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Supplementary Information

Remarkable Electron-Withdrawing Effect of $Ph_2P(O)$-Ethynyl Group: $Ph_2P(O)$-Ethynyl-Substituted Aryl Halides and Copper Acetylides for Tailor-Made Sonogashira Couplings

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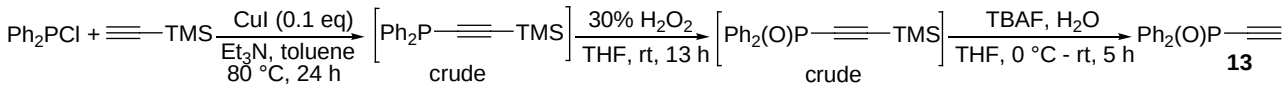
I. General Information.

All reactions were carried out under an atmosphere of nitrogen with freshly distilled solvents, unless otherwise noted. Toluene was distilled from sodium. Diisopropylamine were distilled from CaH_2. Dry tetrahydrofuran (THF) was purchased from Wako Chemicals. $Pd(PPh_3)_4$ was prepared according to the reported method. Silica gel was used for column chromatography. The other materials were purchased from commercial sources and used without additional purification. NMR spectra was recorded at 25 °C on 300 and 500 MHz instruments in $CDCl_3$ and calibrated with tetramethysilane (TMS) as an internal reference. Mass spectra were recorded on MALDI-TOF mass spectrometers. Melting points (m.p.) were measured on a GTO-250RS instrument.

II. Procedure and Characterization.

Synthesis of 13.[1]

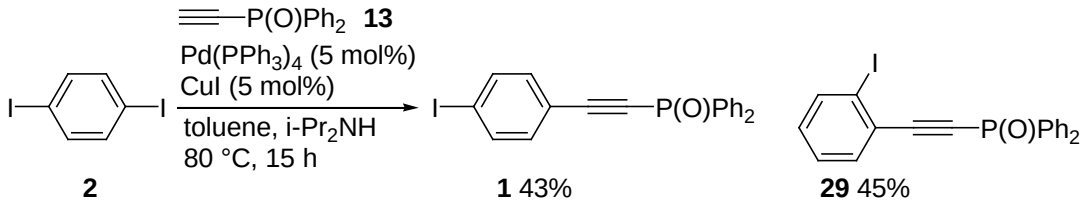


To a flask were added CuI (190.4 mg, 1.0 mmol), Ph_2PCl (1.8 mL, 10.0 mmol), trimethylsilylacetylene (1.7 mL, 12.0 mmol), triethylamine (2.8 mL, 20.0 mmol) and toluene (30.0 mL), and the mixture was stirred under nitrogen at 80 °C for 24 h. After workup with AcOEt/water, the organic layer was washed with aqueous NH_4Cl and brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was used for next step without purification. To the crude product were added THF (20.0 mL) and then 30% H_2O_2 (5.0 mL) at 0 °C, and the mixture was stirred under air at rt for 13 h. After workup with CH_2Cl_2/water, the organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was used for next step without purification. To the crude product were added water (0.5 mL) and THF (50.0 mL), and then TBAF (1.0 M in THF, 1.0 mL, 1.0 mmol) at 0 °C, and the mixture was stirred under air at rt for 5 h. After the solvents were evaporated, the crude product was subjected to

column chromatography on silica gel (hexane/AcOEt, 1:1) to give **13** (1.63 g, 72% yield in 3 steps) in a pure form.

13:[1] white powder; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.33 (d, *J* = 9.8 Hz, 1H), 7.48-7.52 (m, 4H), 7.56-7.59 (m, 2H), 7.83-7.87 (m, 4H).

Synthesis of 1 and 29[2] by Sonogashira coupling (representative procedure for 1).



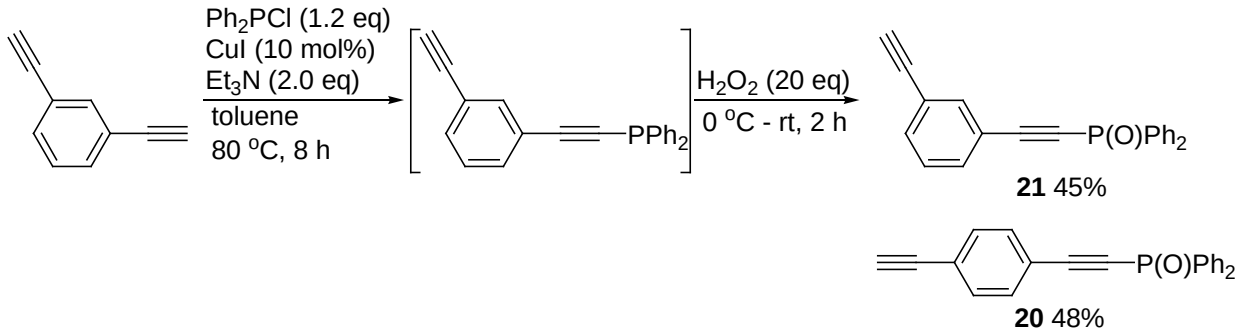
2 **1** 43% **29** 45%

A toluene solution (5 mL) of 1,4-diiodobenzene **2** (659.8 mg, 2.0 mmol), **13** (226.2 mg, 1.0 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:1) to afford **1** in a pure form (184.1 mg, 43% yield).

1: yellow powder, m.p. 163-166 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 (d, *J* = 8.2 Hz, 2H), 7.49-7.52 (m, 4H), 7.56-7.59 (m, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 7.86-7.91 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 83.94 (d, *J* = 167.0 Hz), 97.28, 103.74 (d, *J* = 29.4 Hz), 118.70 (d, *J* = 3.5 Hz), 128.27 (d, *J* = 13.4 Hz), 130.40 (d, *J* = 11.2 Hz), 131.94 (d, *J* = 3.1 Hz), 132.18 (d, *J* = 121.6 Hz), 133.19 (d, *J* = 1.8 Hz), 137.31; ^{31}P NMR (121 MHz, $CDCl_3$): δ 9.87; HRMS (MALDI-TOF) calcd for $C_{20}H_{15}IOP$ ($M+H^+$): 428.9905, found 428.9934.

29:[2] pale-yellow powder, m.p. 112-114 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.14 (t, *J* = 7.8 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.50-7.61 (m, 7H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.96-8.00 (m, 4H).

Synthesis of 20[3] and 21[3] (representative procedure for 21).

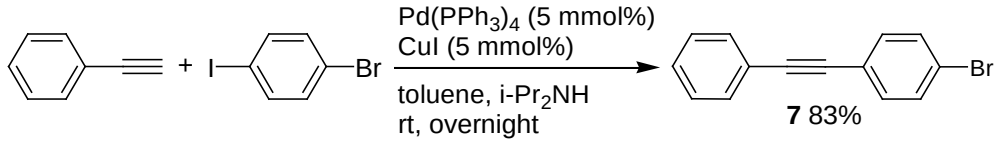


A toluene solution (10 mL) of 1,3-diethynylbenzene (126.2 mg, 1.0 mmol), CuI (19.0 mg, 0.1 mmol), Ph_2PCl (220.6 μL, 1.2 mmol) and Et_3N (277.2 μL, 2.0 mmol) was stirred under nitrogen at 80 °C for 8 h. After usual workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was used for next step without purification. To a THF solution (10.0 mL) of crude product was added H_2O_2aq (30%, 2.5 mL, 20.0 mmol) at 0 °C and the mixture was stirred at rt for 2 h. After workup with CH_2Cl_2 and water, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:1) to afford **21** in a pure form (146.8 mg, 45% yield).

21:[3] white powder; m.p. 111-113 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.12 (s, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.49-7.52 (m, 4H), 7.55-7.58 (m, 4H), 7.71 (s, 1H), 7.87-7.91 (m, 4H).

20:[3] white powder; m.p. 145-147 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.24 (s, 1H), 7.48-7.52 (m, 6H), 7.54-7.58 (m, 4H), 7.87-7.91 (m, 4H).

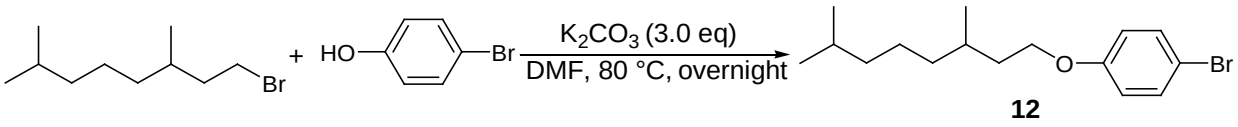
Synthesis of 7.[4]



A toluene solution (5 mL) of ethynylbenzene (102.1 mg, 1.0 mmol), 1-bromo-4-iodobenzene (311.2 mg, 1.1 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at rt

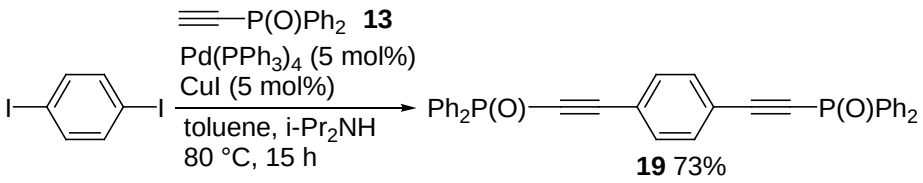
for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane) to afford **7** in a pure form (213.4 mg, 83% yield).

Synthesis of 12.[5]



A DMF solution (5 mL) of 1-bromo-3,7-dimethyloctane (243.3 mg, 1.1 mmol), 4-bromophenol (173.0 mg, 1.0 mmol) and K_2CO_3 (414 mg, 3.0 mmol) was stirred under nitrogen at 80 °C for overnight. After workup with CH_2Cl_2 and dilute HClaq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 6:1) to afford **12** in a pure form (291.3 mg, 93% yield).

Synthesis of 19.

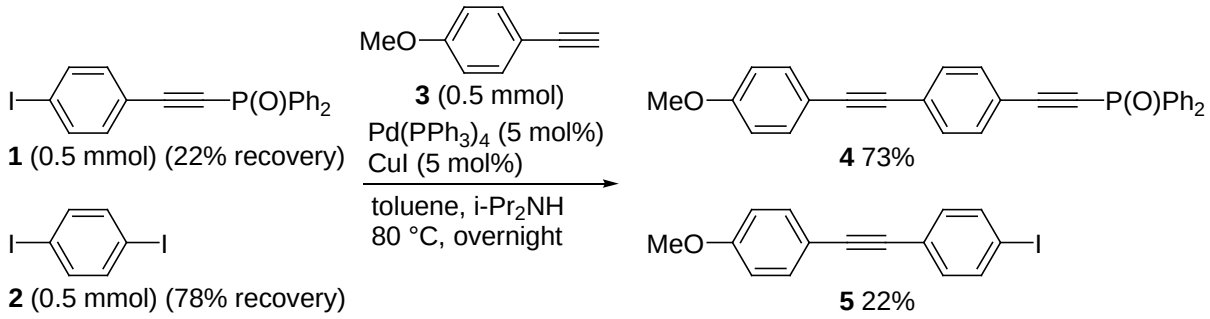


A toluene solution (10 mL) of 1,4-diiodobenzene (329.9 mg, 1.0 mmol), **13** (497.7 mg, 2.2 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (AcOEt) to afford **19** in a pure form (384.3 mg, 73% yield).

19: white powder, m.p. 264-266 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50-7.53 (m, 8H), 7.57-7.61 (m, 8H), 7.87-7.91 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 85.78 (d, *J*

= 164.8 Hz), 103.54 (d, *J* = 28.9 Hz), 122.01 (d, *J* = 4.0 Hz), 128.67 (d, *J* = 13.4 Hz), 130.87 (d, *J* = 11.2 Hz), 132.39 (d, *J* = 3.2 Hz), 132.44 (d, *J* = 122.0 Hz), 132.46 (d, *J* = 1.9 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ 9.88; HRMS (MALDI-TOF) calcd for $C_{34}H_{25}O_2P_2$ ($M+H^+$): 527.1330, found 527.1353.

Competitive Sonogashira coupling of 1 vs 2 with 3.



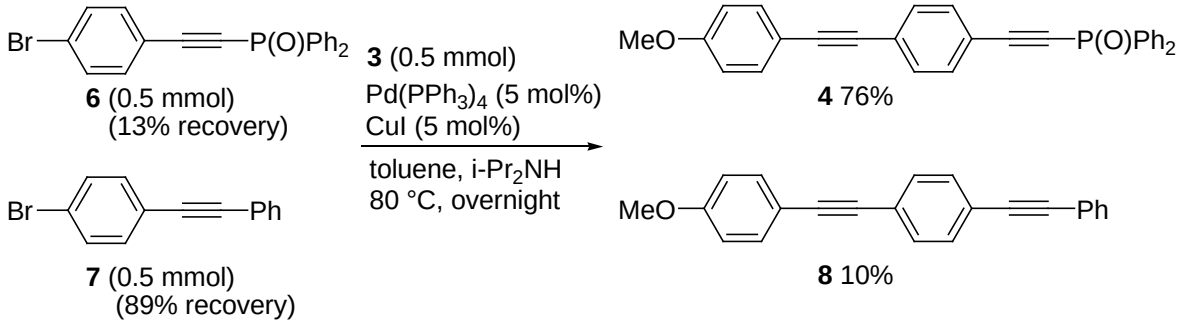
A toluene solution (5.0 mL) of **1** (214.1 mg, 0.5 mmol), **2** (165.0 mg, 0.5 mmol), **3** (66.1 mg, 0.5 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol) and diisopropylamine (0.25 mL) was stirred under nitrogen at 80 °C for overnight. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 10:1, and hexane/AcOEt, 1.5:1) to afford **4** in a pure form (157.8 mg, 73% yield) and **5**[6] in a pure form (36.8 mg, 22% yield) and recover **1** in a pure form (47.1 mg, 22% yield) and **2** in a pure form (128.7 mg, 78% yield) .

4: pale-yellow powder, m.p. 187-189 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.84 (s, 3H), 6.89 (d, *J* = 9.2 Hz, 2H), 7.47-7.53 (m, 8H), 7.56-7.59 (m, 4H), 7.88-7.93 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.25 (d, *J* = 4.9 Hz), 84.25 (d, *J* = 167.9 Hz), 87.36, 92.94, 104.94 (d, *J* = 30.1 Hz), 114.04, 114.54, 118.88 (d, *J* = 4.7 Hz), 126.15, 128.64 (d, *J* = 13.4 Hz), 130.90 (d, *J* = 10.8 Hz), 131.37, 132.26 (d, *J* = 2.8 Hz), 132.36 (d, *J* = 1.9 Hz), 132.83 (d, *J* = 121.9 Hz), 133.16, 159.96; ^{31}P NMR (121 MHz, $CDCl_3$): δ 9.78; HRMS (FAB) calcd for $C_{29}H_{22}O_2P$ ($M+H^+$): 433.1357, found 433.1355.

5:[6] white powder, ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.83 (s, 3H), 6.88 (d, *J* = 8.8 Hz,

2H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 2H).

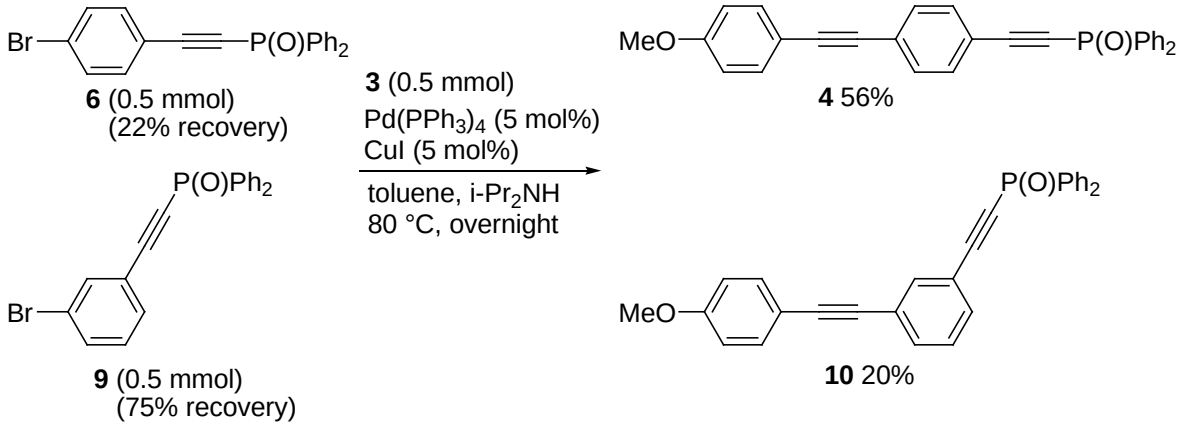
Competitive Sonogashira coupling of 6 vs 7 with 3.



A toluene solution (5.0 mL) of **6** (190.6 mg, 0.5 mmol), **7** (128.6 mg, 0.5 mmol), **3** (66.1 mg, 0.5 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol) and diisopropylamine (0.25 mL) was stirred under nitrogen at 80 °C for overnight. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 10:1, and hexane/AcOEt, 1.5:1) to afford **4** in a pure form (164.3 mg, 76% yield) and **8**[7] in a pure form (15.4 mg, 10% yield) and recover **6** in a pure form (24.8 mg, 13% yield) and **7** in a pure form (114.4 mg, 89% yield) .

8:[7] white powder, ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.84 (s, 3H), 6.89 (d, *J* = 8.8 Hz, 2H), 7.35-7.38 (m, 3H), 7.47-7.55 (m, 8H).

Competitive Sonogashira coupling of 6 vs 9 with 3.

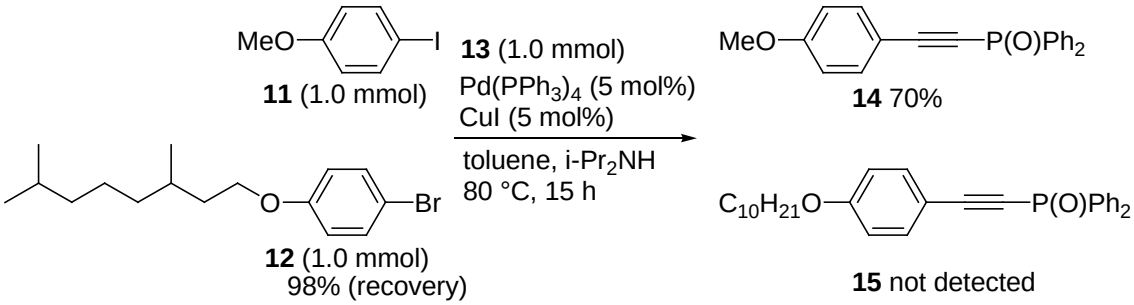


A toluene solution (5.0 mL) of **6** (190.6 mg, 0.5 mmol), **9** (190.6mg, 0.5 mmol), **3**

(66.1 mg, 0.5 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol) and diisopropylamine (0.25 mL) was stirred under nitrogen at 80 °C for overnight. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography silica gel (hexane/AcOEt, 1:1) to afford a mixture of **4**, **10** and remaining starting compounds **6**, **9**. ^{1}H NMR analysis of the mixture indicated the formations of **4** in 56% yield and **10**[3] in 20% yield and recoveries of **6** in 22% and **9** in 75% (using 1, 4-dioxane as an internal standard).

10:[3] white powder; m.p. 151-152 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.84 (s, 3H), 6.89 (d, *J* = 8.8 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.49-7.54 (m, 5H), 7.56-7.58 (m, 3H), 7.74 (s, 1H), 7.88-7.93 (m, 4H).

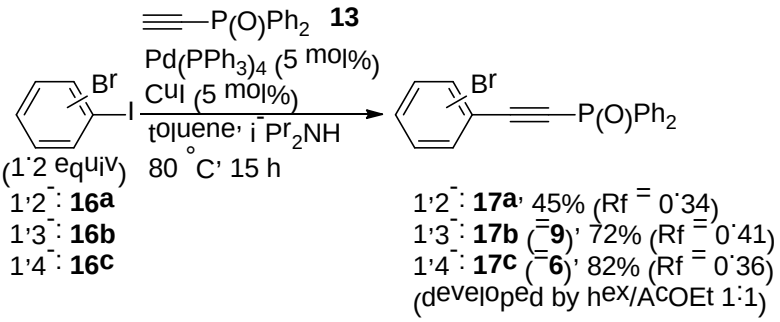
Competitive Sonogashira coupling of 11 vs 12 with 13.



A toluene solution (10.0 mL) of **11** (234.0 mg, 1.0 mmol), **12** (313.3 mg, 1.0 mmol), **13** (226.2 mg, 1.0 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 10:1, and hexane/AcOEt, 1:1) to recover **12** (307.0 mg, 98% yield) and afford **14**[2] in a pure form (232.6 mg, 70% yield).

14:[2] white powder; m.p. 125-126 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.84 (s, 3H), 6.89 (d, *J* = 8.8 Hz, 2H), 7.49-7.56 (m, 8H), 7.88-7.92 (m, 4H).

Synthesis of 17a, 17b and 17c by Sonogashira coupling (representative procedure for 17a).



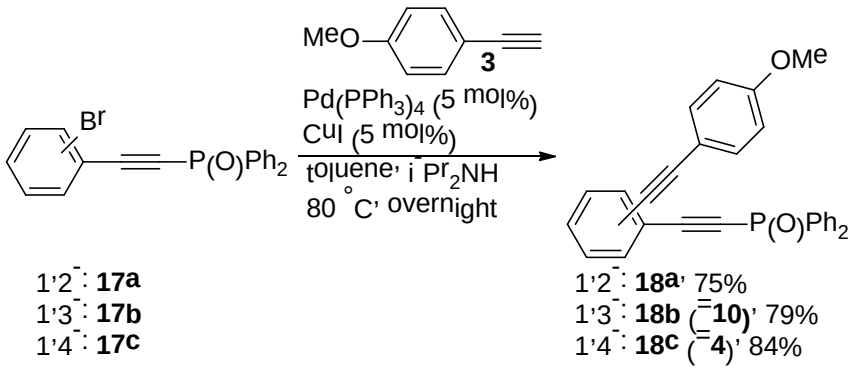
A toluene solution (5 mL) of **16a** (339.5 mg, 1.2 mmol), **13** (226.2 mg, 1.0 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:1) to afford **17a** in a pure form (171.5 mg, 45% yield).

17a: white powder, m.p. 88-89 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.29-7.35 (m, 2H), 7.49-7.52 (m, 4H), 7.55-7.58 (m, 2H), 7.61-7.64 (m, 2H), 7.94-7.98 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 86.76 (d, *J* = 166.3 Hz), 102.63 (d, *J* = 29.5 Hz), 122.05 (d, *J* = 3.8 Hz), 125.87 (d, *J* = 2.1 Hz), 127.04, 128.39 (d, *J* = 13.7 Hz), 130.70 (d, *J* = 11.2 Hz), 131.54, 132.07 (d, *J* = 3.1 Hz), 132.40, 132.46 (d, *J* = 121.9 Hz), 134.16 (d, *J* = 1.9 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ 10.08; HRMS (MALDI-TOF) calcd for $C_{20}H_{15}BrOP$ ($M+H^+$): 381.0044, found 381.0061.

17b (=**9**):[3] white powder; m.p. 98-99 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26 (t, *J* = 8.0 Hz, 1H), 7.49-7.54 (m, 5H), 7.56-7.60 (m, 3H), 7.74 (s, 1H), 7.86-7.91 (m, 4H).

17c (=**6**):[1] white powder; m.p. 154-155 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.6 Hz, 2H), 7.49-7.59 (m, 8H), 7.86-7.91 (m, 4H).

Synthesis of 18a, 18b and 18c by Sonogashira coupling (representative procedure for 19).



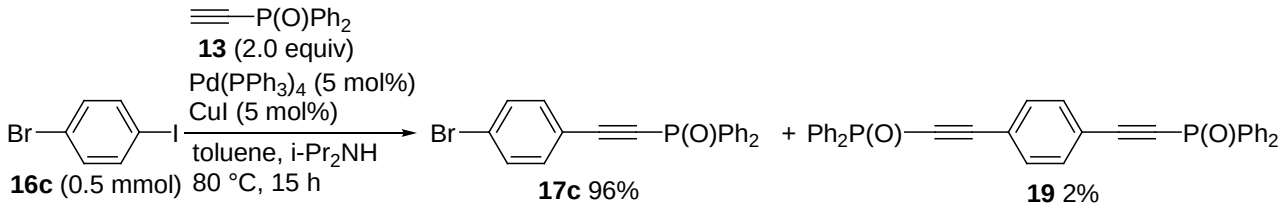
A toluene solution (5.0 mL) of **3** (158.6 mg, 1.2 mmol), **17a** (381.2 mg, 1.0 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1.5:1) to afford **18a** in a pure form (324.3 mg, 75% yield).

18a: yellow powder, m.p.140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.82 (s, 3H), 6.78 (d, *J* = 8.6 Hz, 2H), 7.27-7.34 (m, 3H), 7.35-7.44 (m, 5H), 7.47-7.50 (m, 2H), 7.56 (d, *J* = 7.9 Hz, 1H), 7.63 (d, *J* = 7.4 Hz, 1H), 7.94-7.97 (m, 4H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.18 (d, *J* = 4.6 Hz), 85.90, 85.98 (d, *J* = 169.1 Hz), 94.73, 103.82 (d, *J* = 30.1 Hz), 113.85, 114.37, 121.88 (d, *J* = 4.1 Hz), 127.21 (d, *J* = 1.9 Hz), 127.63, 128.54 (d, *J* = 13.4 Hz), 130.25, 130.84 (d, *J* = 11.2 Hz), 131.81, 132.03 (d, *J* = 2.8 Hz), 132.86 (d, *J* = 1.8 Hz), 132.91 (d, *J* = 121.6 Hz), 133.20, 159.81; ^{31}P NMR (121 MHz, $CDCl_3$): δ 9.86; HRMS (MALDI-TOF) calcd for $C_{29}H_{22}O_2P$ ($M+H^+$): 433.1357, found 433.1362.

18b (=**10**):[3] white powder; m.p. 151-152 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.84 (s, 3H), 6.89 (d, *J* = 8.8 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.49-7.54 (m, 5H), 7.56-7.58 (m, 3H), 7.74 (s, 1H), 7.88-7.93 (m, 4H).

