (EI) *m/z* calcd for $C_{23}H_{24}NOP$ 361.1596, found 361.1593.

***N*-(2,2-Dimethyl-1-phenylpent-3-enyl)-*P*,*P*-diphenylphosphinamide (25)** and ***N*-(2-methyl-1-phenylallyl)-*P*,*P*-diphenylphosphinamide (26).** A solution of 876 mg (3.00 mmol) of Cp_2ZrCl_2 in 10 mL of THF was cooled to –78 °C, treated with 3.80 mL (6.08 mmol) of *n*-BuLi (1.6 M solution in hexanes), stirred at –78 °C for 1 h, treated with 300 μL (3.38 mmol) of 2-bromopropene, warmed to room temperature over 5 min, stirred for 1 h and concentrated *in vacuo*. The residue was suspended in 2 mL of toluene, cooled to –78 °C, treated with 1.50 mL (3.00 mmol) of Me_2Zn (2.0 M solution in toluene), warmed to room temperature over 5 min, stirred for 2 h, and treated with a solution of 153 mg (0.501 mmol) of imine **2** in 0.5 mL of THF and 2 mL of toluene. The reaction mixture was stirred at room temperature for 12 h, quenched with saturated NH_4Cl, diluted with EtOAc and saturated $NaHCO_3$, filtered through Celite, washed with H_2O and brine, dried ($MgSO_4$), filtered through a pad of Florisil and concentrated *in vacuo*. The residue was chromatographed on deactivated SiO_2 (1:4, hexanes/EtOAc containing 1% Et_3N) to yield 88 mg (45%) of **25** and 27 mg (16%) of **26** as colorless solids. **25**:

(R_f 0.61, 1:4, hexanes/EtOAc): mp 134-135 °C (Et_2O/hexane); IR (KBr) 3323, 3059, 3017, 2967, 2937, 2875, 1434, 1182, 1125, 1068, 754, 724, 707, 692 cm^{-1}; ^1H NMR δ 7.80-7.73 (m, 2 H), 7.59-7.52 (m, 2 H), 7.49-7.27 (m, 5 H), 7.21-7.15 (m, 4 H), 6.99-6.96 (m, 2 H), 5.53-5.36 (m, 2 H),[5] 3.83 (t, 1 H, *J* = 10.6 Hz), 3.45 (t, 1 H, *J* = 8.7 Hz), 1.70 (d, 3 H, *J* = 5.1 Hz), 1.22 (s, 3 H), 0.81 (s, 3 H); ^{13}C NMR δ 141.68, 141.64, 136.15, 134.37, 132.84, 132.80, 132.67, 131.91, 131.78, 131.53, 131.50, 131.10, 128.61, 128.45, 128.10, 127.94, 127.49, 126.84, 125.48, 63.77, 40.87, 40.81, 26.06, 25.92, 18.45; EIMS *m/z* 390 ($[M+H]^+$, 1), 306 (100), 201 (93); HRMS (EI) *m/z* calcd for $C_{25}H_{29}NOP$ ($[M+H]^+$) 390.1987, found 390.1971. **26**: (R_f 0.53, 1:4, hexanes/EtOAc): mp 135-136 °C (Et_2O/hexane); IR (KBr) 3192, 