Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

A Synthetic Approach to Kingianin A Based on Biosynthetic Speculation

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Materials and Methods

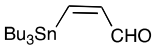
^{1}H and ^{13}C-NMR spectra were recorded on a Bruker AV (III) 400, Bruker AV 400, Bruker DPX 400 (400 MHz (^{1}H), and 100 MHz (^{13}C)), and Bruker DPX 300 (300 MHz (^{1}H) and 75 MHz (^{13}C)) spectrometers. Chemical shifts are expressed in parts per million (ppm) and the spectra calibrated to residual solvent signals of $CDCl_3$ (7.27 ppm (^{1}H) and 77.0 ppm (^{13}C)). Coupling constants are given in hertz (Hz) and the following notations indicate the multiplicity of the signals: s (singlet), d (doublet), brs (broad singlet), brd (broad doublet), t (triplet), q (quartet), m (multiplet).

High Resolution Mass Spectra were recorded on a VG micron Autospec or Bruker microTOF. Fourier Transform Infrared Spectroscopy (FT-IR) spectra were obtained using a Perkin Elmer 1600 series or Bruker Tensor 27 spectrometer.

Melting points were recorded using a STUART SMP3 apparatus and are uncorrected. Thin layer chromatography were carried out on Merck pre-coated silica gel plates (60F-254) and visualised using ultra violet light, $KMnO_4$ solution or *p*-anisaldehyde solution. THF was freshly distilled from sodium-benzophenone; DCM was dried over calcium hydride. Where necessary, reactions requiring anhydrous conditions were performed in dry solvents in flame dried or oven-dried apparatus under nitrogen/argon atmosphere.

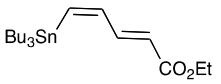
Experimental Procedures

(*Z*)-3-(Tributylstannyl)-2-propenal (19)[1]



To a well-stirred mixture of (*Z*)-3-(tributylstannyl)prop-2-en-1-ol (7.50 g, 21.6 mmol) in anhydrous DCM (100 mL) under nitrogen at 0 °C was added Dess-Martin periodinane (13.7 g, 32.4 mmol) in portions. The mixture was stirred at room temperature until completion of the oxidation (~ 1h, TLC). The reaction was cooled to 0 °C and quenched by addition of a saturated aqueous solution of $NaHCO_3$. The organic layer was separated and the aqueous layer was extracted with DCM (3 x 100 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent was then removed under reduced pressure. The crude residue was purified by flash silica gel column chromatography to give the aldehyde product **14** as clear oil (4.82 g, 65 %). R_f 0.40 (95:5::PE:EA); ν_{max}/cm^{-1}($CHCl_3$): 2959, 1681, 1463, 1378, 1192; δ_H (400 MHz, $CDCl_3$): 9.52 (1H, brd, *J* 6.9 Hz), 7.71 (1H, brd, *J* 13.0 Hz), 7.00 (1H, dd, *J* 6.9, 13.0 Hz), 1.54-1.48 (6H, m), 1.36-1.28 (6H, m), 1.06-1.02 (6H, m), 0.90 (9H, t, *J* 7.3 Hz) ppm; δ_C (100 MHz, $CDCl_3$): 194.5, 162.7, 145.4, 28.9, 27.1, 13.5, 11.3 ppm; HRESIMS calculated for $C_{15}H_{30}NaOSn$ $[M+Na]^+$ 369.1211 obtained 369.1229.

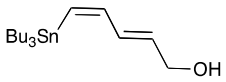
(2*E*,4*Z*)-Ethyl 5-(tributylstannyl)penta-2,4-dienoate (11)[2]



To a well stirred solution of the aldehyde **19** (4.50 g, 13.0 mmol) in anhydrous DCM (50 mL) under nitrogen was added ethyl 2-(triphenylphosphoranylidene)acetate (5.45 g, 15.6 mmol). The mixture was stirred overnight (16 h) at room temperature. The solvent was removed under reduced pressure and the product purified by flash silica gel column chromatography to give the Wittig product **11** as yellow oil (5.0 g, 93 %). R_f 0.4 (95:5::PE:EA); ν_{max}/cm^{-}

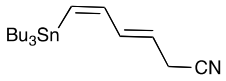
^{1}($CHCl_3$): 2956, 2926, 1704, 1625, 1463, 1368; δ_H (400 MHz, $CDCl_3$): 7.24-7.12 (2H, m), 6.71 (1H, brd, *J* 11.9 Hz), 5.89 (1H, brd, *J* 14.4 Hz), 4.22 (2H, q, *J* 7.1), 1.53-1.49 (6H, m), 1.37-1.28 (9H, m), 1.04-1.00 (6H, m), 0.90 (9H, t *J* 7.1 Hz) ppm; δ_C (100 MHz, $CDCl_3$): 166.9, 147.7, 146.5, 143.9, 122.5, 60.2, 29.0, 27.1, 14.2, 13.6, 10.6 ppm; HRESIMS calculated for $C_{19}H_{37}O_2Sn$ $[M+H]^+$ 417.1810 obtained 417.1803.

(2*E*, 4*Z*)-5-(Tributylstannyl)penta-2,4-dien-1-ol (12)



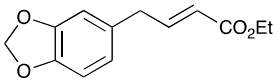
To a well stirred solution of **11** (2.25 g, 5.40 mmol) in anhydrous DCM (20 mL) under argon at -78 ^{o}C, was added a solution of DIBAL-H (1 M) (10.8 mL, 10.8 mmol) dropwise, and mixture was stirred at same temperature for an additional 2 hours. The reaction mixture was then slowly warmed to room temperature and stirred for a further 16 h. After this period the reaction mixture was cooled to 0 ^{o}C and quenched by slowly adding 5 mL of methanol. An aqueous saturated solution of sodium-potassium tartrate (20 mL) was added and the mixture stirred at room temperature until the two layers were clear. The organic layer was separated and the aqueous layer extracted with DCM (3 x 75 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent removed under reduced pressure to give the crude product as yellow oil. Purification using flash silica gel chromatography gave the alcohol product **12** as clear oil (1.3 g, 65 %). R_f 0.12 (95:5::PE:EA); ν_{max}/cm^{-1}($CHCl_3$): 3611, 2917, 2853, 1558, 1456, 1377; δ_H (400 MHz, $CDCl_3$): 7.07 (1H, dd, *J* 12.6, 10.5 Hz), 6.21-6.12 (2H, m), 5.86 (1H, dt, *J* 15.0, 5.7 Hz), 4.22 (2H, brt, *J* 5.7 Hz), 1.56-1.47 (6H, m), 1.37-1.27 (6H, m), 0.98-0.94 (6H, m), 0.90 (9H, t, *J* 7.4 Hz) ppm; δ_C (100 MHz, $CDCl_3$): 145.5, 135.2, 133.9, 133.4, 63.4, 29.1, 27.3, 13.6, 10.4 ppm; HRESIMS calculated for $C_{17}H_{34}NaOSn$ $[M+Na]^+$ 397.1524 obtained 397.1522.

(3*E*, 5*Z*)-6-(Tributylstannyl)hexa-3,5-dienenitrile (7)



To a well-stirred solution of **12** (0.400 g, 1.07 mmol) in degassed dry THF (2 mL) under argon, was slowly added a solution of triphenyl phosphine (0.674 g, 2.50 mmol) in THF (2 mL) dropwise. The reaction mixture was cooled to 0 ^{o}C and stirred for 5 min before adding acetone cyanohydrin (0.228 g, 2.50 mmol) dropwise. The mixture was further stirred for 5 mins. A solution of DEAD (0.448 g, 2.50 mmol) in degassed dry THF (2.2 mL) was added dropwise over 10 mins. The mixture was stirred at 0 ^{o}C until the completion of reaction (TLC). The volatile solvent and reagents were removed under reduced pressure to render the crude product. Purification by flash silica gel column chromatography gave the nitrile product **7** as clear oil (0.224 g, 55 %). R_f 0.26 (95:5::PE:EA); ν_{max}/cm^{-1}($CHCl_3$): 3154, 2925, 2257, 1816, 1793, 1643, 1461, 1378 ; δ_H (400 MHz, $CDCl_3$): 7.04 (1H, dd, *J* 12.7, 10.4 Hz), 6.35-6.27 (1H, m), 6.24 (1H, brd, *J* 12.7 Hz), 5.57 (1H, dt, *J* 15.0, 5.5 Hz), 3.20 (2H, dd, *J* 5.5, 1.5 Hz), 1.58-1.48 (6H, m), 1.39-1.27 (6H, m), 1.02-0.96 (6H, m), 0.90 (9H, t, *J* 7.2 Hz) ppm; δ_C (100 MHz, $CDCl_3$): 144.1, 137.4, 137.1, 121.1, 117.0, 29.0, 27.2, 20.5, 13.6, 10.4 ppm; HRESIMS calculated for $C_{18}H_{33}NNaSn$ $[M+Na]^+$ 406.1527 obtained 406.1530.

(*E*)-Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)but-2-enoate (8)[3,4]

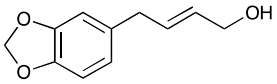


To a solution of safrole (9.0 g, 55.5 mmol) in methanol:H_2O (1.5:1) (450 mL), THF (~50 mL) was added dropwise until the solid has dissolved. OsO_4 (0.17 mL, 4% w/v water, 0.28 mmol) was added followed by the addition of sodium periodate (30.0 g, 13.9 mmol). The reaction mixture was vigorously stirred at room temperature for 16 h, then filtered through celite and washed with ethyl acetate (200 mL). The volume of solvent was then reduced by around a third under reduced pressure. The crude mixture was diluted with water (200 mL) and

extracted with ethyl acetate (3 x 400 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent removed under reduced pressure.

The crude aldehyde product (8.50 g (crude), 51.5 mmol) and ethyl 2-(triphenylphosphoranylidene)acetate (27.0 g, 77.5 mmol) were taken in dry DCM (200 mL) under nitrogen and heated to reflux for 20 h. The solvent was removed under reduced pressure and the crude residue purified by flash silica gel column chromatography to furnish ester **8** as pale yellow viscous oil (10.3 g, 80%, over 2 steps). R_f 0.51 (80:20::PE:EA)ν_{max}/cm^{-1}($CHCl_3$): 3011, 2966, 2893, 1722, 1504, 1491, 1445, 1248; δ_H (400 MHz, $CDCl_3$) 7.06 (1H, dt, *J* 15.5, 6.8 Hz), 6.77-6.62 (3H, m), 5.94 (2H, s), 5.80 (1H, dt, *J* 15.5, 1.6 Hz), 4.19 (2H, q, *J* 7.2 Hz), 3.44 (2H, brd, *J* 6.7 Hz), 1.28 (3H, t, *J* 7.2, Hz); δ_C (100 MHz, $CDCl_3$) 166.3, 147.7, 147.2, 146.2, 131.2, 122.0, 121.6, 109.1, 108.2, 100.8, 60.1, 37.9, 14.1; HRESIMS: Calculated for $C_{13}H_{15}O_4$ $[M+H]^+$ 235.0968 found 235.0965, $C_{13}H_{14}NaO_4$ $[M+Na]^+$ 257.0784 found 257.0782.

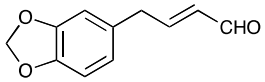
(*E*)-4-(Benzo[d][1,3]dioxo-5-yl)but-2-en-1-ol (9)



The ester **8** (8.70 g, 37.1 mmol) was dissolved in dry DCM (80 mL) under nitrogen and the solution cooled to -78 ^oC. DIBAL-H (81.7 mL, 81.7 mmol, 1M heptanes) was added dropwise over 10 min. The solution was warmed to room temperature and stirred for a further 30 mins. The reaction mixture was cooled to 0 ^oC and methanol (20 mL) added carefully. A saturated solution of sodium potassium tartrate (100 mL) was added and the mixture further stirred at room temperature until both layers were clear. The organic layer was separated and the aqueous layer was extracted with DCM (3 x 200 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent removed under reduced pressure. The residue purified by flash silica gel column chromatography to furnish the allylic alcohol product **9** as viscous pale yellow oil (5.01 g, 71%). R_f 0.31 (60:40::PE:EA)ν_{max}/cm^{-1}($CHCl_3$):

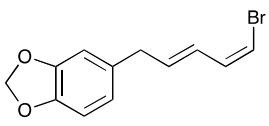
3611, 3011, 2884, 2777, 1504, 1489, 1443, 1245; δ_H (400 MHz, $CDCl_3$) 6.75 - 6.62 (3H, m), 5.91 (2H, s), 5.84-5.76 (1H, m), 5.71-5.64 (1H, m), 4.10 (2H, brd, *J* 5.4 Hz), 3.29 (2H, d, *J* 6.5 Hz); δ_C (100 MHz, $CDCl_3$) 147.5, 145.7, 133.7, 131.4, 130.1, 121.1, 108.9, 108.1, 100.7, 63.2, 38.2. HRMS: Calculated for $C_{11}H_{12}NaO_3$ $[M+Na]^+$ 215.0679 found 215.0678.

(*E*)-4-(Benzo[*d*][1,3]dioxol-5-yl)but-2-enal (10)



To a solution of alcohol **9** (500 mg, 2.60 mmol) in dry DCM (5 mL) under nitrogen was added DMP (1.32 g, 3.12 mmol) at 0 ^oC. The mixture was warmed to room temperature and stirred further for 30 mins. After the completion of reaction (TLC) the mixture was cooled to 0 ^oC and a saturated aqueous solution of $NaHCO_3$ (100 mL) was added. The organic layer was separated and the aqueous layer was extracted with DCM (3 x 100 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent removed under reduced pressure. The crude residue was purified by flash column chromatography to furnish the aldehyde product **10** as pale yellow oil (475 mg, 96%). R_f 0.33 (80:20::PE:EA)ν_{max}/cm^{-1}($CHCl_3$): 3367, 3011, 2824, 1686, 1635, 1489, 1444, 1243; δ_H (400 MHz, $CDCl_3$) 9.53 (1H, d, *J* 7.7 Hz), 6.92 (1H, dt, *J* 15.5, 6.5 Hz), 6.78 – 6.62 (3H, m), 6.12 (1H, ddt, *J* 15.5, 7.7, 1.5 Hz), 5.94 (2H, s), 3.56 (2H, dd, *J* 6.5, 1.5 Hz); δ_C (100 MHz, $CDCl_3$) 193.6, 156.3, 147.9, 146.4, 133.2, 130.5, 121.7, 109.1, 108.4, 100.9, 38.5. HRESIMS: Calculated for $C_{11}H_{10}NaO_3$ $[M+Na]^+$ 213.0522 found 213.0524.

