Zirconium Bis(Amido) Catalysts for Asymmetric Intramolecular Alkene Hydroamination

*Donald A. Watson, Melanie Chiu and Robert G. Bergman**

Department of Chemistry, University of California, Berkeley, California 94720-1460

rbergman@berkeley.edu

Supporting Information

Index:

General Experimental Details.

"Oven-dried" glassware was dried at 180 °C for at least 12 h before use. THF, CH_2Cl_2, and PhMe were dried according to published procedures.[1] Triethylamine, pyridine, F-Ph, and Cl-Ph were distilled from CaH_2 and deoxygenated prior to use. 1,4-Dioxane was distilled from Na/benzophenone ketyl prior to use. For in situ NMR experiments, C_6D_6 and d_8-toluene were sparged with N_2 and stored over 4Å molecular sieves prior to use. Ligands were dried under high vacuum (*ca*. 20 mtorr) for at least 12 h prior to use. Hydroamination substrates were prepared according to literature procedures.[2] All other reagents and solvents were purchased at highest commercial quality and used as received. Unless otherwise noted, reactions were run using standard Shlenk line techniques. Where noted a nitrogen filled glovebox was used.

General Procedures:

General Procedure 1. Preparation of Diarylphosphinic Acids. In an oven-dried flask protected by an atmosphere of N_2, a rapidly stirred solution of aryl bromide in THF was treated dropwise with a solution of either ^{i}PrMgCl or ^{t}BuLi at –78 °C. The solution was stirred for 10 min, warmed to 25 °C and stirred for 30 min. The solution was re-cooled to –78 °C and treated with 0.5 equivalents $Cl_2P(O)NMe_2$ in one portion. The reaction mixture was warmed to 25 °C and stirred for 12 h. The flask was opened to the atmosphere and treated with 3 equivalents of 6 M aqueous HCl. The resulting suspension was rapidly stirred for 3–12 h until the reaction was completed as judged by TLC or ^{31}P NMR analysis[3] and then worked up and purified as indicated.

General Procedure 2. Preparation of Diphosphinic Amide Ligands from Chlorodiarylphosphines or Chlorodialkylphosphines. In an oven-dried 20 mL vial protected by an atmosphere of N_2 and equipped with a septum and a magnetic stirbar, R_2PCl (2.1 equiv) was added to a solution of chiral diamine (1 equiv) and Et_3N (5 equiv) in CH_2Cl_2 at 25 °C. The reaction mixture was stirred for 12 h, opened to the atmosphere, and cooled to 0 °C with an ice bath. A solution of either *t*-BuOOH (5.5 M in decane, 3-4 equiv) or H_2O_2 (30% aqueous, 3-4 equiv) was slowly added (note: oxidation is exothermic). The reaction mixture was stirred at 0 °C for 10 min, then at 25 °C for 30 min before being quenched with aqueous $Na_2S_2O_3$ (1 M, 10 equiv). The resulting biphasic solution was extracted with CH_2Cl_2 and the combined extracts were dried (Na_2SO_4) and concentrated in vacuo. The product was purified as specified below.

---

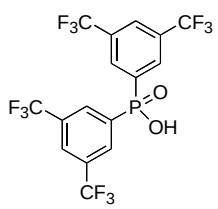
[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

[2] (a) Tamaru, Y.; Hojo, M.; Higashimura, H.; Yoshida, Z. *J. Am. Chem. Soc.* **1988**, *110*, 3994–4002. (b) Harding, K. E.; Burks, S. R. *J. Org. Chem.* **1981**, *46*, 3920–3922. (c) Hurd, C. D.; Jenkins, W. W. *J. Org. Chem.* **1957**, *22*, 1418–1423.

[3] Monitoring by ^{31}P NMR was readily accomplished by transferring a 200 µL sample of the biphasic solution into an NMR tube containing 0.6 mL $CDCl_3$ and obtaining a spectrum directly of the solution once the water layer had risen to top of the biphasic solution.

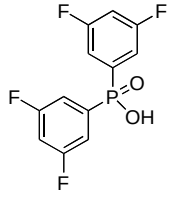
General Procedure 3. Preparation of Diphosphinic Amide Ligands from Diarylphosphinic Acids. In an oven-dried Schlenk flask protected by an atmosphere of N_2 and equipped with a reflux condenser, stirbar and nitrogen inlet, the diarylphosphinic acid was dissolved in an excess of $SOCl_2$ and then heated at reflux for 30 min. The reaction mixture was cooled to 25 °C, the reflux condenser was replaced with a greased glass stopper and the remaining $SOCl_2$ was removed in vacuo. The resulting phosphinic chloride was dried under high vacuum (*ca.* 100 mm Hg) for at least 12 h before continuing. The glass stopper was then replaced with a rubber septum and the phosphinic chloride was dissolved in CH_2Cl_2. Triethylamine (2.5 equiv) and chiral diamine (0.5 equiv) were then added and the reaction mixture was stirred for another 12 h. The flask was then opened to the atmosphere and worked up by the addition of saturated aqueous $NaHCO_3$. Extraction with CH_2Cl_2, followed by drying the combined extracts (Na_2SO_4) and concentration in vacuo provided the crude ligand, which was purified as described below.

Preparation of Diarylphosphinic Acids:



$C_{16}H_7F_{12}O_2P$
Mol. Wt.: 490.18
S1

Bis(3,5-bis(trifluoromethyl)phenyl)phosphinic Acid (**S1**): 3,5-Bis(trifluoromethyl)phenyl bromide (18.09 g, 61.7 mmol, 10.5 mL), isopropyl magnesium chloride (34.1 mL, 61.7 mmol, 1.8 M in Et_2O), *N*,*N*-dimethylphosphoramic dichloride (5.0 g, 30.9 mmol, 3.67 mL), 6 M aqueous HCl (30 mL) and THF (150 mL) were combined as described in General Procedure 1. The reaction mixture was then extracted with CH_2Cl_2, and the combined extracts were dried over Na_2SO_4, concentrated in vacuo and purified by precipitation from THF/hexane solution to give 6.50 g (43%) of **S1** as an off-white powder: IR (solid) cm^{-1} 1281, 1135, 674; ^1H NMR (400 MHz, d_6-DMSO) δ 8.39 (d, *J* = 11.7 Hz, 4 H), 8.23 (s, 4 H), 6.69 (br. s, 1 H); ^{13}C NMR (101 MHz, d_6-DMSO) δ 138.5 (d, *J* = 135.7 Hz), 132.2-132.6 (m), 130.9 (qd, *J* = 33.0, 13.2 Hz), 125.8-126.1 (m), 123.4 (q, *J* = 272.9 Hz); ^{19}F NMR (376 MHz, d_6-DMSO) δ -60.7; ^{31}P NMR (162 MHz, d_6-DMSO) δ 14.0; HRMS (ESI^+) *m/z* 575.9991 [575.9975 calcd for $C_{16}H_7F_{12}O_2P$ $(M+2Na-H+CH_3CN)^+$].[4]

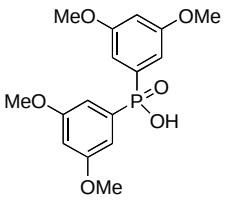


$C_{12}H_7F_4O_2P$
Mol. Wt.: 290.15
S2

Bis(3,5-difluorophenyl)phosphinic Acid (**S2**): 3,5-Difluorophenyl bromide (7.72 g, 40.0 mmol, 4.6 mL), isopropylmagnesium chloride (22.1 mL, 40 mmol, 1.8 M in Et_2O), *N*,*N*-dimethylphosphoramic dichloride (3.8 g, 30.9 mmol, 2.99 mL), 6 M aqueous HCl (20 mL) and THF (120 mL) were combined as described in General Procedure 1. The reaction mixture was extracted with CH_2Cl_2, dried over Na_2SO_4 and concentrated in vacuo. The product was purified by precipitation from CH_2Cl_2/hexane solution to give 1.35 g (10%) of **S2** as a colorless

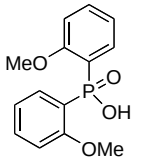
[4] Acetonitrile from electrospray mass spectrometer.

powder: IR (solid) cm^{-1} 1584, 1421, 1928, 977; ^{1}H NMR (400 MHz, d_6-DMSO) δ 8.77 (br. s, 1 H), 7.39-7.57 (m, 6 H); ^{13}C NMR (101 MHz, d_6-DMSO) δ 162.7 (ddd, *J* = 250.3, 20.5, 11.0 Hz), 139.2 (dt, *J* = 138.3, 5.9 Hz), 114.7 (ddd, *J* = 18.3, 10.2, 8.1 Hz), 107.9 (t, *J* = 24.9 Hz); ^{19}F NMR (376 MHz, d_6-DMSO) δ -107.1; ^{31}P NMR (162 MHz, d_6-DMSO) δ 16.9 (t, *J* = 6.9 Hz); HRMS (ESI^+) *m/z* 376.0103 [376.0108 calcd for $C_{12}H_7F_4O_2P$ $(M+2Na-H+CH_3CN)^+$].[4]



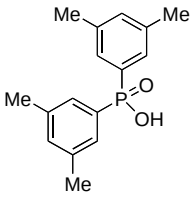
$C_{16}H_{19}O_6P$
Mol. Wt.: 338.29
S3

Bis(3,5-dimethoxyphenyl)phosphinic Acid (**S3**): 3,5-Dimethoxyphenyl bromide (4.34 g, 20 mmol), *t*-BuLi (24.2 mL, 40 mmol, 1.65 M in pentane), *N*,*N*-dimethylphosphoramic dichloride (1.61 g, 10 mmol, 1.19 mL), 6 M aqueous HCl (10 mL) and THF (100 mL) were combined as described in General Procedure 1 to give a thick, white heterogeneous suspension. The suspension was filtered, and the precipitate was washed with water (50 mL) and Et_2O (200 mL) to give 2.0 g (74%) of **S3** as a colorless powder: IR (solid) cm^{-1} 1607, 1409, 1199, 1152; ^{1}H NMR (400 MHz, d_6-DMSO) δ 6.78 (dd, *J* = 13.2, 2.2 Hz, 4 H), 6.61 (t, *J* = 2.2 Hz, 2 H), 3.72 (s, 12 H); ^{13}C NMR (101 MHz, d_6-DMSO) δ 160.8 (d, *J* = 18.3 Hz), 137.3 (d, *J* = 134.2 Hz), 108.9 (d, *J* = 10.3 Hz), 103.4 (d, *J* = 1.5 Hz), 55.8; ^{31}P NMR (162 MHz, d_6-DMSO) δ 23.0; HRMS (ESI^+) *m/z* 383.0636 [383.0636 calcd for $C_{16}H_{19}O_6P$ $(M+2Na-H)^+$].



$C_{14}H_{15}O_4P$
Mol. Wt.: 278.24
S4

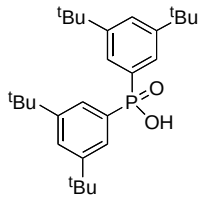
Di(*o*-methoxyphenyl)phosphinic Acid (**S4**): *O*-methoxyphenyl bromide (3.74 g, 20 mmol), *t*-BuLi (24.2 mL, 40 mmol, 1.65 M in pentane), *N*,*N*-dimethylphosphoramic dichloride (1.61 g, 10 mmol, 1.19 mL), 6 M aqueous HCl (10 mL) and THF (100 mL) were combined as described in General Procedure 1 to give a thick, white heterogenous suspension. The suspension was filtered, and the precipitate was washed with water (50 mL) and Et_2O (200 mL) to give 2.2 g (79%) of **S4** as a colorless powder: IR (solid) cm^{-1} 1590, 1479, 1275, 1441, 948; ^{1}H NMR (400 MHz, d_6-DMSO) δ 8.78 (br. s, 1 H), 7.71 (ddd, *J* = 13.7, 7.3, 1.5 Hz, 2 H), 7.43 (t, *J* = 7.3 Hz, 2 H), 6.99 (td, *J* = 7.1, 1.7 Hz, 2 H), 6.92 (dd, *J* = 7.8, 5.9 Hz, 2 H), 3.47 (s, 6 H); ^{13}C NMR (101 MHz, d_6-DMSO) δ 160.8 (d, *J* = 3.7 Hz), 133.7 (d, *J* = 6.6 Hz), 133.4 (d, *J* = 1.5 Hz), 123.7 (d, *J* = 141.6 Hz), 120.3 (d, *J* = 11.7 Hz), 112.0 (d, *J* = 7.3 Hz), 55.8; ^{31}P NMR (162 MHz, d_6-DMSO) δ 19.1; HRMS (ESI^+) *m/z* 323.0434 [323.0425 calcd for $C_{14}H_{15}O_4P$ $(M+2Na-H)^+$].



$C_{16}H_{19}O_2P$
Mol. Wt.: 274.29
S5

Bis(3,5-dimethylphenyl)phosphinic Acid (**S5**): 3,5-Dimethylphenyl bromide (10.0 g, 54 mmol), *t*-BuLi (65.4 mL, 108 mmol, 1.65 M in pentane), *N*,*N*-dimethylphosphoramic dichloride (4.32 g, 27 mmol, 3.21 mL), 6 M aqueous HCl (30 mL) and THF (200 mL) were combined as described in General Procedure 1. The reaction mixture was then extracted with CH_2Cl_2, dried over Na_2SO_4 and concentrated in vacuo. The product was purified by precipitation from CH_2Cl_2/hexane solution to give 5.65 g (76%) of **S5** as a colorless powder: IR (solid) cm^{-1} 2915, 1584, 1275, 1135, 937, 685;

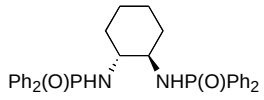
^{1}H NMR (400 MHz, CD_2Cl_2) δ 12.68 (br. s, 1 H), 7.32 (d, *J* = 12.7 Hz, 4 H), 7.10 (s, 2 H), 2.27 (s, 12 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 138.1 (d, *J* = 13.9 Hz), 133.5 (d, *J* = 2.9 Hz), 132.6 (d, *J* = 137.9 Hz), 128.5 (d, *J* = 10.3 Hz), 20.9; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 33.4; HRMS (ESI$^+$) *m/z* 319.0840 [319.0840 calcd for $C_{16}H_{19}O_2P$ (M+2Na-H)$^+$].



$C_{28}H_{43}O_2P$
Mol. Wt.: 442.61
S6

Bis(3,5-di-*t*-Bu-phenyl)phosphinic Acid (**S6**): 3,5-Di-*t*-Bu-phenyl bromide (7.56 g, 28 mmol), *t*-BuLi (39.2 mL, 56 mmol, 1.43 M in pentane), *N*,*N*-dimethylphosphoramic dichloride (2.67 g, 14 mmol, 1.27 mL), 6 M aqueous HCl (15 mL) and THF (100 mL) were combined as described in General Procedure 1. The reaction mixture was then extracted with CH_2Cl_2, dried over Na_2SO_4 and concentrated in vacuo. The product was purified by flash chromatography (Et_2O/hex) to give 5.44 g (87%) of **S6** as an off-white foamy solid: IR (solid) cm^{-1} 2961, 2856, 1590, 1357, 1141, 960; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.90 (br. s, 1 H), 7.67 (dd, *J* = 13.4, 1.8 Hz, 4 H), 7.61 (s, 2 H), 1.31 (s, 36 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 151.0 (d, *J* = 13.2 Hz), 131.8 (d, *J* = 139.0 Hz), 126.3 (d, *J* = 2.2 Hz), 125.1 (d, *J* = 11.7 Hz), 34.9, 31.1; ^{31}P NMR (162 MHz, d_6-DMSO) δ 36.5; HRMS (ESI$^+$) *m/z* 487.2708 [487.2718 calcd for $C_{28}H_{43}O_2P$ (M+2Na-H)$^+$].

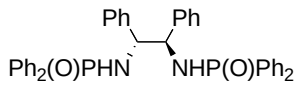
Preparation of Diphosphinic Amide Ligands 3c, 9-12 and 13a-13i:



$C_{30}H_{32}N_2O_2P_2$
Mol. Wt.: 514.53

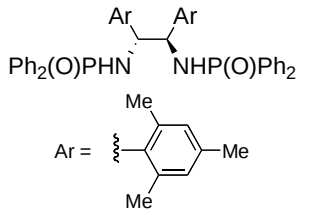
(*R*,*R*)-Diphosphinic Amide Ligand 3c: In an oven-dried 20 mL vial protected by an atmosphere of N_2 and equipped with a septum and a magnetic stirbar, diphenylphosphinic chloride (2.07 g, 3.05 mL, 8.75 mmol) was added dropwise to a solution of (*R*,*R*)-1,2-cyclohexanediamine (500 mg, 4.78 mmol) and Et_3N (2.21 g, 3.05 mL, 21.9 mmol) in CH_2Cl_2 (10 mL) at 0 °C. The reaction mixture was warmed to 25 °C and stirred for 2 h, during which time colorless precipitates were observed. The reaction mixture was quenched by the careful addition of saturated aqueous $NaHCO_3$ and extracted with CH_2Cl_2. The combined extracts were dried over Na_2SO_4 and concentrated in vacuo to give a yellow foam. The product was purified by precipitation from EtOAc/hex solution to give 1.66 g (74%) of **3c** as an extremely fluffy white solid: IR (solid) cm^{-1} 3181, 2922, 1434, 1185; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.99 (ddd, *J* = 11.9, 6.8, 1.3 Hz, 4 H), 7.81-7.88 (m, *J* = 11.6, 7.1 Hz, 4 H), 7.46-7.59 (m, 8 H), 7.39 (td, *J* = 7.6, 3.0 Hz, 4 H), 4.42 (t, *J* = 7.1 Hz, 2 H), 2.83-2.97 (m, 2 H), 2.07 (d, *J* = 13.4 Hz, 2 H), 1.58 (d, *J* = 7.8 Hz, 2 H), 1.32 (q, *J* = 10.4 Hz, 2 H), 1.12 (t, *J* = 9.9 Hz, 2 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 133.66 (d, *J* = 122.9 Hz), 133.62 (d, *J* = 129.2 Hz), 131.58 (d, *J* = 2.9 Hz), 131.55 (d, *J* = 2.9 Hz), 131.51 (d, *J* = 9.5 Hz), 132.4 (d, *J* = 9.5 Hz), 128.34 (d, *J* = 12.4 Hz), 128.31 (d, *J* = 12.4 Hz), 56.1 , 36.0 (d, *J* = 4.4 Hz), 25.0; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 24.3; HRMS (FAB$^+$) *m/z* 515.2010 [515.2017 calcd for $C_{30}H_{32}N_2O_2P_2$ (M+H)$^+$].

.



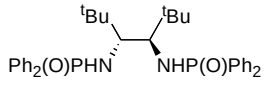
$C_{38}H_{34}N_2O_2P_2$
Mol. Wt.: 612.64

(*R,R*)-Diphosphinic Amide Ligand 9: In an oven-dried 20 mL vial protected by an atmosphere of N_2 and equipped with a septum and a magnetic stirbar, diphenylphosphinic chloride (111 mg, 90 µL, 470 µmol) was added dropwise to a solution of (*R,R)*-1,2-diphenylethylenediamine (50 mg, 235 µmol) and Et_3N (120 mg, 160 µL, 1.17 mmol) in CH_2Cl_2 (5 mL) at 25 °C. The reaction mixture was stirred for 12 h, during which time colorless precipitates were observed. The reaction mixture was quenched by the careful addition of saturated aqueous $NaHCO_3$ and extracted with CH_2Cl_2. The combined extracts were dried over Na_2SO_4 and concentrated to give a yellow foam. The product was purified by precipitation from CH_2Cl_2/hexanes solution to give 140 mg (97%) of **9** as a white powder: IR (solid) cm^{-1} 3210, 3056, 1434, 1188, 1108; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.77 (dd, *J* = 11.3, 7.9 Hz, 4 H), 7.61 (dd, *J* = 11.3, 7.9 Hz, 4 H), 7.53 (t, *J* = 7.0 Hz, 2 H), 7.33-7.45 (m, 8 H), 7.18-7.28 (m, 4 H), 7.07 (d, *J* = 7.3 Hz, 4 H), 6.88 (d, *J* = 6.4 Hz, 4 H), 6.19 (br. s, 2 H), 4.26-4.37 (m, 2 H); ^{13}C NMR (126 MHz, CD_2Cl_2) δ 132.70 (d, *J* = 126.2 Hz), 132.69 (d, *J* = 9.9 Hz), 131.9 (d, *J* = 133.8 Hz), 131.7 (d, *J* = 2.4 Hz), 131.53 (d, *J* = 9.4 Hz), 131.48 (d, *J* = 2.4 Hz), 128.2 (d, *J* = 12.7 Hz), 128.07, 128.05 (d, *J* = 13.2 Hz), 127.8, 127.5, 126, 61.7; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 27.8; HRMS (FAB^+) *m/z* 613.2188 [613.2173 calcd for $C_{38}H_{34}N_2O_2P_2$ $(M+H)^+$].



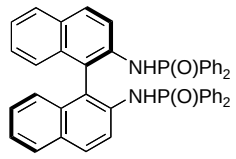
$C_{44}H_{46}N_2O_2P_2$
Mol. Wt.: 696.80

(*R,R*)-Diphosphinic Amide Ligand 10: (*R,R*)-1,2-dimesitylethylenediamine (75 mg, 252 µmol), chlorodiphenylphosphine (117 mg, 98 µL, 531 µmol), Et_3N (128 mg, 176 µL, 1.26 mmol), aqueous H_2O_2 (30%, 171 µL, 1.51 mmol) and CH_2Cl_2 (4 mL) were combined according to General Procedure 2 to produce a crude oil. The product was isolated by flash chromatography (65:35 Et_2O/hexanes) to provide 152 mg (87%) of **10** as a colorless solid: IR (solid) cm^{-1} 3199, 3064, 2965, 1617, 1434, 1185; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.85 (dd, *J* = 11.3, 7.9 Hz, 4 H), 7.72 (dd, *J* = 11.6, 7.9 Hz, 4 H), 7.55 (t, *J* = 7.3 Hz, 2 H), 7.39-7.50 (m, 10 H), 7.28 (td, *J* = 7.6, 3.1 Hz, 4 H), 6.78 (s, 2 H), 6.30 (s, 2 H), 5.91 (d, *J* = 9.2 Hz, 2 H), 4.80-4.92 (m, 2 H), 2.68 (s, 6 H), 2.14 (s, 6 H), 0.73 (s, 6 H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 136.9, 136.6, 134.2 (d, *J* = 6.6 Hz), 133.2 (d, *J* =125.7 Hz), 132.9 (d, *J* = 9.4 Hz), 132.3 (d, *J* = 133.9 Hz), 131.7 (d, *J* = 7.5 Hz), 131.6 (d, *J* = 7.5 Hz), 131.5 (d, *J* = 9.4 Hz), 130.8, 128.32, 128.31 (d, *J* = 12.2 Hz), 128.2 (d, *J* = 12.3 Hz), 53.7 (d, *J* = 1.9 Hz), 21.4, 20.3, 18.5; ^{31}P NMR (202 MHz, CD_2Cl_2) δ 26.7; HRMS (FAB^+) *m/z* 697.3123 [697.3113 calcd for $C_{62}H_{96}N_2O_2P_2$ $(M+H^+)$].



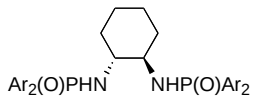
$C_{34}H_{42}N_2O_2P_2$
Mol. Wt.: 572.66

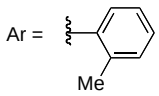
(*R,R*)-Diphosphinic Amide Ligand 11: (*R,R*)-1,2-di-*t*-Bu-ethylenediamine[5] (100 mg, 577 µmol), chlorodiphenylphosphine (380 mg, 320 µL, 1.73 mmol), Et_3N (290 mg, 400 µL, 2.88 mmol), *t*-BuOOH (5.5 M in decane, 420 µL, 2.30 mmol) and CH_2Cl_2 (6 mL) were combined according to General Procedure 2 to produce a crude oil. The product was isolated by flash chromatography (40:60 EtOAc/hexanes) then crystallized from hexanes to provide 90 mg (27%) of **11** as colorless needles: IR (solid) cm^{-1} 3350, 3280, 2958, 1442, 1108; ^1H NMR (400 MHz, CD_2Cl_2) δ 7.89 (dd, *J* = 11.6, 10.4 Hz, 4H), 7.84 (dd, *J* = 12.0, 10.4 Hz, 4H), 7.39–7.52 (m, 8H), 7.26 (t, *J* = 7.6 Hz, 4H), 4.63 (dd, *J* = 12.0, 5.2, Hz, 2H), 3.06 (t, *J* = 9.2 Hz, 2H), 0.88 (s, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 135.7 (d, *J* = 127.0 Hz), 132.5 (d, *J* = 129.0 Hz), 132.4 (d, *J* = 10 Hz), 131.5, 131.3 (d, *J* = 9.0 Hz), 131.2, 128.3 (d, *J* = 13.0 Hz), 128.2 (d, *J* = 12 Hz), 58.6 (d, *J* = 4.0 Hz), 36.8 (d, *J* = 9.0 Hz), 28.1; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 21.2; HRMS (FAB^+) *m/z* 573.2801 [573.2800 calcd for $C_{34}H_{42}N_2O_2P_2$ (M^+)].



$C_{44}H_{34}N_2O_2P_2$
Mol. Wt.: 684.70

(*R,R*)-Diphosphinic Amide Ligand 12: (*R*)-BINAM (56 mg, 196 µmol), chlorodiphenylphosphine (91 mg, 77 µL, 413 µmol), Et_3N (100 mg, 137 µL, 984 µmol), *t*-BuOOH (5.5 M in decane, 200 µL) and CH_2Cl_2 (4 mL) were combined according to General Procedure 2 to produce a crude oil. The product was isolated by flash chromatography (50:50 Et_2O/hexanes) and then crystallized by slow diffusion of hexanes into a toluene solution to provide 94 mg (70%) of **12** as colorless prisms: IR (solid) cm^{-1} 3354, 3058, 1623, 1583, 1211; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.90 (d, *J* = 6.1 Hz, 2 H), 7.82 (d, *J* = 9.2 Hz, 2 H), 7.62-7.72 (m, 6 H), 7.31-7.50 (m, 18 H), 7.29 (d, *J* = 7.6 Hz, 2 H), 7.12-7.21 (m, 4 H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 138.0, 132.7, 132.2 (d, *J* = 129.0 Hz), 132.1 (d, *J* = 128.0 Hz), 132.0 (d, *J* = 2.8 Hz), 131.9 (d, *J* = 2.4 Hz), 131.6 (d, *J* = 9.9 Hz), 131.0 (d, *J* = 9.9 Hz), 130.0, 129.8, 128.7 (d, *J* = 12.7 Hz), 128.6 (d, *J* = 12.5 Hz), 128.5, 127.5, 124.5, 124.4, 119.0 (d, *J* = 3.8 Hz), 116.9 (d, *J* = 8.0 Hz); ^{31}P NMR (162 MHz, CD_2Cl_2) δ 15.8; HRMS (FAB^+) *m/z* 685.2171 [685.2174 calcd for $C_{62}H_{96}N_2O_2P_2$ ($M+H^+$)].



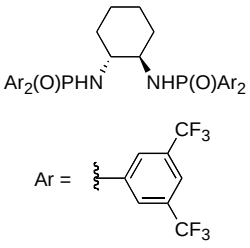


$C_{34}H_{40}N_2O_2P_2$
Mol. Wt.: 570.64

(*R,R*)-Diphosphinic Amide Ligand 13a: (*R,R*)-1,2-cyclohexanediamine (50 mg, 437 µmol), chlorodi(*o*-tolyl) phosphine (228 mg, 919 µmol), Et_3N (443 mg, 610 µL, 4.37 mmol), *t*-BuOOH (5.5 M in decane, 310 µL, 1.78 mmol) and CH_2Cl_2 (10 mL) were combined according to General Procedure 2 to produce a crude oil. The product was purified by precipitation from CH_2Cl_2/hexane solution to provide 92 mg (37%) of **13a** as a colorless powder: IR (solid) cm^{-1} 3467, 3178, 2942, 2853, 1560, 1455, 1163; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.98 (dd, *J* = 13.4, 7.6 Hz, 2 H), 7.59 (dd, *J* = 12.5, 7.9 Hz, 2 H), 7.35-7.44 (m, 4 H), 7.28-7.34 (m, 2 H), 7.08-7.22 (m, 6 H), 4.49-4.56 (m, 2 H), 3.13-3.23 (m, 2 H), 2.32 (s, 6 H), 2.24 (s, 6 H), 2.19 (d, *J* = 12.8 Hz, 2 H), 1.55-1.68 (m, 2 H), 1.40

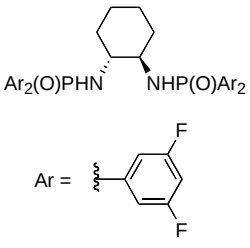
[5] Roland, S.; Mangeney, P.; Alexakis, A. *Synthesis* **1999**, 228-230.

(q, *J* = 10.1 Hz, 2 H), 1.21 (t, *J* = 9.8 Hz, 2 H); ^{13}C NMR (125 MHz, CD_2Cl_2, one aromatic signal partially obscured) δ 142.0 (d, *J* = 10.8 Hz), 141.4 (d, *J* = 9.9 Hz), 133.2 (d, *J* = 9.9 Hz), 132.7 (d, *J* = 10.4 Hz), 131.5 (d, *J* = 118.2 Hz), 131.43, 131.42, 131.41 (d, *J* = 7.6 Hz), 131.3 (d, *J* = 6.1 Hz), 125.2 (d, *J* = 12.2 Hz), 125.1 (d, *J* = 12.2 Hz), 56.4 (d, *J* = 3.8 Hz), 35.7 (d, *J* = 2.8 Hz), 25.1, 21.2 (d, *J* = 3.8 Hz), 21.1 (d, *J* = 3.8 Hz); ^{31}P NMR (162 MHz, CD_2Cl_2) δ 26.7; HMRS (FAB$^+$) *m/z* 571.2652 [571.2643 calcd for $C_{34}H_{40}N_2O_2P_2$ (M+H$^+$)].



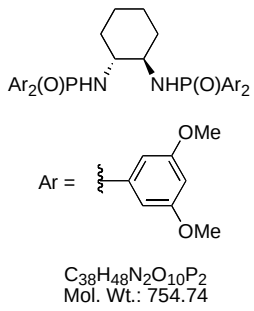
$C_{38}H_{24}F_{24}N_2O_2P_2$
Mol. Wt.: 1058.52

(*R,R*)-Diphosphinic Amide Ligand 13b: Bis(bis(3,5-trifluoromethyl)phenyl)phosphinic acid (1.0 g, 2.04 mmol), $SOCl_2$ (10 mL), (*R,R*)-1,2-cyclohexanediamine (116 mg, 1.02 mmol), Et_3N (516 mg, 710 µL, 5.10 mmol), and CH_2Cl_2 (10 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was dissolved in Et_2O and filtered through a short plug of silica gel. The silica was washed with Et_2O, and the resulting solution was concentrated. The product was further purified by precipitation from Et_2O/hexanes solution to provide 618 mg (57%) of **13b** as a colorless powder: IR (solid) cm^{-1} 3174, 2932, 1621, 1456, 1360, 1277, 1119; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.45 (d, *J* = 12.0 Hz, 4 H), 8.32 (d, *J* = 11.7 Hz, 4 H), 8.08 (d, *J* = 14.2 Hz, 4 H), 4.80-4.98 (m, 2 H), 2.80-2.98 (m, 2 H), 1.92 (d, *J* = 13.0 Hz, 2 H), 1.60 (d, *J* = 8.3 Hz, 2 H), 1.33-1.47 (m, 2 H), 1.05 (t, *J* = 9.5 Hz, 2 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 135.1 (d, *J* = 135.0 Hz), 134.7 (d, *J* = 128.2 Hz), 132.8-133.1 (m), 132.3 (t, *J* = 12.5 Hz), 131.9 (t, *J* = 12.5 Hz), 131.5-131.8 (m), 126.1-126.5 (m), 122.9 (q, *J* = 272.2 Hz), 122.7 (q, *J* = 272.2 Hz), 56.6, 35.6 (d, *J* = 5.9 Hz), 24.7; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 20.2; ^{19}F NMR (376 MHz, CD_2Cl_2) δ -62.57, -62.65; LRMS (FAB$^+$) *m/z* 1059 (M+H$^+$).



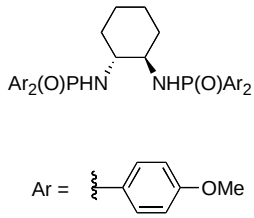
$C_{30}H_{24}F_8N_2O_2P_2$
Mol. Wt.: 658.46

(*R,R*)-Diphosphinic Amide Ligand 13c: Bis(3,5-difluorophenyl)phosphinic acid (300 mg, 1.03 mmol), $SOCl_2$ (5 mL), (*R,R*)-1,2-cyclohexanediamine (59 mg, 516 µmol), Et_3N (253 mg, 350 µL, 2.5 mmol), and CH_2Cl_2 (5 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was purified by flash chromatography (90:10 hexanes/Et_2O) to provide 165 mg (49%) of **13c** as a colorless solid: IR (solid) cm^{-1} 3150, 2907, 2852, 1613, 1591, 1420, 1298, 1999, 1121; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.51 (ddt, *J* = 12.9, 5.6, 2.3 Hz, 4H), 7.02 (ddt, *J* = 12.6, 5.6, 2.3 Hz, 4H), 6.96-7.09 (m, 4 H), 4.79-4.87 (m, 2 H), 2.91-3.01 (m, 1 H), 2.02 (d, *J* = 14.4 Hz, 2 H), 1.59-1.68 (m, 2 H), 1.38 (q, *J* = 9.3 Hz, 6 H), 1.15 (t, *J* = 10.1 Hz, 2 H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 164.2 (dt, *J* = 24.9, 11.7 Hz), 161.7 (ddd, *J* = 21.2, 13.2, 11.0 Hz), 136.8 (dt, *J* = 133.9, 7.3 Hz), 136.2 (dt, *J* = 126.8, 7.3 Hz), 115.4 (ddd, *J* = 18.3, 10.2 Hz), 114.4 (dt, *J* = 18.3, 10.3 Hz), 107.5 (d, *J* = 24.9 Hz), 107.8 (d, *J* = 25.6 Hz), 56.2, 35.6 (d, *J* = 4.4 Hz), 24.9; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 21.0 (td, *J* = 13.8, 5.9 Hz); ^{19}F NMR (376 MHz, CD_2Cl_2) δ -107.04 (q, *J* = 8.2 Hz), -107.26 (q, *J* = 8.3 Hz); HRMS (FAB$^+$) *m/z* 659.1255 [659.1264 calcd for $C_{30}H_{24}F_8N_2O_2P_2$ (M+H$^+$)].