1438, 1184, 1123, 1109, 725, 701 cm^{-1}; ^1H NMR δ 8.04-7.89 (m, 4 H), 7.61-7.31 (m, 11 H), 5.22 (s, 1 H), 5.13 (s, 1 H), 4.77 (t, 1 H, *J* = 10.6 Hz), 3.43 (dd, 1 H, *J* = 10.6, 6.6 Hz), 1.70 (s, 3 H); ^{13}C NMR δ 145.72, 145.66, 141.69, 141.63, 133.50, 133.11, 132.39, 132.27, 132.20, 132.07, 131.89, 131.85, 131.82, 131.79, 131.40, 128.55, 128.51, 128.45, 128.34, 128.29, 127.31, 127.01, 126.68, 112.54, 60.21, 19.79; EIMS *m/z* 347 (M^+, 19), 306 (26), 201 (72), 193 (43), 146 (95), 99 (100), 77 (59); HRMS (EI) *m/z* calcd for $C_{22}H_{22}NOP$ 347.1439, found 347.1431.

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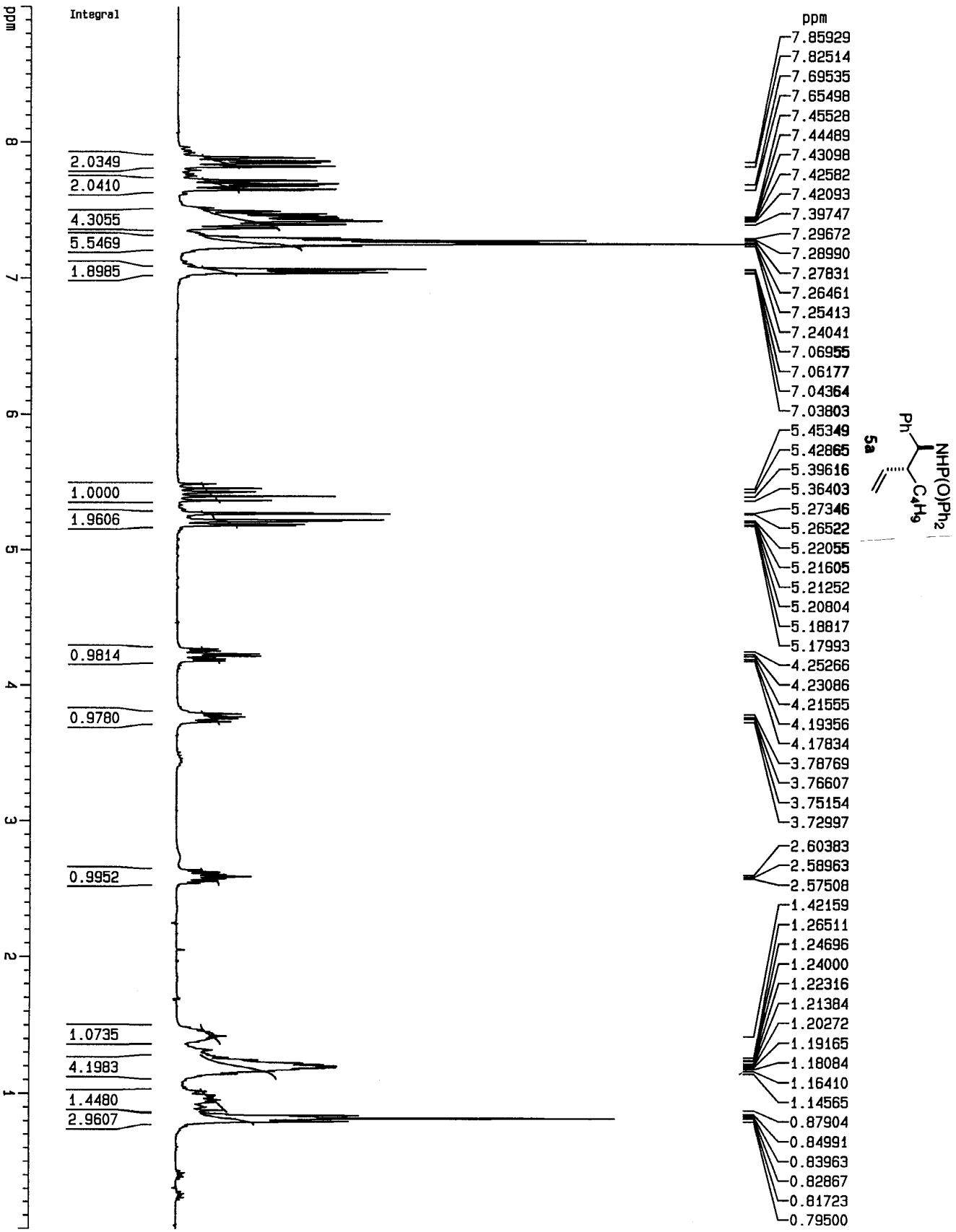
(1) Buchwald, S. L.; LaMaire, S. J.; Nielsen, R. B. *Org. Synth.* **1993**, *71*, 77.

(2) Jennings, W. B.; Lovely, C. J. *Tetrahedron* **1991**, *47*, 5561.

(3) Wipf, P.; Xu, W. *Org. Synth.* **1997**, *74*, 205.

(4) Chemla, F.; Hebbe, V.; Normant, J.-F. *Synthesis* **2000**, 75.

(5) Irradiation of the CH_3 doublet at 1.70 ppm leaves AB doublets at 5.52 and 5.44 ppm for this peak, with *J* = 15.7 Hz, thus the alkene is *trans*-configured.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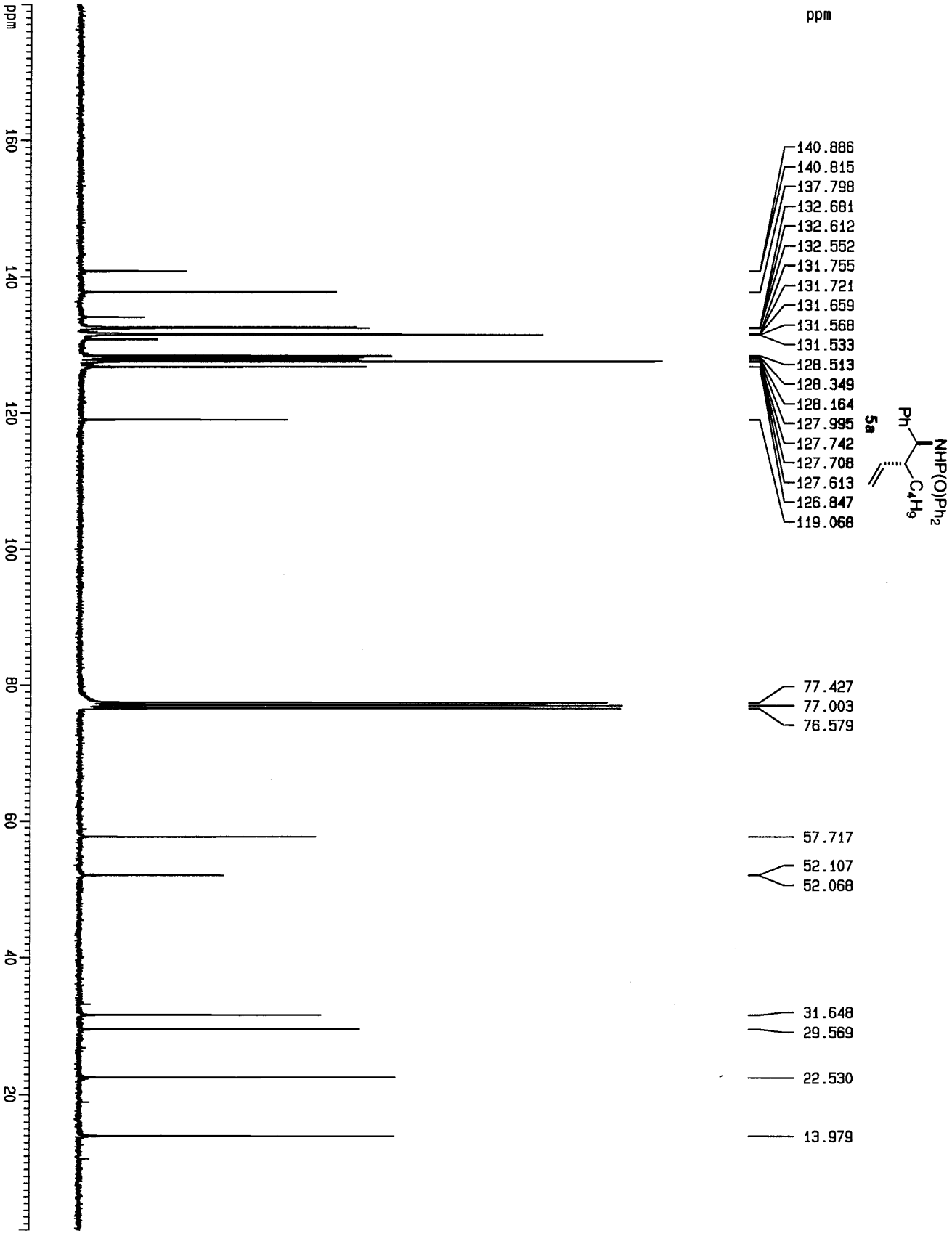
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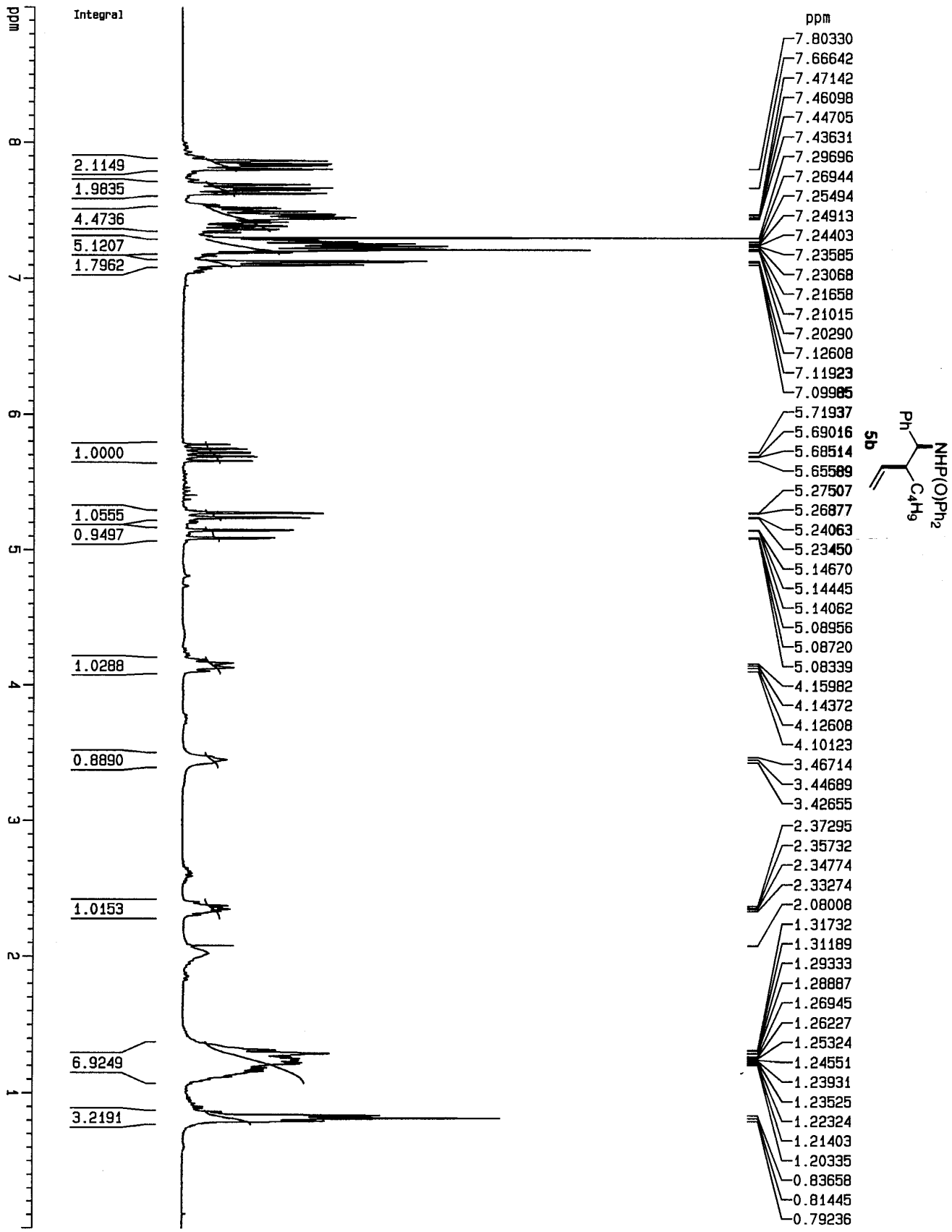
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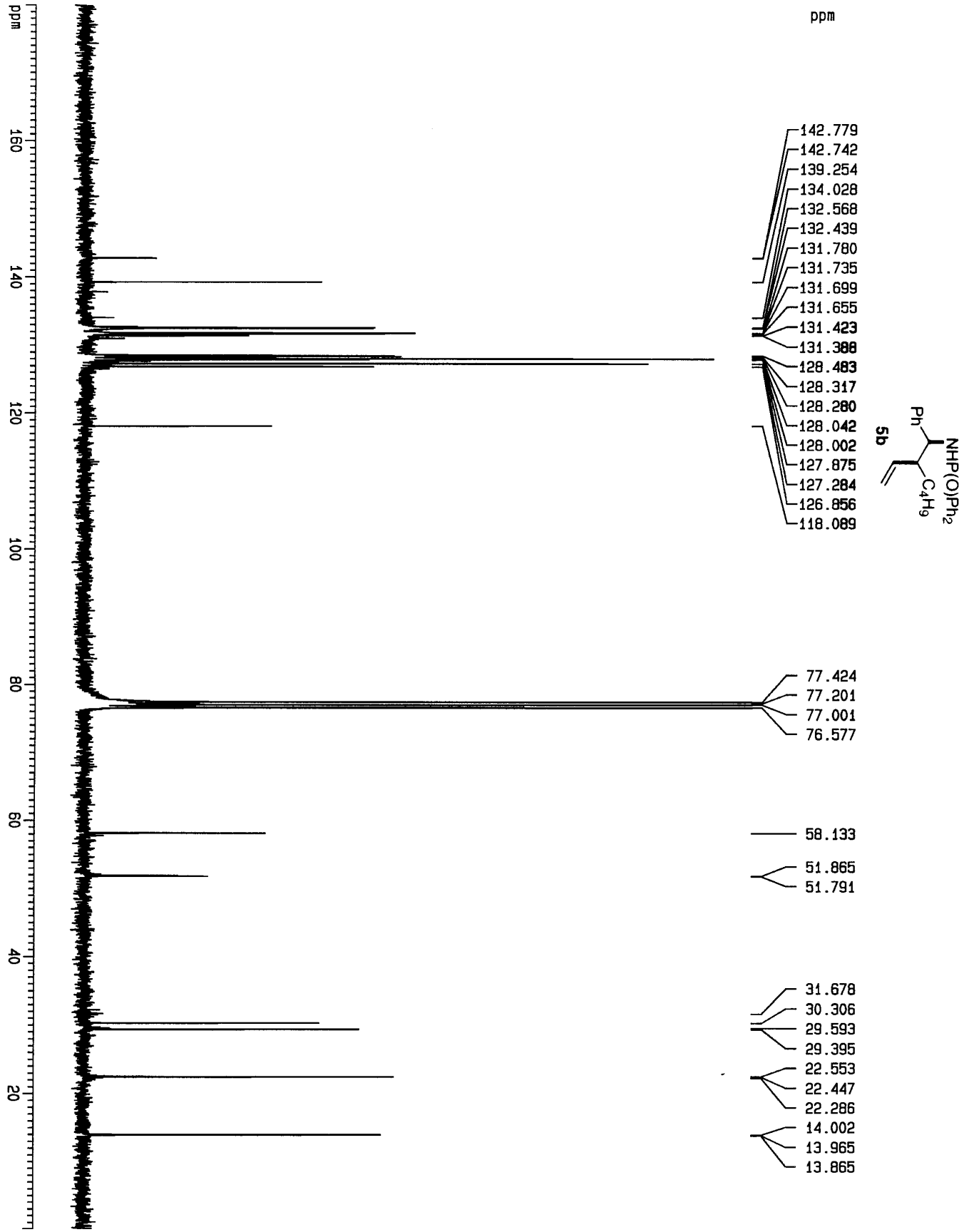
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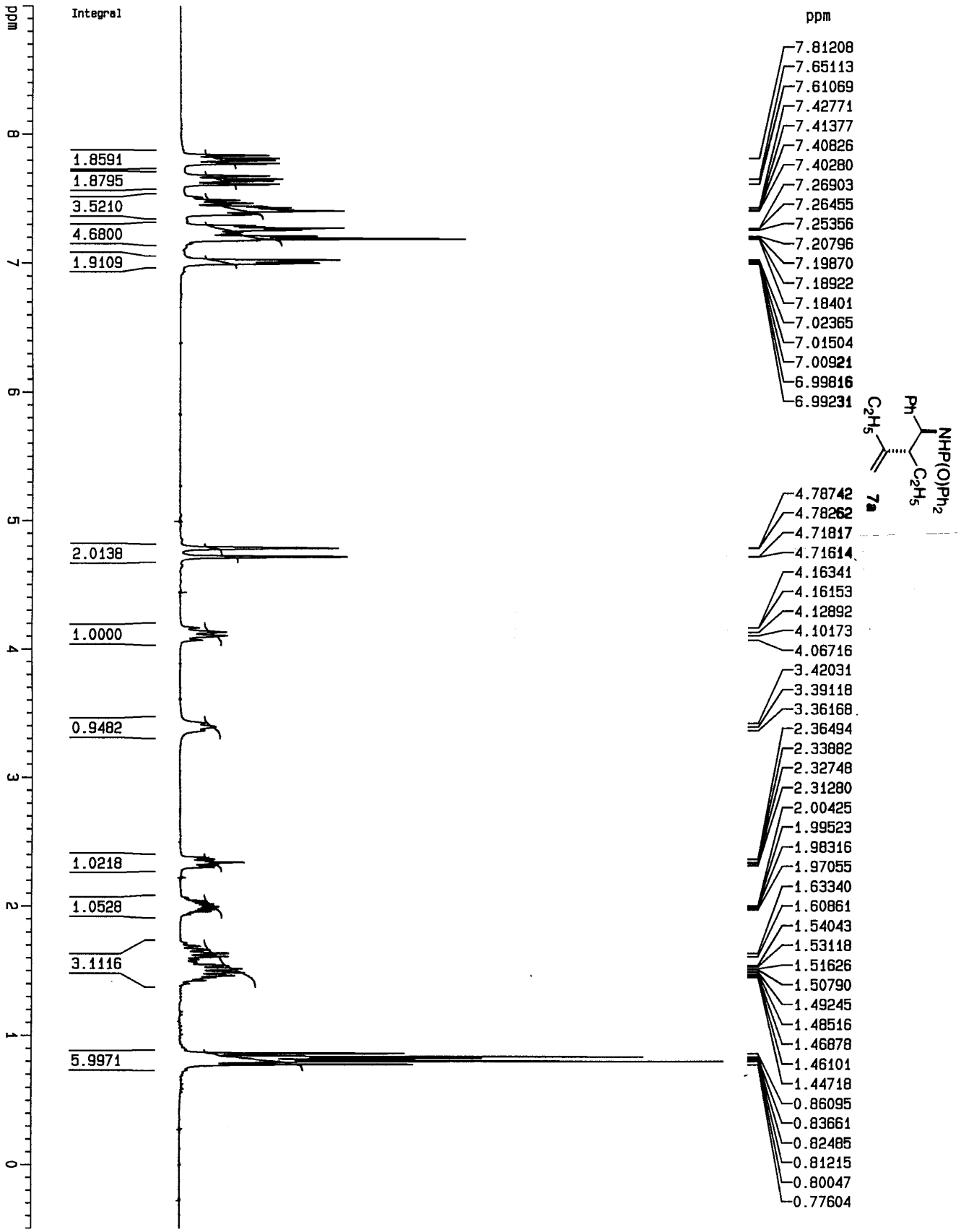
118.089
77.424
77.201
77.001
76.577
58.133
51.865
51.791
31.678
30.306
29.593
29.395
22.553
22.447
22.286
14.002
13.965
13.865
Ph
NHP(O)Ph2
C4H9
5b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3.5210
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2.0138
1.0000
0.9482
1.0218
1.0528
3.1116
5.9971
ppm
7.81208
7.65113
7.61069
7.42771
7.41377
7.40826
7.40280
7.26903
7.26455
7.25356
7.20796
7.19870
7.18922
7.18401
7.02365
7.01504
7.00921
6.99816
6.99231
4.78742
4.78262
4.71817
4.71614
4.16341
4.16153
4.12892
4.10173
4.06716
3.42031
3.39118
3.36168
2.36494
2.33882
2.32748
2.31280
2.00425
1.99523
1.98316
1.97055
1.63340
