"The Dioxanone Approach to (2*S*, 3*R*)-2-*C*-Methylerythritol 4-Phosphate and 2,4-Cyclodiphosphate, and Various MEP Analogues."

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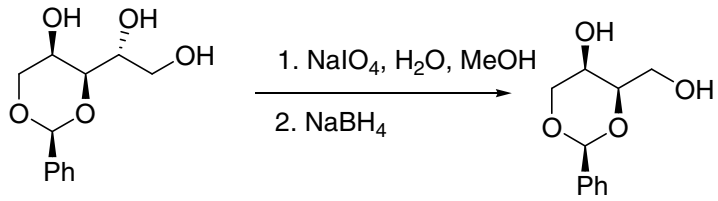
SUPPORTING INFORMATION

Note: Some NMR spectra are available in the Supporting Information accompanying the preliminary communication: Urbansky, M.; Davis, C. E.; Surjan, J. D.; Coates, R. M. *Org. Lett.* **2004,** *4*, 135-138

General Aspects:

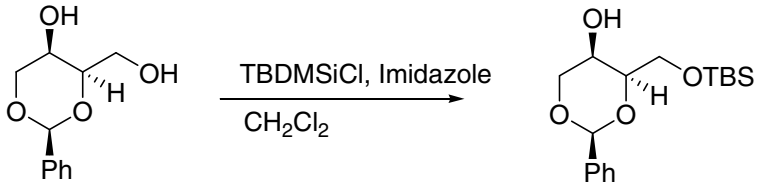
Reactions carried out under anhydrous conditions were performed under N_2 using oven-dried glassware unless specified otherwise. Et_2O, THF and benzene were distilled from sodium / bezophenone ketyl before use. Pentane, toluene, CH_2Cl_2, and Et_3N were distilled from calcium hydride before use. Dibenzyl phosphorochloridate was prepared as described by Buck and Reese.[1] Dibenzyl *N*, *N*-diisopropylphosphoramidite was prepared from PCl_3 using literature procedures.[2] Solvents used for chromatography were distilled prior to use. All other reagents and solvents used were reagent grade. Flash column chromatography was performed according to Still's procedure using 100-700 times excess 32-64 μm grade silica gel.[3] TLC analysis was performed using glass TLC plates (0.25 nm 60 F-254 silica gel). Visualization of the developed plates was accomplished by staining with ethanolic phosphomolybdic acid, ceric ammonium molybdate, or *p*-anisaldehyde followed by heating on a hot plate (ca 120 ^oC).

The following solvents and reference values (ppm) were used for NMR spectroscopy: $CDCl_3$ (^1H: 7.26, ^{13}C: 77.0), C_6D_6 (^1H: 7.16, ^{13}C: 128.0), C_5D_5N (^1H: 7.19, ^{13}C: 123.5), THF-d_8 (^1H: 3.58), acetone-d_6 (^1H: 2.05, ^{13}C: 206.0), CD_3OH (^1H: 3.31, ^{13}C: 49.0), D_2O (^1H: 4.80). ^{13}C and ^{31}P NMR spectral data taken in D_2O were externally referenced to aqueous sodium 2,2-dimethyl-2-silapentane-5-sulfonate (DSS) and 85% phosphoric acid (0.00 ppm), respectively. The abbreviation "app" (apparent) in ^1H NMR data sets refers to the appearance of the multiplet observed, and the coupling constants deduced in these cases were obtained by first-order coupling analysis. Samples for FT IR analysis were prepared as neat films on NaCl plates, and the data are reported in wave-numbers (cm^{-1}). Melting points were determined in open capillary tubes and are uncorrected. Optical rotations were measured using a digital polarimeter with a sodium lamp as MeOH, $CHCl_3$, water, or pyridine solutions in 0.5- or 1-dm cells at 24 ^oC unless noted otherwise. The University of Illinois Mass Spectroscopy Laboratories collected mass spectral data.



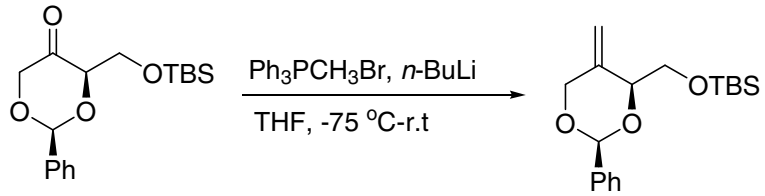
1, 3-Benzylidene-D-threitol (10).

The oxidative cleavage procedure reported by Ueno et al. [4] was followed with some modifications. A solution of 1, 3-benzylidene D-arabitol (**9**)[5] (3.0 g, 12.4 mmol) in methanol (70 mL) was stirred and cooled at 0 ^{o}C as a cold aqueous solution (30 mL) of $NaIO_4$ (2.9 g, 13.7 mmol) was added dropwise over 10 min. The resulting white suspension was allowed to stir for 20 min, at which time the TLC (R_f 0.7, 10:90 EtOH-CH_2Cl_2) indicated the reaction was completed. The precipitated solids were filtered and washed with methanol (15 mL). The filtrate containing aldehyde was stirred as $NaBH_4$ (0.47 g, 12.4 mmol) in water (3 mL) at 0 ^{o}C was added. The resulting milky suspension was stirred for 1 h at 0 ^{o}C after which methanol was removed under reduced pressure, and satd aqueous NH_4Cl (100 mL) was added. The product was extracted with CH_2Cl_2 (5x100 mL), and the combined extracts were dried over $MgSO_4$. Evaporation of the solvent under reduced pressure gave a crude white solid, which was crystallized with 7:3 CH_2Cl_2-acetone (10 mL) to give pure diol **10** (1.25 g) as a white crystalline solid. Concentration of the filtrate and purification by chromatography (65:35 acetone-CH_2Cl_2) gave another 670 mg of pure diol as a white solid; total yield, 1.95 g (76%): mp 130-132 ^{o}C [lit[6] mp 123 ^{o}C]; $[\alpha]_D^{25}$ -3.1^{o} (c = 1.00, MeOH) [lit[6] $[\alpha]_D^{23}$ – 6.0]; TLC R_f 0.5 (10:90 ethanol:CH_2Cl_2); ^{1}H NMR (400 MHz, CD_3OD) δ 3.62 (q, 1H, J = 1.5 Hz) 3.70-3.79 (m, 2H), 4.00 (m, 1H), 4.12 (t, 2H, J = 1.5 Hz), 5.60 (s, 1H), 7.33 (m, 3H), 7.55 (m, 2H); ^{13}C NMR (100 MHz, CD_3OD) δ 63.1, 64.9, 73.7, 81.5, 102.8, 127.6, 129.0, 129.8, 139.9. The ^{1}H NMR (CD_3OH) data correlate well with the literature values.[6]



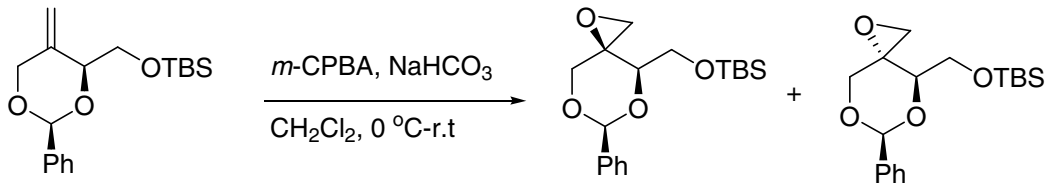
1, 3-Benzylidene-D-threitol, 4-(*t*-Butyldimethylsilyl) Ether (11).

A solution of 1, 3-benzylidene threitol 10 (2.00 g, 9.5 mmol) and imidazole (710 mg, 10.4 mmol) in dry CH_2Cl_2 (170 mL) was stirred and cooled at 0 ^oC as *t*-butyldimethylchlorosilane (1.8 g, 11.9 mmol) in CH_2Cl_2 (10 mL) was added dropwise over 5 min. The resulting milky suspension was stirred for 8 h at ambient temp. Ethanolamine (1.0 mL) was added to scavenge the excess TBDMSCl, and the suspension was allowed to stir for an additional 30 min. Water (150 mL) was added, and the product was extracted with CH_2Cl_2 (4x100 mL). The combined organic layers were washed with brine (1x150 mL) and dried over $MgSO_4$. Evaporation of the solvent under reduced pressure, and purification of the residue by flash chromatography (20:80 acetone-hexane and 50:50 acetone-hexane) gave 2.70 g (92%) of the mono-silyl ether 11 as a colorless solid: mp 43-44 ^oC; TLC R_f 0.48 (30:70, acetone:hexane); $[\alpha]_D^{25}$ –4.46^o (*c* = 1.0, MeOH); ^1H NMR (400 MHz, $CDCl_3$) δ 0.08 (s, 3H), 0.09 (s, 3H), 0.90 (s, 9H), 2.84 (d, 1H, *J* = 10.3 Hz), 3.73 (app d, 1H, *J* = 10.2 Hz), 3.79 (app dd, 1H, *J* = 10.0, 5.1 Hz), 3.90 (app dd, 1H, *J* = 10.0, 6.8 Hz), 3.97 (m, 1H), 4.07 (dd, 1H, *J* = 11.9, 1.5 Hz), 4.25 (dd, 1H, *J* = 11.9, 1.9 Hz), 5.59 (s, 1H), 7.33-7.40 (m, 3H), 7.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.3, -5.2, 18.4, 26.0, 62.6, 63.9, 72.9, 79.9, 101.6, 126.1, 128.4, 129.2, 137.9; IR (neat film) ν 3446, 3019, 2955, 2930, 1857, 1521, 1406, 1215 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{17}H_{28}O_4Si$ $(M+1)^+$: 325.1836, found 325.1835.



(2*S*, 4*S*)-4-(*t*-Butyldimethylsilyloxymethyl)-5-methylene-2-phenyl-1, 3-dioxane (24).

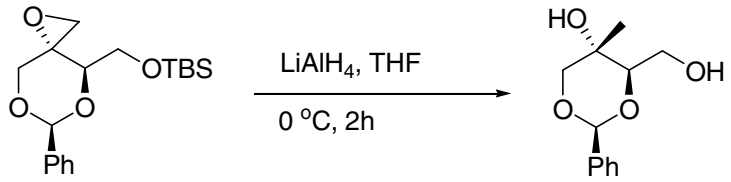
The Wittig reaction was carried out according to the procedure of Acton et al.[7] with some modifications. A suspension of methyltriphenylphosphonium bromide (1.13 g, 3.16 mmol) in dry THF (10 mL) was stirred and cooled at –75 °C as 1.97 mL of *n*-BuLi (3.16 mmol, 1.6 *M* in hexane) was added dropwise over 3 min. The resulting solution gradually turned yellow. After being stirred for 15 min, the solution was warmed to room temp and stirred an additional 30 min. The solution was again cooled to –75 °C, keto silyl ether **12** (340 mg, 1.05 mmol) in THF (1 mL) was added dropwise, and the solution was warmed to room temp. After 6 h the reaction was quenched with cold water (5 mL), and the product was extracted with ether (4x15 mL). The combined extracts were washed with brine (15 mL) and dried over $MgSO_4$. Evaporation of the solvent and purification by silica-gel chromatography (8:92 ethyl acetate:hexane) provided 270 mg (82%) of olefin **24** as a liquid: TLC R_f 0.75 (20% EtOAc in hexane): $[\alpha]^{25}_D$ –30.8° (*c* = 0.5, MeOH); ^{1}H NMR (500 MHz, benzene-d_6) δ 0.00 (s, 6H), 0.91 (s, 9H), 3.87 (υ_B ABX, 1H, J_{AB} = 12.0 Hz, J_{BX} = 3.7 Hz), 3.95 (υ_A ABX, 1H, J_{AB} = 12.0 Hz, J_{AX} = 5.2 Hz), 4.05 and 4.19 (ABdd, 2H, *J* = 12.3 Hz), 4.24 (app t, υ_X ABX, 1H, J_{app} = 5.7 Hz), 4.68 (s, 1H), 4.97(s, 1H), 5.47 (s, 1H), 7.06 (t, 1H, *J* = 7.5 Hz), 7.14 (t, 2H, *J* = 7.2 Hz), 7.61 (d, 2H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, benzene-d_6) δ –5.4, -5.3, 18.2, 25.8, 64.0, 71.5, 78.4, 101.2, 109.2, 126.7, 128.1, 128.7, 139.0, 141.0; IR (neat film) ν 3068, 2955, 2929, 2884, 2856, 1472, 1461, 1403, 1253 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{18}H_{28}O_3Si$ $(M)^+$: 320.1808, found 320.1762.



(2*S*, 4*S*, 5*R*)-*cis*, *cis*- and (2*S*, 4*S*, 5*S*)-*cis*, *trans*-4-(*t*-Butyldimethylsilyloxymethyl)-5, 5-oxymethylene-2-phenyl-1, 3-dioxane (25 and 26).

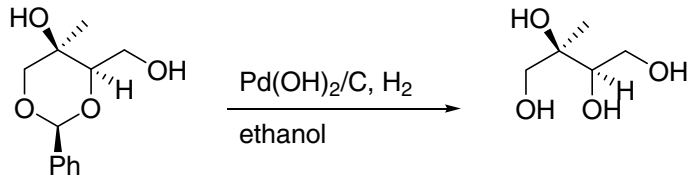
The epoxidation was carried out according to a procedure described by Schneider and Séquin[8] with modifications. A suspension of $NaHCO_3$ (0.91 g, 10.9 mmol) in a solution of olefin **24** (0.7 g, 2.1 mmol) in CH_2Cl_2 (20 mL) was stirred and cooled at 0 ^oC as 2.0 equiv of *m*-chloroperoxybenzoic acid (900 mg, 70%, 4.2 mmol) was added. After 4 h, another 2.0 equiv of peracid (0.9 g, 70%, 4.2 mmol) was added. After 12 h, satd aq Na_2SO_3 (1.0 mL) and satd aq $NaHCO_3$ (30 mL) were added, and the product was extracted with CH_2Cl_2 (3 x 40 mL). The combined extracts were washed with satd aq $NaHCO_3$ (30 mL) and brine (30 mL), and dried over $MgSO_4$. Evaporation of the solvent under reduced pressure gave the crude product, which was a 2:1 mixture of cis,cis and cis,trans epoxides according to ^1H NMR analysis. Purification by flash chromatography (10% ethyl acetate-hexane) yielded 350 mg (48%) of the cis,cis epoxide **25** and 180 mg (25%) of the cis,trans epoxide **26** as liquids. Data for the cis,cis epoxide **25**: TLC R_f 0.4 (20:80. EtOAc: hexane); $[\alpha]_D^{25}$ +10.1° (*c* = 1.00, MeOH); ^1H NMR (400 MHz, benzene-d_6,) δ -0.02 (s, 3H), -0.01 (s, 3H), 0.91 (s, 9H), 1.96 (d, 1H, *J* = 4.7 Hz), 2.85 (d, 1H, *J* = 4.7 Hz), 3.41 (d, 1H, *J* = 12.5 Hz), 3.70 (υ_B ABX, 1H, J_{AB} = 10.4 Hz, J_{BX} = 4.9 Hz), 3.82 (υ_A ABX, 1H, J_{AB} = 10.0 Hz, J_{AX} = 6.9 Hz), 3.85 (d, 1H, *J* = 12.5 Hz), 4.21 (υx ABX, 1H, J_{AX} = 6.9 Hz, J_{BX} = 4.9 Hz), 5.4 (s, 1H), 7.05-7.09 (m, 1H), 7.12-7.16 (m, 2H), 7.60-7.63 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.7, -5.5, 18.2, 25.8, 46.5, 54.2, 61.6, 72.3, 78.1, 101.5, 126.7, 128.1, 128.9, 138.6; IR (neat film) ν 2954, 2929, 2885, 2856, 1471, 1398, 1255 cm^{-1}; HRMS (FAB) *m/z*

Calcd for $C_{18}H_{28}O_4Si$ $(M-H)^+$: 335.1679, found 335.1679. Data for the cis,trans epoxide **26**: TLC R_f 0.6 (20:80, EtOAc:hexane); $[\alpha]_D^{25}$ +49.4° (*c* = 1.1, MeOH); ^{1}H NMR (500 MHz, benzene-d_6,) δ 0.05 (s, 3H), 0.06 (s, 3H), 0.96 (s, 9H), 2.21 (dd, 1H, *J* = 4.6, 1.2 Hz), 2.71 (dd, 1H, *J* = 1.7, 4.5 Hz), 3.44 (d, 1H, *J* = 11.3 Hz), 3.62 (d, 1H, *J* = 1.5 Hz), 3.63 (s, 1H), 3.84 (dd, 1H, *J* = 2.0, 11.3 Hz), 4.05 (app t, 1H, J_{app} = 3.8 Hz), 5.41 (s, 1H), 7.08 (m, 1H), 7.17 (m, 2H), 7.64 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ -5.3, -5.2, 18.3, 25.8, 51.7, 52.0, 61.3, 71.2, 79.6, 101.2, 126.6, 128.2, 128.8, 138.4; IR (neat film) ν 2953, 2929, 2856, 1471, 1461, 1461, 1349, 1253, 1105, 1033, 972 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{18}H_{28}O_4Si$ $(M+H)^+$: 337.1836, found 337.1835.



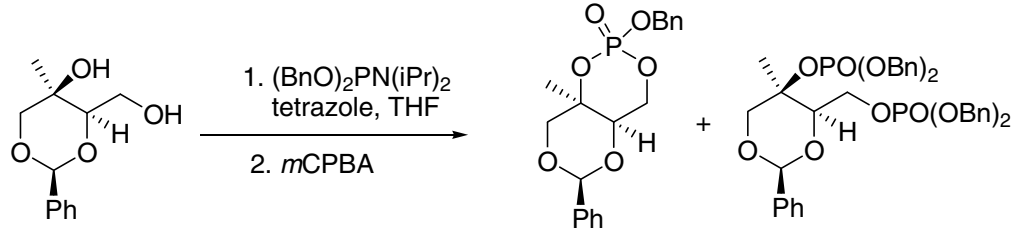
1, 3-Benzylidene-2-*C*-methylerythritol (15).

The $LiAlH_4$ reduction-desilation of cis,trans epoxide **26** was carried out as described in the Experimental Section for the cis, cis epoxide (**25**). The weights of the starting epoxide and reagent were as follows: epoxide (40 mg 0.1 mmol) and $LiAlH_4$ (4.5 mg, 0.1 mmol). After work-up, the crude product was purified by silica-gel chromatography (60% EtOAc in hexane) to give 24 mg (90 %) diol **15** as white solid. The physical and NMR data were in agreement with those reported in the Experimental Section for 1, 3-benzylidene 2-*C*-methylerythritol (**15**).



2-*C*-Methyl-D-threitol (28).

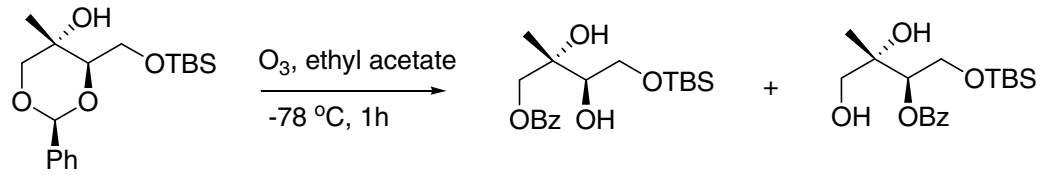
To a solution of diol **27** (34 mg, 0.15 mmol) in ethanol (3 mL) containing HCO_2H (1 drop) was added 20% $Pd(OH)_2/C$ (15 mg). The suspension was allowed to stir under an H_2 atmosphere for 12 h. The mixture was diluted with MeOH (5 mL) and filtered through celite. Concentration of the filtrate at reduced pressure afforded 20 mg (98%) of 2-*C*-methylthreitol (**28**, 20 mg, 98%) as a colorless oil: $[\alpha]_D^{25}$ +9.8° (*c* = 1.00, MeOH); [lit[9] $[\alpha]_D^{25}$ +7.3° (*c* = 0.8, MeOH); ^{1}H NMR (500 MHz, D_2O) δ 1.09 (s, 3H), 3.47 and 3.53 (ABdd, 2H, J_{AB} = 11.6 Hz), 3.50 (bd, 1H, J = 11.6 Hz), 3.63 (dd, 1H, *J* = 8.5, 2.8 Hz), 3.75 (dd, 1H, *J* = 11.8, 2.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.2, 61.9, 66.2, 74.2, 75.2. The spectral data agree with the reported values.[9]



1, 3-Benzylidene-2-*C*-methyl-D-threitol 2, 4-Cyclophosphate, Benzyl Ester (31) and 1, 3-Benzylidene-2-*C*-methyl-D-threitol 2, 4-diphosphate, Tetrabenzyl Ester (32).

The phosphorylation procedure was based on that described by Yu and Fraser-Reid[10] and on the procedure described in the Experimental Section for diol **15**. A solution of dibenzyl *N,N*-diisopropylphosphoramidite[2] (230 mg, 0.66 mmol) and tetrazole (70 mg, 1.0 mmol) in CH_3CN (2.0 mL) was allowed to stir for 30 min at room temp. A solution of diol **27** (50 mg, 0.22 mmol) in CH_3CN (1.5

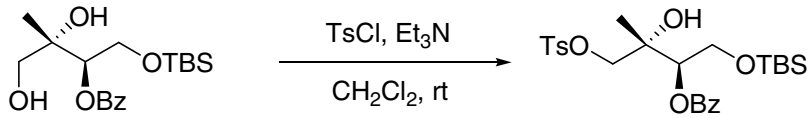
mL) was added dropwise. After 5 h, the reaction mixture was cooled to 0 °C and *m*-chloroperoxybenzoic acid (solid, 220 mg, 2.0 mmol, 77%) was added. The cooling bath was removed, and the reaction mixture was allowed to stir at room temp. After 1h, the mixture was diluted with Et_2O (40 mL). The ethereal layer was washed with 10% $Na_2S_2O_5$ (2x10 mL), satd aq $NaHCO_3$ (10 mL), and brine (10 mL); dried ($MgSO_4$); and evaporated to give the crude product as a white solid. Purification by flash chromatography (60% and 75% EtOAc:hexane) gave 25 mg (35%) of cyclic phosphate **31** as white solid and 15 mg (10%) bis(dibenzyl) diphosphate **32** as a colorless oil. Data for **31**: mp 181-182 °C TLC R_f 0.5 (75:25 EtOAc:hexane); $[\alpha]_D^{25}$ –57.0° (*c* = 0.61, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$), δ 1.35 (s, 3H), 3.72-3.76 (m, 2H), 4.11 (d, 1H, *J* = 12.5 Hz), 4.33 (app dd, 1H, J_{app} = 12.5, 23.5 Hz), 4.49 (d, 1H, *J* = 12.5 Hz), 5.15 (d, 2H, *J* = 8.0 Hz), 5.54 (s, 1H), 7.34-7.40 (m, 8H), 7.55 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.0, 67.3 (d, J_{cp} = 5.4 Hz), 69.4 (d, J_{cp} = 5.4 Hz), 73.6 (d, J_{cp} = 7.2 Hz), 74.7 (d, J_{cp} = 9.1 Hz), 78.2 (d, J_{cp} = 8.1 Hz), 101.6, 126.8, 128.4, 128.6, 128.9, 129.7, 136.1 (d, J_{cp} = 7.4 Hz), 137.1; HRMS (FAB) *m/z* Calcd for $C_{19}H_{22}O_6P$ $(M+1)^+$ 377.1154, found 377.1155. Data for the bis(dibenzyl) diphosphate **32**: TLC R_f 0.31 (75:35 EtOAc-hexane); $[\alpha]_D^{25}$ +12.9° (*c* = 1.38, $CHCl_3$); ^{1}H NMR (500 MHz, acetone d_6), δ 1.47 (s, 3H), 3.94 and 4.77 (ABdd, 2H, *J* = 12.7 Hz), 4.14-4.24 (m, 2H), 4.32-4.37 (m, 1H), 5.02-5.13 (m, 8H), 5.73 (s, 1H), 7.27-7.38 (m, 21H), 7.40 (m, 2H,), 7.50 (m, 2H); ^{13}C NMR (125 MHz, acetone-d_6) δ 18.7, 66.1 (d, J_{cp} = 4.9 Hz), 68.8 (d, J_{cp} = 6.2 Hz), 68.9 (d, J_{cp} = 4.9 Hz), 69.0 (d, J_{cp} = 6.2 Hz), 72.5, 77.4 (d, J_{cp} = 4.9 Hz), 81.9 (d, J_{cp} = 7.0 Hz), 82.0 (d, J_{cp} = 7.0 Hz), 101.0, 126.5, 127.9, 128.0, 128.1, 128.1, 128.2, 128.3, 128.5, 128.5, 128.6, 128.6, 128.6, 128.7, 128.7, 128.9, 136.6, 138.6, 138.7, 138.4; HRMS (FAB) *m/z* Calcd for $C_{40}H_{43}O_{10}P_2$ $(M+1)_+$ 745.2331, found 745.2332.