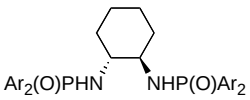
$C_{38}H_{48}N_2O_{10}P_2$
Mol. Wt.: 754.74

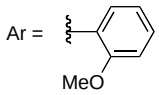
(*R*,*R*)-Diphosphinic Amide Ligand 13d: Bis(3,5-dimethoxyphenyl)phosphinic acid (676 mg, 2.0 mmol), $SOCl_2$ (10 mL), (*R*,*R*)-1,2-cyclohexanediamine (114 mg, 1.0 mmol), Et_3N (506 mg, 700 µL, 5.0 mmol), and CH_2Cl_2 (10 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was purified by flash chromatography (EtOAc) to provide 660 mg (89%) of **13d** as a tan foamy solid: IR (film) cm^{-1} 3174, 2930, 2833, 1587, 1447, 1423, 1151; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.15 (d, *J* = 13.4 Hz, 4 H), 7.02 (d, *J* = 12.8 Hz, 4 H), 6.61 (s, 2 H), 6.55 (s, 2 H), 4.57-4.67 (m, 2 H), 3.84 (s, 12 H), 3.75 (s, 12 H), 2.89-3.03 (m, 2 H), 2.11 (d, *J* = 12.5 Hz, 2 H), 1.52-1.63 (m, 2 H), 1.35 (q, *J* = 9.2 Hz, 2 H), 1.13 (t, *J* = 8.9 Hz, 2 H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 160.8 (d, *J* = 18.4 Hz), 135.6 (d, *J* = 130.5 Hz), 135.5 (d, *J* = 123.9 Hz), 109.9 (d, *J* = 10.4 Hz), 109.0 (d, *J* = 10.8 Hz), 103.8, 103.4, 56.2, 55.4, 35.7 (d, *J* = 3.8 Hz), 25.0; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 24.9; HRMS (FAB^+) *m/z* 755.2869 [755.2862 calcd for $C_{38}H_{48}N_2O_{10}P_2$ ($M+H^+$)].



$C_{34}H_{40}N_2O_6P_2$
Mol. Wt.: 634.64

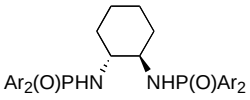
(*R*,*R*)-Diphosphinic Amide Ligand 13e: Using a modification of General Procedure 3, di(*p*-methoxyphenyl)phosphinic acid (2.5 g, 8.9 mmol) was heated at reflux in $SOCl_2$ (15 mL) for 1 h. After concentration, the resulting oil was distilled bulb-to-bulb (300 °C, 0.4 mmHg) to provide *ca*. 2 g of the phosphinic chloride as a thick yellow oil, which was used without further purfication. A portion of the phosphinic chloride (1.10 g, 3.70 mmol) was transferred to an oven-dried 100 mL round bottom flask via pipet and dissolved in CH_2Cl_2 (15 mL). Triethylamine (750 mg, 1.03 mL, 7.40 mmol) and (*R*,*R*)-1,2-cyclohexanediamine (212 mg, 1.89 mmol) were then added sequentially, and the reaction mixture was stirred for 12 h. Saturated aqueous $NaHCO_3$ (10 mL) was added to quench the reaction. Extraction with CH_2Cl_2, followed by drying the combined extracts over Na_2SO_4 and concentration, gave an oily solid. The product was purified by precipitation from CH_2Cl_2/Et_2O solution to provide 1.05 g (89%) of **13e** as a fluffy, colorless solid: IR (solid) cm^{-1} 3192. 2921, 2844, 1595, 1500, 1159, 1122; ^1H NMR (500 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 11.3, 8.9 Hz, 4 H), 7.74 (dd, *J* = 11.0, 8.9 Hz, 4 H), 6.98 (d, *J* = 6.4 Hz, 4 H), 6.87 (d, *J* = 6.7 Hz, 4 H), 4.15-4.28 (m, 2 H), 3.85 (s, 6 H), 3.81 (s, 6 H), 2.71-2.87 (m, 2 H), 2.02 (d, *J* = 12.5 Hz, 2 H), 1.50-1.59 (m, 2 H), 1.21-1.31 (m, 2 H), 1.09 (t, *J* = 9.5 Hz, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.2 (d, *J* = 2.4 Hz), 162.1 (d, *J* = 2.8 Hz), 134.1 (d, *J* = 10.8 Hz), 133.3 (d, *J* = 10.8 Hz), 125.4 (d, *J* = 131.4 Hz), 125.0 (d, *J* = 135.0 Hz), 113.7 (d, *J* = 8.9 Hz), 113.6 (d, *J* = 8.5 Hz), 55.9, 55.3, 55.2, 36.0 (d, *J* = 3.3 Hz), 25.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ 24.4; HRMS (FAB^+) *m/z* 635.2441 [635.2440 calcd for $C_{34}H_{40}N_2O_6P_2$ ($M+H^+$)].

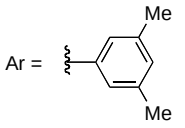




$C_{34}H_{40}N_2O_6P_2$
Mol. Wt.: 634.64

(*R,R*)-Diphosphinic Amide Ligand 13f: Di(*o*-methoxyphenyl)phosphinic acid (556 mg, 2.0 mmol), $SOCl_2$ (10 mL), (*R,R*)-1,2-cyclohexanediamine (114 mg, 1.0 mmol), Et_3N (506 mg, 700 µL, 5.0 mmol), and CH_2Cl_2 (10 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was purified by flash chromatography, first with CH_2CH_2/MeOH, then with acetone/EtOAc to provide 121 mg (19%) of **13f** as a tan foamy solid: IR (film) cm^{-1} 3367, 2939, 2853, 1591, 1479, 1279, 1243; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.87 (dd, *J* = 13.7, 7.3 Hz, 2 H), 7.73-7.80 (m, *J* = 14.0, 7.6 Hz, 2 H), 7.42 (t, *J* = 7.3 Hz, 2 H), 7.37 (t, *J* = 7.6 Hz, 2 H), 7.03 (t, *J* = 7.0 Hz, 2 H), 6.79-6.88 (m, 4 H), 6.74 (t, *J* = 7.3 Hz, 2 H), 4.04-4.19 (m, 2 H), 3.61 (s, 6 H), 3.48 (s, 6 H), 3.15-3.27 (m, 2 H), 2.03 (d, *J* = 9.8 Hz, 2 H), 1.50-1.62 (m, 2 H), 1.15-1.33 (m, 4 H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 160.2 (d, *J* = 3.8 Hz), 160.0 (d, *J* = 3.8 Hz), 133.6 (d, *J* = 7.1 Hz), 133.4 (d, *J* = 7.1 Hz), 132.5, 125.1 (d, *J* = 124.8 Hz), 123.3 (d, *J* = 125.3 Hz), 120.2 (d, *J* = 12.2 Hz), 120.1 (d, *J* = 11.9 Hz), 110.9 (d, *J* = 2.4 Hz), 110.9 (d, *J* = 2.4 Hz), 55.3, 55.3, 55.2, 35.7, 25.1; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 21.2; HRMS (FAB^+) *m/z* 635.2447 [635.2440 calcd for $C_{34}H_{40}N_2O_6P_2$ ($M+H^+$)].

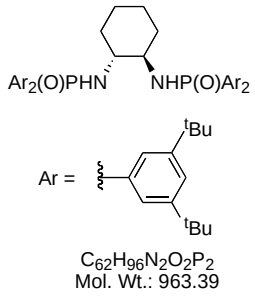




$C_{38}H_{48}N_2O_2P_2$
Mol. Wt.: 626.75

(*R,R*)-Diphosphinic Amide Ligand 13g. (This ligand has been prepared in two ways. Both procedures are presented.)

Method A: (*R,R*)-1,2-cyclohexanediamine (706 mg, 6.18 mmol), chlorodi(3,5-dimethylphenyl)phosphine (3.51 g, 12.9 mmol), Et_3N (6.26 g, 8.62 mL, 61.8 mmol), *t*-BuOOH (5.5 M in decane, 4.5 mL, 24.7 mmol) and CH_2Cl_2 (100 mL) were combined according to General Procedure 2 to produce a crude solid. The product was purified by flash chromatography (60:40 Et_2O/CH_2Cl_2) to provide 1.74 g (45%) of **13g** as an off-white, foamy solid. **Method B:** Di(3,5-dimethylphenyl)phosphinic acid (3.0 g, 10.9 mmol), $SOCl_2$ (20 mL), (*R,R*)-1,2-cyclohexanediamine (624 mg, 5.46 mmol), Et_3N (2.76 g, 3.8 mL, 27.3 mmol), and CH_2Cl_2 (50 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was purified by flash chromatography (60:40 Et_2O/CH_2Cl_2) to provide 2.73 g (80%) of **13g** as a colorless solid: IR (solid) cm^{-1} 3189, 2925, 2859, 1603, 1448, 1189; ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 12.4 Hz, 4 H), 7.47 (d, *J* = 12.1 Hz, 4 H), 7.15 (s, 2 H), 7.10 (s, 2 H), 4.31 (br. s, 2 H), 2.93-3.12 (m, 2 H), 2.38 (s, 12 H), 2.28 (s, 12 H), 2.04 (d, *J* = 12.6 Hz, 2 H), 1.57 (d, *J* = 7.8 Hz, 2 H), 1.24-1.37 (m, 2 H), 1.12 (t, *J* = 9.6 Hz, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.9 (d, *J* = 13.2 Hz), 137.8 (d, *J* = 13.4 Hz), 133.4 (d, *J* = 2.4 Hz), 133.3 (d, *J* = 2.8 Hz), 133.0 (d, *J* = 128.1 Hz), 132.7 (d, *J* = 126.0 Hz), 130.1 (d, *J* = 9.9 Hz), 129.0 (d, *J* = 9.9 Hz), 55.8 (d, *J* = 1.4 Hz), 35.8 (d, *J* = 2.4 Hz), 25.0, 21.3, 21.2; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 26.4; HRMS (FAB^+) *m/z* 627.3266 [627.3269 calcd for $C_{38}H_{48}N_2O_2P_2$ ($M+H^+$)].



$C_{62}H_{96}N_2O_2P_2$
Mol. Wt.: 963.39

(*R,R*)-Diphosphinic Amide Ligand 13h. Bis(3,5-di-*t*-Bu-phenyl)phosphinic acid (5.44 g, 12.3 mmol), $SOCl_2$ (20 mL), (*R,R*)-1,2-cyclohexanediamine (701 mg, 6.13 mmol), Et_3N (3.10 g, 4.3 mL, 30.7 mmol), and CH_2Cl_2 (50 mL) were combined according to General Procedure 3 to provide a light brown solid. The product was purified by flash chromatography (50:50 Et_2O/hexanes) to provide 4.90 g (83%) of **13h** as an off-white foamy solid: IR (solid) cm^{-1} 3189, 2958, 2859, 1602, 1247, 1178, 1119; ^1H NMR (500 MHz, CD_2Cl_2, one aliphatic signal not observed) δ 7.86 (d, *J* = 12.5 Hz, 4 H), 7.77 (d, *J* = 12.2 Hz, 4 H), 7.56 (d, *J* = 10.0 Hz, 4 H), 2.87 (s, 2 H), 2.14 (d, *J* = 12.5 Hz, 2H), 1.55 (m, 2 H), 1.36 (s, 36 H), 1.29 (s, 36 H), 1.08 (t, *J* = 10.0 Hz, 2 H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ 150.8 (d, *J* = 12.0 Hz), 150.7 (d, *J* = 12.0 Hz), 133.6 (d, *J* = 32.5 Hz), 132.6 (d, *J* = 37.5 Hz) 126.4 (d, *J* = 9.9 Hz), 125.6 (d, *J* = 10.4 Hz), 125.4 (d, *J* = 2.4 Hz), 56.5 (d, *J* = 2.8 Hz), 35.8 (d, *J* = 3.3 Hz), 34.9 (d, *J* = 7.5 Hz), 31.1 (d, *J* = 3.3 Hz), 25.0; ^{31}P NMR (162 MHz, CD_2Cl_2) δ 25.5; HRMS (FAB^+) *m/z* 963.7011 [963.7025 calcd for $C_{62}H_{96}N_2O_2P_2$ ($M+H^+$)].

Ligand Screening Experiments (Tables 1 and 2):

In a glovebox, ligand (22 μmol, solid) and a solution of $M(NMe_2)_4$ (20 μmol, 500 μL, 40.0 mM in toluene) were combined in an oven-dried 4 mL (1 dram) vial equipped with an oven-dried stirbar. The vial was sealed[6] and heated[7] for 15 min. The vial was cooled to 25 °C and substrate **1** (15 μL, 11.4 mg, 100 μmol) was added. The vial was resealed and heated for the specified time. The vial was cooled to 25 °C, removed from the glovebox, and opened to the atmosphere. While rapidly stirring the reaction mixture, trifluoroacetic anhydride (50 μL) was added, and the resulting solution was stirred for 5 min. Saturated aqueous Na_2CO_3 (200 μL) was then added, and the resulting suspension was stirred for 1 min. This suspension was then transferred to a 4 mL solid phase extraction (SPE) column (containing approx. 1 g anhydrous $MgSO_4$ atop approx. 0.25 g silica gel)[8] via pipet,

[6] Vials were sealed using specific caps designed to withstand elevated pressure (Kontes Mini-Inert Valve, size 21, Kontes Article Number 749110-0021). Other caps did not perform as well.

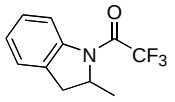
[7] The reactions were heated in an aluminum block set atop a digitally controlled heating magnetic stirrer. Temperature was controlled using a thermocouple controller placed in a hole bored to the diameter of the thermocouple end. The vials were placed in holes bored to the diameter of the vial (15 mm) and to a depth of the shoulder of the vial (35 mm). This depth proved critical in order to minimize non-heated surface area in the vial and prevent solvent reflux at temperatures lower than that of the aluminum block.

[8] For convenience, common household English Standard measuring spoons were used to measure packing materials for SPE columns. For the anhydrous $MgSO_4$, 3/8th of an teaspoon was used; for silica gel, 1/8th teaspoon was used. These materials, silica then $MgSO_4$, were added to the SFE columns in layers using a small funnel and packed down using vacuum (ca. 15 mm Hg).

filtered and washed with EtOAc (3 mL). The filtrate was collected into 2 mL auto-sampler vials[9] and analyzed directly by chiral GC. Conversion was determined by comparison of the area of the sum of the two enantiomers to that of the starting material.

Cyclization Experiments, GC Analysis (Table 3, Entries 1-6):

In a glovebox, a solution of ligand **13g** (10 μmol, 100 μL, 0.1 M in toluene), a solution of $Zr(NMe_2)_4$ (10 μmol, 100 μL, 0.1 M in toluene) and 200 μL of toluene were combined in an oven-dried 4-mL (1 dram) vial equipped with an oven-dried stirbar.[10] The vial was sealed[6] and heated[7] for 15 min. The vial was cooled to 25 °C and a standard solution[11] of the substrate amine and hexamethylbenzene in toluene (100 μL, 1.0 M amine, 100 μmol amine) was added. The vial was resealed and heated for the specified time. The vial was then cooled to 25 °C, removed from the glovebox, and opened to the atmosphere. To this rapidly stirred solution, trifluoroacetic anhydride (50 μL) was added, and the resulting solution was stirred for 5 min. Saturated aqueous Na_2CO_3 (200 μL) was then added, and the resulting suspension was stirred for 1 min. This suspension was then transferred to a 4-mL solid phase extraction (SPE) column (containing approx. 1 g anhydrous $MgSO_4$ atop approx. 0.25 g silica gel)[8] via pipet, filtered and washed with EtOAc (2 mL). The filtrate was collected into 2 mL auto-sampler vials[9] and analyzed directly by chiral GC. For internal standard calibration, 100 μL of the standard amine solution (see above) was diluted to 500 μL using toluene and worked up in the same fashion as the reaction mixture. This latter solution was then analyzed under chiral GC conditions identical to those used for analysis of the reaction mixture.

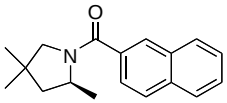


$C_{11}H_{10}F_3NO$
Mol. Wt.: 229.20

Amide 18, Isolated Yield. A solution of ligand **13g** (88.3 μmol, 883 μL, 0.1 M in toluene) and a solution of $Zr(NMe_2)_4$ (88.3 μmol, 883 μL, 0.1 M in toluene) were combined in an oven-dried 4 mL (1 dram) vial equipped with an oven-dried stir bar. The vial was sealed and heated to 135 °C for 15 min. The vial was allowed to cool to room temperature, and a solution of substrate amine (442 μmol, 442 μL, 1.0 M in toluene) was added. The vial was re-sealed and heated to 135 °C for 48 h. The vial was then cooled to 25 °C and removed from the glovebox. The reaction mixture was transferred to a 20 mL scintillation vial. Trifluoroacetic anhydride (185 mg, 123 μL, 883 μmol) was added, and the resulting mixture was stirred 15 min at 25 °C. The reaction mixture was quenched by adding saturated Na_2CO_3 (3 mL). The organic layer was washed with brine (1 x 3 mL), dried over $MgSO_4$, filtered, and concentrated to provide a brown oil. The product was purified by flash chromatography to yield 88 mg (85%) of **18** as a yellow oil: IR (film) 2971,

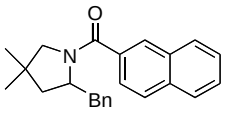
[9] A standard 12-port solid phase extraction vacuum manifold was used.
[10] For reactions involving 20 mol% catalyst, 200 μL each of the ligand and $Zr(NMe_2)_4$ were used. No additional solvent was added.
[11] The standard solution of amine containing hexamethylbenzene as an internal standard was prepared in a 1.0 mL volumetric flask using amine (1.0 mmol), hexamethylbenzene (15 mg), and toluene (to make 1 mL total volume).

1683, 1482, 1137; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8 Hz, 1 H), 7.17-7.21 (m, 2 H), 7.06-7.10 (m, 1 H), 4.72 (m, 1 H), 3.38 (dd, *J* = 13 Hz, 24 Hz, 1 H), 2.63 (d, *J* = 16 Hz, 1 H), 1.24 (d, *J* = 6 Hz, 3 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.3 (q, *J* = 27 Hz), 140.2, 131.2, 128.3, 127.7, 126.0, 125.4, 119.4, 116.3 (q, *J* = 289 Hz), 56.8, 36.9, 21.9; ^{19}F NMR (376 MHz, C_6D_6) δ -69.9; HRMS (ESI^+) *m/z* 252.0608 [252.0612 calcd for $C_{11}H_{10}F_3NO\ (M+Na)^+$].



$C_{18}H_{21}NO$
Mol. Wt.: 267.37

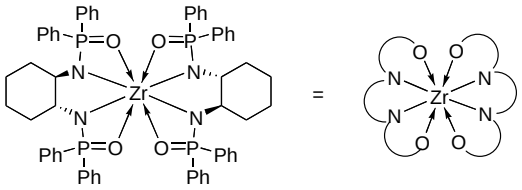
Amide 19. A solution of ligand **13g** (88.3 µmol, 883 µL, 0.1 M in toluene), a solution of $Zr(NMe_2)_4$ (88.3 µmol, 883 µL, 0.1 M in toluene) and 883 µL of toluene were combined in an oven-dried 4 mL (1 dram) vial equipped with an oven-dried stir bar. The vial was sealed and heated to 115 °C for 15 min. The vial was cooled to 25 °C, and a solution of substrate amine (883 µmol, 883 µL, 1.0 M in toluene) was added. The vial was re-sealed and heated at 115 °C for 48 h. The vial was then cooled to 25 °C and removed from the glovebox. The reaction mixture was transferred to a 20 mL scintillation vial. Triethylamine (134 mg, 184 µL, 1.33 mmol) and 2-naphthoyl chloride (168 mg, 883 µmol) were added, and the resulting suspension was stirred for 2 h at 25 °C. The reaction mixture was filtered and washed with saturated $NaHCO_3$ (2 x 3 mL). The organic layer was washed with brine (1 x 3 mL), dried over $MgSO_4$, filtered, and concentrated to provide a brown oil. The product was purified by flash chromatography to yield 184 mg (78%) of **19** as grainy, colorless crystals: IR (solid) cm^{-1} 2957, 2926, 2867, 1619, 1406; ^{1}H NMR (400 MHz, CD_2Cl_2, multiple rotamers observed in solution; major peaks reported) δ 7.89-8.04 (m, 4 H), 7.52-7.64 (m, 3 H), 4.39 (m, 1 H), 3.39 (d, *J* = 12 Hz, 1 H), 3.22 (d, *J* = 12 Hz, 1 H), 1.98 (dd, *J* = 12 Hz, 7 Hz, 1 H), 1.47 (dd, *J* = 16 Hz, 12 Hz, 1 H), 1.43 (d, *J* = 6 Hz, 3 H), 1.05 (s, 3 H), 0.81 (s, 3 H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 169.7, 134.8, 133.8, 132.5, 128.4, 128.3, 127.8, 127.6, 127.2, 127.0, 126.5, 62.6, 47.4, 38.2, 25.4, 25.2, 20.0; HRMS (ESI^+) *m/z* 268.1696 [268.1701 calcd for $C_{18}H_{21}NO\ (M+H)^+$].



$C_{24}H_{25}NO$
Mol. Wt.: 343.46

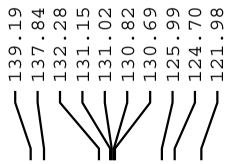
Amide 20. A solution of ligand **13g** (88.3 µmol, 883 µL, 0.1 M in toluene) and a solution of $Zr(NMe_2)_4$ (88.3 µmol, 883 µL, 0.1 M in toluene) were combined in an oven-dried 4 mL (1 dram) vial equipped with an oven-dried stir bar. The vial was sealed and heated at 135 °C for 15 min. After cooling to 25 °C, a solution of substrate amine (442 µmol, 442 µL, 1.0 M in toluene) was added. The vial was re-sealed and heated at 135 °C for 24 h. It was then cooled to 25 °C and removed from the glovebox. The reaction mixture was transferred to a 20 mL scintillation vial. Triethylamine (134 mg, 184 µL, 1.33 mmol) and 2-naphthoyl chloride (168 mg, 883 µmol) were added, and the resulting suspension was stirred for 2 h at 25 °C. The reaction mixture was filtered and washed with saturated $NaHCO_3$ (2 x 3 mL). The organic layer was washed with brine (1 x 3 mL), dried over $MgSO_4$, filtered, and concentrated to provide a brown oil. The product was purified by flash chromatography to yield 115 mg (76%) of **20** as a yellow oil: IR (film) cm^{-1} 3058, 2956, 2867, 1620, 1407; ^{1}H NMR (400 MHz, C_6D_6) δ 8.12 (s, 1 H), 7.80 (d, *J* = 8 Hz, 1 H), 7.32-7.64 (m, 3 H), 7.18-7.32 (m, 2 H), 4.74-4.76 (m, 1 H), 3.48 (d, *J* = 12 Hz, 1 H), 3.01-3.06 (m, 1 H), 2.92 (d, *J* = 8 Hz, 1 H), 2.79 (d, *J* = 8 Hz), 1.35-1.42 (m, 2 H), 0.56

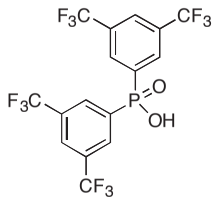
(s, 3H), 0.54 (s, 3 H); ^{13}C NMR (126 MHz, CD_2Cl_2) δ 169.8, 138.6, 134.7, 133.9, 132.5, 129.8, 128.5, 128.1, 127.8, 127.6, 127.5, 127.3, 127.0, 126.5, 126.2, 124.7, 63.1, 57.9, 43.9, 39.0, 37.9, 25.2; HRMS (ESI$^+$) *m/z* 366.1841 [366.1834 calcd for $C_{24}H_{25}NO$ $(M+Na)^+$].



$C_{60}H_{60}N_4O_4P_4Zr$
Mol. Wt.: 1116.2618

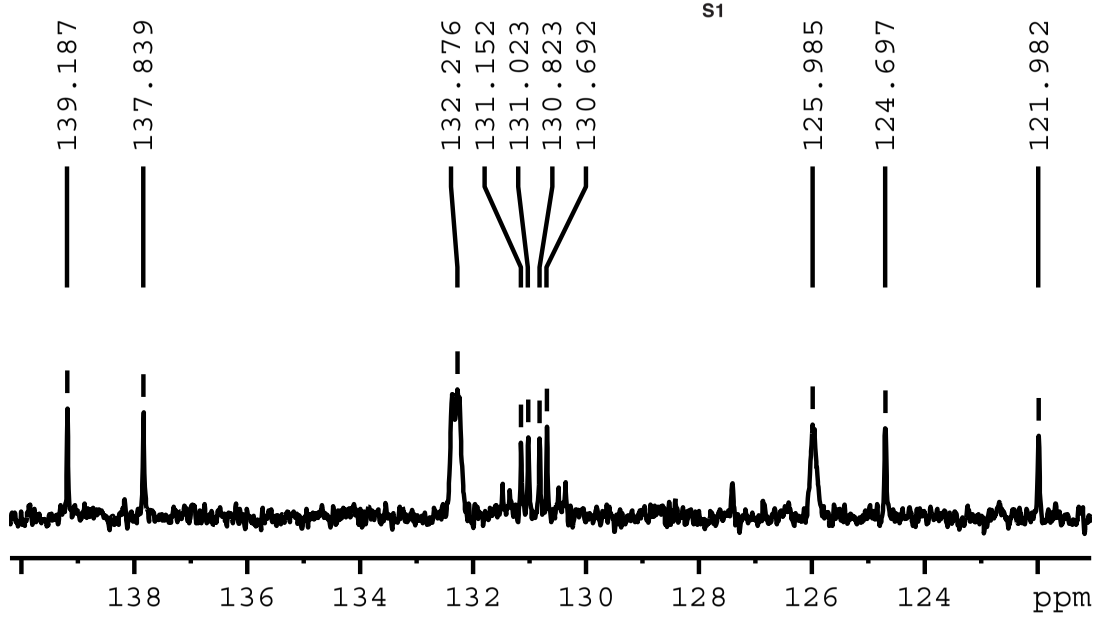
Complex 21a. In a glovebox, (*R*,*R*)-ligand **3c** (50 mg, 97 μmol), $Zr(NMe_2)_4$ (26 mg, 97 μmol) and d_8-toluene (0.5 mL) were combined in a 4 mL vial equipped with a magnetic stirbar. The resulting suspension was stirred for 30 min, during which time the reaction mixture became homogenous. The solution was transferred to a medium-walled NMR tube equipped with a teflon valve (J. Young tube). The tube was sealed and then removed from the glovebox. The tube was heated in an oil bath at 150 °C for 48 h and was occasionally removed to monitor reaction progress by NMR. Once intermediate **20a** was fully converted to **21a** (as judged by NMR), the tube was returned to the glovebox and opened. The solution was transferred to a 4 ml vial and layered with 1 ml of pentane, resulting in formation of microcrystalline **21a**. Decanting of the solvent provided 16 mg (30%) of **21a** as colorless prisms. Recrystallization from hot toluene provided X-ray quality crystals: ^{1}H NMR (400 MHz, C_6D_6) δ 8.03 (ddd, *J* = 20.8, 12.2, 7.3 Hz, 16 H), 7.26 (t, *J* = 7.3 Hz, 4 H), 7.15 (td, *J* = 7.6, 2.2 Hz, 8 H), 6.97-7.03 (m, 4 H), 6.9 (t, *J* = 7.8 Hz, 8 H), 4.07 (d, *J* = 9.5 Hz, 4 H), 1.79 (d, *J* = 11.0 Hz, 4 H), 1.51-1.67 (m, 4 H), 1.35 (t, *J* = 8.6 Hz, 4 H), 1.08 (t, *J* = 9.8 Hz, 3 H); ^{13}C NMR (126 MHz, C_6D_6, 2 resonances obscured) δ 137.1 (d, *J* = 117.3 Hz), 135. 2 (d, *J* = 99.8 Hz), 133.0 (d, *J* = 10.8 Hz), 132.4 (d, *J* = 10.8 Hz), 130.3 (d, *J* = 1.9 Hz), 130.0 (d, *J* = 2.4 Hz), 65.5 (d, *J* = 16.0 Hz), 35.3, 25.1; ^{31}P NMR (162 MHz, C_6D_6) δ 34.3. Anal. Calcd for $C_{60}H_{60}N_4O_4P_4Zr$: C, 64.56; H, 5.42; N, 5.02. Found: C, 64.49; H, 5.22; N, 5.92.



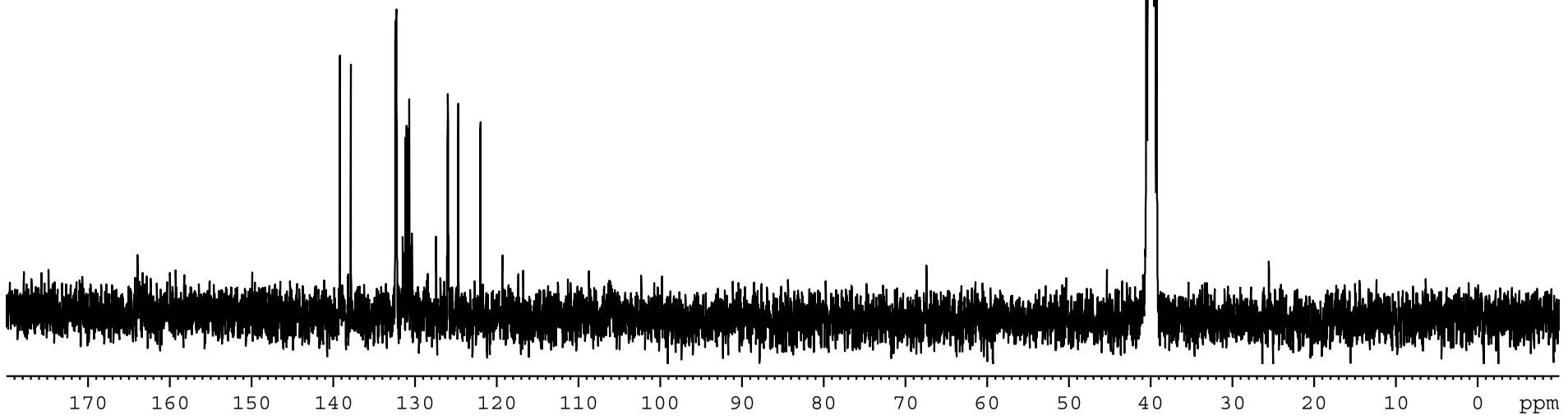


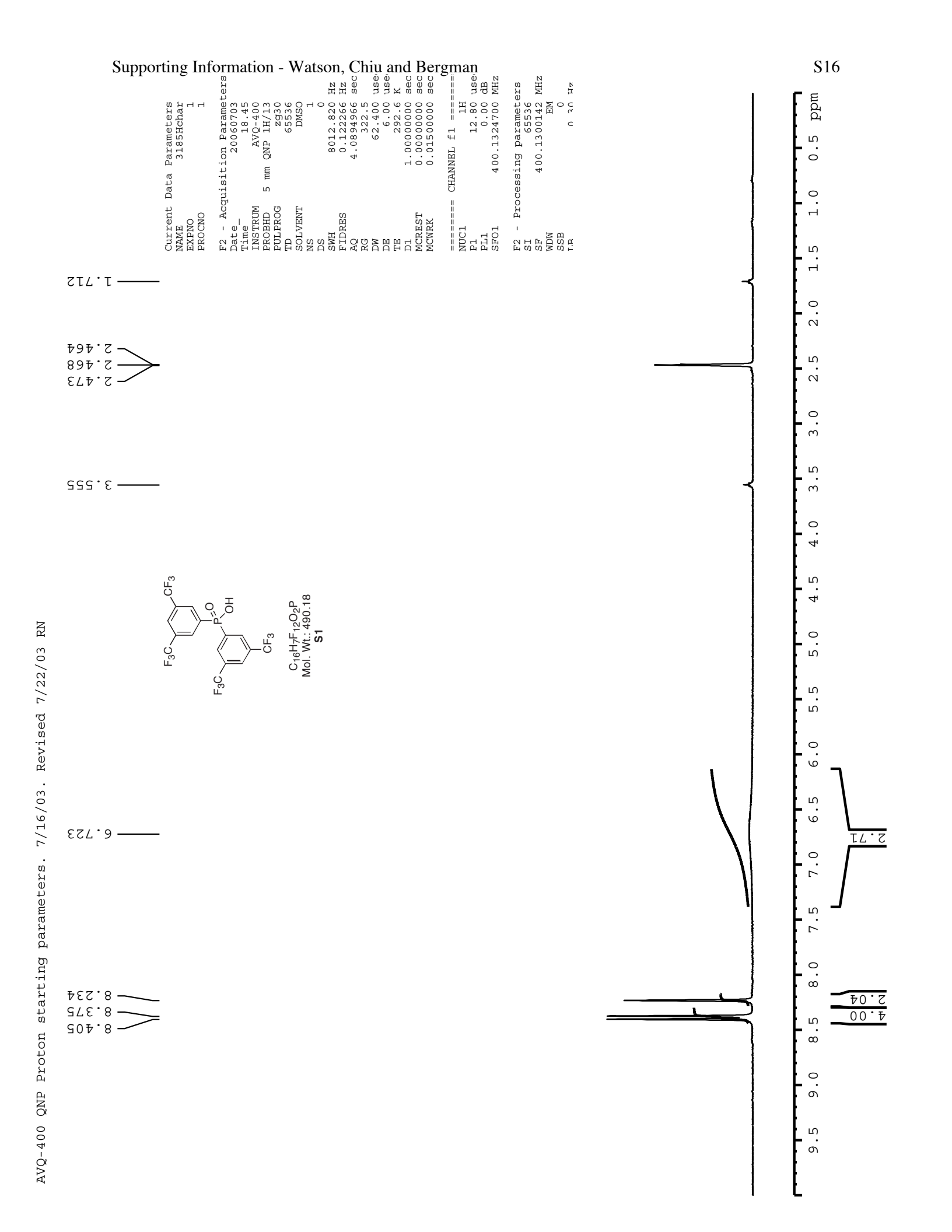
$C_{16}H_7F_{12}O_2P$
Mol. Wt.: 490.18
S1