1.60861
1.54043
1.53118
1.51626
1.50790
1.49245
1.48516
1.46878
1.46101
1.44718
0.86095
0.83661
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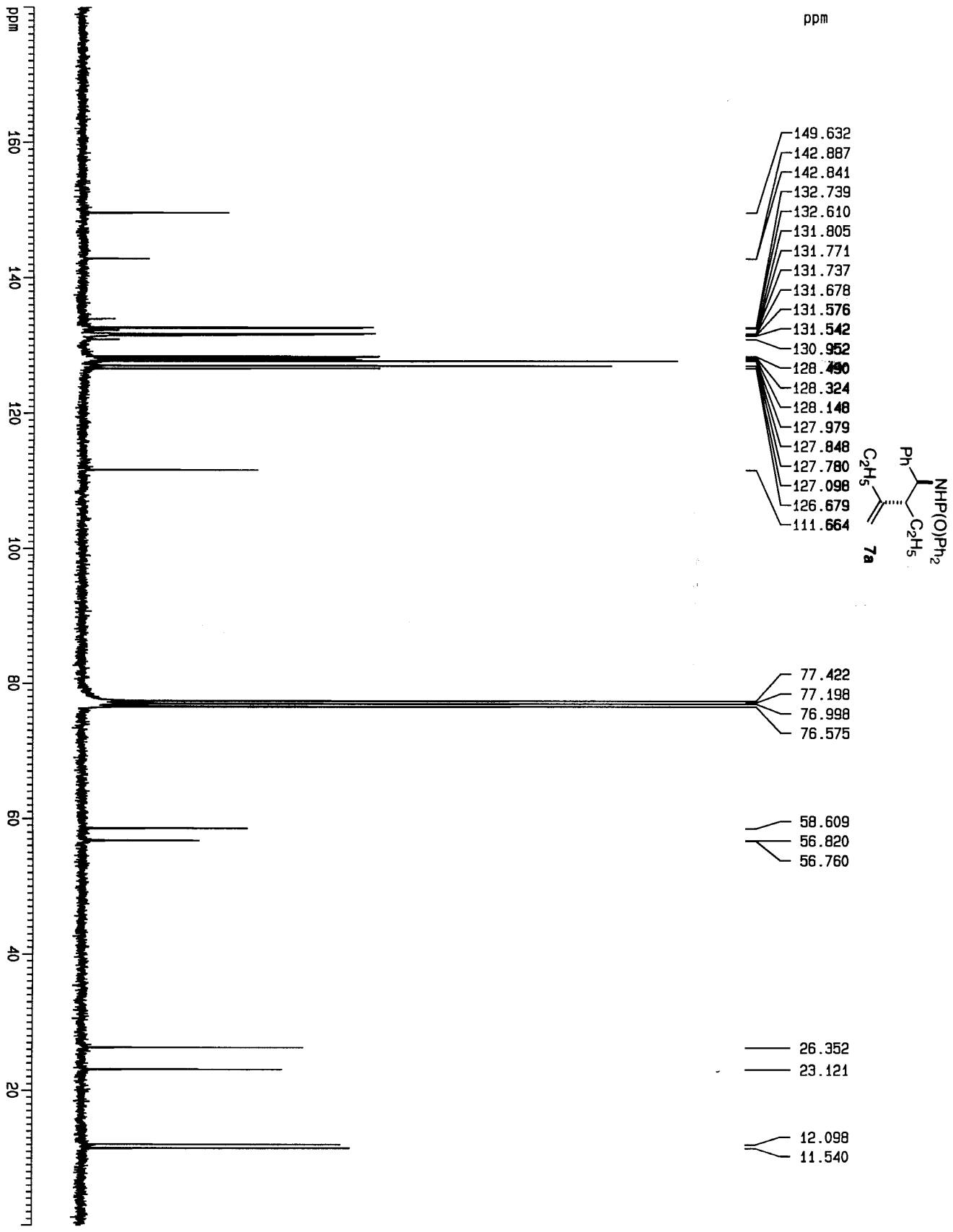
0.81215
0.80047
0.77604
Ph
NHP(O)Ph2
C2H5
C2H5
7a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120
100
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60
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20
ppm
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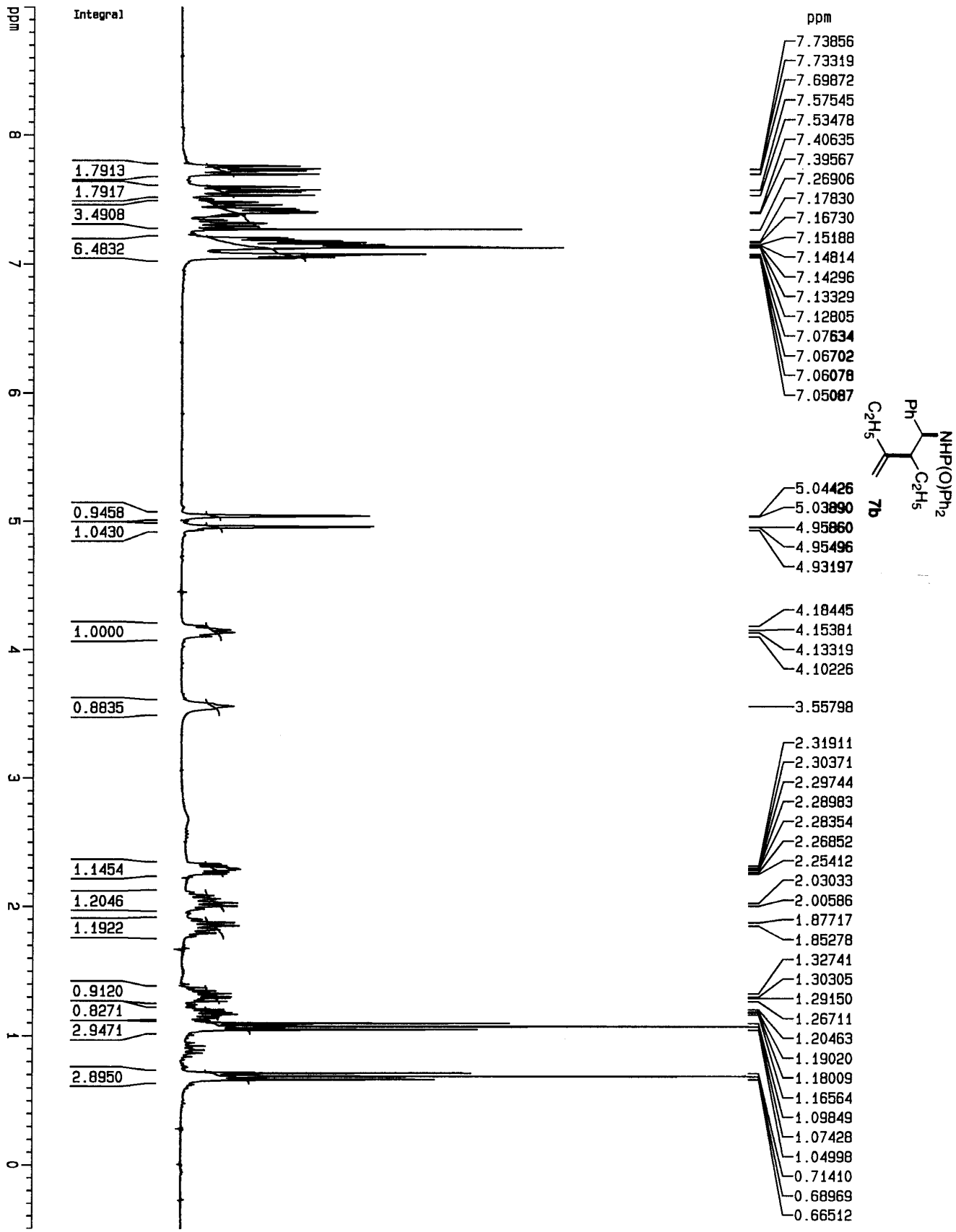
142.841
132.739
132.610
131.805
131.771
131.737
131.678
131.576
131.542
130.952
128.490
128.324
128.148
127.979
127.848
127.780
127.098
126.679
111.664
77.422
77.198
76.998
76.575
58.609
56.820
56.760
26.352
23.121
12.098
11.540
Ph
NHP(O)Ph2
C2H5
C2H5
7a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1.7917
3.4908
6.4832
0.9458
1.0430
1.0000
0.8835
1.1454
1.2046
1.1922
0.9120
0.8271
2.9471
2.8950
ppm
7.73856
7.73319
7.69872
7.57545
7.53478
7.40635
7.39567
7.26906
7.17830
7.16730
7.15188
7.14814
7.14296
7.13329
7.12805
7.07634
7.06702
7.06078
7.05087
5.04426
5.03890
4.95860
4.95496
4.93197
4.18445
4.15381
4.13319
4.10226
3.55798
2.31911
2.30371
2.29744
2.28983
2.28354
2.26852
2.25412
2.03033
2.00586
1.87717
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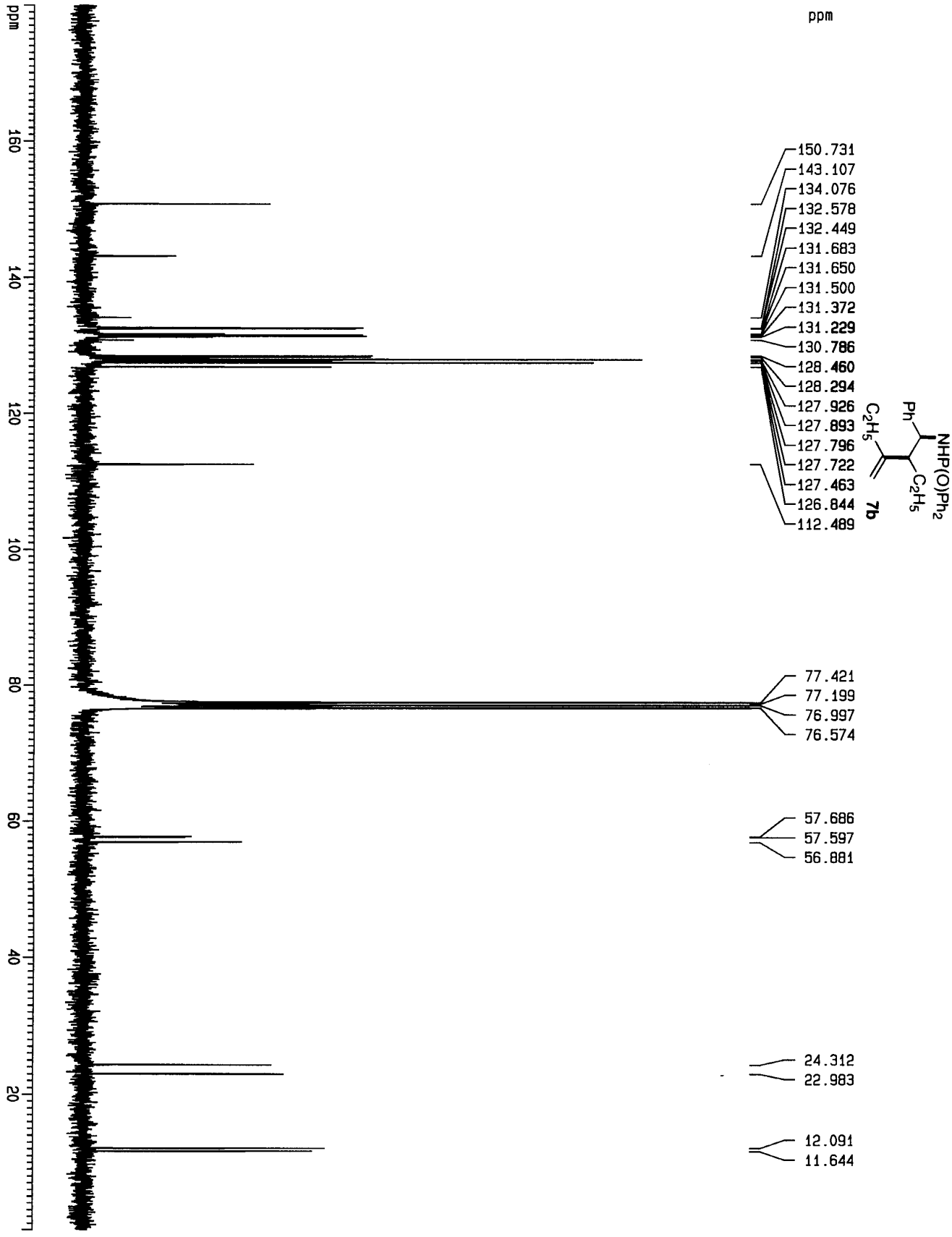
1.29150
1.26711
1.20463
1.19020
1.18009
1.16564
1.09849
1.07428
1.04998
0.71410
0.68969
0.66512
Ph
NHP(O)Ph2
C2H5
C2H5
7b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80
60
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20
ppm
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143.107
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132.578
132.449
131.683
131.650
131.500
131.372
131.229
130.786
128.460
128.294
127.926
127.893
127.796
127.722
127.463
126.844
112.489
77.421
77.199
76.997
76.574
57.686
57.597
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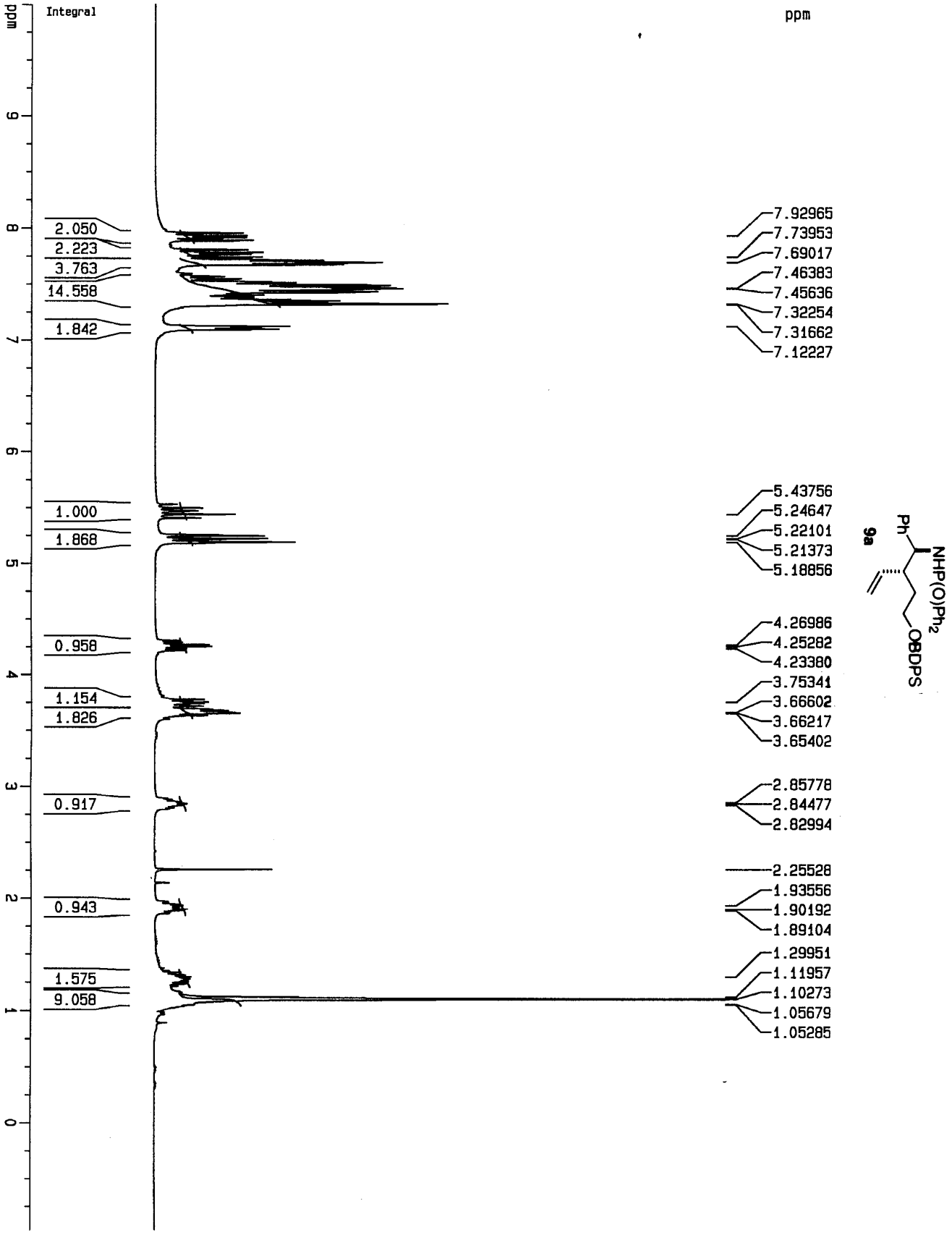
24.312
22.983
12.091
11.644
Ph
C2H5
NHP(O)Ph2
C2H5
7b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1.000
1.868
0.958
1.154
1.826
0.917
0.943
1.575
9.058
7.92965
7.73953
