Electronic Supplementary Information for

Palladium-catalyzed Asymmetric 6-Endo Cyclization of Dienamides with Substituents-driven Activation

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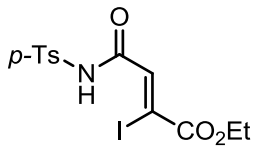
1. Experimental Section

(I) General Experimental

All reactions were carried out under Argon, in flame-dried glassware with magnetic stirring and monitored by thin-layer chromatography (TLC) using Merck precoated TLC plates (silica gel 60 GF_{254}, 0.25 mm). All commercially available reagents were used without further purification. Dioxane was refluxed over and distilled from sodium. Toluene was refluxed over and distilled from CaH_2. Preparative separation was usually performed by column chromatography on silica gel (FUJI silysia LTD, BW-200). ^{1}H

NMR and ^{13}C NMR spectra were recorded on a JEOL α-400 spectrometer. Chemical shifts for proton NMR spectra are reported in parts per million (ppm) downfield from tetramethylsilane and were referenced to residual protonated solvent ($CHCl_3$: d 7.24 ppm). Chemical shifts for carbon NMR spectra are reported in parts per million downfield from tetramethylsilane and referenced to protonated solvent ($CHCl_3$: d 77.0 ppm). Data are represented as follows: chemical shift (multiplicity [br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet], coupling constants in Hertz, integration). Infrared (IR) spectra were recorded on a SHIMADZU FT/IR-8000 Fourier Transform Infrared Spectrometer. Optical rotations were measured on a JASCO DIP-370 digital polarimeter. High resolution mass spectra (HRMS) were measured on a JEOL JMS-T100LC spectrometer. Analytical HPLC was carried out on a JASCO PU-2080 and UV-2070 instrument with UV-VIS detector.

(II) Synthesis of (*Z*)-3-ethoxycarbonyl-3-iodo- *N*-*p*-toluenesulfonylacrylamide **a**

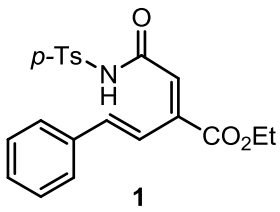


a

To a solution of (*Z*)-3-ethoxycarbonyl-3-iodo-acrylic acid [1)] (1.54 g, 3.31 mmol) in THF (15 mL) was added *N*-methylmorphorine (0.47 mL, 4.31 mmol) at 0 ^oC. After the mixture was stirred at this temperature for 10 min, isobutyl chloroformate (0.52 mL, 3.98 mmol) was added. The mixture was stirred at this temperature for 15 min then a saturated aqueous NH_4Cl solution was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 6.6% to 9.0% ethyl acetate in hexane) gave mixed anhydride (1.76 g, 95%) as a yellow oil. To a suspension of sodium hydride (562 mg, 14.0 mmol, 60% in oil) in THF (45 mL) was added *p*-toluenesulfonamide (2.04 g, 11.9 mmol) at 0 ^oC. The mixture was stirred for 10 min at this temperature then a solution of the crude mixed anhydride (4.00 g, 10.8 mmol) in THF (5 mL) was added. After the mixture was stirred for 2 h at room temperature, a saturated aqueous $NaHCO_3$ solution was added, and the resulting mixture was extracted with saturated aqueous $NaHCO_3$ solution. The aqueous layers were combined, and a 2 N HCl solution (50 mL) was added, and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give *N*-sulfonyl amide **a**

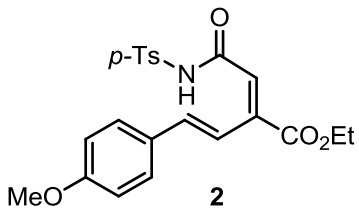
(4.31 g, 94%) as a yellow solid. : R_f 0.22 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}); 3232, 2982, 1693, 1599, 1449, 1367, 1252, 810, 702; ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, 2H, *J* = 8.8 Hz), 7.62 (s, 1H), 7.36 (d, 2H, *J* = 8.4 Hz), 4.31 (q, 2H, *J* = 7.6 Hz), 2.46 (s, 3H), 1.34 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 161.2, 145.6, 136.9, 135.0, 129.7, 128.6, 103.1, 64.1, 21.7, 13.9; ESI HRMS *m*/*z* calcd for $C_{13}H_{14}NO_5SI$ $(M-H)^-$, 421.9559, found 421.9540.

(III) Synthesis of dienamides **1**-**8**



1

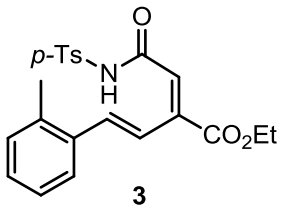
To a solution of *N*-sulfonyl amide **a** (1.00 g, 2.36 mmol) in dioxane (20 mL) were added (*E*)-1-phenyl-2-tributylstannyl ethene [2] (1.21 g, 3.07 mmol) in dioxane (5 mL), dichloro bis(triphenylphosphine)palladium(II) (166 mg, 0.236 mmol) and copper iodide(I) (540 mg, 2.84 mmol) at room temperature. After the mixture was stirred at 80 ^oC for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **1** (915 mg, 97%) as a yellow amorphous solid: R_f 0.36 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}); 3250, 2984, 1721, 1692, 1611, 1443, 1253, 1084, 974, 814; ^1H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 8.01-7.92 (m, 3H), 7.50-7.45 (m, 2H), 7.37-7.31 (m, 5H), 7.21 (d, 1H, *J* = 18.7 Hz), 6.19 (s, 1H), 4.34 (q, 2H, *J* = 7.2 Hz), 2.43 (s, 3H), 1.36 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 162.5, 145.8, 145.4, 141.2, 136.2, 135.7, 129.9, 129.7, 128.9, 128.6, 128.1, 120.6, 120.5, 62.3, 21.9, 14.3; ESI HRMS *m*/*z* calcd for $C_{21}H_{21}NO_5S$ $(M-H)^-$, 398.1062, found 398.1045.



2

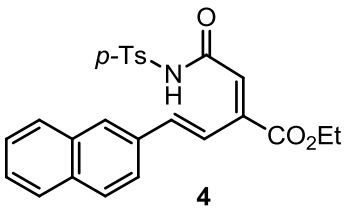
To a solution of *N*-sulfonyl amide **a** (200 mg, 0.472 mmol) in dioxane (2 mL) were added (*E*)-1-*p*-methoxyphenyl-2-tributylstannylethene [2] (260 mg, 0.614 mmol) in dioxane (3 mL), dichlorobis(triphenylphosphine)palladium(II) (33 mg, 0.047 mmol) and copper iodide(I) (108 mg, 0.567 mmol) at room temperature. After the mixture was stirred at 80 ^oC for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude products. Column

chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **2** (132 mg, 65%) as a yellow amorphous solid: R_f 0.67 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3246, 2982, 2922, 1719, 1578, 1510, 1439, 1259, 1146, 974; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13-7.94 (brs, 1H) , 7.98 (d, 2H, *J* = 8.4 Hz), 7.89 (d, 1H, *J* = 16.4 Hz), 7.44 (d, 2H, *J* = 8.8 Hz), 7.35 (d, 2H, *J* = 8.4 Hz), 7.23 (d, 1H, *J* = 16.4 Hz), 6.87 (d, 2H, *J* = 8.8 Hz), 6.06 (s, 1H), 4.33 (q, 2H, *J* = 6.8 Hz), 3.83 (s, 3H), 2.44 (s, 3H), 1.37 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 162.7, 161.1, 146.5, 145.4, 141.0, 135.8, 129.9, 129.7, 129.1, 128.6, 118.5, 114.4, 62.2, 55.5, 21.9, 14.3; ESI HRMS *m*/*z* calcd for $C_{22}H_{23}NO_6S$ $(M-H)^-$, 428.1168, found 428.1159.



3

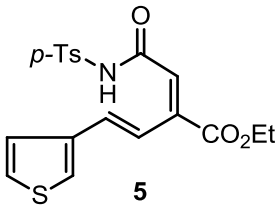
To a solution of *N*-sulfonyl amide **a** (100 mg, 0.236 mmol) in dioxane (1 mL) were added (*E*)-1-*o*-tolyl-2-tributylstannylethene [2)] (163 mg, 0.307 mmol) in dioxane (2 mL), dichloro bis(triphenylphosphine)palladium(II) (17 mg, 0.0236 mmol) and copper iodide(I) (54 mg, 0.284 mmol) at room temperature. After the mixture was stirred at 80 ^{o}C for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **3** (101 mg, 89%) as a yellow amorphous solid: R_f 0.31 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3279, 3054, 1676, 1613, 1580, 1431, 1287, 1159, 1086, 841, 668; ^{1}H NMR (400 MHz, $CDCl_3$, 55 ^{o}C) δ 8.57-8.14 (brs, 1H) , 7.96 (d, 2H, *J* = 8.0 Hz), 7.79-7.69 (m, 1H), 7.60-7.49 (m, 2H), 7.3 (d, 2H, *J* = 8.4 Hz), 7.24-7.11 (m, 2H), 6.28-6.26 (m, 1H), 4.34 (q, 2H, *J* = 7.2 Hz), 2.41 (s, 3H),2.32 (s, 3H), 1.36 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 162.5, 145.8, 145.4, 138.8, 137.3, 135.7, 135.2, 130.7, 129.9, 129.5, 128.6, 126.5, 126.4, 121.3, 120.9, 62.3, 21.9, 19.8, 14.3; ESI HRMS *m*/*z* calcd for $C_{22}H_{23}NO_5S$ $(M-H)^-$, 412.1219, found 412.1202.



4

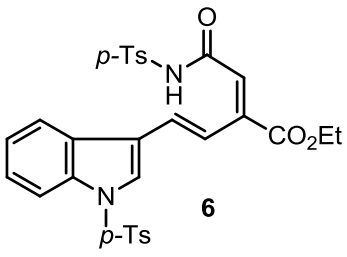
To a solution of *N*-sulfonyl amide **a** (88 mg, 0.21 mmol) in dioxane (1 mL) were added (*E*)-1-(2-naphthalenyl)-2-tributylstannylethene [2)] (120 mg, 0.271 mmol) in dioxane (1 mL), dichloro bis(triphenylphosphine)palladium(II) (15 mg, 0.021 mmol) and copper iodide(I) (48 mg, 0.25 mmol) at room temperature. After the mixture was stirred at 80 ^{o}C for 1 h, cooled to room

temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **4** (45 mg, 48%) as a yellow amorphous solid: R_f 0.41 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3222, 2982, 1676, 1655, 1560, 1509, 1426, 1082, 843, 771; ^1H NMR (400 MHz, $CDCl_3$) δ 8.71-8.52 (brs,1H), 8.12 (d, 1H, *J* = 16.4 Hz), 7.99 (d, 2H, *J* = 8.2 Hz), 7.87-7.73 (m, 4H), 7.68 (dd,1H, *J* = 8.8, 1.2 Hz), 7.52-7.44 (m,2H), 7.43 (d, 1H, *J* = 16.4 Hz), 7.34 (d, 2H, *J* = 8.0 Hz), 6.22 (s, 1H), 4.37 (q, 2H, *J* = 7.2 Hz), 2.42 (s, 3H), 1.39 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 162.5, 146.0, 145.5, 141.4, 135.7, 134.1, 133.8, 133.5, 129.9, 129.4, 128.6, 128.6, 127.9, 127.1, 127.0, 124.0, 120.7, 120.3, 62.3, 21.9, 14.3; ESI HRMS *m*/*z* calcd for $C_{25}H_{23}NO_5S$ $(M+Na)^+$, 472.1195, found 472.1207.



5

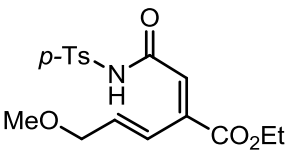
To a solution of *N*-sulfonyl amide **a** (100 mg, 0.236 mmol) in dioxane (1 mL) were added (*E*)-1-(3-thiophenyl)-2-tributyl stannylethene [2] (123 mg, 0.307 mmol) in dioxane (2 mL), dichloro bis(triphenylphosphine)palladium(II) (17 mg, 0.0236 mmol) and copper iodide(I) (54 mg, 0.284 mmol) at room temperature. After the mixture was stirred at 80 ^oC for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **5** (80 mg, 83%) as a yellow amorphous solid: R_f 0.39 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3266, 2978, 1716, 1690, 1609, 1431, 1246, 1084, 970, 774; ^1H NMR (400 MHz, $CDCl_3$) δ 8.53-8.43 (brs,1H), 7.97 (d, 2H, *J* = 8.8 Hz), 7.82 (d, 1H, *J* = 16.4 Hz), 7.38-7.27 (m, 6H), 6.15 (s,1H), 4.32 (q, 2H, *J* = 6.8 Hz), 2.44 (s, 3H), 1.35 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 162.4, 145.8, 145.2, 139.4, 135.5, 134.7, 129.7, 128.4, 126.7, 126.5, 125.3, 120.3, 119.8, 62.1, 21.7, 14.1; ESI HRMS *m*/*z* calcd for $C_{19}H_{19}NO_5S_2$ $(M-H)^-$, 404.0626, found 404.0608.



6

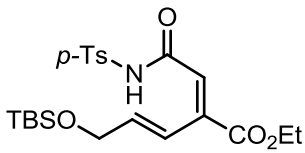
To a solution of *N*-sulfonyl amide **a** (100 mg, 0.236 mmol) in dioxane (1 mL) were added (*E*)-1-(3-*N*-*p*-toluenesulfonyl indolyl)-2-tributyl stannylethene [2] (180 mg, 0.307 mmol) in dioxane (2 mL), dichlorobis(triphenylphosphine)

palladium(II) (17 mg, 0.0236 mmol) and copper iodide(I) (54 mg, 0.284 mmol) at room temperature. After the mixture was stirred at 80 ^{o}C for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **6** (119 mg, 85%) as a yellow amorphous solid: R_f 0.60 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3254, 3024, 1724, 1597, 1447, 1375, 1217, 1127, 978, 754; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65-8.36 (brs,1H), 8.10 (d, 2H, *J* = 16.8 Hz), 8.02-7.94 (m, 3H) 7.87-7.81 (m,1H), 7.81-7.75 (m, 3H), 7.40 (d, 1H, *J* = 16.4 Hz), 7.38-7.29 (m, 5H), 7.23 (d, 2H, *J* = 8.0 Hz), 6.19 (s,1H), 4.35 (q, 2H, *J* = 6.8 Hz), 2.41 (s, 3H), 2.34 (s, 3H), 1.38 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 162.4, 145.5, 145.3, 145.2, 135.5, 135.4, 134.7, 131.8, 130.0, 129.6, 128.4, 128.2, 127.2, 126.9, 125.3, 124.1, 120.8, 120.8, 120.1, 119.7, 113.6, 67.0, 62.1, 21.6, 21.5, 14.1; ESI HRMS *m*/*z* calcd for $C_{30}H_{28}N_2O_7S_2$ $(M+Na)^+$, 615.1236, found 615.1227.



7

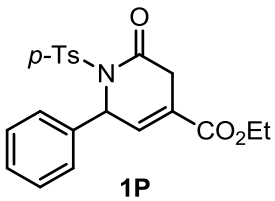
To a solution of *N*-sulfonyl amide **a** (100 mg, 0.236 mmol) in dioxane (1 mL) were added (*E*)-1-methoxymethyl-2-tributyl stannylethene [2)] (111 mg, 0.307 mmol) in dioxane (2 mL), dichloro bis(triphenylphosphine)palladium(II) (17 mg, 0.0236 mmol) and copper iodide(I) (54 mg, 0.284 mmol) at room temperature. After the mixture was stirred at 80 ^{o}C for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **7** (44 mg, 51%) as a yellow amorphous solid: R_f 0.55 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3245, 2926, 2826, 1727, 1634, 1447, 1346, 1121, 851, 666; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, 2H, *J* = 8.8 Hz), 7.35 (d, 2H, *J* = 8.0 Hz), 7.23 (d, 1H, *J* = 14.0 Hz), 6.48 (dt, 1H, *J* = 16.0, 6.0 Hz), 6.24 (s,1H), 4.28 (q, 2H, *J* = 7.2 Hz), 4.03 (dd, 2H, *J* = 5.6, 1.6 Hz), 3.34 (s, 3H), 2.45 (s, 3H), 1.32 (t, 3H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 162.3, 145.1, 144.0, 139.2, 135.5, 129.6, 128.4, 123.0, 122.2, 72.7, 62.1, 58.2, 21.6, 14.0; ESI HRMS *m*/*z* calcd for $C_{17}H_{21}NO_6S$ $(M+Na)^+$, 390.0987, found 390.0976.



8

To a solution of *N*-sulfonyl amide **a** (100 mg, 0.236 mmol) in dioxane (1 mL) were added (*E*)-1-*t*-butyldimethylsilyloxymethyl-2-tributylstannylethene [2)] (142 mg, 0.307 mmol) in dioxane (2 mL), dichloro bis(triphenylphosphine)palladium(II) (17 mg, 0.0236 mmol) and copper iodide(I) (54 mg, 0.284 mmol) at room temperature. After the mixture was stirred at 80 ^{o}C for 1 h, cooled to room temperature, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9.0% to 25% ethyl acetate in hexane) gave *N*-sulfonyl dienamide **8** (88 mg, 79%) as a yellow oil: R_f 0.56 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3249, 2930, 2859, 1721, 1701, 1443, 1254, 1086, 972, 733; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71-8.51 (brs,1H), 7.95 (d, 2H, *J* = 8.4 Hz), 7.33 (d, 2H, *J* = 8.4 Hz), 7.29-7.26 (m, 1H), 6.50 (td, 1H, *J* = 16.0, 4.4 Hz), 6.24 (s,1H), 4.33-4.23 (m, 4H), 2.44 (s, 3H), 1.31 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 162.1, 144.9, 144.2, 142.4, 135.4, 135.4, 129.5, 128.4, 121.5, 120.5, 63.5, 62.0, 25.8, 21.6, 18.2, 13.9, -5.4; ESI HRMS *m*/*z* calcd for $C_{22}H_{33}NO_6SSi$ $(M-H)^-$, 466.1720, found 466.1697.

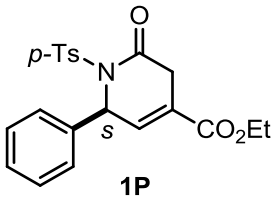
(IV) Cyclization of dienamides **1**-**8** (Synthesis of 2-piperidinones **1P**-**8P**)



1P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (12 mg, 0.013 mmol) in toluene (1 mL) was added 1,2-bis(diphenylphosphino)ethane (10 mg, 0.026 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **1** (26 mg, 0.065 mmol) in toluene (1 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **1P** (22 mg, 85%) as a white solid.

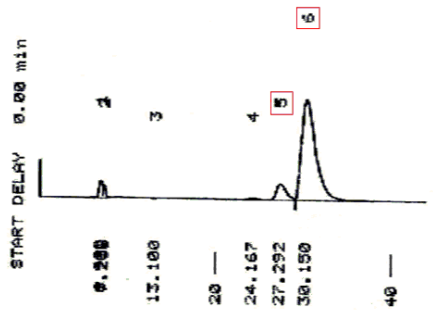
・Enantioselective cyclization of dienamide **1** with (S)-BINAP



1P

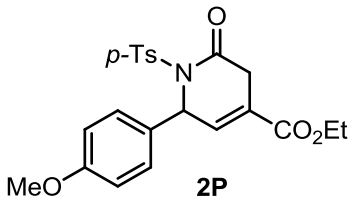
To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (8.3 mg, 0.0090 mmol) in toluene (1.5 mL) was added (S)-(-)-2,2'-bis(diphenylphosphino)-1,1'-binaphtyl (11 mg, 0.018 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **1** (72 mg, 0.18 mmol) in toluene (2.5 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **1P** (62 mg, 86%) as a white solid. The enantiomeric excess was determined to be 81% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 3/1, flow rate = 0.5 mL/min., tR(minor) = 27.3 min, tR(major) = 30.2 min) : R_f 0.62 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3034, 2929, 1721, 1708, 1598, 1456, 1370, 1264, 1173, 893 ; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.31 (m, 5H), 7.24-7.16 (m, 2H), 7.09 (d, 2H, *J* = 8.4 Hz), 7.03 (dd, 1H, *J* = 6, 2.8 Hz), 6.17 (dt, 1H, *J* = 22.0, 2.8 Hz), 4.25-4.13 (m, 2H), 3.56 (dd, 1H, *J* = 22.4, 2.4 Hz), 3.34 (dt, 1H, *J* = 22.0, 2.8 Hz), 2.35 (s, 3H), 1.25 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 164.4, 144.9, 137.9, 135.2, 134.8, 129.3, 129.2, 128.8, 127.4, 123.7, 61.6, 61.4, 33.4, 21.6, 14.1; ESI HRMS *m/z* calcd for $C_{21}H_{21}NO_5S$ $(M+Na)^+$, 422.1062, found 422.1055.

HPLC chromatogram for **1P**



-- % CALCULATION RESULT --

PEAK#	RT(min)	AREA	HEIGHT	MK	AREA%
1	6.900	616272	23600	V	3.4328
2	7.258	310762	17468	V	1.7310
3	13.100	7546	448		4.2033E-02
4	24.167	80833	1479		0.4503
5	27.292	1683687	21949	V	8.9330
6	30.150	15333348	139801	V	85.4109
	TOTAL	17952447	204736		100.0000

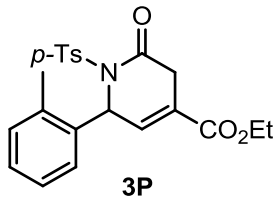


2P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (6 mg, 0.007 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (6 mg, 0.01 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **2** (20 mg, 0.047 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **2P** (14 mg, 70%) as a yellow solid.

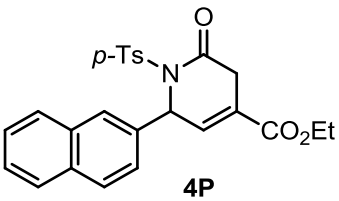
・Enantioselective cyclization of dienamide **2** with (S)-BINAP

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (6.0 mg, 0.007 mmol) in toluene (2 mL) was added (S)-(-)-2,2’-bis(diphenylphosphino)-1,1’-binaphtyl (9 mg, 0.01 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **2** (20 mg, 0.047 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **2P** (18 mg, 90%) as a yellow solid. The enantiomeric excess was determined to be 64% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 3/1, flow rate = 0.5 mL/min., tR(minor) = 29.1 min, tR(major) = 32.3 min) : R_f 0.67 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 2982, 2840, 1713, 1608, 1512, 1360, 1170, 1084, 912, 731; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, 2H, *J* = 8.4 Hz), 7.16-7.08(m, 4H), 7.01 (dd, 1H, *J* = 5.6, 2.4 Hz), 6.89-6.84 (m, 2H), 6.12 (dt, 1H, *J* = 5.2, 2.8 Hz), 4.26-4.15 (m, 2H), 3.83 (s, 3H), 3.54 (dd, 1H, *J* = 22.4, 2.0 Hz), 3.33 (dt, 1H, *J* = 22.4, 2.8 Hz), 2.36 (s, 3H), 1.27 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 164.4, 160.0, 144.8, 135.4, 135.0, 129.8, 129.2, 128.8, 128.8, 123.3, 114.5, 61.3, 61.1, 55.4, 33.4, 21.6, 14.1; ESI HRMS *m/z* calcd for $C_{22}H_{23}NO_6S$ $(M+Na)^{+}$, 452.1144, found 452.1144.



