Total Synthesis of the HDAC inhibitor, (+)-(*R*)-Trichostatin A, via O-Directed Dialkylacetylene Free Radical Hydrostannation with Ph_3SnH/Et_3B. The Unusual Inhibitory Effect of a Proximal α-OPv group on the Course of a Vinyl Iodide Stille Cross-Coupling

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Electronic Supplementary Information

(297 Pages Inclusive of Cover Pages)

Dedicated with admiration and respect to the memory of the great Professor Amos B. Smith III (2014 William H. Nichols Gold Medallist, 2015 RSC Perkin Prize Winner, 2009 RSC Simonsen Medallist, and 2002 RSC Centenary Prize Medallist) in recognition of his numerous magnificent achievements in complex natural product total synthesis, new synthetic methodology development, materials science, and his rational design of a totally new class of HIV-1-neutralising drugs. Sadly, Professor Smith passed away on the morning of Monday February 3rd, 2025, aged 80 years. His landmark contributions to the field of organic synthesis and medicinal chemistry will continue to serve as a source of much future inspiration to us all. He will forever be missed by his many friends, former students, postdoctoral fellows, Associate Editors, and admirers within the world of organic chemistry. Professor Smith was recipient of the Order of the Rising Sun of Japan.

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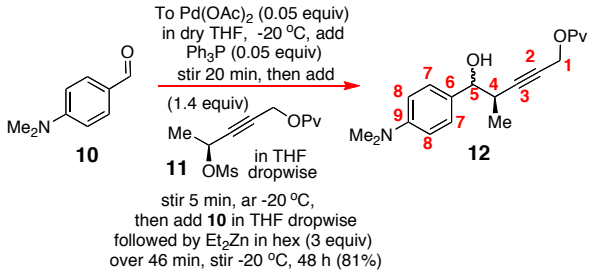
Part A. Experimental Procedures for the Synthesis of Propargyl Alcohol 15 and Additional Experimental Discussion

General Information

Unless stated otherwise, all the reactions in this total synthesis of (+)-(6*R*)-Trichostatin A were run in dry solvents under an N_2 atmosphere. Dry THF and CH_2Cl_2 were freshly distilled from CaH_2 under an N_2 atmosphere. Dry PhMe and MeOH were used as supplied by Sigma-Aldrich. All anhydrous solvents were taken out by dry syringe under an N_2 atmosphere. Ph_3SnH was purchased from Sigma-Aldrich and used as supplied; it was always handled in a glove-bag under N_2. SiO_2 flash chromatography was conducted with Fluorochem silica gel 60Å, and petrol refers to the 40-60 ^oC b.p. fraction; it was distilled prior to use for chromatography. HPLC grade EtOAc was used for all chromatographic purifications. TLC analysis and preparative TLC were performed on Merck glass-backed TLC plates coated with silica gel 60 F_{254}. NMR analyses were carried out using the QUB School of Chemistry Bruker Avance III HD Ascend 600 instrument operating at a frequency of 600.1337 MHz or on the two Bruker Ultrashield 400 NMR spectrometers operating at a frequency of 399.9025 and 400.11 MHz respectively. All ^1H NMR spectra recorded at these frequencies were referenced with respect the solvent peak at δ 7.26 ppm in $CDCl_3$, δ 3.31 ppm in CD_3OD-d_4 or δ 4.79 ppm in D_2O. ^{13}C NMR spectra were recorded at the specified frequency, referenced upon the solvent peak at δ 77.0 pm in $CDCl_3$ or δ 49.0 ppm in CD_3OD-d_4. For the NMR assignments of all the intermediates in this synthetic route, the numbering shown on each structure has been used. IR spectra were recorded on a Perkin – Elmer FT-IR instrument. All IR measurements were performed as neat films on NaCl plates. IR absorptions were recorded in cm^{-1} and the intensity of the absorptions was recorded using following abbreviations: very w – very weak, w – weak, m – medium, s – strong, vs –very strong, br – broad.

Experimental Procedures for the Synthesis of Propargylic Alcohol 15

Preparation of Marshall Adduct 12



To a stirred -20°C solution of $Pd(OAc)_2$ (0.17 g, 0.78 mmol, 0.05 equiv) in dry THF (32 mL) under N_2 was added Ph_3P (0.20 g, 0.78 mmol, 0.05 equiv) in one portion. Immediately the reaction mixture turned a yellow-brown colour. The resulting mixture was stirred for 20 min at -20 °C, whereupon a solution of the freshly made chiral O-mesylate[18] **11** (5.69 g, 21.71 mmol, 1.4 equiv) in dry THF (10 mL) was added dropwise via cannula. After stirring for 5 min, a solution of 4-dimethylaminobenzaldehyde **10** (2.31 g, 15.5 mmol) in THF (10 mL) was added dropwise via cannula. After 10 min, Et_2Zn (1.0 M in hex, 46.53 mL, 46.53 mmol, 3 equiv) was added dropwise via syringe over 46 min. The reaction mixture was maintained at -20°C for 48 h, whereafter the resulting mixture was diluted with EtOAc (50 mL) and quenched with saturated aq. NH_4Cl (100 mL). This led to a white precipitate being formed that was filtered off. The filtrate was then fractionated in a separatory funnel. The aqueous layer was further extracted with EtOAc (50 mL x 3), and the combined organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude residue was purified by SiO_2 flash chromatography with petrol:EtOAc (8:1) as eluent to afford a mixture of C5-alcohols **12** in 1.00:1.12 ratio (4.0 g, 81% based upon aldehyde **10**, 58% based upon mesylate **11**) as an amber oil. IR of **12** (mixture, neat film): 3487 (br m), 2974 (s), 2934 (s), 2803 (m), 2239 (w), 1733 (s), 1616 (s), 1523 (s), 1480 (m), 1459 (m), 1366 (m), 1351 (m), 1283 (m), 1149 (s), 1032 (w), 949 (w), 816 (w) cm^{-1}.

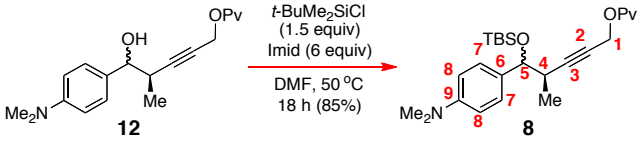
^1H NMR of **12** (Isomer 1) (400.11 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.8 Hz, 2H, H7), 6.70 (d, *J* = 8.4 Hz, 2H, H8), 4.62 (d, *J* = 2.4 Hz, 2H, H1), 4.59 (d, *J* = 6.0 Hz, 1H, H5), 2.94 (s, 6H, $N(C\underline{H}_3)_2$), 2.88 (m, 1H, H4), 2.14 (br, 1H, O<u>H</u>), 1.20 (s, 9H, *t*-<u>Bu</u> of OPv), 1.13 (d, *J* = 7.2 Hz, 3H, C(4)-Me) ppm. ^{13}C NMR of **12** (Isomer 1) (100.57 MHz, $CDCl_3$): δ 177.8 (<u>C</u>=O of Pv), 150.2 (quaternary C at C9), 129.3 (1 x

quaternary C at C6), 127.4 (2 x C8), 112.1 (2 x C7), 88.6 (C2), 76.8 (C3), 76.2 (C5), 52.7 (C1), 40.6 ($N(\underline{C}H_3)_2$), 38.67 ($Me_3\underline{C}O$), 34.2 (C4), 27.1 ($\underline{Me}_3C$ of Pv), 16.0 (C4-$\underline{Me}$) ppm.

^{1}H NMR of **12** (Isomer 2) (400.11 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.8 Hz, 2H, H7), 6.70 (d, *J* = 8.4 Hz, 2H, H8), 4.69 (d, *J* = 2.3 Hz, 2H, H1), 4.41 (br d, *J* = 7.2 Hz, 1H, H5), 2.938 (s, 6H, $N(C\underline{H}_3)_2$), 2.79 (m, 1H, C<u>H</u>-Me), 2.44 (br, 1H, O<u>H</u>), 1.22 (s, 9H, *t*-Bu of OPv), 1.05 (d, *J* = 7.2 Hz, 3H, C(4)-Me) ppm. ^{13}C NMR of **12** (Isomer 2) (100.57 MHz, $CDCl_3$): δ 177.9 (<u>C</u>=O of Pv), 150.4 (1 x quaternary C at C9), 129.1 (1 x quaternary C at C6), 127.5 (2 x C8), 112.3 (2 x C7), 88.5 (C2), 77.3 (C5), 77.2 (C3), 52.7 (C1), 40.6 ($N(\underline{C}H_3)_2$), 38.71 ($Me_3\underline{C}O$), 35.2 (C4), 27.1 ($\underline{Me}_3C$ of Pv), 17.3 (C4-Me) ppm.

TOF ES$^+$ HRMS of **12**: Calcd. for $C_{19}H_{28}NO_3$ $[M+H]^+$: 318.2069. Found: 318.2069.

Preparation of Silyl Ether 8



To a stirred solution of the mixed alcohols **12** (3.79 g, 11.94 mmol) and imidazole (4.88 g, 71.64 mmol, 6 equiv) in dry DMF (60 mL) was added *tert*-butyldimethylsilyl chloride (2.70 g, 17.91 mmol, 1.5 equiv) in one portion. The reactants were then heated at 50°C under N_2 overnight. The reaction mixture was cooled to rt, diluted with EtOAc (50 mL) and excess solid $NaHCO_3$ (about 1 g) was then added followed by addition of saturated aq. $NaHCO_3$ (50 mL) dropwise until CO_2 evolution ceased. The aqueous layer was extracted with EtOAc (50 mL x 3), and the combined organic extracts were washed with H_2O (100 mL), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude residue was purified by gradient elution SiO_2 flash chromatography, transitioning from 30:1→25:1 petrol:EtOAc as eluent, to afford **8** (4.70 g, 85%) as an approx. 1:1 mixture of C5 epimers and as an amber oil. IR of **8** (mixture, neat film): 2960 (s), 2934 (s), 2856 (s), 2240 (w), 1736 (s), 1615

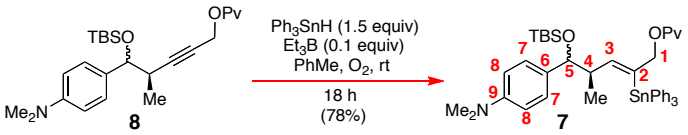
(s), 1521 (s), 1481 (m), 1461 (m), 1360 (m), 1282 (m), 1250 (m), 1146 (s), 1081 (s), 861 (s), 839 (s), 779 (s) cm^{-1}.

^1H NMR of **8** (Isomer 1) (399.9 MHz, $CDCl_3$): δ 7.17 (d, *J* = 8.4 Hz, 2H, H7), 6.66 (d, *J* = 8.8 Hz, 2H, H8), 4.60 (d, *J* = 2.0 Hz, 2H, H1), 4.55 (d, *J* = 6.0 Hz, 1H, H5), 2.93 (s, 6H, $N(C\underline{H}_3)_2$), 2.65 (m, 1H, H4), 1.21 (s, 9H, *t*-Bu of OPv), 1.14 (d, *J* = 6.9 Hz, C4-Me), 0.89 (s, 9H, *t*-Bu of OTBS), 0.05 (s, 3H, $C\underline{H}_3Si$ of OTBS), -0.16 (s, 3H, $C\underline{H}_3Si$ of OTBS) ppm. ^{13}C NMR of **8** (Isomer 1) (100.57 MHz, $CDCl_3$): δ 177.8 (<u>C</u>=O), 149.7 (C9), 131.1 (C6), 127.4 (C7), 111.8 (C8), 90.0 (C2), 77.2 (C5), 75.7 (C3), 52.9 (C1), 40.58 ($N(\underline{C}H_3)_2$), 38.68 ($(CH_3)_3\underline{C}CO$), 35.9 (C4), 27.10 ($(\underline{C}H_3)_3CCO$), 25.84 ($(\underline{C}H_3)_3CSi$), 18.2 ($(CH_3)_3\underline{C}Si$), 16.0 (C4-Me), -4.6 ($\underline{C}H_3Si$), -5.1 ($\underline{C}H_3Si$) ppm.

^1H NMR of **8** (Isomer 2) (399.9 MHz, $CDCl_3$): δ 7.17 (*J* = 8.7 Hz, 2H, H7), 6.66 (*J* = 8.7 Hz, 2H, H8), 4.67 (d, *J* = 2.0 Hz, 2H, H1), 4.48 (d, *J* = 6.8 Hz, 1H, H5), 2.93 (s, 6H, $N(C\underline{H}_3)_2$), 2.71 (m, 1H, H4), 1.23 (s, 9H, *t*-Bu of OPv), 0.96 (d, *J* = 7.0 Hz, $C\underline{H}_3$), 0.87 (s, 9H, *t*-Bu of OTBS), 0.04 (s, 3H, $C\underline{H}_3Si$ of OTBS), -0.14 (s, 3H, $C\underline{H}_3Si$ of OTBS) ppm. ^{13}C NMR of **8** (Isomer 2) (100.57 MHz, $CDCl_3$): δ 177.9 (<u>C</u>=O), 149.9 (C9), 130.3 (C6), 127.7 (C7), 111.8 (C8), 90.1 (C2), 77.7 (C5), 75.5 (C3), 53.0 (C1), 40.56 ($N(\underline{C}H_3)_2$), 38.7 ($(CH_3)_3\underline{C}CO$), 35.8 (C4), 27.12 ($(\underline{C}H_3)_3CCO$), 25.8 ($(\underline{C}H_3)_3CSi$), 18.2 ($(CH_3)_3\underline{C}Si$), 16.5 (C4-Me), -4.7 ($\underline{C}H_3Si$), -5.0 ($\underline{C}H_3Si$) ppm.

TOF ES^+ HRMS of **8**: Calcd. for $C_{25}H_{42}NO_3Si$ $[M+H]^+$: 432.2934. Found: 432.2920.

Vinyl Triphenyltin 7



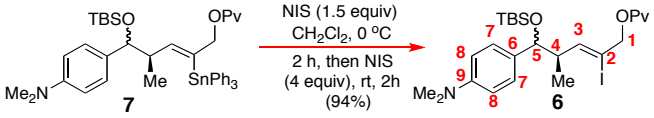
Inside a sealed glove bag containing an N_2 atmosphere, cold solid Ph_3SnH (3.95 g, 11.26 mmol, 1.49 equiv) was weighed into a pear-shaped flask fitted with a rubber septum. When the weighing process

was complete, the septum-sealed flask was removed from the glove bag and fitted with an N_2-filled balloon connected via a Luer-locked needle. Dry PhMe (7.6 mL) was added to a flask containing the pre-dried alkyne **8** (3.27 g, 7.57 mmol) under N_2 and that solution was cannulated into the flask containing the solid Ph_3SnH at rt, maintaining the N_2 atmosphere throughout. A solution of Et_3B (1.0 M in hex, 0.76 mL, 0.76 mmol, 0.1 equiv) was then added slowly to this reaction mixture over 1 min. This was followed by air (10 mL), which was injected into the reaction mixture twice after 5 min and 1 h. The reactants were stirred at rt for 18 h maintaining the N_2 atmosphere throughout. The reaction mixture was then diluted with EtOAc (10 mL) and quenched with H_2O (10 mL). The aqueous layer was extracted with EtOAc (30 mL x 3). The combined organic extracts were then dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude residue was purified by gradient elution SiO_2 flash chromatography initially using petrol:CH_2Cl_2 as eluent (3:1→1:1) to remove tin residues and other impurities, and then finally with petrol:EtOAc 25:1 to elute the vinyl triphenyltin **7** (4.63 g, 78%). It was obtained as a colourless oil and as a mixture of C5-epimers. Data for **7**: ^{1}H NMR of **7** (1:1 mixture at C5) (399.9 MHz, $CDCl_3$): δ 7.58 (m, 6H x 2, ^3J $^{119/117}$Sn-^{1}H = ca. 46.8 Hz, *o*-C<u>H</u>, -$SnPh_3$,), 7.36 (m, 9H x 2, *m*-C<u>H</u> and *p*-C<u>H</u>, -$SnPh_3$), 6.76 (d, *J* = 8.7 Hz, 2H, H7 isomer 1), 6.72 (d, *J* = 10.4 Hz, ^3J ^{119}Sn-^{1}H = 155.2 Hz, ^3J ^{117}Sn-^{1}H = 148.8 Hz, 1H, H3-*syn*-isomer), 6.55 (d, *J* = *ca*. 8.4 Hz, 1H, H3-*anti*-isomer), 6.53 (d, *J* = 8.8 Hz, 2H, H8 isomer 1), 6.44 (d, *J* = 8.8 Hz, 2H, H7 isomer 2), 6.34 (d, *J* = 8.8 Hz, 2H, H8 isomer 2), 4.78 (m, 2H, H1, ^3J $^{119/117}$Sn-^{1}H = 49.2 Hz), 4.45 (d, 1H, *J* = 3.3 Hz, H5-*syn*), 4.23 (d, 1H, *J* = 7.1 Hz, H5-*anti*), 2.93 and 2.88 (s x 2, each 6H, N(C<u>H</u>$_3$)$_2$), 2.48 (m, 1H, H4-*anti*) and 2.26 (m, 1H, H4-*syn*), 0.95 and 0.94 (s x 2, each 9H, *t*-Bu of OPv of *anti* and *syn* isomers), 0.91 and 0.84 (s x 2, each 9H, *t*-Bu of OTBS of *anti* and *syn* isomers), 0.73 (d, *J* = 6.8 Hz, C4-Me-*syn*), 0.56 (d, *J* = 6.8 Hz, C4-Me-*anti*), 0.01, -0.08, -0.22, -0.27 (s x 4, each 3H, C<u>H</u>$_3$Si of *anti* and *syn* isomers) ppm. ^{13}C NMR of **7** (1:1 mixture of isomers at C5) (100.57 MHz, $CDCl_3$): δ 178.14 and 178.07 (<u>C</u>=O of OPv), 154.2 (C3, *syn*-isomer ^2J $^{119/117}$Sn-^{13}C = *ca*. 27.2 Hz), 152.2 (C3, *anti*-isomer, ^2J $^{119/117}$Sn-^{13}C = *ca*. 29.2 Hz), 149.6 and 149.2 (C9, both isomers), 139.0 and 138.7 (quaternary C of -$SnPh_3$, both isomers), 137.14 (*o*-<u>C</u>, -$SnPh_3$, ^3J $^{119/117}$Sn-^{13}C = 37.9 Hz, isomer 1), 137.03 (*o*-<u>C</u>, -$SnPh_3$, ^3J $^{119/117}$Sn-^{13}C = 37.7 Hz, isomer 2), 133.3 and 132.6 (C2, both isomers), 131.36 and 131.33 (C6, both isomers), 129.0 (*p*-<u>C</u>, -$SnPh_3$, ^5J $^{119/117}$Sn-^{13}C = 11.1 Hz) and 128.7 (*p*-<u>C</u>, -$SnPh_3$, ^5J $^{119/117}$Sn-^{13}C = 12.1 Hz), 128.6 (*m*-<u>C</u>, -$SnPh_3$, ^4J $^{119/117}$Sn-^{13}C = *ca.* 52.3 Hz, isomer 1), 128.4 (*m*-<u>C</u>, -$SnPh_3$, ^4J $^{119/117}$Sn-^{13}C = *ca*. 50.3 Hz, isomer 2), 127.9 and 126.7 (C7, both isomers), 111.8 and 111.7 (C8, both isomers), 79.3 and 77.2

(C5, both isomers), 72.2 (C1, ^{2}J $^{119/117}$Sn-^{13}C = 46.0 Hz, isomer 1) and 72.0 (C1, ^{2}J $^{119/117}$Sn-^{13}C = 45.9 Hz, isomer 2), 47.7 and 47.4 (C4, both isomers), 40.62 and 40.6 (N($\underline{C}H_3$)$_2$, both isomers), 38.6 and 38.5 ((CH_3)$_3\underline{C}$CO, both isomers), 27.0 (($\underline{C}H_3$)$_3$CCO, both isomers), 25.9 and 25.86 (($\underline{C}H_3$)$_3$CSi, both isomers), 18.3 ((CH_3)$_3\underline{C}$Si, both isomers), 17.3 (C4-Me, *anti*-isomer) 13.1 (C4-Me, *syn*-isomer), -4.48, -4.54, -4.98 and -5.11 ($\underline{C}H_3$Si, both isomers) ppm.

TOF ES$^+$ HRMS of **7**: Calcd. for $C_{43}H_{58}NO_3SiSn$ $[M+H]^+$: 784.3217. Found: 784.3217.

Vinyl Iodide 6

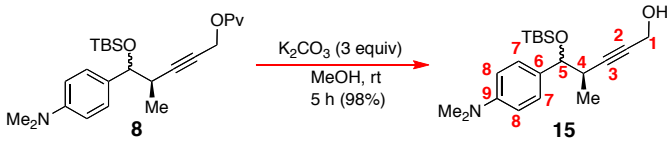


To the vinyl triphenyltin **7** (0.20 g, 0.26 mmol), in an Al-foil-covered pear-shaped flask under N_2, was added dry CH_2Cl_2 (1.28 mL). The resulting solution was cooled to 0 °C and *N*-iodosuccinimide (NIS, 0.09 g, 0.38 mmol, 1.5 equiv) was added in one portion. Stirring was continued at 0°C under N_2 for 2 h and then at rt for 1 h. After 3 h in total, a further quantity of NIS (0.23 g, 1.02 mmol, 4 equiv; making in total 5.5 equiv of NIS) was added to the reaction mixture and the reactants were stirred for another 2 h at rt. under N_2, maintaining darkness throughout. The reaction flask was then diluted with EtOAc (5 mL) and quenched by adding saturated aq. $NaHCO_3$ (10 mL). The aqueous layer was then extracted with EtOAc (30 mL x 3) and the combined organic extracts were successively washed with saturated aq. $Na_2S_2O_3$ (50 mL) and H_2O (50 mL x 2). The organic layer was dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude residue was purified by gradient elution SiO_2 flash chromatography using petrol:CH_2Cl_2 3:1→2:1→1:1 as the eluent to remove tin residues, and petrol:EtOAc 4:1→3:1 to elute the iodide **6** (0.13 g, approx 91%) as a slightly impure amber oil. It contained a small amount of succinimide/NIS according to NMR analysis (^{1}H resonances at around δ 2.70 ppm, and ^{13}C resonances at around δ 177.6, 29.67 and 28.20 ppm, as confirmed by DEPT spectroscopy). Data for **6:** ^{1}H NMR of **6** (mixture of isomers at C5) (399.9 MHz, $CDCl_3$): δ 7.19 (d, *J* =

8.8 Hz, 2H, H7, isomer 1), 7.11 (d, *J* = 8.4 Hz, 2H, H7, isomer 2), 6.95 (d, *J* = 8.8 Hz, 2H, H8, isomer 1), 6.92 (d, *J* = 8.8 Hz, 2H, H8, isomer 2), 5.82 (d, *J* = 8.8 Hz, 1H, H3, isomer 1), 5.73 (d, *J* = 9.2 Hz, 1H, H3, isomer 2), 5.07 (m, 2H, H1, isomer 1), 4.69 (complex m, 3H, H1 of isomer 2 superimposed upon H5 of one isomer). 4.68 (d, 1H, *J* = 6.0 Hz, H5 of one isomer), 4.49 (d, 1H, *J* = 5.9 Hz, C<u>H</u>-OTBS), 3.12 and 3.11 (s, 6H, N(C<u>H</u>$_3$)$_2$, both isomers), 2.73 and 2.63 (m, 1H, H4, both isomers), 1.24 and 1.23 (s, 9H, *t*-Bu of Pv, both isomers), 0.91 (d, *J* = 7.2 Hz, C4-Me, one isomer) superimposed upon 0.87 (d, C4-Me, of other isomer), 0.89 and 0.86 (s, 9H, *t*-Bu of OTBS of *anti* and *syn* isomers), -0.005, -0.02, -0.20 and -0.23 (s x 4, 3H each, C<u>H</u>$_3$Si, both isomers) ppm. ^{13}C NMR of **6** (mixture of isomers at C5) (100.57 MHz, $CDCl_3$): δ 177.54 and 177.51 (<u>C</u>=O of OPv, both isomers), 146.5 and 146.3 (C9, both isomers), 142.7 and 141.3 (C3, both isomers), 132.8 and 132.7 (C6, both isomers), 127.6 and 127.2 (C7, both isomers), 112.7 and 112.6 (C8, both isomers), 99.7 and 99.0 (C2, both isomers), 77.4 and 76.3 (C5, both isomers), 57.62 and 57.58 (C1, both isomers), 49.3 and 48.9 (C4, both isomers), 39.4 (N(<u>C</u>H$_3$)$_2$, both isomers), 38.84 and 38.82 ((CH$_3$)$_3$<u>C</u>CO, both isomers), 28.2 (N(<u>C</u>H$_3$)$_2$), 27.19 and 27.17 ((<u>C</u>H$_3$)$_3$CCO), 25.9 and 25.8 ((<u>C</u>H$_3$)$_3$CSi, both isomers), 18.2 and 18.1 ((CH$_3$)$_3$<u>C</u>Si, both isomers), 15.4 and 12.8 (C4-<u>Me</u>, both isomers), -4.5, -4.6, -5.0, -5.2 (<u>C</u>H$_3$Si, both isomers) ppm.

TOF ES$^+$ HRMS of **6**: Calcd. for $C_{25}H_{42}NIO_3Si$ $[M]^+$: 559.1928. Found: 559.1931.

Preparation of Propargyl Alcohol 15



To a stirred rt solution of the ester **8** (4.70 g, 10.88 mmol) in commercial, undried, MeOH (109 mL) was added K_2CO_3 (4.51 g, 32.21 mmol, 3 equiv) in one portion. After stirring at rt for 5 h, the reaction mixture was diluted with EtOAc (50 mL) and quenched with H_2O (100 mL). The aqueous layer was extracted with EtOAc (50 mL x 3) and the combined organic extracts were washed with H_2O (50 mL x 2), dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude residue was

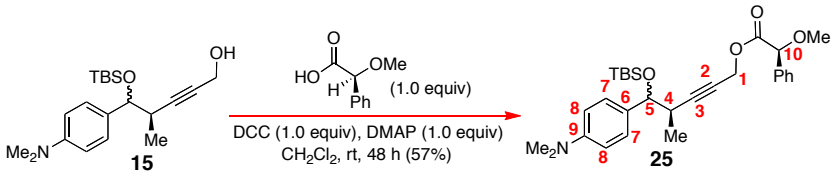
purified by gradient elution SiO_2 flash chromatography with 10:1→6:1 petrol:EtOAc as eluent to afford **15** (3.69 g, 98%) as an oil. Data for **15**: IR of **15** mixture (neat film): 3350 (br, m), 2957 (s), 2932 (s), 2889 (s), 2856 (s), 2235 (w), 1615 (s), 1569 (w), 1524 (s), 1473 (m), 1352 (m), 1254 (m), 1188 (w), 1163 (m), 1080 (s), 946 (m), 868 (s), 838 (s) 780 (s), 674 (m), 565 (m) cm^{-1}.

^1H NMR of **15** (isomer 1) (399.9 MHz, $CDCl_3$, faster moving from the mixture): δ 7.17 (d, *J* = 8.8 Hz, 2H, H7), 6.67 (d, *J* = 8.8 Hz, 2H, H8), 4.49 (d, *J* = 6.5 Hz, 1H, H5), 4.25 (apparent d, *J* = 1.6 Hz, 2H, H1), 2.94 (s, 6H, $N(C\underline{H}_3)_2$), 2.71 (m, 1H, H4), 1.59 (br, 1H, O<u>H</u>), 0.97 (d, *J* = 6.8 Hz, 3H, C4-<u>Me</u>), 0.88 (s, 9H, *t*-<u>Bu</u> of OTBS), 0.03 (s, 3H, $C\underline{H}_3Si$ of OTBS), -0.14 (s, 3H, $C\underline{H}_3Si$ of OTBS) ppm. ^{13}C NMR of **15** (isomer 1) (100.57 MHz, $CDCl_3$): δ 149.9 (C9), 130.4 (C6), 127.7 (C7), 111.8 (C8), 89.4 (C2), 79.7 (C3), 77.7 (C5), 51.5 (C1), 40.6 ($N(\underline{C}H_3)_2$), 35.6 (C4), 25.8 ($(\underline{C}H_3)_3CSi$), 18.3 ($(CH_3)_3\underline{C}Si$), 16.5 (C4-<u>Me</u>), -4.6 ($\underline{C}H_3Si$), -5.1 ($\underline{C}H_3Si$) ppm.

^1H NMR of **15** (isomer 2) (399.9 MHz, $CDCl_3$, slower moving from the mixture): δ 7.17 (d, *J* = 8.4 Hz, 2H, H7), 6.67 (d, *J* = 8.8 Hz, 2H, H8), 4.51 (d, *J* = 6.4 Hz, H5), 4.16 (apparent d, *J* = 2.0 Hz, 2H, H1), 2.94 (s, 6H, $N(C\underline{H}_3)_2$), 2.65 (m, 1H, H4), 1.60 (br, 1H, O<u>H</u>), 1.17 (d, *J* = 6.8 Hz, 3H, C4-<u>Me</u>), 0.88 (s, 9H, *t*-<u>Bu</u> of OTBS), 0.04 (s, 3H, $C\underline{H}_3Si$ of OTBS), -0.17 (s, 3H, $C\underline{H}_3Si$ of OTBS) ppm. ^{13}C NMR of **15** (isomer 2) (100.57 MHz, $CDCl_3$): δ 149.8 (C9), 131.4 (C6), 127.5 (C7), 111.8 (C8), 89.4 (C2), 80.0 (C3), 77.7 (C5), 51.4 (C1), 40.6 ($N(\underline{C}H_3)_2$), 36.0 (C4), 25.8 ($(\underline{C}H_3)_3CSi$), 18.3 ($(CH_3)_3\underline{C}Si$), 16.5 ($\underline{C}H_3$), -4.5 ($\underline{C}H_3Si$), -5.1 ($\underline{C}H_3Si$) ppm.

TOF ES^+ HRMS of **15**: Calcd. for $C_{20}H_{34}NO_2Si$ $[M+H]^+$: 348.2359. Found: 348.2358.

Derivatisation of Chiral Alcohol Mixture 15 as Ester 25



To a stirred solution of the alcohol **15** (50 mg, 0.14 mmol) and (*R*)-methoxyphenylacetic acid (26 mg, 0.14 mmol, 1 equiv) in commercial CH_2Cl_2 (1 mL) was added *N,N'*-dicyclohexyl-carbodiimide (DCC) (33 mg, 0.14 mmol, 1 equiv) and 4-dimethylaminopyridine (DMAP) (2 mg, 0.01 mmol, 0.1 equiv) and stirring was continued at rt for 36 h. The crude reaction mixture (including the CH_2Cl_2 solvent) was applied directly to a short SiO_2 column and gradient elution flash chromatography performed with 18:1→15:1 petrol/EtOAc as eluent, to obtain the product **25** (40 mg, 56%). ^{1}H NMR of **25** subsequently revealed that **15** was a mixture of two C(5)-epimers whose ratio was close to 1:1 and that **15** was of approximately 98% ee. Data for **25**: ^{1}H NMR of **25** (ca. 1:1 mixture of isomers at C5) (600.13 MHz, CD_3OD): δ 7.48-7.42 (m, 2H x 2, Ph of methoxyphenylacetate), 7.39-7.31 (m, 3H x 2, Ph of methoxyphenylacetate), 7.13 (d, *J* = 9.0 Hz, 2H, H7, isomer 1) superimposed upon 7.12 (d, *J* = 8.4 Hz, 2H, H7, isomer 2), 6.65 (d, *J* = 8.4 Hz, H8, both isomers), 4.82 (s, 1H, -(Ph)CH(OMe)/H10) and 4.79 (s, 1H, -(Ph)CH(OMe)/H10) superimposed upon 4.80 (dd, *J* = 15.6 Hz, 1H, H1a, isomer 1), 4.73 (dd, *J* = 15.6, 2.4 Hz, 1H, H1a, isomer 2), 4.64 (dd, *J* = 15.6, 1.8 Hz, 1H, H1b, isomer 1), 4.58 (dd, *J* = 15.0, 2.4 Hz, 1H, H1b, isomer 2), 4.50 (d, *J* = 6.0 Hz, 1H, H5, isomer 1), 4.43 (d, *J* = 6.6 Hz, 1H, H5, isomer 2), 3.428 (s, 3H, OCH_3), isomer 1), 3.425 (s, 3H, OCH_3, isomer 2), 2.94 (s, 6H, $N(CH_3)_2$, isomer 1), 2.93 (s, 6H, $N(CH_3)_2$, isomer 2), 2.67 (m, 1H, C4-Me, isomer 1), 2.60 (m, 1H, C4-Me, isomer 2), 1.10 (d, *J* = 6.6 Hz, 3H, CH_3), 0.91 (d, *J* = 7.2 Hz, 3H, CH_3), 0.88 (s, 9H, *t*-Bu of OTBS of isomer 2), 0.86 (s, 9H, *t*-Bu of OTBS of isomer 1), 0.021 (s, 3H, CH_3Si), 0.013 (s, 3H, CH_3Si), -0.16 (s, 3H, CH_3Si), -0.18 (s, 3H, CH_3Si) ppm. ^{13}C NMR of **25** (ca. 1:1 mixture of isomers at C5) (150.92 MHz, $CDCl_3$): δ 170.1 and 170.0 (C=O), 149.9 and 149.8 (C9), 136.0 (quaternary C of Ph), 131.1 and 130.2 (C6), 128.8 (CH-aromatic ring from the acid), 128.6 (CH-aromatic ring from the acid), 127.7 (CH-aromatic ring from **15**), 127.4 (CH-aromatic ring from the acid), 127.3 (CH-aromatic ring from the acid), 111.8 (CH-aromatic ring from **15**), 91.0 and 90.9 (C2), 82.5 ((-Ph)CH-OMe, both isomers), 77.6 and 77.2 (C5), 74.9 and 74.8 (C3), 57.40 and 57.39 (-(Ph)CH-OMe), 53.6 and 53.5 (C1), 40.60 and

40.58 ($N(\underline{C}H_3)_2$), 35.9 and 35.7 (C4), 25.83 and 25.78 ($(\underline{C}H_3)_3CSi$), 18.23 and 18.22 ($(CH_3)_3\underline{C}Si$), 16.3 and 15.9 (C4-Me), -4.6 ($\underline{C}H_3Si$), -4.7 ($\underline{C}H_3Si$), -5.07 ($\underline{C}H_3Si$), -5.09 ($\underline{C}H_3Si$) ppm.

TOF ES^+ HRMS of **25**: Calcd. for $C_{29}H_{42}NO_4Si$ $[M+H]^+$: 496.2883. Found: 496.2880.

Extra Commentary on The O-Desilylation of Enantiomeric (-)-24 to (-)-(*S*)-Trichostatin A in the Presence of 3Å Molecular Sieves

It is worthy of mention that when Dr Manaviazar conducted the synthesis of enantiomeric (-)-(6*S*)-trichostatin A by an identical route, using the enantiomer of **11**, synthetically pure (-)-**24** was isolated in 61% yield when 140 mg of (-)-(6*S*)-trichostatic acid was employed for the Helquist siloxyamination procedure.[17e] In this instance, Dr Manaviazar isolated pure (-)-**24** by extractive work-up and by gradient elution SiO_2 flash chromatographic purification using petrol:EtOAc (6:1→3:1) as eluent.

Dr Manaviazar subsequently observed that when purified **24** was converted through to (-)-(6*S*)-trichostatin A with CsF in MeOH ***in the presence of 3Å molecular sieves***, a much lower 44% yield of (-)-(6*S*)-trichostatin A was obtained than that recorded in the literature, despite this deprotection being very clean according to TLC analysis. We believe that the lower yield of (-)-**1a** obtained in the presence of 3Å molecular sieves could potentially be due to irreversible trapping and adsorption of some of the product **(-)-TSA** within the zeolite.

In this regard we note that when Professor Helquist's team[17e] conducted the (+)-(6*R*)-trichostatic acid (**22**)→(+)-(6*R*)-trichostatin A (**1**) conversion on 173 mg scale, ***without 3Å sieves added at the CsF/MeOH step***, they obtained 61 mg (+)-(6*R*)-trichostatin A (**1**) in 35% yield ***for the two steps***. This procedure also did not purify the intermediary TBSO-trichostatin A **24** by SiO_2 flash chromatography but instead used crude **24** for the CsF/MeOH deprotection step. This appears to be essential to obtain high yields of **1a**.

It is also important to note that when the Helquist team[17e] performed the conversion of (+)-(6*R*)-trichostatic acid (**22**) into (+)-(3*R*)-trichostatin A (**1**) on much larger 1.02 g scale with respect to **22**, and their crude procedure was followed (***in the absence of 3Å*** sieves for the CsF/MeOH deprotection step), 0.98 g of (+)-(3*R*)-trichostatin A (**1**) was isolated after trituration of the crude reaction mixture with Hex/Et_2O (4:1), which represents a 92% yield for the overall (**22**)→(+)-(6*R*)-trichostatin A 2 step conversion.

We therefore ***strongly recommend*** that, in future, workers follow Professor Paul Helquist's exact endgame[17e] for effecting the (+)-(6*R*)-trichostatic acid (**22**)→(+)-(6*R*)-trichostatin A (**1a**) conversion,[17e] when using our newly developed, ***improved***, intersecting route. The Helquist protocol performs the final CsF deprotection step in MeOH at rt ***without any added molecular sieves***.

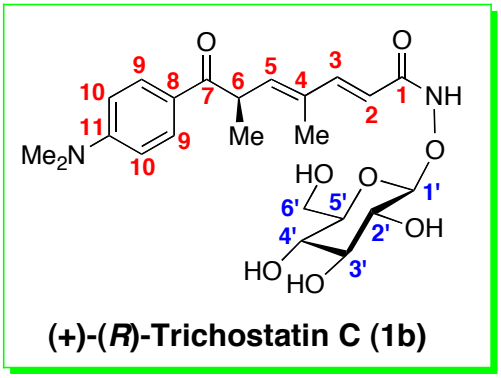
Proof of the (6*R*)-Stereochemistry and Optical Purity of Our Synthetic (+)-(*R*)-Trichostatin A (1a). Conversion of Our Synthetic (+)-Trichostatic Acid (22) into (+)-Trichostatin C (1b), and Synthetic Verification of the Structure Assigned to (+)-Trichostatin C (1b)

Apart from measuring the optical rotation of our fully synthetic (+)-(*R*)-trichostatin A (**1a**) ($[\alpha]_D$ = +88.8° (*c* 0.26, MeOH)), in order for us confirm that our synthetic material was of high enantiomeric purity, we synthetically converted its (+)-(*R*)-trichostatic acid precursor **22** into its β-glucopyranoside derivative of (+)-trichostatin A, namely, (+)-trichostatin C (**1b**).[20a,16] The NMR data of our synthetic (+)-trichostatin C (**1b**) nicely matched the NMR data that had previously been reported in the literature by Tsuji,[20a] so confirming that the original structure that had been assigned to **1b** by Tsuji and Kobayashi,[20a] and later by Thorsen,[20b] and Xu and SI,[16] was indeed correct. The details of our first total synthesis of trichostatin C (**1b**) will be reported in a separate paper, but the NMR data and ^{1}H and ^{13}C NMR spectra for our synthetic **1b** are included here in this SI, in order to support and confirm the integrity of the present total synthesis of (+)-(*R*)-trichostatin A (**1a**).

With regard to our synthetic (+)-(*R*)-trichostatin A (**1a**), the $[\alpha]_D$ value of +88.8° (*c* 0.26, MeOH, 20 °C) that was recorded for **1a** compares very favourably with earlier $[\alpha]_D$ data reported by Mori[17b] for (*R*)- and (*S*)-trichostatin A in MeOH. Thus, Mori recorded an $[\alpha]_D$ +96 ° (*c* 0.31, MeOH, 22 °C) for optically pure (+)-(*R*)-trichostatin A, synthesised from methyl (*R*)-3-hydroxy-2-methyl-propionate of 99% ee. Mori also reported an $[\alpha]_D$ -82 ° (*c* 0.24, MeOH, 17 °C) for optically pure (-)-(*S*)-trichostatin A synthesised by the same route from methyl (*S*)-3-hydroxy-2-methyl-propionate of 99% ee. Thus our $[\alpha]_D$ value for **1a** lies well within the margins of error that Mori reported for these two measurements. In light of this, and our derivatisation of our synthetic (+)-(*R*)-trichostatic acid (**22**) as (+)-trichostatin C (**1b**), and our earier derivatisation of propargylic alcohol **15** as chiral ester **25**, we consider that the synthetic (+)-(*R*)-trichostatin A (**1a**) that we have prepared is of approximately 98-100 % ee. However, we also recognise that there will always be errors associated with weighing small samples, much of which often depends on the accuracy of the balance used, and this could also explain the slightly higher $[\alpha]_D$ recorded by Mori et al. for their synthetic (+)-**1a**. Additionally, Mori has also recorded his $[\alpha]_D$ of -82 ° (*c* 0.24, MeOH) at 17 °C for the optically pure (*S*)-enantiomer, while we recorded our $[\alpha]_D$ value of +88.8° (*c* 0.26, MeOH) at 20 °C for the (*R*)-enantiomer, which compares well with the even

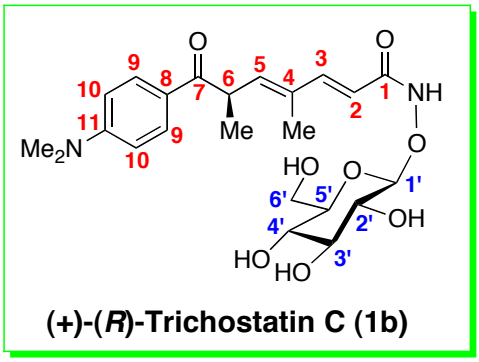
higher $[\alpha]_D$ of +96 ^{o} (*c* 0.31, MeOH) Mori recorded for (+)-**1a** at the higher temperature of 22 ^{o}C. Given the aforementioned conversion of (+)-trichostatin A into (+)-trichostatin C (**1b**), and us not detecting any (6*S*)-diastereomer in that reaction mixture, we consider that our synthetic (+)-**1a** is essentially optically pure.

The ^{1}H and ^{13}C spectral data that we obtained for fully synthetic (+)-trichostatin C (**1b**) derived from the synthetic (+)-trichostatic acid (**22**) of this route is shown opposite, in Tabular format, along with the 2D and DEPT ^{13}C NMR assignments, and comparisons with the spectral data reported previously for (+)-trichostatin C (**1b**).



(+)-(*R*)-Trichostatin C (1b)

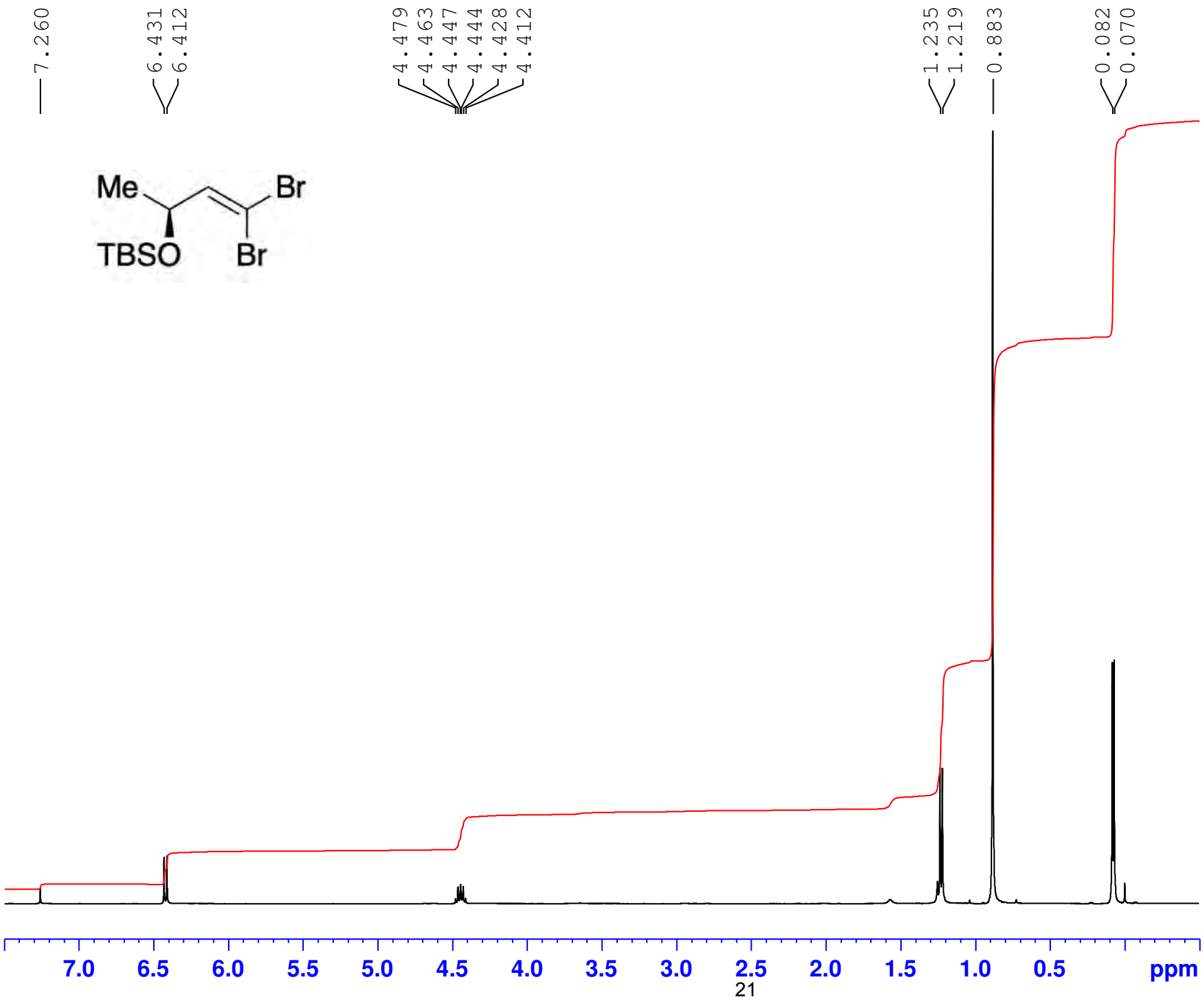
^{1}H Assignment	This work Hale, Pan and Manaviazar 600.13 MHz ^{1}H NMR (CD_3OD) (ref. δ 3.31 ppm)	Xu and Si[16] 600 MHz ^{1}H NMR (CD_3OD)	Thorsen[20b] 400 MHz ^{1}H NMR (CD_3OD)
H9	7.86 (d, *J* = 9.6 Hz, 2H)	7.86 (d, *J* = 9.2 Hz, 2H)	7.87 (d, *J* = 8.0 Hz, 2H)
H2	7.26 (d, *J* = 15.6 Hz, 1H)	7.27 (d, *J* = 9.2 Hz, 1H)	7.27 (d, *J* = 8.0 Hz, 1H)
H10	6.73 (d, *J* = 9.0 Hz, 2H)	6.72 (d, *J* = 9.2 Hz, 2H)	6.74 (d, *J* = 9.2 Hz, 2H)
H5	5.97 (d, *J* = 9.0 Hz, 1H)	5.97 (d, *J* = 9.6 Hz, 1H)	5.98 (d, *J* = 9.2 Hz, 1H)
H3	5.87 (d, *J* = 15.6 Hz, 1H)	5.87 (d, *J* = 15.6 Hz, 1H)	5.88 (d, *J* = 15.2 Hz, 1H)
H6	4.55 (d, *J* = 9.0 Hz, 1H)	-	4.54 (d, *J* = 8.4 Hz, 1H)
H1'	4.54 (dq, *J* = 9.6, 6.6 Hz, 1H)	4.54 (dq, *J* = 9.6, 6.0 Hz, 1H)	4.55 (m, 1H)
H6a'	3.91 (dd, *J* = 11.4, 2.4 Hz, 1H)	3.91 (dd, *J* = 12.0, 2.4 Hz, 1H)	3.91 (dd, *J* = 12.4, 2.4 Hz, 1H)
H6b'	3.67 (dd, *J* = 12.0, 6.6 Hz, 1H)	3.67 (dd, *J* = 12.0, 6.0 Hz, 1H)	3.66 (dd, *J* = 11.4, 6.4 Hz, 1H)
H3'	3.41 (dd, *J* = 9.0, 9.0 Hz, 1H)	3.40 (t, *J* = 9.0 Hz, 1H)	3.42 (t, *J* = 9.2 Hz, 1H)
H5'	3.33 (m, 1H)	3.34 (m, 1H)	3.34 (m, 1H)
H2'	3.32 (m, 1H)	3.32 (m, 1H)	3.31 (m, 1H)
H4'	3.28 (m, 1H)	3.28 (m, 1H)	3.25 (m, 1H)
NMe_2	3.06 (s, 3H)	3.06 (s, 3H)	3.07 (s, 3H)
C4-Me	1.93 (s, 3H)	1.93 (s, 3H)	1.94 (s, 3H)
C6-Me	1.28 (d, *J* = 7.2 Hz, 3H)	1.27 (d, *J* = 6.0 Hz, 3H)	1.29 (d, *J* = 6.8 Hz, 3H)



(+)-(*R*)-Trichostatin C (1b)

^{13}C Assignment	This work Hale, Pan and Manaviazar 150.92 MHz ^{13}C NMR (CD_3OD) (ref. δ 49.0 ppm)	Xu and Si[16] 150 MHz ^{13}C NMR (CD_3OD)	Thorsen[20b] 100 MHz ^{13}C NMR (CD_3OD)	Tsuji and Kobayashi[20a] 15 MHz ^{13}C NMR (CD_3OD)
C7	201.3	201.4	201.5	201.3
C1	166.9	167.1	167.0	168.0
C11	155.5	155.6	155.6	155.6
C2	147.6	147.8	147.8	147.8
C5	142.5	142.7	142.6	142.3
C8	134.3	134.4	134.5	134.5
C9	131.9	132.1	132.1	131.9
C4	124.7	124.8	124.9	124.9
C3	116.2	116.3	116.3	116.3
C10	111.9	112.1	112.1	112.0
C1'	107.7	107.8	107.8	107.9
C5'	78.4	78.5	78.5	78.4
C3'	77.5	77.6	77.7	77.7
C2'	73.1	73.2	73.3	73.2
C4'	71.3	71.4	71.5	71.5
C6'	62.8	62.9	62.9	62.9
C6	41.7	41.8	41.9	41.9
NMe_2	40.0	40.2	40.3	40.1
C6-Me	18.3	18.4	18.4	18.3
C4-Me	12.7	12.9	12.9	12.8

Part B. Copies of the ^{1}H NMR, ^{13}C NMR, IR and HRMS Spectra For All Synthetic Intermediates in the Route to (+)-(*R*)-Trichostatin A. Spectra for the (+)-(*R*)-Trichostatin C Synthesised from the (+)-(*R*)-Trichostatic Acid Prepared By This Route. TLC Analyses of Key Reactions



BRUKER

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AQ 5.4525952 sec
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DE 9.85 usec
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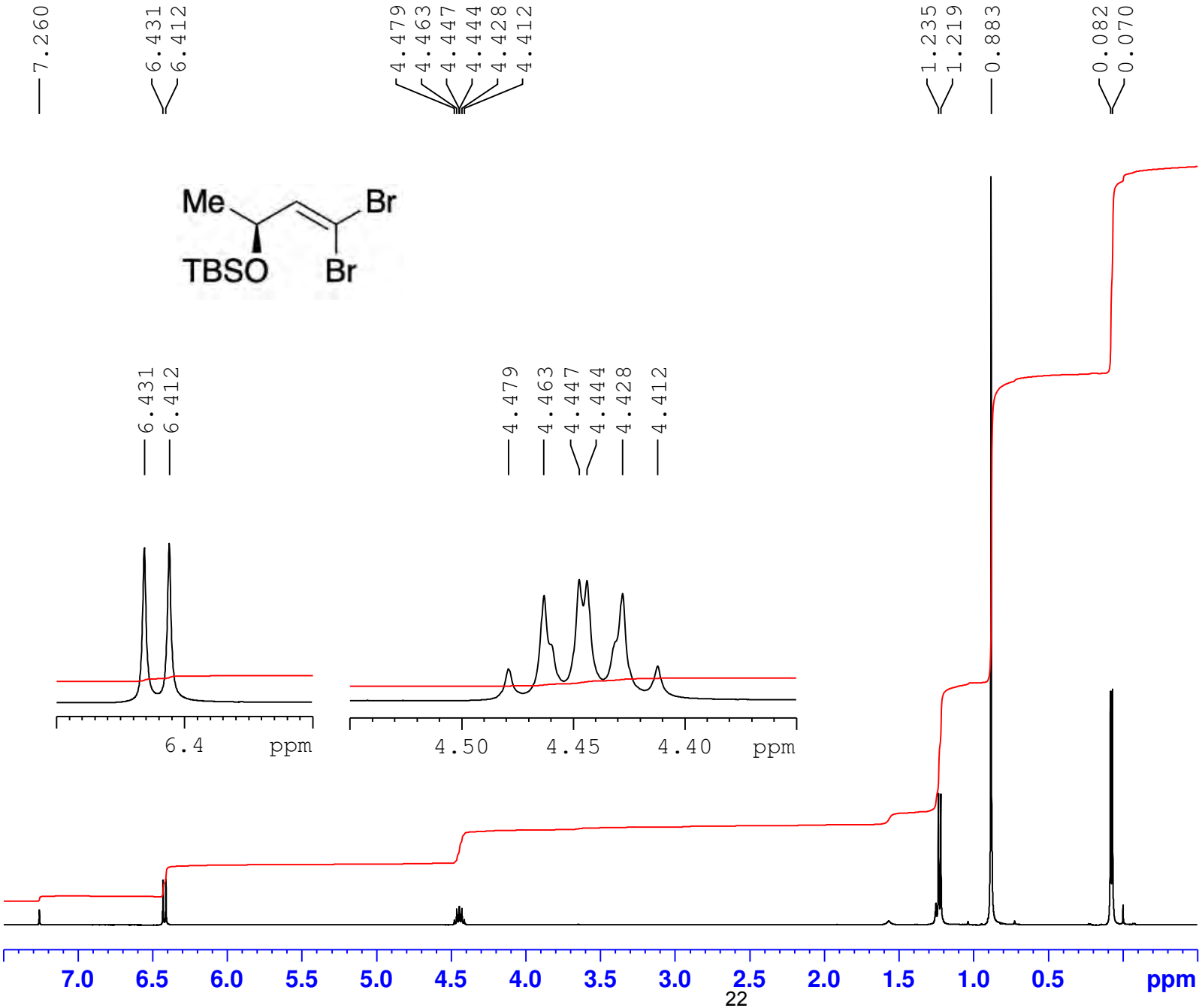

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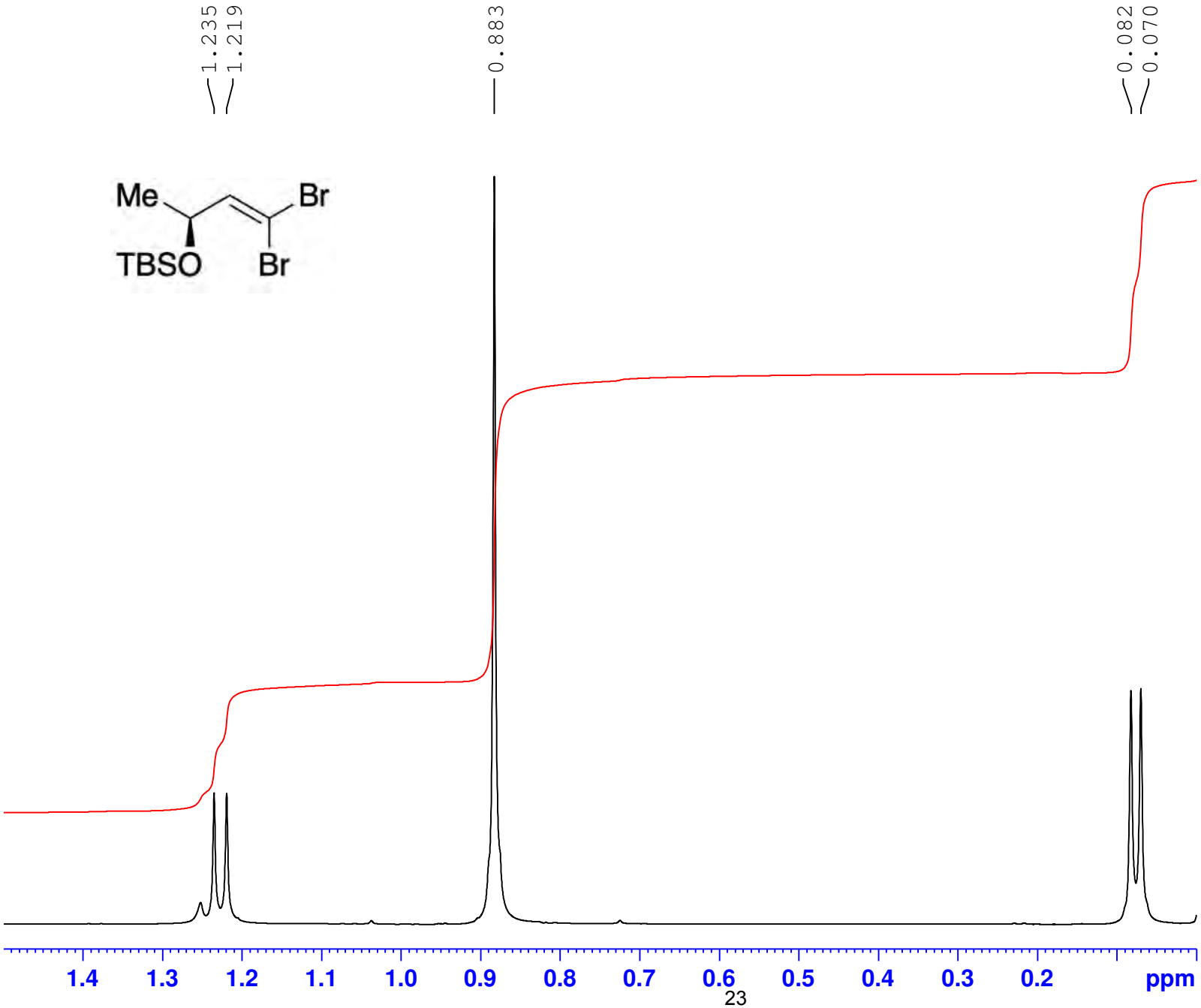