7.69017
7.46383
7.45636
7.32254
7.31662
7.12227
5.43756
5.24647
5.22101
5.21373
5.18856
4.26986
4.25282
4.23380
3.75341
3.66602
3.66217
3.65402
2.85778
2.84477
2.82994
2.25528
1.93556
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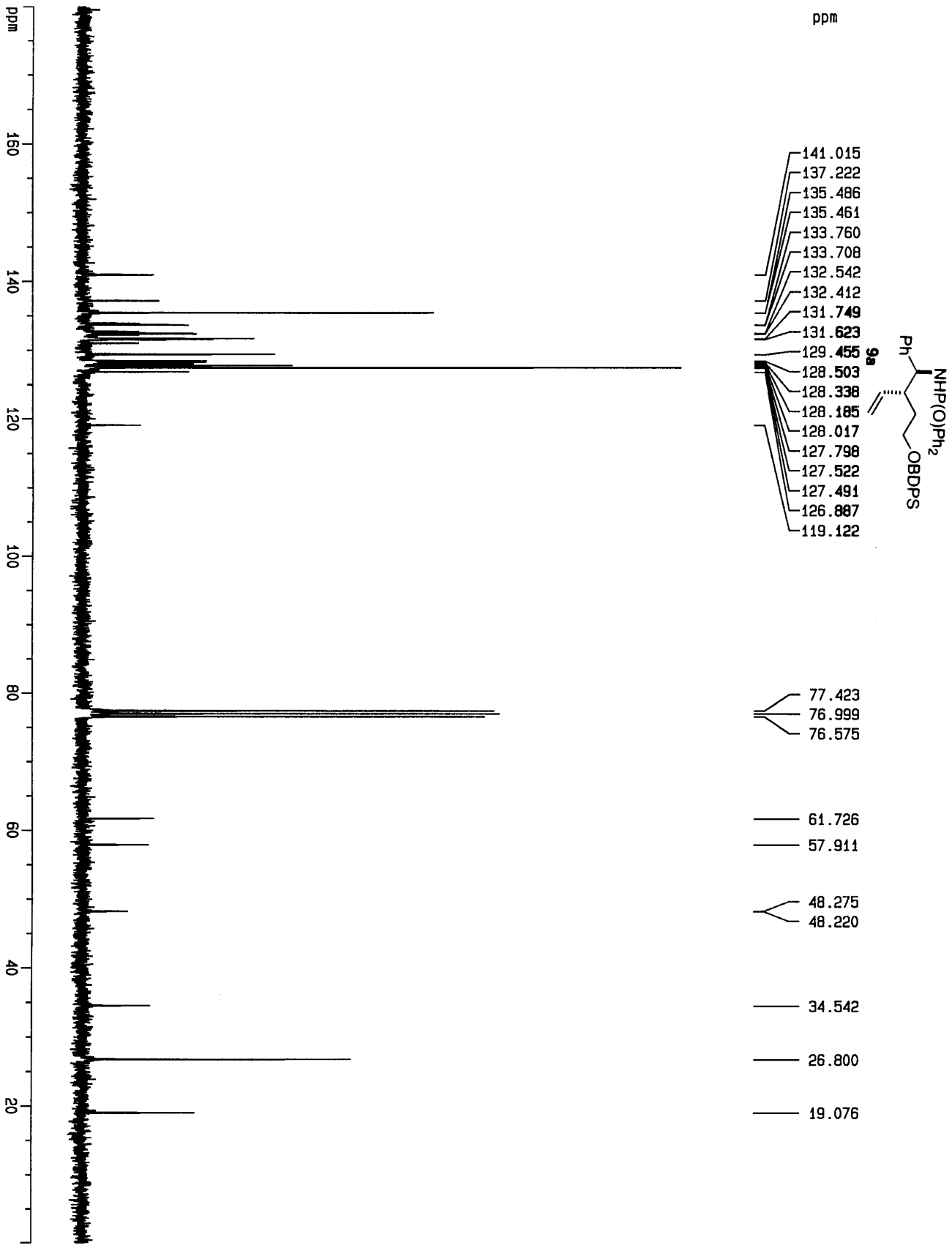
1.29951
1.11957
1.10273
1.05679
1.05285
9a
Ph
NHP(O)Ph2
OTBDPS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80
60
40
20
ppm
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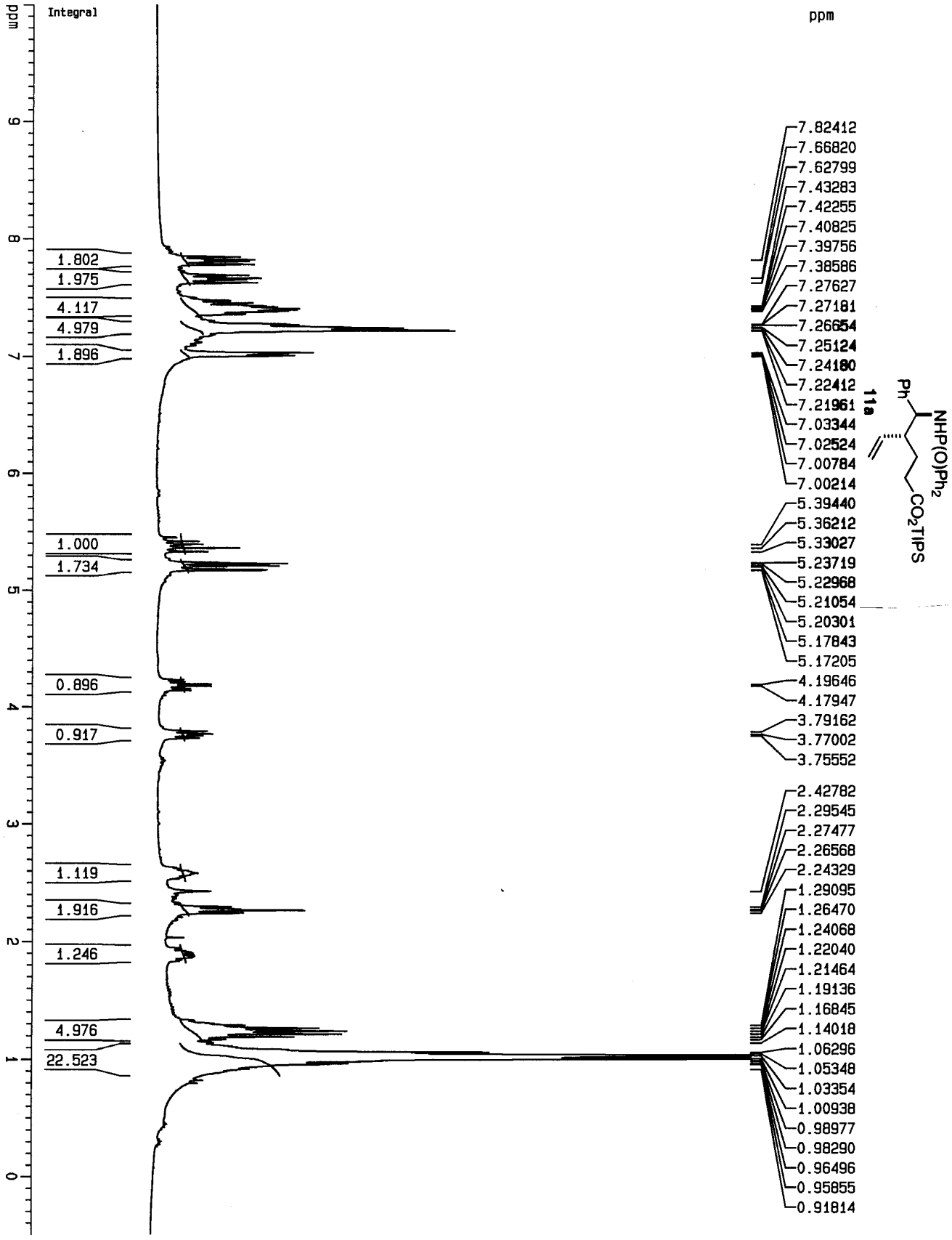
135.461
133.760
133.708
132.542
132.412
131.749
131.623
129.455
128.503
128.338
128.185
128.017
127.798
127.522
127.491
126.887
119.122
9a
Ph
NHP(O)Ph2
OBDPS
77.423
76.999
76.575
61.726
57.911
48.275
48.220
34.542
26.800
19.076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Integral
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4.117
4.979
1.896
1.000
1.734
0.896
0.917
1.119
1.916
1.246
4.976
22.523
ppm
7.82412
7.66820
7.62799
7.43283
7.42255
7.40825
7.39756
7.38586
7.27627
7.27181
7.26654
7.25124
7.24180
7.22412
7.21961
7.03344
7.02524
7.00784
7.00214
5.39440
5.36212
5.33027
5.23719
5.22968
5.21054
5.20301
5.17843
5.17205
4.19646
4.17947
3.79162
3.77002
3.75552
2.42782
2.29545
2.27477
2.26568
2.24329
1.29095
1.26470
1.24068
1.22040
1.21464
1.19136
1.16845
1.14018
1.06296
1.05348
1.03354
1.00938
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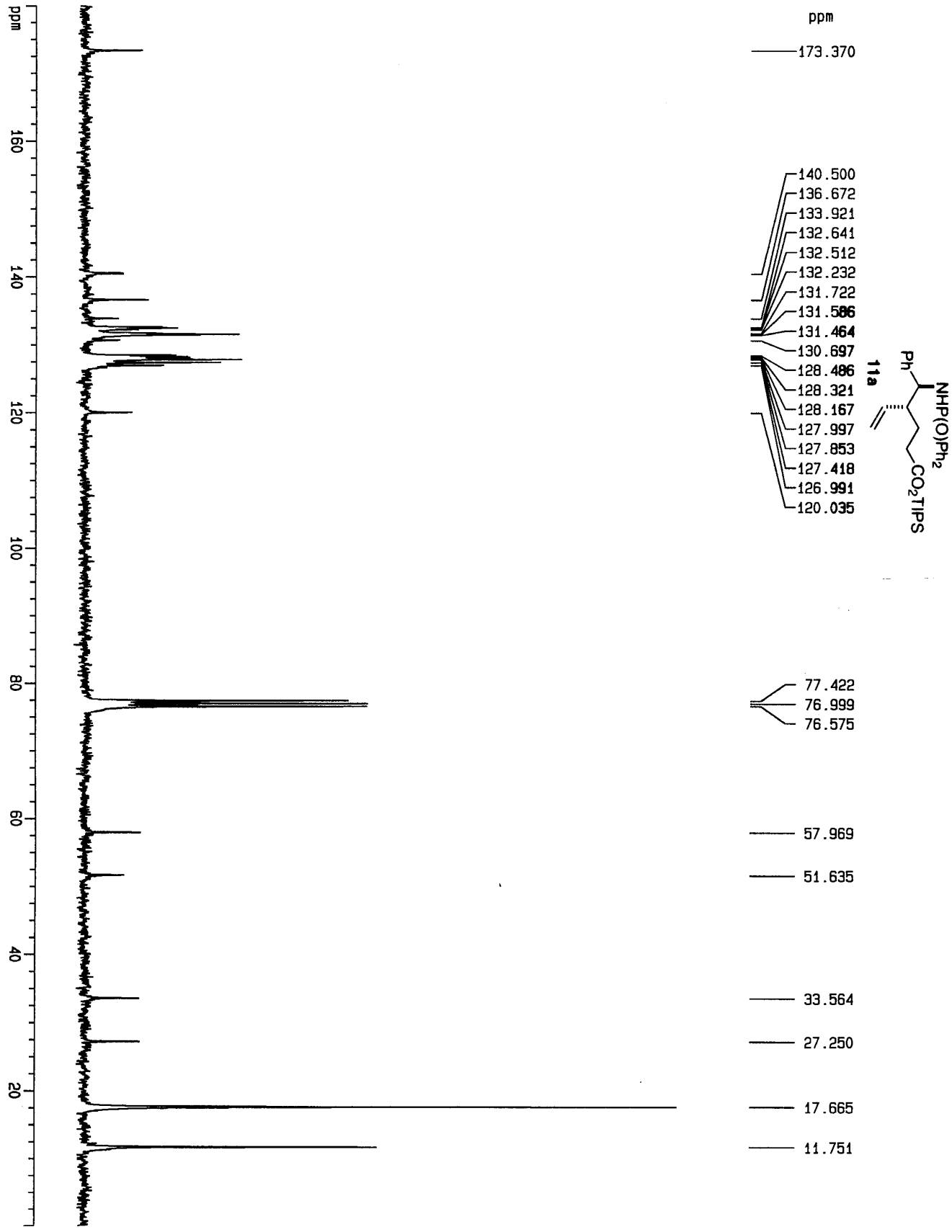
0.98290
0.96496
0.95855
0.91814
11a
Ph
NHP(O)Ph2
CO2TIPS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140
120
100
80
60
40
20
ppm
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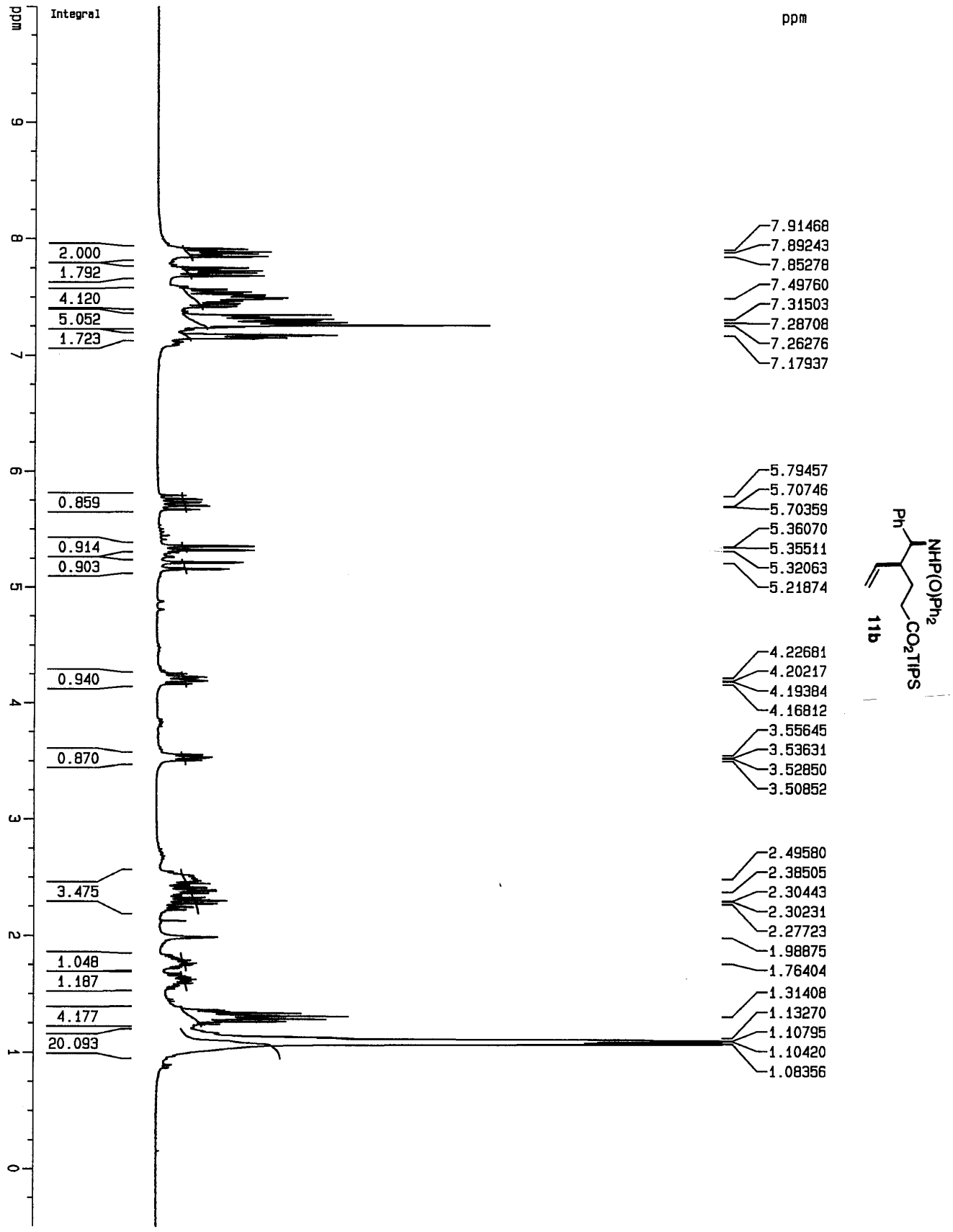
133.921
132.641
132.512
132.232
131.722
131.586
131.464
130.697
128.486
128.321
128.167
127.997
127.853
127.418
126.991
120.035
77.422
76.999
76.575
57.969
51.635
33.564
27.250
17.665
11.751
11a
Ph
NHP(O)Ph2
CO2TIPS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1.723
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0.914
0.903
0.940
0.870
3.475
1.048
1.187
4.177
20.093
7.91468
7.89243
7.85278
7.49760
7.31503
7.28708
7.26276
7.17937
5.79457
5.70746
5.70359
5.36070