3P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (13 mg, 0.015 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (12 mg, 0.029 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **3** (30 mg, 0.073 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **3P** (28 mg, 90%) as a white solid: R_f 0.62 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3230, 1680, 1582, 1437, 1289, 1194, 1086, 1021, 841, 668; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, 2H, *J* = 8.4 Hz), 7.26-7.19(m, 3H), 7.09 (d, 2H, *J* = 8.0 Hz), 7.06-7.00 (m, 1H), 6.98-6.92 (m,1H), 6.77 (d, 1H, *J* = 8.0), 6.41 (dt, 1H, *J* = 5.2, 2.8 Hz) 4.25-4.13 (m, 2H), 3.56 (ddd, 1H, *J* = 24.0, 2.4, 0.8 Hz), 3.36 (dt, 1H, *J* = 22.4, 2.8 Hz), 2.54 (s, 3H), 2.36 (s, 3H), 1.27 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 164.5, 144.9, 136.4, 135.3, 135.0, 133.6, 131.2, 129.5, 128.7, 128.5, 126.8, 126.5, 123.2, 61.4, 58.5, 33.3, 21.6, 19.1, 14.1; ESI HRMS *m*/*z* calcd for $C_{22}H_{23}NO_5S$ $(M+Na)^+$, 436.1195, found 436.1173.

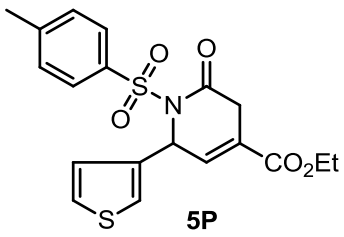


4P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (8 mg, 0.009 mmol) in toluene (2 mL) was added 1,4-bis(diphenylphosphino)butane (8 mg, 0.02 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **4** (20 mg, 0.044 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 2 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **4P** (16 mg, 80%) as a yellow solid.

・Enantioselective cyclization of dienamide **4** with (S)-BINAP

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (9.0 mg, 0.009 mmol) in toluene (2 mL) was added (S)-(-)-2,2'-bis(diphenylphosphino)-1,1'-binaphtyl (12 mg, 0.019 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **4** (21 mg, 0.047 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **4P** (15 mg, 71%) as a yellow solid. The enantiomeric excess was determined to be 80% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 3/1, flow rate = 0.5 mL/min., tR(minor) = 27.7 min, tR(major) = 32.7 min) : R_f 0.71 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3058, 2926, 1701, 1597, 1509, 1356, 1173, 1084, 906, 814; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.82 (m, 1H), 7.81-7.75 (m, 2H), 7.71 (s, 1H), 7.56-7.50 (m, 2H) 7.37 (d, 2H, *J* = 8.0 Hz), 7.19 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.09 (dd, 1H, *J* = 6.0, 2.8 Hz), 6.96 (d, 1H, *J* = 8.4 Hz), 6.34 (dt, 1H, *J* = 5.6, 2.0 Hz), 4.26-4.13 (m, 2H), 3.63 (dd,1H, *J* = 22.8, 2.0 Hz), 3.42 (dt, 1H, *J* = 22.4, 2.8 Hz), 2.29 (s, 3H), 1.26 (t, 3H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 164.4, 144.9, 135.2, 135.0, 134.7, 133.2, 133.1, 129.3, 128.8, 127.7, 126.9, 126.8, 126.7, 124.1, 123.9, 61.9, 61.5, 33.5, 21.5, 14.1; ESI HRMS *m*/*z* calcd for $C_{25}H_{23}NO_5S$ $(M+Na)^+$, 472.1195, found 472.1189.

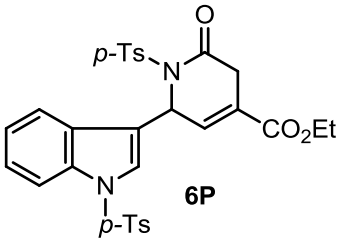


5P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (10 mg, 0.011 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (9 mg, 0.02 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **5** (30 mg, 0.074 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **5P** (25 mg, 83%) as a white solid.

・Enantioselective cyclization of dienamide **5** with (S)-BINAP

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (10 mg, 0.011 mmol) in toluene (2 mL) was added (S)-(-)-2,2'-bis(diphenylphosphino)-1,1'-binaphtyl (14 mg, 0.022 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **5** (30 mg, 0.074 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **5P** (24 mg, 80%) as a white solid. The enantiomeric excess was determined to be 65% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 3/1, flow rate = 0.5 mL/min., tR(minor) = 30.8 min, tR(major) = 34.3 min) : R_f 0.76 (67% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3119, 2990, 1719, 1678, 1539, 1393, 1285, 1051, 873; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, 2H, *J* = 8.4 Hz), 7.33-7.28 (m, 2H), 7.15 (d, 2H, *J* = 8.8 Hz), 7.09 (dd, 1H, *J* = 5.6, 2.8 Hz), 6.86 (dd, 1H, *J* = 5.2, 1.6 Hz), 6.32(dt, 1H, *J* = 6.0, 1.6 Hz), 4.28-4.17 (m, 2H), 3.55 (dd,1H, *J* = 22.0, 1.6 Hz), 3.27 (dt, 1H, *J* = 22.0, 2.8 Hz), 2.37 (s, 3H), 1.29 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 164.4, 144.9, 138.1, 135.4, 134.4, 129.1, 129.0, 127.3, 126.0, 124.7, 124.5, 61.5, 56.6, 33.4, 21.6, 14.1; ESI HRMS *m*/*z* calcd for $C_{19}H_{19}NO_5S_2$ $(M+Na)^+$, 428.0602, found 428.0616.

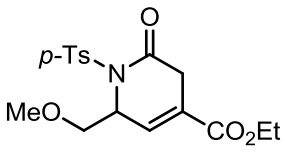


6P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (7 mg, 0.08 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (6 mg, 0.02 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **6** (30 mg, 0.051 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1.5 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **6P** (26 mg, 78%) as a yellow solid.

・Enantioselective cyclization of dienamide **6** with (S)-BINAP

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (7 mg, 0.007 mmol) in toluene (2 mL) was added (S)-(-)-2,2'-bis(diphenylphosphino)-1,1'-binaphtyl (9 mg, 0.015 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **6** (30 mg, 0.051 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 17% to 25% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **6P** (21 mg, 70%) as a yellow solid. The enantiomeric excess was determined to be 60% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 2/1, flow rate = 0.5 mL/min., tR(minor) = 29.5 min, tR(major) = 46.6 min) : R_f 0.63 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 2986, 2928, 1693, 1597, 1447, 1372, 1172, 1092, 905, 814; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, 2H, *J* = 8.4 Hz), 7.82 (d, 2H, *J* = 8.8 Hz), 7.64 (s, 1H), 7.40 (d, 2H, *J* = 8.4 Hz), 7.34-7.27 (m, 2H) 7.24 (d, 2H, *J* = 8.8 Hz), 7.20-7.08 (m, 2H), 7.05 (dd, 1H, *J* = 5.2, 2.8 Hz), 6.88 (d, 1H, *J* = 8.4 Hz), 6.42 (dt, 1H, *J* = 5.2, 2.4 Hz), 4.25-4.12 (m, 2H), 3.59 (dd,1H, *J* = 21.6, 2.0 Hz), 3.38 (dt, 1H, *J* = 22.4, 2.8 Hz), 2.31 (s, 3H), 2.28 (s, 3H), 1.26 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 164.2, 145.3, 144.9, 135.1, 135.0, 134.8, 133.1, 130.1, 128.9, 128.8, 127.5, 127.1, 125.5, 125.2, 124.9, 123.7, 119.0, 118.9, 113.9, 61.5, 54.7, 33.4, 21.5, 14.1; ESI HRMS *m*/*z* calcd for $C_{30}H_{28}N_2O_7S_2$ $(M+Na)^+$, 615.1236, found 615.1208.

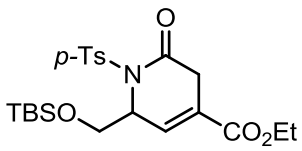


7P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (9 mg, 0.01 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (8 mg, 0.02 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **7** (18 mg, 0.049 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9% to 11% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **7P** (13 mg, 72%) as a yellow solid.

・Enantioselective cyclization of dienamide **7** with (S)-BINAP

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (14 mg, 0.015 mmol) in toluene (2 mL) was added (S)-(-)-2,2'-bis(diphenylphosphino)-1,1'-binaphtyl (19 mg, 0.030 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **7** (28 mg, 0.076 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude products. Column chromatography on silica gel (from 9% to 11% ethyl acetate in hexane) gave (*6S*)-*N*-sulfonyl-2-piperidinone **7P** (21 mg, 85%) as a yellow solid. The enantiomeric excess was determined to be 73% by chiral HPLC analysis (Daicel Chiralcel OD-H column, hexane/2-propanol = 3/1, flow rate = 0.5 mL/min., tR(minor) = 21.9 min, tR(major) = 23.4 min) : R_f 0.66 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 3023, 2930, 1713, 1597, 1356, 1258, 1086, 901, 814; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, 2H, *J* = 8.4 Hz), 7.31 (d, 2H, *J* = 8.0 Hz), 7.03-6.97 (m, 1H), 5.27-5.21 (m, 1H), 4.21 (q, 2H, *J* = 7.6 Hz), 3.82 (dd,1H, *J* = 8.8, 4.4 Hz), 3.68 (dd, 1H, *J* = 10.4, 2.0 Hz), 3.31 (s, 3H), 3.29-3.25 (m, 2H), 2.43 (s, 3H), 1.30 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2, 164.3, 145.0, 135.9, 132.9, 129.3, 129.0, 128.5, 74.0, 61.3, 59.4, 57.4, 34.2, 21.7, 14.1; ESI HRMS *m*/*z* calcd for $C_{17}H_{21}NO_6S$ $(M+Na)^+$, 390.0987, found 390.0991.



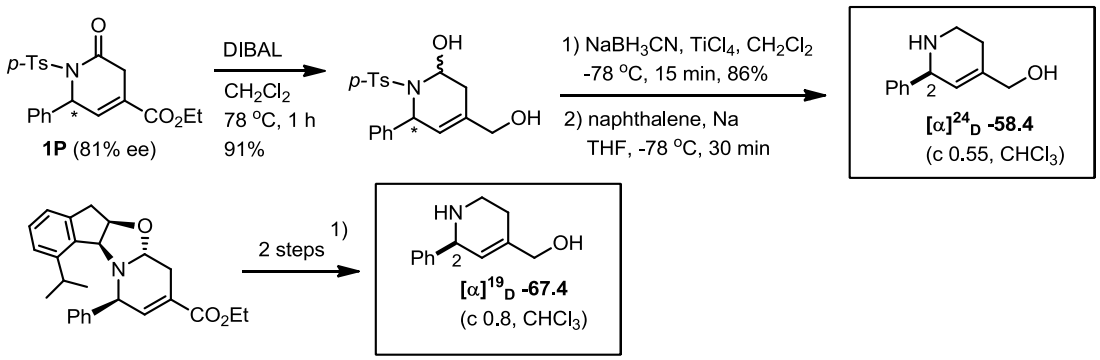
8P

To a suspension of the tris(dibenzylideneacetone)dipalladium(0) (12 mg, 0.013 mmol) in toluene (2 mL) was added 1,2-bis(diphenylphosphino)ethane (10 mg, 0.026 mmol) at room temperature. After the mixture was stirred at 100 ^{o}C for 30 min, *N*-sulfonyl dienamide **8** (30 mg, 0.064 mmol) in toluene (2 mL) was added. After the mixture was stirred at 100 ^{o}C for 1 h, H_2O was added and the resulting mixture was extracted with ethyl acetate. The organic layers were combined, washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo* to give the crude product. Column chromatography on silica gel (from 9% to 11% ethyl acetate in hexane) gave *N*-sulfonyl-2-piperidinone **8P** (19 mg, 63%) as a yellow solid: R_f 0.80 (50% ethyl acetate in hexane); IR (KBr disk, cm^{-1}) 2930, 2859, 1719, 1701, 1597, 1358, 1256, 1171, 1084, 895, 839; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, 2H, *J* = 8.4 Hz), 7.30 (d, 2H, *J* = 8.0 Hz), 6.97 (dd, 1H, *J* = 6.0, 2.8 Hz), 5.21 (ddd, 1H, *J* = 5.6,

4.0, 2.4 Hz), 4.23 (q, 2H, *J* = 7.2 Hz), 4.12 (dd,1H, *J* = 10.4, 3.6 Hz), 3.84 (dd, 1H, *J* = 10.8, 2.0 Hz), 3.29 (dd,1H, *J* = 22.0, 1.2 Hz), 3.21 (dt, 1H, *J* = 21.6, 2.4 Hz), 2.42 (s, 3H), 1.28 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.0, 164.3, 144.9, 136.2, 133.2, 129.3, 128.8, 128.4, 65.0, 61.2, 59.0, 34.2, 25.6, 21.6, 18.1, 14.1, -5.7, -5.7; ESI HRMS *m*/*z* calcd for $C_{22}H_{33}NO_6SSi$ $(M+Na)^+$, 490.1696, found 490.1673.

(V) Determination of the absolute configuration of 6-phenyl-2-piperidinone **1P**

1P obtained from the reaction with (S)-BINAP (81% ee) was converted to the piperidine compound in 3 steps and then the optical rotation was compared with the identical product obtained from the known compound.[1)] The absolute configuration at C-6 position of **1P** was determined to be *6S*.



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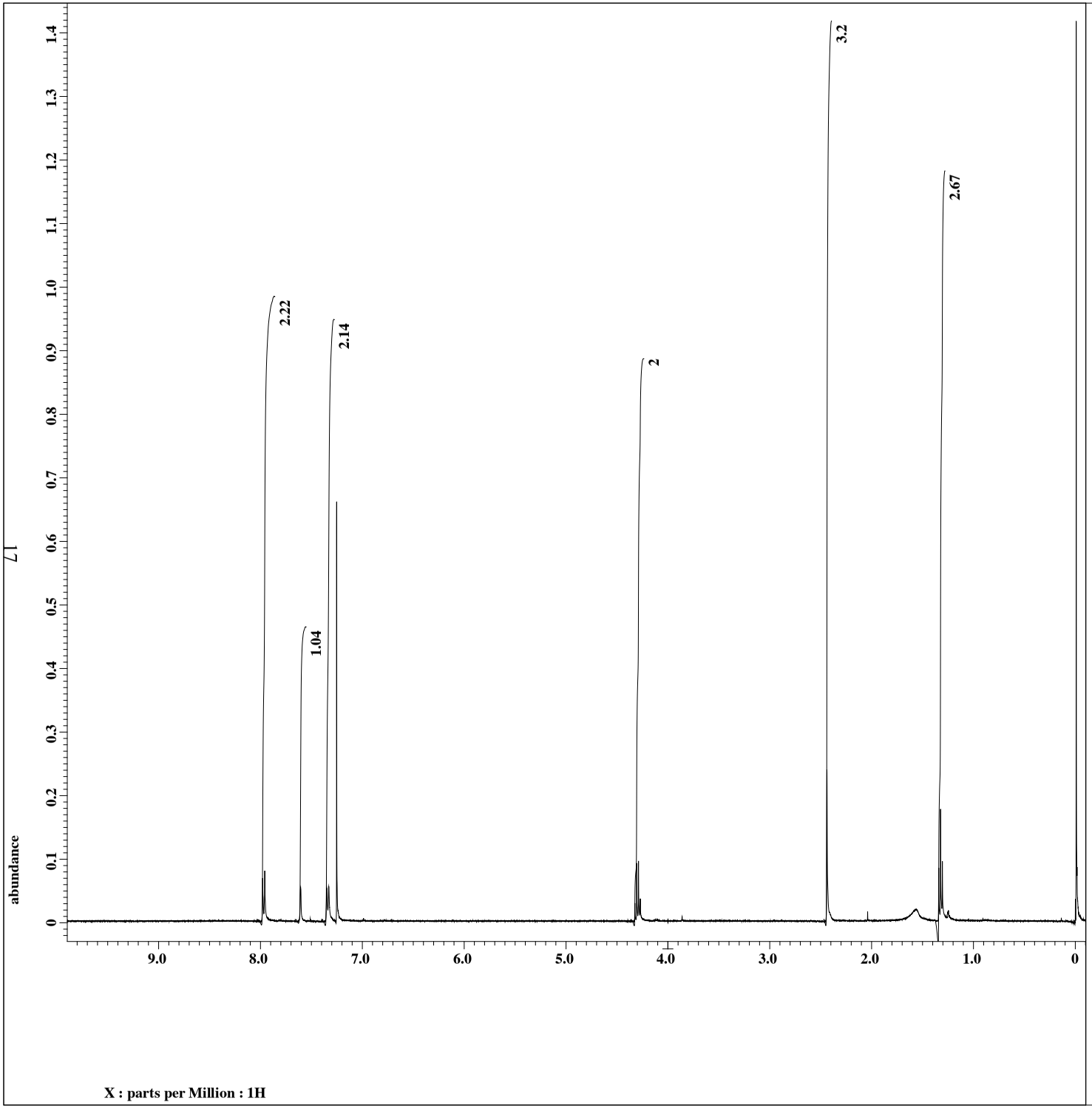
(1) This compound was easily prepared by the oxidation of the known aldehyde. See: Kobayashi, T.; Nakashima, M.; Hakogi, T.; Tanaka, K.; Katsumura, S. *Org. Lett.* **2006**, *8*, 3809-3812.

(2) All vinylstannanes are known compounds. See:(a) Kikukawa, K.; Umekawa, H.; Wada, F.; Matsuda, T. *Chem. Lett.* **1988**, pp881-884. (b) Parkinson, C. J.; Stoermer, M. J. *J. Organomet. Chem.* **1996**, *507*, 207-214. (c) Sakaguchi, T.; Kobayashi, T.; Hatano, S.; Tsuchikawa, H.; Fukase, K.; Tanaka, K.; Katsumura, S. *Chem. Asian J.* **2009**, *4*, 1573-1577. All vinylstannanes were prepared by either the coupling between the corresponding aryl halides and *trans*-1,2-bis(tributylstannyl)-ethene or the hydrostannation of the corresponding acetylene compounds.

2. Analytical Data

(I) ^{1}H and ^{13}C NMR Spectra



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Revision_time	= 15-NOV-2010 14:01:44
Current_time	= 15-NOV-2010 14:02:24
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Dim_title	= 1H
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Dimensions	= X
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Spectrometer	= DELTA2_NMR
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X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
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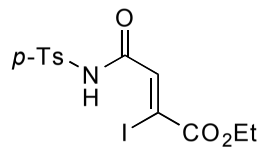
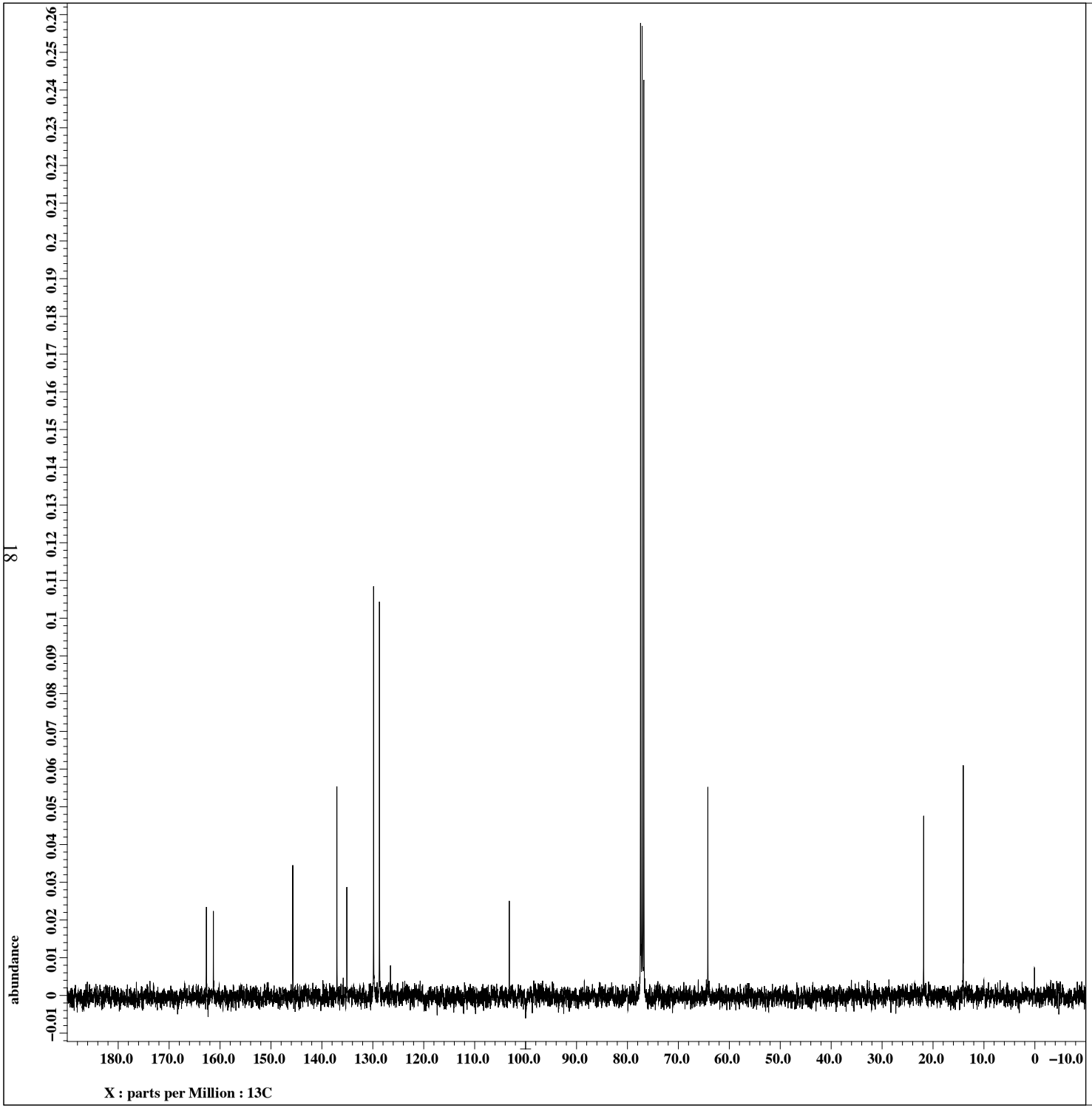
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 24.3[dC]



a

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Experiment        = single_pulse_dec
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Revision_time     = 15-NOV-2010 14:03:25
Current_time      = 15-NOV-2010 14:04:02

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Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