(2*R*, 3*S*)-3-Benzoyloxy-4-(*t*-butyldimethylsilyloxy)-2-methylbutan-1, 2-diol (34) and (2*R*, 3*S*)-1-Benzoyloxy-4-(*t*-butyldimethylsilyloxy)-2-methylbutan-2, 3-diol (35)

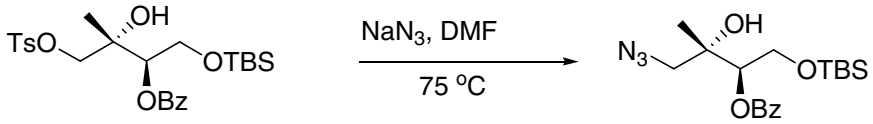
The ozonolysis was carried out according to a procedure described by Deslongchamps et al.[11] A solution of silyl ether (250 mg, 0.74 mmol) in ethyl acetate (8.0 mL) stirred and cooled at –75 °C as an ozone-oxygen mixture was bubbled through the solution for 1.5 h. The residual ozone was removed by flushing with nitrogen. Evaporation of the solvent under reduced pressure gave the crude product, as a 4:1 mixture of primary and secondary alcohol isomers according to ^{1}H NMR analysis. Purification by silica-gel column chromatography (20% ethyl acetate/hexane) afforded 40 mg (20%) of the less polar secondary alcohol **35** and 195 mg (75%) of the more polar primary alcohol **34** as liquids. Physical data for **35**: TLC R_f 0.35 (20:80 ethyl acetate: hexane); $[\alpha]_D^{25}$ +7.2° (c = 1.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.09 (s, 6H), 0.90 (s, 9H), 1.33 (s, 3H), 3.66 (t, 1H, J = 5.0 Hz), 3.86 (d, 2H, J = 5.0 Hz), 4.27 and 4.33 (ABdd, 2H, J_{AB} = 11.7 Hz), 7.43 (app t, 2H, J_{app} = 6.5 Hz), 7.58 (app t, 1H, J_{app} = 7.5 Hz), 8.05 (app d, 2H, J_{app} = 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ –5.3, -5.4, 18.3, 21.0, 26.0, 64.1, 69.5, 73.1, 73.9, 128.7, 129.9, 133.5, 167.0; IR (neat film) ν 3473, 2955, 2885, 1722, 1463, 1452, 1274, 1112, 837 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{18}H_{30}O_5Si$ $(M+1)^+$ 355.1942, found 355.1940. Physical data for **34**: TLC R_f 0.22 (20:80 ethyl acetate: hexane); $[\alpha]_D^{25}$ +6.5° (c = 1.6, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.07 (s, 6H), 0.86 (s, 9H), 1.22 (s, 3H), 3.39 and 3.55 (ABdd, 2H, J_{AB} = 12.0 Hz), 4.00 (υ_B ABX, 1H, J_{AB} = 11.0 Hz, J_{BX} = 5.5 Hz), 4.00 (υ_A ABX, 1H, J_{AB}= 11.0 Hz, J_{AX} = 5.0 Hz), 5.12 (t, 1H, J = 5.5 Hz), 7.47 (app t, 2H, J_{app} = 7.5 Hz), 7.59 (app t, 1H, J_{app} = 7.5 Hz), 8.06

(app d, 2H, J_{app} = 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ –5.4, -5.3, 18.2, 19.8, 25.8, 61.9, 67.6, 73.8, 75.5, 128.7, 129.7, 130.0, 133.7, 166.9; IR (neat film) ν 3444, 2954, 2885, 1722, 1471, 1452, 1273, 1125, 837 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{18}H_{30}O_5Si$ $(M+1)^+$ 355.1942, found 355.1940.



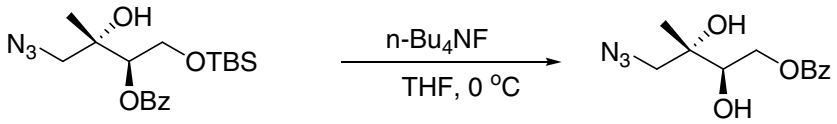
(2*S*, 3*R*)-3-Benzyloxy, 4-(*t*-butyldimethylsilyloxy)-2-hydroxy-2-methylbut-1-yl Tosylate.

A solution of alcohol **34** (145 mg, 0.41 mmol) and triethylamine (0.6 mL, 4.6 mmol) in methylene chloride (4.0 mL) was stirred and cooled at 0 °C as *p*-toluenesulfonyl chloride (167 mg, 0.88 mmol) and a catalytic amount of 4-dimethylaminopyridine (4.0 mg) were added. The solution was warmed to room temp and stirred for 10 h. Water (5.0 mL) was added, and the product was extracted with methylene chloride (2 x 30 mL). The combined extracts were washed with water (10 mL) and brine (10 mL) and dried over $MgSO_4$. Evaporation of the solvent and purification by flash chromatography (20% ethyl acetate: hexane) gave 180 mg (82%) of the tosylate intermediate as a viscous liquid: TLC R_f 0.75 (30:70 ethyl acetate- hexane); $[\alpha]_D^{25}$ -18.2° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.00 (s, 3H), 0.01 (s, 3H), 0.83 (s, 9H), 1.26 (s, 3H), 2.39 (s, 3H), 3.96 (d, 1H, *J* = 9.6 Hz), 3.91-4.01 (m, 2H), 4.06 (d, 1H, *J* = 9.6 Hz), 5.08 (t, 1H, *J* = 4.3 Hz), 7.28 (d, 2H, *J* = 8.0 Hz), 7.47 (app t, 2H, J_{app} = 7.5 Hz), 7.58 (app t, 1H, J_{app} = 7.5 Hz), 7.76 (d, 2H, *J* = 8.0 Hz), 8.00 (app d, 2H, J_{app} = 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ –5.6, -5.4, 18.1, 18.1, 21.5, 21.8, 25.8, 62.5, 73.5, 73.7, 128.2, 128.6, 129.8, 129.9, 130.1, 132.5, 133.5, 145.2, 165.7; IR (neat film) ν 3472, 2930, 2857, 1722, 1452, 1361, 1270, 1177, 1097, 836 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{25}H_{36}O_7SSi$ $(M+1)^+$ 509.2027, found 509.2029.



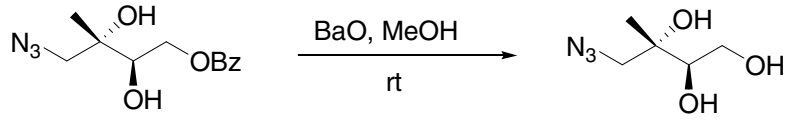
(2*R*, 3*S*)- 1-Azido-3-benzoyloxy-4-(*t*-butyldimethylsilyloxy)-3-methylbutan-3-ol (38)

A solution of the tosylate (160 mg, 0.32 mmol) and sodium azide (42 mg, 0.64 mmol) in dry DMF (2.0 mL) was stirred and heated at 75 ^{o}C. After 2.5 h, suspension was cooled to 0 ^{o} C and diluted with ether (10 mL) and water (5.0 L). The aqueous layer was separated and extracted with ether (2 x 15 mL). The combined ethereal layers were washed with water (2 x 5 mL) and brine (5 mL), and dried over $MgSO_4$. Concentration and purification by the flash chromatography (10% ethyl acetate: hexane) afforded 93 mg (76%) of azide **38** as a liquid: TLC R_f 0.47 (15:85 ethyl acetate: hexane); $[\alpha]_D^{25}$ -40.4° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.07 (s, 3H), 0.09 (s, 3H), 0.89 (s, 9H), 1.35 (s, 3H), 3.35 (s, 2H), 3.97 (υ_B ABX, 1H, J_{AB} = 10.0 Hz, J_{BX} = 6.0 Hz), 4.01 (bs, 1H), 4.09 (υ_A ABX, 1H, J_{AB} = 10.0 Hz, J_{AX} = 4.0 Hz), 5.16 (dd, 1H, *J* = 4.0, 5.5 Hz), 7.46 (app t, 2H, J_{app} = 8.0 Hz), 7.46 (app t, 1H, J_{app} = 7.5 Hz), 8.05 (app d, 2H, J_{app} = 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ –5.5, -5.4, 18.2, 21.9, 25.8, 58.6, 62.5, 73.5, 73.8, 75.1, 128.7, 129.8, 129.9, 133.6, 165.7; IR (neat film) ν 3477, 2930, 2858, 2104, 1723, 1452, 1269, 1096, 837 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{18}H_{29}N_3O_4Si$ $(M+1)^+$ 380.2006, found 380.2005.



(2*R*, 3*S*)-1-Azido-4-benzoyloxy-2-methylbutan-2, 3-diol (39).

A solution of silyl-protected azide **38** (60 mg, 0.15 mmol) in dry THF (1.0 mL) was stirred and cooled at 0 °C as nBu_4NF (0.16 mL, 0.15 mmol, 1 *M* in THF) was added dropwise. After 25 min, water (4 mL) was added, and the product was extracted with ether (3x5 mL). The ethereal extracts were combined and dried over $MgSO_4$. Evaporation of the solvent and purification of the residual oil by flash chromatography (40% EtOAc:hexane) afforded 37 mg (91%) of azido diol **39** as a liquid: TLC R_f 0.2 (30:70 EtOAc: hexane); $[\alpha]_D^{25}$ +13.0° (*c* = 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.28 (s, 3H), 2.49 (s, 1H), 2.93 (bd, 1H, *J* = 3.5 Hz), 3.34 (d, 1H, *J* = 12.4 Hz), 3.64 (d, 1H, *J* = 12.0 Hz), 3.94 (bd, 1H, *J* = 7.4 Hz), 4.41 (dd, 1H, *J* = 7.5, 12.0 Hz), 4.61 (dd, 1H, *J* = 2.5, 12.0 Hz), 7.46 (m, 2H), 7.58 (m, 1H), 8.05 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.7, 58.2, 66.4, 73.7, 74.3, 128.7, 129.7, 129.9, 133.6, 165.7; IR (neat film) ν 3448, 2932, 2106, 1704, 1602, 1451, 1278, 1122, 1026 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_{12}H_{15}N_3O_4$ $(M+1)^+$ 266.1149; found 266.1141.



(2*R*, 3*S*)-4-Azido-3-methylbutane-1, 2, 3-triol (40).

A solution of azido diol **39** (25 mg, 0.09 mmol) and BaO (29 mg, 0.18 mmol) in MeOH[12] was stirred for 40 min at room temp with TLC monitoring. Evaporation of the MeOH and purification of the residue by silica-gel chromatography (80% ethyl acetate/hexane) gave 12 mg (92 %) of azido triol 40 as a viscous oil: TLC R_f 0.17 (80:20 EtOAc:hexane); $[\alpha]_D^{25}$ +13.8° (*c* = 0.78,

MeOH); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.25 (s, 3H), 3.32 (d, 1H, *J* = 12.0 Hz), 3.53 (d, 1H, *J* = 12.3 Hz), 3.63 (dd, 1H, *J* = 4.0, 6.2 Hz), 3.72 (dd, 1H, *J* = 6.0, 11.2 Hz), 3.83 (dd, 1H, *J* = 3.7, 11.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.7, 58.1, 62.9, 74.3, 75.0; IR (neat film) ν 3391, 2932, 2106, 1289, 1088, 1024 cm^{-1}; HRMS (FAB) *m/z* Calcd for $C_5H_{11}N_3O_3$ $(M+1)^+$ 162.0881, found 162.0879.

References

1. Buck, I. M.; Reese, C. B. *J. Chem. Soc., Perkin Trans. 1* **1990**, 2937-2942.

2. (a) Tanaka, T.; Tamatsukuri, S.; Ikehara, M. *Tetrahedron Lett.* **1986**, *27*, 199-202. (b) Dreef, C. E.; Tulnman, R. J.; Lefeber, A. W. M.; Elle, C. J. J.; van der Marel, G. A.; van Boom, J. H. *Tetrahedron* **1991**, *47*, 4709-4722.

3. Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

4. Ueno, Y.; Tadano, K.; Ogawa, S.; McLaughlin, J. L.; Alkofahi, A. *Bull. Chem. Soc. Jpn***. 1989**, *62*, 2328-2337.

5. Haskins, W. T.; Hann, R. M.; Hudson, C. S. *J. Am. Chem. Soc.* **1943**, *65*, 1663-1667.

6. Lehmann, J.; Wagenknecht, H.-A. *Carbohydr. Res.* **1995**, *276*, 215-218.

7. Acton, E. M.; Goerner, R. N.; Uh, H. S.; Ryan, K. J.; Henry, D. W.; Cass, C. E.; LePage, G. A. *J. Med. Chem.*, **1979**, 22, 518-525.

8. Schneider, A.; Séquin, U. *Tetrahedron* **1985**, *41*, 949-953.

9. Fontana, A.; Messina, R.; Spinella, A.; Cimino, G.; *Tetrahedron Lett.* **2000,** *41*, 7559-7562.

10. Yu, K.-L.; Fraser-Reid, B. *Tetrahedron Lett.* **1988**, *29*, 979-982.

11. Deslongchamps, P; Moreu, C.; Fréhel; D.; Chênevert, R. *Can. J. Chem.* **1975**, *53*, 1204-1210.

12. Hsu, D-S.; Matsumoto, T.; Suzuki, K. *Synlett* **2005**, 801-804.

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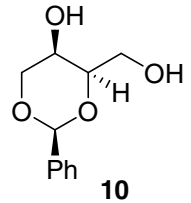

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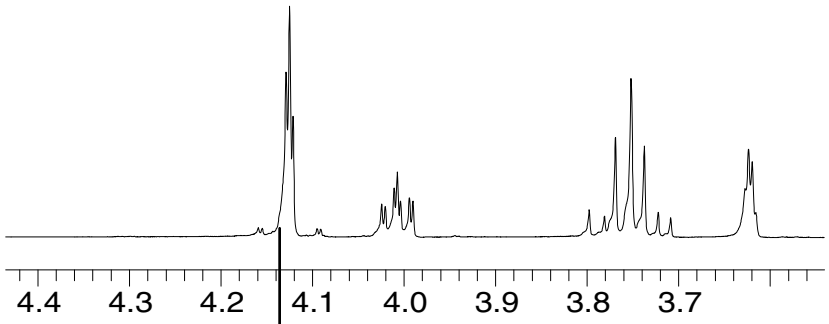

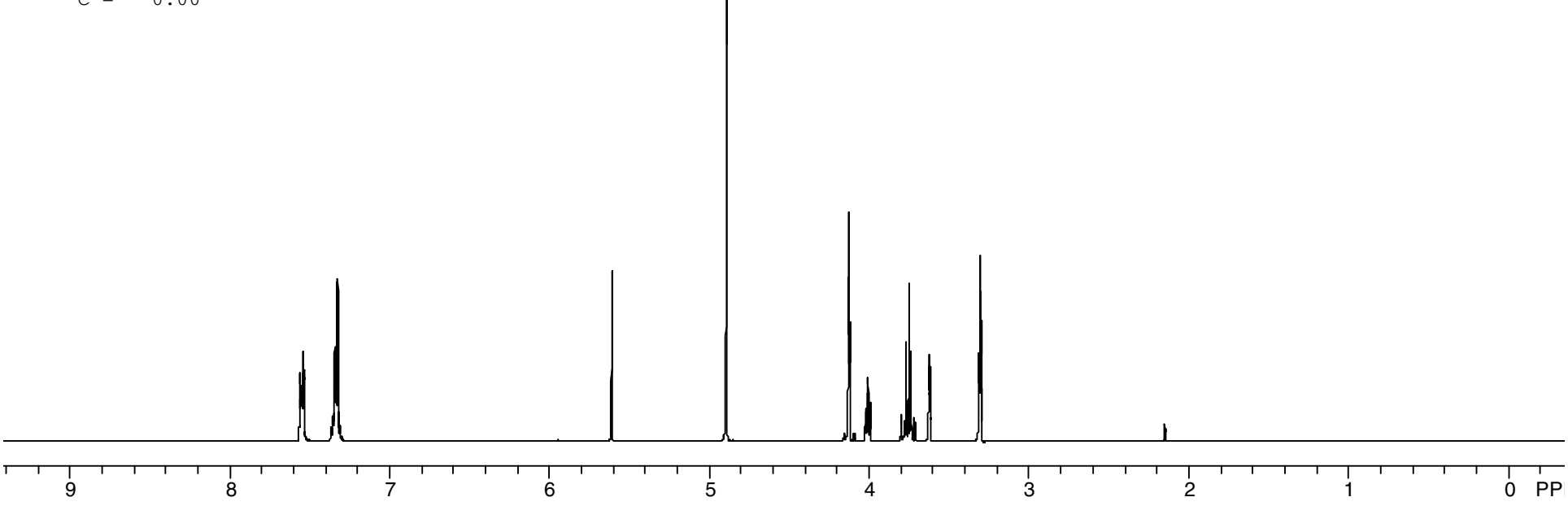

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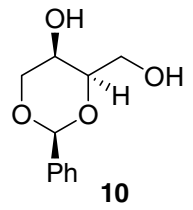

10

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Hz per Pt 2ndD = 1.00 Hz
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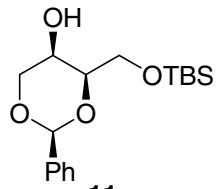
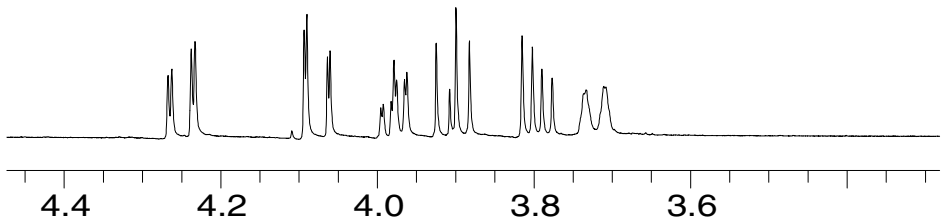


11



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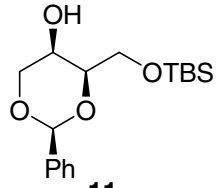
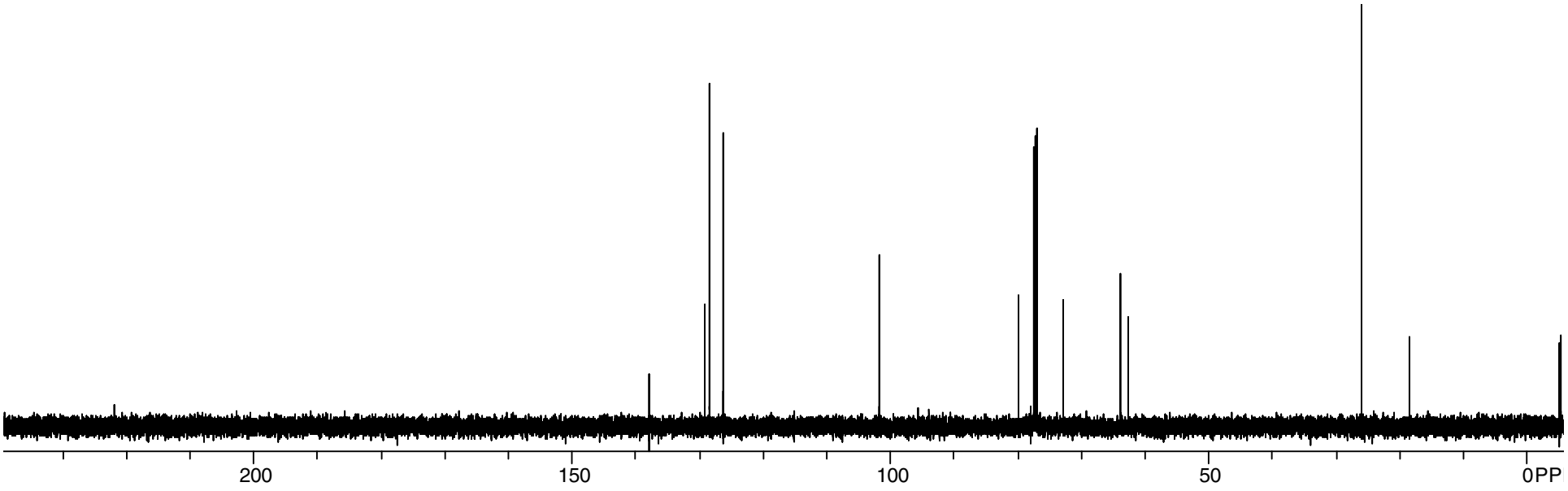
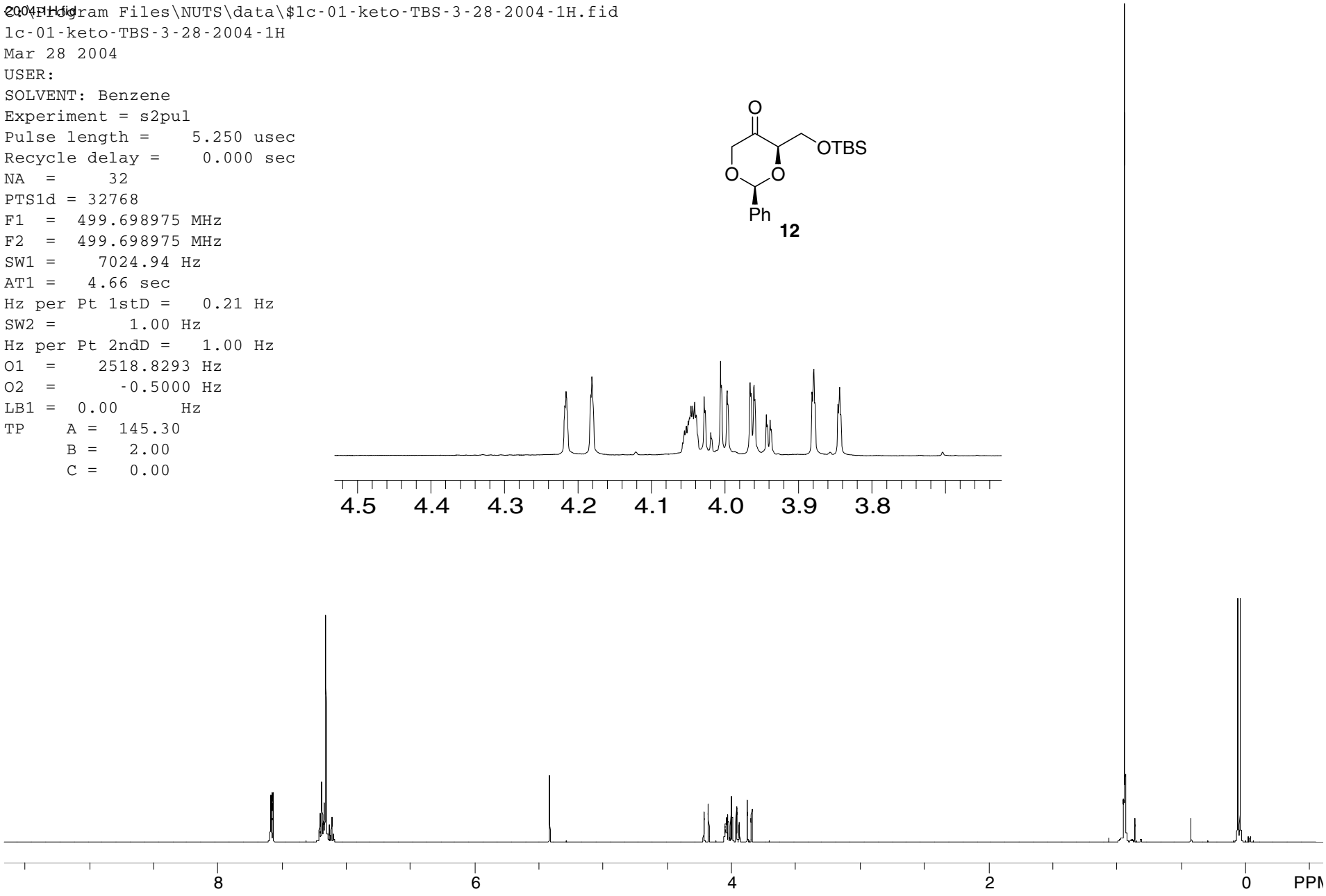