5.35511
5.32063
5.21874
4.22681
4.20217
4.19384
4.16812
3.55645
3.53631
3.52850
3.50852
2.49580
2.38505
2.30443
2.30231
2.27723
1.98875
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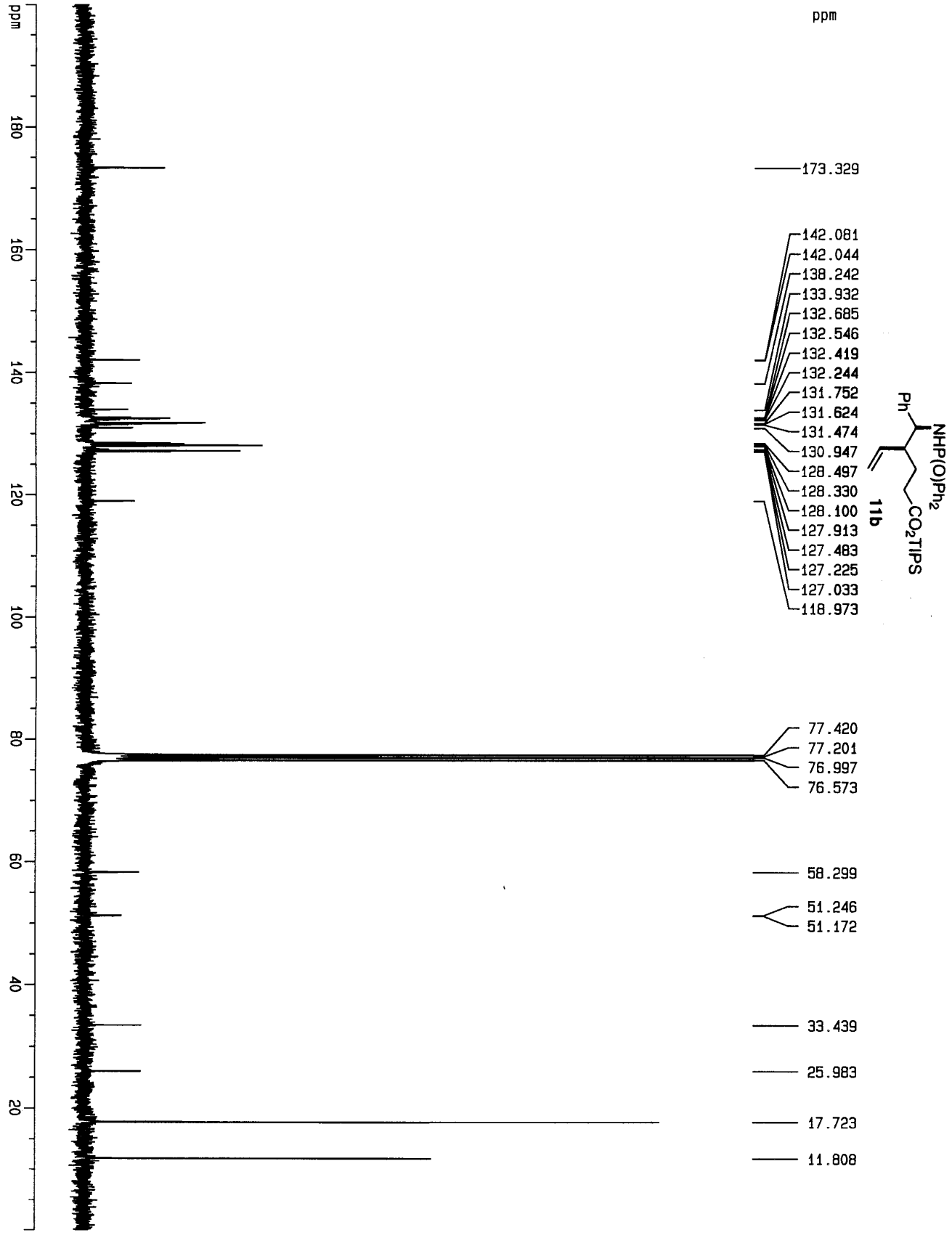
1.13270
1.10795
1.10420
1.08356
Ph
NHP(O)Ph2
CO2TIPS
11b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140
120
100
80
60
40
20
ppm
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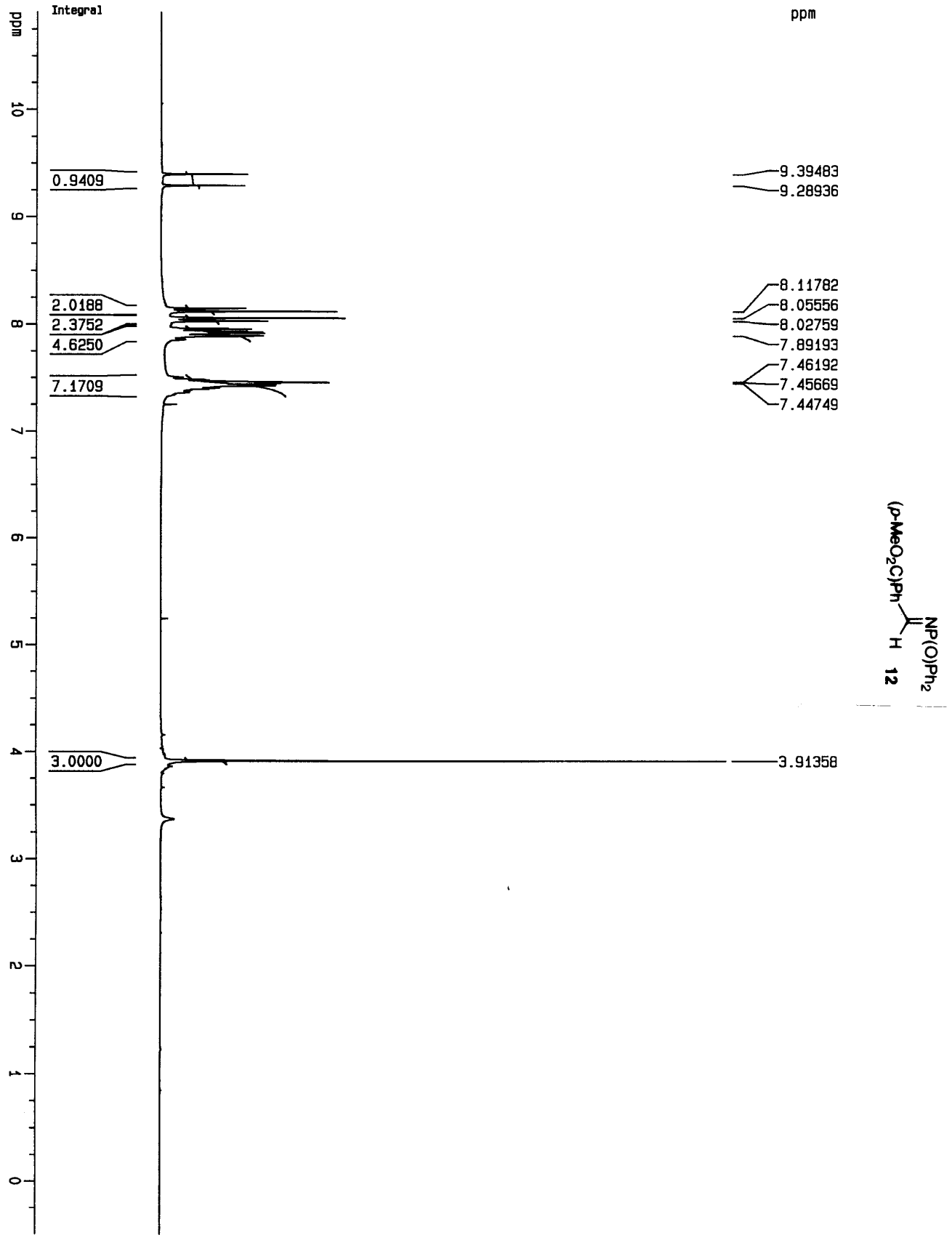
142.044
138.242
133.932
132.685
132.546
132.419
132.244
131.752
131.624
131.474
130.947
128.497
128.330
128.100
127.913
127.483
127.225
127.033
118.973
77.420
77.201
76.997
76.573
58.299
51.246
51.172
33.439
25.983
17.723
11.808
Ph
NHP(O)Ph2
CO2TIPS
11b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2.3752
4.6250
7.1709
3.0000
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9.28936
8.11782
8.05556
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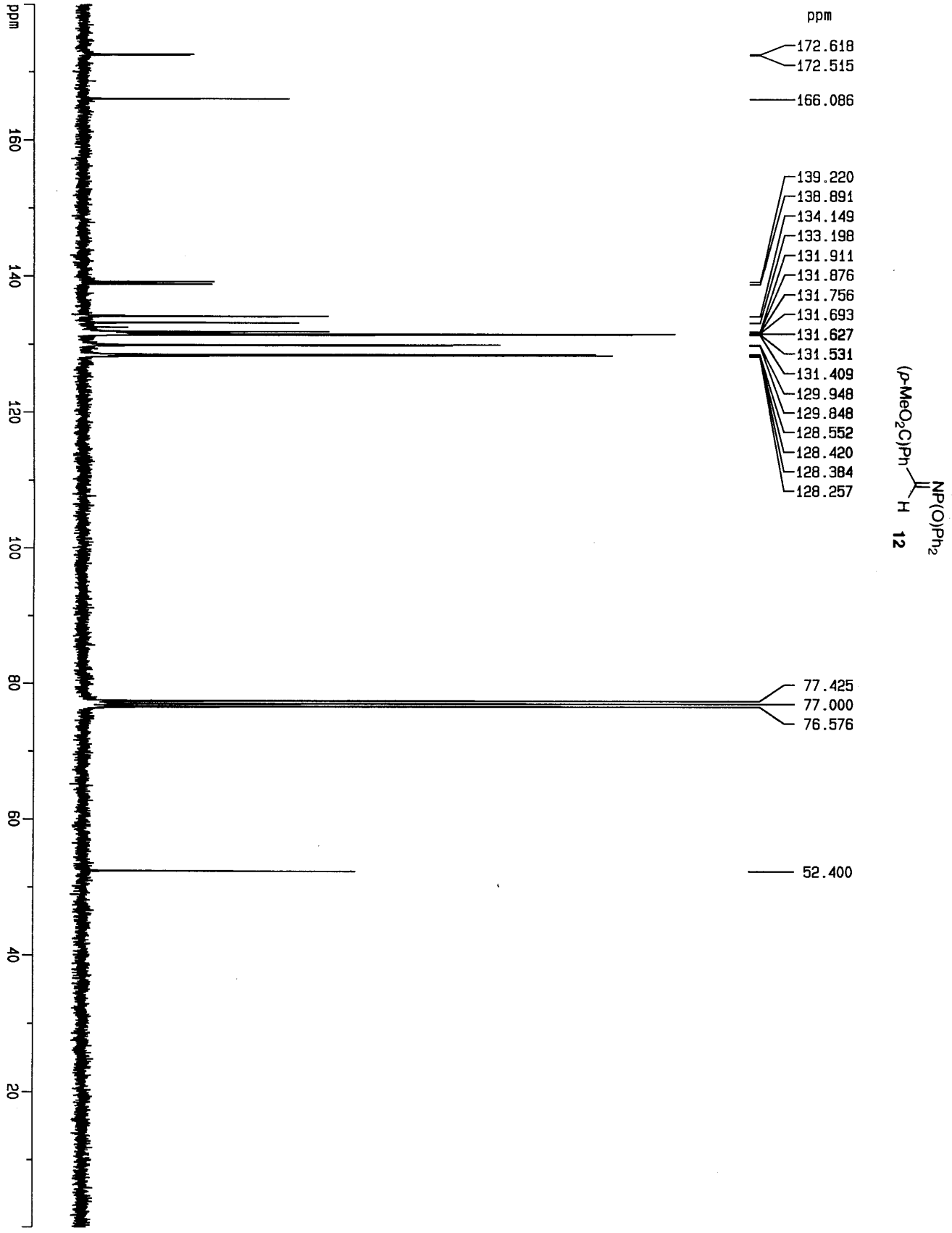
7.46192
7.45669
7.44749
3.91358
(p-MeO2C)Ph
H
NP(O)Ph2
12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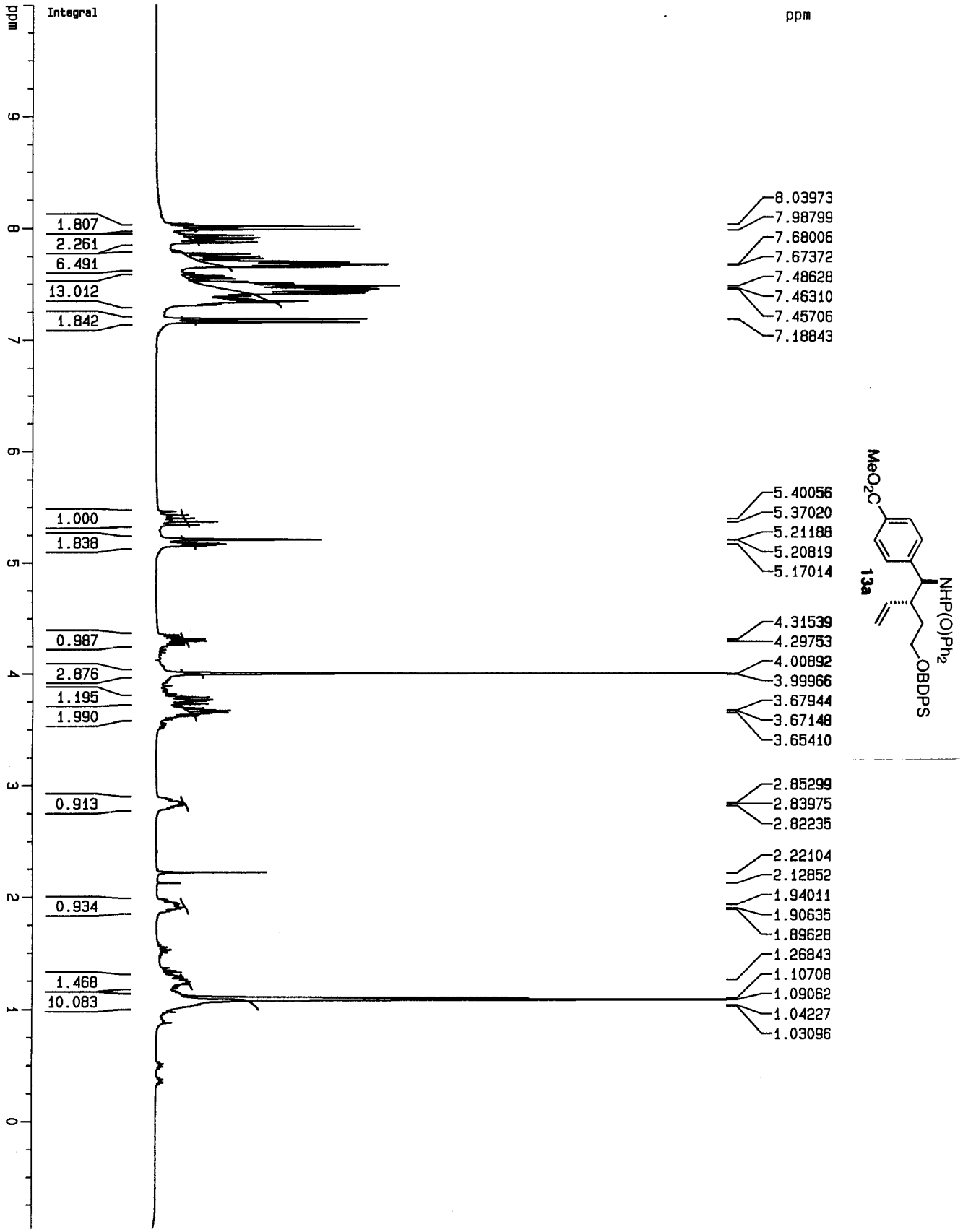
138.891
134.149
133.198
131.911
131.876
131.756
131.693
131.627
131.531
131.409
129.948
129.848
128.552
128.420
128.384
128.257
77.425
77.000
76.576
52.400
(p-MeO2C)Ph
NP(O)Ph2
H
12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2.876
1.195
1.990
0.913
0.934
1.468
10.083
8.03973
7.98799
7.68006
7.67372
7.48628
7.46310
7.45706
7.18843
5.40056
5.37020
5.21188
5.20819
5.17014
4.31539
4.29753
4.00892
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3.67944
3.67148
3.65410
2.85299
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1.90635
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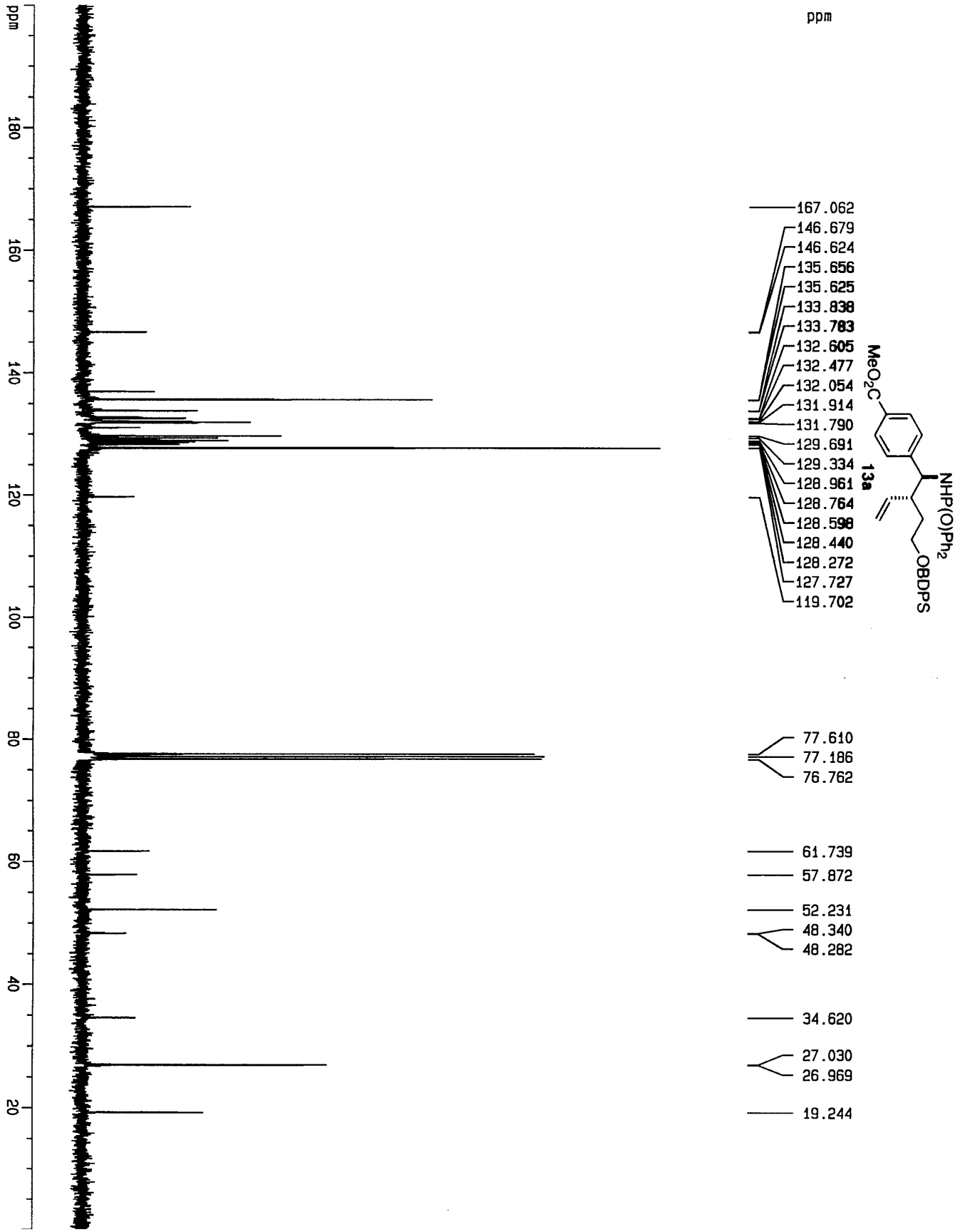
1.09062
1.04227
1.03096
MeO2C
13a
NHP(O)Ph2
OBDPS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120
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80
60
40
20
ppm
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146.624
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135.625
133.838
133.783
132.605
132.477
132.054
131.914
131.790
129.691