5-((2*E*,4*Z*)-5-Bromopenta-2,4-dien-1-yl)benzo[*d*][1,3]dioxole (6)

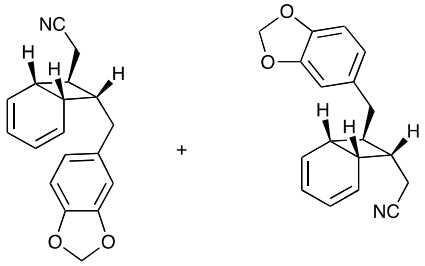


To a solution of carbon tetrabromide (1.90 g, 5.70 mmol) in anhydrous DCM (20 mL) at 0 ^oC was added a solution of triphenyl phosphine (3.00 g, 11.4 mmol) in anhydrous DCM (15 mL)

under nitrogen. The mixture was stirred for 10 mins followed by addition of triethylamine (2.00 g, 20 mmol). To this mixture was added the aldehyde **11** (0.390 g, 2.0 mmol) in dry DCM (5 mL) dropwise. The mixture was stirred at room temperature for 15 min. A saturated solution of ammonium chloride (20 mL) was added and the organic layer separated. The aqueous layer was further extracted with DCM (3 x 100 mL) and the combined organic extracts dried over anhydrous Na_2SO_4, filtered and the solvent removed under reduced pressure. The residue was purified using flash column chromatography to give the dibromo product as viscous yellow oil. The compound was put forward to the next step without further delay.

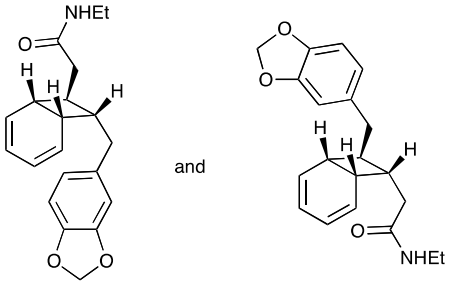
To a solution of dibromo (0.576, 1.6 mmol) in degassed and anhydrous toluene (5.0 mL) was added $Pd(PPh_3)_4$ (0.192 g, 10 mol%) under argon. To this was added tributyltin hydride (0.533 g, 1.83 mmol) dropwise. The mixture was stirred at room temperature until the completion of reaction (~ 1h, TLC). The reaction mixture was filtered through a pad of silica gel and the solvent removed under reduced pressure. The residue was purified by flash silica gel column chromatography to give the bromo **6** product as an opaque viscous oil which solidified on standing at lower temperature (0.360 g, 67 % yield over 2 steps). R_f 0.6 (75:25::PE:EA); mp: 38-40 ^oC; $\nu_{max}/cm^{-1}(CHCl_3)$: 3690, 3606, 1602, 1504, 1488, 1442 ; δ_H (400 MHz, $CDCl_3$): 6.77-6.75 (3H, m), 6.62 (1H, dd, *J* 10.1, 7.1 Hz), 6.46 (1H, dd, *J* 15.0, 10.1 Hz), 6.10 (1H, brd, *J* 7.1 Hz), 6.02 (1H, dt, *J* 15.0, 7.1 Hz), 5.94 (2H, s), 3.40 (2H, brd, *J* 7.1 Hz) ppm; δ_C (100 MHz, $CDCl_3$): 147.7, 146.0, 137.4, 133.1, 132.2, 126.9, 121.3, 109.0, 108.2, 106.6, 100.8, 39.0 ppm; HRMS: Calculated for $C_{12}H_{11}BrO_2$ $[M+H]^+$ 265.9943 and 267.9922 obtained 265.9937 and 267.9925.

Bicyclo[4.2.0]octadiene 16 and 17



To a solution of the vinyl bromide **6** (0.144 g, 0.540 mmol) in degassed anhydrous toluene (1.5 mL) under argon was added the vinyl stannane **7** (0.228 g, 0.590 mmol), and the mixture heated at 35 ^{o}C. In a separate flask, $Pd_2(dba)_3$ (0.250 g, 0.027 mmol) and tri-(2-furyl)phosphine (0.370 g, 0.160 mmol) were stirred in degassed toluene for 30 minutes. The catalyst mixture was added to the above reaction dropwise over 10 mins and the mixture heated at 100 ^{o}C for 10 h. After completion of the reaction (TLC) the mixture was filtered through a pad of silica gel and the solvent removed under reduced pressure. The residue was purified by flash silica gel column chromatography to give a mixture of diastereoisomers **16** and **17** (1:1) as pale yellow viscous oil (0.090 g, 60%). R_f 0.24 (90:10::PE:EA); ν_{max}/cm^{-1}($CHCl_3$): 3154, 2901, 2256, 1816, 1793, 1643, 1461, 1380 ; δ_H (400 MHz, $CDCl_3$): 6.75-6.60 (6H, m), 5.97-5.92 (2H, m), 5.94 (4H, s), 5.78 (1H, dd, *J* 9.6, 5.2 Hz), 5.69-5.61 (3H, m), 5.57 (1H, dd, *J* 10.0, 4.1 Hz), 5.44 (1H, dd, *J* 9.7, 5.5 Hz), 3.33-3.24 (2H, m), 2.92-2.52 (10H, m), 2.47 (1H, dd, *J* 16.6, 9.4 Hz), 2.25 (1H, dd, *J* 16.7, 6.2 Hz), 2.17 (1H, dd, *J* 17.0, 4.7 Hz), 2.01 (1H, dd, *J* 17.0, 7.0 Hz); δ_C (100 MHz, $CDCl_3$): 147.75, 147.69, 145.99, 145.92, 133.6, 133.3, 126.8, 125.9, 125.7, 125.1, 124.5, 123.6, 122.8, 121.5, 121.4, 121.1, 119.1, 118.4, 108.9, 108.3, 108.26, 100.9, 53.0, 50.8, 46.1, 45.4, 40.8, 36.1, 35.6, 35.5, 35.2, 33.7, 21.5, 17.9 ppm; HRESIMS calculated for $C_{18}H_{17}NNaO_2$ $[M+Na]^+$ 302.1151 obtained 302.1156.

Pre-kingianin A (2) and bicylo[4.2.0]octadiene 18

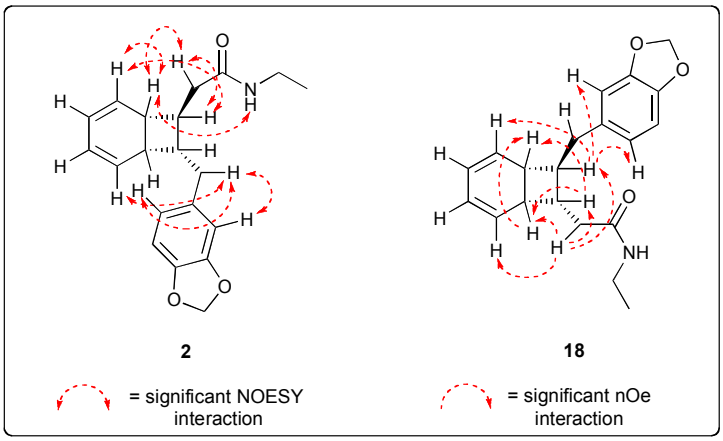


2 **18**

To a solution of a diastereomeric mixture of **16** and **17** (0.042 g, 0.15 mmol) in EtOH (1mL) was added 7 (M) aqueous KOH (0.05 mL) at 0 ^{o}C. To the above well stirred solution was added 30 % H_2O_2 (0.7 mL) dropwise and the mixture was stirred at room temperature for 1 h followed by heating at 70 ^{o}C for 4 h. A change in colour of the mixture was observed from pale yellow to brown. After completion of the reaction, the mixture was extracted with diethyl ether (3 x 5 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and the solvent was then removed under reduced pressure. The crude product was carried forward to the next step without further purification.

The crude mixture was then taken in dry toluene (1.0 mL) and CH_3CHO (0.015 g, 0.35 mmol) was added. This was followed by the addition of Et_3SiH (0.041 g, 0.35 mmol) and TFA (0.040 g, 0.35 mmol) and the mixture was then heated at 120 ^{o}C for 1 h. The mixture was cooled to room temperature, diluted with ethyl acetate (4.0 mL) and washed with saturated aqueous solution of $NaHCO_3$ (4.0 mL). The organic layer was dried over anhydrous Na_2SO_4, filtered and the solvent was then evaporated to dryness under reduced pressure. The residue was purified *via* preparative TLC to furnish the compound **2** (0.015 g) and **18** (0.015 g) in 62% combined yield.

Observed nOe and NOESY correlation diagram for 2 and 18



T1: Reaction conditions screened for dimerization studies of **2** and **18**

Entry	Substrate	Reaction condition	Results[a]
1.	**18**	Neat, Room temperature, 14 days	Isomerisation **18**:**2**::1:0.30
2.	**2**	Ethyl acetate:Pet ether, Room temperature, 21 days	Isomerisation **2**:**18**::1:0.45
3.	**18/2**	Toluene, microwave, 120 ^{o}C, 30 min	No change
5.	**18/2**	Ethyl acetate, 40 ^{o}C, 48 h, sealed tube	Isomerisation **18**:**2**::1:1
5.	**18**	Sublimation at 195 ^{o}C, 1-5mbar vacuum for 5 h.[5]	Sublimed product **18**:**2**::1:1
6.	**2**	Sublimation at 195 ^{o}C, 1-5 mbar vacuum for 5h	No sublimation, substrate charred
7.	**2**	Sublimation after melting the substrate at 120 ^{o}C. Heated at 130 ^{o}C under 1-5 mbar vacuum sealed for 16 h	No sublimation occurred, residue not conclusive
8.	**18/2**	5.0 (M) $LiClO_4$,[6] 60 ^{o}C, 16 h	Isomerisation **18**:**2**::1:1

[a] Ratio based on ^{1}H NMR integration

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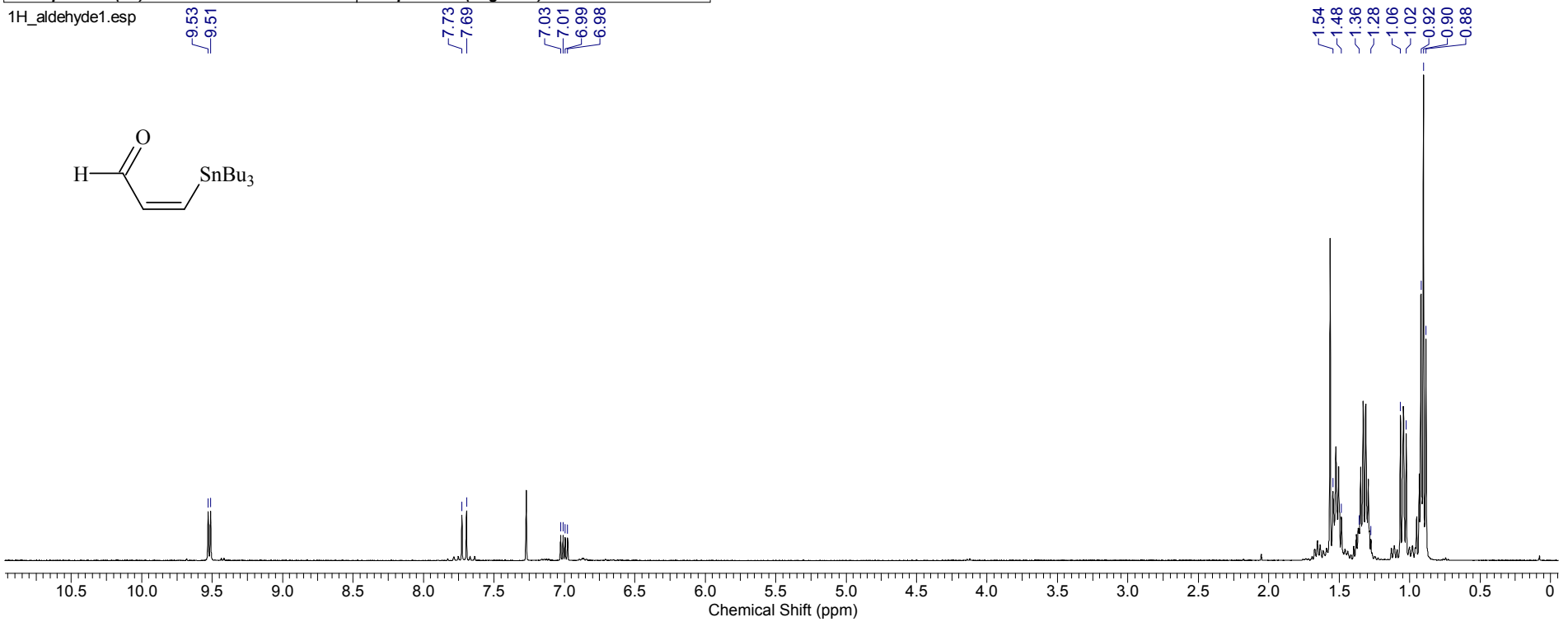
^{1}H NMR: (*Z*)-3-(Tributylstannyl) acrylaldehyde (19)

06/04/2011 11:11:25

Formula $C_{15}H_{30}OSn$	***FW***	345.1081	

Acquisition Time (sec)	3.9846	***Comment***	UserID p_sha SampleID Pal667 SupervisorID moses Lab Phone No. 13540 Slot Number 50				
Date	16 Sep 2010 15:28:16			***Date Stamp***	16 Sep 2010 15:28:16		
File Name	C:\Users\psharma\Desktop\Kingainin\nmr-kingianin-3400\aldehyde 1\p_sha.PAl667\1\pdata\1\1r					***Frequency (MHz)***	400.07
Nucleus	1H	***Number of Transients***	16	***Origin***	av3400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	256.00
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2461.3486	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	24.925				

1H_aldehyde1.esp



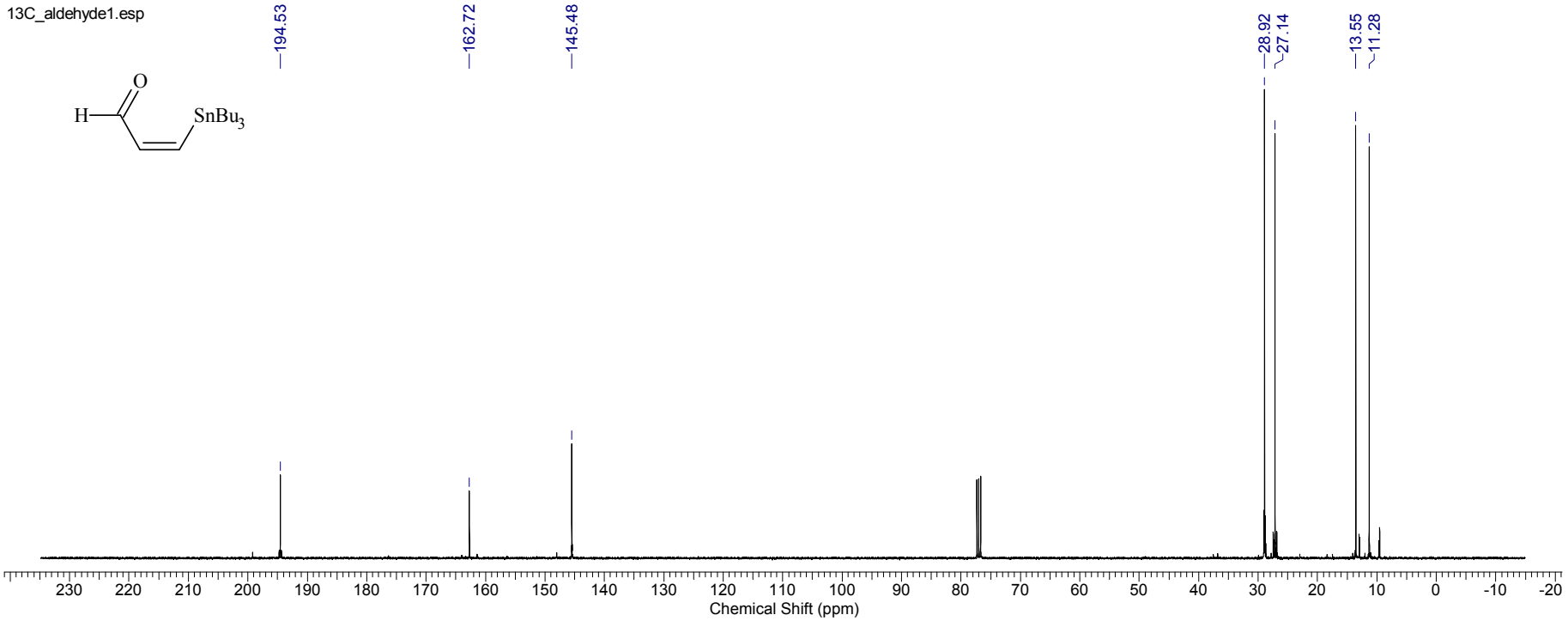
^{13}C NMR: (*Z*)-3-(Tributylstannyl) acrylaldehyde (19)

