SUPPLEMENTARY MATERIAL

Synthesis of Homoallylic Amines by Hydrozirconation-Imine Addition of Allenes

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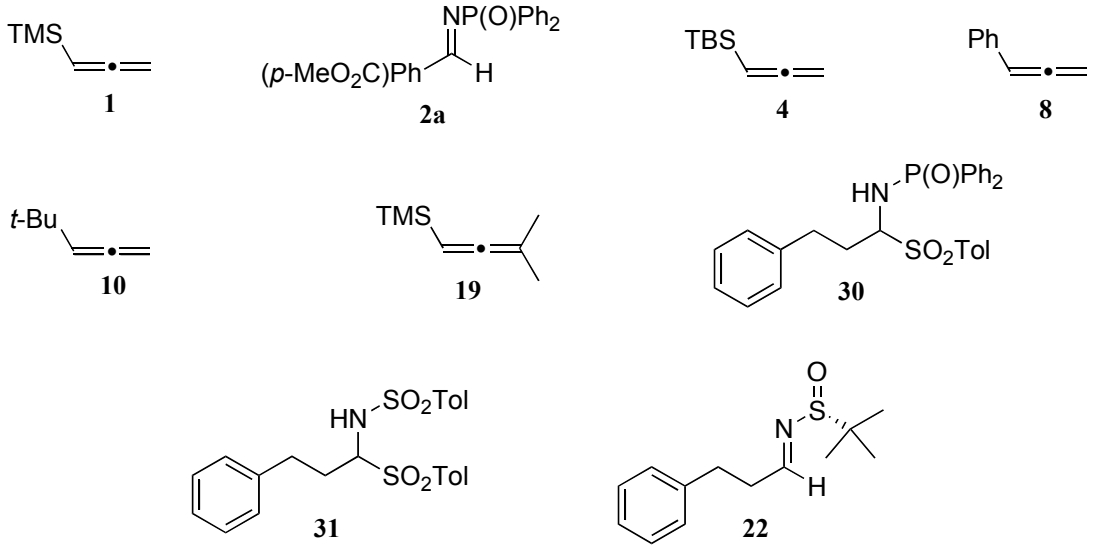
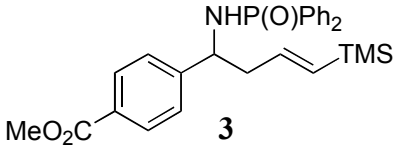
Experimental procedures and spectral data for all new compounds, including copies of ^{1}H and ^{13}C NMR spectra.

General. All reactions were performed 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, Et_3N was distilled from CaH_2, and CH_2Cl_2 and toluene were purified using an alumina filtration system. Me_2Zn, Et_2Zn, and allene **15** and **17** were purchased from Aldrich Company. Cp_2ZrHCl,[1] imines **2a**[2] and **22**,[3] sulfinyl adducts **30**[4] and **31**,[5] and allenes **1**,[6] **4**,[6] **8**,[7] **10**,[7] and **19**[8] were prepared according to literature procedures.



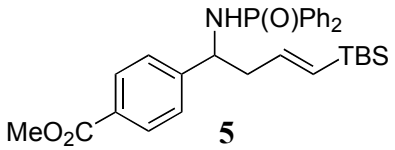
Reactions were monitored by TLC analysis (EM Science pre-coated silica gel 60 F_{254} plates, 250 μm layer thickness) and visualization was accomplished with a 254 nm UV light and by staining with a PMA solution (5 g of phosphomolybdic acid in 100 mL of 95% EtOH), *p*-anisaldehyde solution (2.5 mL of *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 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 $CDCl_3$ unless otherwise noted. Chemical shifts were reported in parts per million with the residual solvent peak

used as an internal standard. ^{1}H NMR spectra were run at 300 MHz and are tabulated as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), number of protons, and coupling constant(s). ^{13}C NMR spectra were run at 76 MHz using a proton-decoupled pulse sequence with a d_1 of 3 sec, and are tabulated by observed peak. Mass spectra were obtained on a Micromass Autospec double focusing instrument.



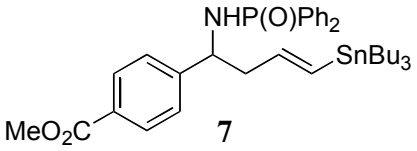
3

(*E*)-Methyl 4-[1-(diphenylphosphinamido)-4-(trimethylsilyl)but-3-enyl]benzoate (3). General Protocol A. A solution of 23.2 mg (0.207 mmol) of allene **1** in 1 mL of CH_2Cl_2 was treated at –78 °C with 53.4 mg (0.207 mmol) of Cp_2ZrHCl and allowed to warm to room temperature over 20 min. After approximately 90% of the CH_2Cl_2 was removed under reduced pressure, 1 mL of toluene was added. The resulting red solution was cooled to –78 °C, and treated with 104 μL (0.207 mmol) of Me_2Zn (2.0 M in toluene) and 50.0 mg (0.138 mmol) of imine **2a**. The reaction mixture was warmed to room temperature and stirred for 3 h, quenched with saturated aqueous $NaHCO_3$, extracted with EtOAc (3x), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by chromatography on SiO_2 (7:3, EtOAc:hexanes) to yield 51.6 mg (78%) of **3** as a colorless solid: mp 147-148 °C (CH_2Cl_2); IR (KBr) 3169, 2952, 1721, 1611, 1437, 1280, 1186, 1110, 838, 697 cm^{-1}; ^{1}H NMR (CD_2Cl_2) δ 7.93 (d, 2 H, *J* = 8.3 Hz), 7.88-7.70 (m, 4 H), 7.58-7.41 (m, 4 H), 7.40-7.30 (m, 2 H), 7.28 (d, 2 H, *J* = 8.3 Hz), 5.87 (dt, 1 H, *J* = 18.5, 6.3 Hz), 5.75 (d, 1 H, *J* = 18.6 Hz), 4.34 (tt, 1 H, *J* = 9.4, 6.5 Hz), 3.88 (s, 3 H), 4.46 (dd, 1 H, *J* = 9.4, 6.8 Hz), 2.73-2.56 (m, 2 H), 0.00 (s, 9 H); ^{13}C NMR (CD_2Cl_2) δ 166.59, 148.70, 141.35, 135.66, 133.97, 133.67, 132.11, 131.99, 131.84, 131.72, 129.38, 129.02, 128.52, 128.35, 128.18, 126.54, 54.34, 51.76, 46.55, 46.48, -1.80; ESIMS *m/z* 500 ([M+Na]$^{+}$, 100), 478 ([M+H]$^{+}$, 33); HRMS (ESI) *m/z* calcd for $C_{27}H_{32}NO_3PSiNa$ 500.1787 (M+Na), found 500.1778.



5

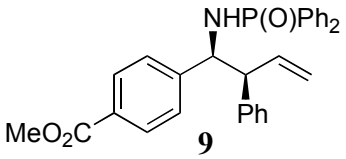
(*E*)-Methyl 4-[4-(*tert*-butyldimethylsilyl)-1-(diphenylphosphinamido)-but-3-enyl]benzoate (5). According to the General Protocol A, 31.9 mg (0.207 mmol) of allene **4**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 104 μL (0.207 mmol) of Me_2Zn (2.0 M in toluene), and 50.0 mg (0.138 mmol) of imine **2a** afforded 57.2 mg (80%) of **5** as a colorless solid: mp 114-115 °C (CH_2Cl_2); IR (KBr) 3167, 2951, 2854, 1723, 1611, 1437, 1279, 1187, 1110, 828, 725, 698 cm^{-1}; ^1H NMR (CD_2Cl_2) δ 7.93 (d, 2 H, J = 8.3 Hz), 7.89-7.70 (m, 4 H), 7.58-7.40 (m, 4 H), 7.40-7.30 (m, 2 H), 7.28 (d, 2 H, J = 8.3 Hz), 5.83 (dt, 1 H, J = 18.6, 6.4 Hz), 5.73 (d, 1 H, J = 18.6 Hz), 4.38 (tt, 1 H, J = 9.8, 6.5 Hz), 3.88 (s, 3 H), 3.50 (dd, 1 H, J = 9.4, 6.3 Hz), 2.81-2.95 (m, 2 H), 0.753 (s, 9 H), -0.046 (s, 3 H), -0.072 (s, 3 H); ^{13}C NMR (CD_2Cl_2) δ 167.13, 149.08, 143.02, 134.49, 134.18, 132.67, 132.55, 132.35, 129.95, 129.60, 129.08, 128.91, 128.71, 127.14, 54.79, 52.28, 47.16, 47.11, 26.55, 16.60, -5.91, -6.08; ESIMS *m/z* 542 ($[M+Na]^+$, 100), 520 ($[M+H]^+$, 45); HRMS (ESI) *m/z* calcd for $C_{30}H_{38}NO_3PSiNa$ 542.2256 (M+Na), found 542.2283.



7

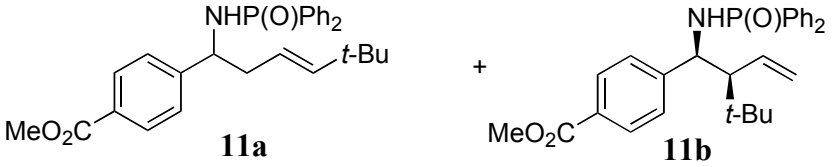
(*E*)-Methyl 4-[1-(diphenylphosphinamido)-4-(tributylstannyl)but-3-enyl]benzoate (7). According to the General Protocol A, 68.1 mg (0.207 mmol) of allene **6**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 104 μL (0.207 mmol) of Me_2Zn (2.0 M in toluene), and 50.0 mg (0.138 mmol) of imine **2a** afforded 77.6 mg (81%) of **7** as a colorless solid: mp 63-64 °C (CH_2Cl_2); IR (KBr) 3168, 2955, 2925, 2871, 2852, 1723, 1437, 1278, 1186, 1109, 696 cm^{-1}; ^1H NMR δ 7.98 (d, 2 H, J = 8.3 Hz), 7.95-7.73 (m, 4 H), 7.58-7.41 (m, 4 H), 7.38-7.26 (m, 2 H), 7.28 (d, 2 H, J = 7.3 Hz), 6.04 (d, 1 H, $^2J_{H\text{-}Sn}$ = 74.0, $J_{H\text{-}H}$ = 16.5 Hz), 5.64 (dt, 1 H, J = 18.8, 7.4 Hz), 4.53-4.41 (m, 1 H), 3.92 (s, 3 H), 3.49 (dd, 1 H, J = 9.6, 5.8 Hz), 2.90-2.65 (m, 2 H), 1.56-1.17 (m, 12 H), 0.97-0.70 (m, 15

H); ^{13}C NMR δ 166.87, 148.46, 142.92, 134.80, 133.12, 132.42, 132.29, 131.83, 131.39, 129.64, 128.98, 128.62, 128.45, 128.24, 126.65, 54.13, 51.95, 47.55, 47.50, 29.03, 27.16, 13.56, 9.49; ESIMS *m/z* 718 ([M+Na]$^+$, 85), 696 ([M+H]$^+$, 100); HRMS (ESI) *m/z* calcd for $C_{36}H_{50}NO_3PSnNa$ 718.2448 (M+Na), found 718.2469.

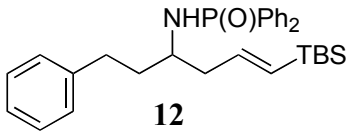


9

Methyl 4-[(1*S,2*S**)-1-(diphenylphosphinamido)-2-phenylbut-3-enyl]benzoate (9).** According to the General Protocol A, 24.0 mg (0.207 mmol) of allene **8**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 104 μL (0.207 mmol) of Me_2Zn (2.0 M in toluene), and 50.0 mg (0.138 mmol) of imine **2a** (1.5 h reaction time) afforded 55.1 mg (83%) of **9** as a colorless solid: mp 192-193 °C (CH_2Cl_2); IR (KBr) 3176, 3059, 2924, 2854, 1721, 1437, 1281, 1184, 1109, 698 cm^{-1}; ^{1}H NMR δ 7.88 (d, 2 H, *J* = 8.3 Hz), 7.70-7.56 (m, 4 H), 7.55-7.47 (m, 1 H), 7.46-7.37 (m, 3 H), 7.34-7.23 (m, 5 H), 7.08-7.02 (m, 2 H), 6.98 (d, 2 H, *J* = 8.4 Hz), 6.01 (dt, 1 H, *J* = 19.6, 10.2 Hz), 5.18 (d, 1 H, *J* = 10.2 Hz), 5.15 (d, 1 H, *J* = 17.4 Hz), 4.56 (ddd, 1 H, *J* = 9.8, 6.3 Hz), 3.94 (s, 3 H), 3.85 (dd, 1 H, *J* = 9.1, 6.3 Hz), 3.54 (dd, 1 H, *J* = 9.7, 6.2 Hz); ^{13}C NMR δ 166.90, 146.23, 139.91, 136.15, 133.70, 132.97, 132.39, 132.26, 131.73, 131.60, 129.11, 128.60, 128.37, 128.28, 128.11, 127.72, 127.12, 118.83, 59.36, 57.95, 57.89, 51.95; ESIMS *m/z* 504 ([M+Na]$^+$, 100), 482 ([M+H]$^+$, 98); HRMS (ESI) *m/z* calcd for $C_{30}H_{28}NO_3PNa$ 504.1705 (M+Na), found 504.1703.

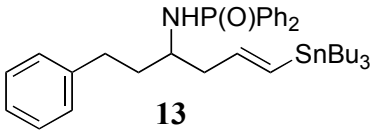


(*E*)-Methyl 4-[1-(diphenylphosphinamido)-5,5-dimethylhex-3-enyl]benzoate (11a) and methyl 4-[(1*S,2*S**)-2-*tert*-butyl-1-(diphenylphosphinamido)but-3-enyl]benzoate (11b).** According to the General Protocol A, 19.9 mg (0.207 mmol) of allene **10**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 104 μL (0.207 mmol) of Me_2Zn (2.0 M in toluene), and 50.0 mg (0.138 mmol) of imine **2a** (12 h reaction time) afforded 40.6 mg (64%) of a 1.5:1 mixture of **11a**:**11b**: IR (KBr) 3183, 3058, 2954, 1721, 1611, 1437, 1280, 1190, 1109, 1019, 972, 914, 725, 698 cm^{-1}; ^1H NMR δ 7.96-7.71 (m, 6 H), 7.55-7.37 (m, 4 H), 7.34-7.25 (m, 3.2 H), 7.12 (d, 0.8 H, *J* = 8.3 Hz), 5.87 (dt, 0.4 H, *J* = 16.9, 10.5 Hz), 5.53 (d, 0.6 H, *J* = 15.6 Hz), 5.39 (dd, 0.4 H, *J* = 10.4, 2.2 Hz), 5.36 (dd, 0.4 H, *J* = 16.6, 2.2 Hz), 5.09 (dt, 0.6 H, *J* = 15.7, 7.0 Hz), 4.54 (dt, 0.4 H, *J* = 10.8, 3.6 Hz), 4.38 (tt, 0.6 H, *J* = 10.0, 6.4 Hz), 3.93 (s, 1.2 H), 3.92 (s, 1.8 H), 3.75 (dd, 0.4 H, *J* = 10.4, 7.1 Hz), 3.47-3.37 (m, 0.6 H), 2.65-2.53 (m, 1.2 H), 2.47 (dd, 0.4 H, *J* = 11.2, 4.0 Hz), 0.91 (s, 5.6 H), 0.70 (s, 3.4 H); ^{13}C NMR δ 166.91, 166.84, 148.62, 148.55, 147.86, 147.74, 146.99, 134.85, 133.85, 133.58, 132.80, 132.64, 132.55, 132.42, 132.37, 132.24, 132.16, 131.90, 131.86, 131.80, 131.74, 131.62, 131.51, 131.07, 130.91, 130.15, 130.02, 129.54, 129.25, 128.78, 128.72, 128.57, 128.40, 128.36, 128.22, 128.19, 128.06, 126.53, 120.96, 118.76, 62.85, 62.81, 54.41, 53.93, 52.06, 52.03, 42.46, 42.39, 33.10, 32.65, 31.54, 30.89, 29.47, 29.25, 28.19, 25.22, 22.65, 22.61, 14.09; EIMS *m/z* 430 ([M-OCH_3]$^+$, 20), 364 (95), 201 (100); HRMS (EI) *m/z* calcd for $C_{27}H_{29}NO_2P$ 430.1936 (M-OCH_3), found 430.1920.



(*E*)-*N*-[6-(*tert*-Butyldimethylsilyl)-1-phenylhex-5-en-3-yl]-*P*,*P*-diphenylphosphinamide (12). General Protocol B. A solution of 23.2 mg (0.207

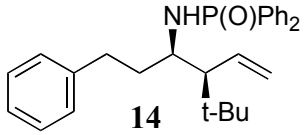
mmol) of allene **4** in 1 mL of CH_2Cl_2 was treated at –78 °C with 53.4 mg (0.207 mmol) of Cp_2ZrHCl and allowed to warm to room temperature over 20 min. After approximately 90% of the CH_2Cl_2 was removed under reduced pressure, 1 mL of toluene was added. The resulting red solution was cooled to –78 °C, treated with 173 μL (0.345 mmol) of Me_2Zn (2.0 M in toluene) and 67.6 mg (0.138 mmol) of sulfinyl adduct **30**, and allowed to stir at –78 °C for 15 min. The reaction mixture was then warmed to room temperature and stirred for an additional 3 h, quenched with saturated aqueous $NaHCO_3$, extracted with EtOAc (3x), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by chromatography on SiO_2 (6:4, EtOAc:hexanes) to yield 53.3 mg (79%) of **12** as a colorless solid: mp 109-110 °C (CH_2Cl_2); IR (KBr) 3180, 2926, 2854, 1438, 1186, 1122, 828, 697 cm^{-1}; ^1H NMR δ 8.02-7.85 (m, 4 H), 7.60-7.41 (m, 6 H), 7.32-7.21 (m, 2 H), 7.21-7.11 (m, 3 H), 6.05 (dt, 1 H, J = 18.5, 7.1 Hz), 5.83 (d, 1 H, J = 18.5 Hz), 3.38-3.20 (m, 1 H), 2.87 (dd, 1 H, J = 10.3, 6.3 Hz), 2.81-2.61 (m, 2 H), 2.61-2.41 (m, 2 H), 1.96-1.84 (m, 2 H), 0.81 (s, 9 H), 0.46 (s, 6 H); ^{13}C NMR δ 143.46, 141.82, 134.32, 134.10, 132.40, 132.26, 132.21, 132.17, 132.12, 132.04, 131.67, 131.64, 128.53, 128.36, 125.79, 50.83, 44.27, 44.21, 38.23, 38.17, 32.03, 26.44, 16.37, -5.98, -6.08; EIMS *m/z* 490 ($[M+H]^+$, 3), 432 (100), 335 (75), 201 (52); HRMS (EI) *m/z* calcd for $C_{30}H_{40}NOPSi$ 489.2617, found 489.2626.



13

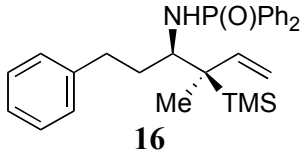
(*E*)-*P*,*P*-Diphenyl-*N*-[1-phenyl-6-(tributylstannyl)hex-5-en-3-yl]phosphinamide (13). According to the General Protocol B, 68.1 mg (0.207 mmol) of allene **6**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 173 μL (0.345 mmol) of Me_2Zn (2.0 M in toluene), and 67.6 mg (0.138 mmol) of sulfinyl adduct **30** afforded 72.1 mg (79%) of **13** as a colorless solid: mp 57.9-58.4 °C (CH_2Cl_2); IR (KBr) 3183, 3059, 2925, 1438, 1376, 1190, 1122, 991, 723, 698 cm^{-1}; ^1H NMR δ 8.00-7.82 (m, 4 H), 7.58-7.40 (m, 6 H), 7.29-7.20 (m, 2 H), 7.20-7.09 (m, 3 H), 6.09 (d, 1 H, $J_{H\text{-}H}$ = 18.8, $^2J_{H\text{-}Sn}$ = 75.0 Hz), 5.92 (dt, 1 H, J = 18.6, 6.6 Hz), 3.35-3.19 (m, 1 H), 2.88 (dd, 1 H, J = 10.6, 6.3 Hz), 2.82-2.61 (m, 2

H), 2.60-2.39 (m, 2 H), 1.95-1.82 (m, 2 H), 1.71-1.22 (m, 12 H), 1.10-0.75 (m, 15 H); ^{13}C NMR δ 144.15, 141.85, 134.11, 133.94, 133.24, 132.20, 132.12, 132.00, 131.67, 128.49, 128.32, 125.73, 50.65, 45.02, 44.97, 38.05, 37.99, 32.04, 29.10, 27.20, 13.61, 9.46; ESI *m/z* 672 ([M+Li]$^+$, 45), 666 ([M+H]$^+$, 100); HRMS (ESI) *m/z* calcd for $C_{36}H_{52}NOPSnLi$ 672.2969 (M+Li), found 672.2968.



14

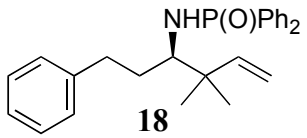
***N*-[(3*R**,4*S**)-4-*tert*-Butyl-1-phenylhex-5-en-3-yl]-*P*,*P*-diphenylphosphinamide (14).** According to the General Protocol B, 19.1 mg (0.207 mmol) of allene **10**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 173 μL (0.345 mmol) of Me_2Zn (2.0 M in toluene), and 67.6 mg (0.138 mmol) of sulfinyl adduct **30** afforded 47.5 mg (80%) of **14** as a colorless solid: mp 109-110 °C (CH_2Cl_2); IR (KBr) 3216, 3060, 2960, 1438, 1365, 1198, 1122, 1057, 918, 725, 699 cm^{-1}; ^{1}H NMR δ 8.04-7.92 (m, 4 H), 7.55-7.41 (m, 6 H), 7.30-7.21 (m, 2 H), 7.21-7.13 (m, 3 H), 5.74 (dt, 1 H, *J* = 16.9, 10.4 Hz), 5.23 (dd, 1 H, *J* = 16.9, 2.5 Hz), 5.21 (dd, 1 H, 10.1, 2.5 Hz), 3.32 (q, 1 H, *J* = 10.8 Hz), 3.10-2.95 (m, 2 H), 2.63-2.51 (m, 1 H), 2.23 (dd, 1 H, *J* = 10.7, 1.7 Hz), 2.0-1.8 (m, 1 H), 1.56-1.40 (m, 1 H), 0.70 (s, 9 H); ^{13}C NMR δ 142.51, 135.48, 132.47, 132.36, 131.85, 128.74, 128.60, 128.41, 125.84, 119.48, 62.15, 51.62, 36.83, 36.62, 33.29, 32.49, 28.73; EIMS *m/z* 374 ([M-C_3H_9]$^+$, 4), 335 (80), 201 (65); HRMS (EI) *m/z* calcd for $C_{24}H_{25}NOP$ 374.1674 (M-C_3H_9), found 374.1665.