18c (=**4**): as described in above

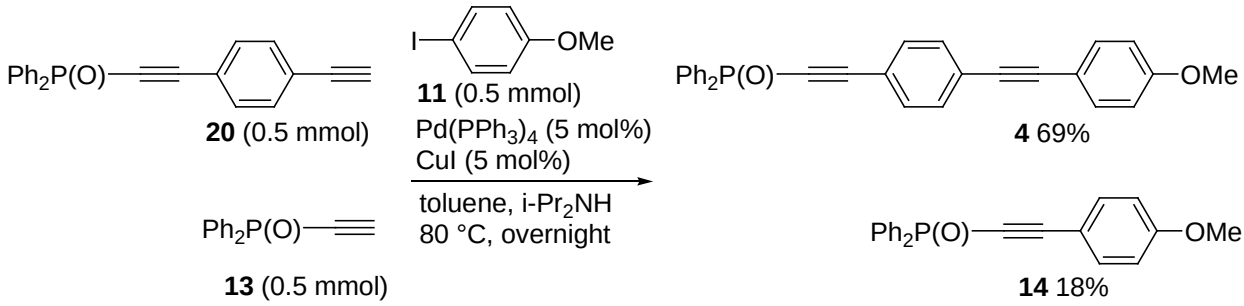
Sonogashira coupling of 16c with an excess amount of 13.



A toluene solution (5 mL) of **16c** (141.4 mg, 0.5 mmol), **13** (226.2 mg, 1.0 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol) and diisopropylamine (0.25 mL) was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (AcOEt) to afford **17c** in a pure form (183.0 mg, 96% yield) and **19** (0.01 mmol, 2% NMR yield (using 1, 4-dioxane as an internal standard)).

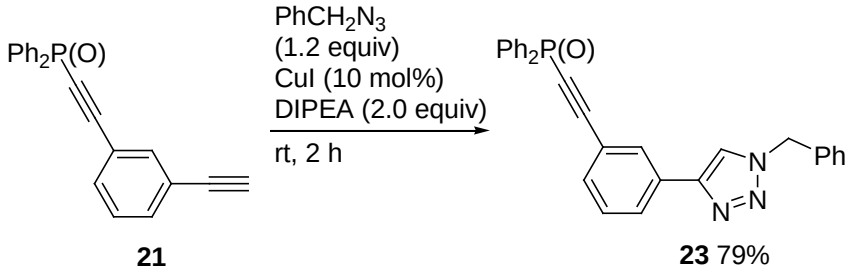
17c: as described in above

Competitive Sonogashira coupling of 20 vs 13 with 11.

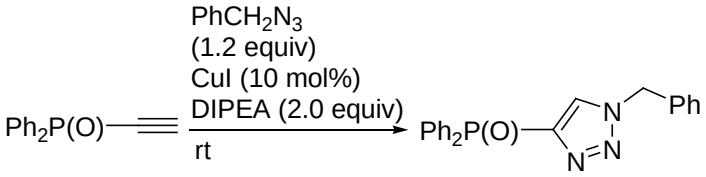


A toluene solution (5.0 mL) of **20** (163.2 mg, 0.5 mmol), **13** (113.1 mg, 0.5 mmol), **11** (117.0 mg, 0.5 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol) and diisopropylamine (0.25 mL) was stirred under nitrogen at 80 °C for overnight. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1.5:1) to afford **4** in a pure form (149.2 mg, 69% yield) and **14** in a pure form (29.9 mg, 18% yield).

Synthesis of 23[2] and 24 by click reaction (representative procedure for 23).



21 **23** 79%



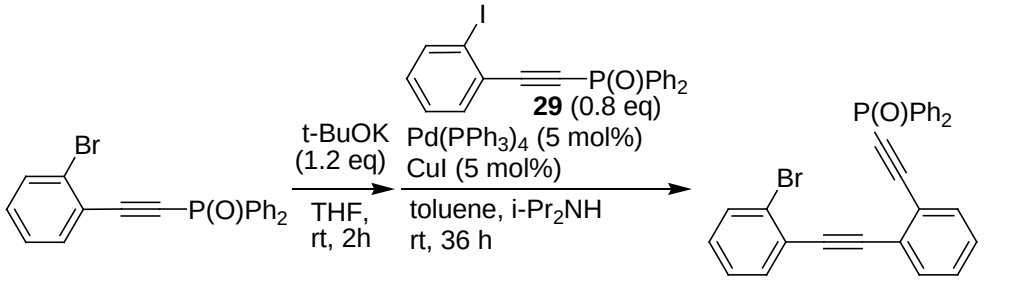
13 **24** 73% (6 h)
42% (2 h)

An acetone solution (10 mL) of **21** (326.3 mg, 1.0 mmol), benzyl azide (159.8 mg, 1.2 mmol), CuI (19.0 mg, 0.1 mmol) and DIPEA (340.1 μL, 2.0 mmol) was stirred under nitrogen at rt for 2 h. After workup with CH_2Cl_2 and water, the organic layer was dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:2) to give **23** in a pure form (363.0 mg, 79% yield).

23:[2] white powder; m.p. 114-115 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.59 (s, 2H), 7.32-7.33 (m, 2H), 7.38-7.44 (m, 4H), 7.48-7.58 (m, 7H), 7.68 (s, 1H), 7.88-7.92 (m, 5H), 8.02 (s, 1H).

24: white powder; m.p. 157-158˚C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.57 (s, 2H), 7.29-7.32 (m, 2H), 7.36-7.39 (m, 3H), 7.43-7.48 (m, 4H), 7.49-7.55 (m, 2H), 7.85-7.91 (m, 4H), 8.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 54.41, 128.53 (d, J = 13.4 Hz), 129.27 (d, J = 13.4 Hz), 130.77 (d, J = 23.9 Hz), 131.49 (d, J = 10.5 Hz), 131.85, 132.14 (d, J = 2.8 Hz), 132.95, 133.44, 141.52, 142.88; MRMS (MALDI-TOF) calcd for $C_{21}H_{19}N_3OP$ ($M+H^+$): 360.1266, found 360.1262.

Synthesis of 28.

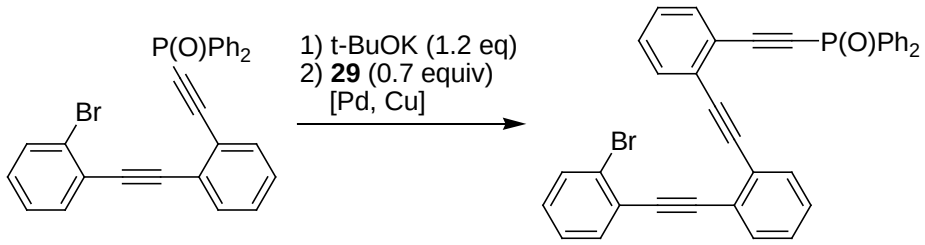


17a **28** 62% (based on iodide)

To a THF solution (10 mL) of **17a** (381.2 mg, 1.0 mmol) was added t-BuOK (134.6 mg, 1.2 mmol). After the mixture was stirred for 2 h under nitrogen at rt, **29** (342.6 mg, 0.8 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol), toluene (16 mL) and diisopropylamine (0.5 mL) was added sequentially. The mixture was stirred under nitrogen at rt for 36 h. After workup with CH_2Cl_2 and NH_4Claq, the organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 2:1) to give **28** in a pure form (238.7 mg, 62% yield, based on iodide).

28: white powder; m.p. 126-129 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.17-7.20 (m, 2H), 7.28-7.30 (m, 1H), 7.33-7.38 (m, 5H), 7.44-7.48 (m, 3H), 7.58-7.60 (m, 1H), 7.65 (d, *J* = 3.4 Hz, 1H), 7.66 (d, *J* = 4.2 Hz, 1H), 7.90-7.95 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 86.33 (d, *J* = 168.6 Hz), 91.19, 92.66, 103.37 (d, *J* = 30.4 Hz), 122.15 (d, *J* = 4.1 Hz), 124.55, 125.50, 126.39 (d, *J* = 1.8 Hz), 126.94, 128.39, 128.44 (d, *J* = 13.7 Hz), 129.78, 130.28, 130.85 (d, *J* = 11.2 Hz), 132.04 (d, *J* = 2.8 Hz), 132.28, 132.32, 132.79 (d, *J* = 121.9 Hz), 133.00 (d, *J* = 2.2 Hz), 133.55; ^{31}P NMR (121 MHz, $CDCl_3$): δ 9.91; HRMS (MALDI-TOF) calcd for $C_{28}H_{19}BrOP$ ($M+H^+$): 481.0357, found 481.0396.

Synthesis of 30.

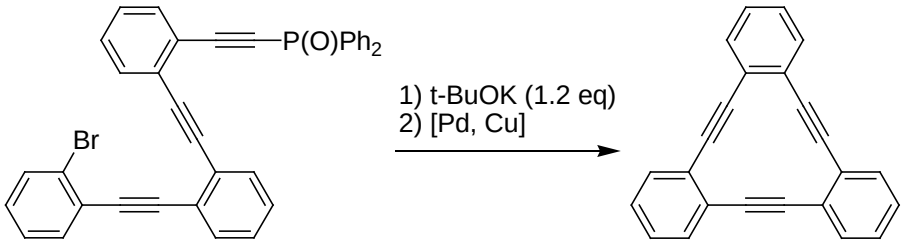


28 **30** 56% (based on iodide)

To a THF solution (10 mL) of **28** (240.6 mg, 0.5 mmol) was added t-BuOK (67.3 mg, 0.6 mmol). After the mixture was stirred for 2 h under nitrogen at rt, **29** (149.9 mg, 0.35 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol), toluene (16 mL) and diisopropylamine (0.25 mL) was added sequentially. The mixture was stirred under nitrogen at rt for 48 h. After workup with CH_2Cl_2 and NH_4Claq, the organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1.5:1) and recrystallization from THF/hexane to give **30** in a pure form (114.0 mg, 56% yield, based on iodide).

30: white powder; m.p. 129-131 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.16-7.31 (m, 8H), 7.34-7.38 (m, 2H), 7.40-7.44 (m, 3H), 7.54 (dd, *J* = 1.6 Hz, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.62-7.66 (m, 3H), 7.88-7.92 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 86.46 (d, *J* = 168.5 Hz), 91.14, 92.11, 92.36, 93.03, 103.53 (d, *J* = 30.0 Hz), 122.48 (d, *J* = 4.1 Hz), 124.98, 125.28, 125.44, 125.51, 126.39 (d, *J* = 2.0 Hz), 127.03, 127.06, 128.33, 128.40-128.48 (m), 128.53 (d, *J* = 11.3 Hz), 128.56, 129.62 (d, *J* = 3.5 Hz), 130.30, 130.94 (d, *J* = 11.3 Hz), 132.06, 132.24, 132.47, 132.92 (d, *J* = 122.0 Hz), 133.10, 133.63; ^{31}P NMR (121 MHz, $CDCl_3$): δ 6.89; HRMS (MALDI-TOF) calcd for $C_{36}H_{23}BrOP$ ($M+H^+$): 581.0670, found 581.0641.

Synthesis of 25[8].

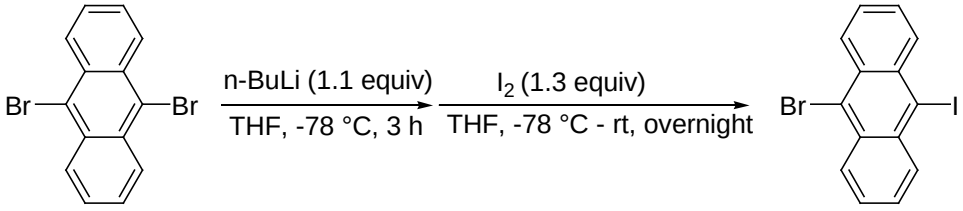


30 **25** 68%

To a THF solution (5 mL) of **30** (58.1 mg, 0.1 mmol) was added t-BuOK (13.5 mg, 0.12 mmol). After the mixture was stirred for 2 h under nitrogen at rt, the resulted reaction mixture was added to a toluene solution (8 mL) of $Pd(PPh_3)_4$ (5.8 mg, 0.005 mmol), CuI (1 mg, 0.005 mmol) and diisopropylamine (0.05 mL) via syringe over 6 minutes. The mixture was stirred under nitrogen at 80 °C for 15 h. After workup with CH_2Cl_2 and NH_4Claq, the organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 8:1) to give **25** in a pure form (20.4 mg, 68% yield).

25:[8] pale-green powder; m.p. 209-210 ^oC; ^1H NMR (500 MHz, $CDCl_3$): δ 7.17-7.20 (m, 6H), 7.33-7.37 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 92.79, 126.61, 128.52, 131.98.

Synthesis of starting compound 31[9].



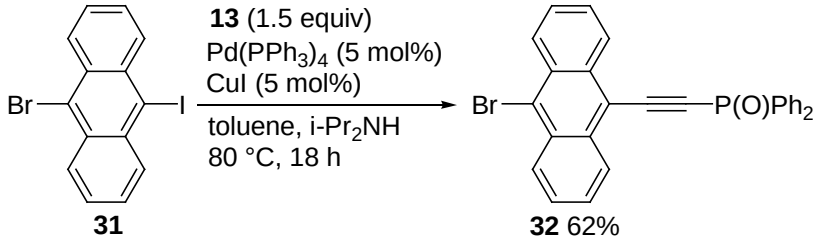
31 79%

A solution of BuLi (1.6 M in hexane, 2.1 mL, 3.3 mmol) was added dropwise to a stirring suspension of 9,10-dibromoanthrancene (1008.1 mg, 3.0 mmol) in 40 mL of THF at -78 °C, and the resulting mixture was stirred for 3 h at -78 °C. To the resulting clear red solution, a solution of iodine (989.8 mg, 3.9 mmol) in 10.0 mL of THF was added dropwise, and the resulting mixture was stirred for 1 h at -78 °C, and then allowed to warm to room temperature for overnight. The resulting mixture was

concentrated in vacuo to approximately 10% of the initial volume, and 20% aqueous solution of $Na_2S_2O_3$ was added. A yellow crystalline precipitate was filtered, washed successively with dilute $Na_2S_2O_3$ solution, water, and cold ethanol, and dried in vacuo. Recrystallization from toluene afforded **31** in a pure form (907.8 mg, 79% yield).

31:[9] yellow fiber-like crystal, ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.59-7.63 (m, 4H), 8.52-8.56 (m, 4H).

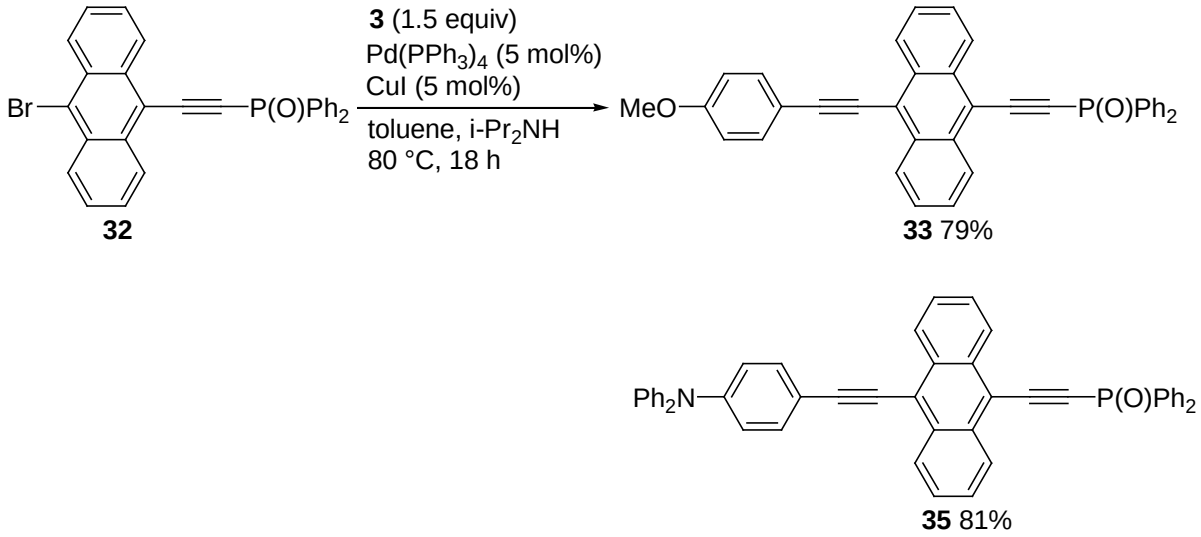
Synthesis of 32.



A toluene solution (20.0 mL) of **31** (383.0 mg, 1.0 mmol), **13** (339.3 mg, 1.5 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 18 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:1) to afford **32** in a pure form (298.4 mg, 62% yield).

32: white powder, m.p. 195-196 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.54-7.58 (m, 4H), 7.60-7.67 (m, 6H), 8.03-8.07 (m, 4H), 8.47 (d, *J* = 8.6 Hz, 2H), 8.59 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 94.87 (d, *J* = 165.8 Hz), 102.44 (d, *J* = 29.5 Hz), 113.90 (d, *J* = 4.1 Hz), 126.42, 127.65, 127.83, 128.05, 128.50, 128.79 (d, *J* = 12.9 Hz), 129.99, 131.06 (d, *J* = 11.4 Hz), 132.41, 133.05 (d, *J* = 121.9 Hz), 133.94 (d, *J* = 2.1 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ 6.33; HRMS (MALDI-TOF) calcd for $C_{28}H_{19}BrOP$ ($M+H^+$): 481.0357, found 481.0323.

Synthesis of 33 and 35 (representative procedure for 33).



32 **33** 79% **35** 81%

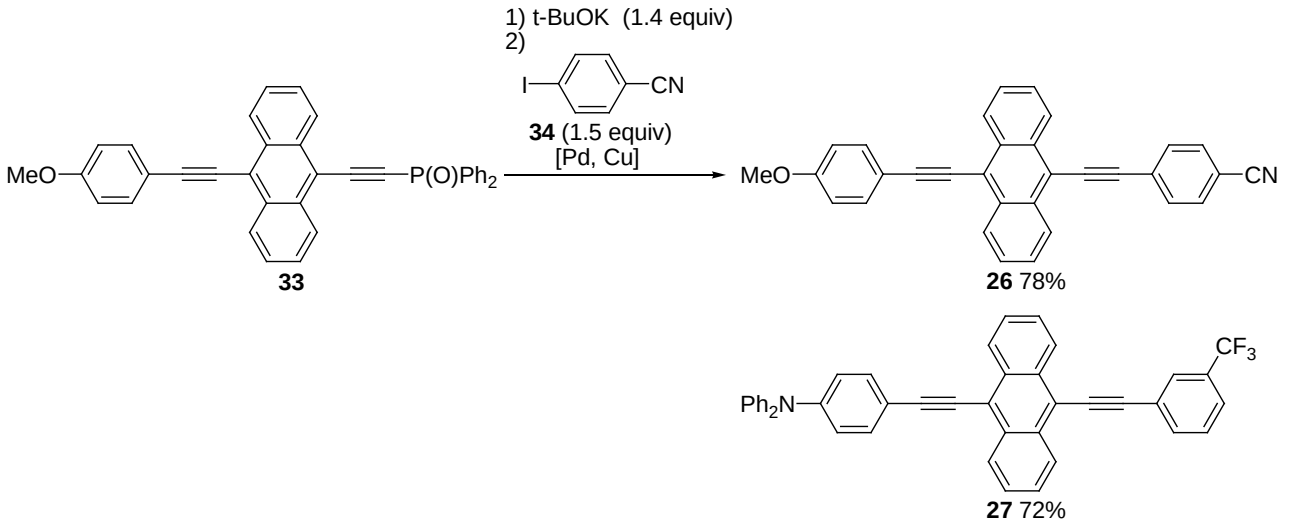
A toluene solution (20.0 mL) of **3** (198.2 mg, 1.5 mmol), **32** (481.3 mg, 1.0 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), CuI (9.5 mg, 0.05 mmol) and diisopropylamine (0.5 mL) was stirred under nitrogen at 80 °C for 18 h. After workup with CH_2Cl_2 and NH_4Claq, the combined organic layer was washed with brine and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/AcOEt, 1:1) to afford **33** in a pure form (420.7 mg, 79% yield).

33: white powder, m.p. 180-181 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.89 (s, 3H), 7.00 (d, *J* = 8.8 Hz, 2H), 7.54-7.58 (m, 4H), 7.60-7.65 (m, 6H), 7.72 (d, *J* = 8.6 Hz, 2H), 8.04-8.08 (m, 4H), 8.45-8.47 (m, 2H), 8.69-8.70 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.36 (d, *J* = 4.6 Hz), 84.94, 94.89 (d, *J* = 167.5 Hz), 103.14 (d, *J* = 29.4 Hz), 104.35, 113.22 (d, *J* = 4.6 Hz), 114.22 (d, *J* = 8.3 Hz), 114.94, 122.28, 126.40, 126.78, 127.53 (d, *J* = 3.6 Hz), 127.94, 128.76 (d, *J* = 13.4 Hz), 131.07 (d, *J* = 11.3 Hz), 131.40, 132.35, 133.21 (d, *J* = 122.0 Hz), 133.24 (d, *J* = 1.5 Hz), 133.32, 160.26; ^{31}P NMR (121 MHz, $CDCl_3$): δ 7.31; HRMS (MALDI-TOF) calcd for $C_{37}H_{26}O_2P$ ($M+H^+$): 533.1670, found 533.1642.

35: white powder, m.p. 249-150 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.11 (t, *J* = 7.3 Hz, 4H), 7.17 (d, *J* = 7.6 Hz, 4H), 7.32 (t, *J* = 7.8 Hz, 4H), 7.54-7.58 (m, 4H), 7.60-7.64 (m, 8H), 8.04-8.08 (m, 4H), 8.45-8.47 (m, 2H), 8.68-8.70 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 85.51, 94.92 (d, *J* = 167.5 Hz), 103.20 (d, *J* = 29.5 Hz),

104.84, 113.19, 115.27, 121.94 (d, J = 8.2 Hz), 122.38, 123.87 (m), 125.22 (m), 126.46, 126.82, 127.59 (d, J = 5.2 Hz), 127.98, 128.78 (d, J = 13.3 Hz), 129.48 (m), 131.10 (d, J = 11.3 Hz), 131.46, 132.78 (d, J = 3.0 Hz), 132.84 (d, J = 121.0 Hz), 133.31, 133.72, 146.93, 148.70; ^{31}P NMR (121 MHz, $CDCl_3$): δ 7.26; HRMS (MALDI-TOF) calcd for $C_{48}H_{32}NOP$ (M^+): 669.2222, found 669.2263.

Synthesis of 26 and 27:



To a THF solution (15.0 mL) of **33** (266.3 mg, 0.5 mmol) was added t-BuOK (78.5 mg, 0.7 mmol). After the mixture was stirred for 3.5 h under nitrogen at rt, 4-iodobenzonitrile **34** (171.8 mg, 0.75 mmol), $Pd(PPh_3)_4$ (28.9 mg, 0.025 mmol), CuI (4.8 mg, 0.025 mmol), toluene (20.0 mL) and diisopropylamine (0.25 mL) was added sequentially. The mixture was stirred under nitrogen at 80 °C overnight. After workup with CH_2Cl_2 and NH_4Claq, the organic layer was washed with brine, and dried over $MgSO_4$. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography on silica gel (hexane/CH_2Cl_2, 4:1) to give **26** in a pure form (169.1 mg, 78% yield).

26: white powder, m.p. 234-235 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.89 (s, 3H), 6.98 (d, J = 8.8 Hz, 2H), 7.60-7.66 (m, 4H), 7.69-7.72 (m, 4H), 7.80 (d, J = 8.2 Hz, 2H), 8.56-8.60 (m, 2H), 8.65-8.70 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 55.40 (d, J = 5.3 Hz), 85.17, 91.02, 100.17, 103.40, 111.55, 114.23, 115.28, 116.35, 118.58,

120.28, 126.71, 127.18, 127.48, 128.26, 131.75, 131.98, 132.18, 132.28, 133.24, 160.13; HRMS (MALDI-TOF) calcd for $C_{32}H_{19}NO$ (M^+): 433.1467, found 433.1422.

27: white powder, m.p. 190-191 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.09-7.12 (m, 4H), 7.18 (d, *J* = 7.6 Hz, 4H), 7.32 (t, *J* = 7.9 Hz, 4H), 7.58-7.69 (m, 8H), 7.95 (d, *J* = 7.6 Hz, 1H), 8.02 (s, 1H), 8.65-8.67 (m, 2H), 8.69-8.71 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 85.77, 88.09, 100.28, 103.42, 115.84, 116.82, 119.71, 122.15, 122.20, 123.75, 123.76 (q, *J* = 272.3 Hz), 124.38, 125.11 (q, *J* = 2.1 Hz), 126.66, 126.92, 127.02, 127.41, 128.26 (q, *J* = 3.6 Hz), 129.07, 129.46 (m), 131.12 (q, *J* = 32.5 Hz), 131.79, 132.20, 132.67, 134.69 (d, *J* = 1.5 Hz), 147.04, 148.39; HRMS (MALDI-TOF) calcd for $C_{43}H_{26}F_3N$ (M^+): 613.2017, found 613.2003.