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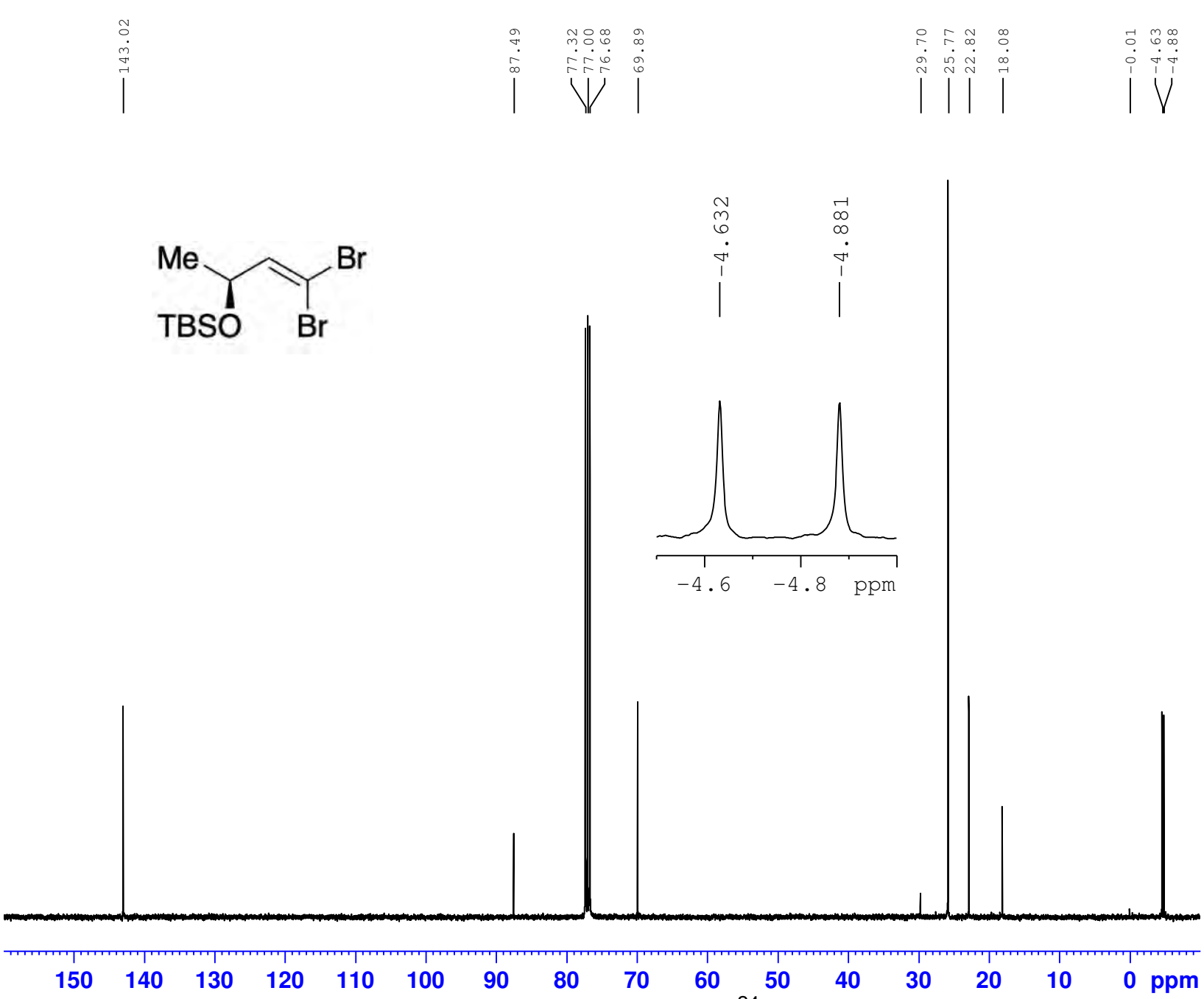


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PC                 1.40
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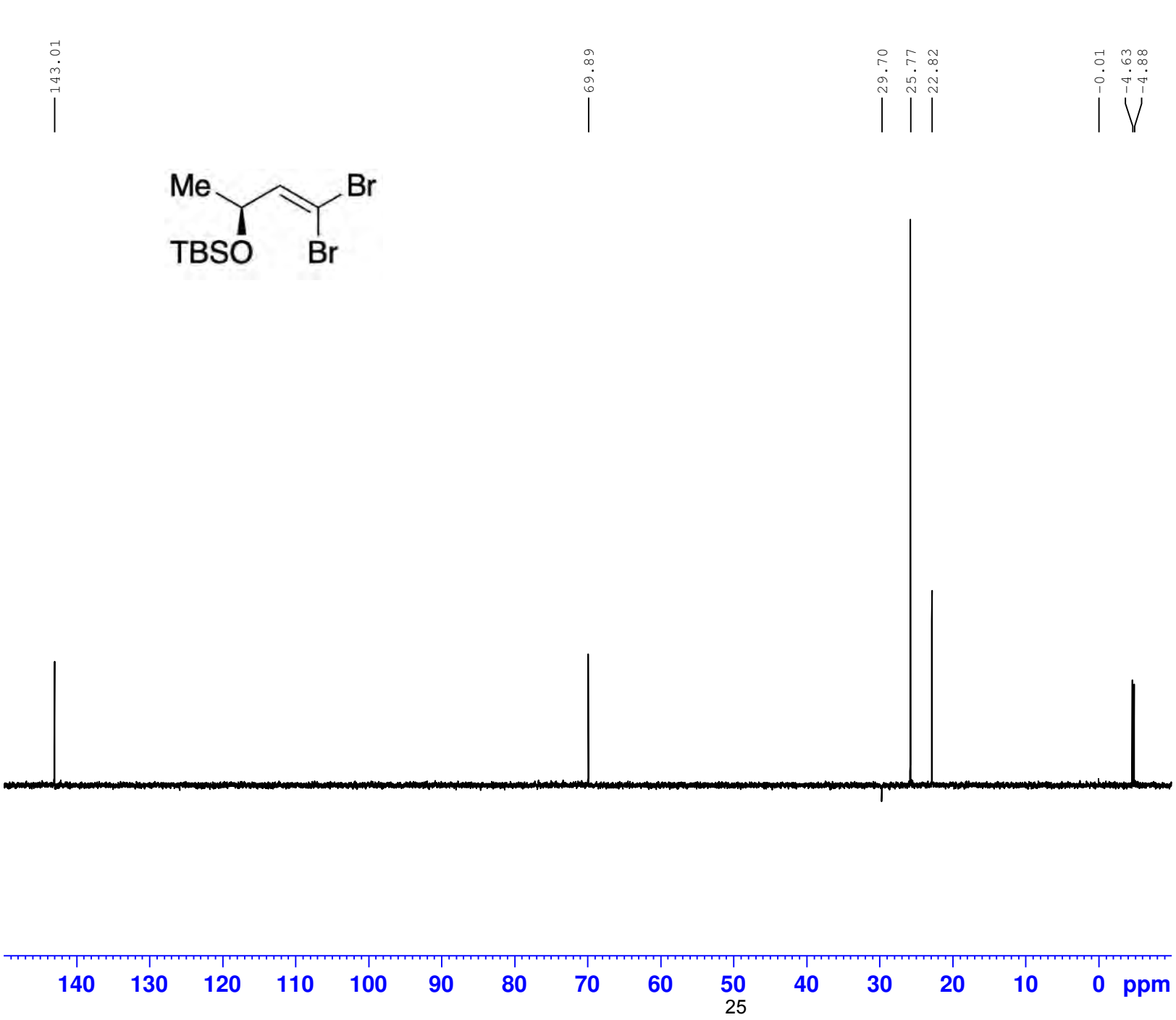


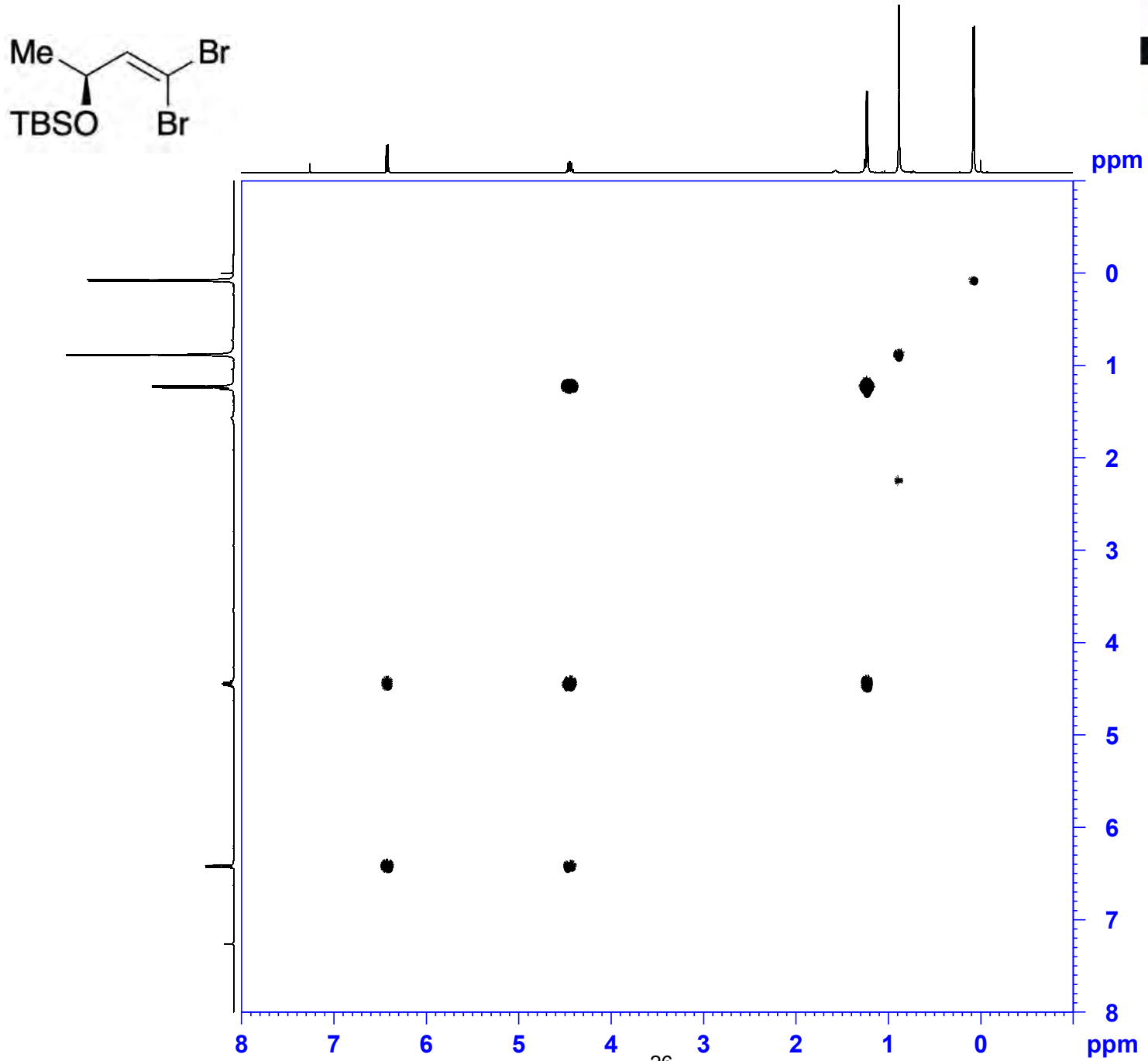
Current Data Parameters
NAME I-PK-33PURE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180116
Time 7.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.2 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549369 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME         I-PK-33PURE
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180116
Time                7.09
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                  2048
SOLVENT            CDCl3
NS                     1
DS                     8
SWH             4708.098 Hz
FIDRES          2.298876 Hz
AQ              0.2174976 sec
RG                  2050
DW                106.200 usec
DE                  6.50 usec
TE                 295.7 K
D0            0.00000300 sec
D1            0.93364388 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00021260 sec

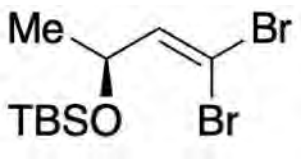
======== CHANNEL f1 ========
SFO1         399.9008997 MHz
NUC1                   1H
P1                 14.88 usec
P17              2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1                16.00 %
GPZ2                12.00 %
GPZ3                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            399.9009 MHz
FIDRES         36.747414 Hz
SW                11.762 ppm
FnMODE                QF

F2 - Processing parameters
SI                   1024
SF           399.9000106 MHz
WDW                  SINE
SSB       0
LB        0 Hz
GB        0
PC                   1.40

F1 - Processing parameters
SI                   1024
MC2                    QF
SF           399.9000107 MHz
WDW                  SINE
SSB       0
LB        0 Hz
GB        0
```




```
Current Data Parameters
NAME          I-PK-33PURE
EXPNO                  14
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180116
Time                7.16
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT             CDCl3
NS                     2
DS                    32
SWH              4807.692 Hz
FIDRES           4.695012 Hz
AQ             0.1064960 sec
RG                  2050
DW                104.000 usec
DE                  6.50 usec
TE                 295.8 K
CNST2         145.0000000
D0              0.00000300 sec
D1              0.80000001 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
D21             0.00360000 sec
IN0             0.00001910 sec

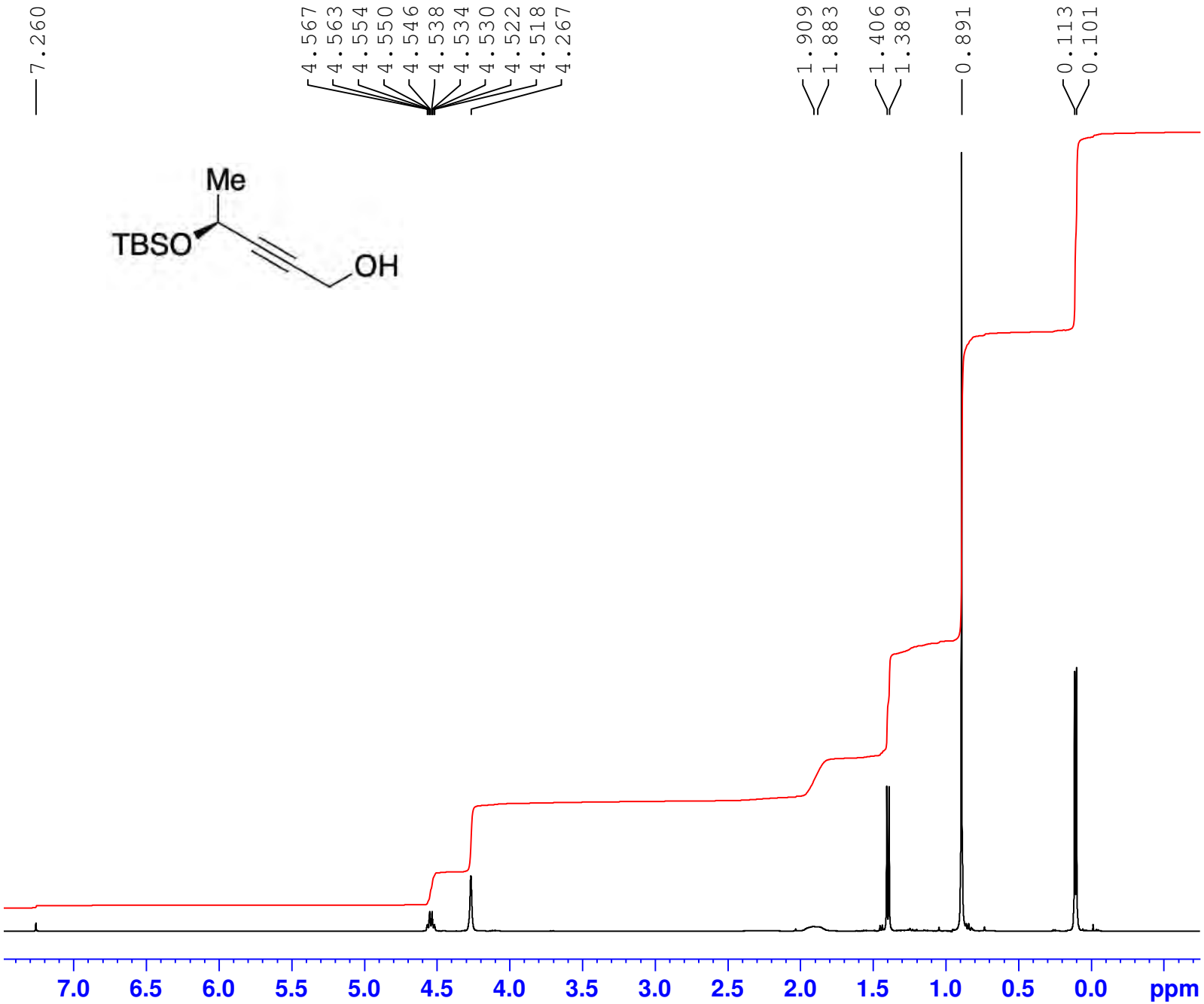
======== CHANNEL f1 ========
SFO1          399.9018806 MHz
NUC1                  1H
P1                 14.88 usec
P2                 29.76 usec
P28       0 usec
PLW1           7.59999990 W

======== CHANNEL f2 ========
SFO2          100.5670016 MHz
NUC2                 13C
CPDPRG[2            garp4
P3                 10.00 usec
P14               500.00 usec
P31              1900.00 usec
PCPD2              80.00 usec
PLW0       0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3   0 Hz
SPW3           6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18             0.500
SPOFFS18 0 Hz
SPW18          1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1             100.567 MHz
FIDRES        204.515701 Hz
SW               260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           399.9000099 MHz
WDW                 OSINE
```



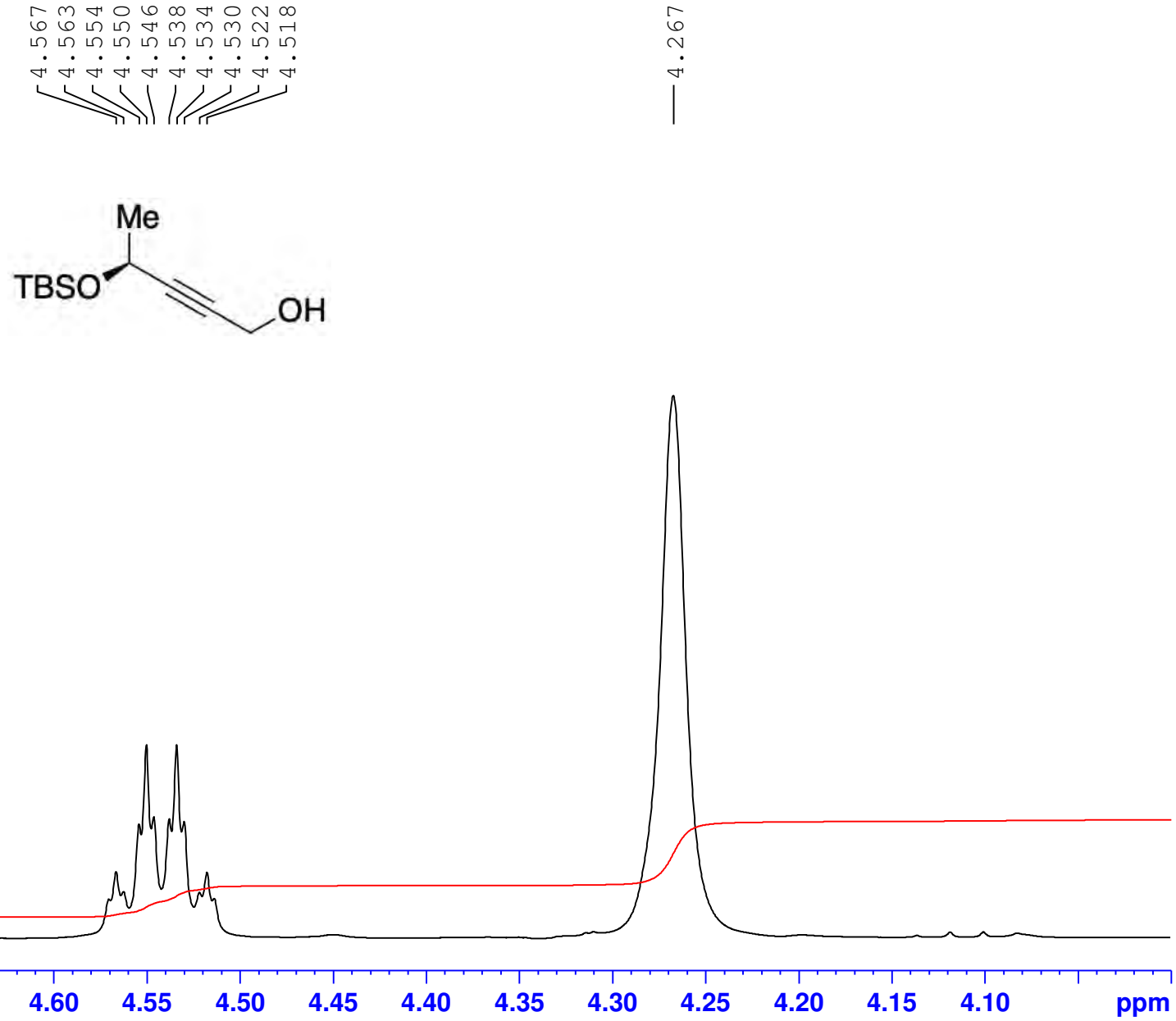
BRUKER

Current Data Parameters
NAME I-PK-13
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171108
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 32
DW 41.600 usec
DE 10.58 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 11.40 usec
PLW1 4.09999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



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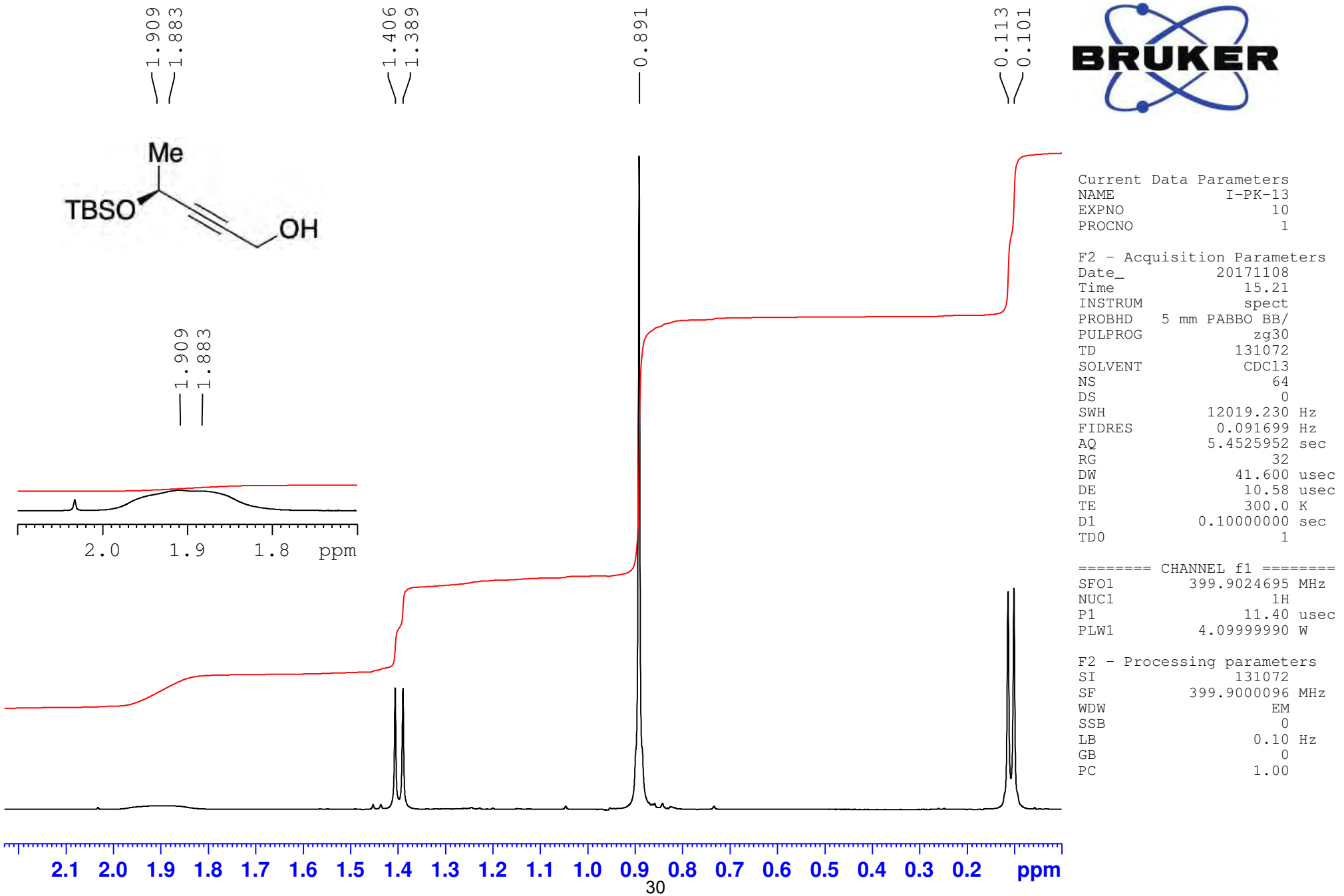
```
Current Data Parameters
NAME                  I-PK-13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20171108
Time                    15.21
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                    131072
SOLVENT                CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                 5.4525952 sec
RG                         32
DW                    41.600 usec
DE                    10.58 usec
TE                    300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                    11.40 usec
PLW1              4.09999990 W

F2 - Processing parameters
SI                    131072
SF               399.9000096 MHz
WDW                        EM
SSB                         0
LB                     0.10 Hz
GB                          0
PC                     1.00
```



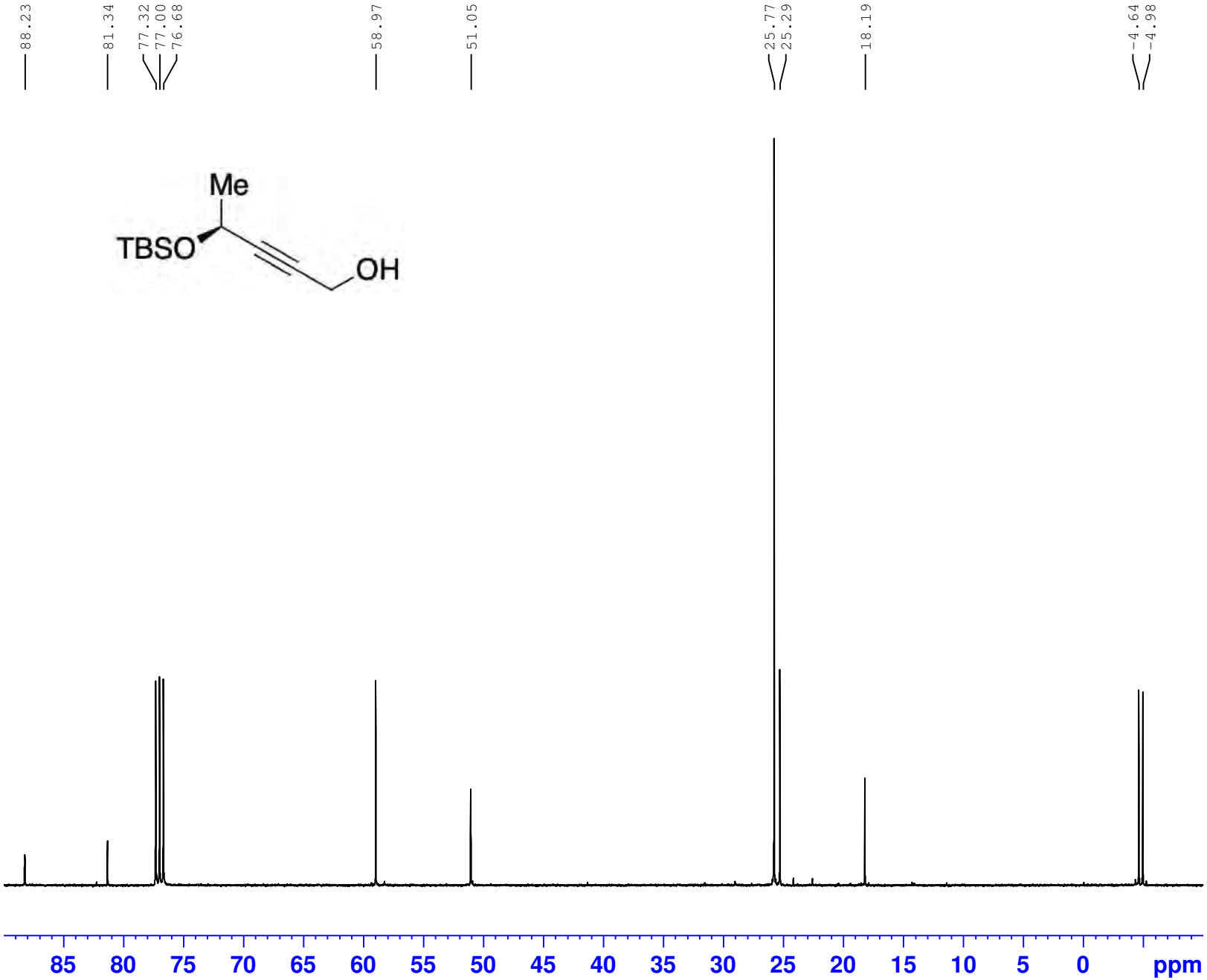
BRUKER

```
Current Data Parameters
NAME                  I-PK-13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20171108
Time                   15.21
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                   131072
SOLVENT               CDCl3
NS                        64
DS                         0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                 5.4525952 sec
RG                        32
DW                    41.600 usec
DE                    10.58 usec
TE                    300.0 K
D1                 0.10000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                    11.40 usec
PLW1              4.09999990 W

F2 - Processing parameters
SI                   131072
SF               399.9000096 MHz
WDW                      EM
SSB                       0
LB                     0.10 Hz
GB                        0
PC                     1.00
```



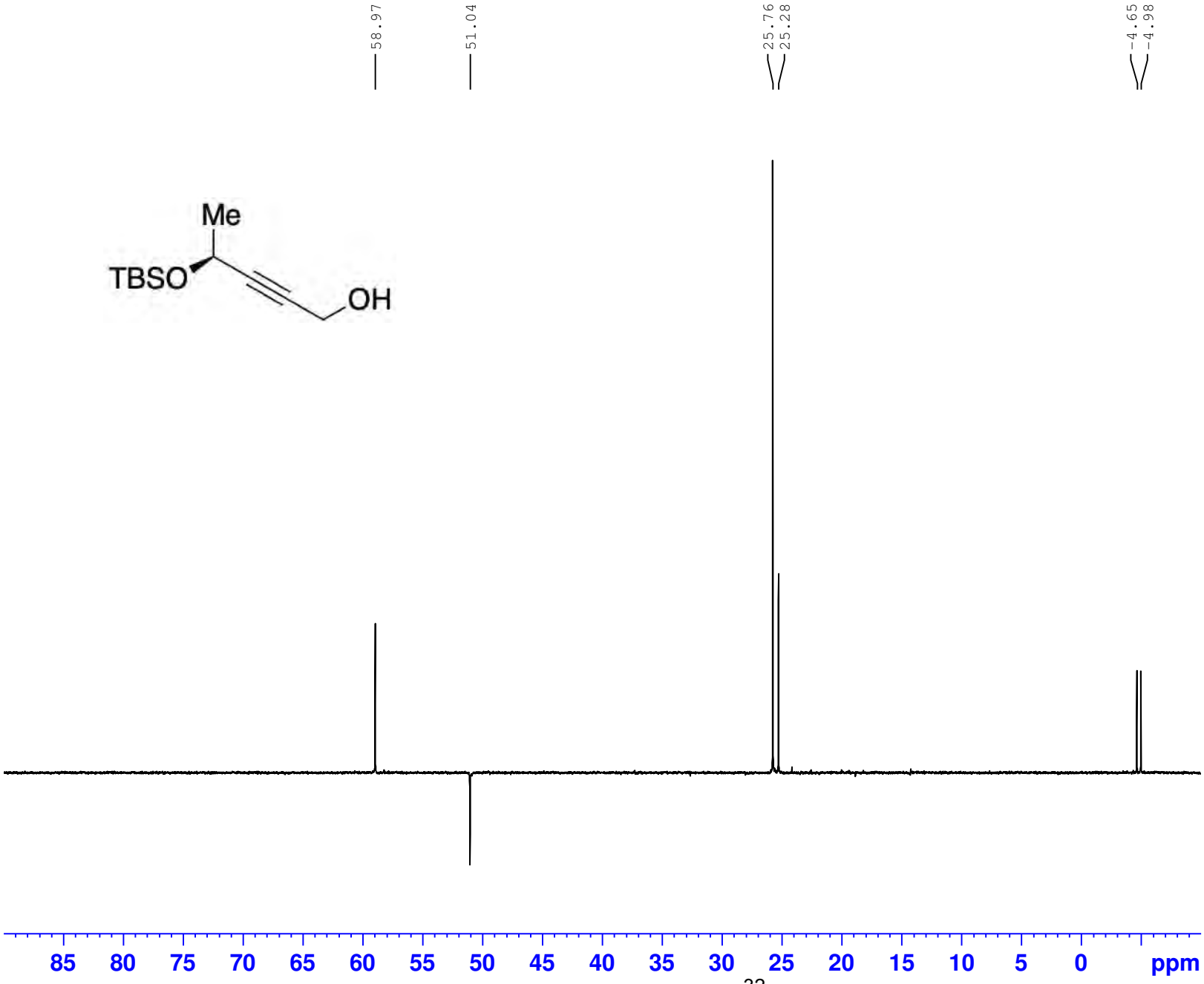
Current Data Parameters
NAME I-PK-13
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171108
Time 16.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1300
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 17.37999916 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 4.09999990 W
PLW12 0.06578200 W
PLW13 0.05328400 W

F2 - Processing parameters
SI 131072
SF 100.5549380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



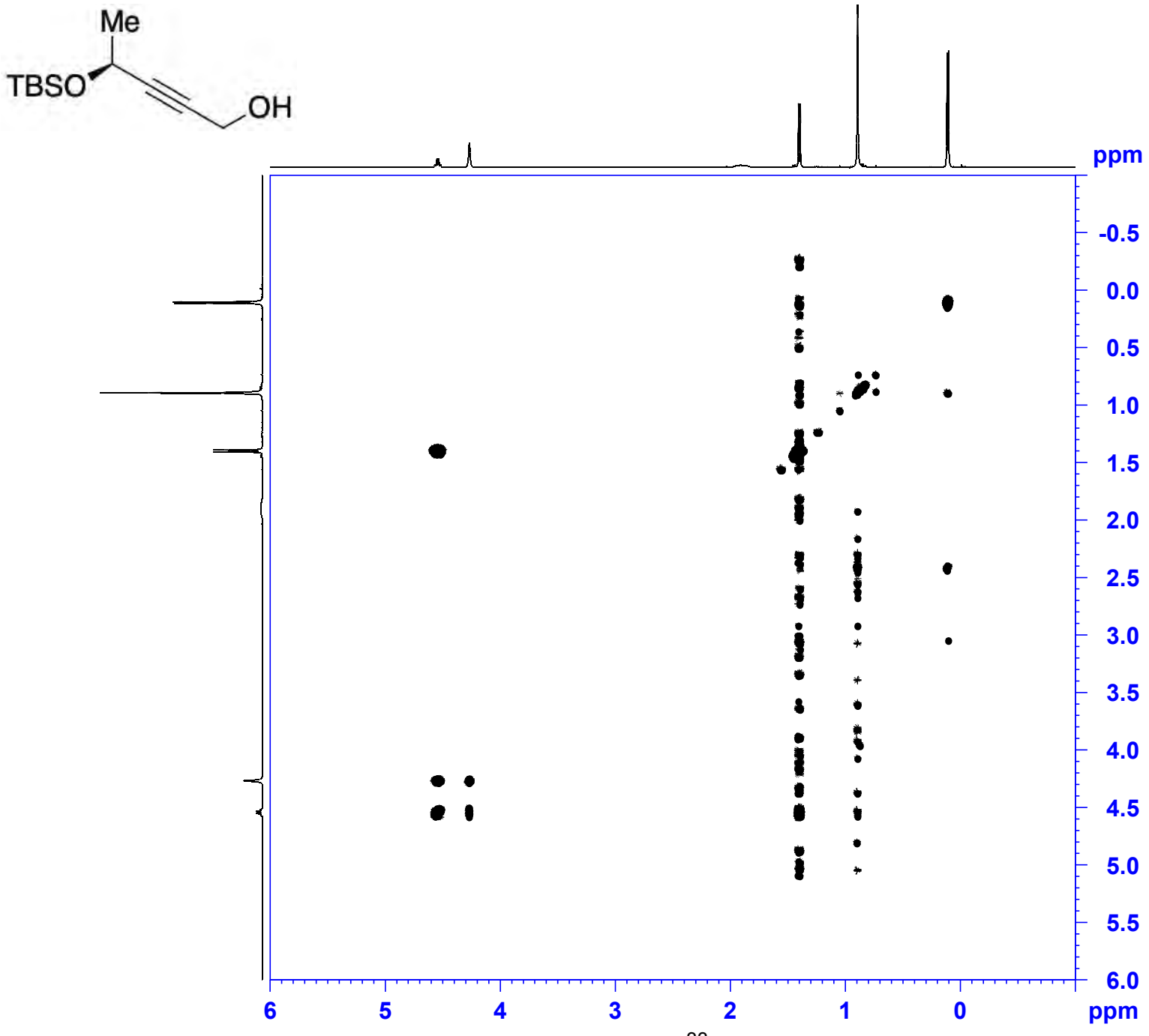
Current Data Parameters
NAME I-PK-13
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171108
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 17.37999916 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 2.65549994 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 11.40 usec
P4 22.80 usec
PCPD2 90.00 usec
PLW2 4.09999990 W
PLW12 0.06578200 W

F2 - Processing parameters
SI 32768
SF 100.5549386 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                  I-PK-13
EXPNO                      13
PROCNO                      1

F2 - Acquisition Parameters
Date_                20171108
Time                    16.56
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                       2048
SOLVENT                 CDCl3
NS                          1
DS                          8
SWH                2167.630 Hz
FIDRES             1.058413 Hz
AQ                 0.4724053 sec
RG                       2050
DW                  230.667 usec
DE                    6.50 usec
TE                   300.0 K
D0                 0.00000300 sec
D1                 0.64077991 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00046120 sec

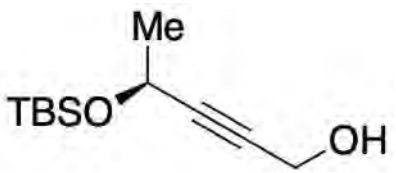
======== CHANNEL f1 ========
SFO1            399.9009750 MHz
NUC1                      1H
P1                     11.40 usec
P17                  2500.00 usec
PLW1             4.09999990 W
PLW10            0.78821999 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPNAM[3]       SMSQ10.100
GPZ1                  16.00 %
GPZ2                  12.00 %
GPZ3                  40.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                399.901 MHz
FIDRES            16.939507 Hz
SW                    5.422 ppm
FnMODE                   QF

F2 - Processing parameters
SI                       1024
SF              399.9000087 MHz
WDW                      SINE
SSB          0
LB           0 Hz
GB           0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2                      QF
SF              399.9000101 MHz
WDW                      SINE
SSB          0
LB           0 Hz
GB           0
```



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```
Current Data Parameters
NAME                  I-PK-13
EXPNO                      14
PROCNO                      1

F2 - Acquisition Parameters
Date_              20171108
Time                  17.03
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                      1024
SOLVENT               CDCl3
NS                         2
DS                        32
SWH                4807.692 Hz
FIDRES             4.695012 Hz
AQ                0.1064960 sec
RG                     2050
DW                  104.000 usec
DE                    6.50 usec
TE                   300.0 K
CNST2           145.0000000
D0                0.00000300 sec
D1                0.80000001 sec
D4                0.00172414 sec
D11               0.03000000 sec
D16               0.00020000 sec
D21               0.00360000 sec
IN0               0.00001910 sec

======== CHANNEL f1 ========
SFO1            399.9018806 MHz
NUC1                    1H
P1                    11.40 usec
P2                    22.80 usec
P28         0 usec
PLW1             4.09999990 W

======== CHANNEL f2 ========
SFO2            100.5670016 MHz
NUC2                   13C
CPDPRG[2              garp4
P3                    10.00 usec
P14                  500.00 usec
P31                 1900.00 usec
PCPD2                 80.00 usec
PLW0        0 W
PLW2           17.37999916 W
PLW12           0.27156001 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                0.500
SPOFFS3   0 Hz
SPW3            2.65549994 W
SPNAM[18  Crp60_xfilt.2
SPOAL18               0.500
SPOFFS18 0 Hz
SPW18           0.63628000 W

====== GRADIENT CHANNEL =====
GPNAM[1]        SMSQ10.100
GPNAM[2]        SMSQ10.100
GPZ1                  80.00 %
GPZ2                  20.10 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                100.567 MHz
FIDRES          204.515701 Hz
SW                  260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      1024
SF           399.9000095 MHz
```

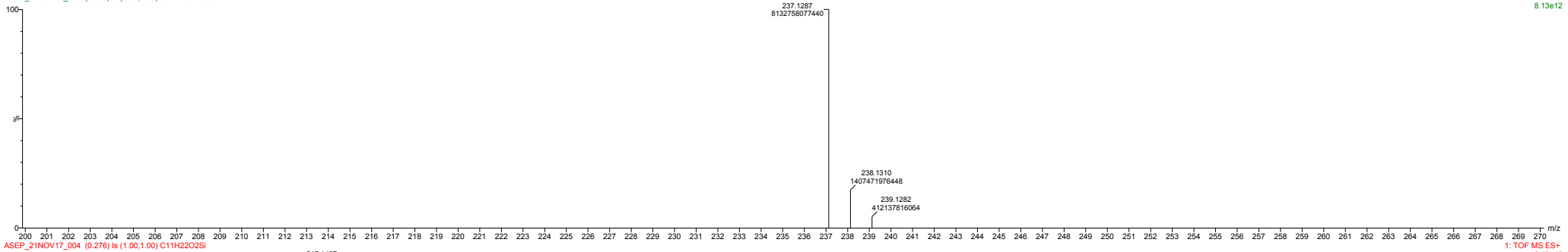
Mass Spectrometry Result Sheet

Waters Xevo G2-XS QTof Mass Spectrometer

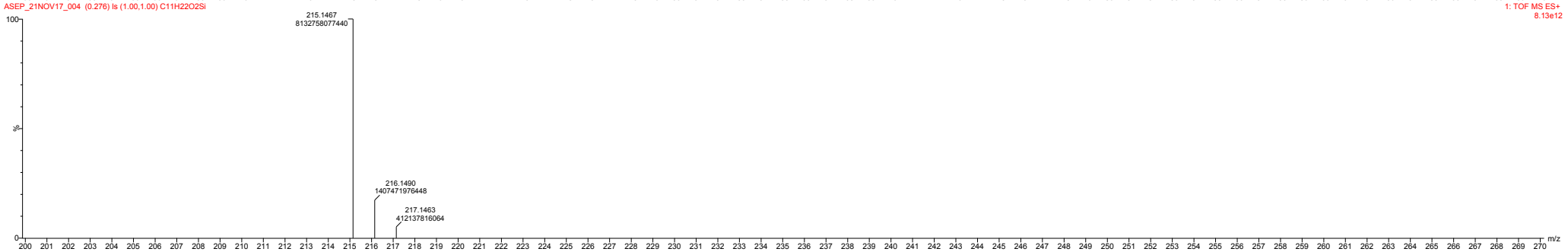
09-11-2017

I-PK-13

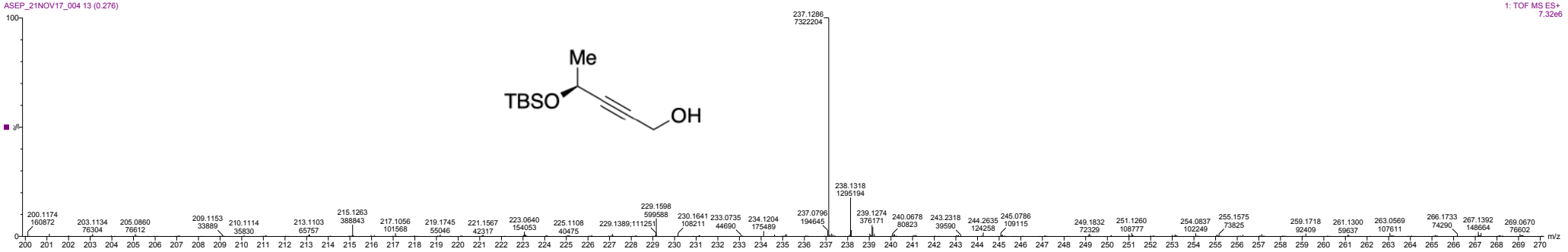
ASEP_21NOV17_004 (0.053) Is (1.00,1.00) C11H22O2SiNa

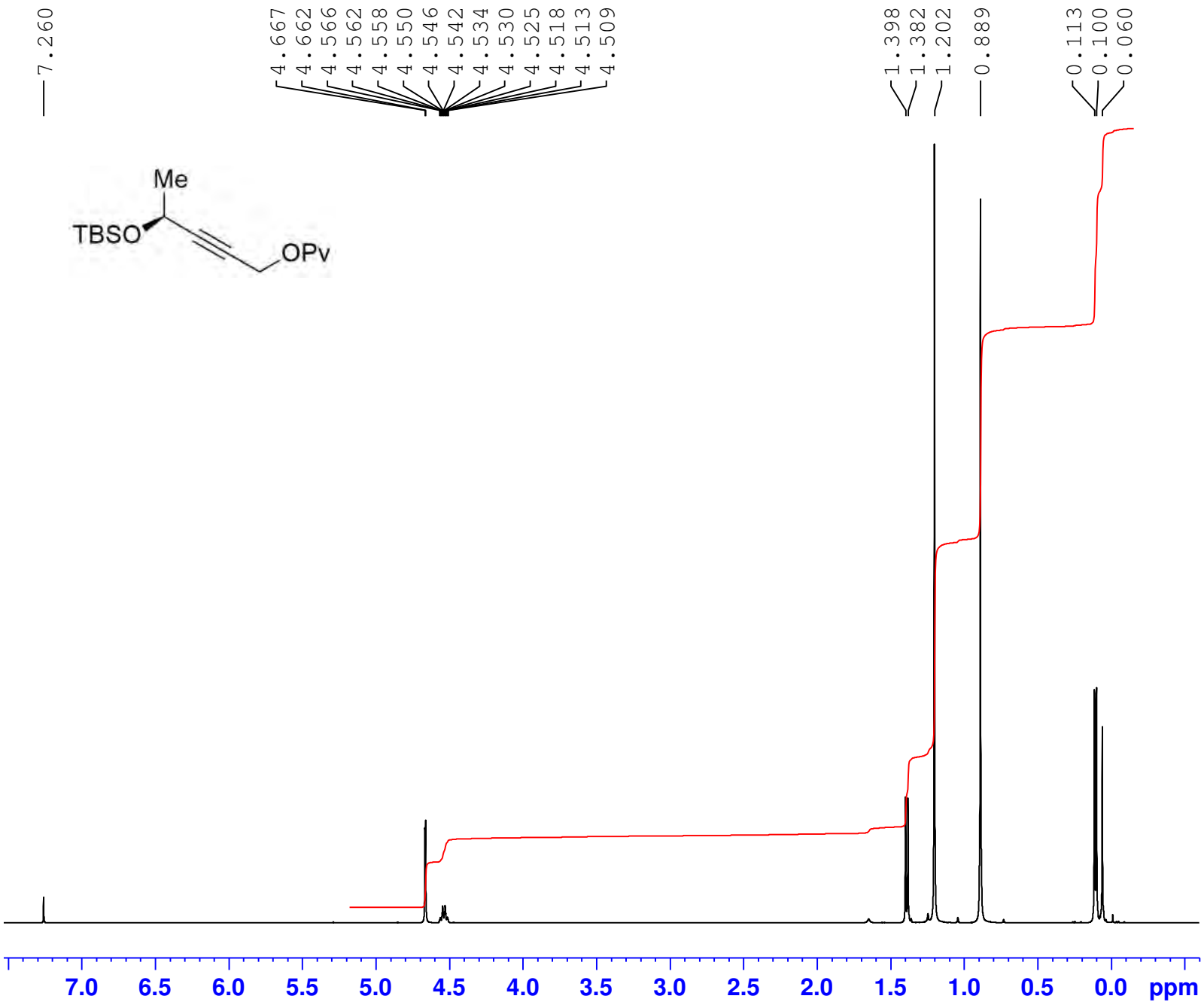


ASEP_21NOV17_004 (0.276) Is (1.00,1.00) C11H22O2Si



ASEP_21NOV17_004 13 (0.276)





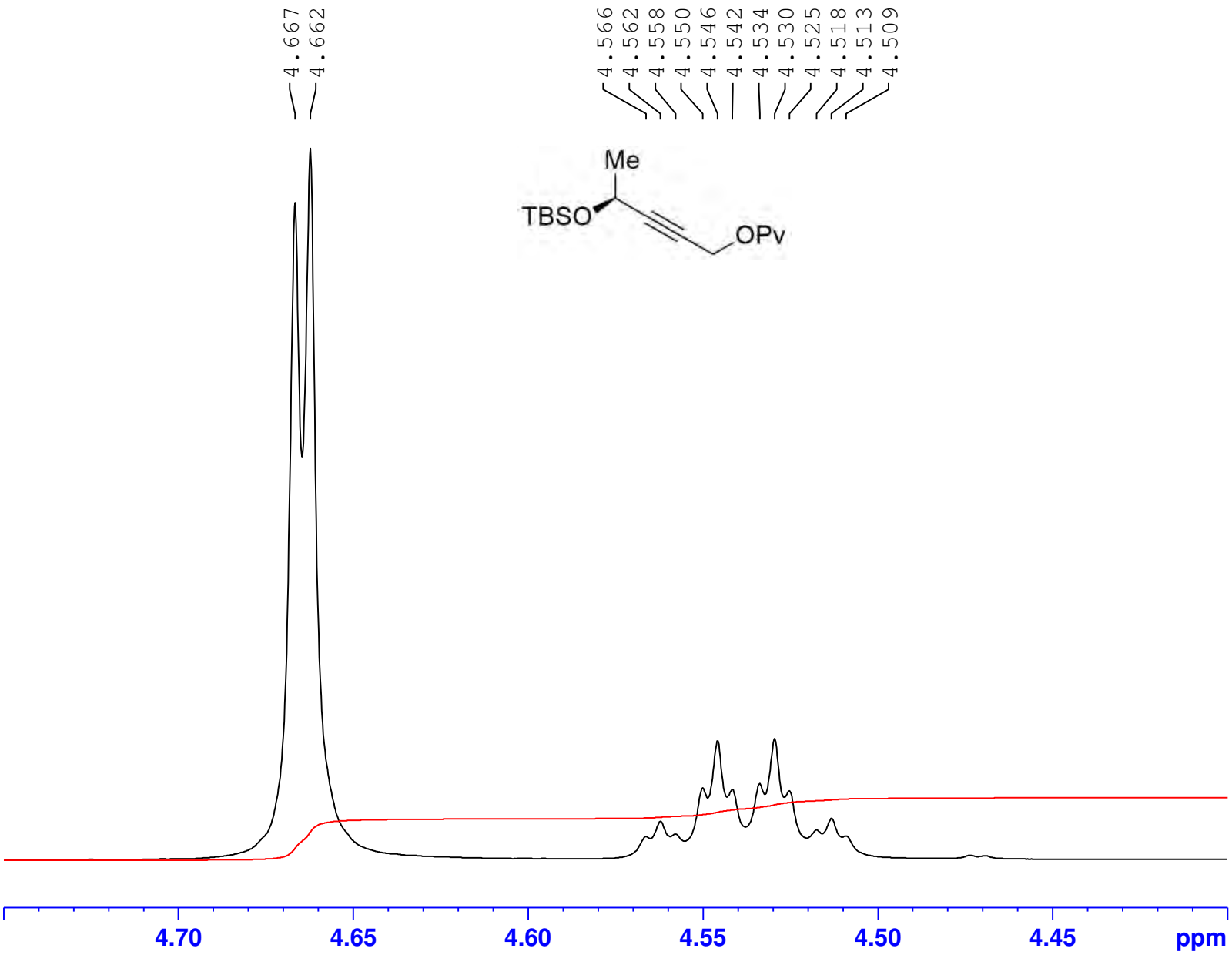
BRUKER

Current Data Parameters
NAME I-PK-49PURRE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180315
Time 18.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 32
DW 41.600 usec
DE 9.85 usec
TE 297.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000098 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

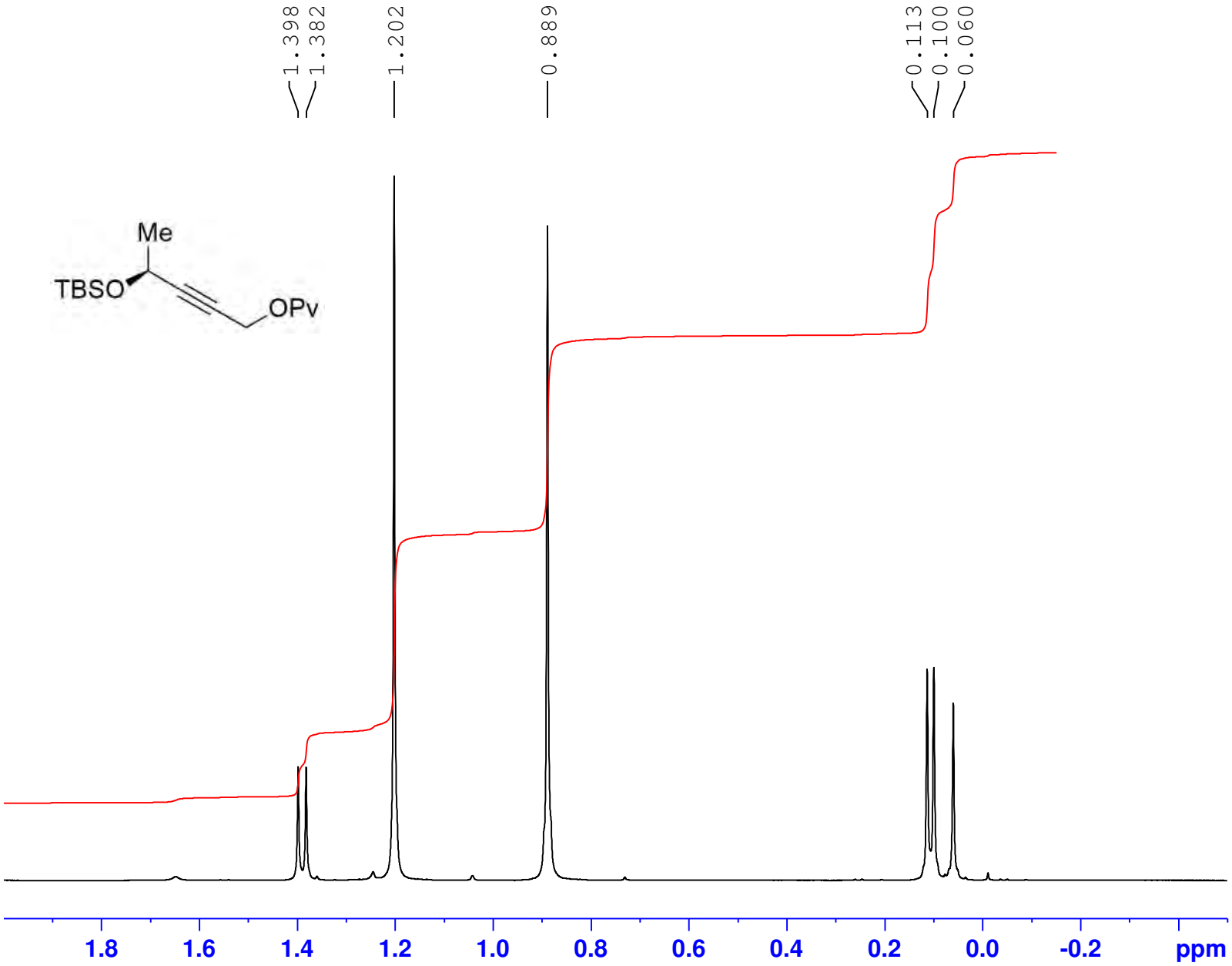


Current Data Parameters
NAME I-PK-49PURRE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180315
Time 18.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 32
DW 41.600 usec
DE 9.85 usec
TE 297.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000098 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

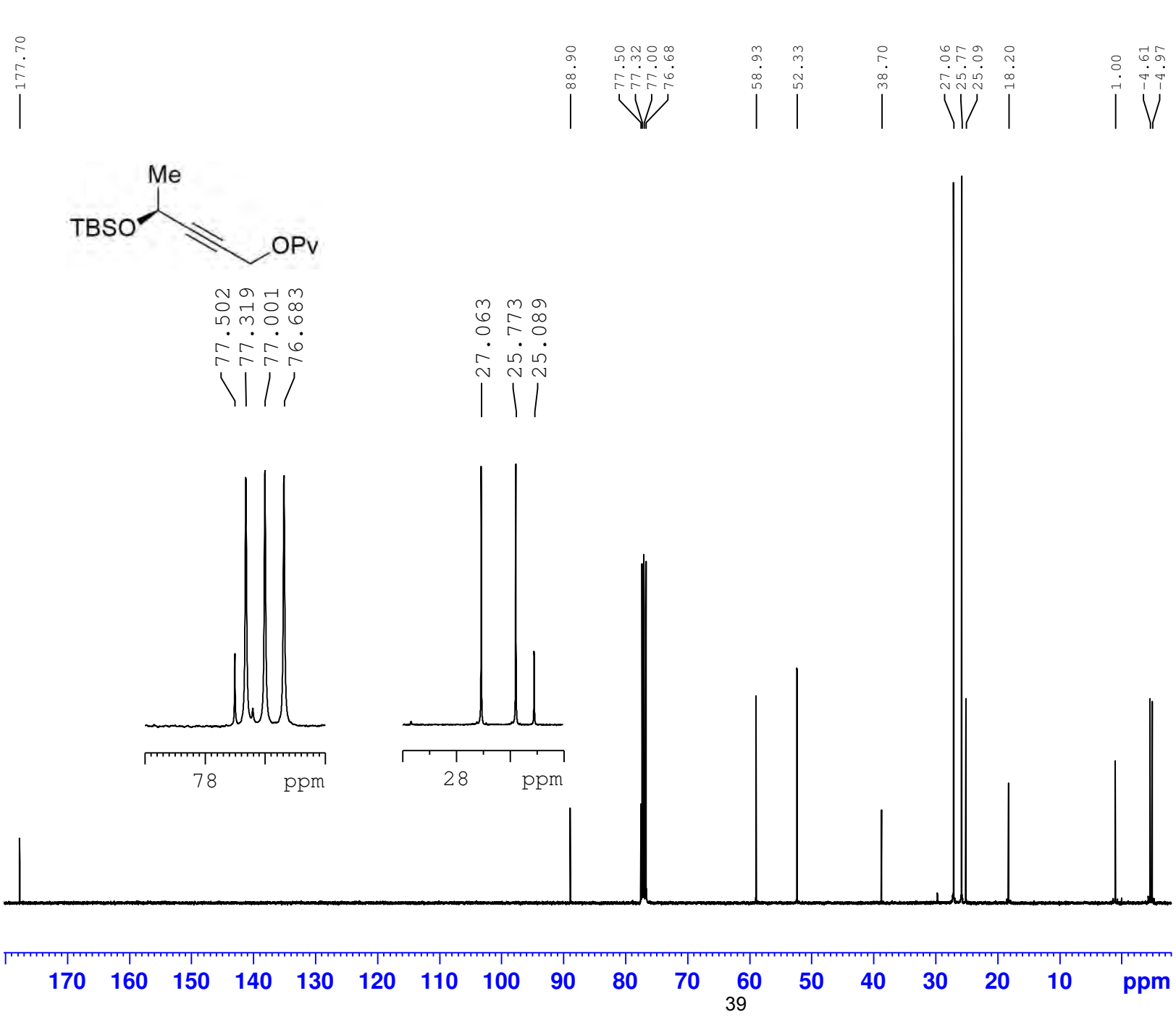



```
Current Data Parameters
NAME        I-PK-49PURRE
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180315
Time               18.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            12019.230 Hz
FIDRES          0.091699 Hz
AQ             5.4525952 sec
RG                    32
DW                41.600 usec
DE                 9.85 usec
TE                297.4 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                   1H
P1                 14.88 usec
PLW1          7.59999990 W

F2 - Processing parameters
SI                131072
SF          399.9000098 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