11




2004-1H.fid
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8
6
4
2
0
PPM

Interpolated Peak Listing

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17	55746	51067K	6.26	3245.15	25.824
18	55748	23084K	2.83	3244.30	25.818
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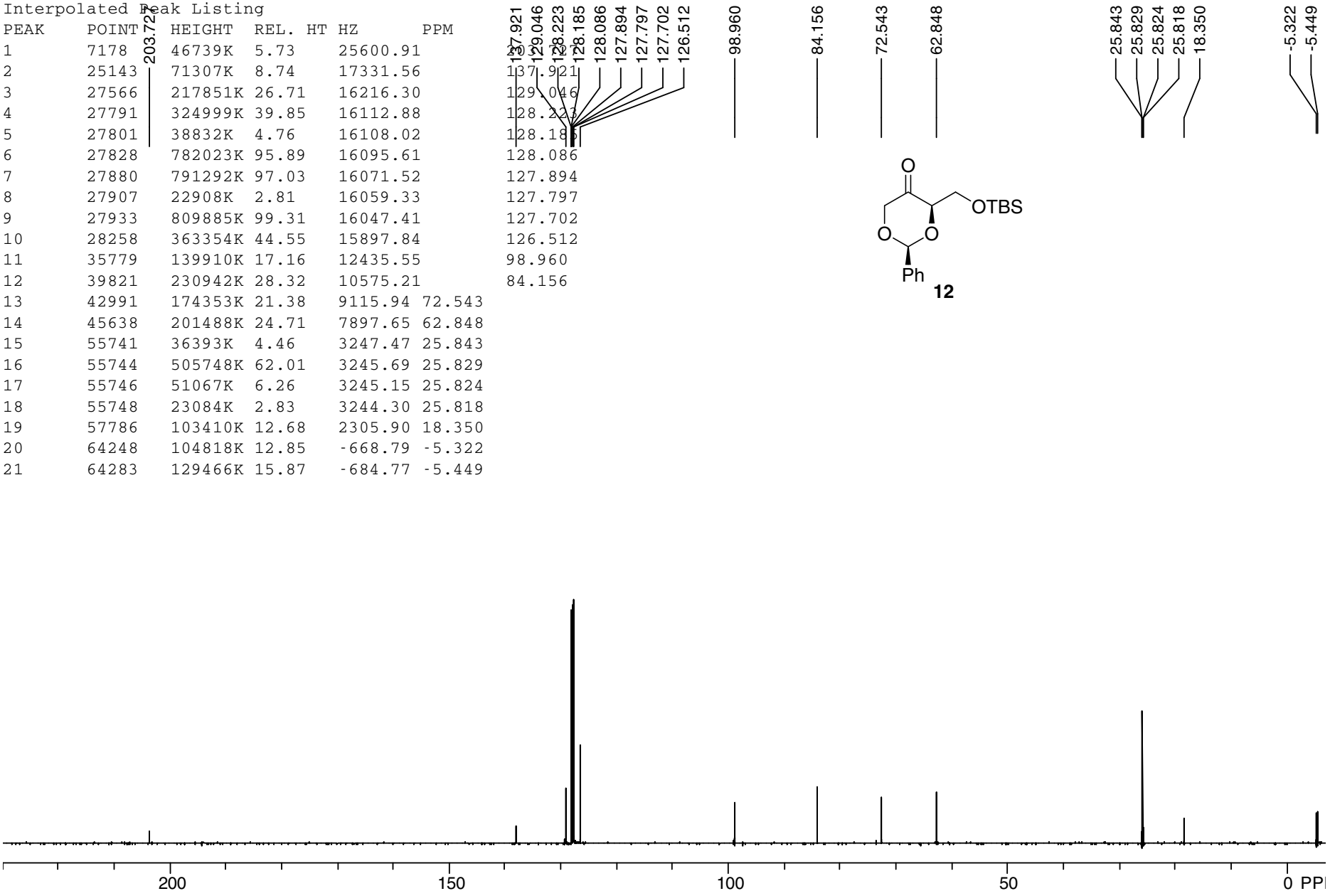

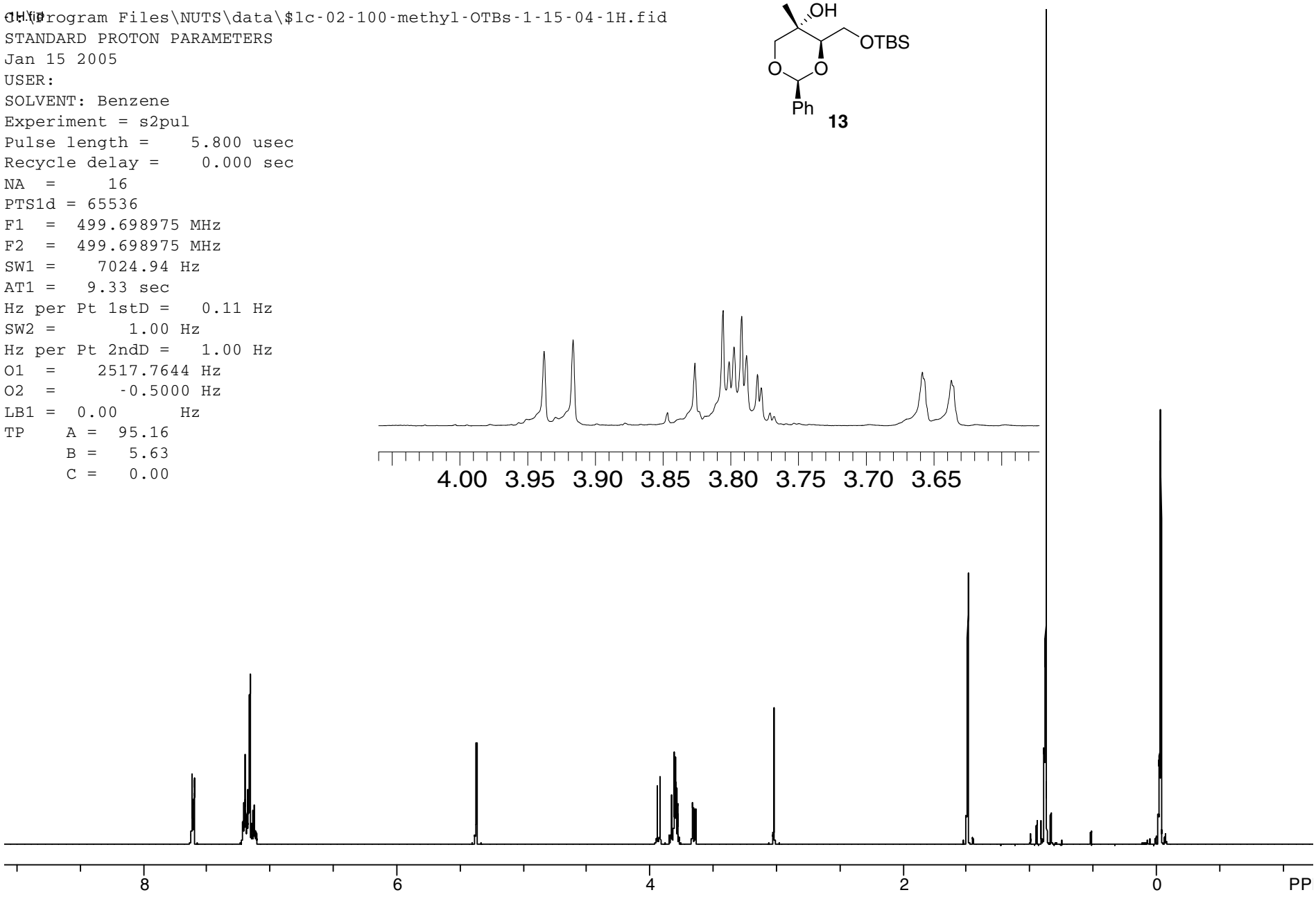
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8
6
4
2
0
PP

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Jan 15 2005
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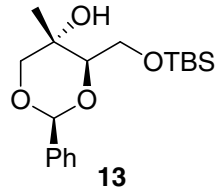
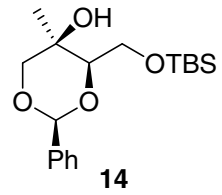
B = 129.00
C = 0.00

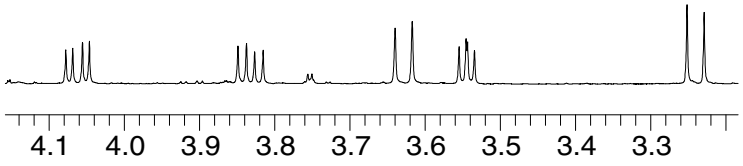


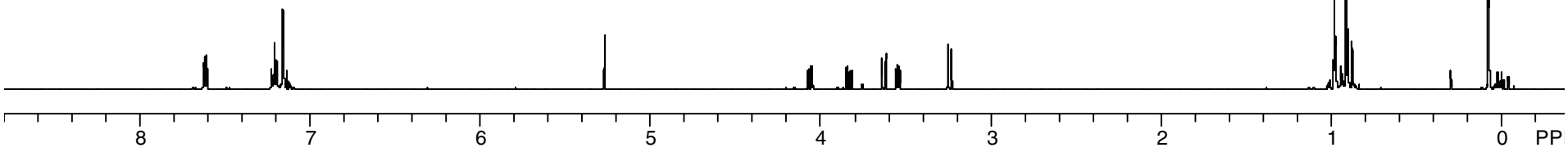
13

C:\Program Files\NUTS\data\$upper-Me-OH-4-20-07-1H.fid
upper-Me-OH-4-20-07-1H
Apr 20 2007
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 32
PTS1d = 65536
F1 = 499.698975 MHz
F2 = 499.698975 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2518.8926 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 175.31
  B = -7.03
  C = 0.00

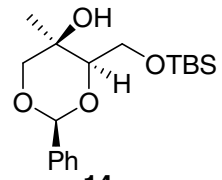


14

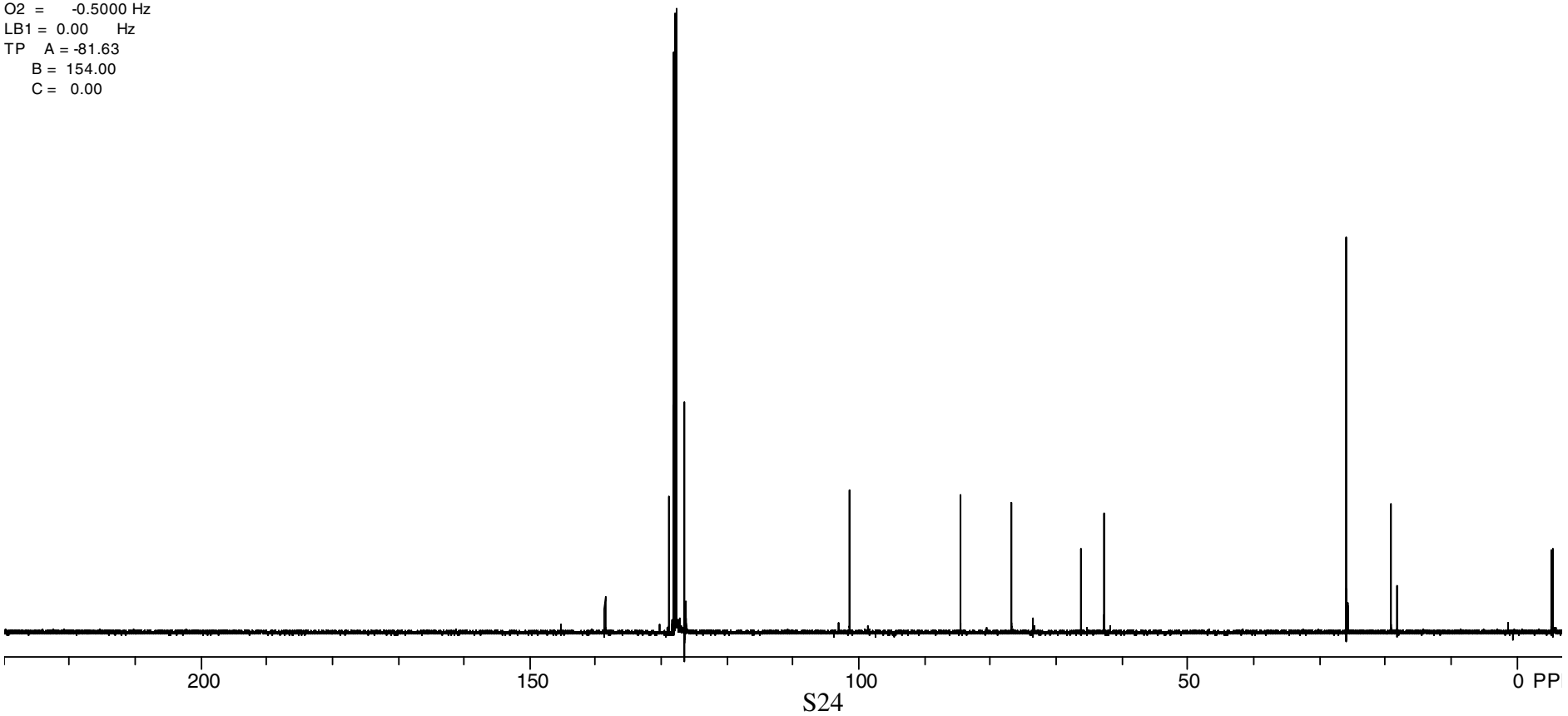


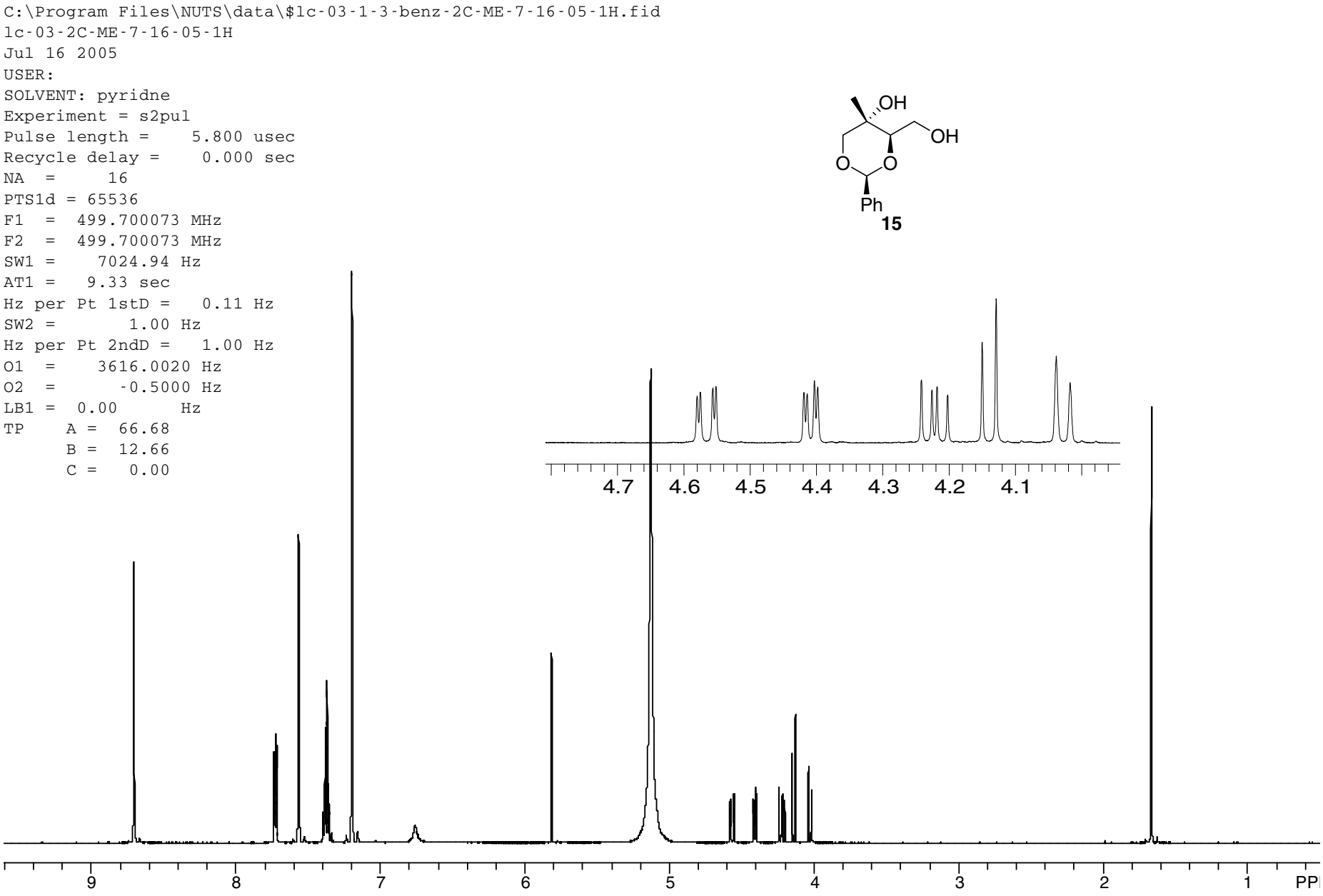


C:\Program Files\NUTS\data\$upper-MeOH-TBS-4-20-07-13C.fid
upper-Me-OH-4-20-07-13C
Apr 20 2007
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 6.000 usec
Recycle delay = 1.000 sec
NA = 245
PTS1d = 65536
F1 = 125.662575 MHz
F2 = 499.698151 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7314 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -81.63
B = 154.00
C = 0.00

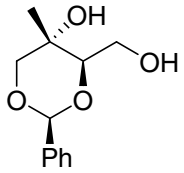


14

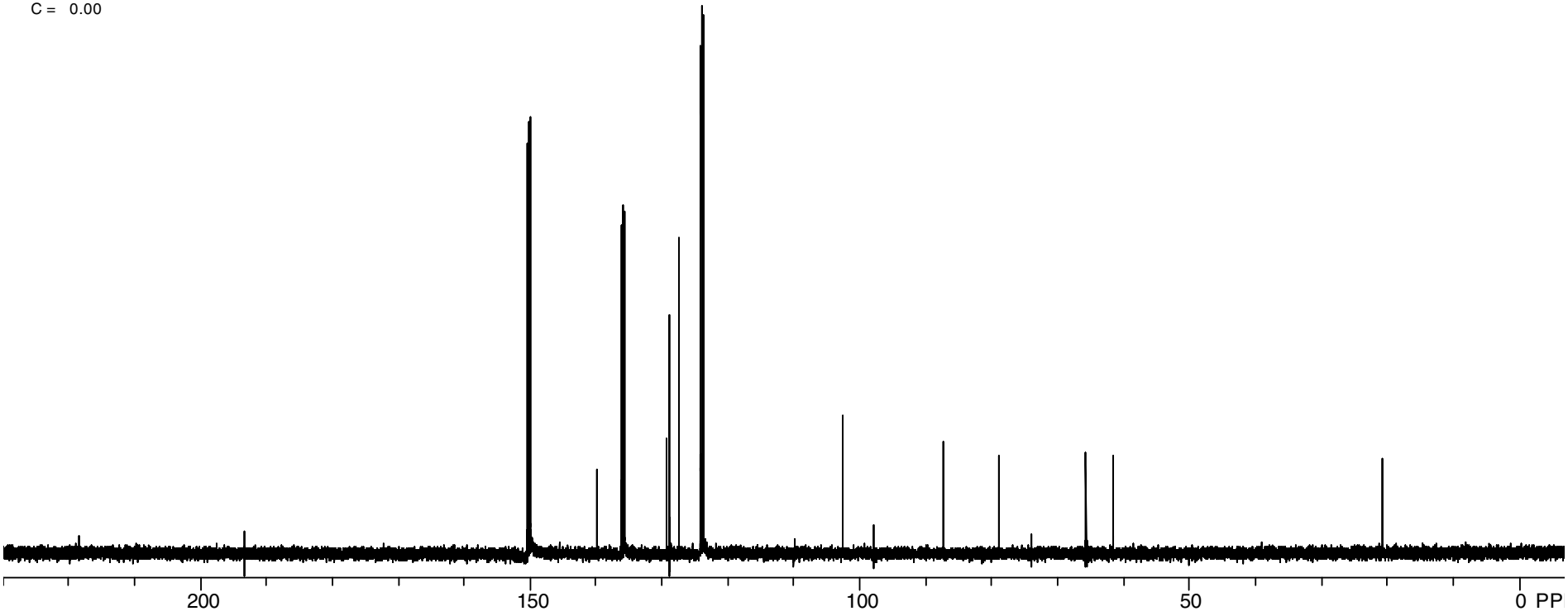



C:\Program Files\NUTS\data\$1c-03-1-3-benz-2C-ME-7-16-05-1H.fid
1c-03-2C-ME-7-16-05-1H
Jul 16 2005
USER:
SOLVENT: pyridne
Experiment = s2pul
Pulse length = 5.800 usec
Recycle delay = 0.000 sec
NA = 16
PTS1d = 65536
F1 = 499.700073 MHz
F2 = 499.700073 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 3616.0020 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 66.68
B = 12.66
C = 0.00
OH
OH
O
O
Ph
15
4.7
4.6
4.5
4.4
4.3
4.2
4.1
9
8
7
6
5
4
3
2
1
PP