III. References

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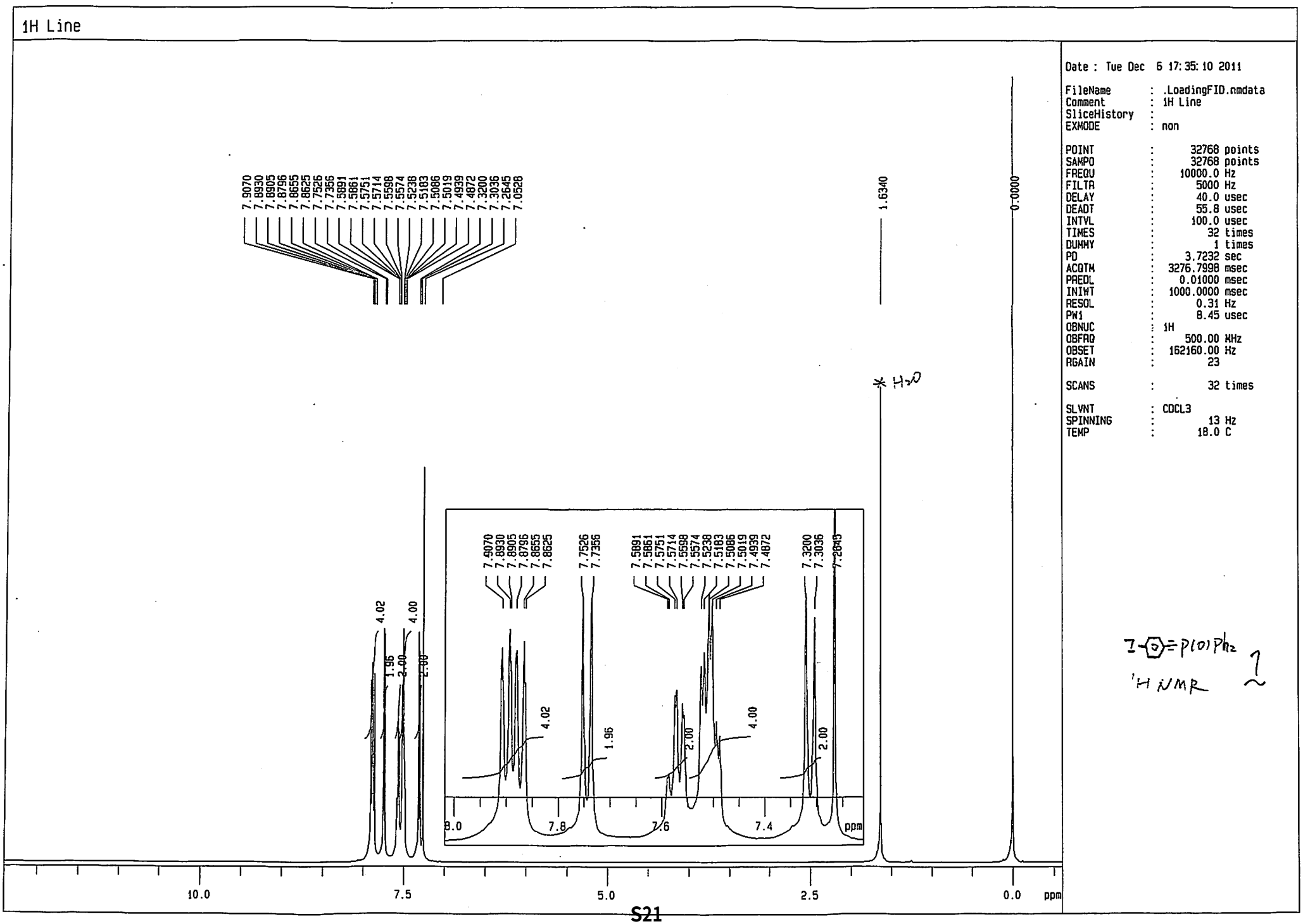
IV. ^{1}H NMR and ^{13}C NMR Charts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7.8655
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7.5086
7.5019
7.4939
7.4872
7.3200
7.3036
7.2645
7.0528
1.6340
0.0000
* H2O
4.02
1.96
2.00
4.00
2.00
8.0
7.8
7.6
7.4
ppm
10.0
7.5
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2.5
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ppm
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DUMMY : 1 times
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INIWT : 1000.0000 msec
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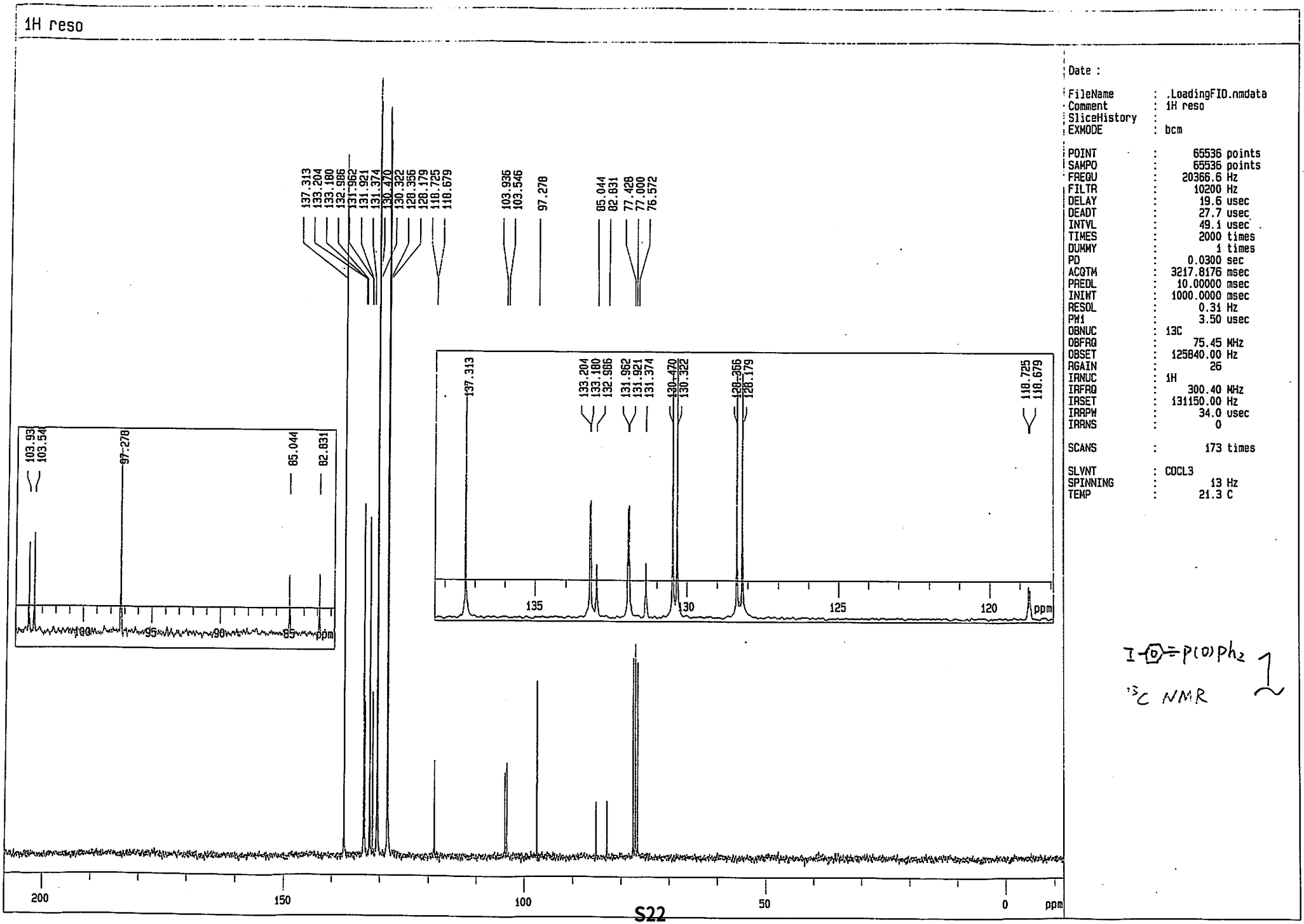
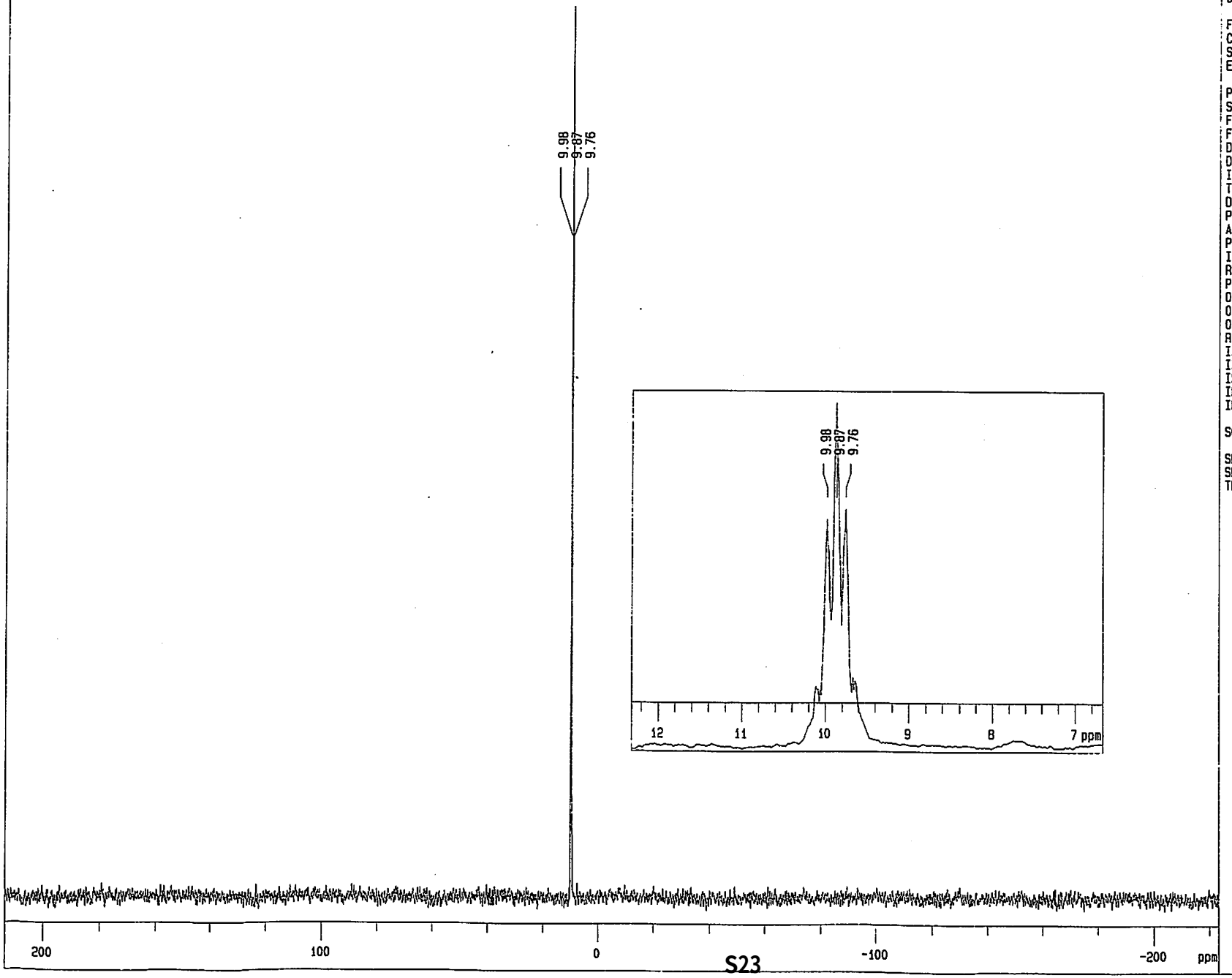
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 23
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 18.0 C

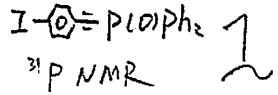

1H reso
137.313
133.204
133.180
132.986
131.962
131.921
131.374
130.470
130.322
128.366
128.179
118.725
118.679
103.936
103.546
97.278
85.044
82.831
77.428
77.000
76.572
Date :
FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
ININT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRRPW : 34.0 usec
IRRNS : 0
SCANS : 173 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.3 C
200
150
100
50
0
ppm
135
130
125
120
ppm
100
95
90
85
ppm
I-C6H4-P(O)Ph2
13C NMR
1

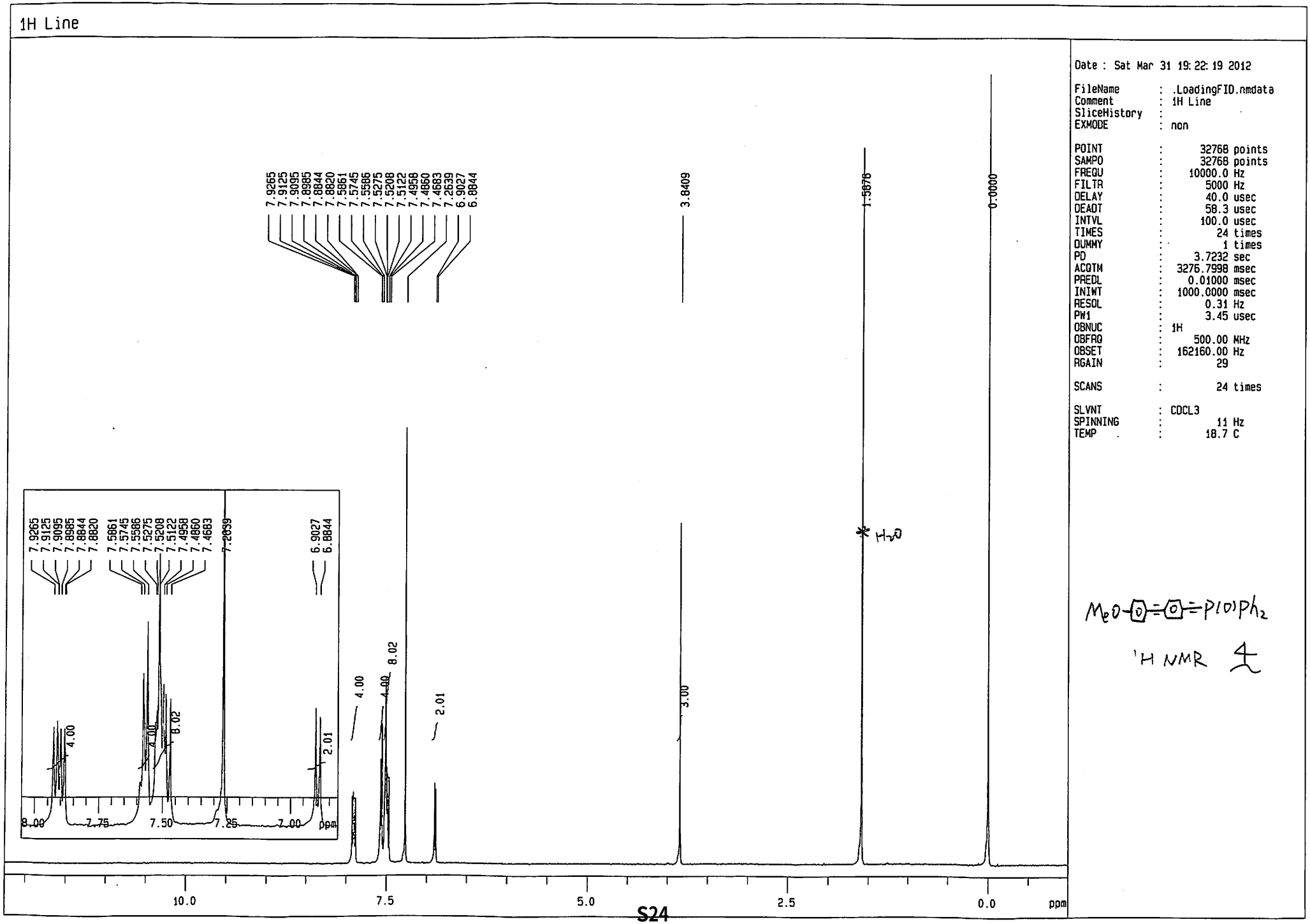
1H reso

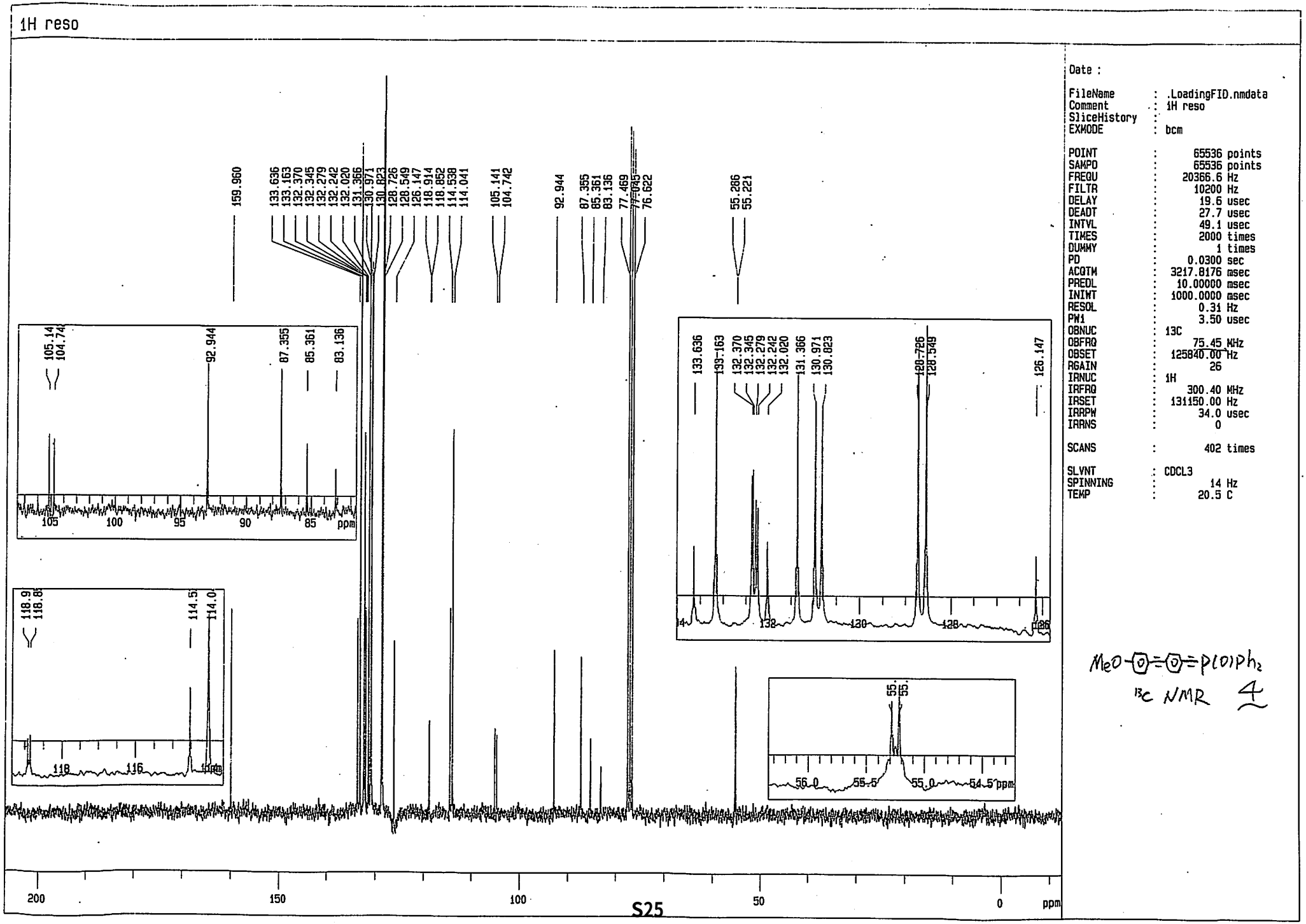


Date : Wed Aug 27 04:05:37 2003

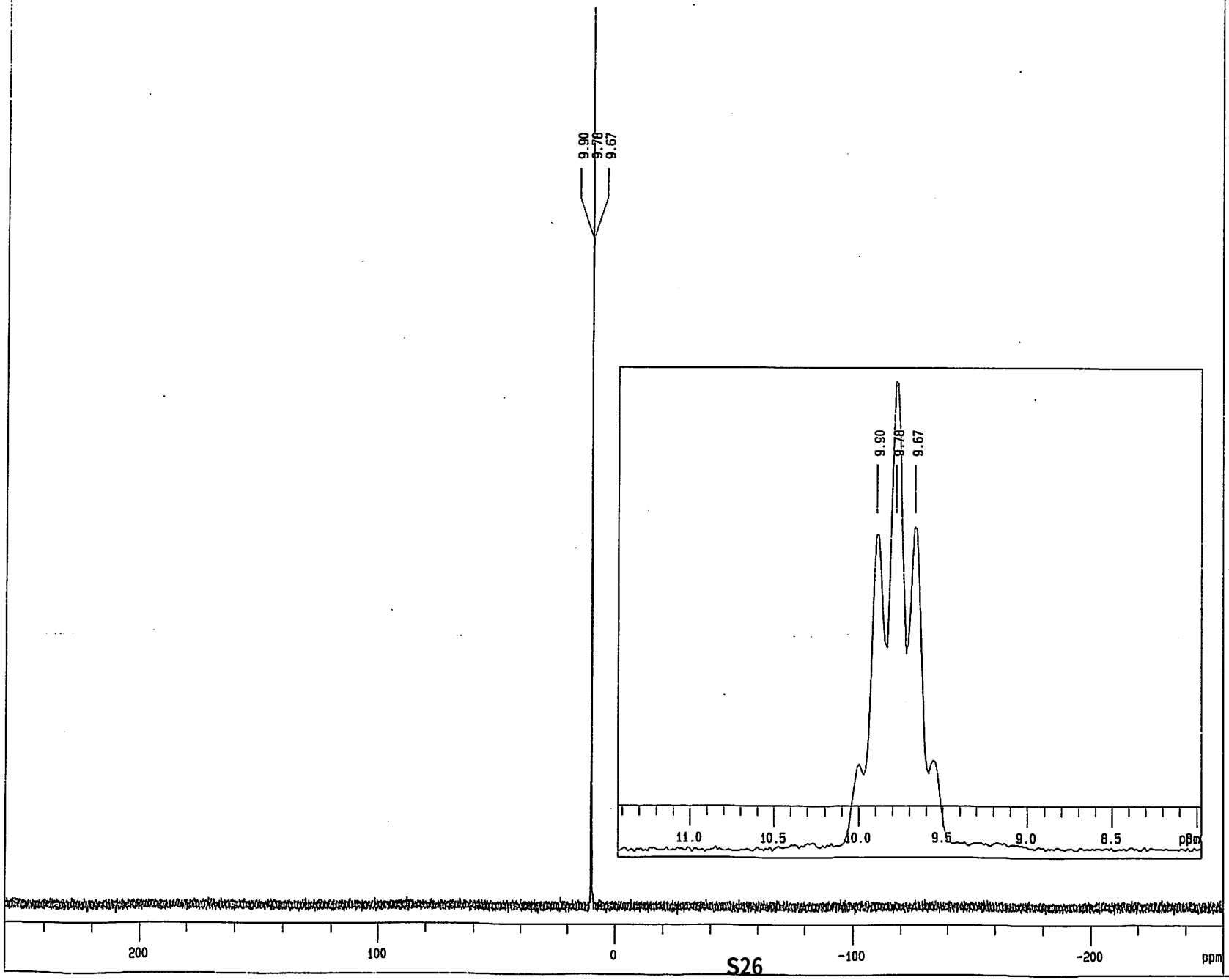
FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPD	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	80 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
ININT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	80 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	18.0 C




1H Line
7.9265
7.9125
7.9095
7.8985
7.8844
7.8820
7.5861
7.5745
7.5586
7.5275
7.5208
7.5122
7.4958
7.4660
7.4683
7.2639
6.9027
6.8844
3.8409
1.5676
0.0000
* H2O
4.00
4.00
8.02
2.01
3.00
10.0
7.5
5.0
2.5
0.0
ppm
8.00
7.75
7.50
7.25
7.00
Date : Sat Mar 31 19:22:19 2012
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 58.3 usec
INTVL : 100.0 usec
TIMES : 24 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 29
SCANS : 24 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 18.7 C
MeO
P(O)Ph2
1H NMR


1H reso
159.960
133.636
133.163
132.370
132.345
132.279
132.242
132.020
131.366
130.971
130.823
128.726
128.549
126.147
118.914
118.852
114.538
114.041
105.141
104.742
92.944
87.355
85.361
83.136
77.469
77.045
76.622
55.286
55.221
Date :
FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPD : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
ININT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRRPW : 34.0 usec
IRRNS : 0
SCANS : 402 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 20.5 C
MeO
P(O)Ph2
13C NMR
4
200
150
100
50
0
ppm

1H reso

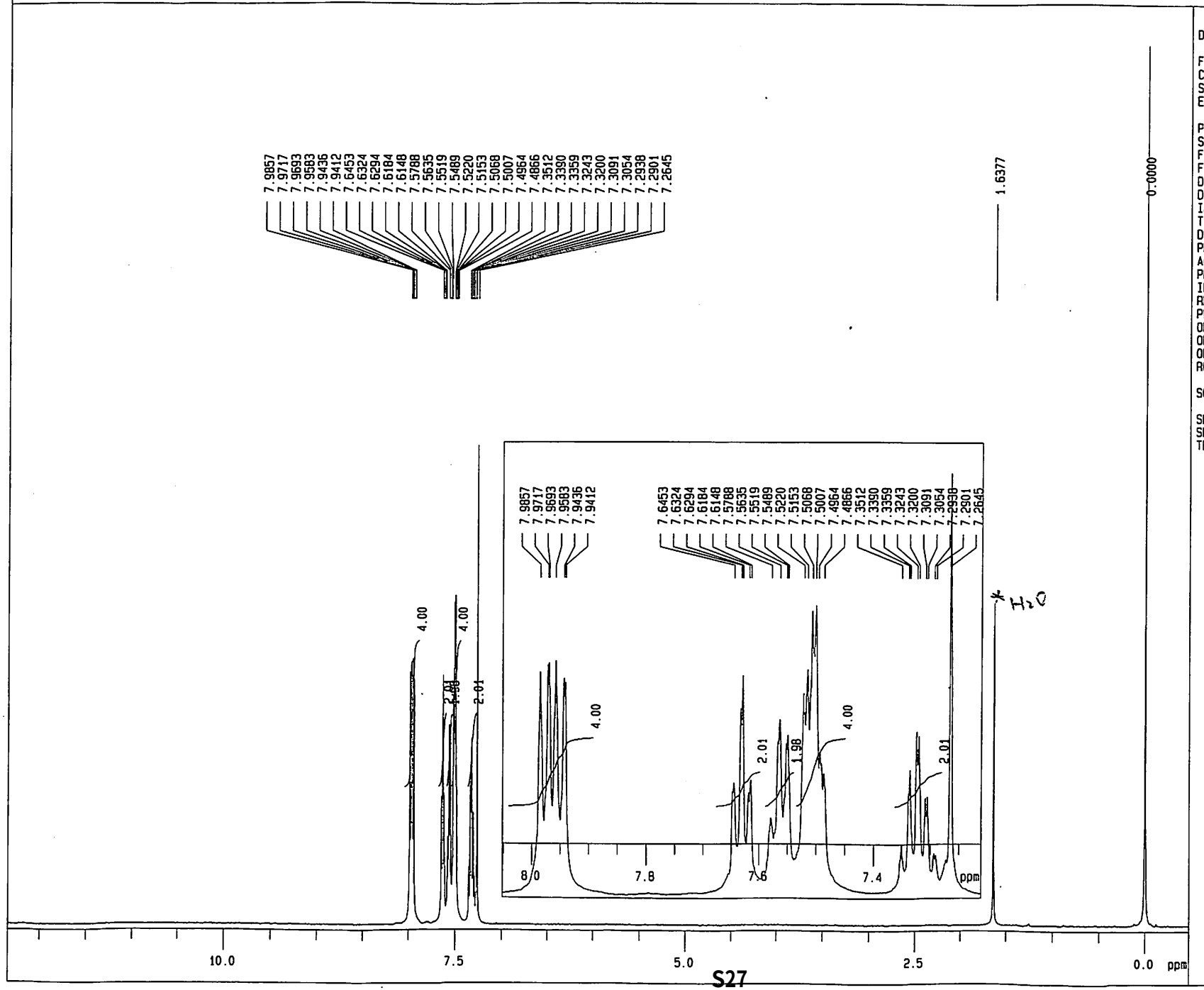


Date : Thu Aug 21 02:42:49 2003

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	400 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIWT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRAPW	:	1.0 usec
IRRNS	:	0
SCANS	:	400 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	17.4 C