16

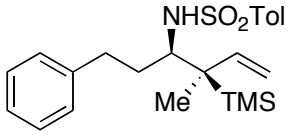
***N*-[(3*R**,4*S**)-4-Methyl-1-phenyl-4-(trimethylsilyl)hex-5-en-3-yl]-*P*,*P*-diphenylphosphinamide (16).** According to the General Protocol B, 26.1 mg (0.207 mmol) of allene **15**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 173 μL (0.345 mmol) of Me_2Zn

(2.0 M in toluene), and 67.6 mg (0.138 mmol) of sulfinyl adduct **30** afforded 51.8 mg (81%) of **16** as a colorless solid: mp 155-156 °C (CH_2Cl_2); IR (KBr) 3230, 3058, 2954, 1619, 1437, 1248, 1190, 1122, 1108, 838, 697 cm^{-1}; ^1H NMR δ 8.10-7.82 (m, 4 H), 7.60-7.42 (m, 6 H), 7.31-7.22 (m, 2 H), 7.21-7.10 (m, 3 H), 6.04 (dd, 1 H, *J* = 17.2, 10.7 Hz), 5.19 (dd, 1 H, *J* = 10.7, 1.2 Hz), 4.99 (dd, 1 H, *J* = 17.2, 1.4 Hz), 3.24 (q, 1 H, *J* = 8.6 Hz), 2.99 (ddd, 1 H, *J* = 11.8, 4.8, 1.8 Hz), 2.80 (t, 1 H, *J* = 9.5 Hz), 2.50 (ddd, 1 H, *J* = 11.4, 5.8, 2.1 Hz), 2.15-1.98 (m, 1 H), 1.9-1.7 (m, 1 H), 1.10 (s, 3 H), -0.02 (s, 9 H); ^{13}C NMR δ 142.33, 139.38, 131.94, 131.49, 128.30, 128.13, 125.50, 114.58, 57.64, 38.72, 38.10, 33.67, 17.57, -2.50; EIMS *m/z* 461 ($[M]^+$, 10), 356 (75), 201 (95); HRMS (EI) *m/z* calcd for $C_{28}H_{36}NOPSi$ 461.2304, found 461.2284.



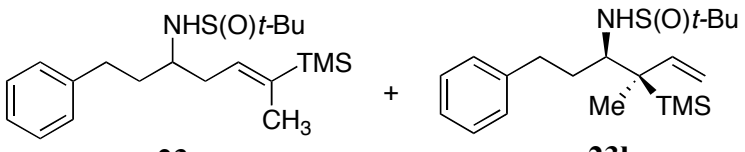
18

***N*-(4,4-Dimethyl-1-phenylhex-5-en-3-yl)-*P*,*P*-diphenylphosphinamide (18).** According to the General Protocol B, 14.1 mg (0.207 mmol) of allene **17**, 53.4 mg (0.207 mmol) of Cp_2ZrHCl, 173 μL (0.345 mmol) of Me_2Zn (2.0 M in toluene), and 67.6 mg (0.138 mmol) of sulfinyl adduct **30** afforded 42.0 mg (76%) of **18** as a colorless solid: mp 173-174 °C (CH_2Cl_2); IR (KBr) 3207, 3058, 2962, 1437, 1185, 1122, 985, 910, 721, 697 cm^{-1}; ^1H NMR δ 8.00-7.82 (m, 4 H), 7.58-7.40 (m, 6 H), 7.29-7.21 (m, 2 H), 7.20-7.07 (m, 3 H), 5.77 (dd, 1 H, *J* = 17.3, 10.9 Hz), 5.05 (d, 1 H, 10.8 Hz), 5.02 (d, 1 H, *J* = 17.7 Hz), 3.00-2.82 (m, 2 H), 2.79-2.68 (m, 1 H), 2.55-2.42 (m, 1 H), 2.10-1.92 (m, 1 H), 1.65-1.42 (m, 1 H), 1.04 (s, 3 H), 1.01 (s, 3 H); ^{13}C NMR δ 145.00, 142.37, 132.31, 132.26, 132.18, 132.14, 131.78, 131.74, 131.66, 131.62, 130.85, 128.53, 128.44, 128.38, 128.21, 125.63, 113.38, 59.73, 41.98, 41.91, 36.09, 33.64, 25.44, 23.27; EIMS *m/z* 403 (M^+, 20), 334 (100), 201 (100); HRMS (EI) *m/z* calcd for $C_{26}H_{30}NOP$ 403.2065, found 403.2050.



21

4-Methyl-*N*-[(3*R,4*R**)-4-methyl-1-phenyl-4-(trimethylsilyl)hex-5-en-3-yl]benzenesulfonamide (21).** A solution of 210 mg (1.65 mmol) of allene **15** in 5 mL of CH_2Cl_2 was treated at –78 °C with 425 mg (1.65 mmol) of Cp_2ZrHCl and allowed to warm to room temperature over 20 min. After approximately 90% of the CH_2Cl_2 was removed under reduced pressure, 5 mL of toluene was added. The resulting red solution was cooled to –78 °C, treated with 2.47 mL (2.47 mmol) of Et_2Zn (1.0 M in hexane) and 500 mg (1.10 mmol) of sulfinyl adduct **31**, and allowed to stir at –78 °C for 15 min. The reaction mixture was then warmed to room temperature and stirred for an additional 3 h, quenched with saturated aqueous $NaHCO_3$, extracted with EtOAc (3x), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by chromatography on SiO_2 (6:4, EtOAc:hexanes) to yield 401 mg (85%) of **21** as a colorless solid: mp 131.2-132.4 °C (CH_2Cl_2); IR (KBr) 3286, 3027, 2953, 1496, 1323, 1157, 840 cm^{-1}; ^1H NMR δ 7.84-7.75 (m, 2 H), 7.35-7.21 (m, 4 H), 7.21-7.13 (m, 1 H), 7.03-6.96 (m, 2 H), 5.89 (dd, 1 H, *J* = 17.2, 10.7 Hz), 5.14 (dd, 1 H, *J* = 10.7, 1.3 Hz), 4.89 (dd, 1 H, *J* = 17.2, 1.4 Hz), 4.31 (d, 1 H, *J* = 9.3 Hz), 3.56 (dt, 1 H, *J* = 9.5, 2.7 Hz), 2.58-2.43 (m, 2 H), 2.43 (s, 3 H), 1.95-1.84 (m, 1 H), 1.73-1.58 (m, 1 H), 0.88 (s, 3 H), 0.01 (s, 9 H); ^{13}C NMR δ 142.83, 141.78, 139.44, 138.81, 129.40, 128.20, 128.16, 128.12, 126.74, 125.68, 114.76, 60.51, 37.28, 36.65, 33.57, 21.36, 16.56, -2.67; ESIMS *m/z* 438 ($[M+Na]^+$, 100); HRMS (ESI) *m/z* calcd for $C_{23}H_{33}NO_2SSiNa$ (M+Na) 438.1899, found 438.1891.

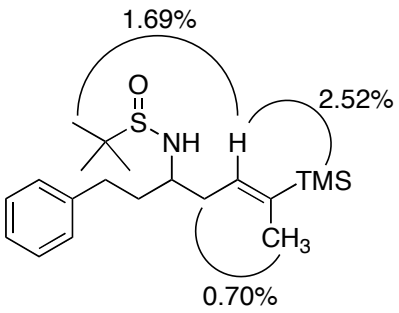


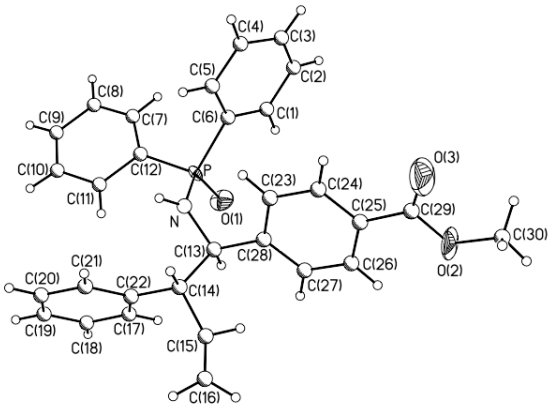
23a **23b**

(*E*)-2-Methyl-*N*-(1-phenyl-6-(trimethylsilyl)hept-5-en-3-yl)propane-2-sulfinamide (23a) and 2-methyl-*N*-((3*R,4*R**)-4-methyl-1-phenyl-4-(trimethylsilyl)-**

hex-5-en-3-yl)propane-2-sulfinamide (23b). A solution of 222 mg (1.75 mmol) of allene **15** in 6 mL of CH_2Cl_2 was treated at –78 °C with 450 mg (1.75 mmol) of Cp_2ZrHCl and allowed to warm to room temperature over 20 min. After approximately 90% of the CH_2Cl_2 was removed under reduced pressure, 6 mL of toluene was added. The resulting red solution was cooled to –78 °C, treated with 1.75 mL (1.75 mmol) of Et_2Zn (1.0 M in hexane) and 312 mg (1.31 mmol) of imine **22** in 6 mL of toluene, and allowed to stir at –78 °C for 15 min. The reaction mixture was then warmed to room temperature and stirred for an additional 3 h, quenched with saturated aqueous $NaHCO_3$, extracted with EtOAc (3x), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by chromatography on SiO_2 (1:4, EtOAc:hexanes to 1:2 EtOAc:hexanes) to yield 167 mg (36%) of **23a** and 221 mg (45%) of **23b** as colorless oils. **23a**: IR (neat) 3213, 3026, 2953, 1618, 1496, 1247, 1055, 836 cm^{-1}; ^1H NMR δ 7.35-7.25 (m, 2 H), 7.24-7.15 (m, 3 H), 5.72 (dt, 1 H, *J* = 7.0, 1.6 Hz), 3.45-3.31 (m, 1 H), 3.24 (d, 1 H, *J* = 5.85 Hz), 2.82-2.60 (m, 2 H), 2.54-2.41 (m, 2 H), 1.92-1.81 (m, 2 H), 1.73 (d, 3 H, *J* = 1.0 Hz), 1.23 (s, 9 H), 0.06 (s, 9 H); ^{13}C NMR δ 141.73, 140.57, 133.68, 128.34, 128.24, 125.80, 55.56, 54.75, 36.86, 34.73, 31.70, 22.56, 14.73, -2.23; ESIMS *m/z* 388 ($[M+Na]^+$, 100), 332 (10), 260 (30); HRMS (ESI) *m/z* calcd for $C_{20}H_{35}NOSSiNa$ (M+Na) 388.2106, found 388.2087. **23b**: IR (neat) 3312, 3084, 3026, 2955, 1618, 1454, 1250, 1077, 889 cm^{-1}; ^1H NMR δ 7.31-7.22 (m, 2 H), 7.20-7.13 (m, 3 H), 5.96 (dd, 1 H, *J* = 17.2, 10.7 Hz), 5.15 (dd, 1 H, *J* = 10.7, 1.4), 4.94 (dd, 1 H, *J* = 17.2, 1.5 Hz), 3.48 (d, 1 H, *J* = 6.3 Hz), 3.18 (ddd, 1 H, *J* = 10.4, 6.3, 1.6 Hz), 2.86 (ddd, 1 H, *J* = 14.2, 10.0, 4.8 Hz), 2.54 (ddd, 1 H, *J* = 16.5, 9.8, 6.8 Hz), 2.05-1.92 (m, 1 H), 1.86-1.71 (m, 1 H), 1.27 (s, 9 H), 1.12 (s, 3 H), -0.02 (s, 9 H); ^{13}C NMR δ 141.79, 139.37, 128.32, 125.78, 114.59, 61.13, 56.64, 37.69, 37.07, 33.44, 23.09, 17.15, -2.59; ESIMS *m/z* 388 ($[M+Na]^+$, 100), 366 (20), 332 (25); HRMS (ESI) *m/z* calcd for $C_{20}H_{35}NOSSiNa$ (M+Na) 388.2106, found 388.2090.

NOE Data for **23a**:





X-ray Structrure and Data for **9**

Table 1. Crystal data and structure refinement for **9**.

Identification code	9
Empirical formula	C30 H28 N O3 P
Formula weight	481.50
Temperature	295(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	a = 10.4622(16) Å α= 90°.
	b = 24.020(4) Å β= 99.629(4)°.
	c = 10.5456(17) Å γ = 90°.
Volume	2612.8(7) $Å^3$
Z	4
Density (calculated)	1.224 Mg/m^3
Absorption coefficient	0.136 mm^{-1}
F(000)	1016
Crystal size	0.37 x 0.03 x 0.03 mm^3
Theta range for data collection	1.70 to 22.50°.
Index ranges	-11<=h<=11, -25<=k<=25, -11<=l<=11
Reflections collected	16213
Independent reflections	3414 [R(int) = 0.3255]
Completeness to theta = 22.50°	100.0 %
Absorption correction	sadabs
Max. and min. transmission	0.9959 and 0.9513
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3414 / 0 / 161
Goodness-of-fit on F^2	1.156
Final R indices [I>2sigma(I)]	R1 = 0.2114, wR2 = 0.3576
R indices (all data)	R1 = 0.2941, wR2 = 0.3990
Largest diff. peak and hole	0.537 and -0.545 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P	4252(4)	2444(2)	2(3)	15(1)
N	5334(10)	2294(4)	1261(10)	13(3)
O(1)	4832(10)	2431(5)	-1173(9)	41(3)
C(1)	3486(16)	3496(7)	-765(16)	37(5)
O(2)	9718(13)	4709(5)	1589(15)	67(5)
C(2)	2950(20)	4030(10)	-650(20)	79(7)
C(3)	2560(20)	4154(11)	430(20)	76(7)
O(3)	8385(17)	4885(6)	2962(19)	103(7)
C(4)	2550(20)	3789(9)	1430(20)	65(6)
C(5)	3044(19)	3260(9)	1320(20)	58(6)
C(6)	3510(14)	3133(6)	211(14)	23(4)
C(7)	1726(16)	2035(8)	-513(16)	40(5)
C(8)	804(19)	1660(8)	-586(17)	46(5)
C(9)	1110(20)	1136(9)	-120(18)	57(6)
C(10)	2290(20)	1008(9)	432(19)	60(6)
C(11)	3290(18)	1400(7)	480(16)	41(5)
C(12)	2977(16)	1961(7)	-6(15)	30(4)
C(13)	6738(16)	2363(7)	1314(16)	38(5)
C(14)	7427(15)	1935(6)	2284(15)	26(4)
C(15)	8860(30)	1946(14)	2370(30)	122(11)
C(16)	9840(20)	1751(10)	2650(20)	72(7)
C(17)	6930(20)	1060(9)	1000(20)	61(6)
C(18)	6480(20)	516(10)	830(20)	73(7)
C(19)	6010(20)	270(10)	1780(20)	75(7)
C(20)	5970(20)	558(9)	2920(20)	64(6)
C(21)	6434(18)	1097(8)	3080(19)	52(6)
C(22)	6912(17)	1351(7)	2060(16)	37(5)
C(23)	6667(17)	3306(7)	2227(16)	36(4)
C(24)	7190(17)	3835(8)	2462(17)	42(5)
C(25)	8235(16)	4009(7)	1896(15)	29(4)
C(26)	8793(17)	3640(7)	1148(16)	37(5)
C(27)	8287(14)	3121(6)	936(14)	25(4)
C(28)	7241(14)	2943(6)	1494(14)	22(4)
C(29)	8750(19)	4569(9)	2263(19)	47(5)
C(30)	10300(20)	5233(10)	1940(20)	78(7)

Table 3. Bond lengths [Å] and angles [°] for **9**.

P-O(1)	1.468(10)	C(3)-C(2)-C(1)	118(2)
P-N	1.635(11)	C(2)-C(3)-C(4)	124(3)
P-C(12)	1.767(16)	C(3)-C(4)-C(5)	118(2)
P-C(6)	1.858(15)	C(6)-C(5)-C(4)	118(2)
N-C(13)	1.470(19)	C(1)-C(6)-C(5)	123.0(17)
C(1)-C(6)	1.35(2)	C(1)-C(6)-P	116.2(12)
C(1)-C(2)	1.41(3)	C(5)-C(6)-P	120.8(14)
O(2)-C(29)	1.37(2)	C(8)-C(7)-C(12)	126.2(19)
O(2)-C(30)	1.42(2)	C(7)-C(8)-C(9)	119(2)
C(2)-C(3)	1.30(3)	C(10)-C(9)-C(8)	121(2)
C(3)-C(4)	1.37(3)	C(9)-C(10)-C(11)	120(2)
O(3)-C(29)	1.16(2)	C(10)-C(11)-C(12)	119.1(17)
C(4)-C(5)	1.38(3)	C(7)-C(12)-C(11)	114.2(16)
C(5)-C(6)	1.37(2)	C(7)-C(12)-P	126.9(14)
C(7)-C(8)	1.31(2)	C(11)-C(12)-P	118.7(13)
C(7)-C(12)	1.34(2)	N-C(13)-C(28)	116.1(13)
C(8)-C(9)	1.37(2)	N-C(13)-C(14)	107.7(13)
C(9)-C(10)	1.32(3)	C(28)-C(13)-C(14)	115.0(13)
C(10)-C(11)	1.40(2)	C(15)-C(14)-C(22)	110.8(18)
C(11)-C(12)	1.46(2)	C(15)-C(14)-C(13)	112.6(18)
C(13)-C(28)	1.49(2)	C(22)-C(14)-C(13)	113.7(13)
C(13)-C(14)	1.54(2)	C(16)-C(15)-C(14)	151(3)
C(14)-C(15)	1.48(3)	C(22)-C(17)-C(18)	123(2)
C(14)-C(22)	1.51(2)	C(19)-C(18)-C(17)	119(2)
C(15)-C(16)	1.12(3)	C(18)-C(19)-C(20)	120(2)
C(17)-C(22)	1.32(2)	C(21)-C(20)-C(19)	121(2)
C(17)-C(18)	1.39(3)	C(20)-C(21)-C(22)	118.4(19)
C(18)-C(19)	1.32(3)	C(17)-C(22)-C(21)	118.6(18)
C(19)-C(20)	1.40(3)	C(17)-C(22)-C(14)	124.7(17)
C(20)-C(21)	1.38(3)	C(21)-C(22)-C(14)	116.7(16)
C(21)-C(22)	1.40(2)	C(28)-C(23)-C(24)	119.1(17)
C(23)-C(28)	1.37(2)	C(23)-C(24)-C(25)	120.7(17)
C(23)-C(24)	1.39(2)	C(26)-C(25)-C(24)	119.4(16)
C(24)-C(25)	1.39(2)	C(26)-C(25)-C(29)	124.4(16)
C(25)-C(26)	1.38(2)	C(24)-C(25)-C(29)	115.9(16)
C(25)-C(29)	1.48(2)	C(27)-C(26)-C(25)	119.5(16)
C(26)-C(27)	1.36(2)	C(26)-C(27)-C(28)	121.7(15)
C(27)-C(28)	1.39(2)	C(23)-C(28)-C(27)	119.4(15)
O(1)-P-N	110.7(6)	C(23)-C(28)-C(13)	119.3(14)
O(1)-P-C(12)	113.6(7)	C(27)-C(28)-C(13)	121.3(14)
N-P-C(12)	106.2(7)	O(3)-C(29)-O(2)	121.2(19)
O(1)-P-C(6)	111.0(7)	O(3)-C(29)-C(25)	128.0(19)
N-P-C(6)	110.3(6)	O(2)-C(29)-C(25)	110.8(16)
C(12)-P-C(6)	104.8(7)		
C(13)-N-P	123.9(10)		
C(6)-C(1)-C(2)	118.5(18)		
C(29)-O(2)-C(30)	113.8(16)		

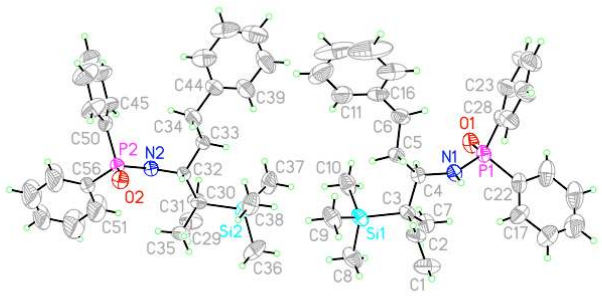
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P	26(2)	11(2)	7(2)	2(2)	3(1)	5(2)
O(1)	44(7)	57(8)	25(6)	9(6)	19(5)	9(7)
O(2)	65(10)	30(8)	115(13)	-29(8)	42(9)	-32(7)
O(3)	110(15)	42(10)	175(19)	-32(12)	78(14)	-27(10)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^{\ 3}$) for **9**.

	x	y	z	U(eq)
H(0A)	5066	2167	1933	16
H(1B)	3813	3397	-1501	44
H(2A)	2884	4286	-1315	95
H(3A)	2259	4514	525	92
H(4A)	2230	3895	2162	78
H(5A)	3058	2999	1975	70
H(7A)	1488	2386	-844	48
H(8A)	-44	1749	-949	55
H(9A)	463	866	-195	68
H(10A)	2469	657	790	72
H(11A)	4140	1304	818	50
H(13A)	6914	2244	471	45
H(14A)	7249	2048	3130	31
H(15)	9024	2285	2006	146
H(16A)	9912	1402	3046	87
H(16B)	10573	1937	2498	87
H(17A)	7255	1229	325	73
H(18A)	6515	328	68	88
H(19A)	5708	-94	1681	90
H(20A)	5628	386	3577	76
H(21A)	6427	1287	3848	63
H(23A)	5937	3200	2563	43
H(24A)	6839	4076	3002	50
H(26A)	9510	3747	791	45
H(27A)	8647	2879	405	29
H(30A)	10965	5304	1434	117
H(30B)	10675	5228	2835	117
H(30C)	9654	5520	1787	117



X-ray Structrure and Data for **16**

Table 1. Crystal data and structure refinement for **16**.