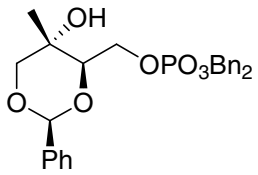
C:\Program Files\NUTS\data\$Ic-03-1-3-benz-2C-ME-7-16-05-13C.fid
Ic-03-1,3-benz-2C-ME-7-16-05-13C
Jul 16 2005
USER:
SOLVENT: pyridine
Experiment = s2pul
Pulse length = 4.300 usec
Recycle delay = 1.000 sec
NA = 203
PTS1d = 65536
F1 = 125.662521 MHz
F2 = 499.697968 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.6904 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -59.06
B = 172.00
C = 0.00



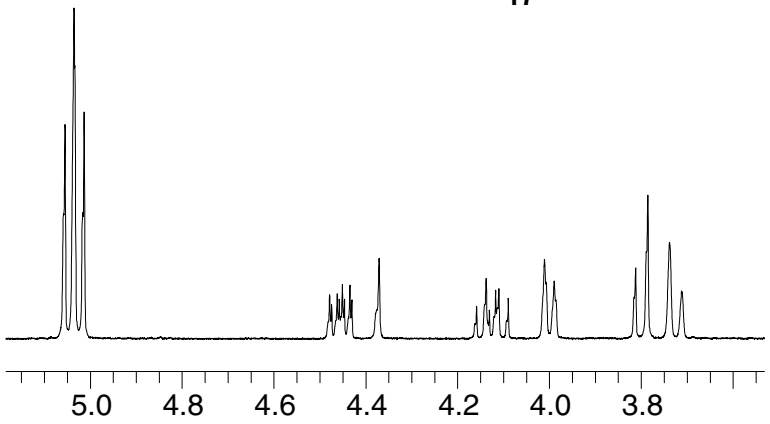
15

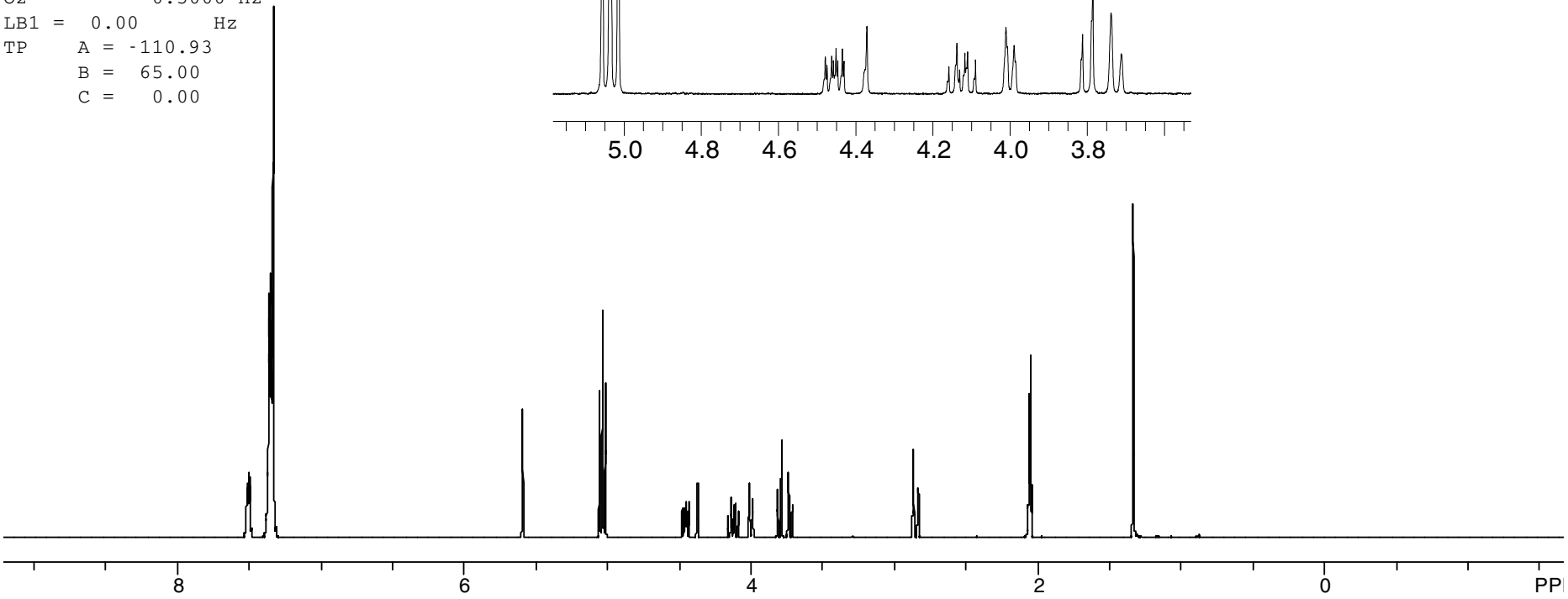


C:\Program Files\NUTS\data\$lc-03--benz-ery-mono-phosho-5-24-05-1H.fid
lc-03--benz-ery-mono-phosho-5-24-05-1H
May 24 2005
USER:
SOLVENT: Acetone
Experiment = s2pul
Pulse length = 8.975 usec
Recycle delay = 0.000 sec
NA = 8
PTS1d = 65536
F1 = 399.952759 MHz
F2 = 399.953186 MHz
SW1 = 8000.00 Hz
AT1 = 8.19 sec
Hz per Pt 1stD = 0.12 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 1999.8096 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -110.93
B = 65.00
C = 0.00

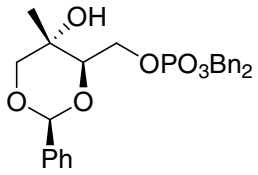


17

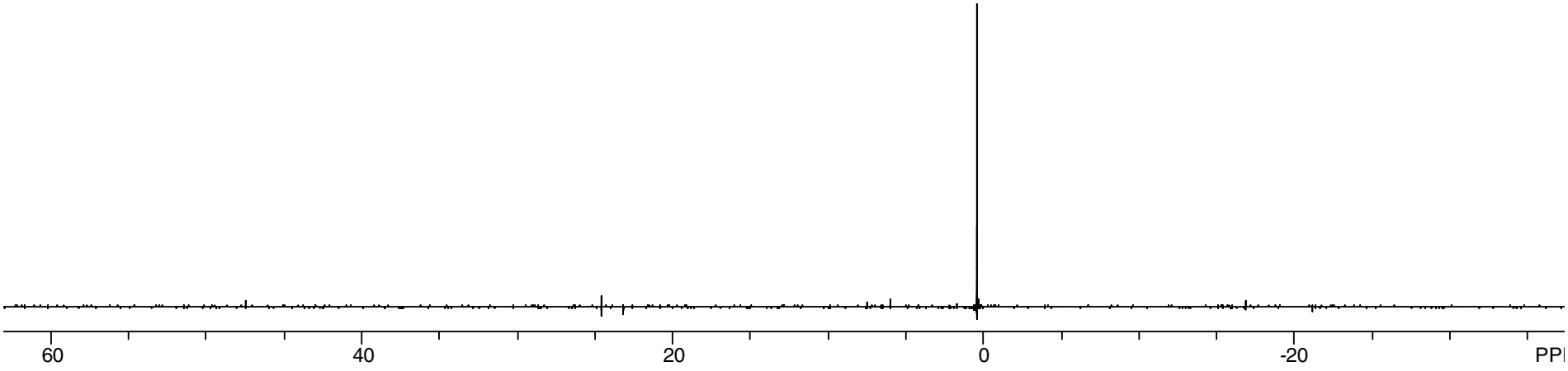




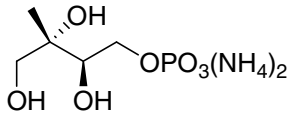
```
C:\Program Files\NUTS\data\$lc-03--benz-ery-mono-phosho-5-24-05-31P.fid
lc-03--benz-ery-mono-phosho-5-24-05-31P
May 24 2005
USER:
SOLVENT: Acetone
Experiment = s2pul
Pulse length =    4.700 usec
Recycle delay =    1.000 sec
NA  =    141
PTS1d = 65536
F1  =  161.903473 MHz
F2  =  399.952087 MHz
SW1 =   40000.00 Hz
AT1 =   1.64 sec
Hz per Pt 1stD =   0.61 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =     625.2930 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  140.43
      B =   5.00
      C =   0.00
```



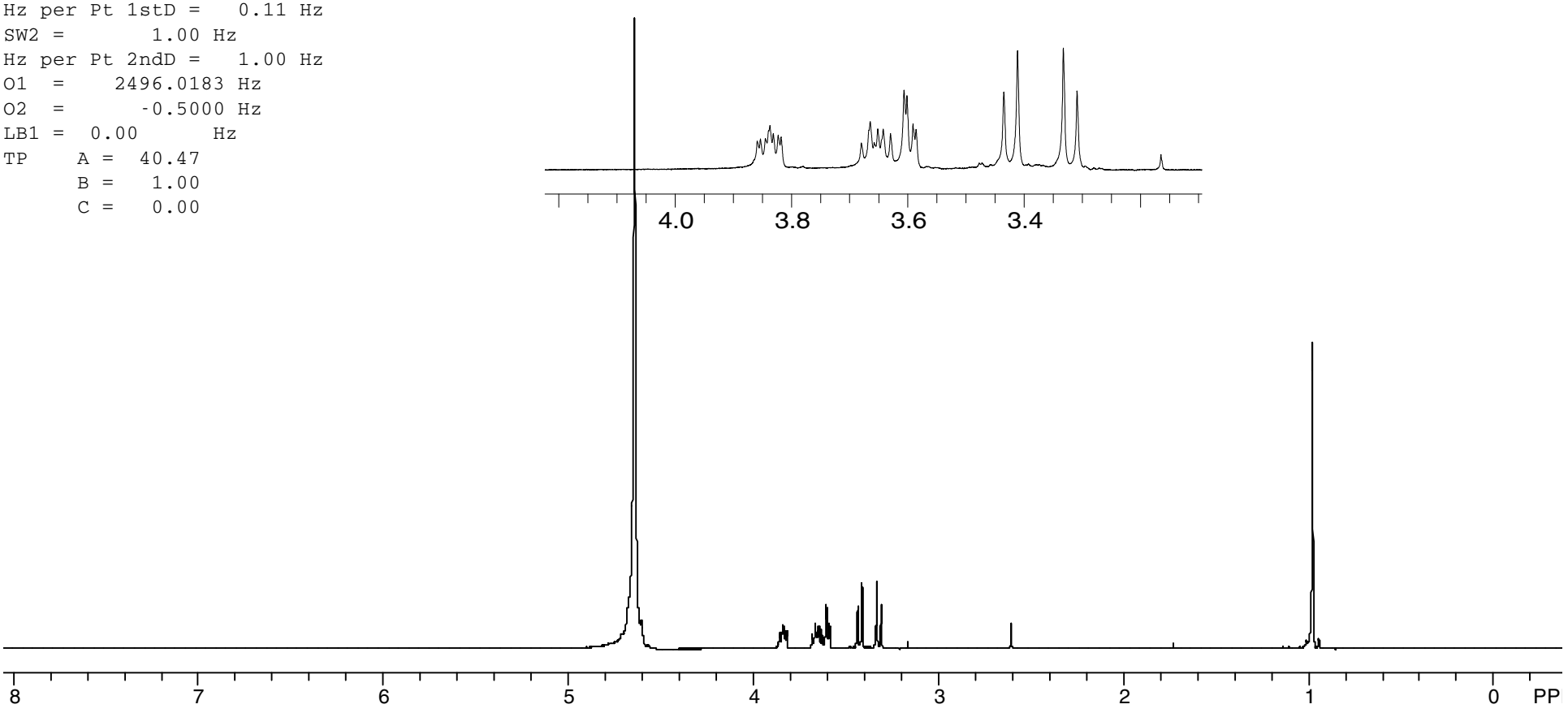
17



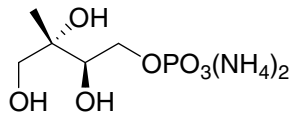
```
C:\Program Files\NUTS\data\$lc-03--2C-MEP-5-24-05-1H.fid
lc-03--2C-MEP-5-24-05-1H
May 24 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length =    5.800 usec
Recycle delay =    0.000 sec
NA  =     16
PTS1d = 65536
F1  =  499.700226 MHz
F2  =  499.700226 MHz
SW1 =    7024.94 Hz
AT1 =   9.33 sec
Hz per Pt 1stD =   0.11 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =    2496.0183 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  40.47
      B =   1.00
      C =   0.00
```



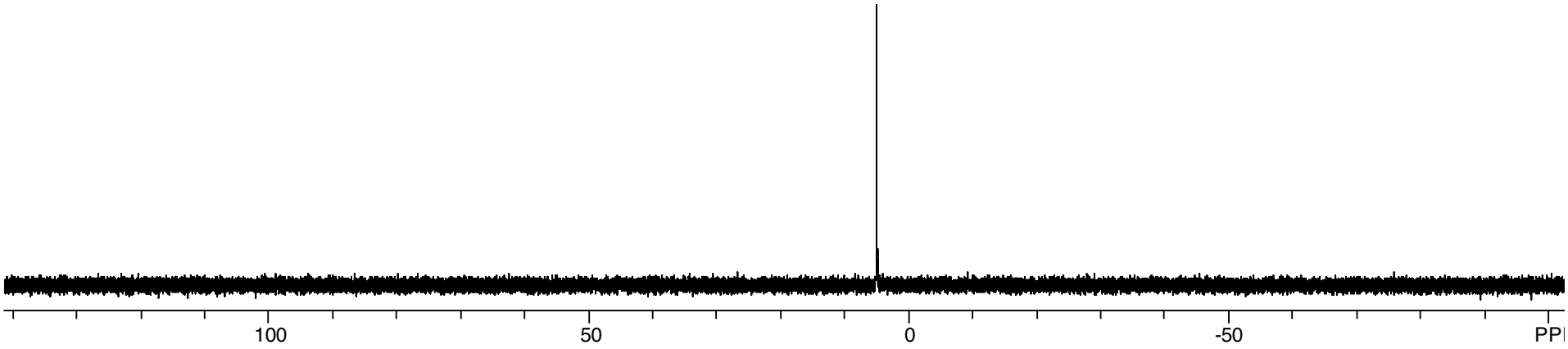
2

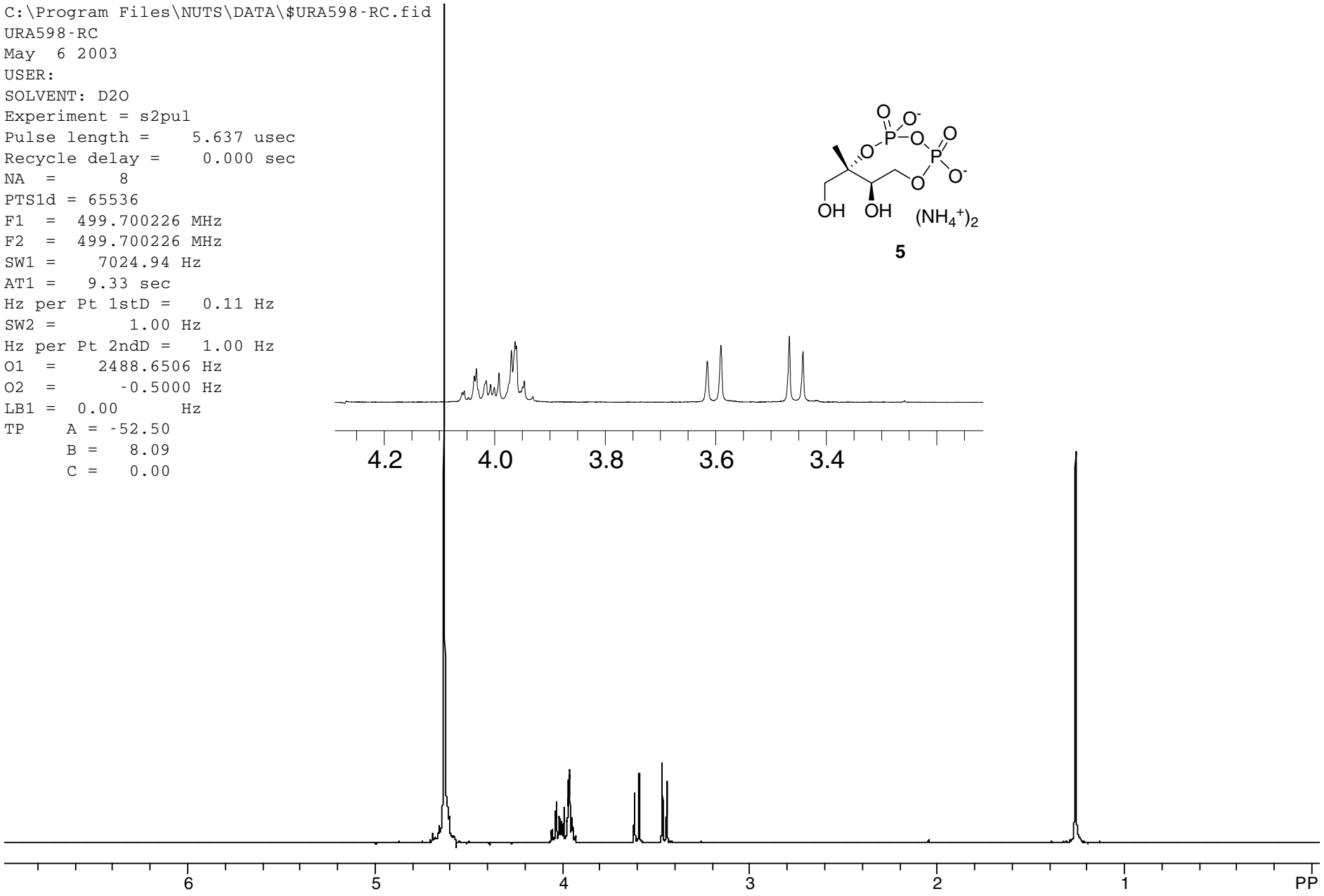


C:\Program Files\NUTS\data\$Ic-03--2C-MEP-5-24-05-31P.fid
Ic-03--2C-MEP-5-24-05-31P
May 24 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 4.825 usec
Recycle delay = 1.000 sec
NA = 117
PTS1d = 65536
F1 = 202.284744 MHz
F2 = 499.699402 MHz
SW1 = 50000.00 Hz
AT1 = 1.31 sec
Hz per Pt 1stD = 0.76 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 3622.5781 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 374.06
B = -378.28
C = 0.00

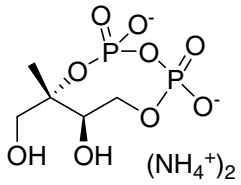


2

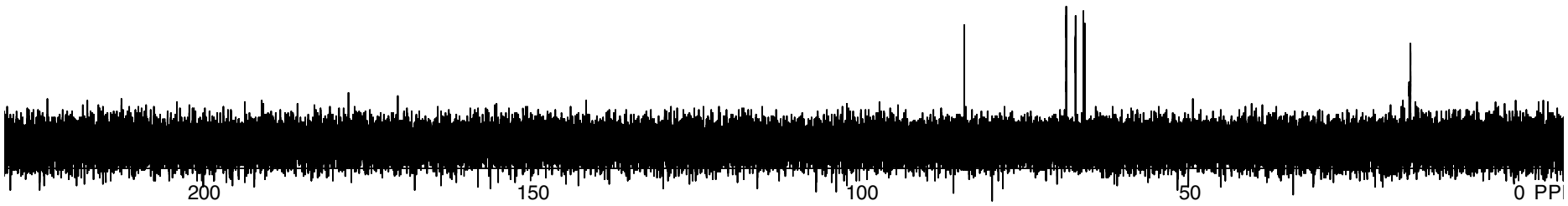



C:\Program Files\NUTS\DATA\$URA598-RC.fid
URA598-RC
May 6 2003
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 5.637 usec
Recycle delay = 0.000 sec
NA = 8
PTS1d = 65536
F1 = 499.700226 MHz
F2 = 499.700226 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2488.6506 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -52.50
B = 8.09
C = 0.00
OH OH
(NH4+)2
5
4.2
4.0
3.8
3.6
3.4
6
5
4
3
2
1
PP

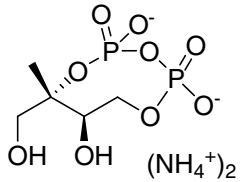
C:\Program Files\NUTS\DATA\$URA598-RC_13C.fid
URA598-RC
May 6 2003
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 4.500 usec
Recycle delay = 1.000 sec
NA = 499
PTS1d = 65536
F1 = 125.662888 MHz
F2 = 499.699402 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7246 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -101.72
B = 56.25
C = 0.00