BRUKER

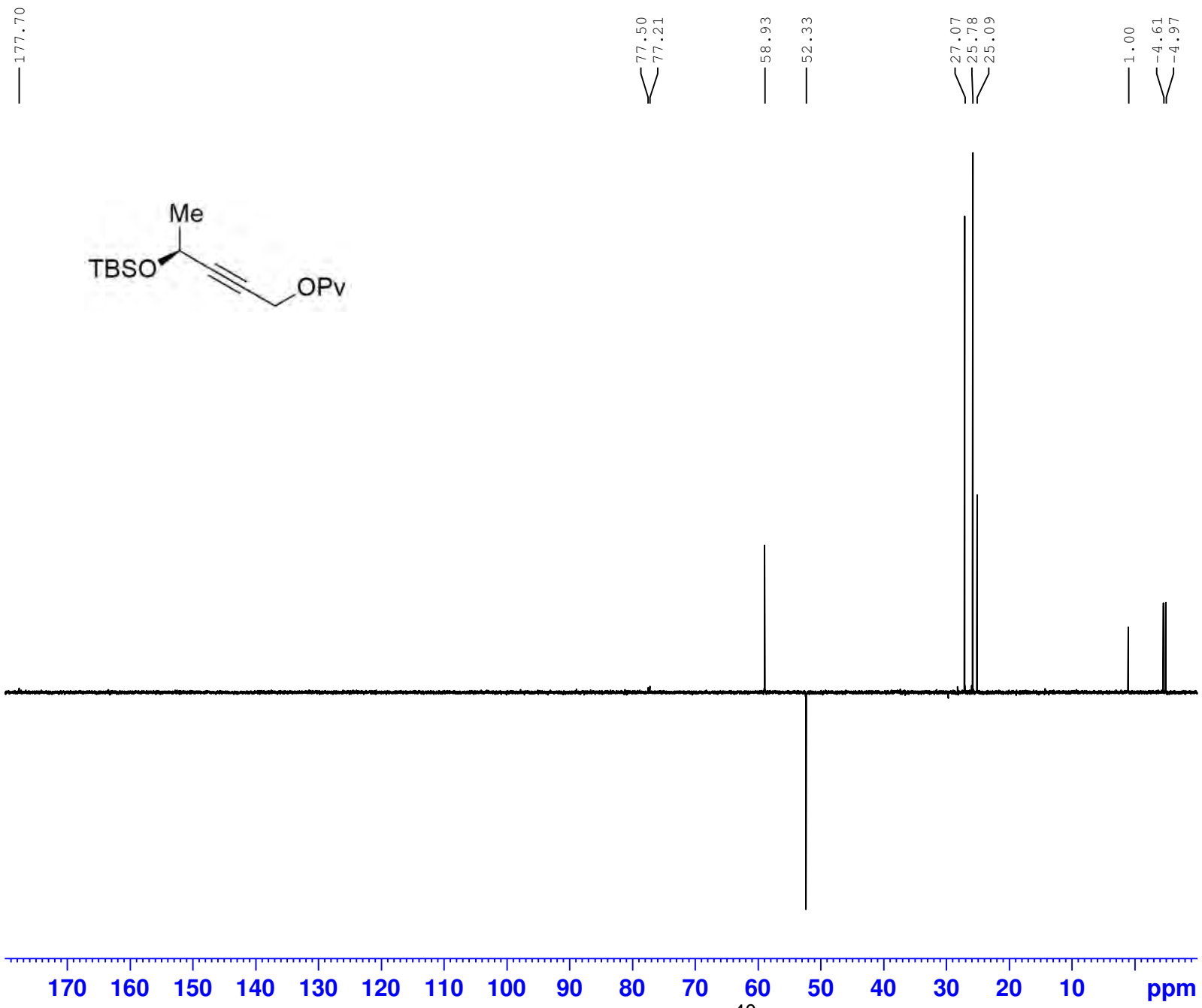
```
Current Data Parameters
NAME        I-PK-49PURRE
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180315
Time               19.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH          25000.000 Hz
FIDRES        0.210006 Hz
AQ            2.3808801 sec
RG                  2050
DW               20.000 usec
DE                9.12 usec
TE                298.5 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.5659947 MHz
NUC1                13C
P1               10.00 usec
PLW1        44.46300125 W

======== CHANNEL f2 ========
SFO2        399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2            90.00 usec
PLW2          7.59999990 W
PLW12         0.20774999 W
PLW13         0.16827001 W

F2 - Processing parameters
SI                131072
SF          100.5549364 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



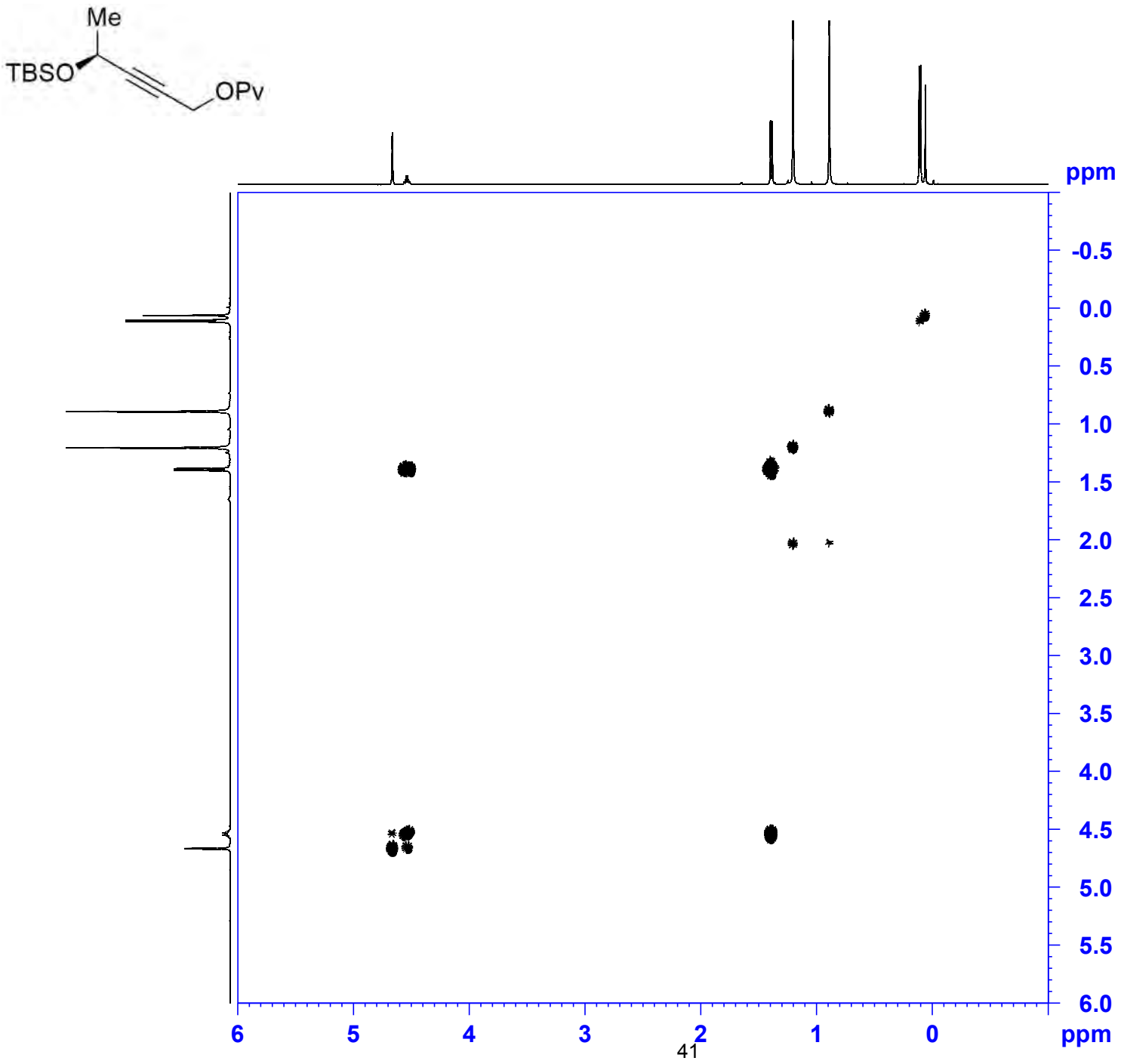
Current Data Parameters
NAME I-PK-49PURRE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180315
Time 20.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1820
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549361 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

```
Current Data Parameters
NAME        I-PK-49PURRE
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180315
Time                20.09
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                  2048
SOLVENT             CDCl3
NS                     1
DS                     8
SWH              4921.260 Hz
FIDRES           2.402959 Hz
AQ               0.2080768 sec
RG                  2050
DW                101.600 usec
DE                  6.50 usec
TE                297.4 K
D0             0.00000300 sec
D1             0.94388402 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00020340 sec

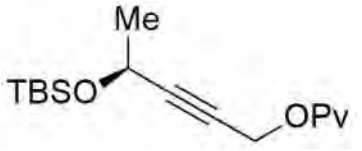
======== CHANNEL f1 ========
SFO1         399.9008189 MHz
NUC1                 1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1             16.00 %
GPZ2             12.00 %
GPZ3             40.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            399.9008 MHz
FIDRES         38.409538 Hz
SW                12.294 ppm
FnMODE               QF

F2 - Processing parameters
SI                  1024
SF           399.9000086 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           399.9000081 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```




```
Current Data Parameters
NAME        I-PK-49PURRE
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180315
Time              20.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                 1024
SOLVENT            CDC13
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES          4.695012 Hz
AQ            0.1064960 sec
RG                  2050
DW               104.000 usec
DE                 6.50 usec
TE                297.5 K
CNST2        145.0000000
D0            0.00000300 sec
D1            0.80000001 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D21           0.00360000 sec
IN0           0.00001910 sec

======== CHANNEL f1 ========
SFO1         399.9018806 MHz
NUC1                   1H
P1                  14.88 usec
P2                  29.76 usec
P28       0 usec
PLW1          7.59999990 W

======== CHANNEL f2 ========
SFO2         100.5670016 MHz
NUC2                  13C
CPDPRG[2            garp4
P3                  10.00 usec
P14                500.00 usec
P31               1900.00 usec
PCPD2               80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3   0 Hz
SPW3          6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18             0.500
SPOFFS18 0 Hz
SPW18         1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1             100.567 MHz
FIDRES        204.515701 Hz
SW                260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
```

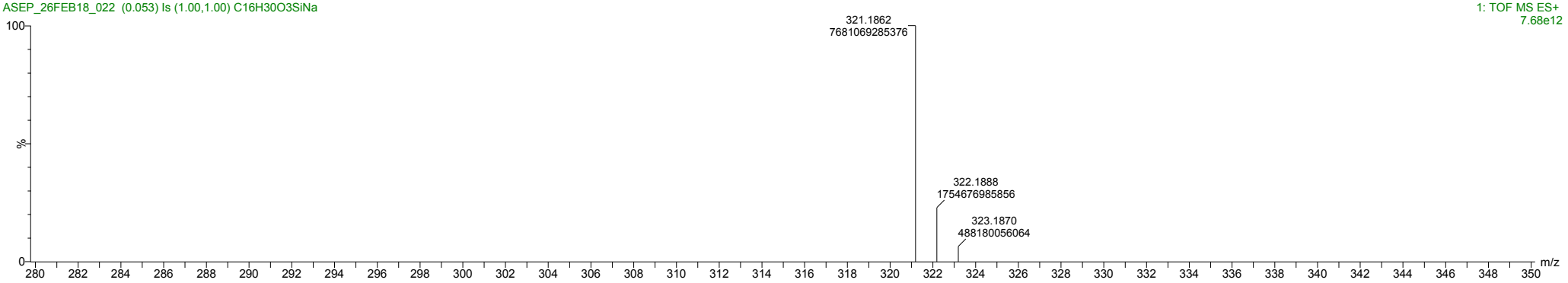
Mass Spectrometry Result Sheet

Waters Xevo G2-XS QTof Mass Spectrometer

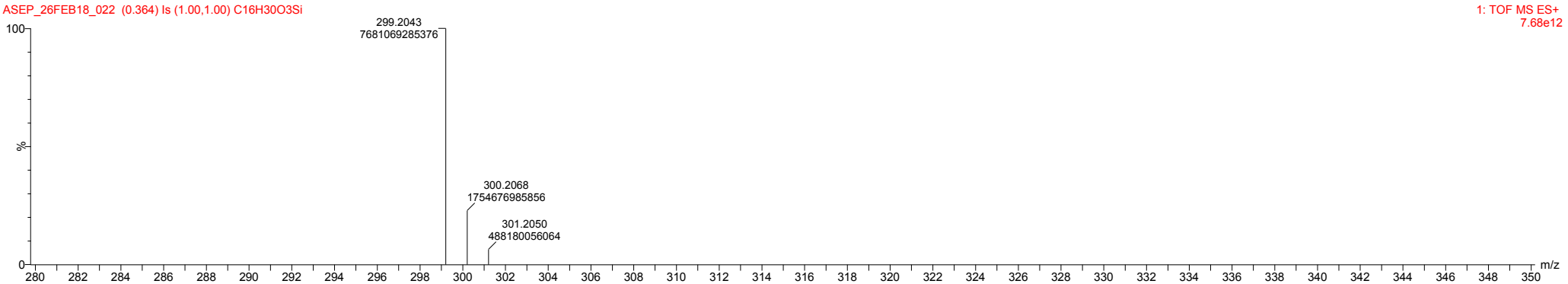
23-02-2018

I-PK-49

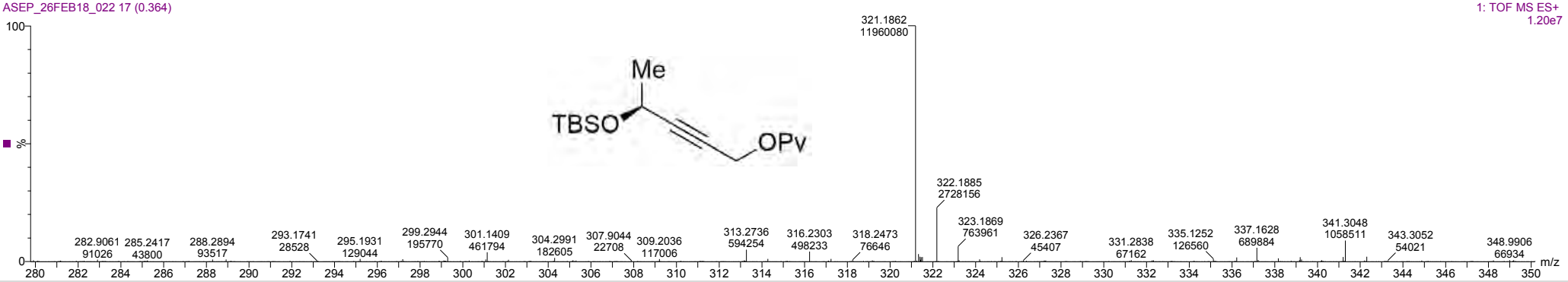
ASEP_26FEB18_022 (0.053) Is (1.00,1.00) C16H30O3SiNa

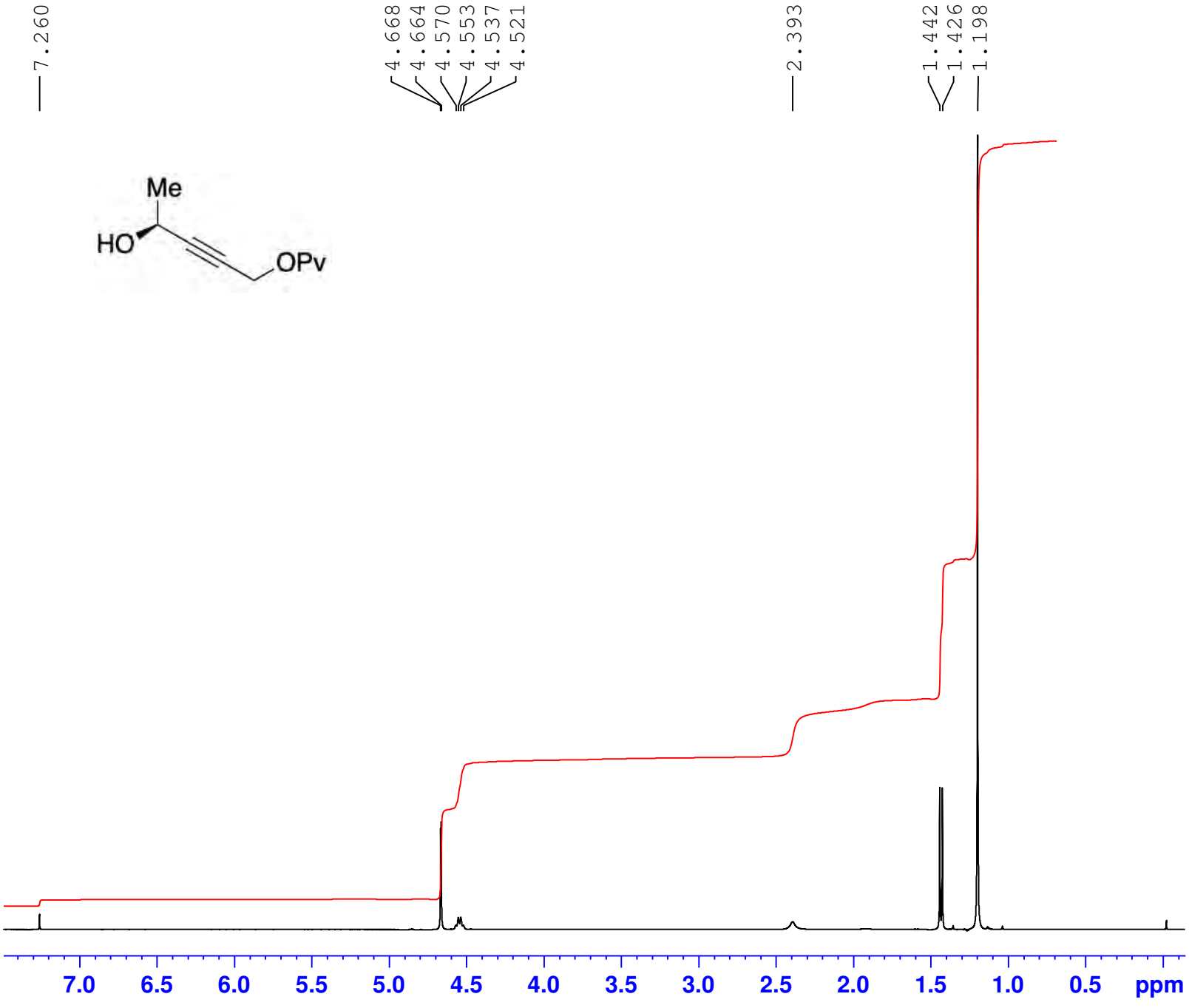


ASEP_26FEB18_022 (0.364) Is (1.00,1.00) C16H30O3Si



ASEP_26FEB18_022 17 (0.364)



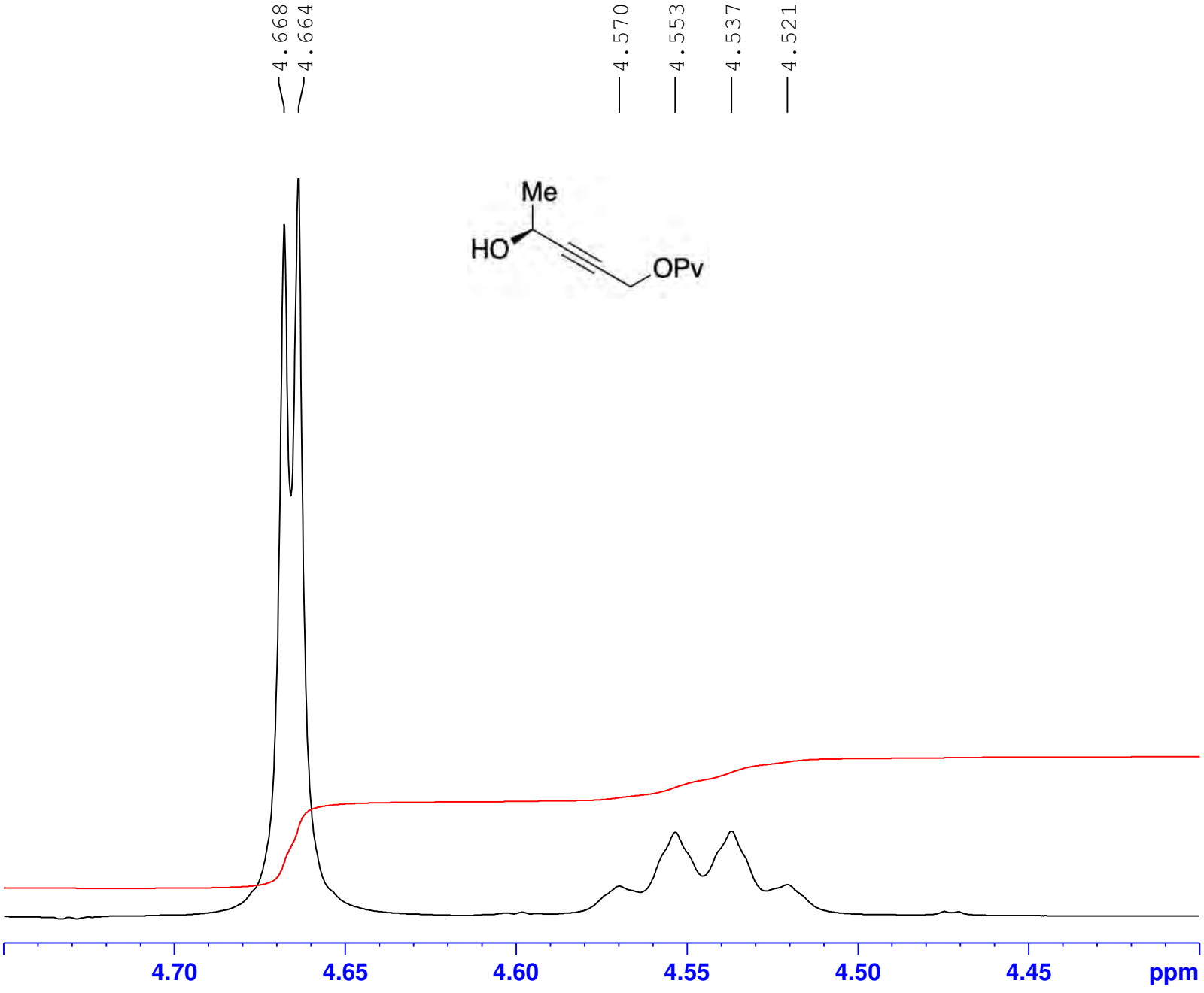



```
Current Data Parameters
NAME          I-PK-51PURE
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180220
Time                 8.03
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 131072
SOLVENT             CDCl3
NS                     64
DS                      0
SWH             12019.230 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    45.2
DW                  41.600 usec
DE                   9.85 usec
TE                  295.9 K
D1             0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          399.9024695 MHz
NUC1                    1H
P1                   14.88 usec
PLW1           7.59999990 W

F2 - Processing parameters
SI                 131072
SF            399.9000097 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```

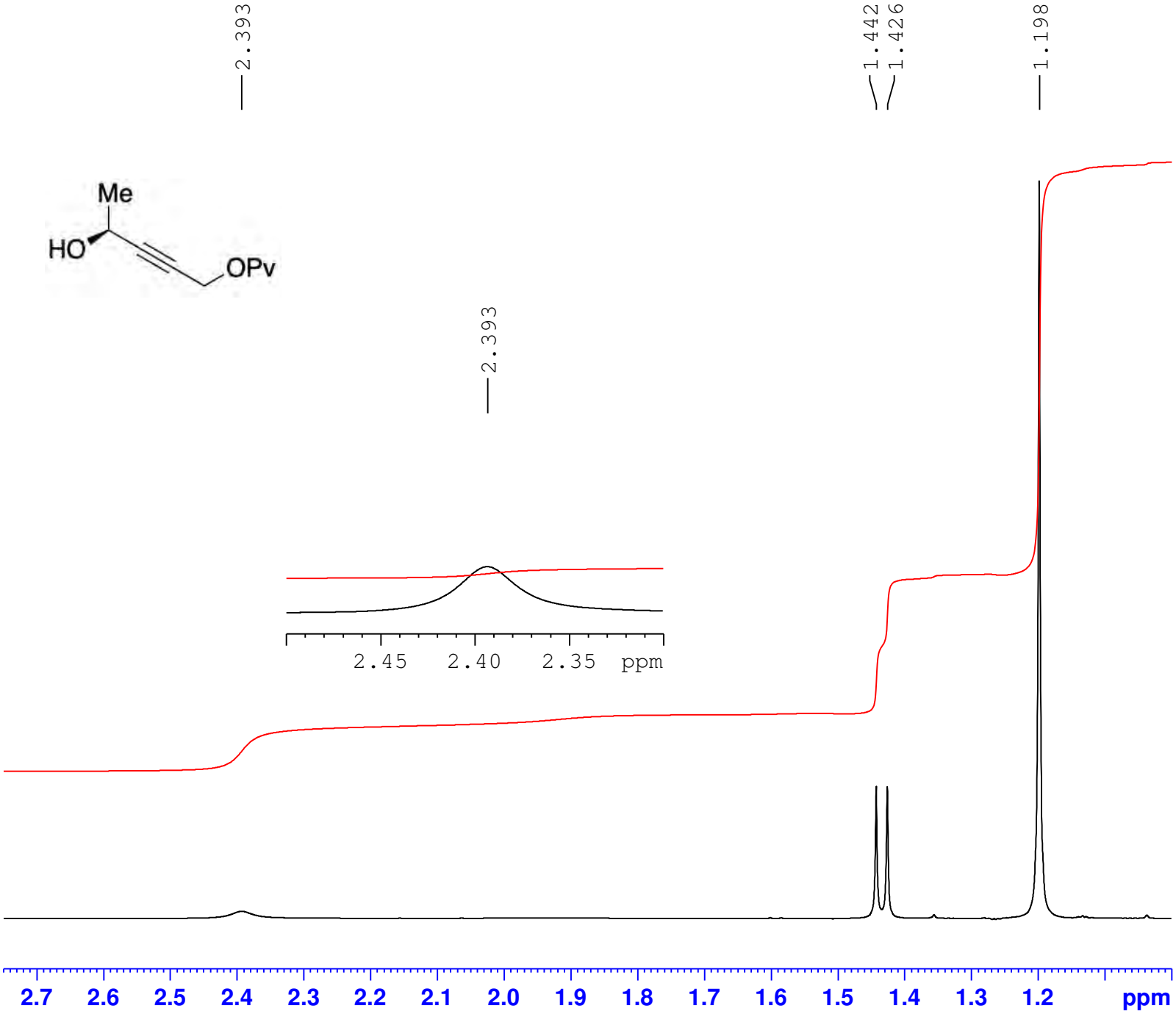


Current Data Parameters
NAME I-PK-51PURE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180220
Time 8.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 295.9 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000097 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



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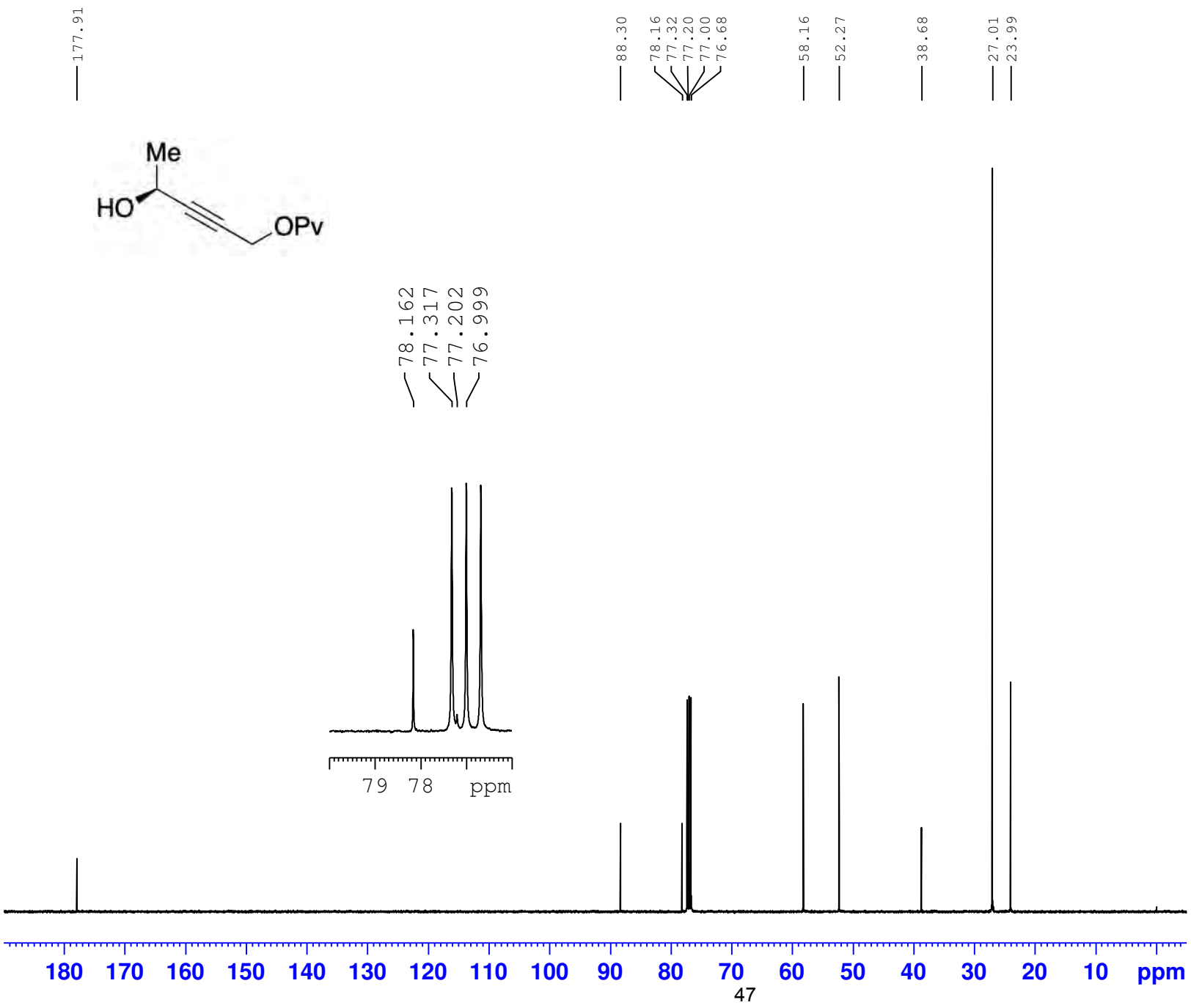
Current Data Parameters
NAME I-PK-51PURE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180220
Time 8.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 295.9 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000097 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



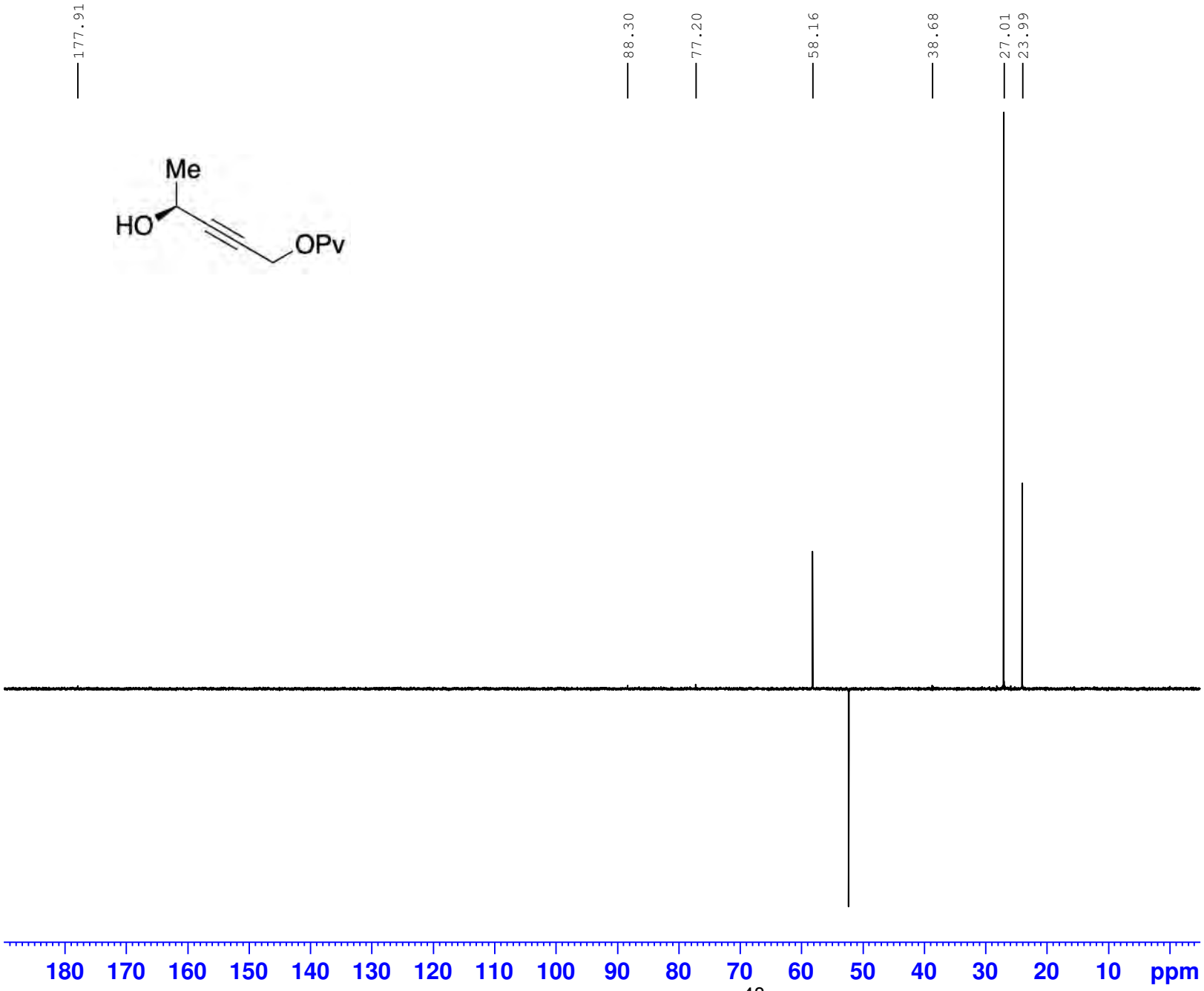
Current Data Parameters
NAME I-PK-51PURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180220
Time 9.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.4 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549399 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



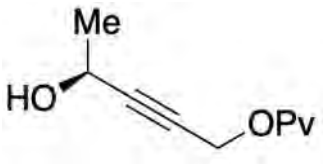
Current Data Parameters
NAME I-PK-51PURE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180220
Time 9.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1820
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549398 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME         I-PK-51PURE
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180220
Time                9.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH             4472.272 Hz
FIDRES          2.183727 Hz
AQ              0.2289664 sec
RG                 2050
DW               111.800 usec
DE                 6.50 usec
TE                296.7 K
D0            0.00000300 sec
D1            0.92135590 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00022360 sec

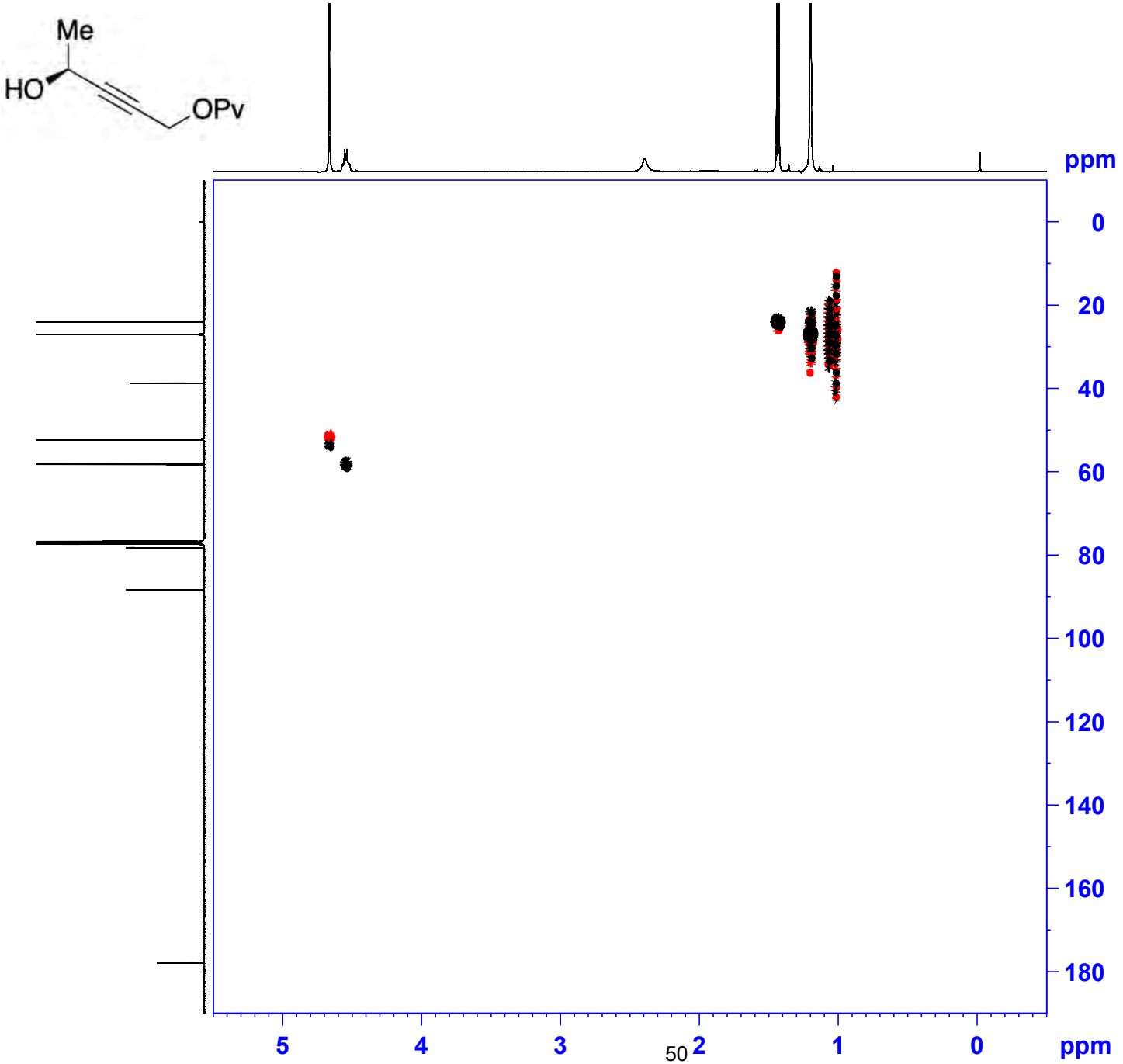
======== CHANNEL f1 ========
SFO1         399.9010158 MHz
NUC1                1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1             16.00 %
GPZ2             12.00 %
GPZ3             40.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1            399.901 MHz
FIDRES        34.939625 Hz
SW               11.183 ppm
FnMODE              QF

F2 - Processing parameters
SI                 1024
SF          399.9000094 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                 QF
SF          399.9000090 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
```




```
Current Data Parameters
NAME         I-PK-51PURE
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180220
Time                9.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT            CDC13
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES             4.695012 Hz
AQ              0.1064960 sec
RG                  2050
DW                104.000 usec
DE                  6.50 usec
TE                297.0 K
CNST2         145.0000000
D0             0.00000300 sec
D1             0.80000001 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D21            0.00360000 sec
IN0            0.00001910 sec

======== CHANNEL f1 ========
SFO1          399.9018806 MHz
NUC1                  1H
P1                 14.88 usec
P2                 29.76 usec
P28       0 usec
PLW1           7.59999990 W

======== CHANNEL f2 ========
SFO2          100.5670016 MHz
NUC2                 13C
CPDPRG[2             garp4
P3                 10.00 usec
P14               500.00 usec
P31              1900.00 usec
PCPD2              80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3               0.500
SPOFFS3   0 Hz
SPW3           6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18              0.500
SPOFFS18 0 Hz
SPW18          1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1              100.567 MHz
FIDRES        204.515701 Hz
SW                260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           399.9000087 MHz
WDW                 QSINE
```

Mass Spectrometry Result Sheet

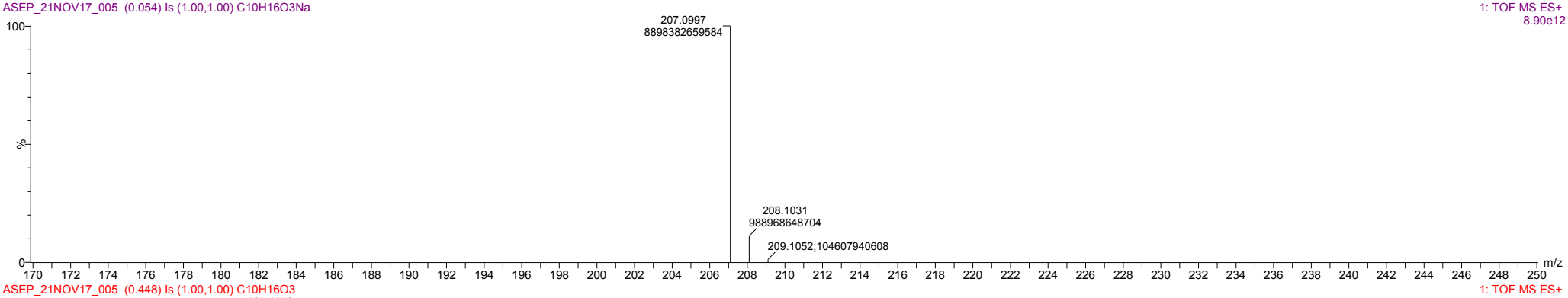
Waters Xevo G2-XS QTof Mass Spectrometer

15-11-2017

I-PK-15

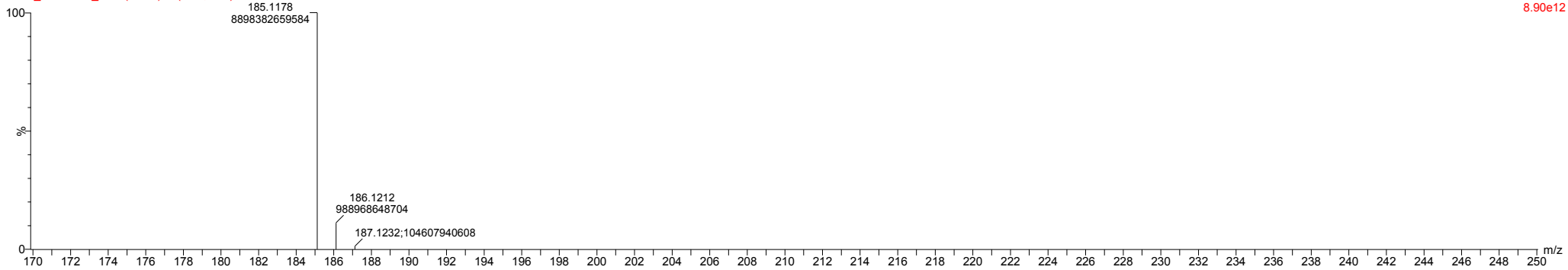
ASEP_21NOV17_005 (0.054) Is (1.00,1.00) C10H16O3Na

1: TOF MS ES+
8.90e12



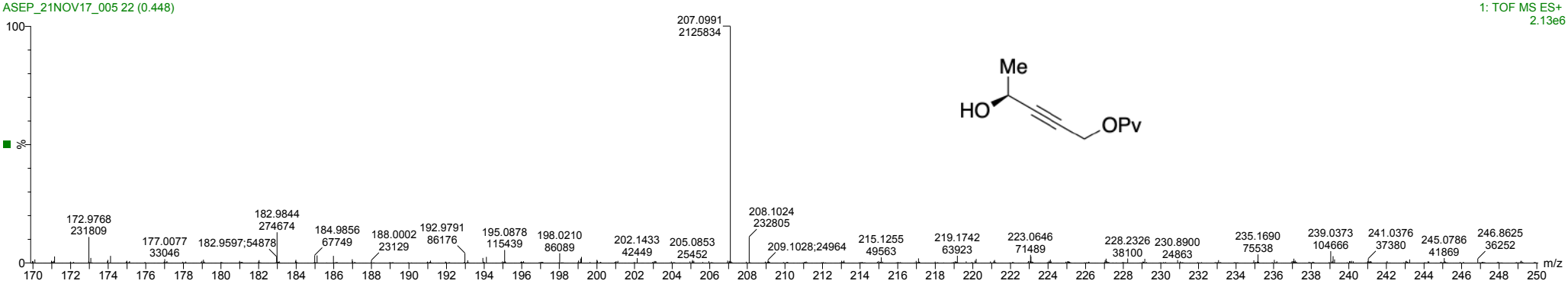
ASEP_21NOV17_005 (0.448) Is (1.00,1.00) C10H16O3

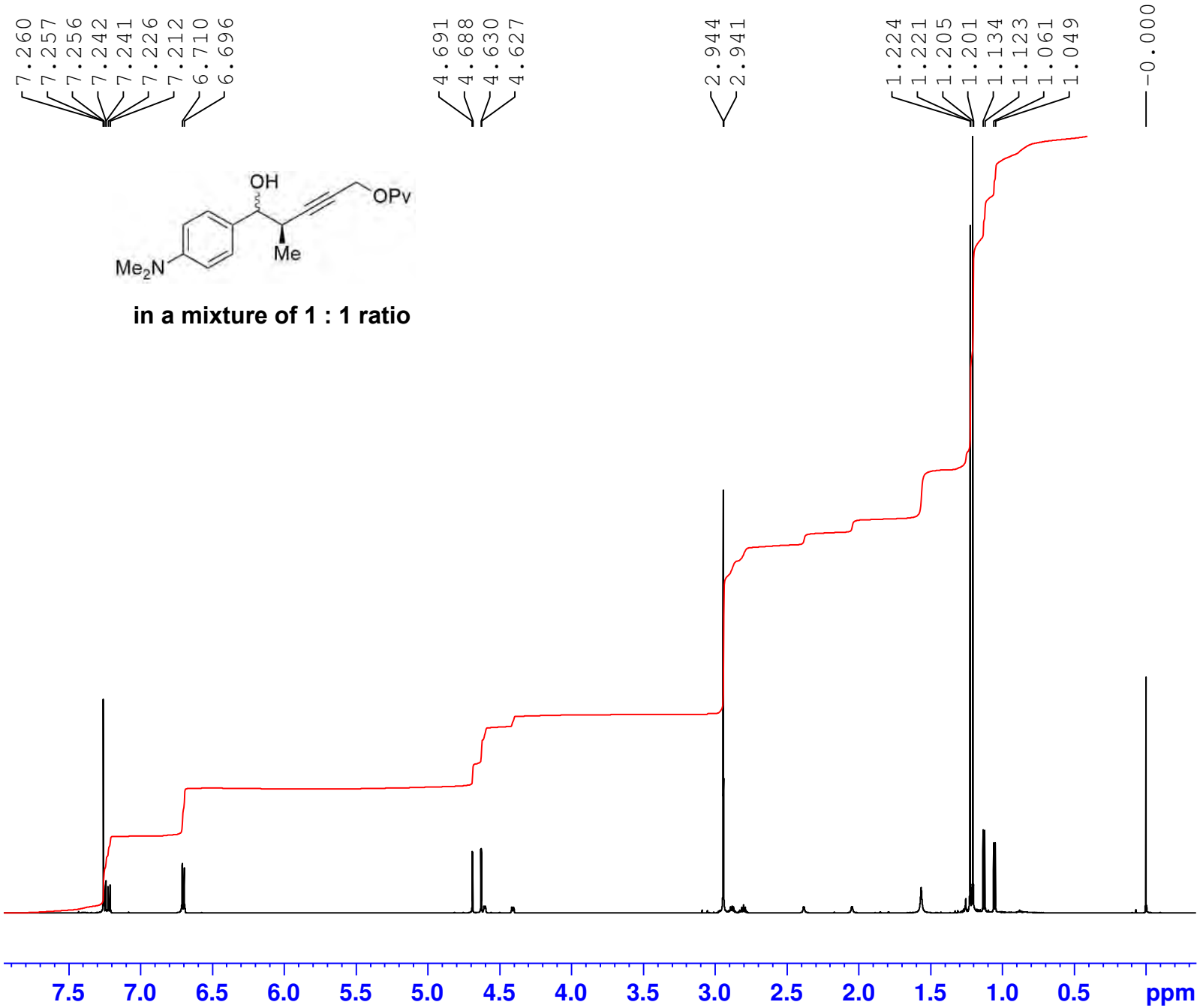
1: TOF MS ES+
8.90e12



ASEP_21NOV17_005 22 (0.448)

1: TOF MS ES+
2.13e6





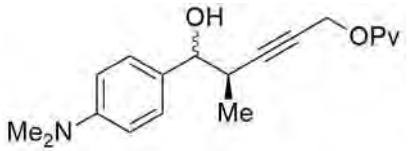
BRUKER

```
Current Data Parameters
NAME                  III-PK-66
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20191107
Time                     10.06
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                      180286
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  18028.846 Hz
FIDRES                0.100001 Hz
AQ                   4.9999318 sec
RG                        97.5
DW                      27.733 usec
DE                        7.60 usec
TE                       300.0 K
D1                  0.10000000 sec
TD0                          1

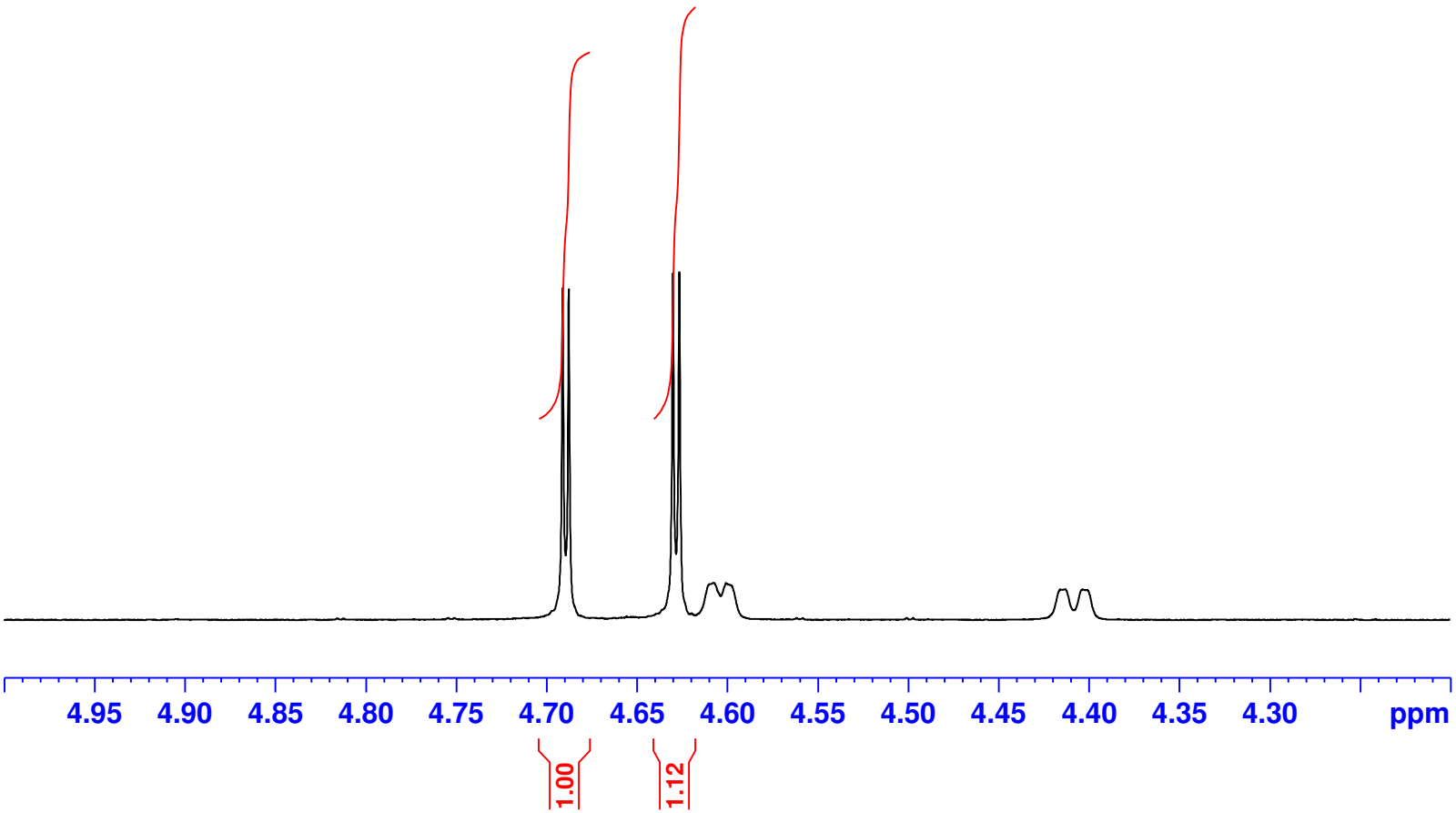
======== CHANNEL f1 ========
SFO1                600.1337060 MHz
NUC1                        1H
P1                       10.00 usec
PLW1                26.60000038 W

F2 - Processing parameters
SI                       262144
SF                  600.1300144 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00
```



in a mixture of 1 : 1 ratio

4.691
4.688
4.630
4.627

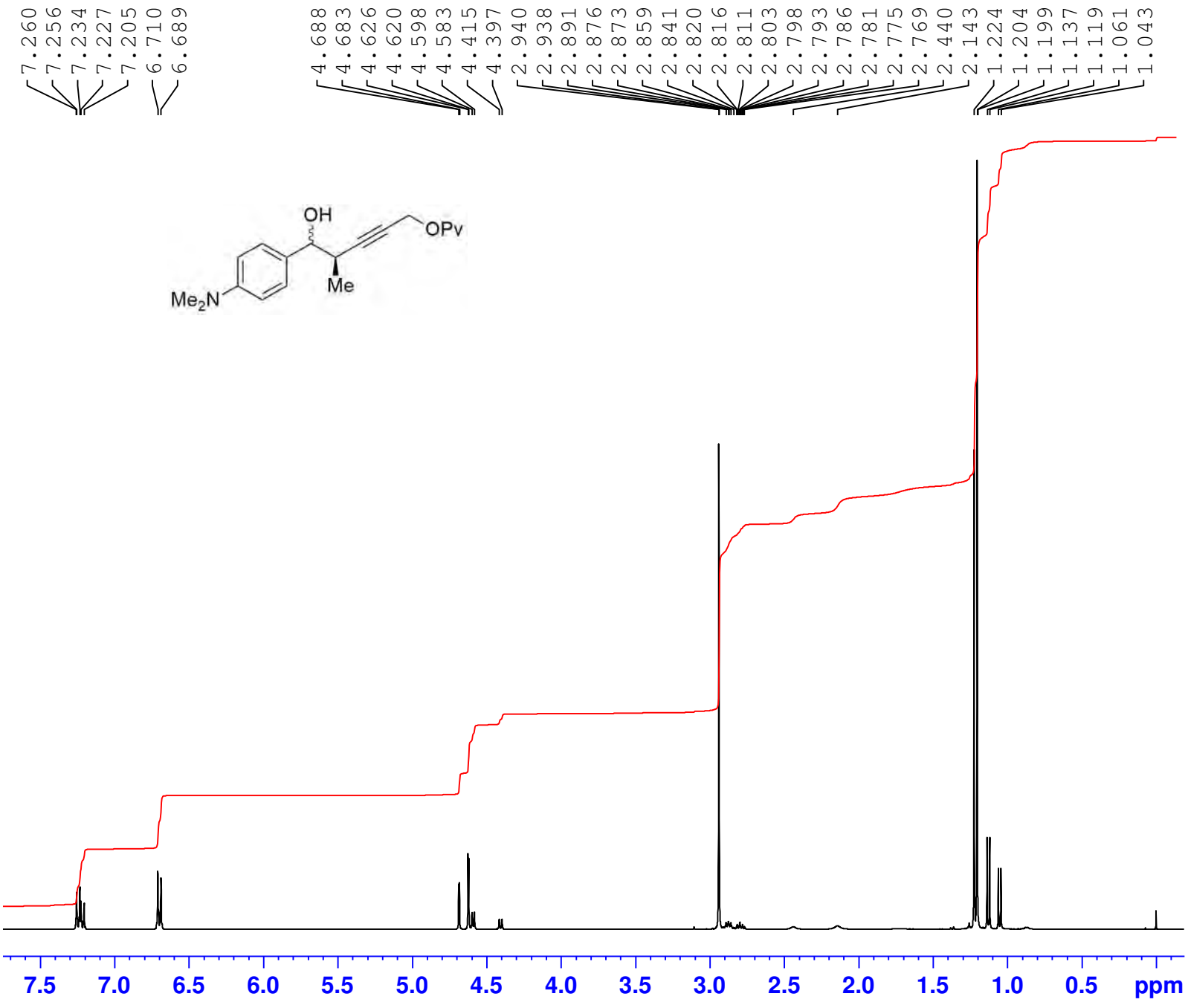



```
Current Data Parameters
NAME          III-PK-66
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20191107
Time              10.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD              180286
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           18028.846 Hz
FIDRES         0.100001 Hz
AQ            4.9999318 sec
RG                97.5
DW               27.733 usec
DE                7.60 usec
TE               300.0 K
D1           0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.1337060 MHz
NUC1                1H
P1                10.00 usec
PLW1        26.60000038 W

F2 - Processing parameters
SI                262144
SF          600.1300144 MHz
WDW                 EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```



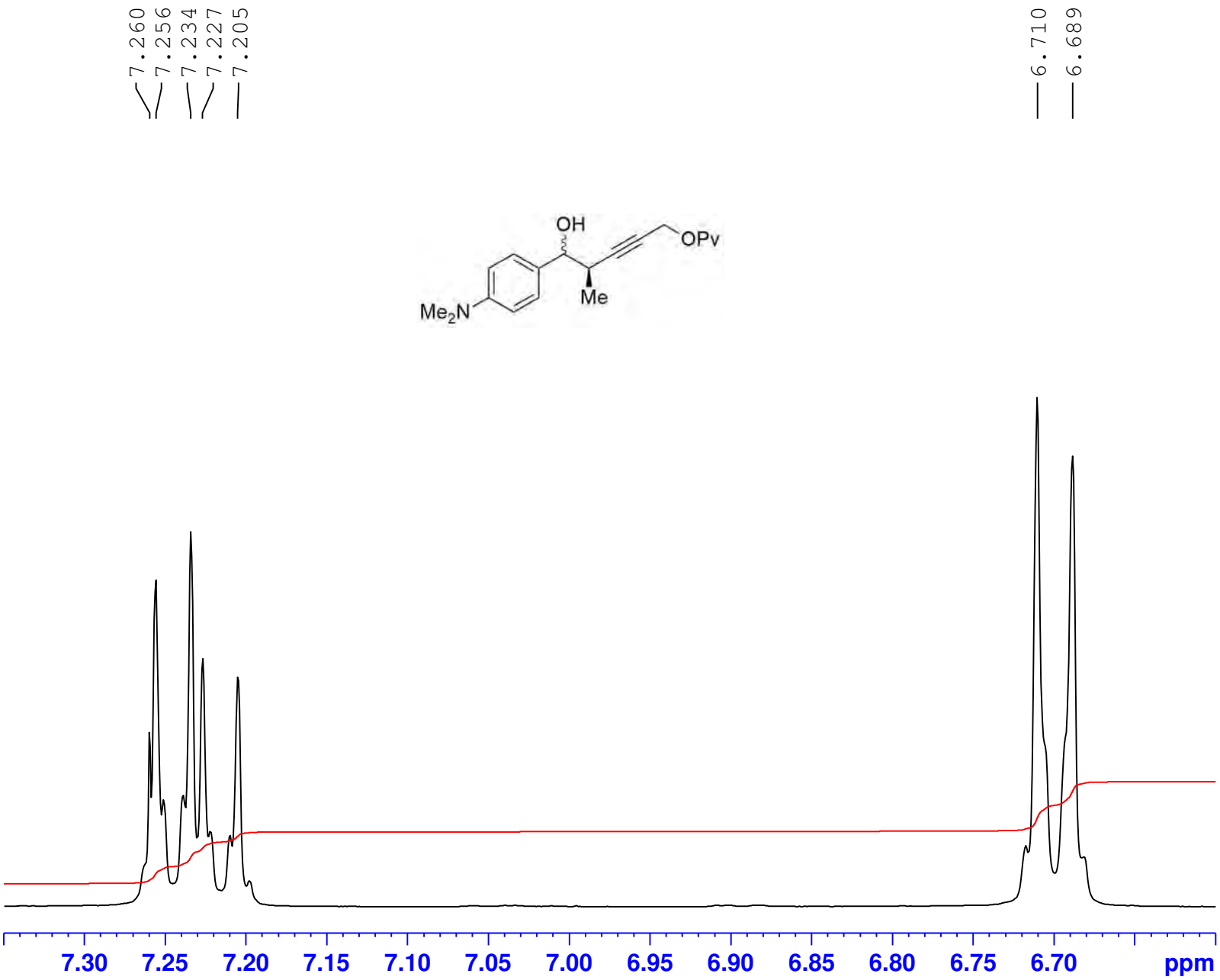
BRUKER

Current Data Parameters
NAME I-PK-20-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171128
Time 13.19
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 36
DW 60.800 usec
DE 16.82 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1124708 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 400.1100099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

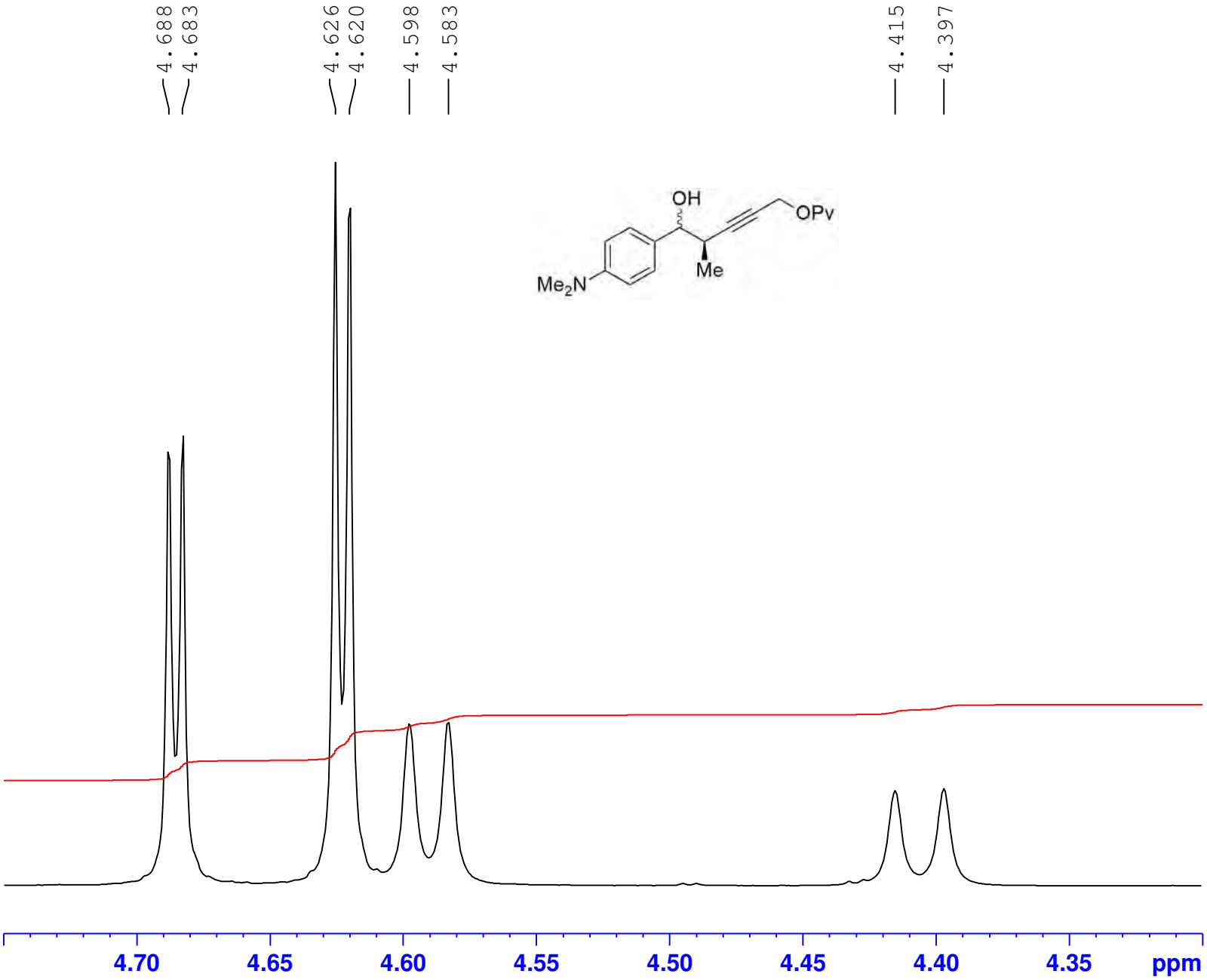