Identification code	16	
Empirical formula	C28 H36 N O P Si	
Formula weight	461.64	
Temperature	295(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.9313(5) Å	α= 90.8080(10)°.
	b = 13.4272(7) Å	β= 90.8930(10)°.
	c = 19.5603(10) Å	γ = 105.9390(10)°.
Volume	2759.8(2) $Å^3$	
Z	4	
Density (calculated)	1.111 Mg/m^3	
Absorption coefficient	0.162 mm^{-1}	
F(000)	992	
Crystal size	0.35 x 0.04 x 0.04 mm^3	
Theta range for data collection	1.58 to 25.00°.	
Index ranges	-12<=h<=12, -15<=k<=15, -23<=l<=23	
Reflections collected	22360	
Independent reflections	9704 [R(int) = 0.0408]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	Sadabs	
Max. and min. transmission	0.9936 and 0.9455	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9704 / 0 / 565	
Goodness-of-fit on F^2	1.275	
Final R indices [I>2sigma(I)]	R1 = 0.0813, wR2 = 0.1862	
R indices (all data)	R1 = 0.1219, wR2 = 0.1998	
Largest diff. peak and hole	0.521 and -0.405 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **16**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	9192(1)	7401(1)	5595(1)	62(1)
Si(2)	6738(1)	5634(1)	3248(1)	66(1)
P(1)	8485(1)	7854(1)	8352(1)	54(1)
P(2)	6458(1)	2094(1)	1686(1)	48(1)
O(1)	7149(2)	7654(2)	8117(1)	71(1)
O(2)	7823(2)	2347(2)	1865(1)	64(1)
N(1)	9427(3)	7660(2)	7755(1)	53(1)
N(2)	5751(2)	2890(2)	2042(1)	49(1)
C(1)	11643(5)	9329(4)	6467(3)	108(2)
C(2)	10950(4)	8416(3)	6574(2)	67(1)
C(3)	9530(3)	7992(3)	6499(2)	52(1)
C(4)	9011(3)	7204(3)	7075(2)	48(1)
C(5)	9315(3)	6158(3)	7017(2)	53(1)
C(6)	8432(4)	5334(3)	7439(2)	76(1)
C(7)	8855(4)	8854(3)	6557(2)	76(1)
C(8)	9401(5)	8439(4)	4956(2)	99(2)
C(9)	10305(4)	6630(4)	5337(2)	85(1)
C(10)	7515(4)	6597(4)	5542(2)	95(2)
C(11)	8381(5)	3741(4)	6755(3)	100(2)
C(12)	8612(8)	2757(6)	6716(5)	160(4)
C(13)	9034(11)	2354(9)	7283(8)	205(9)
C(14)	9235(10)	2855(8)	7851(8)	195(7)
C(15)	9042(5)	3821(5)	7911(3)	115(2)
C(16)	8634(4)	4276(3)	7374(3)	69(1)
C(17)	10276(6)	9786(4)	8418(3)	106(2)
C(18)	10793(8)	10777(5)	8681(4)	137(2)
C(19)	10234(9)	11137(5)	9179(4)	126(2)
C(20)	9131(8)	10564(6)	9435(3)	123(2)
C(21)	8575(5)	9538(5)	9177(3)	108(2)
C(22)	9181(4)	9167(3)	8666(2)	66(1)
C(23)	7557(5)	6564(4)	9431(3)	96(2)
C(24)	7683(6)	5995(5)	10009(3)	113(2)
C(25)	8837(7)	5901(4)	10203(3)	105(2)
C(26)	9876(6)	6383(4)	9862(2)	94(2)
C(27)	9786(5)	6955(3)	9296(2)	76(1)
C(28)	8628(4)	7062(3)	9071(2)	60(1)
C(29)	4839(5)	5399(4)	1707(3)	101(2)
C(30)	5065(4)	4795(3)	2188(2)	63(1)
C(31)	6307(3)	4703(3)	2469(2)	50(1)
C(32)	6217(3)	3573(3)	2637(2)	44(1)
C(33)	5445(3)	3131(3)	3265(2)	50(1)
C(34)	5748(5)	2173(4)	3528(2)	85(1)
C(35)	7390(4)	5067(3)	1956(2)	77(1)
C(36)	7284(5)	6982(3)	2946(3)	113(2)
C(37)	5351(4)	5589(4)	3806(3)	100(2)
C(38)	8055(4)	5322(4)	3742(3)	89(2)
C(39)	5674(5)	2325(4)	4799(3)	90(2)

C(40)	5218(8)	1985(6)	5431(3)	122(2)
C(41)	4253(8)	1146(7)	5450(5)	132(3)
C(42)	3699(6)	556(6)	4913(5)	126(3)
C(43)	4204(5)	908(4)	4250(3)	100(2)
C(44)	5187(4)	1789(4)	4200(2)	67(1)
C(45)	4333(4)	464(4)	1951(3)	88(1)
C(46)	3707(6)	-499(4)	2146(3)	116(2)
C(47)	4341(7)	-1156(4)	2331(3)	109(2)
C(48)	5615(8)	-887(5)	2316(3)	126(2)
C(49)	6277(5)	102(4)	2101(3)	103(2)
C(50)	5631(4)	791(3)	1922(2)	54(1)
C(51)	5919(5)	3035(3)	515(2)	179(4)
C(52)	5761(6)	3101(4)	-188(2)	215(5)
C(53)	5831(6)	2285(5)	-616(1)	157(3)
C(54)	6060(6)	1402(4)	-342(2)	216(5)
C(55)	6218(5)	1335(3)	361(2)	189(4)
C(56)	6147(3)	2152(3)	789(1)	62(1)

Table 3. Bond lengths [Å] and angles [°] for **16**.

Si(1)-C(10)	1.854(4)	C(5)-H(5B)	0.9700
Si(1)-C(8)	1.855(5)	C(6)-C(16)	1.500(6)
Si(1)-C(9)	1.871(4)	C(6)-H(6A)	0.9700
Si(1)-C(3)	1.917(4)	C(6)-H(6B)	0.9700
Si(2)-C(36)	1.852(5)	C(7)-H(7A)	0.9600
Si(2)-C(38)	1.866(4)	C(7)-H(7B)	0.9600
Si(2)-C(37)	1.870(5)	C(7)-H(7C)	0.9600
Si(2)-C(31)	1.925(4)	C(8)-H(8A)	0.9600
P(1)-O(1)	1.475(2)	C(8)-H(8B)	0.9600
P(1)-N(1)	1.633(3)	C(8)-H(8C)	0.9600
P(1)-C(28)	1.805(4)	C(9)-H(9A)	0.9600
P(1)-C(22)	1.813(4)	C(9)-H(9B)	0.9600
P(2)-O(2)	1.473(2)	C(9)-H(9C)	0.9600
P(2)-N(2)	1.635(3)	C(10)-H(10A)	0.9600
P(2)-C(56)	1.788(3)	C(10)-H(10B)	0.9600
P(2)-C(50)	1.805(4)	C(10)-H(10C)	0.9600
N(1)-C(4)	1.468(4)	C(11)-C(16)	1.384(7)
N(1)-H(1B)	0.8600	C(11)-C(12)	1.414(8)
N(2)-C(32)	1.467(4)	C(11)-H(11A)	0.9300
N(2)-H(2B)	0.8600	C(12)-C(13)	1.368(15)
C(1)-C(2)	1.274(5)	C(12)-H(12A)	0.9300
C(1)-H(1C)	0.9300	C(13)-C(14)	1.275(18)
C(1)-H(1D)	0.9300	C(13)-H(13A)	0.9300
C(2)-C(3)	1.503(5)	C(14)-C(15)	1.374(11)
C(2)-H(2C)	0.9300	C(14)-H(14A)	0.9300
C(3)-C(7)	1.539(5)	C(15)-C(16)	1.353(7)
C(3)-C(4)	1.560(5)	C(15)-H(15A)	0.9300
C(4)-C(5)	1.532(5)	C(17)-C(22)	1.357(6)
C(4)-H(4A)	0.9800	C(17)-C(18)	1.383(7)
C(5)-C(6)	1.516(5)	C(17)-H(17A)	0.9300
C(5)-H(5A)	0.9700	C(18)-C(19)	1.312(9)

C(18)-H(18A)	0.9300	C(43)-C(44)	1.369(6)
C(19)-C(20)	1.348(9)	C(43)-H(43A)	0.9300
C(19)-H(19A)	0.9300	C(45)-C(46)	1.350(6)
C(20)-C(21)	1.425(8)	C(45)-C(50)	1.367(5)
C(20)-H(20A)	0.9300	C(45)-H(45A)	0.9300
C(21)-C(22)	1.368(6)	C(46)-C(47)	1.314(7)
C(21)-H(21A)	0.9300	C(46)-H(46A)	0.9300
C(23)-C(24)	1.399(7)	C(47)-C(48)	1.341(8)
C(23)-C(28)	1.387(6)	C(47)-H(47A)	0.9300
C(23)-H(23A)	0.9300	C(48)-C(49)	1.400(7)
C(24)-C(25)	1.351(7)	C(48)-H(48A)	0.9300
C(24)-H(24A)	0.9300	C(49)-C(50)	1.355(5)
C(25)-C(26)	1.335(7)	C(49)-H(49A)	0.9300
C(25)-H(25A)	0.9300	C(51)-C(52)	1.3900
C(26)-C(27)	1.374(6)	C(51)-C(56)	1.3900
C(26)-H(26A)	0.9300	C(51)-H(51A)	0.9300
C(27)-C(28)	1.379(5)	C(52)-C(53)	1.3900
C(27)-H(27A)	0.9300	C(52)-H(52A)	0.9300
C(29)-C(30)	1.315(5)	C(53)-C(54)	1.3900
C(29)-H(29A)	0.9300	C(53)-H(53A)	0.9300
C(29)-H(29B)	0.9300	C(54)-C(55)	1.3900
C(30)-C(31)	1.494(5)	C(54)-H(54A)	0.9300
C(30)-H(30A)	0.9300	C(55)-C(56)	1.3900
C(31)-C(32)	1.533(5)	C(55)-H(55A)	0.9300
C(31)-C(35)	1.544(5)	C(10)-Si(1)-C(8)	108.3(2)
C(32)-C(33)	1.533(5)	C(10)-Si(1)-C(9)	111.0(2)
C(32)-H(32A)	0.9800	C(8)-Si(1)-C(9)	105.3(2)
C(33)-C(34)	1.508(5)	C(10)-Si(1)-C(3)	108.50(18)
C(33)-H(33A)	0.9700	C(8)-Si(1)-C(3)	110.3(2)
C(33)-H(33B)	0.9700	C(9)-Si(1)-C(3)	113.34(17)
C(34)-C(44)	1.496(6)	C(36)-Si(2)-C(38)	109.3(2)
C(34)-H(34A)	0.9700	C(36)-Si(2)-C(37)	105.9(3)
C(34)-H(34B)	0.9700	C(38)-Si(2)-C(37)	111.0(2)
C(35)-H(35A)	0.9600	C(36)-Si(2)-C(31)	109.1(2)
C(35)-H(35B)	0.9600	C(38)-Si(2)-C(31)	108.41(18)
C(35)-H(35C)	0.9600	C(37)-Si(2)-C(31)	113.07(19)
C(36)-H(36A)	0.9600	O(1)-P(1)-N(1)	113.32(15)
C(36)-H(36B)	0.9600	O(1)-P(1)-C(28)	111.43(18)
C(36)-H(36C)	0.9600	N(1)-P(1)-C(28)	108.24(16)
C(37)-H(37A)	0.9600	O(1)-P(1)-C(22)	113.57(17)
C(37)-H(37B)	0.9600	N(1)-P(1)-C(22)	105.71(19)
C(37)-H(37C)	0.9600	C(28)-P(1)-C(22)	103.94(18)
C(38)-H(38A)	0.9600	O(2)-P(2)-N(2)	112.78(14)
C(38)-H(38B)	0.9600	O(2)-P(2)-C(56)	113.35(16)
C(38)-H(38C)	0.9600	N(2)-P(2)-C(56)	104.89(15)
C(39)-C(40)	1.375(7)	O(2)-P(2)-C(50)	111.65(16)
C(39)-C(44)	1.386(6)	N(2)-P(2)-C(50)	108.39(16)
C(39)-H(39A)	0.9300	C(56)-P(2)-C(50)	105.26(17)
C(40)-C(41)	1.316(9)	C(4)-N(1)-P(1)	125.2(2)
C(40)-H(40A)	0.9300	C(4)-N(1)-H(1B)	117.4
C(41)-C(42)	1.339(9)	P(1)-N(1)-H(1B)	117.4
C(41)-H(41A)	0.9300	C(32)-N(2)-P(2)	126.4(2)
C(42)-C(43)	1.453(9)	C(32)-N(2)-H(2B)	116.8
C(42)-H(42A)	0.9300	P(2)-N(2)-H(2B)	116.8

C(2)-C(1)-H(1C)	120.0	C(16)-C(11)-C(12)	117.5(7)
C(2)-C(1)-H(1D)	120.0	C(16)-C(11)-H(11A)	121.3
H(1C)-C(1)-H(1D)	120.0	C(12)-C(11)-H(11A)	121.3
C(1)-C(2)-C(3)	129.0(4)	C(13)-C(12)-C(11)	120.0(9)
C(1)-C(2)-H(2C)	115.5	C(13)-C(12)-H(12A)	120.0
C(3)-C(2)-H(2C)	115.5	C(11)-C(12)-H(12A)	120.0
C(2)-C(3)-C(7)	111.6(3)	C(14)-C(13)-C(12)	121.2(14)
C(2)-C(3)-C(4)	110.2(3)	C(14)-C(13)-H(13A)	119.4
C(7)-C(3)-C(4)	107.3(3)	C(12)-C(13)-H(13A)	119.4
C(2)-C(3)-Si(1)	106.7(2)	C(13)-C(14)-C(15)	120.9(14)
C(7)-C(3)-Si(1)	107.4(2)	C(13)-C(14)-H(14A)	119.5
C(4)-C(3)-Si(1)	113.6(2)	C(15)-C(14)-H(14A)	119.5
N(1)-C(4)-C(5)	108.7(3)	C(16)-C(15)-C(14)	121.5(8)
N(1)-C(4)-C(3)	111.4(3)	C(16)-C(15)-H(15A)	119.2
C(5)-C(4)-C(3)	116.3(3)	C(14)-C(15)-H(15A)	119.2
N(1)-C(4)-H(4A)	106.6	C(15)-C(16)-C(11)	118.8(5)
C(5)-C(4)-H(4A)	106.6	C(15)-C(16)-C(6)	121.5(5)
C(3)-C(4)-H(4A)	106.6	C(11)-C(16)-C(6)	119.7(4)
C(6)-C(5)-C(4)	112.2(3)	C(22)-C(17)-C(18)	120.9(6)
C(6)-C(5)-H(5A)	109.2	C(22)-C(17)-H(17A)	119.5
C(4)-C(5)-H(5A)	109.2	C(18)-C(17)-H(17A)	119.5
C(6)-C(5)-H(5B)	109.2	C(19)-C(18)-C(17)	120.5(7)
C(4)-C(5)-H(5B)	109.2	C(19)-C(18)-H(18A)	119.8
H(5A)-C(5)-H(5B)	107.9	C(17)-C(18)-H(18A)	119.8
C(16)-C(6)-C(5)	114.8(3)	C(18)-C(19)-C(20)	121.1(7)
C(16)-C(6)-H(6A)	108.6	C(18)-C(19)-H(19A)	119.4
C(5)-C(6)-H(6A)	108.6	C(20)-C(19)-H(19A)	119.4
C(16)-C(6)-H(6B)	108.6	C(19)-C(20)-C(21)	119.8(6)
C(5)-C(6)-H(6B)	108.6	C(19)-C(20)-H(20A)	120.1
H(6A)-C(6)-H(6B)	107.5	C(21)-C(20)-H(20A)	120.1
C(3)-C(7)-H(7A)	109.5	C(22)-C(21)-C(20)	118.4(6)
C(3)-C(7)-H(7B)	109.5	C(22)-C(21)-H(21A)	120.8
H(7A)-C(7)-H(7B)	109.5	C(20)-C(21)-H(21A)	120.8
C(3)-C(7)-H(7C)	109.5	C(17)-C(22)-C(21)	119.3(5)
H(7A)-C(7)-H(7C)	109.5	C(17)-C(22)-P(1)	122.5(4)
H(7B)-C(7)-H(7C)	109.5	C(21)-C(22)-P(1)	118.2(4)
Si(1)-C(8)-H(8A)	109.5	C(24)-C(23)-C(28)	119.8(5)
Si(1)-C(8)-H(8B)	109.5	C(24)-C(23)-H(23A)	120.1
H(8A)-C(8)-H(8B)	109.5	C(28)-C(23)-H(23A)	120.1
Si(1)-C(8)-H(8C)	109.5	C(25)-C(24)-C(23)	120.2(5)
H(8A)-C(8)-H(8C)	109.5	C(25)-C(24)-H(24A)	119.9
H(8B)-C(8)-H(8C)	109.5	C(23)-C(24)-H(24A)	119.9
Si(1)-C(9)-H(9A)	109.5	C(26)-C(25)-C(24)	120.4(5)
Si(1)-C(9)-H(9B)	109.5	C(26)-C(25)-H(25A)	119.8
H(9A)-C(9)-H(9B)	109.5	C(24)-C(25)-H(25A)	119.8
Si(1)-C(9)-H(9C)	109.5	C(25)-C(26)-C(27)	120.8(5)
H(9A)-C(9)-H(9C)	109.5	C(25)-C(26)-H(26A)	119.6
H(9B)-C(9)-H(9C)	109.5	C(27)-C(26)-H(26A)	119.6
Si(1)-C(10)-H(10A)	109.5	C(26)-C(27)-C(28)	121.1(5)
Si(1)-C(10)-H(10B)	109.5	C(26)-C(27)-H(27A)	119.5
H(10A)-C(10)-H(10B)	109.5	C(28)-C(27)-H(27A)	119.5
Si(1)-C(10)-H(10C)	109.5	C(27)-C(28)-C(23)	117.7(4)
H(10A)-C(10)-H(10C)	109.5	C(27)-C(28)-P(1)	122.3(3)
H(10B)-C(10)-H(10C)	109.5	C(23)-C(28)-P(1)	120.0(4)

C(30)-C(29)-H(29A)	120.0	C(40)-C(39)-C(44)	122.1(6)
C(30)-C(29)-H(29B)	120.0	C(40)-C(39)-H(39A)	118.9
H(29A)-C(29)-H(29B)	120.0	C(44)-C(39)-H(39A)	118.9
C(29)-C(30)-C(31)	129.6(4)	C(41)-C(40)-C(39)	117.7(7)
C(29)-C(30)-H(30A)	115.2	C(41)-C(40)-H(40A)	121.1
C(31)-C(30)-H(30A)	115.2	C(39)-C(40)-H(40A)	121.1
C(30)-C(31)-C(32)	110.5(3)	C(40)-C(41)-C(42)	126.2(8)
C(30)-C(31)-C(35)	111.8(3)	C(40)-C(41)-H(41A)	116.9
C(32)-C(31)-C(35)	107.6(3)	C(42)-C(41)-H(41A)	116.9
C(30)-C(31)-Si(2)	106.7(2)	C(41)-C(42)-C(43)	115.7(7)
C(32)-C(31)-Si(2)	113.7(2)	C(41)-C(42)-H(42A)	122.2
C(35)-C(31)-Si(2)	106.5(3)	C(43)-C(42)-H(42A)	122.2
N(2)-C(32)-C(33)	109.5(3)	C(44)-C(43)-C(42)	120.4(6)
N(2)-C(32)-C(31)	110.9(3)	C(44)-C(43)-H(43A)	119.8
C(33)-C(32)-C(31)	116.7(3)	C(42)-C(43)-H(43A)	119.8
N(2)-C(32)-H(32A)	106.4	C(43)-C(44)-C(39)	117.8(5)
C(33)-C(32)-H(32A)	106.4	C(43)-C(44)-C(34)	122.2(5)
C(31)-C(32)-H(32A)	106.4	C(39)-C(44)-C(34)	120.0(5)
C(34)-C(33)-C(32)	112.9(3)	C(46)-C(45)-C(50)	122.3(5)
C(34)-C(33)-H(33A)	109.0	C(46)-C(45)-H(45A)	118.8
C(32)-C(33)-H(33A)	109.0	C(50)-C(45)-H(45A)	118.8
C(34)-C(33)-H(33B)	109.0	C(47)-C(46)-C(45)	120.3(6)
C(32)-C(33)-H(33B)	109.0	C(47)-C(46)-H(46A)	119.8
H(33A)-C(33)-H(33B)	107.8	C(45)-C(46)-H(46A)	119.8
C(44)-C(34)-C(33)	116.1(3)	C(46)-C(47)-C(48)	120.5(6)
C(44)-C(34)-H(34A)	108.3	C(46)-C(47)-H(47A)	119.7
C(33)-C(34)-H(34A)	108.3	C(48)-C(47)-H(47A)	119.7
C(44)-C(34)-H(34B)	108.3	C(47)-C(48)-C(49)	119.8(5)
C(33)-C(34)-H(34B)	108.3	C(47)-C(48)-H(48A)	120.1
H(34A)-C(34)-H(34B)	107.4	C(49)-C(48)-H(48A)	120.1
C(31)-C(35)-H(35A)	109.5	C(50)-C(49)-C(48)	120.0(5)
C(31)-C(35)-H(35B)	109.5	C(50)-C(49)-H(49A)	120.0
H(35A)-C(35)-H(35B)	109.5	C(48)-C(49)-H(49A)	120.0
C(31)-C(35)-H(35C)	109.5	C(49)-C(50)-C(45)	117.0(4)
H(35A)-C(35)-H(35C)	109.5	C(49)-C(50)-P(2)	121.2(4)
H(35B)-C(35)-H(35C)	109.5	C(45)-C(50)-P(2)	121.8(3)
Si(2)-C(36)-H(36A)	109.5	C(52)-C(51)-C(56)	120.0
Si(2)-C(36)-H(36B)	109.5	C(52)-C(51)-H(51A)	120.0
H(36A)-C(36)-H(36B)	109.5	C(56)-C(51)-H(51A)	120.0
Si(2)-C(36)-H(36C)	109.5	C(53)-C(52)-C(51)	120.0
H(36A)-C(36)-H(36C)	109.5	C(53)-C(52)-H(52A)	120.0
H(36B)-C(36)-H(36C)	109.5	C(51)-C(52)-H(52A)	120.0
Si(2)-C(37)-H(37A)	109.5	C(54)-C(53)-C(52)	120.0
Si(2)-C(37)-H(37B)	109.5	C(54)-C(53)-H(53A)	120.0
H(37A)-C(37)-H(37B)	109.5	C(52)-C(53)-H(53A)	120.0
Si(2)-C(37)-H(37C)	109.5	C(53)-C(54)-C(55)	120.0
H(37A)-C(37)-H(37C)	109.5	C(53)-C(54)-H(54A)	120.0
H(37B)-C(37)-H(37C)	109.5	C(55)-C(54)-H(54A)	120.0
Si(2)-C(38)-H(38A)	109.5	C(56)-C(55)-C(54)	120.0
Si(2)-C(38)-H(38B)	109.5	C(56)-C(55)-H(55A)	120.0
H(38A)-C(38)-H(38B)	109.5	C(54)-C(55)-H(55A)	120.0
Si(2)-C(38)-H(38C)	109.5	C(55)-C(56)-C(51)	120.0
H(38A)-C(38)-H(38C)	109.5	C(55)-C(56)-P(2)	119.6(2)
H(38B)-C(38)-H(38C)	109.5	C(51)-C(56)-P(2)	120.3(2)