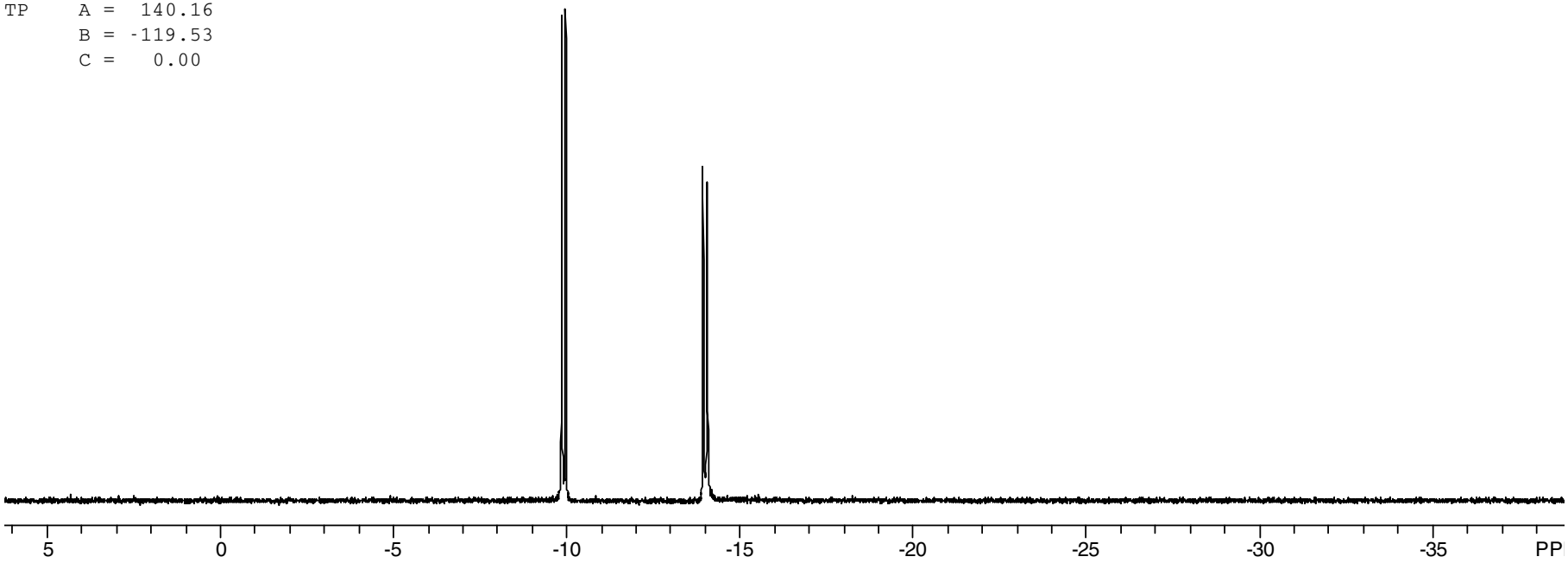
5

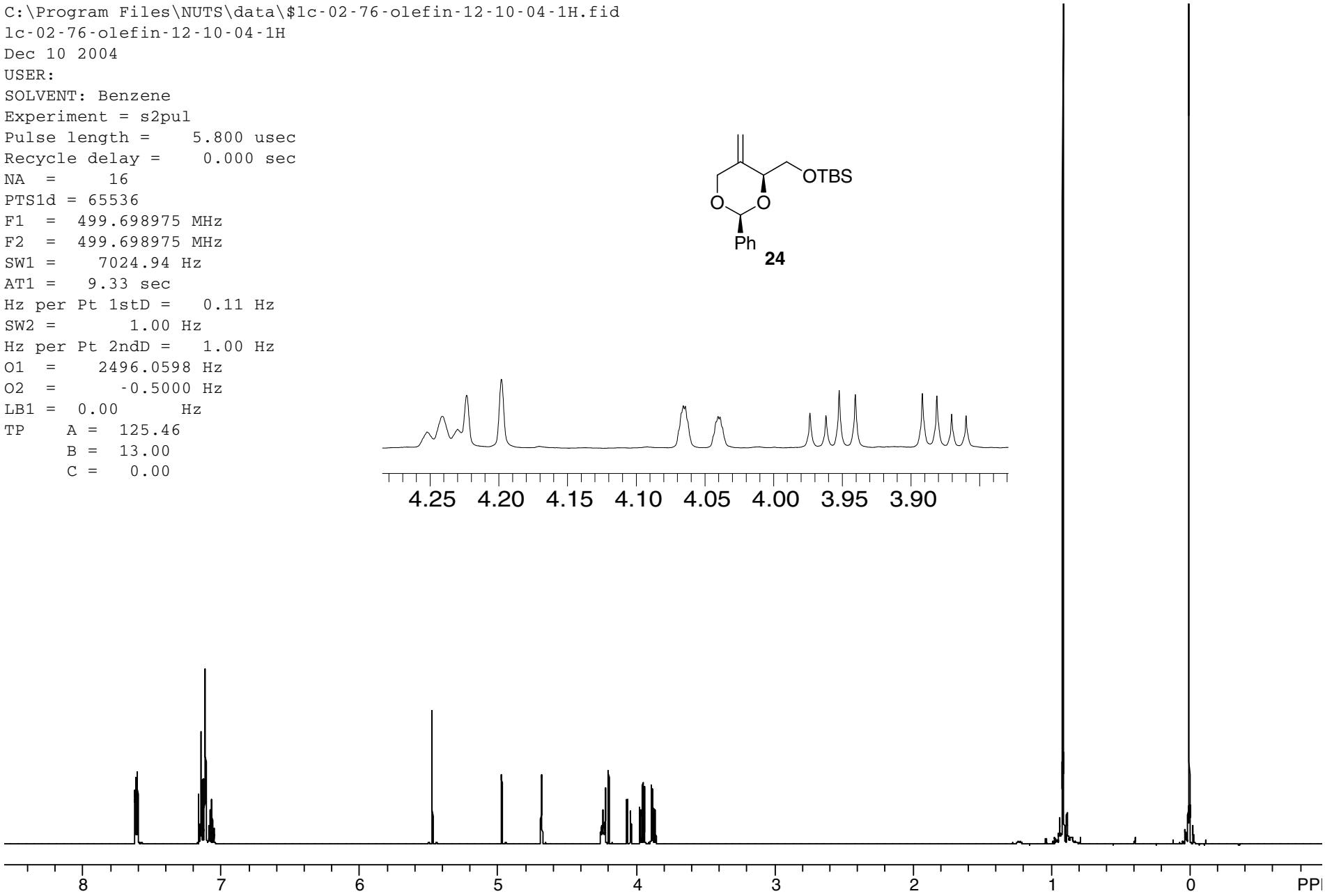


```
C:\Program Files\NUTS\DATA\$URA598-RC_31P.fid
URA598-RC
May  6 2003
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length =    4.375 usec
Recycle delay =    1.000 sec
NA  =    209
PTS1d = 65536
F1  =  202.284744 MHz
F2  =  499.699402 MHz
SW1 =   50000.00 Hz
AT1 =   1.31 sec
Hz per Pt 1stD =   0.76 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =    3622.5781 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  140.16
      B = -119.53
      C =   0.00
```

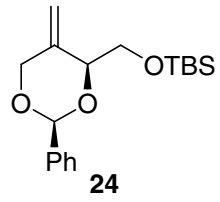


5

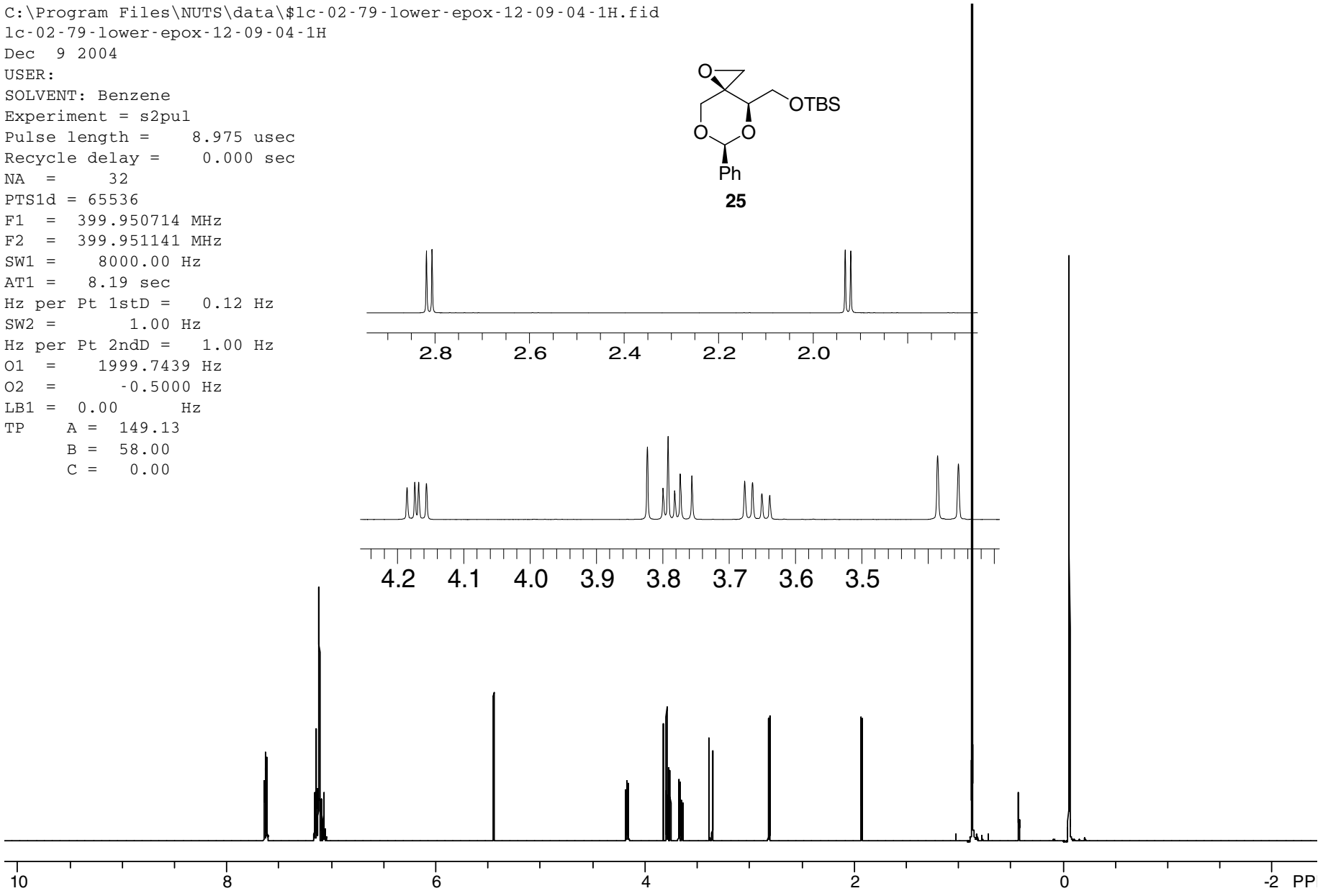



C:\Program Files\NUTS\data\$1c-02-76-olefin-12-10-04-1H.fid
1c-02-76-olefin-12-10-04-1H
Dec 10 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 5.800 usec
Recycle delay = 0.000 sec
NA = 16
PTS1d = 65536
F1 = 499.698975 MHz
F2 = 499.698975 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0598 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 125.46
B = 13.00
C = 0.00
OTBS
O
O
Ph
24
4.25 4.20 4.15 4.10 4.05 4.00 3.95 3.90
8 7 6 5 4 3 2 1 0 PPI

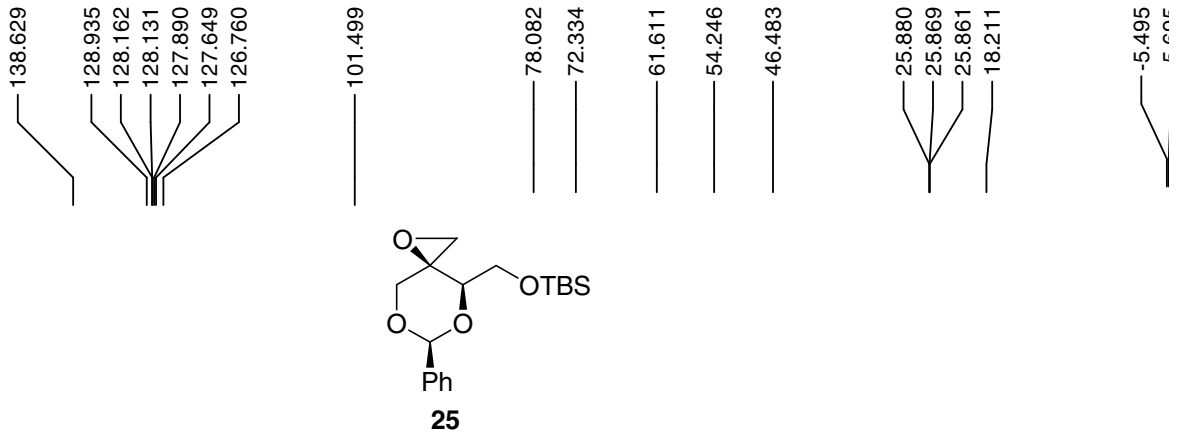
C:\Program Files\NUTS\data\$lc-02-76-olefin-12-10-04-13C.fid
lc-02-76-olefin-12-10-04-13C
Dec 10 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 4.300 usec
Recycle delay = 1.000 sec
NA = 232
PTS1d = 65536
F1 = 125.662575 MHz
F2 = 499.698151 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7314 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -154.77
B = 129.00
C = 0.00



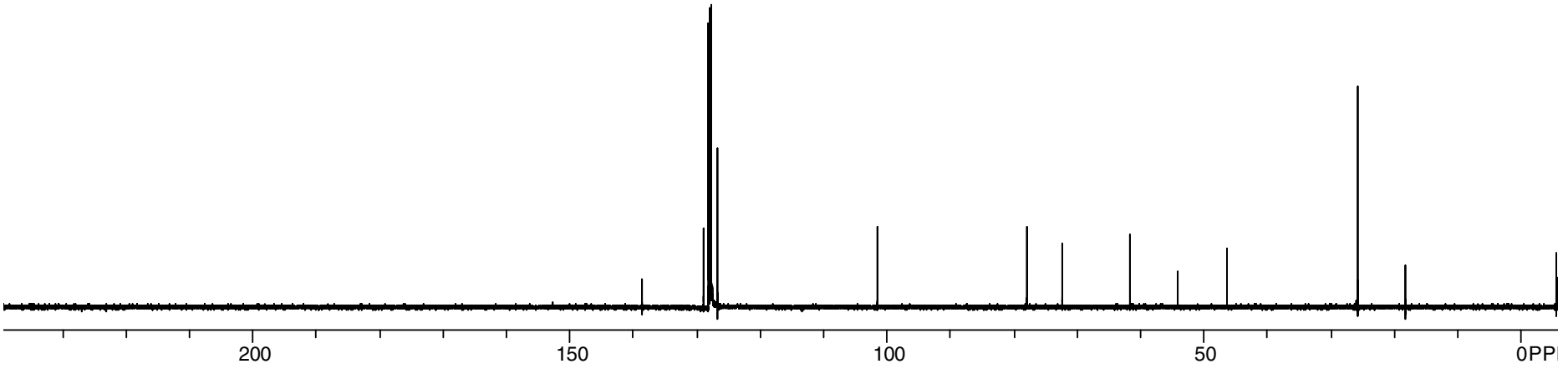
24

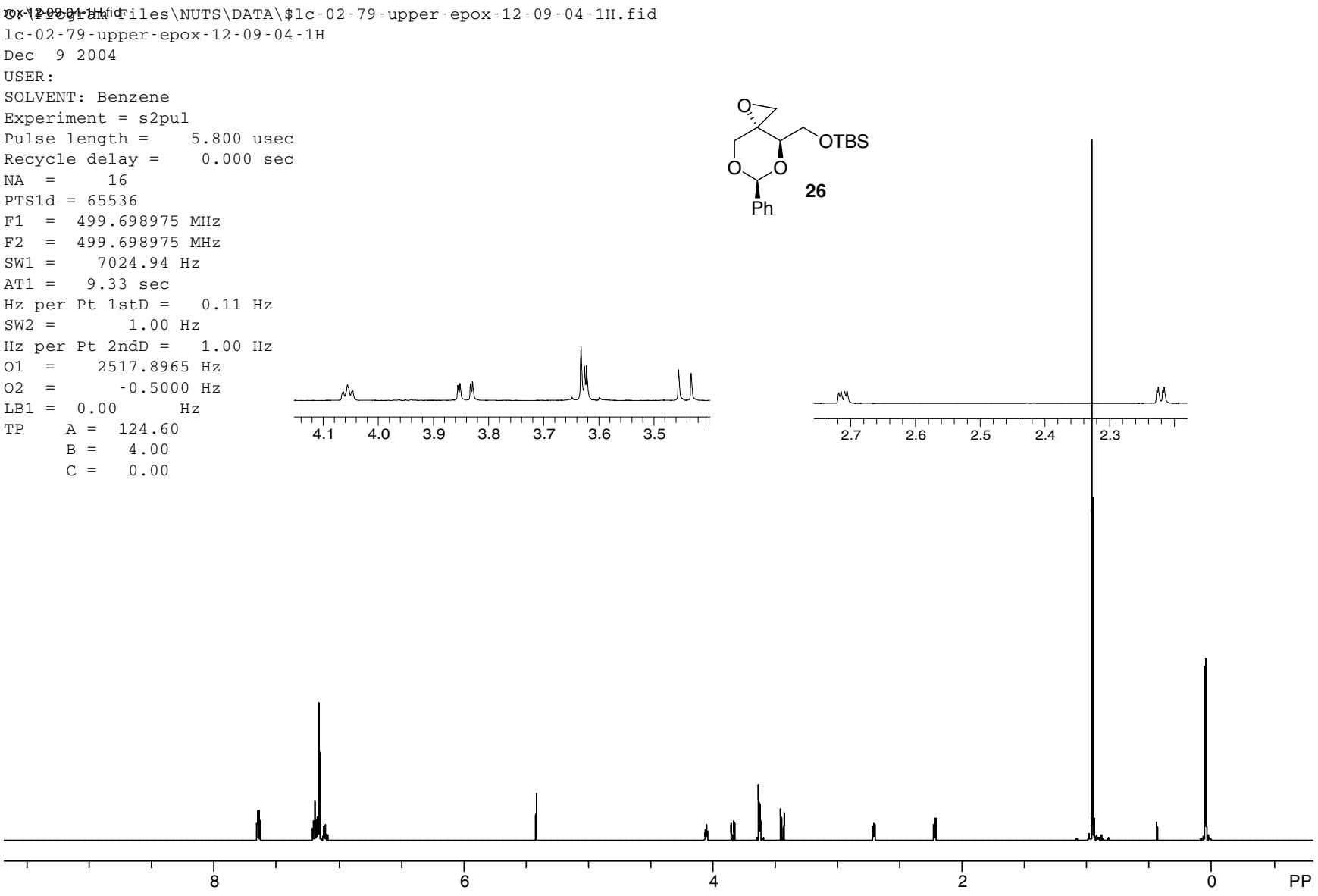

C:\Program Files\NUTS\data\$lc-02-79-lower-epox-12-09-04-1H.fid
lc-02-79-lower-epox-12-09-04-1H
Dec 9 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 8.975 usec
Recycle delay = 0.000 sec
NA = 32
PTS1d = 65536
F1 = 399.950714 MHz
F2 = 399.951141 MHz
SW1 = 8000.00 Hz
AT1 = 8.19 sec
Hz per Pt 1stD = 0.12 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 1999.7439 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 149.13
B = 58.00
C = 0.00
O
OTBS
O
O
Ph
25
2.8
2.6
2.4
2.2
2.0
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
10
8
6
4
2
0
-2
PP

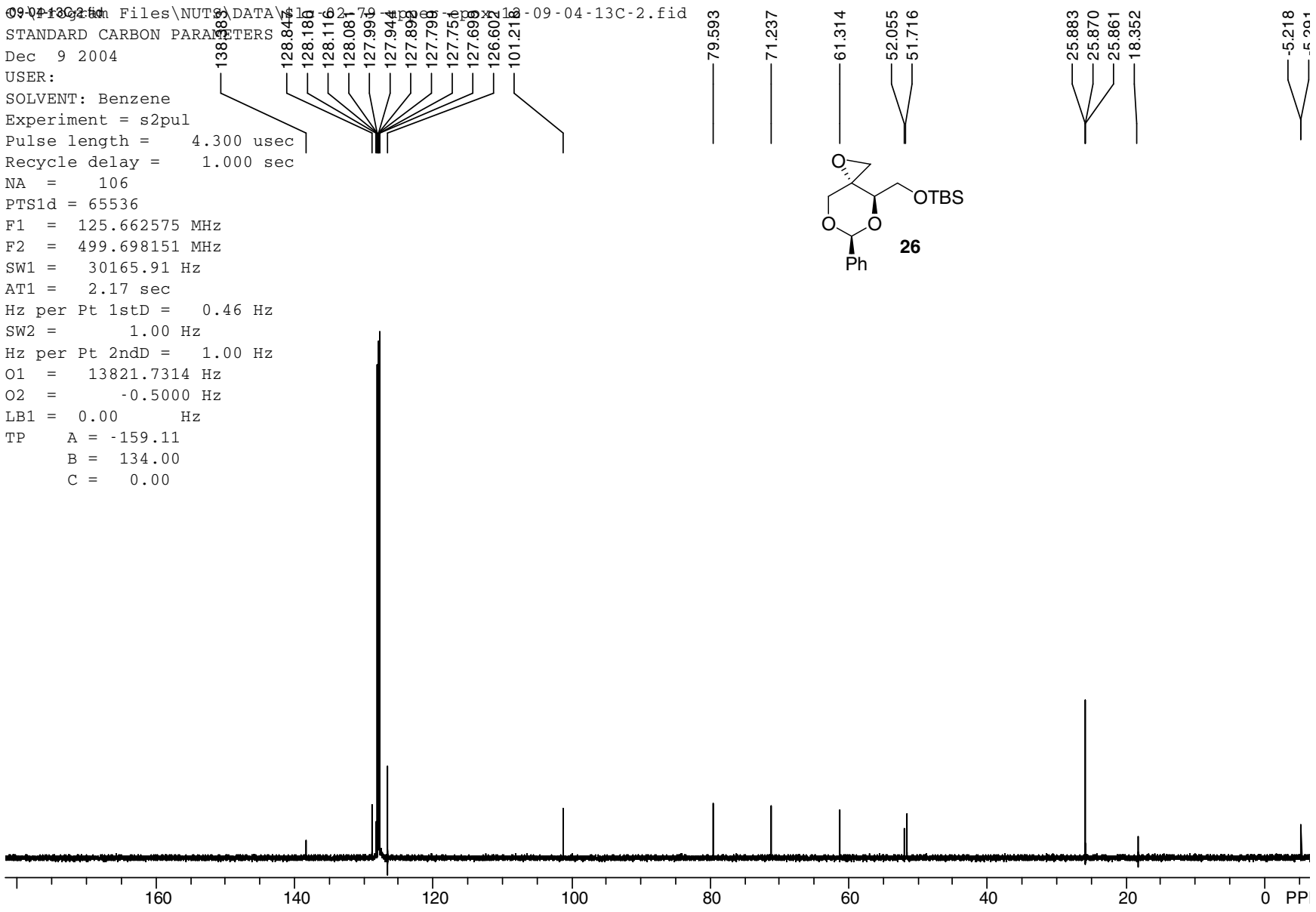
C:\Program Files\NUTS\DATA\$lc-02-79-lower-epox-12-09-04-13C.fid
lc-02-79-lower-epox-12-09-04-13C
Dec 9 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 7.075 usec
Recycle delay = 1.000 sec
NA = 248
PTS1d = 65536
F1 = 100.578751 MHz
F2 = 399.950043 MHz
SW1 = 25000.00 Hz
AT1 = 2.62 sec
Hz per Pt 1stD = 0.38 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 11589.9160 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 62.54
B = 221.00
C = 0.00



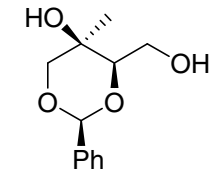
25



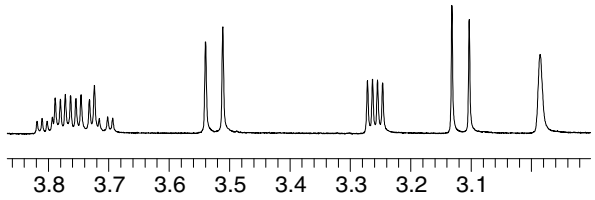

C:\Program Files\NUTS\DATA\$lc-02-79-upper-epox-12-09-04-1H.fid
lc-02-79-upper-epox-12-09-04-1H
Dec 9 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 5.800 usec
Recycle delay = 0.000 sec
NA = 16
PTS1d = 65536
F1 = 499.698975 MHz
F2 = 499.698975 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2517.8965 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 124.60
B = 4.00
C = 0.00
O
OTBS
O
O
Ph
26
4.1
4.0
3.9
3.8
3.7
3.6
3.5
2.7
2.6
2.5
2.4
2.3
8
6
4
2
0
PP