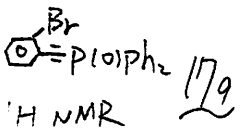
^{31}P NMR 4

1H Line

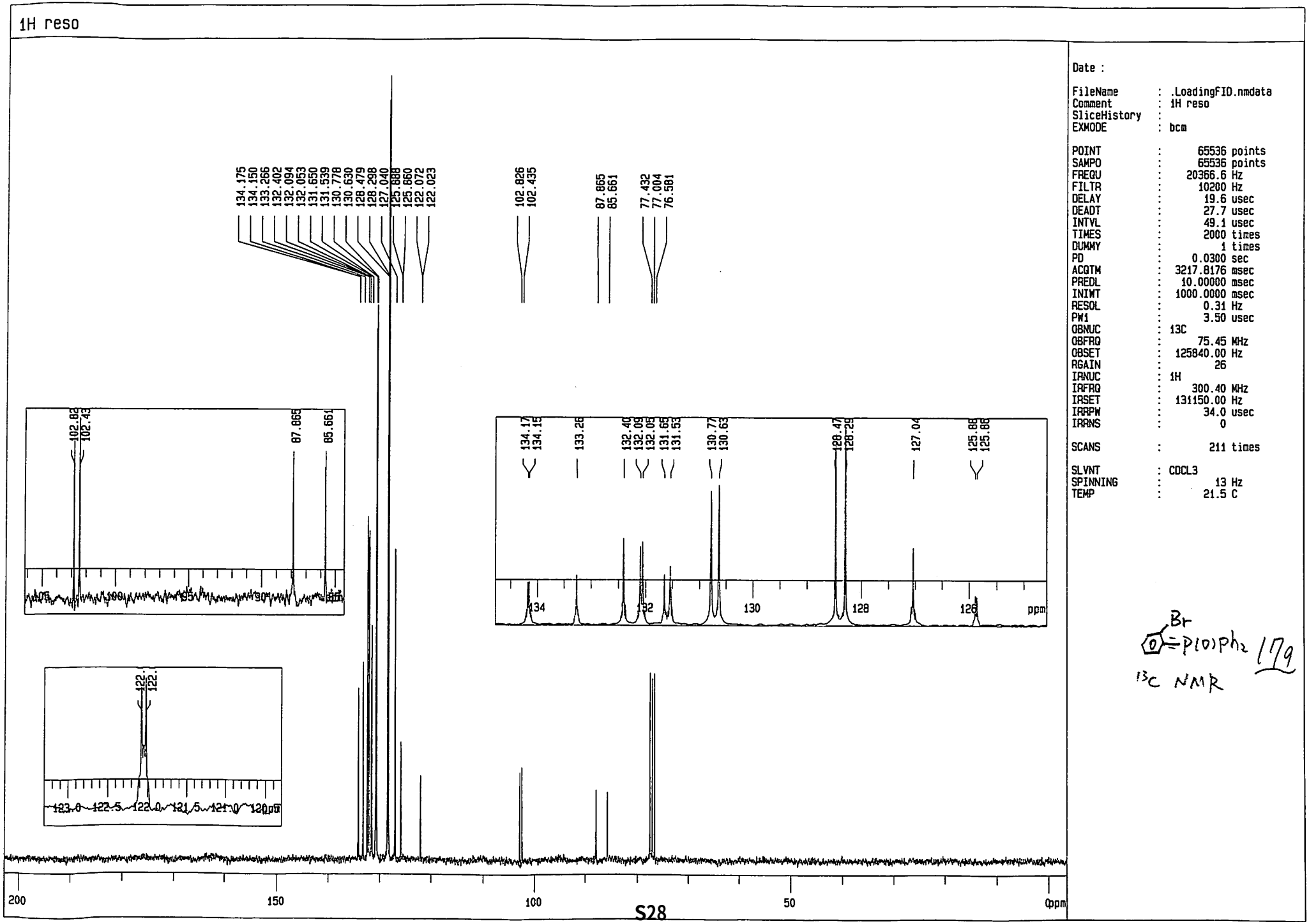


Date : Fri Dec 9 14:59:43 2011

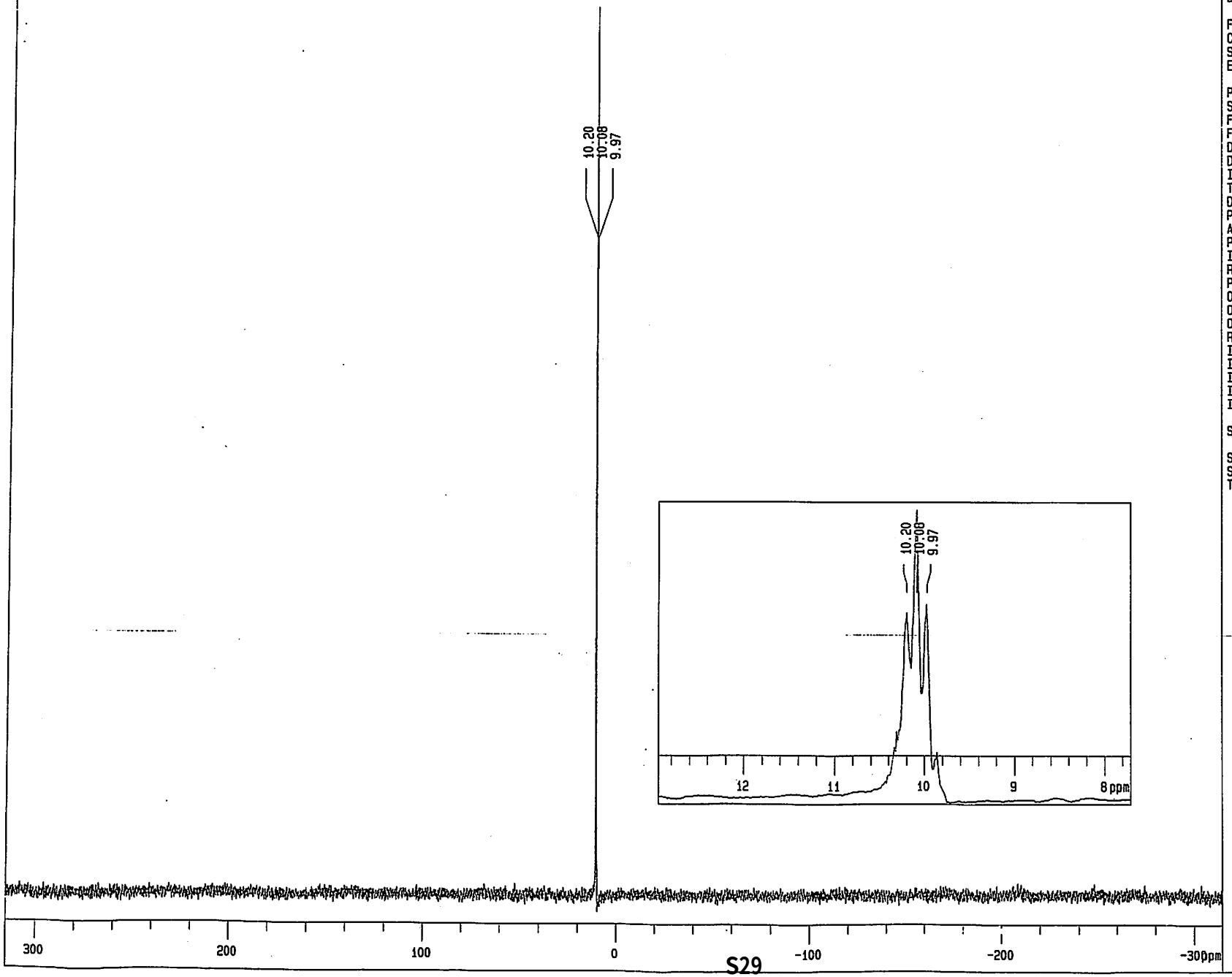
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	10000.0 Hz
FILTR	:	5000 Hz
DELAY	:	40.0 usec
DEADT	:	58.3 usec
INTVL	:	100.0 usec
TIMES	:	45 times
DUMMY	:	1 times
PD	:	3.7232 sec
ACQTM	:	3276.7998 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.31 Hz
PW1	:	3.45 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162160.00 Hz
RGAIN	:	26
SCANS	:	45 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	16.7 C



^{1}H NMR 17g

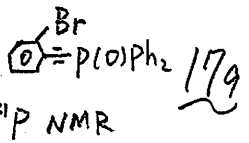

1H reso
134.175
134.150
133.266
132.402
132.094
132.053
131.650
131.539
130.778
130.630
128.479
128.298
127.040
125.888
125.860
122.072
122.023
102.826
102.435
87.865
85.661
77.432
77.004
76.581
Date :
FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRRPW : 34.0 usec
IRRNS : 0
SCANS : 211 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.5 C
200
150
100
50
0ppm
134
132
130
128
126
ppm
123.0
122.5
122.0
121.5
121.0
120ppm
Br
P(O)Ph2
179
13C NMR

1H reso



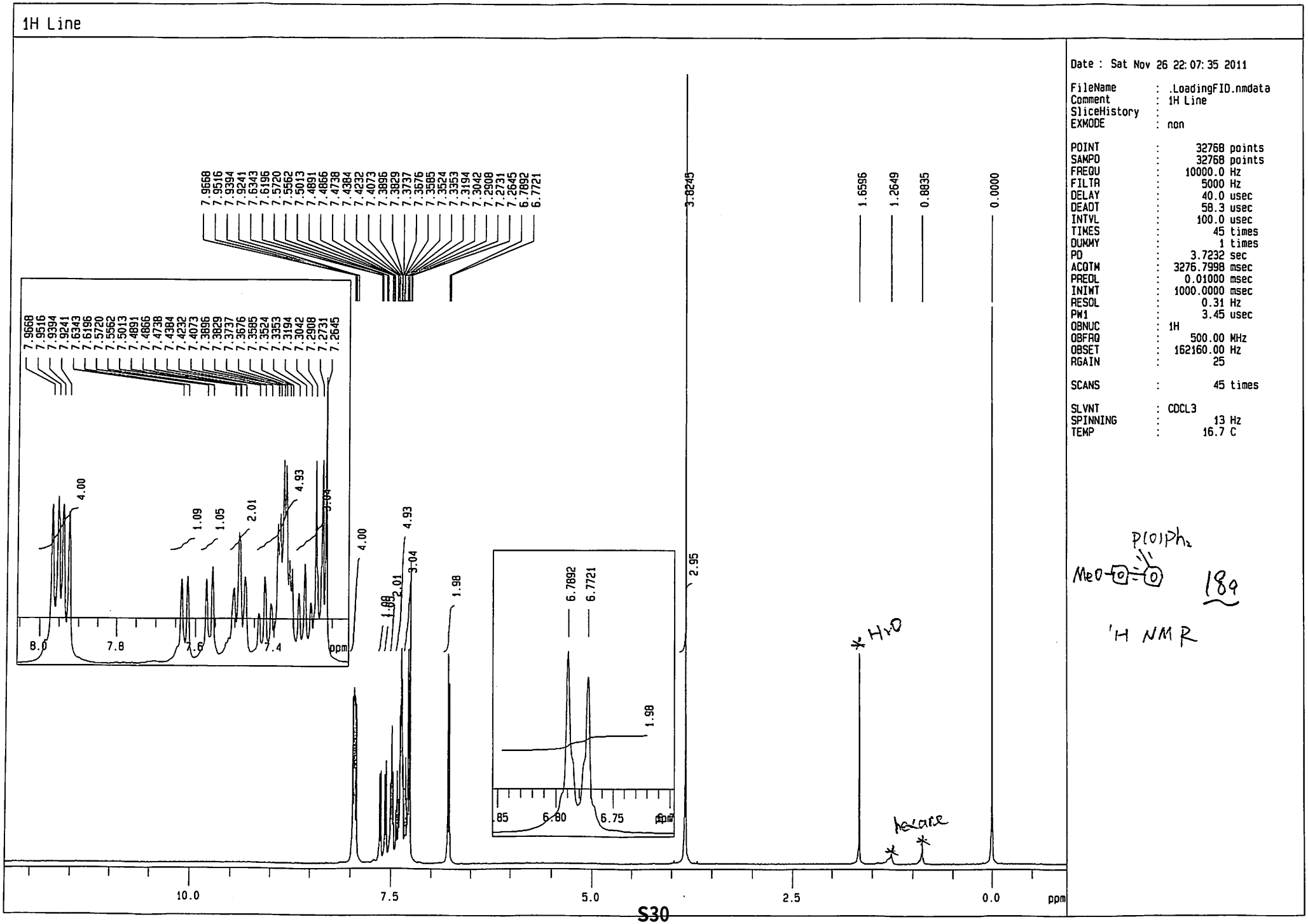
Date : Tue Sep 2 06:58:18 2003

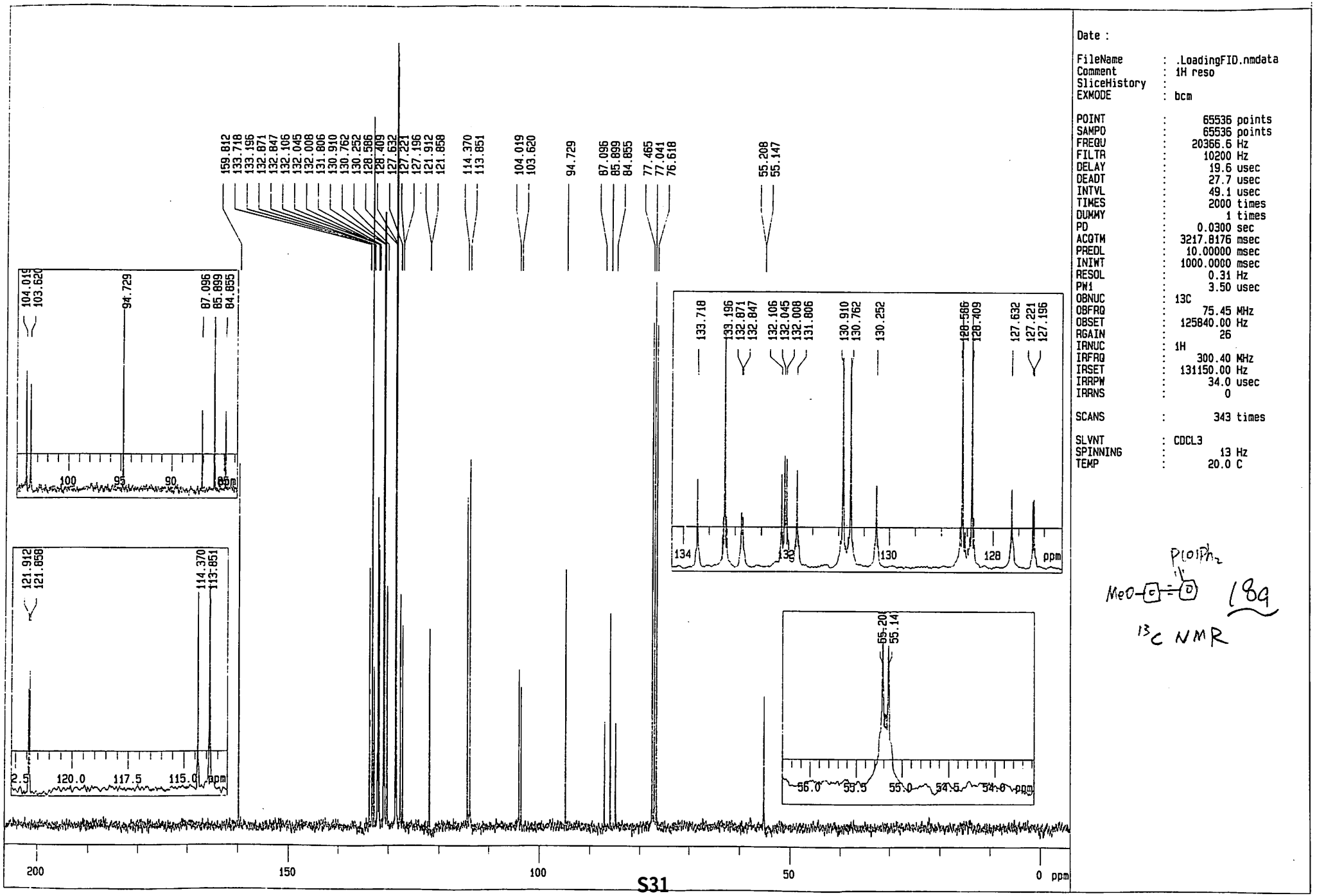
FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	60 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
ININT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	60 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	17.5 C



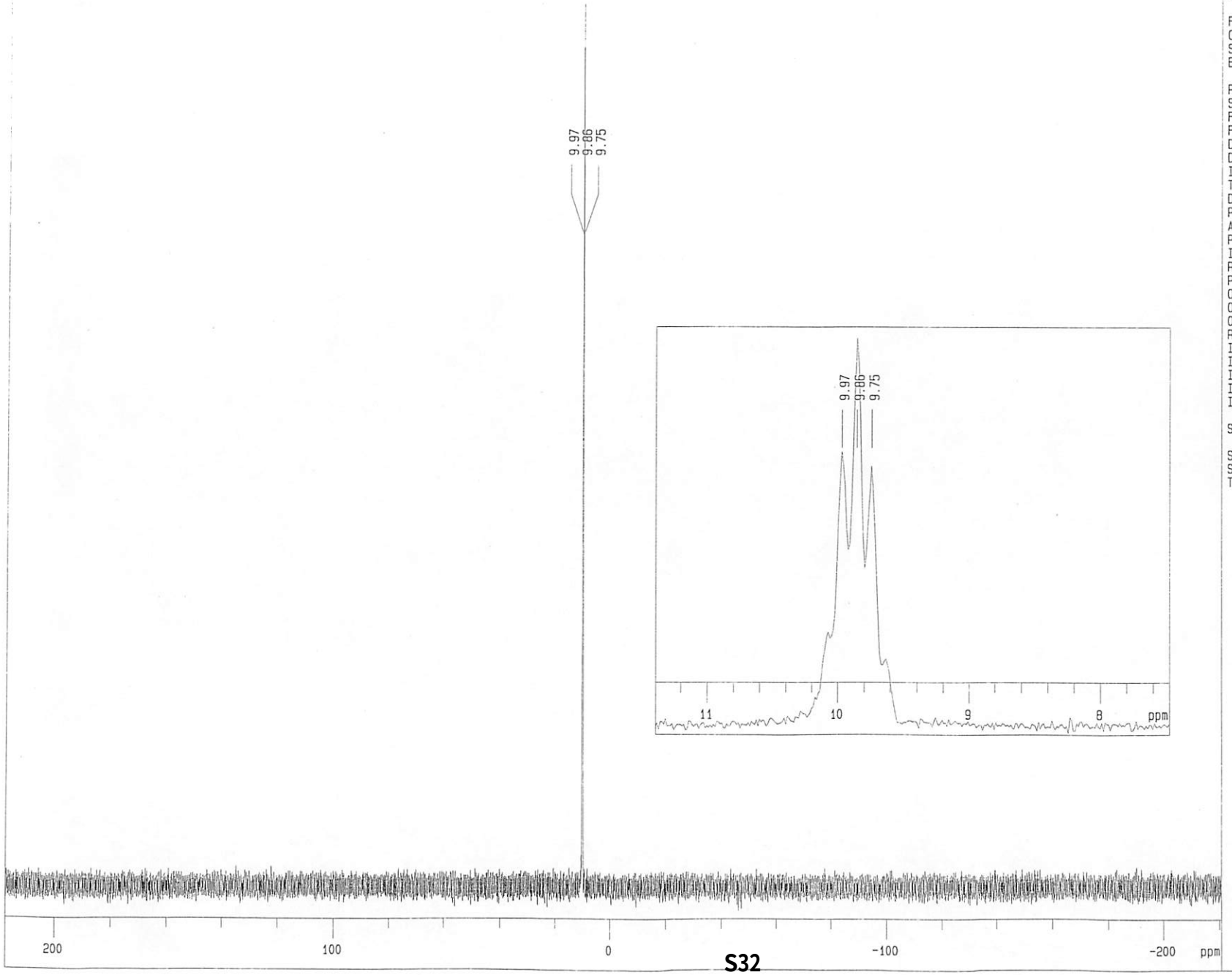
179

^{31}P NMR


1H Line
Date : Sat Nov 26 22:07:35 2011
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 58.3 usec
INTVL : 100.0 usec
TIMES : 45 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 25
SCANS : 45 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 16.7 C
7.9668 7.9516 7.9394 7.9241 7.6343 7.6196 7.5720 7.5562 7.5013 7.4891 7.4866 7.4738 7.4384 7.4232 7.4073 7.3896 7.3829 7.3737 7.3676 7.3585 7.3524 7.3353 7.3194 7.3042 7.2908 7.2731 7.2645 6.7892 6.7721
3.8245
1.6596 1.2649 0.8835 0.0000
4.00 1.09 1.05 2.01 4.93 3.04
8.0 7.8 7.6 7.4 ppm
4.00 1.88 2.01 4.93 3.04 1.98
6.7892 6.7721 1.98
.85 6.80 6.75
2.95
* H2O
hexane
10.0 7.5 5.0 2.5 0.0 ppm
P(O)Ph2
MeO
189
1H NMR

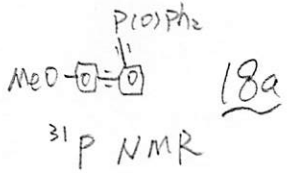

1H reso
159.812
133.718
133.196
132.871
132.847
132.106
132.045
132.008
131.806
130.910
130.762
130.252
128.586
128.409
127.632
127.221
127.196
121.912
121.858
114.370
113.851
104.019
103.620
94.729
87.096
85.899
84.855
77.465
77.041
76.618
55.208
55.147
Date :
FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRRPW : 34.0 usec
IRRNS : 0
SCANS : 343 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 20.0 C
200
150
100
50
0 ppm
ppm
MeO
PPh2
184
13C NMR

1H reso

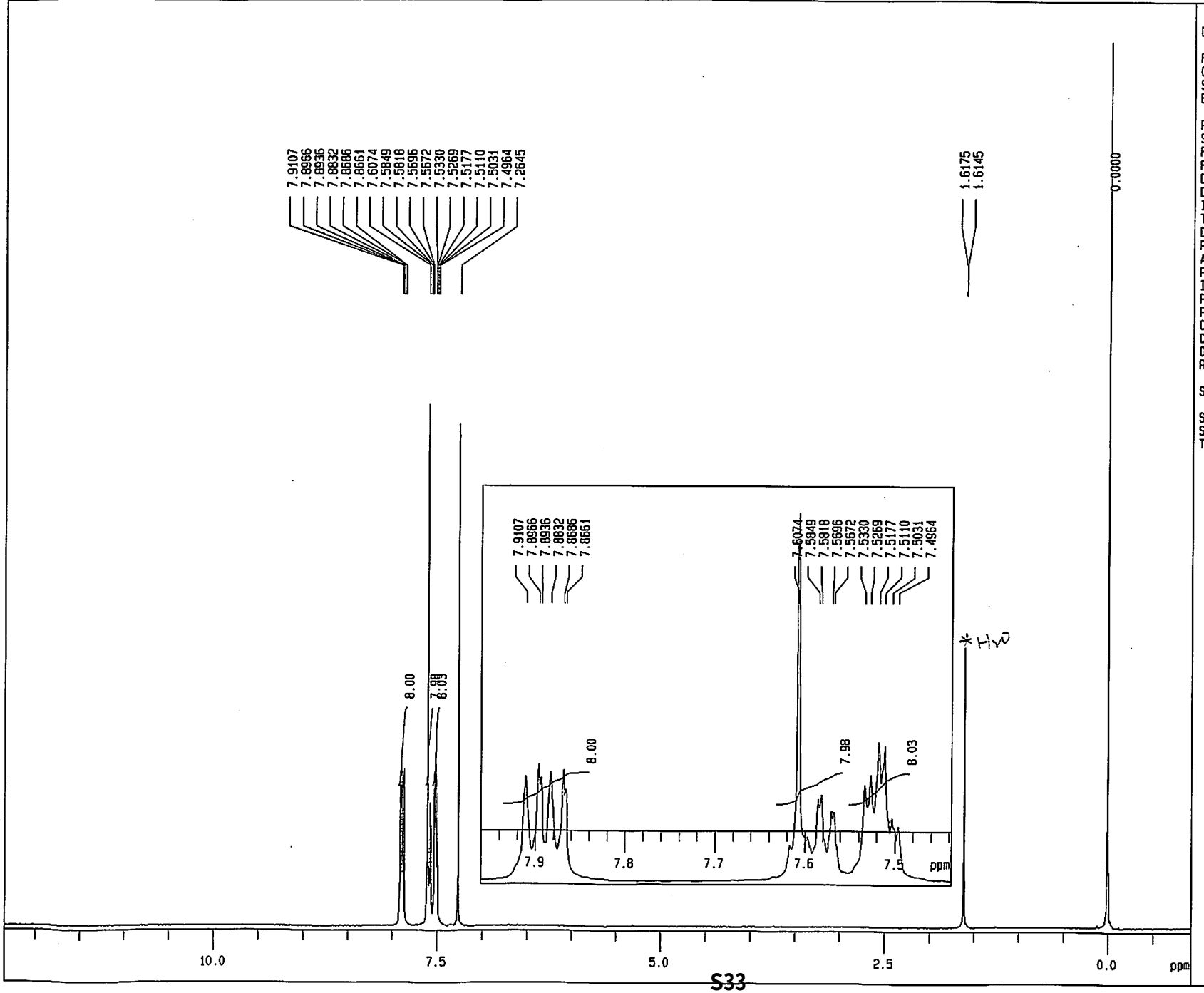


Date : Sun Aug 24 04:32:19 2003

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	100 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIWT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	22
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	100 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	17.1 C