_____ Symmetry transformations used to generate equivalent
_____ atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **16**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	57(1)	89(1)	47(1)	7(1)	2(1)	29(1)
Si(2)	53(1)	58(1)	87(1)	-11(1)	-6(1)	19(1)
P(1)	47(1)	68(1)	50(1)	-7(1)	-1(1)	22(1)
P(2)	41(1)	55(1)	50(1)	0(1)	2(1)	16(1)
O(1)	47(2)	103(2)	66(2)	-14(2)	-1(1)	29(2)
O(2)	44(1)	77(2)	75(2)	-7(1)	0(1)	23(1)
N(1)	38(2)	73(2)	48(2)	-4(2)	-2(1)	15(2)
N(2)	39(2)	58(2)	53(2)	0(1)	-5(1)	18(1)
C(1)	82(3)	106(4)	118(4)	52(3)	-7(3)	-8(3)
C(2)	62(3)	72(3)	62(3)	27(2)	-1(2)	9(2)
C(3)	52(2)	62(2)	48(2)	11(2)	2(2)	25(2)
C(4)	36(2)	61(2)	50(2)	-2(2)	-1(2)	20(2)
C(5)	48(2)	61(2)	53(2)	7(2)	9(2)	18(2)
C(6)	78(3)	73(3)	75(3)	11(2)	29(2)	15(2)
C(7)	89(3)	80(3)	72(3)	7(2)	-7(2)	44(3)
C(8)	126(4)	124(4)	60(3)	21(3)	4(3)	55(4)
C(9)	77(3)	127(4)	65(3)	3(3)	10(2)	52(3)
C(10)	69(3)	149(5)	64(3)	-18(3)	-8(2)	25(3)
C(11)	113(4)	72(4)	113(5)	9(3)	43(3)	17(3)
C(12)	153(7)	75(5)	231(10)	-30(5)	115(7)	-12(4)
C(13)	111(8)	101(8)	410(30)	116(11)	109(12)	32(6)
C(14)	93(5)	141(11)	350(20)	146(10)	-6(9)	24(7)
C(15)	83(4)	96(4)	153(6)	60(4)	-13(3)	0(3)
C(16)	50(2)	64(3)	91(3)	23(3)	19(2)	9(2)
C(17)	126(5)	63(3)	114(4)	-17(3)	14(4)	-1(3)
C(18)	188(7)	77(4)	125(6)	-11(4)	5(5)	4(4)
C(19)	195(8)	75(4)	116(6)	-6(4)	-22(5)	55(5)
C(20)	169(7)	126(6)	99(5)	-41(4)	-21(5)	89(5)
C(21)	114(4)	119(5)	106(4)	-47(4)	-21(3)	63(4)
C(22)	83(3)	73(3)	55(3)	-7(2)	-20(2)	45(3)
C(23)	84(3)	114(4)	76(3)	15(3)	9(3)	4(3)
C(24)	125(5)	120(5)	76(4)	28(3)	27(4)	-2(4)
C(25)	157(6)	91(4)	75(4)	21(3)	14(4)	48(4)
C(26)	135(5)	98(4)	70(3)	20(3)	14(3)	65(4)
C(27)	90(3)	84(3)	67(3)	13(2)	16(2)	47(3)
C(28)	70(3)	63(3)	44(2)	-7(2)	13(2)	17(2)
C(29)	89(4)	105(4)	112(4)	42(3)	-9(3)	30(3)
C(30)	55(2)	59(3)	73(3)	15(2)	1(2)	15(2)
C(31)	41(2)	49(2)	59(2)	13(2)	8(2)	10(2)
C(32)	33(2)	55(2)	45(2)	0(2)	1(2)	13(2)
C(33)	49(2)	53(2)	52(2)	4(2)	5(2)	20(2)
C(34)	121(4)	90(3)	65(3)	21(2)	33(3)	60(3)
C(35)	74(3)	68(3)	82(3)	17(2)	21(2)	7(2)

C(36)	108(4)	56(3)	173(6)	-10(3)	-14(4)	19(3)
C(37)	83(3)	110(4)	117(4)	-42(3)	6(3)	47(3)
C(38)	71(3)	96(4)	104(4)	-31(3)	-25(3)	30(3)
C(39)	116(4)	102(4)	69(3)	16(3)	20(3)	55(3)
C(40)	186(7)	149(6)	62(4)	19(4)	18(4)	97(6)
C(41)	134(7)	121(7)	159(8)	59(5)	69(6)	60(5)
C(42)	88(4)	89(5)	210(9)	65(5)	35(5)	33(4)
C(43)	84(4)	92(4)	135(5)	38(4)	-6(3)	41(3)
C(44)	77(3)	73(3)	66(3)	21(2)	17(2)	47(3)
C(45)	75(3)	60(3)	124(4)	14(3)	19(3)	8(2)
C(46)	103(4)	64(4)	168(6)	12(4)	38(4)	-2(3)
C(47)	155(6)	66(4)	90(4)	9(3)	-6(4)	6(4)
C(48)	157(6)	79(4)	148(6)	19(4)	-58(5)	45(4)
C(49)	87(3)	71(3)	155(5)	8(3)	-31(3)	30(3)
C(50)	60(2)	55(2)	51(2)	-3(2)	-6(2)	21(2)
C(51)	346(12)	137(6)	67(4)	-3(4)	-49(5)	94(7)
C(52)	428(15)	175(8)	62(4)	11(5)	-42(6)	121(9)
C(53)	285(9)	152(7)	45(3)	-4(4)	1(4)	79(6)
C(54)	403(15)	183(9)	65(5)	-20(5)	-11(6)	89(9)
C(55)	361(12)	160(7)	63(4)	-15(4)	-10(5)	101(7)
C(56)	69(3)	66(3)	52(2)	-1(2)	4(2)	19(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{3}) for **16**.

	x	y	z	U(eq)
H(1B)	10231	7840	7847	64
H(2B)	5036	2911	1862	59
H(1C)	11272	9838	6322	129
H(1D)	12520	9487	6534	129
H(2C)	11382	7946	6718	80
H(4A)	8082	7056	7056	57
H(5A)	9241	5932	6542	64
H(5B)	10186	6242	7169	64
H(6A)	8542	5553	7916	91
H(6B)	7561	5289	7304	91
H(7A)	9006	9167	7006	114
H(7B)	7957	8564	6481	114
H(7C)	9180	9370	6222	114
H(8A)	9229	8133	4505	148
H(8B)	10260	8874	4983	148
H(8C)	8824	8847	5048	148
H(9A)	10090	6356	4882	128
H(9B)	10235	6070	5647	128
H(9C)	11163	7069	5350	128
H(10A)	7331	6295	5092	143
H(10B)	6959	7022	5637	143
H(10C)	7388	6056	5872	143
H(11A)	8071	4019	6379	120