06/04/2011 11:14:59

Formula $C_{15}H_{30}OSn$	***FW***	345.1081	

Acquisition Time (sec)	0.6521	***Comment***	UserID p_sha SampleID Pal744 SupervisorID moses Lab Phone No. 13540 Slot Number 58				
Date	06 Nov 2010 17:04:00	***Date Stamp***	06 Nov 2010 17:04:00				
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-400\aldehyde 1\p_sha.Pal744\1\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	512	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	16384.00
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11061.5605	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.160				

13C_aldehyde1.esp



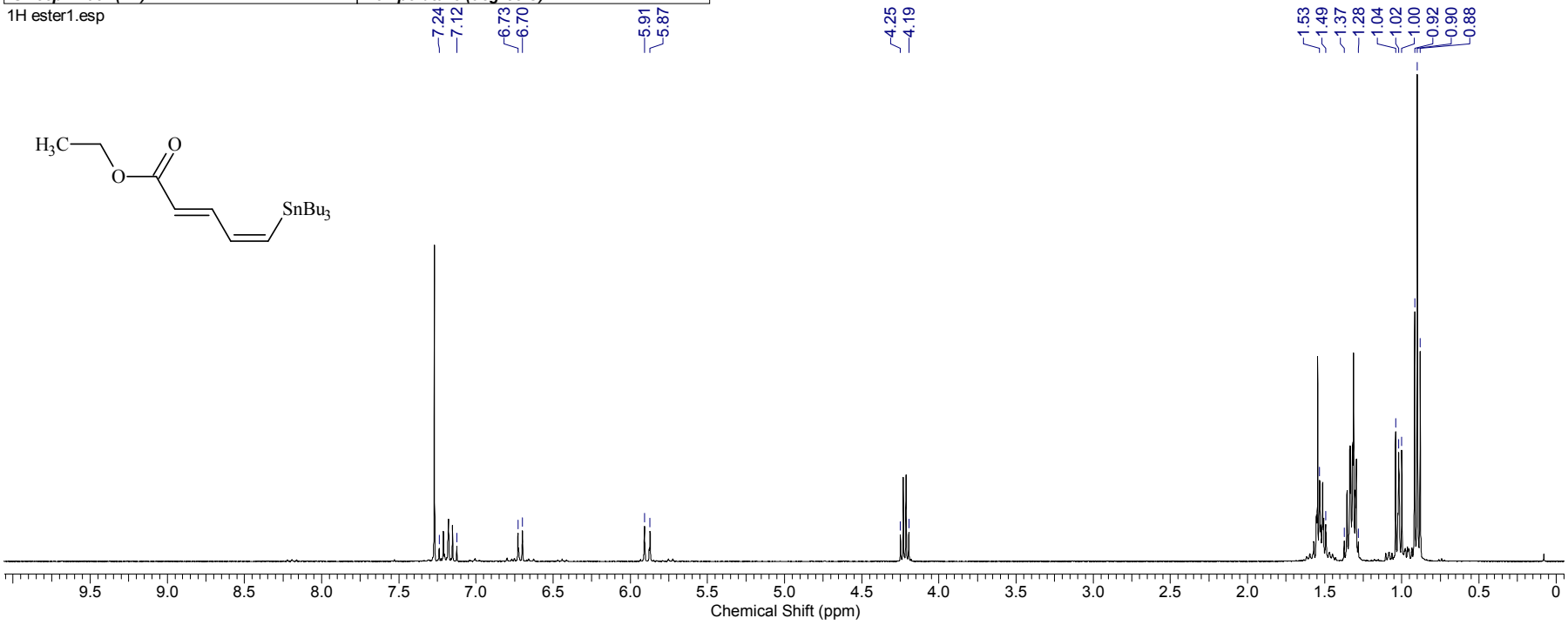
^{1}H NMR: (2*E*,4*Z*)-Ethyl 5-(tributylstannyl)penta-2,4-dienoate (11)

06/04/2011 11:20:34

Formula $C_{19}H_{36}O_2Sn$	***FW***	415.1980	

Acquisition Time (sec)	3.9846	***Comment***	UserID p_sha SampleID PAl802 SupervisorID moses Lab Phone No. 13540 Slot Number 42				
Date	22 Jan 2011 15:36:32			***Date Stamp***	22 Jan 2011 15:36:32		
File Name	C:\Users\psharma\Desktop\Kingainin\nmr-kingianin-3400\wittig 1\p_sha.Pal802\1\pdata\1\1r					***Frequency (MHz)***	400.07
Nucleus	1H	***Number of Transients***	16	***Origin***	av3400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	362.00
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2462.4780	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	24.925				

1H ester1.esp

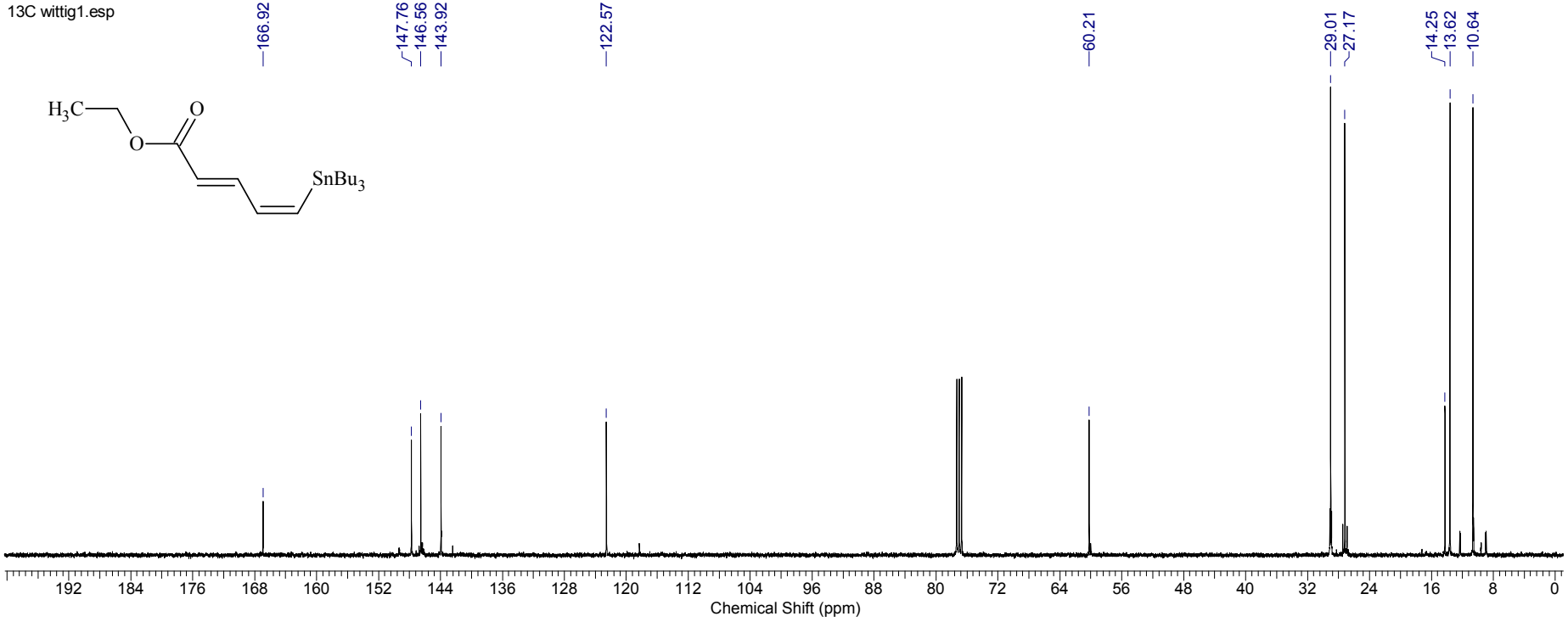


^{13}C NMR: (2*E*,4*Z*)-Ethyl 5-(tributylstannyl)penta-2,4-dienoate (11)

06/04/2011 11:31:44

Formula $C_{19}H_{36}O_2Sn$	***FW*** 415.1980						
Acquisition Time (sec)	0.6521	***Comment***	UserID p_sha SampleID Pa668re SupervisorID moses Lab Phone No. 13540 Slot Number 10				
Date	06 Nov 2010 11:03:28	***Date Stamp***	06 Nov 2010 11:03:28				
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-400\wittig 1\p_sha.Pal668re\2\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	512	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	18390.40
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11063.0947	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.160				

13C wittig1.esp

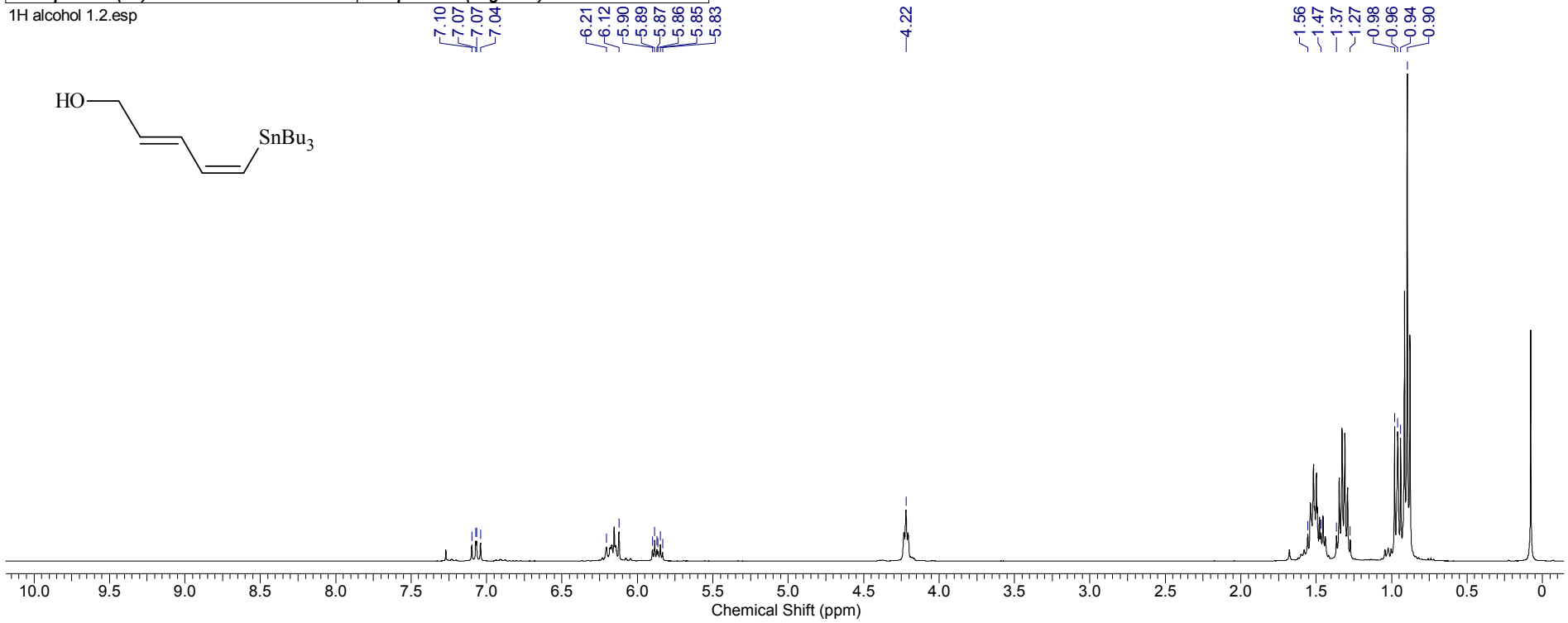


^{1}H NMR: (2*E*, 4*Z*)-5-(Tributylstannyl)penta-2,4-dien-1-ol (12)

06/04/2011 11:27:11

Formula $C_{17}H_{34}OSn$	***FW*** 373.1613						
Acquisition Time (sec)	3.9846	***Comment***	Slot No. 42 Sample ID Pal717 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	30 Mar 2011 16:13:04			***Date Stamp***	30 Mar 2011 16:13:04		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\alcohol 1\p_sha.Pal717\1\pdata\1\1r					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	80.60
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2462.2937	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				