1H Line

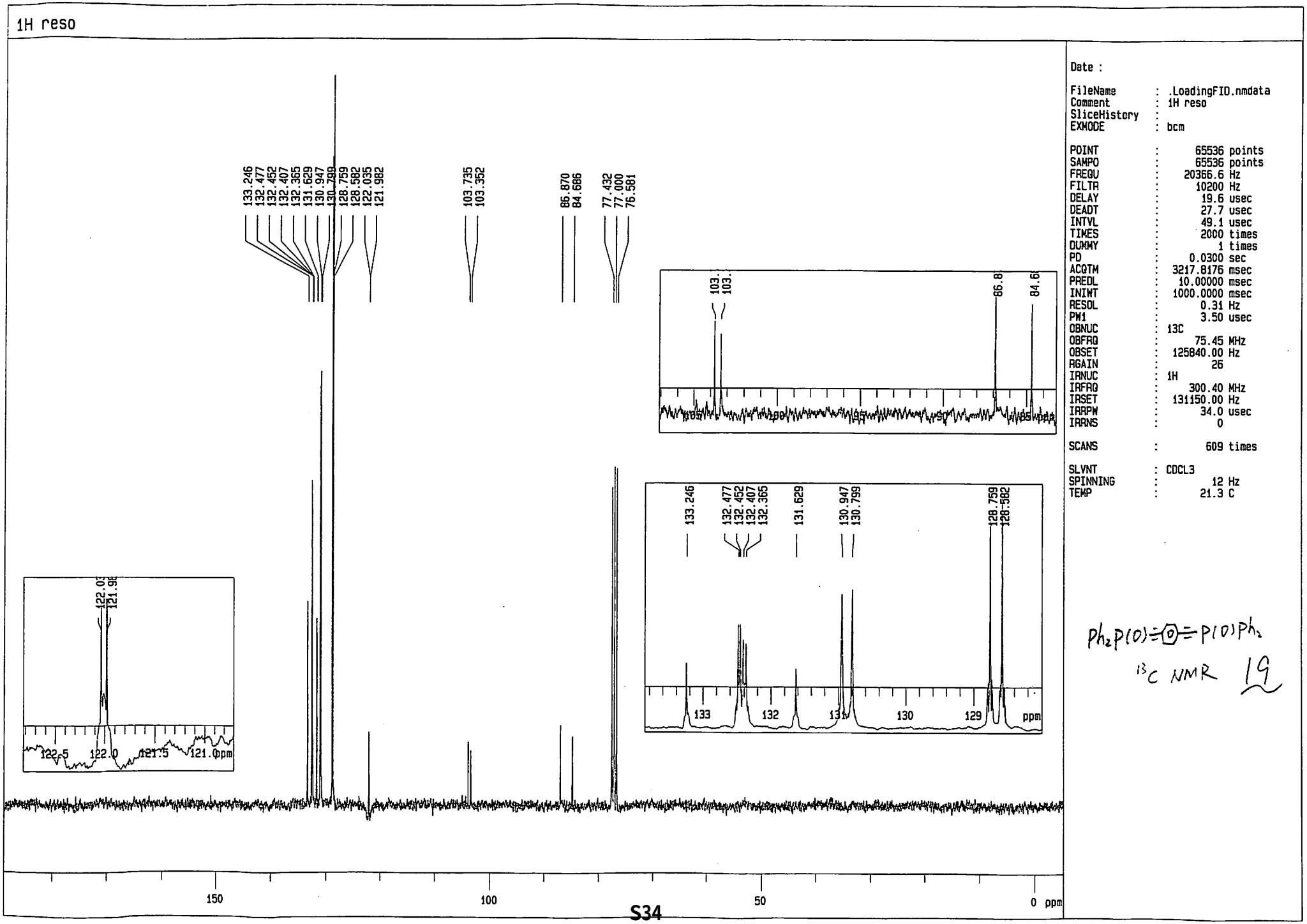


Date : Mon Nov 28 16:43:59 2011

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	10000.0 Hz
FILTR	:	5000 Hz
DELAY	:	40.0 usec
DEADT	:	58.3 usec
INTVL	:	100.0 usec
TIMES	:	35 times
DUMMY	:	1 times
PD	:	3.7232 sec
ACQTM	:	3276.7998 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.31 Hz
PW1	:	3.45 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162160.00 Hz
AGAIN	:	28
SCANS	:	35 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	16.7 C

$Ph_2P(O)$=⬡=$P(O)Ph_2$

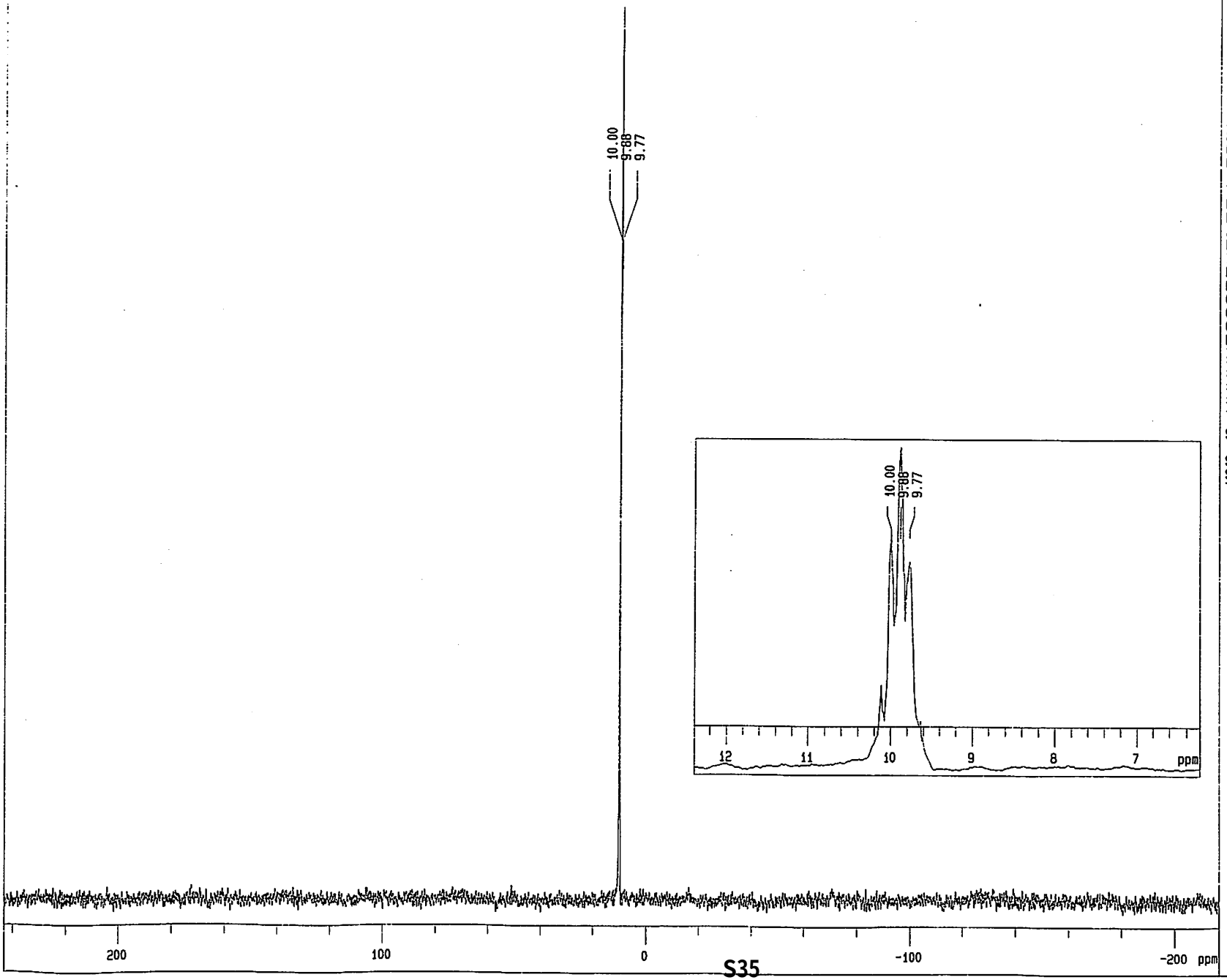
^1HNMR 19



$Ph_2P(O)\text{-}\boxed{O}\text{-}P(O)Ph_2$

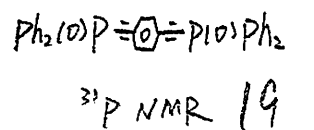
^{13}C NMR 19

1H reso

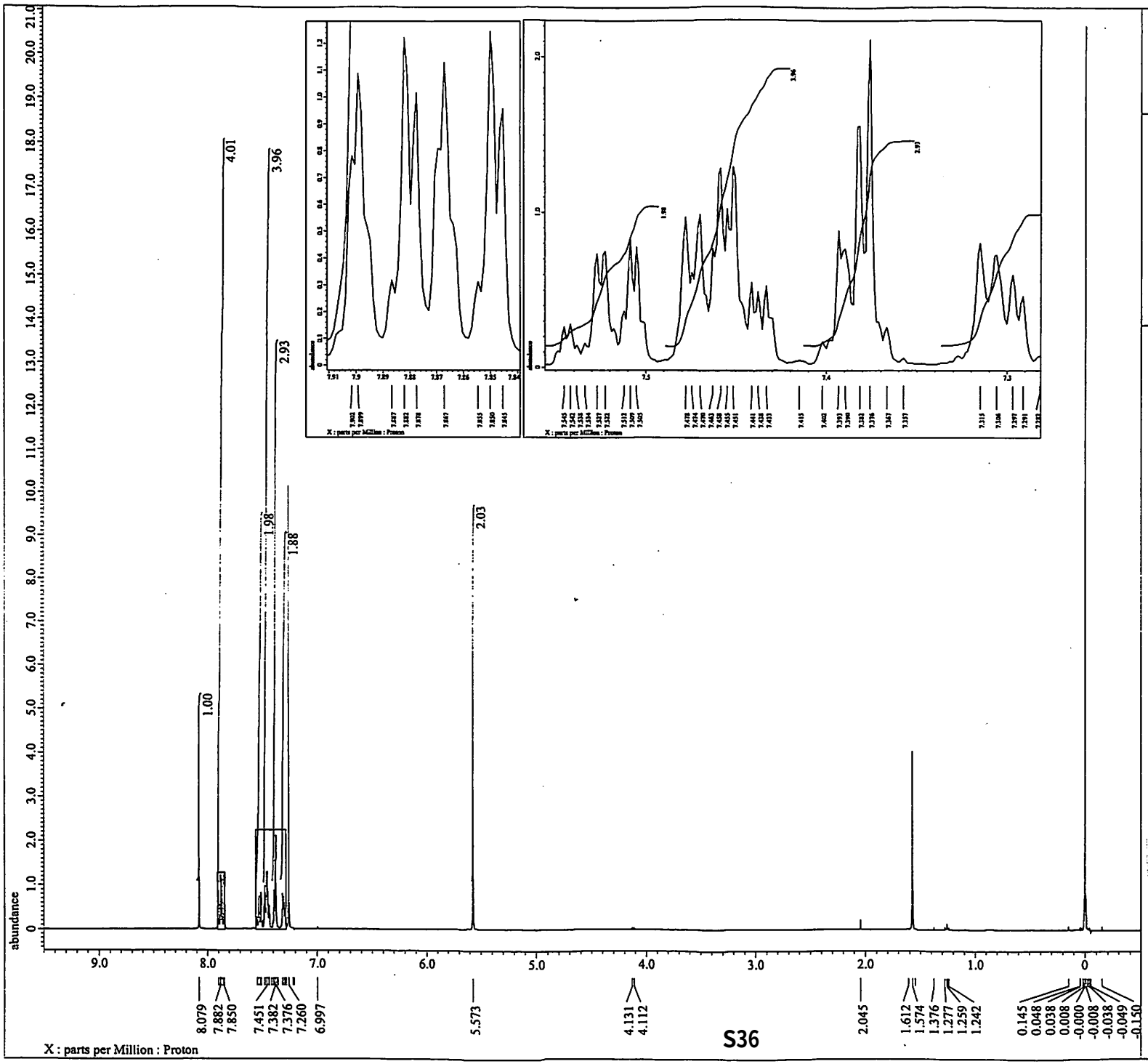


Date : Wed Aug 27 03:58:00 2003

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	50 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
ININT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	50 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	18.0 C



^{31}P NMR 19

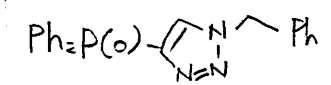


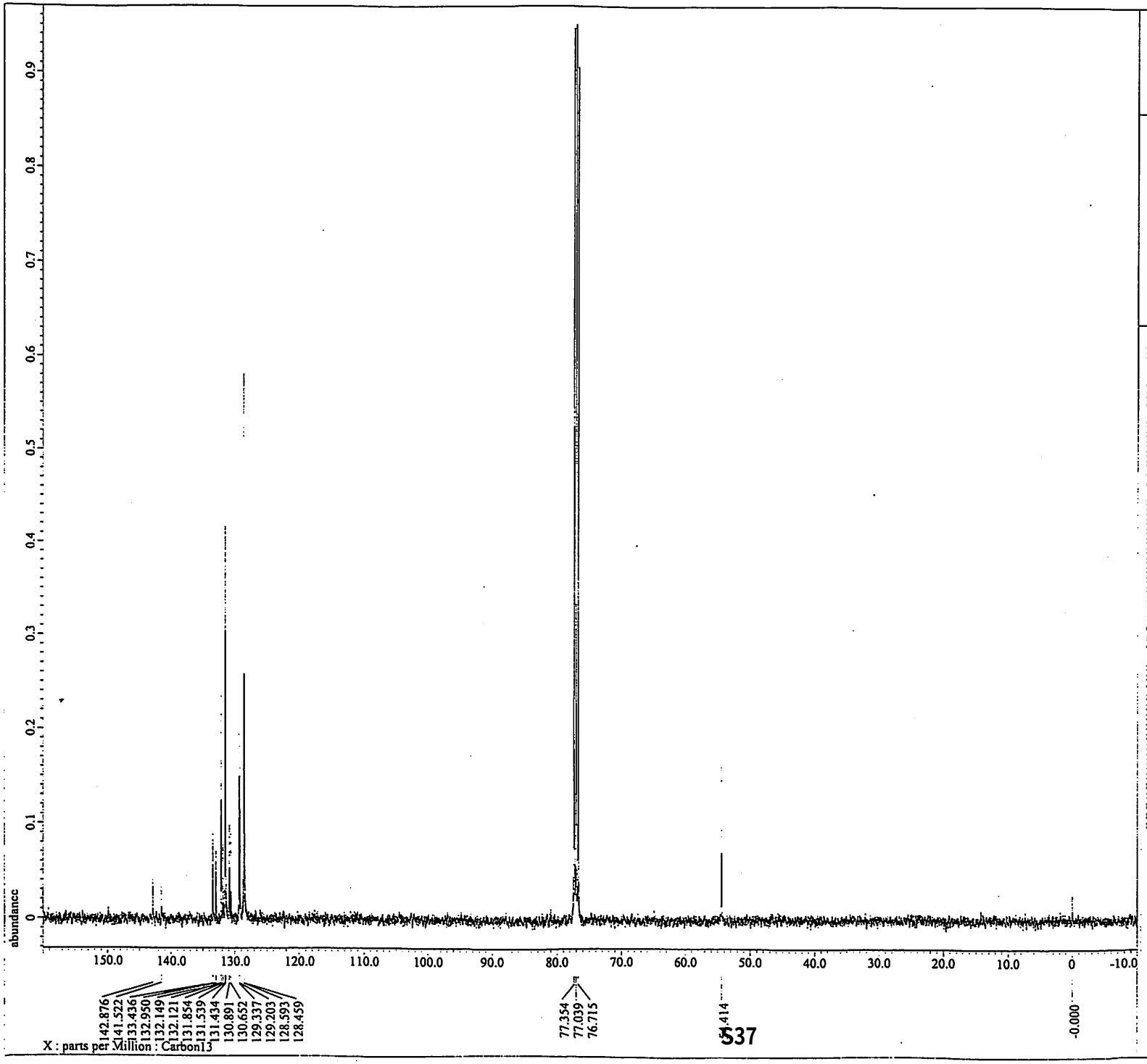
JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
nachinephase
ppm
auto_reference( 5[%], TRUE )

以下に由来:: _non_1H-107-1.jdf
```

Filename	= _non_1H-107-4.jdf
Author	= delta
Experiment	= single_pulse.jxp
Sample_Id	= 88662264
Solvent	= CHLOROFORM-D
Creation_Time	= 10-JUL-2014 18:24:18
Revision_Time	= 10-JUL-2014 18:03:36
Current_Time	= 10-JUL-2014 18:07:13
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 54
Temp_Get	= 24.5[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.7[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

^{1}H NMR



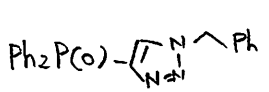


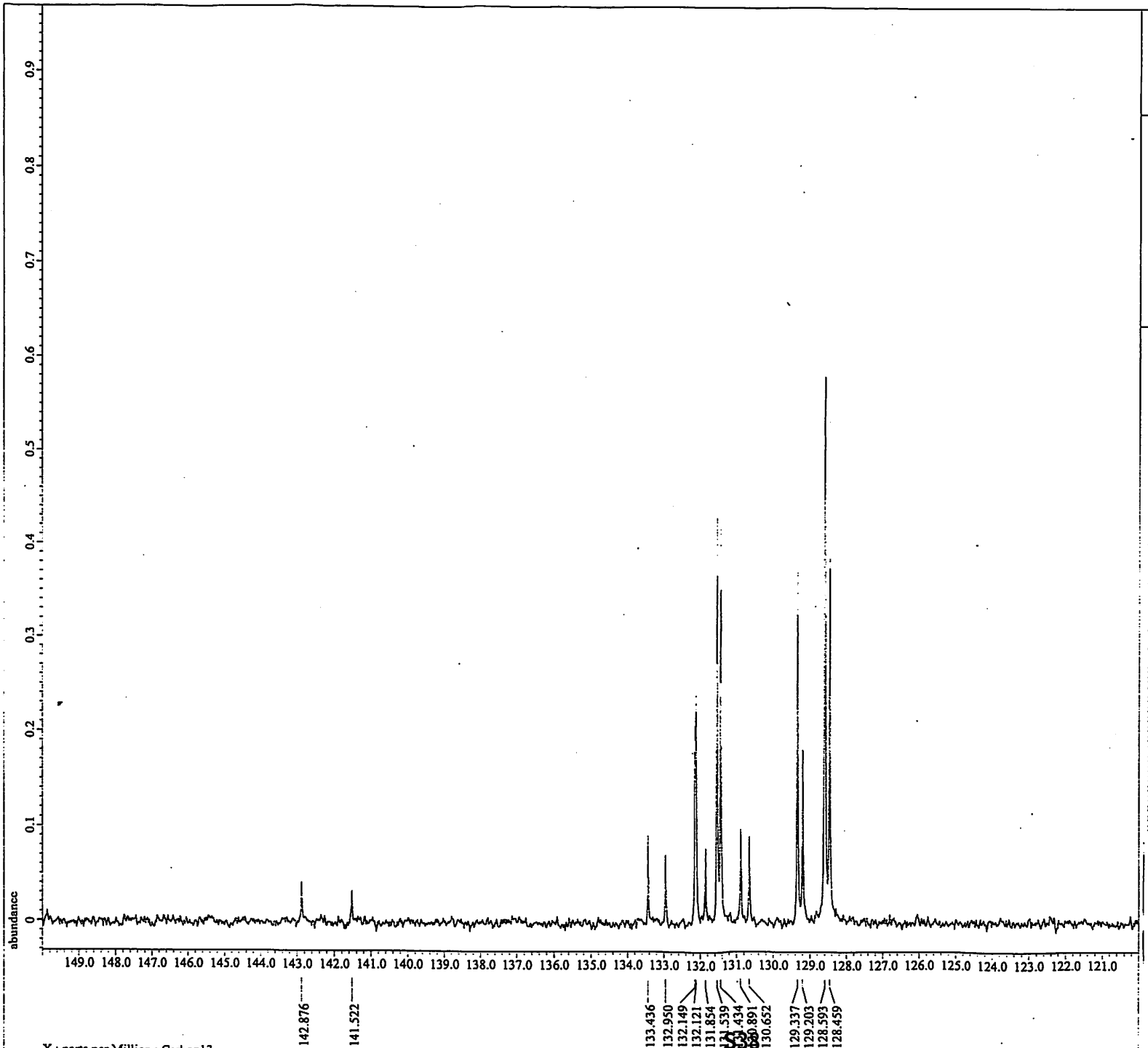
JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
nachinephase
ppm
auto_reference( 5[%], TRUE )

以下に由来:: __cm-13C-3-1.jdf
```

Filename	= __cm-13C-3-4.jdf
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= 88767383
Solvent	= CHLOROFORM-D
Creation_Time	= 22-SEP-2014 21:19:17
Revision_Time	= 14-OCT-2014 21:12:14
Current_Time	= 14-OCT-2014 21:12:25
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 500
Total_Scans	= 500
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 24.9[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6.2[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 22.03[dB]
Irr_Atn_Noe	= 22.03[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

^{13}C NMR



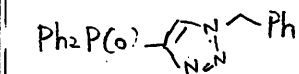


JEOL RESONANCE

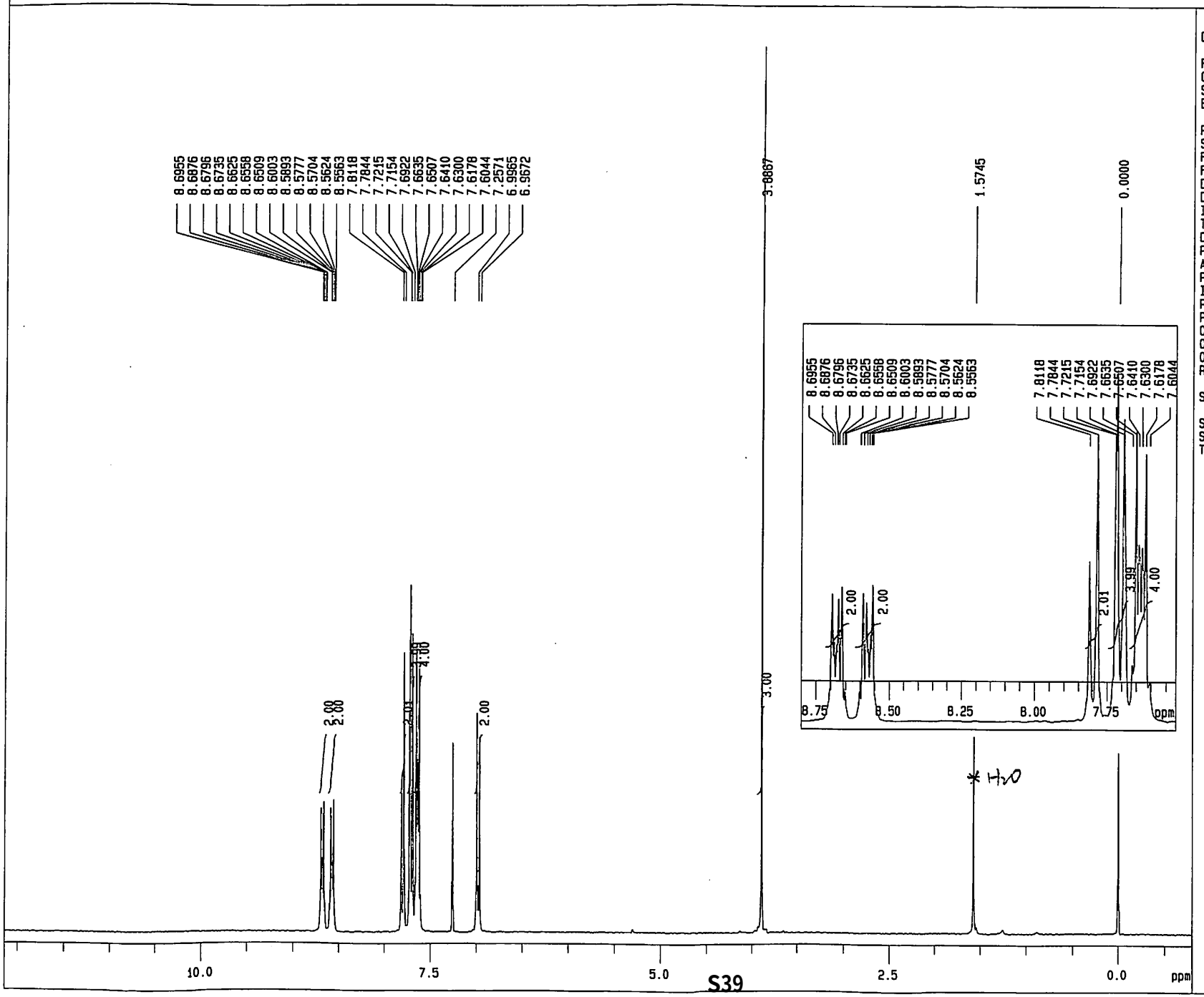
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
auto_reference( 5[%], TRUE )