H(12A)	8477	2382	6305	193
H(13A)	9178	1704	7253	245
H(14A)	9515	2562	8230	234
H(15A)	9197	4171	8331	138
H(17A)	10684	9540	8067	128
H(18A)	11543	11191	8504	164
H(19A)	10606	11798	9358	151
H(20A)	8733	10837	9778	147
H(21A)	7819	9131	9352	129
H(23A)	6758	6607	9289	115
H(24A)	6970	5680	10260	136
H(25A)	8909	5498	10576	126
H(26A)	10668	6331	10010	113
H(27A)	10519	7276	9062	91
H(29A)	5512	5854	1490	121
H(29B)	4005	5372	1584	121
H(30A)	4346	4361	2382	75
H(32A)	7090	3543	2731	53
H(33A)	5616	3656	3626	60
H(33B)	4546	2964	3146	60
H(34A)	5455	1622	3189	102
H(34B)	6665	2315	3570	102
H(35A)	7216	4622	1557	115
H(35B)	8181	5042	2164	115
H(35C)	7446	5765	1826	115
H(36A)	7497	7450	3333	170
H(36B)	6617	7138	2680	170
H(36C)	8020	7057	2669	170
H(37A)	5619	6067	4184	150
H(37B)	5023	4901	3975	150
H(37C)	4698	5775	3545	150
H(38A)	8281	5779	4133	134
H(38B)	8782	5407	3456	134
H(38C)	7781	4618	3890	134
H(39A)	6329	2934	4774	108
H(40A)	5580	2335	5830	146
H(41A)	3926	945	5878	158
H(42A)	3032	-41	4962	151
H(43A)	3859	532	3856	120
H(45A)	3866	920	1831	106
H(46A)	2823	-698	2149	140
H(47A)	3902	-1813	2474	130
H(48A)	6059	-1356	2447	151
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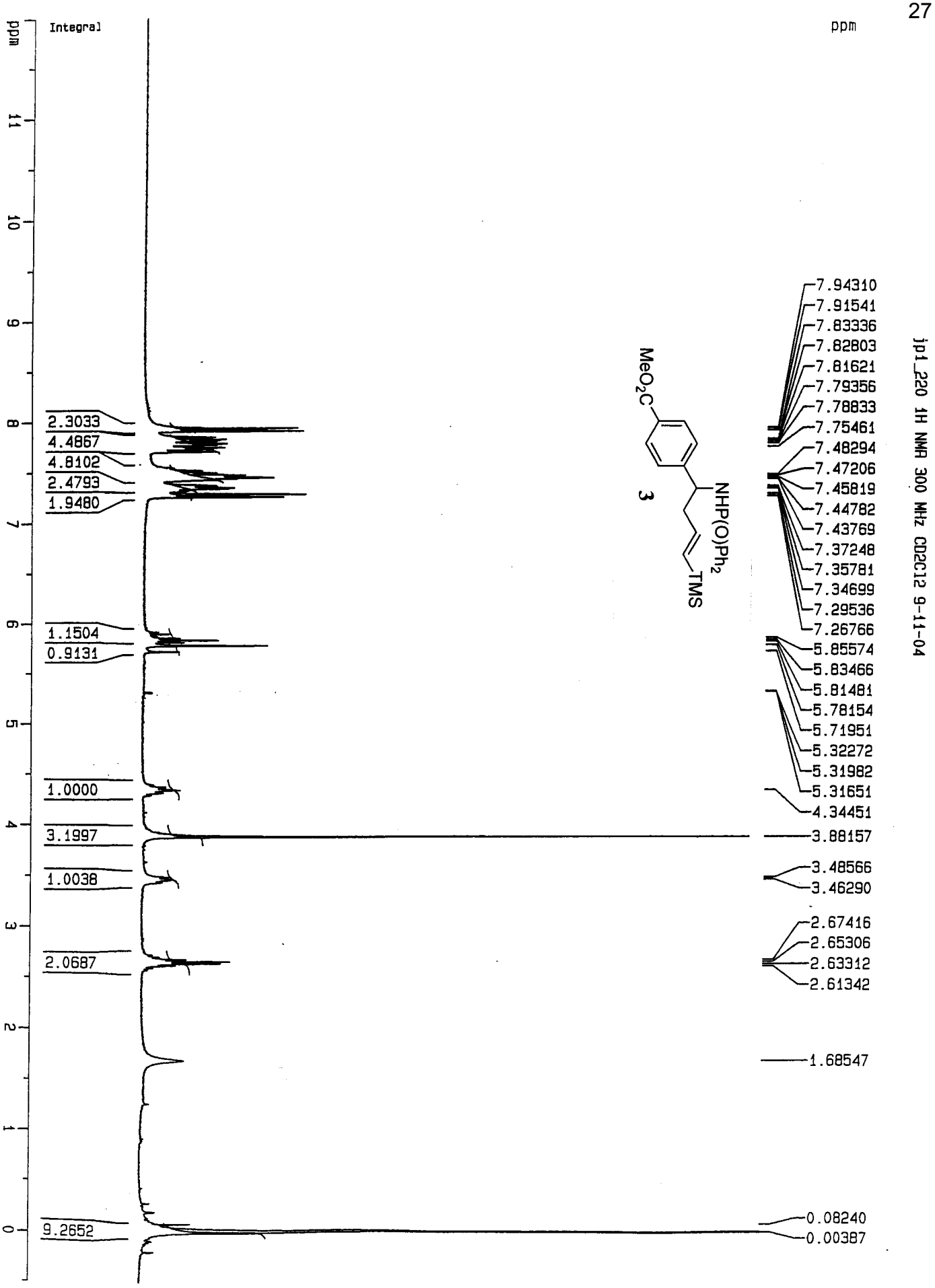
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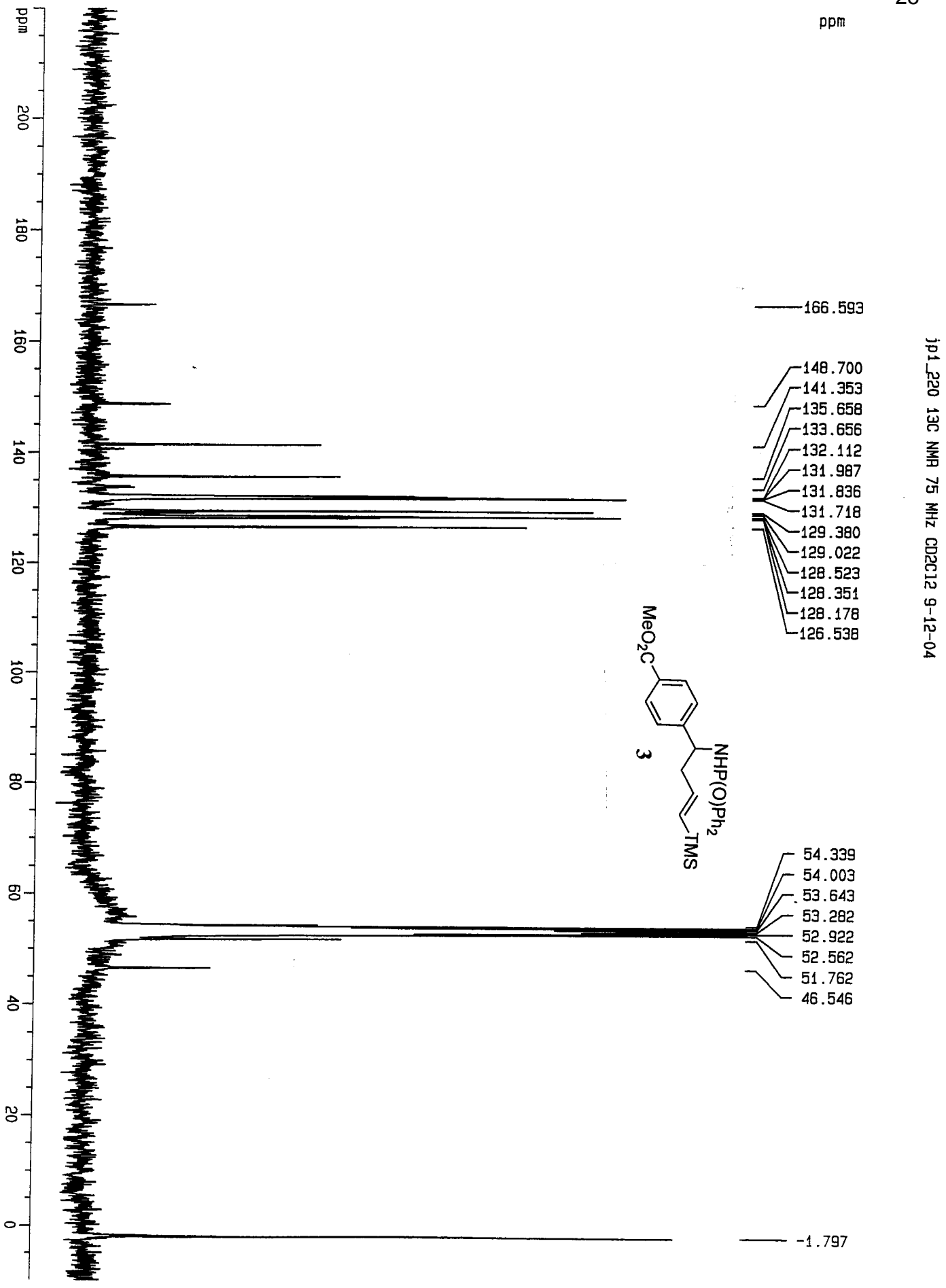
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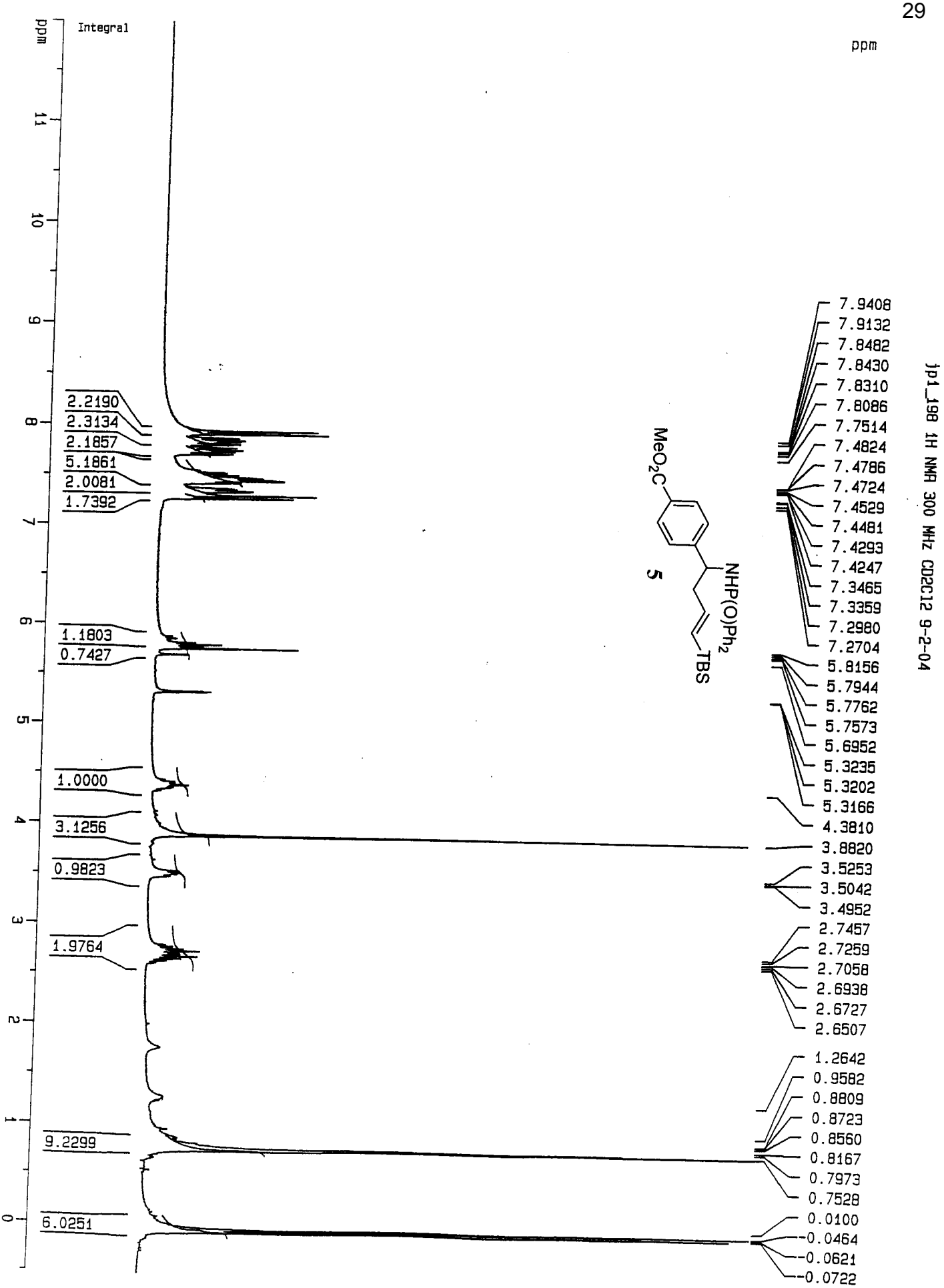
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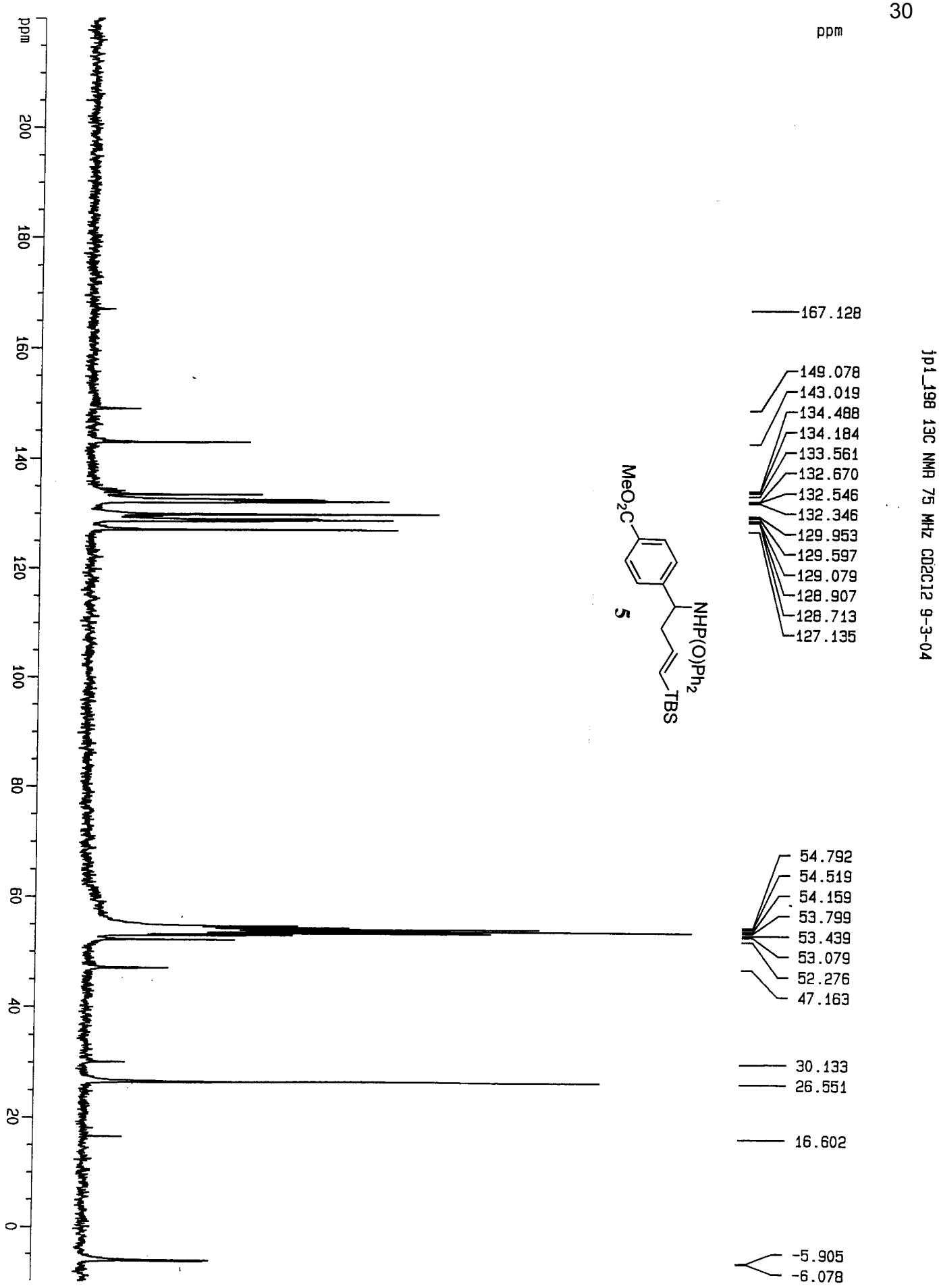
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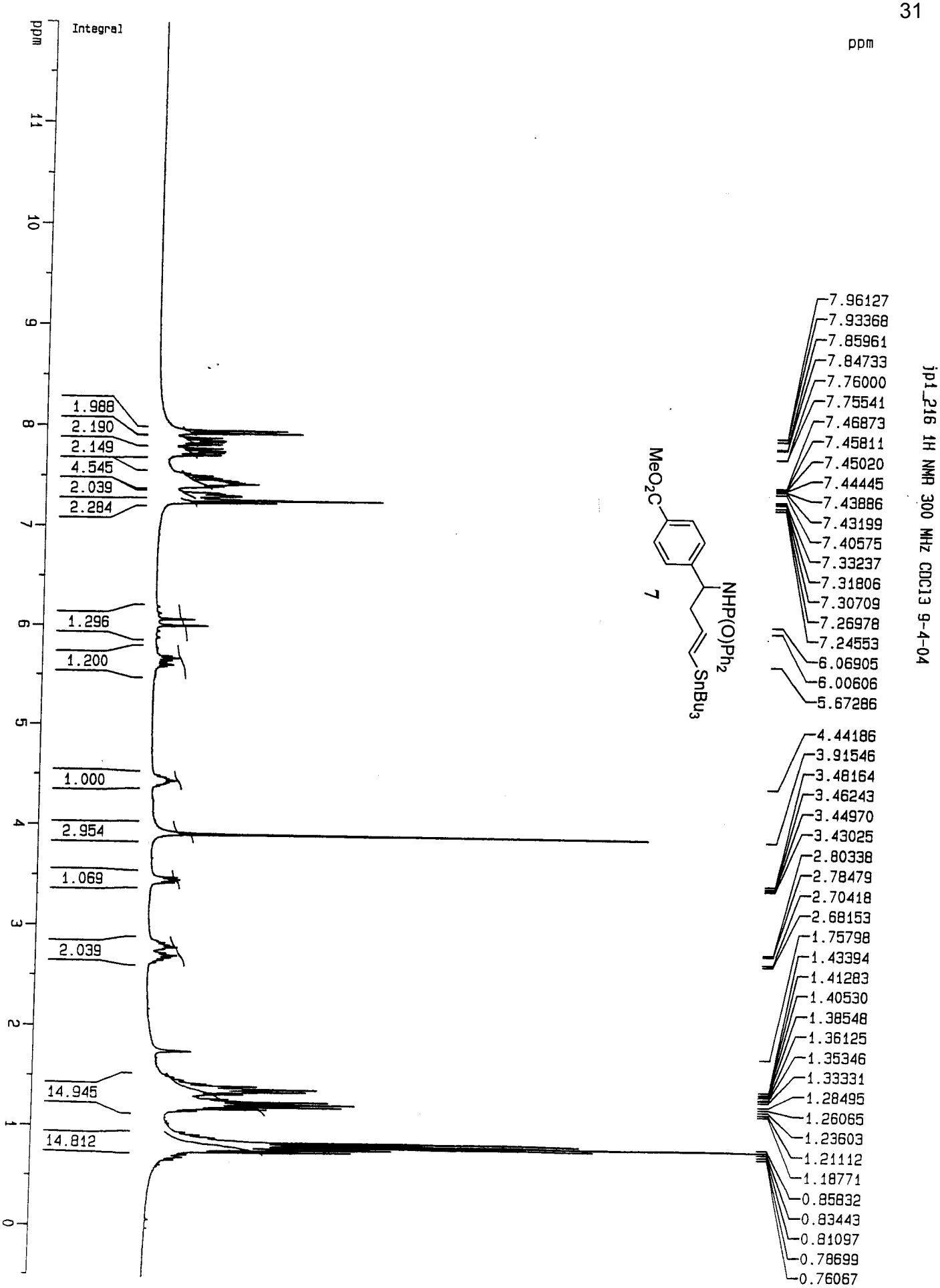
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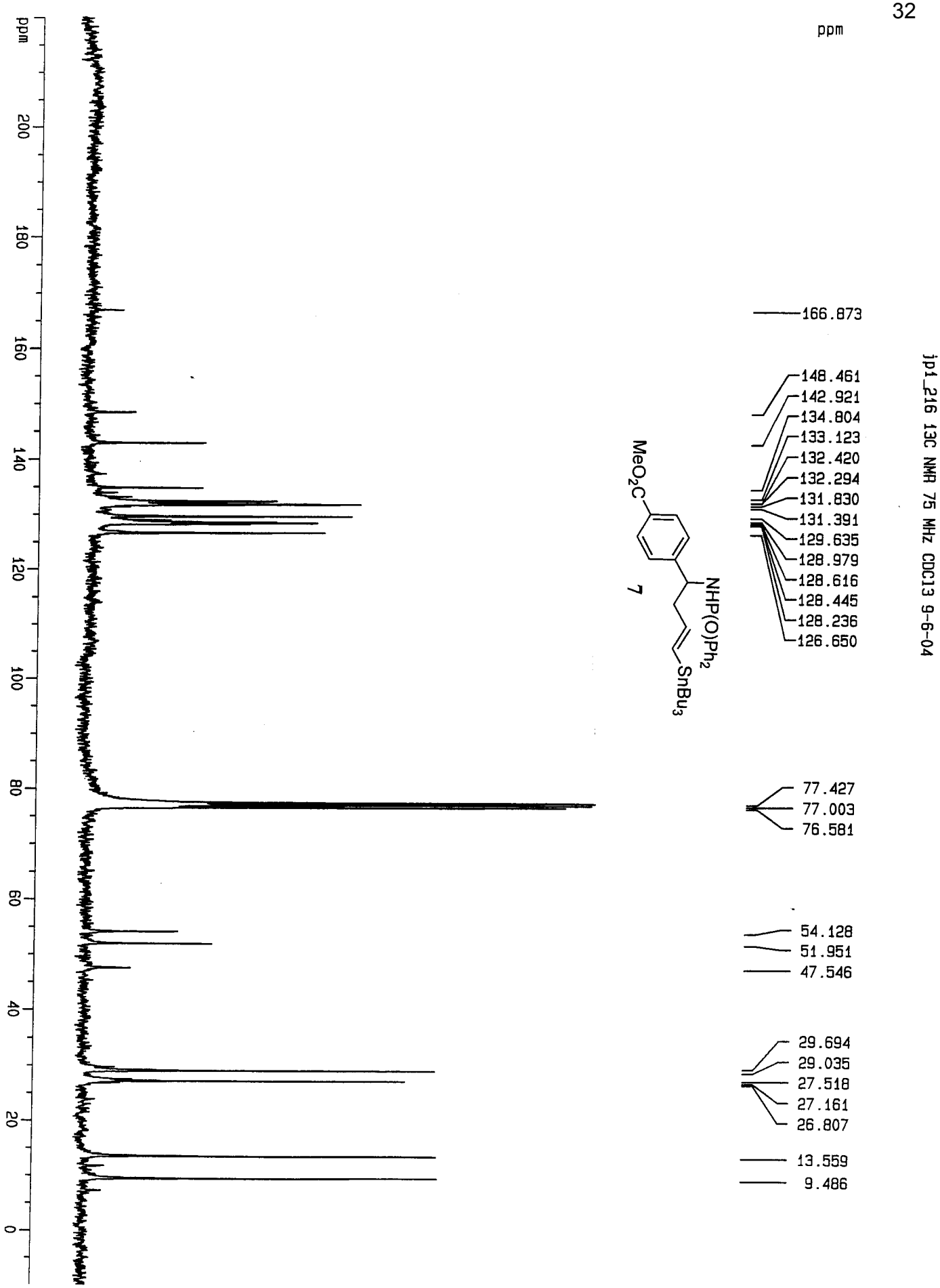
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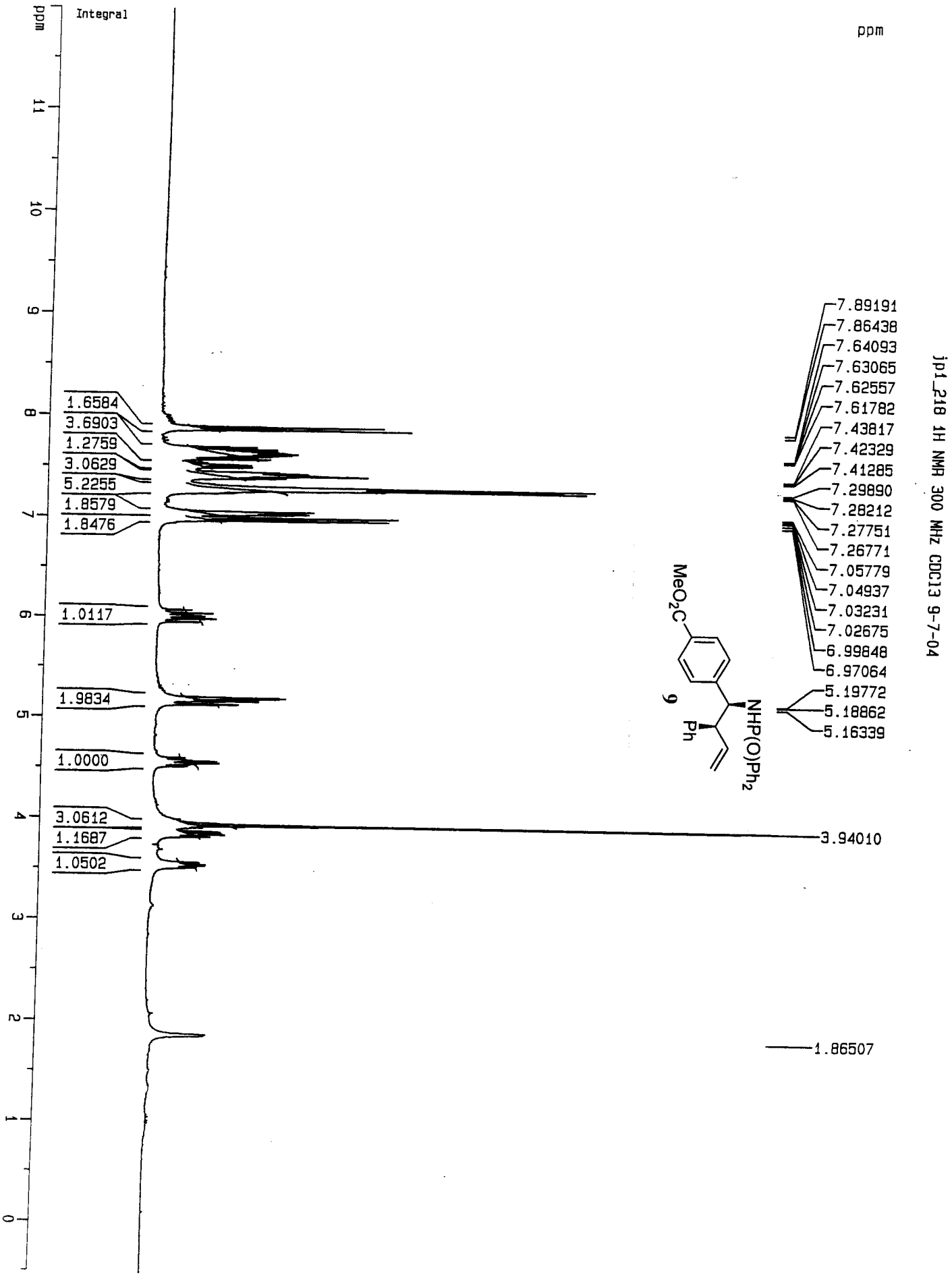
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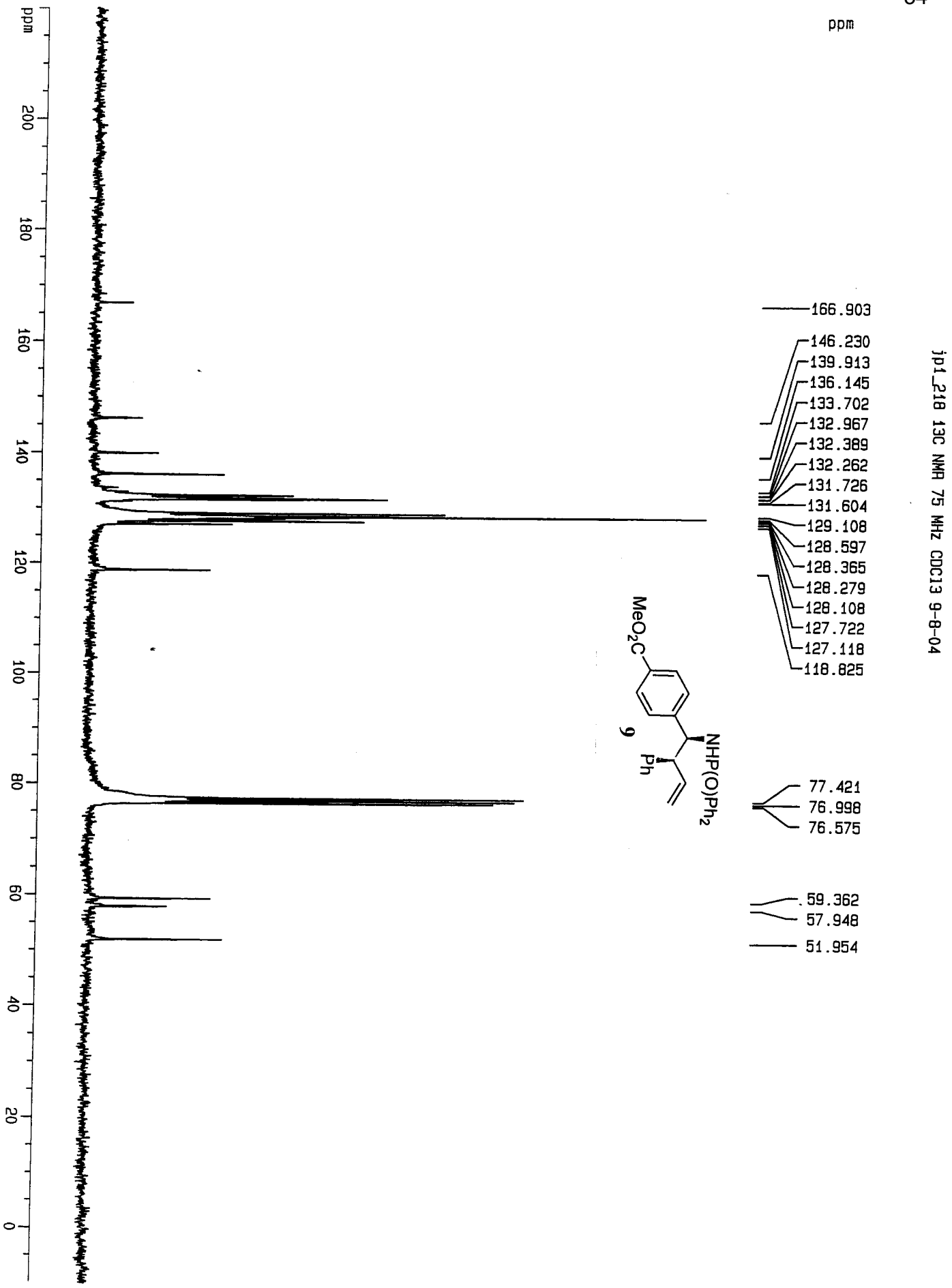
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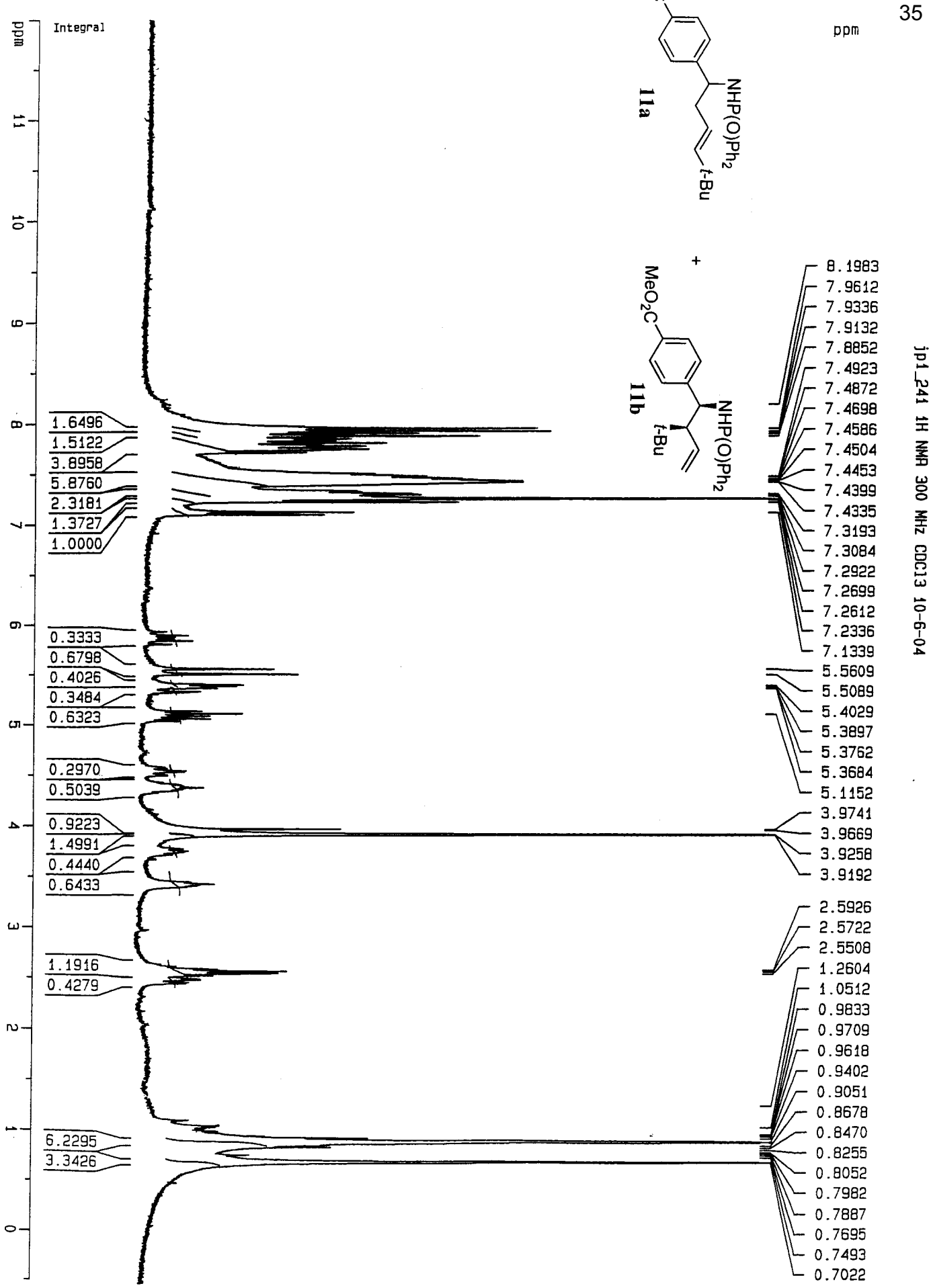
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0.4440
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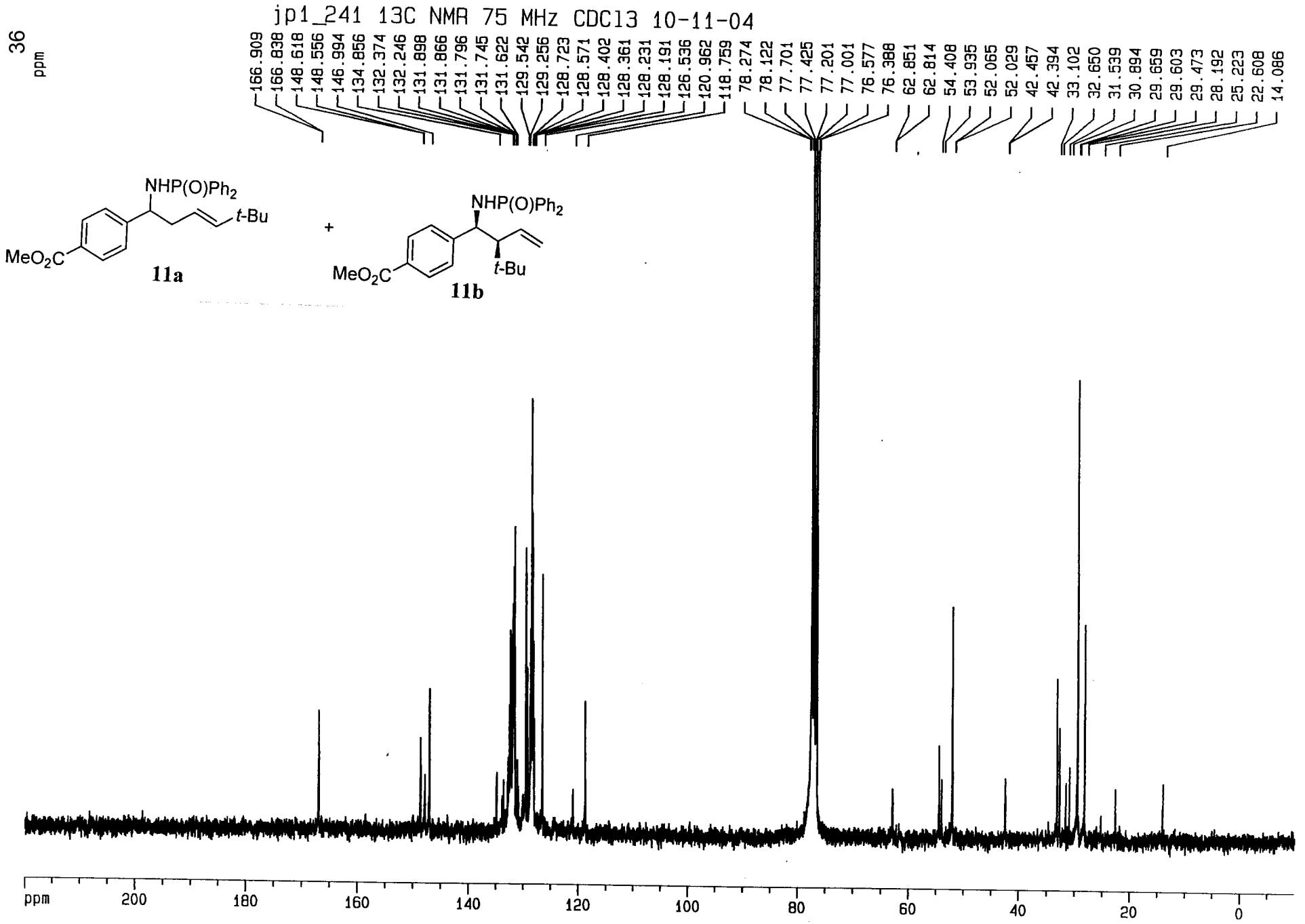
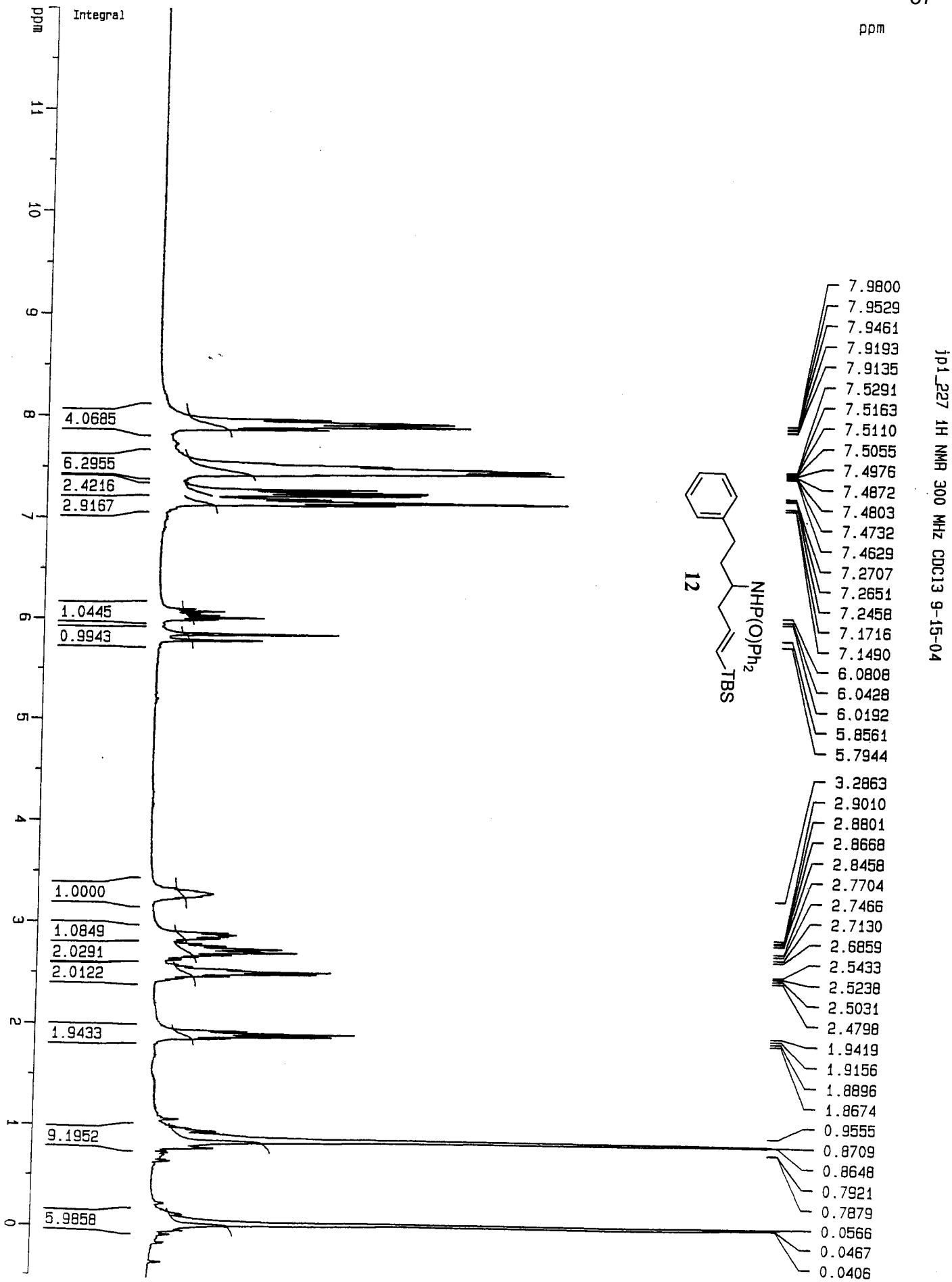
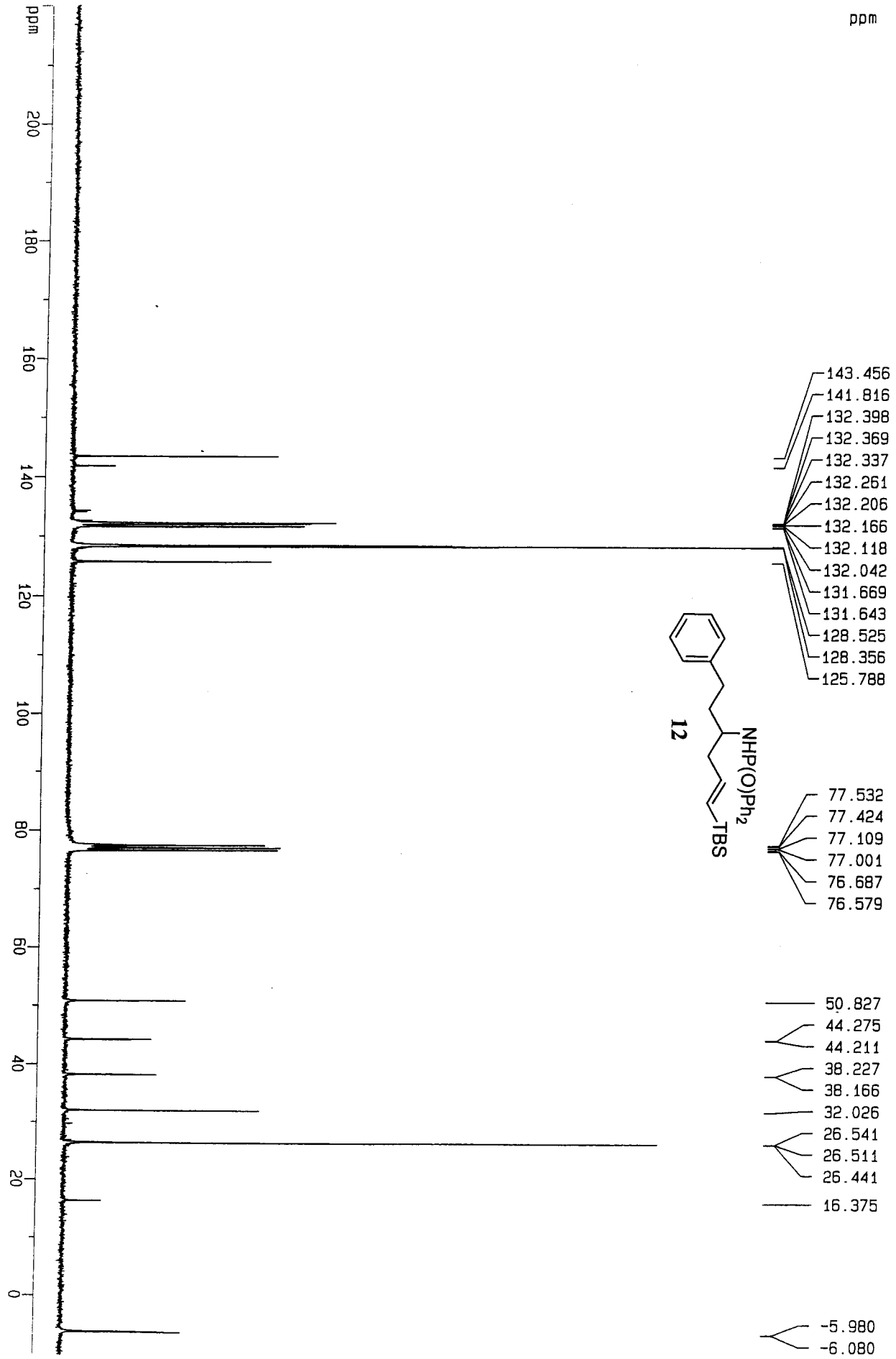
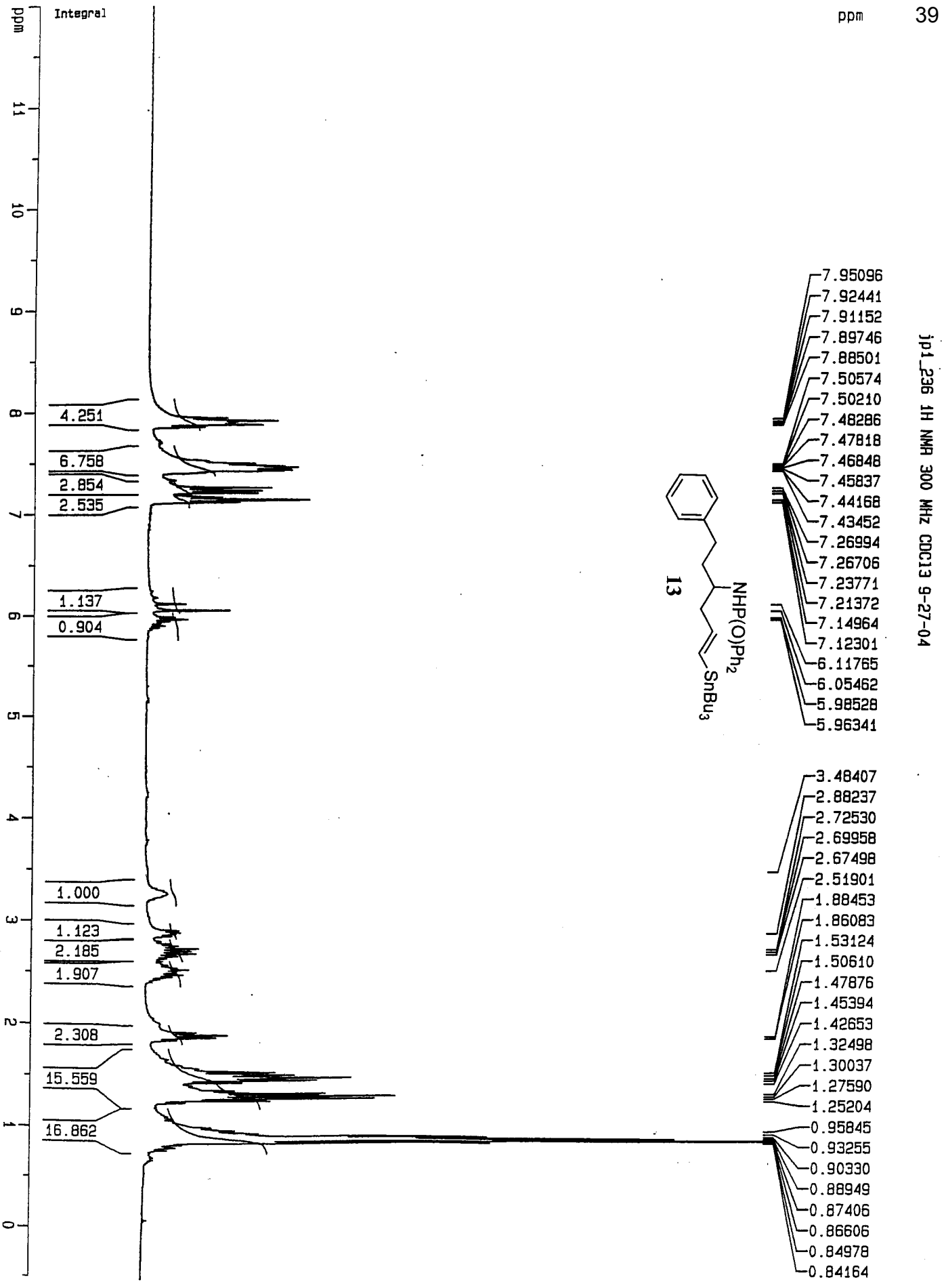
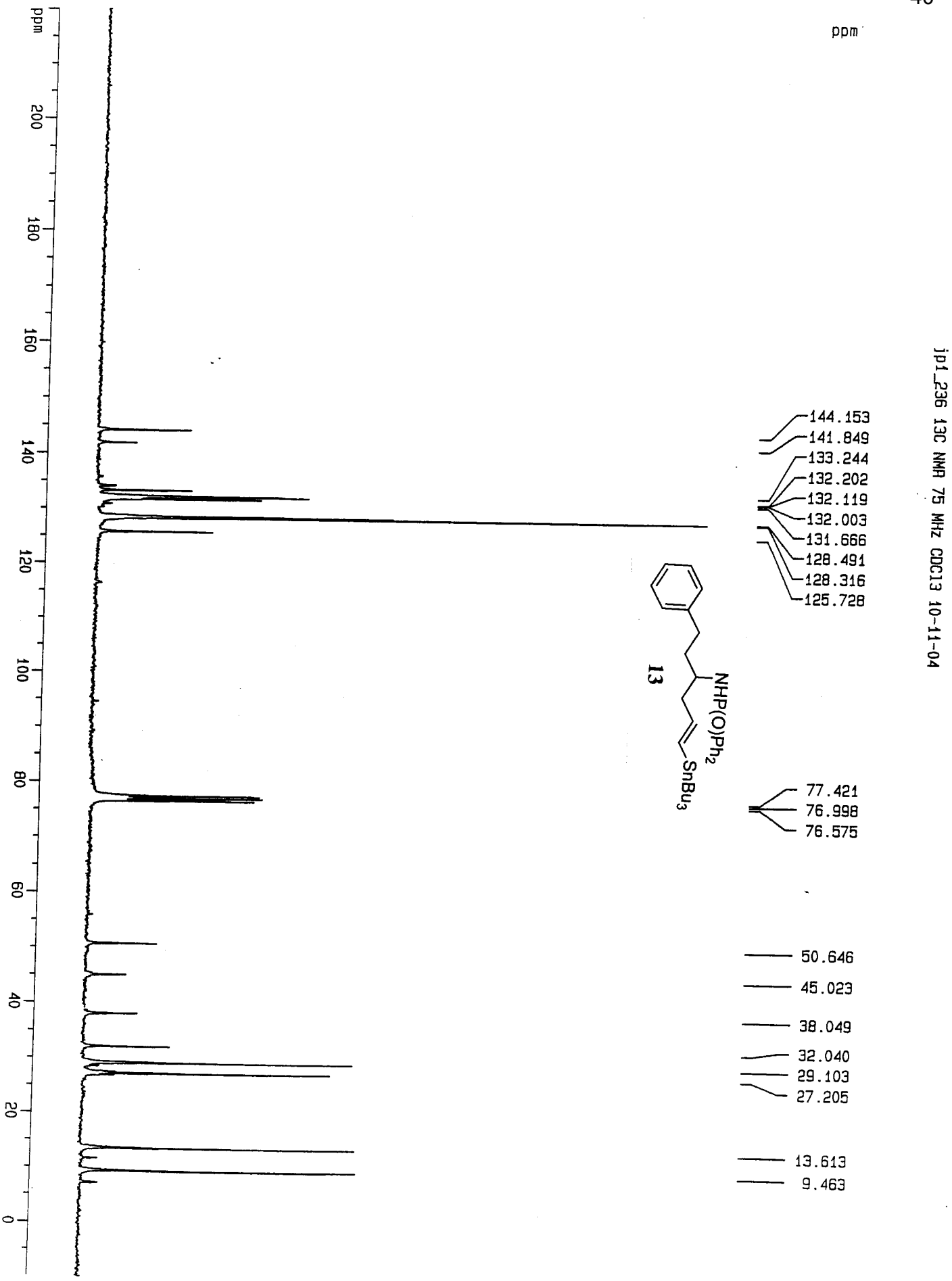
1.1916
0.4279
6.2295
3.3426
8.1983
7.9612
7.9336
7.9132
7.8852
7.4923
7.4872
7.4698
7.4586
7.4504
7.4453
7.4399
7.4335
7.3193
7.3084
7.2922
7.2699
7.2612
7.2336
7.1339
5.5609
5.5089
5.4029
5.3897
5.3762
5.3684
5.1152
3.9741
3.9669
3.9258
3.9192
2.5926
2.5722
2.5508
1.2604
1.0512
0.9833
0.9709
0.9618
0.9402
0.9051
0.8678
0.8470
0.8255
0.8052
0.7982
0.7887
0.7695
0.7493
0.7022
jp1_241 1H NMR 300 MHz CDCl3 10-6-04