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X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
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X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
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Repetition_time   = 6.04333312[s]
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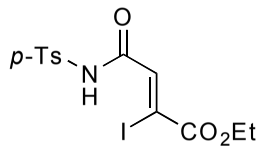

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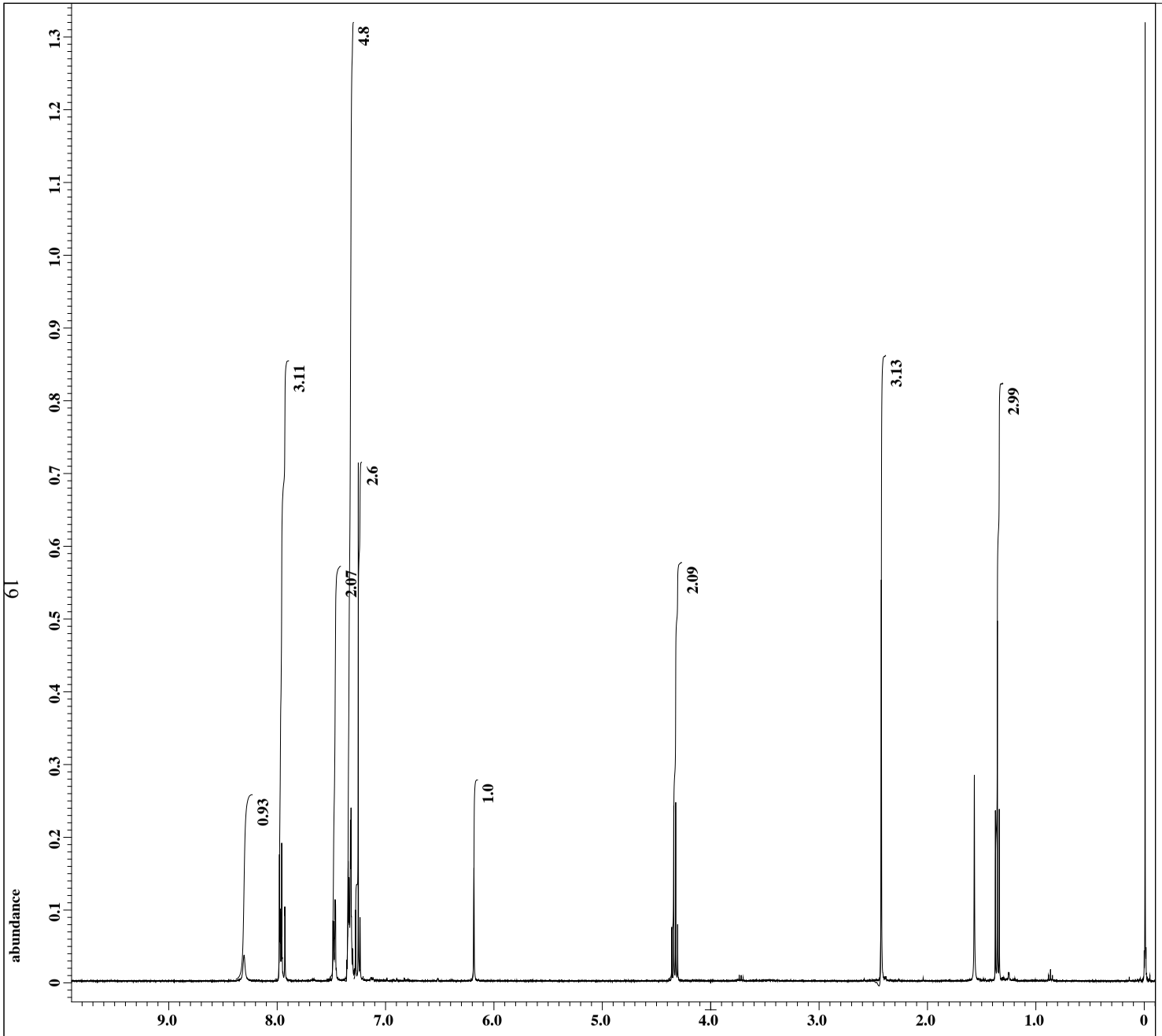

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Author	= delta
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Creation_time	= 14-JUN-2010 15:49:34
Revision_time	= 15-NOV-2010 14:07:02
Current_time	= 15-NOV-2010 14:09:39
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Dim_title	= 1H
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Spectrometer	= DELTA2_NMR
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X_acq_duration	= 4.36731904[s]
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X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
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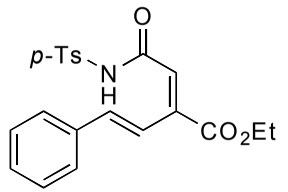
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 26[dC]



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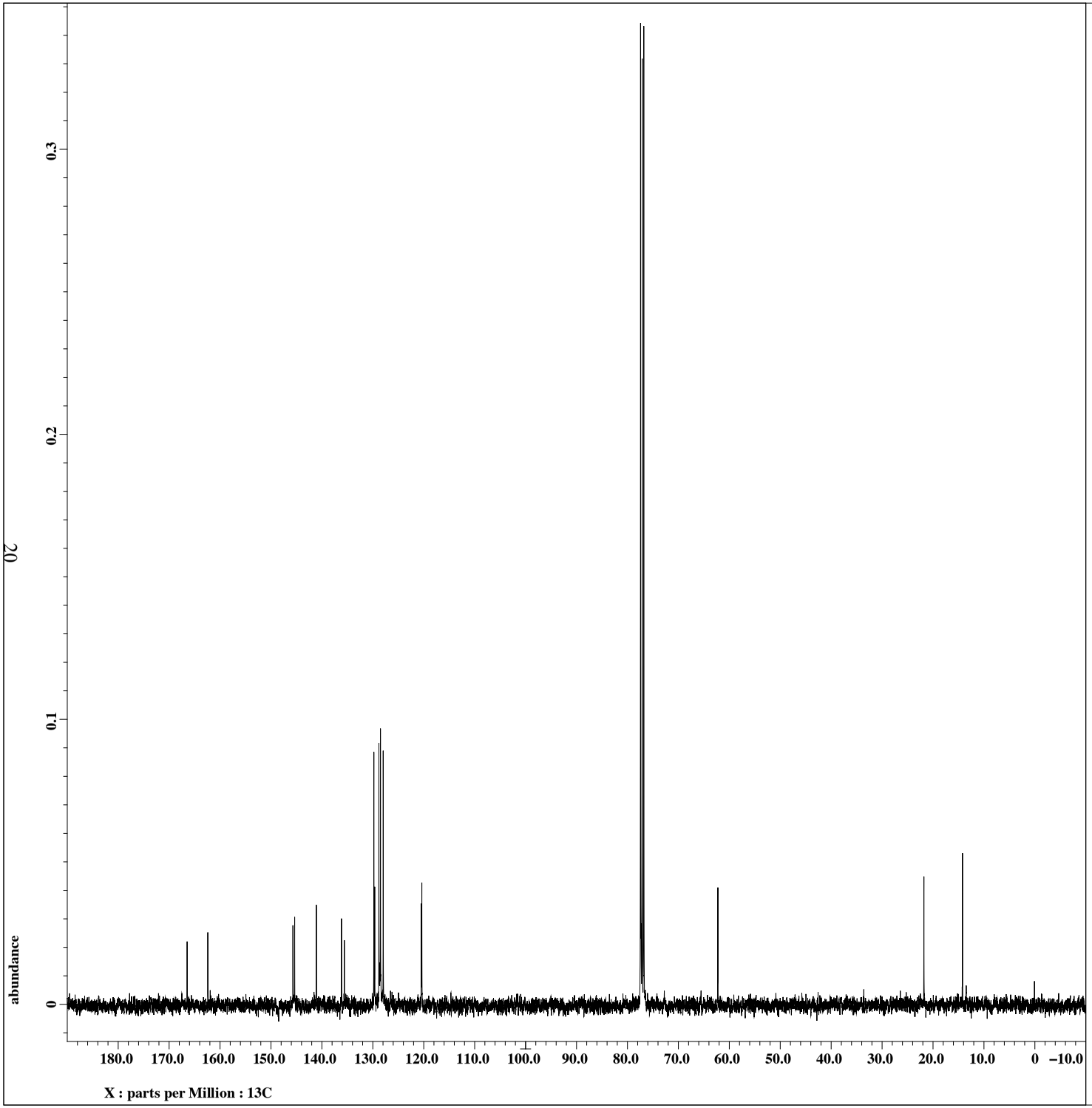


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Author            = delta
Experiment        = single_pulse_dec
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Revision_time     = 15-NOV-2010 14:10:36
Current_time      = 15-NOV-2010 14:10:53

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Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 183
Total_scans       = 183

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
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Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
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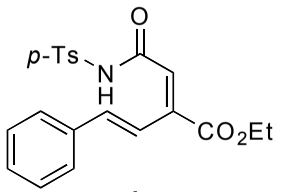
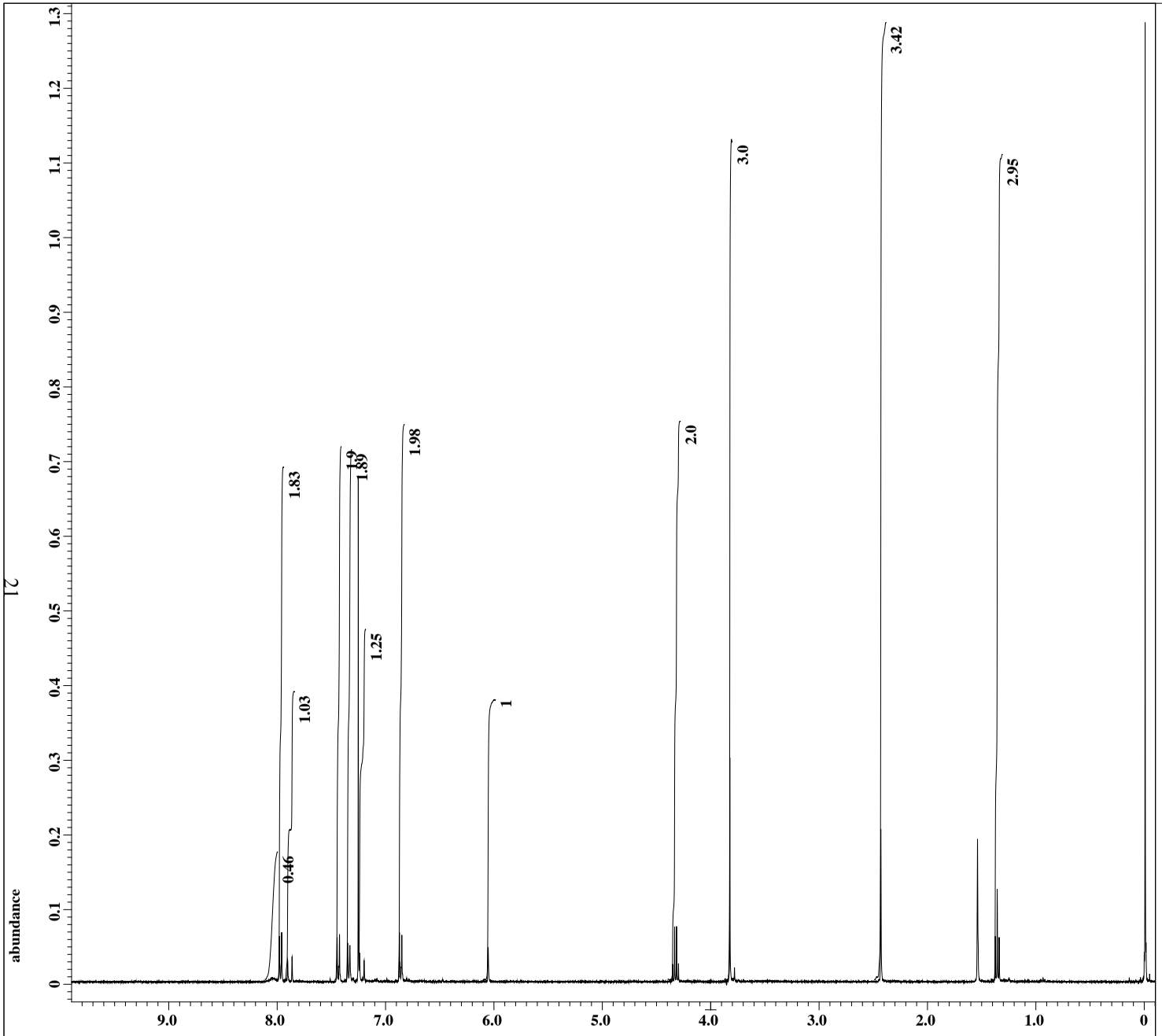


1



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Revision_time	= 15-NOV-2010 14:13:28
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Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
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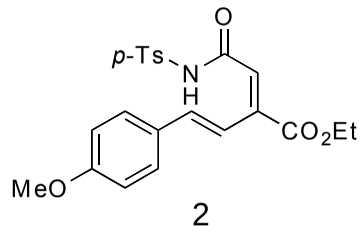
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 25.5[dC]



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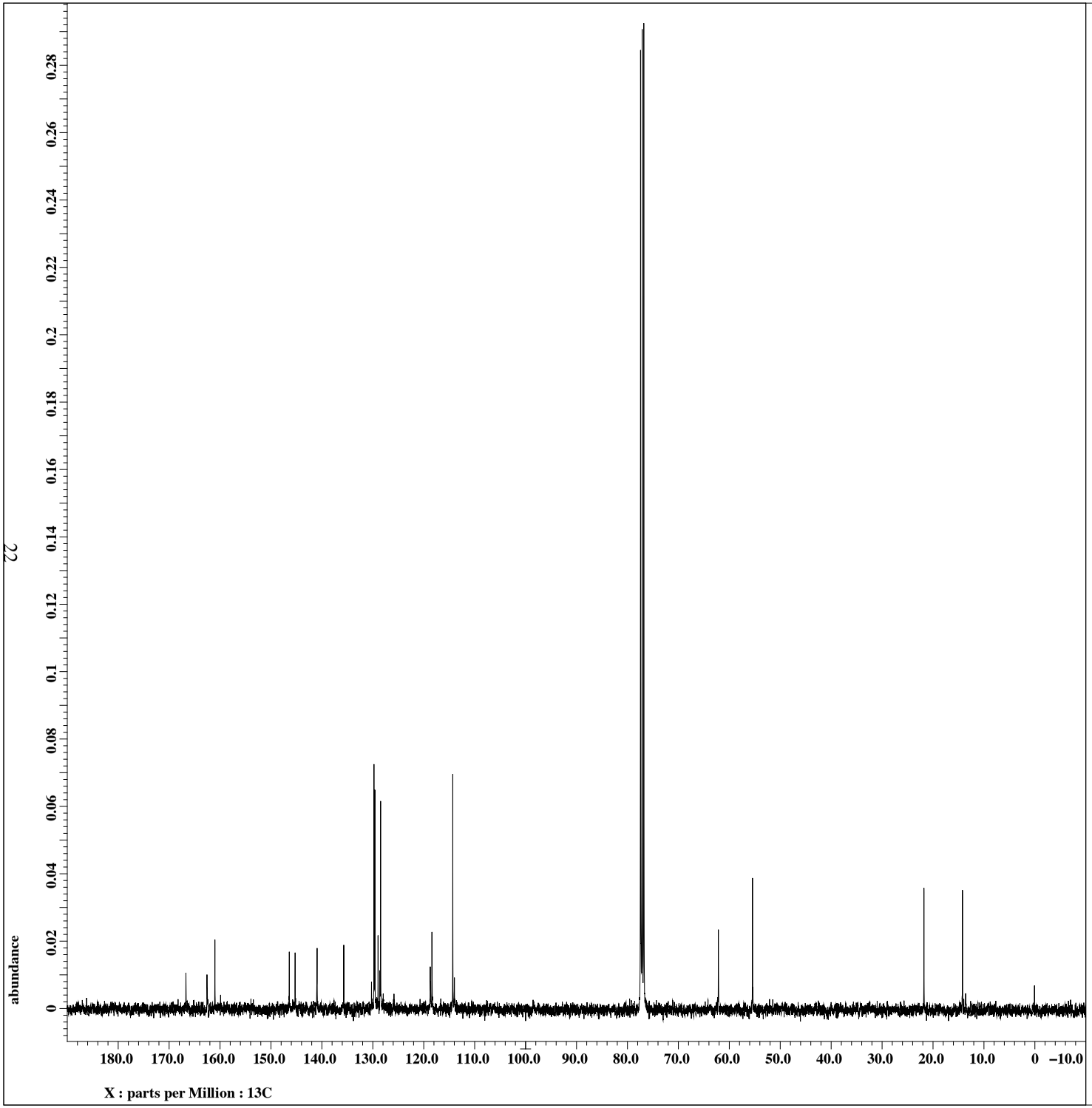

22

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Revision_time     = 15-NOV-2010 14:14:33
Current_time      = 15-NOV-2010 14:15:11

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Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
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X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 417
Total_scans       = 417

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.6[dC]
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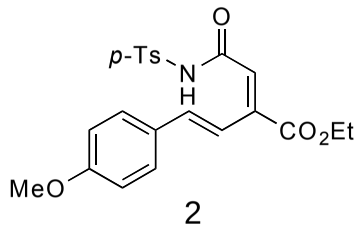

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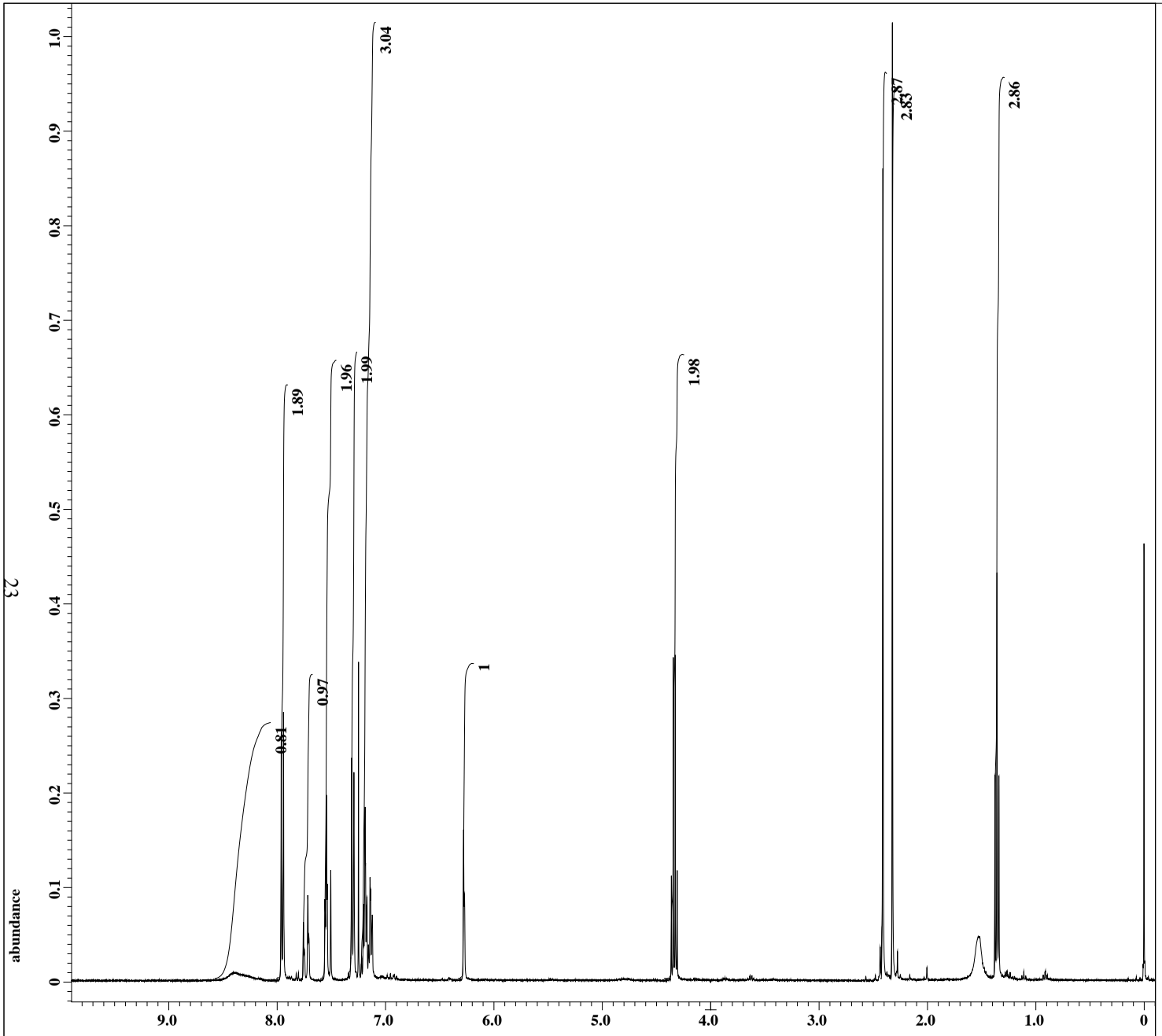

23

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Author	= delta
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Creation_time	= 5-JUL-2010 09:57:47
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Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
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X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
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Tri_mode	= Off
Dante_presat	= FALSE
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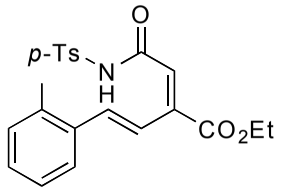
Temp_get	= 55[dC]