```
Current Data Parameters
NAME          I-PK-20-03
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171128
Time               13.19
INSTRUM         AVIII_400
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9845889 sec
RG                     36
DW                60.800 usec
DE                16.82 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                  32768
SF           400.1100099 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

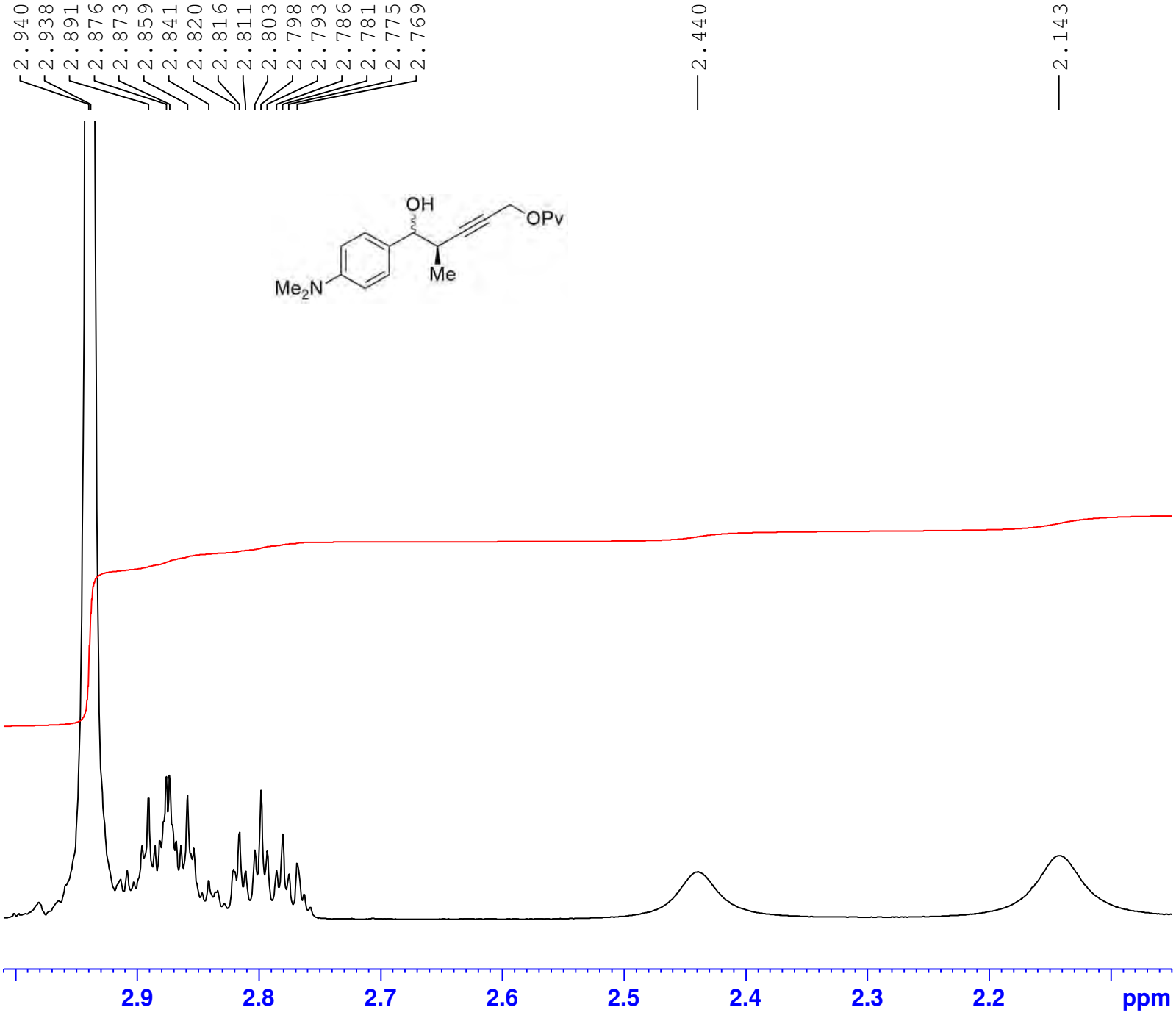



```
Current Data Parameters
NAME          I-PK-20-03
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171128
Time               13.19
INSTRUM        AVIII_400
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                    36
DW                60.800 usec
DE                16.82 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                  1H
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

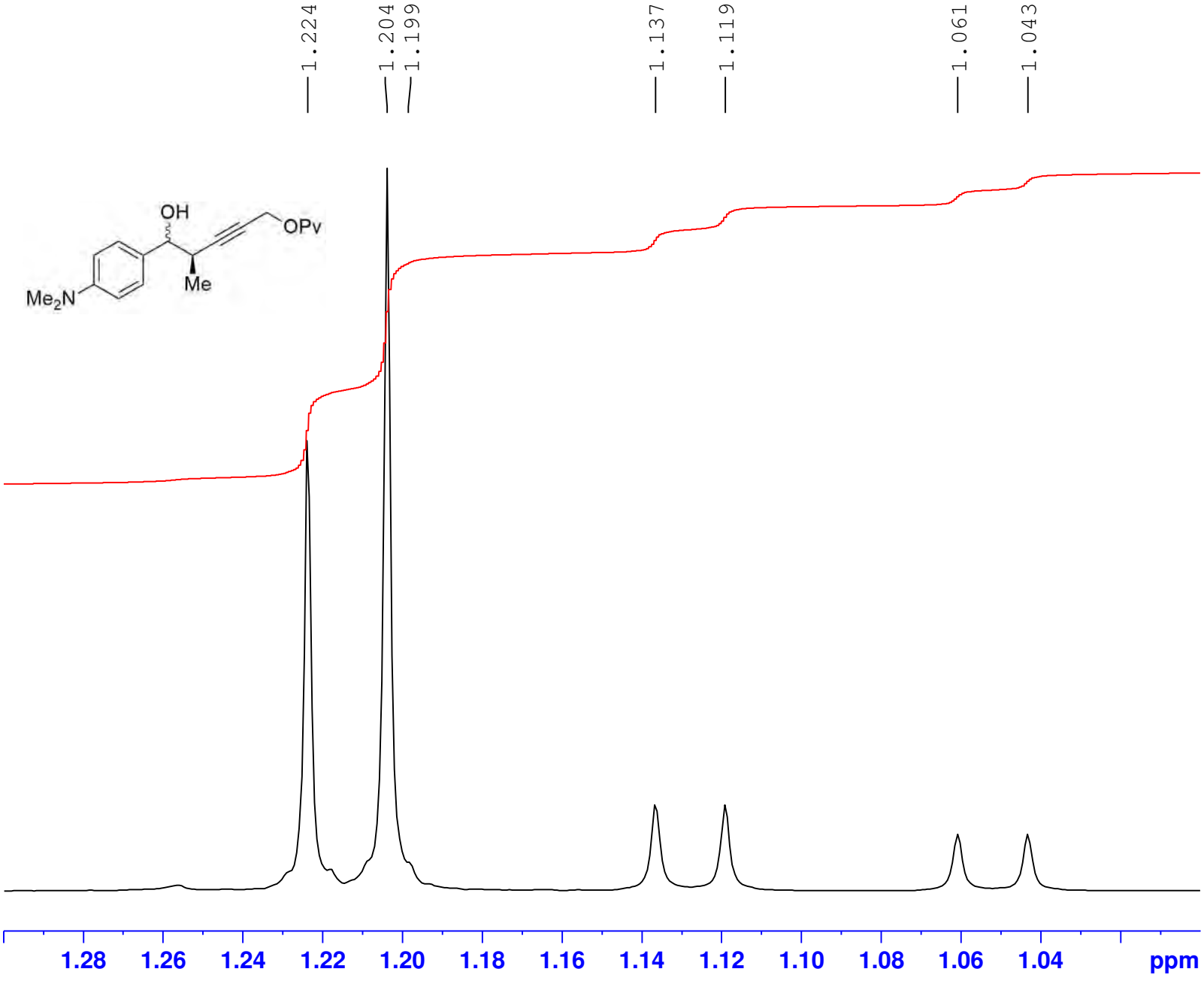


Current Data Parameters
NAME I-PK-20-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171128
Time 13.19
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 36
DW 60.800 usec
DE 16.82 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1124708 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 400.1100099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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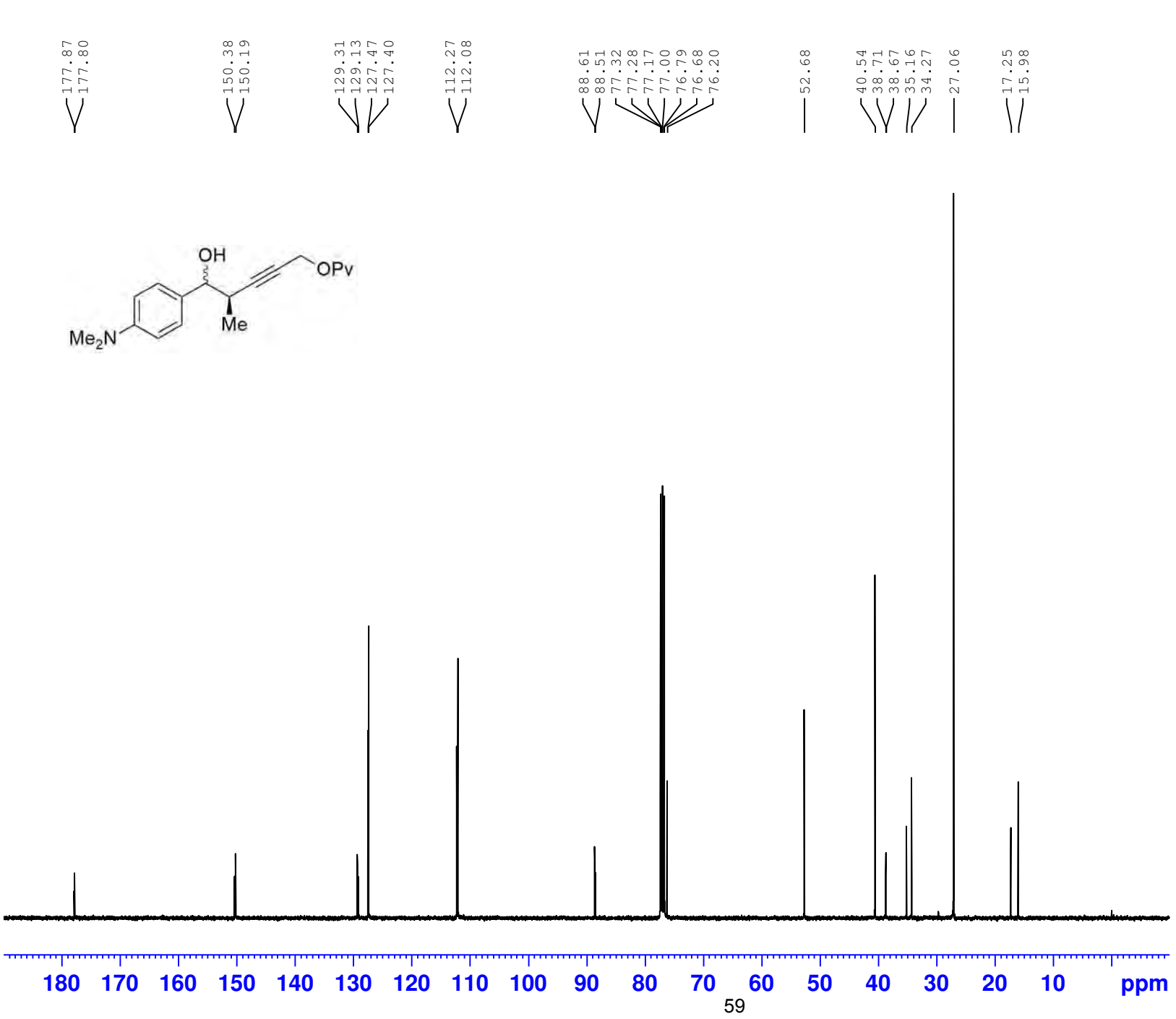
```
Current Data Parameters
NAME          I-PK-20-03
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171128
Time               13.19
INSTRUM       AVIII_400
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                    36
DW                60.800 usec
DE                16.82 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1124708 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



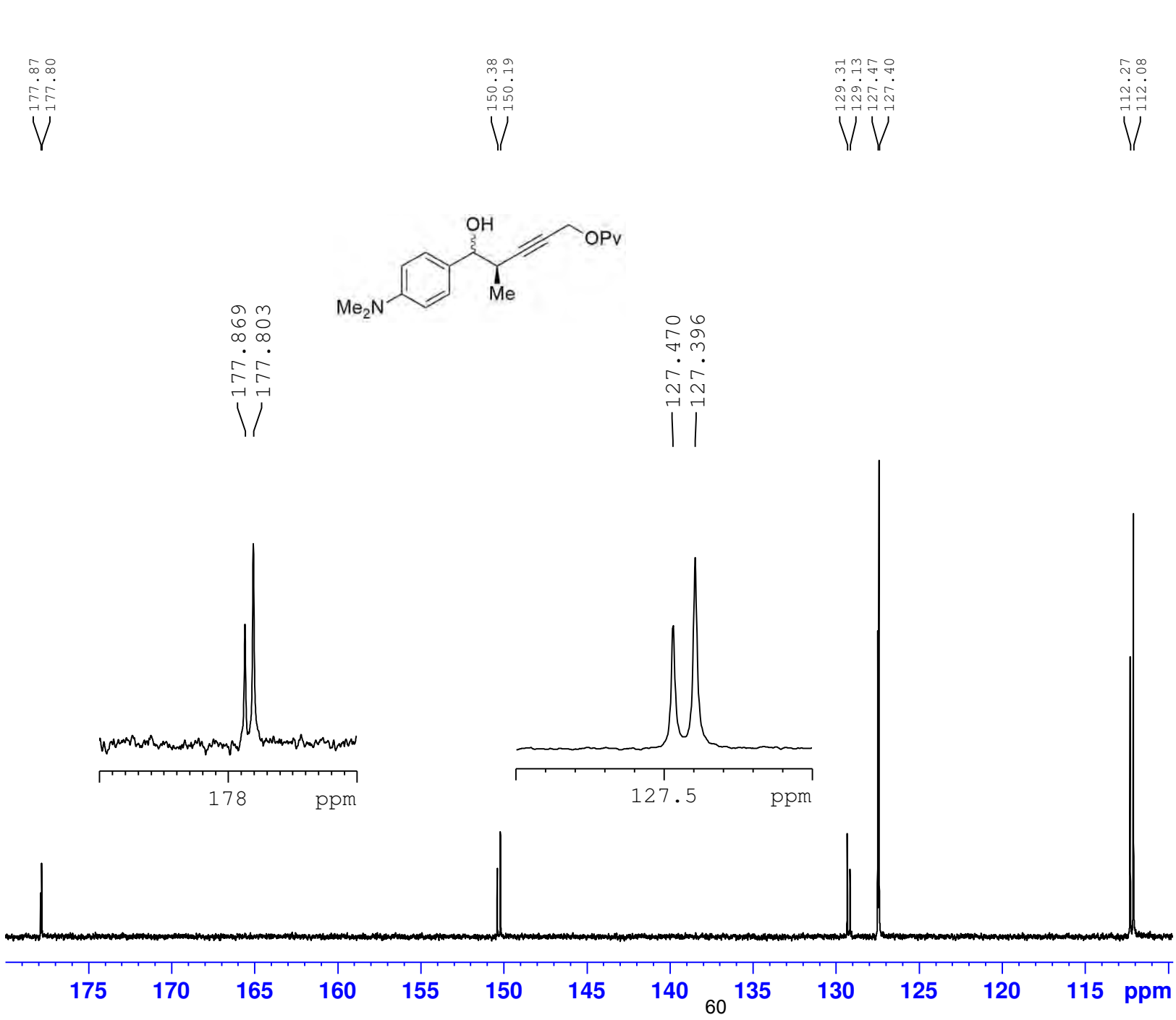
Current Data Parameters
NAME I-PK-20-04
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171129
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549404 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



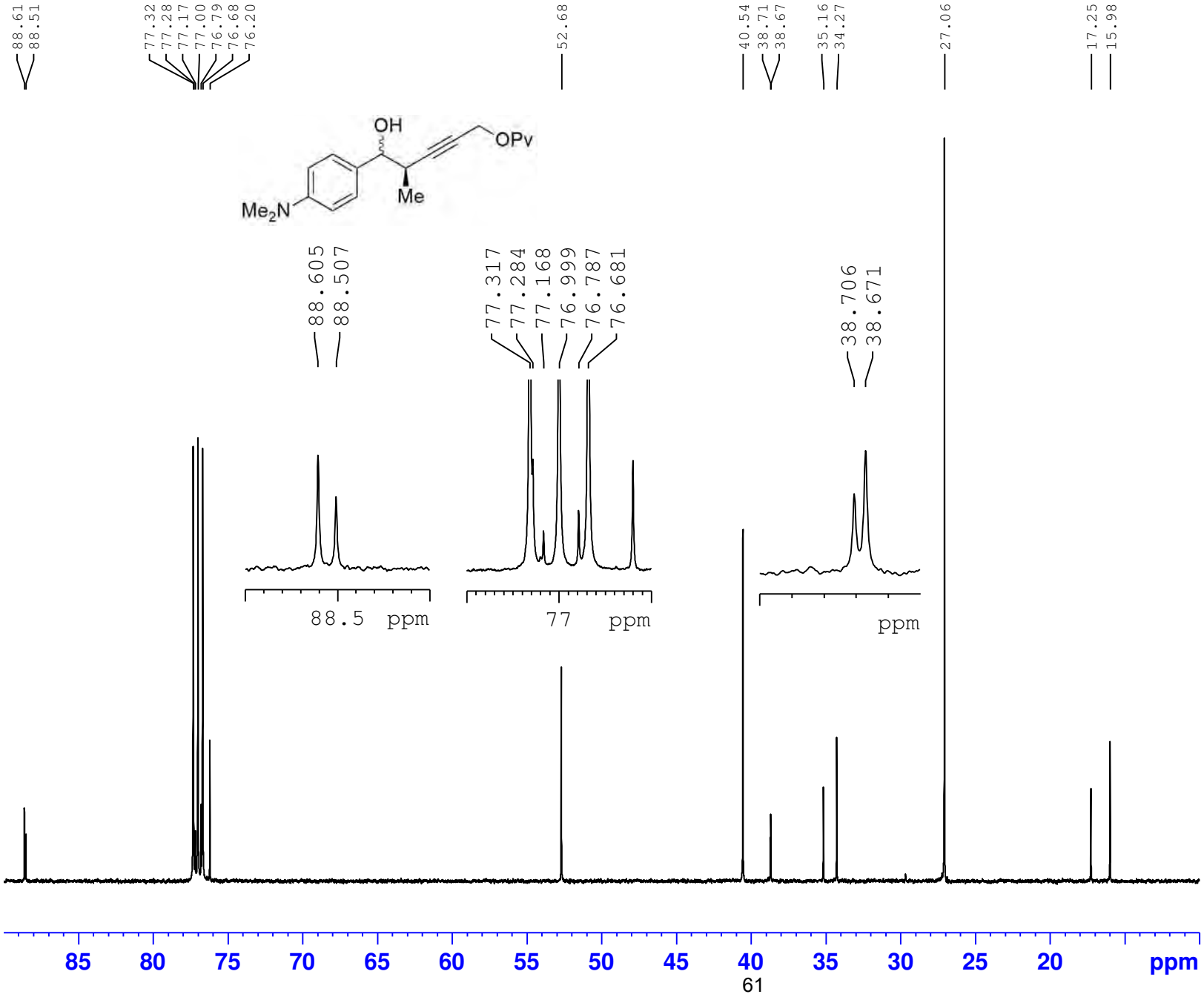
Current Data Parameters
NAME I-PK-20-04
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171129
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549404 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



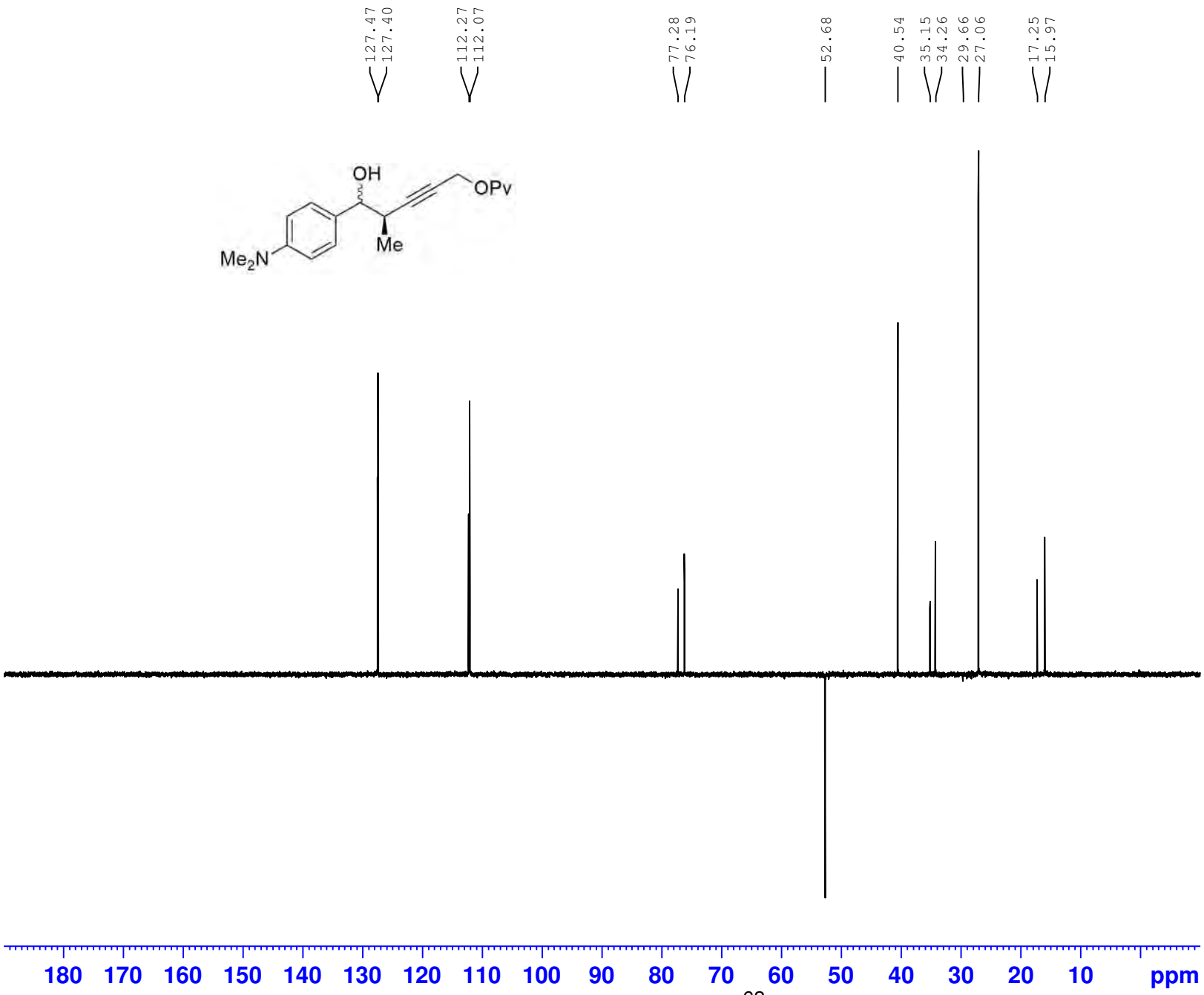
Current Data Parameters
NAME I-PK-20-04
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171129
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 298.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549404 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



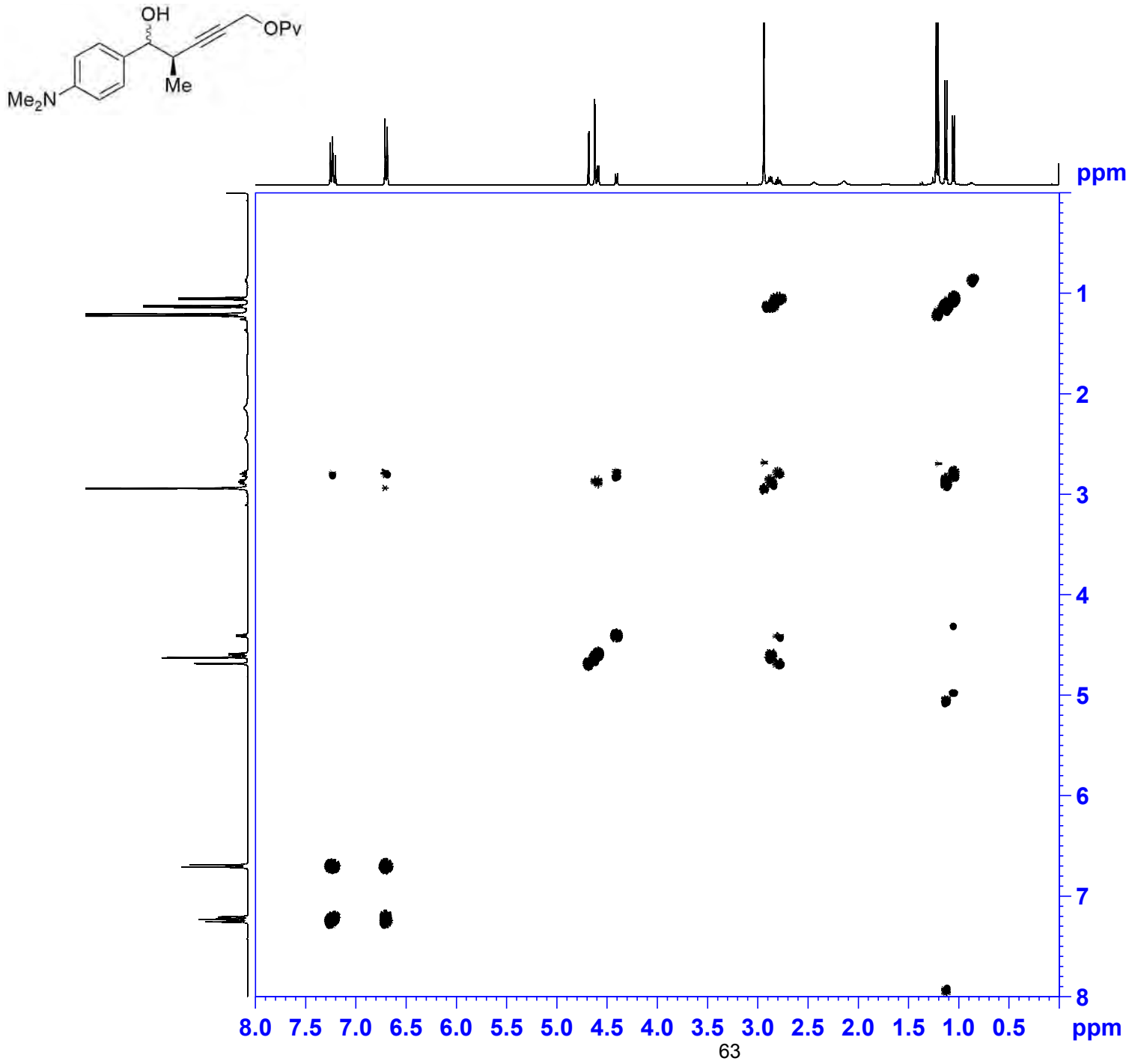
Current Data Parameters
NAME I-PK-20-04
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171129
Time 11.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549404 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME          I-PK-20-04
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171129
Time              11.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                2048
SOLVENT           CDCl3
NS                   1
DS                   8
SWH           4300.458 Hz
FIDRES        2.099833 Hz
AQ            0.2381141 sec
RG                2050
DW             116.267 usec
DE               6.50 usec
TE               297.1 K
D0            0.00000300 sec
D1            0.91111588 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00023260 sec

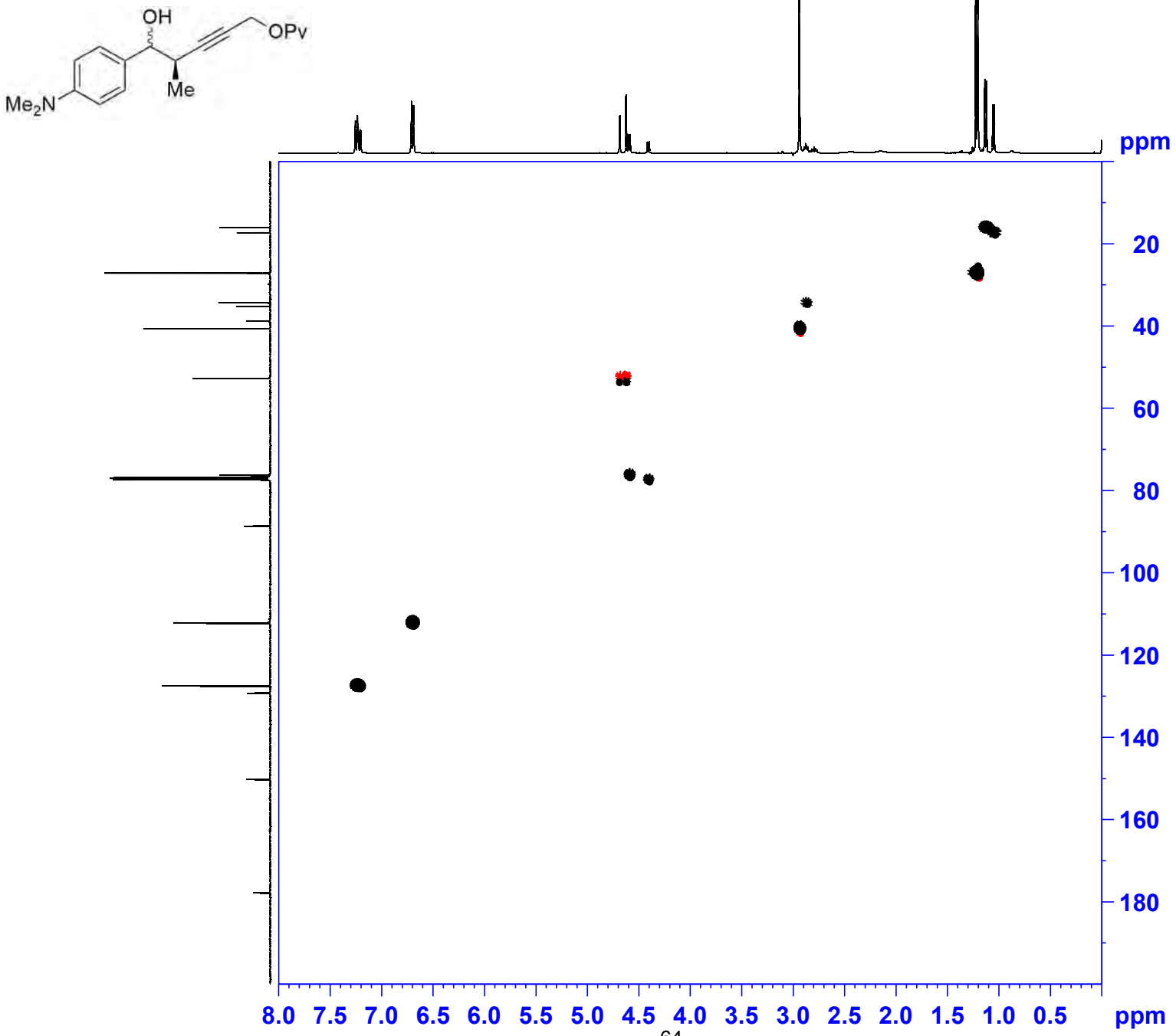
======== CHANNEL f1 ========
SFO1         399.9010913 MHz
NUC1                 1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1             16.00 %
GPZ2             12.00 %
GPZ3             40.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                 256
SFO1           399.9011 MHz
FIDRES        33.587704 Hz
SW               10.751 ppm
FnMODE              QF

F2 - Processing parameters
SI                1024
SF          399.9000100 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                 QF
SF          399.9000100 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```




```
Current Data Parameters
NAME          I-PK-20-04
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171129
Time              11.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT            CDCl3
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES          4.695012 Hz
AQ             0.1064960 sec
RG                  2050
DW               104.000 usec
DE                 6.50 usec
TE                297.2 K
CNST2        145.0000000
D0            0.00000300 sec
D1            0.80000001 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D21           0.00360000 sec
IN0           0.00001910 sec

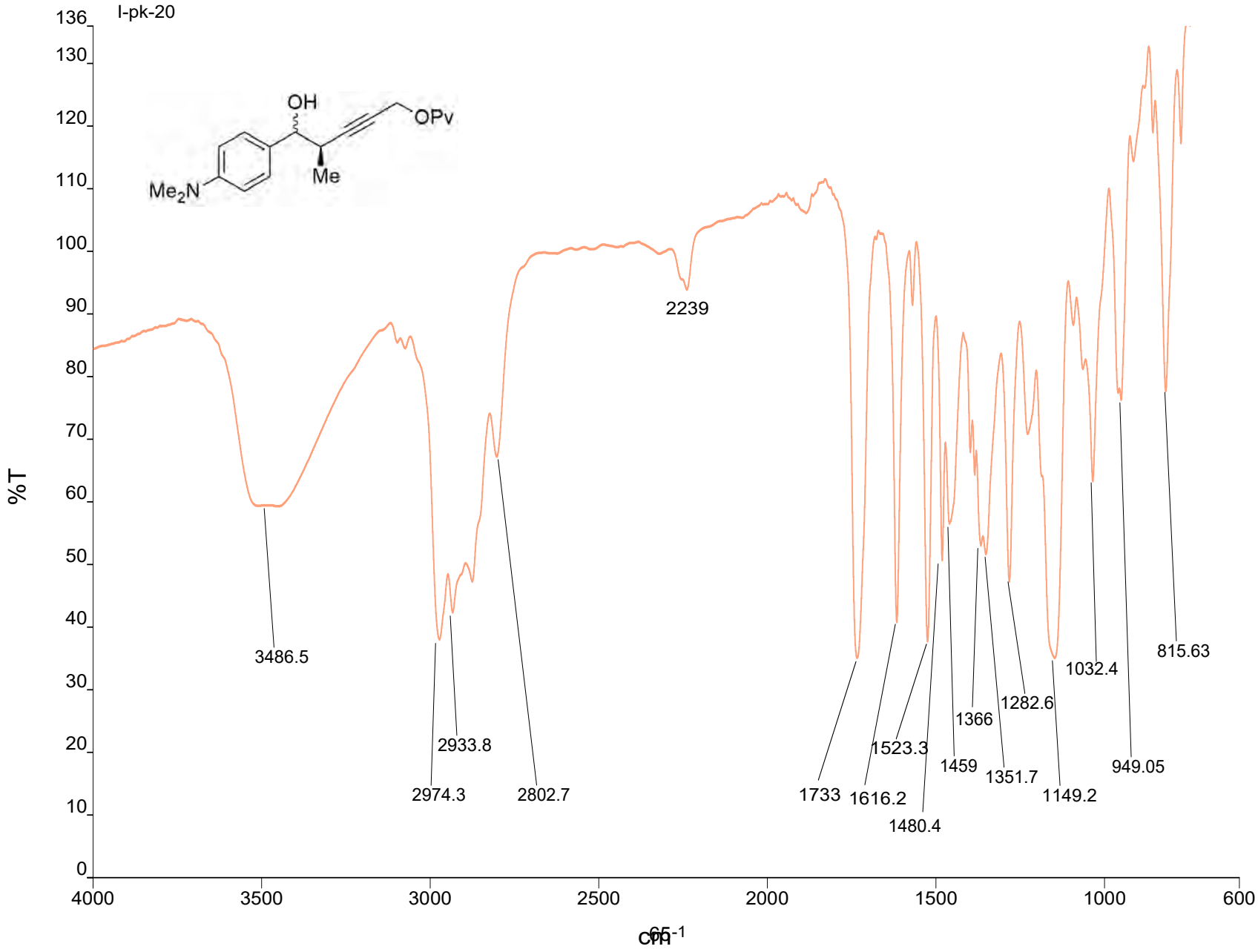
======== CHANNEL f1 ========
SFO1         399.9018806 MHz
NUC1                 1H
P1                14.88 usec
P2                29.76 usec
P28       0 usec
PLW1          7.59999990 W

======== CHANNEL f2 ========
SFO2         100.5670016 MHz
NUC2                13C
CPDPRG[2           garp4
P3                10.00 usec
P14              500.00 usec
P31             1900.00 usec
PCPD2             80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3   0 Hz
SPW3          6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18            0.500
SPOFFS18 0 Hz
SPW18         1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1            100.567 MHz
FIDRES        204.515701 Hz
SW              260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           399.9000124 MHz
```


I-pk-20
OH
OPv
Me
Me2N
%T
cm-1
3486.5
2974.3
2933.8
2802.7
2239
1733
1616.2
1523.3
1480.4
1459
1366
1351.7
1282.6
1149.2
1032.4
949.05
815.63

Mass Spectrometry Result Sheet

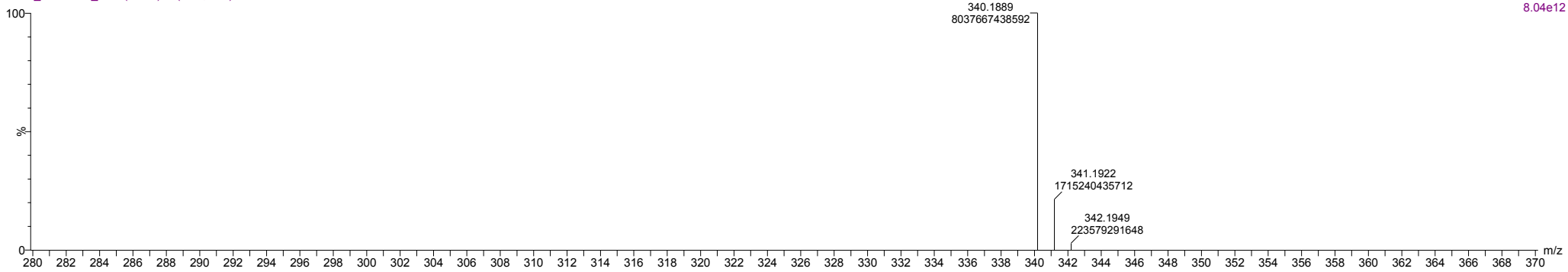
Waters Xevo G2-XS QTof Mass Spectrometer

28-11-2017

I-PK-20

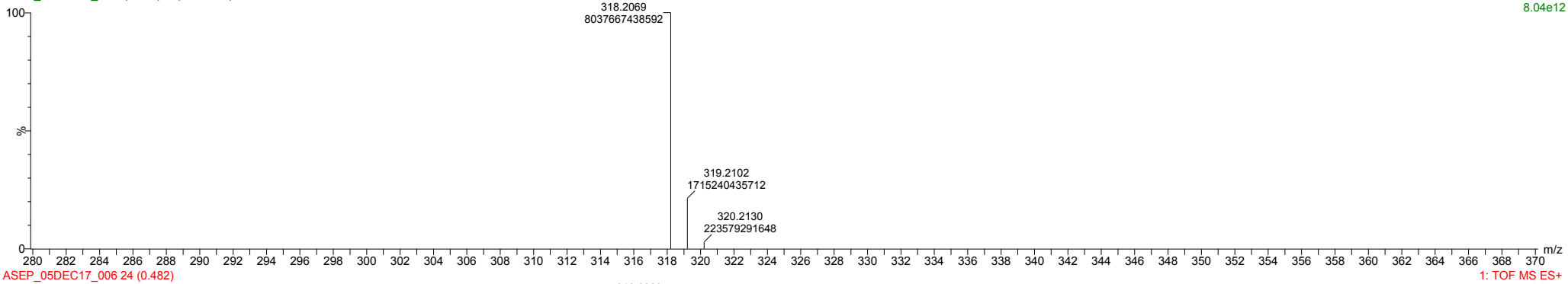
ASEP_05DEC17_006 (0.053) Is (1.00,1.00) C19H27NO3Na

1: TOF MS ES+
8.04e12



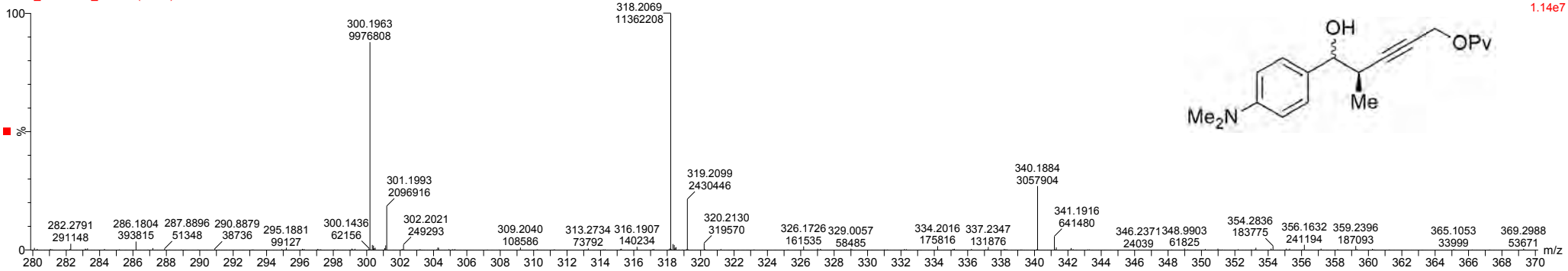
ASEP_05DEC17_006 (0.482) Is (1.00,1.00) C19H27NO3

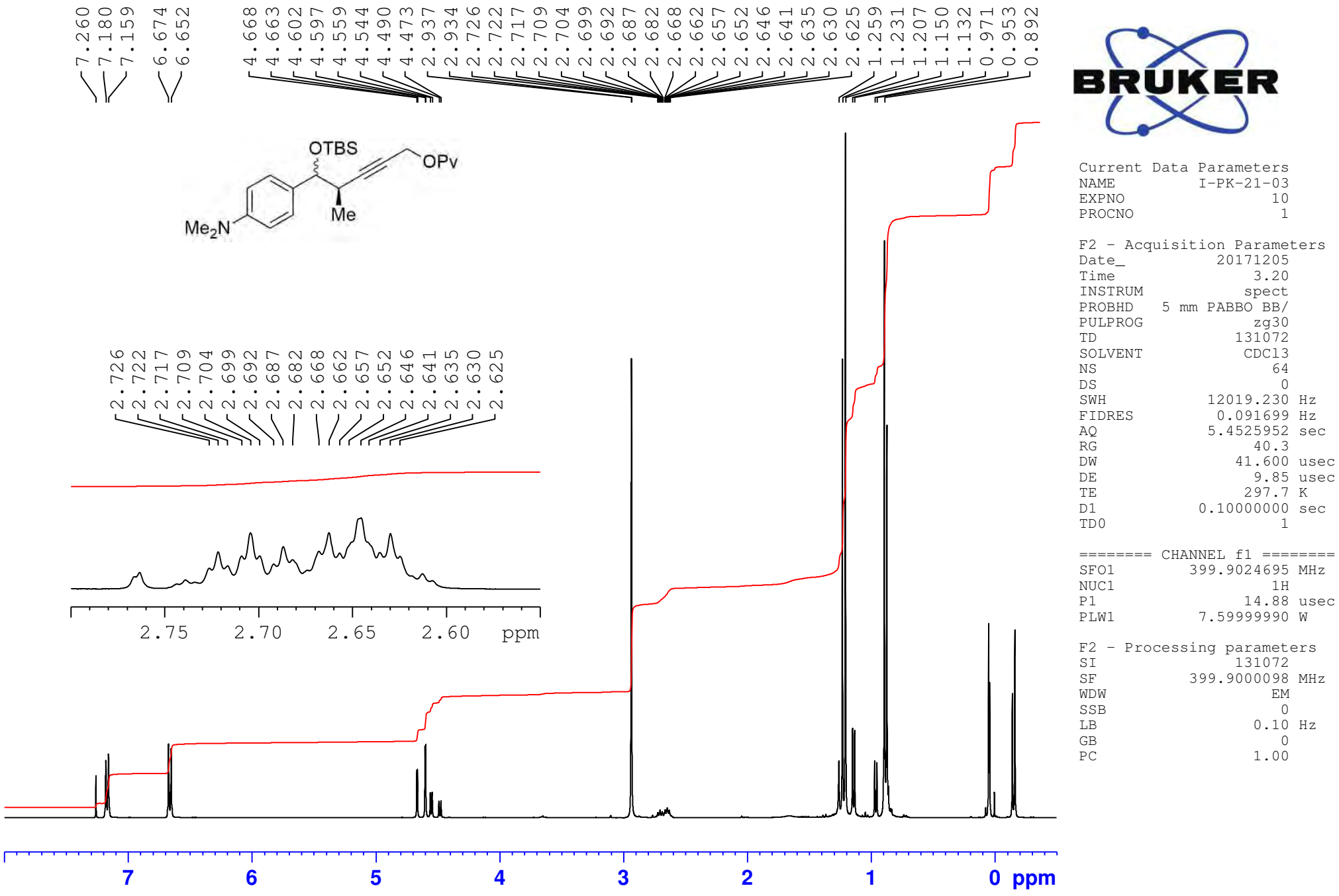
1: TOF MS ES+
8.04e12



ASEP_05DEC17_006 24 (0.482)

1: TOF MS ES+
1.14e7



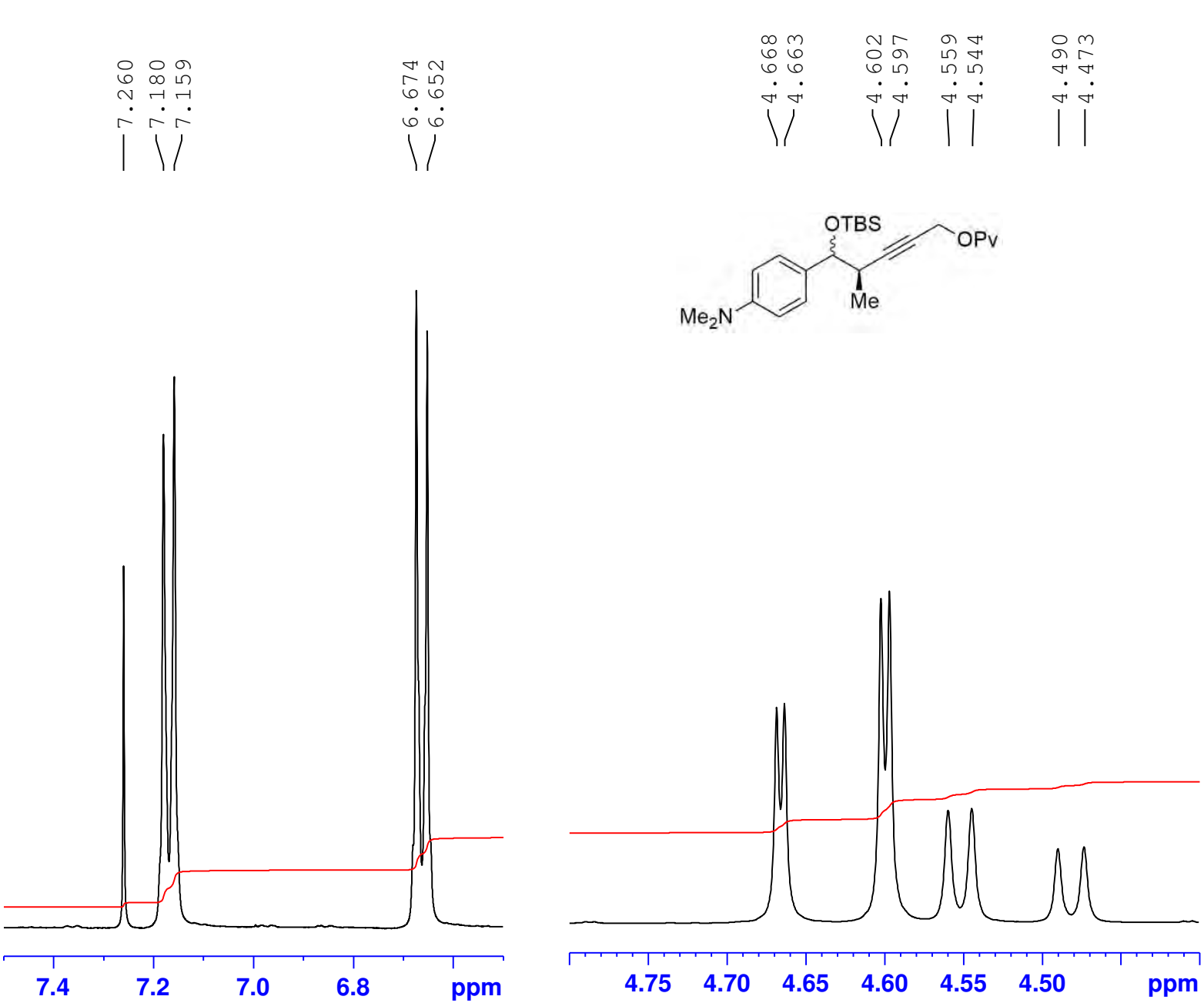


Current Data Parameters
NAME I-PK-21-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 3.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 40.3
DW 41.600 usec
DE 9.85 usec
TE 297.7 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000098 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

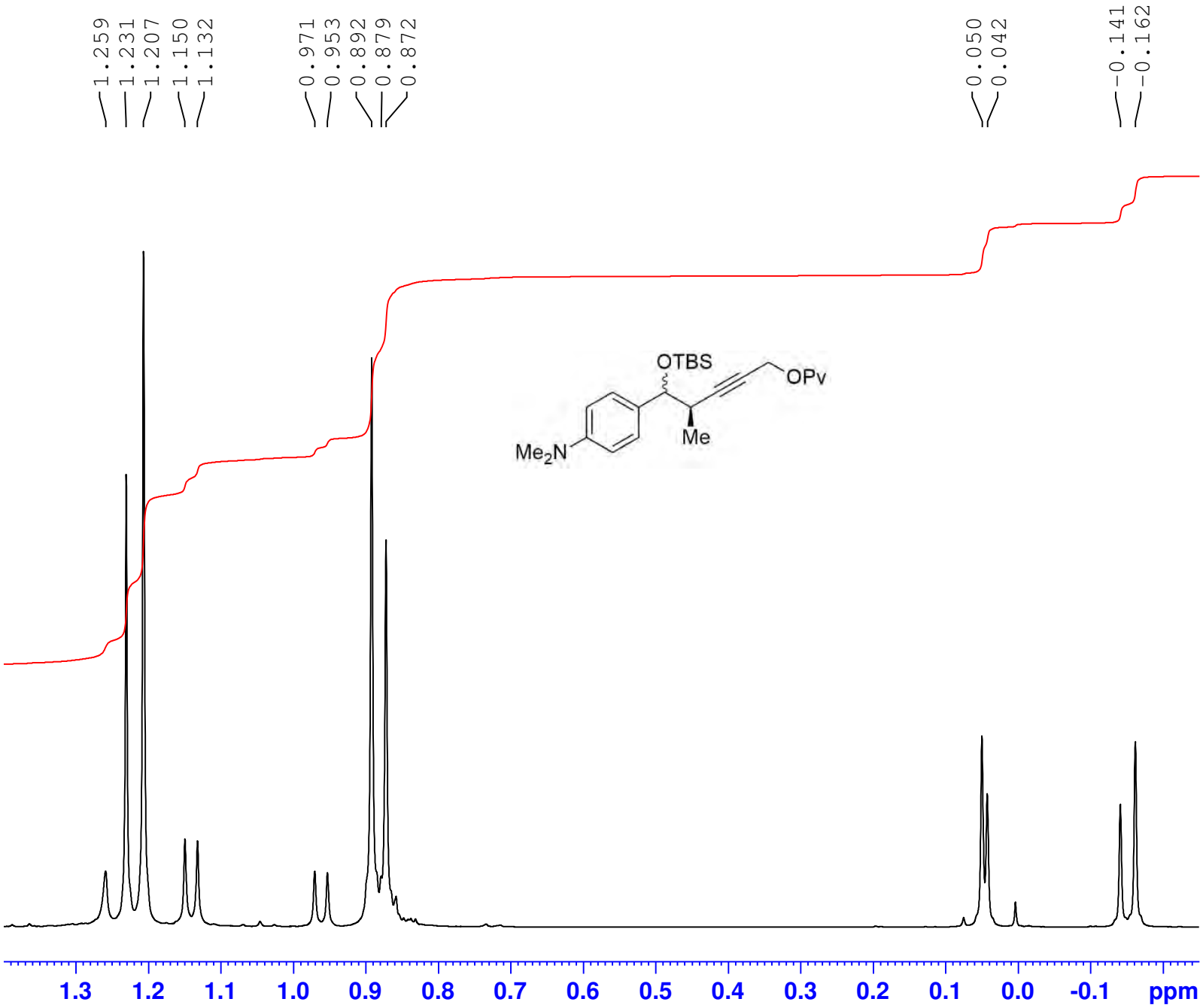


Current Data Parameters
NAME I-PK-21-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 3.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 40.3
DW 41.600 usec
DE 9.85 usec
TE 297.7 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000098 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