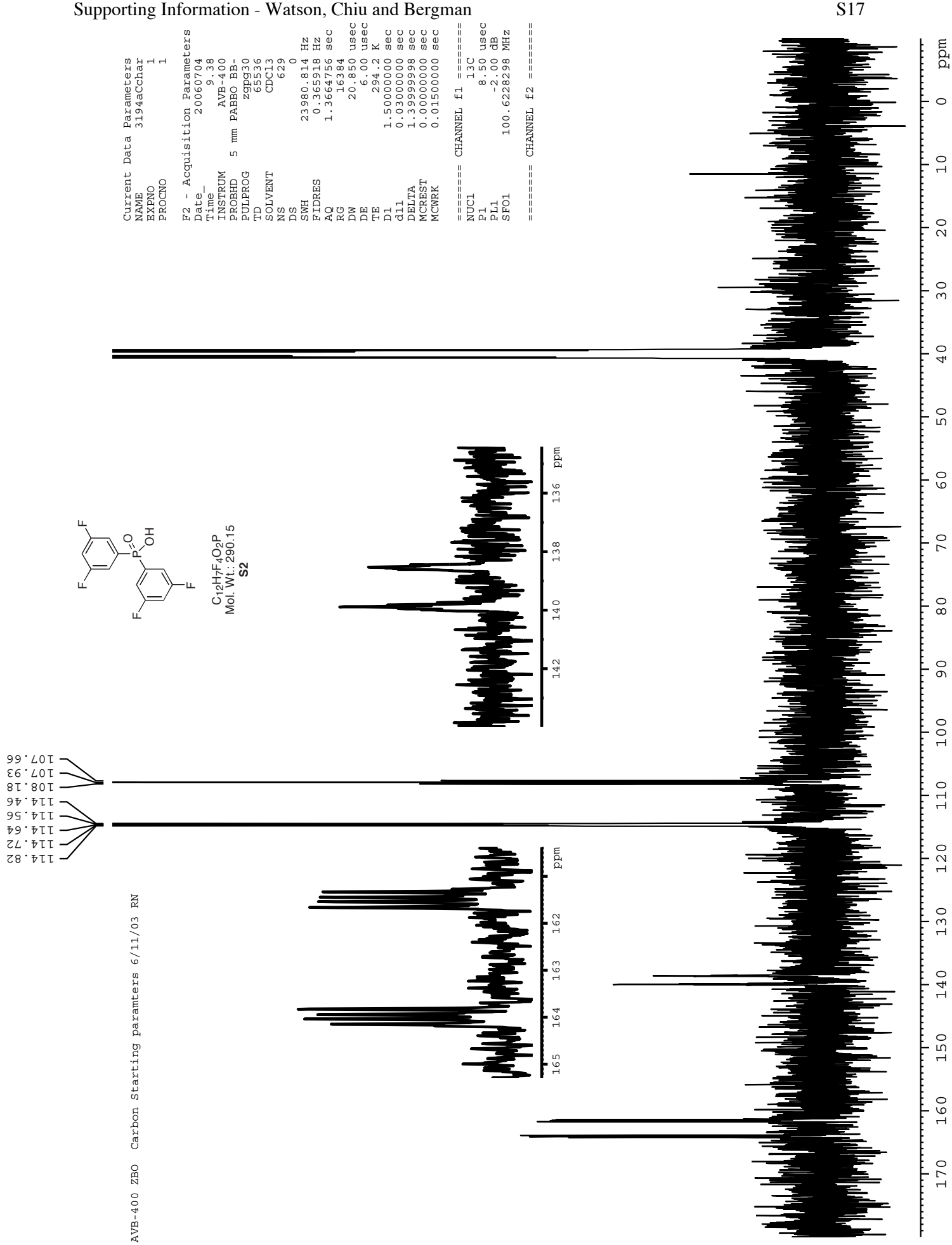
AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7



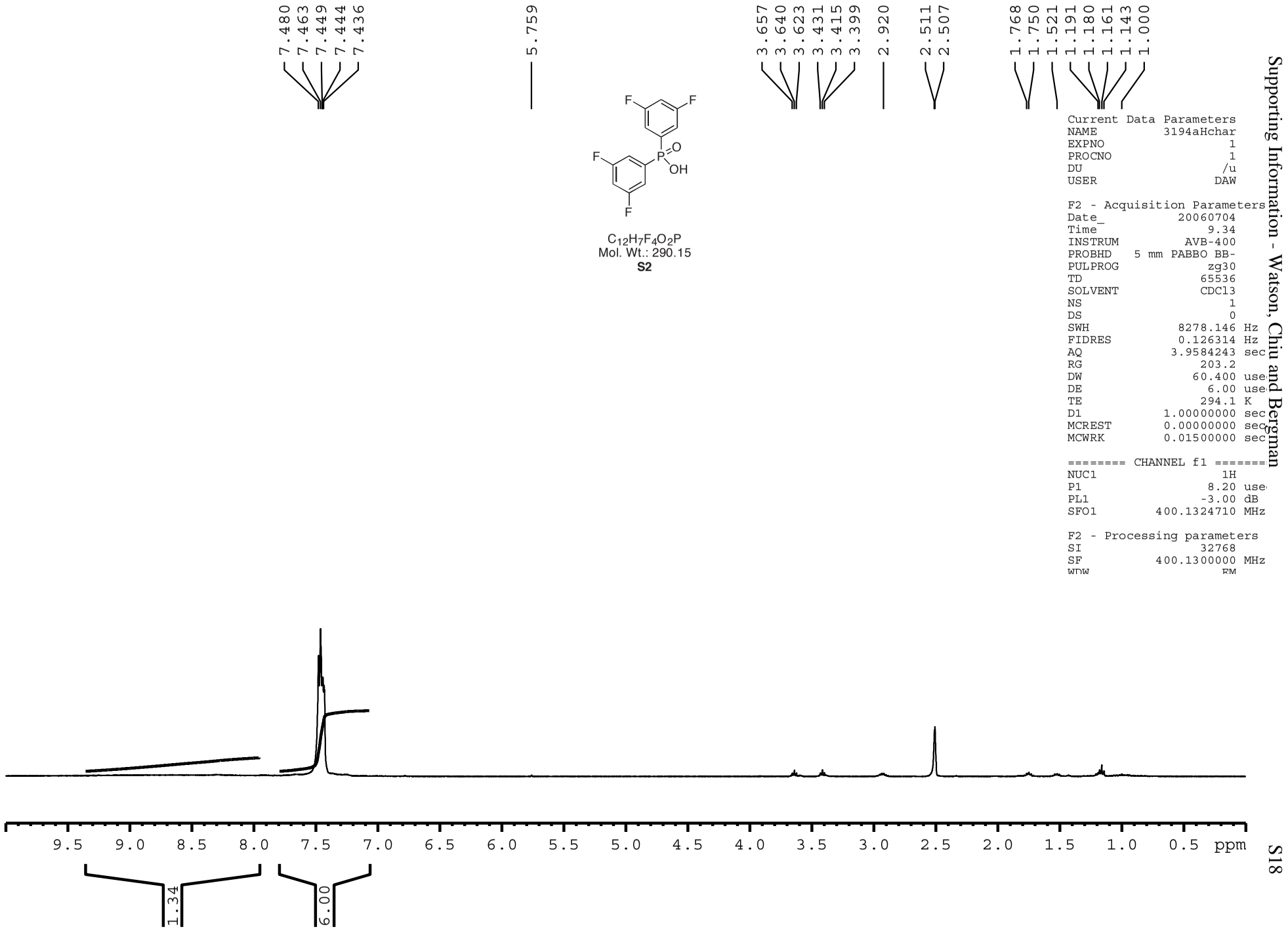

```
Current Data Parameters
NAME                  3185Cchar
EXPNO                         1
PROCNO                        1
DU                           /u
USER                        DAW

F2 - Acquisition Parameters
Date_                  20060703
Time                      18.49
INSTRUM                 AVQ-400
PROBHD    5 mm QNP 1H/13
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         557
DS                           0
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                   1.3632196 sec
RG                       16384
DW                    20.800 usec
DE                     6.00 usec
TE                     292.6 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      8.50 usec
PL1                     -2.00 dB
SFO1               100.6228298 MHz
```

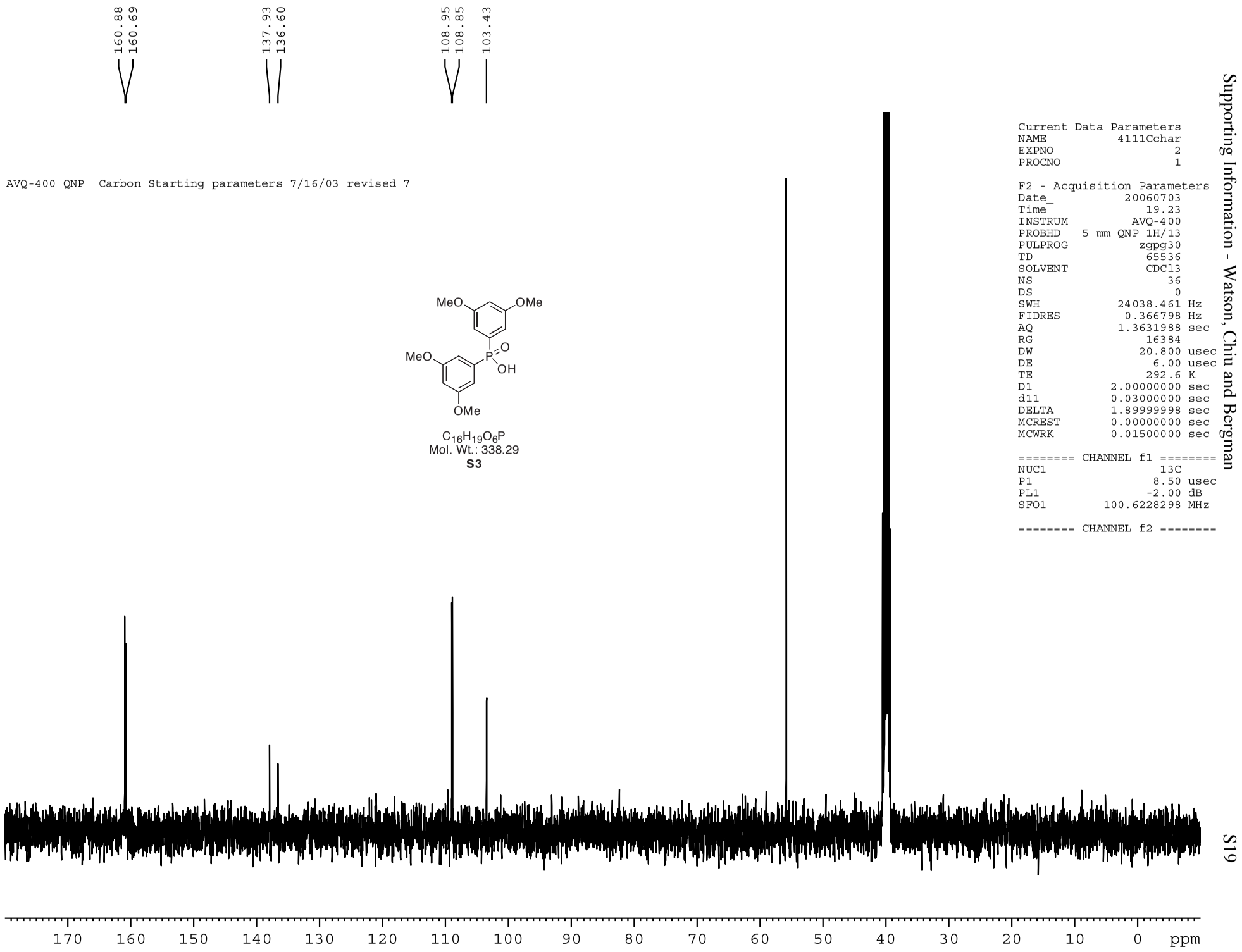


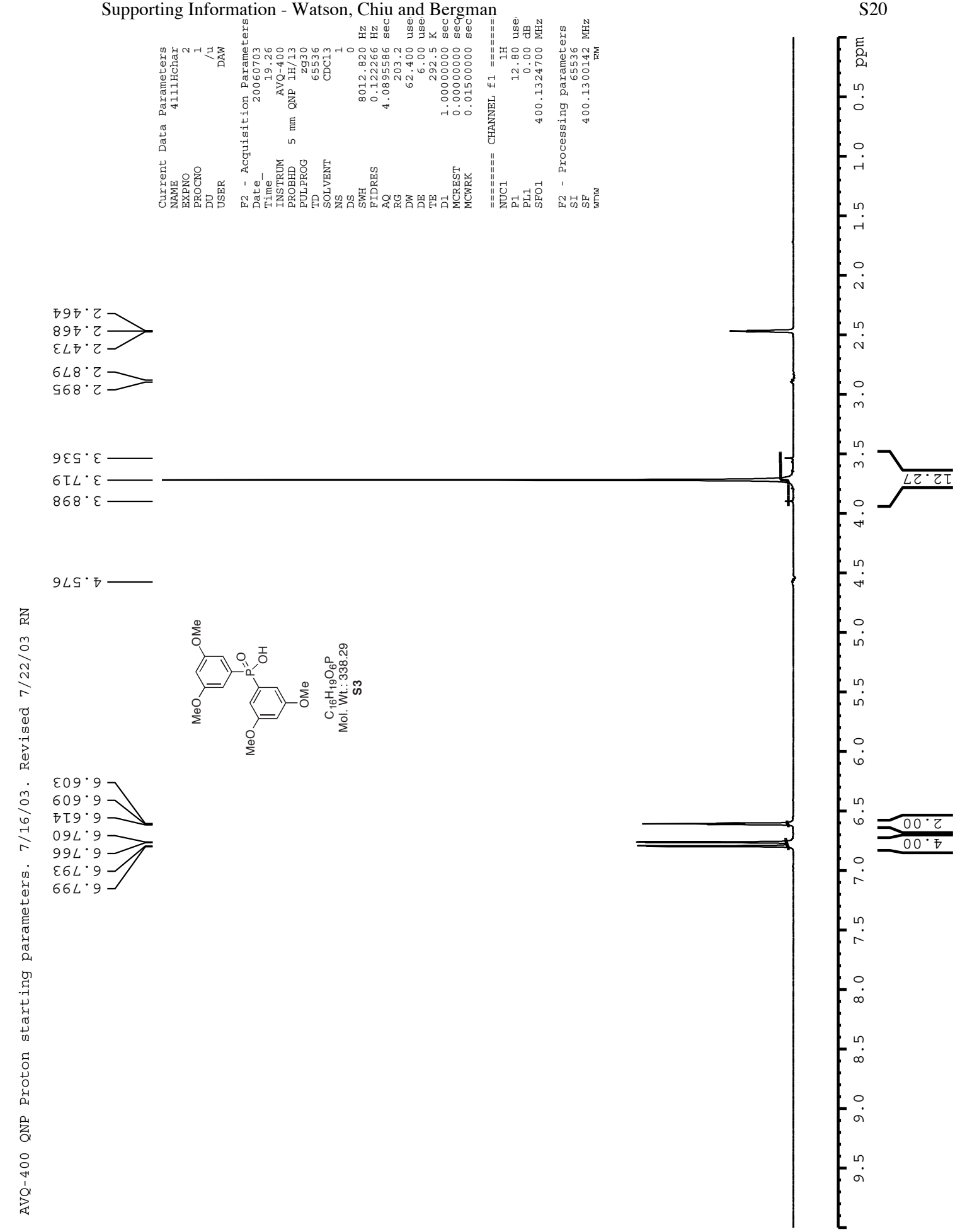

AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN
Current Data Parameters
NAME 3185Hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060703
Time_ 18.45
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 322.5
DW 62.400 use
DE 6.00 use
TE 292.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
8.405
8.375
8.234
6.723
3.555
2.473
2.468
2.464
1.712
CF3
F3C
P
O
OH
CF3
F3C
C16H7F12O2P
Mol. Wt.: 490.18
S1
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.00
2.04
2.71

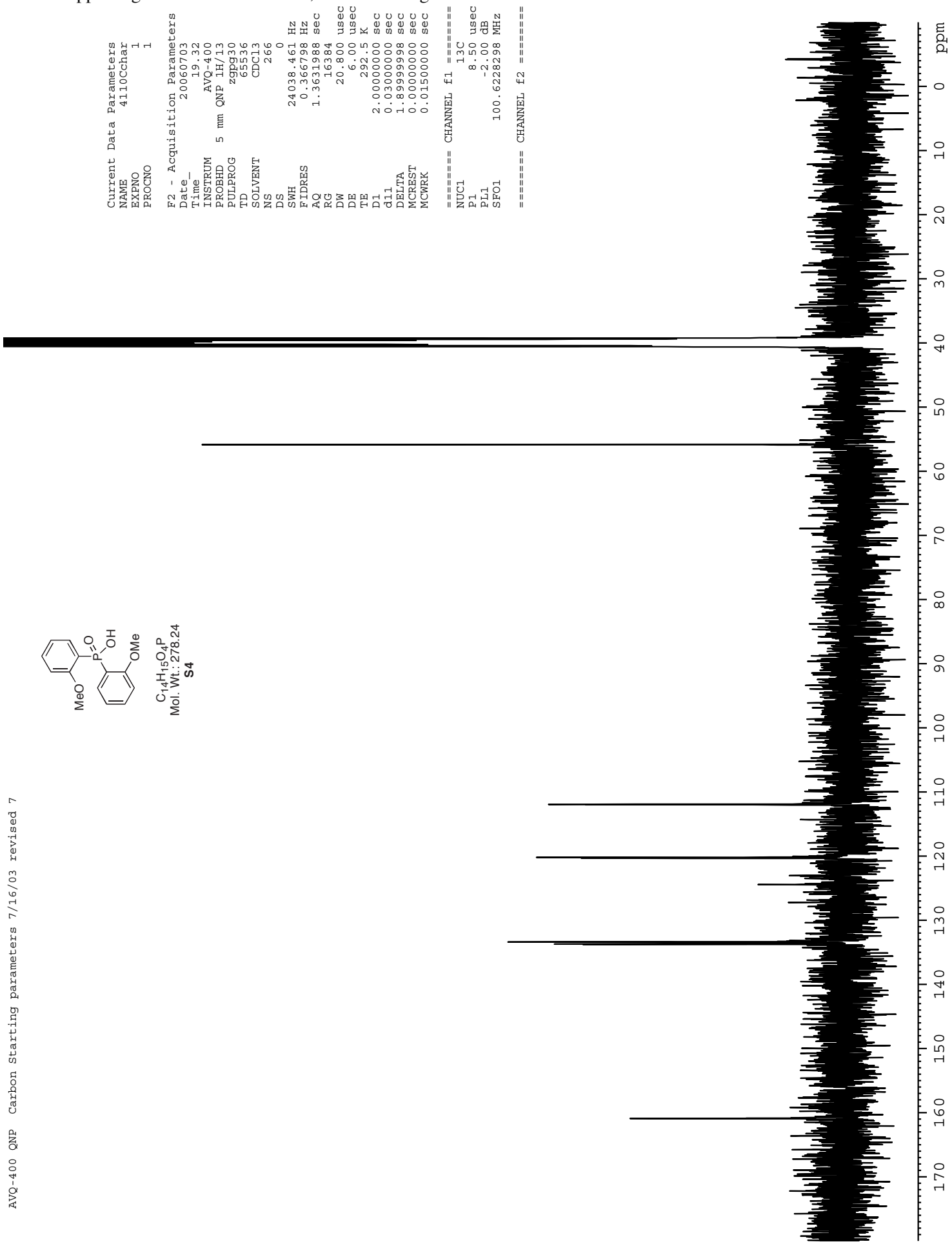

AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
Current Data Parameters
NAME 3194aCchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060704
Time_ 9.38
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 629
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 294.2 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
C12H7F4O2P
Mol. Wt.: 290.15
S2
114.82
114.72
114.64
114.56
114.46
108.18
107.93
107.66
ppm

AVB-400 ZBO Proton starting parameters. 6/11/03 RN

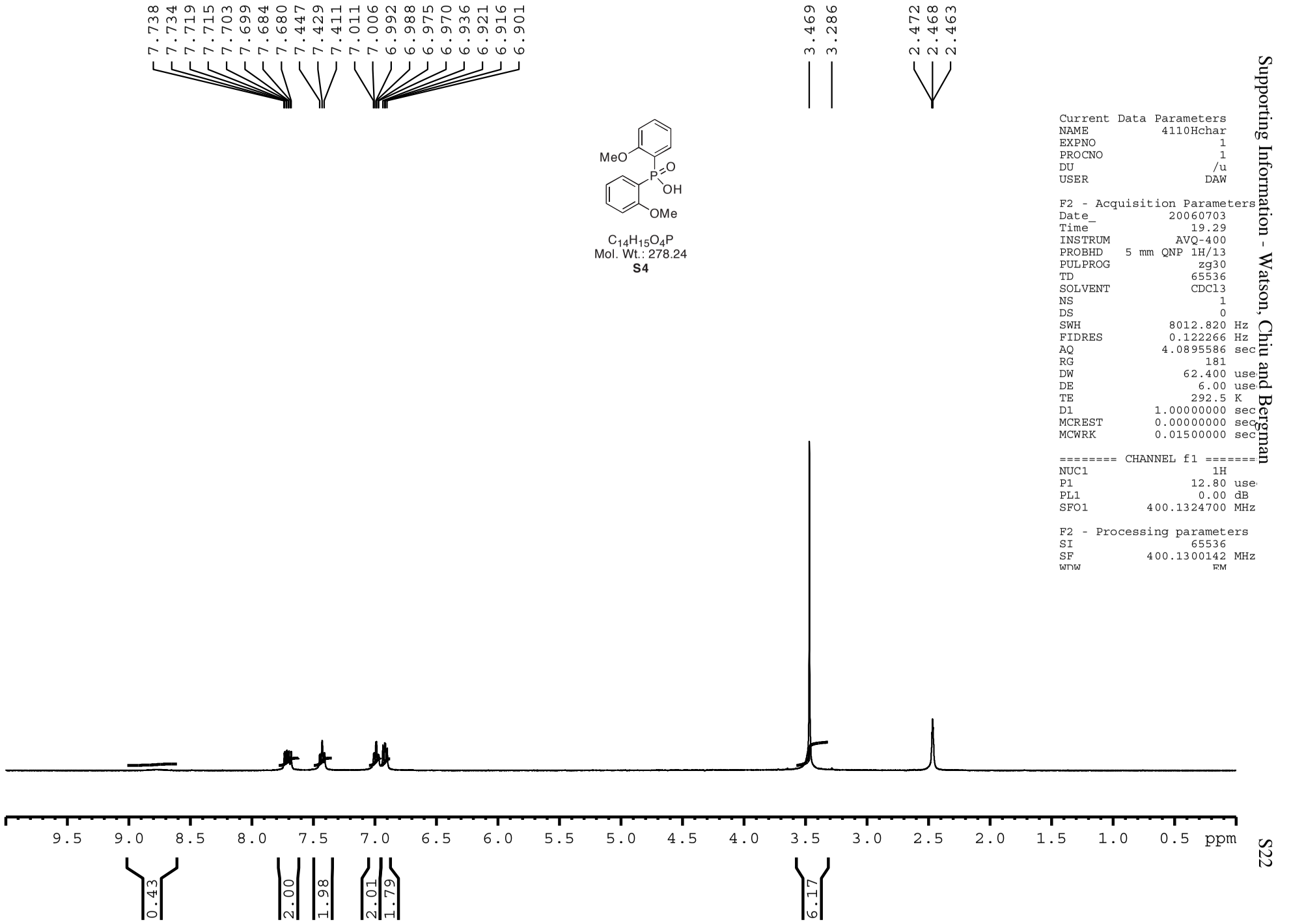


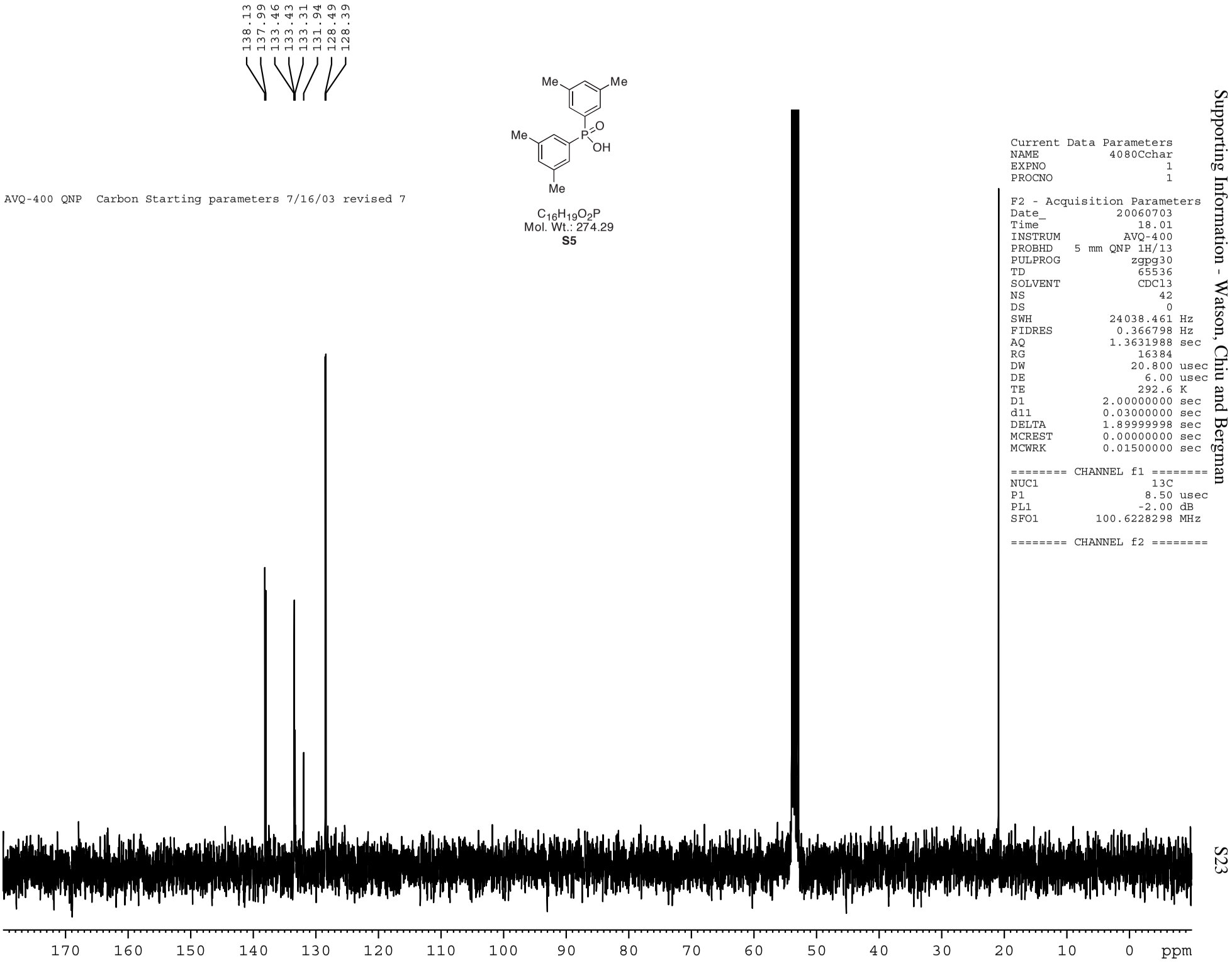

160.88
160.69
137.93
136.60
108.95
108.85
103.43
AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7
MeO
OMe
MeO
P
O
OH
OMe
C16H19O6P
Mol. Wt.: 338.29
S3
Current Data Parameters
NAME 4111Cchar
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060703
Time 19.23
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN
Current Data Parameters
NAME 4111Hchar
EXPNO 2
PROCNO 1
DU /u
USER DAW
F2 - Acquisition Parameters
Date_ 20060703
Time_ 19.26
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 203.2
DW 62.400 use
DE 6.00 use
TE 292.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
6.799
6.793
6.766
6.760
6.614
6.609
6.603
4.576
3.898
3.719
3.536
2.895
2.879
2.473
2.468
2.464
OMe
MeO
OMe
MeO
OH
P
O
C16H19O6P
Mol. Wt.: 338.29
S3
12.27
4.00
2.00
ppm

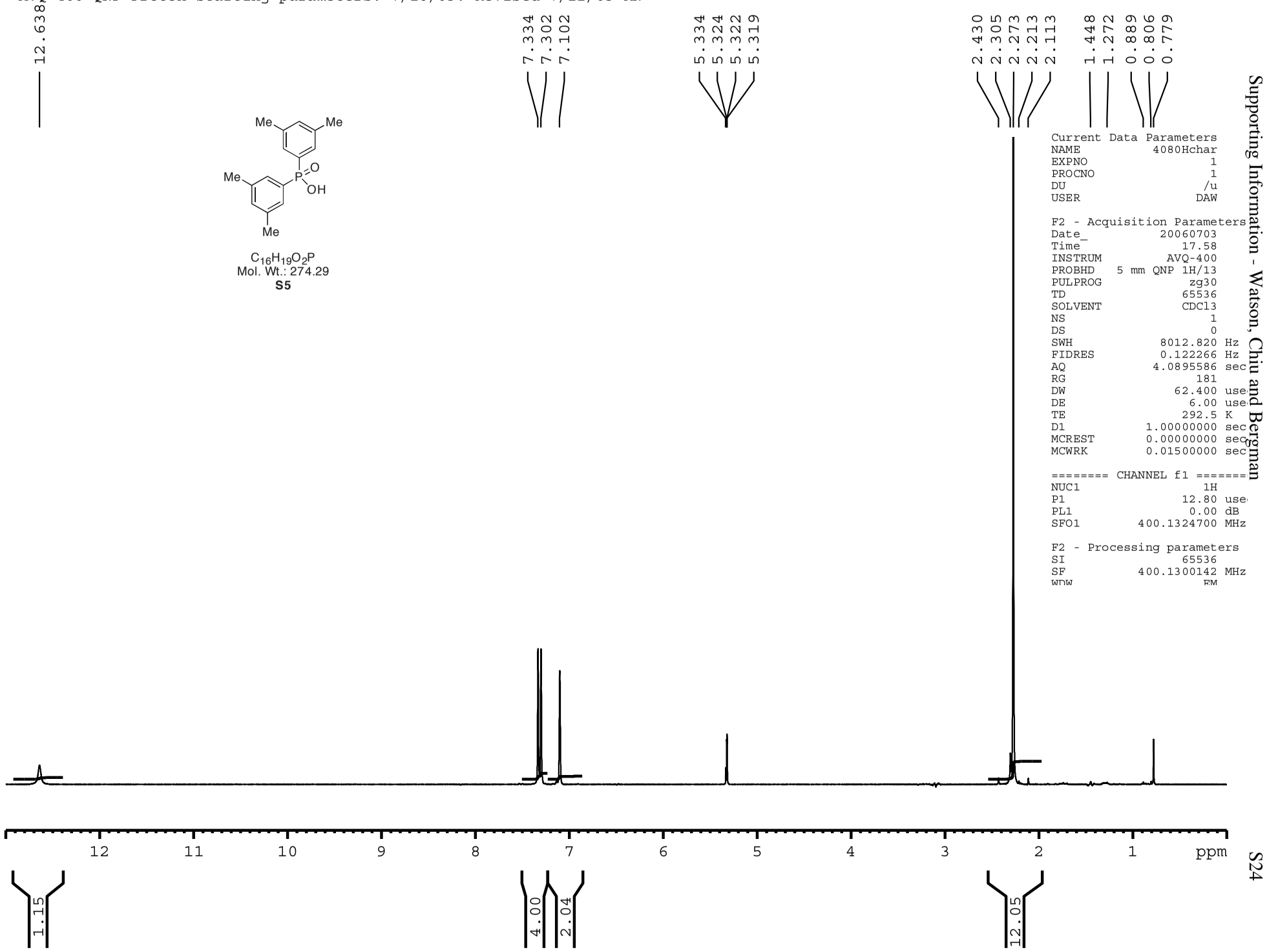

AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7
Current Data Parameters
NAME 4110Cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060703
Time_ 19.32
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 266
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
MeO
OMe
OH
P
O
C14H15O4P
Mol. Wt.:278.24
S4
ppm

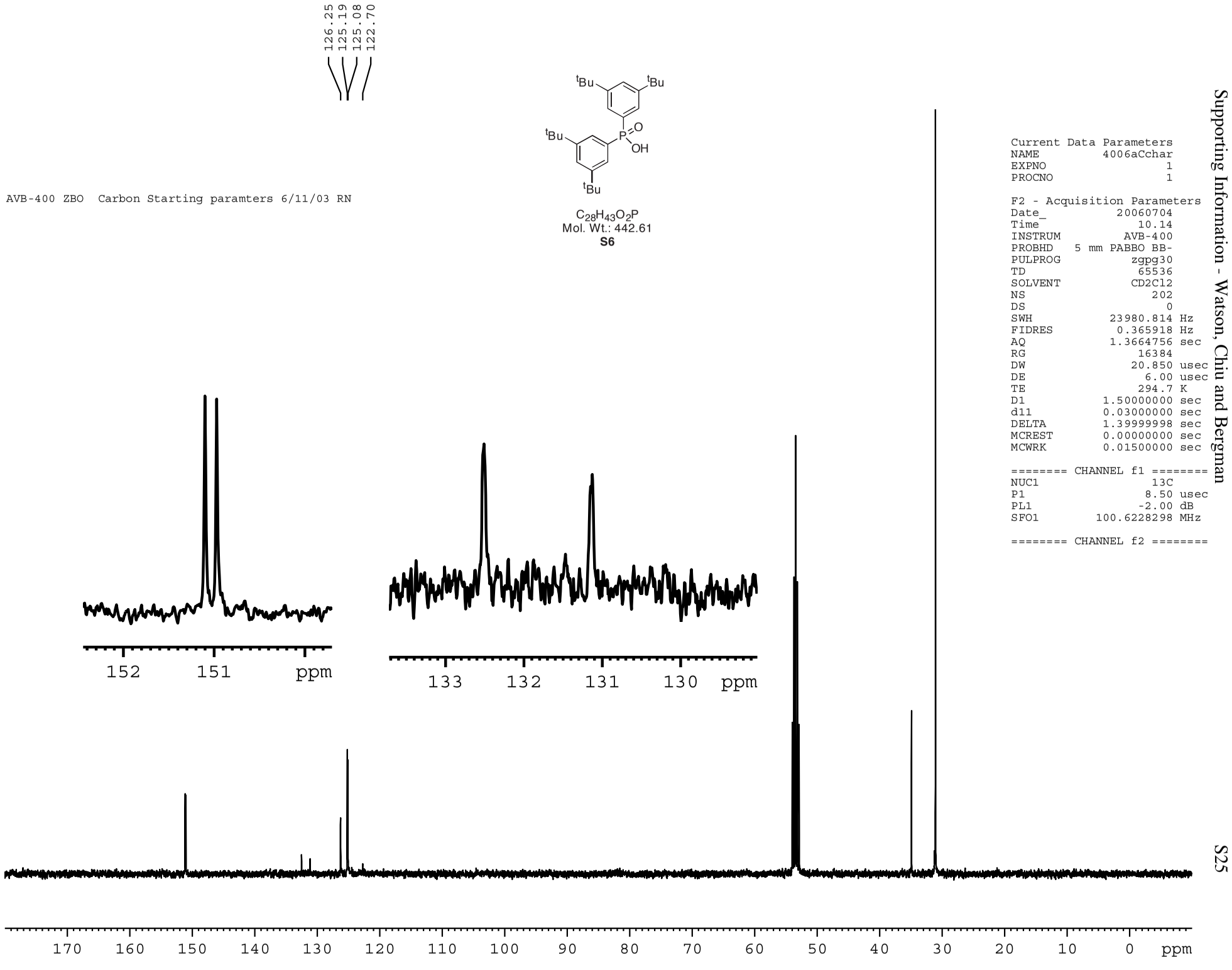
AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