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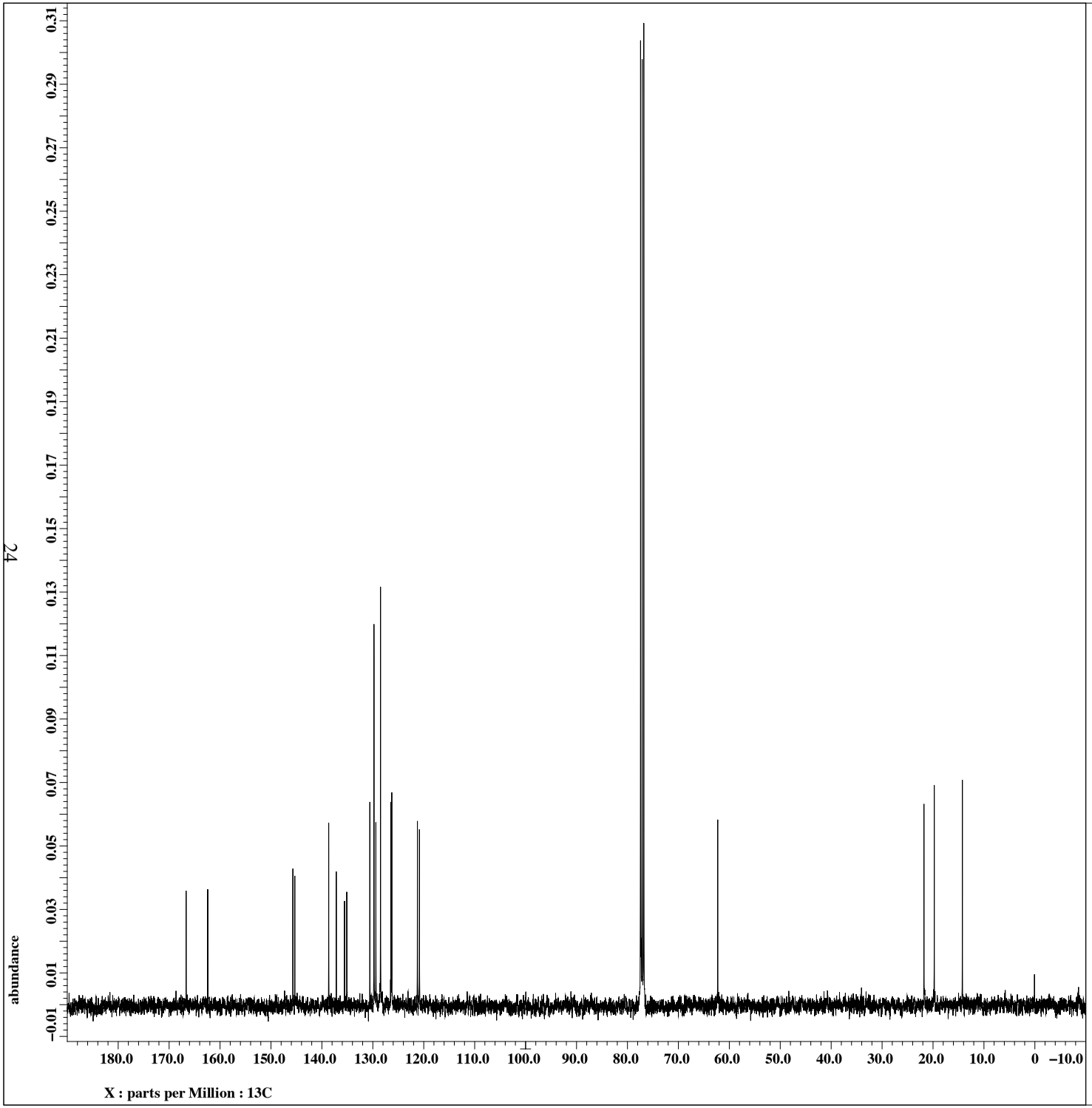

24

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Author	= delta
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Revision_time	= 15-NOV-2010 14:56:39
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Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 1.04333312[s]
X_domain	= 13C
X_freq	= 100.52530333[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95846665[Hz]
X_sweep	= 31.40703518[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 193
Total_scans	= 193
X_90_width	= 9.2[us]
X_acq_time	= 1.04333312[s]
X_angle	= 45[deg]
X_atn	= 6.6[dB]
X_pulse	= 4.6[us]
Irr_atn_dec	= 22.2[dB]
Irr_atn_noe	= 22.2[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 5[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 6.04333312[s]
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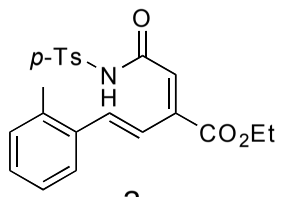

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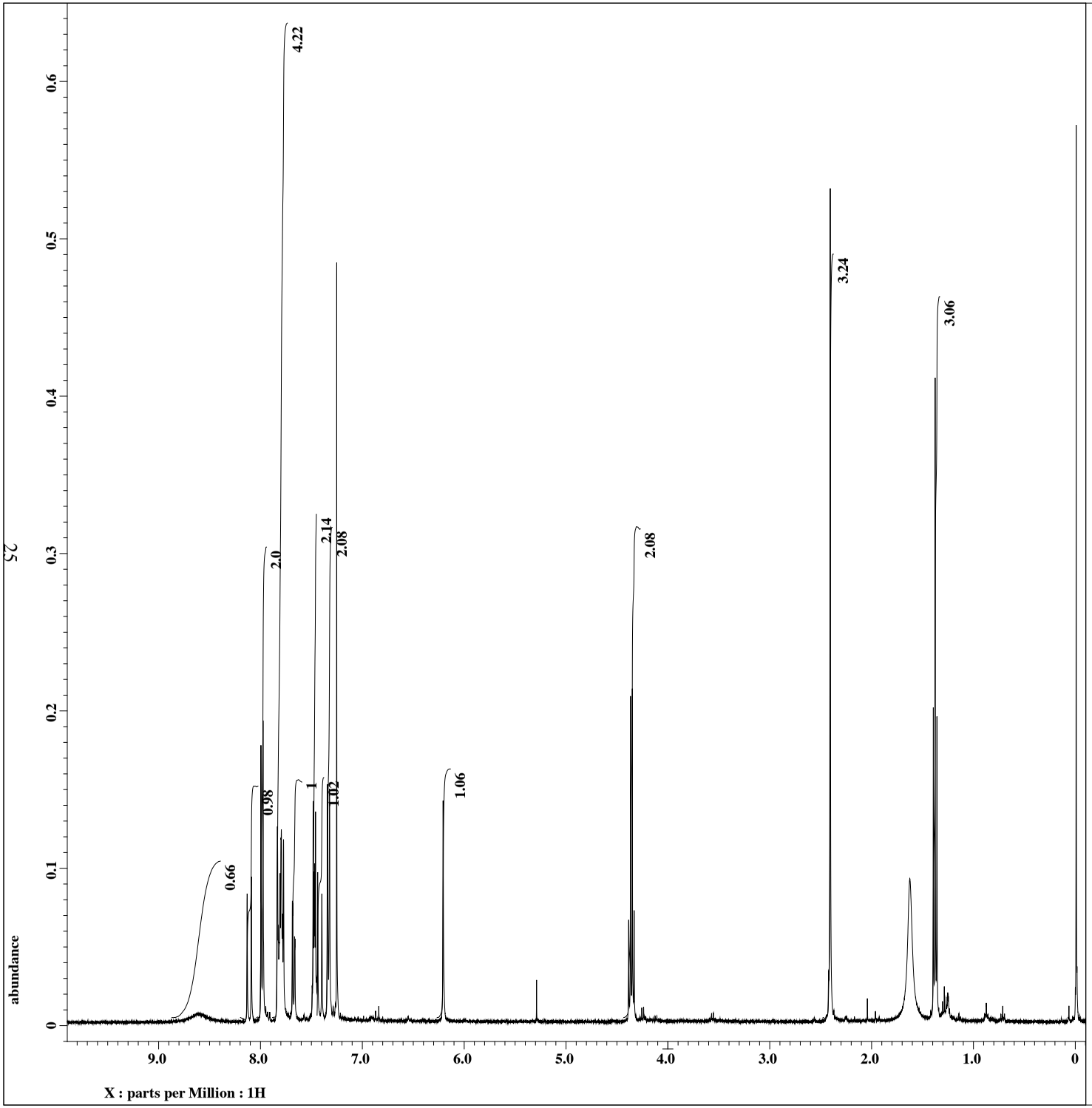


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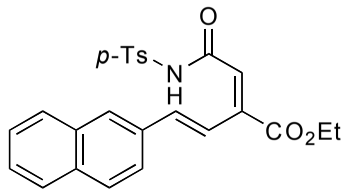
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  2-JUL-2010 16:39:19
Revision_time     = 15-NOV-2010 14:24:55
Current_time      = 15-NOV-2010 14:25:06

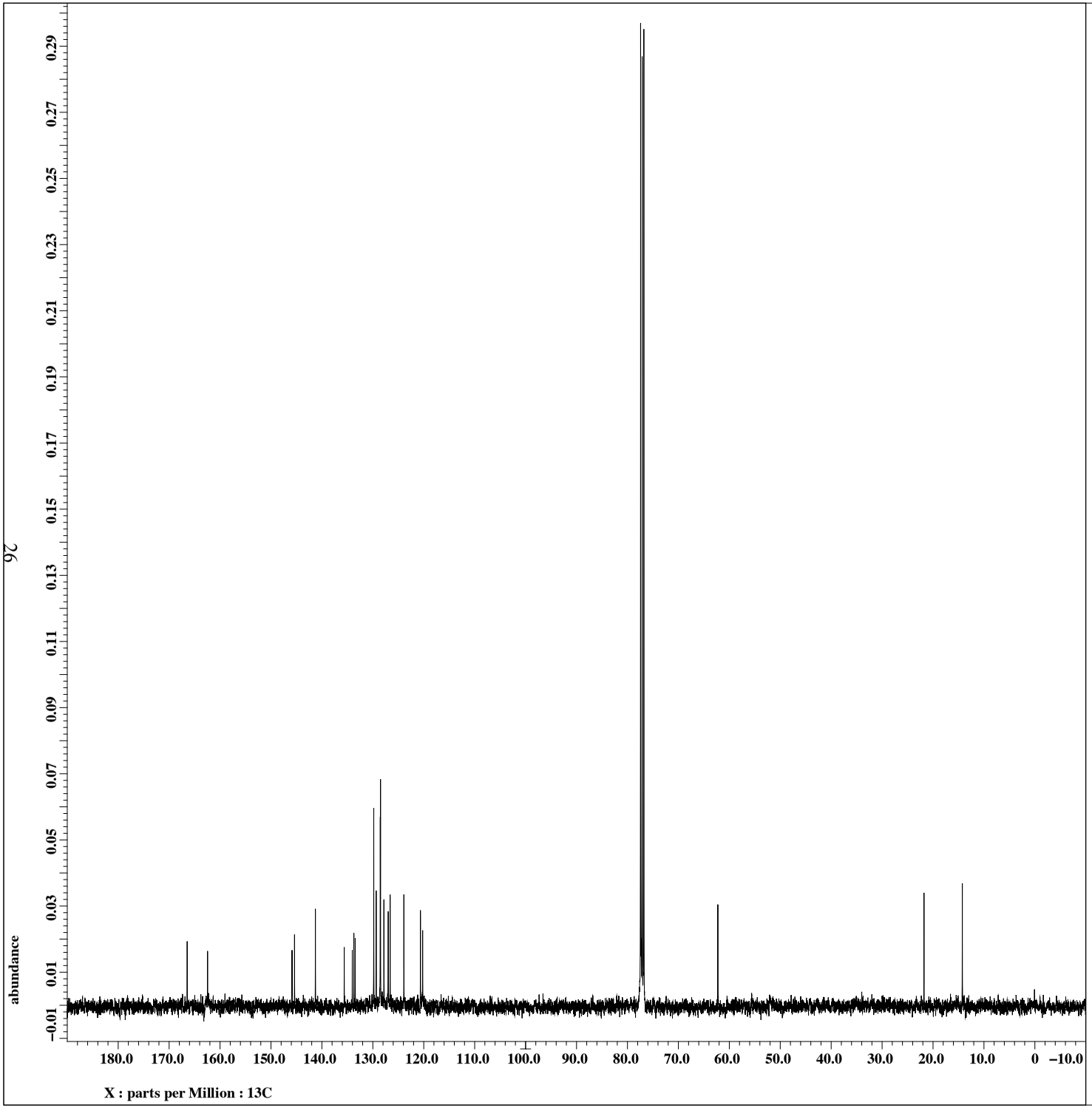
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.9[dC]
```



4

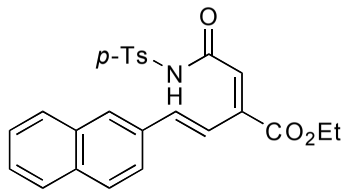



```
Filename          = 601 13C Stille (Sulfo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#649827
Solvent           = CHLOROFORM-D
Creation_time     =  2-JUL-2010 17:38:20
Revision_time     = 15-NOV-2010 14:26:01
Current_time      = 15-NOV-2010 14:26:14

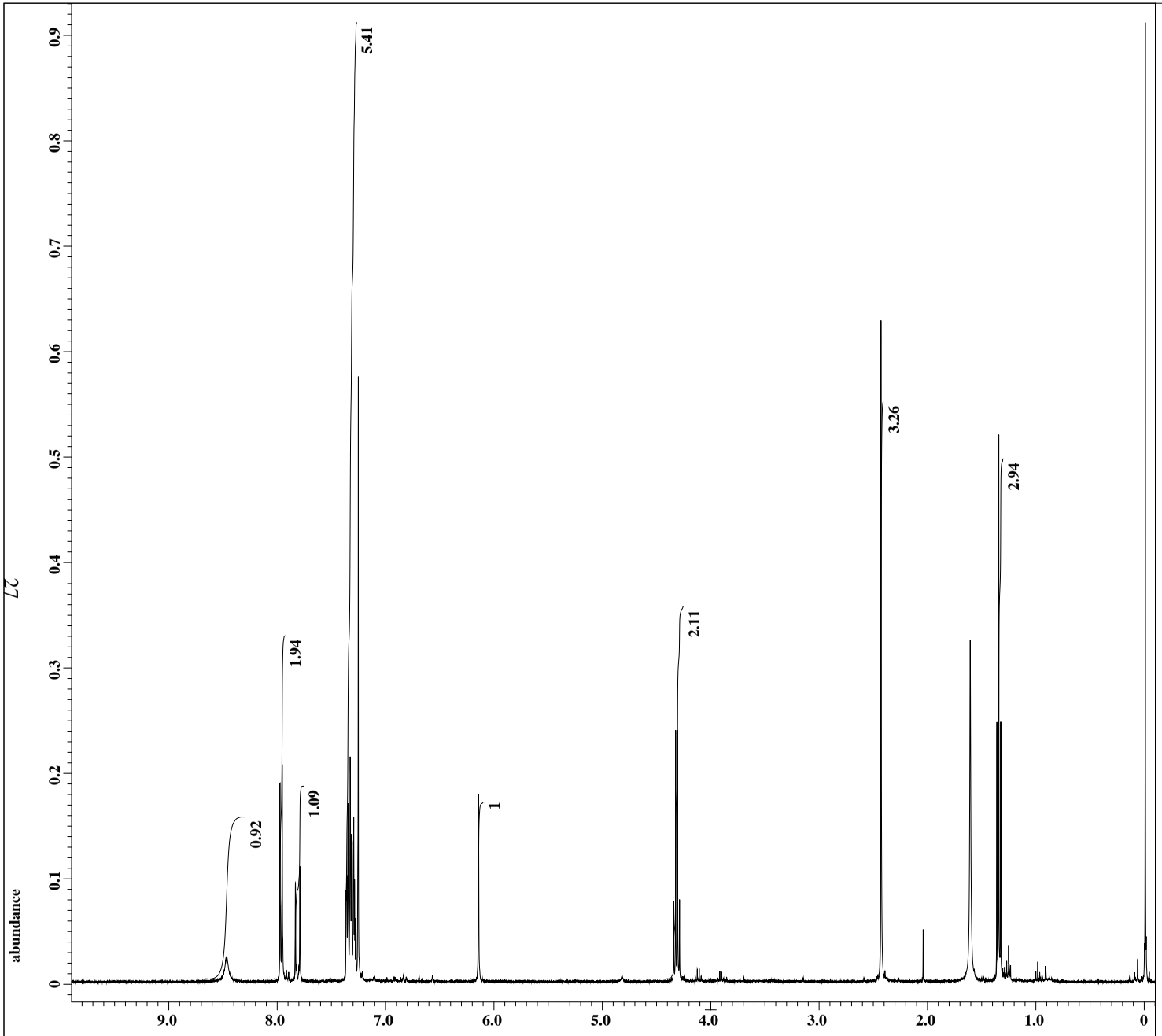
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 270
Total_scans       = 270

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 26[dC]
```



4

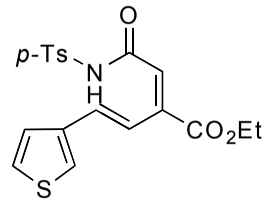



```
Filename          = data thiophen dienami
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 14:19:17
Revision_time     = 15-NOV-2010 14:18:37
Current_time      = 15-NOV-2010 14:18:50

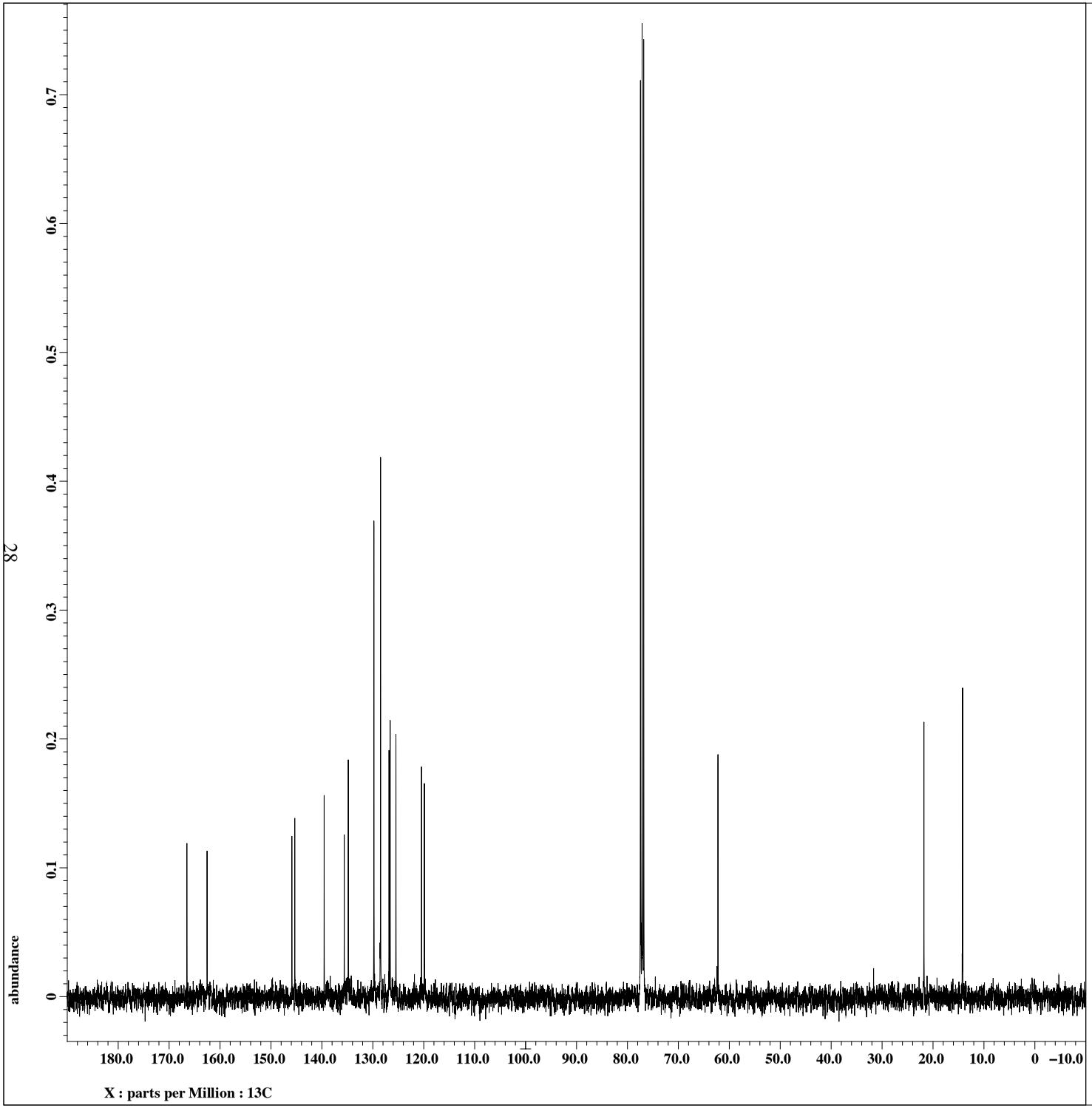
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.6[dC]
```



5



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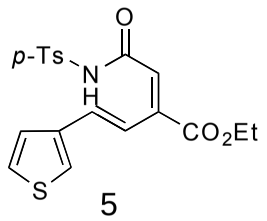
```
Filename          = data 13C thiophen-2.j
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#606224
Solvent           = CHLOROFORM-D
Creation_time     =  6-OCT-2010 16:59:57
Revision_time     = 15-NOV-2010 14:20:21
Current_time      = 15-NOV-2010 14:20:37

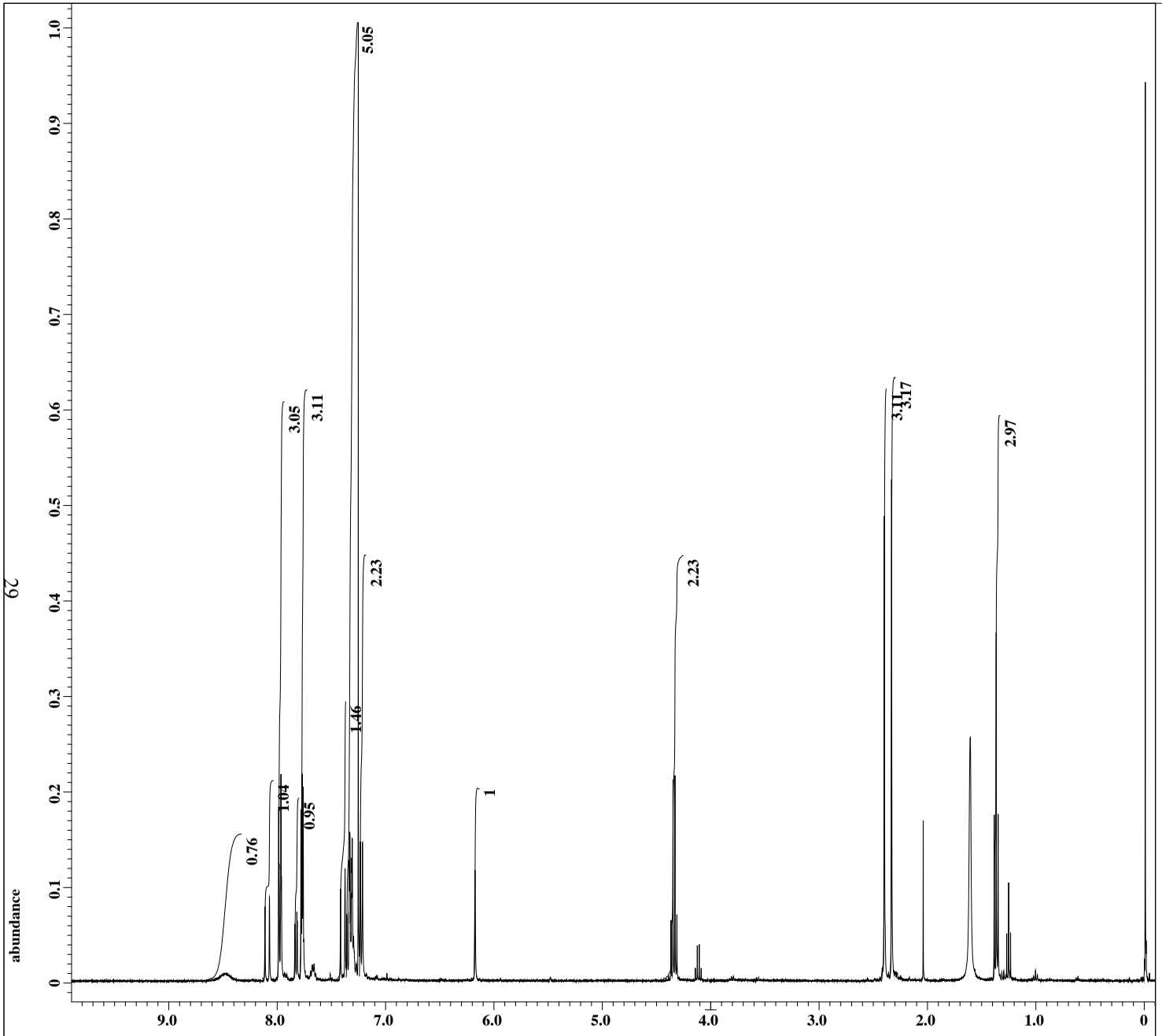
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 89
Total_scans       = 89