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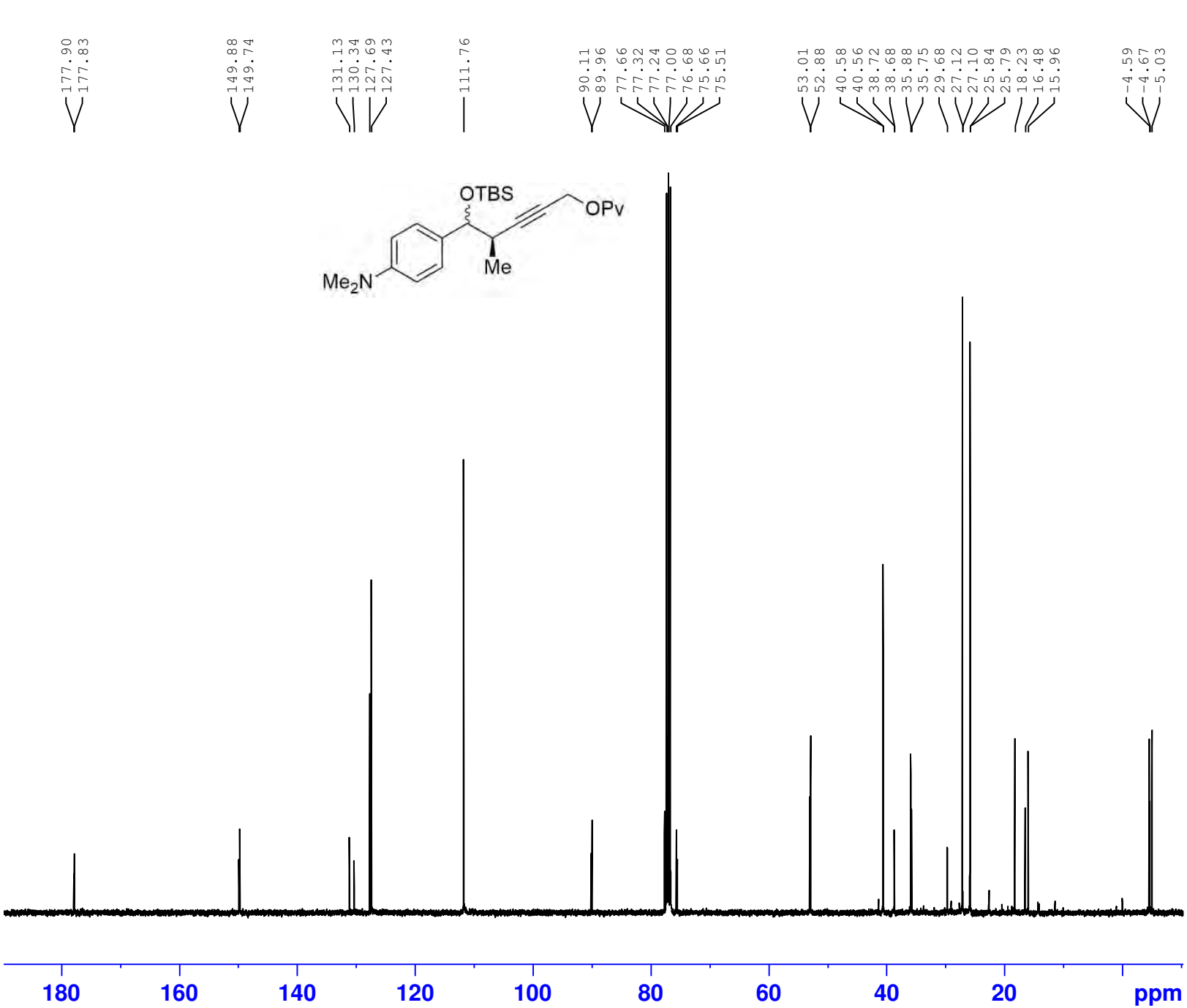
Current Data Parameters
NAME I-PK-21-03
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 3.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 40.3
DW 41.600 usec
DE 9.85 usec
TE 297.7 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000098 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



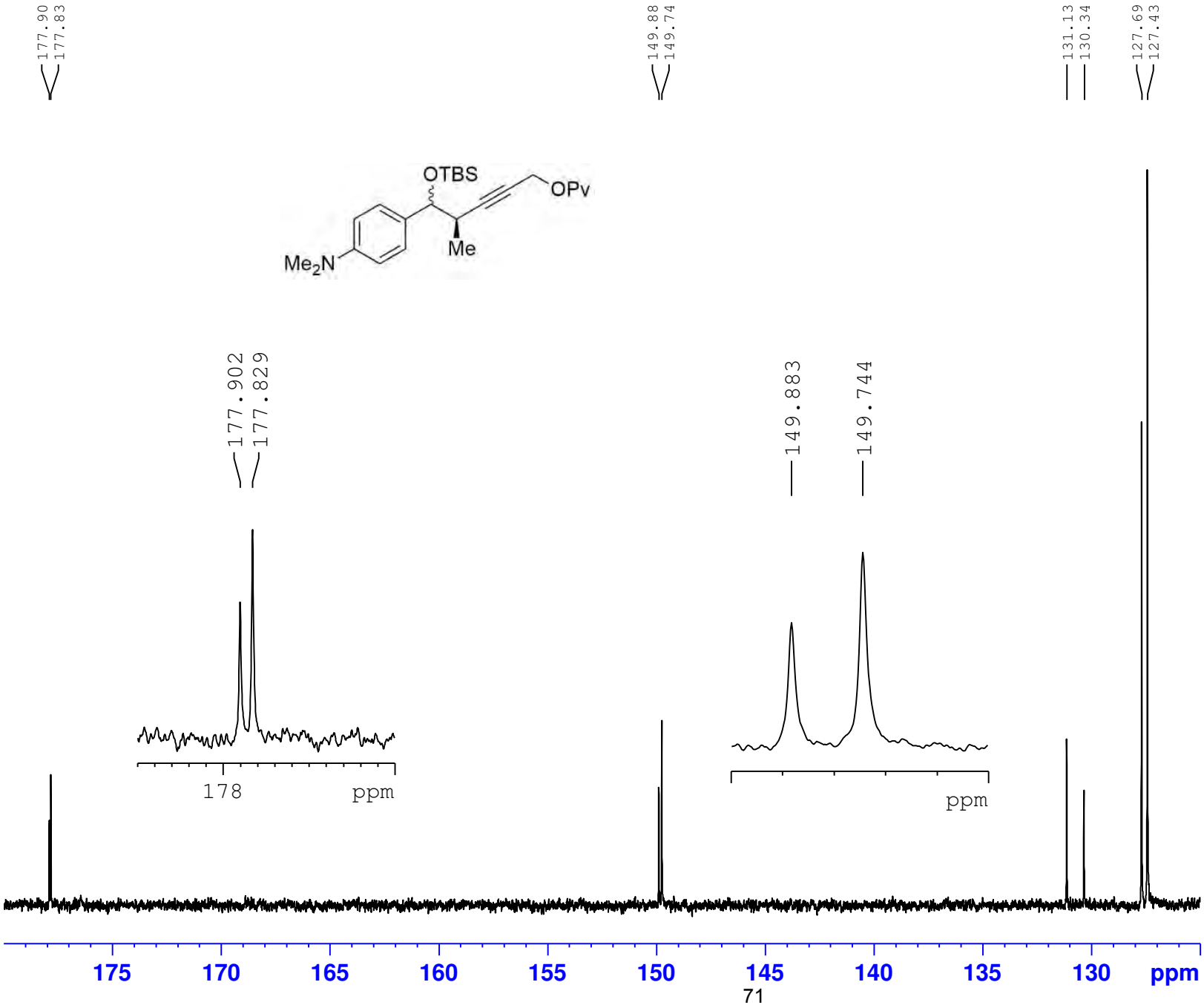
Current Data Parameters
NAME I-PK-21-03
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 4.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



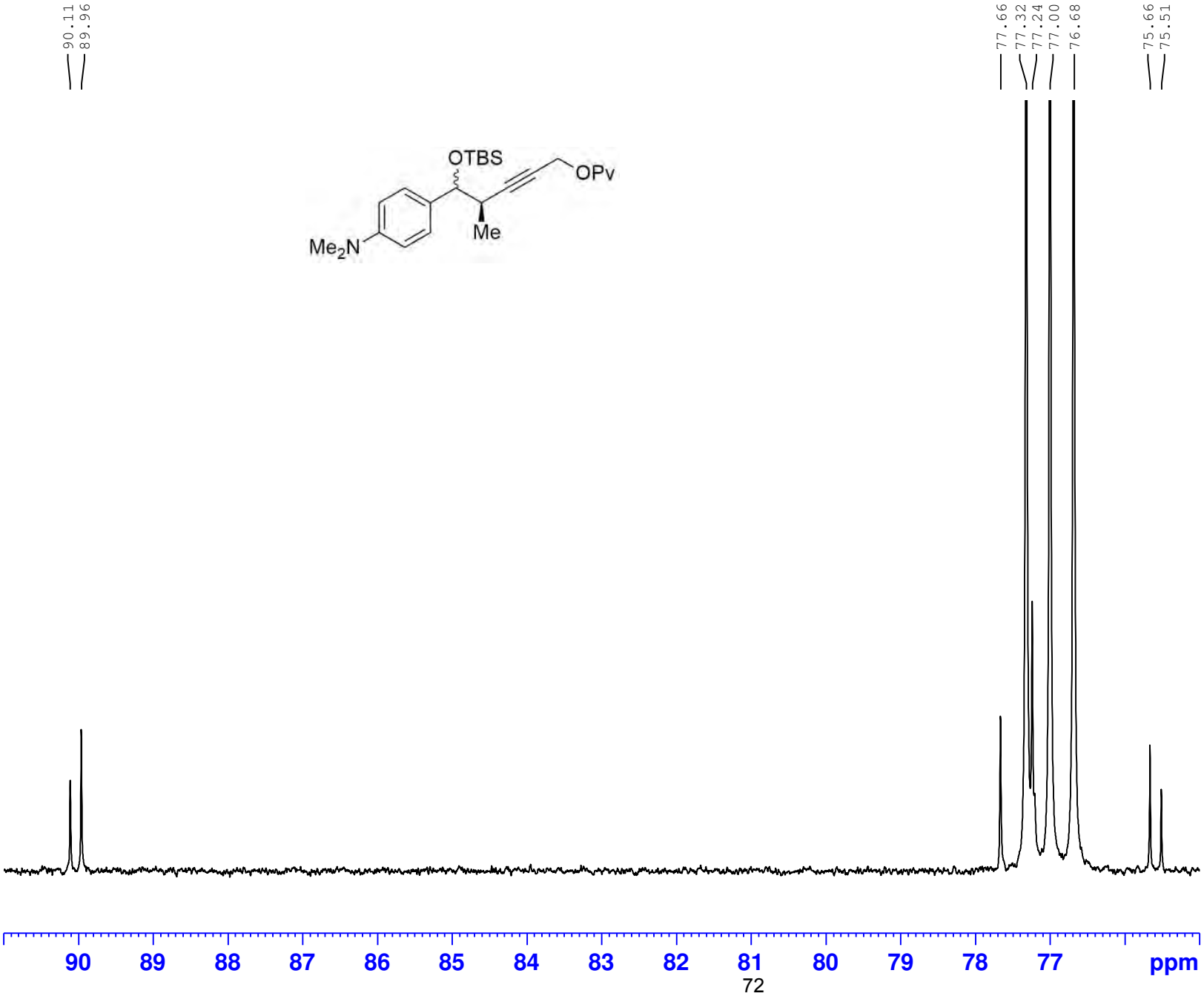
Current Data Parameters
NAME I-PK-21-03
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 4.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



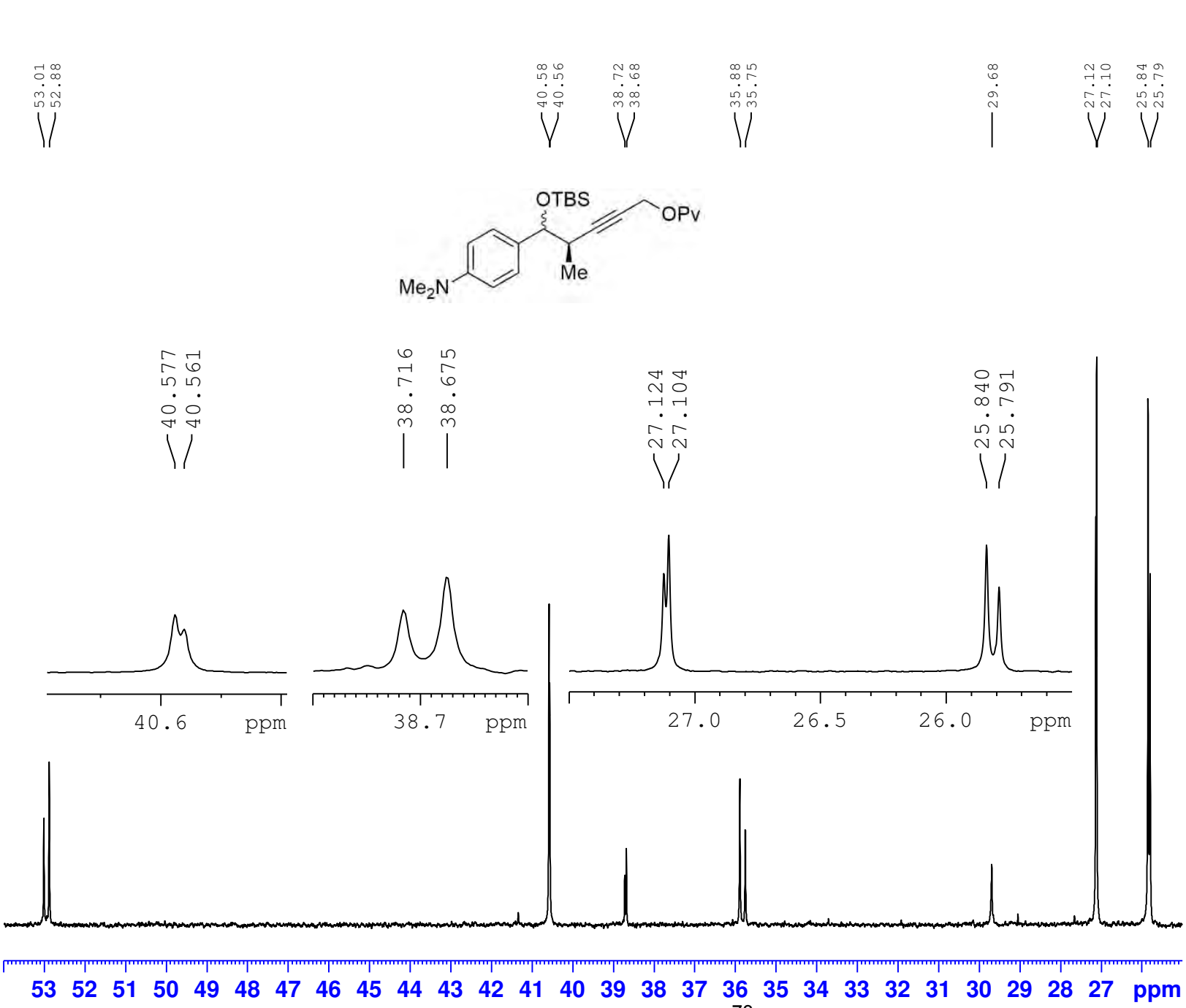
Current Data Parameters
NAME I-PK-21-03
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 4.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

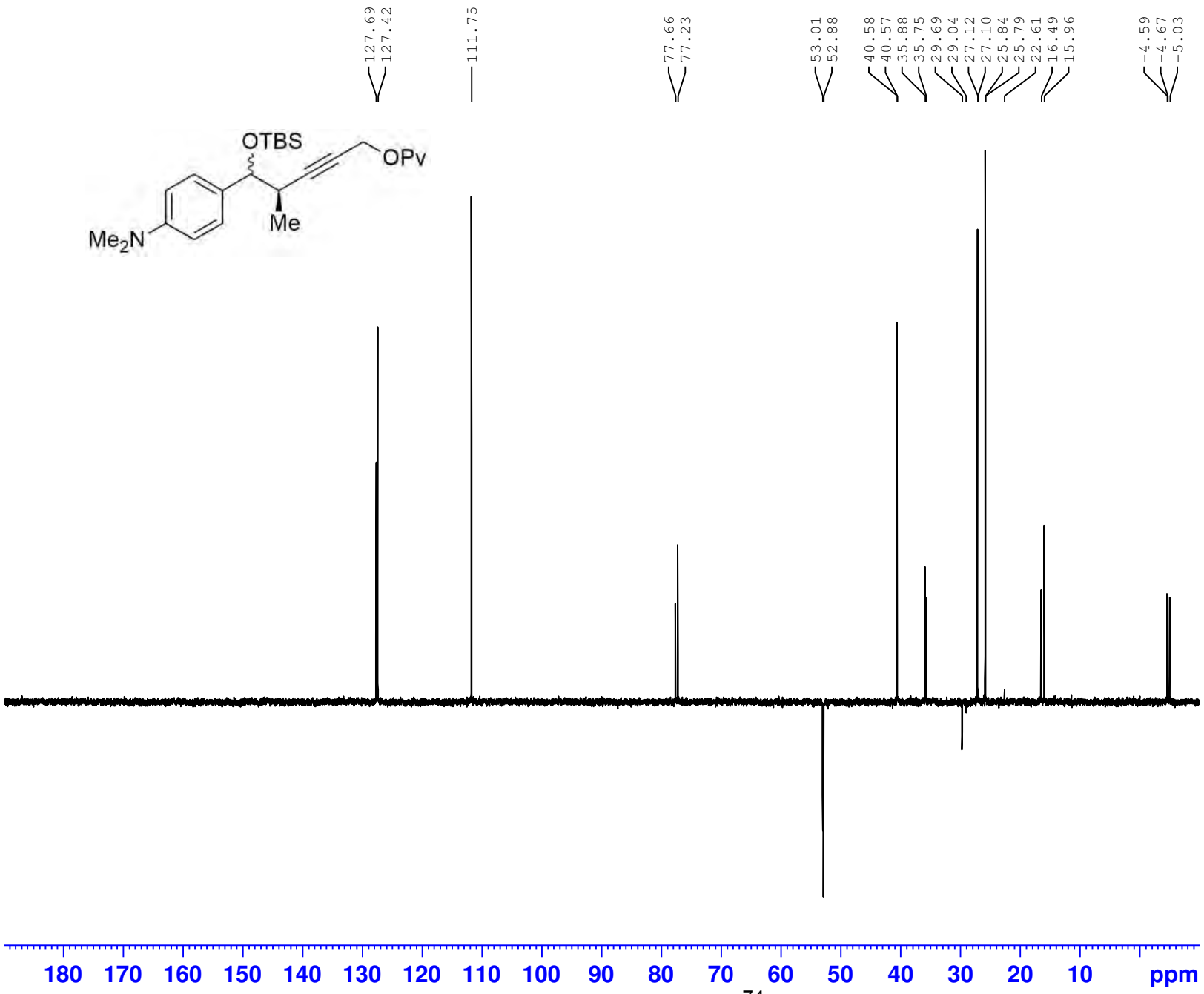
Current Data Parameters
NAME I-PK-21-03
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 4.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



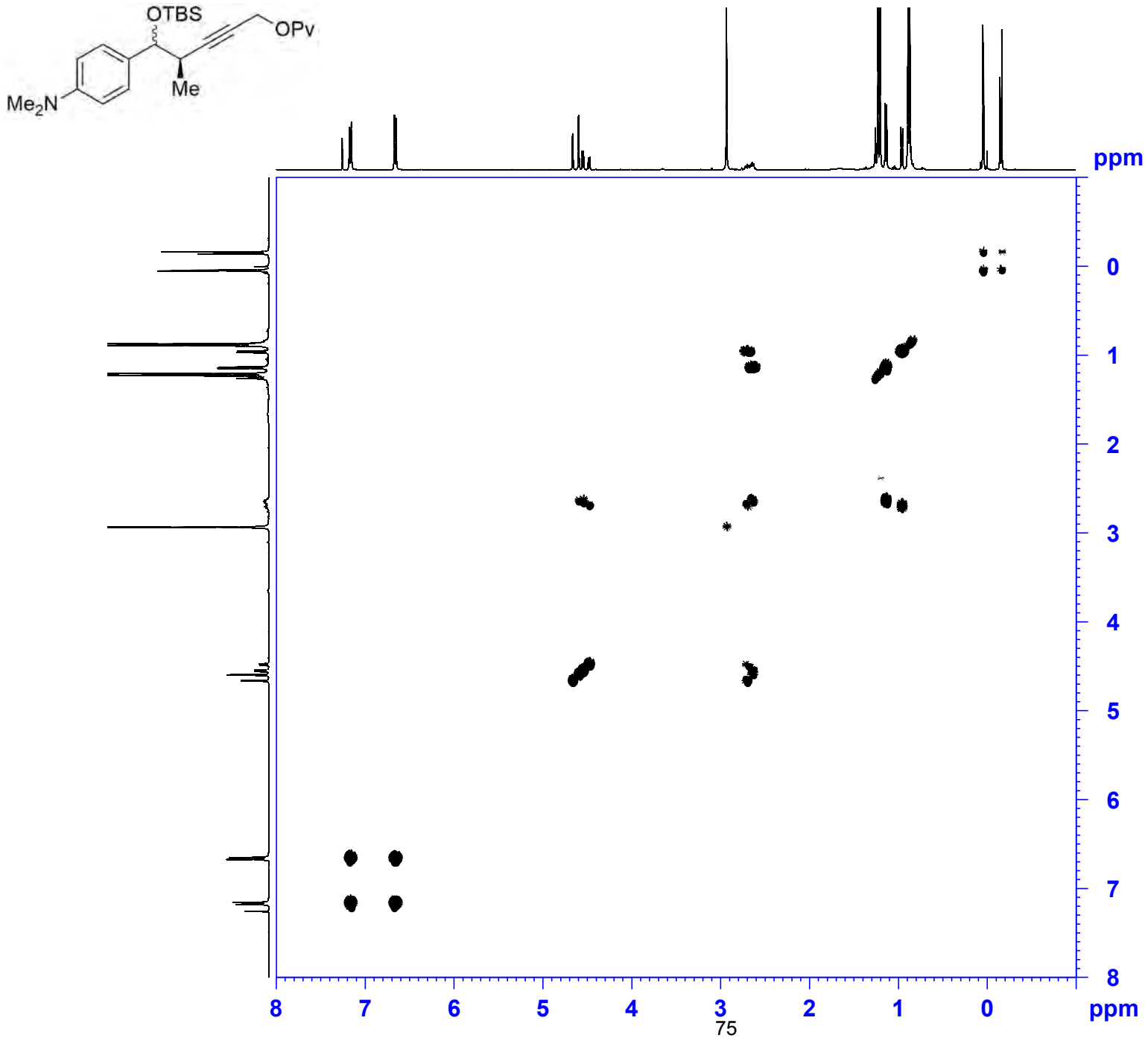
Current Data Parameters
NAME I-PK-21-03
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171205
Time 4.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME          I-PK-21-03
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171205
Time                4.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH             4472.272 Hz
FIDRES          2.183727 Hz
AQ              0.2289664 sec
RG                 2050
DW               111.800 usec
DE                 6.50 usec
TE                298.1 K
D0            0.00000300 sec
D1            0.92135590 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00022360 sec

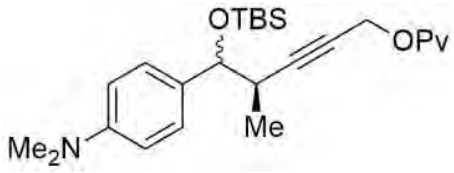
======== CHANNEL f1 ========
SFO1        399.9009709 MHz
NUC1               1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1            399.901 MHz
FIDRES         34.939625 Hz
SW                11.183 ppm
FnMODE               QF

F2 - Processing parameters
SI                 1024
SF          399.9000107 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF          399.9000122 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
```




```
Current Data Parameters
NAME          I-PK-21-03
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171205
Time                4.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    hmbcetgpl3nd
TD                  4096
SOLVENT            CDCl3
NS                     4
DS                    16
SWH           5208.333 Hz
FIDRES        1.271566 Hz
AQ            0.3932160 sec
RG                  2050
DW              96.000 usec
DE               6.50 usec
TE                298.0 K
CNST6       120.0000000
CNST7       175.0000000
CNST13        8.0000000
CNST30        0.5981122
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06250000 sec
D16         0.00020000 sec
IN0         0.00002070 sec

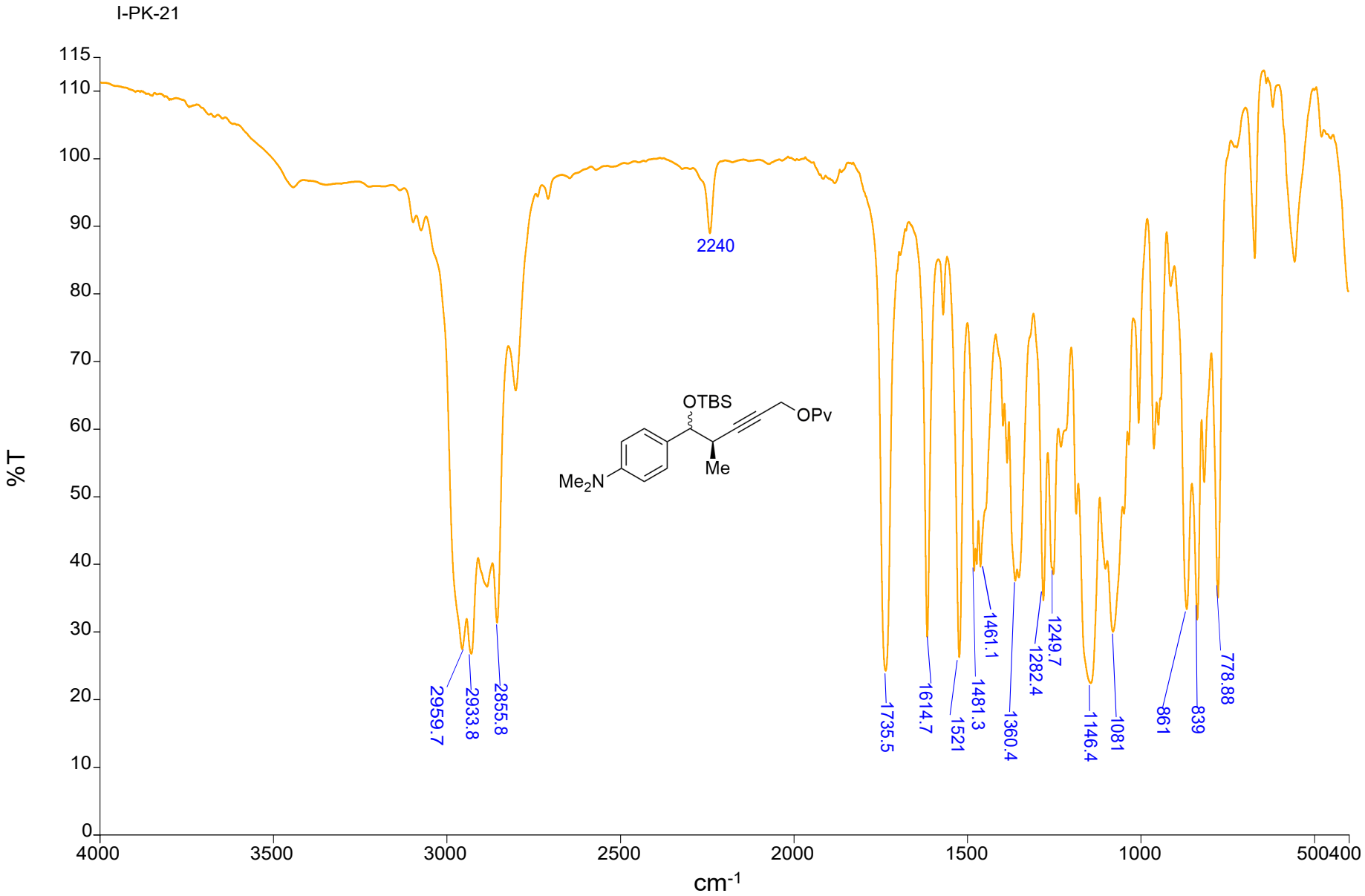
======== CHANNEL f1 ========
SFO1        399.9019995 MHz
NUC1                1H
P1                14.88 usec
P2                29.76 usec
PLW1         7.59999990 W

======== CHANNEL f2 ========
SFO2        100.5659947 MHz
NUC2                13C
P3               10.00 usec
P24            2000.00 usec
PLW2        44.46300125 W
SPNAM[7]    Crp60comp.4
SPOAL7            0.500
SPOFFS7  0 Hz
SPW7         6.79339981 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1             80.00 %
GPZ3             14.00 %
GPZ4             -8.00 %
GPZ5             -4.00 %
GPZ6             -2.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                  512
SFO1          100.566 MHz
FIDRES       94.353867 Hz
SW             240.186 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                 4096
SF          399.9000000 MHz
WDW                QSINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```


I-PK-21
%T
115
110
100
90
80
70
60
50
40
30
20
10
0
4000
3500
3000
2500
2000
1500
1000
500400
cm-1
2240
2959.7
2933.8
2855.8
1735.5
1614.7
1521
1481.3
1461.1
1360.4
1282.4
1249.7
1146.4
1081
861
839
778.88
OTBS
OPv
Me
Me2N

I-PK-21

5-12-2017

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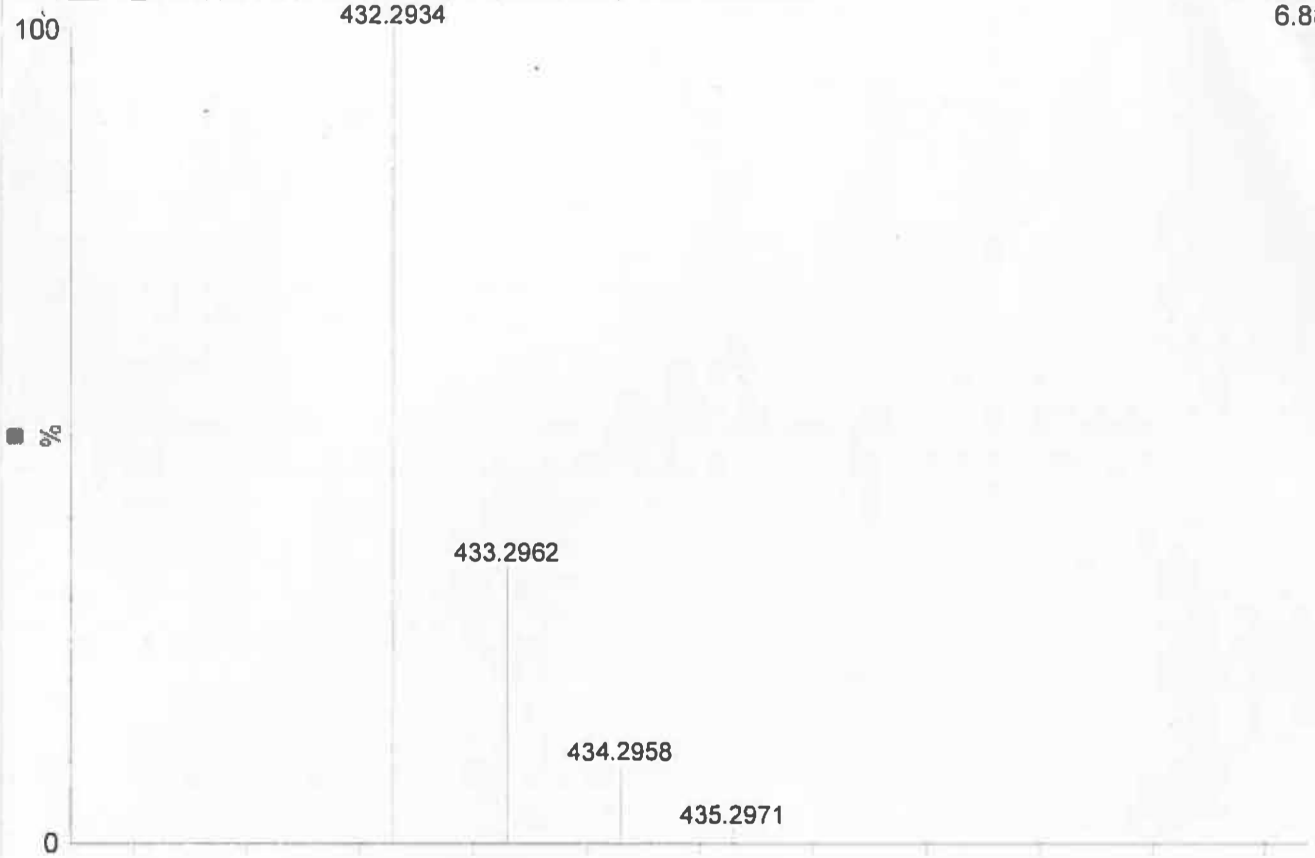
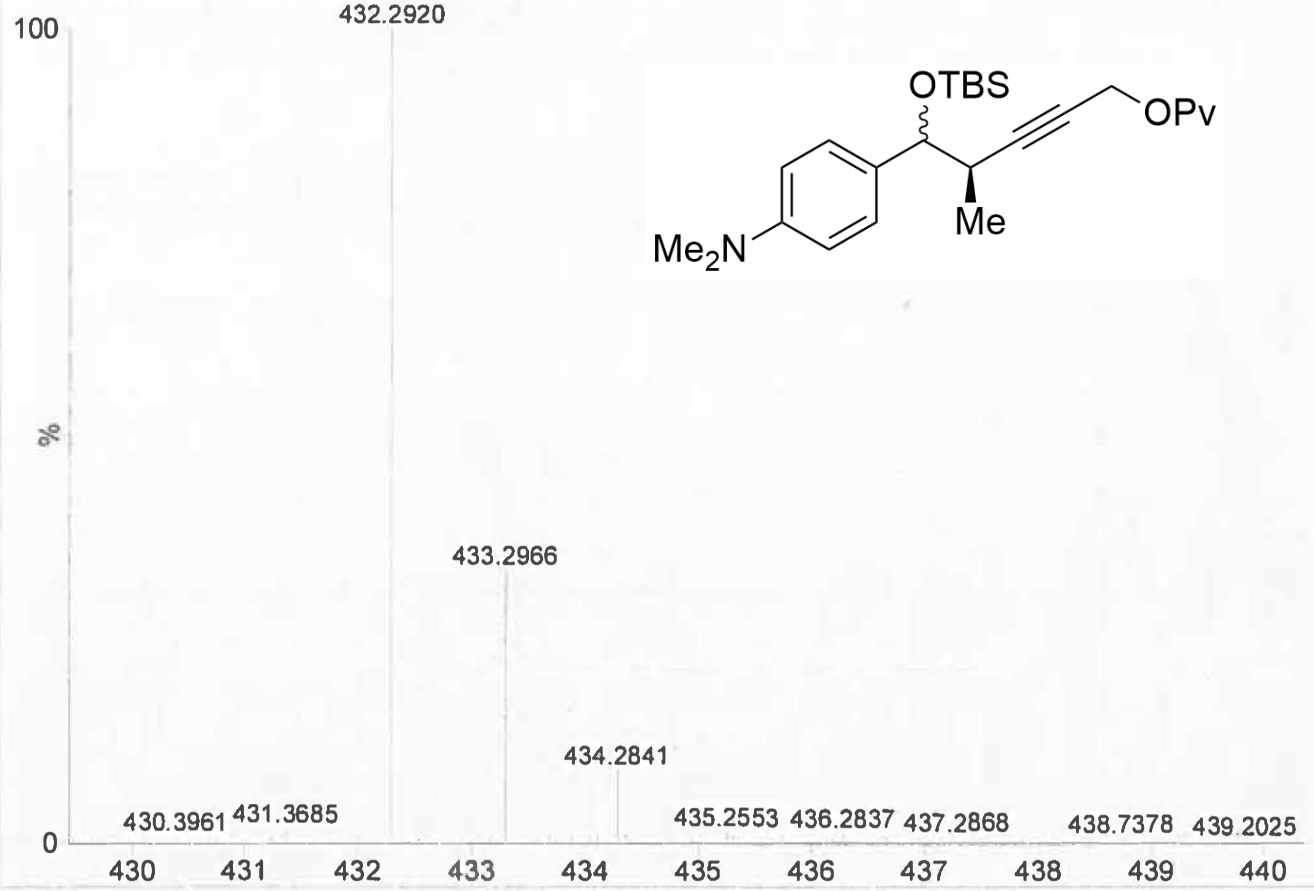
6.88e12

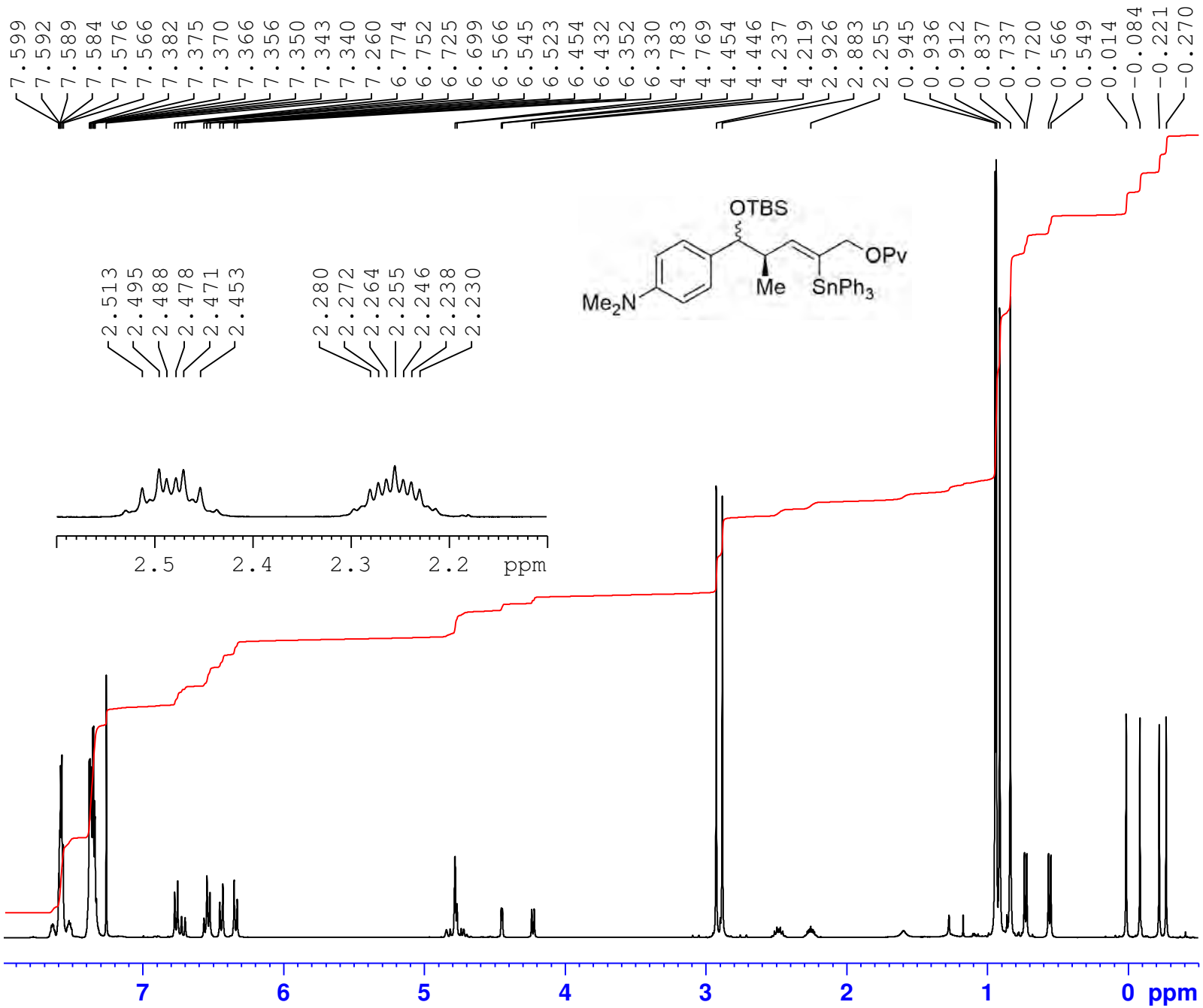


asep__15_NOV_2017_106 9 (0.429) Cm (9:10)

1: TOF MS ES+

652



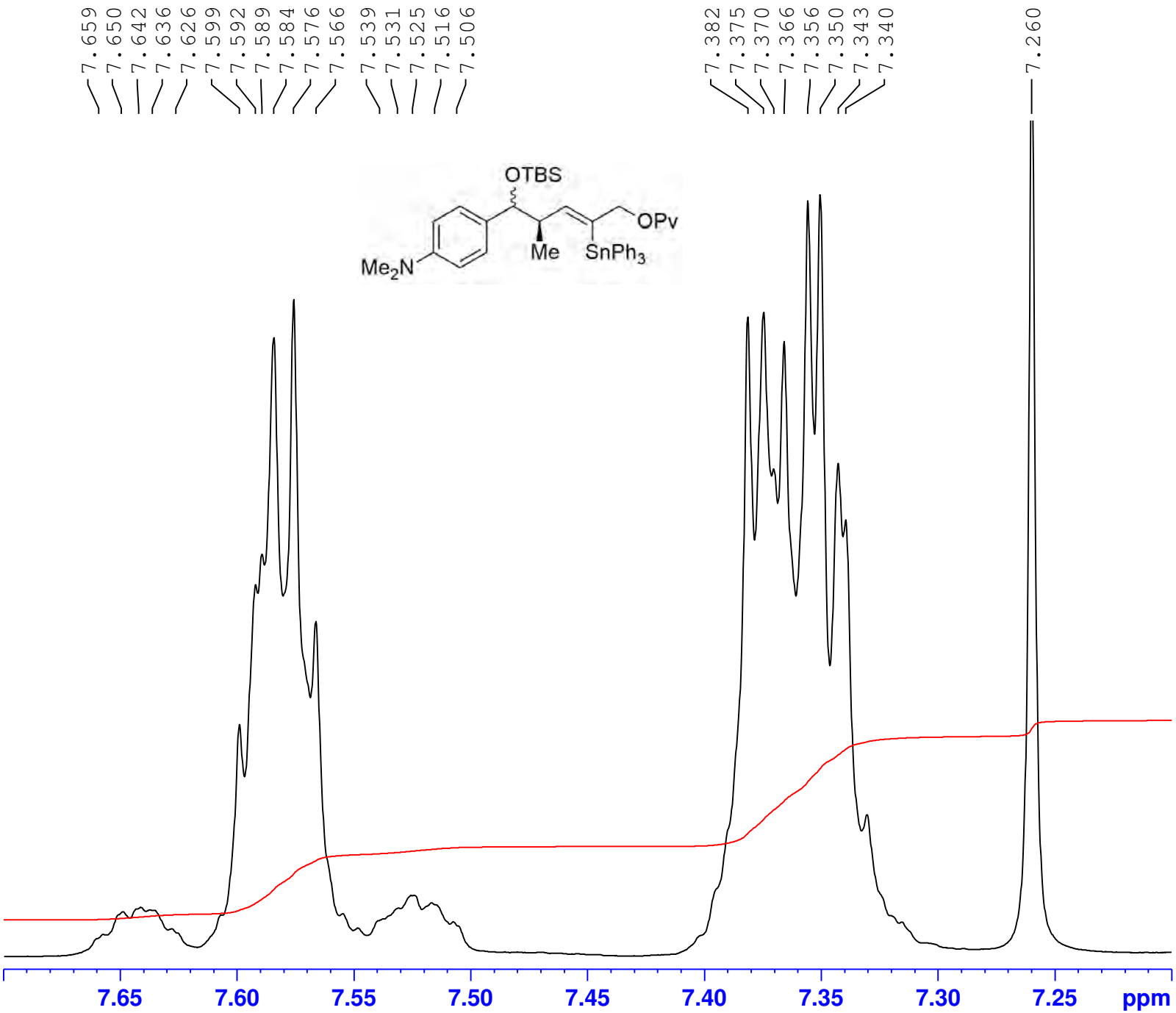


Current Data Parameters
NAME I-PK-48
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 7.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 296.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

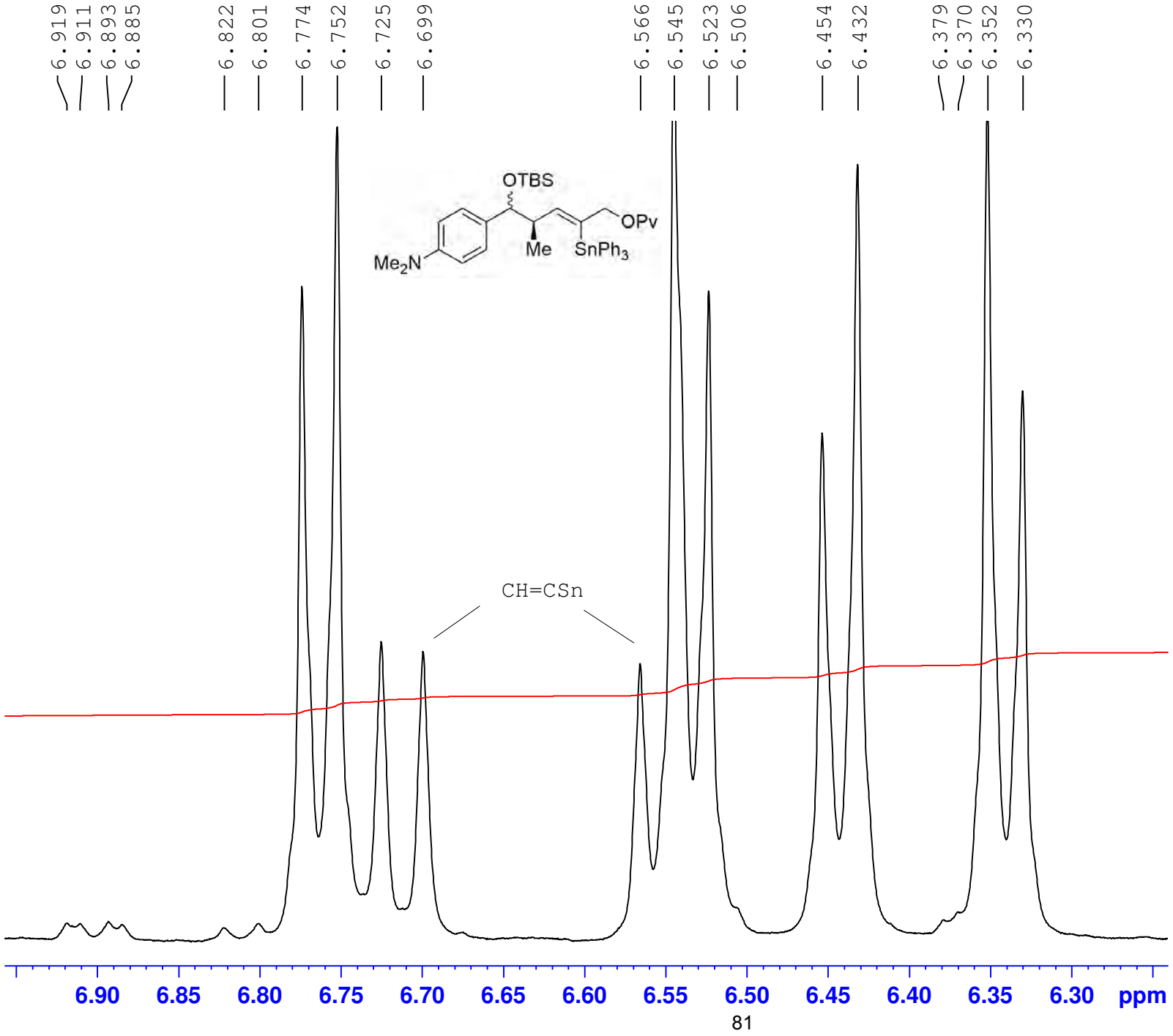


Current Data Parameters
NAME I-PK-48
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 7.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 296.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

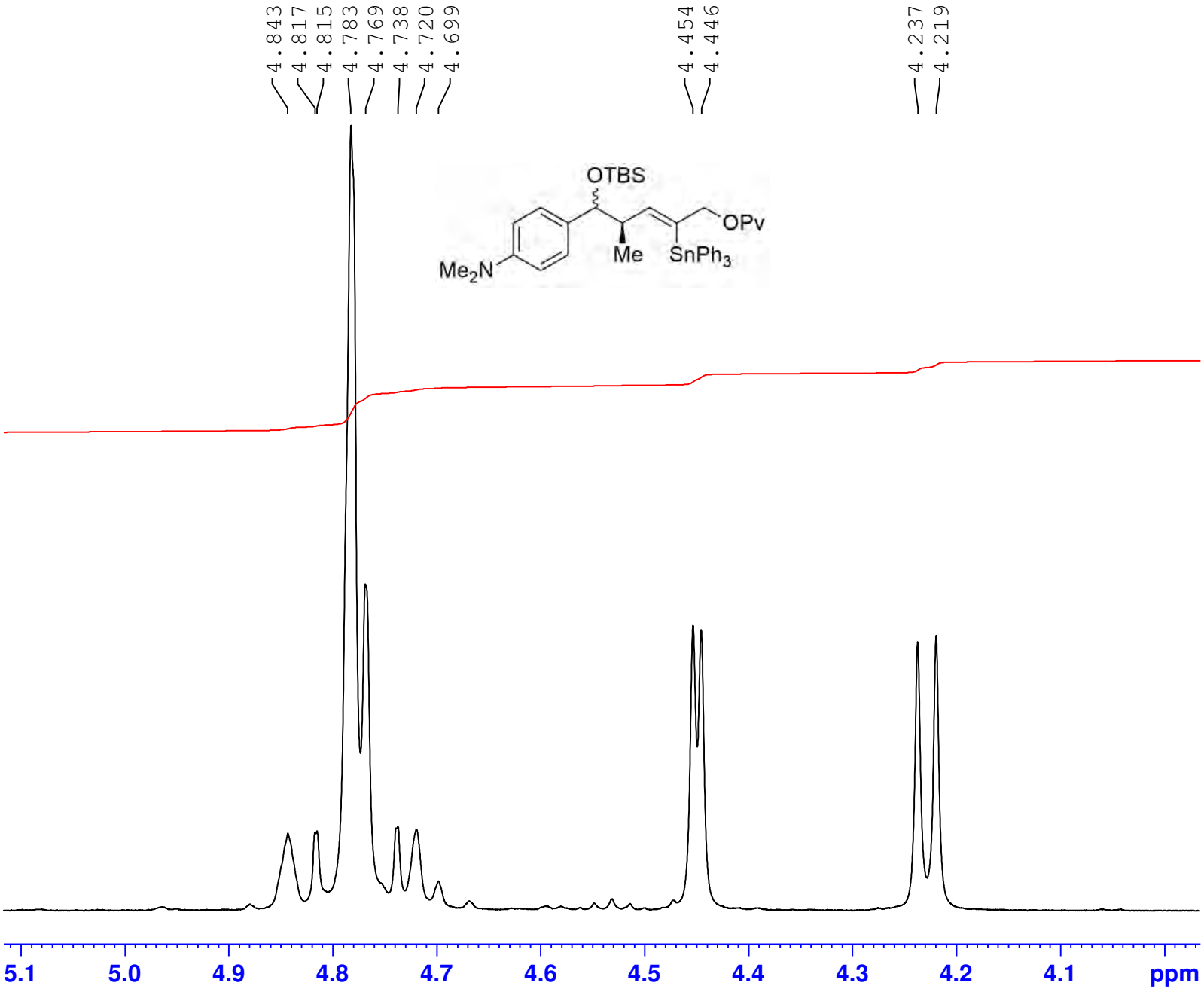


Current Data Parameters
NAME I-PK-48
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 7.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 296.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

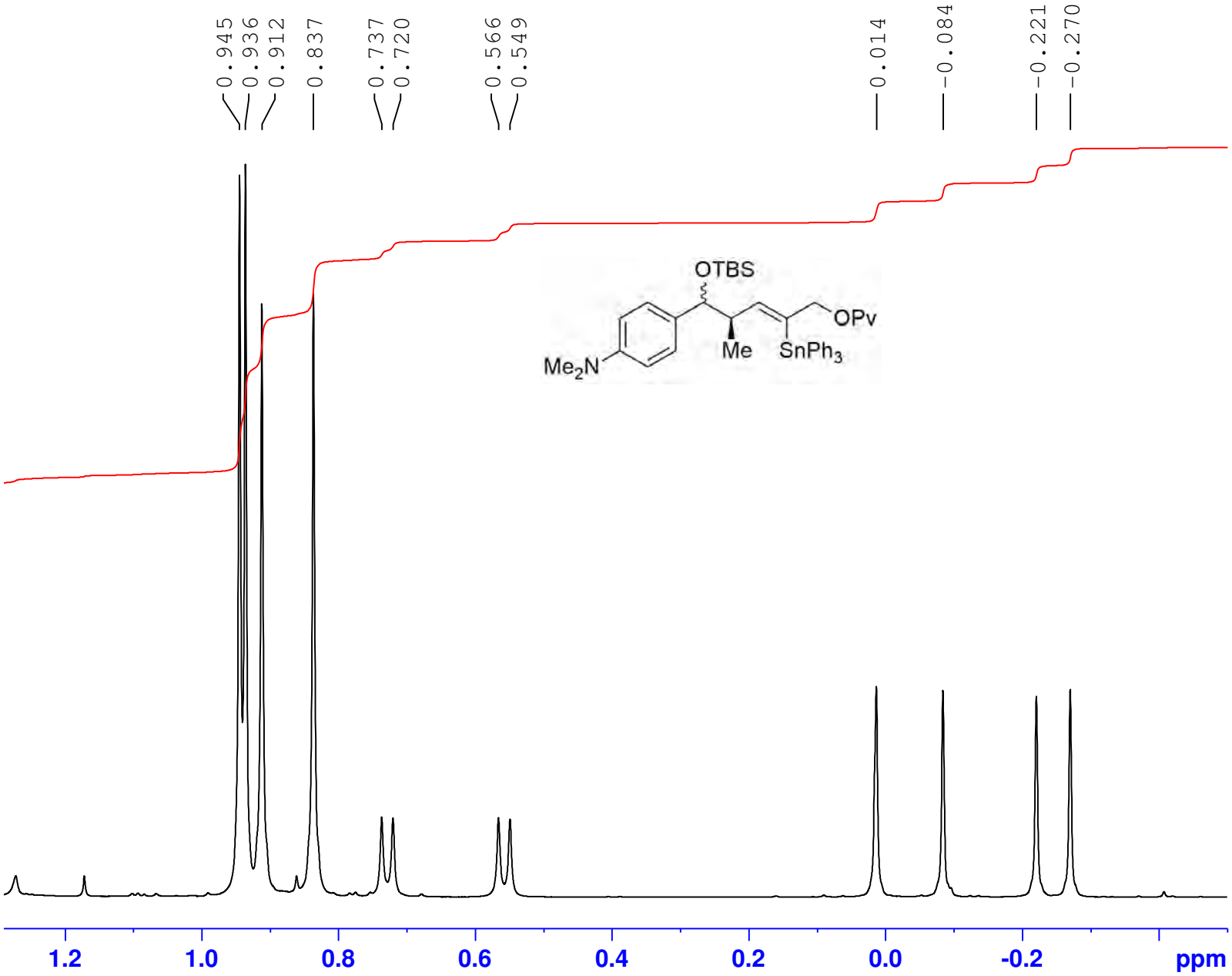


Current Data Parameters
NAME I-PK-48
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 7.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 45.2
DW 41.600 usec
DE 9.85 usec
TE 296.4 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