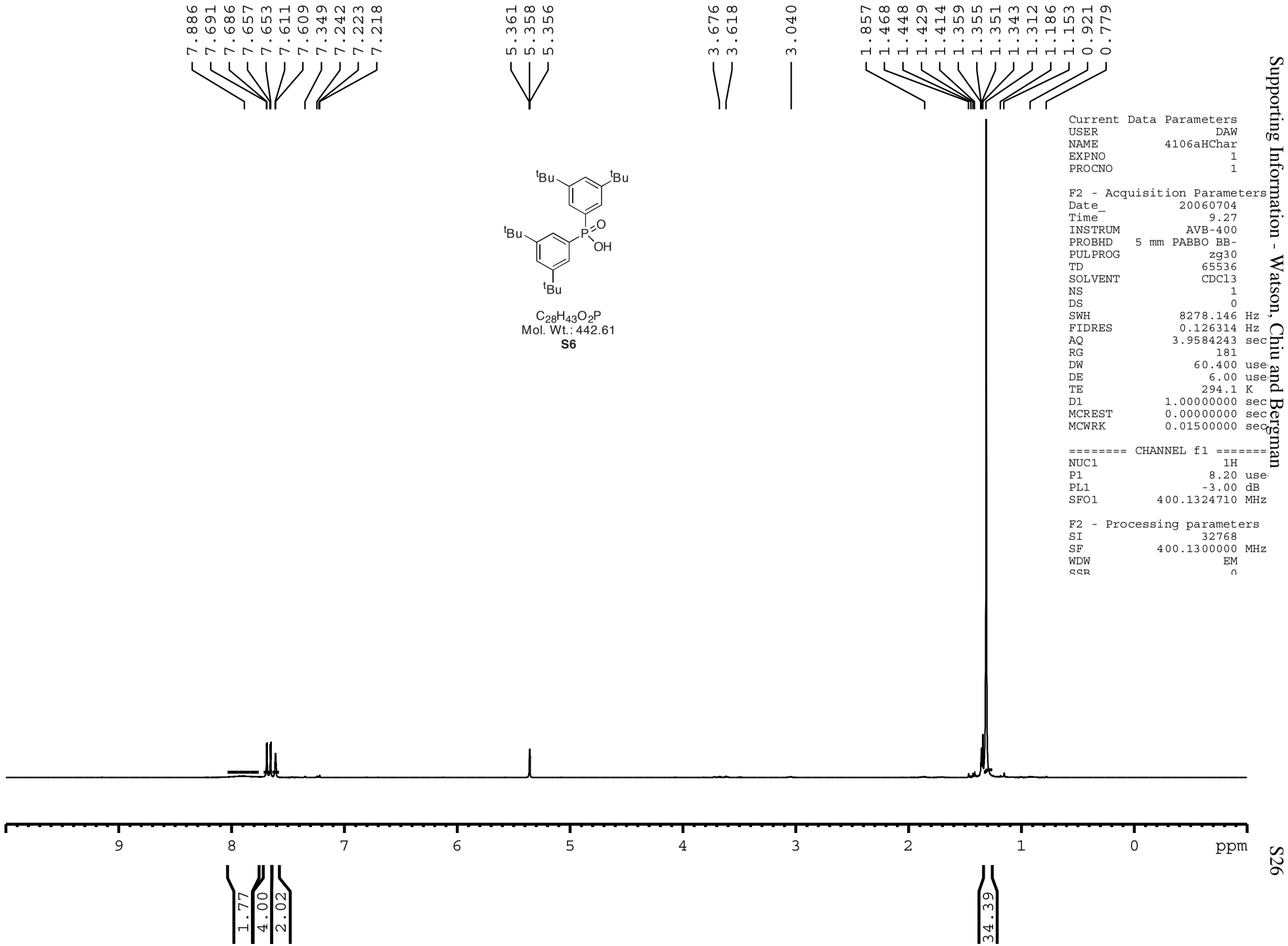

138.13
137.99
133.46
133.43
133.31
131.94
128.49
128.39
Me
Me
Me
P
O
OH
Me
C16H19O2P
Mol. Wt.: 274.29
S5
AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7
Current Data Parameters
NAME 4080Cchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060703
Time 18.01
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

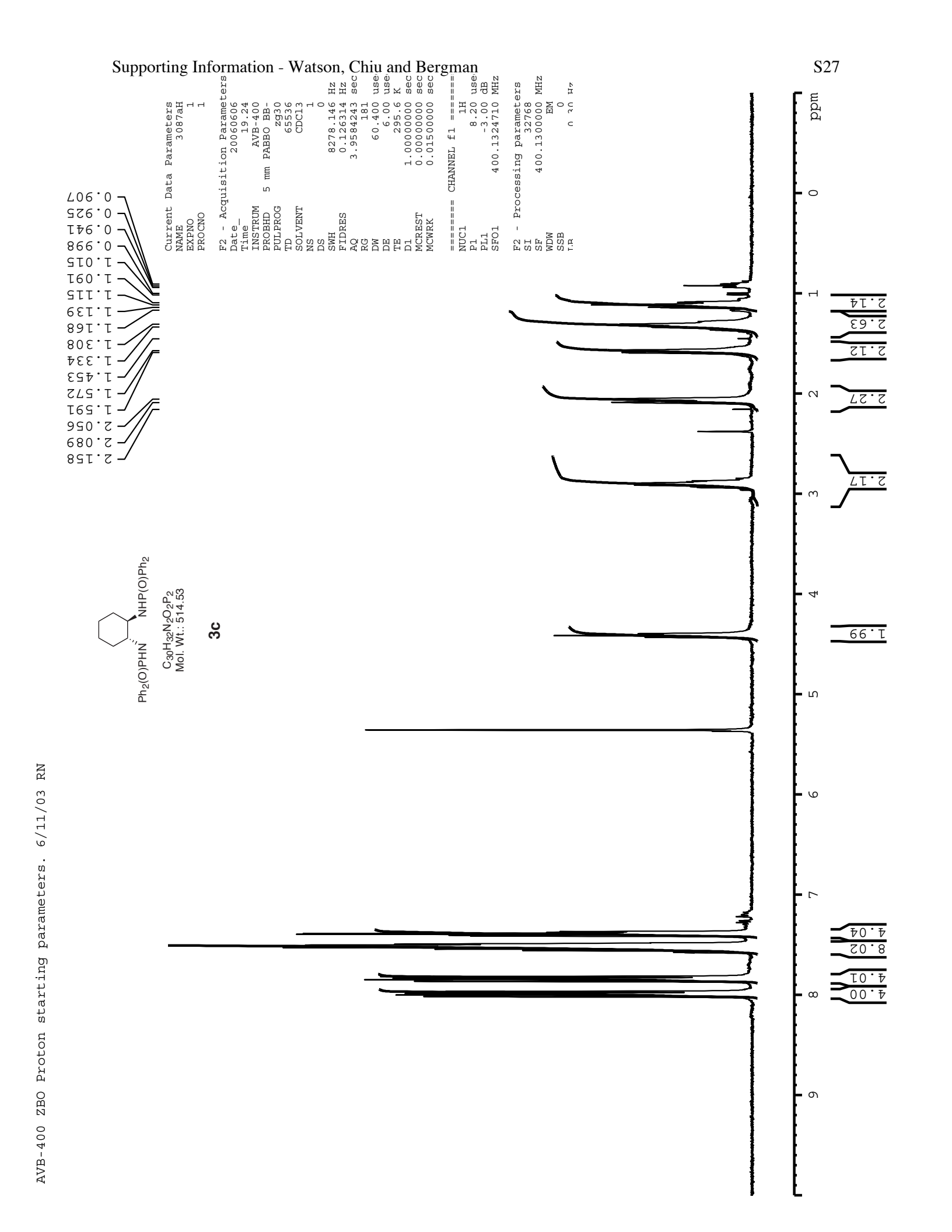
AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



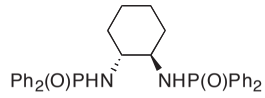

126.25
125.19
125.08
122.70
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
$C_{28}H_{43}O_2P$
Mol. Wt.: 442.61
S6
Current Data Parameters
NAME 4006aCchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060704
Time 10.14
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 202
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 294.7 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
152
151
ppm
133
132
131
130
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

AVB-400 ZBO Proton starting parameters. 6/11/03 RN




AVB-400 ZBO Proton starting parameters. 6/11/03 RN
Current Data Parameters
NAME 3087aH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time_ 19.24
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 use
DE 6.00 use
TE 295.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 8.20 use
PL1 -3.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
NHP(O)Ph2
Ph2(O)PHN
C30H32N2O2P2
Mol. Wt.: 514.53
3c
ppm
0.907
0.925
0.941
0.998
1.015
1.091
1.115
1.139
1.168
1.308
1.334
1.453
1.572
1.591
2.056
2.089
2.158
1.99
2.17
2.27
2.12
2.63
2.14
4.00
4.01
8.02
4.04

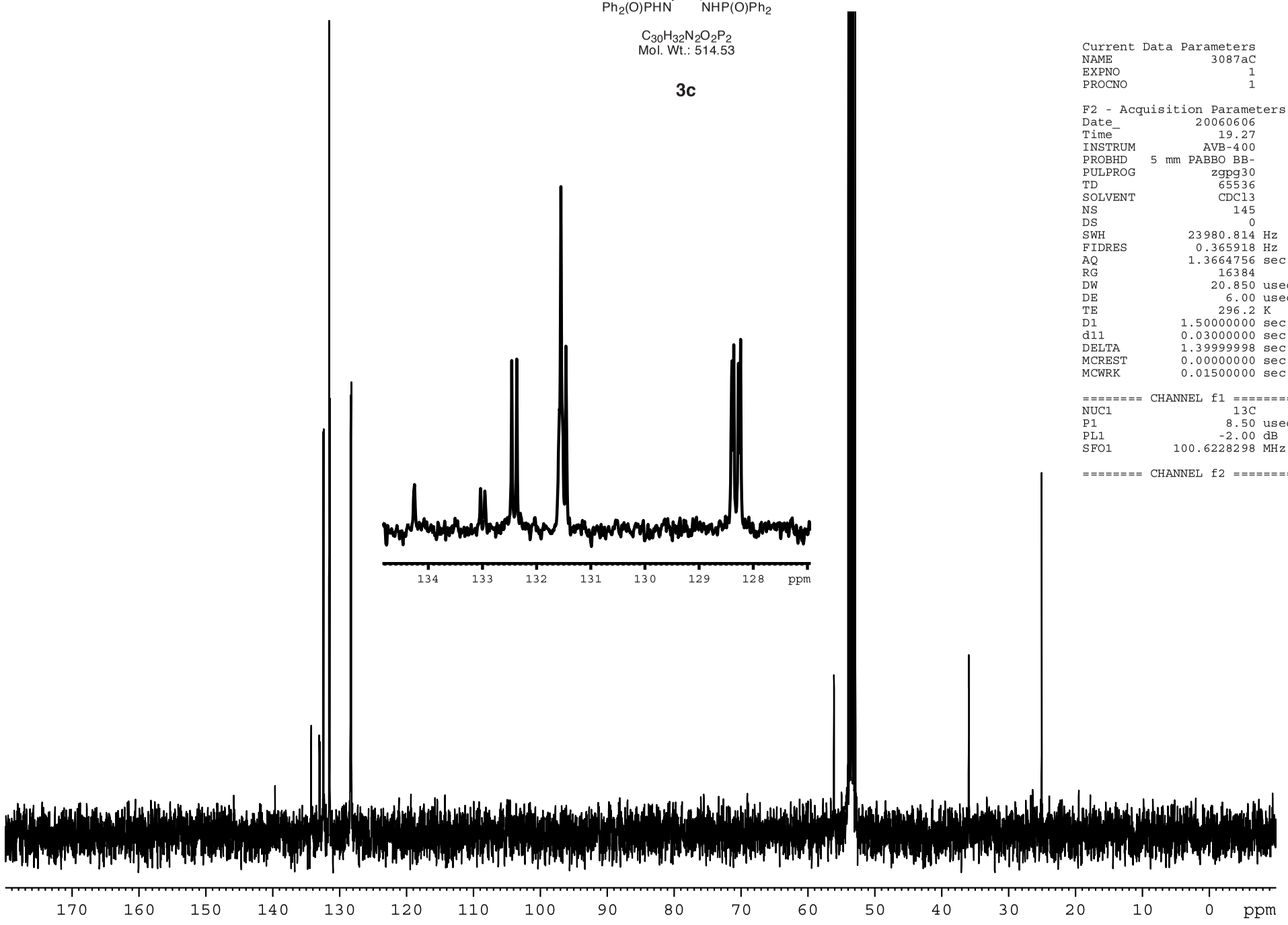
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN

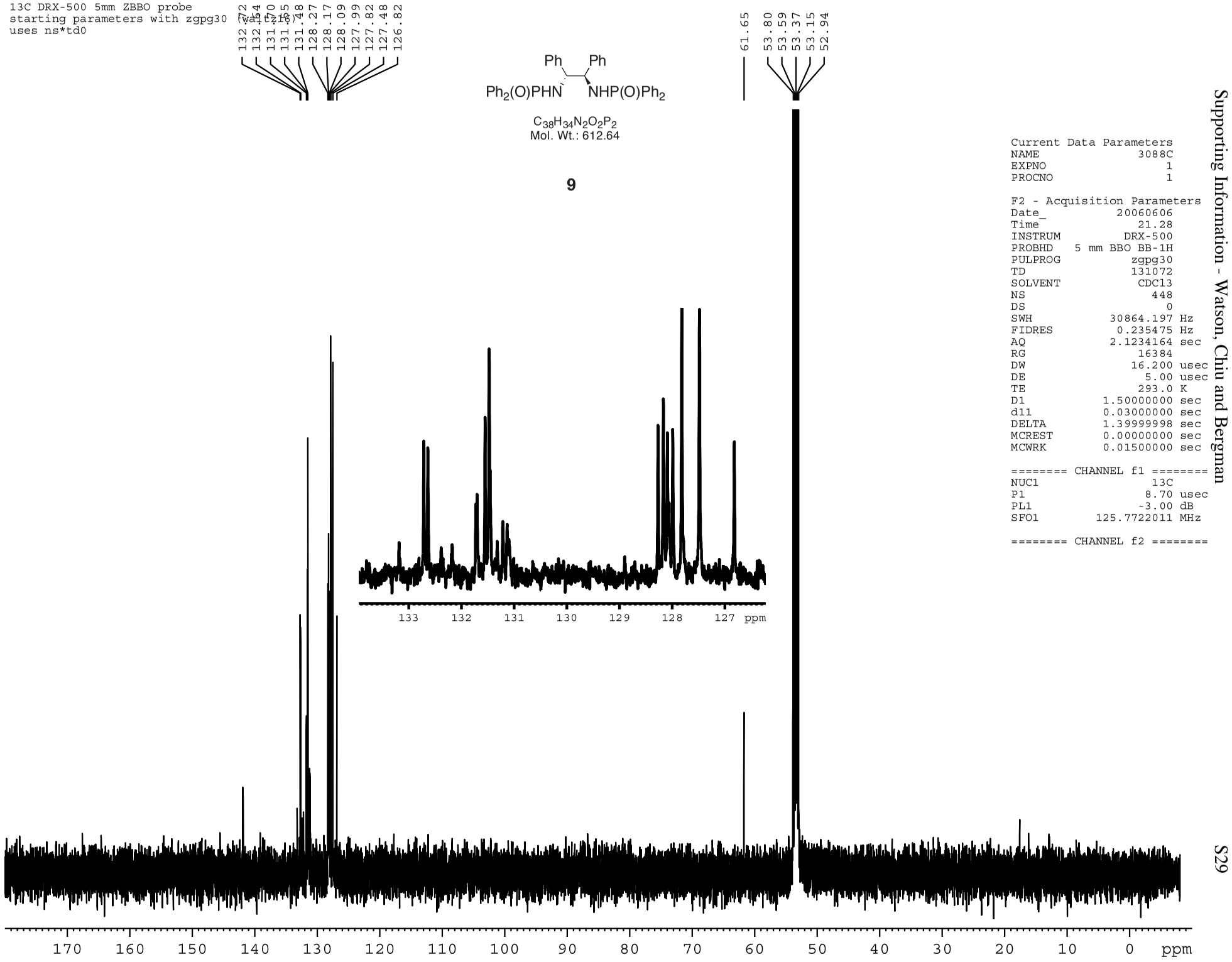


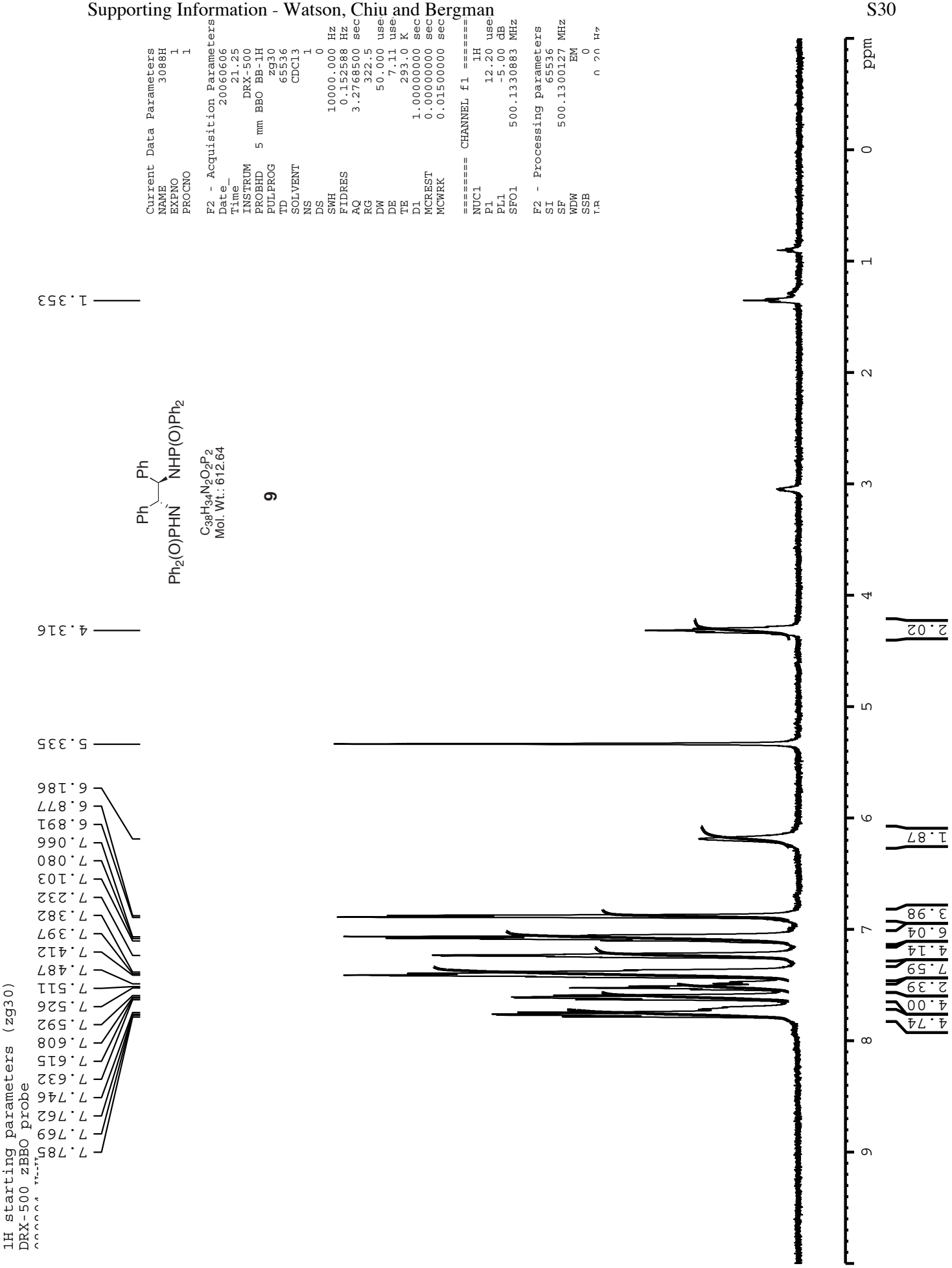
$C_{30}H_{32}N_2O_2P_2$
Mol. Wt.: 514.53

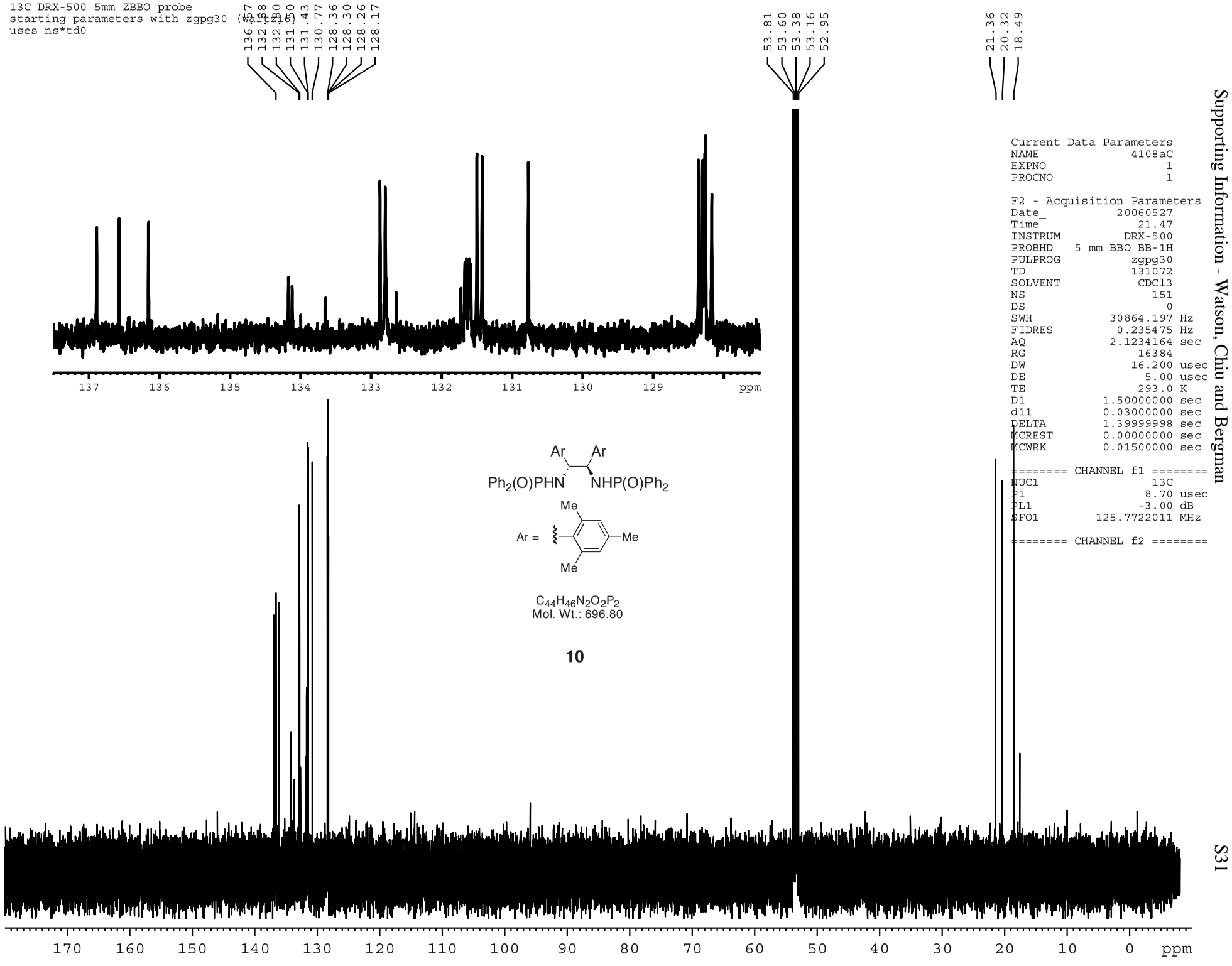
3c

```
Current Data Parameters
NAME                 3087aC
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060606
Time                 19.27
INSTRUM            AVB-400
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     145
DS                       0
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                   16384
DW                  20.850 usec
DE                   6.00 usec
TE                  296.2 K
D1              1.50000000 sec
d11             0.03000000 sec
DELTA           1.39999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.50 usec
PL1                  -2.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
```

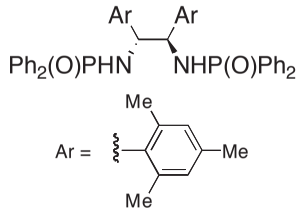



13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30
uses ns*td0
132.72
132.54
131.70
131.15
131.48
128.27
128.17
128.09
127.99
127.82
127.48
126.82
61.65
53.80
53.59
53.37
53.15
52.94
Ph
Ph
Ph2(O)PHN
NHP(O)Ph2
C38H34N2O2P2
Mol. Wt.: 612.64
9
Current Data Parameters
NAME 3088C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time 21.28
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 448
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
======== CHANNEL f2 ========
133
132
131
130
129
128
127 ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


1H starting parameters (zg30)
DRX-500 zBBO probe
Current Data Parameters
NAME 3088H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time_ 21.25
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 322.5
DW 50.000 use
DE 7.11 use
TE 293.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.20 use
PL1 -5.00 dB
SFO1 500.1330883 MHz
F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
Ph
Ph
Ph2(O)PHN
NHP(O)Ph2
C38H34N2O2P2
Mol. Wt.: 612.64
9
7.785
7.769
7.762
7.746
7.632
7.615
7.608
7.592
7.526
7.511
7.487
7.412
7.397
7.382
7.232
7.103
7.080
7.066
6.891
6.877
6.186
5.335
4.316
1.353
4.74
4.00
2.39
7.59
4.14
6.04
3.98
1.87
2.02
ppm

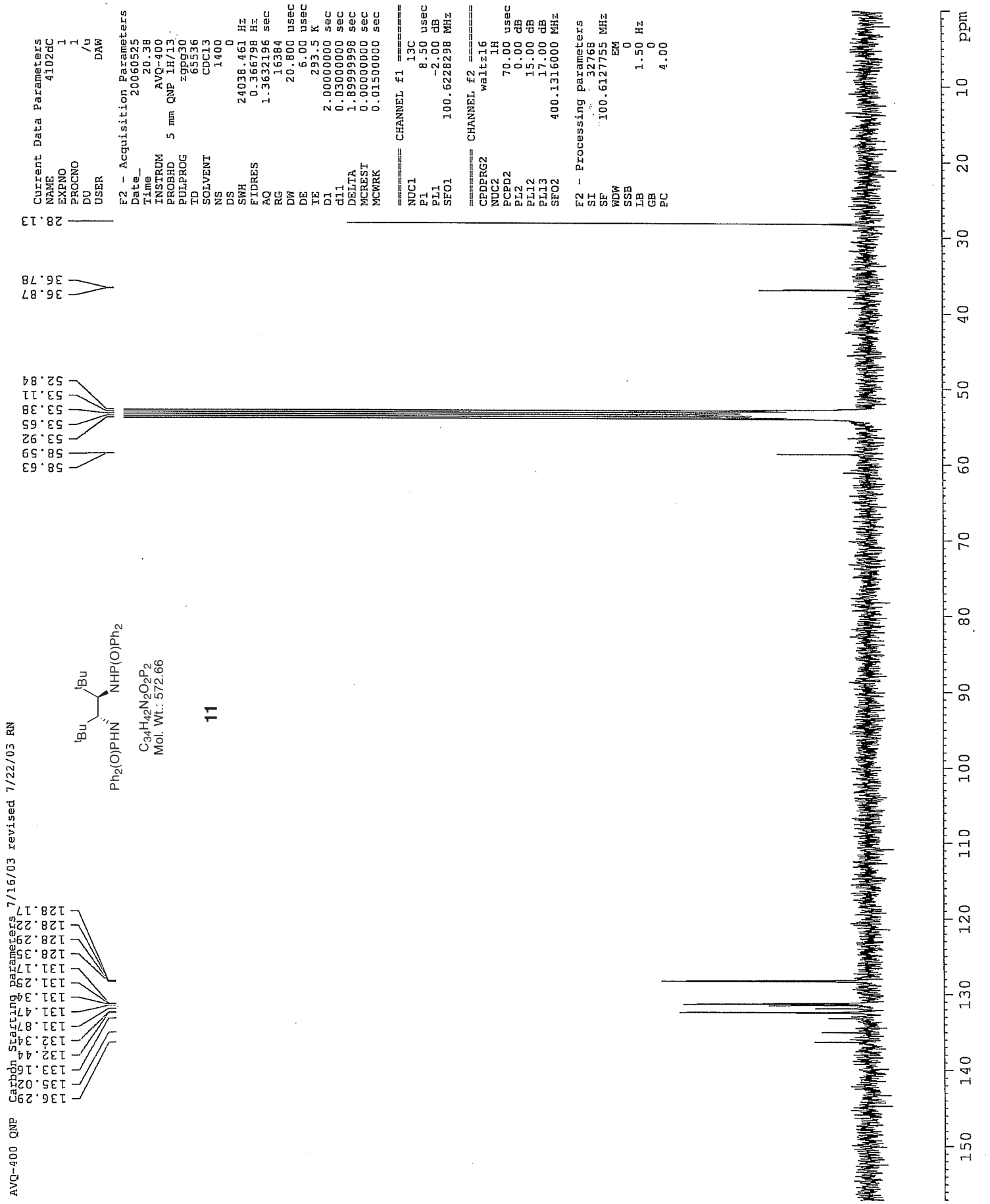

13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
136.97
132.18
132.00
131.50
131.43
130.77
128.36
128.30
128.26
128.17
53.81
53.60
53.38
53.16
52.95
21.36
20.32
18.49
Current Data Parameters
NAME 4108aC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060527
Time 21.47
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 151
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
======== CHANNEL f2 ========
Ar Ar
Ph2(O)PHN NHP(O)Ph2
Me
Ar =
Me
Me
C44H46N2O2P2
Mol. Wt.: 696.80
10
137 136 135 134 133 132 131 130 129 ppm
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

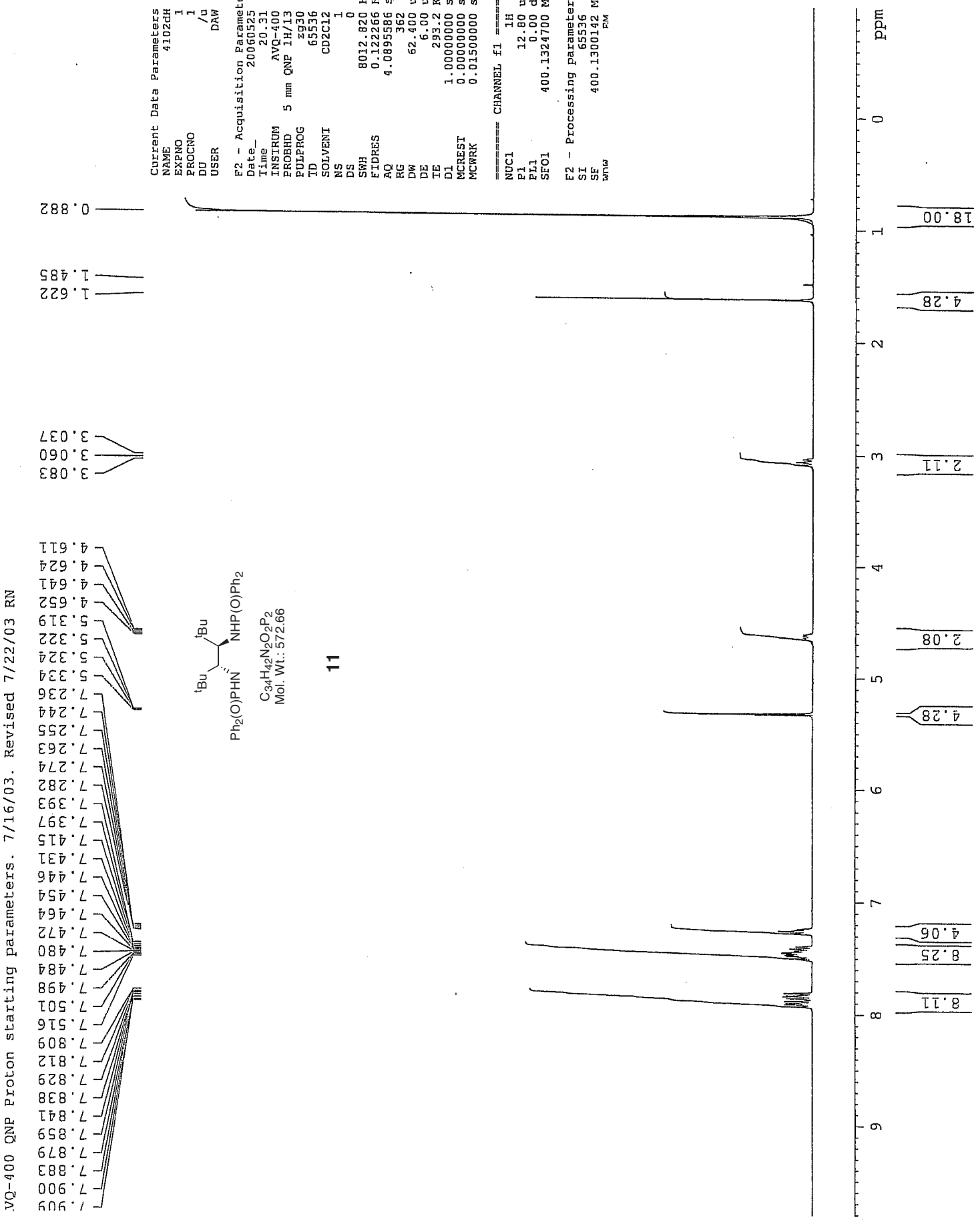
1H starting parameters (zg30)
DRX-500 zBBO probe