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 58
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 24.8[dC]
```



5

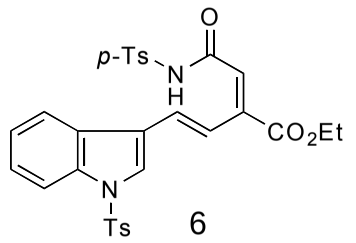



```
Filename          = 606 Stille (Sulfonami
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  9-JUL-2010 20:02:52
Revision_time     = 15-NOV-2010 14:30:05
Current_time      = 15-NOV-2010 14:30:17

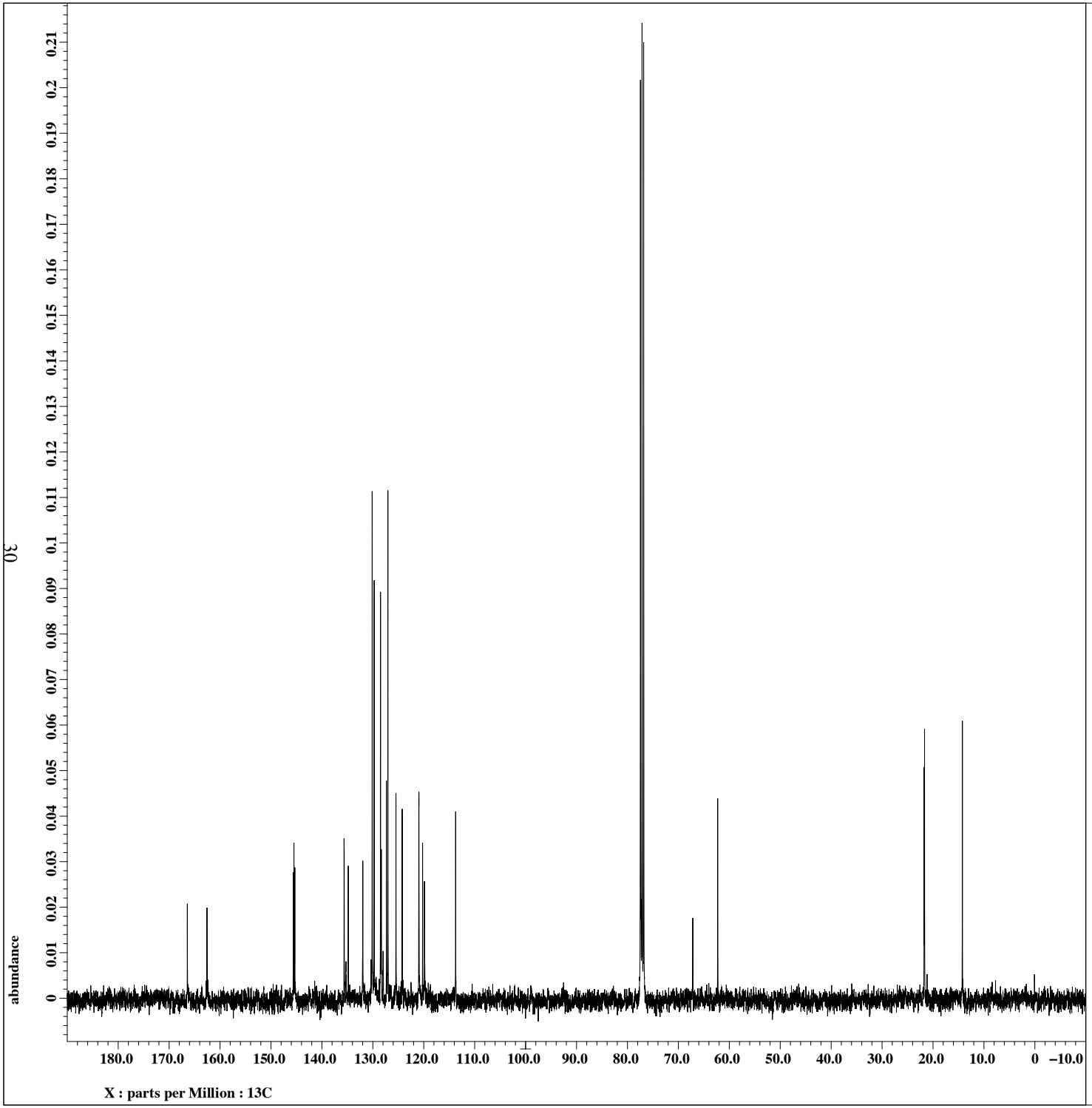
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.9[dC]
```



6



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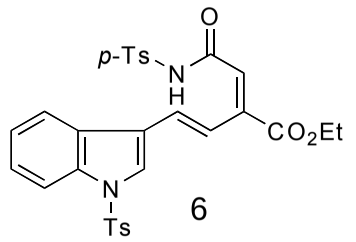
```
Filename          = 606 13C Stille (Sulfo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#507069
Solvent           = CHLOROFORM-D
Creation_time     = 10-JUL-2010 13:29:29
Revision_time     = 15-NOV-2010 14:30:47
Current_time      = 15-NOV-2010 14:30:59

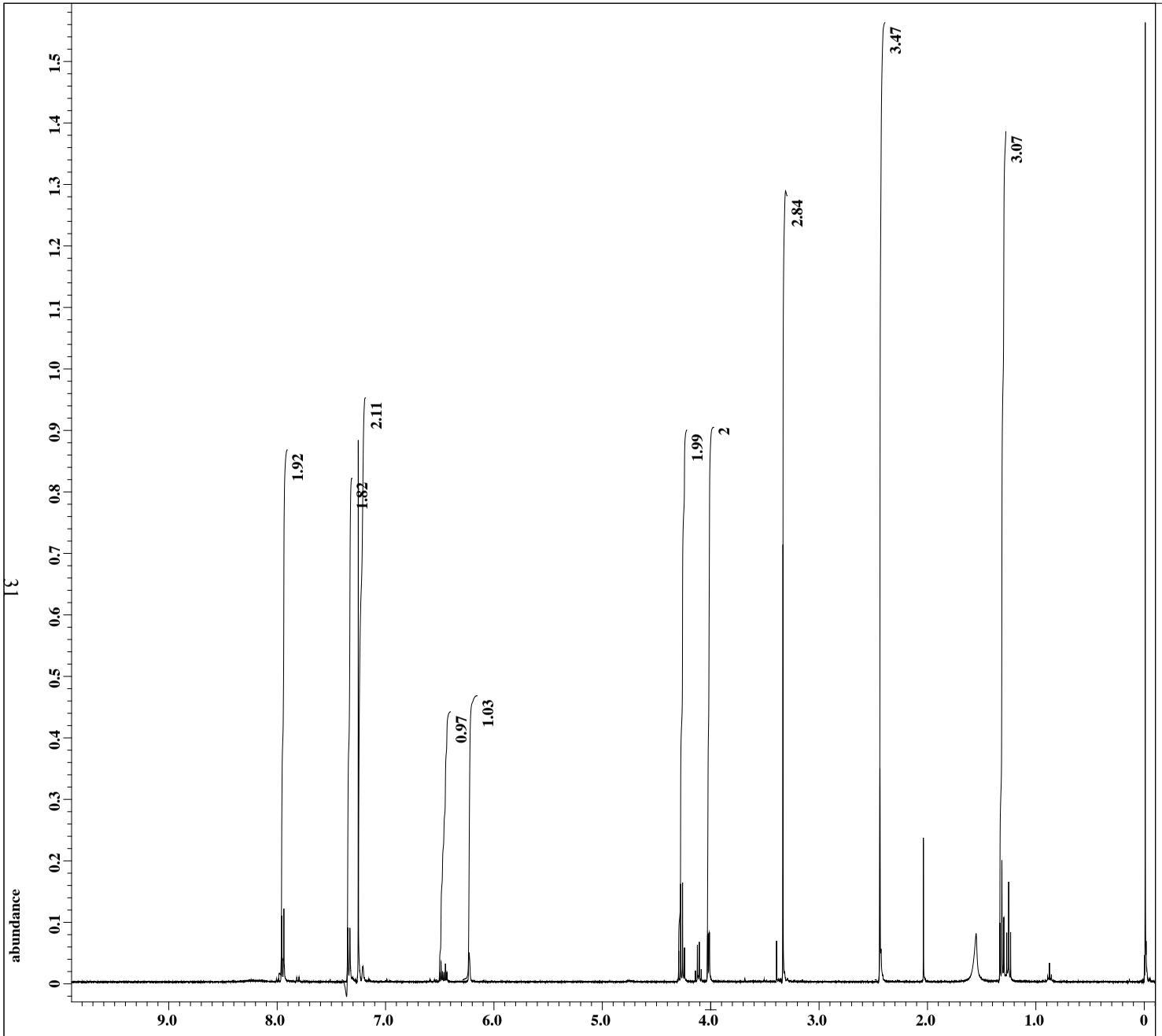
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 163
Total_scans       = 163

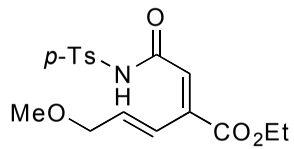
X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.2[dC]
```



6



Filename	= 503 Stille (Sulfonami
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 5-DEC-2009 15:40:04
Revision_time	= 15-NOV-2010 14:40:26
Current_time	= 15-NOV-2010 14:40:41
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 25.9[dC]



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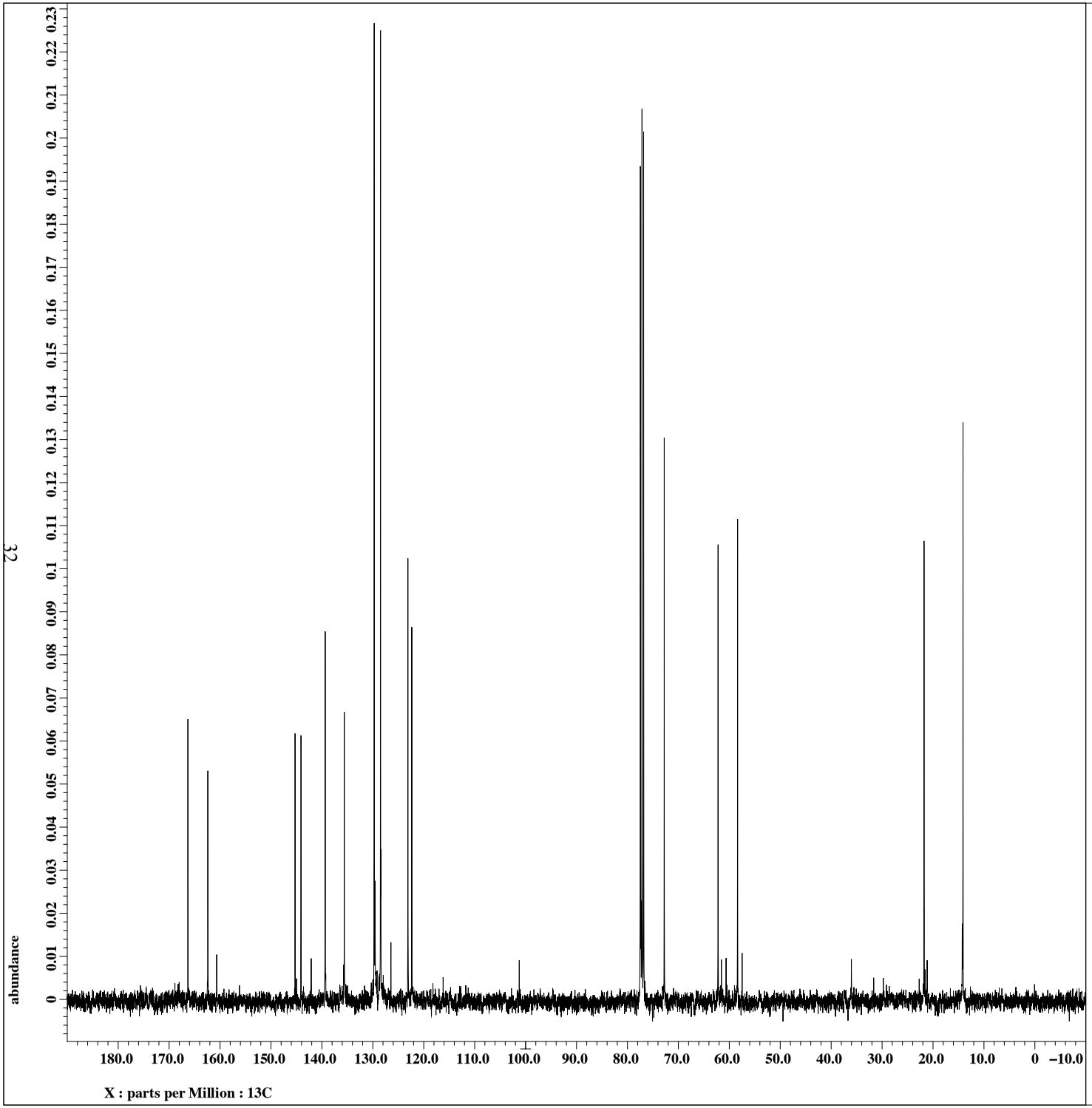

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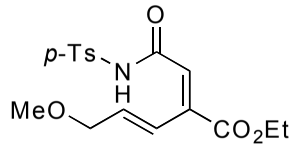
```
Filename          = 607 13C Stille (Sulfo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#518727
Solvent           = CHLOROFORM-D
Creation_time     = 12-JUL-2010 13:49:09
Revision_time     = 15-NOV-2010 14:41:11
Current_time      = 15-NOV-2010 14:41:25

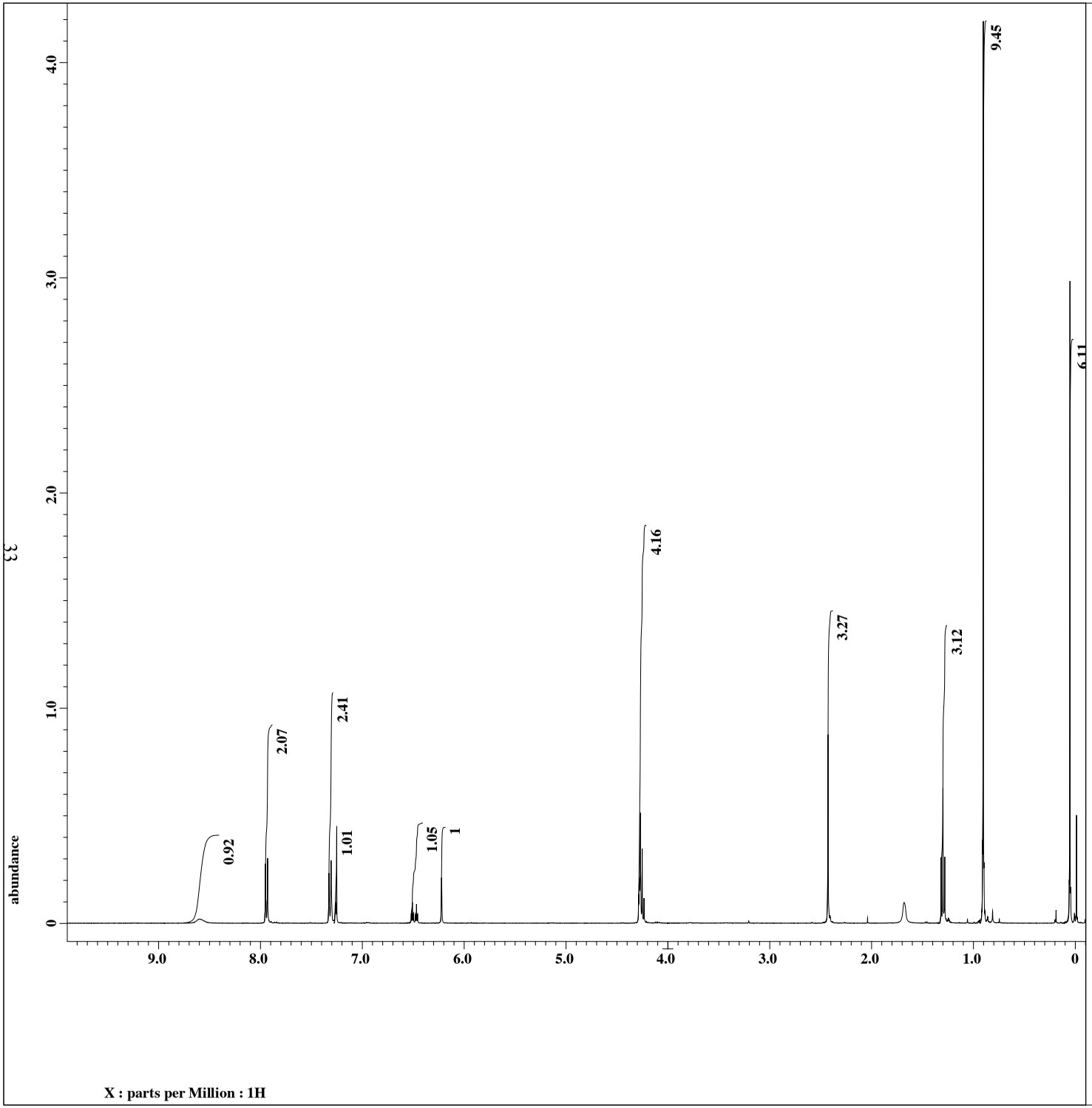
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 165
Total_scans       = 165

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.3[dC]
```



7

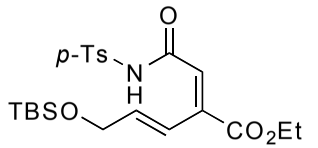



```
Filename          = 604 Stille (Sulfonami
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  6-JUL-2010 12:32:54
Revision_time     = 15-NOV-2010 14:36:10
Current_time      = 15-NOV-2010 14:36:39

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.8[dC]
```



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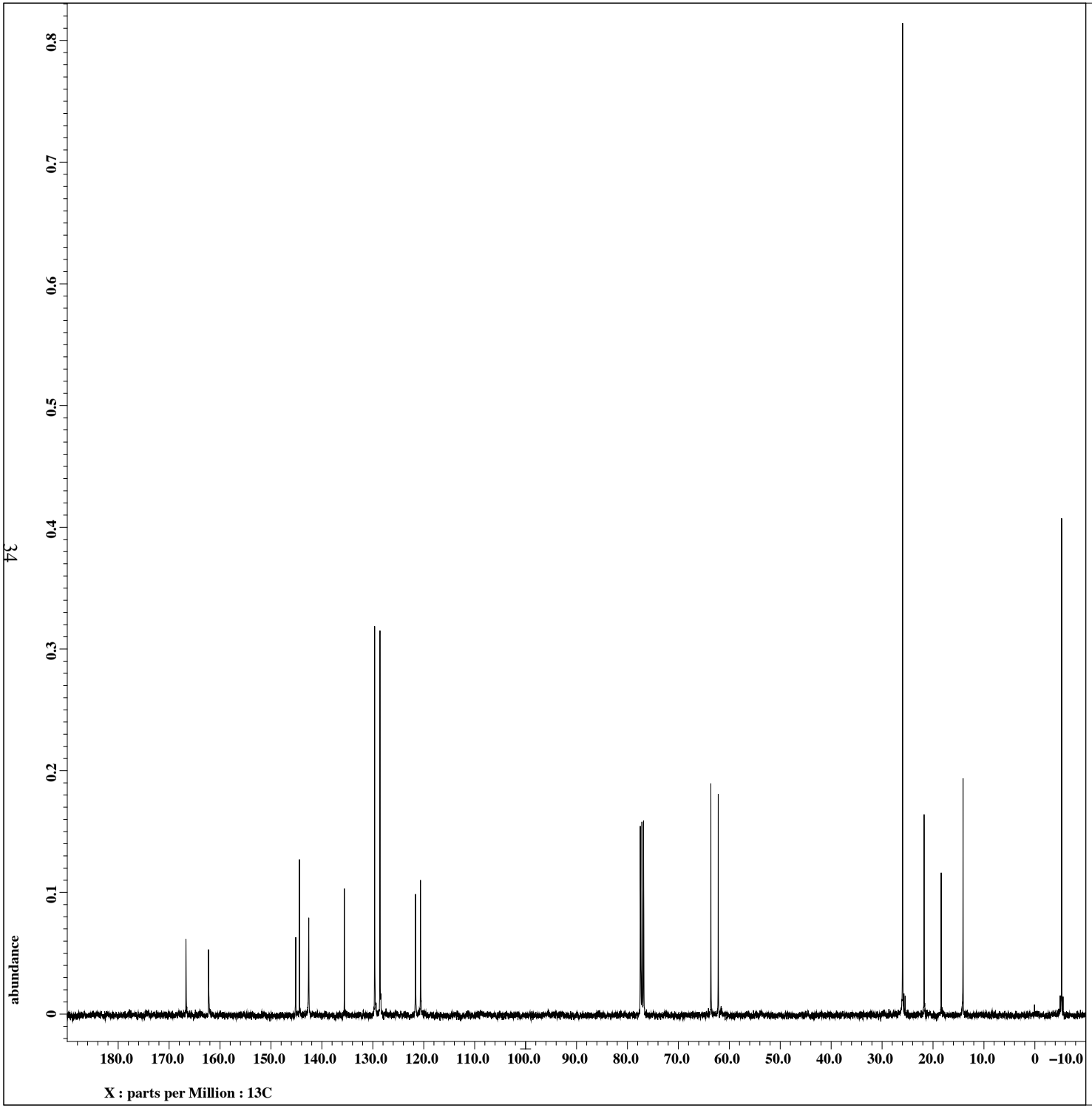
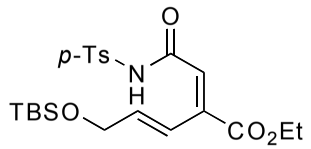



```
Filename          = 604 13C Stille (Sulfo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#491458
Solvent           = CHLOROFORM-D
Creation_time     =  6-JUL-2010 13:06:21
Revision_time     = 15-NOV-2010 14:37:12
Current_time      = 15-NOV-2010 14:37:26

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 193
Total_scans       = 193