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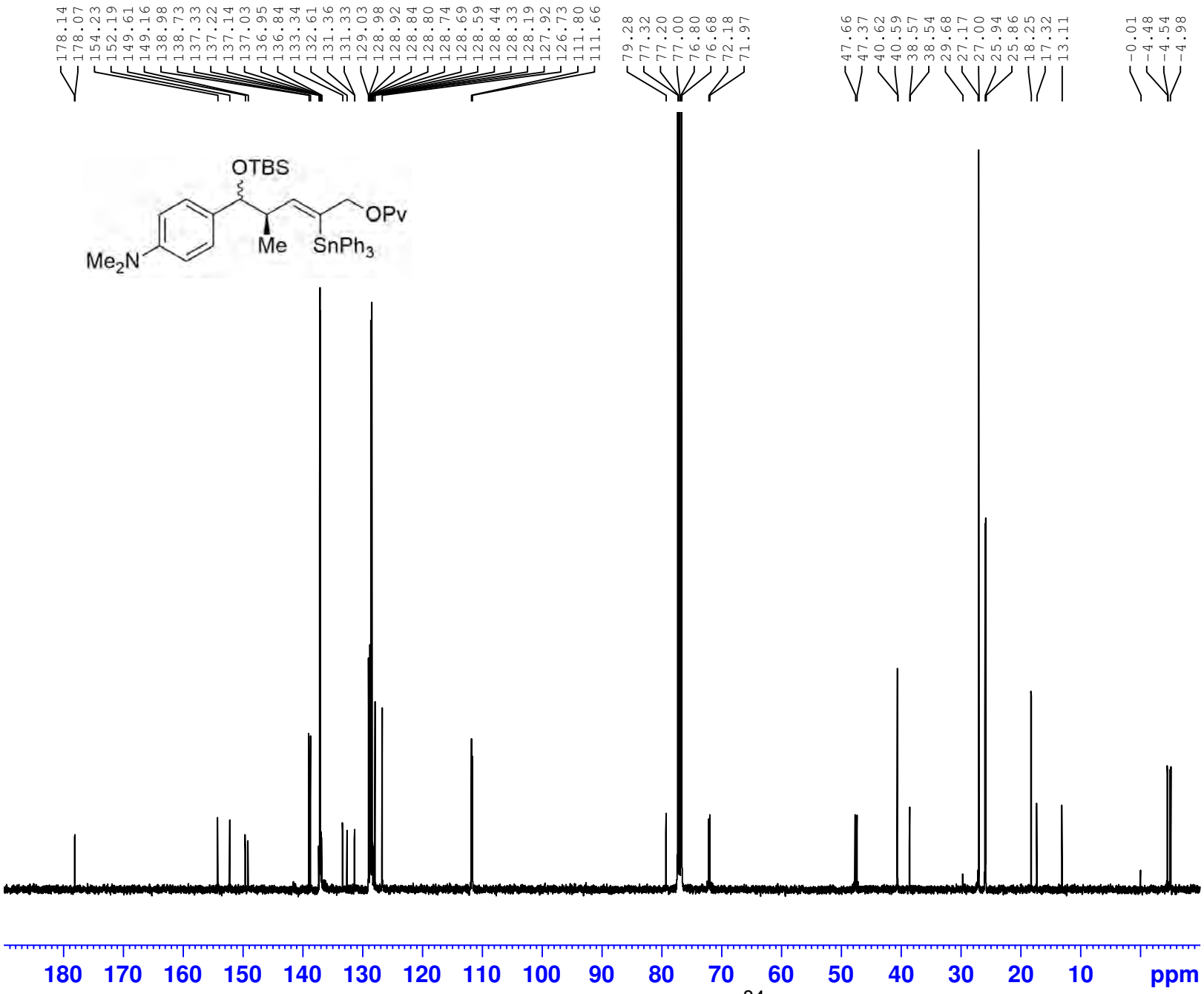
Current Data Parameters
NAME                  I-PK-48
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180224
Time                    7.58
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                    131072
SOLVENT                CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                       45.2
DW                     41.600 usec
DE                      9.85 usec
TE                     296.4 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                    131072
SF               399.9000096 MHz
WDW                       EM
SSB                        0
LB                      0.10 Hz
GB                         0
PC                      1.00
```



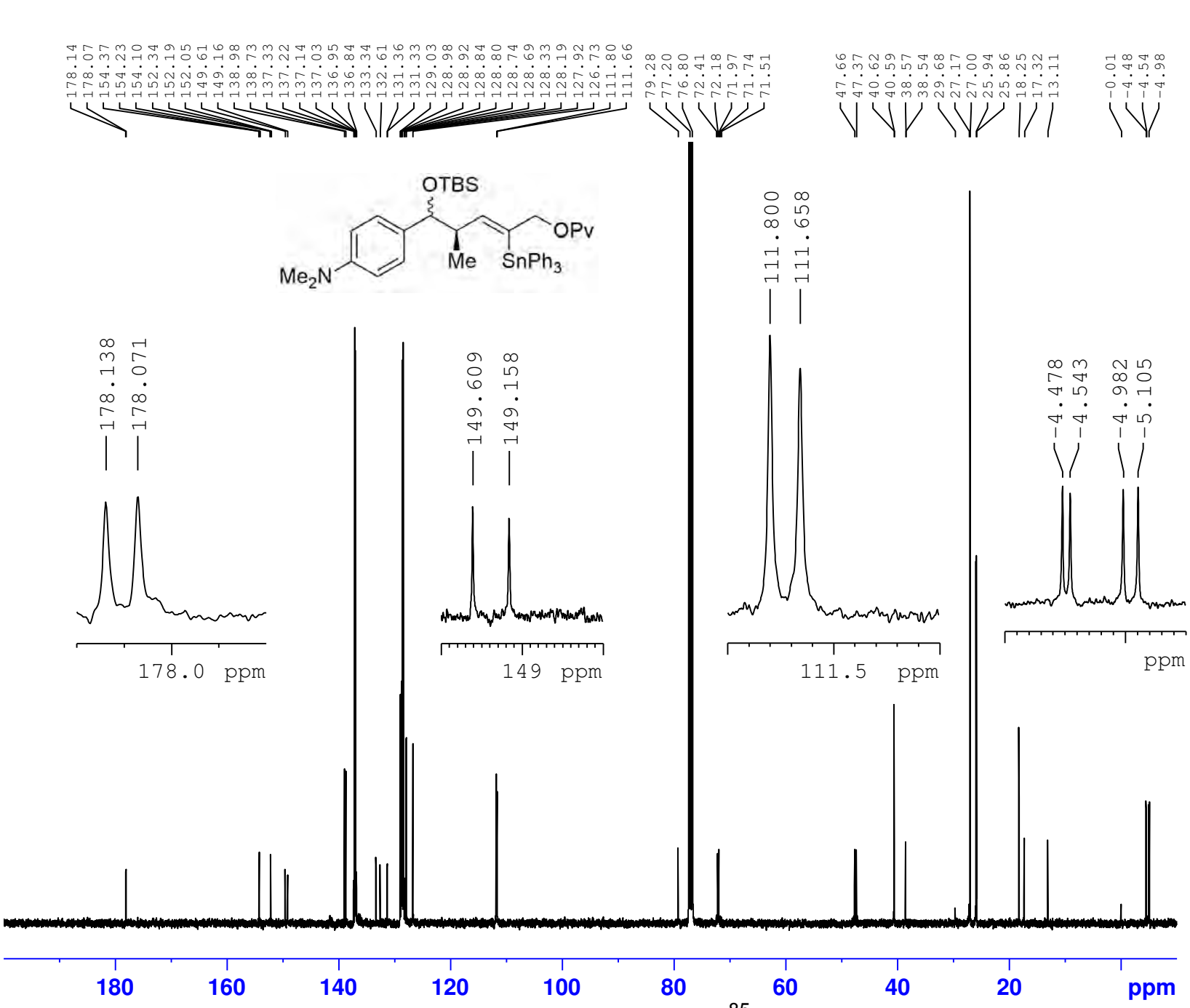
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



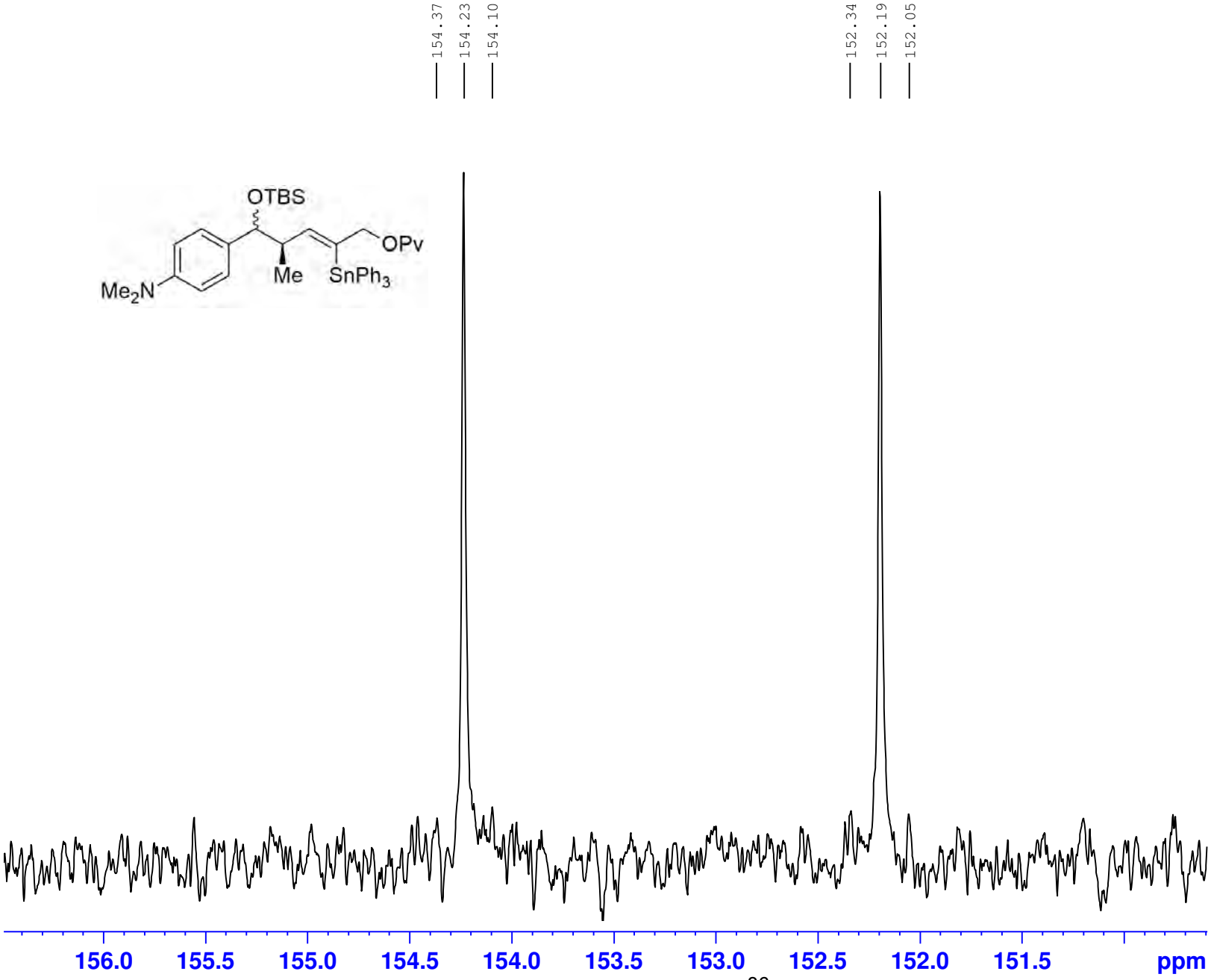
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



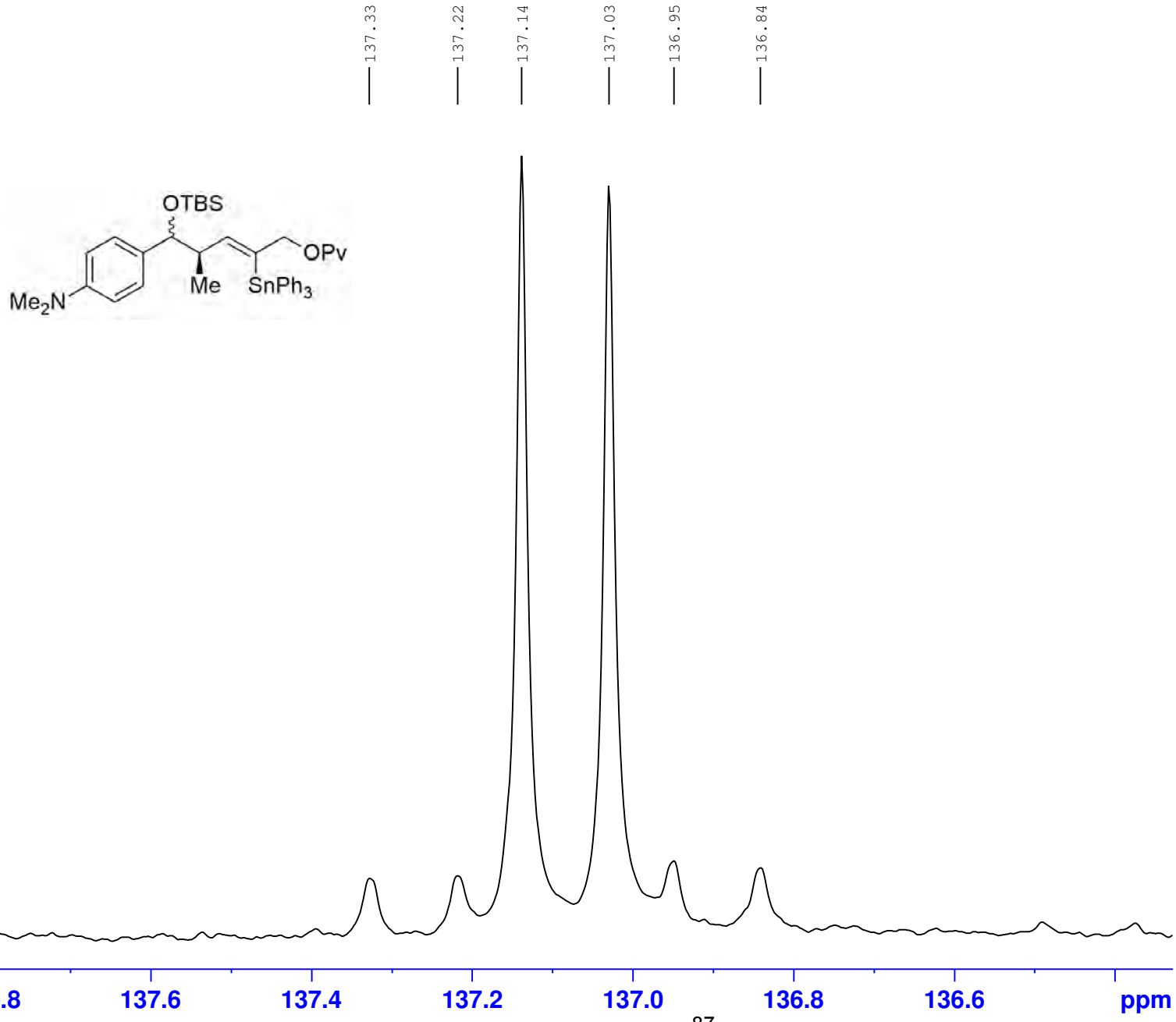
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



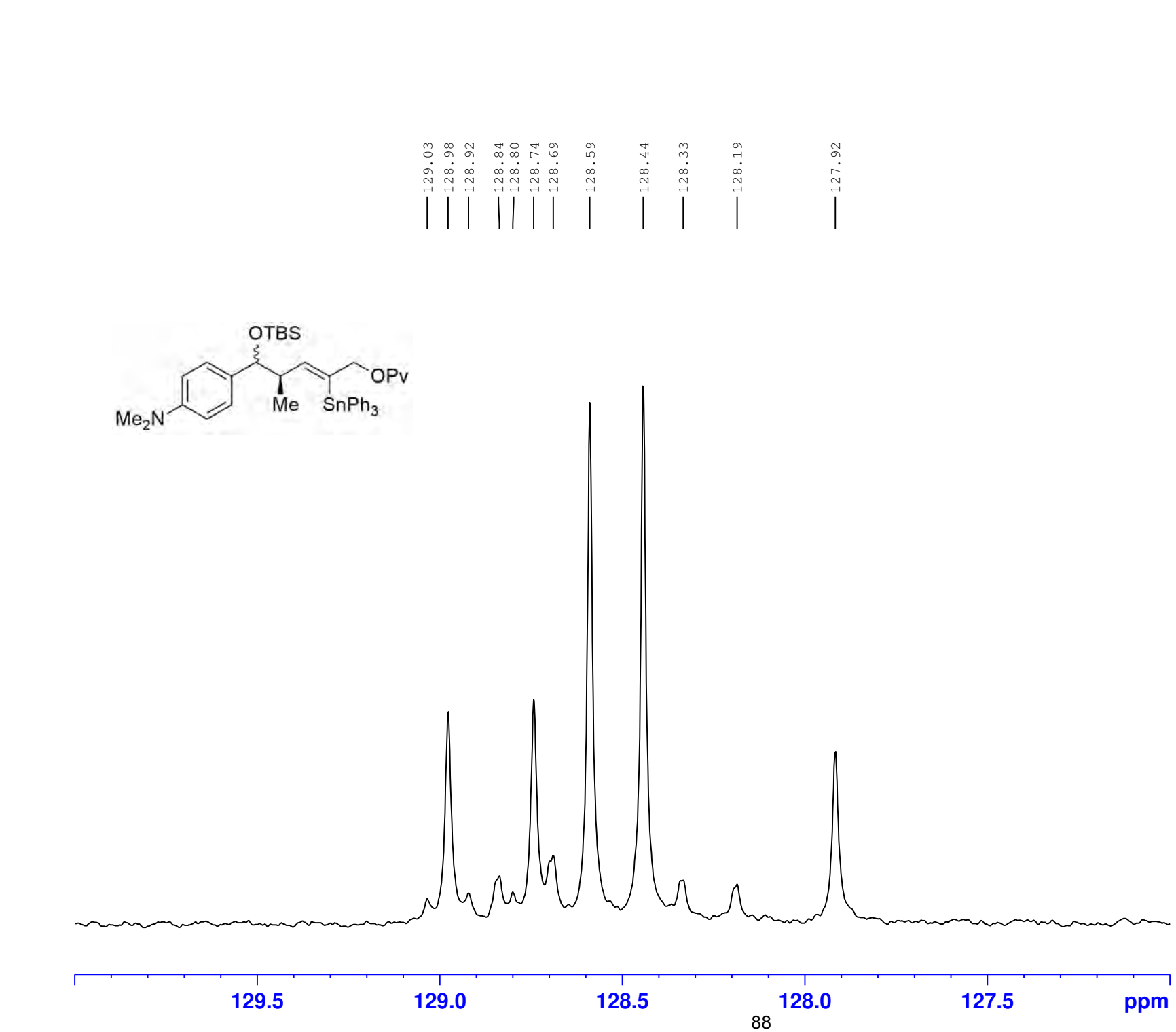
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



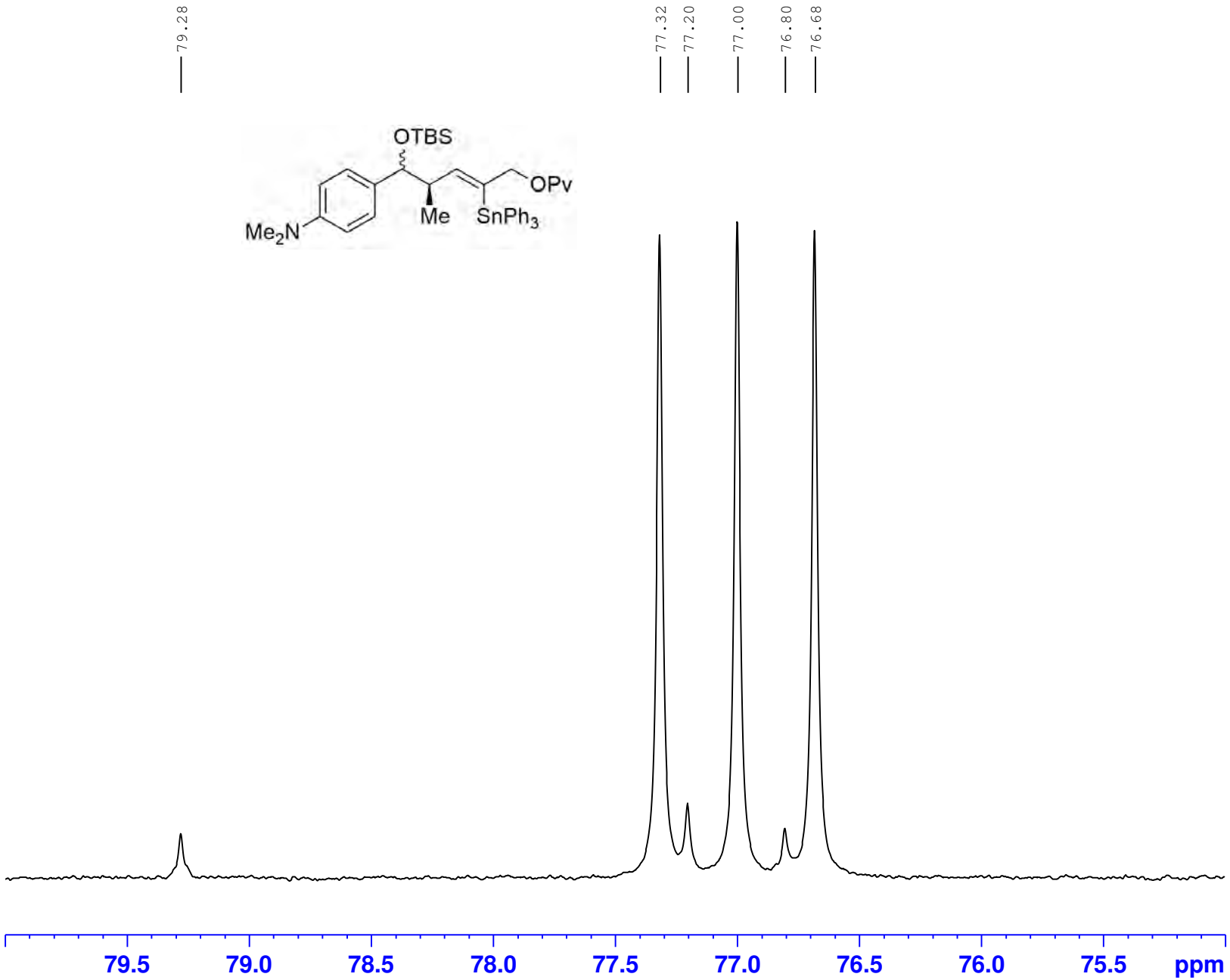
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



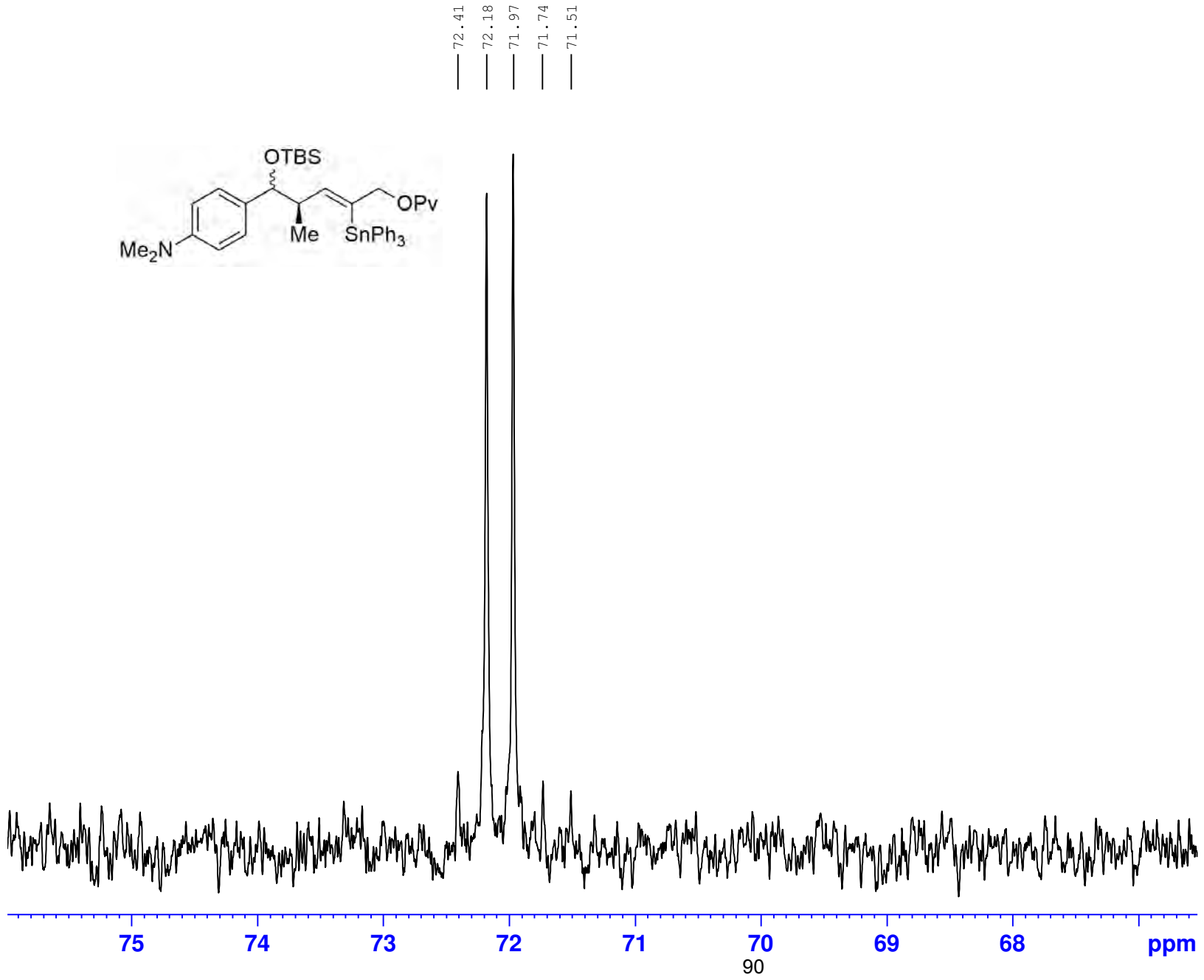
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



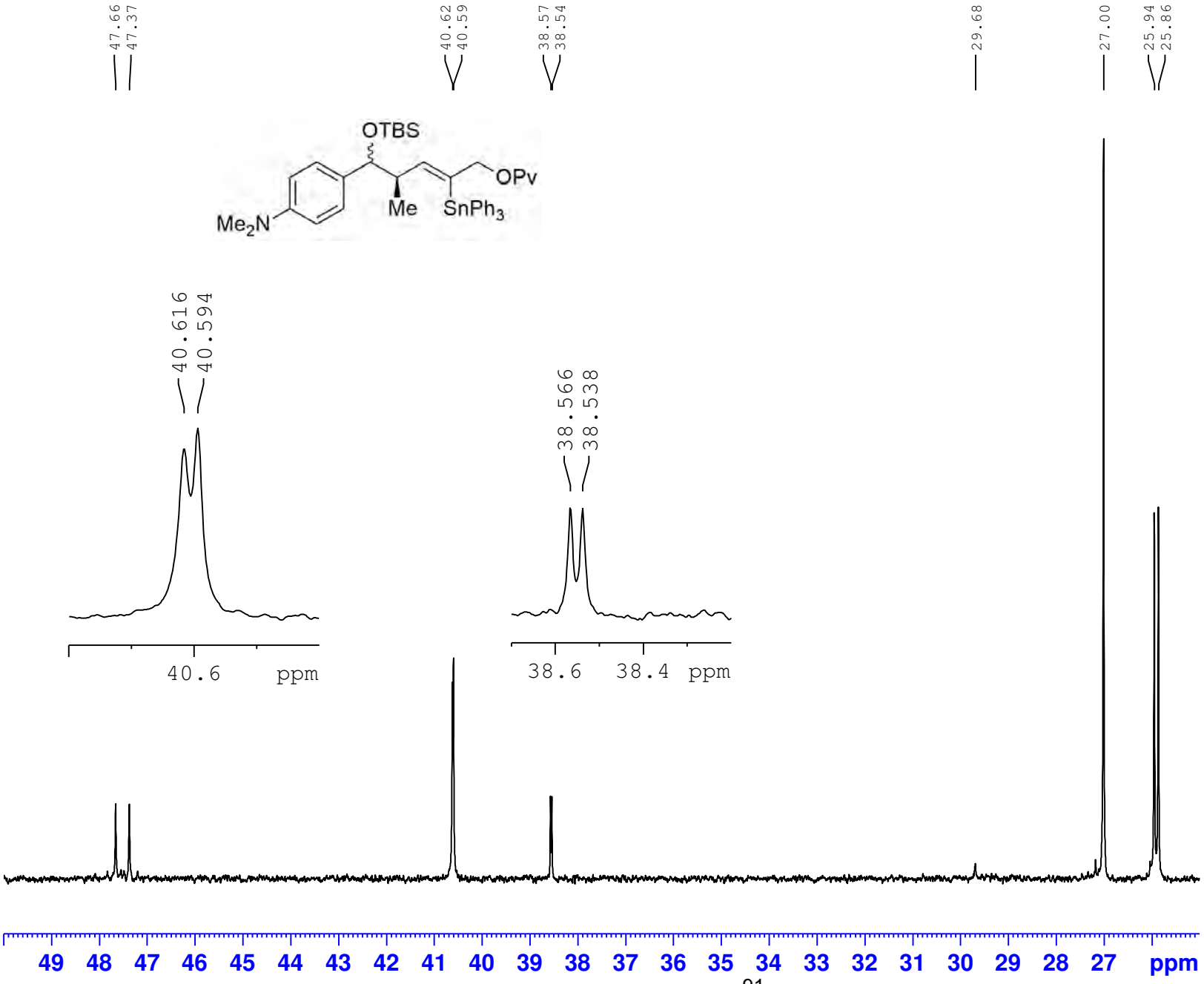
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



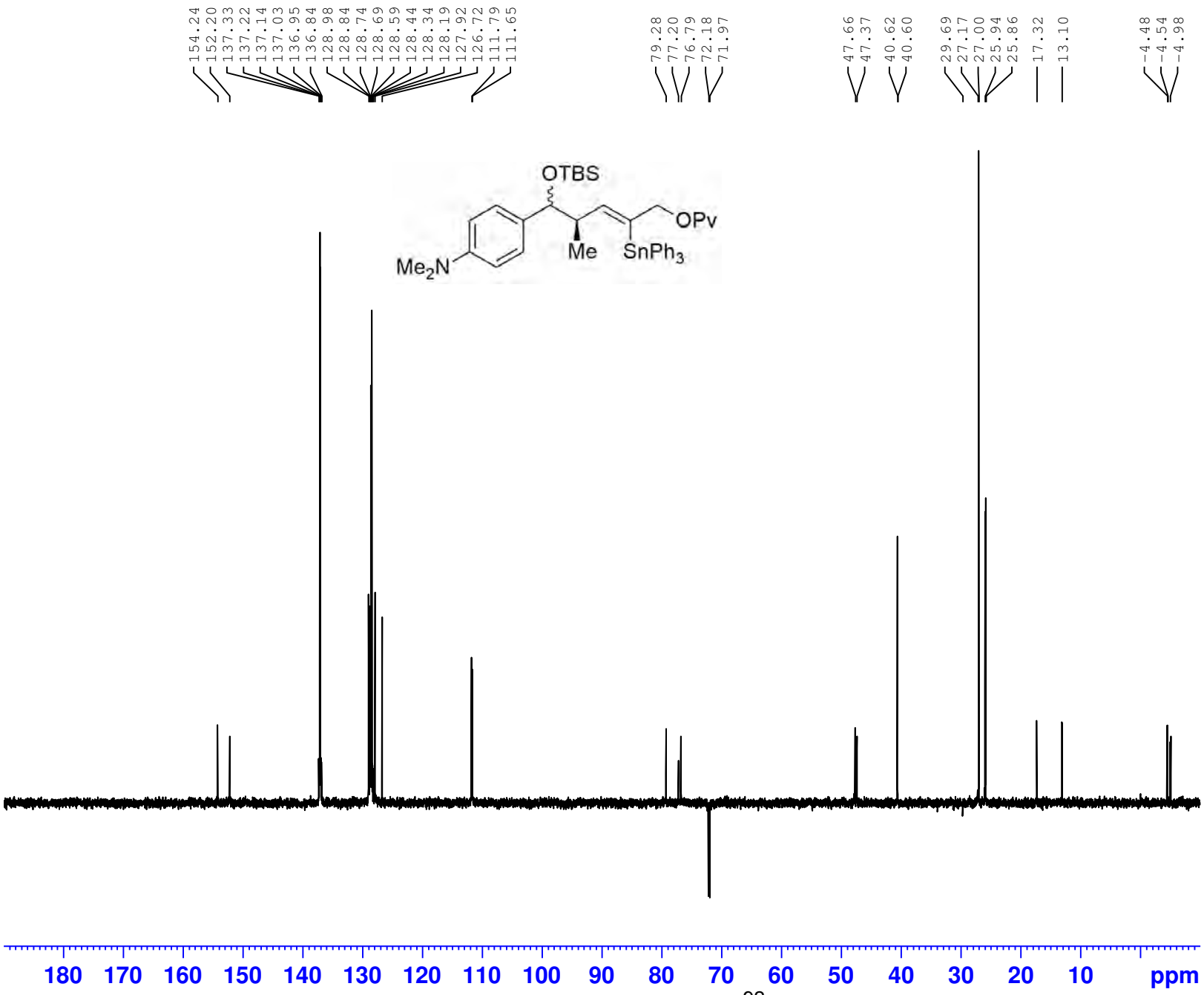
Current Data Parameters
NAME I-PK-48
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180224
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	I-PK-48
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20180224
Time	9.27
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	296.9 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

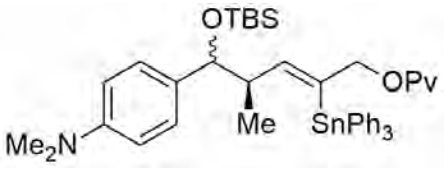
SFO1	100.5649905 MHz
NUC1	13C
P1	10.00 usec
P13	2000.00 usec
PLW0	0 W
PLW1	44.46300125 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	6.79339981 W

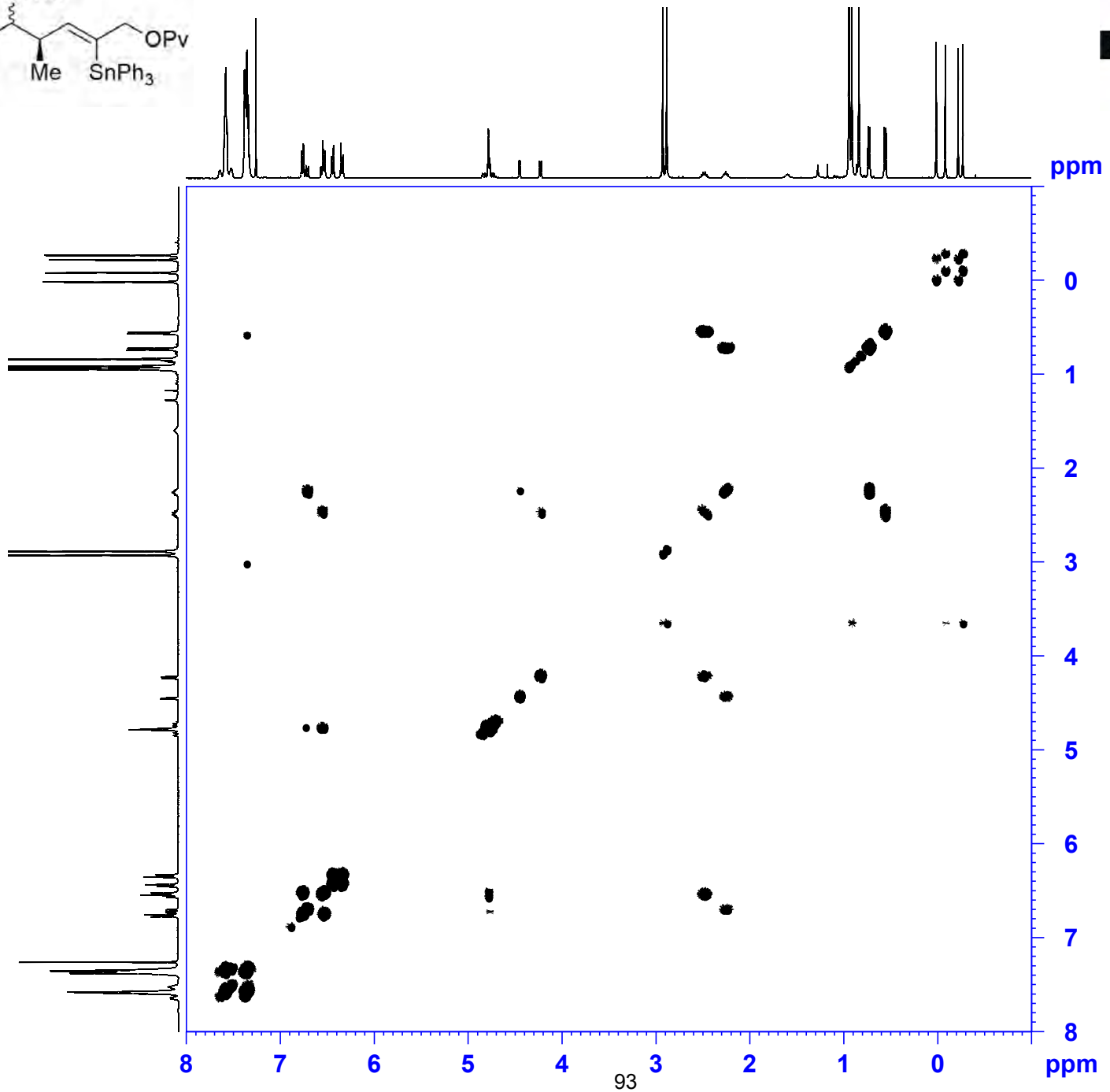
======== CHANNEL f2 ========

SFO2	399.9012789 MHz
NUC2	1H
CPDPRG[2	waltz16
P3	14.88 usec
P4	29.76 usec
PCPD2	90.00 usec
PLW2	7.59999990 W
PLW12	0.20774999 W

F2 - Processing parameters

SI	32768
SF	100.5549395 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40






```
Current Data Parameters
NAME                  I-PK-48
EXPNO                      14
PROCNO                      1

F2 - Acquisition Parameters
Date_                20180224
Time                     9.30
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                       2048
SOLVENT                 CDCl3
NS                          1
DS                          8
SWH                  3795.547 Hz
FIDRES               1.853294 Hz
AQ                  0.2697899 sec
RG                       2050
DW                    131.733 usec
DE                      6.50 usec
TE                     296.6 K
D0                  0.00000300 sec
D1                  0.87629992 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D13                 0.00000400 sec
D16                 0.00020000 sec
IN0                 0.00026340 sec

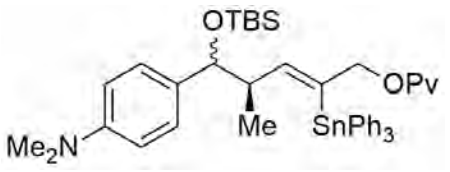
======== CHANNEL f1 ========
SFO1             399.9014763 MHz
NUC1                       1H
P1                      14.88 usec
P17                   2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

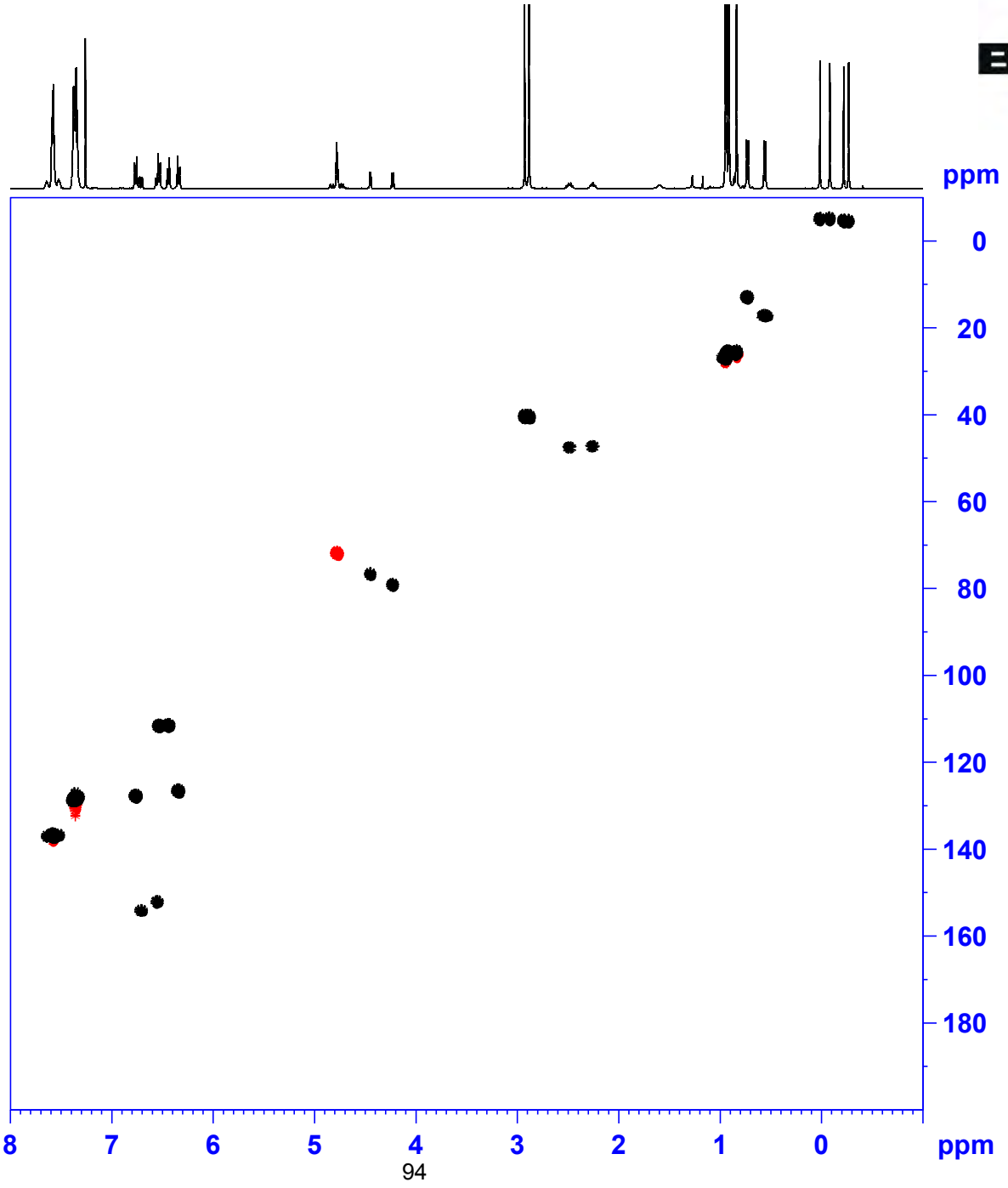
====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPNAM[3]          SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1                399.9015 MHz
FIDRES             29.660213 Hz
SW                      9.494 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                399.9000100 MHz
WDW                       SINE
SSB          0
LB           0 Hz
GB           0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                399.9000144 MHz
WDW                       SINE
SSB          0
LB           0 Hz
GB           0
```






```
Current Data Parameters
NAME                  I-PK-48
EXPNO                      15
PROCNO                      1

F2 - Acquisition Parameters
Date_                20180224
Time                     9.37
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                       1024
SOLVENT                  CDCl3
NS                          2
DS                         32
SWH                  4807.692 Hz
FIDRES               4.695012 Hz
AQ                   0.1064960 sec
RG                        2050
DW                    104.000 usec
DE                      6.50 usec
TE                     296.6 K
CNST2               145.0000000
D0                   0.00000300 sec
D1                   0.80000001 sec
D4                   0.00172414 sec
D11                  0.03000000 sec
D16                  0.00020000 sec
D21                  0.00360000 sec
IN0                  0.00001910 sec

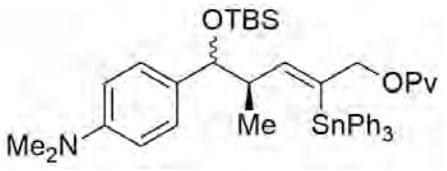
======== CHANNEL f1 ========
SFO1               399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28            0 usec
PLW1                7.59999990 W

======== CHANNEL f2 ========
SFO2               100.5670016 MHz
NUC2                       13C
CPDPRG[2                 garp4
P3                      10.00 usec
P14                    500.00 usec
P31                   1900.00 usec
PCPD2                   80.00 usec
PLW0           0 W
PLW2               44.46300125 W
PLW12               0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                   0.500
SPOFFS3  0 Hz
SPW3                6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                  0.500
SPOFFS18 0 Hz
SPW18               1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                    80.00 %
GPZ2                    20.10 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1                 100.567 MHz
FIDRES               204.515701 Hz
SW                   260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF                 399.9000091 MHz
WDW                      OSINE
```




```
Current Data Parameters
NAME                 I-PK-48
EXPNO                     16
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180224
Time                   9.48
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG     hmbcetgpl3nd
TD                      4096
SOLVENT              CDCl3
NS                         4
DS                        16
SWH                5208.333 Hz
FIDRES             1.271566 Hz
AQ                 0.3932160 sec
RG                      2050
DW                   96.000 usec
DE                    6.50 usec
TE                   296.5 K
CNST6            120.0000000
CNST7            175.0000000
CNST13             8.0000000
CNST30             0.5981122
D0                 0.00000300 sec
D1                 1.50000000 sec
D6                 0.06250000 sec
D16                0.00020000 sec
IN0                0.00002070 sec

======== CHANNEL f1 ========
SFO1             399.9019995 MHz
NUC1                     1H
P1                    14.88 usec
P2                    29.76 usec
PLW1              7.59999990 W

======== CHANNEL f2 ========
SFO2             100.5659947 MHz
NUC2                    13C
P3                    10.00 usec
P24                 2000.00 usec
PLW2             44.46300125 W
SPNAM[7]      Crp60comp.4
SPOAL7                0.500
SPOFFS7   0 Hz
SPW7              6.79339981 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[3]       SMSQ10.100
GPNAM[4]       SMSQ10.100
GPNAM[5]       SMSQ10.100
GPNAM[6]       SMSQ10.100
GPZ1                 80.00 %
GPZ3                 14.00 %
GPZ4                 -8.00 %
GPZ5                 -4.00 %
GPZ6                 -2.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       512
SFO1               100.566 MHz
FIDRES           94.353867 Hz
SW                 240.186 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      4096
SF               399.9000097 MHz
WDW                    QSINE
SSB       0
LB        0 Hz
GB        0
PC                     1.40
```

Mass Spectrometry Result Sheet

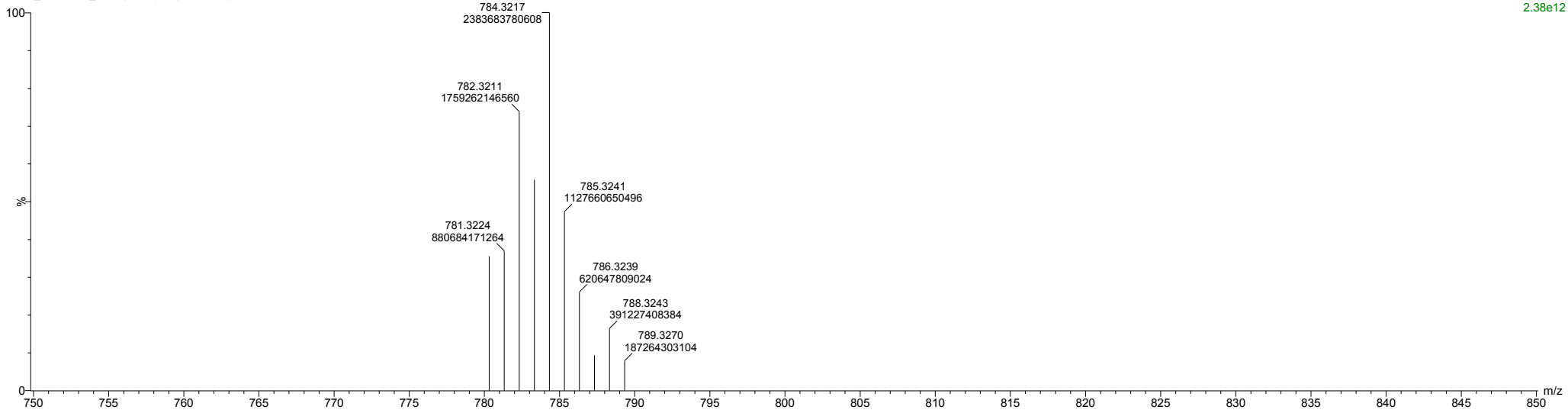
Waters Xevo G2-XS QTof Mass Spectrometer

23-02-2018

I-PK-48

ASEP_26FEB18_021 (0.364) Is (1.00,1.00) C43H57NO3SiSn

1: TOF MS ES+
2.38e12



ASEP_26FEB18_021 17 (0.364)

1: TOF MS ES+
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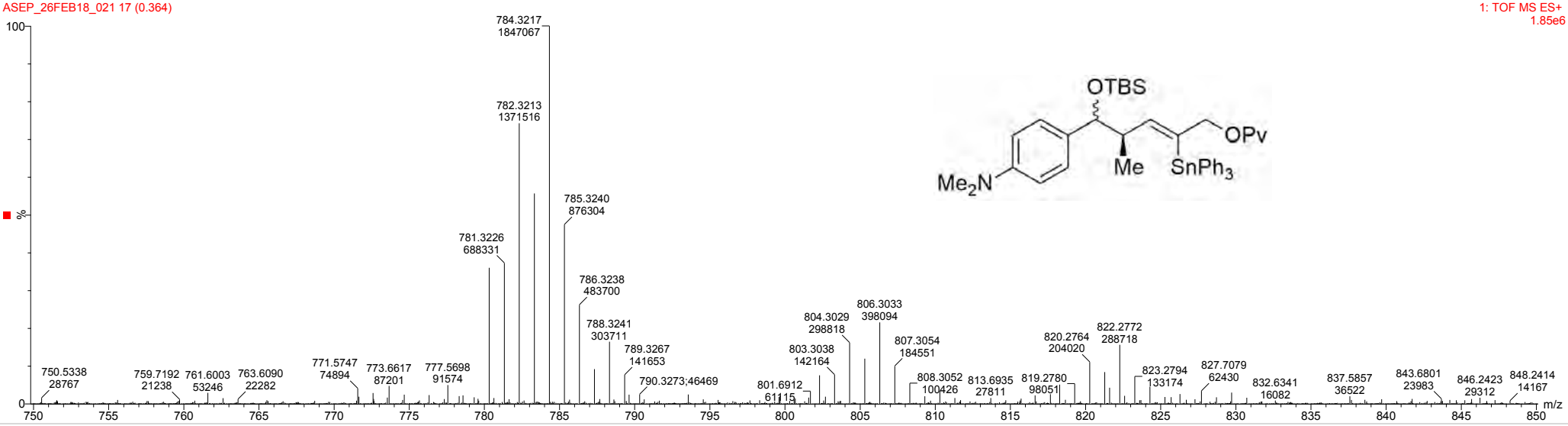
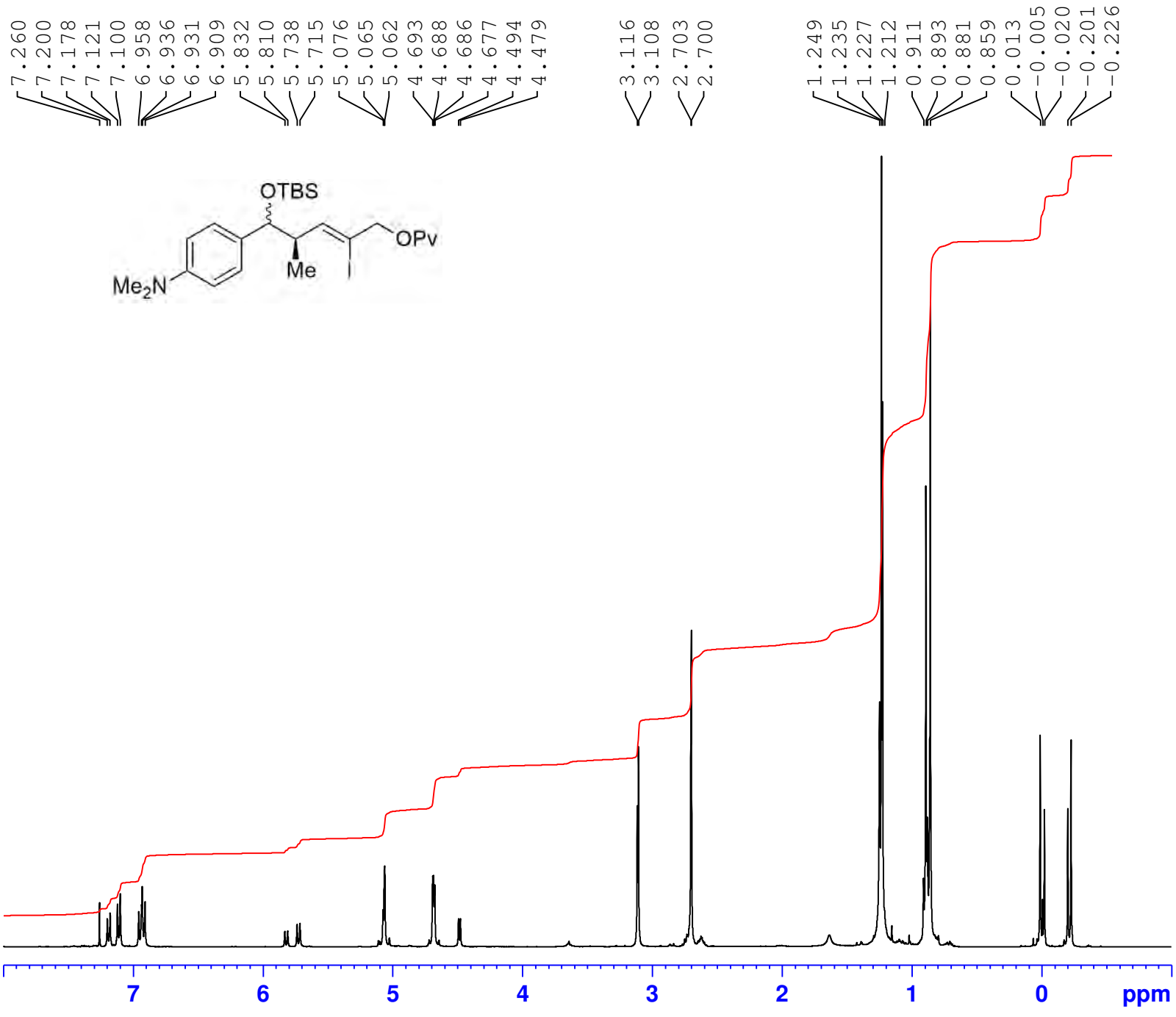




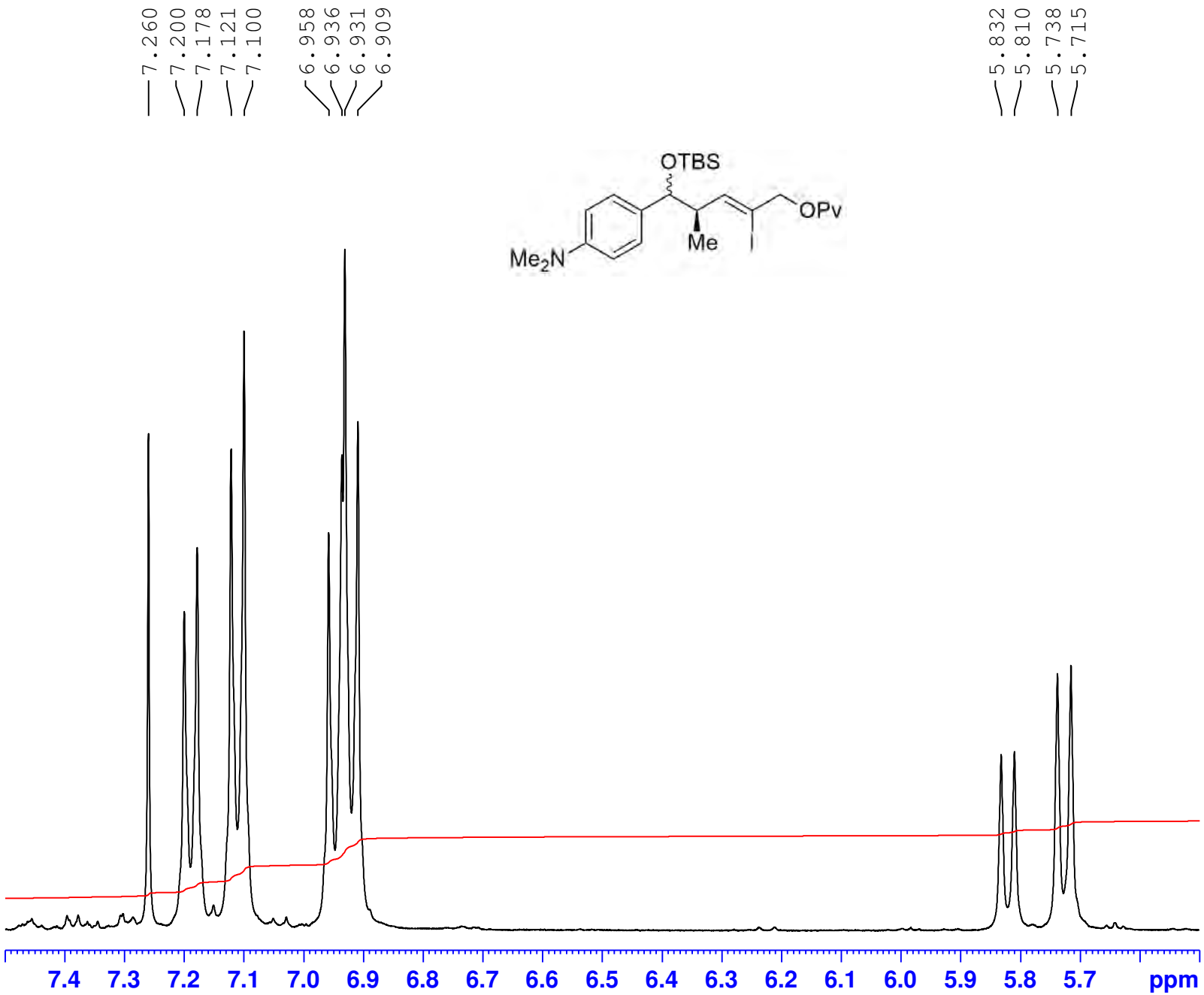
BRUKER

Current Data Parameters
NAME I-PK-27DRY
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 4.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 50.8
DW 41.600 usec
DE 9.85 usec
TE 295.8 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME          I-PK-27DRY
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180111
Time                4.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           12019.230 Hz
FIDRES         0.091699 Hz
AQ            5.4525952 sec
RG                 50.8
DW                41.600 usec
DE                 9.85 usec
TE                295.8 K
D1           0.10000000 sec
TD0                    1

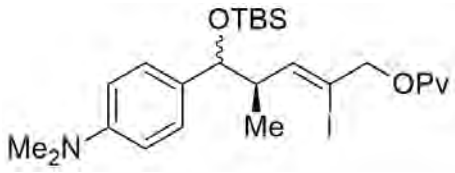
======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                   1H
P1                 14.88 usec
PLW1          7.59999990 W

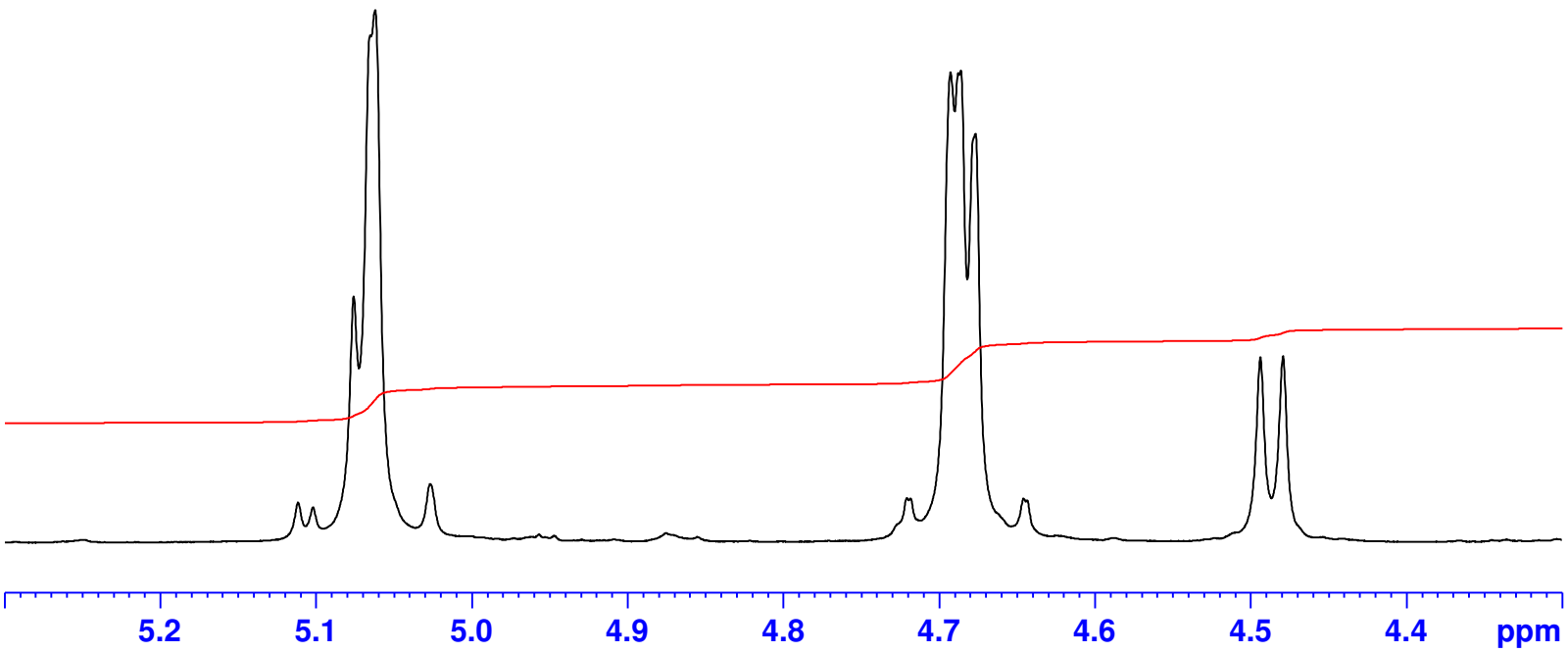
F2 - Processing parameters
SI                131072
SF           399.9000096 MHz
WDW                   EM
SSB                    0
LB                 0.10 Hz
GB                     0
PC                 1.00
```