$C_{44}H_{46}N_2O_2P_2$
Mol. Wt.: 696.80

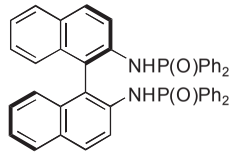
10


AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7/22/03 RN
136.29
135.02
133.16
132.44
132.34
131.87
131.47
131.34
131.25
131.17
128.35
128.29
128.22
128.17
58.63
58.59
53.92
53.65
53.38
53.11
52.84
36.87
36.78
28.13
Current Data Parameters
NAME 4102dC
EXPNO 1
PROCNO 1
DU /u
USER DAW
F2 - Acquisition Parameters
Date_ 20060525
Time 20.38
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1400
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
SFO2 400.1316000 MHz
F2 - Processing parameters
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 4.00
11
$C_{34}H_{42}N_2O_2P_2$
Mol. Wt.: 572.66
$Ph_2(O)PHN$
$NHP(O)Ph_2$
^tBu
^tBu
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


VQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN
Current Data Parameters
NAME 4102dH
EXPNO 1
PROCNO 1
DU /u
USER DAW
F2 - Acquisition Parameters
Date_ 20060525
Time_ 20.31
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 362
DW 62.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
tBu
tBu
NHP(O)Ph2
Ph2(O)PHN
C34H42N2O2P2
Mol. Wt.: 572.66
11
ppm
0.882
1.485
1.622
3.037
3.060
3.083
4.611
4.624
4.641
4.652
5.319
5.322
5.324
5.334
7.236
7.244
7.255
7.263
7.274
7.282
7.393
7.397
7.415
7.431
7.446
7.454
7.464
7.472
7.480
7.484
7.498
7.501
7.516
7.809
7.812
7.829
7.838
7.841
7.859
7.879
7.883
7.900
18.00
4.28
2.11
2.08
4.28
4.06
8.25
8.11

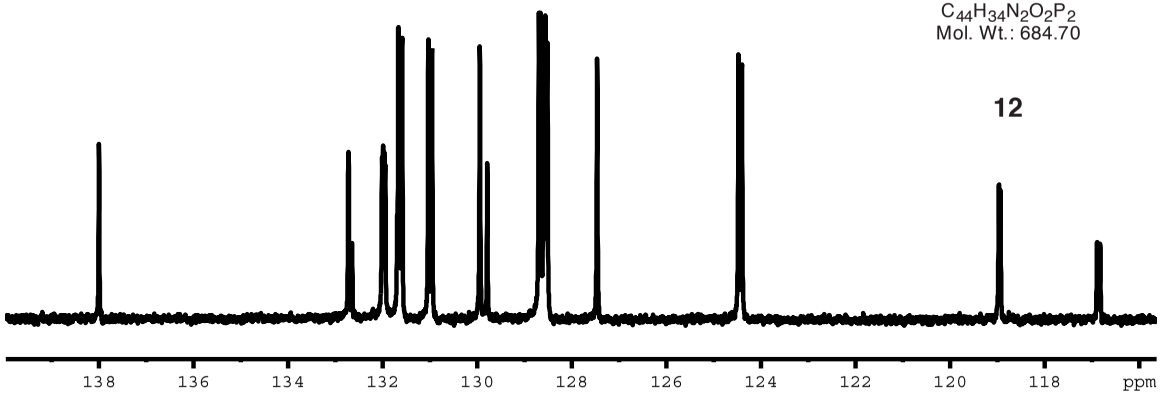
13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0

124.47
124.40
118.96
118.93
116.89
116.82

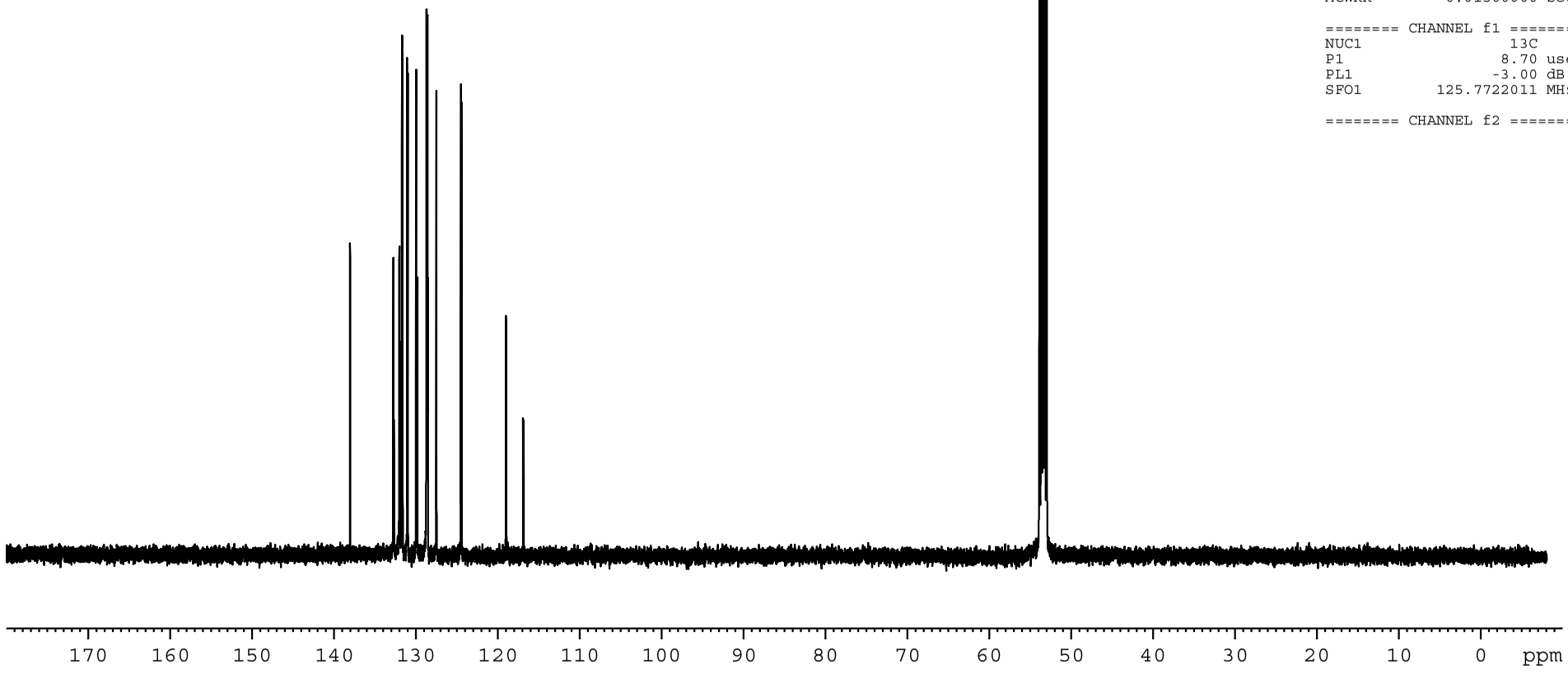


$C_{44}H_{34}N_2O_2P_2$
Mol. Wt.: 684.70

12

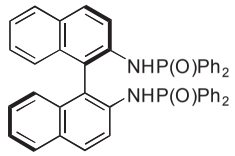



```
Current Data Parameters
NAME                  3064C
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060607
Time                   0.09
INSTRUM             DRX-500
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                  131072
SOLVENT               CDCl3
NS                     6842
DS                        0
SWH              30864.197 Hz
FIDRES           0.235475 Hz
AQ               2.1234164 sec
RG                    16384
DW                 16.200 usec
DE                  5.00 usec
TE                  293.0 K
D1             1.50000000 sec
d11            0.03000000 sec
DELTA          1.39999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   8.70 usec
PL1                 -3.00 dB
SFO1          125.7722011 MHz

======== CHANNEL f2 ========
```



1H starting parameters (zg30)
DRX-500 zBBO probe

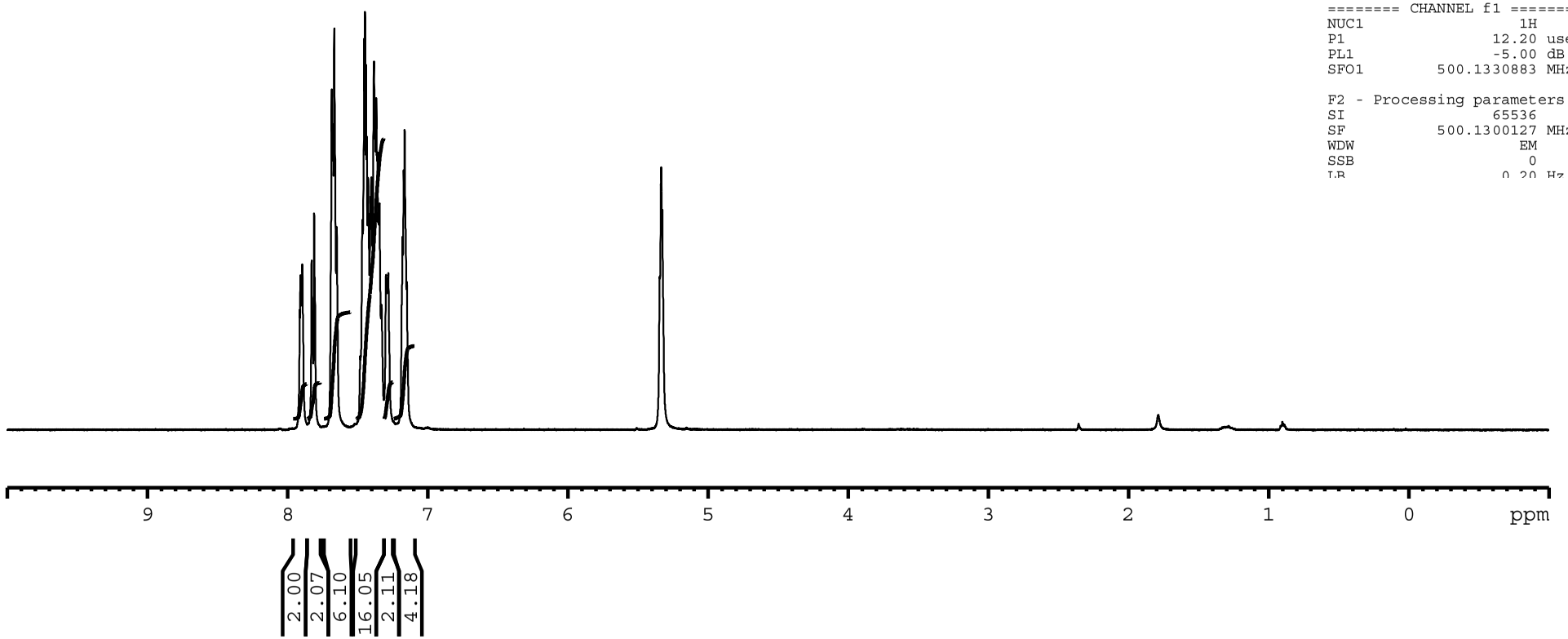
7.896 7.832 7.815 7.685 7.678 7.675 7.667 7.653 7.650 7.468 7.456 7.448 7.441 7.436 7.426 7.410 7.402 7.385 7.375 7.368 7.361 7.353 7.346 7.333 7.330 7.299 7.293 7.282 7.181 7.172 7.166 7.150



$C_{44}H_{34}N_2O_2P_2$
Mol. Wt.: 684.70

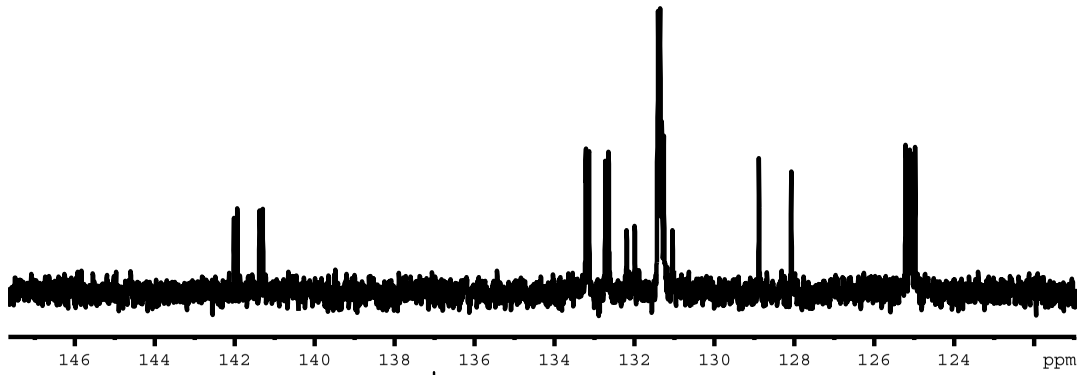
12

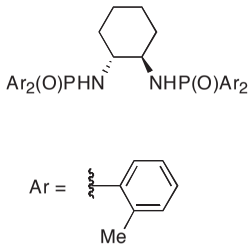
Current Data Parameters	
NAME	3064H
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20060607
Time	0.07
INSTRUM	DRX-500
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	256
DW	50.000 use
DE	7.11 use
TE	293.0 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	12.20 use
PL1	-5.00 dB
SFO1	500.1330883 MHz
F2 - Processing parameters	
SI	65536
SF	500.1300127 MHz
WDW	EM
SSB	0
LB	0.20 Hz



13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0

133.22
133.14
132.73
132.65
131.41
131.36
131.27

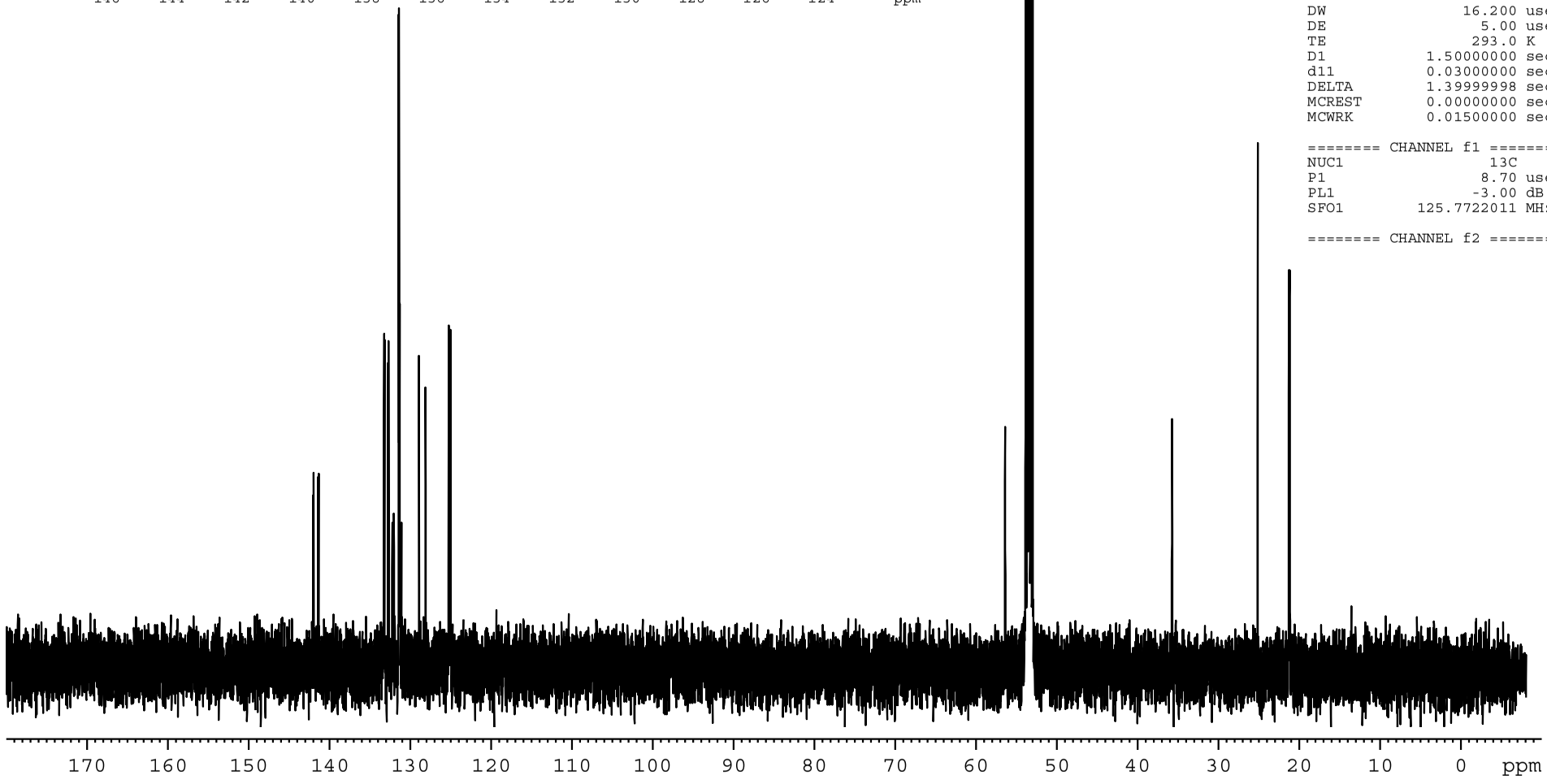


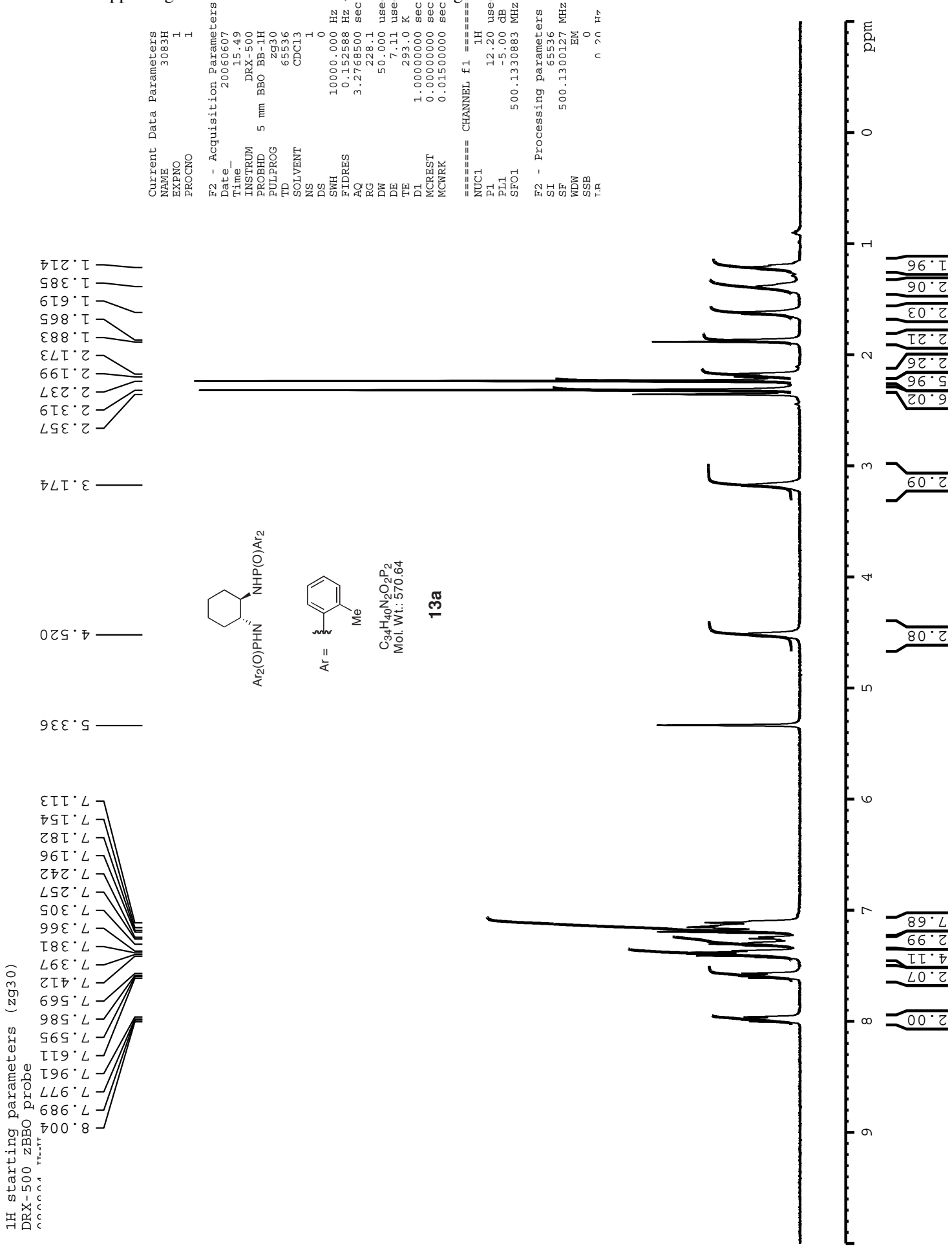


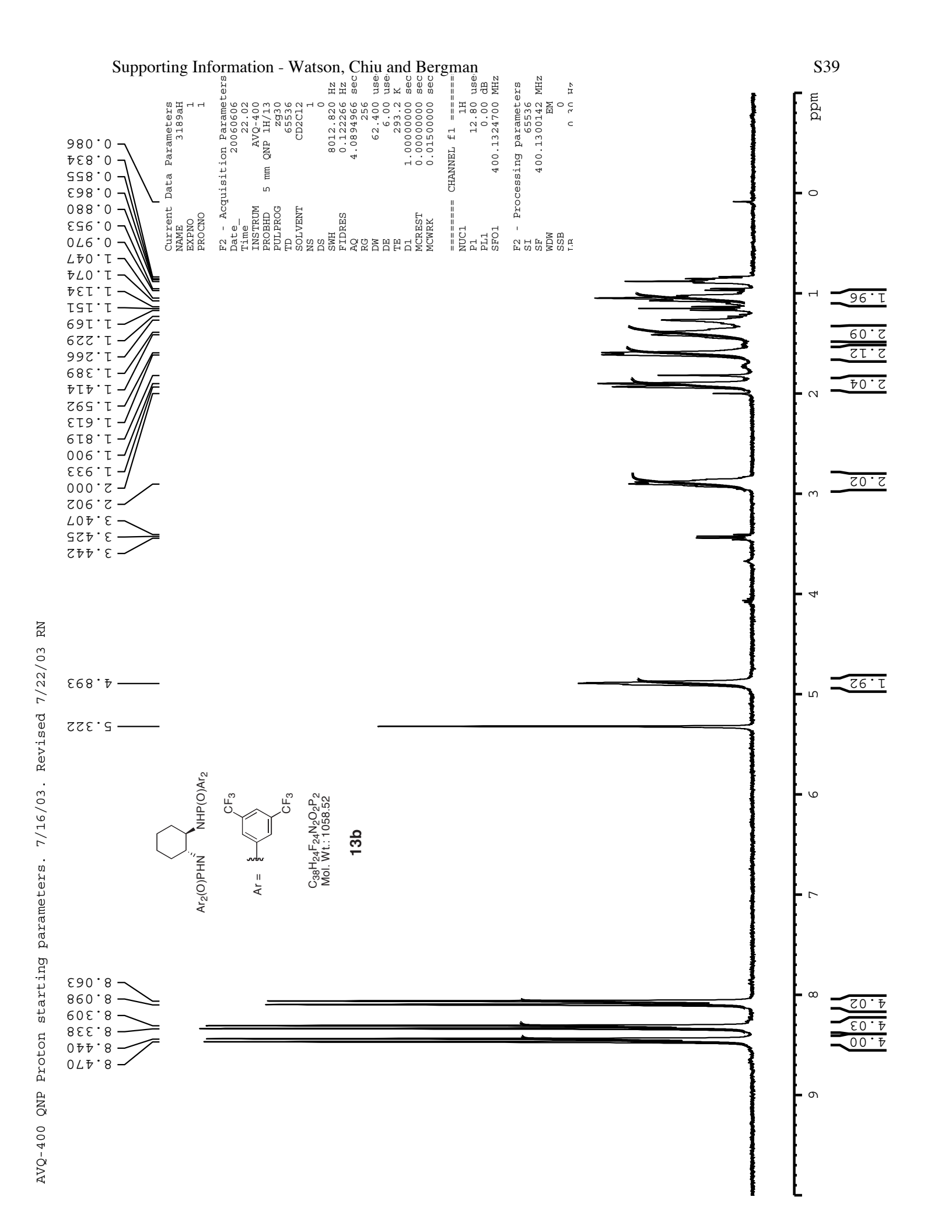
$C_{34}H_{40}N_2O_2P_2$
Mol. Wt.: 570.64

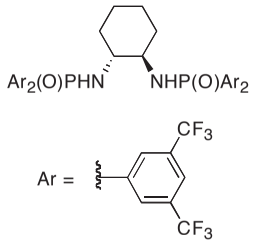
13a

```
Current Data Parameters
NAME                  3083C
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060607
Time                 15.52
INSTRUM             DRX-500
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                  131072
SOLVENT              CDCl3
NS                     577
DS                       0
SWH              30864.197 Hz
FIDRES           0.235475 Hz
AQ               2.1234164 sec
RG                   16384
DW                 16.200 usec
DE                  5.00 usec
TE                  293.0 K
D1             1.50000000 sec
d11            0.03000000 sec
DELTA          1.39999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  8.70 usec
PL1                -3.00 dB
SFO1          125.7722011 MHz

======== CHANNEL f2 ========
```




1H starting parameters (zg30)
DRX-500 zBBO probe
Current Data Parameters
NAME 3083H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060607
Time_ 15.49
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 228.1
DW 50.000 use
DE 7.11 use
TE 293.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 use
PL1 -5.00 dB
SFO1 500.1330883 MHz
F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
NHP(O)Ar2
Ar2(O)PHN
Ar =
Me
C34H40N2O2P2
Mol. Wt.: 570.64
13a
ppm


AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN
Current Data Parameters
NAME 3189aH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time_ 22.02
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 use
DE 6.00 use
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
SSB 0
NHP(O)Ar2
Ar2(O)PHN
Ar =
CF3
CF3
C38H24F24N2O2P2
Mol. Wt.:1058.52
13b
ppm



$C_{38}H_{24}F_{24}N_2O_2P_2$
Mol. Wt.: 1058.52

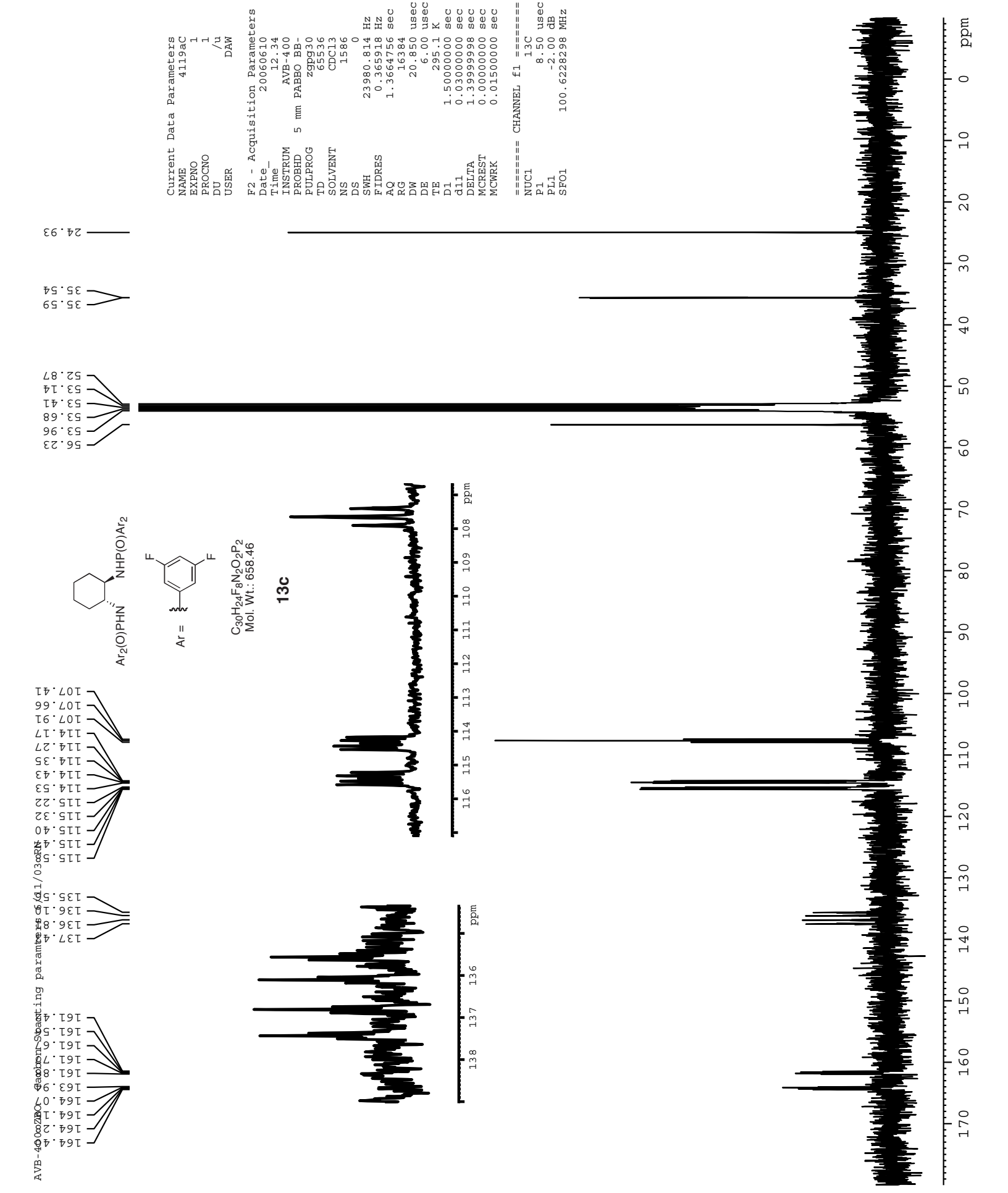
13b

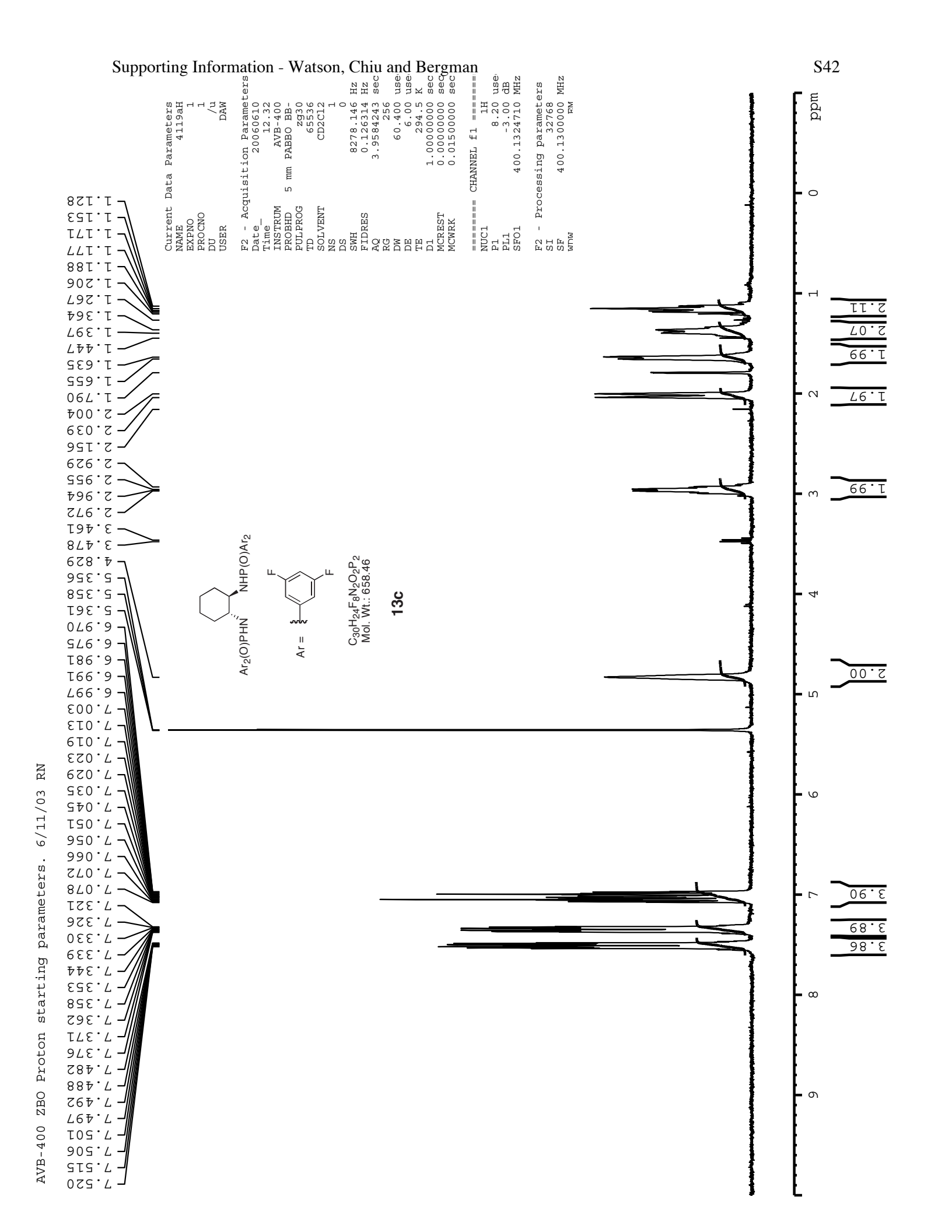
Current Data Parameters
NAME 3189aC
EXPNO 1
PROCNO 1