1H alcohol 1.2.esp



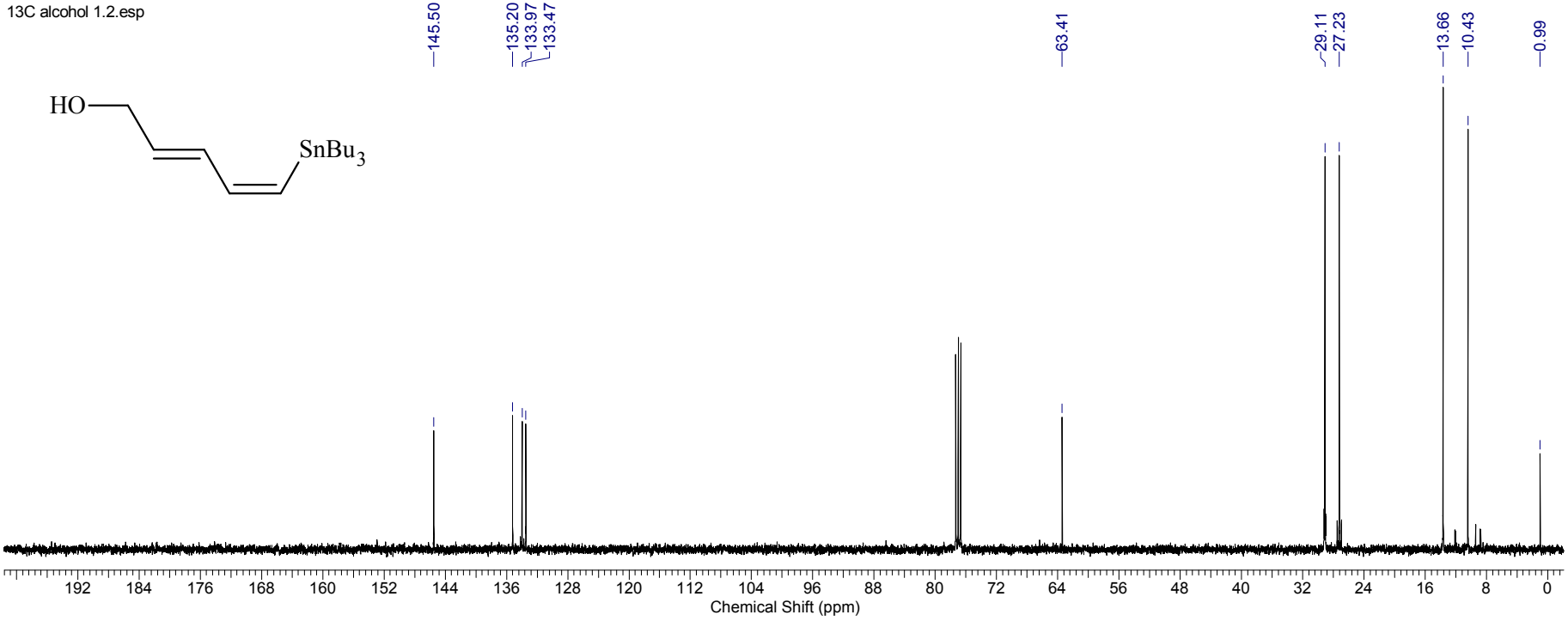
^{13}C NMR: (2*E*, 4*Z*)-5-(Tributylstannyl)penta-2,4-dien-1-ol (12)

06/04/2011 11:36:24

Formula $C_{17}H_{34}OSn$	***FW***	373.1613	

Acquisition Time (sec)	0.6521	***Comment***	Slot No. 42 Sample ID PAI717 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	30 Mar 2011 16:28:00			***Date Stamp***	30 Mar 2011 16:28:00		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\alcohol 1\p_sha.Pal717\3\pdata\1\1r					***Frequency (MHz)***	100.63
Nucleus	13C	***Number of Transients***	128	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	9195.20
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11065.2168	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.000				

13C alcohol 1.2.esp

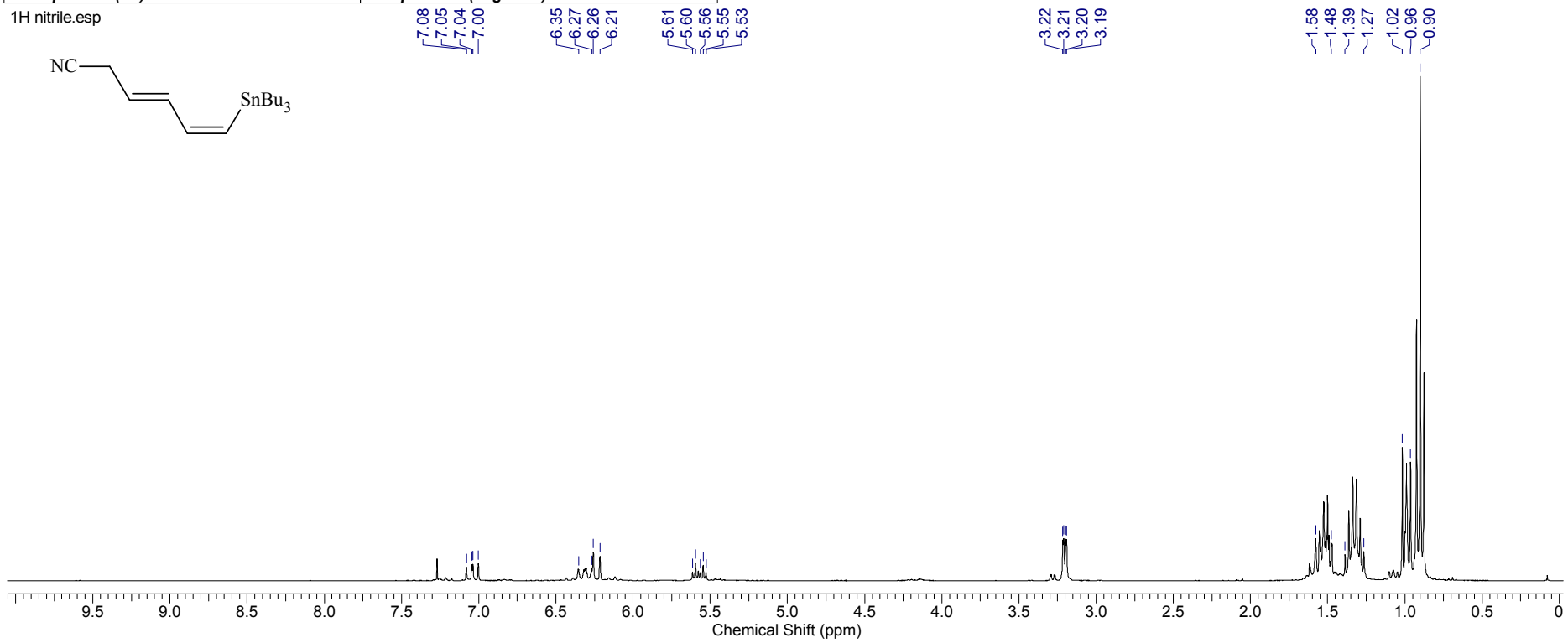


^{1}H NMR: (3*E*, 5*Z*)-6-(Tributylstannyl)hexa-3,5-dienenitrile (7)

Formula $C_{18}H_{33}NSn$	***FW*** 382.1713

Acquisition Time (sec)	4.5613	***Comment***	Slot No. 54 Sample ID Pal664 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	16 Sep 2010 20:22:56			***Date Stamp***	16 Sep 2010 20:22:56		
File Name	\\npcjm\Public\Moses Group Work\Dr Pallavi Sharma\NMR\NMR_Feb'11\DPX300\p_sha.Pal664\1\pdata\1\1r					***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx300	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zg30	***Receiver Gain***	101.60
SW(cyclical) (Hz)	3591.95	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	1647.5039	***Spectrum Type***	STANDARD
Sweep Width (Hz)	3591.84	***Temperature (degree C)***	25.000				

1H nitrile.esp

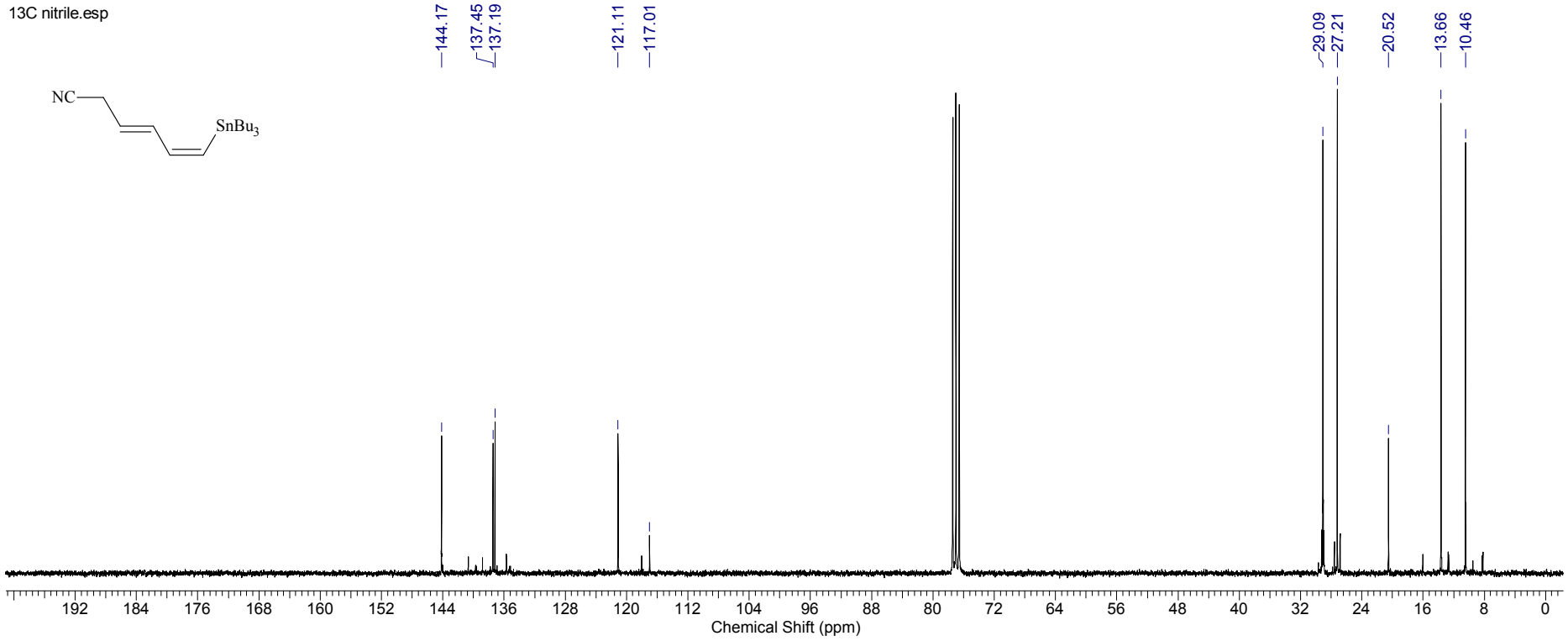


^{13}C NMR: (3*E*, 5*Z*)-6-(Tributylstannyl)hexa-3,5-dienenitrile (7)

Formula $C_{18}H_{33}NSn$	***FW***	382.1713	

Acquisition Time (sec)	0.8700	***Comment***	Slot No. 54 Sample ID Pal664 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	16 Sep 2010 22:41:36			***Date Stamp***	16 Sep 2010 22:41:36		
File Name	C:\Users\psharma\Desktop\Papers\Unpublished\Kingianin A\Kingainin\NMR\p_sha.Pal664\3\pdata\1\1r					***Frequency (MHz)***	75.47
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx300	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	9195.20
SW(cyclical) (Hz)	18832.39	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	8299.8379	***Spectrum Type***	STANDARD
Sweep Width (Hz)	18831.82	***Temperature (degree C)***	25.000				

13C nitrile.esp

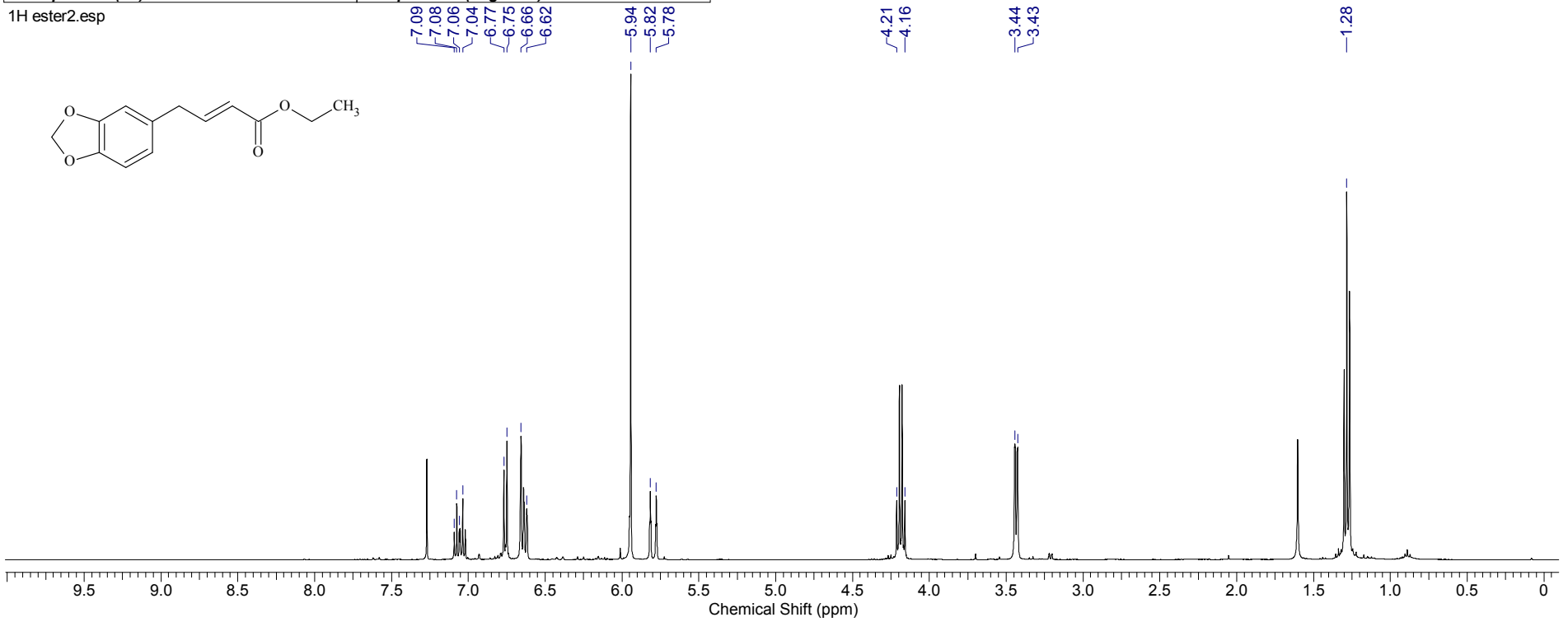


^{1}H NMR: (*E*)-Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)but-2-enoate (8)

06/04/2011 11:48:27

Formula $C_{13}H_{14}O_4$	***FW***	234.2479					
Acquisition Time (sec)	3.9846	***Comment***	UserID j_bur SampleID jb243 SupervisorID moses Lab Phone No. 13540 Slot Number 45				
Date	16 Aug 2010 17:10:40			***Date Stamp***	16 Aug 2010 17:10:40		
File Name	C:\Users\psharma\Desktop\Kingianin A NMR for Pallavi\wittig ester\proton\j_bur.jb243\1\pdata\1\1r					***Frequency (MHz)***	400.07
Nucleus	1H	***Number of Transients***	16	***Origin***	av3400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	181.00
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2462.6035	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	24.925				