09-04-13C-2.fid
C:\Program Files\NUTS\DATA\sla-02-79-4pmeo-epox\13-09-04-13C-2.fid
STANDARD CARBON PARAMETERS
Dec 9 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 4.300 usec
Recycle delay = 1.000 sec
NA = 106
PTS1d = 65536
F1 = 125.662575 MHz
F2 = 499.698151 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7314 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -159.11
B = 134.00
C = 0.00
138.483
128.844
128.180
128.116
128.081
127.991
127.944
127.890
127.798
127.751
127.695
126.602
101.218
79.593
71.237
61.314
52.055
51.716
25.883
25.870
25.861
18.352
-5.218
O
OTBS
O
O
Ph
26
160
140
120
100
80
60
40
20
0
PPM

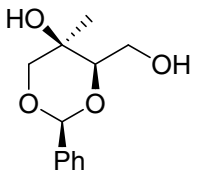
C:\Program Files\NUTS\DATA\$lc-02-82-LAH=threo-diol-12-16-04-1H.fid
lc-02-82-LAH=threo-diol-12-16-04-1H
Dec 16 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 8.975 usec
Recycle delay = 0.000 sec
NA = 8
PTS1d = 65536
F1 = 399.950714 MHz
F2 = 399.951141 MHz
SW1 = 8000.00 Hz
AT1 = 8.19 sec
Hz per Pt 1stD = 0.12 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2017.5828 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 148.74
B = 59.00
C = 0.00



27

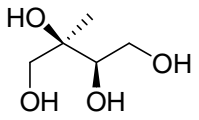


C:\Program Files\NUTS\data\$lc-02-83-lah-threo-12-16-04-13C.fid
lc-02-83-lah-threo-12-16-04-13C
Dec 16 2004
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 7.075 usec
Recycle delay = 1.000 sec
NA = 279
PTS1d = 65536
F1 = 100.578751 MHz
F2 = 399.950043 MHz
SW1 = 25000.00 Hz
AT1 = 2.62 sec
Hz per Pt 1stD = 0.38 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 11589.9160 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 70.84
B = 207.00
C = 0.00

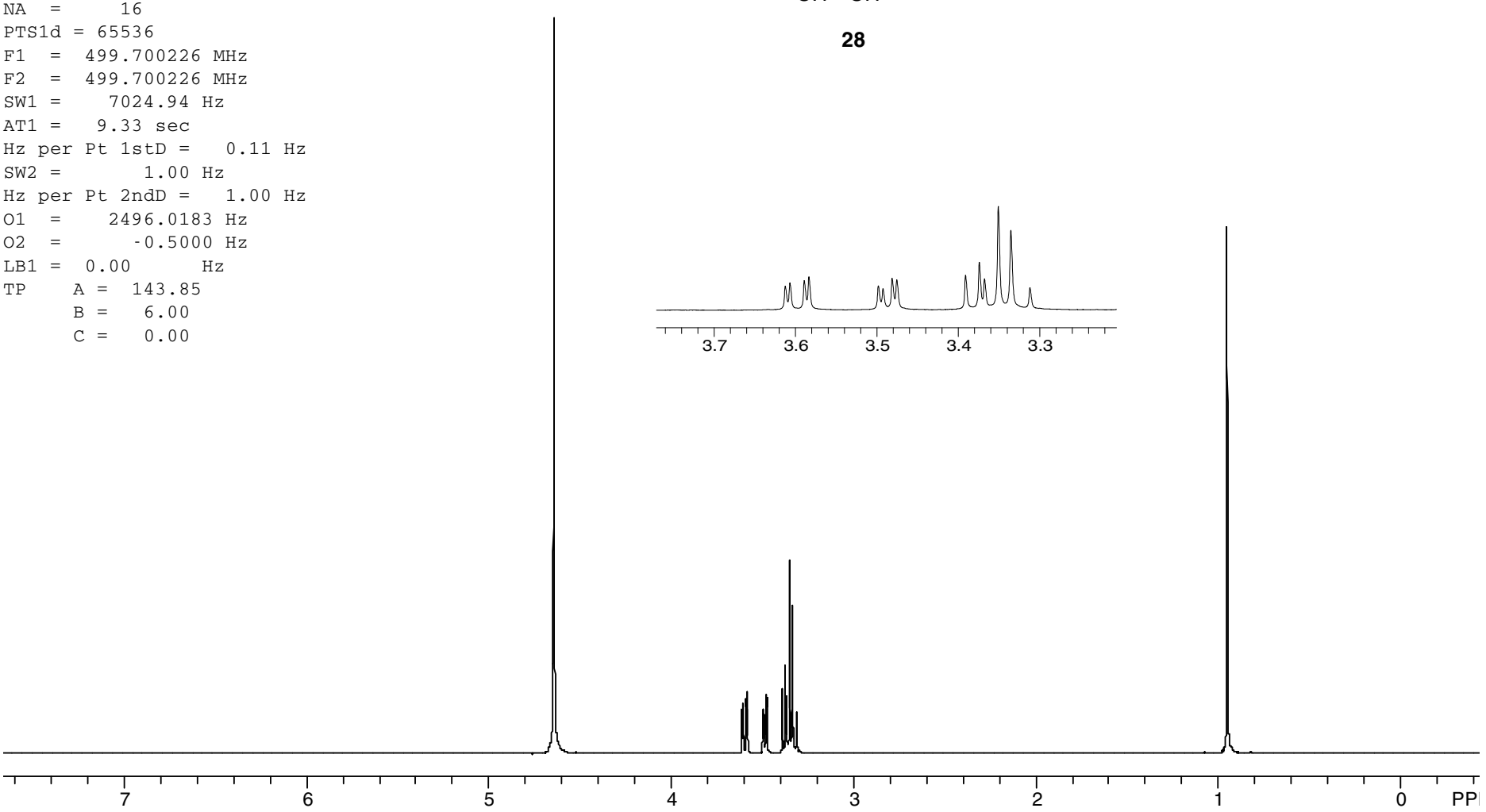


27

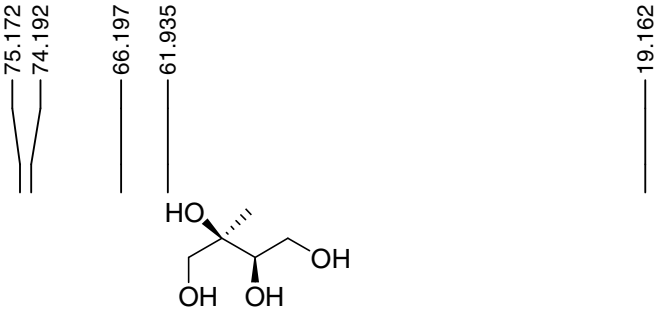
C:\Program Files\NUTS\DATA\$lc-02-88-threio-tetrol-12-21-04-1H.fid
lc-02-88-threio-tetrol-12-21-04-1H
Dec 21 2004
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 5.800 usec
Recycle delay = 0.000 sec
NA = 16
PTS1d = 65536
F1 = 499.700226 MHz
F2 = 499.700226 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0183 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 143.85
B = 6.00
C = 0.00



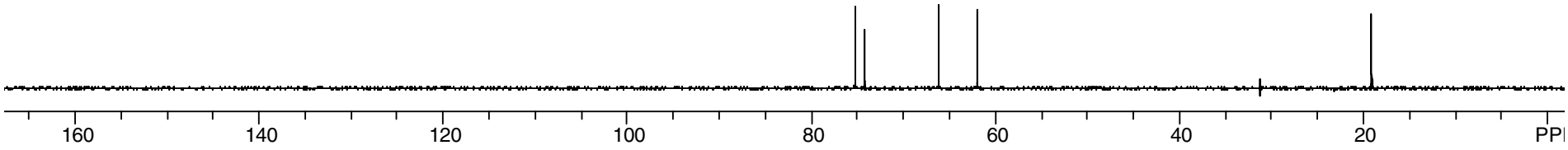
28



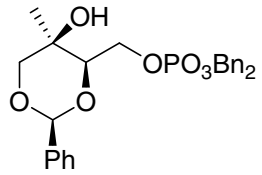
```
C:\Program Files\NUTS\DATA\$lc-02-86-threio-tetrol-12-21-04-13C.fid
lc-02-86-threio-tetrol-12-21-04-13C
Dec 21 2004
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length =    4.300 usec
Recycle delay =    1.000 sec
NA  =    483
PTS1d = 65536
F1  =  125.662888 MHz
F2  =  499.699402 MHz
SW1 =   30165.91 Hz
AT1 =   2.17 sec
Hz per Pt 1stD =   0.46 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =   13821.7246 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  21.20
      B =  117.00
      C =   0.00
```



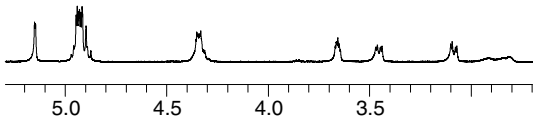
28

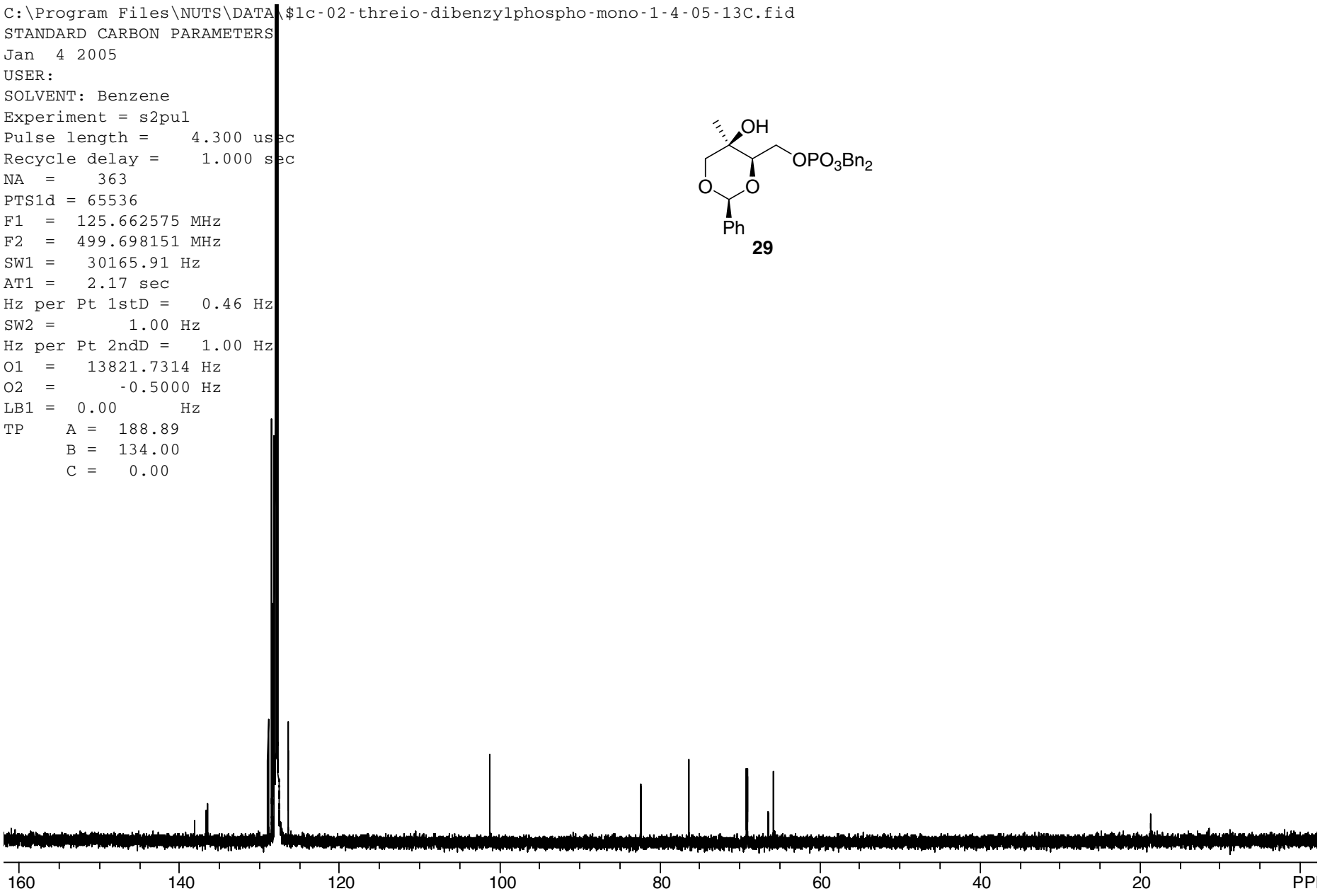


C:\Program Files\NUTS\DATA\$lc-02-threio-dibenzylphosph-1-4-04=1h.fid
STANDARD PROTON PARAMETERS
Jan 4 2005
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 5.800 usec
Recycle delay = 0.000 sec
NA = 4
PTS1d = 65536
F1 = 499.698975 MHz
F2 = 499.698975 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2517.0835 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 108.30
B = 2.00
C = 0.00

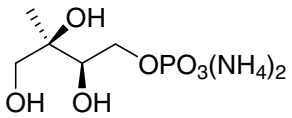


29

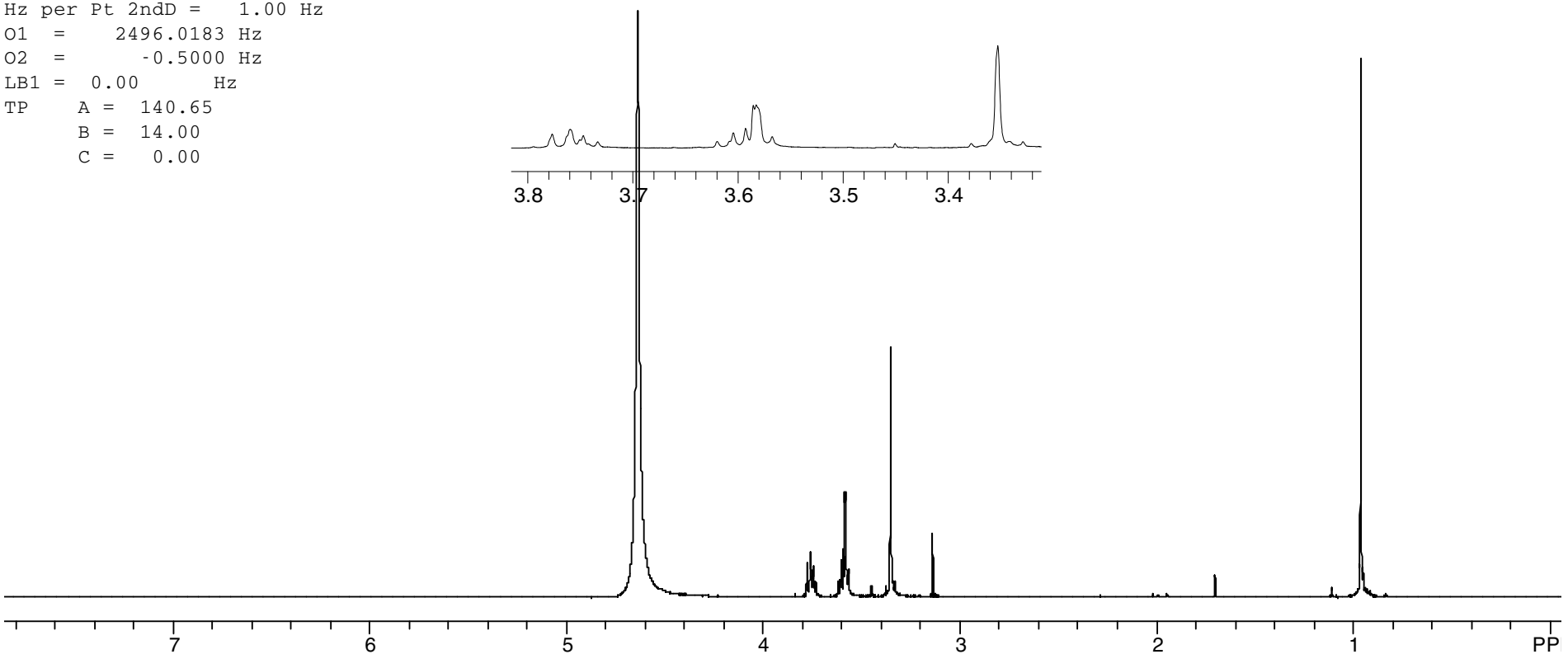



C:\Program Files\NUTS\DATA\$1c-02-threio-dibenzylphospho-mono-1-4-05-13C.fid
STANDARD CARBON PARAMETERS
Jan 4 2005
USER:
SOLVENT: Benzene
Experiment = s2pul
Pulse length = 4.300 usec
Recycle delay = 1.000 sec
NA = 363
PTS1d = 65536
F1 = 125.662575 MHz
F2 = 499.698151 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7314 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 188.89
B = 134.00
C = 0.00
OH
OPO3Bn2
O
O
Ph
29
160
140
120
100
80
60
40
20
PP

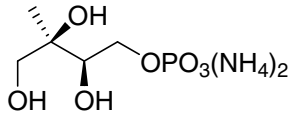
C:\Program Files\NUTS\data\$lc-02-91-threio-4-phospho-ammon-1-4-05-1H.fid
lc-02-91-threio-4-phospho-ammon-1-4-05-1H
Jan 4 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length =   5.800 usec
Recycle delay =   0.000 sec
NA =   8
PTS1d = 65536
F1 = 499.700226 MHz
F2 = 499.700226 MHz
SW1 =   7024.94 Hz
AT1 =   9.33 sec
Hz per Pt 1stD =   0.11 Hz
SW2 =   1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1 =   2496.0183 Hz
O2 =   -0.5000 Hz
LB1 = 0.00   Hz
TP   A = 140.65
      B = 14.00
      C =   0.00



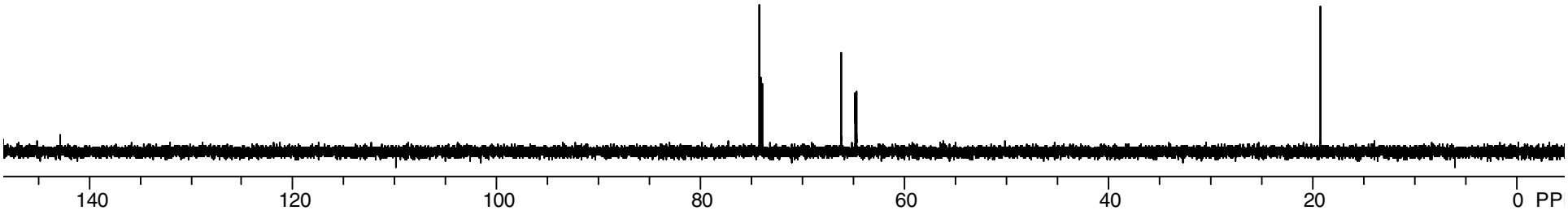
30



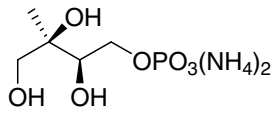
```
C:\Program Files\NUTS\data\$lc-02-91-threio-4-phosphoammo-1-4-04-13C.fid
STANDARD CARBON PARAMETERS
Jan  4 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length =    4.300 usec
Recycle delay =    1.000 sec
NA  =    396
PTS1d = 65536
F1  =  125.662888 MHz
F2  =  499.699402 MHz
SW1 =   30165.91 Hz
AT1 =   2.17 sec
Hz per Pt 1stD =   0.46 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =   13821.7246 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  53.24
      B =  69.00
      C =   0.00
```



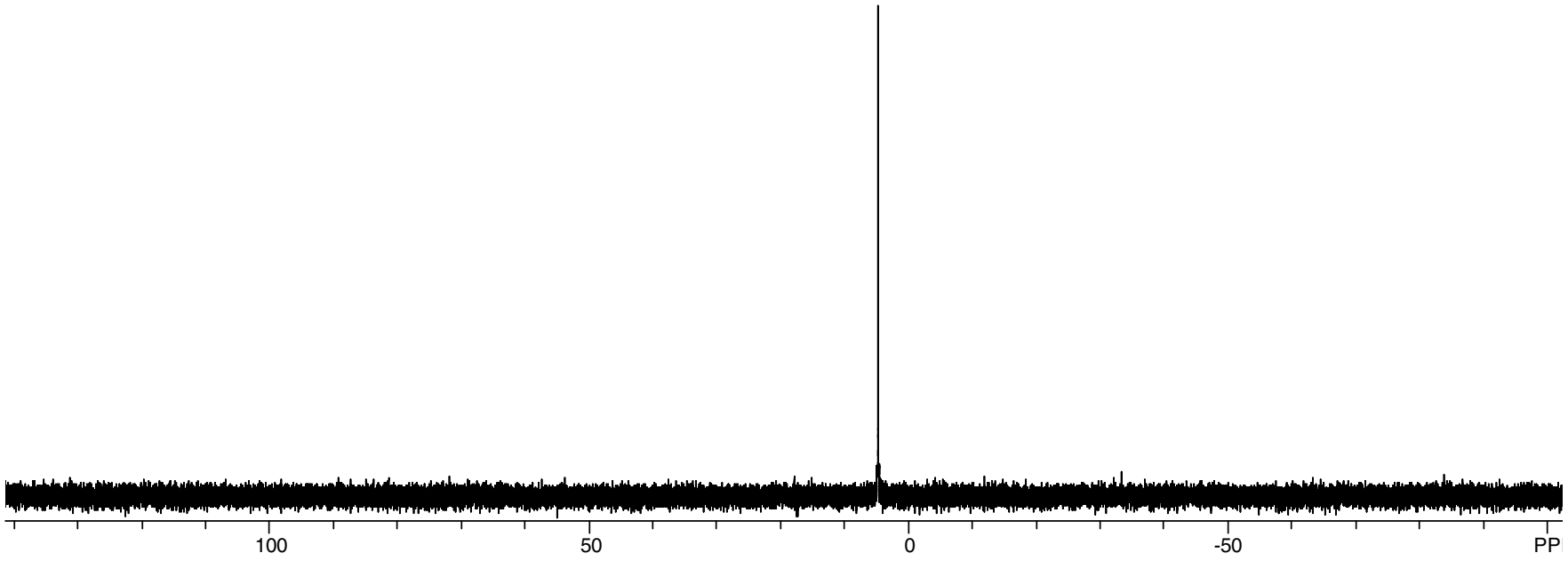
30



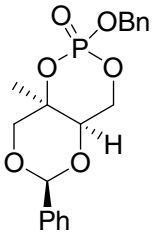
C:\Program Files\NUTS\data\$lc-02-91-threio-4-phosphoammo-1-4-04-31P.fid
P31 TRIPHENYLPHOSPHATE PARAMETERS
Jan 4 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 4.825 usec
Recycle delay = 1.000 sec
NA = 102
PTS1d = 65536
F1 = 202.284744 MHz
F2 = 499.699402 MHz
SW1 = 50000.00 Hz
AT1 = 1.31 sec
Hz per Pt 1stD = 0.76 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 3622.5781 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 226.00
B = 0.00
C = 0.00