129.334
128.961
128.764
128.598
128.440
128.272
127.727
119.702
77.610
77.186
76.762
61.739
57.872
52.231
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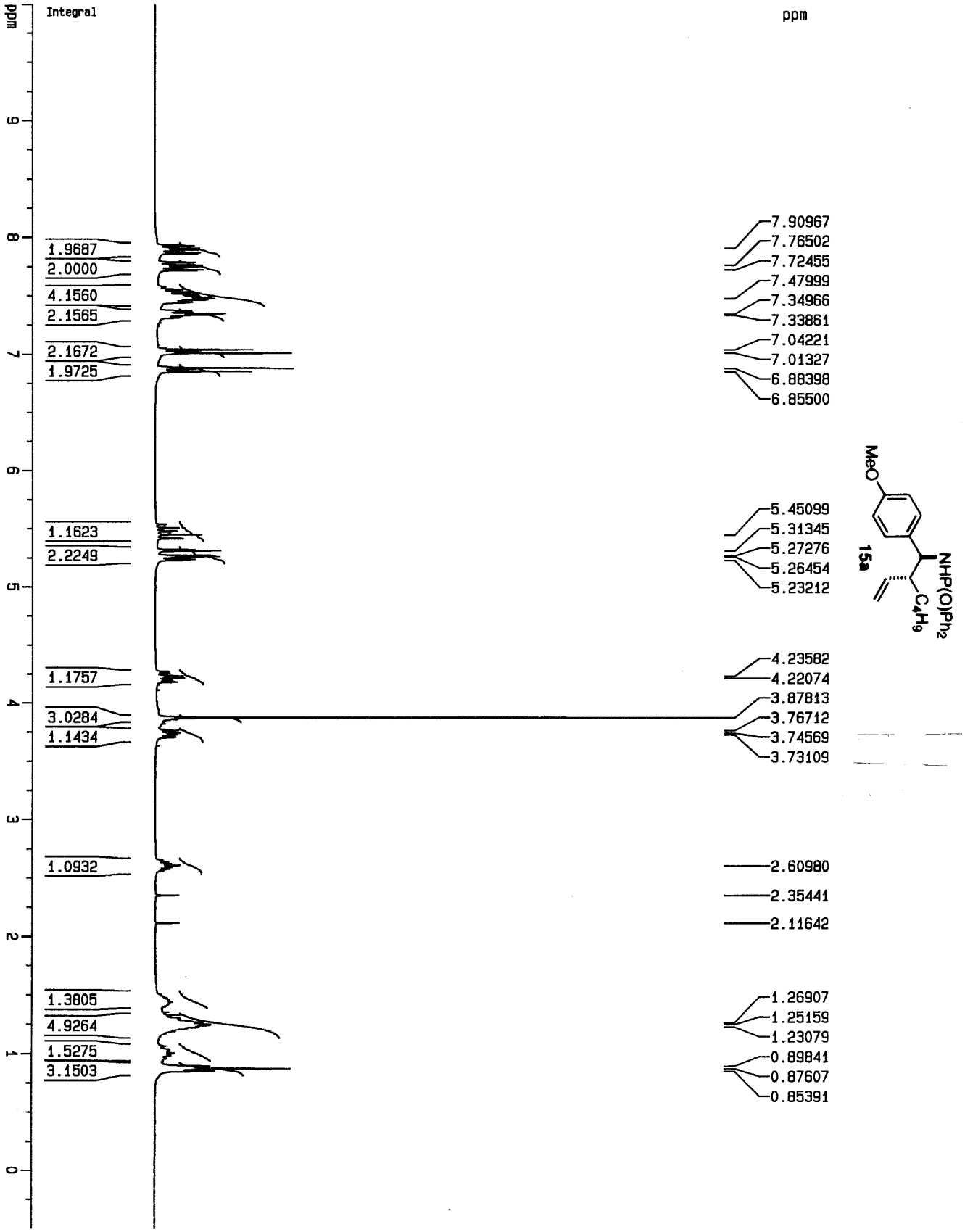
48.282
34.620
27.030
26.969
19.244
MeO2C
13a
NHP(O)Ph2
OBDPS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1.1623
2.2249
1.1757
3.0284
1.1434
1.0932
1.3805
4.9264
1.5275
3.1503
7.90967
7.76502
7.72455
7.47999
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7.04221
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6.88398
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5.26454
5.23212
4.23582
4.22074
3.87813
3.76712
3.74569
3.73109
2.60980
2.35441
2.11642
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MeO
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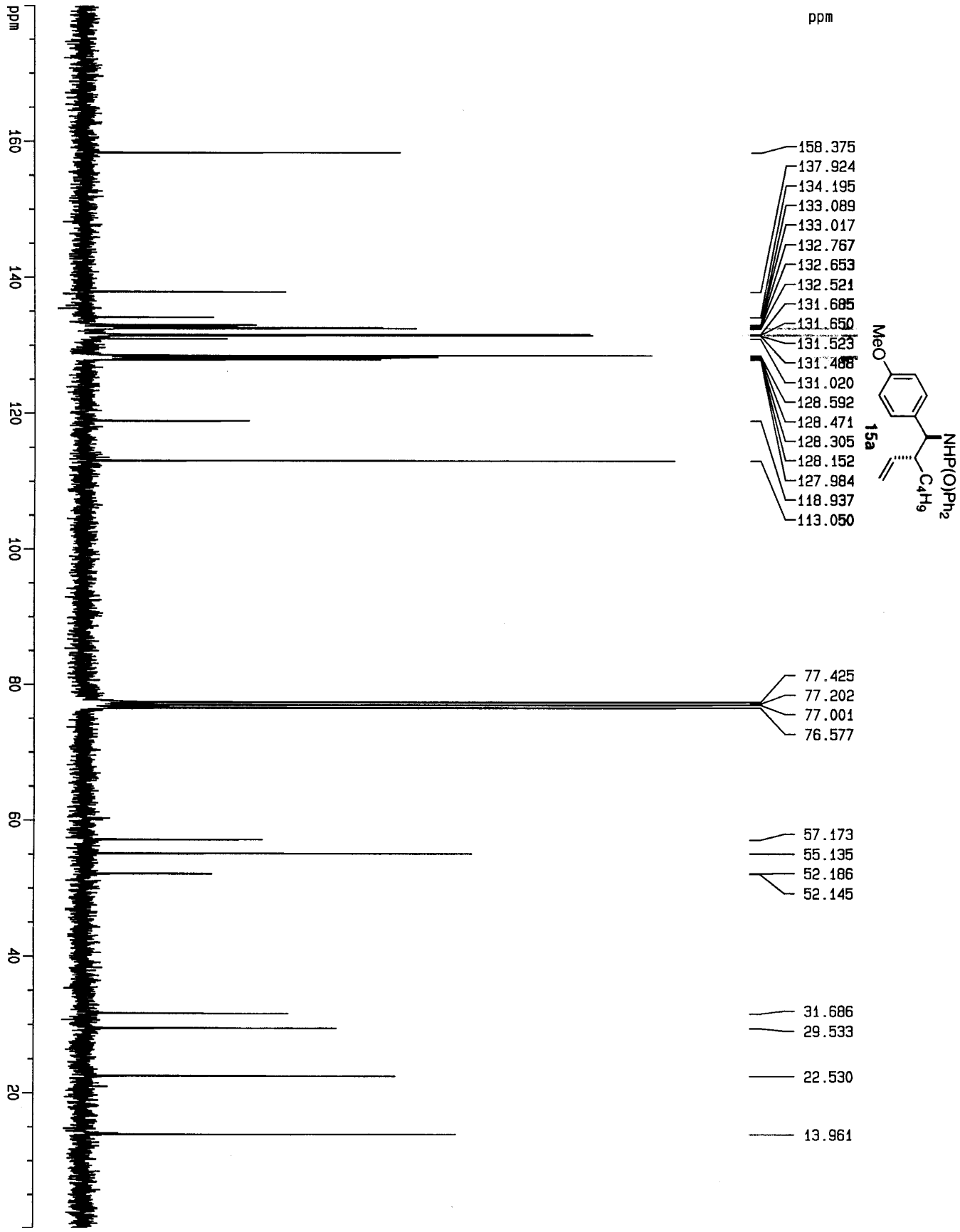
C4H9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20
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131.685
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131.523
131.488
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128.592
128.471
128.305
128.152
127.984
118.937
113.050
77.425
77.202
77.001
76.577
57.173
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31.686
29.533
22.530
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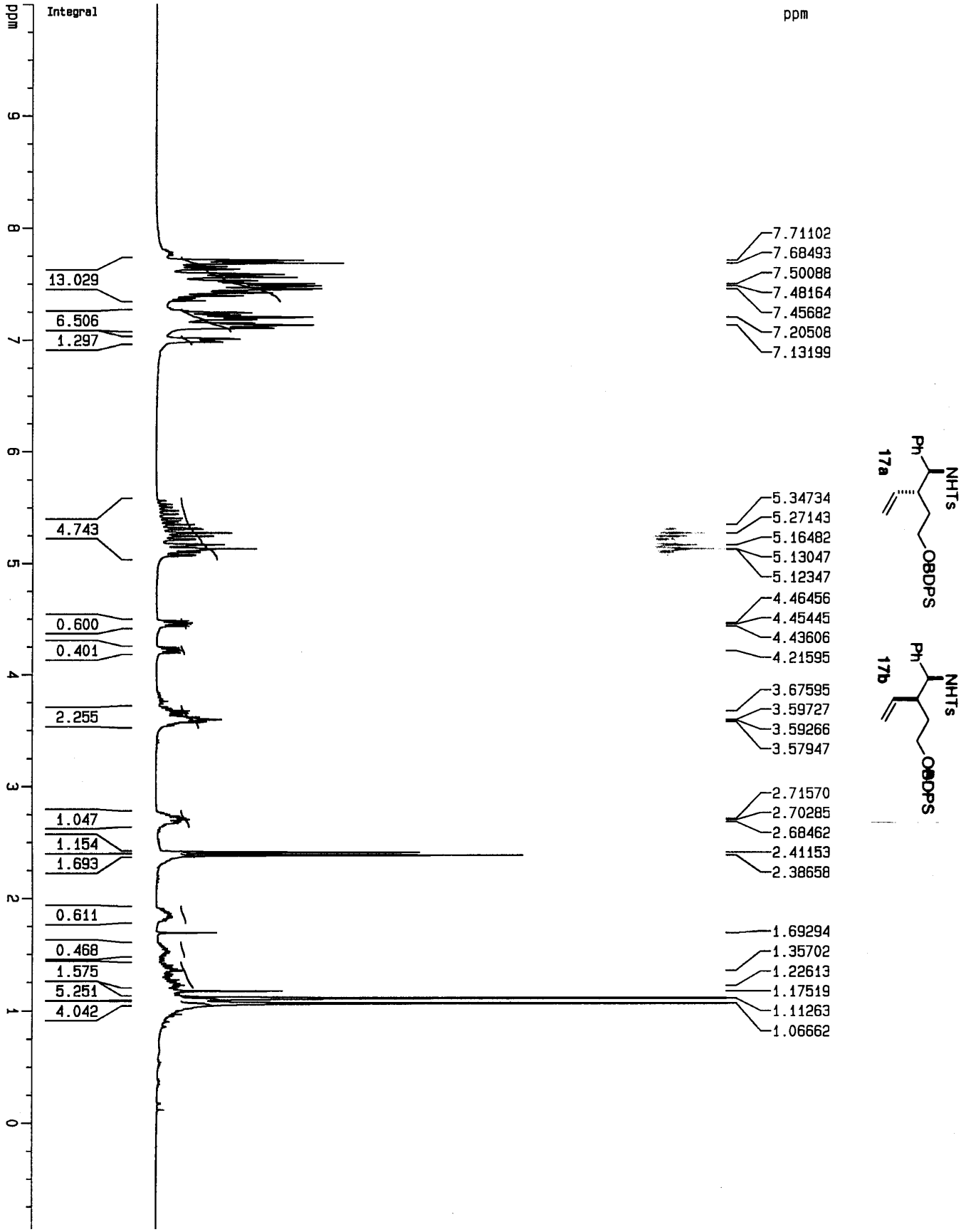
MeO
15a
NHP(O)Ph2
C4H9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1.154
1.693
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0.468
1.575
5.251
4.042
ppm
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7.48164
7.45682
7.20508
7.13199
5.34734
5.27143
5.16482
5.13047
5.12347
4.46456
4.45445
4.43606
4.21595
3.67595
3.59727
3.59266
3.57947
2.71570
2.70285
2.68462
2.41153
2.38658
1.69294
1.35702
1.22613
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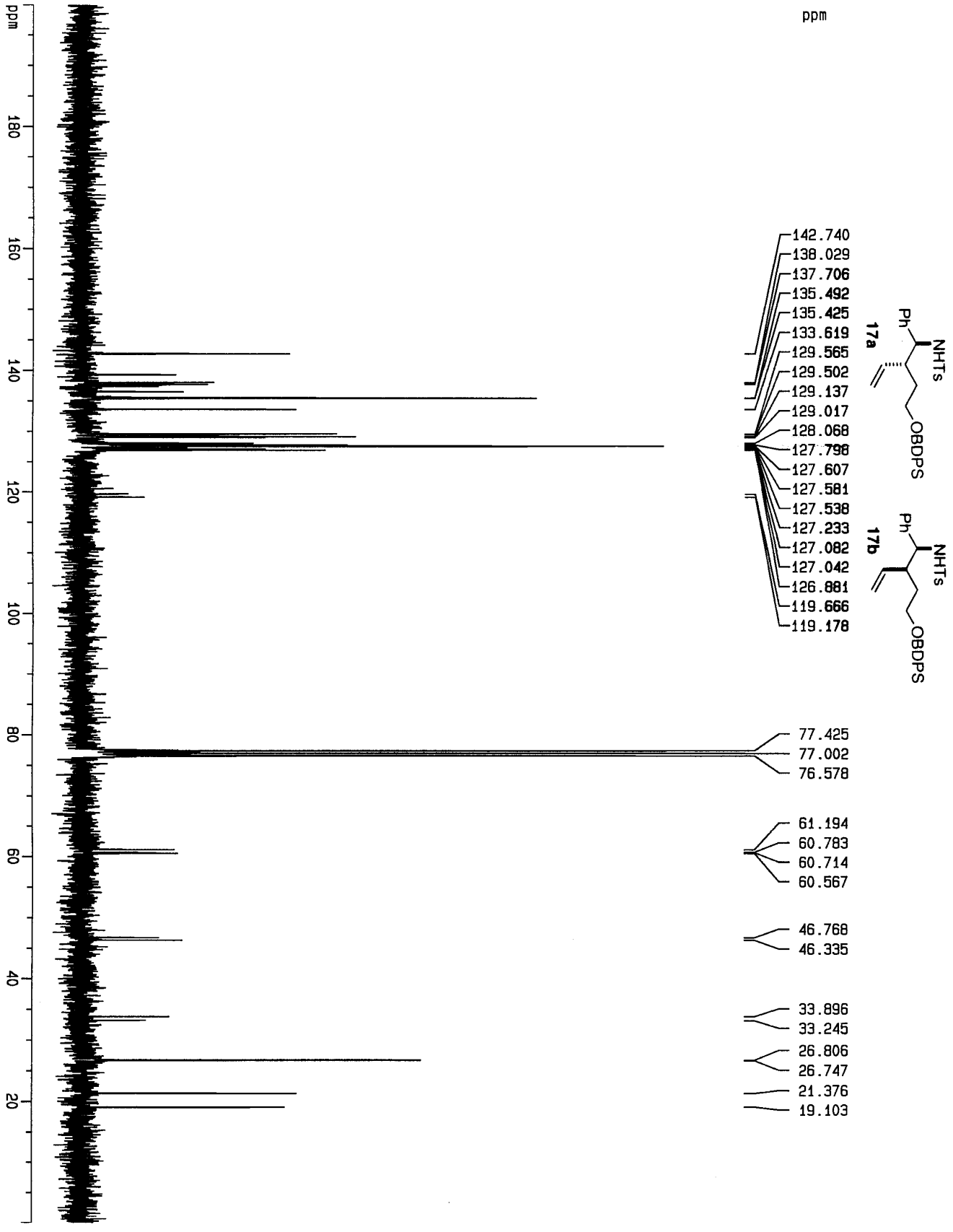
1.11263
1.06662
17a
Ph
NHTs
OBDPS
17b
Ph
NHTs
OBDPS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80
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40
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135.425
133.619
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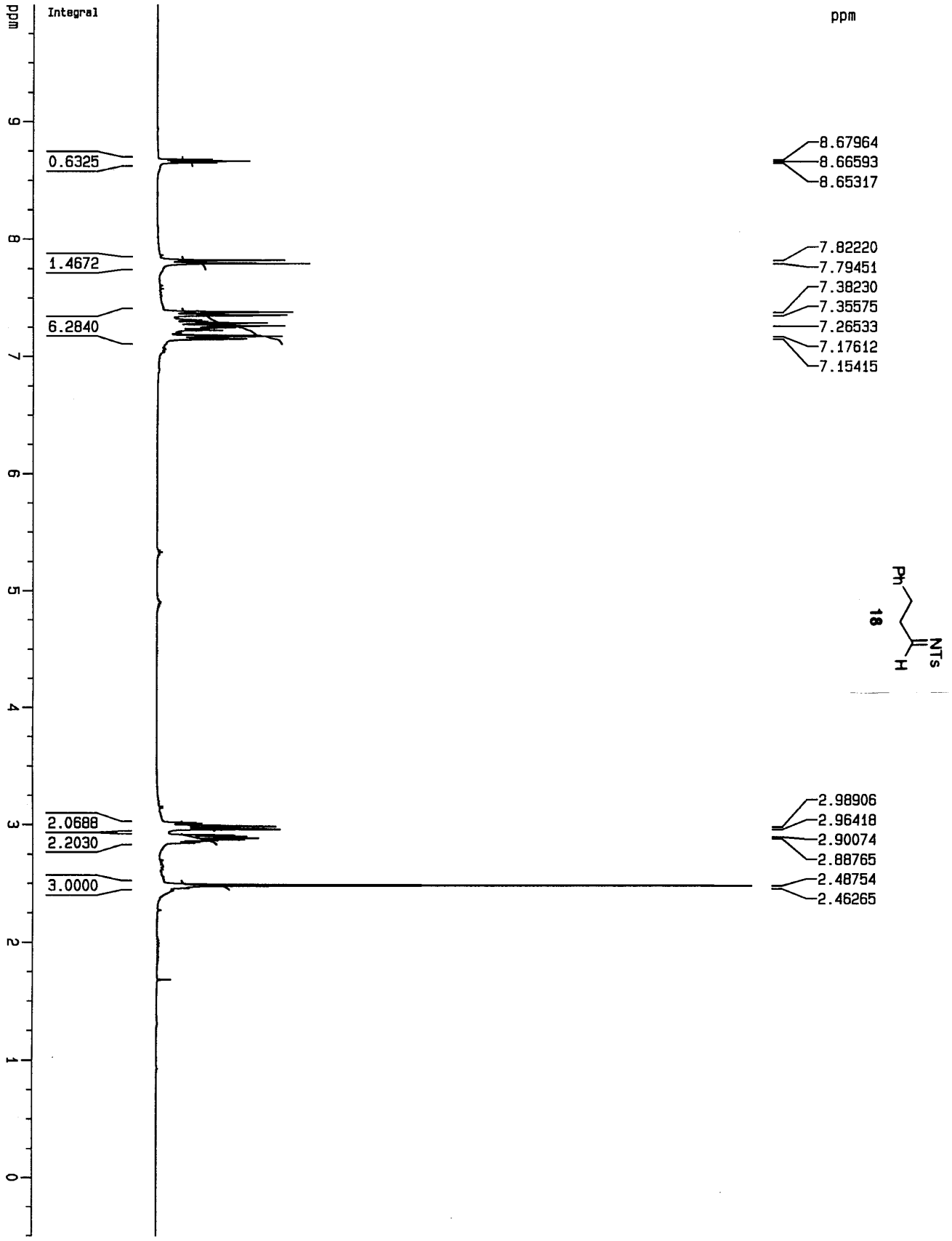