1H ester2.esp



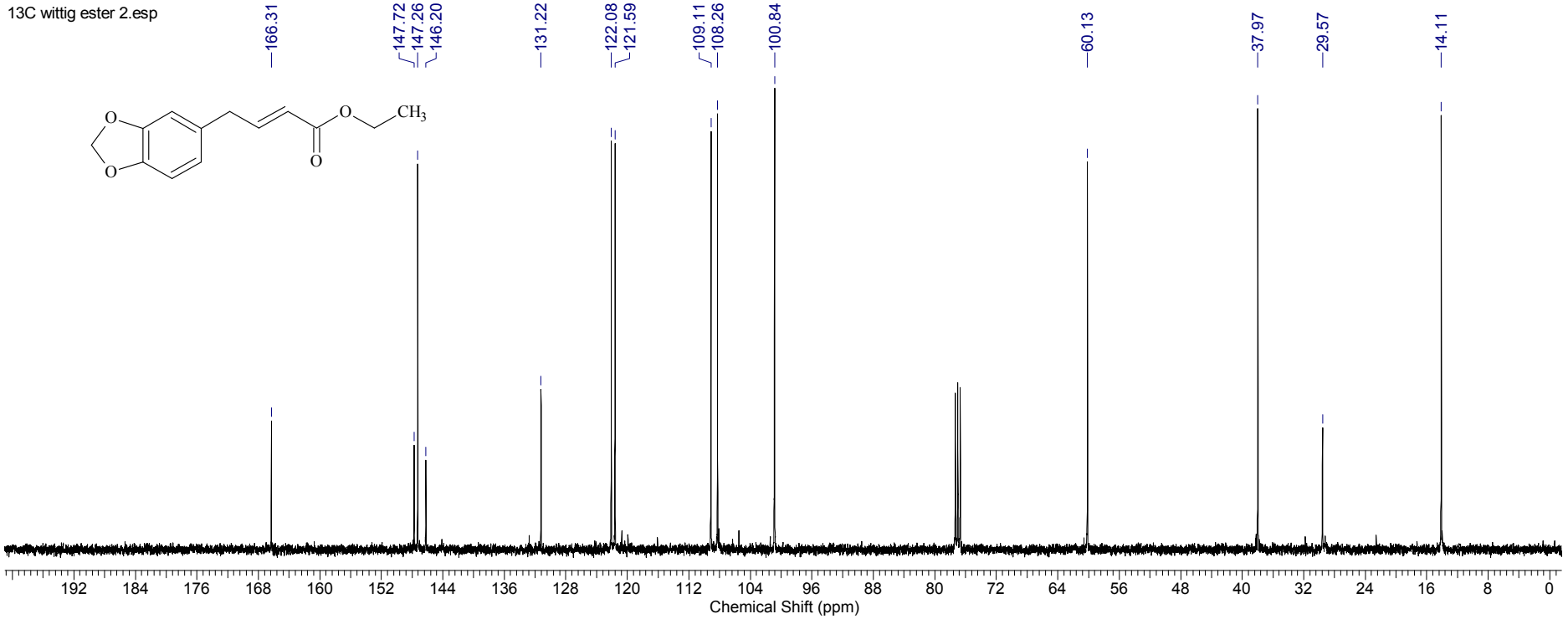
^{13}C NMR: (*E*)-Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)but-2-enoate (8)

06/04/2011 11:52:05

Formula $C_{13}H_{14}O_4$	***FW***	234.2479	

Acquisition Time (sec)	0.6832	***Comment***	UserID j_bur SampleID jb243 clean SupervisorID moses Lab Phone No. 13540 Slot Number 29				
Date	17 Aug 2010 12:41:52			***Date Stamp***	17 Aug 2010 12:41:52		
File Name	C:\Users\psharma\Desktop\Kingianin A NMR for Pallavi\wittig ester\carbon\1\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	128	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	20642.50
SW(cyclical) (Hz)	23980.81	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11053.2539	***Spectrum Type***	STANDARD
Sweep Width (Hz)	23980.08	***Temperature (degree C)***	25.160				

13C wittig ester 2.esp



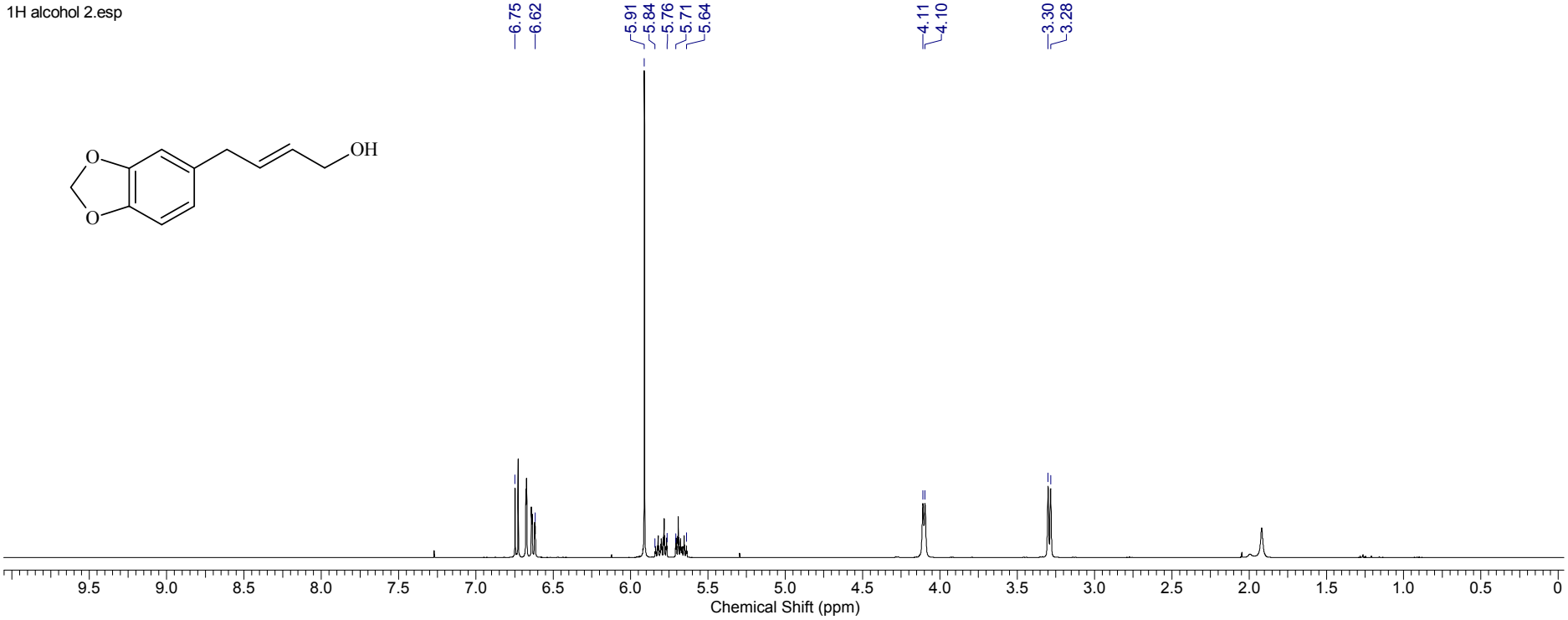
^{1}H NMR: (*E*)-4-(Benzo[d][1,3]dioxo-5-yl)but-2-en-1-ol (9)

06/04/2011 11:56:26

Formula $C_{11}H_{12}O_3$	***FW*** 192.2112

Acquisition Time (sec)	3.4210	***Comment***	Slot No. 40 Sample ID jb249 column SupervisorID moses Lab Phone No. 13540 UserID j_bur				
Date	18 Aug 2010 18:01:52			***Date Stamp***	18 Aug 2010 18:01:52		
File Name	C:\Users\psharma\Desktop\Kingianin A NMR for Pallavi\allyl alcohol\proton\3\pdata\1\1r					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zg30	***Receiver Gain***	90.50
SW(cyclical) (Hz)	4789.27	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2192.1887	***Spectrum Type***	STANDARD
Sweep Width (Hz)	4789.13	***Temperature (degree C)***	23.500				

1H alcohol 2.esp



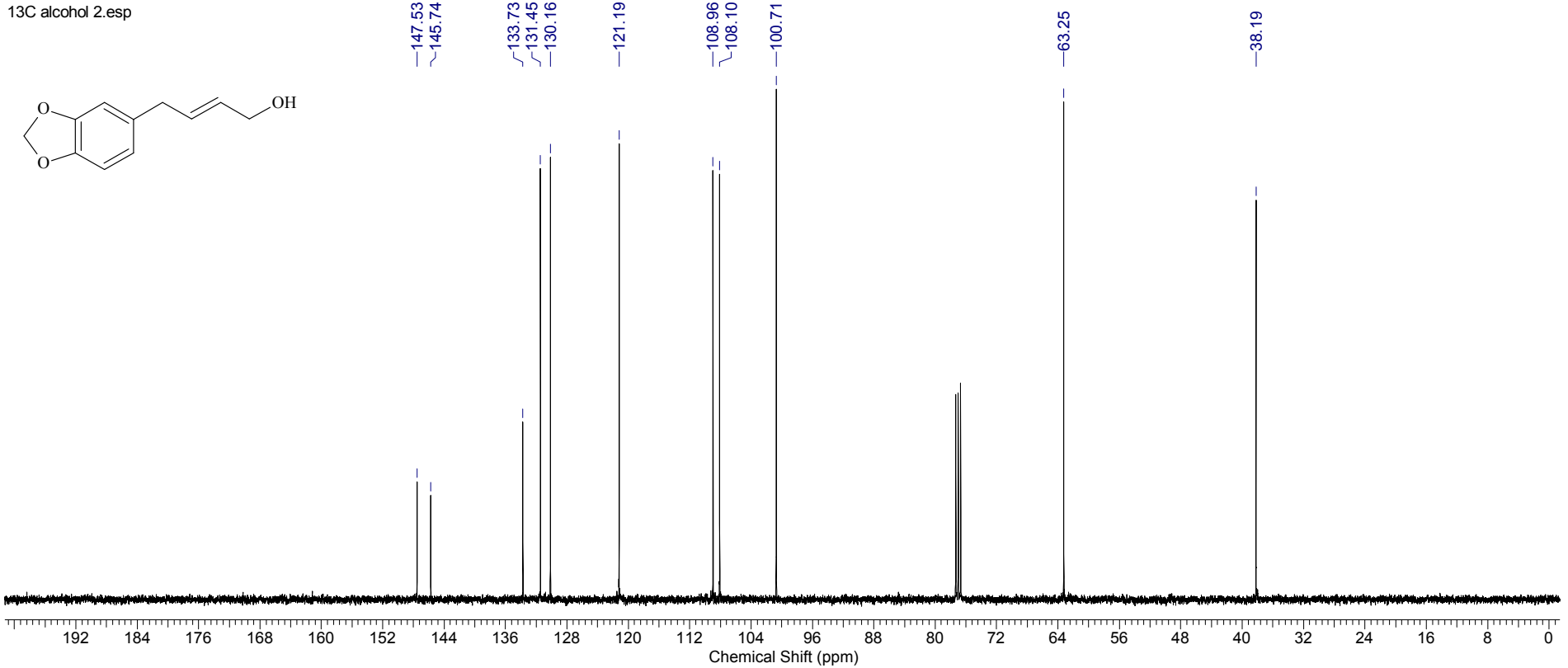
^{13}C NMR: (*E*)-4-(Benzo[d][1,3]dioxo-5-yl)but-2-en-1-ol (9)

06/04/2011 11:59:10

Formula $C_{11}H_{12}O_3$	***FW***	192.2112	

Acquisition Time (sec)	1.3042	***Comment***	Slot No. 40 Sample ID jb249 column SupervisorID moses Lab Phone No. 13540 UserID j_bur				
Date	18 Aug 2010 17:55:58	***File Name***	C:\Users\psharma\Desktop\Kingianin A NMR for Pallavi\allyl alcohol\carbon\1\1r			***Frequency (MHz)***	100.63
Nucleus	13C	***Original Points Count***	32768	***Points Count***	32768	***SW(cyclical) (Hz)***	25125.63
Solvent	CHLOROFORM-d	***Spectrum Offset (Hz)***	11056.0156	***Sweep Width (Hz)***	25124.86	***Temperature (degree C)***	0.000

13C alcohol 2.esp



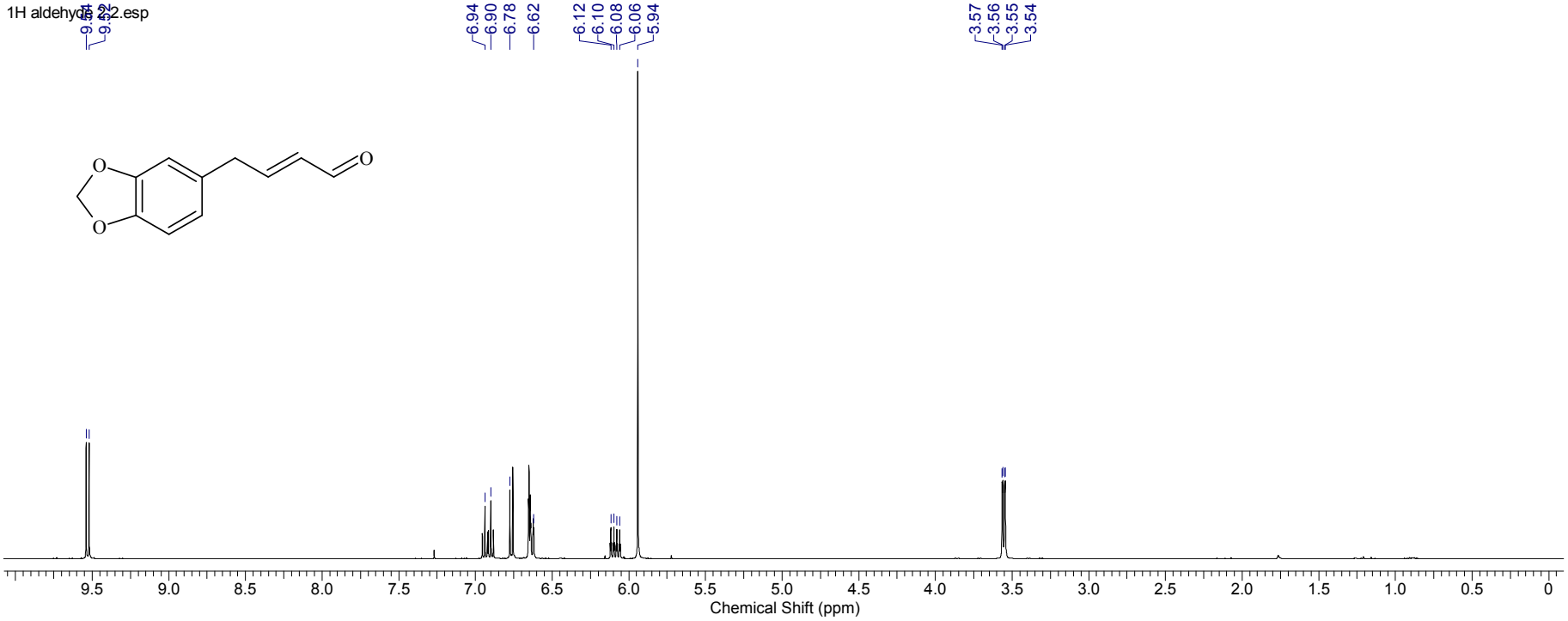
^{1}H NMR: (*E*)-4-(Benzo[*d*][1,3]dioxol-5-yl)but-2-enal (10)

06/04/2011 14:07:55

Formula $C_{11}H_{10}O_3$	***FW***	190.1953	

Acquisition Time (sec)	3.9846	***Comment***	Slot No. 42 Sample ID Pal749 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	09 Nov 2010 18:22:56			***Date Stamp***	09 Nov 2010 18:22:56		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\aldehyde 2\p_sha.Pal749\4\pdata\1\1r					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	143.70
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2462.4192	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				