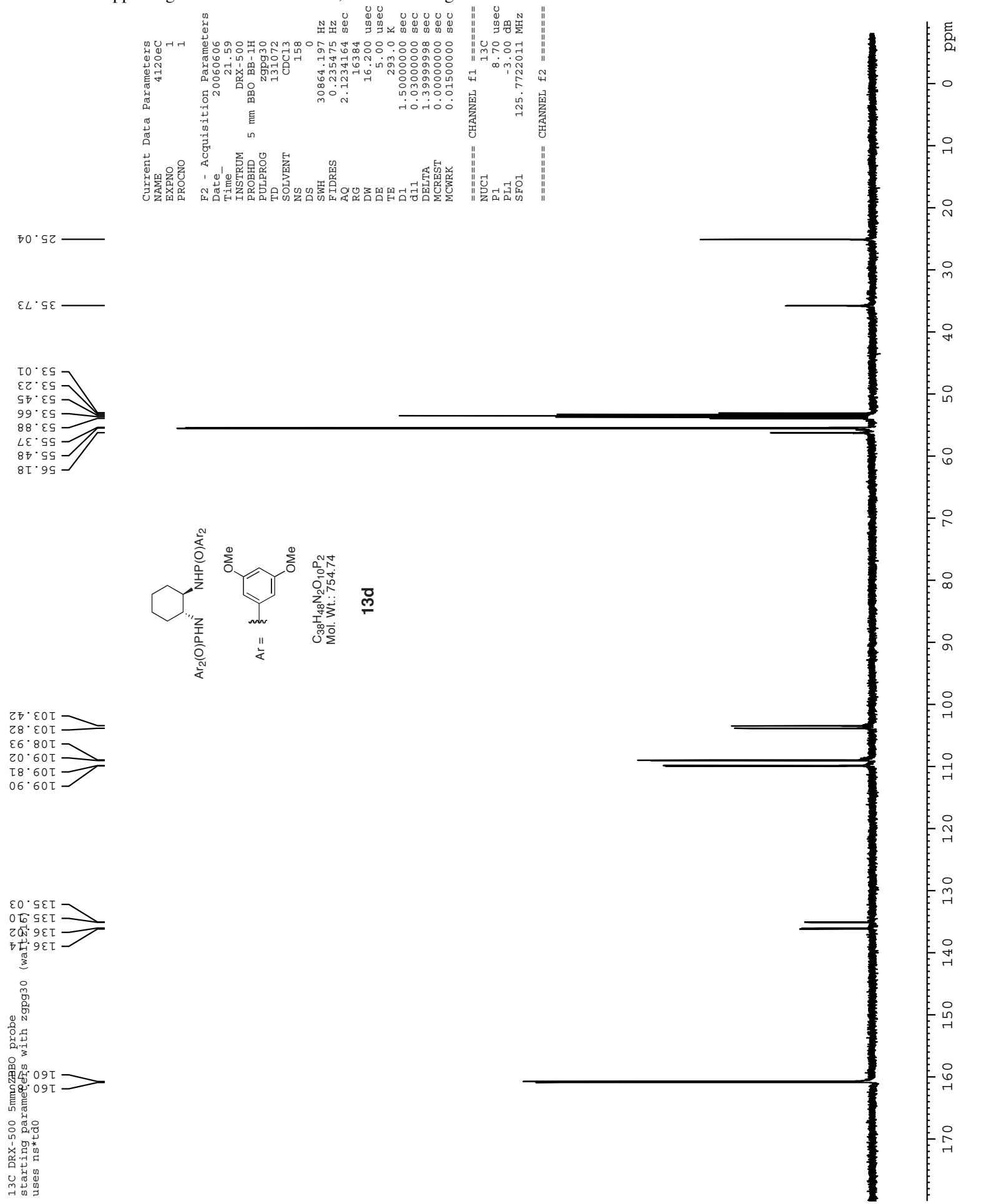
F2 - Acquisition Parameters
Date_ 20060606
Time 22.09
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 829
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

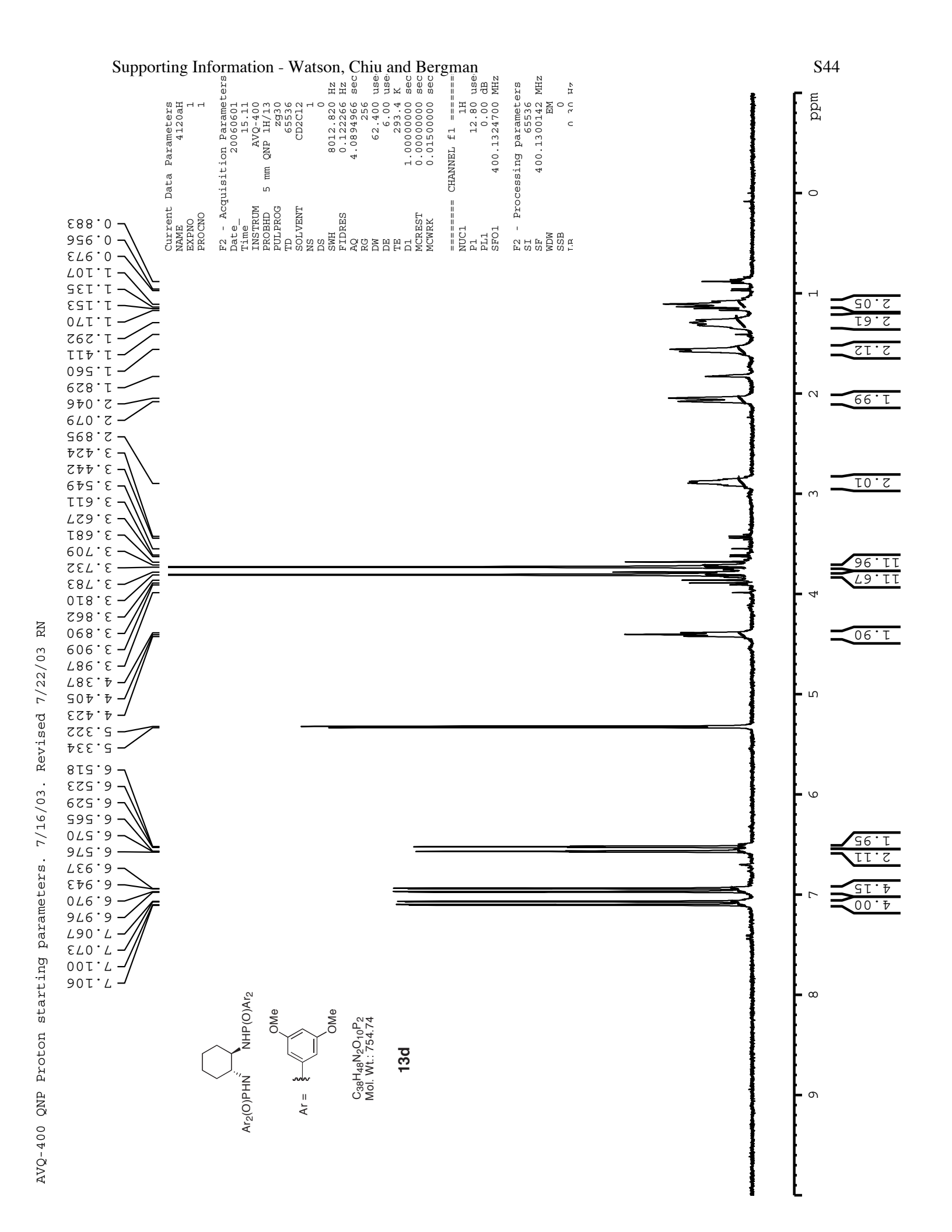
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

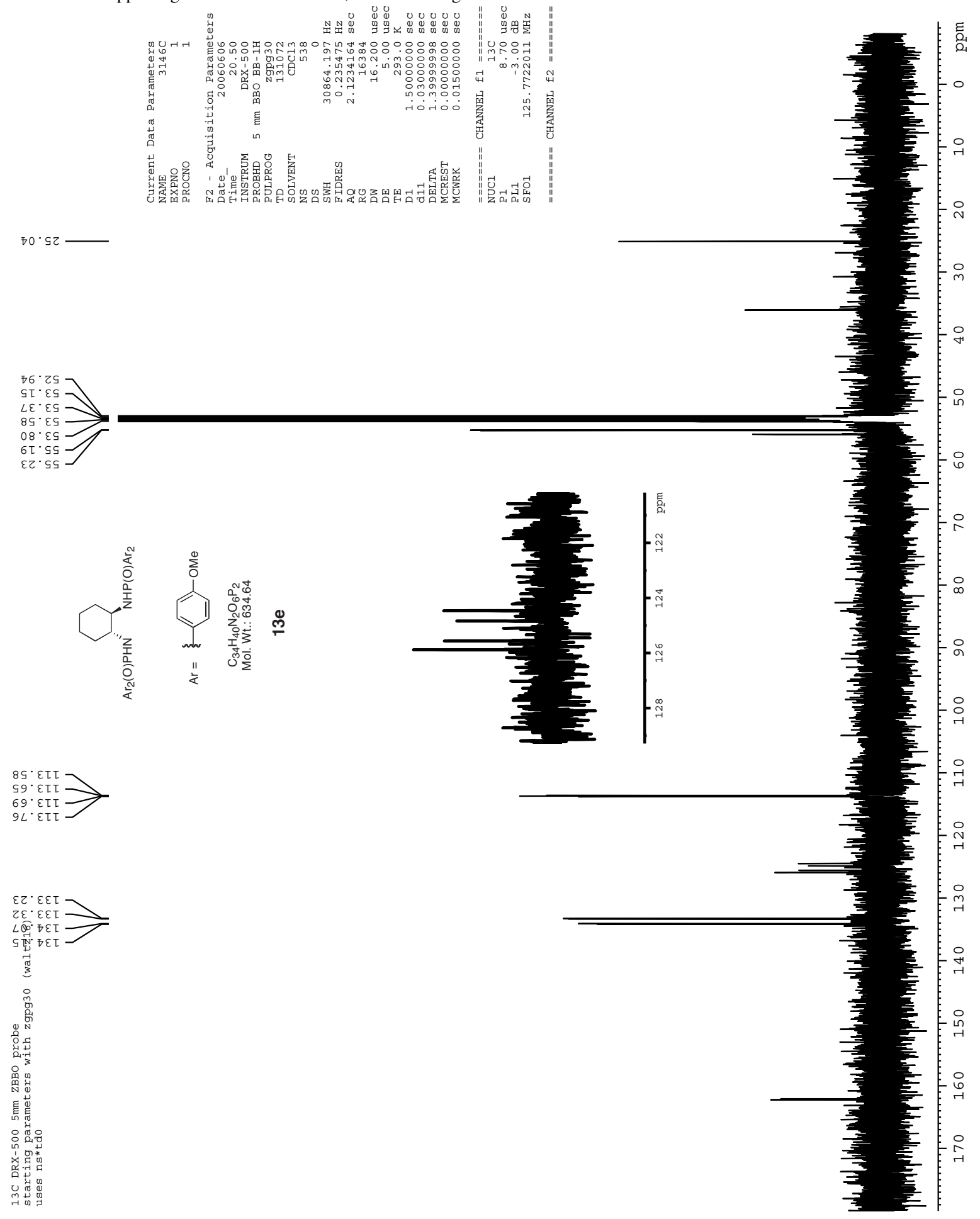
======== CHANNEL f2 ========

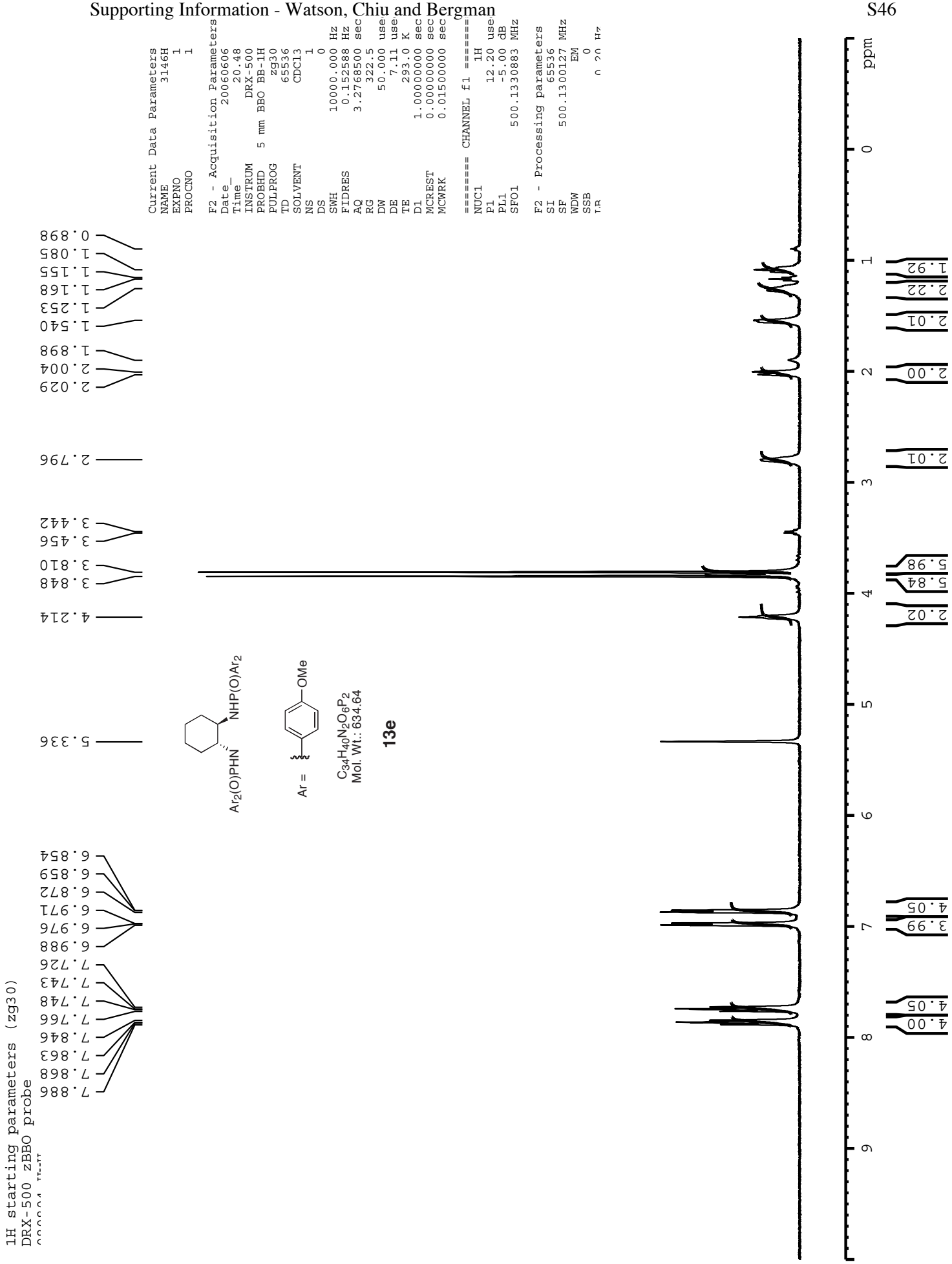

Current Data Parameters
NAME 4119aC
EXPNO 1
PROCNO 1
DU /u
USER DAW
F2 - Acquisition Parameters
Date_ 20060610
Time_ 12.34
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1586
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.36664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.1 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
13c
C30H24F8N2O2P2
Mol. Wt.: 658.46
Ar =
Ar2(O)PHN
NHP(O)Ar2
ppm

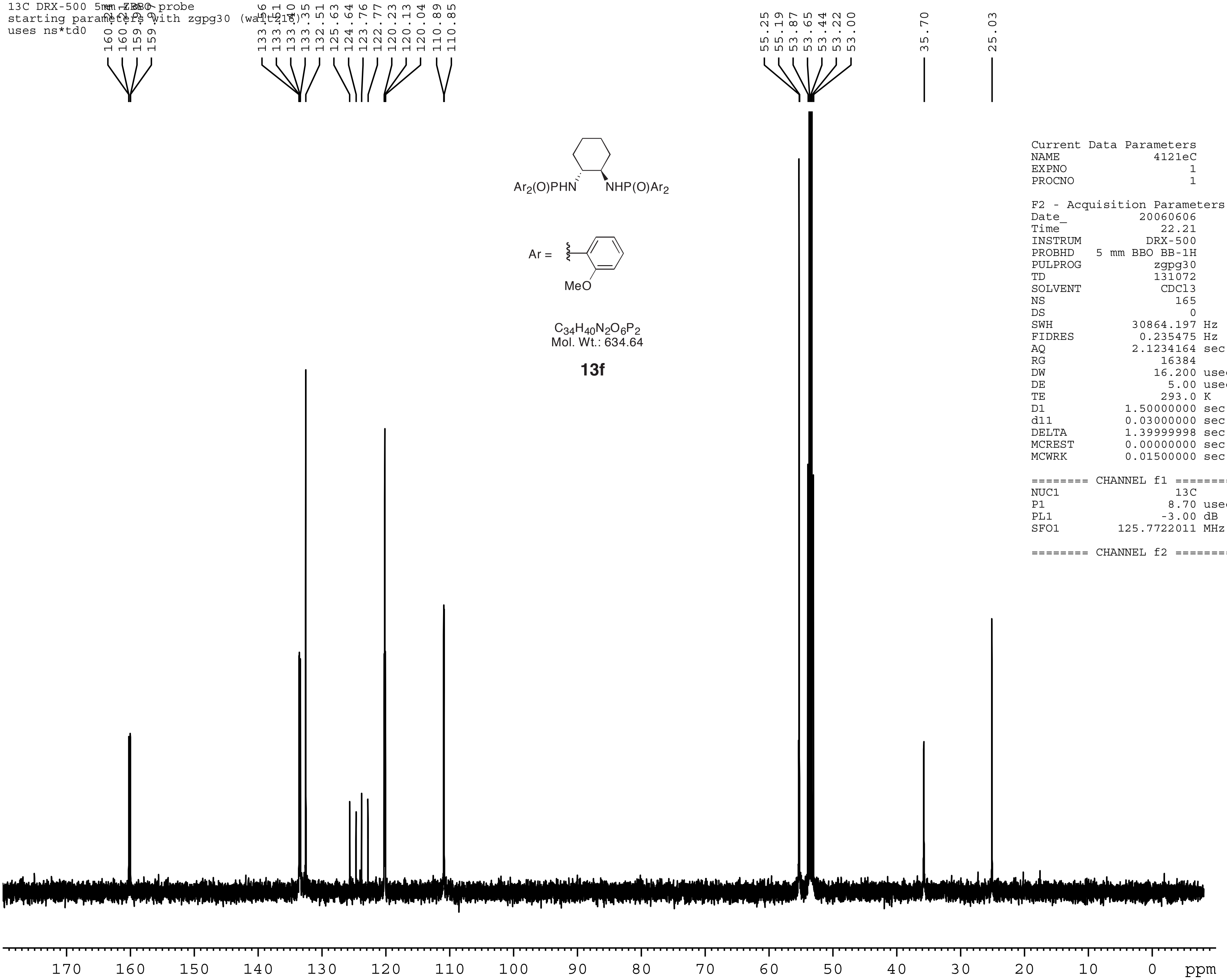

AVB-400 ZBO Proton starting parameters. 6/11/03 RN
Current Data Parameters
NAME 4119aH
EXPNO 1
PROCNO 1
DU /u
USER DAW
F2 - Acquisition Parameters
Date_ 20060610
Time_ 12.32
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 use
DE 6.00 use
TE 294.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 use
PL1 -3.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
NHP(O)Ar2
Ar2(O)PHN
Ar =
F
F
C30H24F8N2O2P2
Mol. Wt.: 658.46
13c
ppm
0
1
2
3
4
5
6
7
8
9
1.99
1.97
1.99
2.00
3.90
3.89
3.86
2.11
2.07
1.99
1.97
1.99
2.00
3.90
3.89
3.86


Current Data Parameters
NAME 4120eC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time 21.59
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 158
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
CHANNEL f2
13C DRX-500 5mm ZBBO probe
starting parameter set with zgpg30 (waltz16)
uses ns*td0
25.04
35.73
53.01
53.23
53.45
53.66
53.88
55.37
55.48
56.18
103.42
103.82
108.93
109.02
109.81
109.90
135.03
135.16
136.24
160.02
160.18
OMe
OMe
Ar =
NHP(O)Ar2
Ar2(O)PHN
C38H48N2O10P2
Mol. Wt.:754.74
13d
ppm


AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN
Current Data Parameters
NAME 4120aH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060601
Time_ 15.11
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 use
DE 6.00 use
TE 293.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
7.106
7.100
7.073
7.067
6.976
6.970
6.943
6.937
6.576
6.570
6.565
6.529
6.523
6.518
5.334
5.322
4.423
4.405
4.387
3.987
3.909
3.890
3.862
3.810
3.783
3.732
3.709
3.681
3.627
3.611
3.549
3.442
3.424
2.895
2.079
2.046
1.829
1.560
1.411
1.292
1.170
1.153
1.135
1.107
0.973
0.956
0.883
ppm
0
1
2
3
4
5
6
7
8
9
4.00
4.15
2.11
1.95
1.90
11.67
11.96
2.01
1.99
2.12
2.61
2.05
NHP(O)Ar2
Ar2(O)PHN
OMe
OMe
Ar =
C38H48N2O10P2
Mol. Wt.: 754.74
13d


13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (walt_4_18)
uses ns*td0
Current Data Parameters
NAME 3146C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time_ 20.50
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 538
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
======== CHANNEL f2 ========
Ar2(O)PHN
NHP(O)Ar2
Ar =
OMe
C34H40N2O6P2
Mol. Wt.: 634.64
13e
ppm

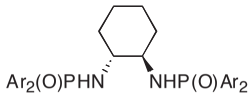

1H starting parameters (zg30)
DRX-500 zBBO probe
Current Data Parameters
NAME 3146H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time_ 20.48
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 322.5
DW 50.000 use
DE 7.11 use
TE 293.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 use
PL1 -5.00 dB
SFO1 500.1330883 MHz
F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.20 Hz
Ar2(O)PHN
NHP(O)Ar2
Ar =
OMe
C34H40N2O6P2
Mol. Wt.: 634.64
13e
ppm

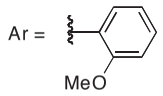

13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
133.56
133.51
133.40
133.35
132.51
125.63
124.64
123.76
122.77
120.23
120.13
120.04
110.89
110.85
55.25
55.19
53.87
53.65
53.44
53.22
53.00
35.70
25.03
Ar2(O)PHN
NHP(O)Ar2
Ar =
MeO
C34H40N2O6P2
Mol. Wt.: 634.64
13f
Current Data Parameters
NAME 4121eC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time 22.21
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 165
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
======== CHANNEL f2 ========
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

1H starting parameters (zg30)
DRX-500 zBBO probe

7.888 7.873 7.860 7.845 7.784 7.769 7.756 7.741 7.436 7.421 7.406 7.383 7.368 7.352 7.042 7.028 6.832 6.755 6.739 6.728 6.103 5.348

4.115 3.727 3.607 3.524 3.502 3.478 3.216 2.639 2.134 2.122 2.041 2.021 1.884 1.572 1.244





$C_{34}H_{40}N_2O_6P_2$
Mol. Wt.: 634.64

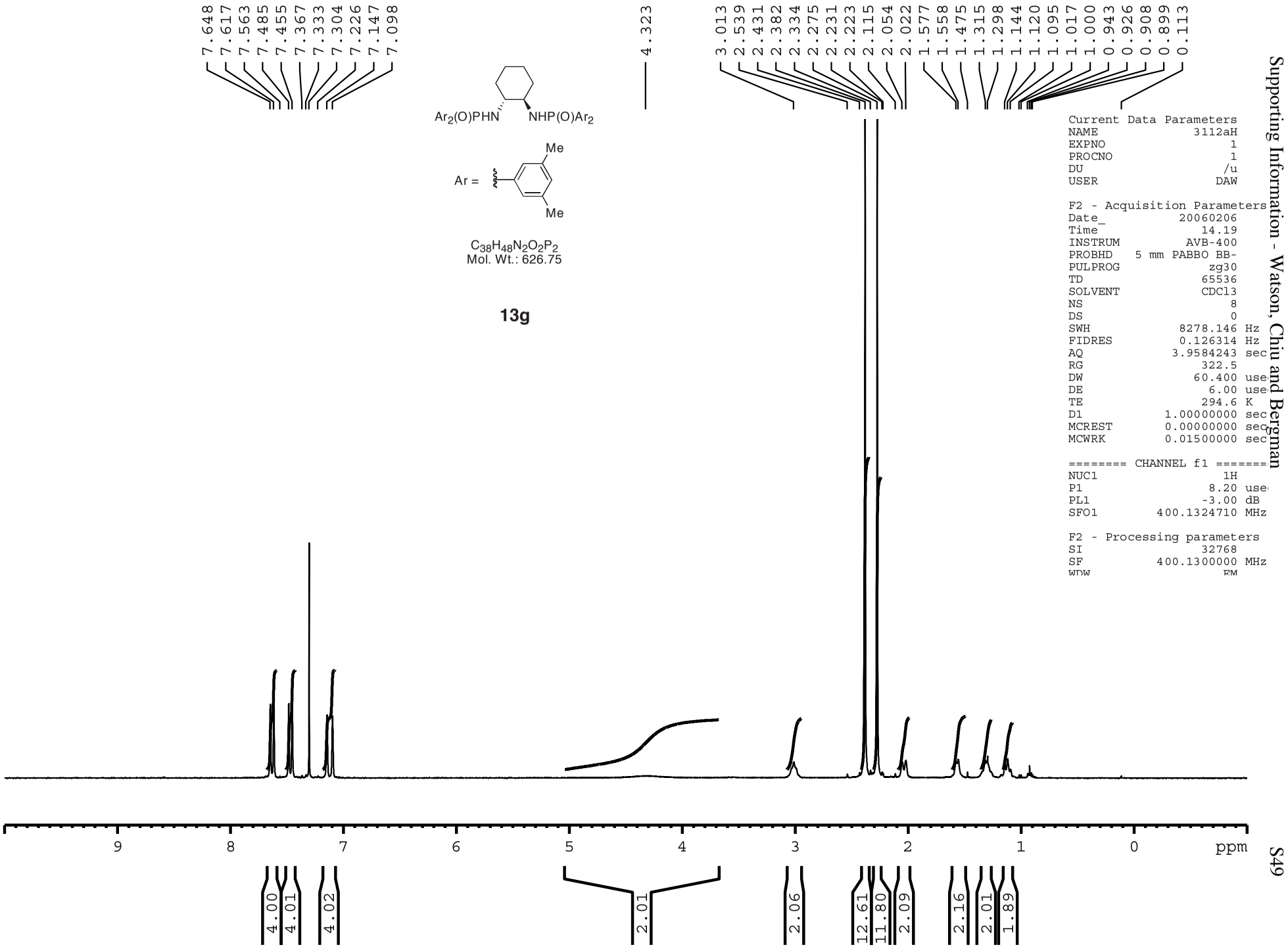
13f

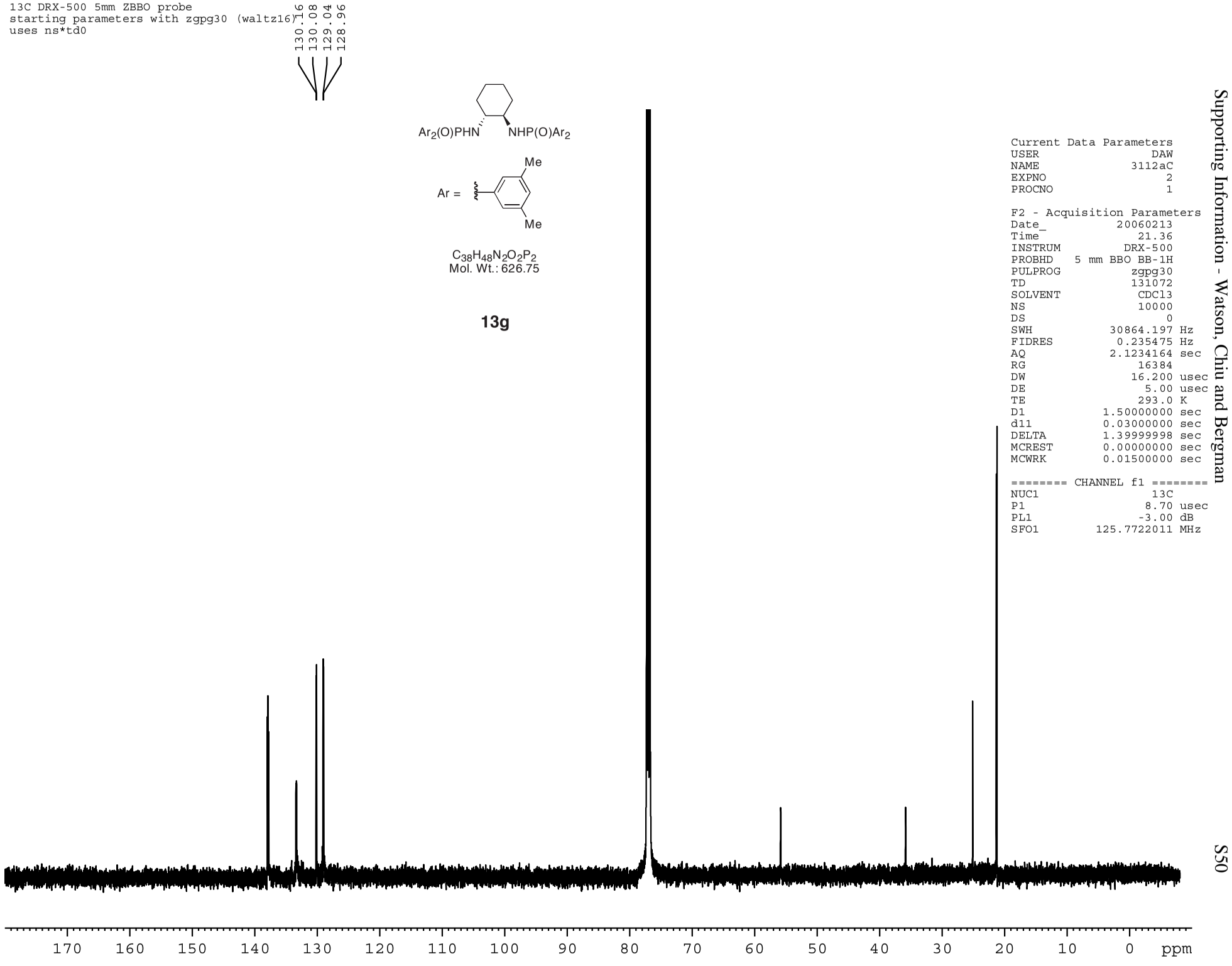
```
Current Data Parameters
NAME                  4121eH
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20060606
Time                  22.19
INSTRUM             DRX-500
PROBHD    5 mm BBO BB-1H
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                         1
DS                         0
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ                3.2768500 sec
RG                      45.3
DW                   50.000 use
DE                    7.11 use
TE                   293.0 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    12.20 use
PL1                   -5.00 dB
SFO1          500.1330883 MHz

F2 - Processing parameters
SI                        65536
SF            500.1300127 MHz
WDW                        EM
SSB                         0
LB                       0.20 Hz
```

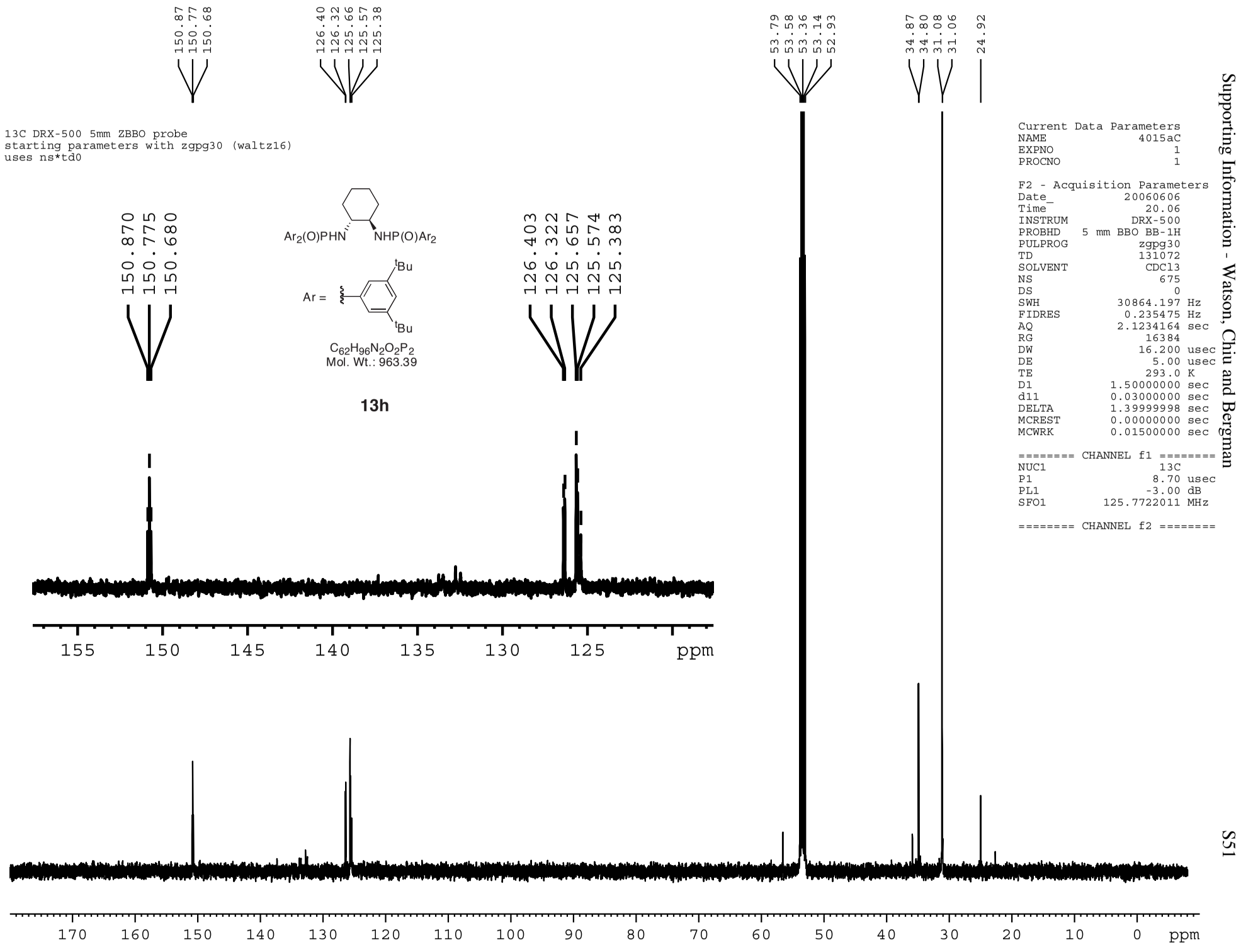
9 8 7 6 5 4 3 2 1 0 ppm

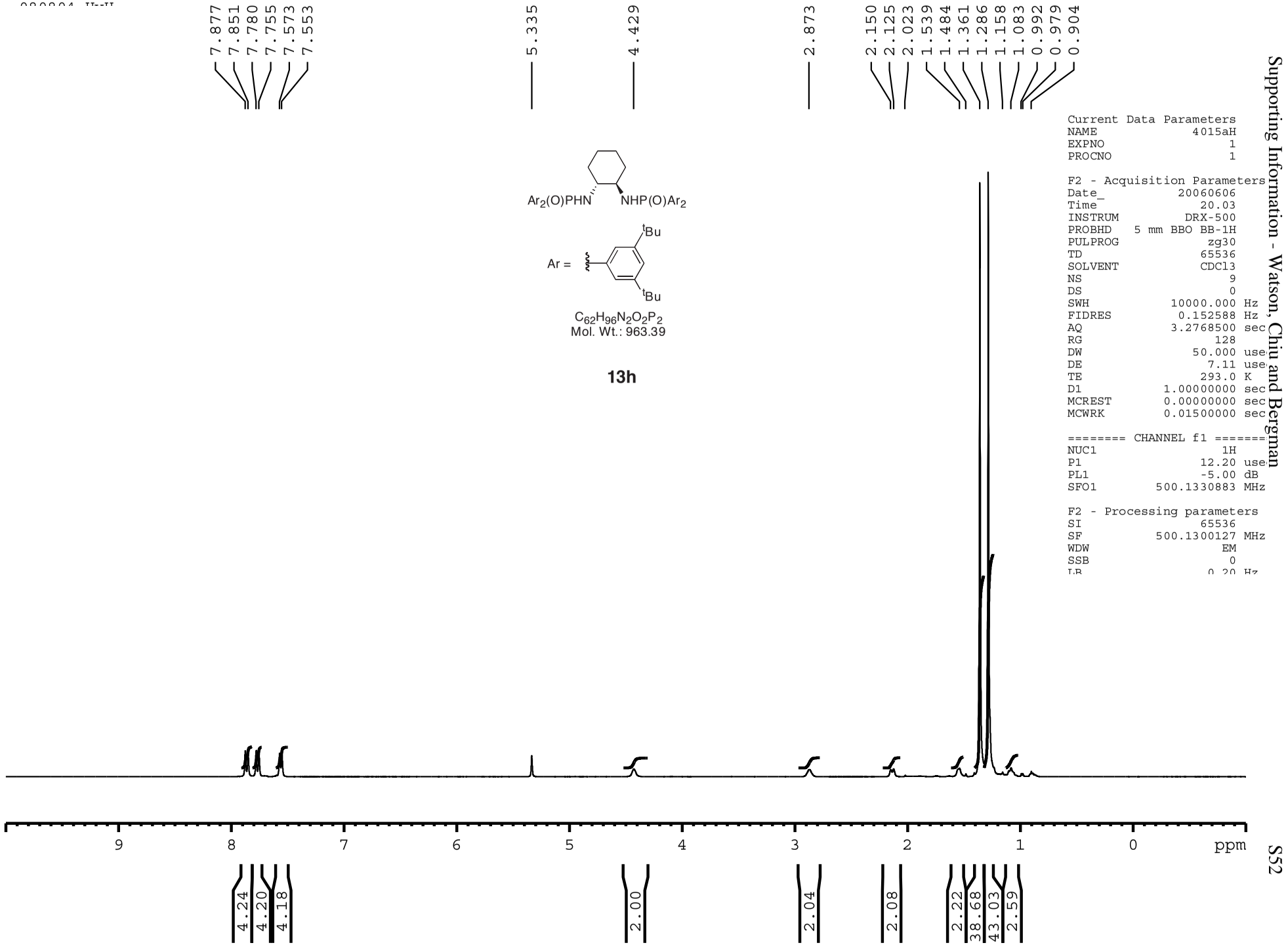
1.99 2.00 2.04 2.05 2.09 4.00 2.06 1.80 6.04 5.90 2.03 1.95 2.08 4.28