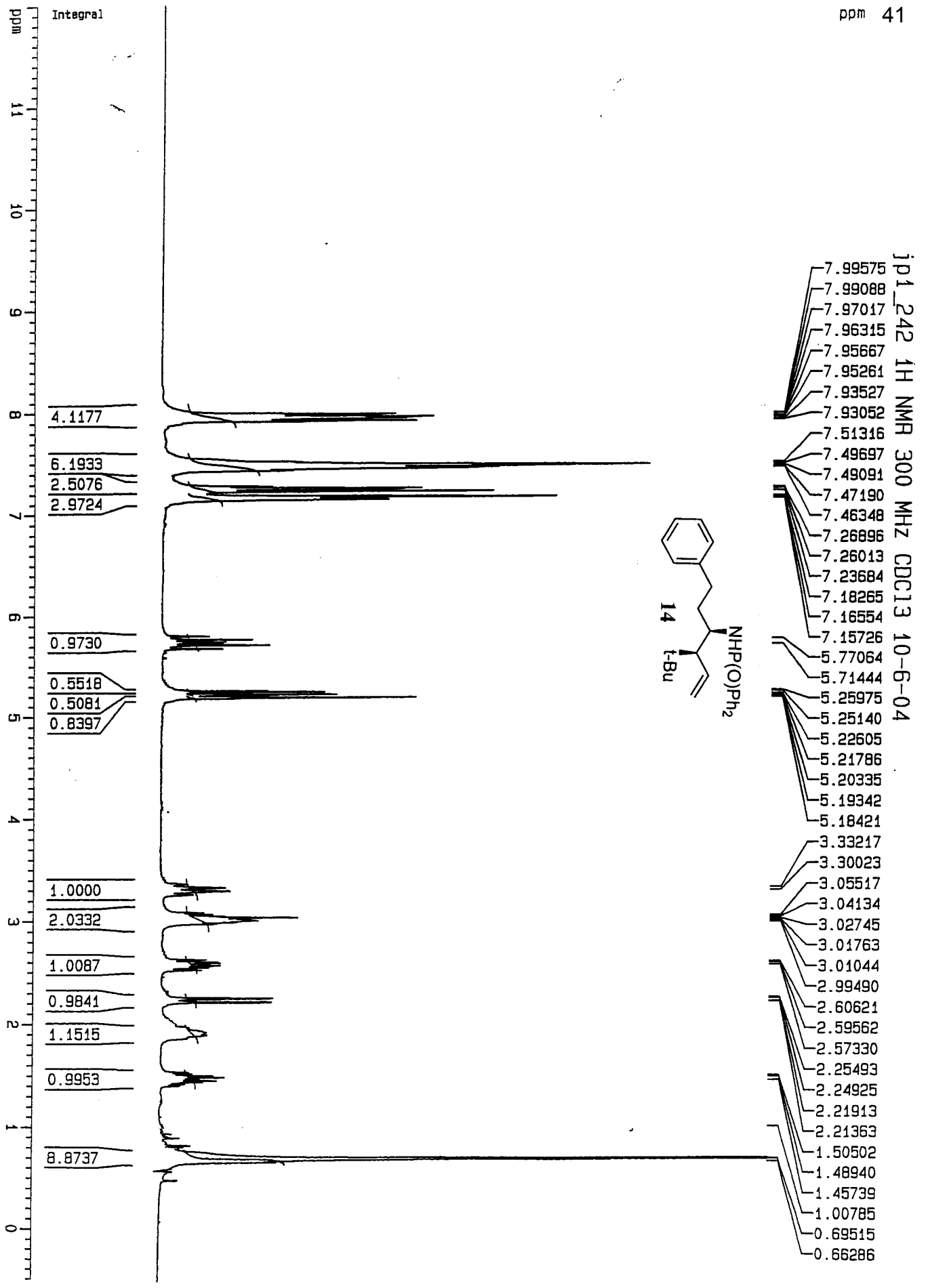

jp1_241 13C NMR 75 MHz CDCl3 10-11-04
36
ppm
166.909
166.838
148.618
148.556
146.994
134.856
132.374
132.246
131.898
131.866
131.796
131.745
131.622
129.542
129.256
128.723
128.571
128.402
128.361
128.231
128.191
126.536
120.962
118.759
78.274
78.122
77.701
77.425
77.201
77.001
76.577
76.388
62.851
62.814
54.408
53.935
52.065
52.029
42.457
42.394
33.102
32.650
31.539
30.894
29.659
29.603
29.473
28.192
25.223
22.608
14.086
NHP(O)Ph2
t-Bu
MeO2C
11a
+
NHP(O)Ph2
t-Bu
MeO2C
11b
ppm
200
180
160
140
120
100
80
60
40
20
0


Integral
ppm
4.0685
6.2955
2.4216
2.9167
1.0445
0.9943
1.0000
1.0849
2.0291
2.0122
1.9433
9.1952
5.9858
12
NHP(O)Ph2
TBS
ppm
7.9800
7.9529
7.9461
7.9193
7.9135
7.5291
7.5163
7.5110
7.5055
7.4976
7.4872
7.4803
7.4732
7.4629
7.2707
7.2651
7.2458
7.1716
7.1490
6.0808
6.0428
6.0192
5.8561
5.7944
3.2863
2.9010
2.8801
2.8668
2.8458
2.7704
2.7466
2.7130
2.6859
2.5433
2.5238
2.5031
2.4798
1.9419
1.9156
1.8896
1.8674
0.9555
0.8709
0.8648
0.7921
0.7879
0.0566
0.0467
0.0406
jp1_227 1H NMR 300 MHz CDCl3 9-15-04