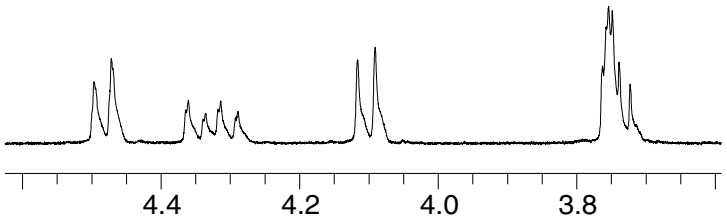
30

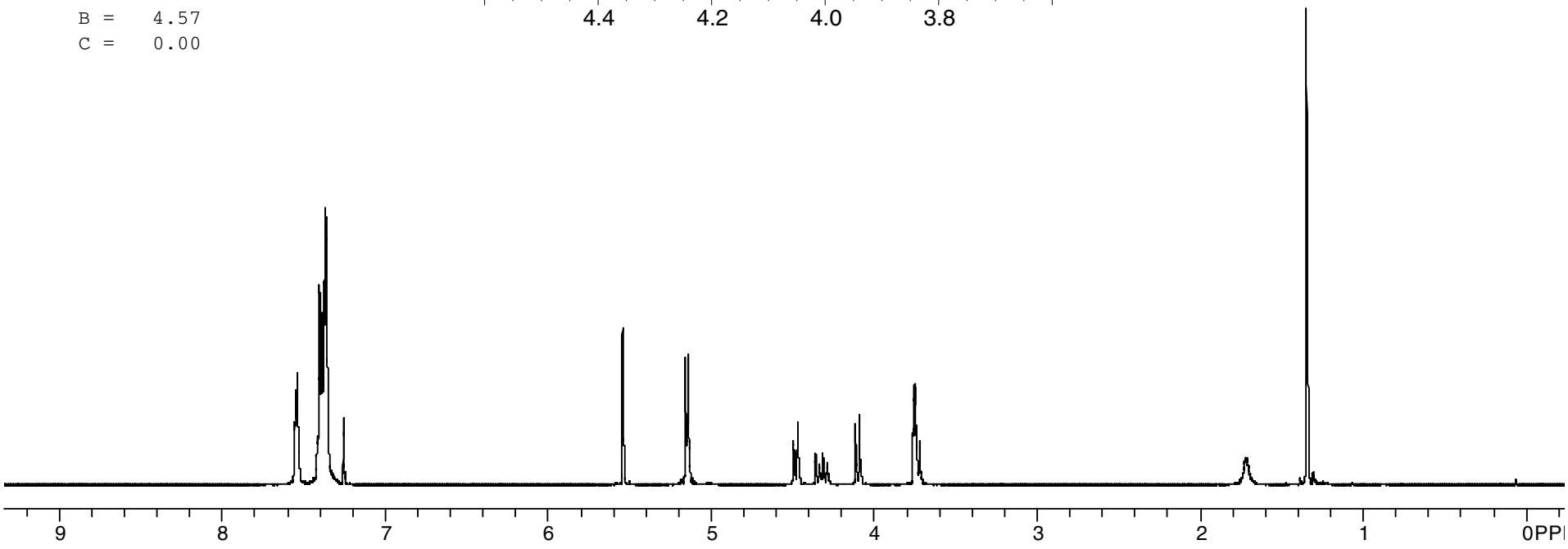


C:\Program Files\NUTS\DATA\$threi-mono-cyclophos-5-27-07-1H.fid
threi-mono-cyclophos-5-27-07-1H
May 27 2007
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 8
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0322 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -123.75
  B = 4.57
  C = 0.00

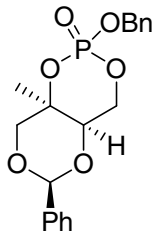


31

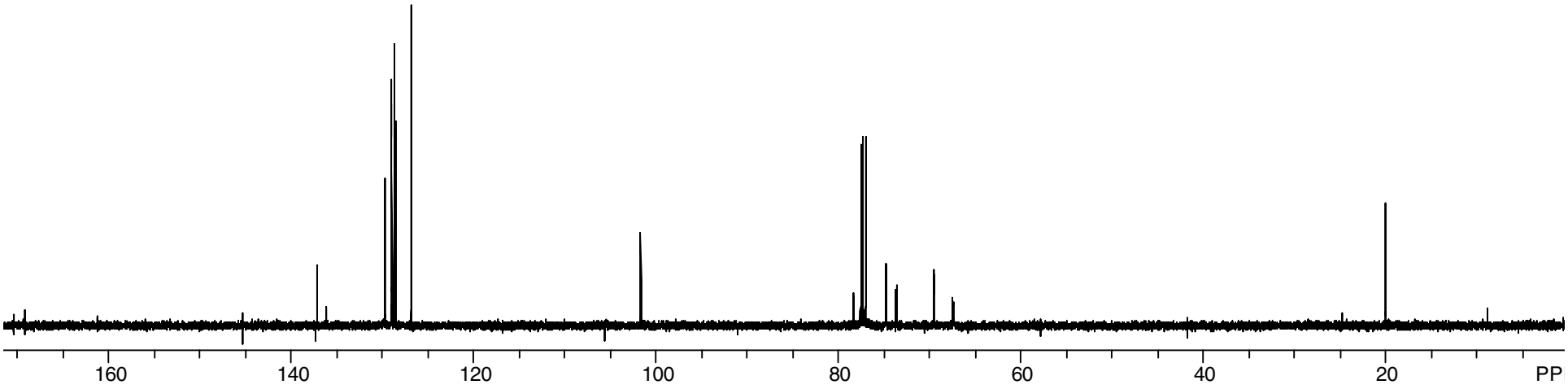




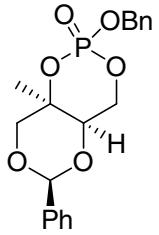
```
C:\Program Files\NUTS\DATA\$threi-mono-cyclophos-5-27-07-13C.fid
threi-mono-cyclophos-5-27-07-13C
May 27 2007
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length =    6.000 usec
Recycle delay =    1.000 sec
NA  =    558
PTS1d = 65536
F1  =  125.662560 MHz
F2  =  499.698120 MHz
SW1 =   30165.91 Hz
AT1 =   2.17 sec
Hz per Pt 1stD =   0.46 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =   13821.7402 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A = -10.31
      B =  125.16
      C =   0.00
```



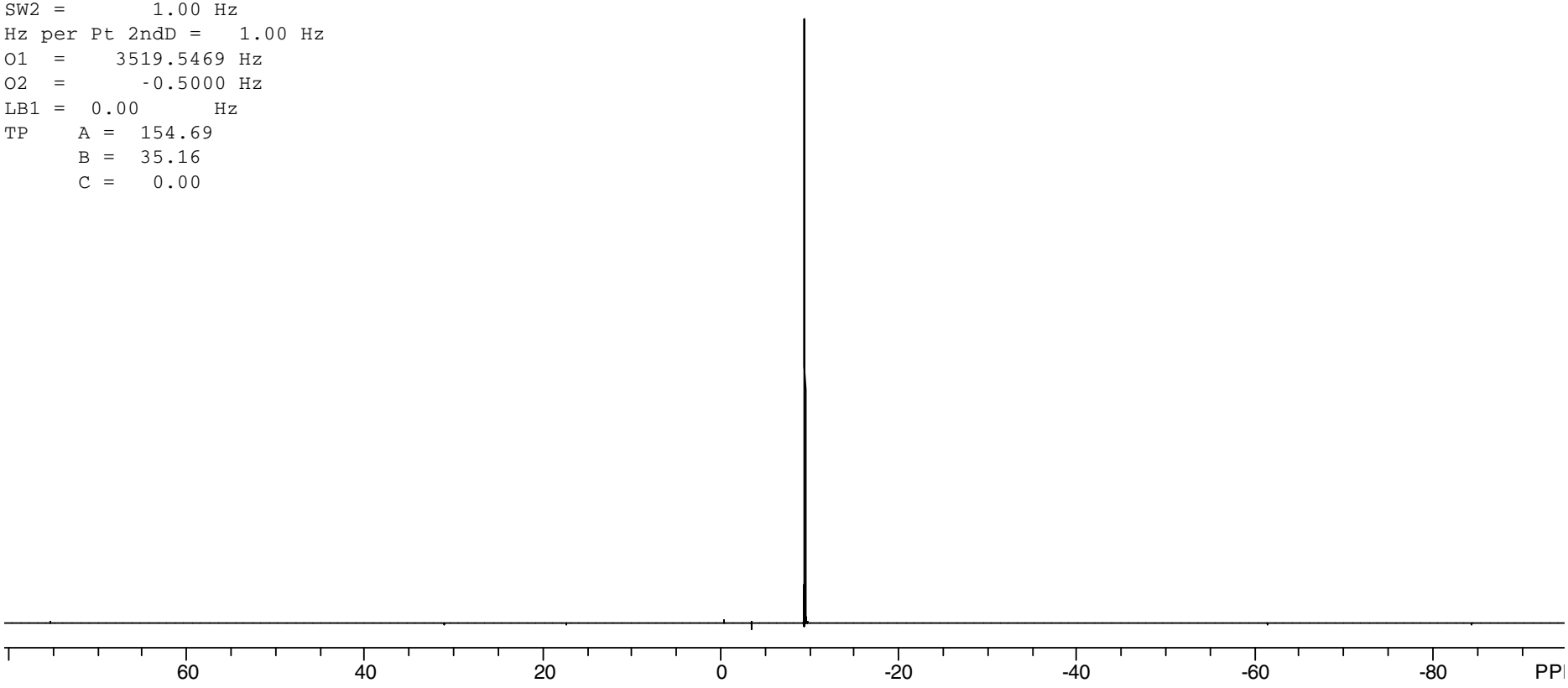
31



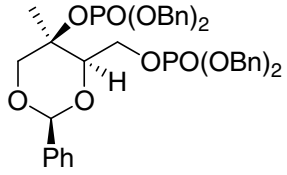
```
C:\Program Files\NUTS\DATA\$threi-mono-cyclo-phos-5-27-07-31P.fid
threi-mono-cyclo-phos-5-27-07-31P
May 27 2007
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length =    3.875 usec
Recycle delay =    1.000 sec
NA  =     32
PTS1d = 65536
F1  =  202.284225 MHz
F2  =  499.698120 MHz
SW1 =   50000.00 Hz
AT1 =   1.31 sec
Hz per Pt 1stD =   0.76 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =    3519.5469 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  154.69
      B =  35.16
      C =   0.00
```



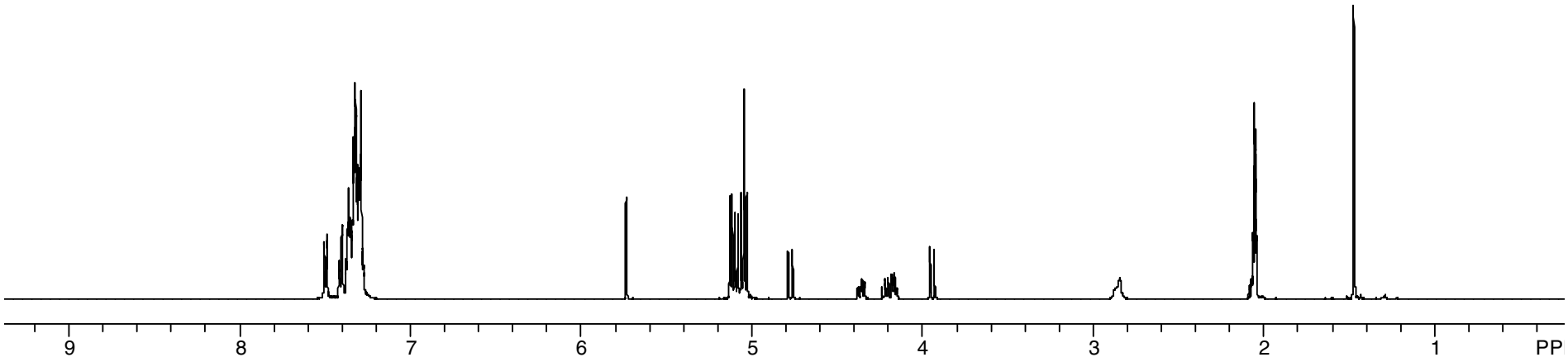
31

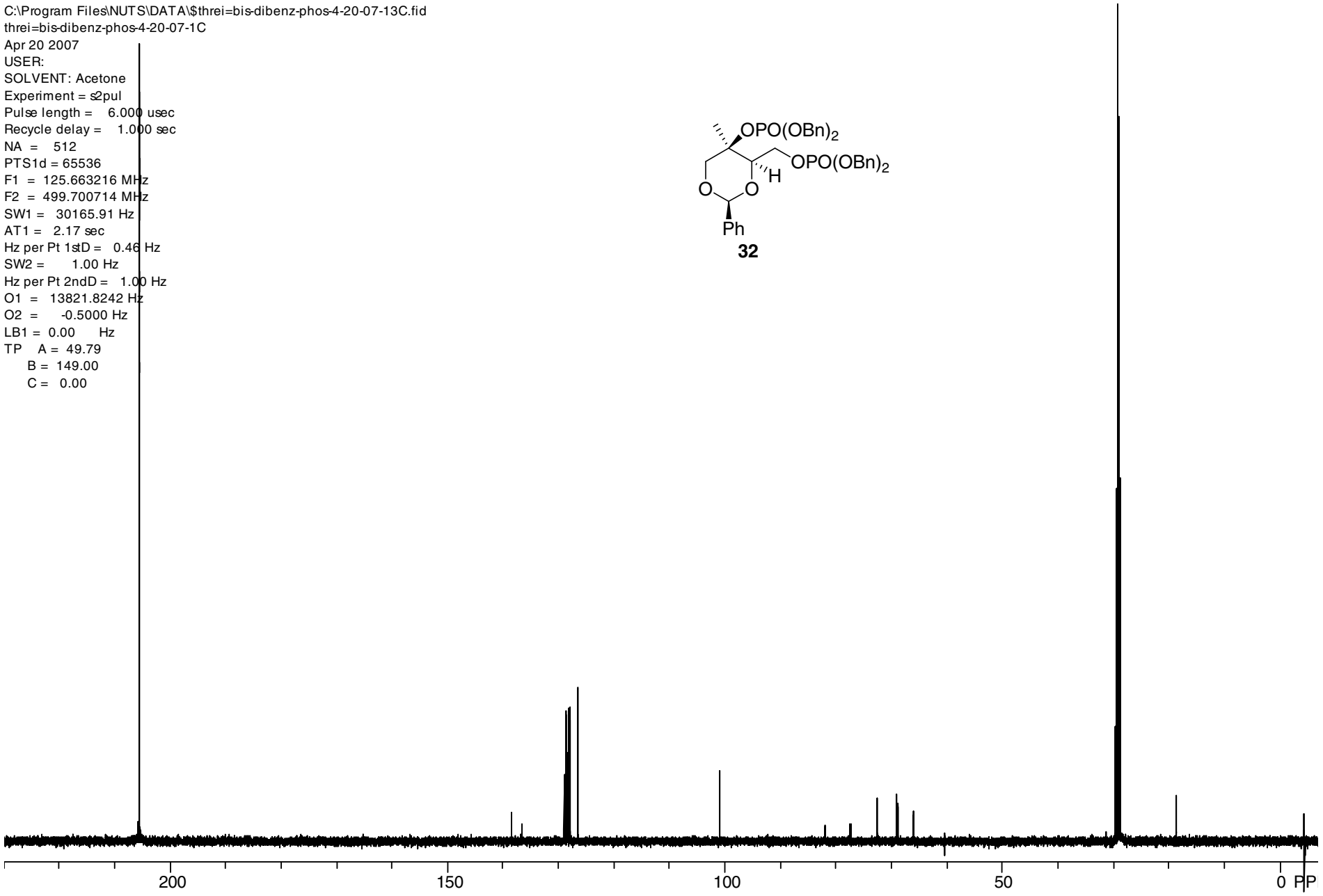


C:\Program Files\NUTS\DATA\$threi=bis-dibenz-phos-4-20-07-1H.fid
threi=bis-dibenz-phos-4-20-07-1H
Apr 20 2007
USER:
SOLVENT: Acetone
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 32
PTS1d = 65536
F1 = 499.701538 MHz
F2 = 499.701538 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0129 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 145.31
  B = 5.63
  C = 0.00

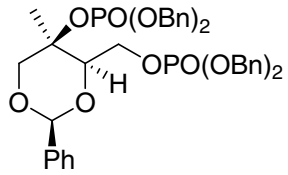


32

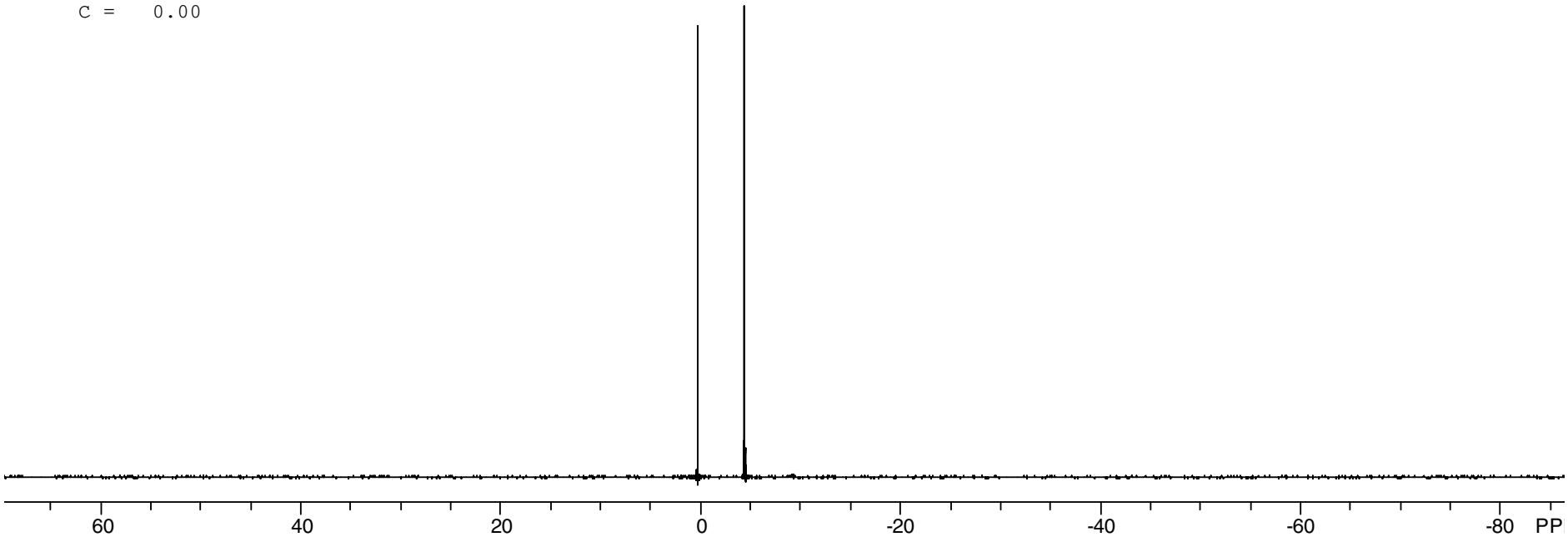


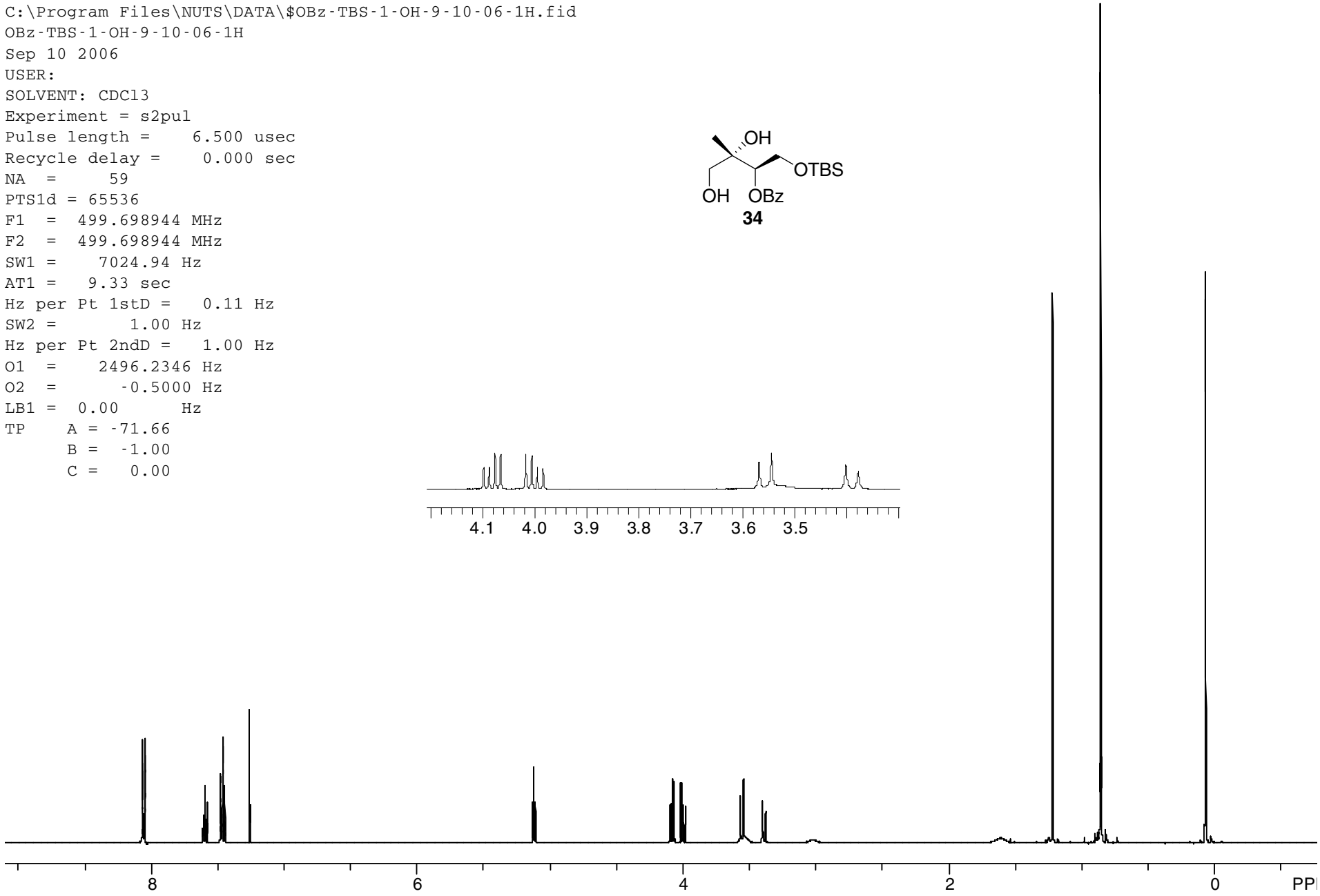

C:\Program Files\NUTS\DATA\$threi=bis-dibenz-phos-4-20-07-13C.fid
threi=bis-dibenz-phos-4-20-07-1C
Apr 20 2007
USER:
SOLVENT: Acetone
Experiment = s2pul
Pulse length = 6.000 usec
Recycle delay = 1.000 sec
NA = 512
PTS1d = 65536
F1 = 125.663216 MHz
F2 = 499.700714 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.8242 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 49.79
B = 149.00
C = 0.00
OPO(OBn)2
OPO(OBn)2
H
O
O
Ph
32
200
150
100
50
0 PP

```
C:\Program Files\NUTS\DATA\$threi=bis-dibenz-phos-4-20-07-31P.fid
P31 TRIPHENYLPHOSPHATE PARAMETERS
Apr 20 2007
USER:
SOLVENT: Acetone
Experiment = s2pul
Pulse length =    3.875 usec
Recycle delay =    1.000 sec
PTS1d = 65536
F1  =  202.285278 MHz
F2  =  499.700714 MHz
SW1 =   50000.00 Hz
AT1 =   1.31 sec
Hz per Pt 1stD =   0.76 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =    3622.5039 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A = -95.86
      B =  61.17
      C =   0.00
```