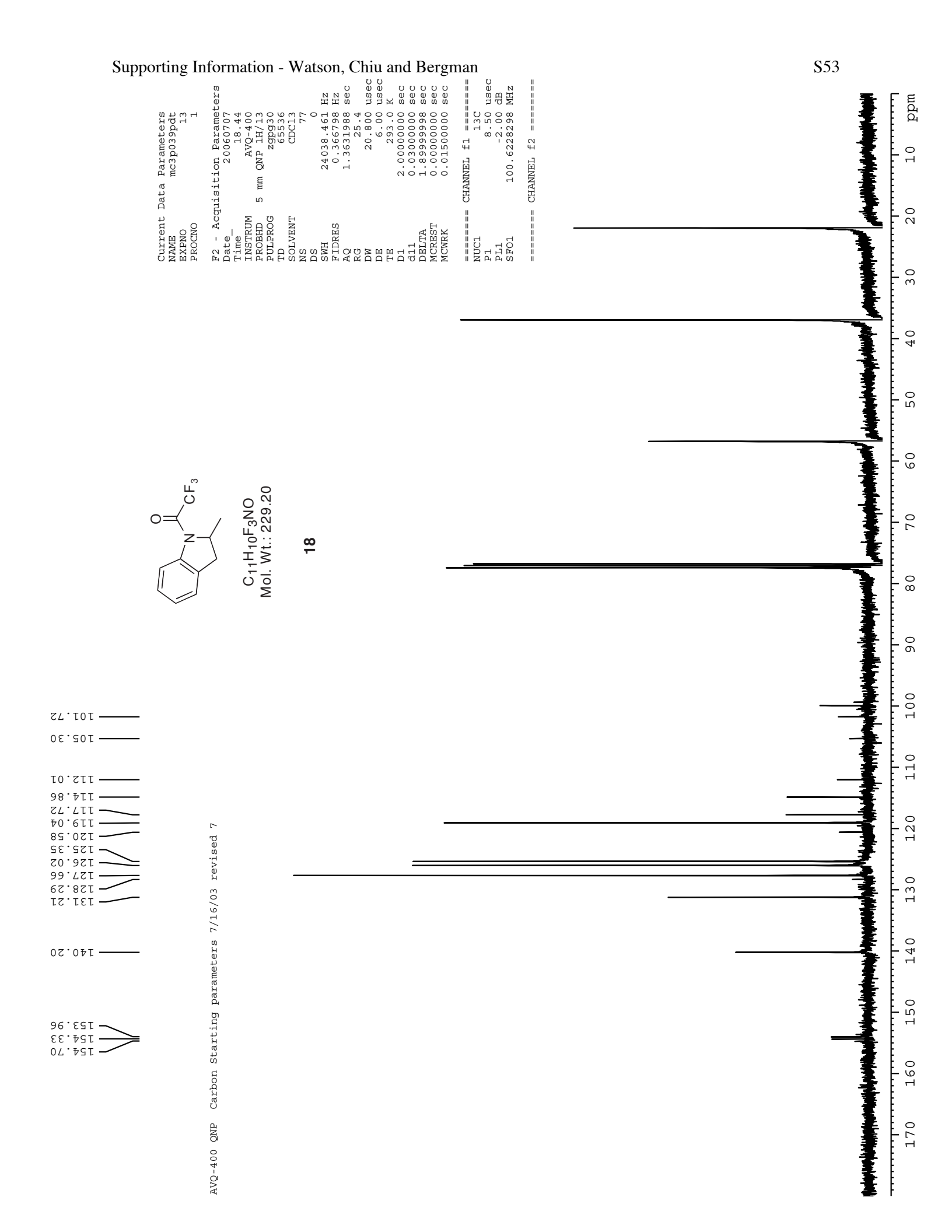
AVB-400 ZBO Proton starting parameters. 6/11/03 RN



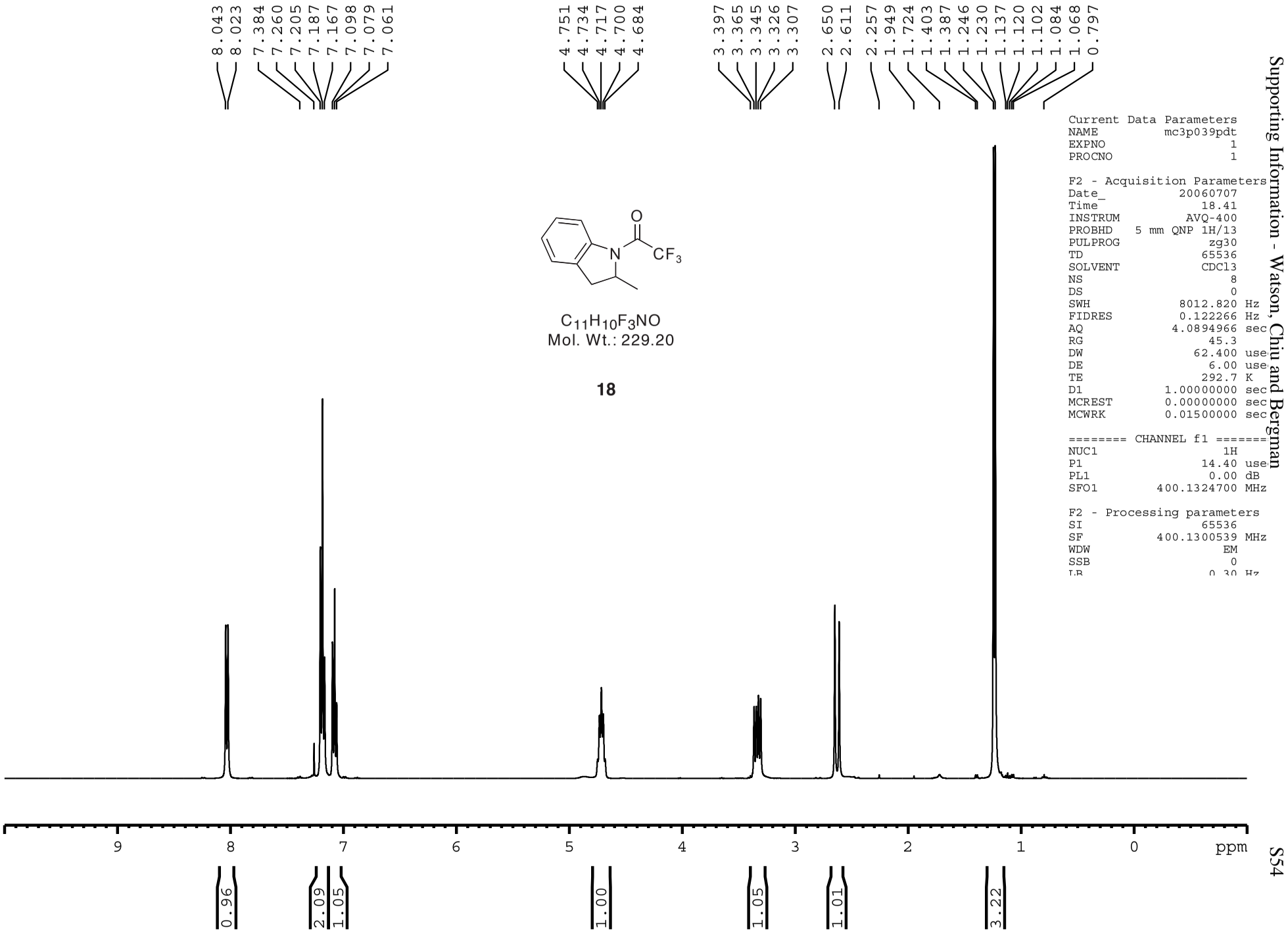

13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
130.16
130.08
129.04
128.96
Ar2(O)PHN
NHP(O)Ar2
Ar =
Me
Me
C38H48N2O2P2
Mol. Wt.: 626.75
13g
Current Data Parameters
USER DAW
NAME 3112aC
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060213
Time 21.36
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 10000
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

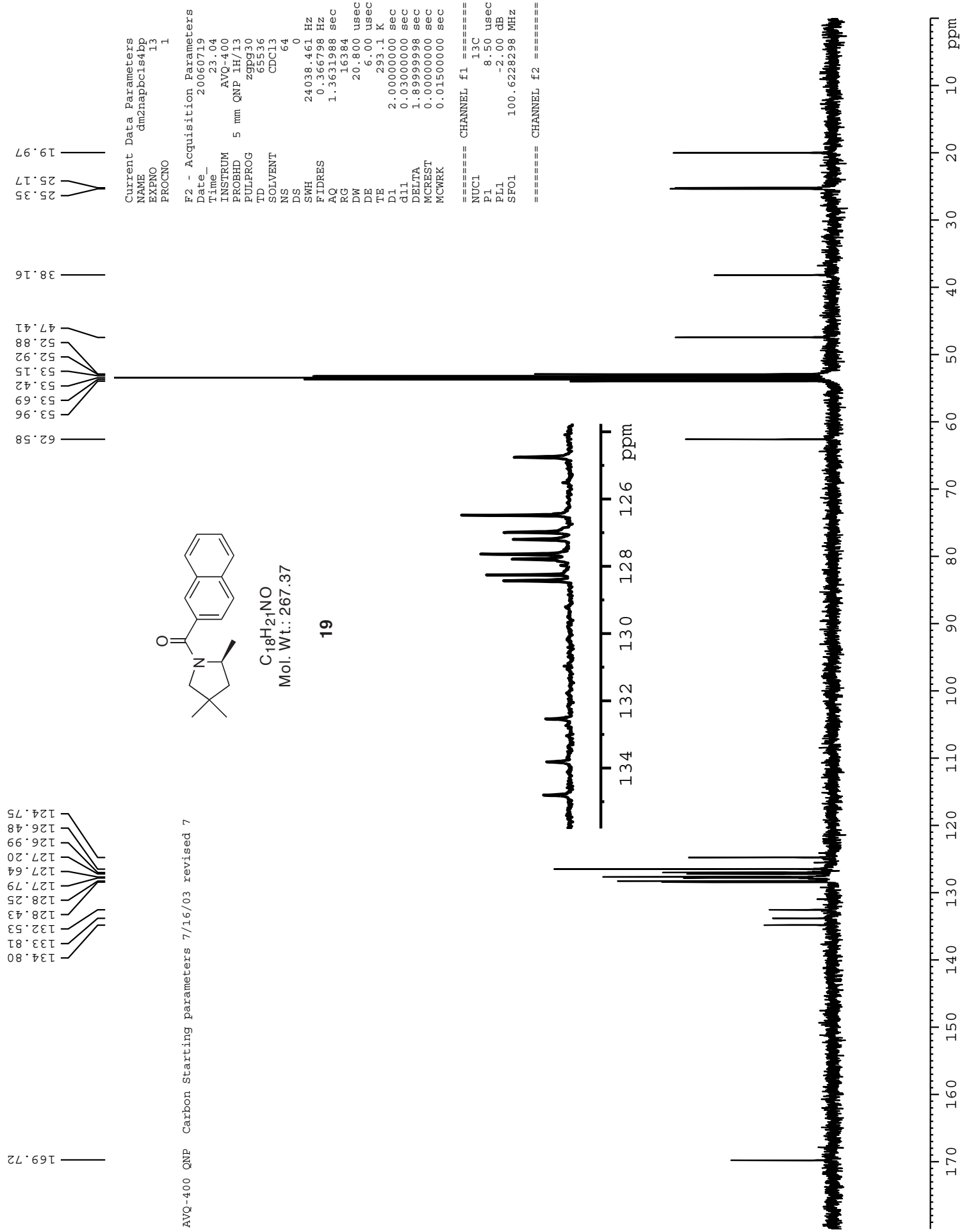

150.87
150.77
150.68
126.40
126.32
125.66
125.57
125.38
53.79
53.58
53.36
53.14
52.93
34.87
34.80
31.08
31.06
24.92
13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
150.870
150.775
150.680
126.403
126.322
125.657
125.574
125.383
Ar2(O)PHN
NHP(O)Ar2
Ar =
tBu
tBu
C62H96N2O2P2
Mol. Wt.: 963.39
13h
Current Data Parameters
NAME 4015aC
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time 20.06
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 675
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz
======== CHANNEL f2 ========
155 150 145 140 135 130 125 ppm
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


1H starting parameters (zg30)
DRX-500 zBBO probe
7.877
7.851
7.780
7.755
7.573
7.553
5.335
4.429
2.873
2.150
2.125
2.023
1.539
1.484
1.361
1.286
1.158
1.083
0.992
0.979
0.904
Current Data Parameters
NAME 4015aH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060606
Time 20.03
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 128
DW 50.000 use
DE 7.11 use
TE 293.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 use
PL1 -5.00 dB
SFO1 500.1330883 MHz
F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.20 Hz
Ar2(O)PHN
NHP(O)Ar2
Ar =
tBu
tBu
C62H96N2O2P2
Mol. Wt.: 963.39
13h
9
8
7
6
5
4
3
2
1
0
ppm
4.24
4.20
4.18
2.00
2.04
2.08
2.22
38.68
43.03
2.59

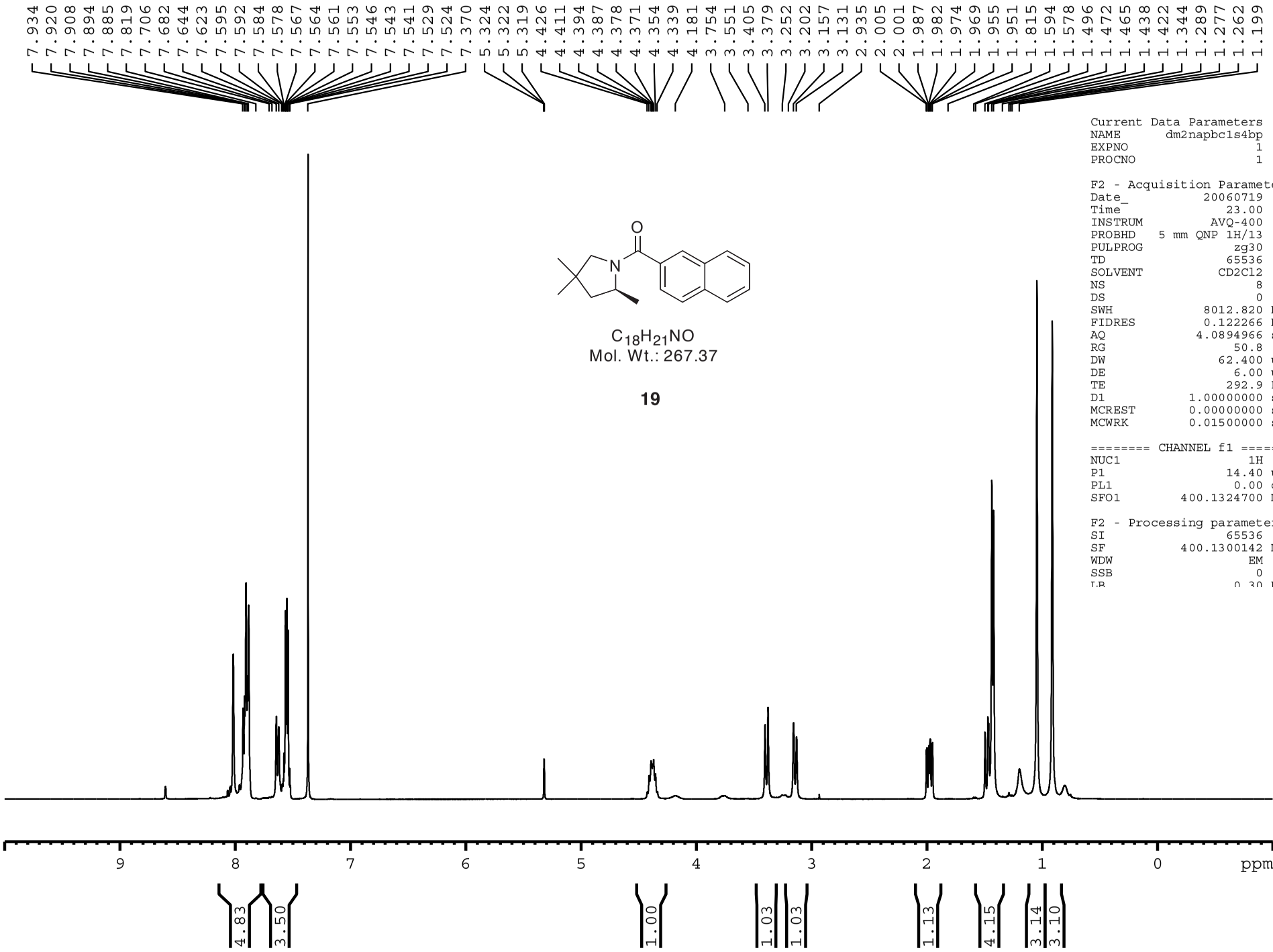

Current Data Parameters
NAME mc3p039pdt
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060707
Time_ 18.44
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 77
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 25.4
DW 20.800 usec
DE 6.00 usec
TE 293.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
O
CF3
N
C11H10F3NO
Mol. Wt.: 229.20
18
AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7
154.70
154.33
153.96
140.20
131.21
128.29
127.66
126.02
125.35
120.58
119.04
117.72
114.86
112.01
105.30
101.72
ppm

AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN

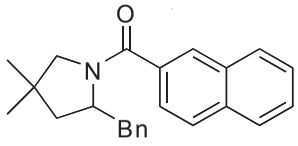



AVQ-400 QNP Carbon Starting parameters 7/16/03 revised 7
Current Data Parameters
NAME dm2napbc1s4bp
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060719
Time_ 23.04
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 64
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
C18H21NO
Mol. Wt.: 267.37
19
ppm
ppm

AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
012504 HBH



$C_{24}H_{25}NO$
Mol. Wt.: 343.46

20

Current Data Parameters

NAME	bn2napc2s3bp3
EXPNO	13
PROCNO	1
DU	/u
USER	labrat

F2 - Acquisition Parameters

Date_	20060722
Time	16.28
INSTRUM	DRX-500
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	131072
SOLVENT	CDCl3
NS	608
DS	0
SWH	30864.197 Hz
FIDRES	0.235475 Hz
AQ	2.1234164 sec
RG	16384
DW	16.200 usec
DE	5.00 usec
TE	293.0 K
D1	1.50000000 sec
d11	0.03000000 sec
DELTA	1.39999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

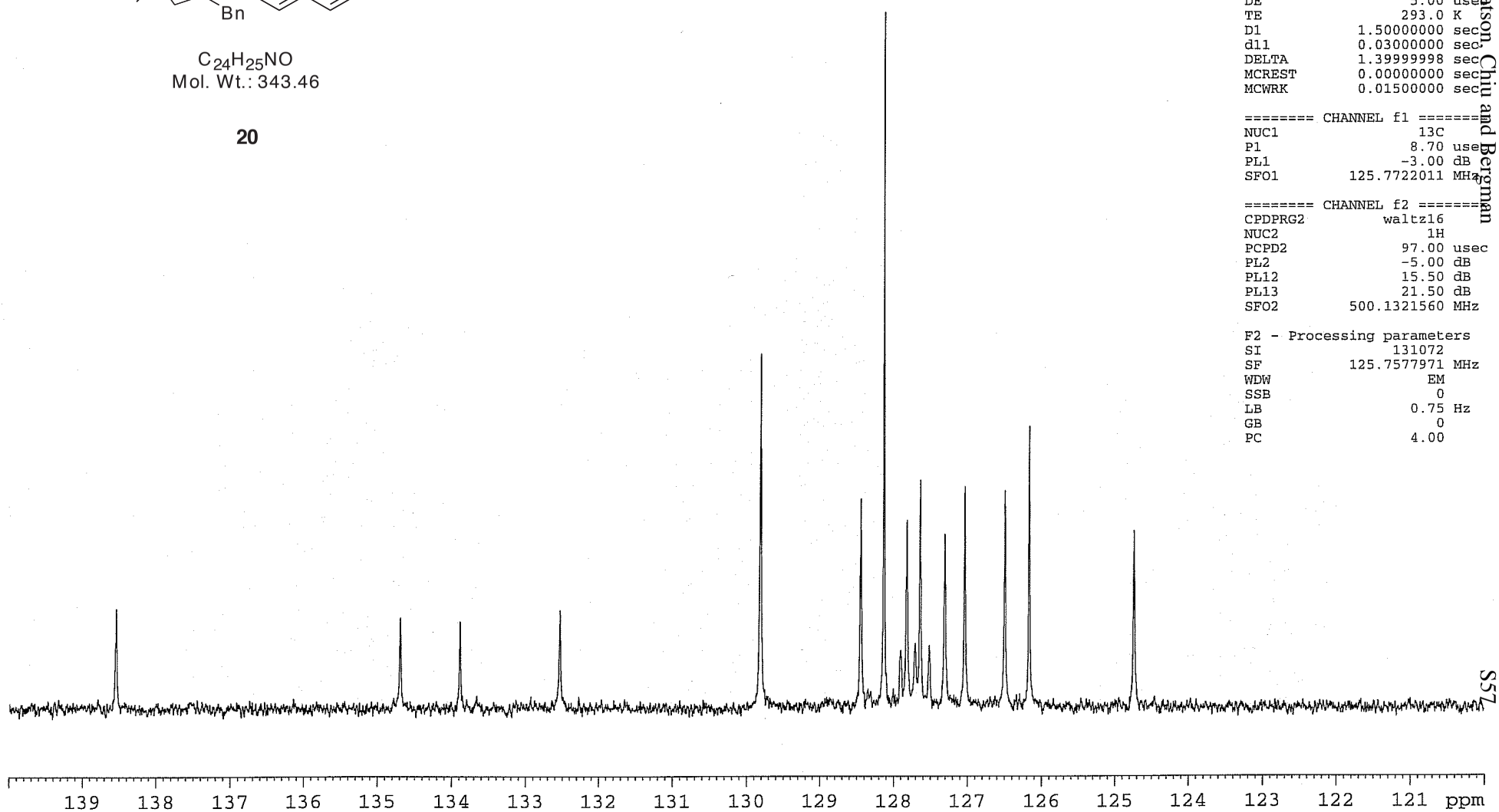
NUC1	13C
P1	8.70 usec
PL1	-3.00 dB
SFO1	125.7722011 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	97.00 usec
PL2	-5.00 dB
PL12	15.50 dB
PL13	21.50 dB
SFO2	500.1321560 MHz

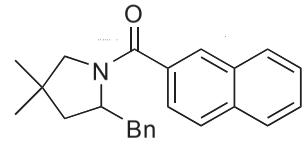
F2 - Processing parameters

SI	131072
SF	125.7577971 MHz
WDW	EM
SSB	0
LB	0.75 Hz
GB	0
PC	4.00



13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0
012504 HvH

169.82
138.55
134.70
133.89
132.54
129.82
128.45
128.13
127.82
127.64
127.52
127.31
127.04
126.50
126.17
124.74
63.14
57.86
53.80
53.58
53.36
53.15
52.93
43.92
39.00
37.91
25.11
25.17



$C_{24}H_{25}NO$
Mol. Wt.: 343.46

20

Current Data Parameters
NAME bn2napc2s3bp3
EXPNO 13
PROCNO 1
DU /u
USER labrat

F2 - Acquisition Parameters
Date_ 20060722
Time 16.28
INSTRUM DRX-500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 592
DS 0
SWH 30864.197 Hz
FIDRES 0.235475 Hz
AQ 2.1234164 sec
RG 16384
DW 16.200 usec
DE 5.00 usec
TE 293.0 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.39999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

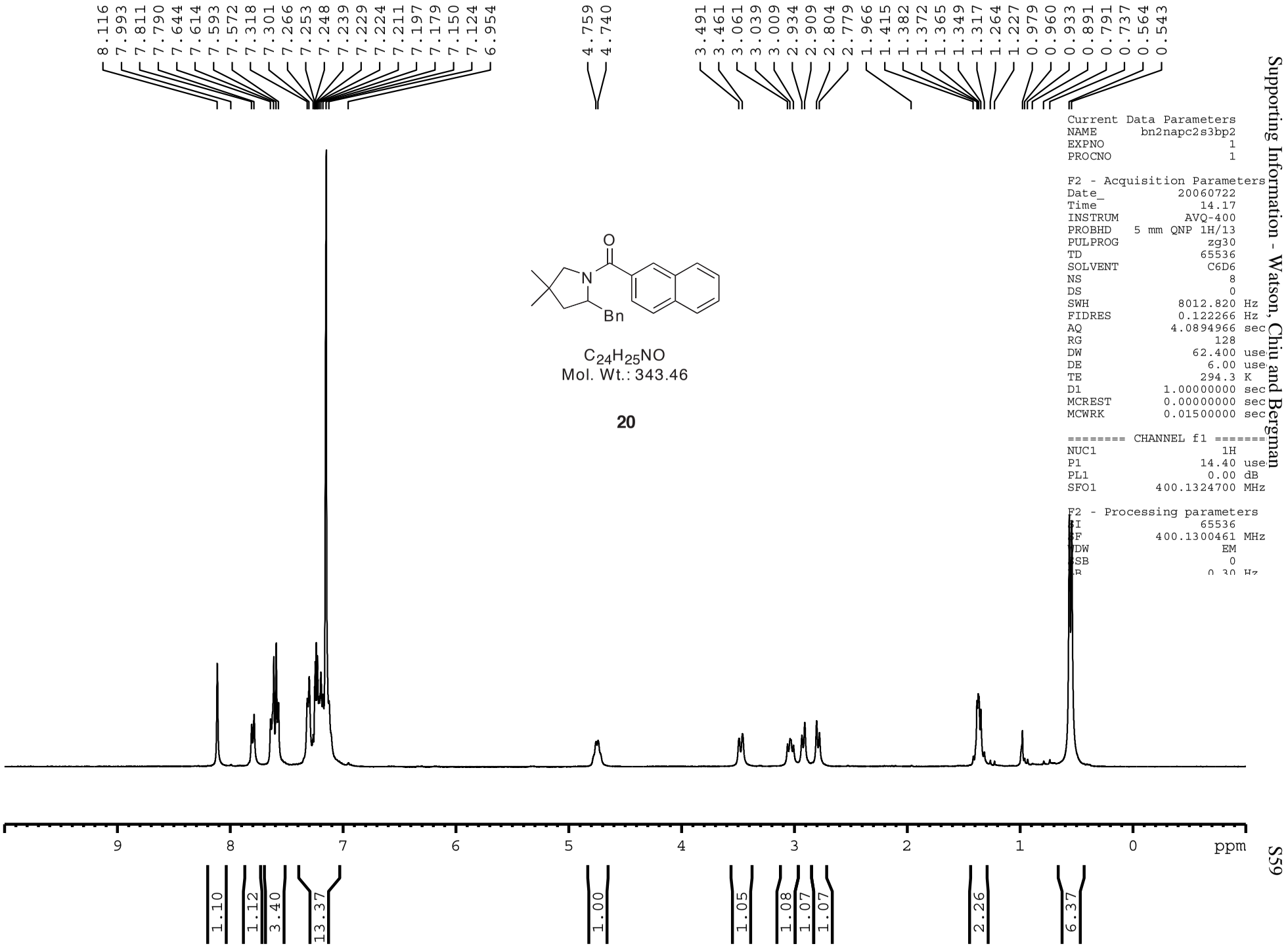
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 125.7722011 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 -5.00 dB
PL12 15.50 dB
PL13 21.50 dB
SFO2 500.1321560 MHz

F2 - Processing parameters
SI 131072
SF 125.7577971 MHz
WDW EM
SSB 0
LB 0.75 Hz
GB 0
PC 4.00

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

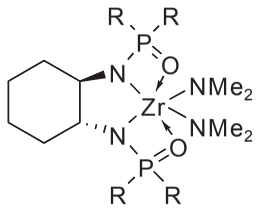
AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



AVB-400 ZBO Proton starting parameters. 6/11/03 RN

7.990 7.973 7.937 7.928 7.921 7.915 7.907 7.890 7.823 7.809 7.798 7.779 7.181 7.152 7.124 7.110 7.102 7.093 7.065 6.921 6.903 6.859 6.846

4.271 3.997 3.470 3.306 3.231 3.211 3.136 3.129 3.106 3.087 3.003 2.947 2.866 2.855 2.755 2.382 2.147 2.132 2.104 1.778 1.749 1.370 1.354 0.964 0.944 0.897 0.880 0.862 0.842 0.422



20a R = Ph
Generated In Situ
Contains Traces of 21a

20a

Current Data Parameters

USER	DAW
NAME	3170aH13515m
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060214
Time	14.09
INSTRUM	AVB-400
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	1
DS	0
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	203.2
DW	60.400 use
DE	6.00 use
TE	295.5 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

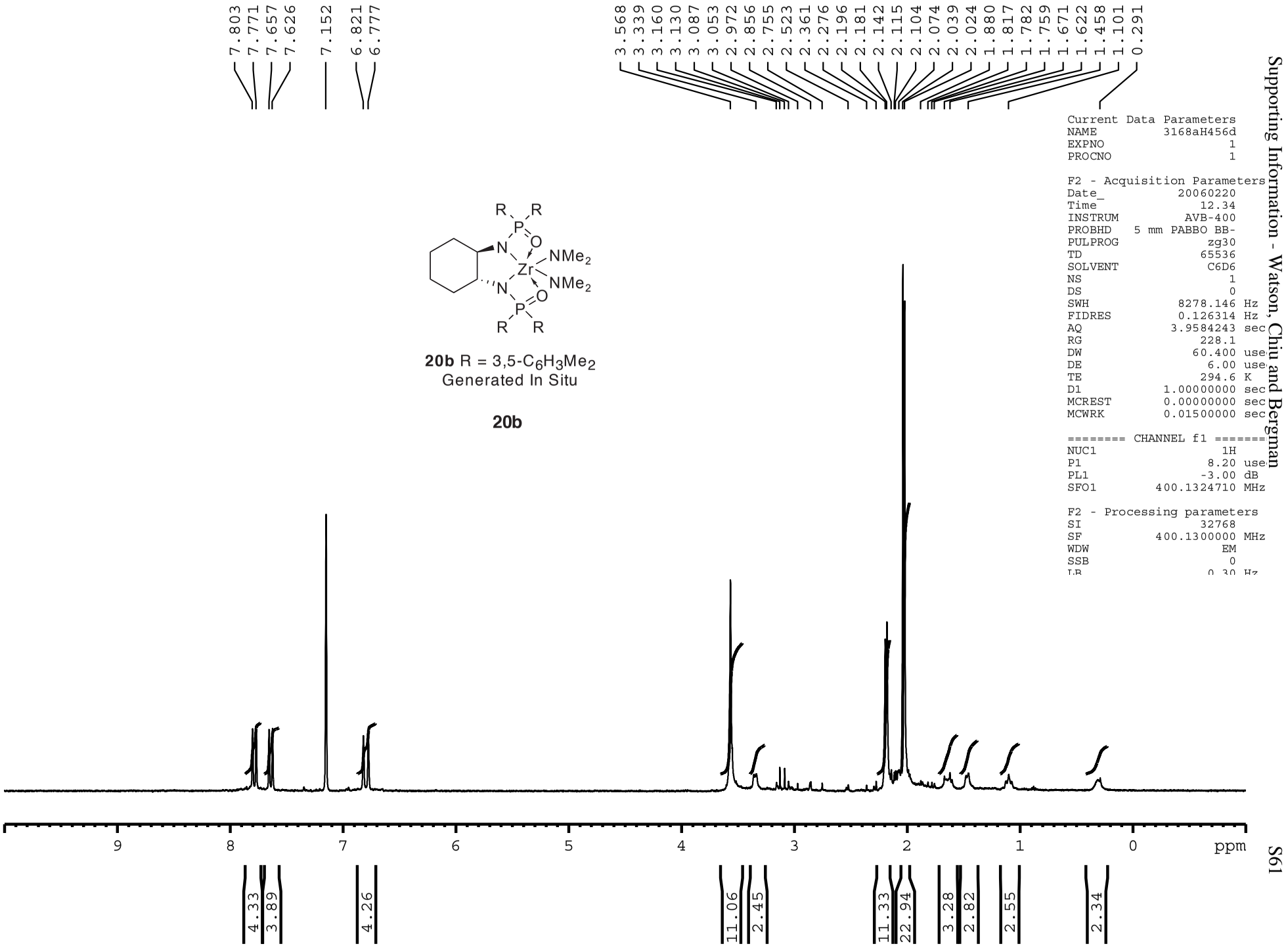
======== CHANNEL f1 ========

NUC1	1H
P1	8.20 use
PL1	-3.00 dB
SFO1	400.1324710 MHz

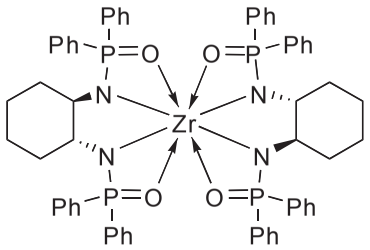
F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0