以下に由来:: __cm-13C-3-1.jdf
```

Filename	= __cm-13C-3-3.jdf
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= 88767383
Solvent	= CHLOROFORM-D
Creation_Time	= 22-SEP-2016 21:19:17
Revision_Time	= 23-SEP-2016 09:22:25
Current_Time	= 23-SEP-2016 09:23:35
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 500
Total_Scans	= 500
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 24.9[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6.2[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 22.03[dB]
Irr_Atn_Noe	= 22.03[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

^{13}C NMR

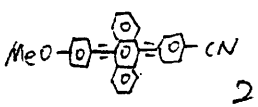


1H reso

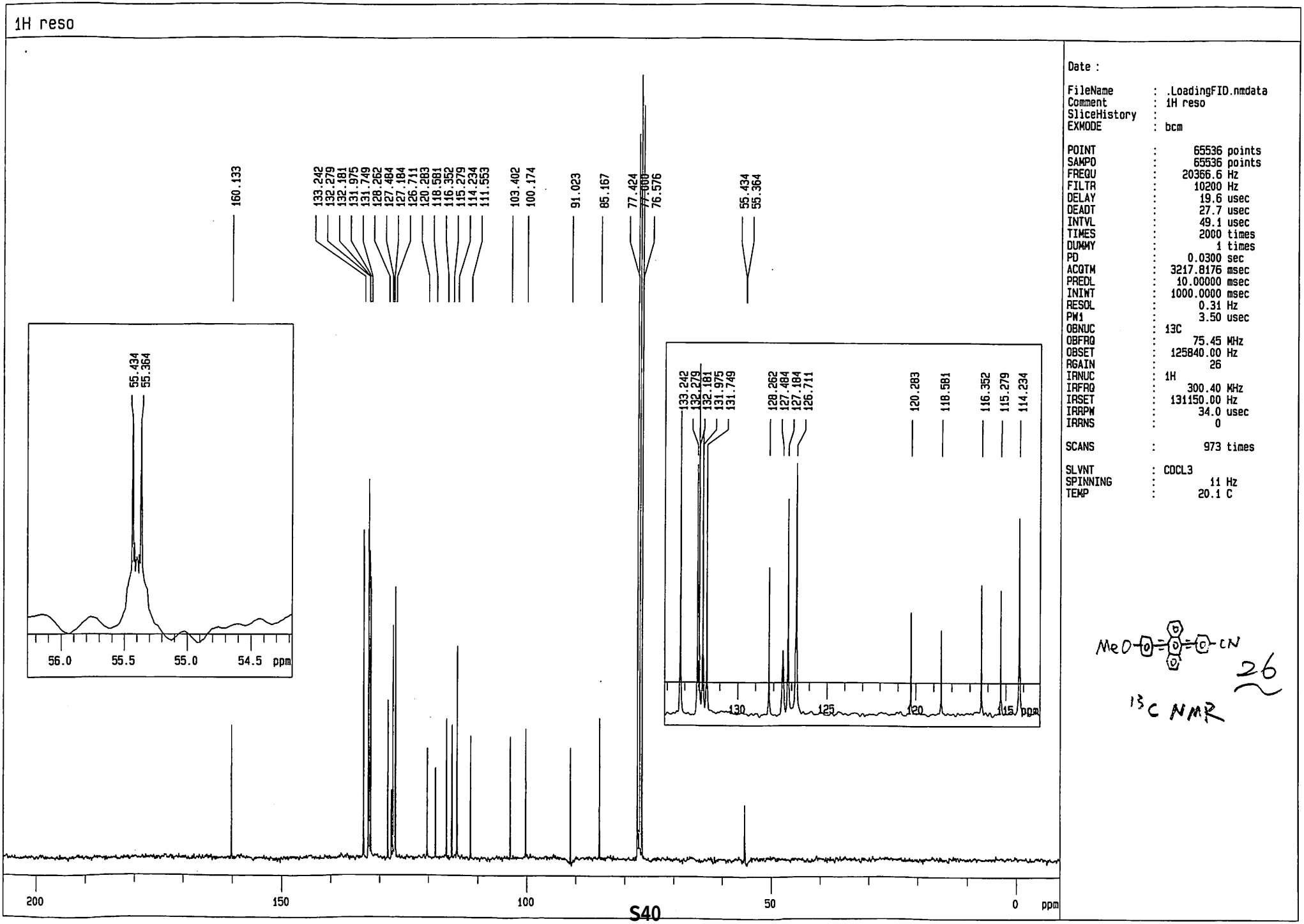


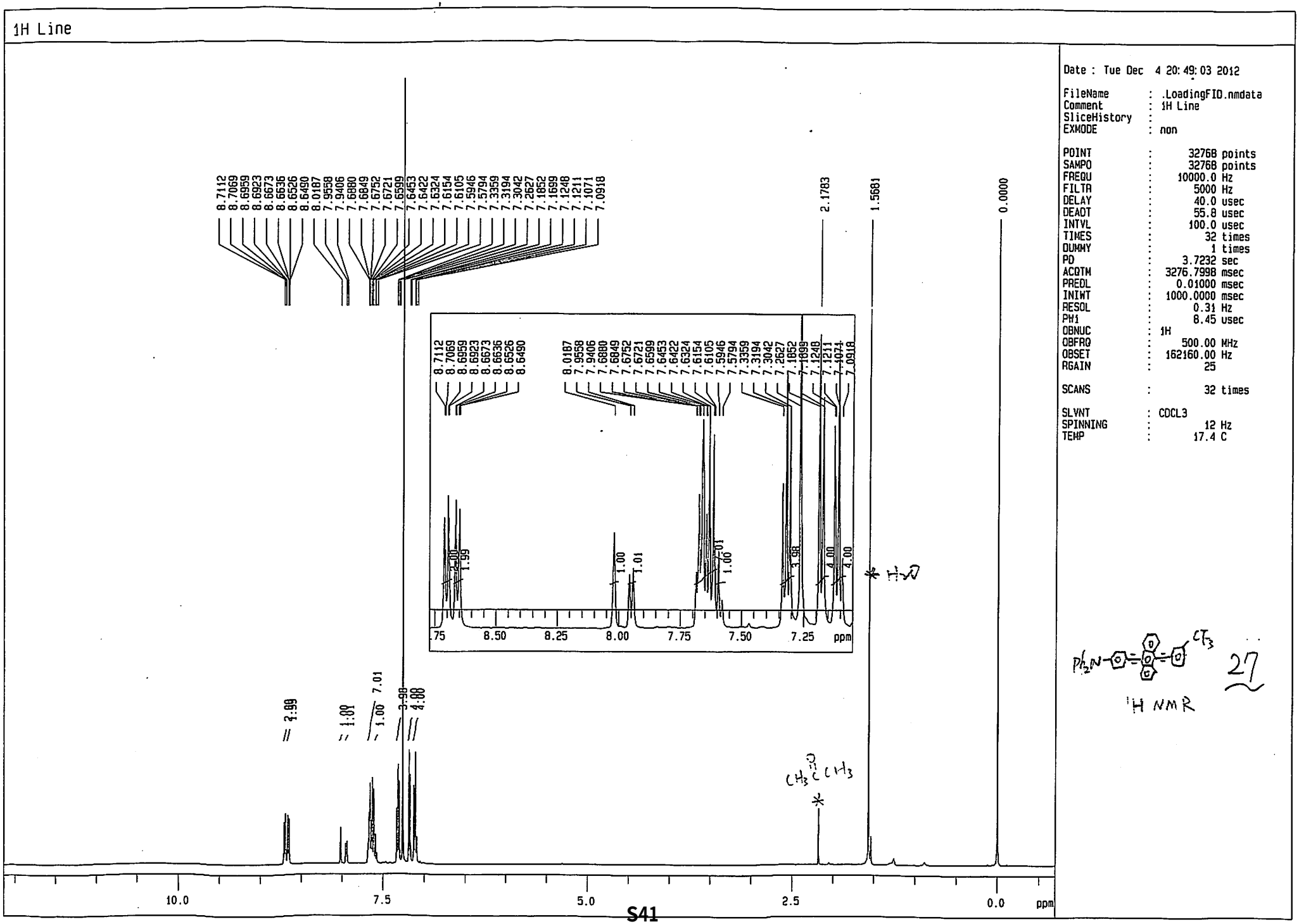
Date : Fri Aug 20 04:00:14 2004

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	6009.6 Hz
FILTR	:	3000 Hz
DELAY	:	66.7 usec
DEADT	:	97.5 usec
INTVL	:	166.4 usec
TIMES	:	4 times
DUMMY	:	1 times
PD	:	1.5474 sec
ACQTM	:	5452.5952 msec
PREDL	:	10.00000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.18 Hz
PW1	:	5.00 usec
OBNUC	:	1H
OBFRQ	:	300.40 MHz
OBSET	:	131150.00 Hz
RGAIN	:	21
SCANS	:	4 times
SLVNT	:	CDCL3
SPINNING	:	8 Hz
TEMP	:	19.2 C

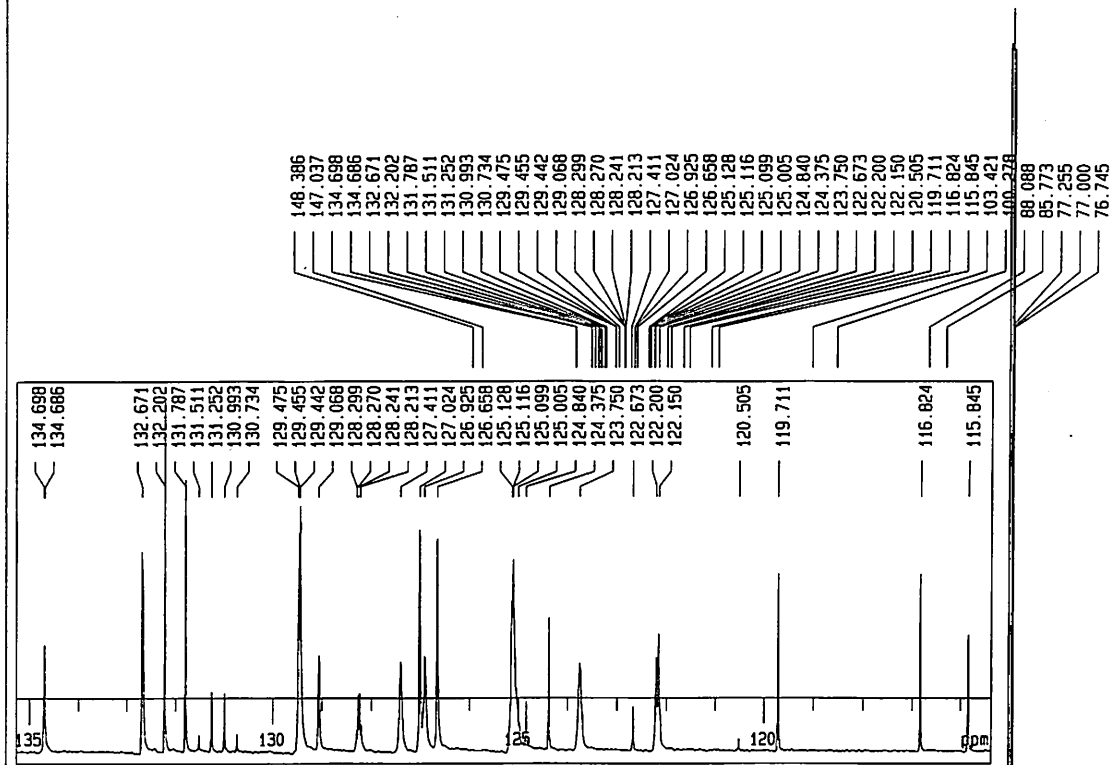


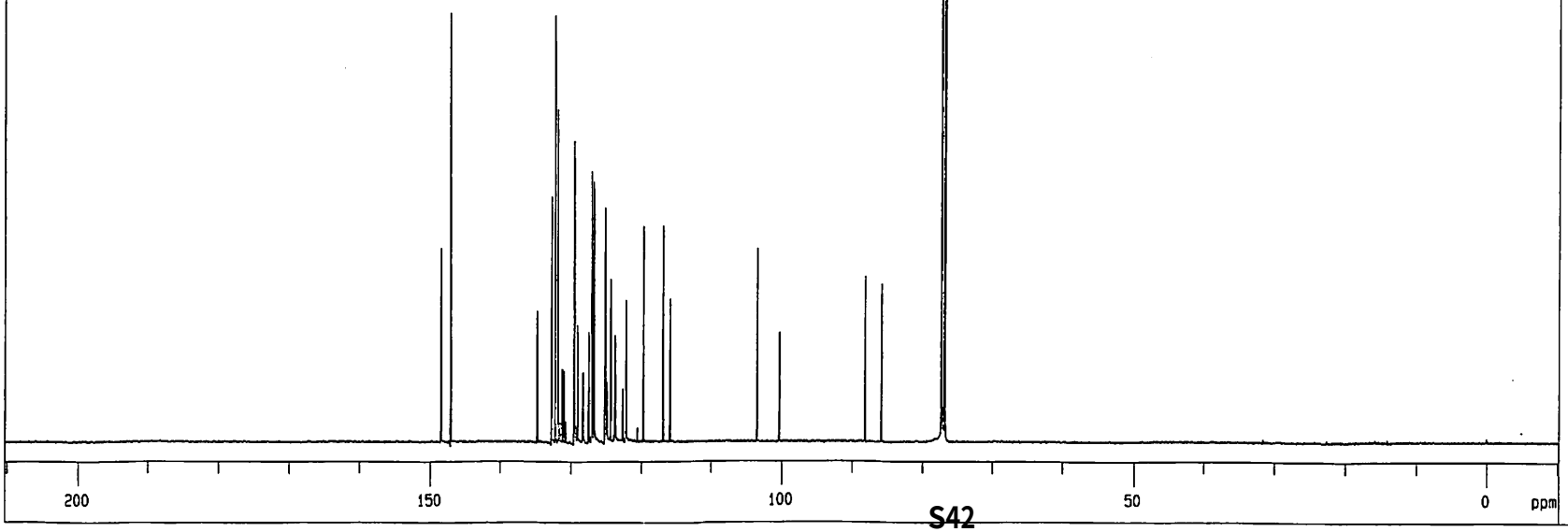
^{1}HNMR 26


1H reso
160.133
133.242
132.279
132.181
131.975
131.749
128.262
127.484
127.184
126.711
120.283
118.581
116.352
115.279
114.234
111.553
103.402
100.174
91.023
85.167
77.424
77.000
76.576
55.434
55.364
56.0
55.5
55.0
54.5 ppm
130
125
120
115 ppm
200
150
100
50
0 ppm
Date :
FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
ININT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRRPW : 34.0 usec
IRRNS : 0
SCANS : 973 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 20.1 C
MeO
CN
26
13C NMR


1H Line
Date : Tue Dec 4 20:49:03 2012
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 55.8 usec
INTVL : 100.0 usec
TIMES : 32 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 8.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 25
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 17.4 C
8.7112 8.7069 8.6959 8.6923 8.6673 8.6636 8.6526 8.6490 8.0187 7.9558 7.9406 7.6880 7.6849 7.6752 7.6721 7.6599 7.6453 7.6422 7.6324 7.6154 7.6105 7.5946 7.5794 7.3359 7.3194 7.3042 7.2627 7.1852 7.1699 7.1248 7.1211 7.1071 7.0918
2.1783
1.5681
0.0000
1.99
2.00
1.00
1.01
1.00
1.01
4.98
4.00
4.00
7.01
3.98
4.00
10.0
7.5
5.0
2.5
0.0
ppm
27
1H NMR

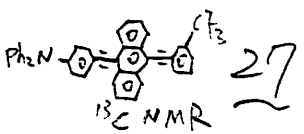
1H Line





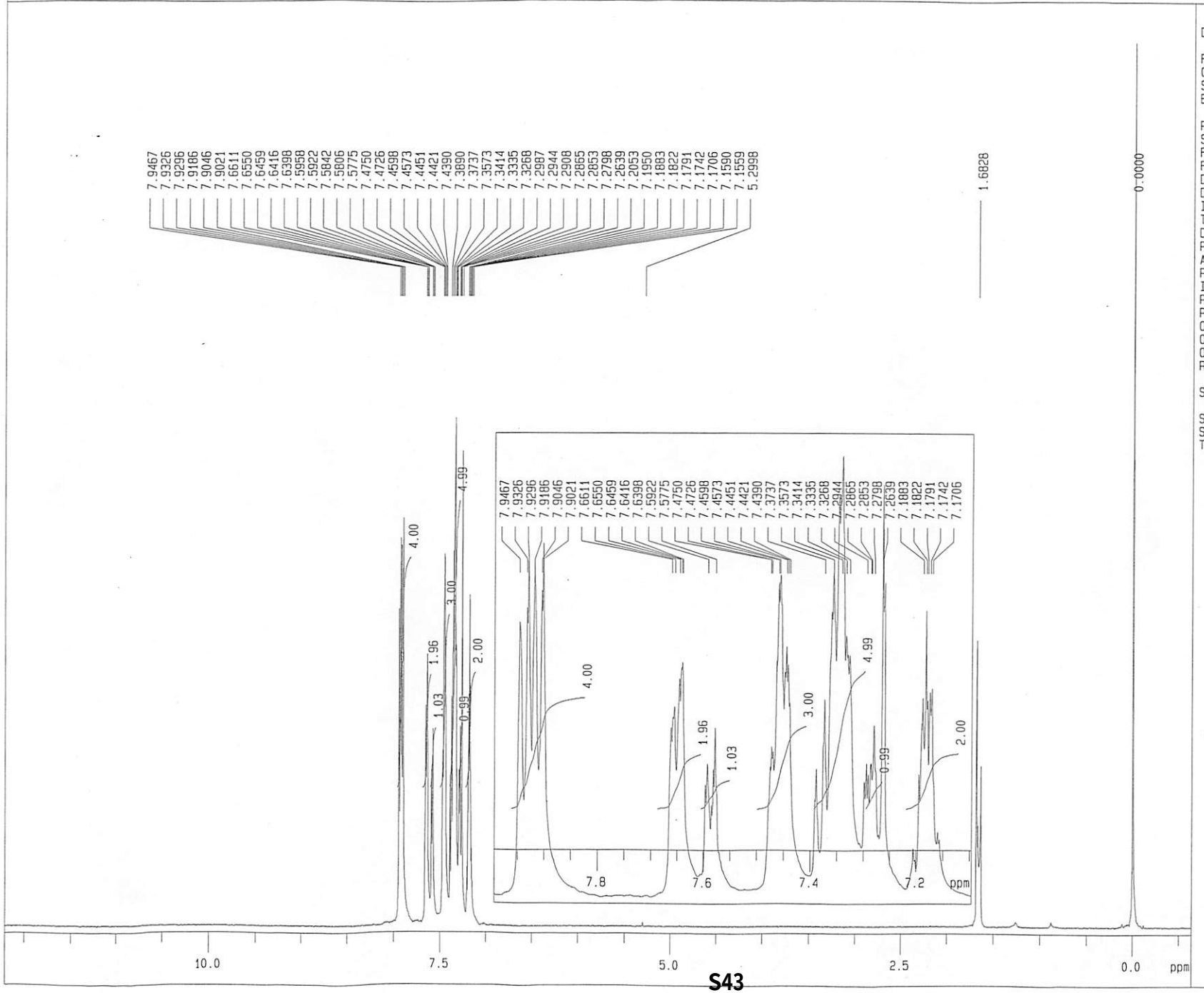
Date :

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	33898.3 Hz
FILTR	:	16950 Hz
DELAY	:	11.8 usec
DEADT	:	10.0 usec
INTVL	:	29.5 usec
TIMES	:	15000 times
DUMMY	:	1 times
PD	:	1.0667 sec
ACQTM	:	1933.3120 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.52 Hz
PW1	:	11.00 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
AGAIN	:	14
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRRPW	:	44.0 usec
IRRNS	:	0
SCANS	:	14227 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	18.5 C



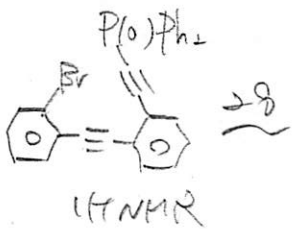
27

1H Line

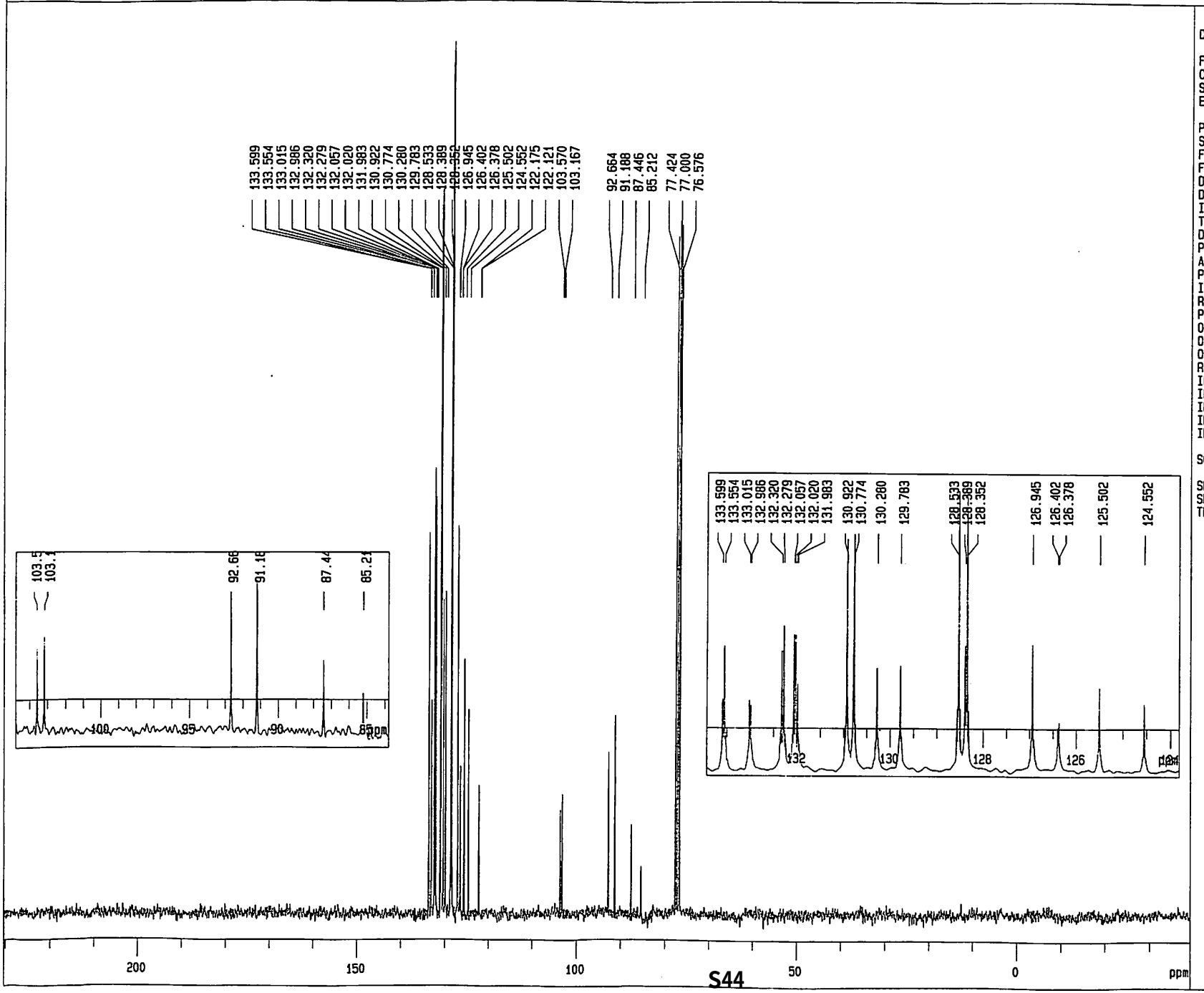


Date : Mon Dec 5 18:34:17 2011

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	10000.0 Hz
FILTR	:	5000 Hz
DELAY	:	40.0 usec
DEADT	:	58.3 usec
INTVL	:	100.0 usec
TIMES	:	45 times
DUMMY	:	1 times
PD	:	3.7232 sec
ACQTM	:	3276.7998 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.31 Hz
PW1	:	3.45 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162160.00 Hz
RGAIN	:	25
SCANS	:	45 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	17.3 C



1H reso



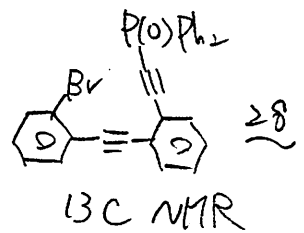
Date :

FileName : .LoadingFID.nmdata
Comment : 1H reso
SliceHistory :
EXMODE : bcm

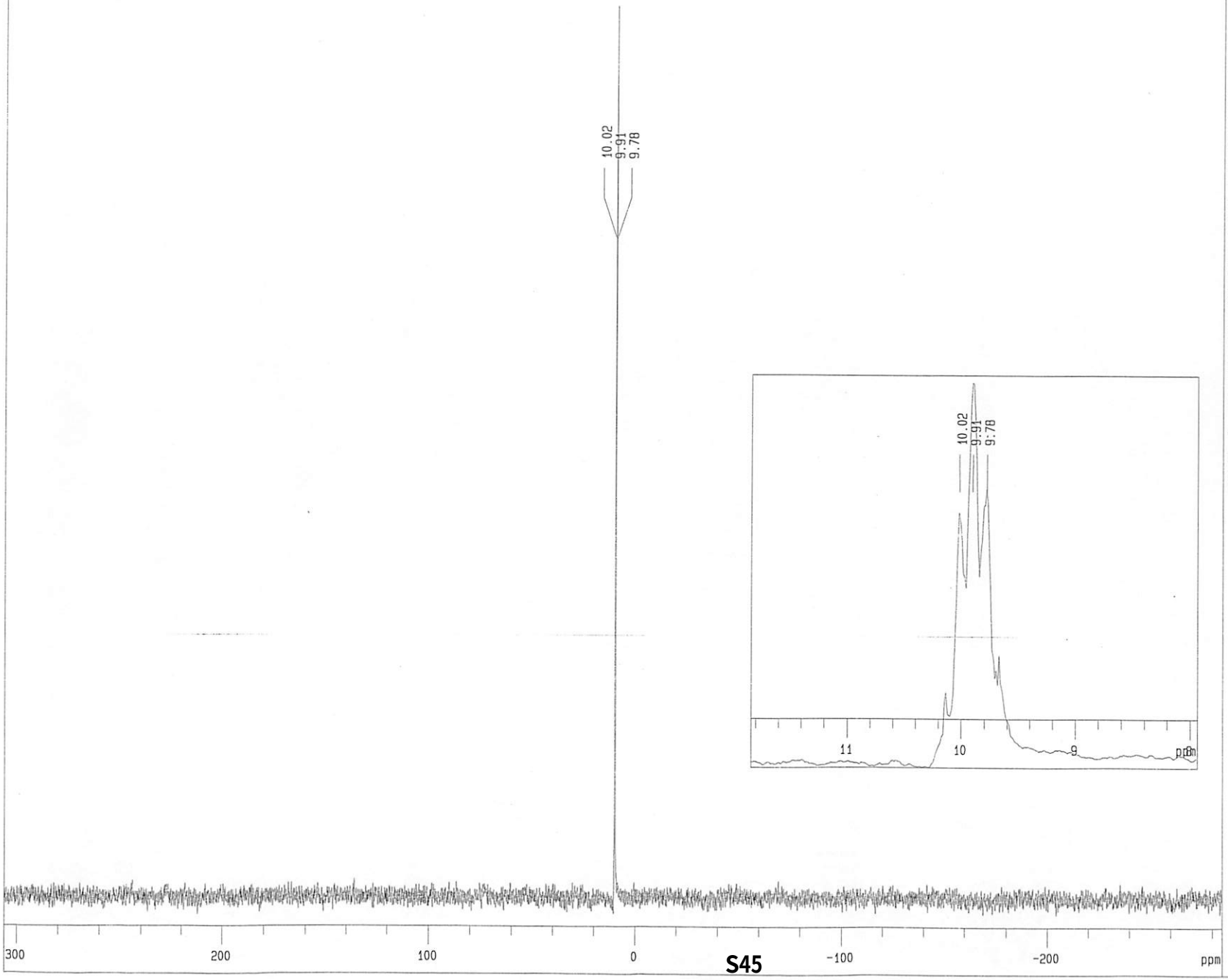
POINT : 65536 points
SAMPO : 65536 points
FREQU : 20366.6 Hz
FILTR : 10200 Hz
DELAY : 19.6 usec
DEADT : 27.7 usec
INTVL : 49.1 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 0.0300 sec
ACQTM : 3217.8176 msec
PREDL : 10.00000 msec
ININT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.50 usec
OBNUC : 13C
OBFRQ : 75.45 MHz
OBSET : 125840.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 300.40 MHz
IRSET : 131150.00 Hz
IRPPW : 34.0 usec
IRRNS : 0

SCANS : 377 times

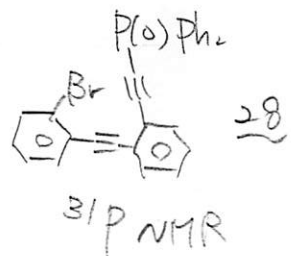
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.5 C

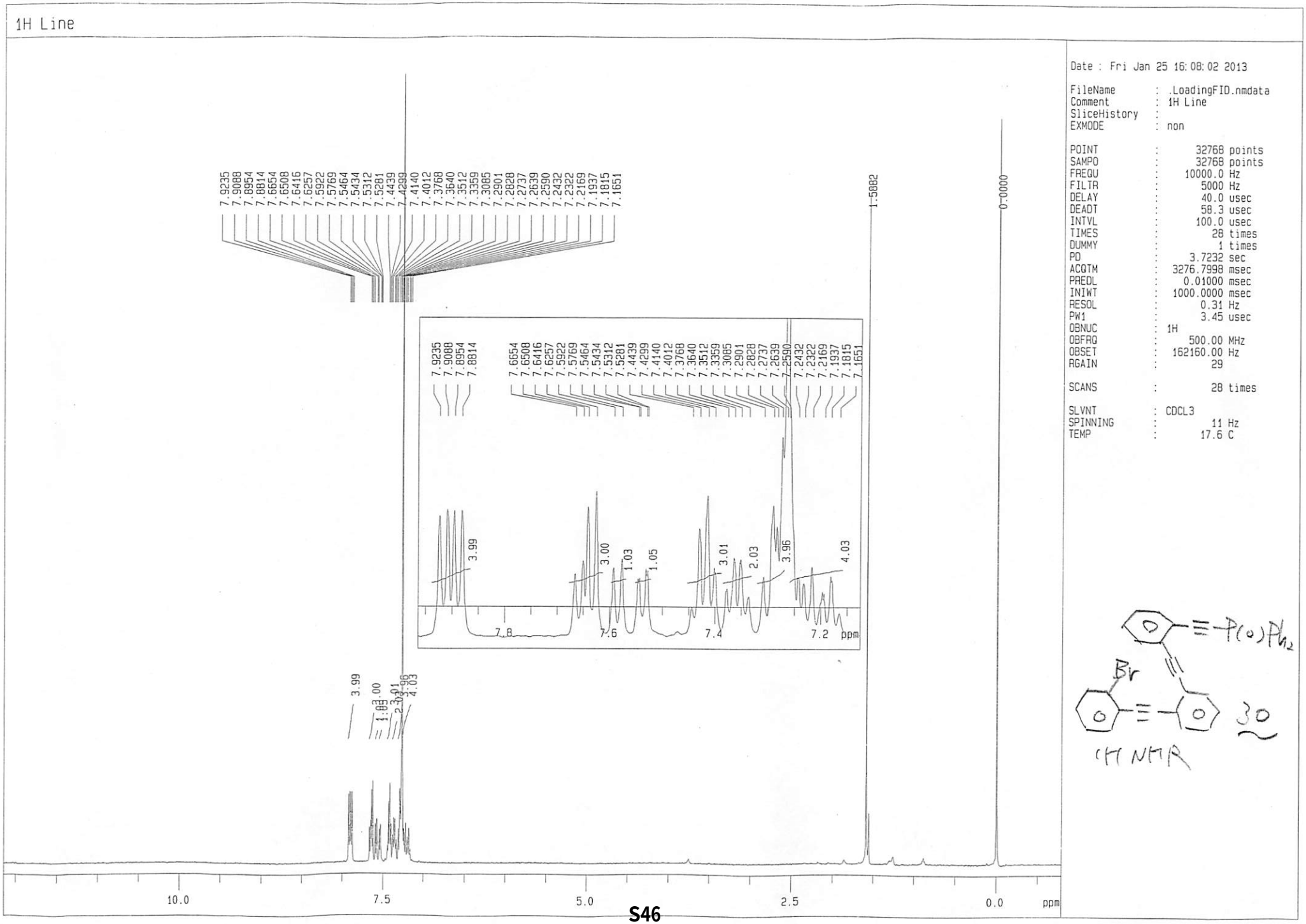


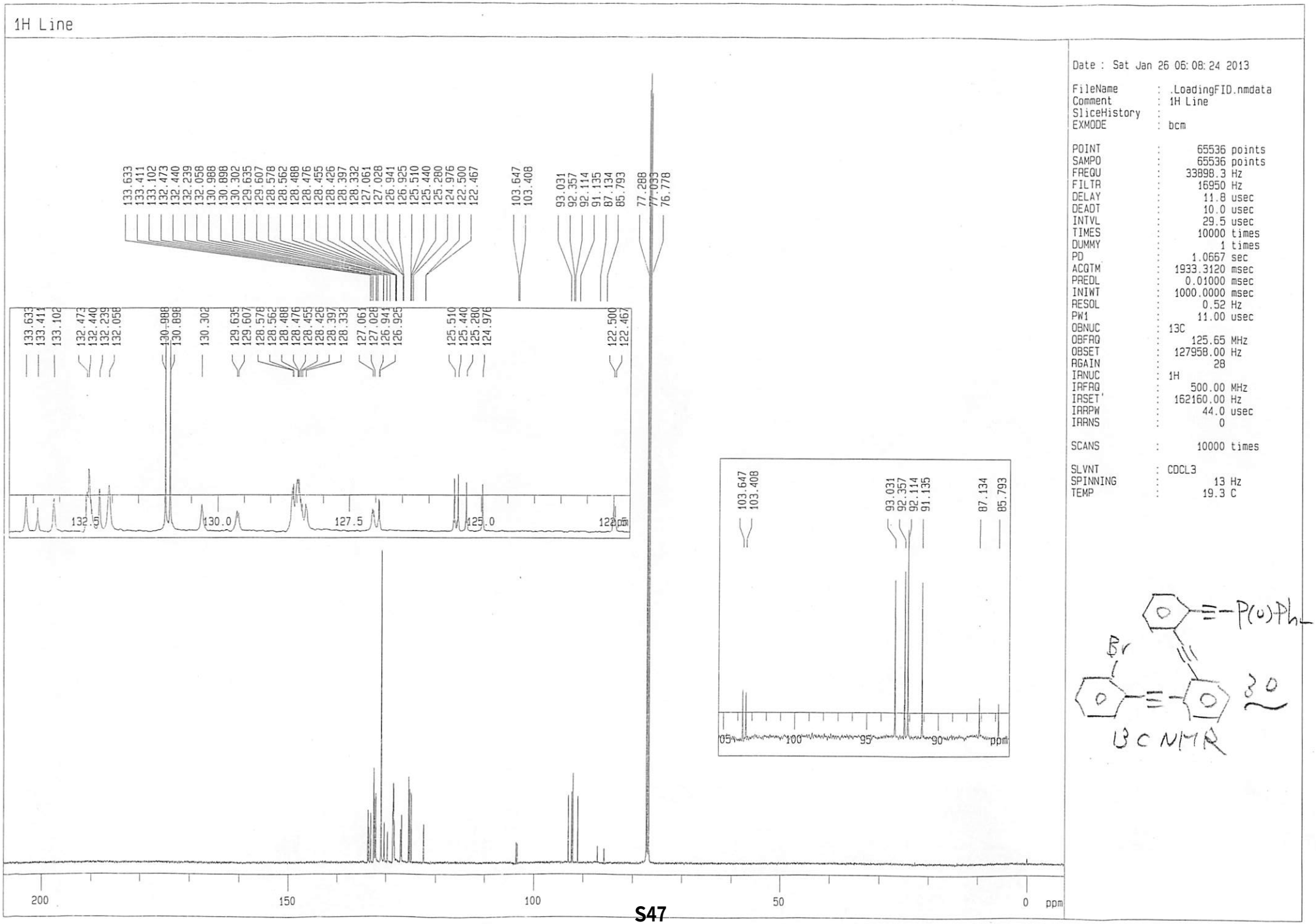
1H reso



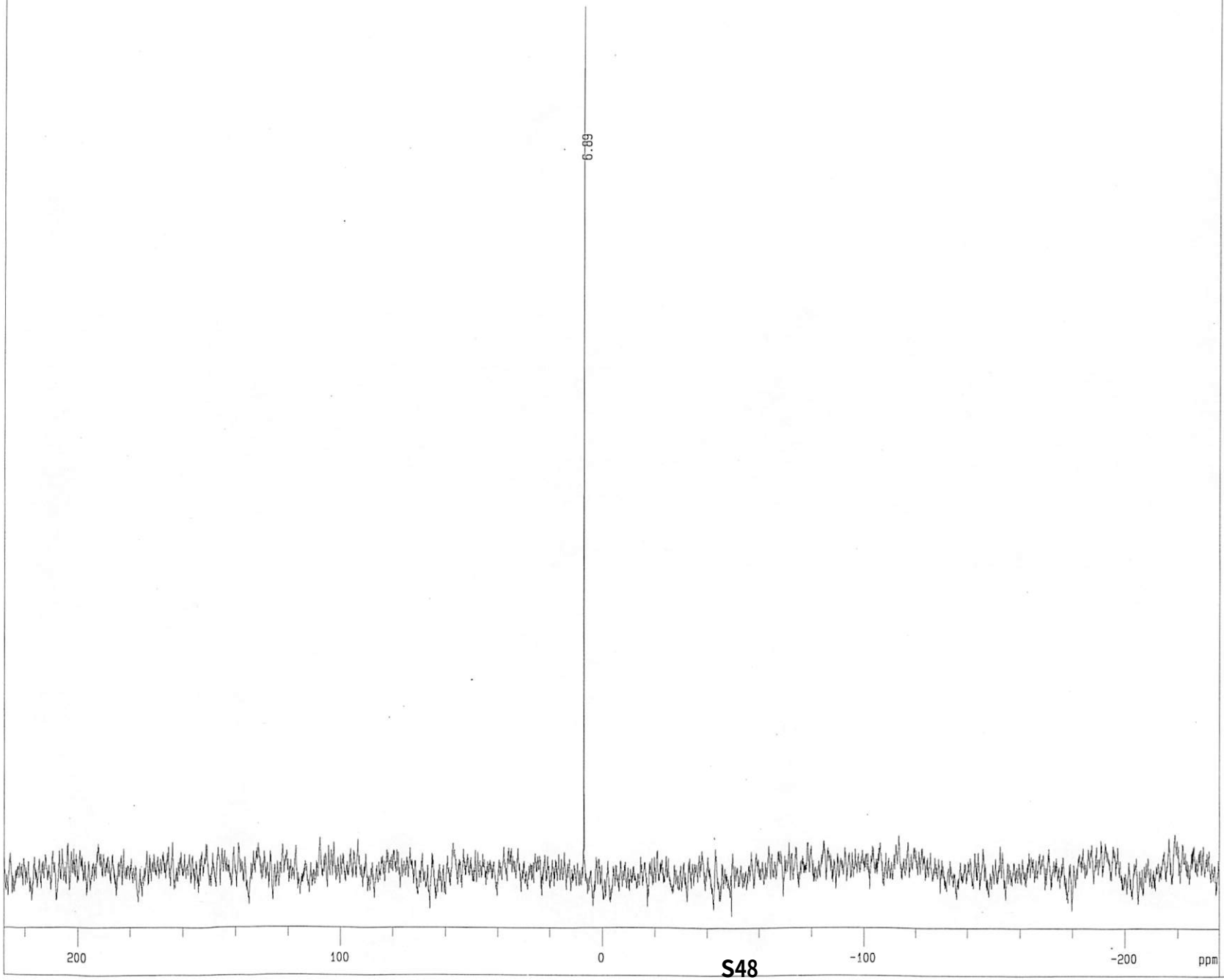