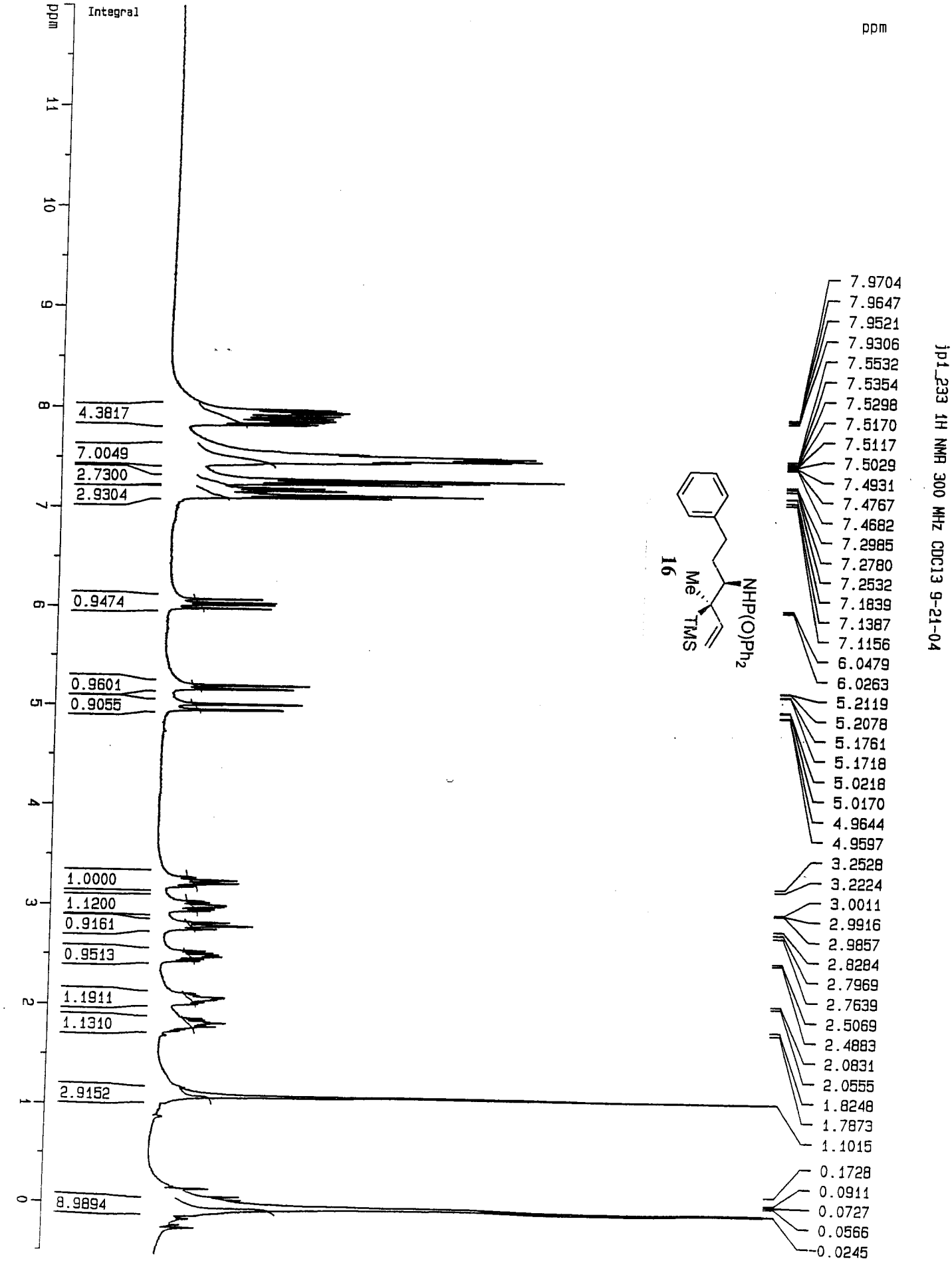

ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
143.456
141.816
132.398
132.369
132.337
132.261
132.206
132.166
132.118
132.042
131.669
131.643
128.525
128.356
125.788
77.532
77.424
77.109
77.001
76.687
76.579
50.827
44.275
44.211
38.227
38.166
32.026
26.541
26.511
26.441
16.375
-5.980
-6.080
12
NHP(O)Ph2
TBS
jp1_227 13C NMR 75MHz CDCl3 9-22-04

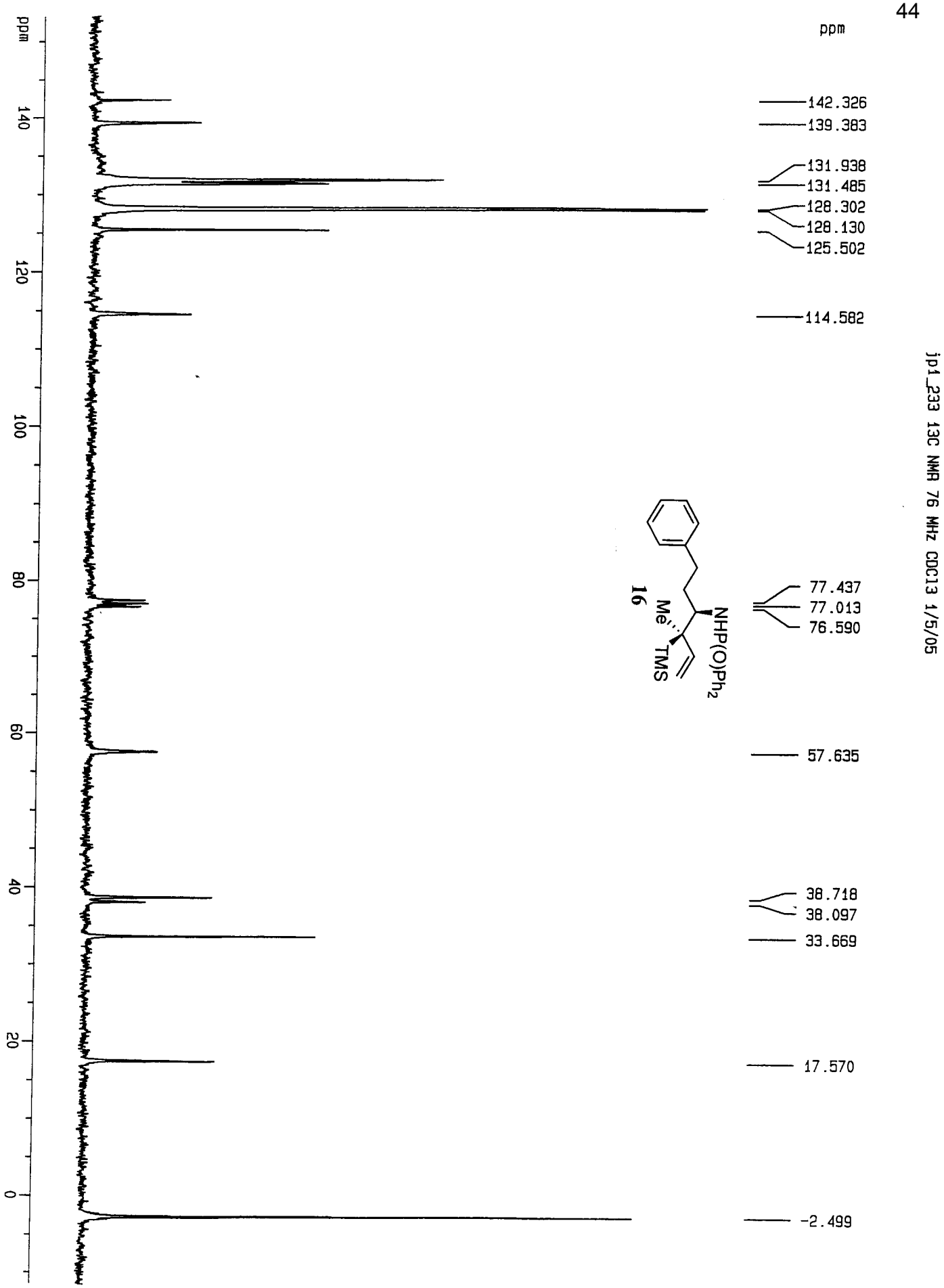

ppm
Integral
ppm
4.251
6.758
2.854
2.535
1.137
0.904
1.000
1.123
2.185
1.907
2.308
15.559
16.862
13
NHP(O)Ph2
SnBu3
7.95096
7.92441
7.91152
7.89746
7.88501
7.50574
7.50210
7.48286
7.47818
7.46848
7.45837
7.44168
7.43452
7.26994
7.26706
7.23771
7.21372
7.14964
7.12301
6.11765
6.05462
5.98528
5.96341
3.48407
2.88237
2.72530
2.69958
2.67498
2.51901
1.88453
1.86083
1.53124
1.50610
1.47876
1.45394
1.42653
1.32498
1.30037
1.27590
1.25204
0.95845
0.93255
0.90330
0.88949
0.87406
0.86606
0.84978
0.84164
jp1_236 1H NMR 300 MHz CDCl3 9-27-04

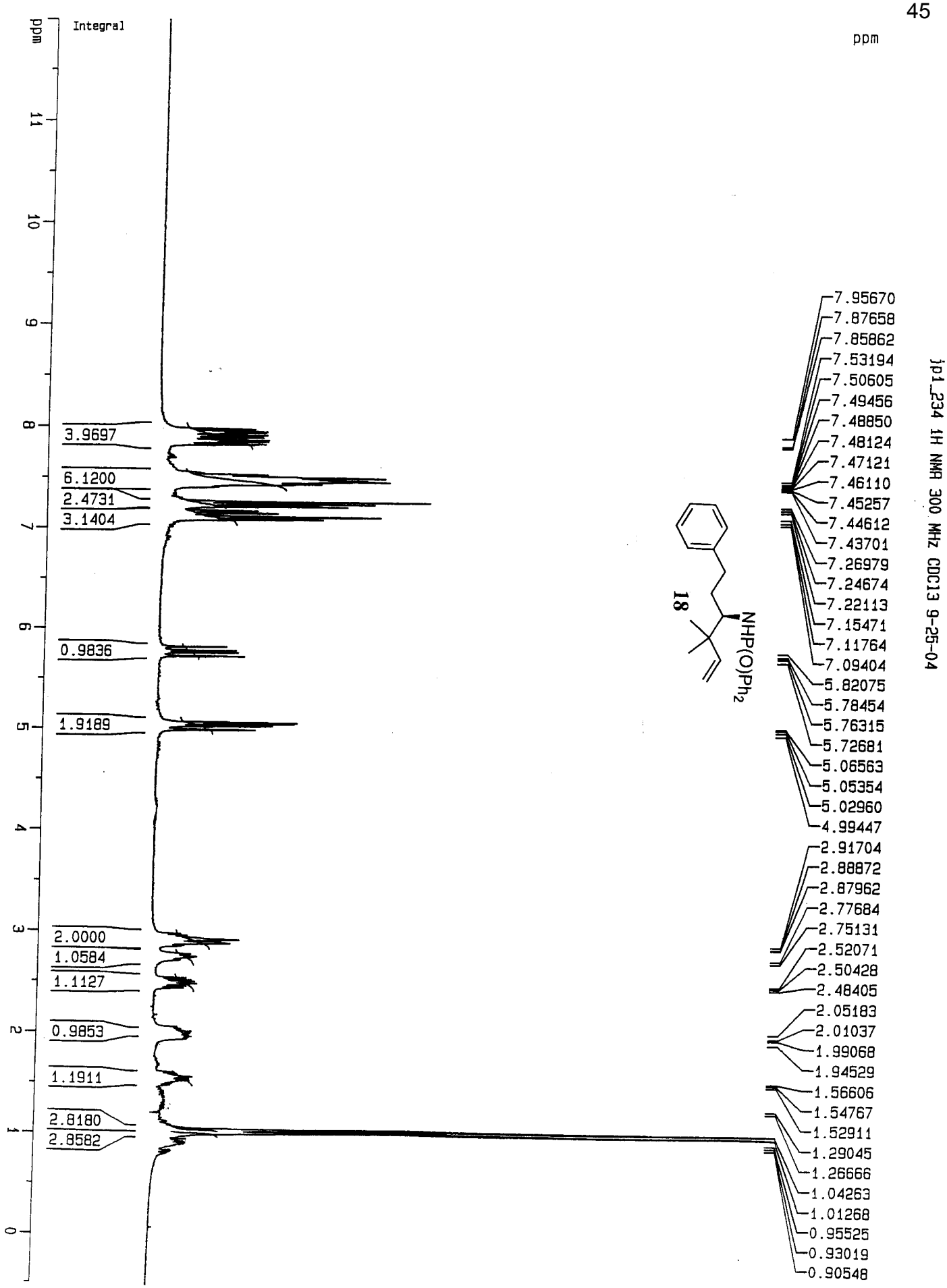

ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
jp1_236 13C NMR 75 MHz CDCl3 10-11-04
144.153
141.849
133.244
132.202
132.119
132.003
131.666
128.491
128.316
125.728
77.421
76.998
76.575
50.646
45.023
38.049
32.040
29.103
27.205
13.613
9.463
NHP(O)Ph2
SnBu3
13

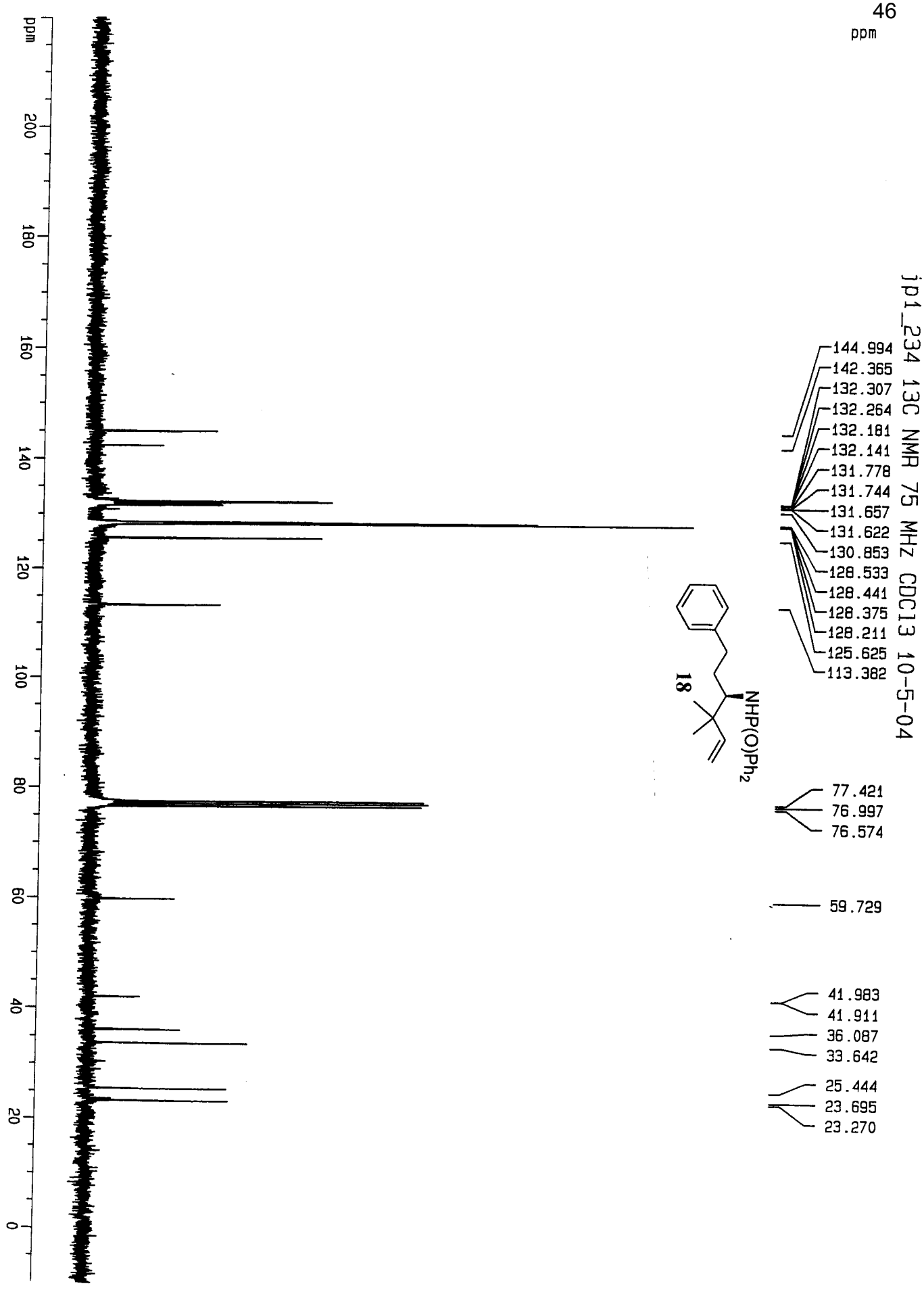

jp1_242 1H NMR 300 MHz CDCl3 10-6-04
Integral
ppm
4.1177
6.1933
2.5076
2.9724
0.9730
0.5518
0.5081
0.8397
1.0000
2.0332
1.0087
0.9841
1.1515
0.9953
8.8737
14
t-Bu
NHP(O)Ph2
7.99575
7.99088
7.97017
7.96315
7.95667
7.95261
7.93527
7.93052
7.51316
7.49697
7.49091
7.47190
7.46348
7.26896
7.26013
7.23684
7.18265
7.16554
7.15726
5.77064
5.71444
5.25975
5.25140
5.22605
5.21786
5.20335
5.19342
5.18421
3.33217
3.30023
3.05517
3.04134
3.02745
3.01763
3.01044
2.99490
2.60621
2.59562
2.57330
2.25493
2.24925
2.21913
2.21363
1.50502
1.48940
1.45739
1.00785
0.69515
0.66286

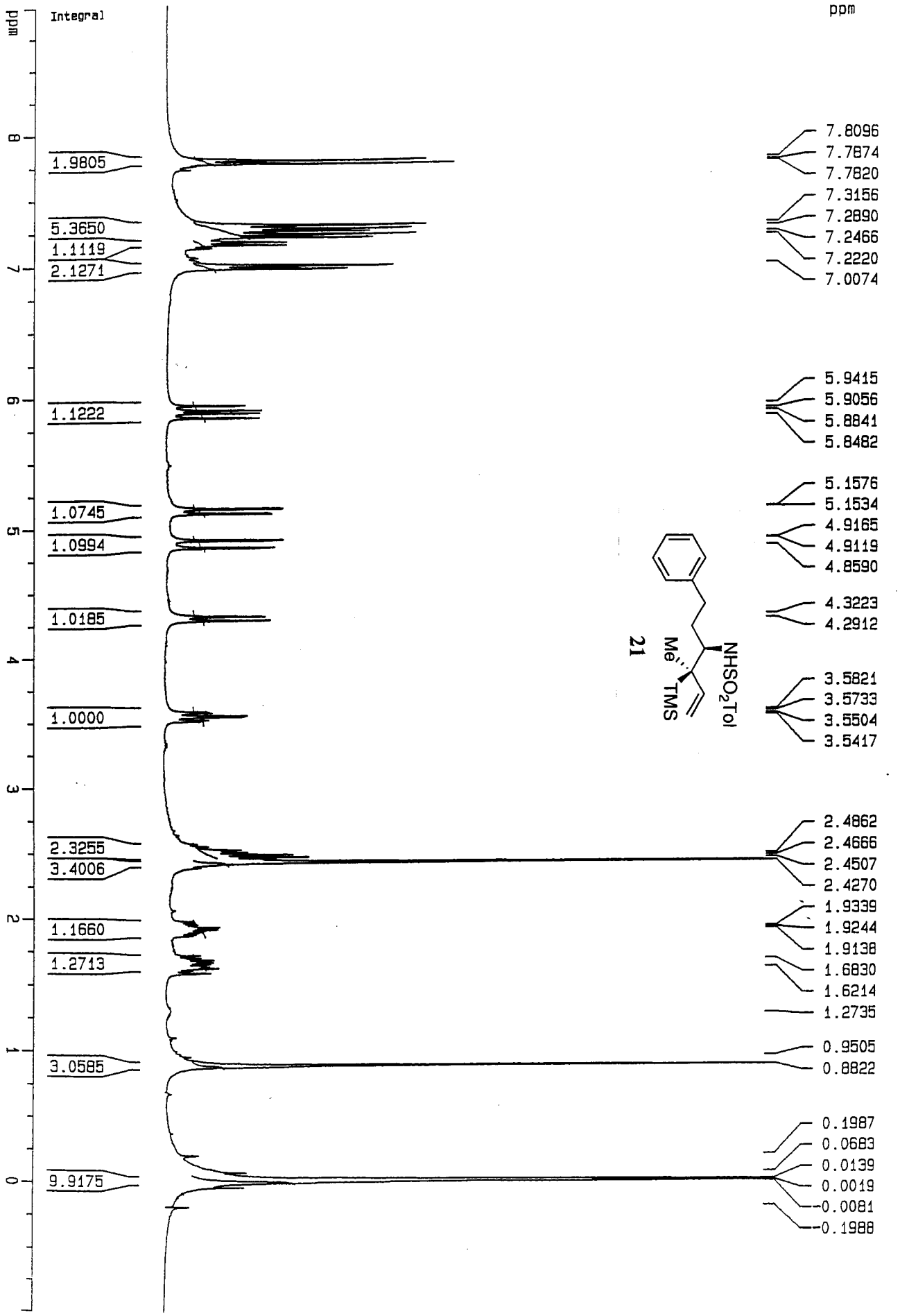
ppm
jp1_242 13C NMR 75 MHz CDCl3 10-7-04
142.505
135.475
132.473
132.360
131.850
128.741
128.604
128.405
125.835
119.480
14
t-Bu
NHP(O)Ph2
77.565
77.141
76.717
62.150
51.620
36.623
33.290
32.488
28.730


ppm
Integral
4.3817
7.0049
2.7300
2.9304
0.9474
0.9601
0.9055
1.0000
1.1200
0.9161
0.9513
1.1911
1.1310
2.9152
8.9894
16
NHP(O)Ph2
Me
TMS
ppm
7.9704
7.9647
7.9521
7.9306
7.5532
7.5354
7.5298
7.5170
7.5117
7.5029
7.4931
7.4767
7.4682
7.2985
7.2780
7.2532
7.1839
7.1387
7.1156
6.0479
6.0263
5.2119
5.2078
5.1761
5.1718
5.0218
5.0170
4.9644
4.9597
3.2528
3.2224
3.0011
2.9916
2.9857
2.8284
2.7969
2.7639
2.5069
2.4883
2.0831
2.0555
1.8248
1.7873
1.1015
0.1728
0.0911
0.0727
0.0566
-0.0245
jp1_233 1H NMR 300 MHz CDCl3 9-21-04


ppm
140
120
100
80
60
40
20
0
ppm
142.326
139.383
131.938
131.485
128.302
128.130
125.502
114.582
77.437
77.013
76.590
57.635
38.718
38.097
33.669
17.570
-2.499
NHP(O)Ph2
Me
TMS
16
jp1_233 13C NMR 76 MHz CDCl3 1/5/05


ppm
Integral
3.9697
6.1200
2.4731
3.1404
0.9836
1.9189
2.0000
1.0584
1.1127
0.9853
1.1911
2.8180
2.8582
18
NHP(O)Ph2
ppm
7.95670
7.87658
7.85862
7.53194
7.50605
7.49456
7.48850
7.48124
7.47121
7.46110
7.45257
7.44612
7.43701
7.26979
7.24674
7.22113
7.15471
7.11764
7.09404
5.82075
5.78454
5.76315
5.72681
5.06563
5.05354
5.02960
4.99447
2.91704
2.88872
2.87962
2.77684
2.75131
2.52071
2.50428
2.48405
2.05183
2.01037
1.99068
1.94529
1.56606
1.54767
1.52911
1.29045
1.26666
1.04263
1.01268
0.95525
0.93019
0.90548
jp1_234 1H NMR 300 MHz CDCl3 9-25-04


ppm
jp1_234 13C NMR 75 MHz CDCl3 10-5-04
144.994
142.365
132.307
132.264
132.181
132.141
131.778
131.744
131.657
131.622
130.853
128.533
128.441
128.375
128.211
125.625
113.382
77.421
76.997
76.574
59.729
41.983
41.911
36.087
33.642
25.444
23.695
23.270
18
NHP(O)Ph2