1H aldehyde 2.esp



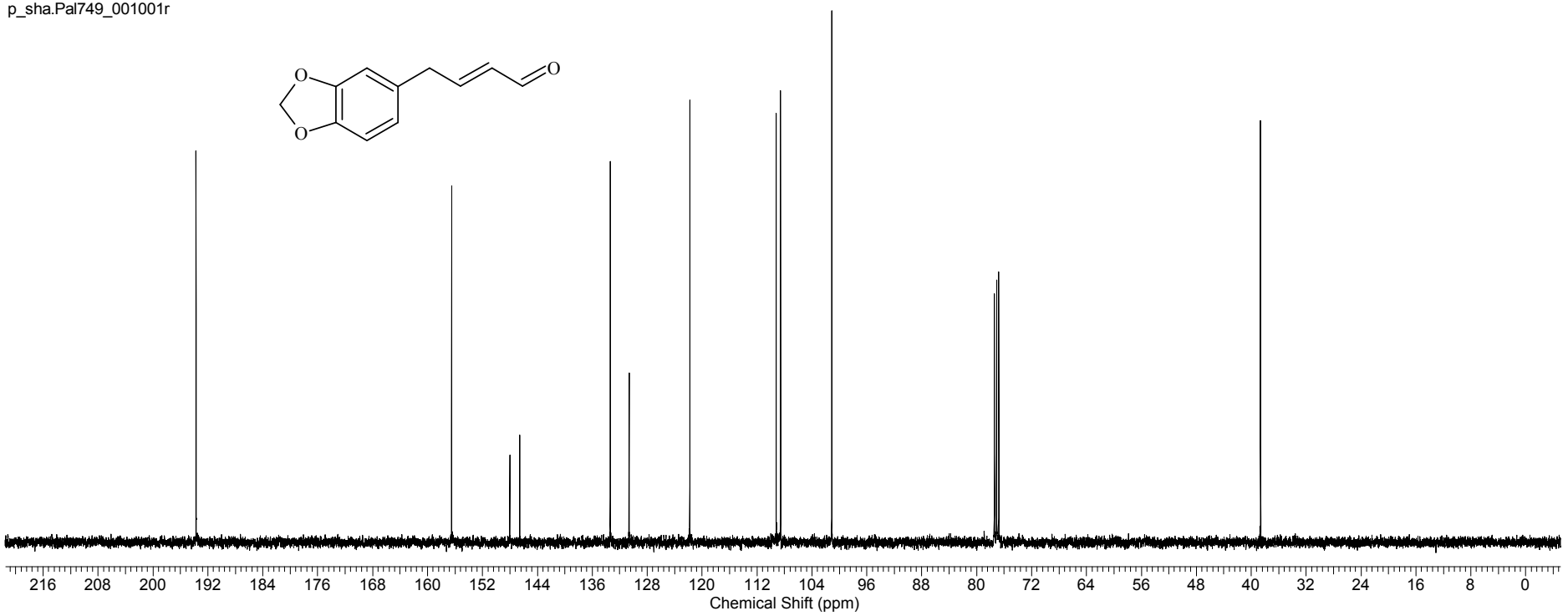
^{13}C NMR: (*E*)-4-(Benzo[*d*][1,3]dioxol-5-yl)but-2-enal (10)

06/04/2011 14:12:04

Formula $C_{11}H_{10}O_3$	***FW***	190.1953	

Acquisition Time (sec)	0.6521	***Comment***	Slot No. 42 Sample ID Pal749 SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	09 Nov 2010 18:14:24			***Date Stamp***	09 Nov 2010 18:14:24		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\aldehyde 2\p_sha.Pal749\1\pdata\1\1r					***Frequency (MHz)***	100.63
Nucleus	13C	***Number of Transients***	128	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	14596.50
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11069.0000	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.000				

p_sha.Pal749_001001r



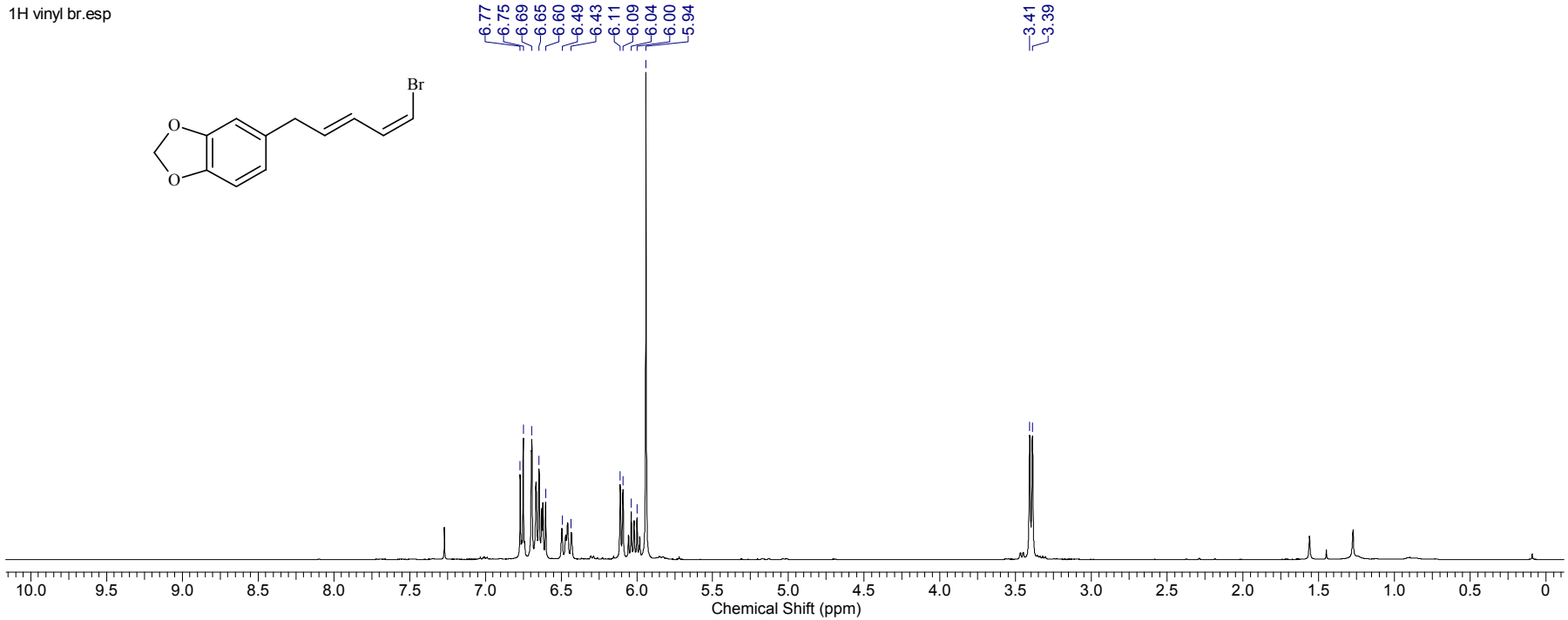
^{1}H NMR: 5-((2*E*,4*Z*)-5-Bromopenta-2,4-dien-1-yl)benzo[*d*][1,3]dioxole (6)

06/04/2011 14:16:10

Formula $C_{12}H_{11}BrO_2$	***FW***	267.1185	

Acquisition Time (sec)	3.9846	***Comment***	Slot No. 48 Sample ID Palsaf-Br SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	29 Mar 2011 16:45:04			***Date Stamp***	29 Mar 2011 16:45:04		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\vinyl bromide\p_sha.Palsaf-br\1\pdata\1\1r					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	287.40
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2462.4192	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				

1H vinyl br.esp

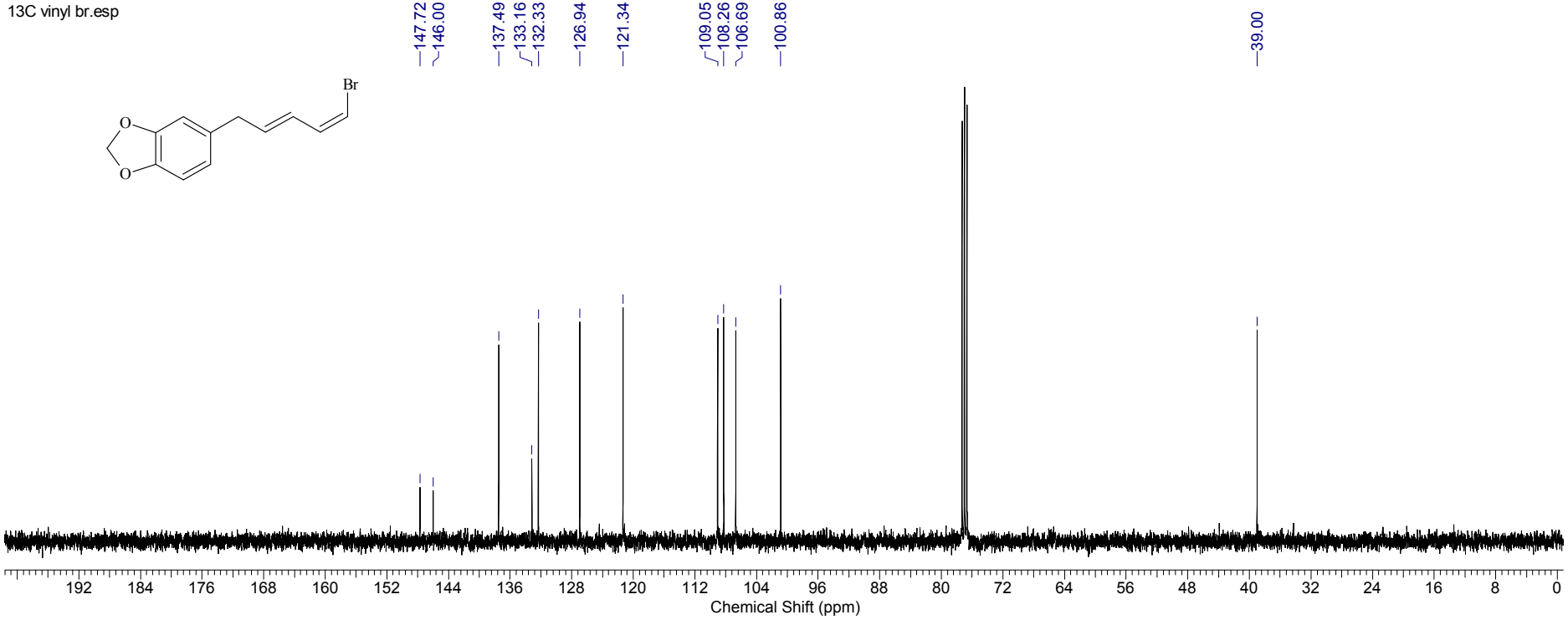


^{13}C NMR: 5-((2*E*,4*Z*)-5-Bromopenta-2,4-dien-1-yl)benzo[*d*][1,3]dioxole (6)

06/04/2011 14:20:36

Formula $C_{12}H_{11}BrO_2$	***FW***	267.1185					
Acquisition Time (sec)	0.6521	***Comment***	Slot No. 48 Sample ID Palsaf-Br SupervisorID moses Lab Phone No. 13540 UserID p_sha				
Date	29 Mar 2011 16:49:20	***Date Stamp***	29 Mar 2011 16:49:20				
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-dpx400\vinyl bromide\p_sha.Palsaf-br\2\pdata\1\1r					***Frequency (MHz)***	100.63
Nucleus	13C	***Number of Transients***	128	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	18390.40
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11064.4502	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.000				

13C vinyl br.esp

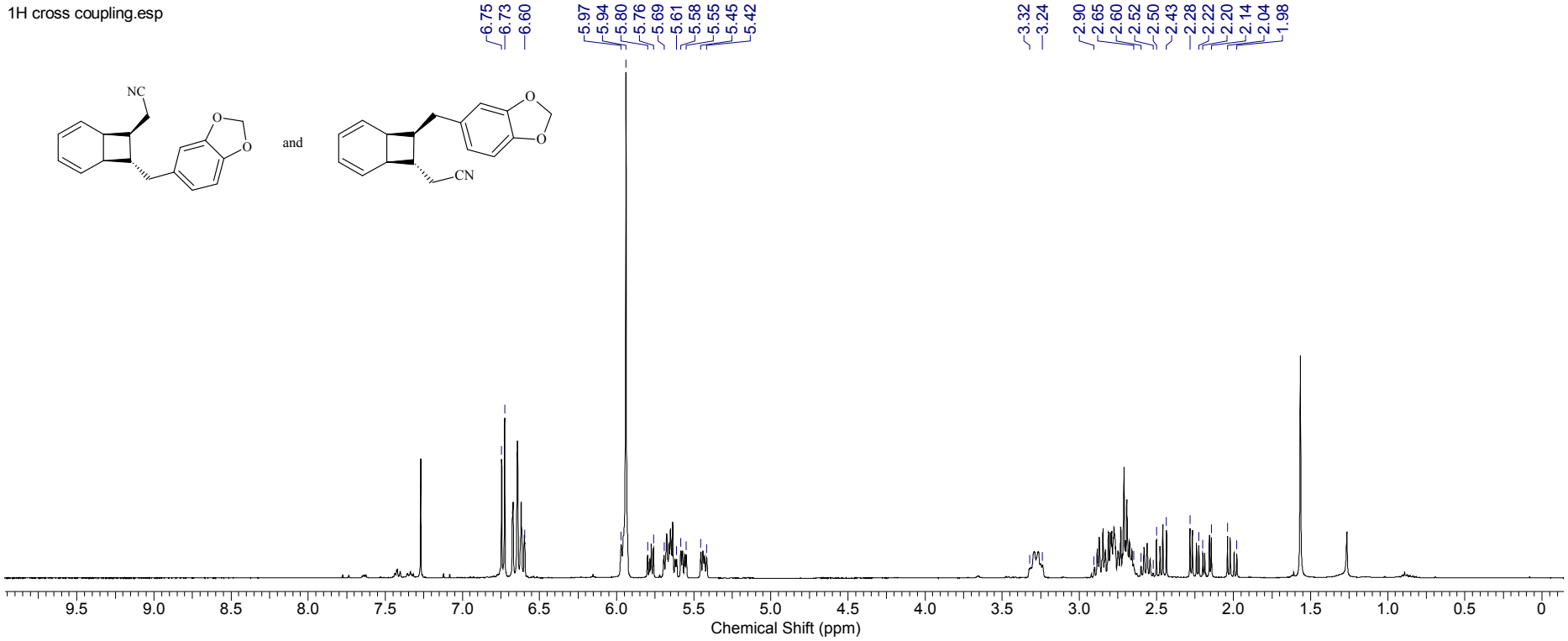


^{1}H NMR: Bicyclo[4.2.0]octadiene 16 and 17

Formula $C_{36}H_{34}N_2O_4$?	***FW***	558.6662+? (279.3331+279.3331+?)	