AVB-400 ZBO Proton starting parameters. 6/11/03 RN



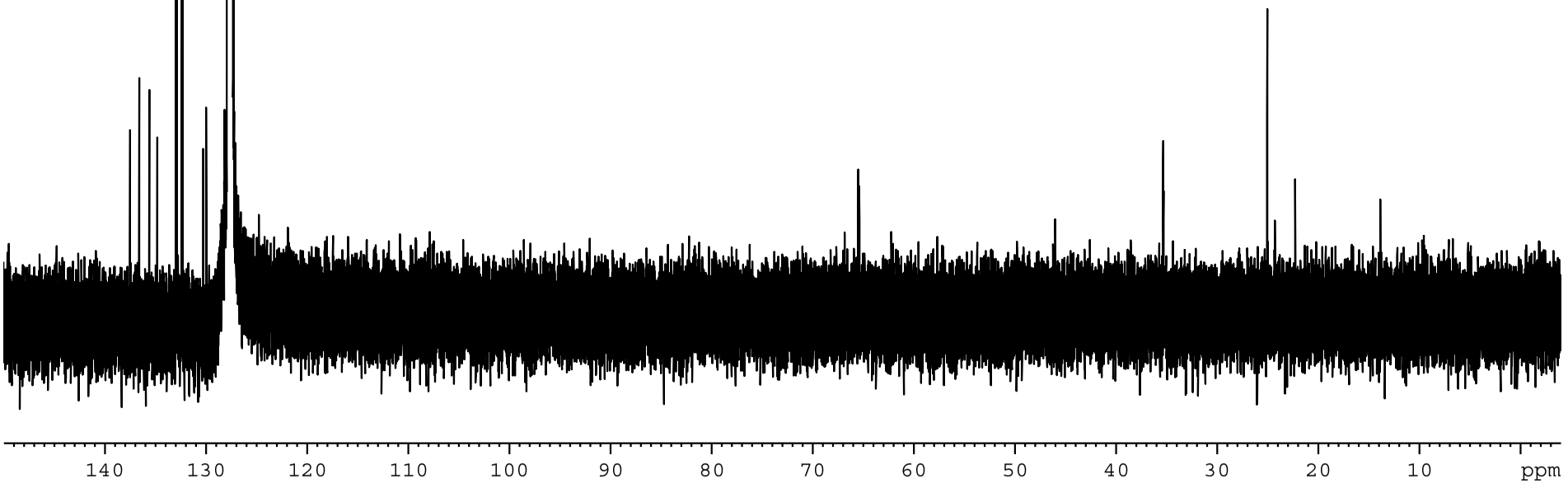
13C DRX-500 5mm ZBBO probe
starting parameters with zgpg30 (waltz16)
uses ns*td0

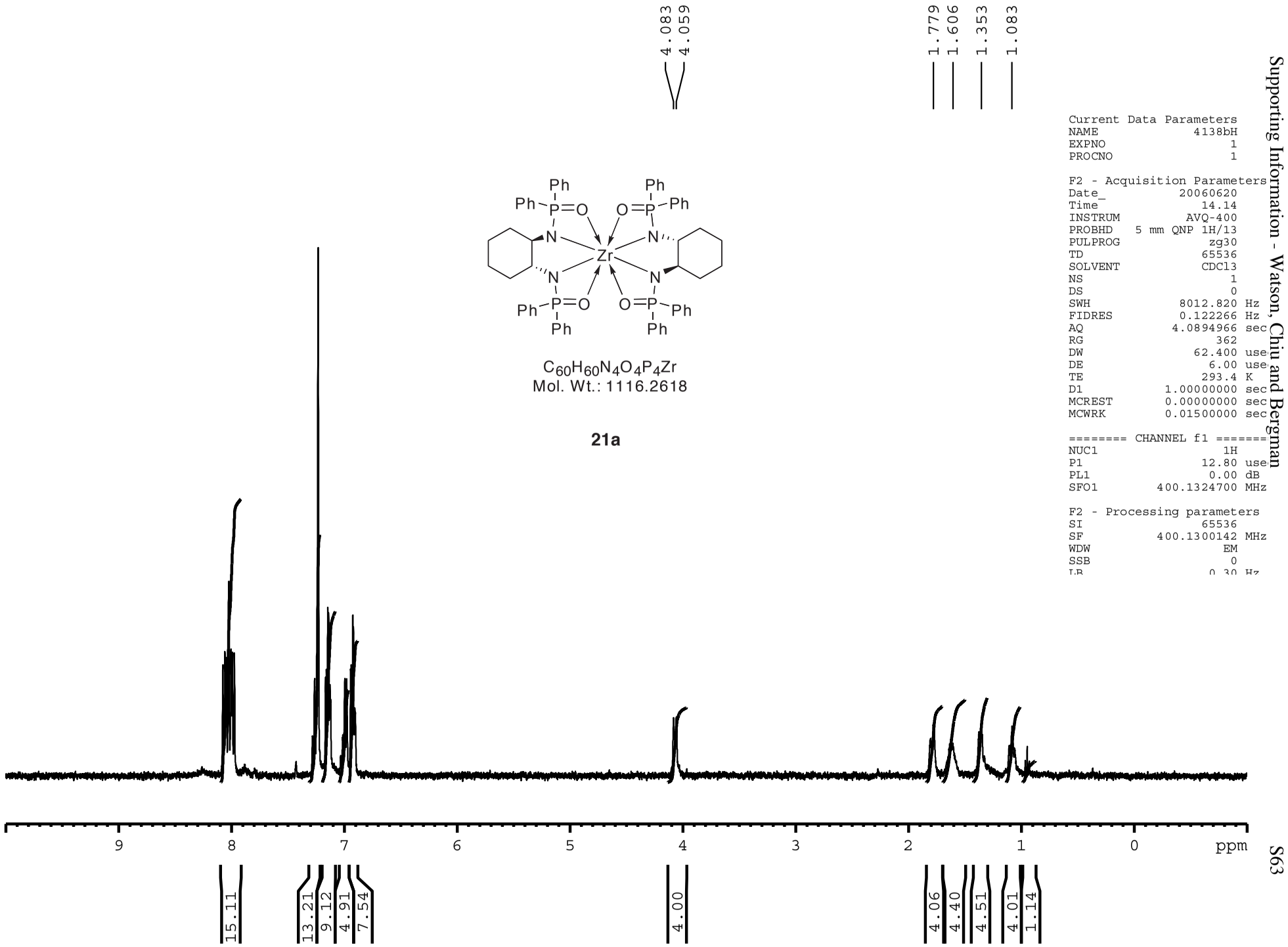


$C_{60}H_{60}N_4O_4P_4Zr$
Mol. Wt.: 1116.2618

21a

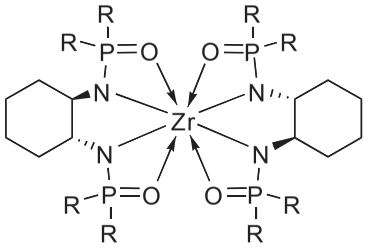
```
Current Data Parameters
NAME                  4138bC
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20060620
Time                   20.10
INSTRUM              DRX-500
PROBHD     5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT                CDCl3
NS                     11309
DS                         0
SWH               30864.197 Hz
FIDRES             0.235475 Hz
AQ                2.1234164 sec
RG                     16384
DW                   16.200 usec
DE                    5.00 usec
TE                   293.0 K
D1              1.50000000 sec
d11             0.03000000 sec
DELTA           1.39999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    8.70 usec
PL1                  -3.00 dB
SFO1           125.7722011 MHz

======== CHANNEL f2 ========
```




4.083
4.059
1.779
1.606
1.353
1.083
Current Data Parameters
NAME 4138bH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060620
Time 14.14
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 362
DW 62.400 use
DE 6.00 use
TE 293.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 use
PL1 0.00 dB
SFO1 400.1324700 MHz
F2 - Processing parameters
SI 65536
SF 400.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
Ph
P=O
O=P
N
Zr
C60H60N4O4P4Zr
Mol. Wt.: 1116.2618
21a
9
8
7
6
5
4
3
2
1
0
ppm
15.11
13.21
9.12
4.91
7.54
4.00
4.06
4.40
4.51
4.01
1.14

AVB-400 ZBO Proton starting parameters. 6/11/03 RN

7.810 7.779 7.152 6.868 6.652 4.086 3.438 2.558 2.524 2.496 2.260 2.199 2.181 2.176 2.095 2.039 2.022 1.994 1.949 1.882 1.361 1.126 0.881 0.115



21b R = 3,5-$C_6H_3Me_2$
Generated In Situ

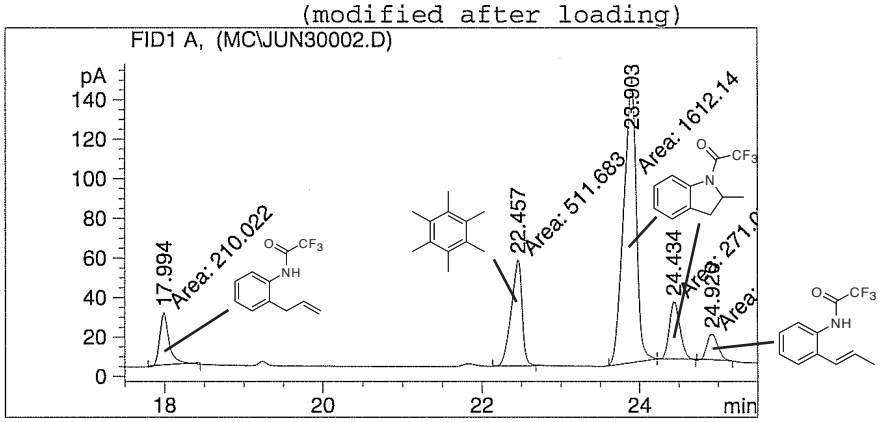
21b

```
Current Data Parameters
NAME          3169aH15024h
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20060216
Time                 14.39
INSTRUM            AVB-400
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        1
DS                        0
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                     287.4
DW                    60.400 use
DE                     6.00 use
TE                    294.8 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.20 use
PL1                   -3.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF            400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
```

9 8 7 6 5 4 3 2 1 0 ppm

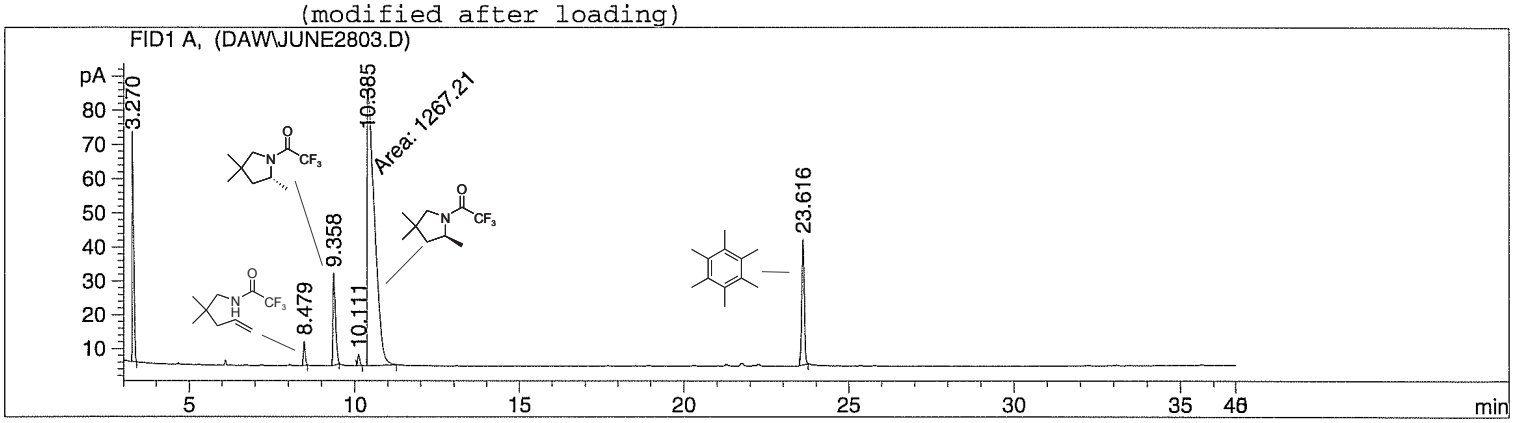
7.03 2.56 2.30 1.96 10.31 13.34 11.58

```
==========================================================================
Injection Date  : 6/30/06 11:39:42 AM          Seq. Line :   2
Sample Name     : AN1048                         Vial :   2
Acq. Operator   : MC                              Inj :   1
                                              Inj Volume : 2 µl

Acq. Method     : D:\HPCHEM\1\METHODS\HA_SUBFM.M
Last changed    : 6/16/06 10:32:54 PM by MC
Analysis Method : D:\HPCHEM\1\METHODS\DYW.M
Last changed    : 7/23/06 10:16:15 PM by MC
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   1.592 BP    0.0152    6.52783    6.82050  0.00247
   2   1.652 VV S  0.0277 1.38883e5  6.56908e4  52.45353
   3   1.943 VB S  0.0255 1.22989e5  8.02763e4  46.45082
   4   2.356 PP    0.0260   10.88994    6.67721  0.00411
   5   2.479 BP    0.0269    4.44667    2.74909  0.00168
   6   3.214 PB    0.0385  148.28819   53.24548  0.05601
   7  17.994 MM    0.1326  210.02168   26.40137  0.07932
   8  22.457 MM    0.1586  511.68347   53.78419  0.19325
   9  23.903 MM    0.1863 1612.14307  144.22220  0.60888
  10  24.434 MM    0.1560  271.07623   28.95608  0.10238
  11  24.920 MM    0.1614  125.91330   13.00030  0.04756

Totals :                  2.64773e5  1.46303e5

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

```
Injection Date  : 6/28/06 4:14:45 PM              Seq. Line :   1
Sample Name     : DAW414?d                          Vial :   3
Acq. Operator   : DAW                               Inj :   1
                                                 Inj Volume : 2 µl

Acq. Method     : D:\HPCHEM\1\METHODS\X_SUBA.M
Last changed    : 6/9/06 10:01:56 AM by MC
Analysis Method : D:\HPCHEM\1\METHODS\SHUTDWN.M
Last changed    : 6/29/06 9:22:36 AM by DAW
                  (modified after loading)
```



Area Percent Report

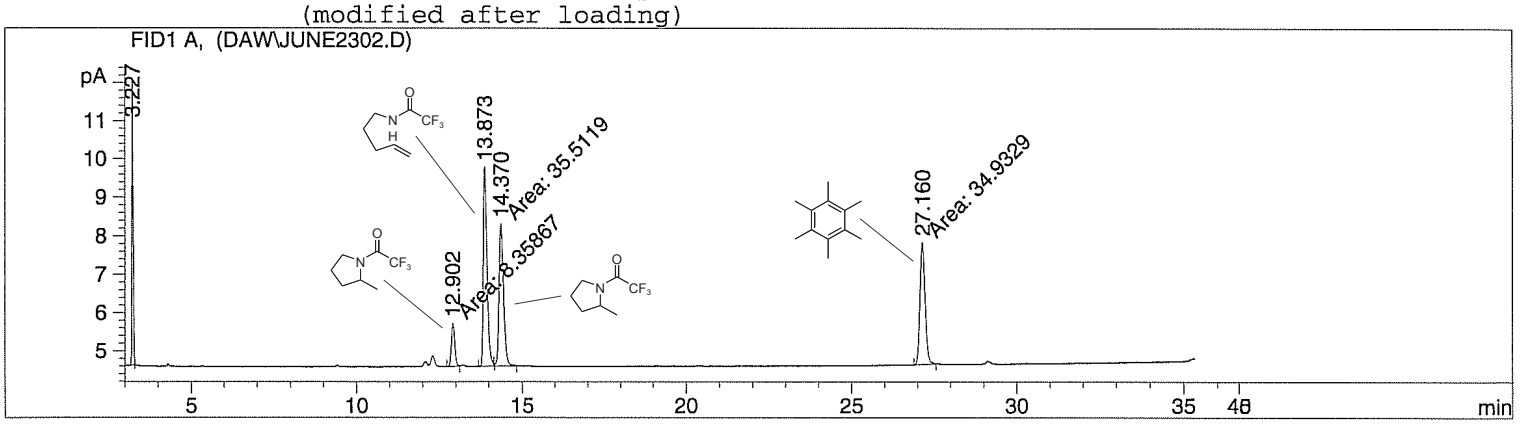
```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.571	PV	0.0110	2.62242	3.84290	0.00115
2	1.593	VV	0.0111	2.74232	3.93816	0.00120
3	1.618	VV	0.0118	26.58545	35.18530	0.01165
4	1.652	VB S	0.0284	1.56971e5	6.93598e4	68.79561
5	1.952	PB S	0.0255	6.92840e4	4.14704e4	30.36496
6	2.371	PP	0.0280	6.25669	3.65211	0.00274
7	3.270	PB	0.0442	228.19263	66.27210	0.10001
8	8.479	PB	0.0570	26.70593	7.08096	0.01170
9	9.358	PB	0.0717	141.81641	27.26009	0.06215
10	10.111	PB	0.0665	14.35946	3.38737	0.00629
11	10.385	MM T	0.2495	1267.21167	84.64605	0.55538
12	23.616	BB	0.0806	198.84915	37.03812	0.08715
Totals :				2.28171e5	1.11102e5	

```
 Results obtained with enhanced integrator!
```

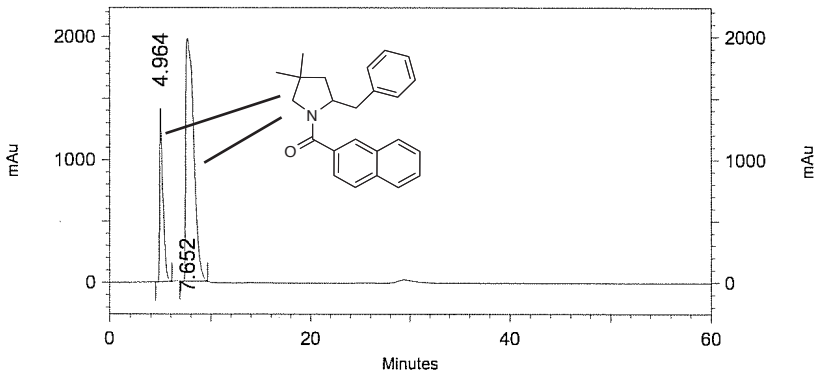
*** End of Report ***

```
==========================================================================
Injection Date  : 6/23/06 10:33:13 AM          Seq. Line :   2
Sample Name     : DAW4131f                          Vial :   8
Acq. Operator   : DAW                               Inj :   1
                                                Inj Volume : 2 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 0.1 µl
Acq. Method     : D:\HPCHEM\1\METHODS\X_SUBE.M
Last changed    : 6/21/06 3:26:59 PM by dg
Analysis Method : D:\HPCHEM\1\METHODS\SHUTDWN.M
Last changed    : 6/23/06 11:58:56 AM by DAW
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]       [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   0.692 BP    0.0115    7.55765   10.37646  0.03686
   2   0.786 BB S  0.0165 1.29078e4  1.11437e4  62.95304
   3   1.008 PB S  0.0117 7446.75293 1.00331e4  36.31869
   4   3.227 BB    0.0420   20.50775    7.48248  0.10002
   5  12.902 MM T  0.1226    8.35867    1.13644  0.04077
   6  13.873 MM R  0.1363   42.45444    5.19316  0.20706
   7  14.370 MM T  0.1594   35.51193    3.71375  0.17320
   8  27.160 MM T  0.1816   34.93287    3.20565  0.17037

Totals :                  2.05039e4  2.12079e4

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

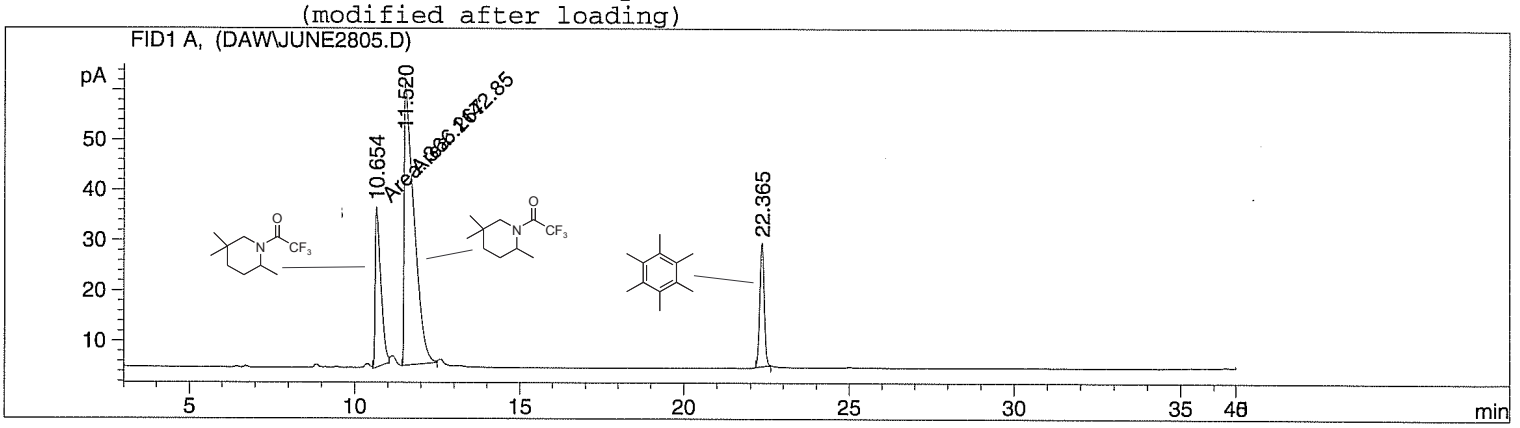
Sample ID: bn2nc2s3c
Filename:
C:\EZStart\Projects\Default\Data\MC\bn2nc2s3c7-23-2006 10-29-52
PMOD9010IP60min15mL.met.dat Method:
C:\EZStart\Projects\Default\Method\MC\OD9010IP60min15mL.met
Injection volume: 5 uL



3: 280 nm, 4 nm Results

Retention Time	Area	Area Percent
4.964	31663378	22.336
7.652	110098768	77.664

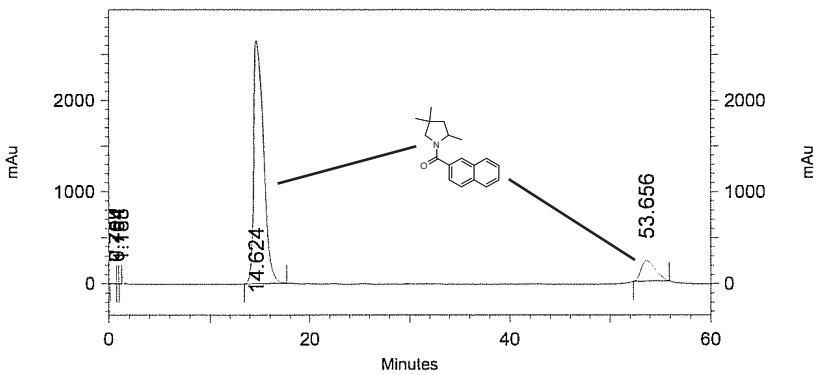
```
==========================================================================
Injection Date  : 6/28/06 5:46:12 PM          Seq. Line :   3
Sample Name     : DAW4148d                        Vial :   5
Acq. Operator   : DAW                             Inj :   1
                                              Inj Volume : 2 µl

Acq. Method     : D:\HPCHEM\1\METHODS\X_SUBB.M
Last changed    : 6/9/06 10:03:13 AM by MC
Analysis Method : D:\HPCHEM\1\METHODS\SHUTDWN.M
Last changed    : 6/29/06 9:21:46 AM by DAW
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1   0.727 BP S  0.0175 1.75196e5  1.41437e5  70.38090
   2   0.876 VB S  0.0184 7.17398e4  6.50759e4  28.81977
   3   1.096 BP    0.0206    6.31058    5.04526  0.00254
   4   1.169 BP    0.0190    1.73517    1.55988  0.00070
   5   1.496 BP    0.0222    1.66162    1.12613  0.00067
   6   1.695 BB    0.0326  227.40259   95.95634  0.09135
   7  10.654 MM T  0.1909  366.26709   31.97196  0.14714
   8  11.520 MM T  0.3327 1142.85339   57.24689  0.45911
   9  22.365 BB    0.1194  243.50099   24.61148  0.09782

Totals :                  2.48926e5  2.06731e5

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

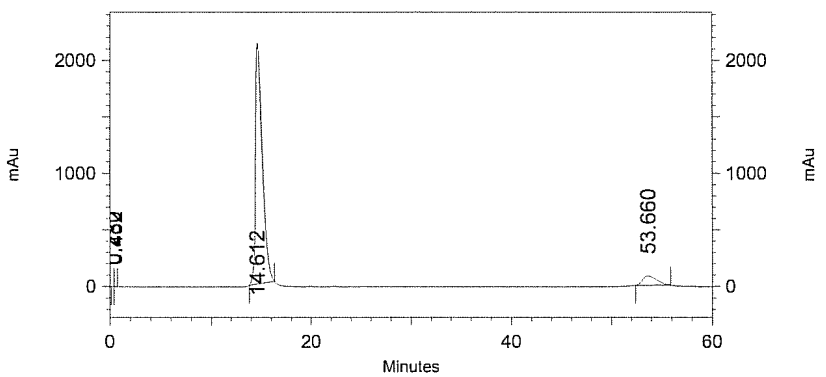
Sample ID: **dm2nap2c1s4a**
Filename:
C:\EZStart\Projects\Default\Data\MC\dm2nap2c1s4a7-20-2006 8-04-49
PMMCWH9010IP2mL.met.dat Method:
C:\EZStart\Projects\Default\Method\MC\MCWH9010IP2mL.met
Injection volume: 5 uL

Description: {Data Description}



1: 230 nm, 4 nm Results

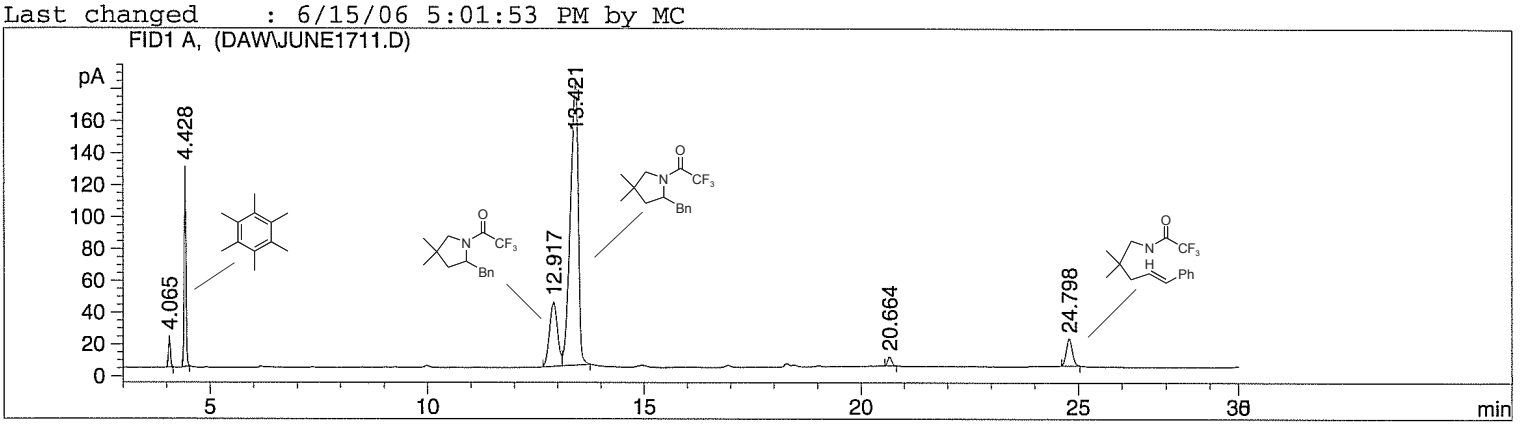
Retention Time	Area	Area Percent
0.280	42145	0.020
0.764	12103	0.006
1.188	6015	0.003
14.624	183288888	89.144
53.656	22260222	10.826



2: 240 nm, 4 nm Results

Retention Time	Area	Area Percent
0.280	2239	0.002
0.432	3770	0.003
14.612	102679324	92.651
53.660	8137998	7.343

```
==========================================================================
Injection Date  : 6/17/06 3:38:51 PM              Seq. Line :   2
Sample Name     : DAW4132e                          Vial :   3
Acq. Operator   : DAW                               Inj :   1
                                                 Inj Volume : 2 µl

Sequence File   : D:\HPCHEM\1\SEQUENCE\MC4SUB.S
Method          : D:\HPCHEM\1\METHODS\X_SUBC.M
Last changed    : 6/15/06 5:01:53 PM by MC
```

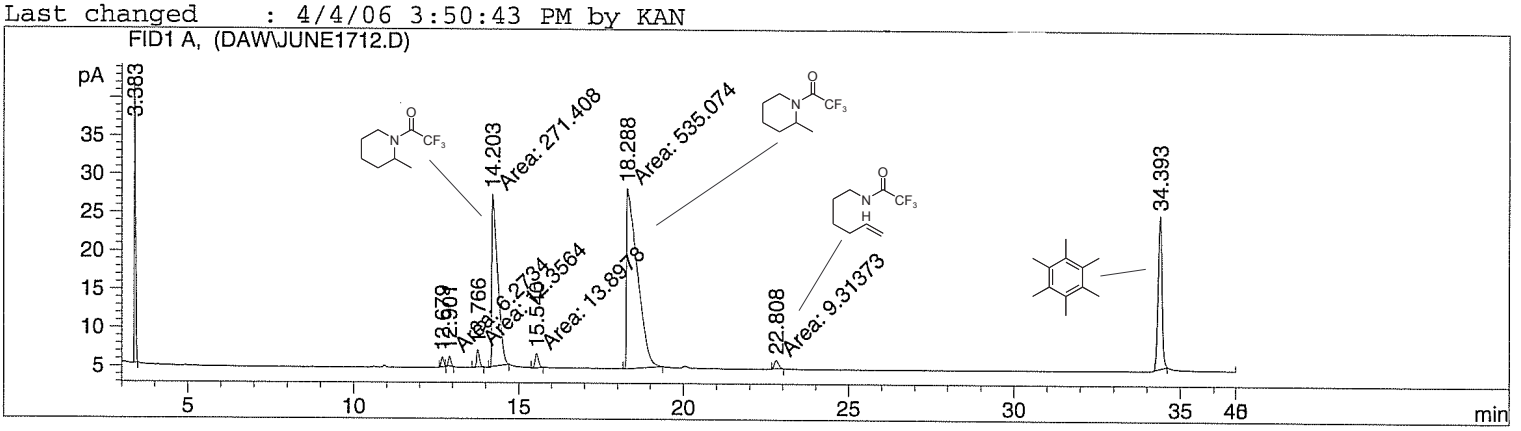



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	0.732	BP S	0.0153	1.58869e5	1.63699e5	59.46644
2	0.780	VB S	0.0190	1.04573e5	9.15890e4	39.14297
3	0.859	BV X	0.0197	264.03189	242.12094	0.09883
4	1.015	VV X	0.0148	4.17706	4.14583	0.00156
5	1.060	VB X	0.0151	1.61738	1.70197	0.00061
6	1.198	PB	0.0169	2.04290	1.85430	0.00076
7	1.794	BP	0.0219	12.37843	9.08484	0.00463
8	2.714	BB	0.0321	6.01821	2.91041	0.00225
9	4.065	BB	0.0466	58.25863	19.61882	0.02181
10	4.428	BB	0.0486	394.07022	125.71962	0.14751
11	12.917	BV	0.1534	521.16260	40.68647	0.19508
12	13.421	VB	0.1676	2249.70703	179.04912	0.84209
13	20.664	BB	0.0824	37.22778	5.61200	0.01393
14	24.798	BB	0.1311	164.34703	17.11080	0.06152
Totals :				2.67157e5	2.55937e5	

```
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

```
==========================================================================
Injection Date  : 6/17/06 4:11:57 PM              Seq. Line :   3
Sample Name     : DAW4133f                           Vial :   4
Acq. Operator   : DAW                                Inj :   1
                                                  Inj Volume : 2 µl

Acq. Method     : D:\HPCHEM\1\METHODS\X_SUBD.M
Last changed    : 6/9/06 10:05:13 AM by MC
Analysis Method : D:\HPCHEM\1\METHODS\GORIN.M
Last changed    : 4/4/06 3:50:43 PM by KAN
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FID1 A,
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.580	BV	0.0186	7.89778	5.91077	0.00575
2	1.628	VP	0.0161	46.23575	41.20645	0.03367
3	1.677	VB S	0.0334	7.13816e4	3.56321e4	51.98667
4	1.961	PB S	0.0211	6.47520e4	4.70268e4	47.15844
5	2.388	PB	0.0265	6.55903	3.92605	0.00478
6	3.383	PB	0.0391	96.69431	36.30813	0.07042
7	12.679	MM R	0.0876	6.78294	1.29008	0.00494
8	12.901	MM T	0.0821	6.27340	1.27406	0.00457
9	13.766	MM T	0.0930	12.35639	2.21524	0.00900
10	14.203	MM T	0.2018	271.40753	22.41681	0.19766
11	15.540	MM T	0.1245	13.89779	1.86032	0.01012
12	18.288	MM T	0.3799	535.07355	23.47433	0.38969
13	22.808	MM T	0.1448	9.31373	1.07188	0.00678
14	34.393	BB	0.1259	161.33351	19.82506	0.11750

```
Totals :                    1.37307e5  8.28196e4

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```