5.112
5.102
5.076
5.065
5.062
5.027

4.721
4.718
4.693
4.688
4.686
4.677
4.646
4.643

4.494
4.479



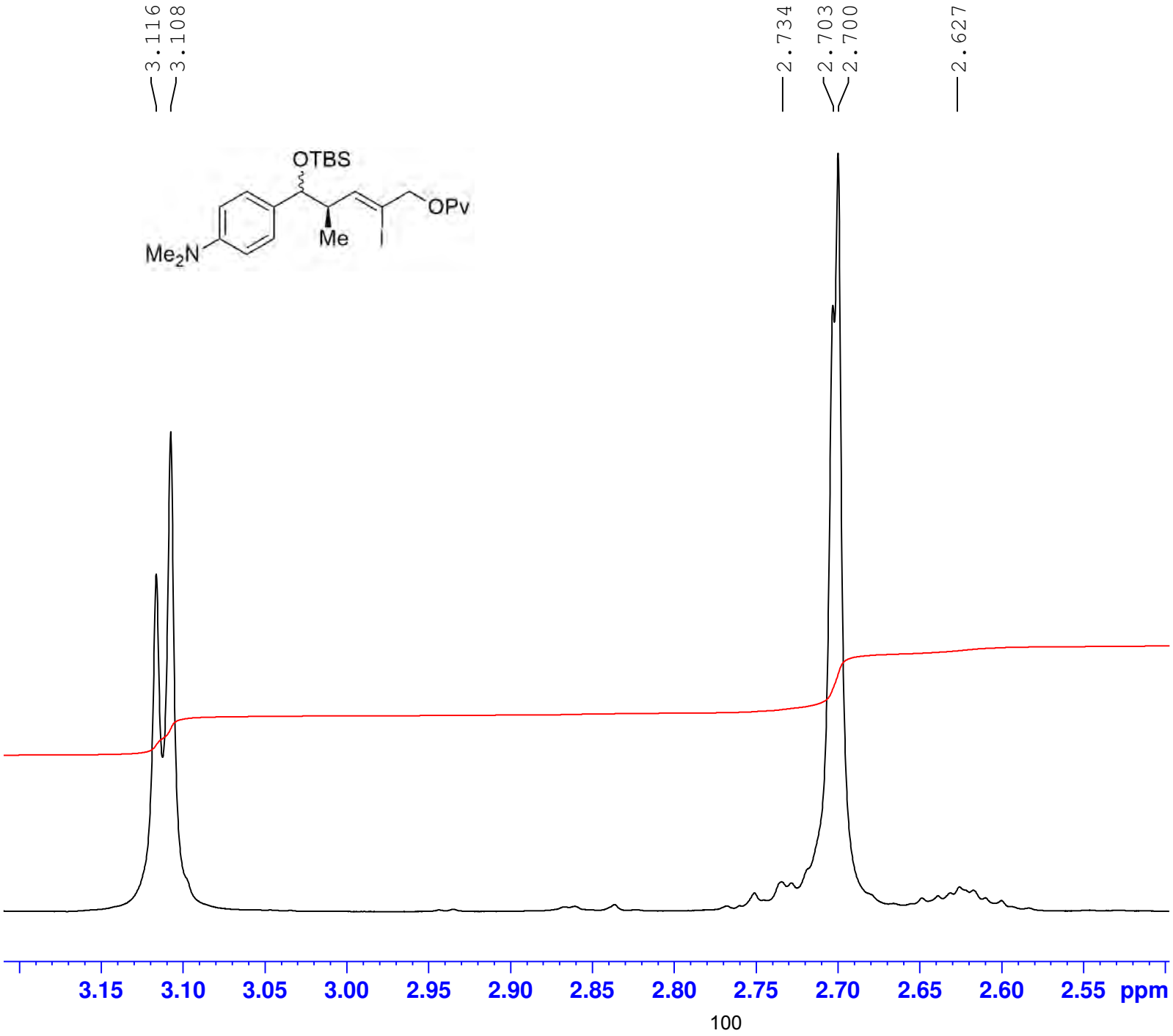



```
Current Data Parameters
NAME          I-PK-27DRY
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180111
Time                4.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH            12019.230 Hz
FIDRES          0.091699 Hz
AQ             5.4525952 sec
RG                  50.8
DW                41.600 usec
DE                 9.85 usec
TE                295.8 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                   1H
P1                 14.88 usec
PLW1          7.59999990 W

F2 - Processing parameters
SI                131072
SF           399.9000096 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

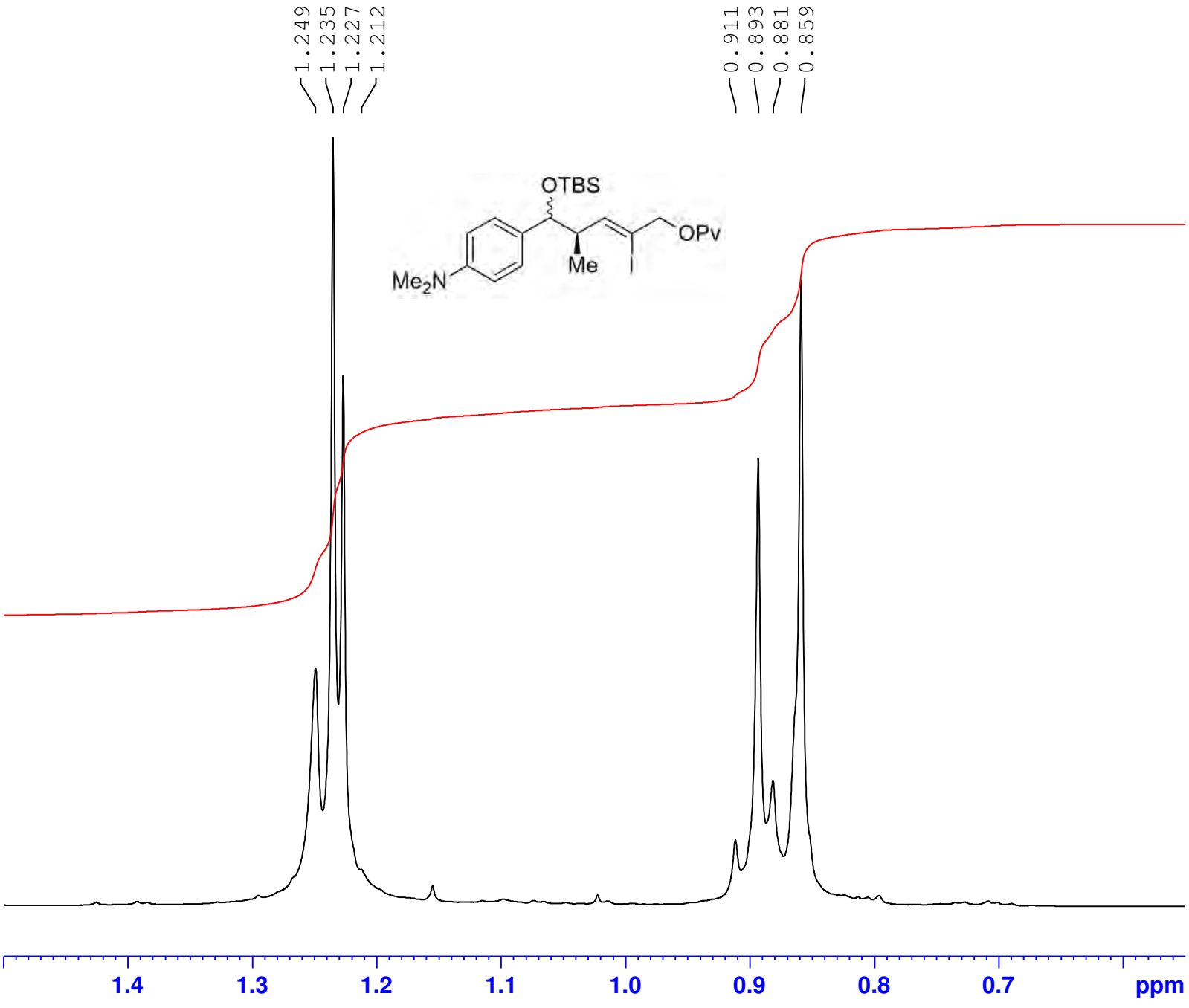


Current Data Parameters
NAME I-PK-27DRY
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 4.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 50.8
DW 41.600 usec
DE 9.85 usec
TE 295.8 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

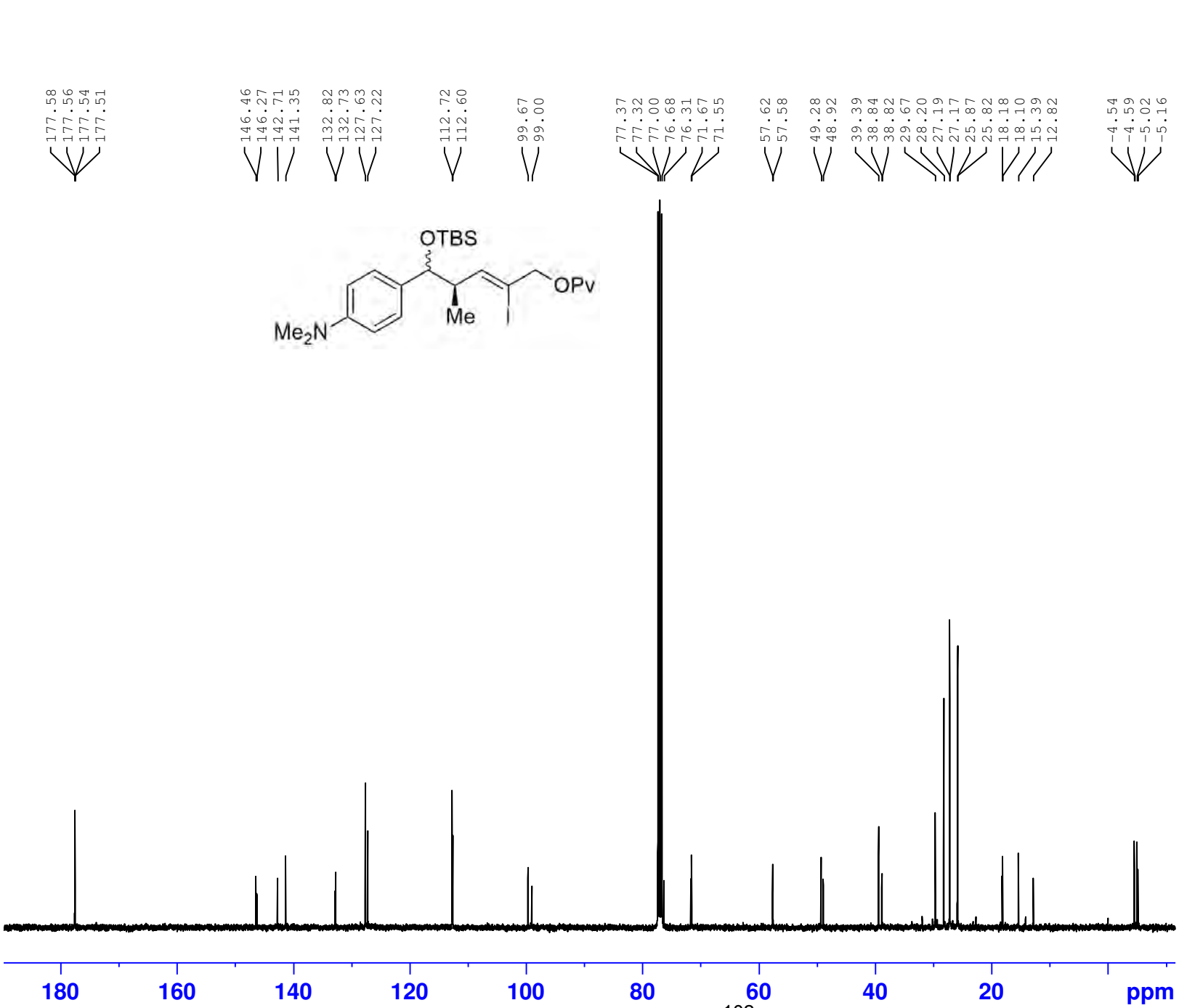



```
Current Data Parameters
NAME          I-PK-27DRY
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180111
Time                4.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           12019.230 Hz
FIDRES         0.091699 Hz
AQ            5.4525952 sec
RG                 50.8
DW                41.600 usec
DE                 9.85 usec
TE                295.8 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                   1H
P1                 14.88 usec
PLW1          7.59999990 W

F2 - Processing parameters
SI                131072
SF           399.9000096 MHz
WDW                   EM
SSB                    0
LB                 0.10 Hz
GB                     0
PC                 1.00
```



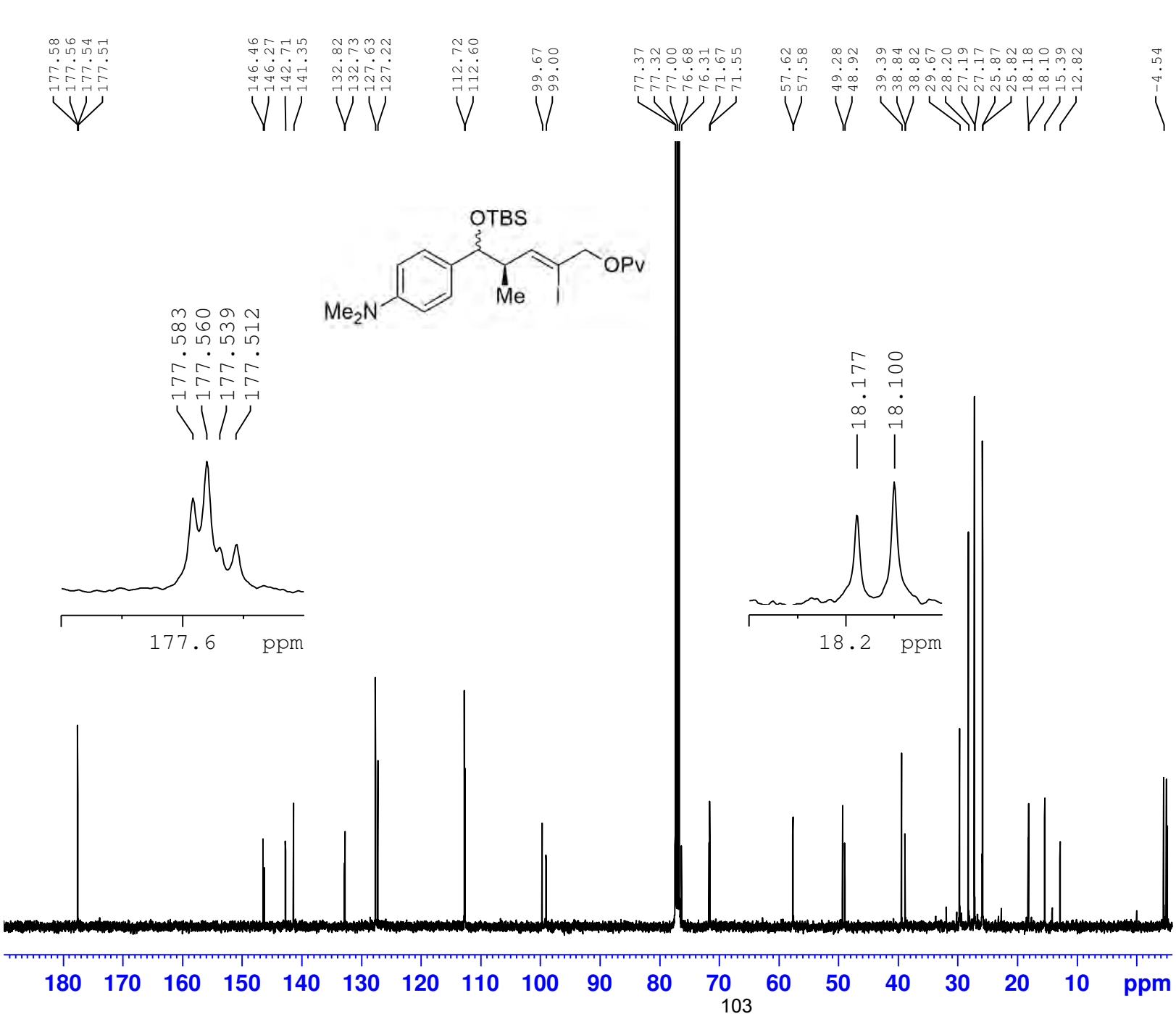
Current Data Parameters
NAME I-PK-27DRY
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 5.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



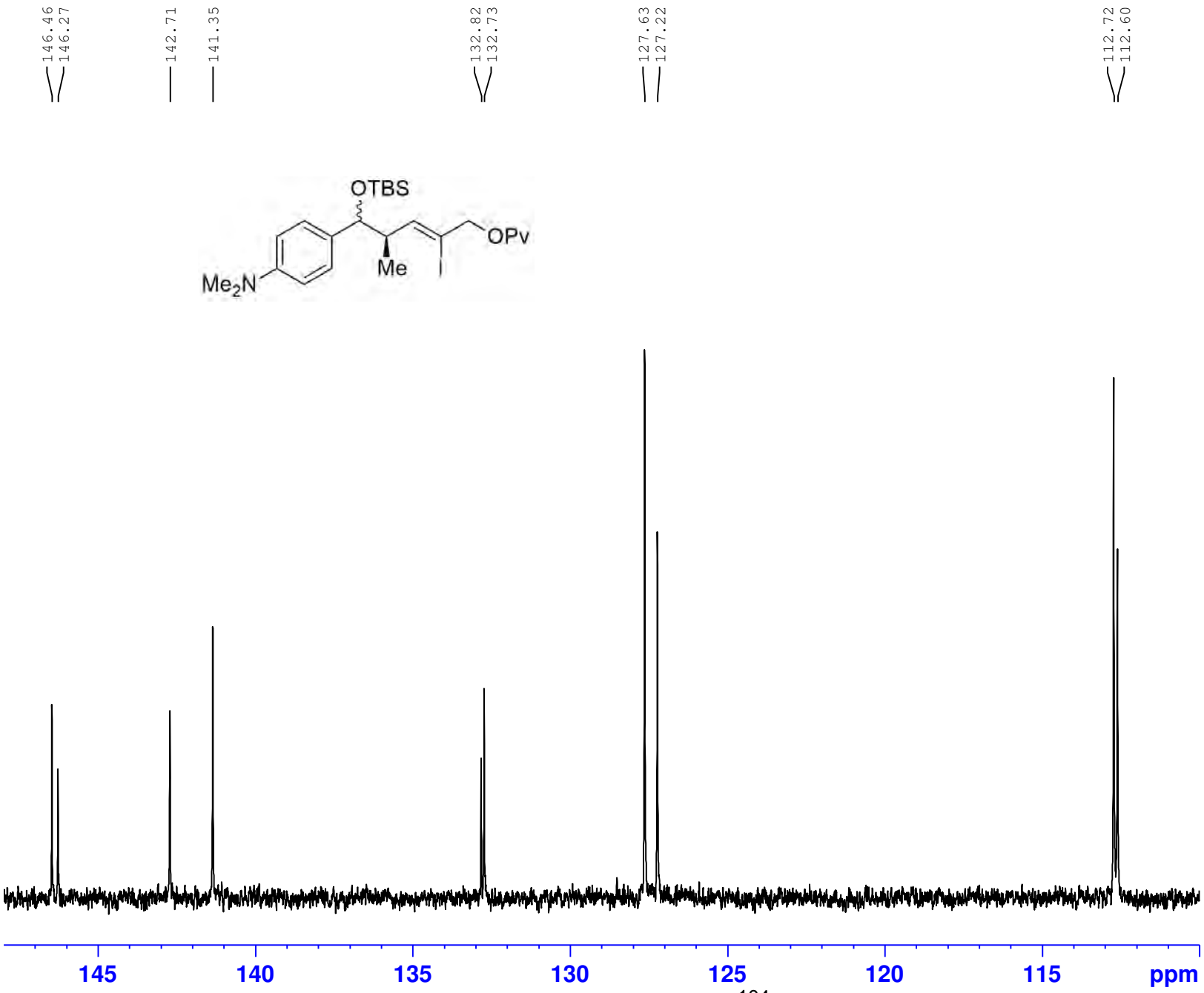
Current Data Parameters
NAME I-PK-27DRY
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 5.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



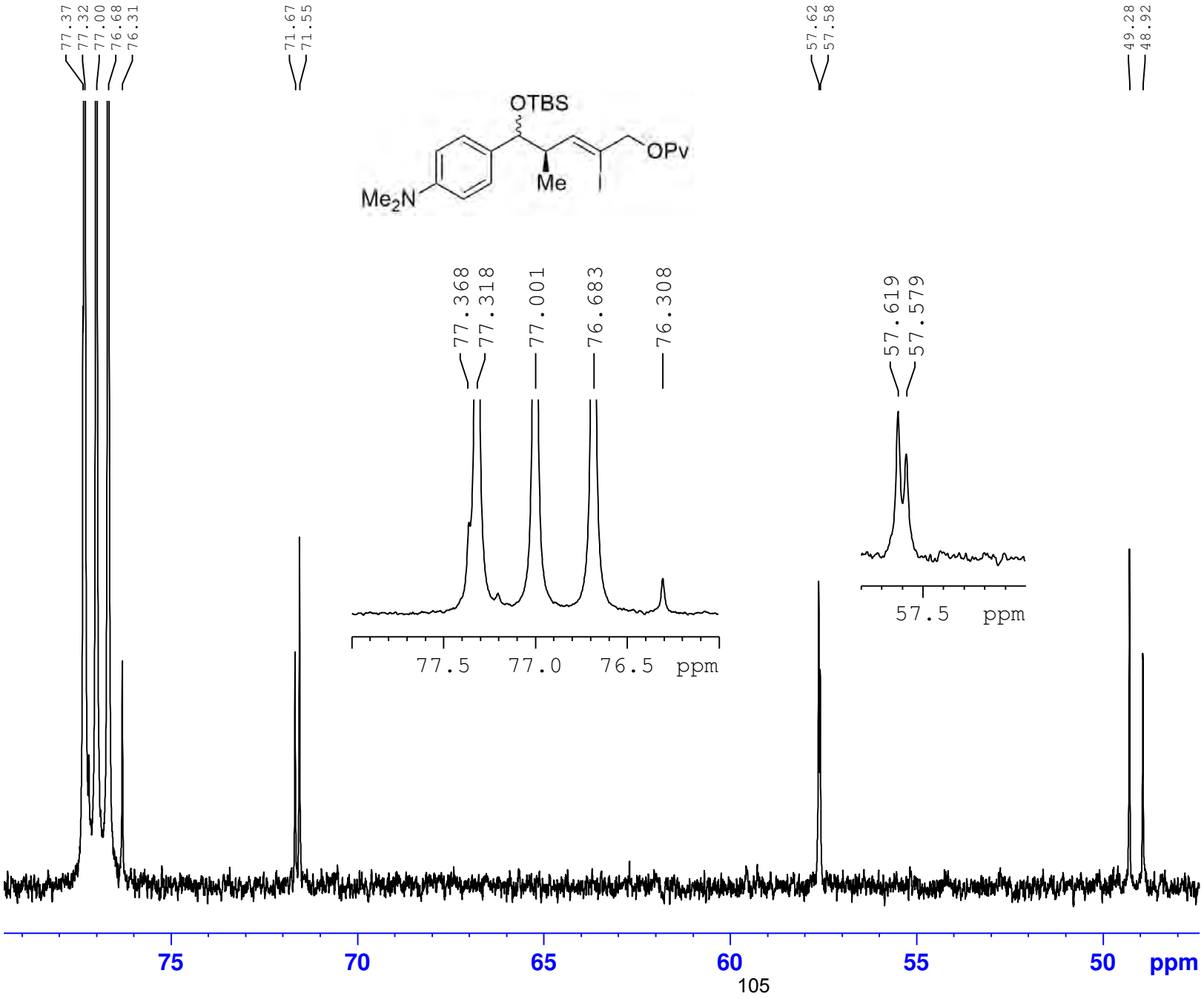
Current Data Parameters
NAME I-PK-27DRY
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 5.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



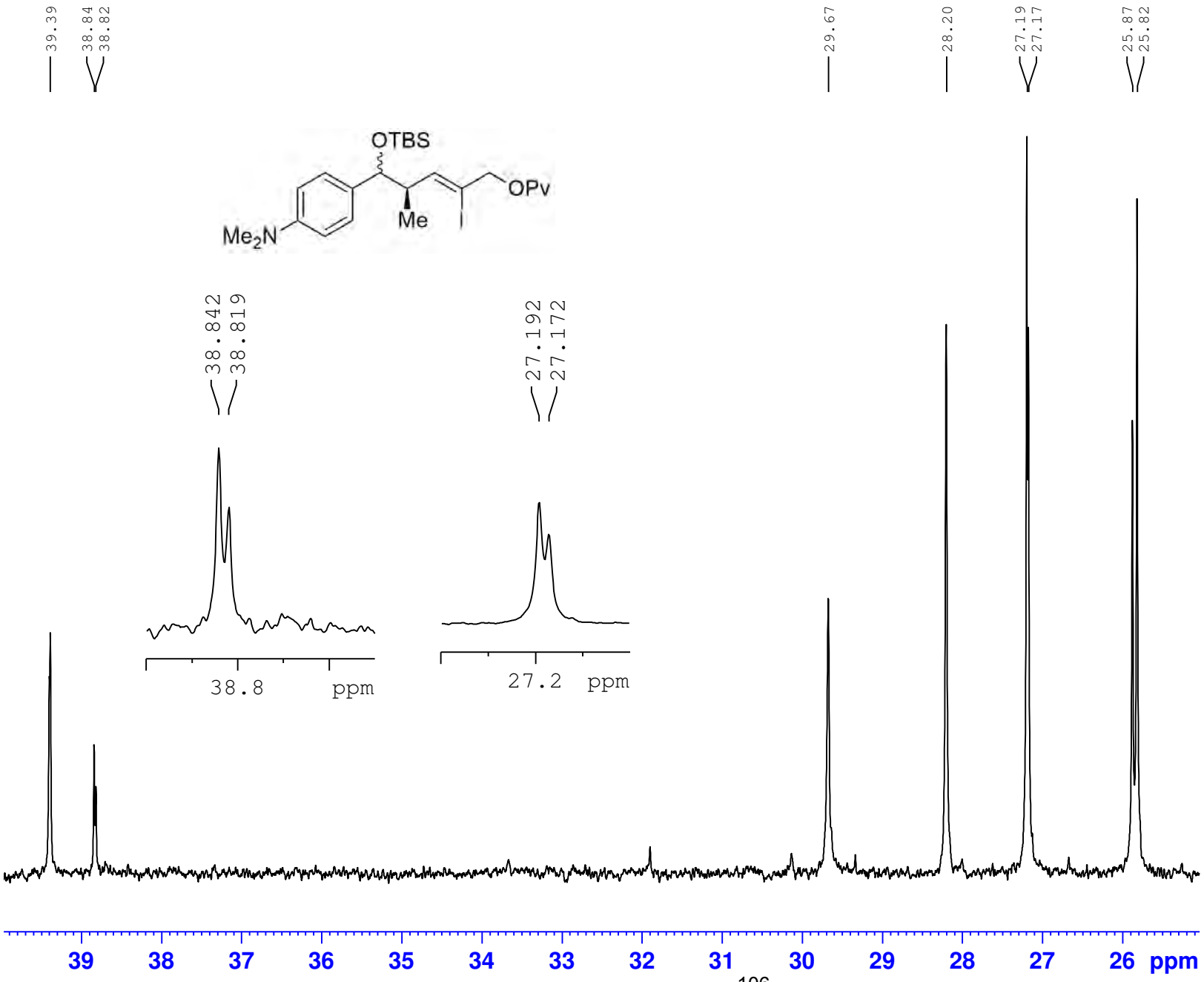

```
Current Data Parameters
NAME          I-PK-27DRY
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180111
Time                5.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH           25000.000 Hz
FIDRES         0.210006 Hz
AQ             2.3808801 sec
RG                  2050
DW               20.000 usec
DE                9.12 usec
TE                297.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.5659947 MHz
NUC1                13C
P1                10.00 usec
PLW1        44.46300125 W

======== CHANNEL f2 ========
SFO2        399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2          7.59999990 W
PLW12         0.20774999 W
PLW13         0.16827001 W

F2 - Processing parameters
SI                131072
SF          100.5549391 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



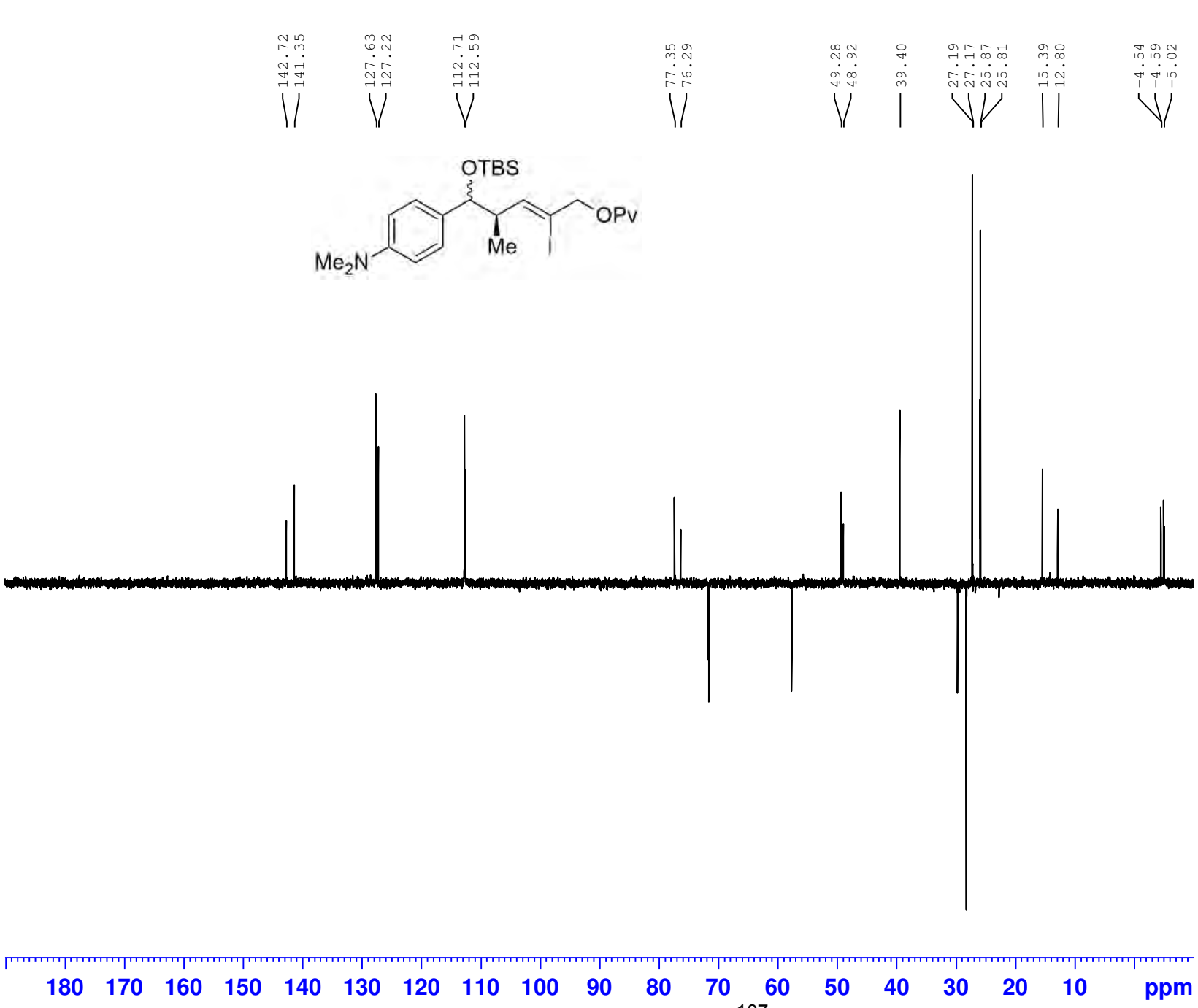
Current Data Parameters
NAME I-PK-27DRY
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180111
Time 5.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	I-PK-27DRY
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20180111
Time	4.29
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	296.4 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

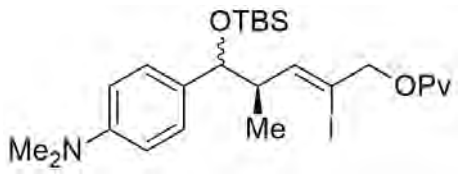
SFO1	100.5649905 MHz
NUC1	13C
P1	10.00 usec
P13	2000.00 usec
PLW0	0 W
PLW1	44.46300125 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	6.79339981 W

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

SFO2	399.9012789 MHz
NUC2	1H
CPDPRG[2	waltz16
P3	14.88 usec
P4	29.76 usec
PCPD2	90.00 usec
PLW2	7.59999990 W
PLW12	0.20774999 W

F2 - Processing parameters

SI	32768
SF	100.5549391 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40




```
Current Data Parameters
NAME          I-PK-27DRY
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180111
Time                4.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH            4076.087 Hz
FIDRES         1.990277 Hz
AQ             0.2512213 sec
RG                 2050
DW              122.667 usec
DE                6.50 usec
TE               296.0 K
D0            0.00000300 sec
D1            0.89678001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00024540 sec

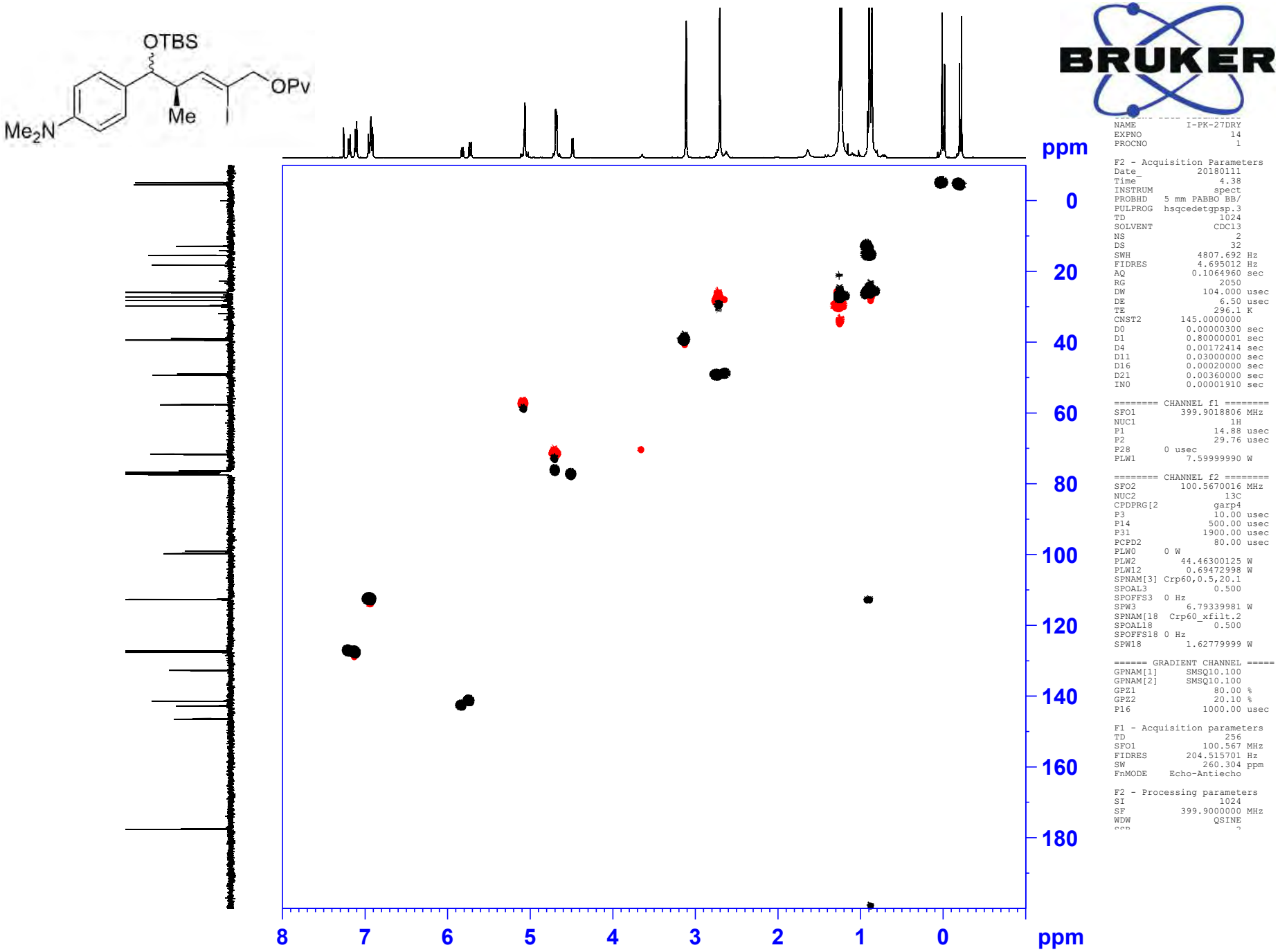
======== CHANNEL f1 ========
SFO1         399.9015543 MHz
NUC1                1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1             16.00 %
GPZ2             12.00 %
GPZ3             40.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1           399.9016 MHz
FIDRES         31.835779 Hz
SW                10.190 ppm
FnMODE               QF

F2 - Processing parameters
SI                 1024
SF          399.9000100 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                  QF
SF          399.8999774 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
```



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```
NAME            I-PK-27DRY
EXPNO                   14
PROCNO                   1

F2 - Acquisition Parameters
Date_             20180111
Time                 4.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                   1024
SOLVENT             CDC13
NS                       2
DS                      32
SWH              4807.692 Hz
FIDRES           4.695012 Hz
AQ               0.1064960 sec
RG                    2050
DW                104.000 usec
DE                  6.50 usec
TE                 296.1 K
CNST2         145.0000000
D0             0.00000300 sec
D1             0.80000001 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D21            0.00360000 sec
IN0            0.00001910 sec

======== CHANNEL f1 ========
SFO1          399.9018806 MHz
NUC1                    1H
P1                  14.88 usec
P2                  29.76 usec
P28       0 usec
PLW1           7.59999990 W

======== CHANNEL f2 ========
SFO2          100.5670016 MHz
NUC2                   13C
CPDPRG[2             garp4
P3                  10.00 usec
P14                500.00 usec
P31               1900.00 usec
PCPD2               80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                0.500
SPOFFS3   0 Hz
SPW3           6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18               0.500
SPOFFS18 0 Hz
SPW18          1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                     256
SFO1             100.567 MHz
FIDRES         204.515701 Hz
SW                260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                    1024
SF            399.9000000 MHz
WDW                   QSINE
```

Mass Spectrometry Result Sheet

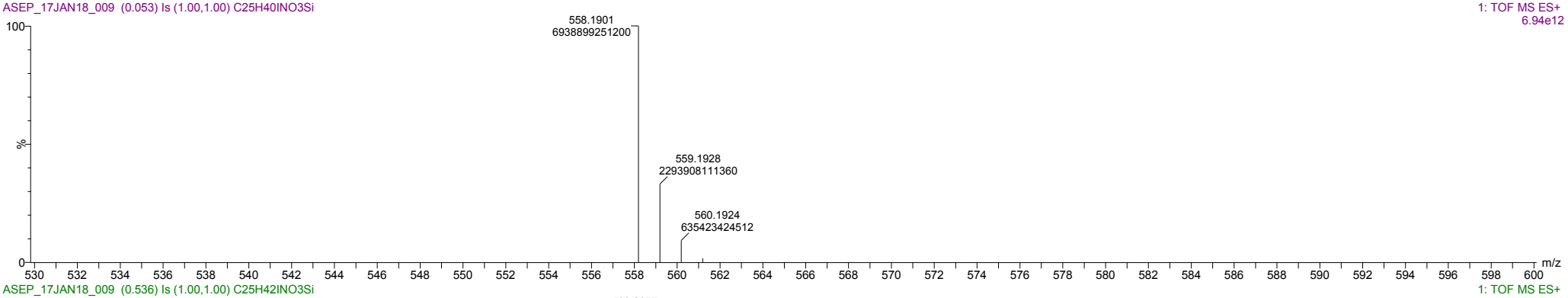
Waters Xevo G2-XS QTof Mass Spectrometer

12-01-2018

I-PK-27

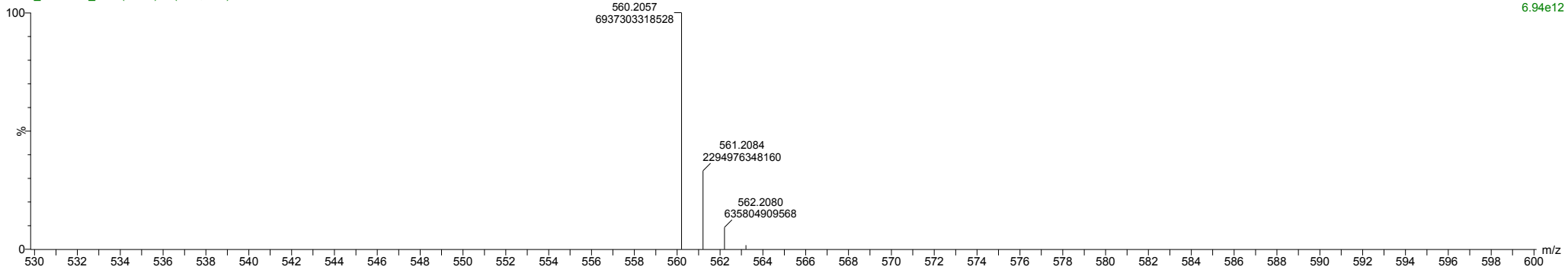
ASEP_17JAN18_009 (0.053) Is (1.00,1.00) C25H40INO3Si

1: TOF MS ES+
6.94e12



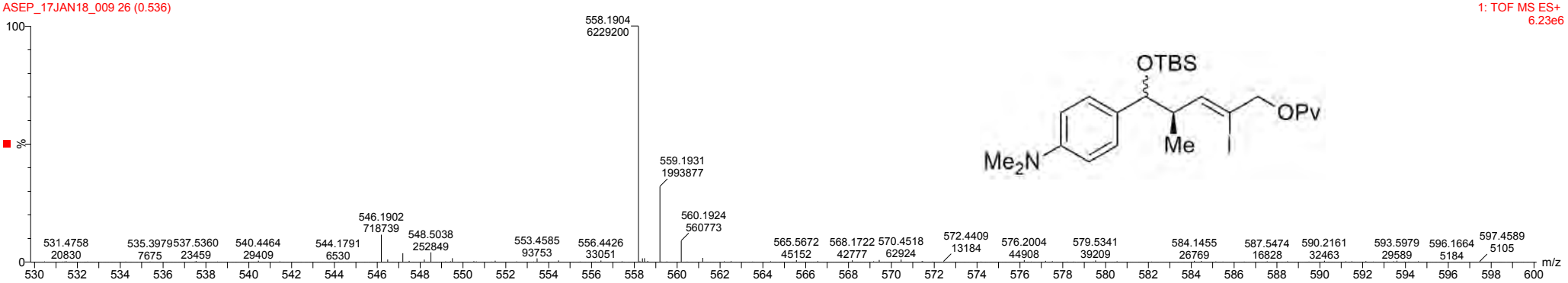
ASEP_17JAN18_009 (0.536) Is (1.00,1.00) C25H42INO3Si

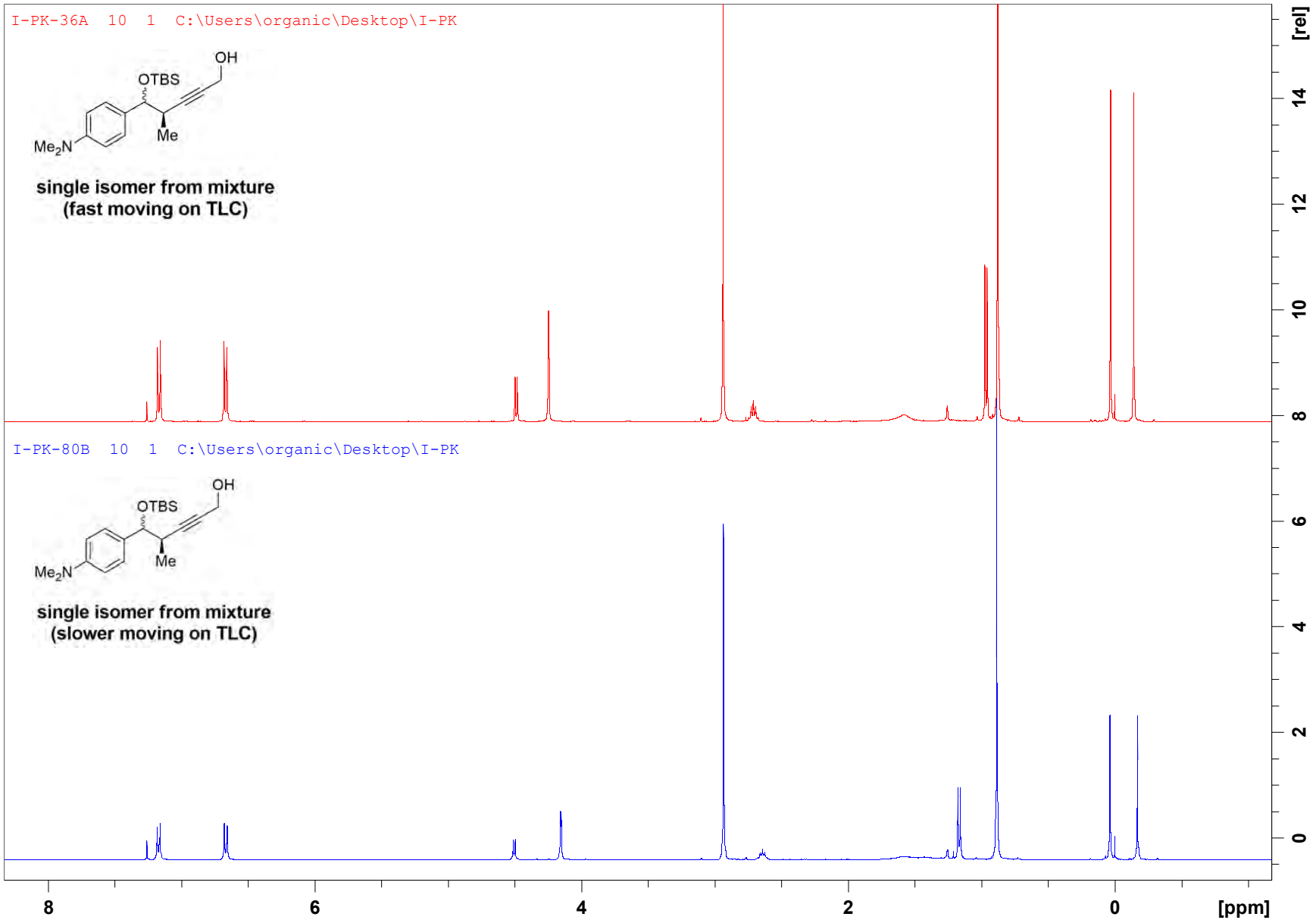
1: TOF MS ES+
6.94e12

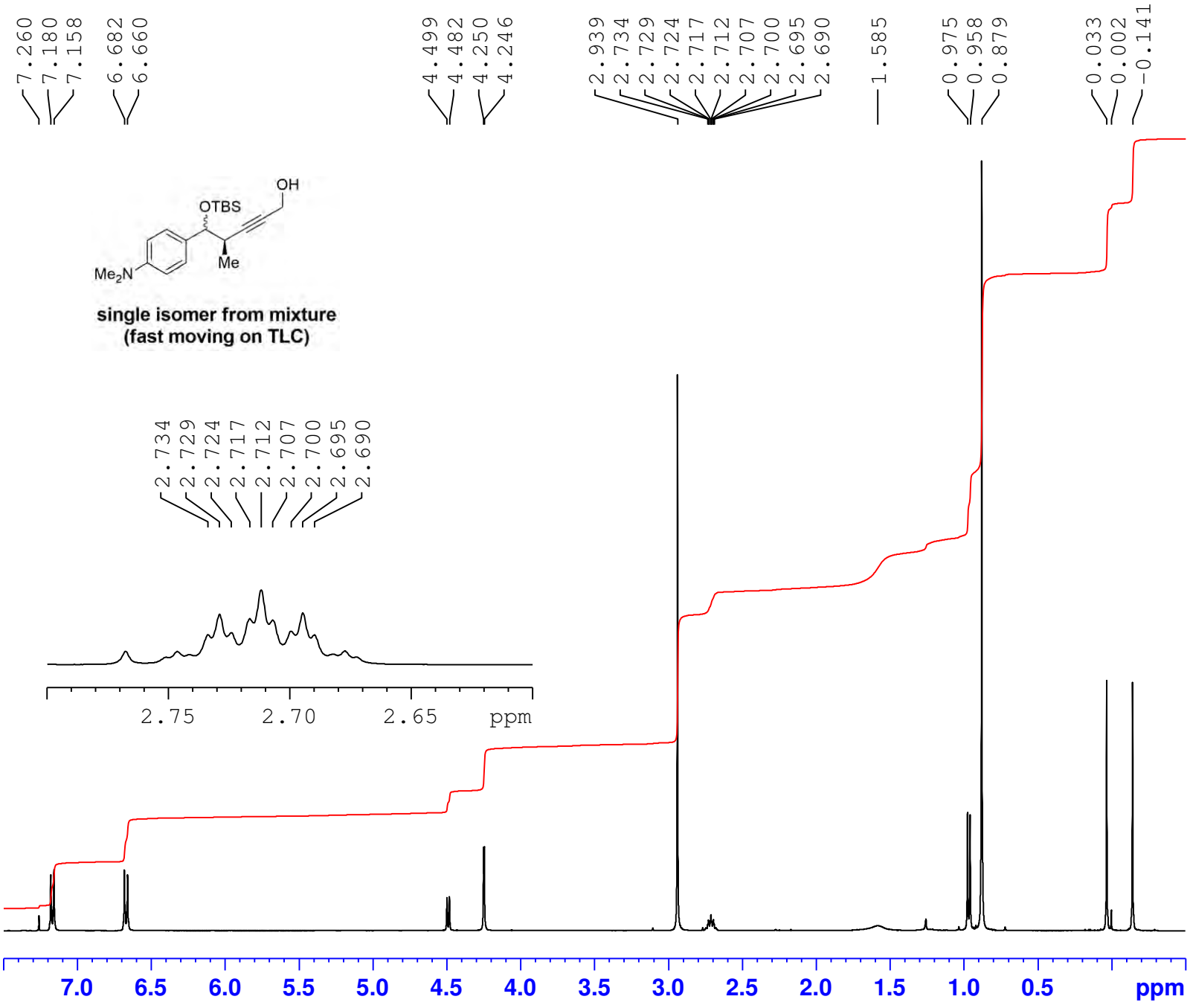


ASEP_17JAN18_009 26 (0.536)

1: TOF MS ES+
6.23e6




I-PK-36A 10 1 C:\Users\organic\Desktop\I-PK
OH
OTBS
Me
Me2N
single isomer from mixture
(fast moving on TLC)
I-PK-80B 10 1 C:\Users\organic\Desktop\I-PK
OH
OTBS
Me
Me2N
single isomer from mixture
(slower moving on TLC)
[rel]
14
12
10
8
6
4
2
0
8
6
4
2
0
[ppm]



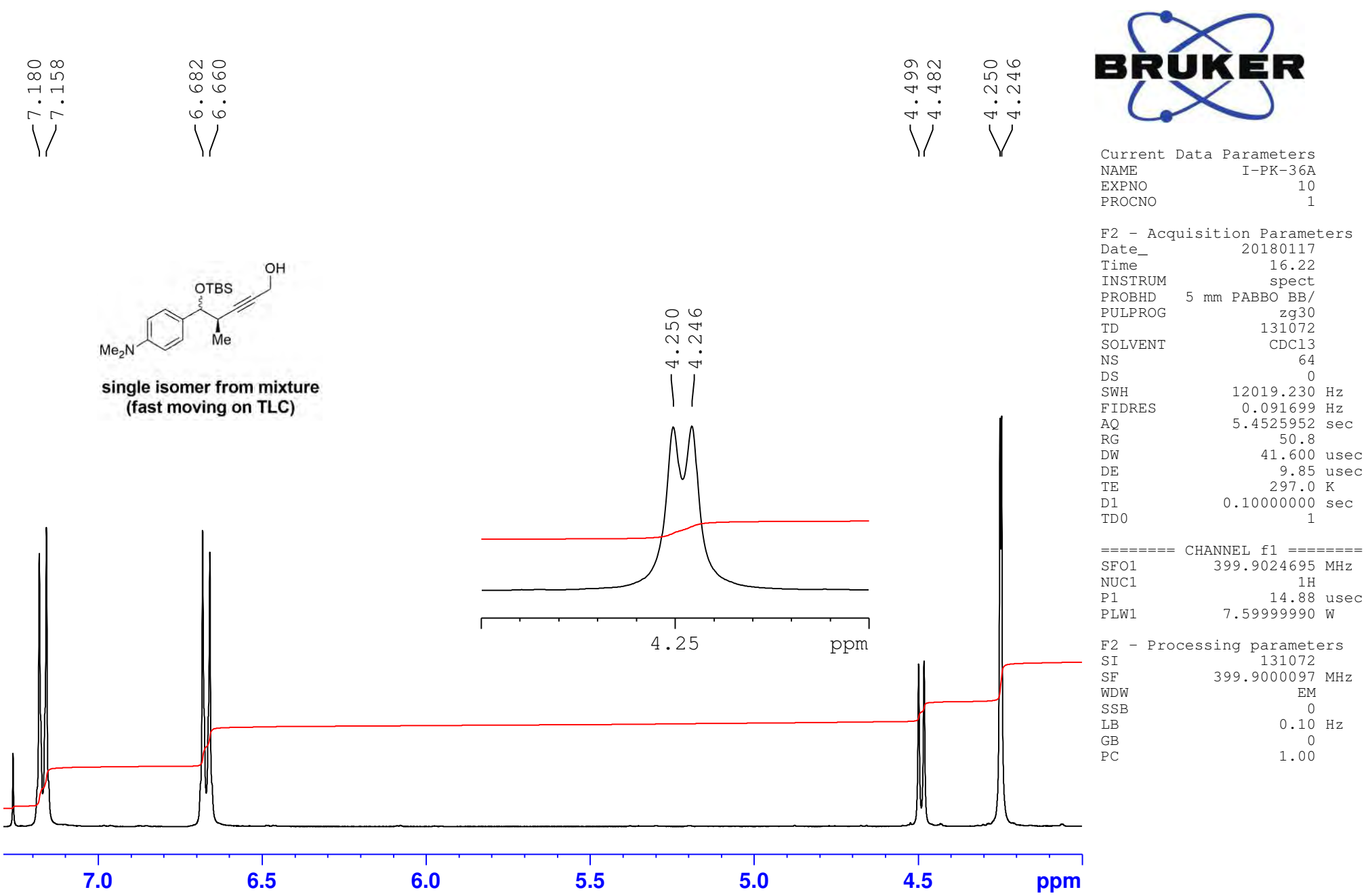
BRUKER

```
Current Data Parameters
NAME            I-PK-36A
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180117
Time               16.22
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           12019.230 Hz
FIDRES         0.091699 Hz
AQ             5.4525952 sec
RG                  50.8
DW                41.600 usec
DE                 9.85 usec
TE                297.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                 1H
P1                14.88 usec
PLW1           7.59999990 W

F2 - Processing parameters
SI                131072
SF           399.9000097 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```



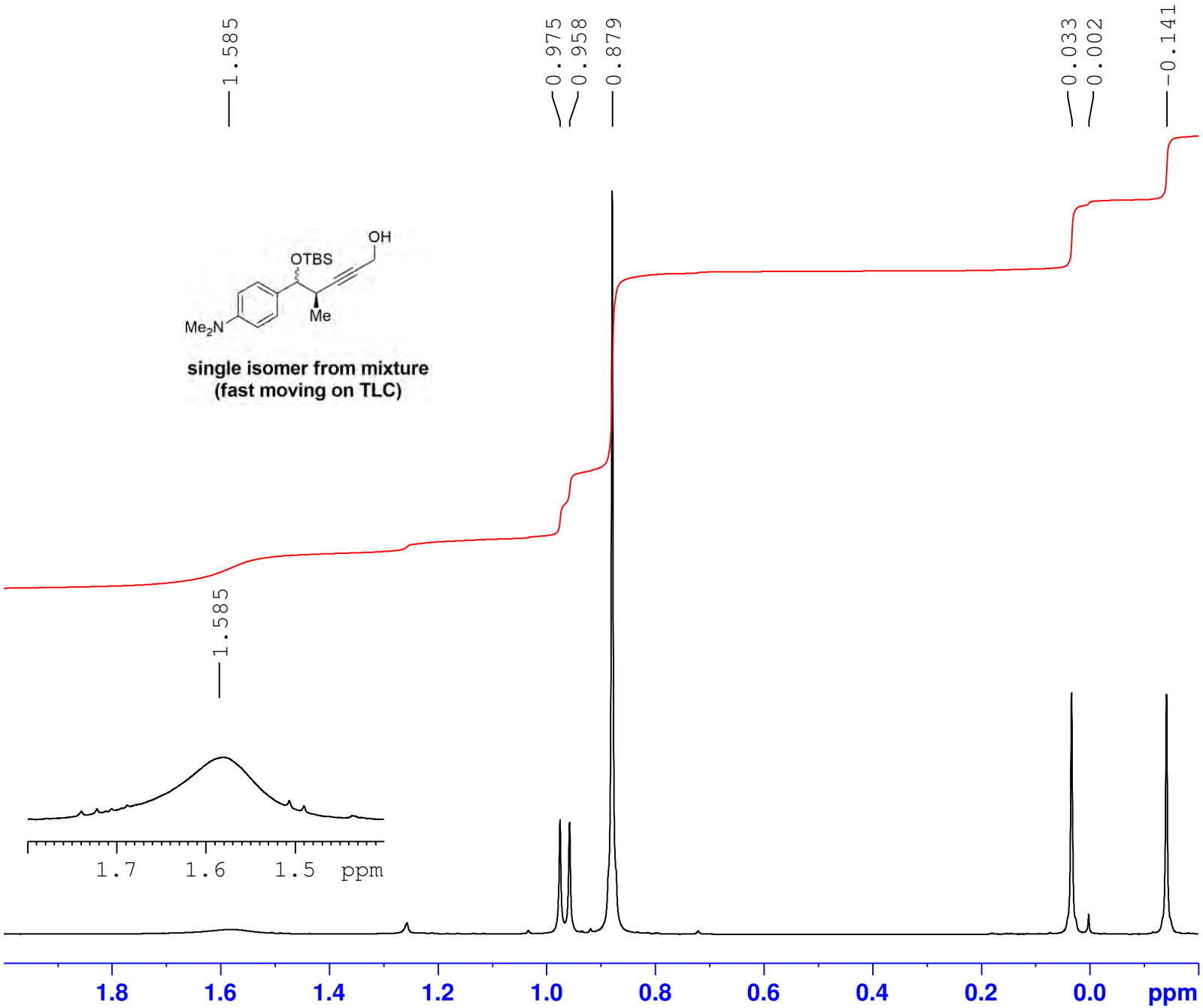
BRUKER

```
Current Data Parameters
NAME                  I-PK-36A
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180117
Time                     16.22
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                       50.8
DW                     41.600 usec
DE                      9.85 usec
TE                     297.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                     131072
SF               399.9000097 MHz
WDW                        EM
SSB                         0
LB                      0.10 Hz
GB                          0
PC                       1.00
```