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.4[dC]
```



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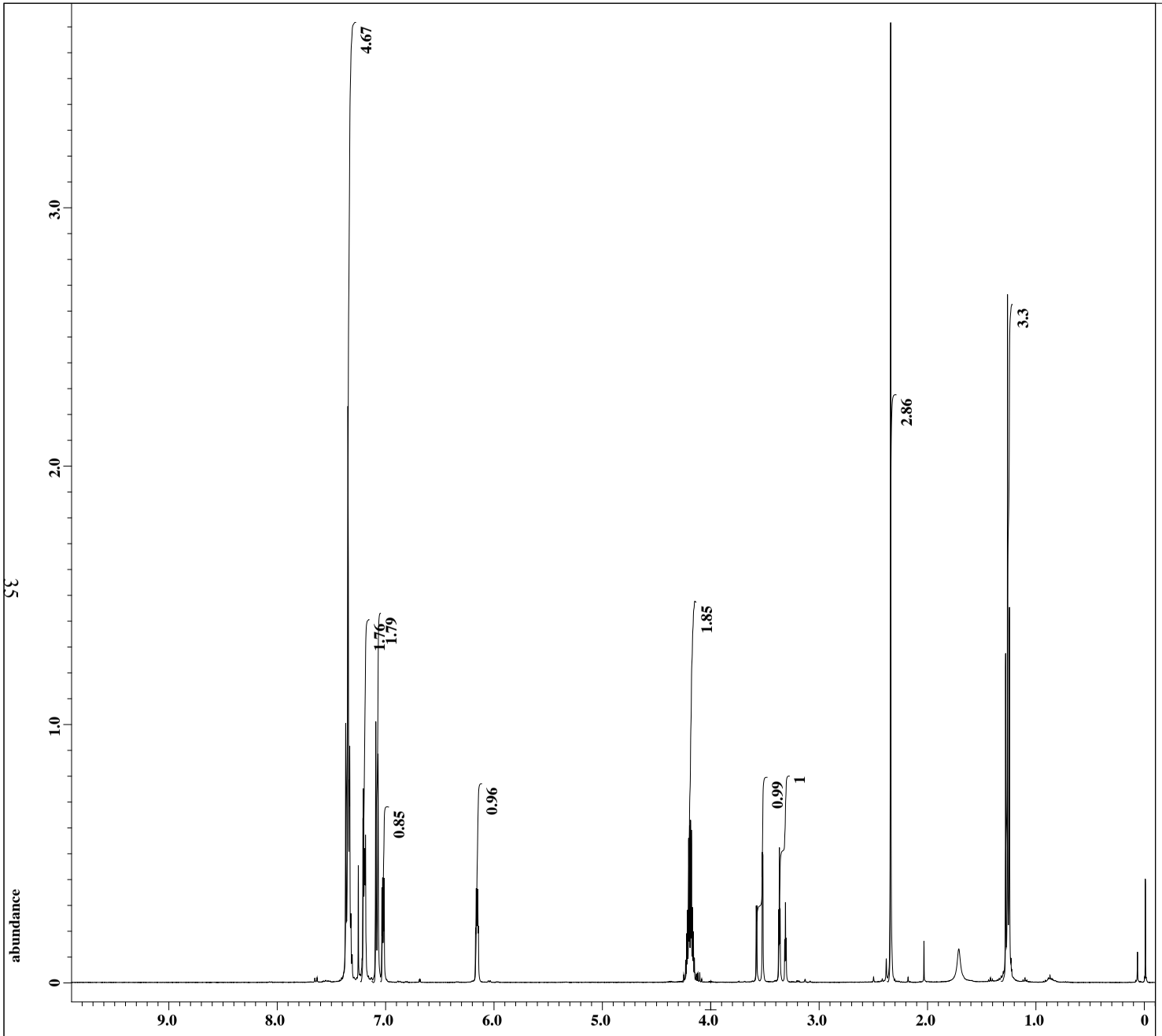
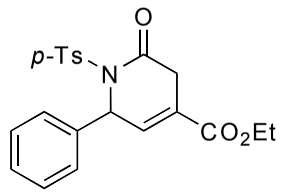
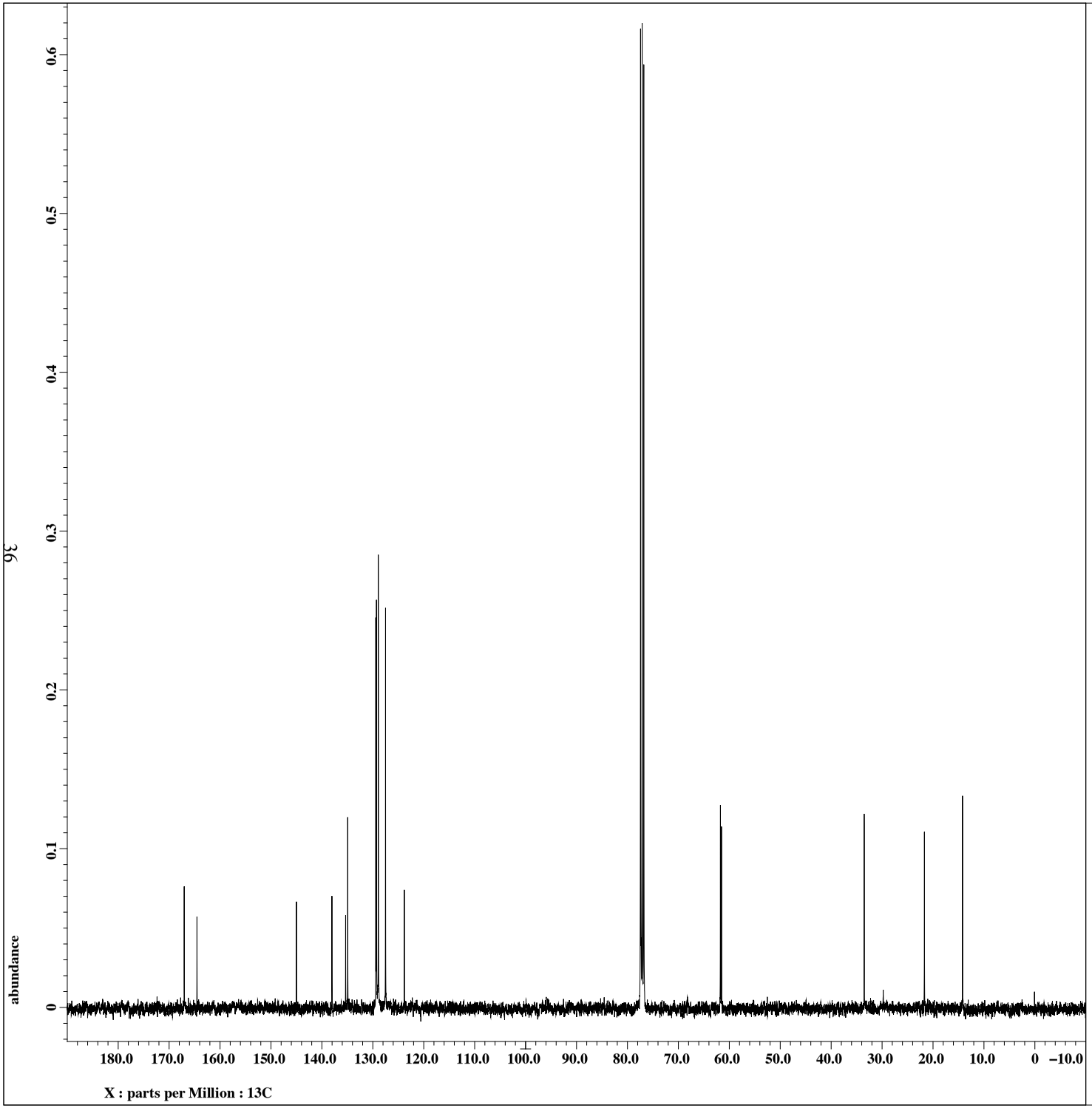


Filename	= V-97.F1-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 22-OCT-2008 13:41:36
Revision_time	= 15-NOV-2010 14:58:39
Current_time	= 15-NOV-2010 14:58:54
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 2.18365952[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45794685[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.45[us]
X_acq_time	= 2.18365952[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.225[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 1[s]
Repetition_time	= 3.18365952[s]
Temp_get	= 25.3[dC]



1P

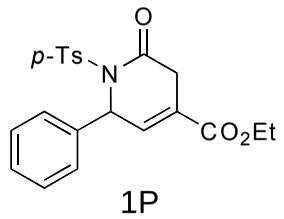



```
Filename          = data 13C Ph lactam-3.
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#749770
Solvent           = CHLOROFORM-D
Creation_time     =  7-OCT-2010 21:23:44
Revision_time     = 15-NOV-2010 14:59:24
Current_time      = 15-NOV-2010 14:59:41

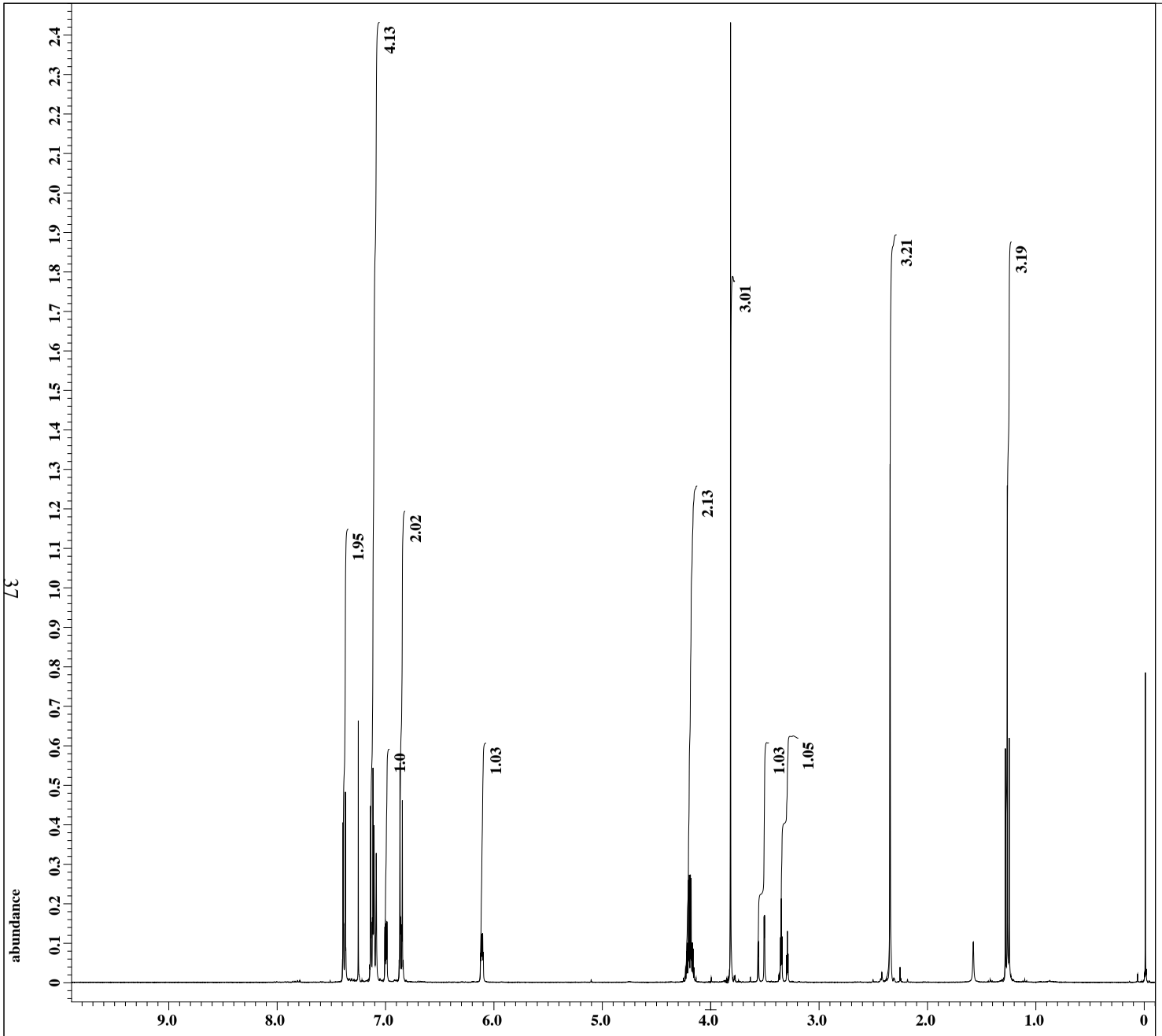
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 338
Total_scans       = 338

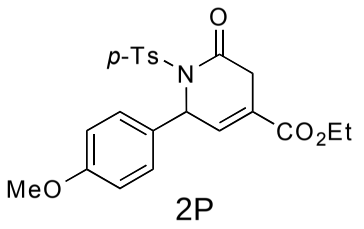
X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25[dC]
```



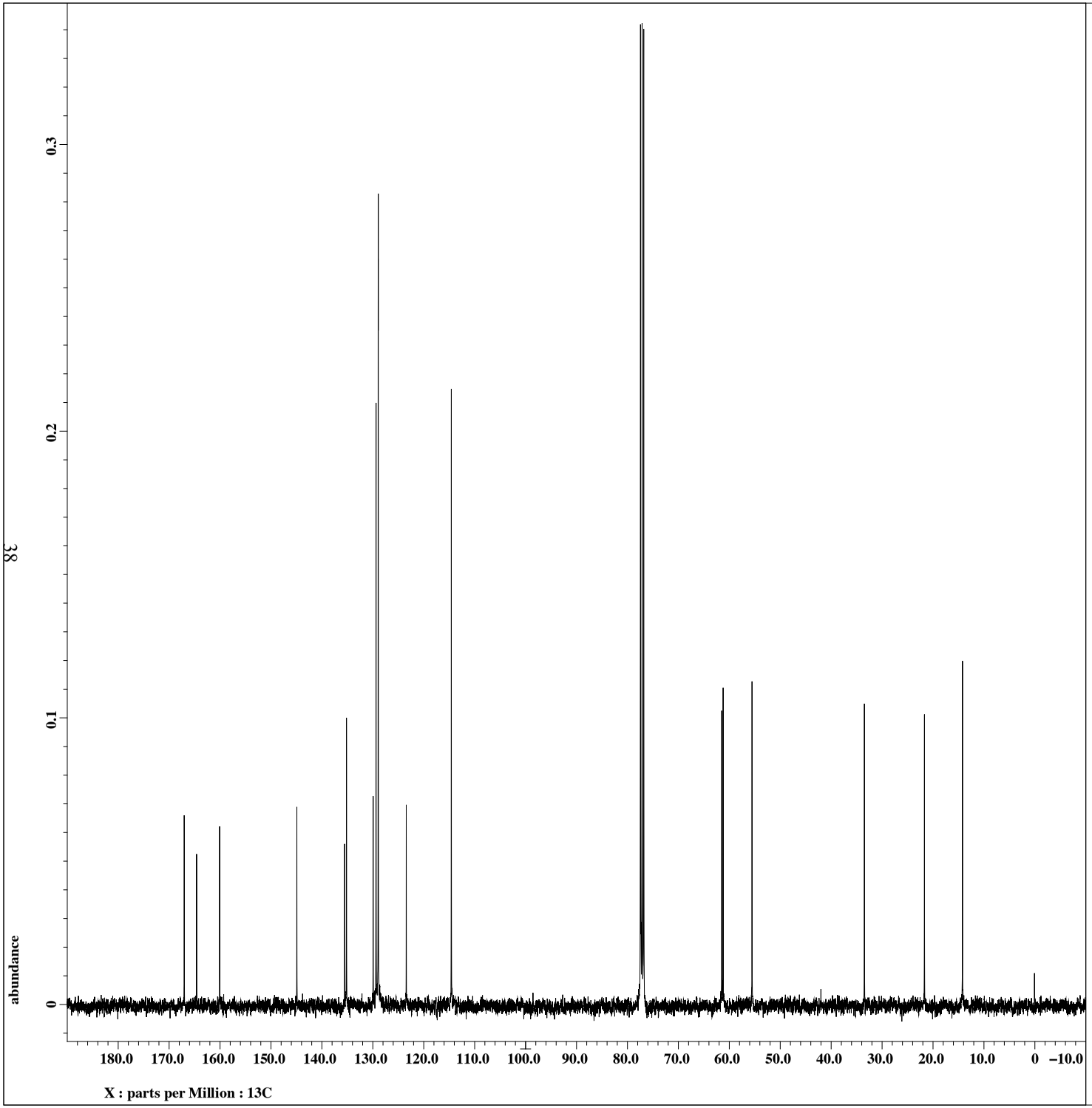
1P



Filename	= 591 cyclization(Sulfo
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 16-JUN-2010 18:03:42
Revision_time	= 15-NOV-2010 15:01:40
Current_time	= 15-NOV-2010 15:01:53
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 25.1[dC]



2P



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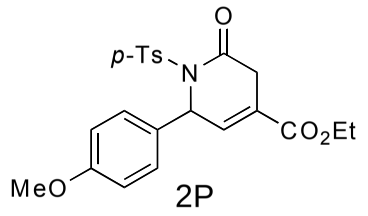
```
Filename          = 591 13C cyclization(S
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#688822
Solvent           = CHLOROFORM-D
Creation_time     = 16-JUN-2010 18:47:17
Revision_time     = 15-NOV-2010 15:02:25
Current_time      = 15-NOV-2010 15:02:36

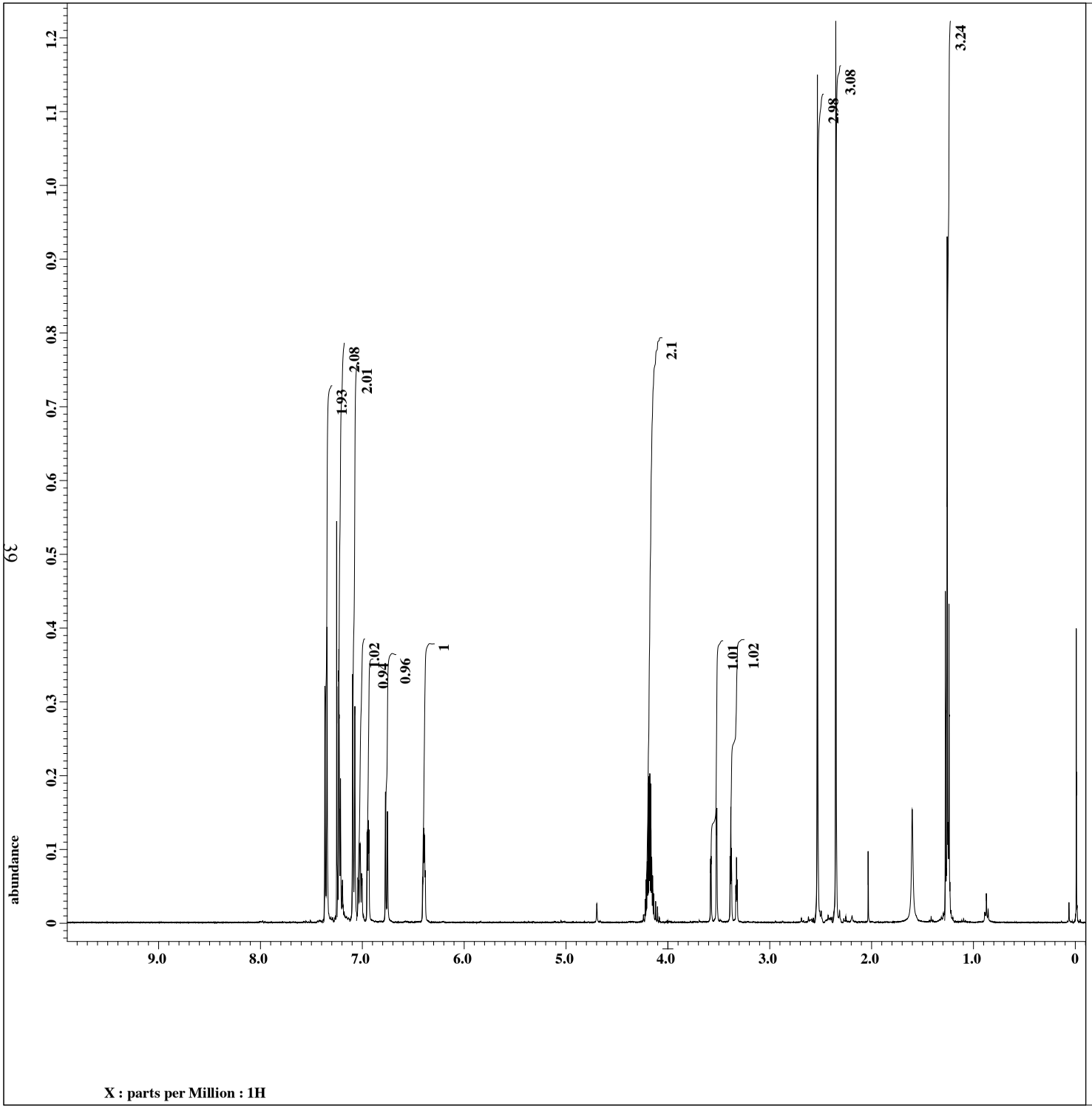
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 294
Total_scans       = 294

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 52
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.6[dC]
```



2P

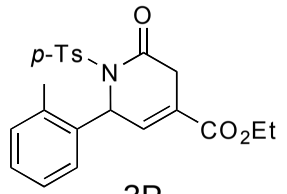



```
Filename          = 603 cyclization (Sulf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  6-JUL-2010 14:29:27
Revision_time     = 15-NOV-2010 15:25:49
Current_time      = 15-NOV-2010 15:26:03