```
Date : Tue Sep  2 06:46:44 2003

FileName        : .LoadingFID.nmdata
Comment         : 1H reso
SliceHistory    :
EXMODE          : bcm

POINT           :      65536 points
SAMPO           :      65536 points
FREQU           :   100000.0 Hz
FILTR           :      50000 Hz
DELAY           :        4.0 usec
DEADT           :       10.0 usec
INTVL           :       10.0 usec
TIMES           :         60 times
DUMMY           :          0 times
PD              :     1.0000 sec
ACQTM           :   655.3600 msec
PREDL           :   10.00000 msec
INIWT           :  1000.0000 msec
RESOL           :       1.53 Hz
PW1             :       3.50 usec
OBNUC           : 31P
OBFRQ           :     121.50 MHz
OBSET           :  153873.56 Hz
RGAIN           :         23
IRNUC           : 1H
IRFRQ           :     300.40 MHz
IRSET           :  131150.00 Hz
IRRPW           :        1.0 usec
IRRNS           :          0

SCANS           :         60 times

SLVNT           : CDCL3
SPINNING        :         12 Hz
TEMP            :       17.5 C
```




1H Line
7.9235
7.9088
7.8954
7.8814
7.6654
7.6508
7.6416
7.6257
7.5922
7.5769
7.5464
7.5434
7.5312
7.5281
7.4439
7.4299
7.4140
7.4012
7.3768
7.3640
7.3512
7.3359
7.3085
7.2901
7.2828
7.2737
7.2639
7.2590
7.2432
7.2322
7.2169
7.1937
7.1815
7.1651
1.5882
0.0000
3.99
3.00
1.03
1.05
3.01
2.03
3.96
4.03
7.8
7.6
7.4
7.2
ppm
10.0
7.5
5.0
2.5
0.0
ppm
Date : Fri Jan 25 16:08:02 2013
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 58.3 usec
INTVL : 100.0 usec
TIMES : 28 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 29
SCANS : 28 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 17.6 C
P(O)Ph2
Br
30
1H NMR

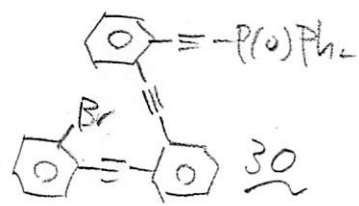

1H Line
133.633
133.411
133.102
132.473
132.440
132.239
132.058
130.988
130.898
130.302
129.635
129.607
128.578
128.562
128.488
128.476
128.455
128.426
128.397
128.332
127.061
127.028
126.941
126.925
125.510
125.440
125.280
124.976
122.500
122.467
103.647
103.408
93.031
92.357
92.114
91.135
87.134
85.793
77.288
77.033
76.778
132.5
130.0
127.5
125.0
122.5
ppm
105
100
95
90
ppm
200
150
100
50
0
ppm
Date : Sat Jan 26 06:08:24 2013
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 10000 times
DUMMY : 1 times
PD : 1.0667 sec
ACQTM : 1933.3120 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.52 Hz
PW1 : 11.00 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 44.0 usec
IRRNS : 0
SCANS : 10000 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 19.3 C
P(O)Ph2
Br
30
13C NMR

1H reso

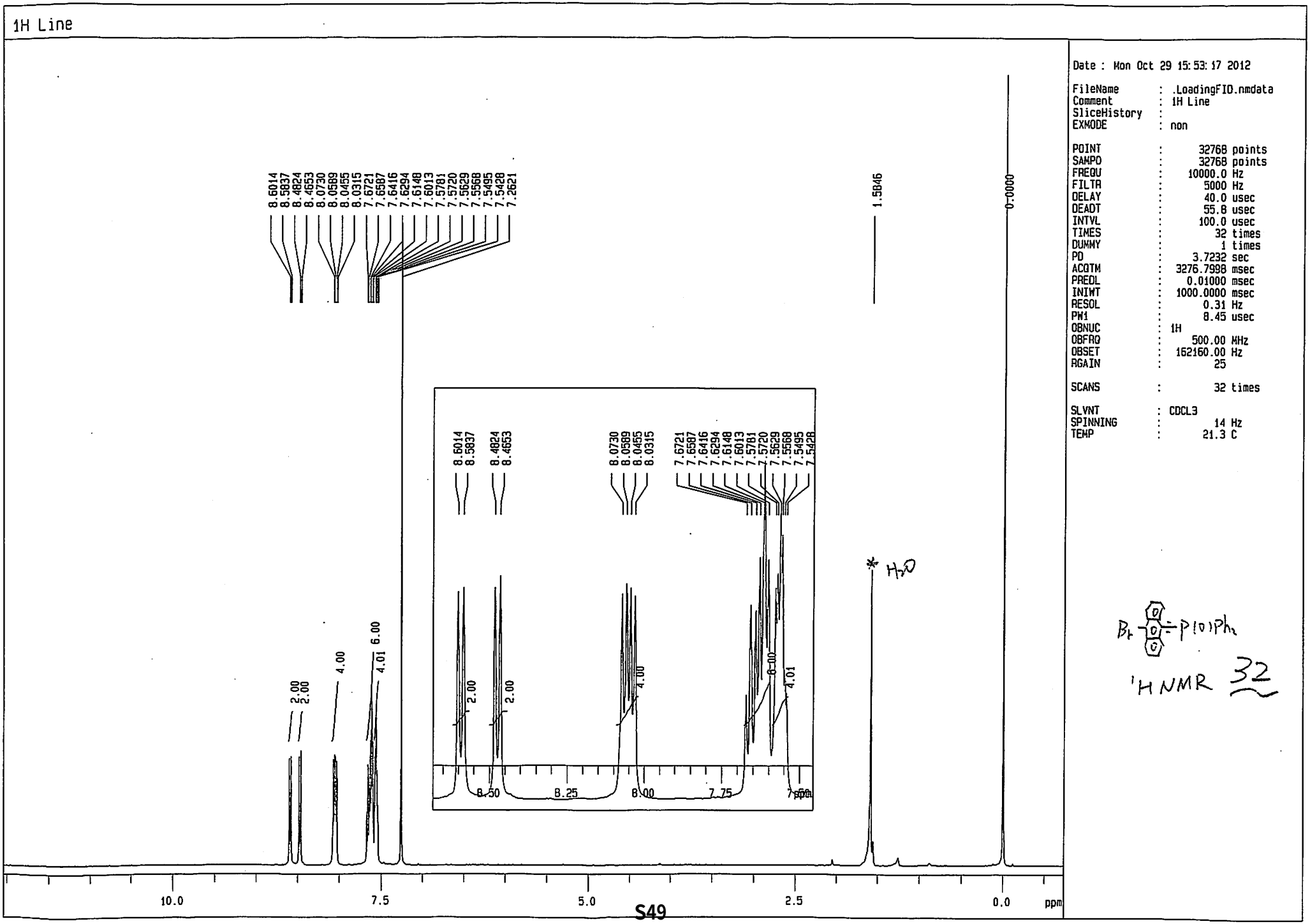


Date :

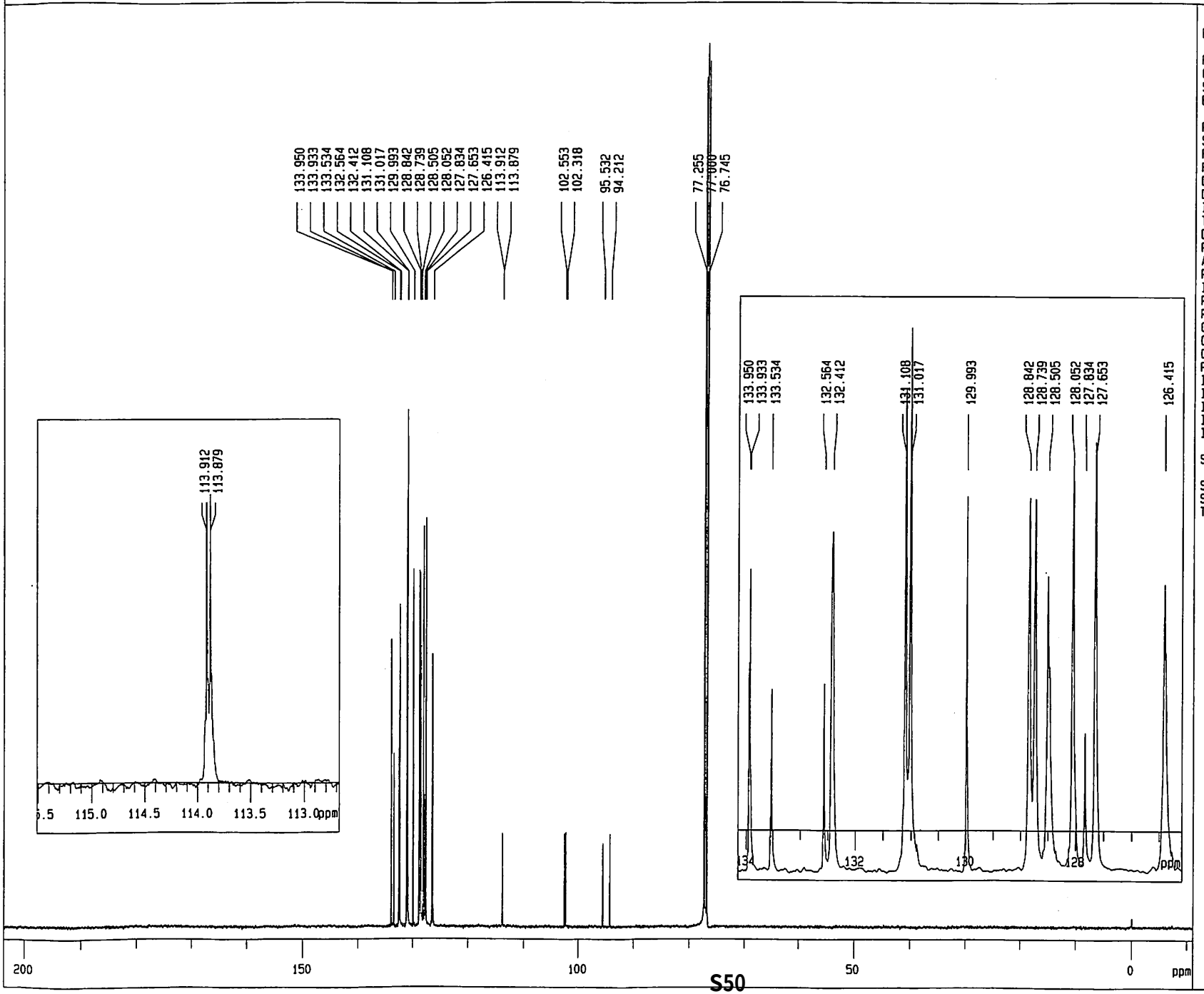
FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	80 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIWT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	24
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	33 times
SLVNT	:	D2O
SPINNING	:	12 Hz
TEMP	:	18.6 C



^{31}P NMR

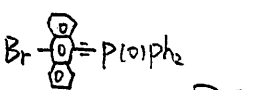

1H Line
8.6014
8.5837
8.4824
8.4653
8.0730
8.0589
8.0455
8.0315
7.6721
7.6587
7.6416
7.6294
7.6148
7.6013
7.5781
7.5720
7.5629
7.5568
7.5495
7.5428
7.2621
1.5846
0.0000
2.00
2.00
4.00
6.00
4.01
* H2O
8.50
8.25
8.00
7.75
10.0
7.5
5.0
2.5
0.0
ppm
Date : Mon Oct 29 15:53:17 2012
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 55.8 usec
INTVL : 100.0 usec
TIMES : 32 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 8.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 25
SCANS : 32 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 21.3 C
Bt
P(O)Ph2
1HNMR 32

1H Line



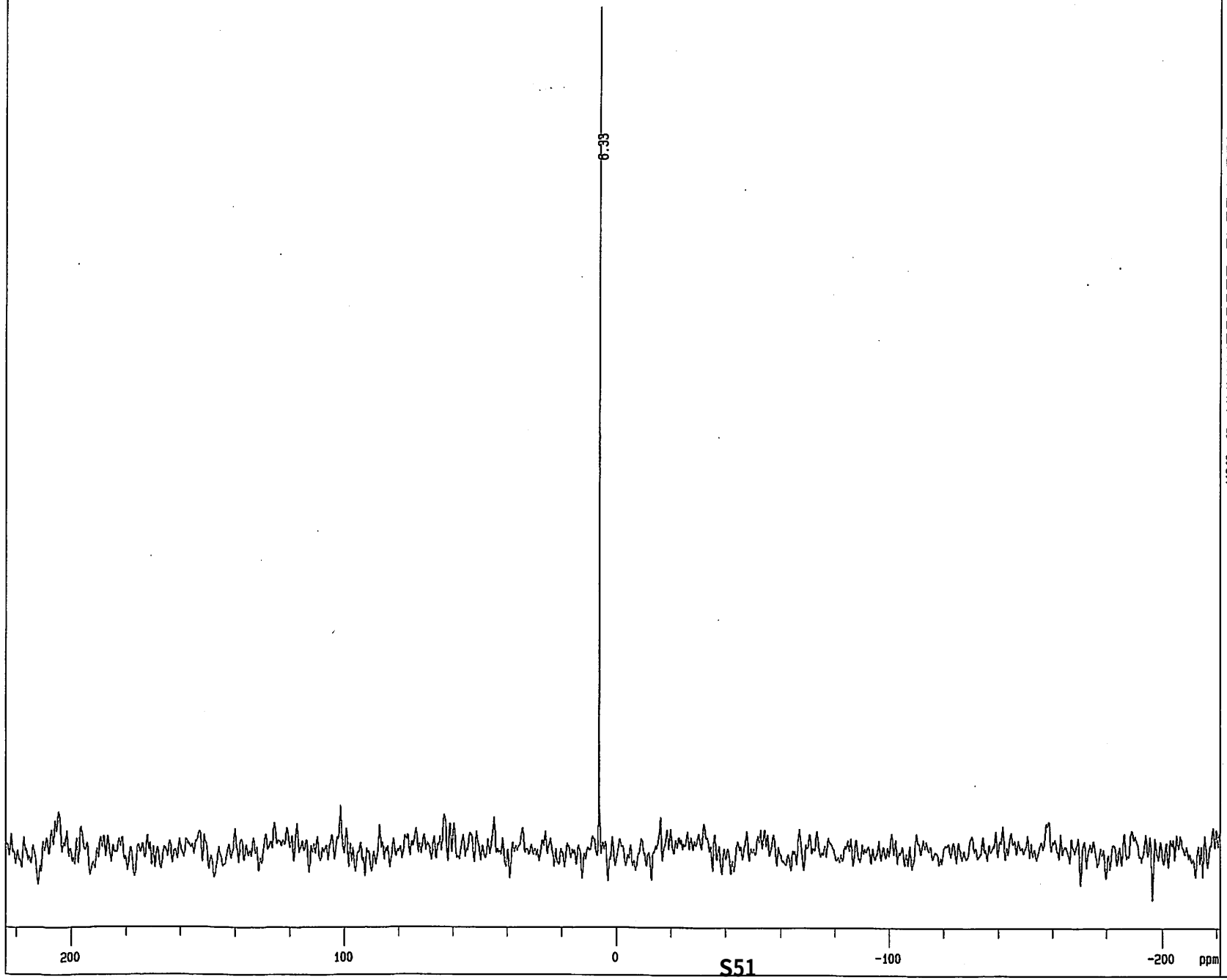
Date : Tue Oct 30 08:01:39 2012

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPD	:	65536 points
FREQU	:	33898.3 Hz
FILTR	:	16950 Hz
DELAY	:	11.8 usec
DEADT	:	10.0 usec
INTVL	:	29.5 usec
TIMES	:	12000 times
DUMMY	:	1 times
PD	:	1.0667 sec
ACQTM	:	1933.3120 msec
PREDL	:	0.01000 msec
ININT	:	1000.0000 msec
RESOL	:	0.52 Hz
PW1	:	11.00 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	29
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRPW	:	44.0 usec
IRNS	:	0
SCANS	:	12000 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	21.5 C



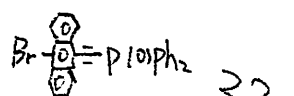
^{13}C NMR 32

1H reso



Date :

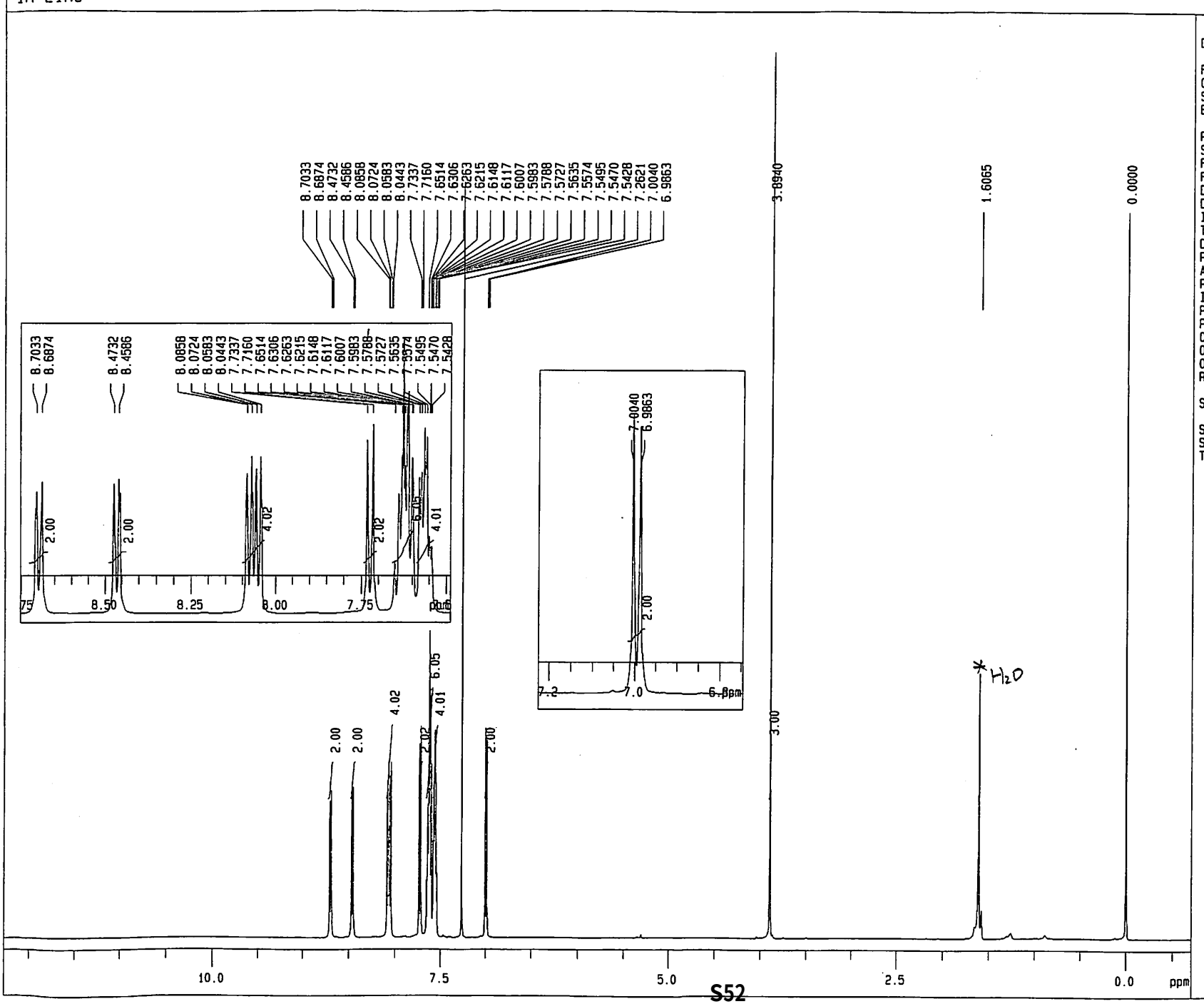
FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	180 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIMT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	129 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	18.3 C



32

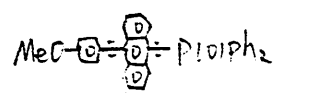
^{31}P NMR

1H Line

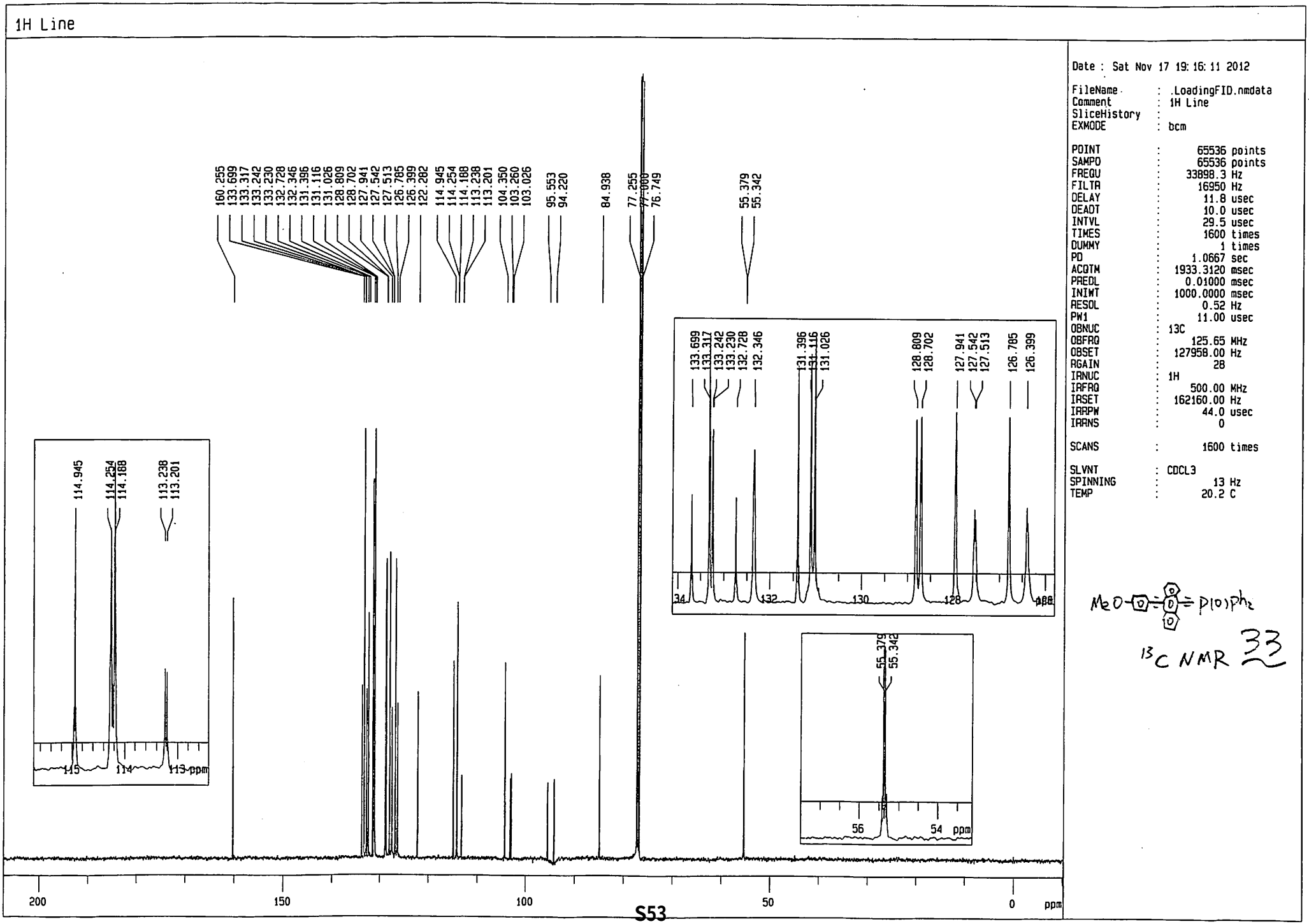


Date : Sat Nov 17 17:16:15 2012

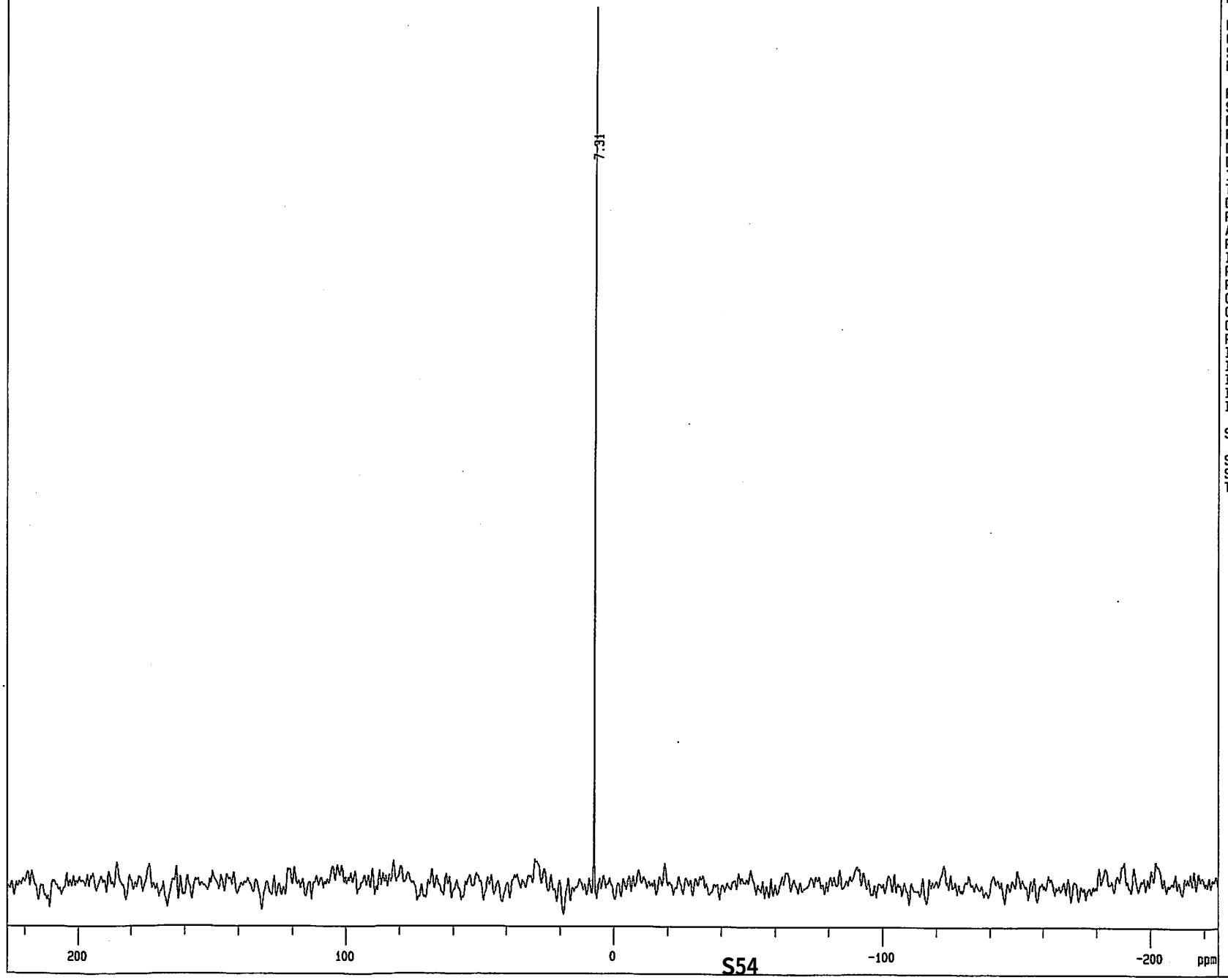
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	10000.0 Hz
FILTR	:	5000 Hz
DELAY	:	40.0 usec
DEADT	:	55.8 usec
INTVL	:	100.0 usec
TIMES	:	36 times
DUMMY	:	1 times
PD	:	3.7232 sec
ACQTM	:	3276.7998 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.31 Hz
PW1	:	8.45 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162160.00 Hz
RGAIN	:	23
SCANS	:	36 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	19.5 C



^{1}H NMR 33

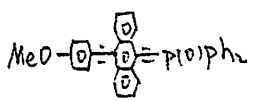

1H Line
160.255
133.699
133.317
133.242
133.230
132.728
132.346
131.396
131.116
131.026
128.809
128.702
127.941
127.542
127.513
126.785
126.399
122.282
114.945
114.254
114.188
113.238
113.201
104.350
103.260
103.026
95.553
94.220
84.938
77.255
77.000
76.749
55.379
55.342
Date : Sat Nov 17 19:16:11 2012
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 1600 times
DUMMY : 1 times
PD : 1.0667 sec
ACQTM : 1933.3120 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.52 Hz
PW1 : 11.00 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 44.0 usec
IRNS : 0
SCANS : 1600 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 20.2 C
MeO
P(O)Ph2
13C NMR 33
200
150
100
50
0
ppm

1H reso

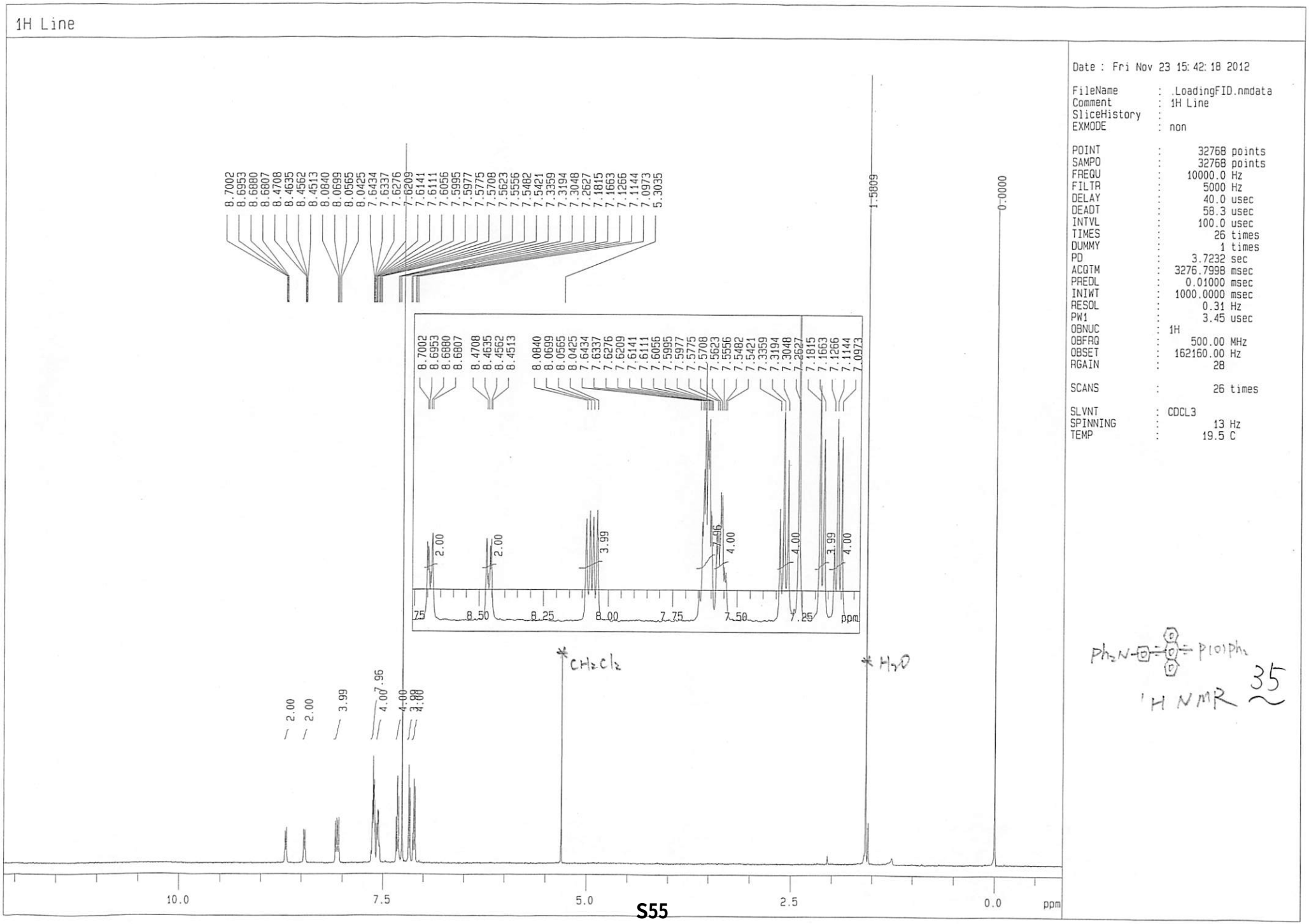


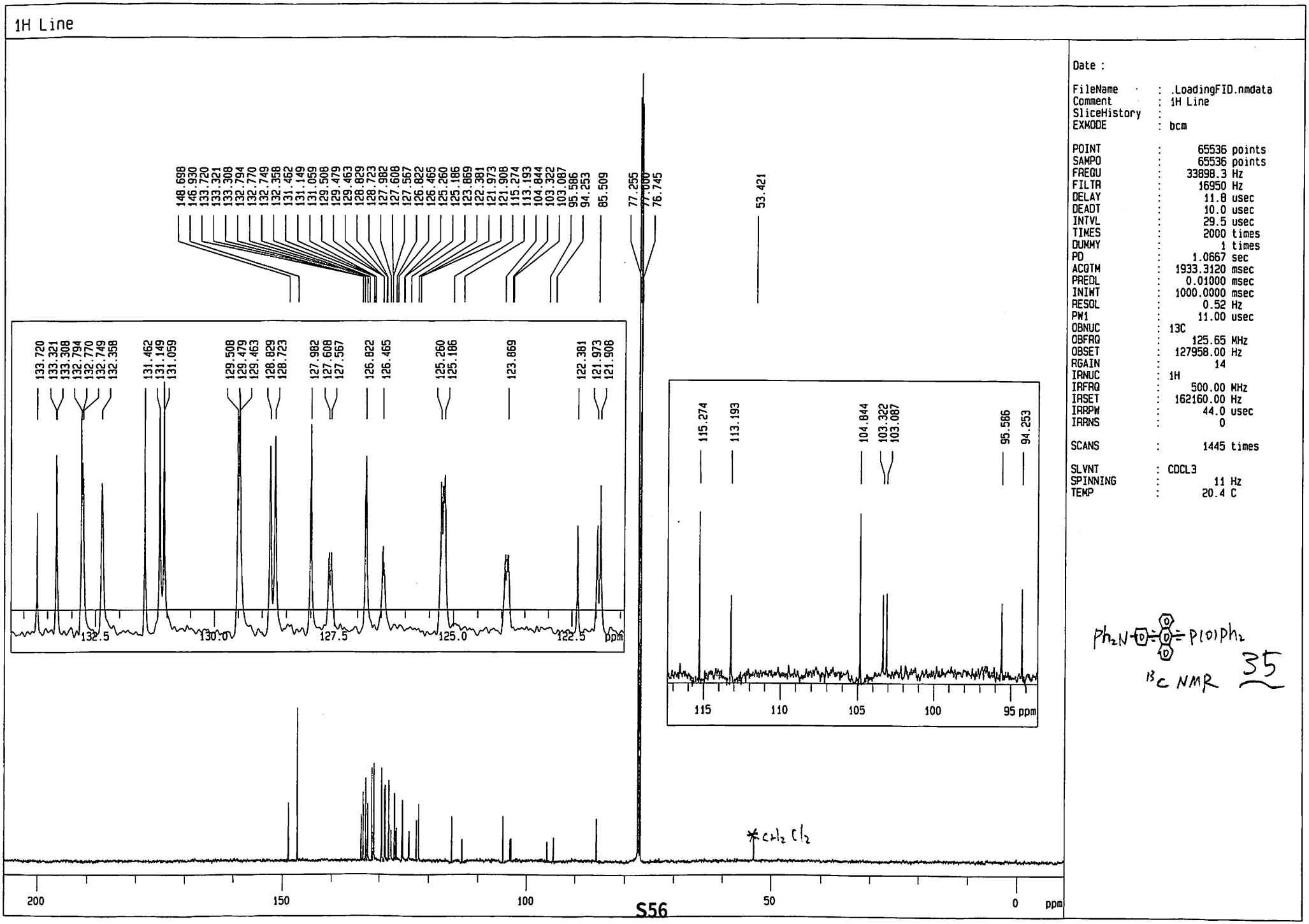
Date :

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	190 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIWT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	24
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRRPW	:	1.0 usec
IRRNS	:	0
SCANS	:	74 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	18.9 C

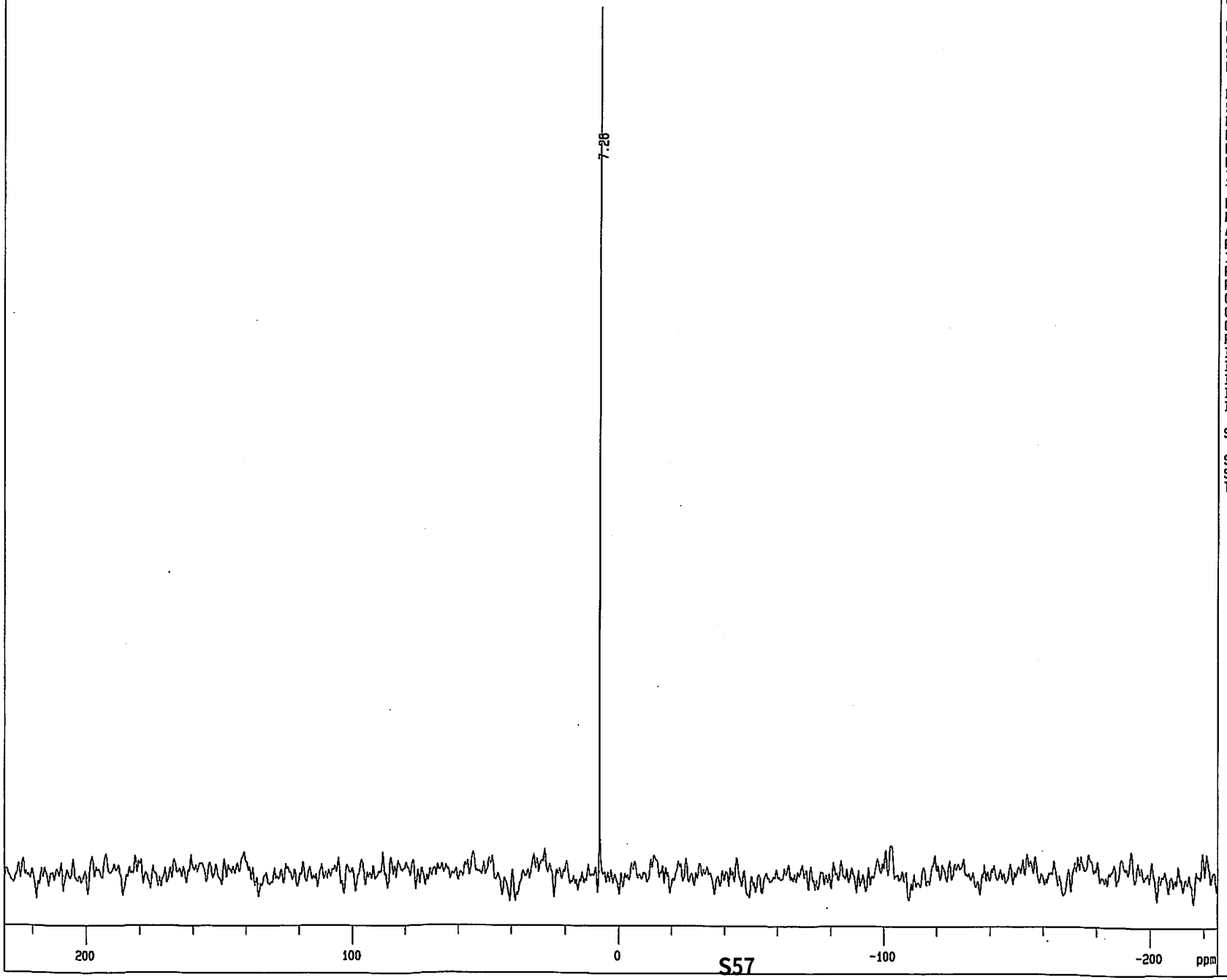


31P NMR 33


1H Line
8.7002
8.6953
8.6880
8.6807
8.4708
8.4635
8.4562
8.4513
8.0840
8.0699
8.0565
8.0425
7.6434
7.6337
7.6276
7.6209
7.6141
7.6111
7.6056
7.5995
7.5977
7.5775
7.5708
7.5623
7.5556
7.5482
7.5421
7.3359
7.3194
7.3048
7.2627
7.1815
7.1663
7.1266
7.1144
7.0973
5.3035
1.5809
0.0000
2.00
2.00
3.99
4.00
7.96
4.00
4.00
3.99
4.00
*CH2Cl2
*H2O
10.0
7.5
5.0
2.5
0.0
ppm
Date : Fri Nov 23 15:42:18 2012
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 58.3 usec
INTVL : 100.0 usec
TIMES : 26 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 3.45 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 28
SCANS : 26 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 19.5 C
Ph2N
P(O)Ph2
1H NMR
35

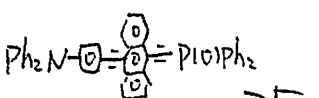

1H Line
148.698
146.930
133.720
133.321
133.308
132.794
132.770
132.749
132.358
131.462
131.149
131.059
129.508
129.479
129.463
128.829
128.723
127.982
127.608
127.567
126.822
126.465
125.260
125.186
123.869
122.381
121.973
121.908
115.274
113.193
104.844
103.322
103.087
95.586
94.253
85.509
77.255
77.000
76.745
53.421
132.5
130.0
127.5
125.0
122.5
ppm
115
110
105
100
95 ppm
200
150
100
50
0
ppm
*CH2Cl2
Date :
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 2000 times
DUMMY : 1 times
PD : 1.0667 sec
ACQTM : 1933.3120 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.52 Hz
PW1 : 11.00 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 14
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 44.0 usec
IRRNS : 0
SCANS : 1445 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 20.4 C
Ph2N P(O)Ph2
13C NMR
35

1H reso



Date :

FileName	:	.LoadingFID.nmdata
Comment	:	1H reso
SliceHistory	:	
EXMODE	:	bcm
POINT	:	65536 points
SAMPO	:	65536 points
FREQU	:	100000.0 Hz
FILTR	:	50000 Hz
DELAY	:	4.0 usec
DEADT	:	10.0 usec
INTVL	:	10.0 usec
TIMES	:	170 times
DUMMY	:	0 times
PD	:	1.0000 sec
ACQTM	:	655.3600 msec
PREDL	:	10.00000 msec
INIMT	:	1000.0000 msec
RESOL	:	1.53 Hz
PW1	:	3.50 usec
OBNUC	:	31P
OBFRQ	:	121.50 MHz
OBSET	:	153873.56 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	300.40 MHz
IRSET	:	131150.00 Hz
IRPPW	:	1.0 usec
IRRNS	:	0
SCANS	:	92 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	19.0 C



^{31}P NMR 35