129.502
129.137
129.017
128.068
127.798
127.607
127.581
127.538
127.233
127.082
127.042
126.881
119.666
119.178
77.425
77.002
76.578
61.194
60.783
60.714
60.567
46.768
46.335
33.896
33.245
26.806
26.747
21.376
19.103
17a
Ph
NHTs
OBDPS
17b
Ph
NHTs
OBDPS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8.66593
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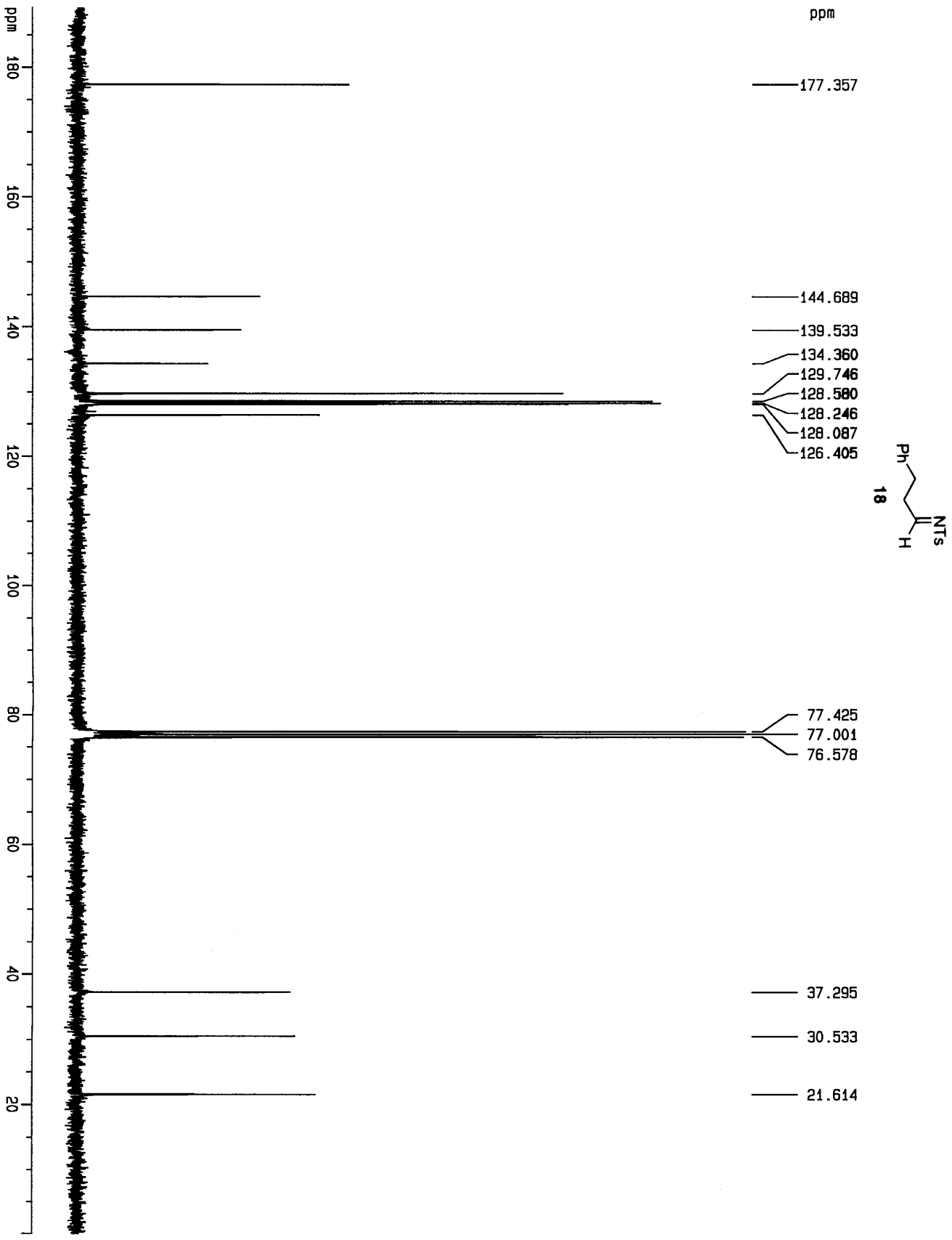
7.79451
7.38230
7.35575
7.26533
7.17612
7.15415
2.98906
2.96418
2.90074
2.88765
2.48754
2.46265
18
Ph
NTs
H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140
120
100
80
60
40
20
ppm
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134.360
129.746
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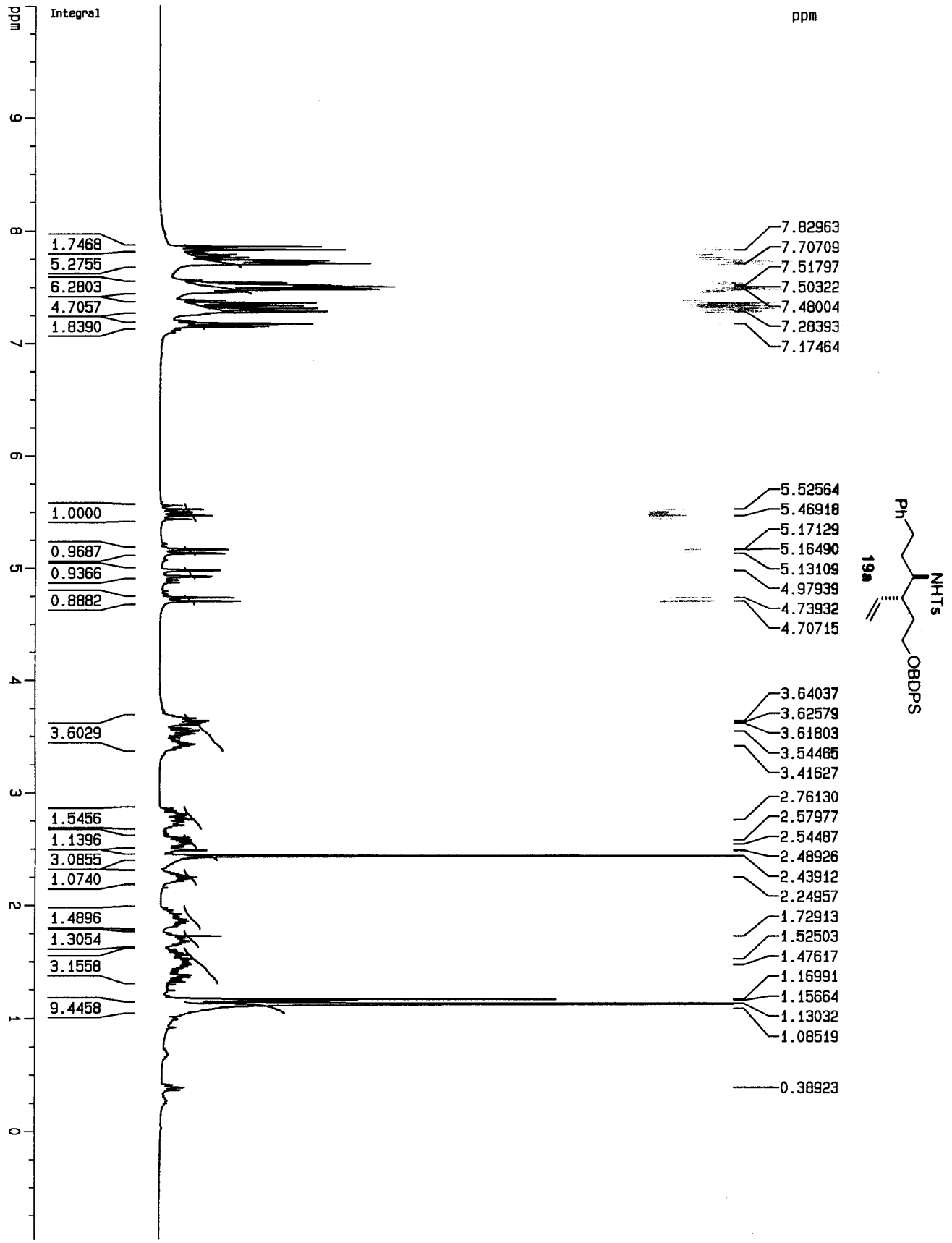
128.246
128.087
126.405
77.425
77.001
76.578
37.295
30.533
21.614
Ph
NTs
H
18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1.0000
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0.9366
0.8882
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1.5456
1.1396
3.0855
1.0740
1.4896
1.3054
3.1558
9.4458
7.82963
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7.51797
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7.28393
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5.13109
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2.43912
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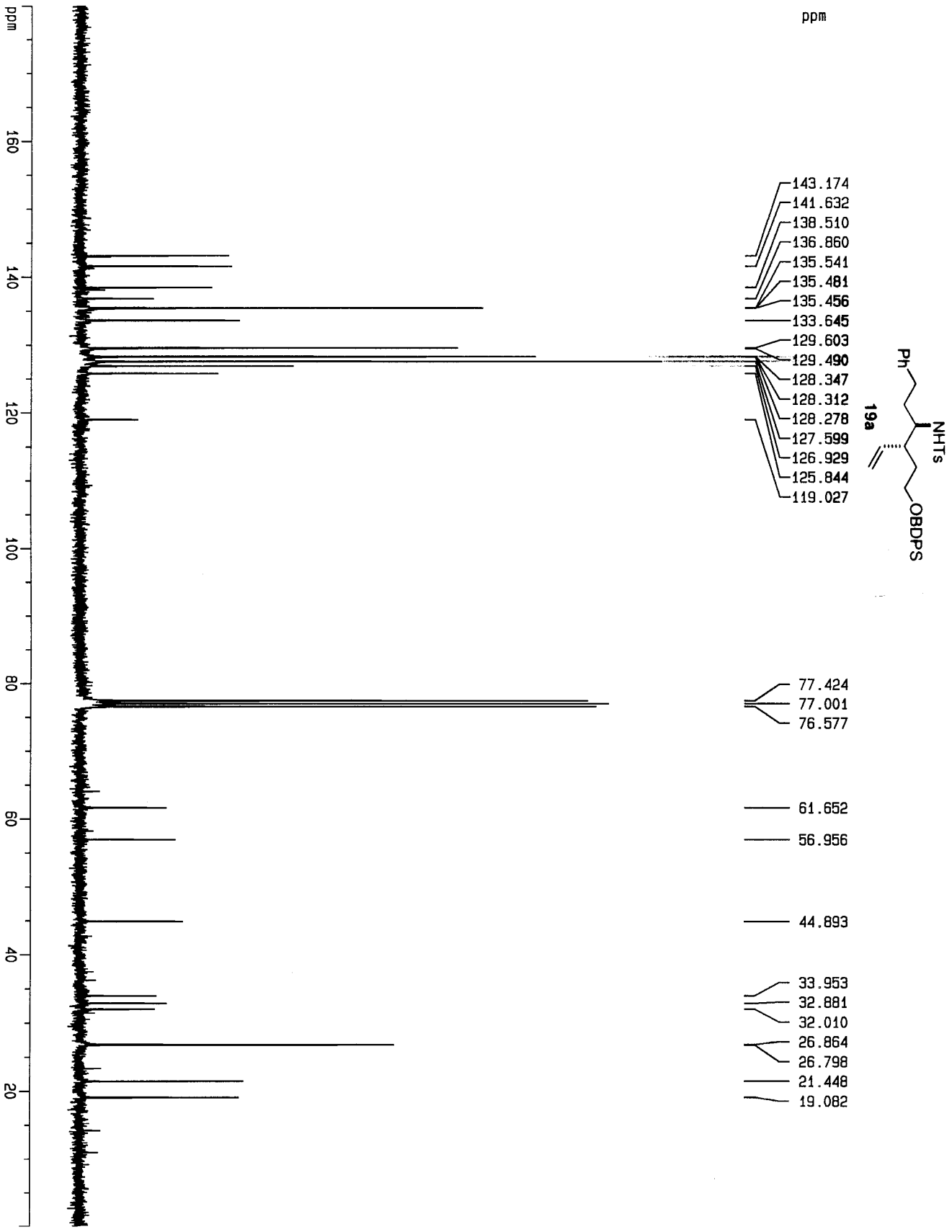
NHTs
OBDPS
19a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129.490
128.347
128.312
128.278
127.599
126.929
125.844
119.027
77.424
77.001
76.577
61.652
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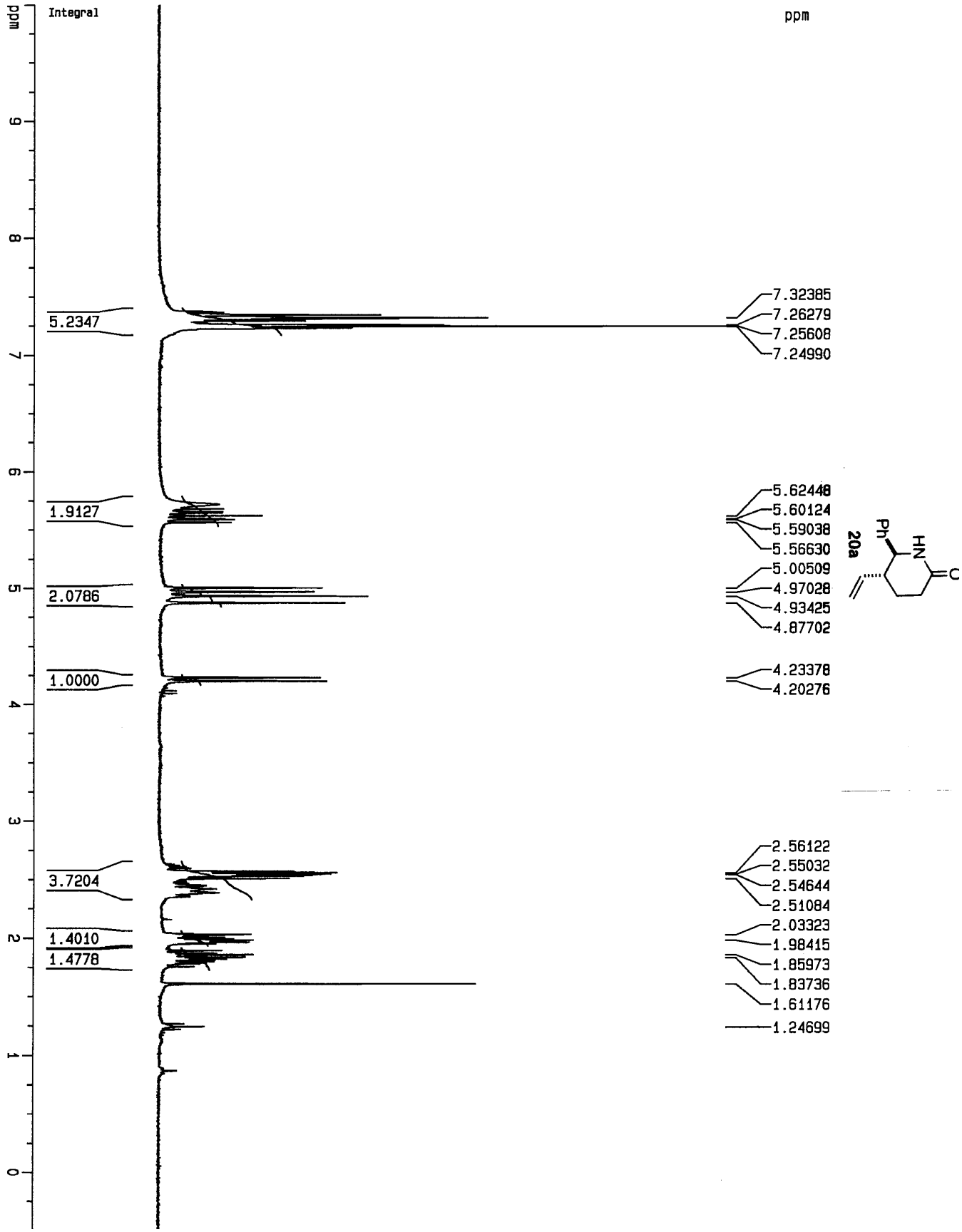
OBDPS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5.00509
4.97028
4.93425
4.87702
4.23378
4.20276
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HN
O
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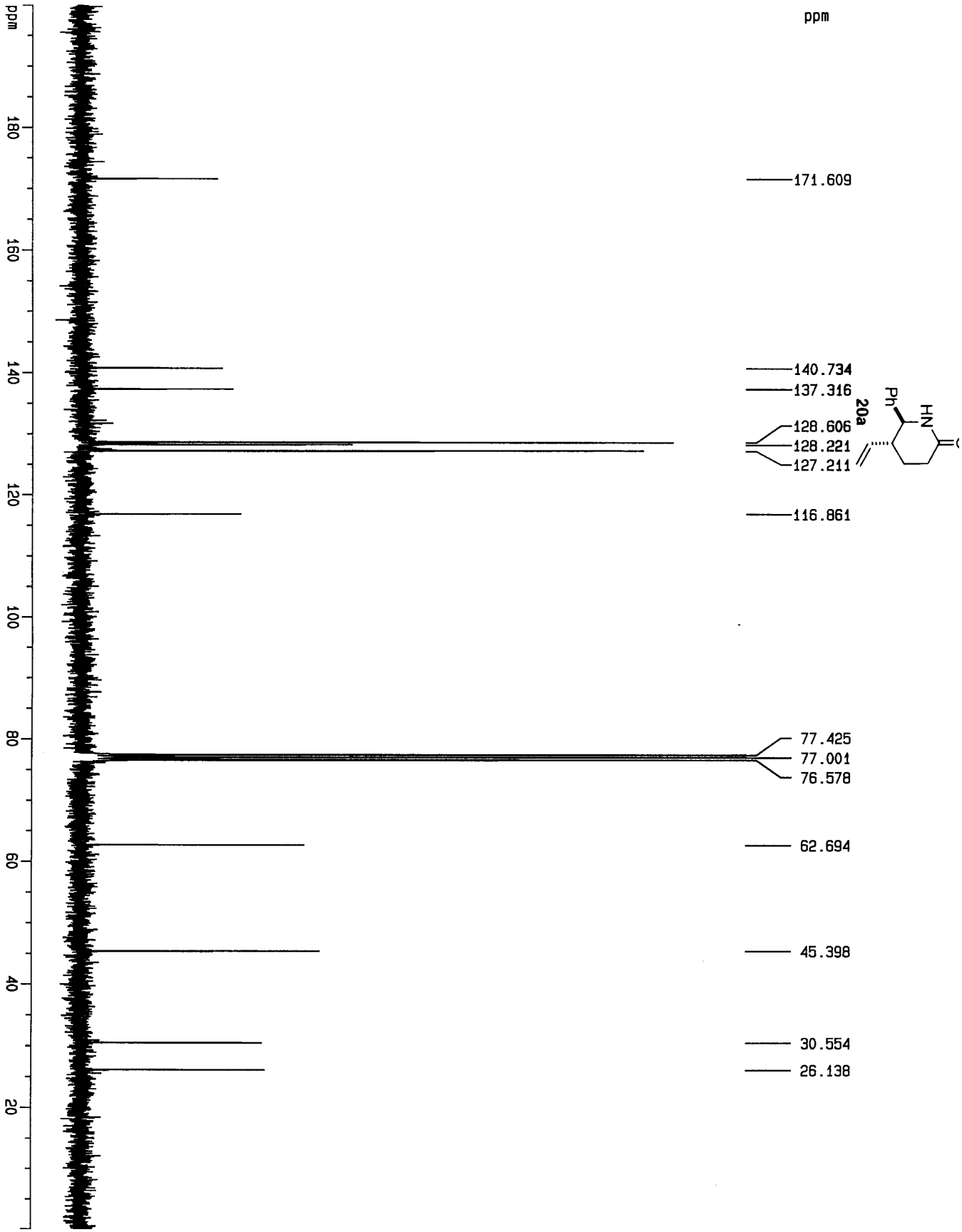
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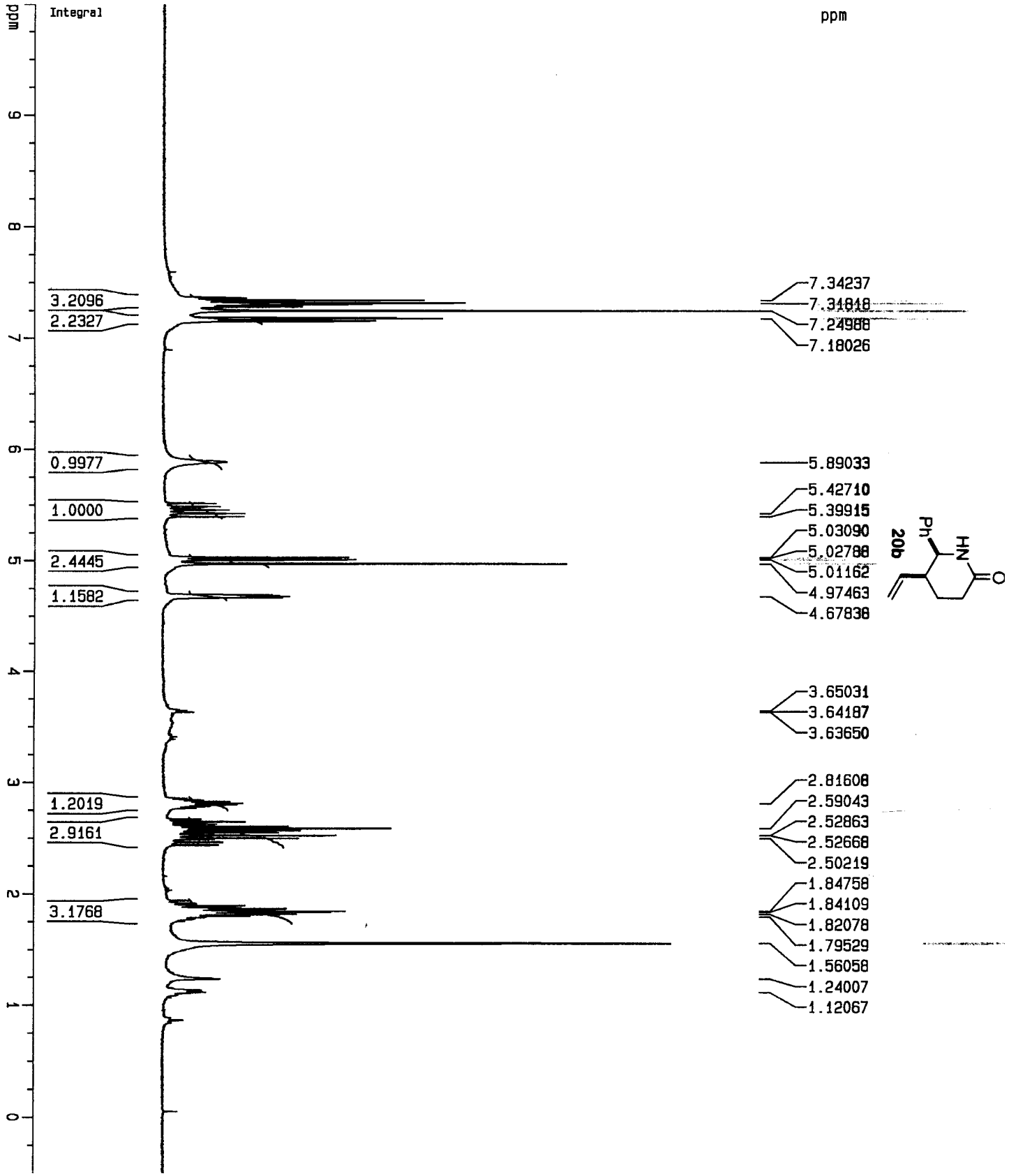
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7.31818