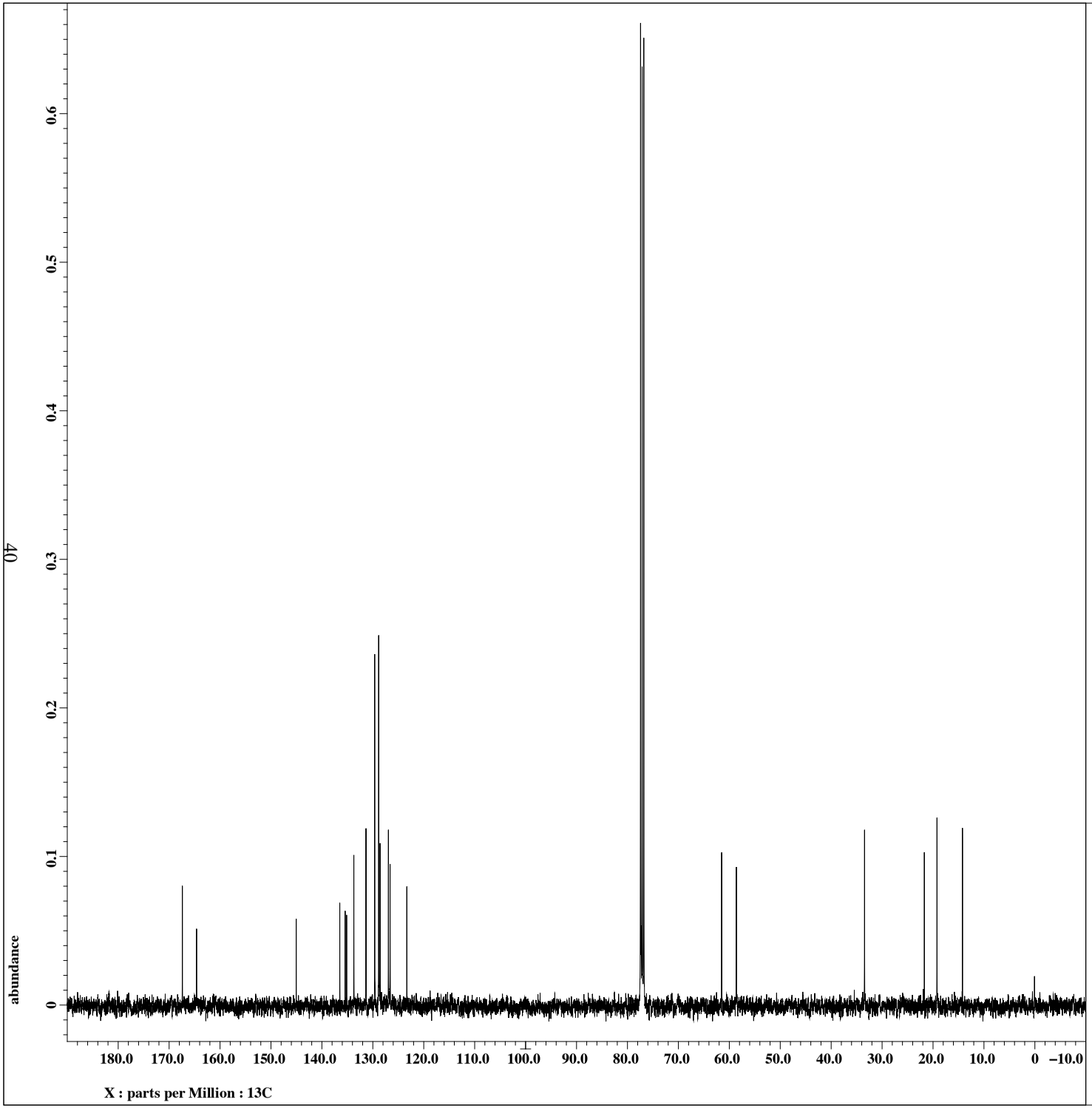
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.8[dC]
```



3P



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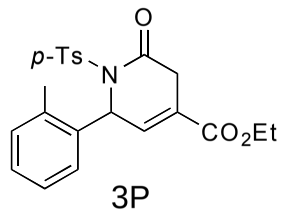
```
Filename          = 603 13C cyclization (
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#556034
Solvent           = CHLOROFORM-D
Creation_time     =  6-JUL-2010 14:51:37
Revision_time     = 15-NOV-2010 15:22:16
Current_time      = 15-NOV-2010 15:22:28

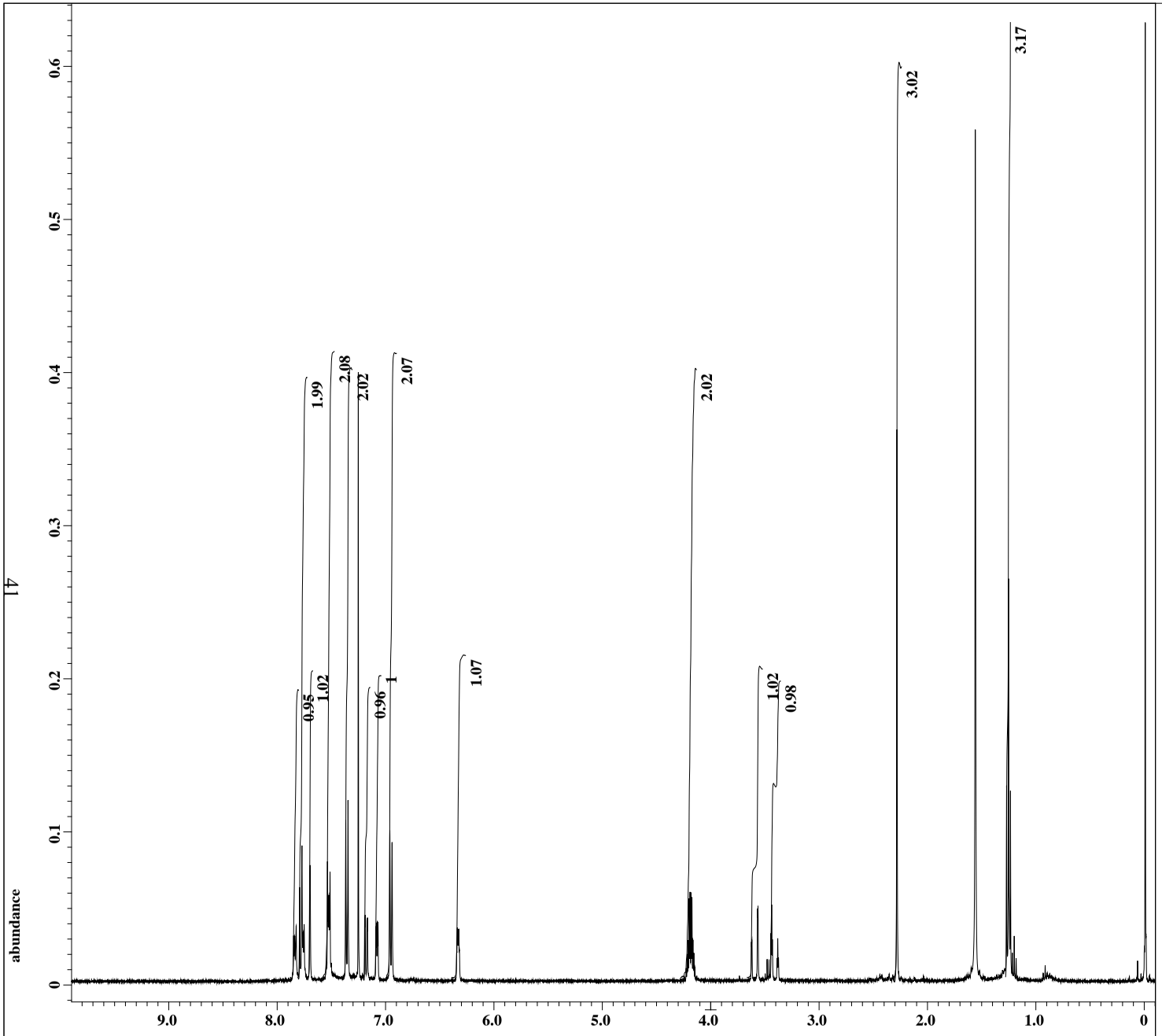
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 160
Total_scans       = 160

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.2[dC]
```



3P

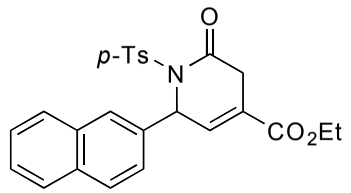



```
Filename          = data naphtarene racta
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 15:12:42
Revision_time     = 15-NOV-2010 15:08:39
Current_time      = 15-NOV-2010 15:08:55

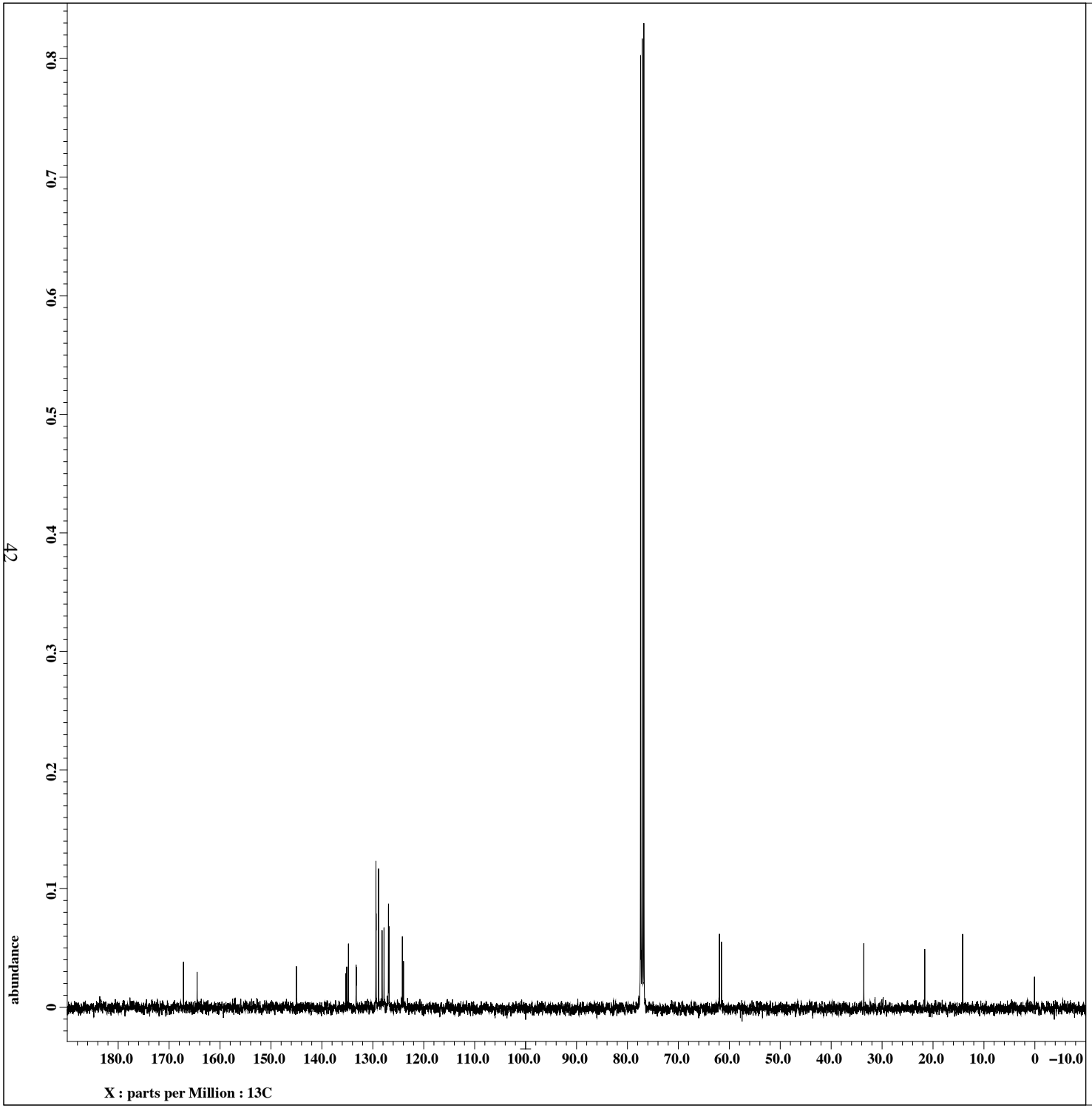
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 25[dC]
```



4P



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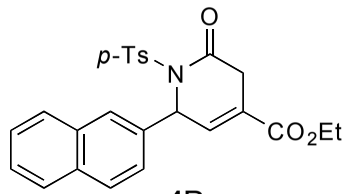
```
Filename          = data 13C naphtarene r
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#604192
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 16:29:36
Revision_time     = 15-NOV-2010 15:09:24
Current_time      = 15-NOV-2010 15:09:38

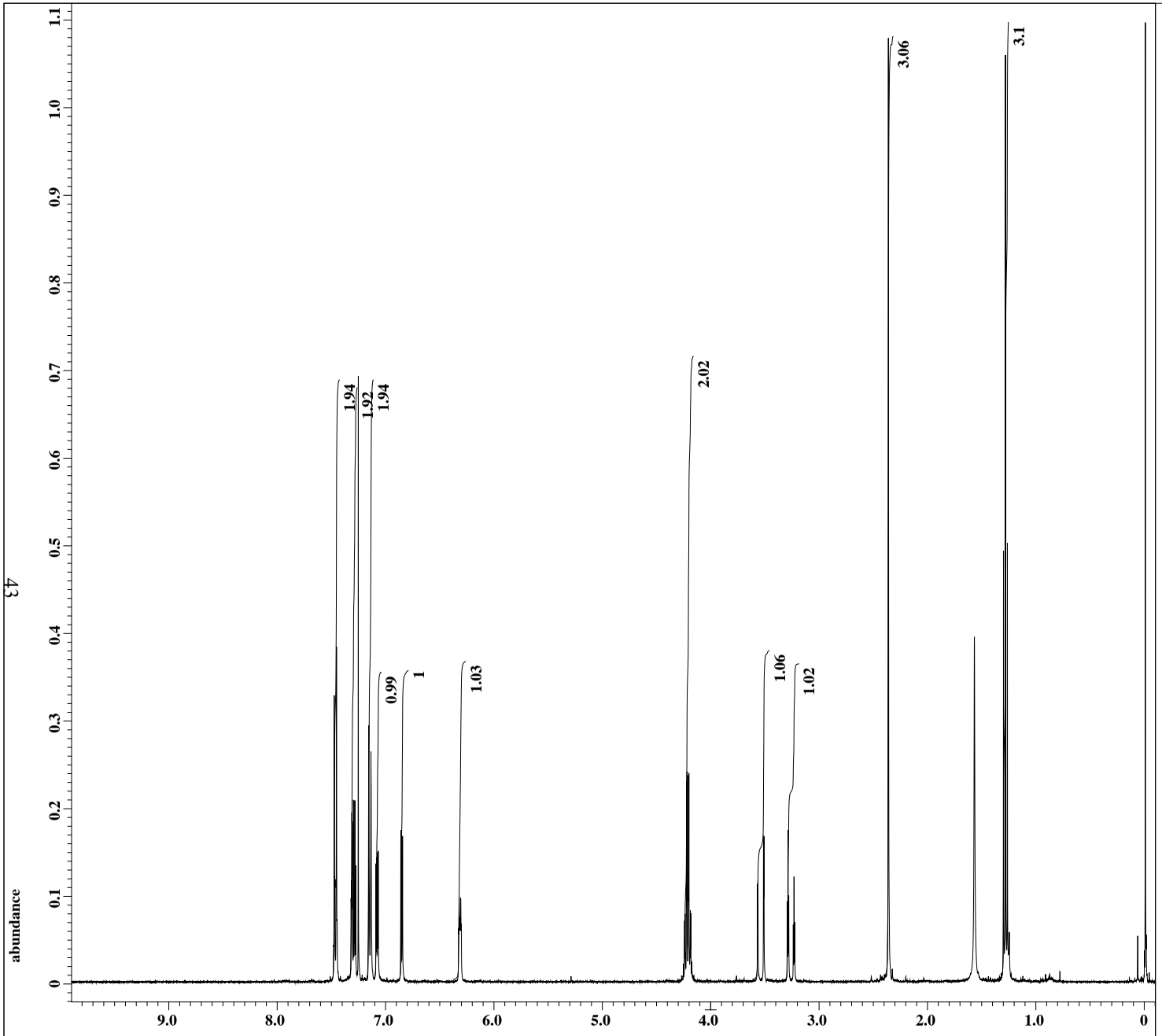
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 333
Total_scans       = 333

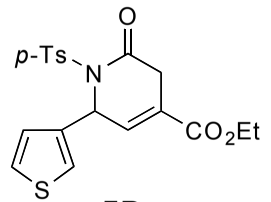
X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 58
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.4[dC]
```



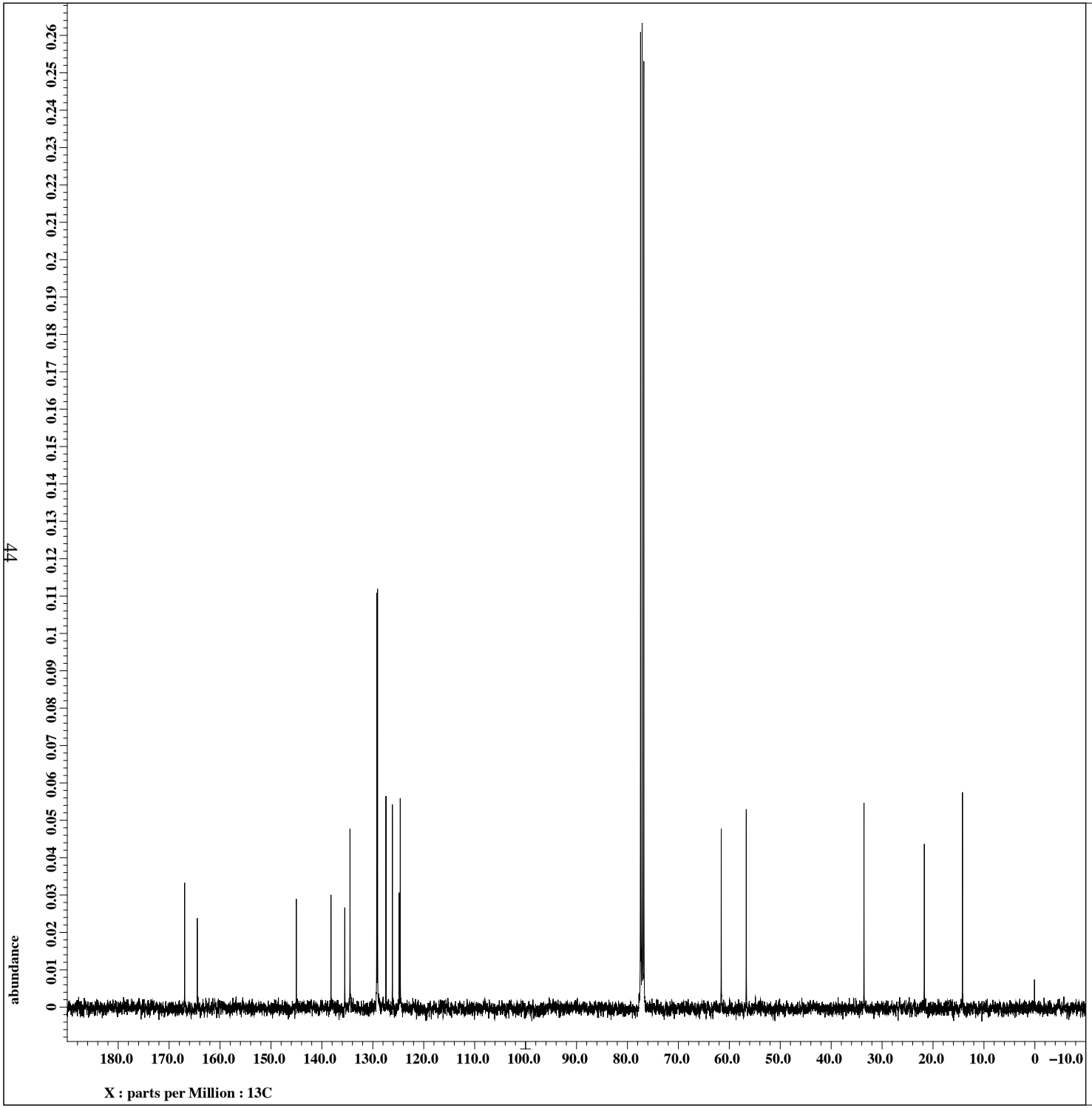
4P



Filename	= data thiophen ractam-
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 27-JUN-2010 14:25:22
Revision_time	= 15-NOV-2010 15:04:38
Current_time	= 15-NOV-2010 15:04:52
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 1[s]
Repetition_time	= 5.36731904[s]
Temp_get	= 25.2[dC]



5P

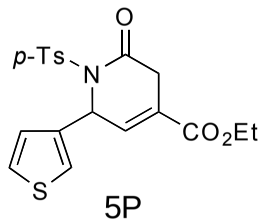



```
Filename          = data 13C thiophen rac
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#582355
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 15:43:43
Revision_time     = 15-NOV-2010 15:05:25
Current_time      = 15-NOV-2010 15:05:36

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 243
Total_scans       = 243

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.5[dC]
```



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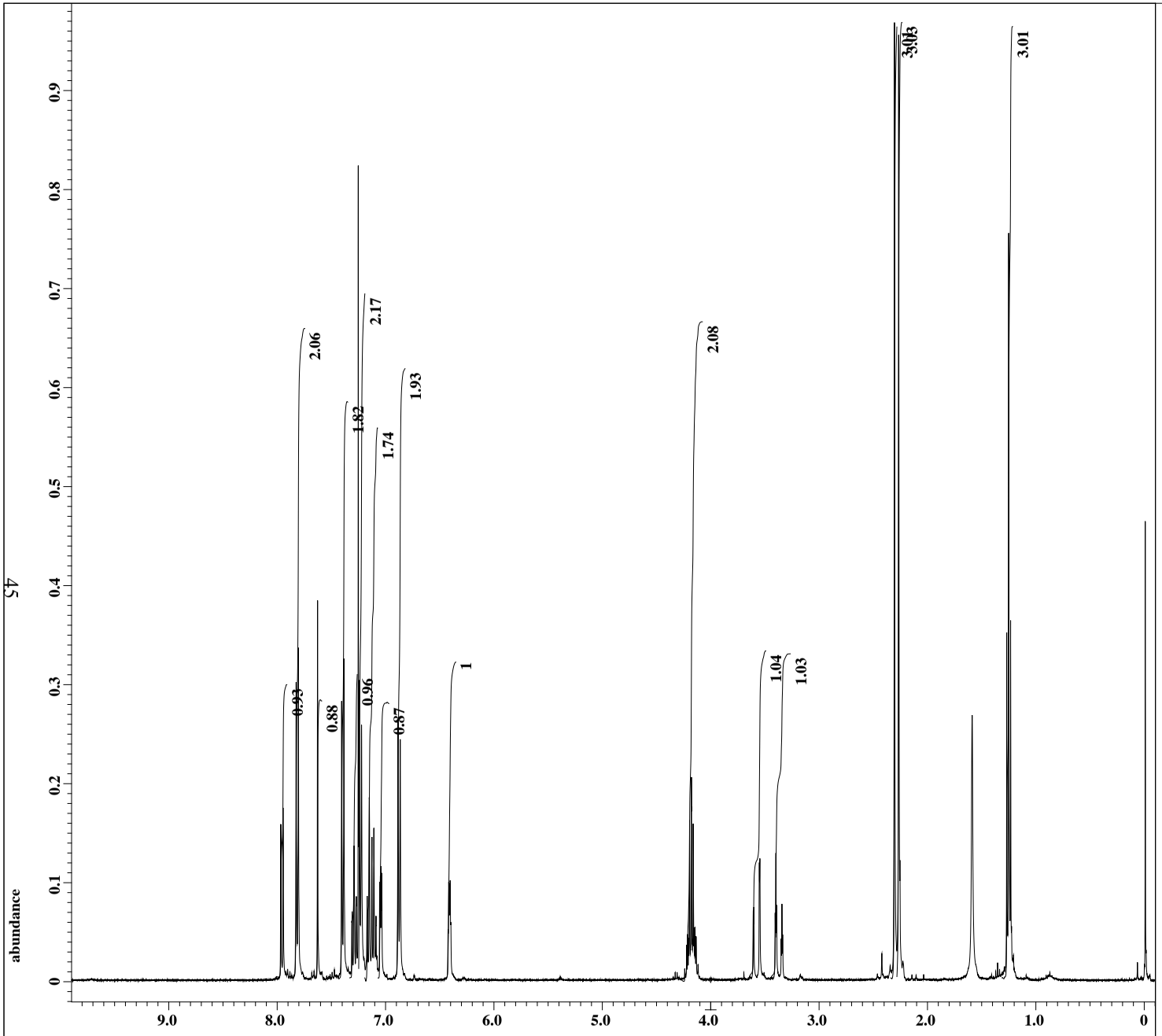
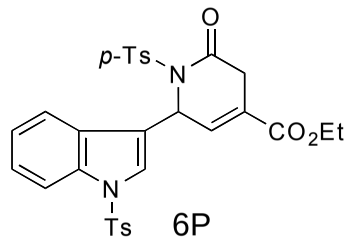