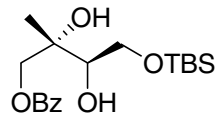


32

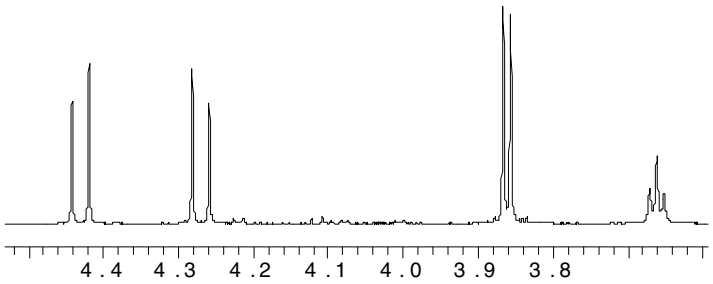



C:\Program Files\NUTS\DATA\$OBz-TBS-1-OH-9-10-06-1H.fid
OBz-TBS-1-OH-9-10-06-1H
Sep 10 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 59
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.2346 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -71.66
B = -1.00
C = 0.00
OH
OTBS
OH
OBz
34
4.1
4.0
3.9
3.8
3.7
3.6
3.5
8
6
4
2
0
PP

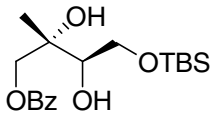
C:\Program Files\NUTS\DATA\$OBz-2nd-OH-TBS-9-12-06-1H.fid
OBz-2nd-OH-TBS-9-12-06-1H
Sep 12 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 31
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.1350 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -109.93
B = 9.00
C = 0.00



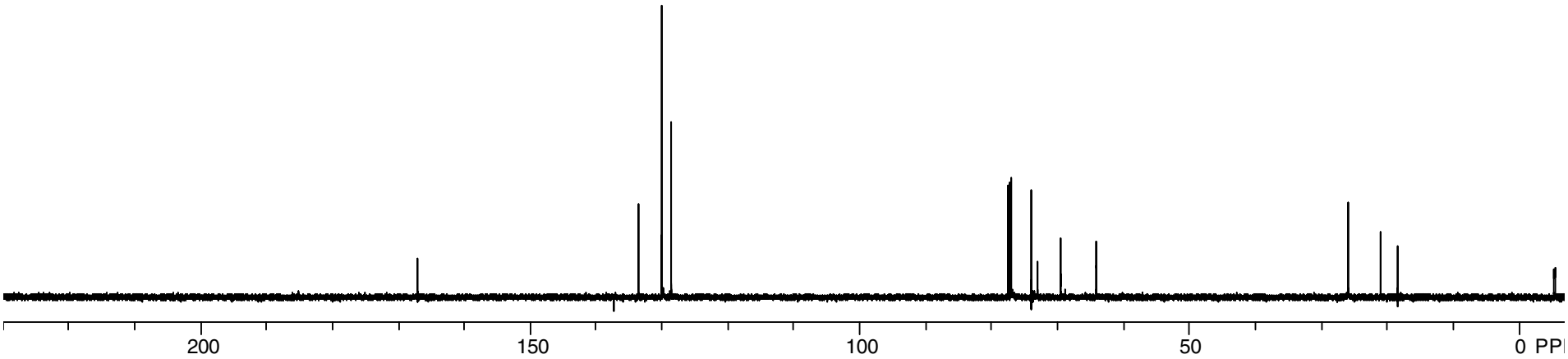
35



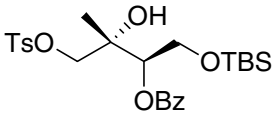
C:\Program Files\NUTS\DATA\$OBz-2nd-OH-TBs-9-12-06-13C.fid
OBz-2nd-OH-9-12-06-13C
Sep 12 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.325 usec
Recycle delay = 1.000 sec
NA = 290
PTS1d = 65536
F1 = 125.662560 MHz
F2 = 499.698120 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7402 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 68.05
B = 127.00
C = 0.00



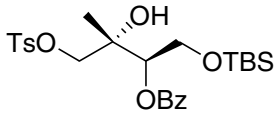
35

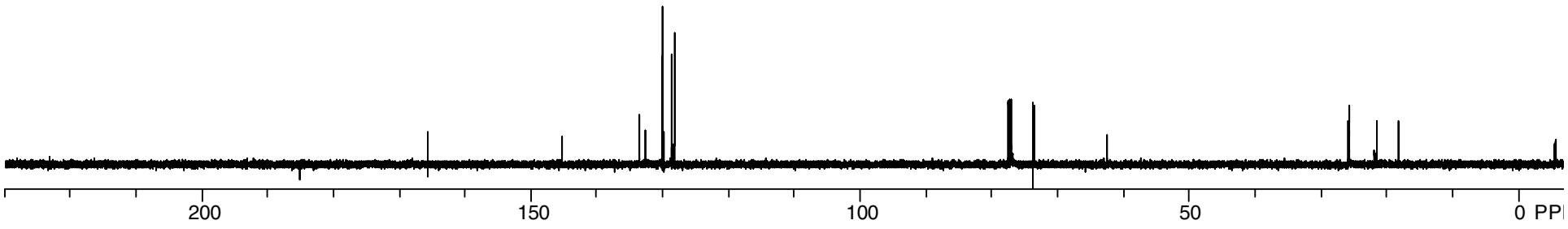


C:\Program Files\NUTS\DATA\$OBz-OTs-TBS-9-12-06-1H.fid
OBz-OTs-TBS-9-12-06-1H
Sep 12 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 16
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2495.9207 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 257.45
  B = 15.00
  C = 0.00

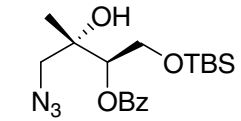


C:\Program Files\NUTS\DATA\$OBz-OTs-TBS-9-12-06-13C.fid
OBz-OTs-TBS-9-12-06-13C
Sep 12 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.325 usec
Recycle delay = 1.000 sec
NA = 201
PTS1d = 65536
F1 = 125.662560 MHz
F2 = 499.698120 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7402 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 142.48
B = 53.00
C = 0.00

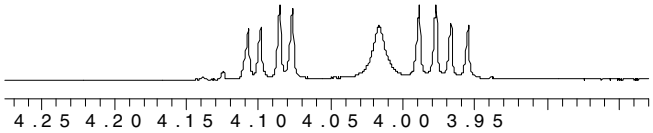




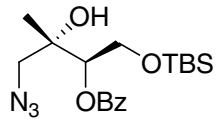
C:\Program Files\NUTS\DATA\$OBz-N3-TBS-9-14-06-1H.fid
OBz-N3-TBS-9-14-06-1H
Sep 14 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 32
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0322 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 270.58
  B = 29.00
  C = 0.00



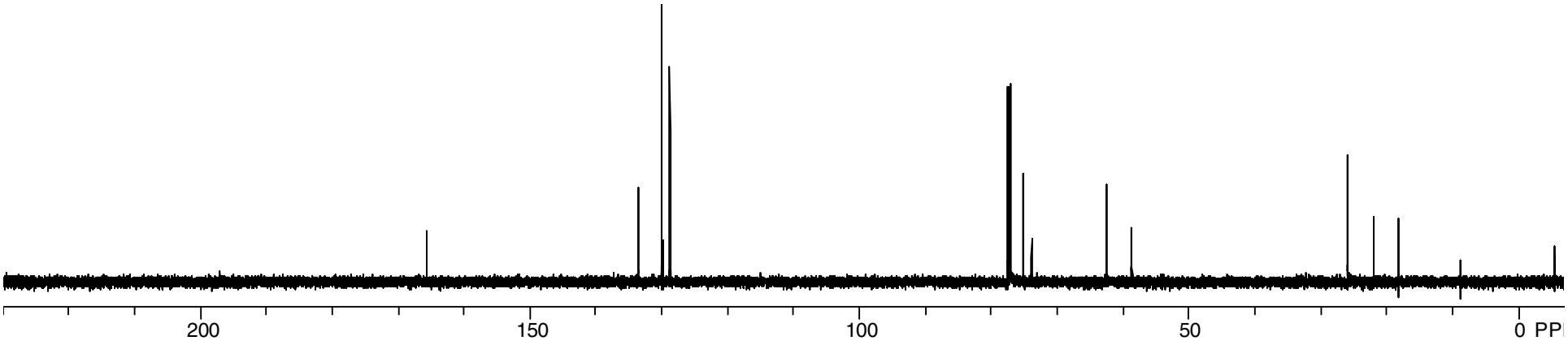
38



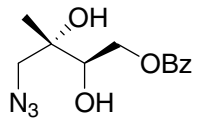
C:\Program Files\NUTS\DATA\$OBz-N3-TBS-9-14-06-13C.fid
OBz-N3-TBS-9-14-06-13C
Sep 14 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.325 usec
Recycle delay = 1.000 sec
NA = 198
PTS1d = 65536
F1 = 125.662560 MHz
F2 = 499.698120 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7402 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 139.33
B = 64.00
C = 0.00



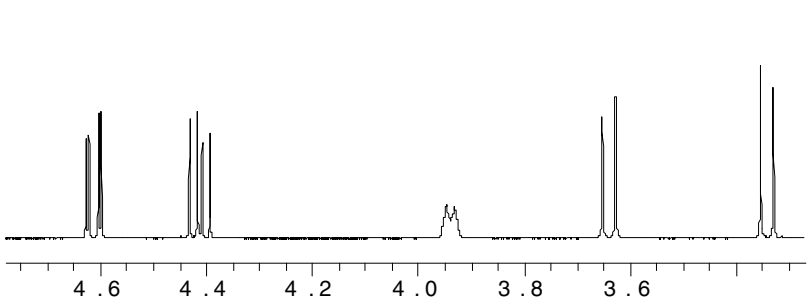
38



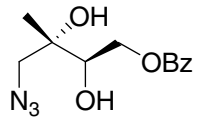
C:\Program Files\NUTS\DATA\$OBz-N3-diol-9-14-06-1H.fid
OBz-N3-diol-9-14-06-1H
Sep 15 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.500 usec
Recycle delay = 0.000 sec
NA = 28
PTS1d = 65536
F1 = 499.698944 MHz
F2 = 499.698944 MHz
SW1 = 7024.94 Hz
AT1 = 9.33 sec
Hz per Pt 1stD = 0.11 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2496.0322 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -66.57
  B = 2.00
  C = 0.00



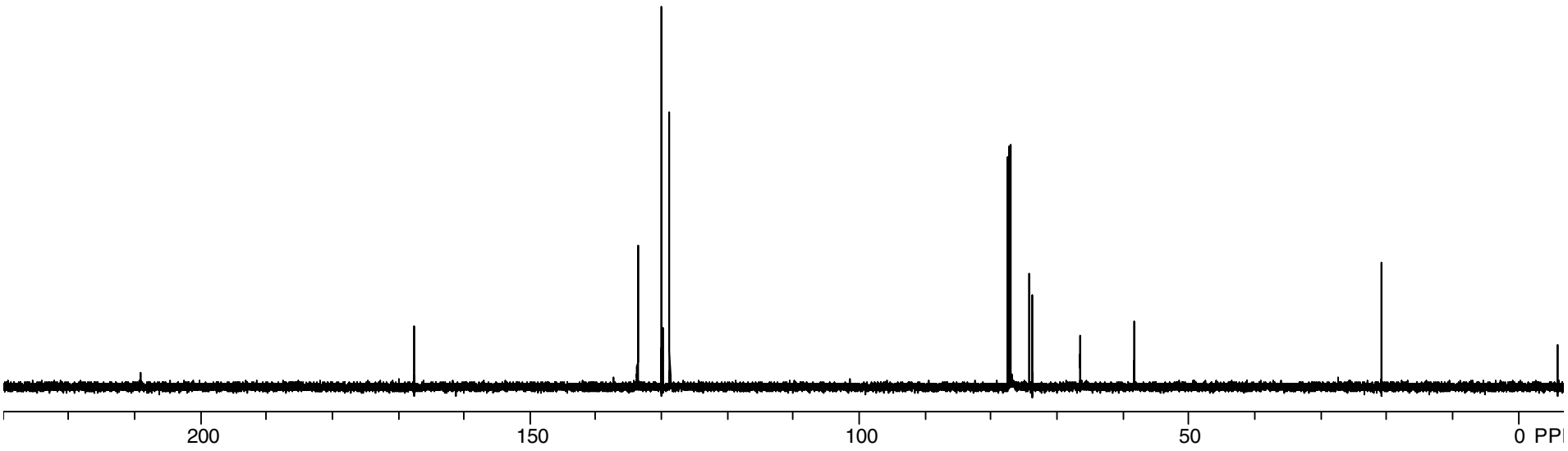
39



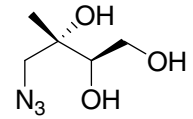
C:\Program Files\NUTS\DATA\$Obz-N3-diol-9-14-06-13C.fid
Obz-N3-diol-9-14-06-13C
Sep 15 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.325 usec
Recycle delay = 1.000 sec
NA = 451
PTS1d = 65536
F1 = 125.662560 MHz
F2 = 499.698120 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7402 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 143.24
B = 69.00
C = 0.00



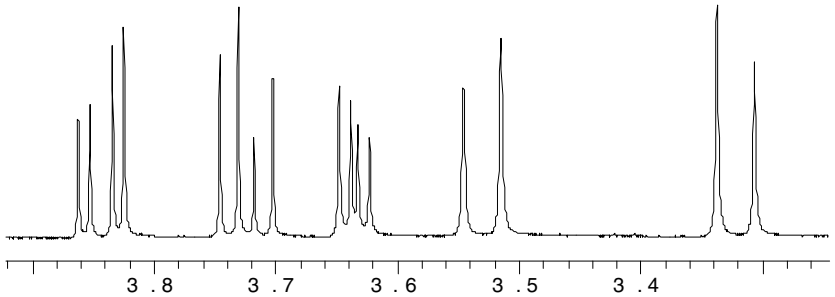
39

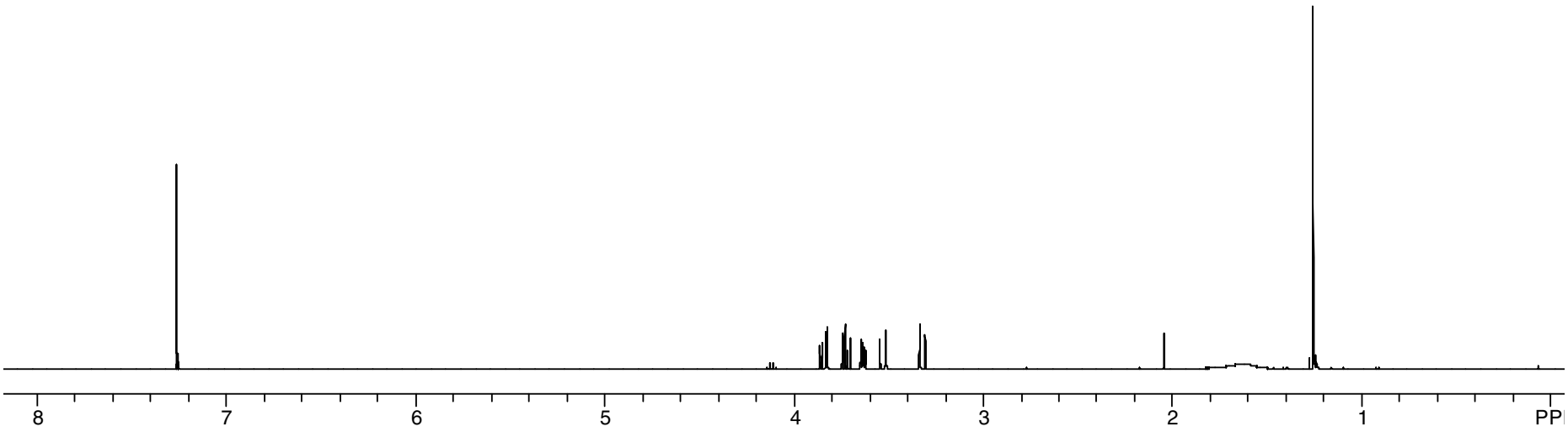


```
C:\Program Files\NUTS\DATA\$N3-triol-9-17-06-1H.fid
N3-triol-9-17-06-1H
Sep 17 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length =    5.825 usec
Recycle delay =    0.000 sec
NA  =     88
PTS1d = 65536
F1  =  399.950684 MHz
F2  =  399.951111 MHz
SW1 =    8000.00 Hz
AT1 =   8.19 sec
Hz per Pt 1stD =   0.12 Hz
SW2 =       1.00 Hz
Hz per Pt 2ndD =   1.00 Hz
O1  =    2000.4019 Hz
O2  =      -0.5000 Hz
LB1 =  0.00      Hz
TP    A =  37.84
      B =  65.00
      C =   0.00
```

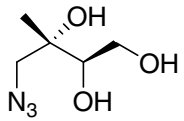


40

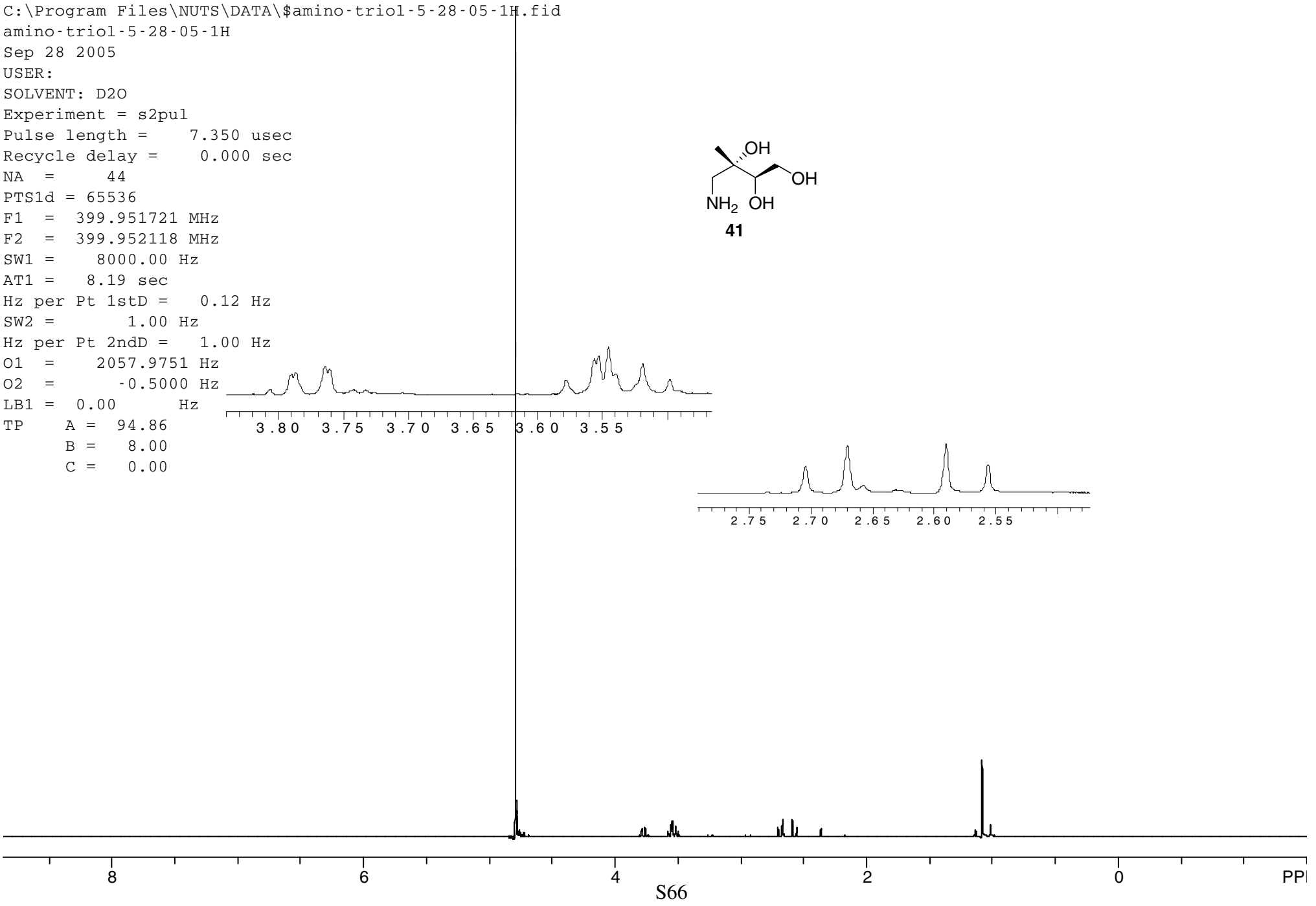




C:\Program Files\NUTS\DATA\$N3-triol-9-17-06-13C.fid
N3-triol-9-17-06-13C
Sep 17 2006
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.325 usec
Recycle delay = 1.000 sec
NA = 1000
PTS1d = 65536
F1 = 125.662560 MHz
F2 = 499.698120 MHz
SW1 = 30165.91 Hz
AT1 = 2.17 sec
Hz per Pt 1stD = 0.46 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 13821.7402 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -7.98
B = 268.00
C = 0.00



40


C:\Program Files\NUTS\DATA\$amino-triol-5-28-05-1H.fid
amino-triol-5-28-05-1H
Sep 28 2005
USER:
SOLVENT: D2O
Experiment = s2pul
Pulse length = 7.350 usec
Recycle delay = 0.000 sec
NA = 44
PTS1d = 65536
F1 = 399.951721 MHz
F2 = 399.952118 MHz
SW1 = 8000.00 Hz
AT1 = 8.19 sec
Hz per Pt 1stD = 0.12 Hz
SW2 = 1.00 Hz
Hz per Pt 2ndD = 1.00 Hz
O1 = 2057.9751 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 94.86
B = 8.00
C = 0.00
OH
OH
NH2
OH
41
3.80
3.75
3.70
3.65
3.60
3.55
2.75
2.70
2.65
2.60
2.55
8
6
4
2
0
PP