7.24988
7.18026
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5.42710
5.39915
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5.02788
5.01162
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HN
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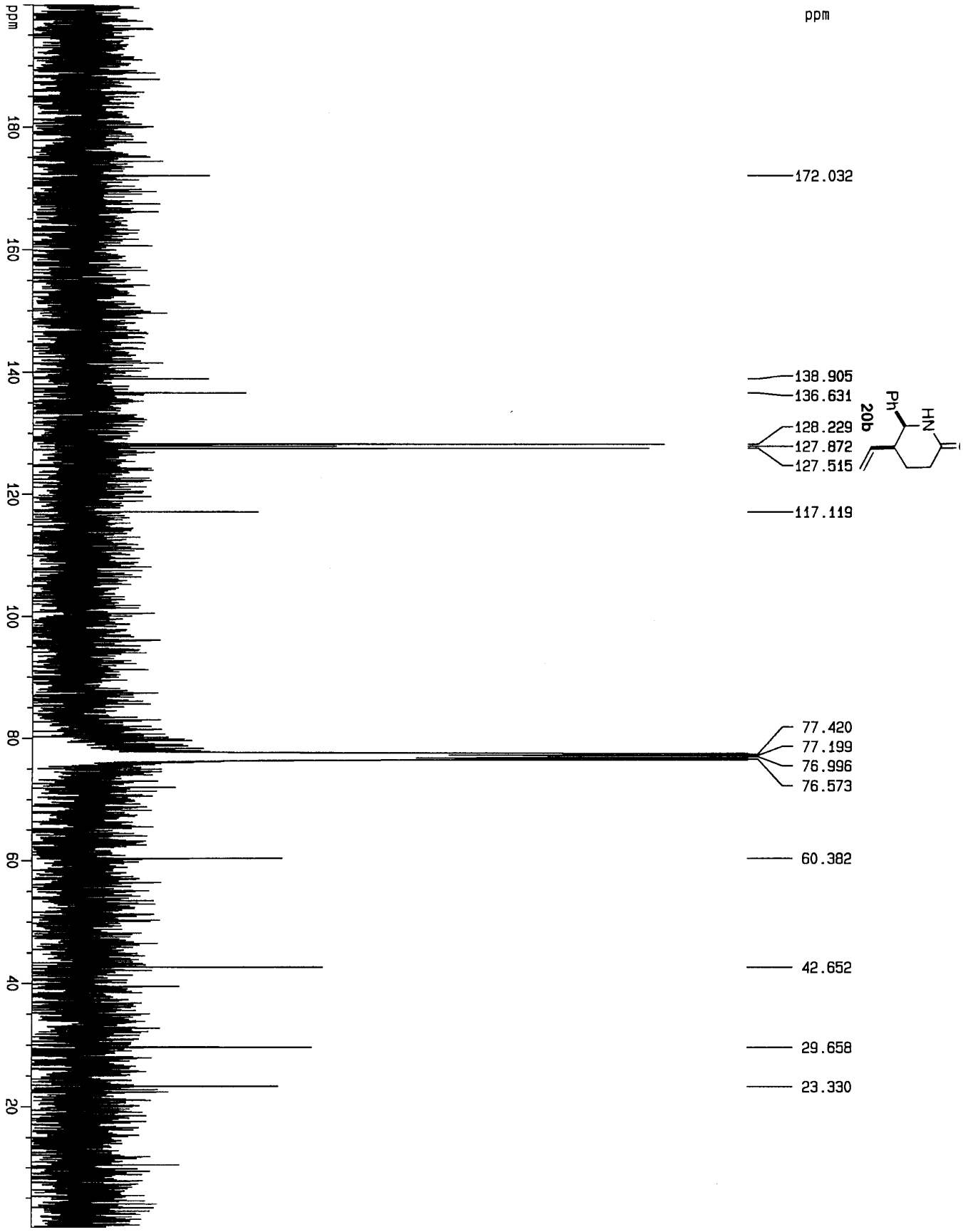
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77.199
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76.573
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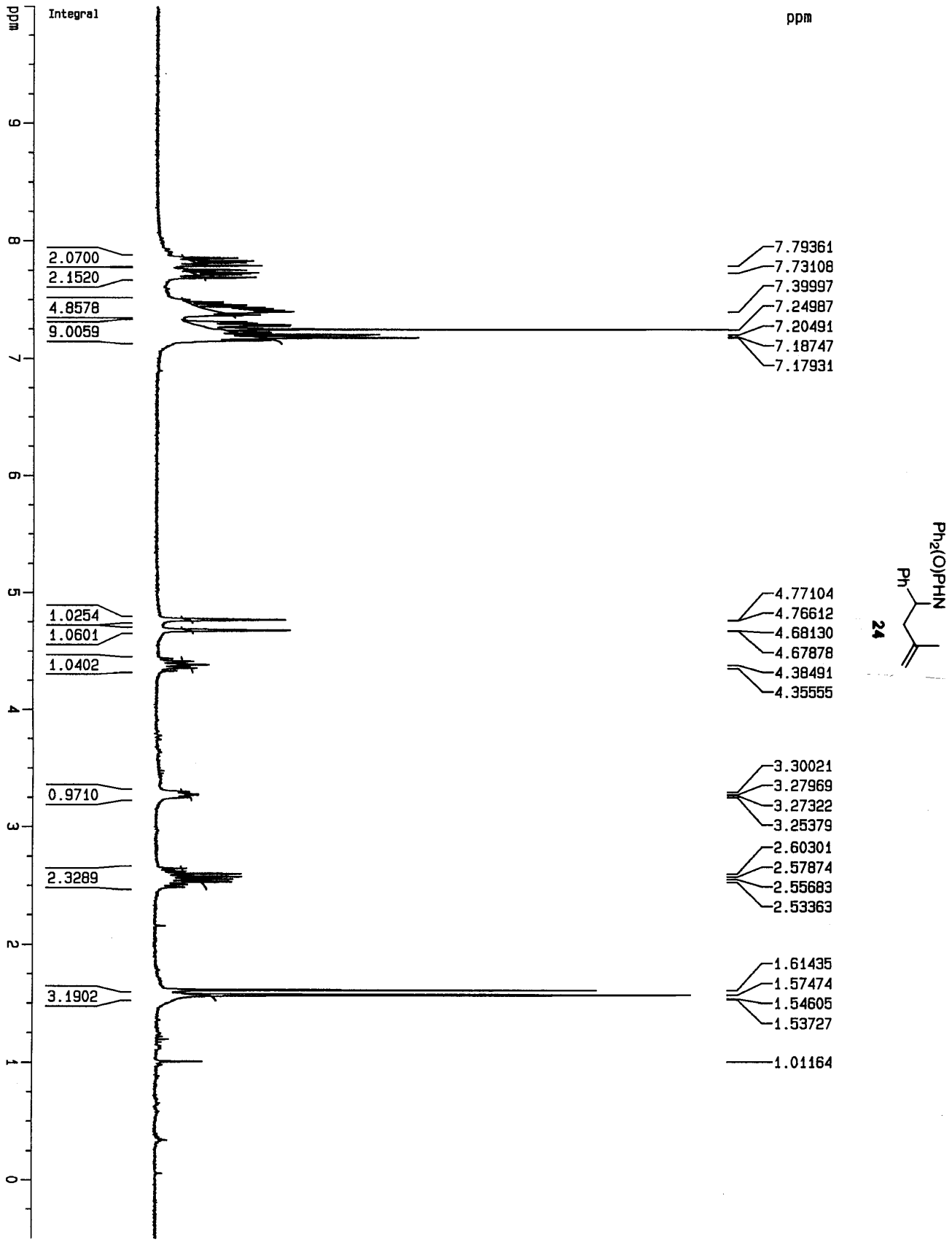
HN
20b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7.73108
7.39997
7.24987
7.20491
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3.27969
3.27322
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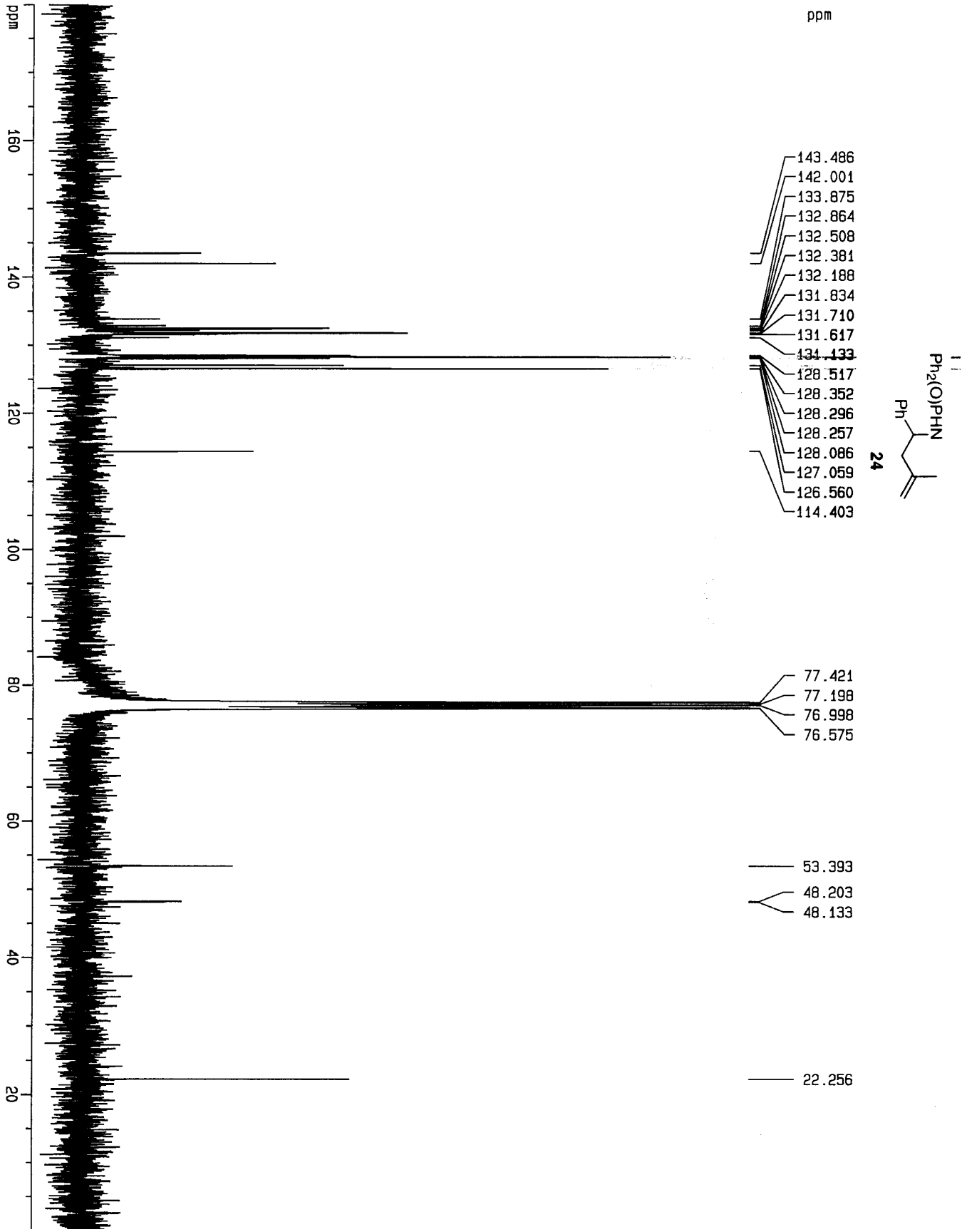
Ph2(O)PHN
Ph
24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131.617
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128.352
128.296
128.257
128.086
127.059
126.560
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77.198
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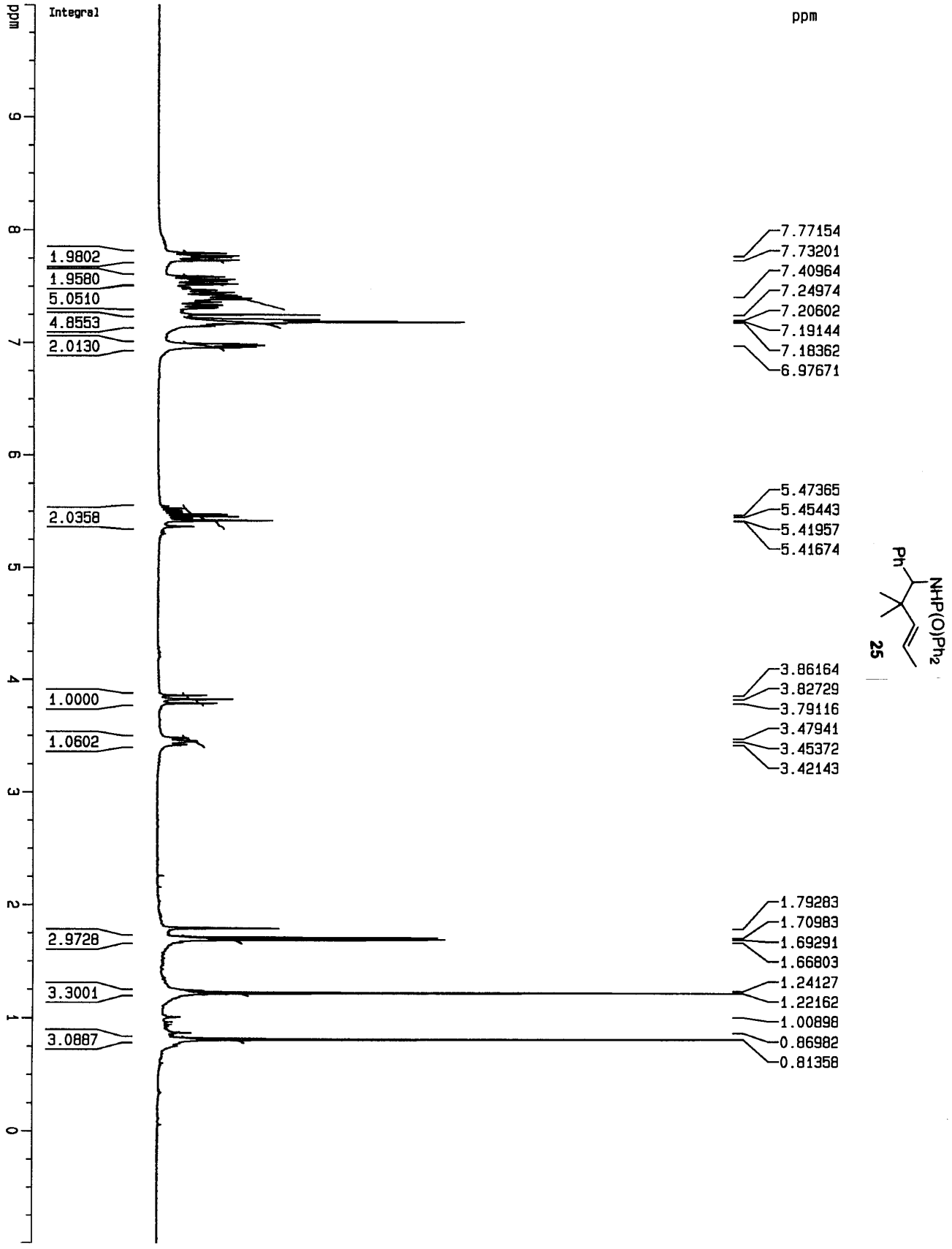
53.393
48.203
48.133
22.256
Ph
Ph2(O)PHN
24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3.0887
ppm
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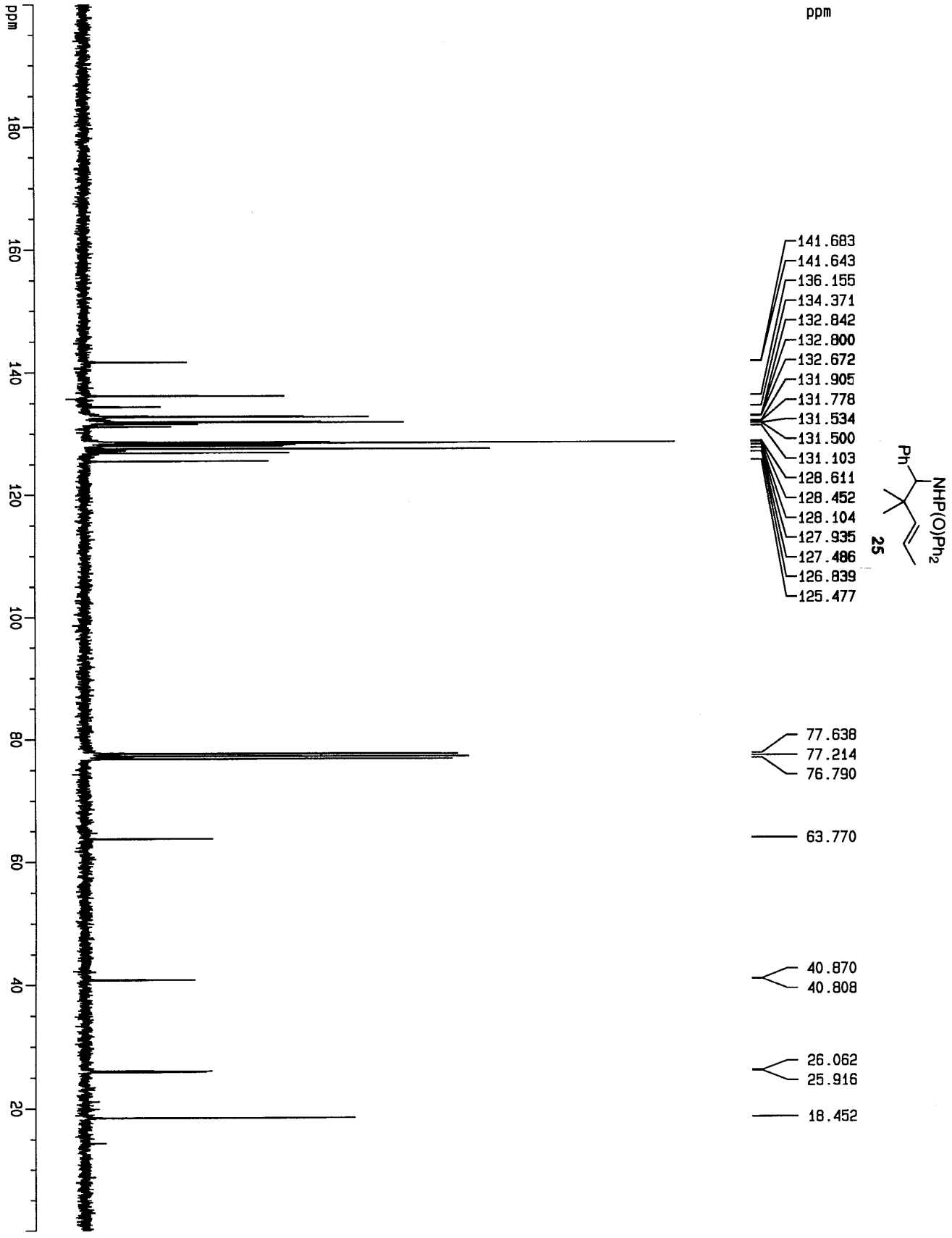
NHP(O)Ph2
25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128.452
128.104
127.935
127.486
126.839
125.477
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NHP(O)Ph2
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77.214
76.790
63.770
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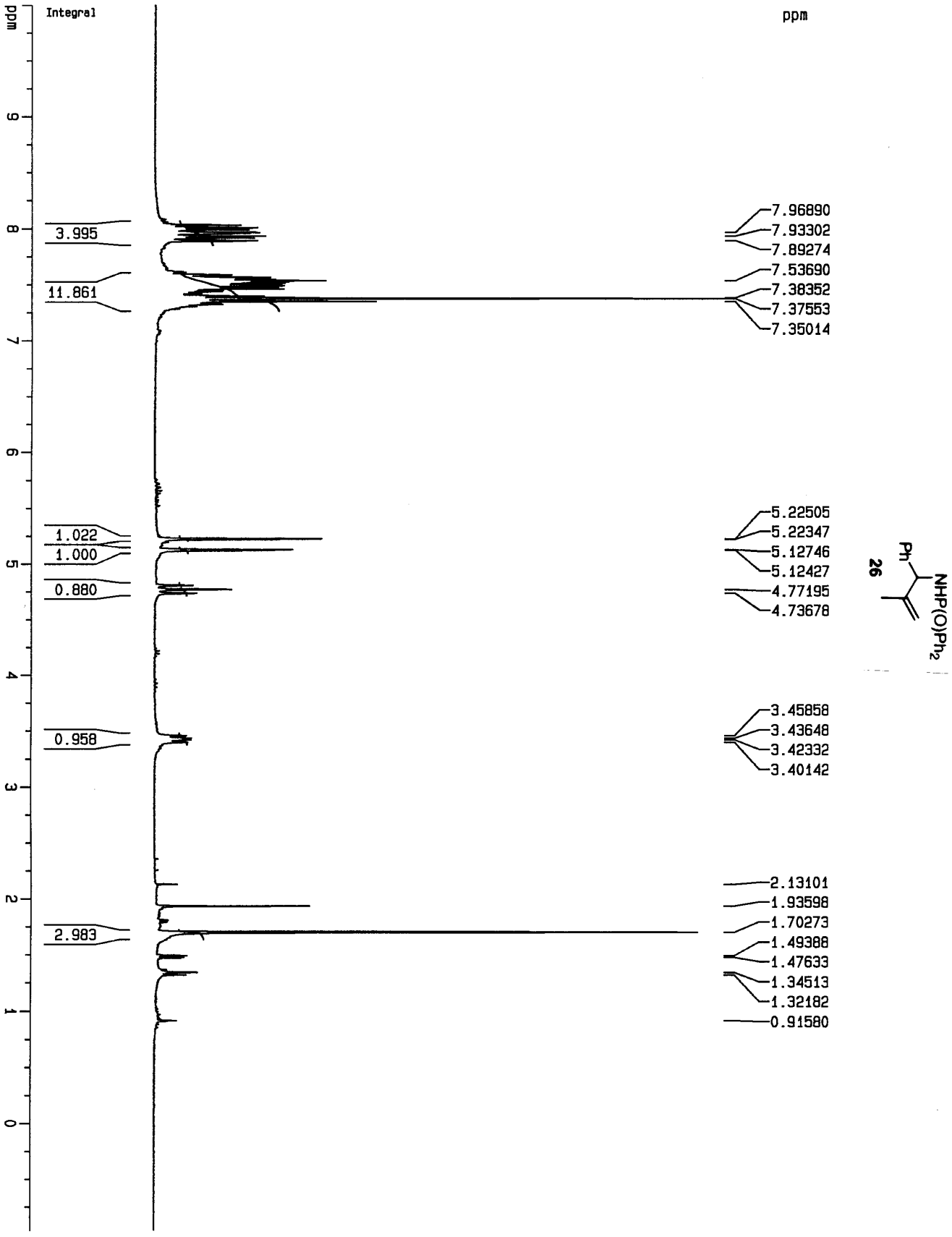
40.808
26.062
25.916
18.452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7.35014
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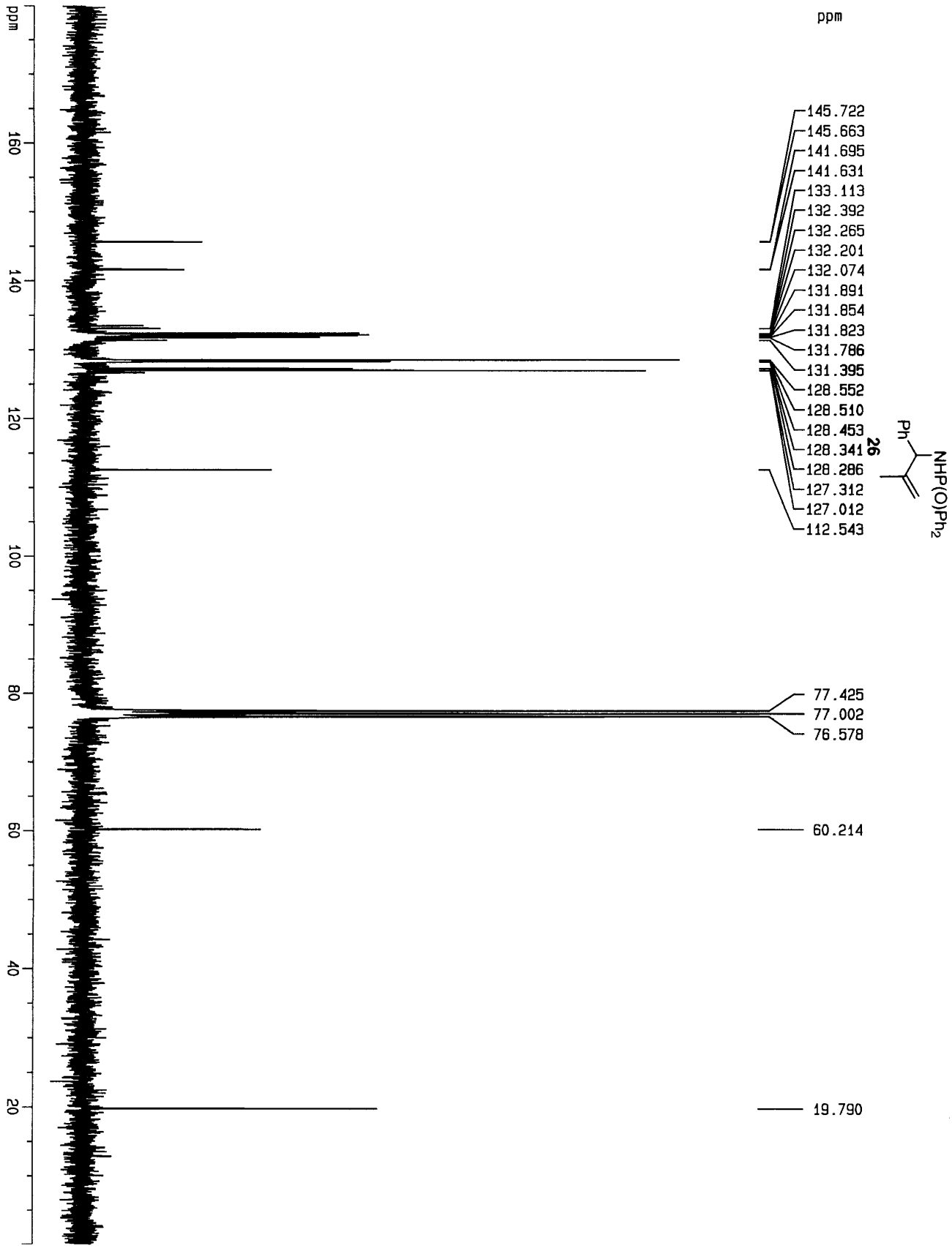
Ph
NHP(O)Ph2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132.074
131.891
131.854
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131.786
131.395
128.552
128.510
128.453
128.341
128.286
127.312
127.012
112.543
26
Ph
NHP(O)Ph2
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19.790