Acquisition Time (sec)	3.9846	***Comment***	UserID p_sha SampleID Pal713-1b-re SupervisorID moses Lab Phone No. 13540 Slot Number 28				
Date	14 Oct 2010 17:38:24	***Date Stamp***	14 Oct 2010 17:38:24				
File Name	C:\Users\psharma\Desktop\Kingainin\nmr-kingianin-3400\cross coupling\p_sha.PAI713-1b-re\1\pdata\1\1r					***Frequency (MHz)***	400.07
Nucleus	1H	***Number of Transients***	64	***Origin***	av3400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	256.00
SW(cyclical) (Hz)	8223.68	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	2461.9761	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	24.925				

1H cross coupling.esp

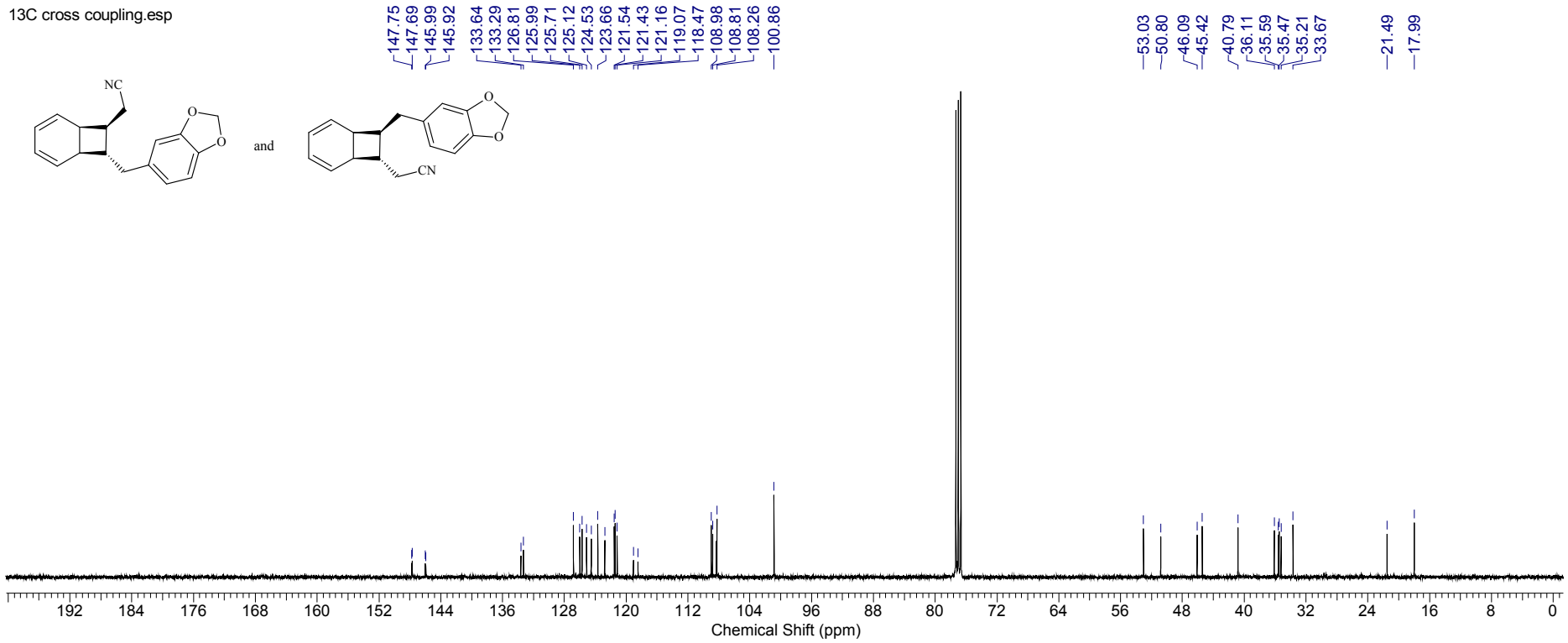


^{13}C NMR: Bicyclo[4.2.0]octadiene 16 and 17

Formula $C_{36}H_{34}N_2O_4$?	***FW***	558.6662+? (279.3331+279.3331+?)	

Acquisition Time (sec)	0.6521	***Comment***	UserID p_sha SampleID PAl713-1b-re SupervisorID moses Lab Phone No. 13540 Slot Number 34				
Date	16 Oct 2010 01:23:28	***Date Stamp***	16 Oct 2010 01:23:28				
File Name	C:\Users\psharma\Desktop\Kingainin\NMR-kingianin-400\cross coupling\p_sha.Pal713-1b-re\1\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	4096	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	20642.50
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11063.0947	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.160				

13C cross coupling.esp

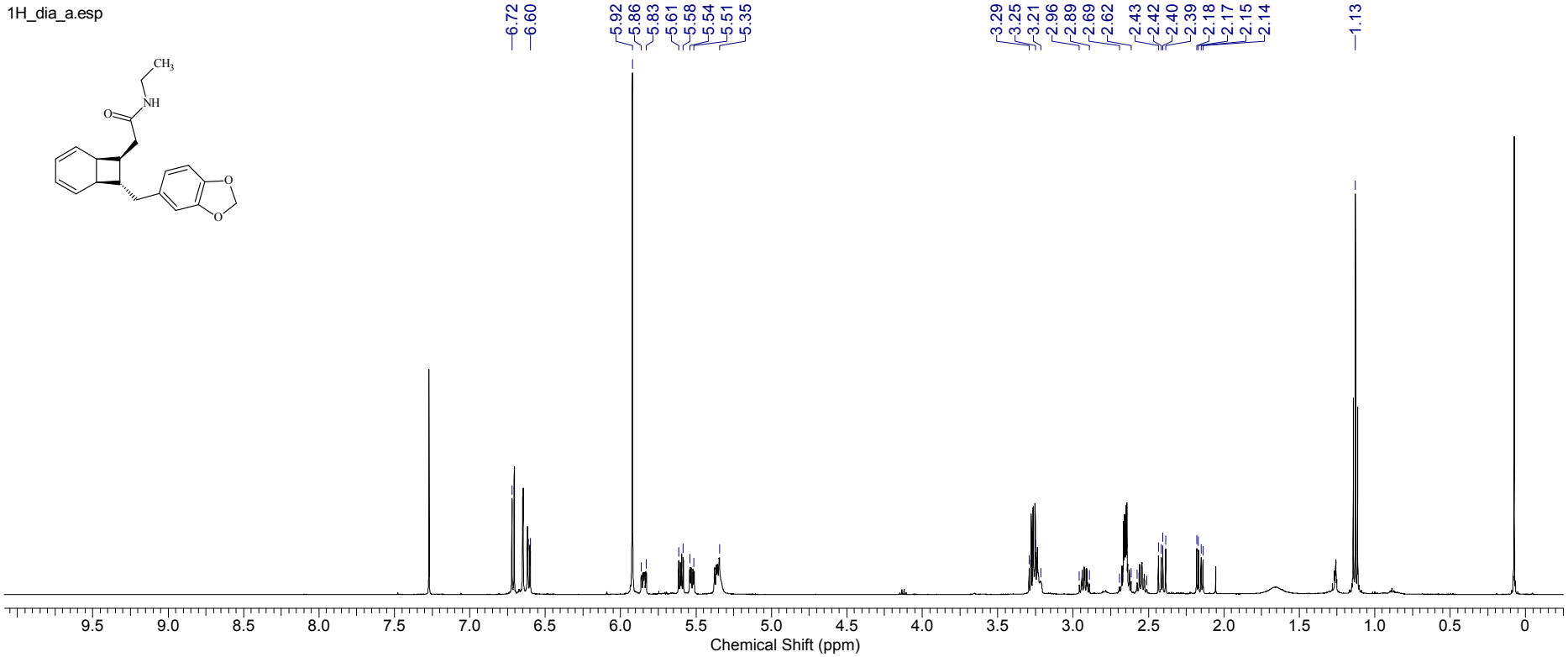


^{1}H NMR: Bicyclo[4.2.0]octadiene 18

Formula $C_{20}H_{23}NO_3$	***FW***	325.4015	

Acquisition Time (sec)	3.1719	***Comment***	5 mm CPDCH 13C/D-1H Z-GRD Z107909/0002			***Date***	22 Feb 2011 13:24:16
Date Stamp	22 Feb 2011 13:24:16			***File Name***	C:\Users\psharma\Desktop\Kingainin\NMR\500\p_sha.Pal741-1\1\pdata\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	4	***Origin***	av3500
Original Points Count	32768	***Owner***	service	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	287.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	948.1602	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

1H_dia_a.esp

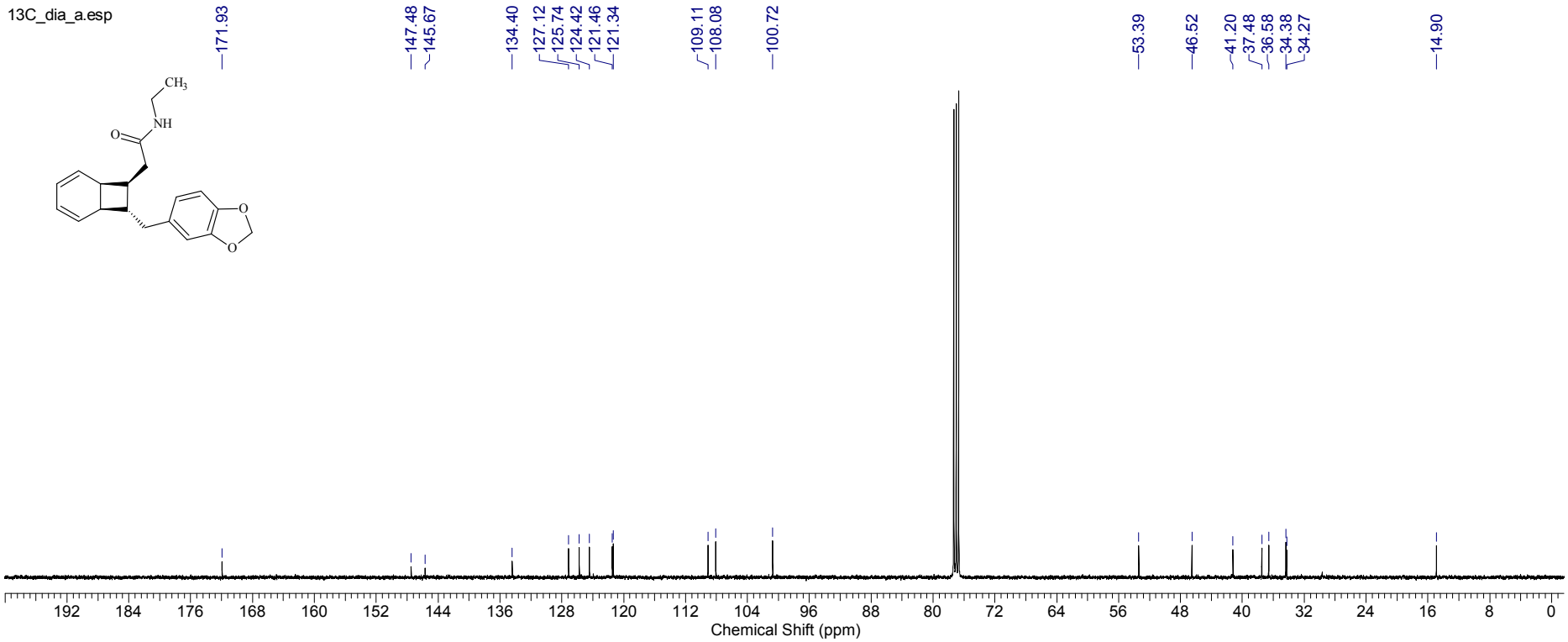


^{13}C NMR: Bicyclo[4.2.0]octadiene 18

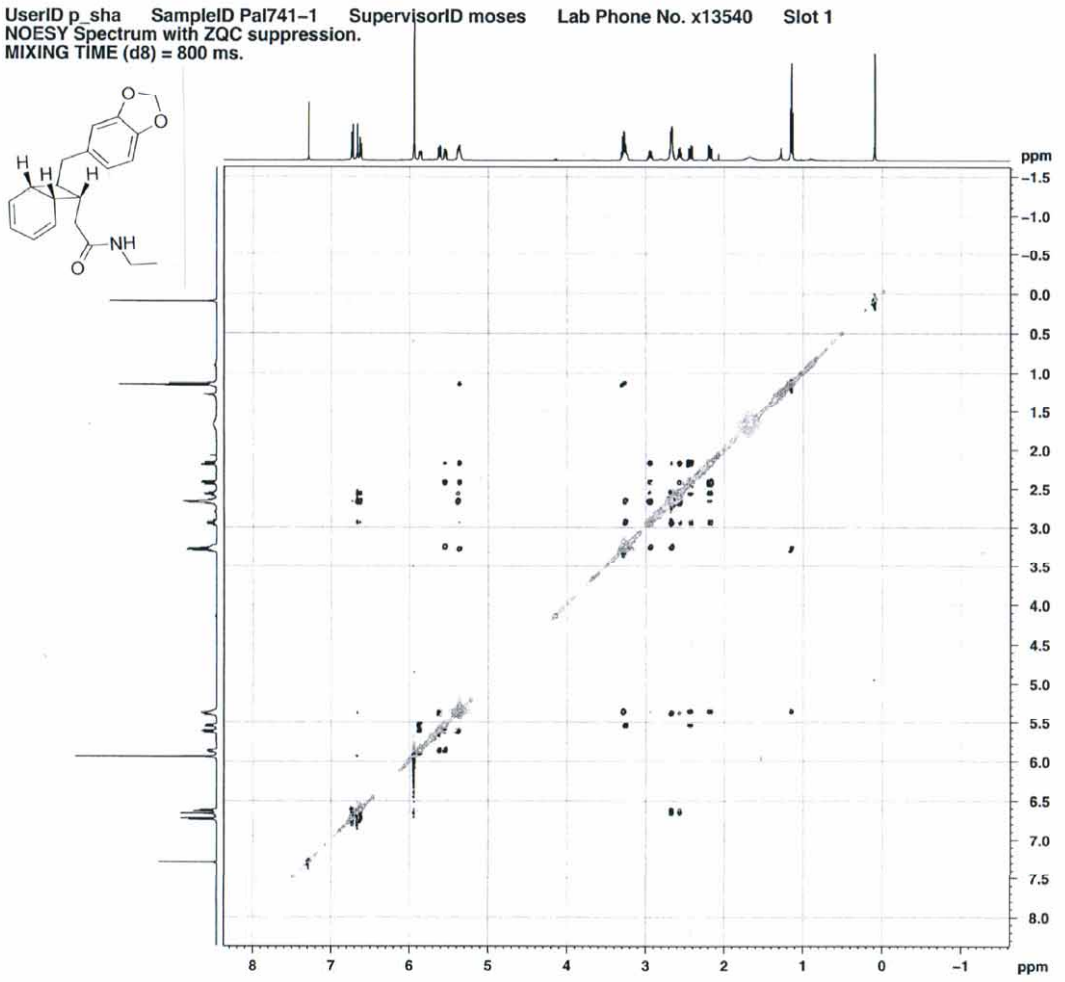
Formula $C_{20}H_{23}NO_3$	***FW***	325.4015	