Integral
ppm
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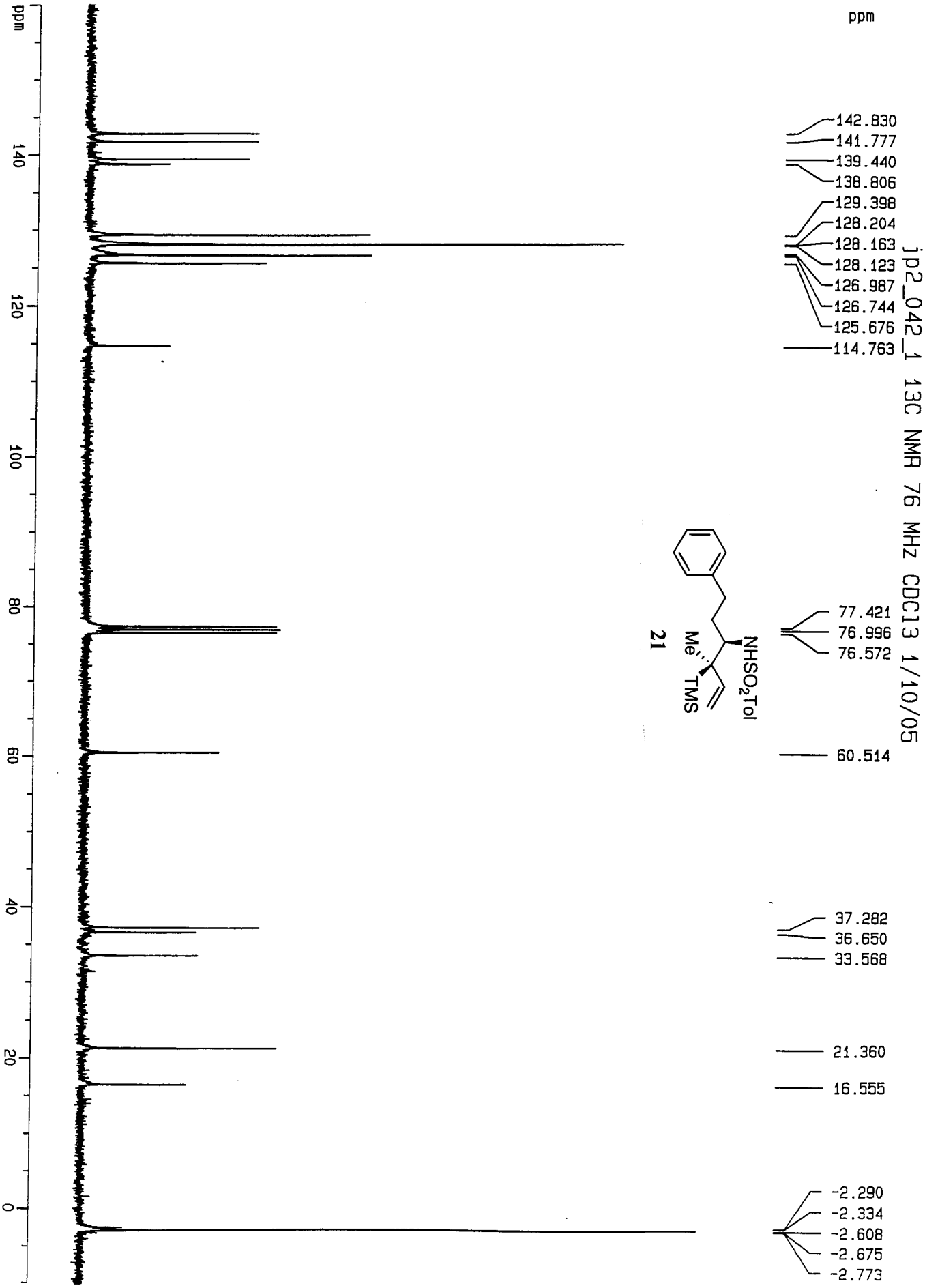
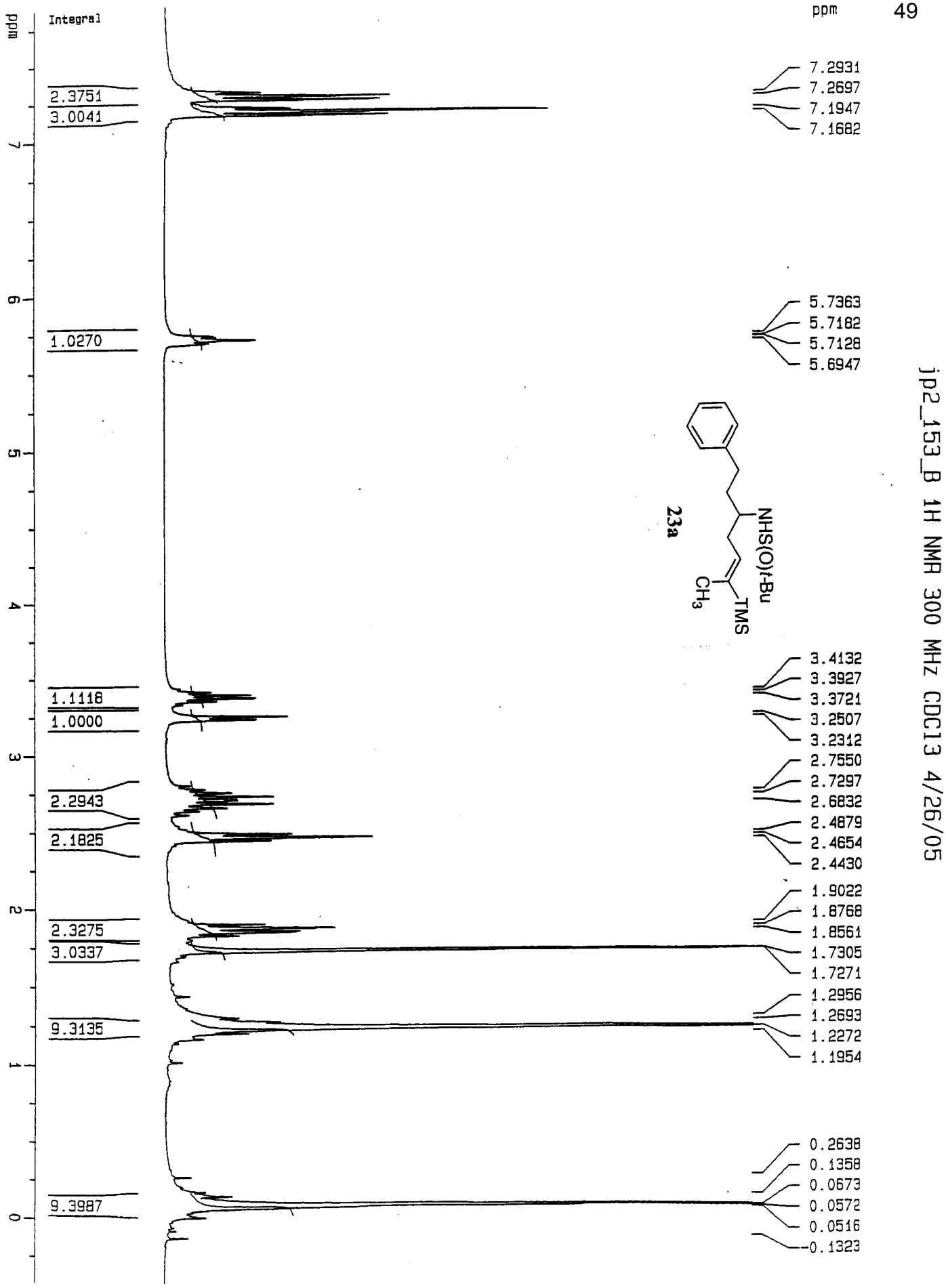
2.1271
1.1222
1.0745
1.0994
1.0185
1.0000
2.3255
3.4006
1.1660
1.2713
3.0585
9.9175
7.8096
7.7874
7.7820
7.3156
7.2890
7.2466
7.2220
7.0074
5.9415
5.9056
5.8841
5.8482
5.1576
5.1534
4.9165
4.9119
4.8590
4.3223
4.2912
3.5821
3.5733
3.5504
3.5417
2.4862
2.4666
2.4507
2.4270
1.9339
1.9244
1.9138
1.6830
1.6214
1.2735
0.9505
0.8822
0.1987
0.0683
0.0139
0.0019
-0.0081
-0.1988
NHSO2Tol
Me
TMS
21
jp2_042_1 1H NMR 300 MHz CDCl3 1/10/05


ppm
140
120
100
80
60
40
20
0
ppm
142.830
141.777
139.440
138.806
129.398
128.204
128.163
128.123
126.987
126.744
125.676
114.763
77.421
76.996
76.572
60.514
37.282
36.650
33.568
21.360
16.555
-2.290
-2.334
-2.608
-2.675
-2.773
jp2_042_1 13C NMR 76 MHz CDCl3 1/10/05
NHSO2Tol
Me
TMS
21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Integral
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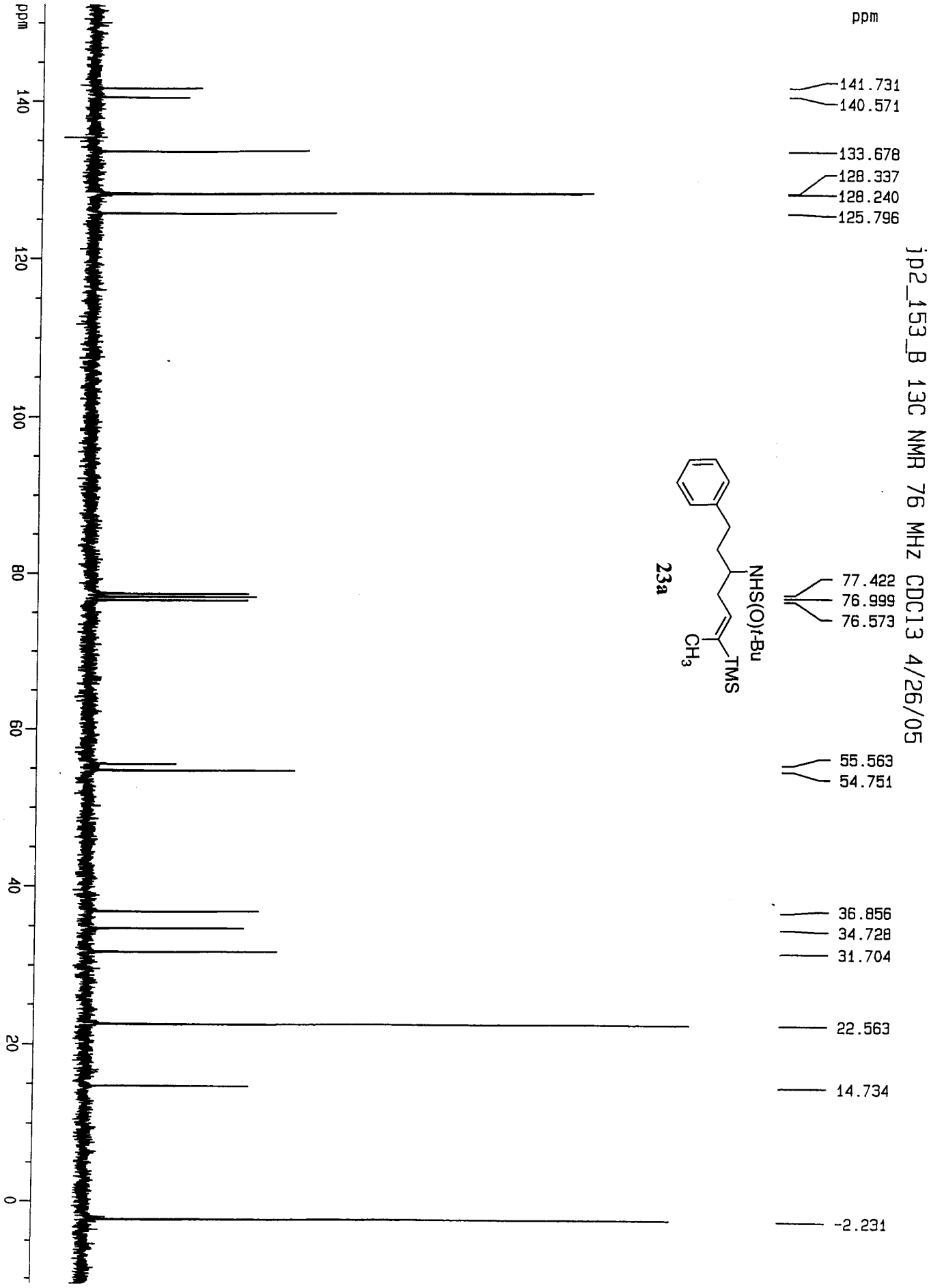
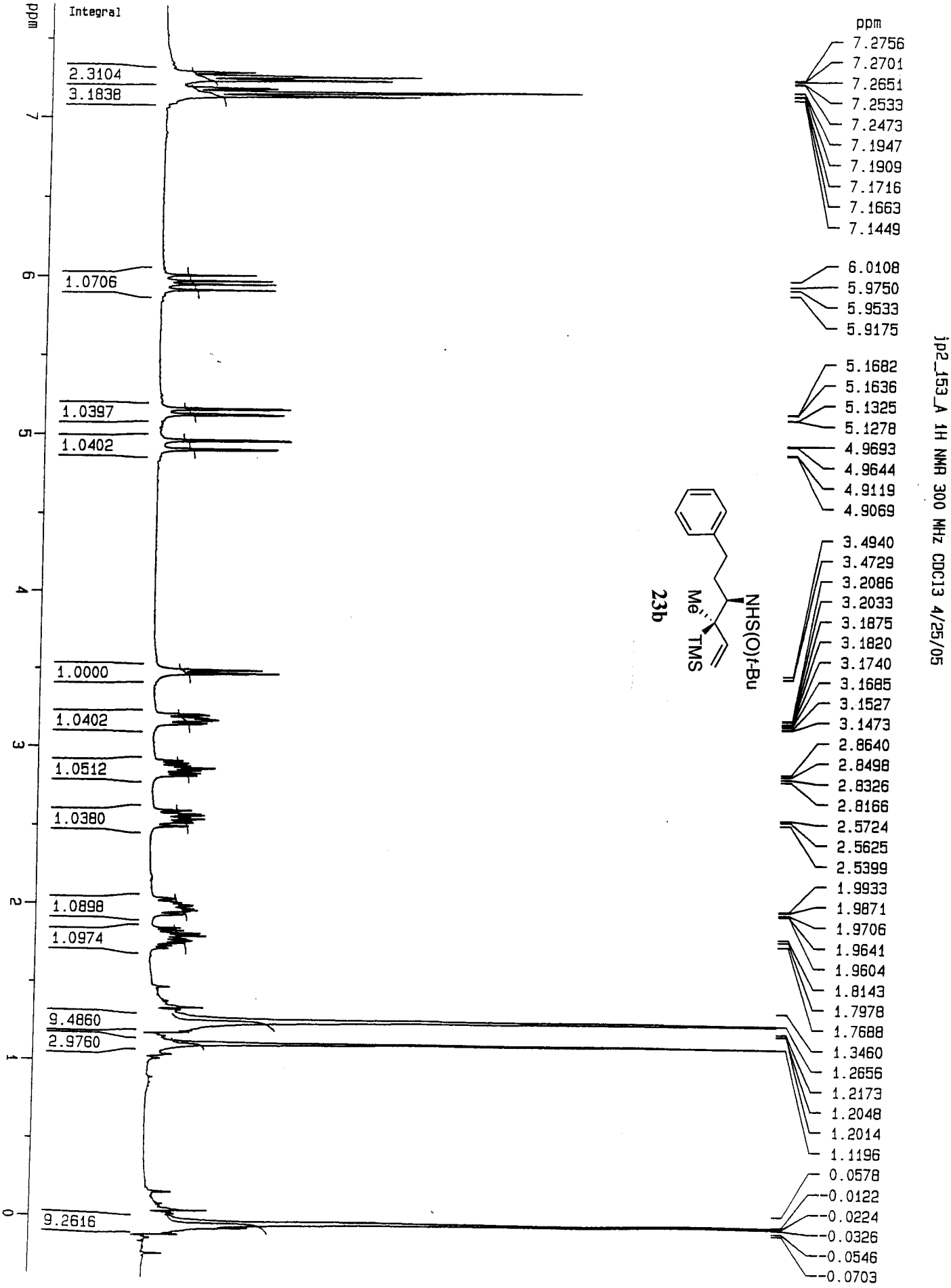
3.0041
1.0270
1.1118
1.0000
2.2943
2.1825
2.3275
3.0337
9.3135
9.3987
7.2931
7.2697
7.1947
7.1682
5.7363
5.7182
5.7128
5.6947
NHS(O)t-Bu
TMS
CH3
23a
3.4132
3.3927
3.3721
3.2507
3.2312
2.7550
2.7297
2.6832
2.4879
2.4654
2.4430
1.9022
1.8768
1.8561
1.7305
1.7271
1.2956
1.2693
1.2272
1.1954
0.2638
0.1358
0.0673
0.0572
0.0516
-0.1323
jp2_153_B 1H NMR 300 MHz CDCl3 4/26/05


ppm
140
120
100
80
60
40
20
0
ppm
141.731
140.571
133.678
128.337
128.240
125.796
77.422
76.999
76.573
55.563
54.751
36.856
34.728
31.704
22.563
14.734
-2.231
NHS(O)t-Bu
TMS
CH3
23a
jp2_153_B 13C NMR 76 MHz CDCl3 4/26/05


ppm
Integral
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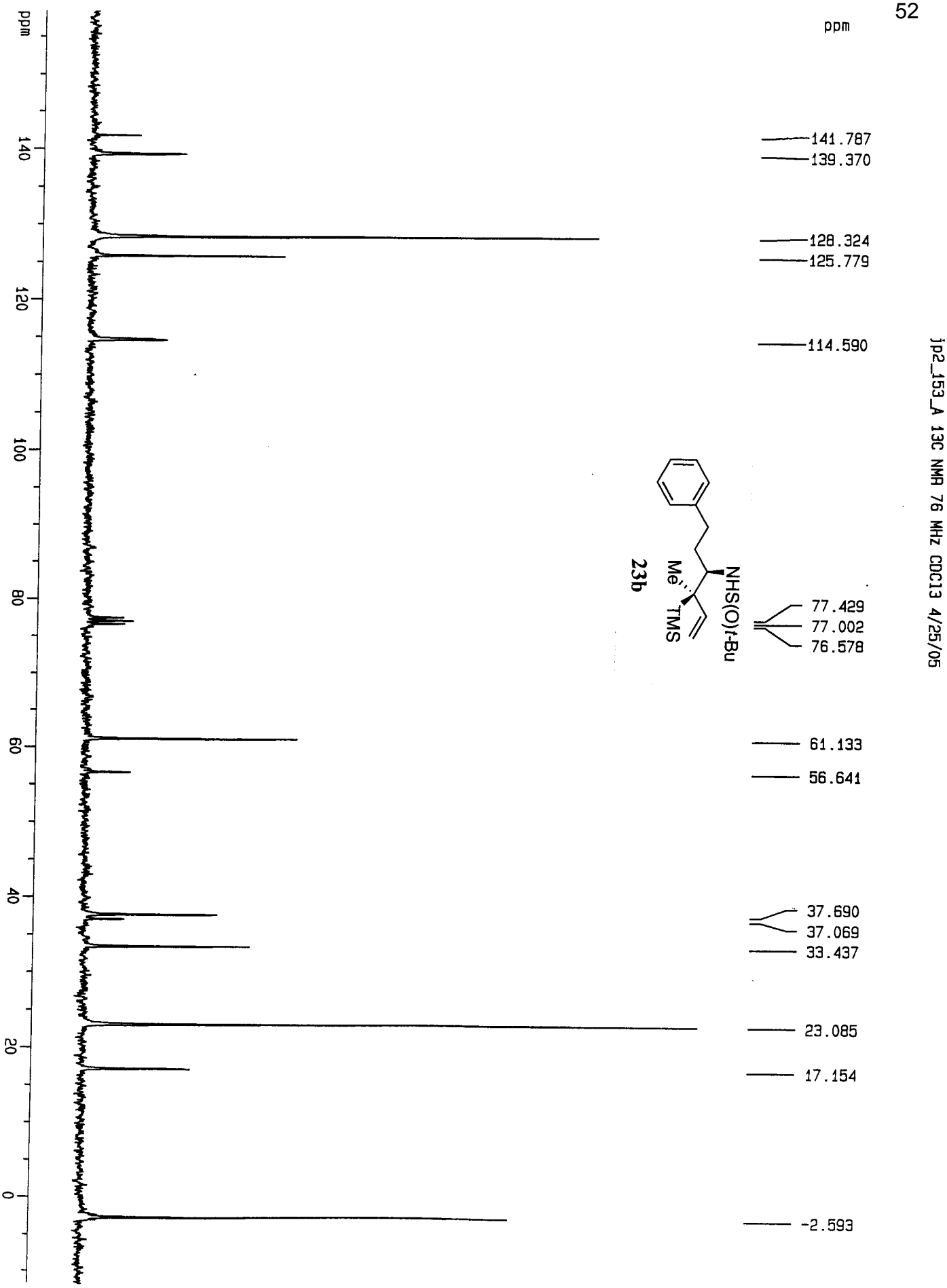
3.1838
1.0706
1.0397
1.0402
1.0000
1.0402
1.0512
1.0380
1.0898
1.0974
9.4860
2.9760
9.2616
ppm
7.2756
7.2701
7.2651
7.2533
7.2473
7.1947
7.1909
7.1716
7.1663
7.1449
6.0108
5.9750
5.9533
5.9175
5.1682
5.1636
5.1325
5.1278
4.9693
4.9644
4.9119
4.9069
3.4940
3.4729
3.2086
3.2033
3.1875
3.1820
3.1740
3.1685
3.1527
3.1473
2.8640
2.8498
2.8326
2.8166
2.5724
2.5625
2.5399
1.9933
1.9871
1.9706
1.9641
1.9604
1.8143
1.7978
1.7688
1.3460
1.2656
1.2173
1.2048
1.2014
1.1196
0.0578
-0.0122
-0.0224
-0.0326
-0.0546
-0.0703
NHS(O)t-Bu
Me
TMS
23b
jp2_153_A 1H NMR 300 MHz CDCl3 4/25/05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128.324
125.779
114.590
77.429
77.002
76.578
61.133
56.641
37.690
37.069
33.437
23.085
17.154
-2.593
NHS(O)t-Bu
Me
TMS
23b
jp2_153_A 13C NMR 76 MHz CDCl3 4/25/05