```
Filename          = data indole ractam-4.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  7-JUL-2010 16:07:07
Revision_time     = 15-NOV-2010 15:13:20
Current_time      = 15-NOV-2010 15:13:32

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.8[dC]
```



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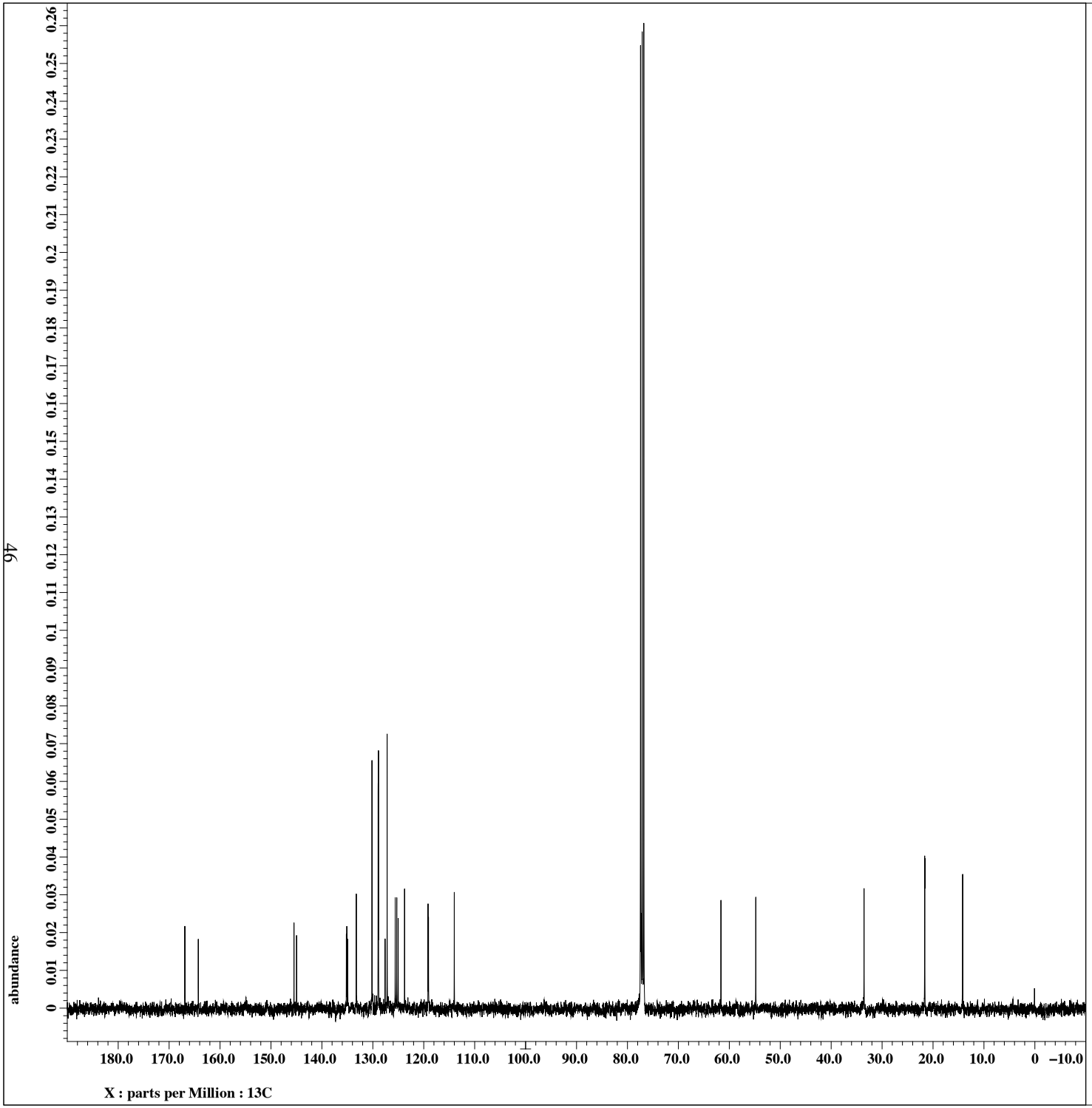
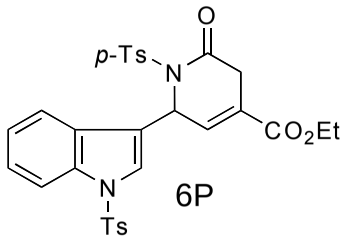


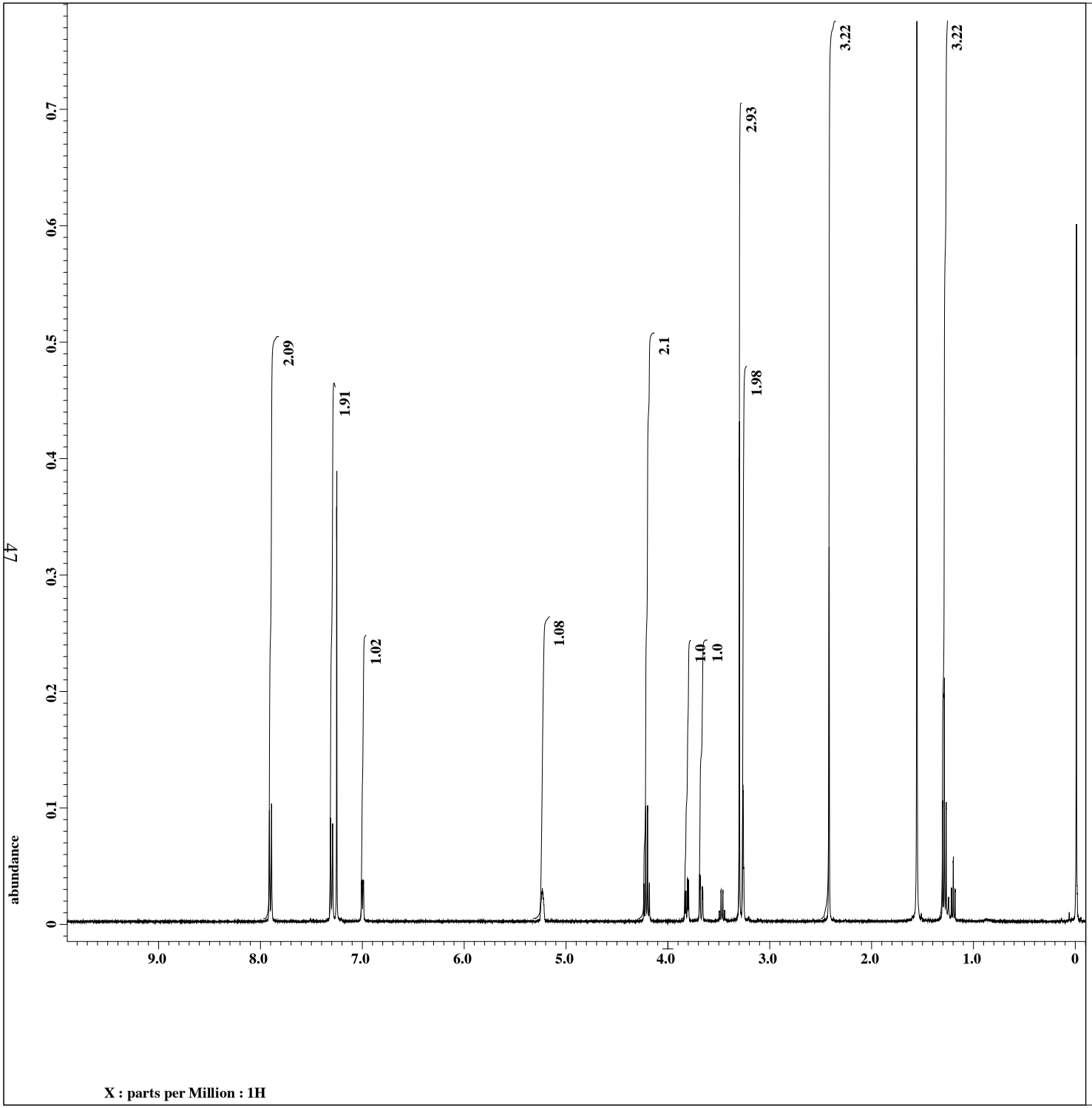

```
Filename          = data 13C indole racta
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#625380
Solvent           = CHLOROFORM-D
Creation_time     =  7-JUL-2010 16:58:53
Revision_time     = 15-NOV-2010 15:13:57
Current_time      = 15-NOV-2010 15:14:09

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

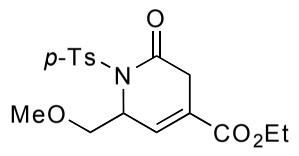
Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 281
Total_scans       = 281

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.5[dC]
```





Filename	= data OMe ractam-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 27-JUN-2010 15:48:27
Revision_time	= 15-NOV-2010 15:21:00
Current_time	= 15-NOV-2010 15:21:16
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX400M
Spectrometer	= DELTA2_NMR
Field_strength	= 9.389766[T] (400[MHz]
X_acq_duration	= 4.36731904[s]
X_domain	= 1H
X_freq	= 399.78219838[MHz]
X_offset	= 4[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22897343[Hz]
X_sweep	= 7.5030012[kHz]
Irr_domain	= 1H
Irr_freq	= 399.78219838[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 399.78219838[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.5[us]
X_acq_time	= 4.36731904[s]
X_angle	= 45[deg]
X_atn	= 1.4[dB]
X_pulse	= 5.25[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 9.36731904[s]
Temp_get	= 25.1[dC]



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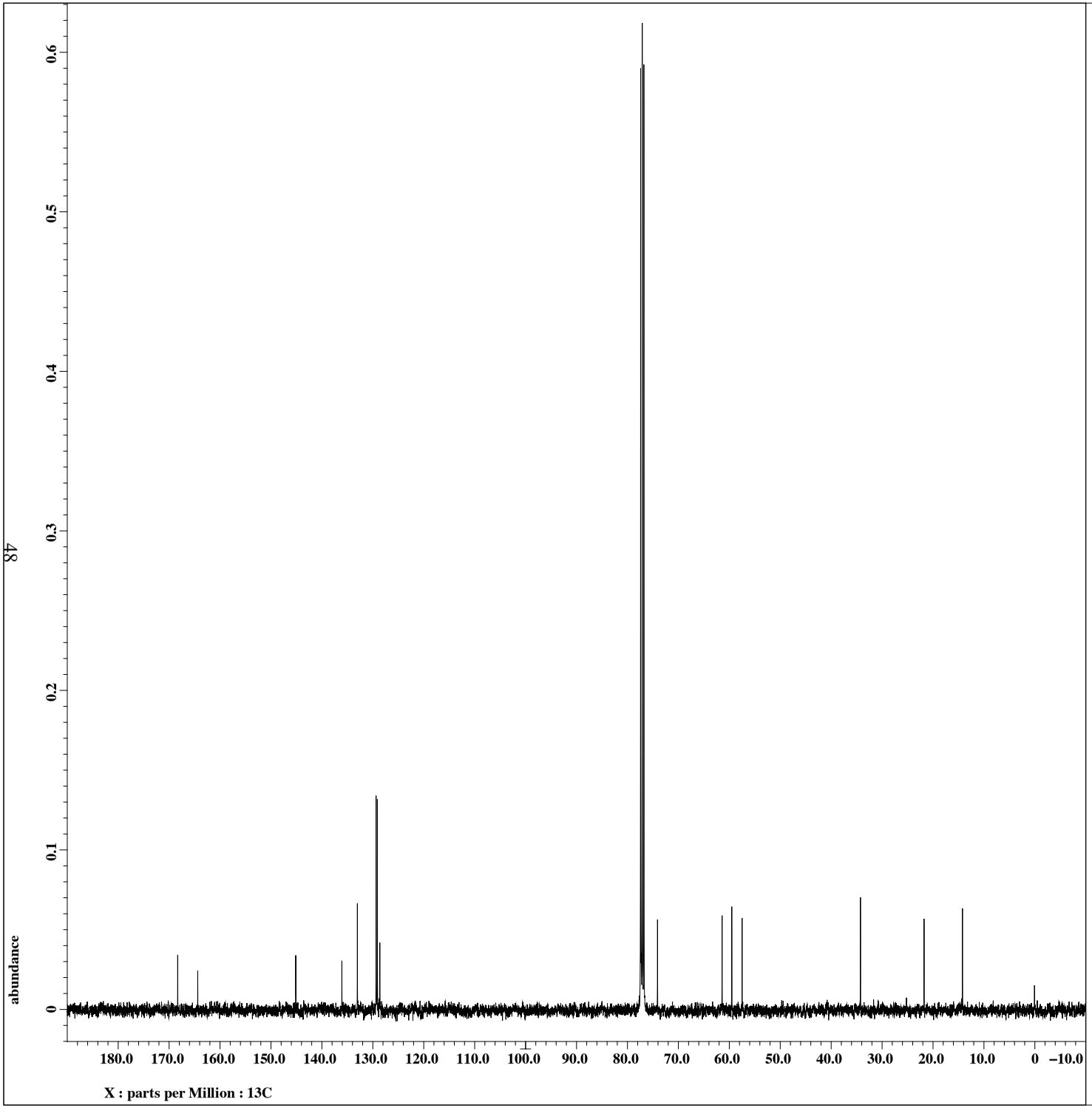
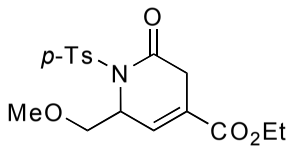



```
Filename          = 498 13C Cyclization (
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#627553
Solvent           = CHLOROFORM-D
Creation_time     =  2-DEC-2009 16:56:37
Revision_time     = 15-NOV-2010 15:21:45
Current_time      = 15-NOV-2010 15:21:59

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 344
Total_scans       = 344

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 56
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.5[dC]
```



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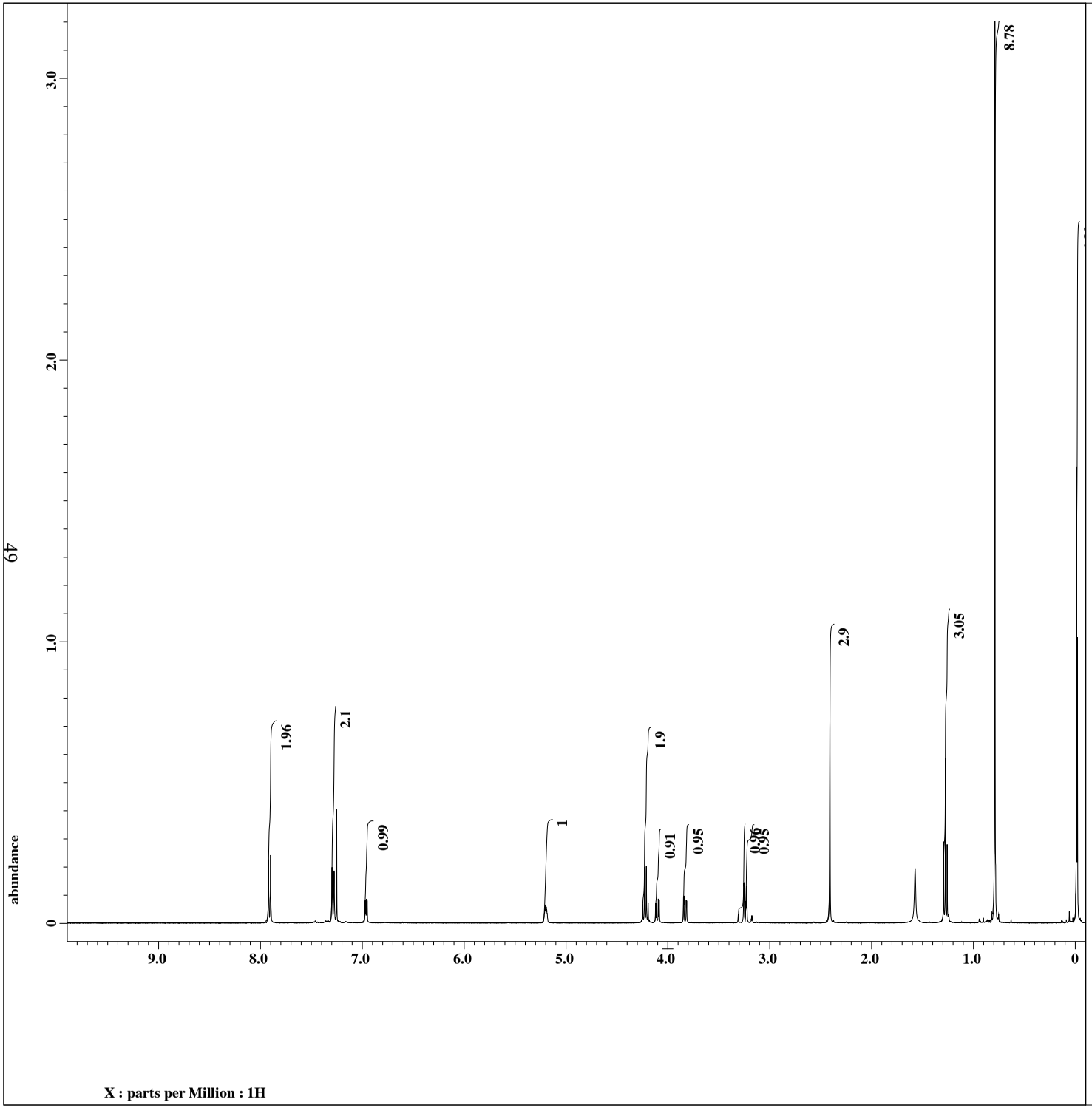
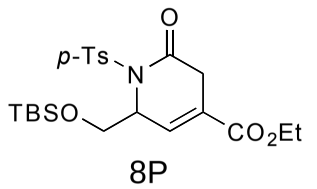



```
Filename          = data TBS ractam-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 14:13:56
Revision_time     = 15-NOV-2010 15:17:17
Current_time      = 15-NOV-2010 15:17:26

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.36731904[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 4[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22897343[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 4.36731904[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 5.36731904[s]
Temp_get          = 24.5[dC]
```



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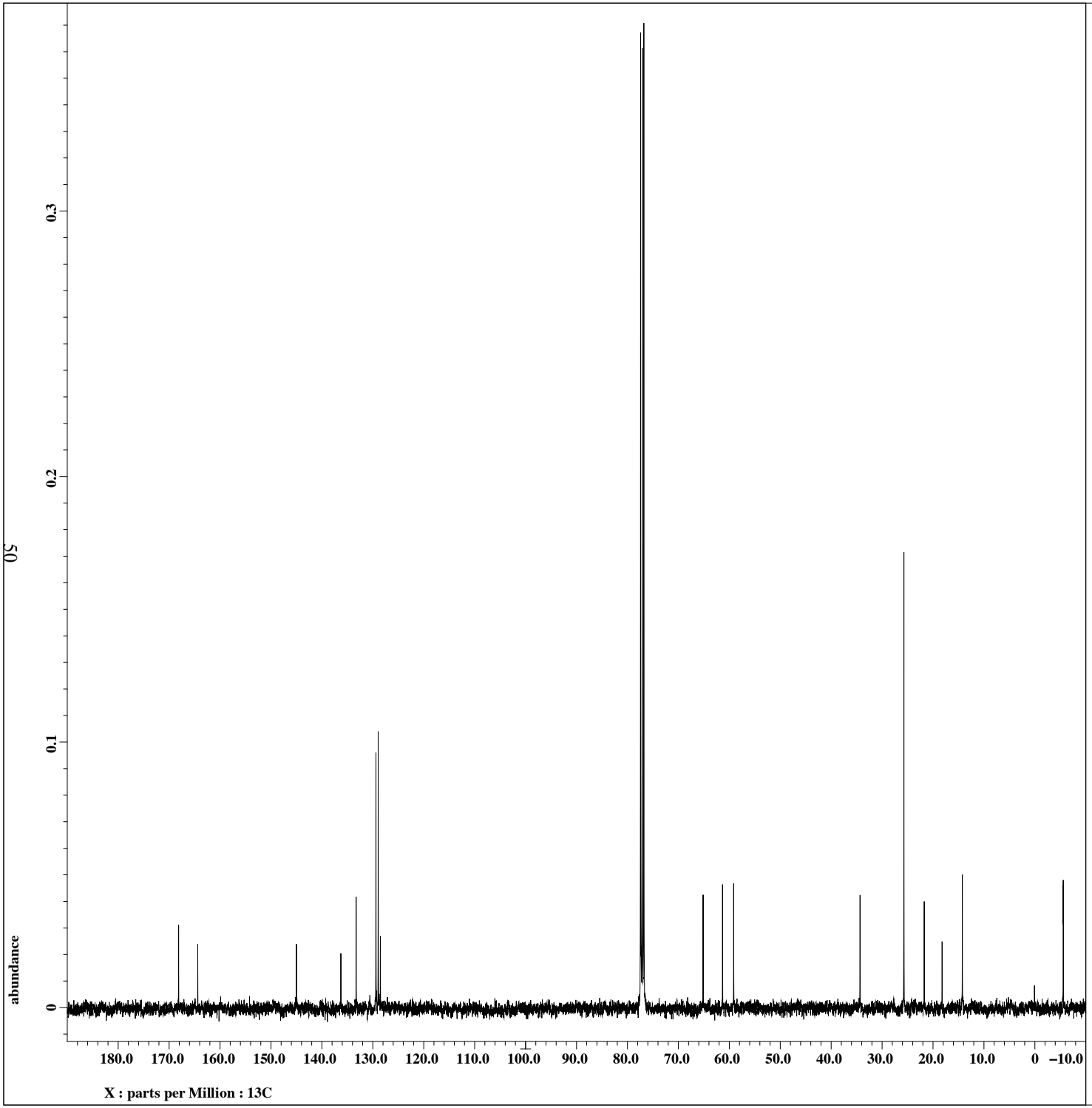
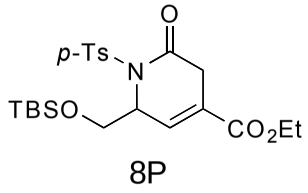



```
Filename          = data 13C TBS ractam-2
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#554222
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUN-2010 15:03:59
Revision_time     = 15-NOV-2010 15:17:50
Current_time      = 15-NOV-2010 15:17:59

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 312
Total_scans       = 312

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 45[deg]
X_atn             = 6.6[dB]
X_pulse           = 4.6[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 5[s]
Recvr_gain        = 52
Relaxation_delay  = 5[s]
Repetition_time   = 6.04333312[s]
Temp_get          = 25.2[dC]
```



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