Acquisition Time (sec)	0.6521	***Comment***	UserID p_sha SampleID Pal741-1 SupervisorID moses Lab Phone No. 13540 Slot Number 52				
Date	04 Nov 2010 03:03:28			***Date Stamp***	04 Nov 2010 03:03:28		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR\p_sha.Pal741-1-13C\1\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	4096	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	20642.50
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11063.8613	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.160				

13C_dia_a.esp



NOESY Bicyclo[4.2.0]octadiene 18



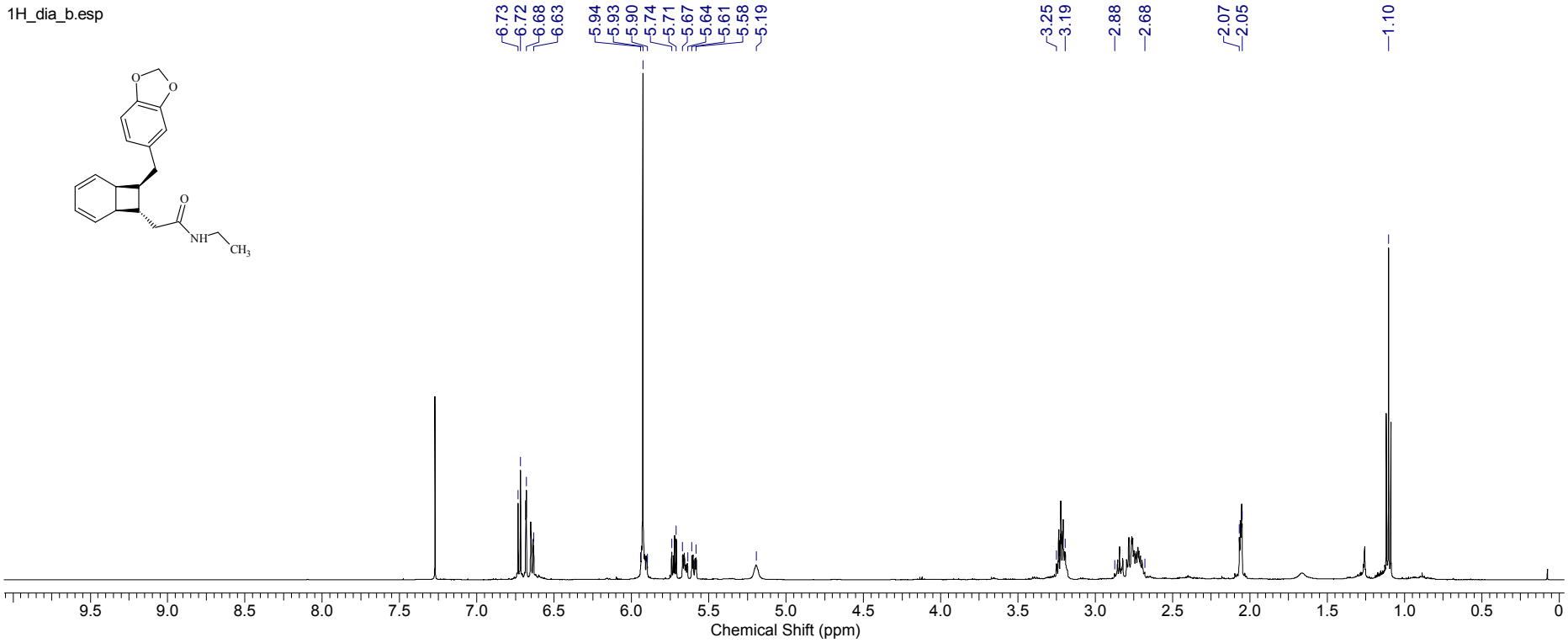
NAME	p_sha.Pal741-1
EXPNO	11
PROCNO	1
Date_	20110222
Time	15.54
INSTRUM	av3500
PROBHD	5 mm CPDCH 13C
PULPROG	noesygpphzs
TD	2048
SOLVENT	CDCl3
NS	16
DS	64
SWH	5000.000 Hz
FIDRES	2.441406 Hz
AQ	0.2048500 sec
RG	128
DW	100.000 usec
DE	90.90 usec
TE	298.0 K
D0	0.00008994 sec
D1	1.46579802 sec
D8	0.80000001 sec
D16	0.00020000 sec
IN0	0.00020000 sec
========	CHANNEL f1 ========
NUC1	1H
P1	7.90 usec
P32	20000.00 usec
PL1	1.60 dB
PL1W	20.50605011 W
SFO1	500.1316881 MHz
SP29	27.83 dB
SPNAM29	Crp60.20.20.10
SPOAL29	0.500
SPOFFS29	0.00 Hz
======	GRADIENT CHANNEL =====
GPNAM1	SINE.100
GPZ0	10.00 %
GPZ1	40.00 %
P31	5000.00 usec
ND0	1
TD	512
SFO1	500.1317 MHz
FIDRES	9.765625 Hz
SW	9.997 ppm
FnMODE	States-TPPI
SI	2048
SF	500.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

^{1}H NMR:Bicyclo[4.2.0]octadiene 2 (pre-kingianin A)

Formula $C_{20}H_{23}NO_3$	***FW***	325.4015	

Acquisition Time (sec)	3.1719	***Comment***	UserID p_sha SampleID Pal741-2 SupervisorID moses Lab Phone No. x13540 Slot 4				
Date	25 Mar 2011 20:54:24			***Date Stamp***	25 Mar 2011 20:54:24		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR\500\p_sha.Pal741-2\1\pdata\1\1r					***Frequency (MHz)***	500.13
Nucleus	1H	***Number of Transients***	16	***Origin***	av3500	***Original Points Count***	32768
Owner	service	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	181.00
SW(cyclical) (Hz)	10330.58	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	3080.3706	***Spectrum Type***	STANDARD
Sweep Width (Hz)	10330.42	***Temperature (degree C)***	25.000				

1H_dia_b.esp

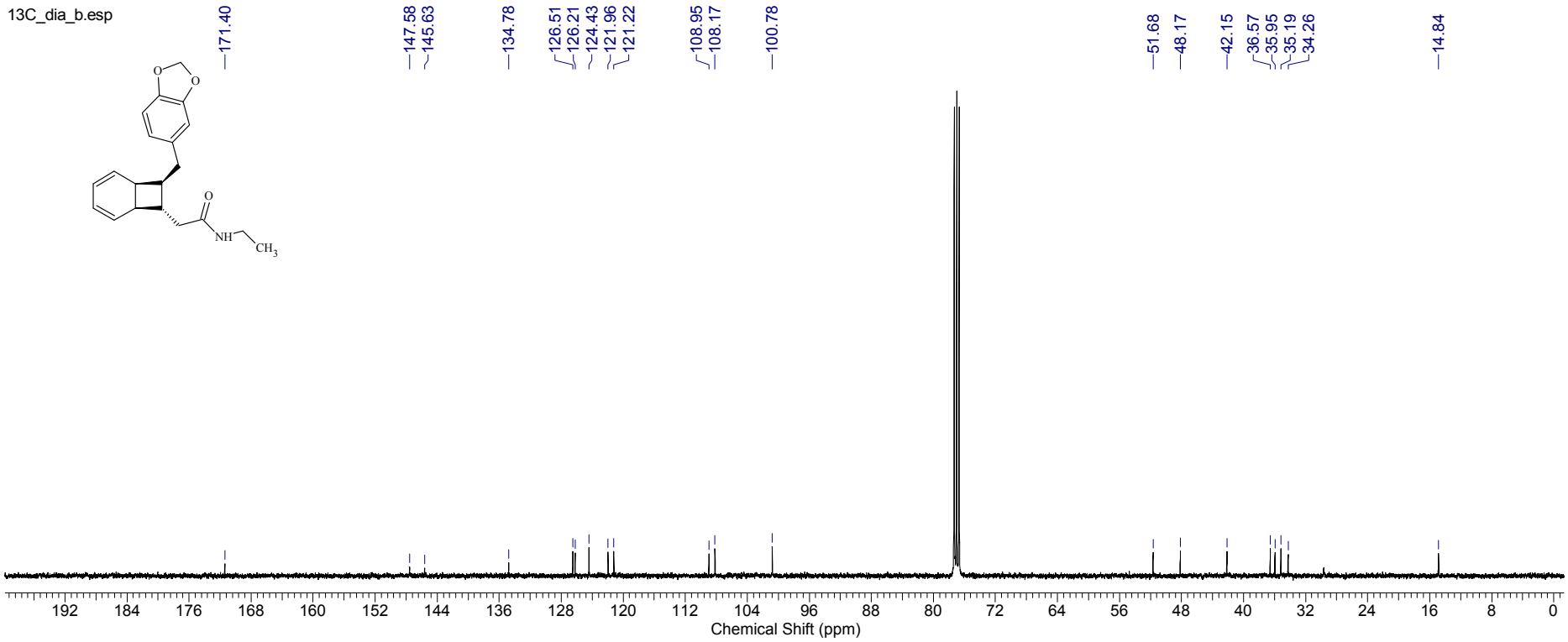


^{13}C NMR: Bicyclo[4.2.0]octadiene 2 (pre-kingianin A)

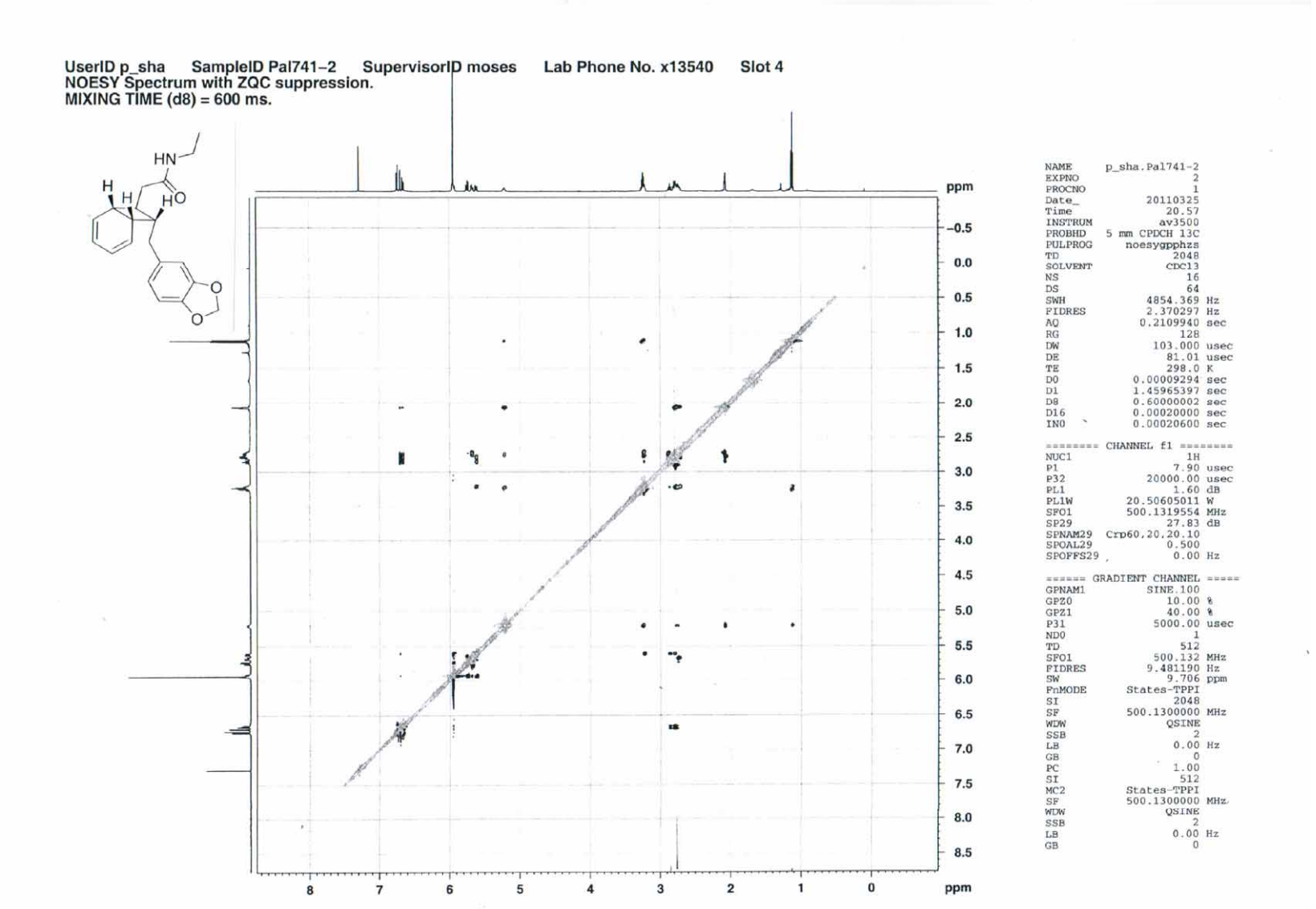
Formula $C_{20}H_{23}NO_3$	***FW***	325.4015	

Acquisition Time (sec)	0.6521	***Comment***	UserID p_sha SampleID Pal741-2 SupervisorID moses Lab Phone No. 13540 Slot Number 54				
Date	04 Nov 2010 06:06:56			***Date Stamp***	04 Nov 2010 06:06:56		
File Name	C:\Users\psharma\Desktop\Kingainin\NMR\p_sha.Pal741-2-13C\1\pdata\1\1r					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	4096	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	20642.50
SW(cyclical) (Hz)	25125.63	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11063.8613	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.160				

13C_dia_b.esp



NOESY Bicyclo[4.2.0]octadiene 2 (pre-kingianin A)

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EXPNO                      2
PROCNO                     1
Date_               20110325
Time                   20.57
INSTRUM                av3500
PROBHD    5 mm CPDCH 13C
PULPROG       noesygpphzs
TD                      2048
SOLVENT                CDC13
NS                        16
DS                        64
SWH                4854.369 Hz
FIDRES             2.370297 Hz
AQ                 0.2109940 sec
RG                       128
DW                   103.000 usec
DE                    81.01 usec
TE                    298.0 K
D0                 0.00009294 sec
D1                 1.45965397 sec
D8                 0.60000002 sec
D16                0.00020000 sec
IN0                0.00020600 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       7.90 usec
P32                  20000.00 usec
PL1                      1.60 dB
PL1W               20.50605011 W
SFO1              500.1319554 MHz
SP29                    27.83 dB
SPNAM29        Crp60.20.20.10
SPOAL29                 0.500
SPOFFS29                0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1                SINE.100
GPZ0                     10.00 %
GPZ1                     40.00 %
P31                    5000.00 usec
ND0                        1
TD                       512
SFO1              500.132 MHz
FIDRES            9.481190 Hz
SW                    9.706 ppm
PnMODE            States-TPPI
SI                       2048
SF                500.1300000 MHz
WDW                     QSINE
SSB                        2
LB                      0.00 Hz
GB                         0
PC                      1.00
SI                       512
MC2               States-TPPI
SF                500.1300000 MHz
WDW                     QSINE
SSB                        2
LB                      0.00 Hz
GB                         0
```