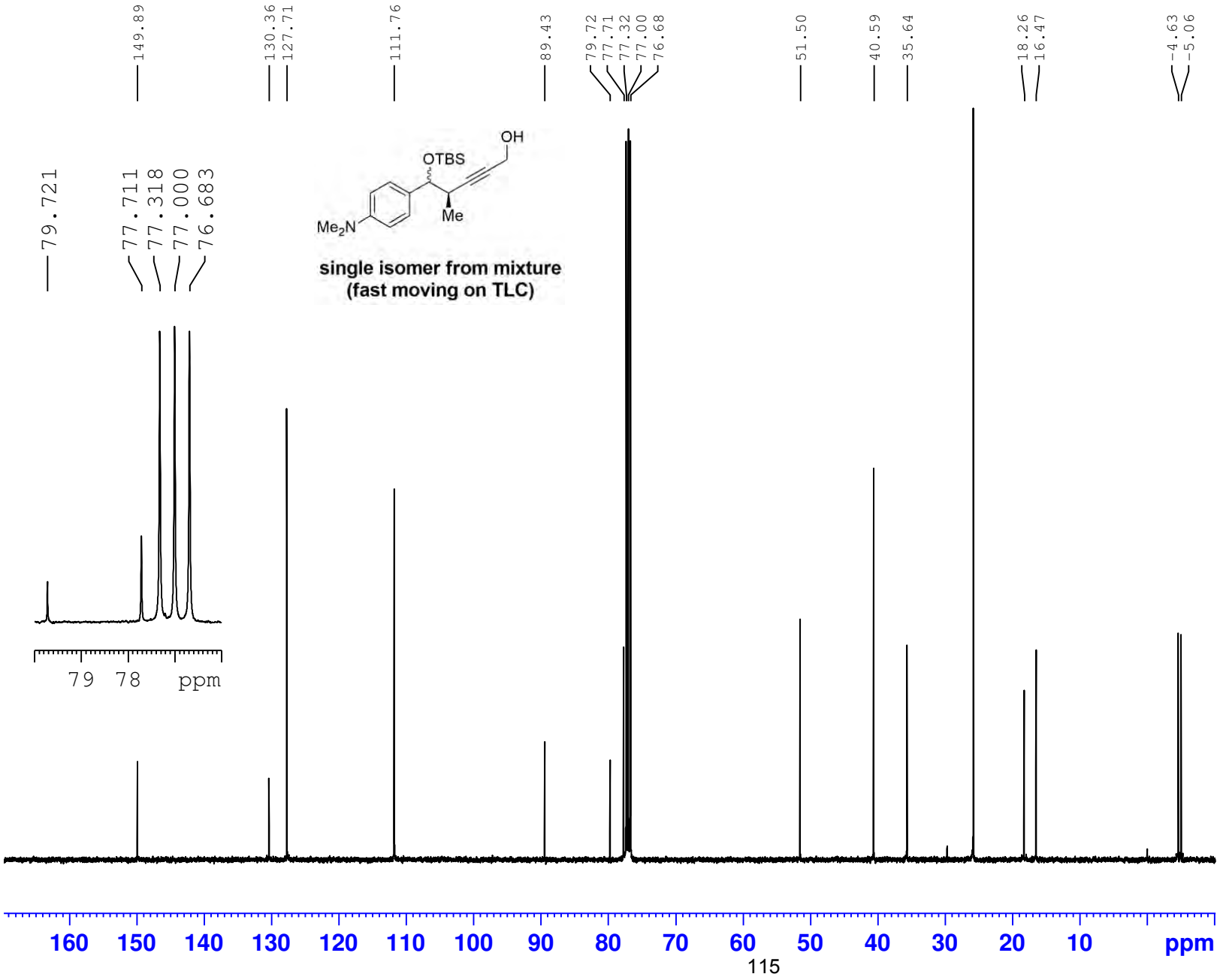
BRUKER

```
Current Data Parameters
NAME                  I-PK-36A
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180117
Time                     16.22
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         50.8
DW                       41.600 usec
DE                        9.85 usec
TE                       297.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000097 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



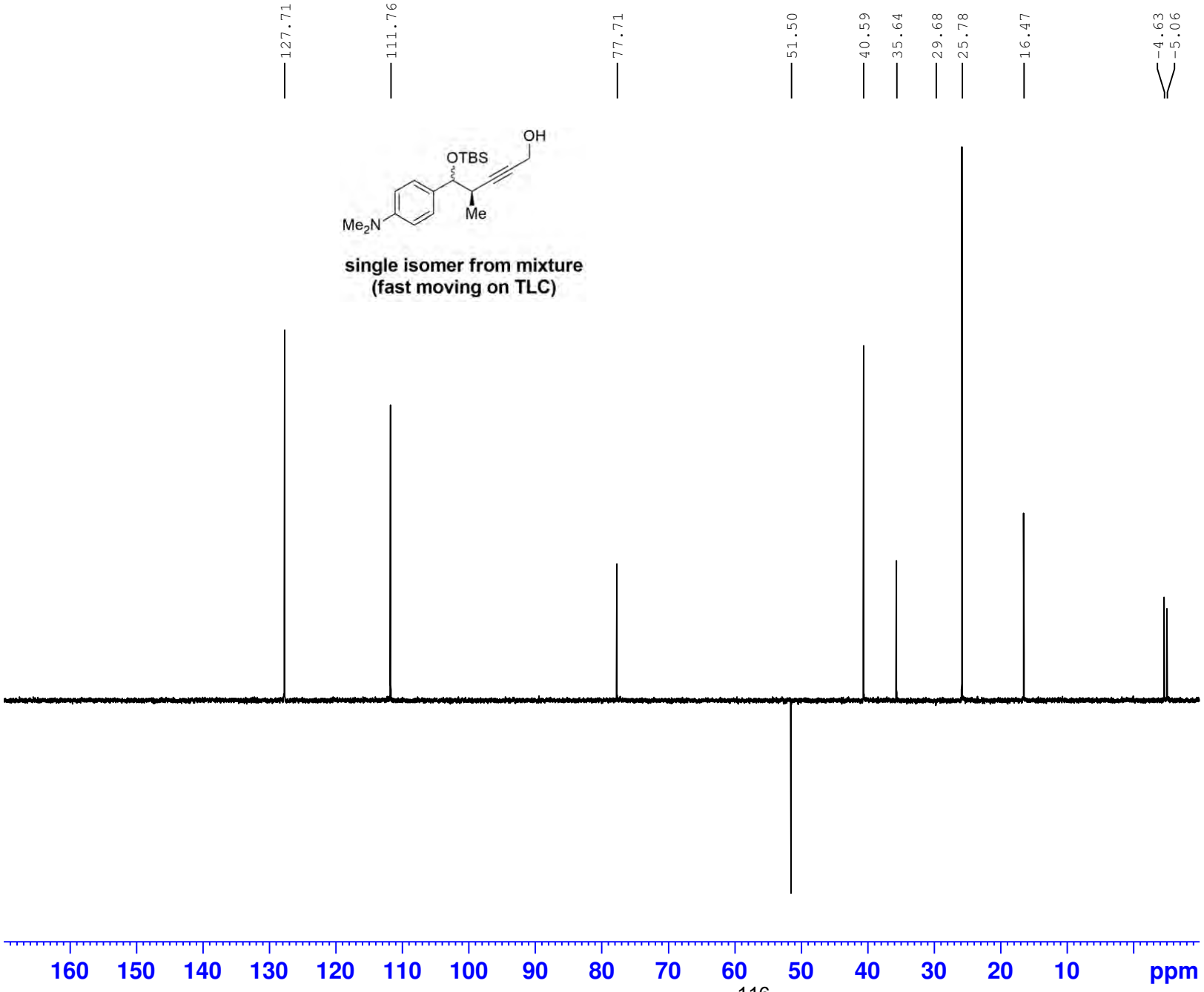

```
Current Data Parameters
NAME                 I-PK-36A
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_              20180117
Time                   17.33
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                     119044
SOLVENT                CDCl3
NS                     1200
DS                     4
SWH                25000.000 Hz
FIDRES             0.210006 Hz
AQ                 2.3808801 sec
RG                     2050
DW                 20.000 usec
DE                 9.12 usec
TE                 298.4 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           100.5659947 MHz
NUC1                   13C
P1                 10.00 usec
PLW1           44.46300125 W

======== CHANNEL f2 ========
SFO2           399.9015996 MHz
NUC2                   1H
CPDPRG[2           waltz64
PCPD2              90.00 usec
PLW2           7.59999990 W
PLW12          0.20774999 W
PLW13          0.16827001 W

F2 - Processing parameters
SI                     131072
SF             100.5549386 MHz
WDW                    EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



BRUKER

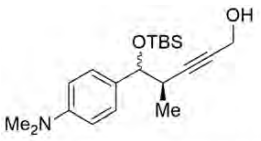
Current Data Parameters
NAME I-PK-36A
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180117
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

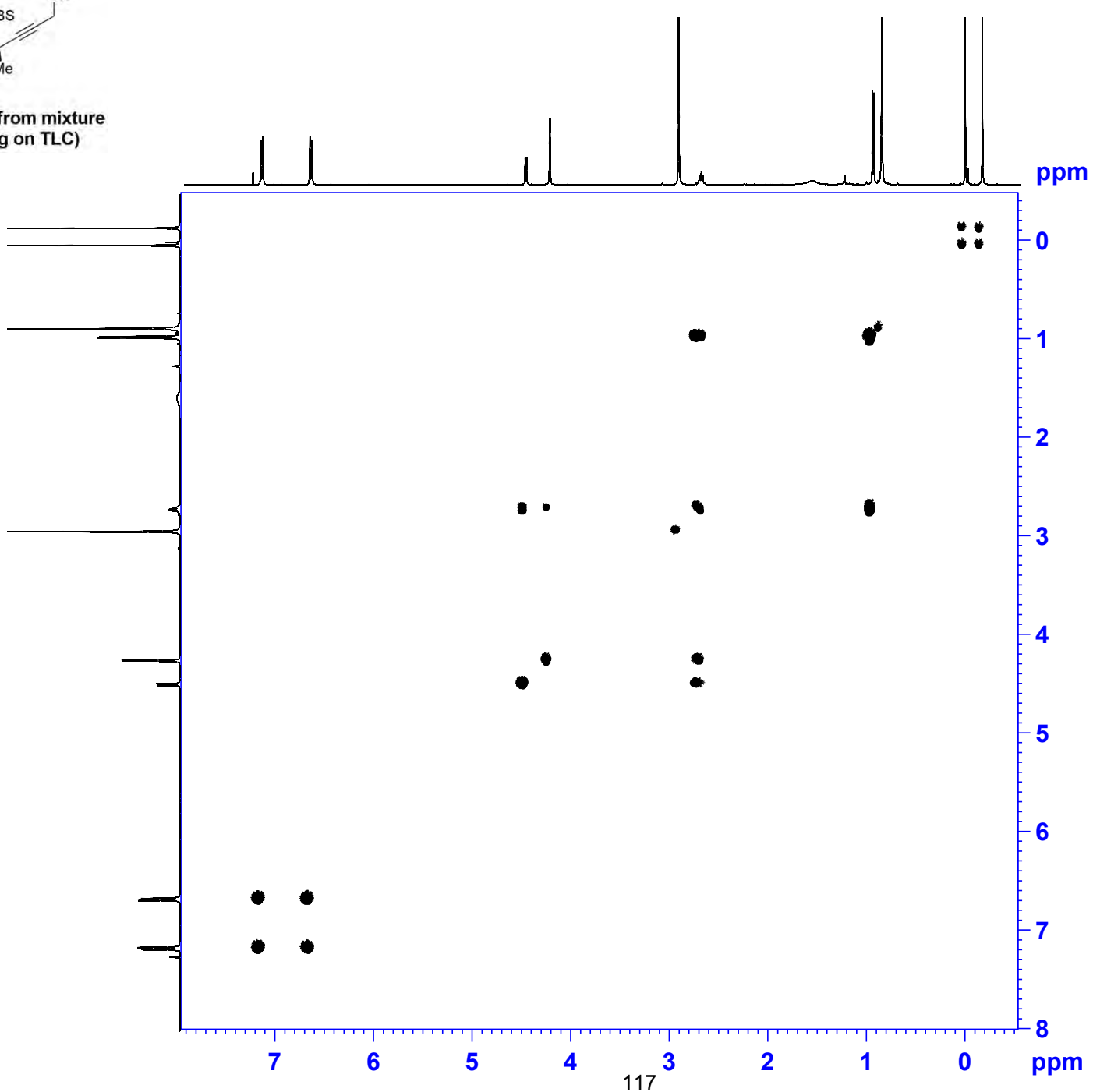
======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549386 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



single isomer from mixture (fast moving on TLC)




```
Current Data Parameters
NAME                  I-PK-36A
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180117
Time                     17.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                       2048
SOLVENT                 CDCl3
NS                           1
DS                           8
SWH                 3578.244 Hz
FIDRES              1.747190 Hz
AQ                  0.2861739 sec
RG                        2050
DW                     139.733 usec
DE                       6.50 usec
TE                      297.4 K
D0                  0.00000300 sec
D1                  0.85786802 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D13                 0.00000400 sec
D16                 0.00020000 sec
IN0                 0.00027940 sec

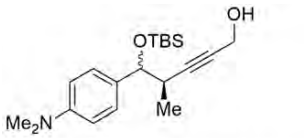
======== CHANNEL f1 ========
SFO1               399.9014143 MHz
NUC1                        1H
P1                       14.88 usec
P17                    2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]           SMSQ10.100
GPNAM[2]           SMSQ10.100
GPNAM[3]           SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1                399.9014 MHz
FIDRES             27.961704 Hz
SW                       8.950 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                399.9000097 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                399.9000097 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
```



single isomer from mixture
(fast moving on TLC)


```
Current Data Parameters
NAME            I-PK-36A
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180117
Time_              17.59
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT            CDCl3
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES          4.695012 Hz
AQ              0.1064960 sec
RG                  2050
DW                104.000 usec
DE                  6.50 usec
TE                297.4 K
CNST2          145.0000000
D0              0.00000300 sec
D1              0.80000001 sec
D4              0.00172414 sec
D11             0.03000000 sec
D16             0.00020000 sec
D21             0.00360000 sec
IN0             0.00001910 sec

======== CHANNEL f1 ========
SFO1          399.9018806 MHz
NUC1                  1H
P1                  14.88 usec
P2                  29.76 usec
P28       0 usec
PLW1           7.59999990 W

======== CHANNEL f2 ========
SFO2          100.5670016 MHz
NUC2                 13C
CPDPRG[2            garp4
P3                  10.00 usec
P14                500.00 usec
P31               1900.00 usec
PCPD2               80.00 usec
PLW0      0 W
PLW2          44.46300125 W
PLW12          0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3               0.500
SPOFFS3   0 Hz
SPW3           6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18              0.500
SPOFFS18 0 Hz
SPW18          1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1             100.567 MHz
FIDRES         204.515701 Hz
SW               260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                   1024
SF            399.9000095 MHz
```

Mass Spectrometry Result Sheet

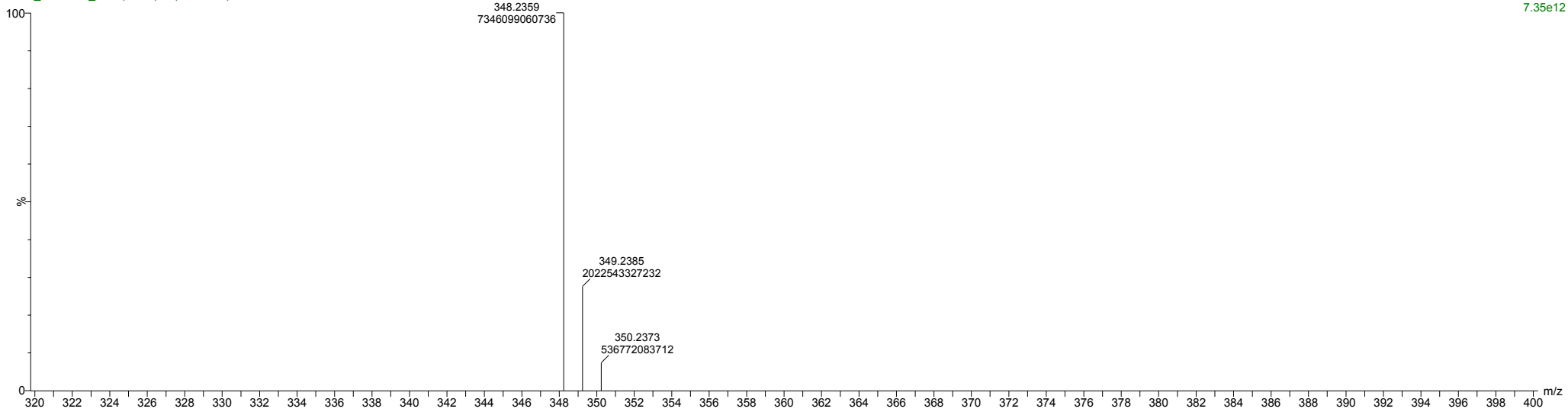
Waters Xevo G2-XS QTof Mass Spectrometer

17-01-2018

I-PK-36A

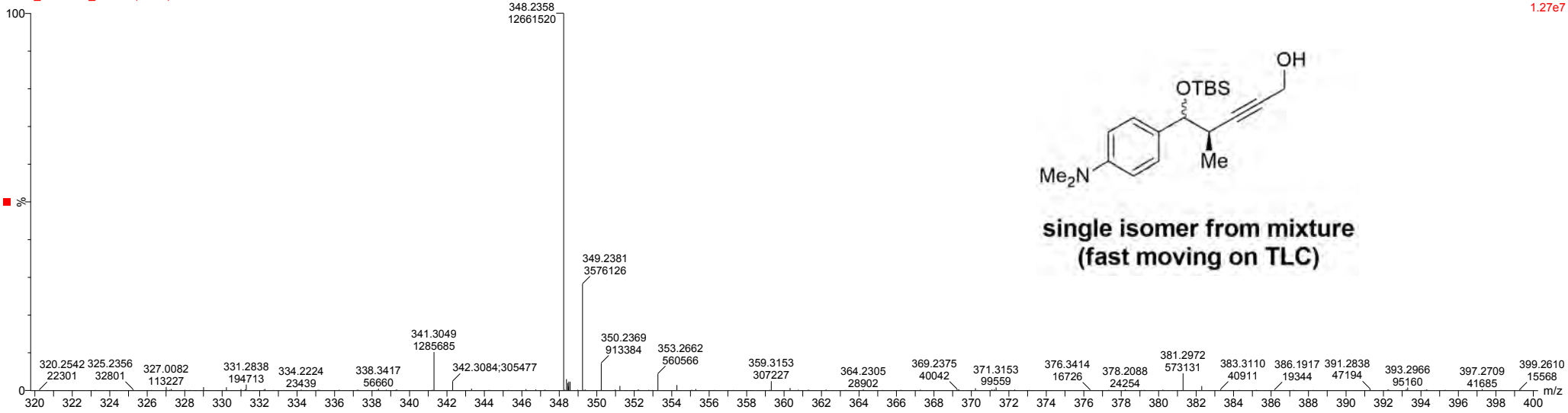
ASEP_19JAN18_006 (0.465) Is (1.00,1.00) C20H33NO2Si

1: TOF MS ES+
7.35e12

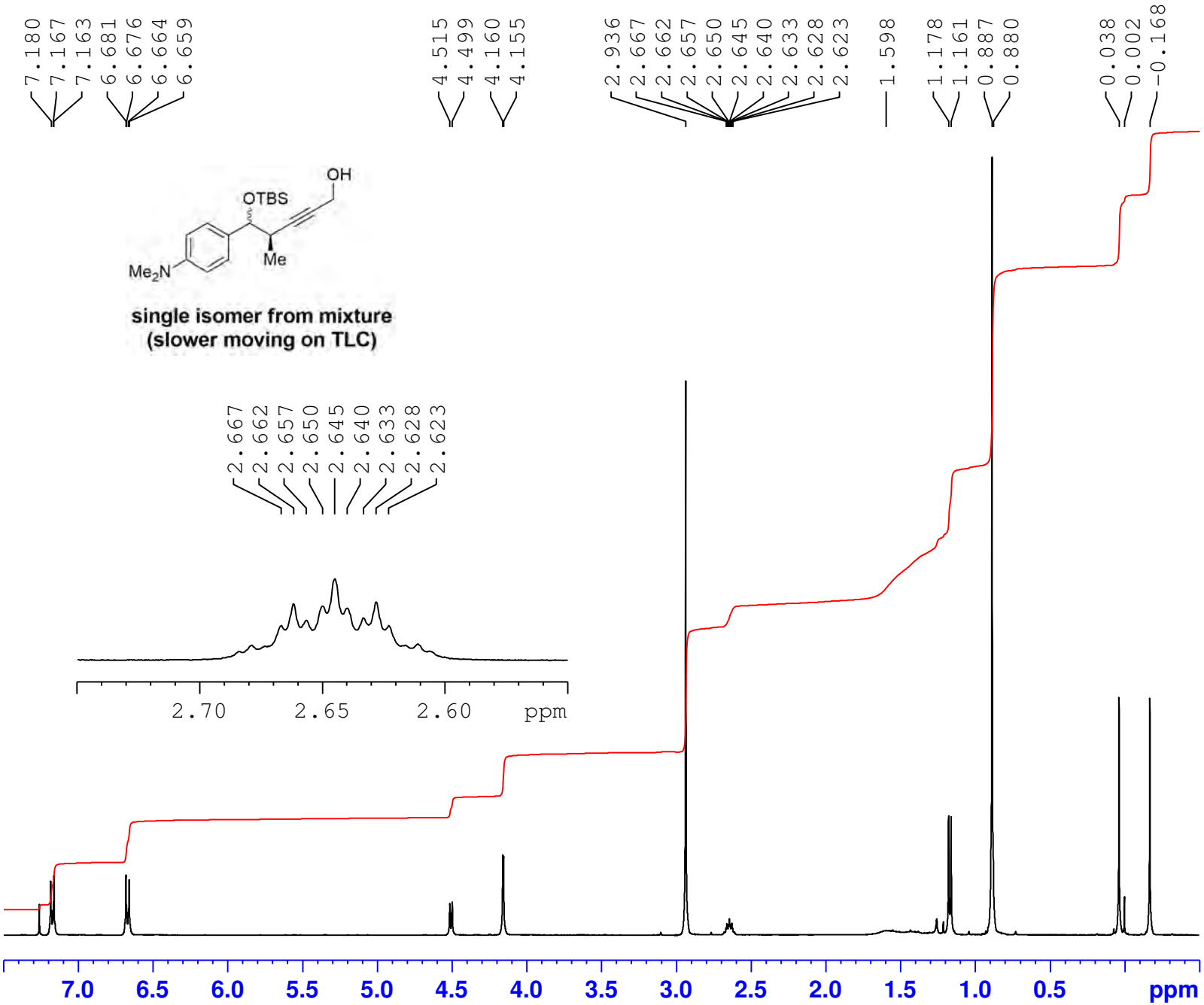


ASEP_19JAN18_006 23 (0.465)

1: TOF MS ES+
1.27e7



single isomer from mixture (fast moving on TLC)



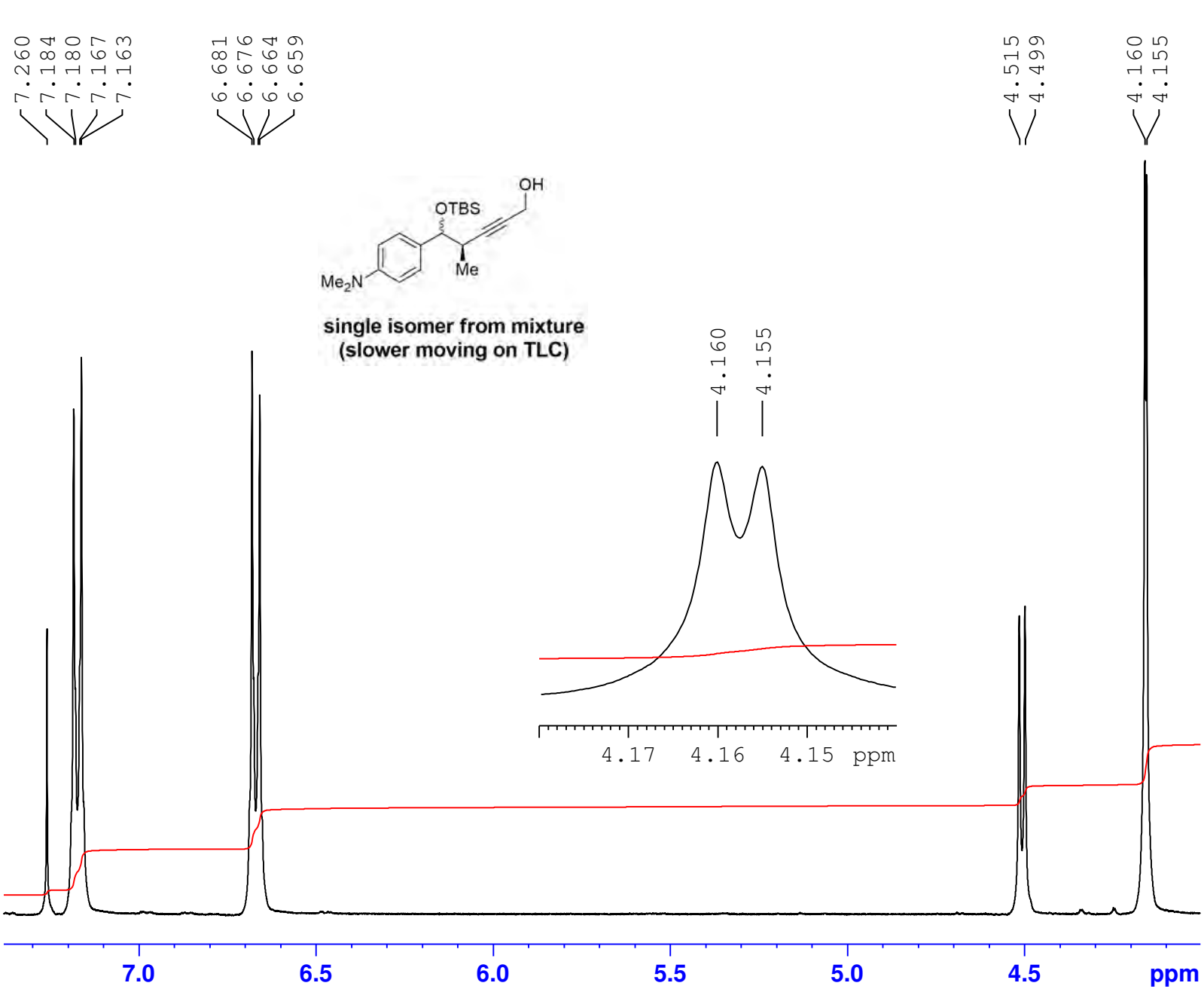
BRUKER

```
Current Data Parameters
NAME                  I-PK-80B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190515
Time                     12.57
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                       71.8
DW                     41.600 usec
DE                      9.85 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                     131072
SF               399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

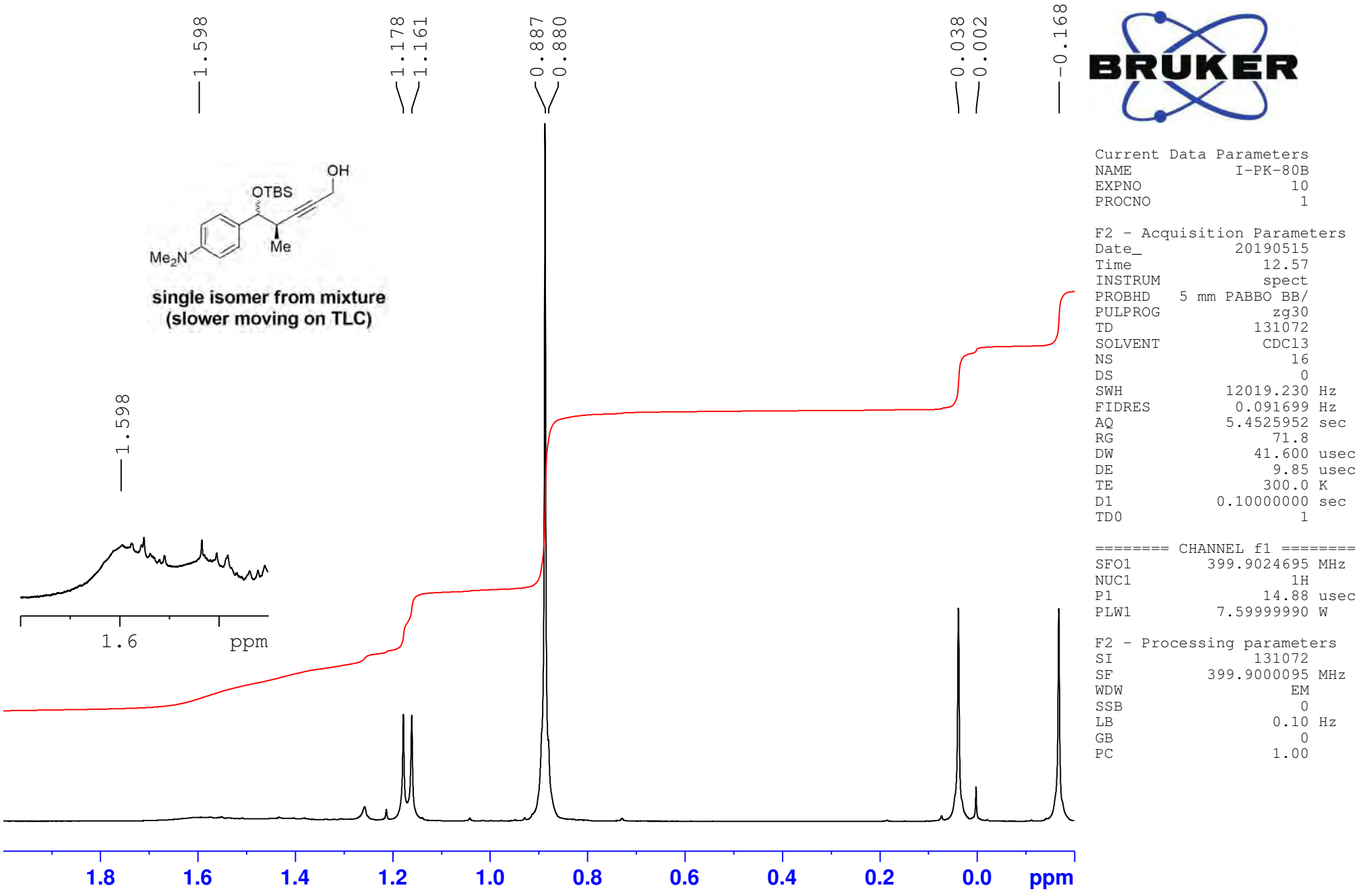



```
Current Data Parameters
NAME                  I-PK-80B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190515
Time                     12.57
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                        41.600 usec
DE                         9.85 usec
TE                        300.0 K
D1                   0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                        14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

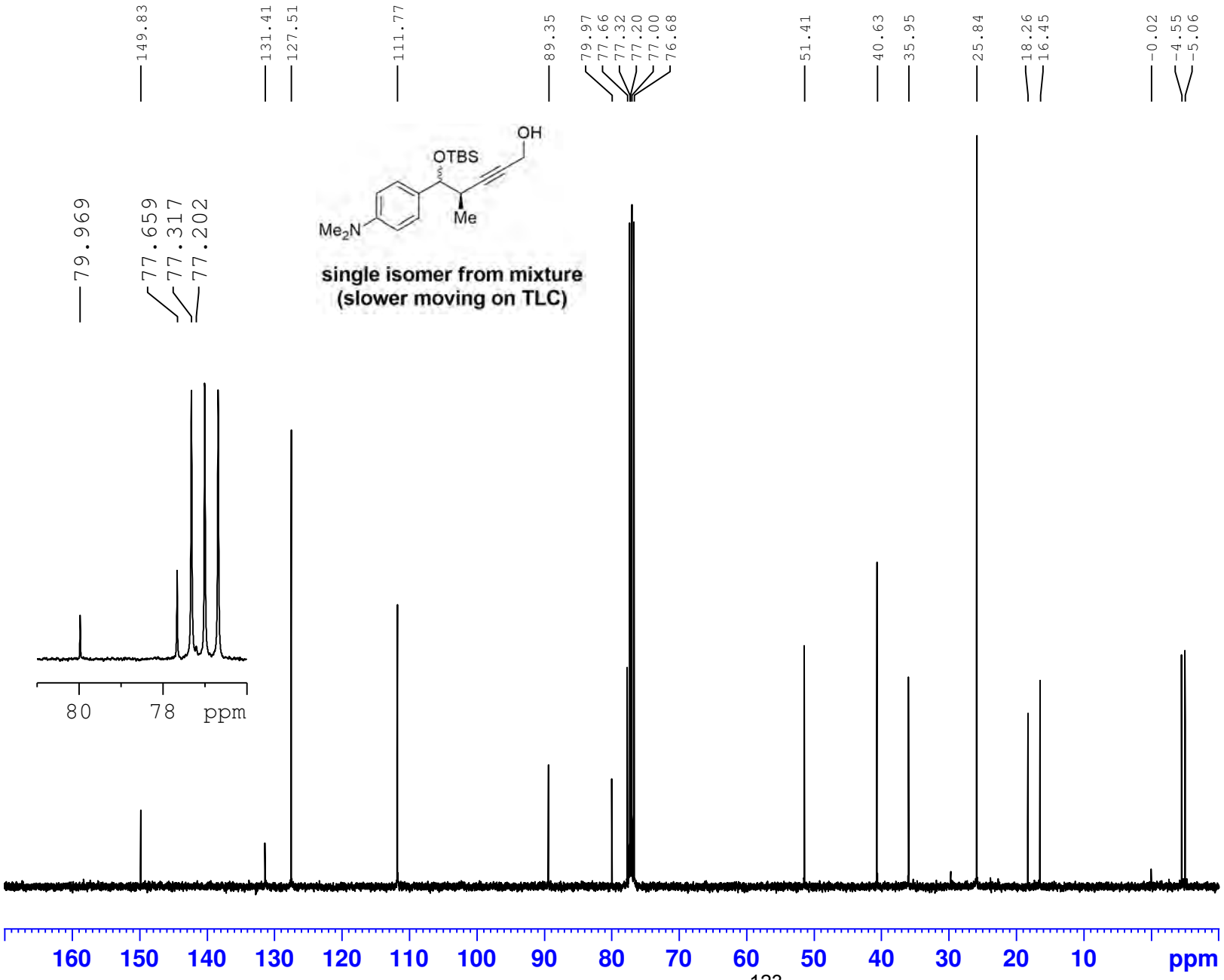



```
Current Data Parameters
NAME                  I-PK-80B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190515
Time                     12.57
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                    0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



BRUKER

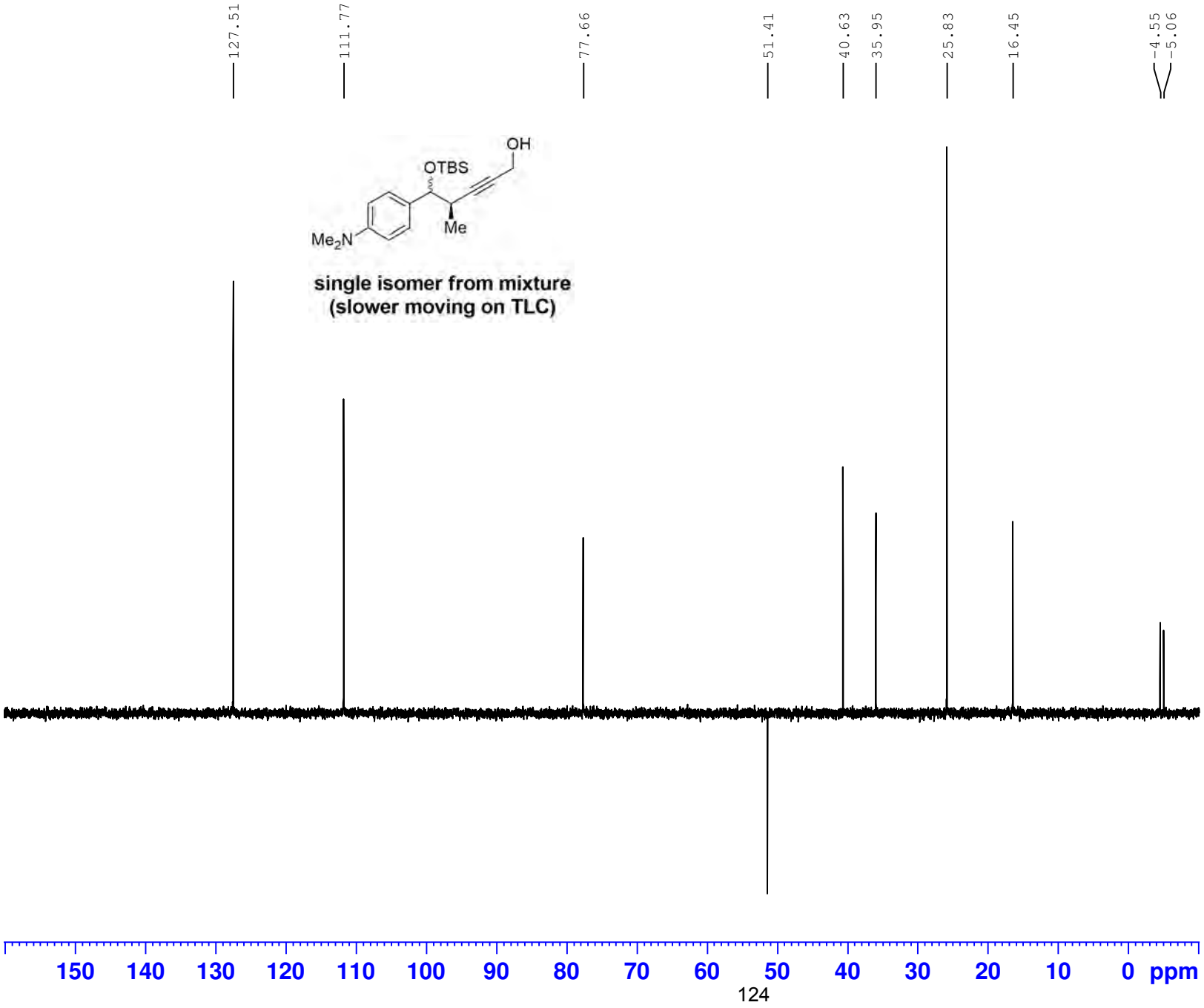
```
Current Data Parameters
NAME                 I-PK-80B
EXPNO                      21
PROCNO                      1

F2 - Acquisition Parameters
Date_               20190516
Time                    9.15
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                     119044
SOLVENT                 CDCl3
NS                       1200
DS                          4
SWH                25000.000 Hz
FIDRES              0.210006 Hz
AQ                  2.3808801 sec
RG                       2050
DW                     20.000 usec
DE                      9.12 usec
TE                     300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             100.5659947 MHz
NUC1                     13C
P1                     10.00 usec
PLW1            44.46300125 W

======== CHANNEL f2 ========
SFO2             399.9015996 MHz
NUC2                     1H
CPDPRG[2              waltz64
PCPD2                  90.00 usec
PLW2             7.59999990 W
PLW12            0.20774999 W
PLW13            0.16827001 W

F2 - Processing parameters
SI                      131072
SF               100.5549363 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



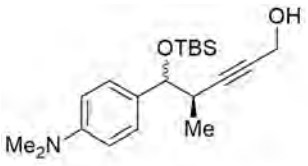
Current Data Parameters
NAME I-PK-80B
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190516
Time 9.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

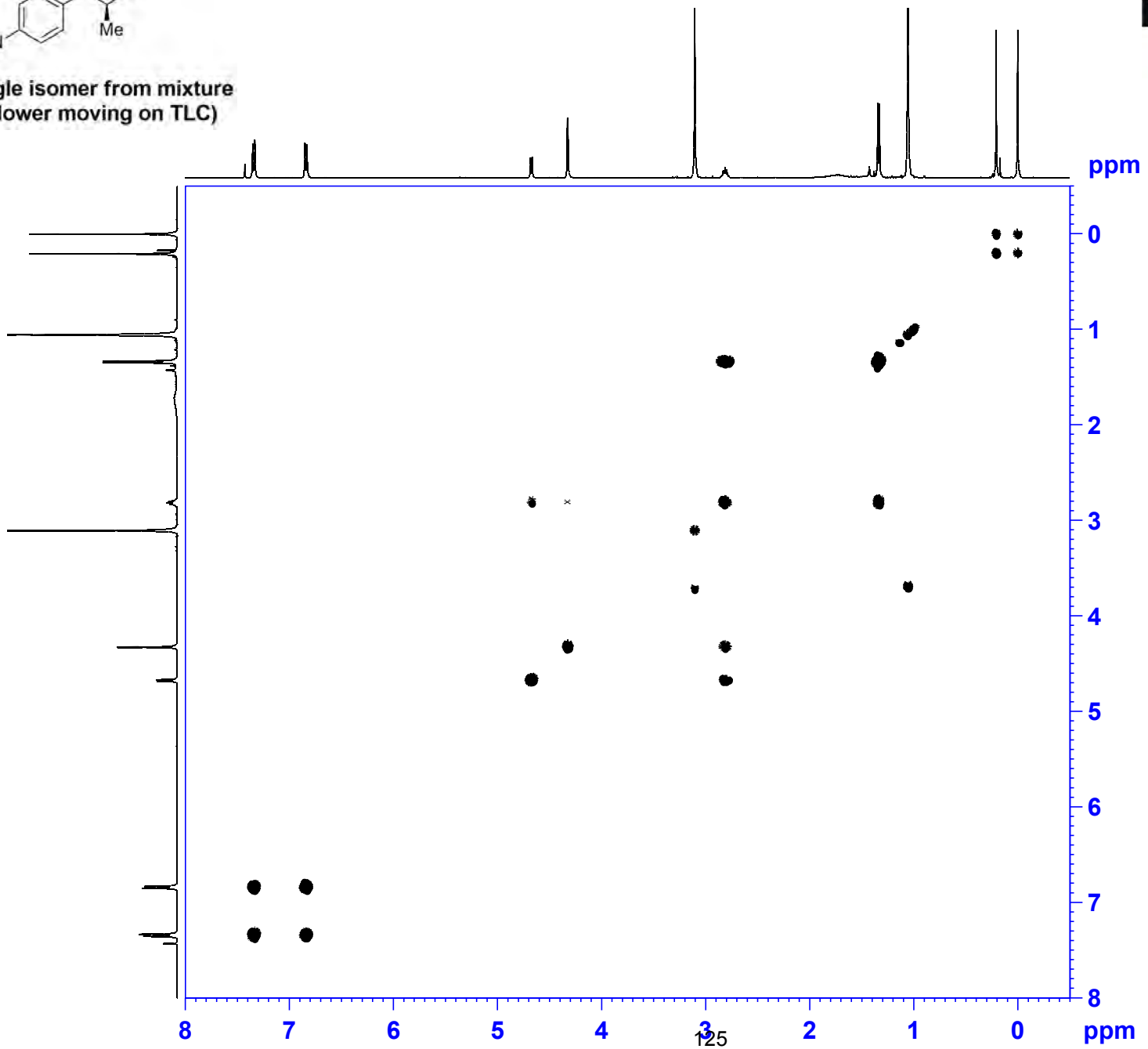
======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549361 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



single isomer from mixture
(slower moving on TLC)



BRUKER

```
Current Data Parameters
NAME                  I-PK-80B
EXPNO                       23
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190516
Time                      9.33
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                        2048
SOLVENT                 CDCl3
NS                           1
DS                           8
SWH                  3556.188 Hz
FIDRES               1.736420 Hz
AQ                   0.2879488 sec
RG                        2050
DW                    140.600 usec
DE                      6.50 usec
TE                      300.0 K
D0                 0.00000300 sec
D1                 0.85581988 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00028120 sec

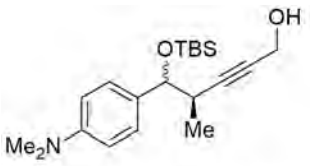
======== CHANNEL f1 ========
SFO1               399.9014201 MHz
NUC1                       1H
P1                      14.88 usec
P17                   2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]           SMSQ10.100
GPNAM[2]           SMSQ10.100
GPNAM[3]           SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

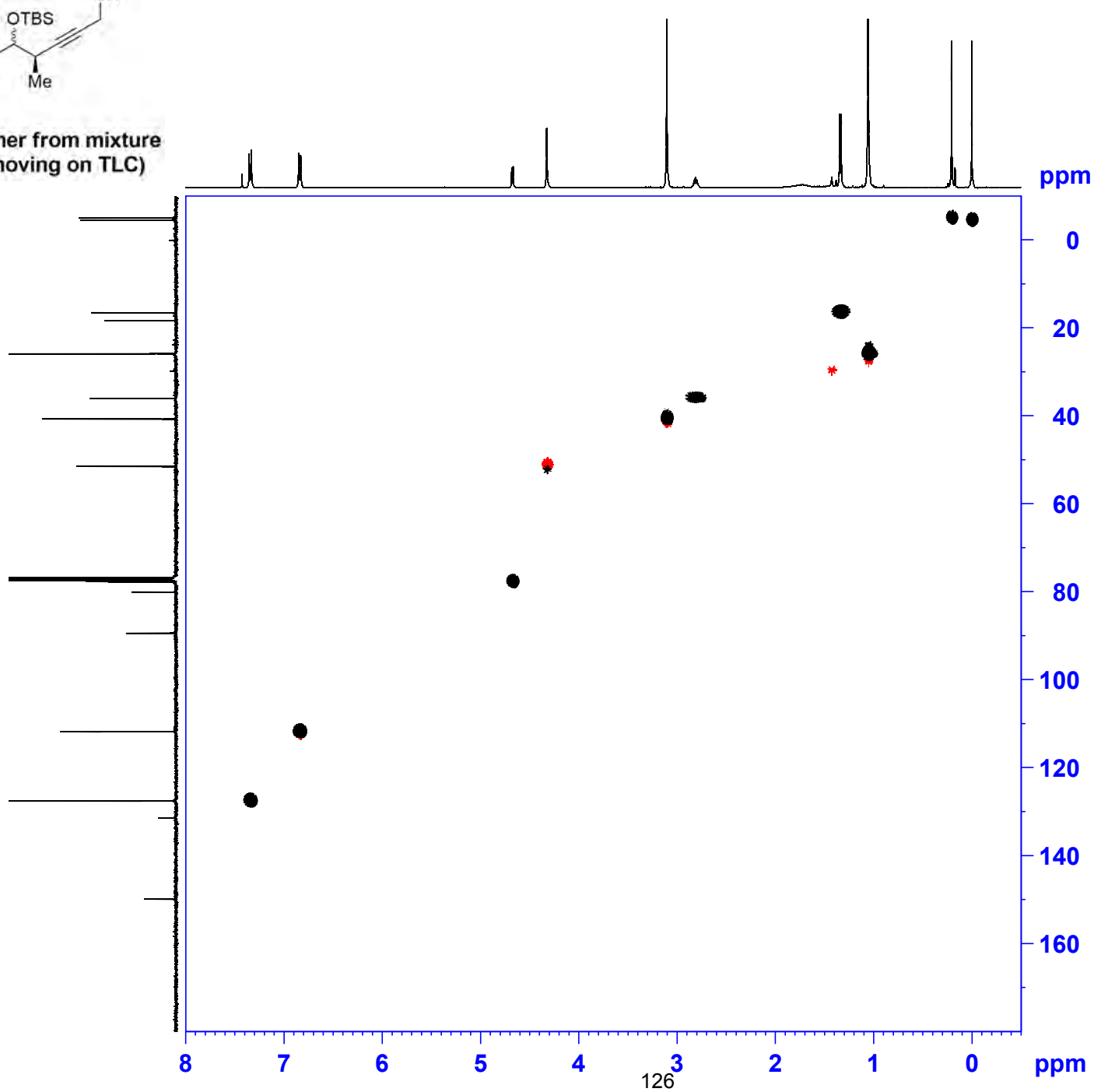
F1 - Acquisition parameters
TD                         256
SFO1                399.9014 MHz
FIDRES              27.782717 Hz
SW                      8.893 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                 399.8999424 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                 399.8999424 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
```



single isomer from mixture
(slower moving on TLC)



Current Data Parameters
NAME I-PK-80B
EXPNO 24
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190516
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG hsqcedetgpsp.3
TD 1024
SOLVENT CDCl3
NS 2
DS 32
SWH 4807.692 Hz
FIDRES 4.695012 Hz
AQ 0.1064960 sec
RG 2050
DW 104.000 usec
DE 6.50 usec
TE 300.1 K
CNST2 145.0000000
D0 0.00000300 sec
D1 0.80000001 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D21 0.00360000 sec
IN0 0.00001910 sec

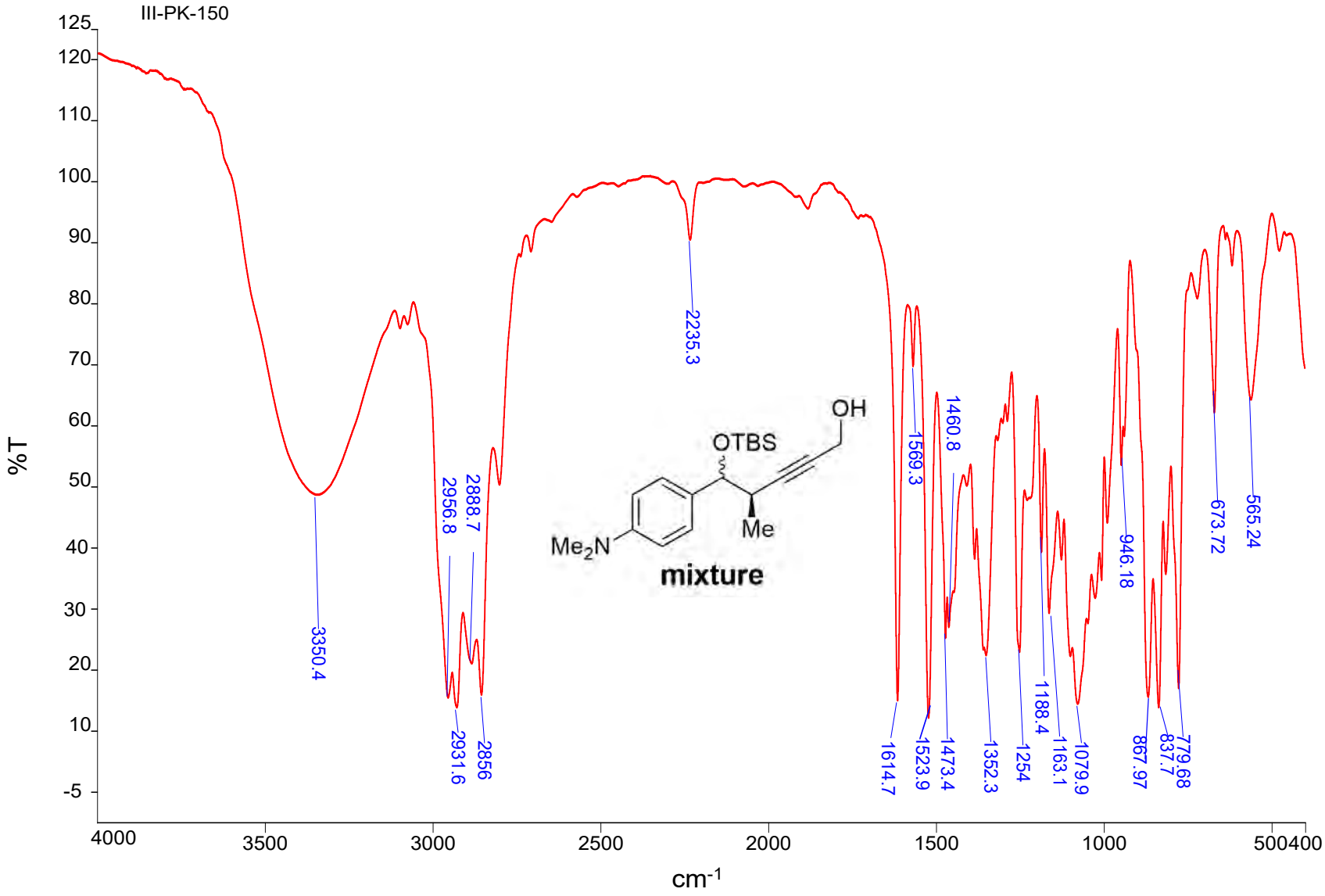
======== CHANNEL f1 ========
SFO1 399.9018806 MHz
NUC1 1H
P1 14.88 usec
P2 29.76 usec
P28 0 usec
PLW1 7.59999990 W

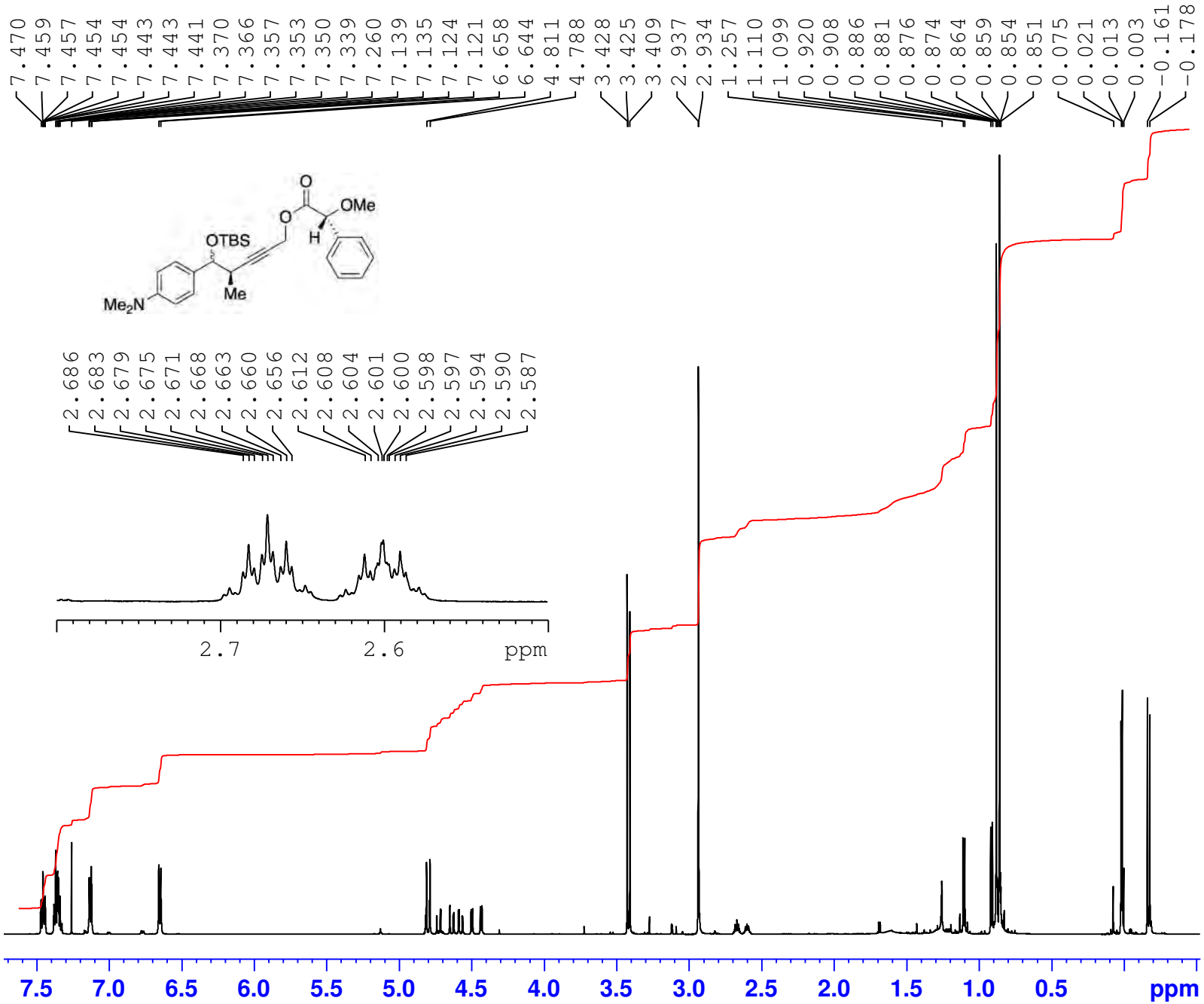
======== CHANNEL f2 ========
SFO2 100.5670016 MHz
NUC2 13C
CPDPRG[2 garp4
P3 10.00 usec
P14 500.00 usec
P31 1900.00 usec
PCPD2 80.00 usec
PLW0 0 W
PLW2 44.46300125 W
PLW12 0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
SPW3 6.79339981 W
SPNAM[18 Crp60_xfilt.2
SPOAL18 0.500
SPOFFS18 0 Hz
SPW18 1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 100.567 MHz
FIDRES 204.515701 Hz
SW 260.304 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 399.8999432 MHz
WDW QSINE


III-PK-150
%T
cm-1
4000
3500
3000
2500
2000
1500
1000
500400
125
120
110
100
90
80
70
60
50
40
30
20
10
-5
3350.4
2956.8
2931.6
2888.7
2856
2235.3
1614.7
1569.3
1523.9
1473.4
1460.8
1352.3
1254
1188.4
1163.1
1079.9
946.18
867.97
837.7
779.68
673.72
565.24
OH
OTBS
Me
Me2N
mixture



Current Data Parameters

NAME	III-PK-68
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

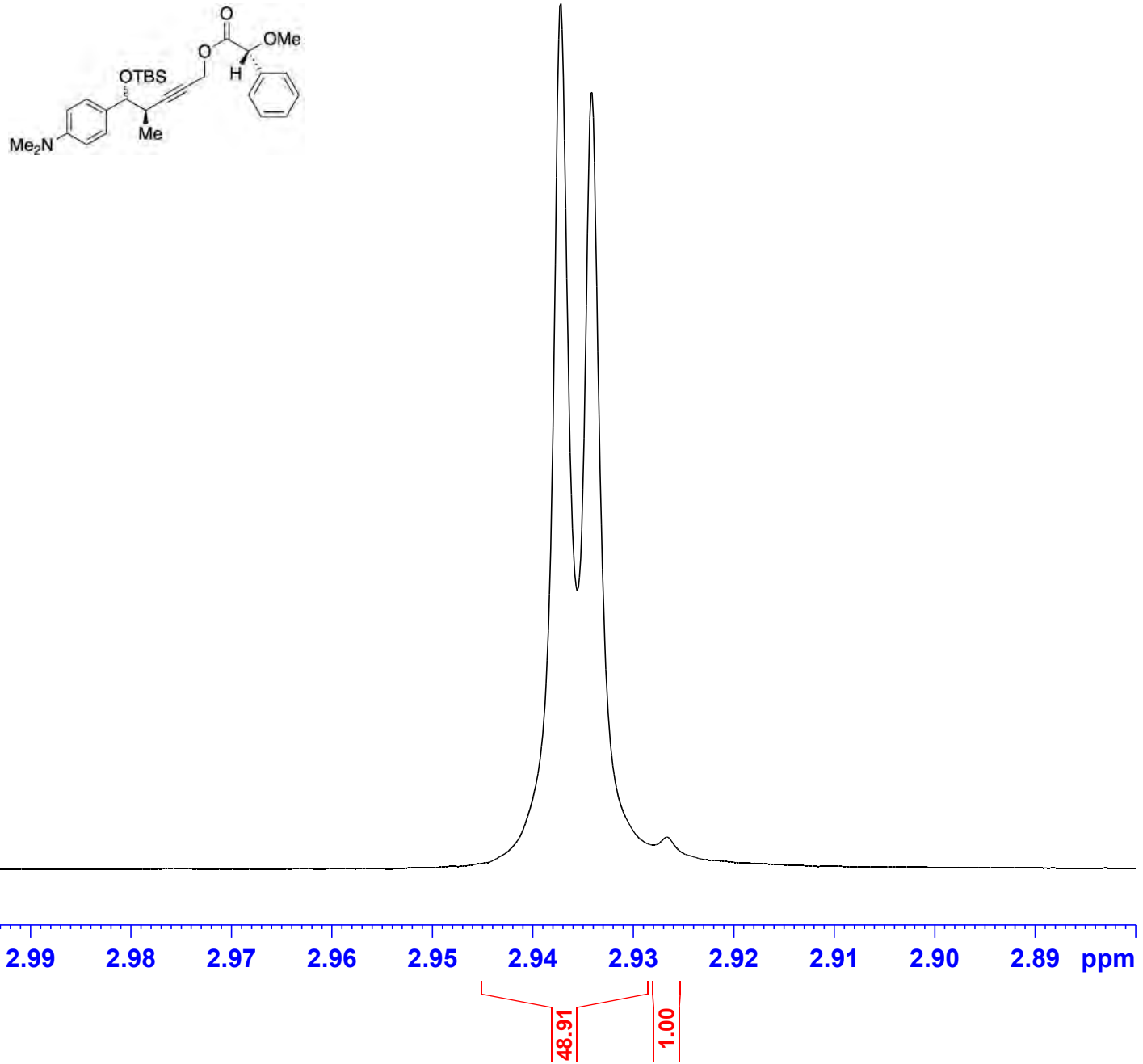
Date_	20191104
Time	15.49
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	180286
SOLVENT	CDCl3
NS	16
DS	0
SWH	18028.846 Hz
FIDRES	0.100001 Hz
AQ	4.9999318 sec
RG	49.63
DW	27.733 usec
DE	7.60 usec
TE	297.3 K
D1	0.10000000 sec
TD0	1

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

SFO1	600.1337060 MHz
NUC1	1H
P1	10.00 usec
PLW1	26.60000038 W

F2 - Processing parameters

SI	262144
SF	600.1300145 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

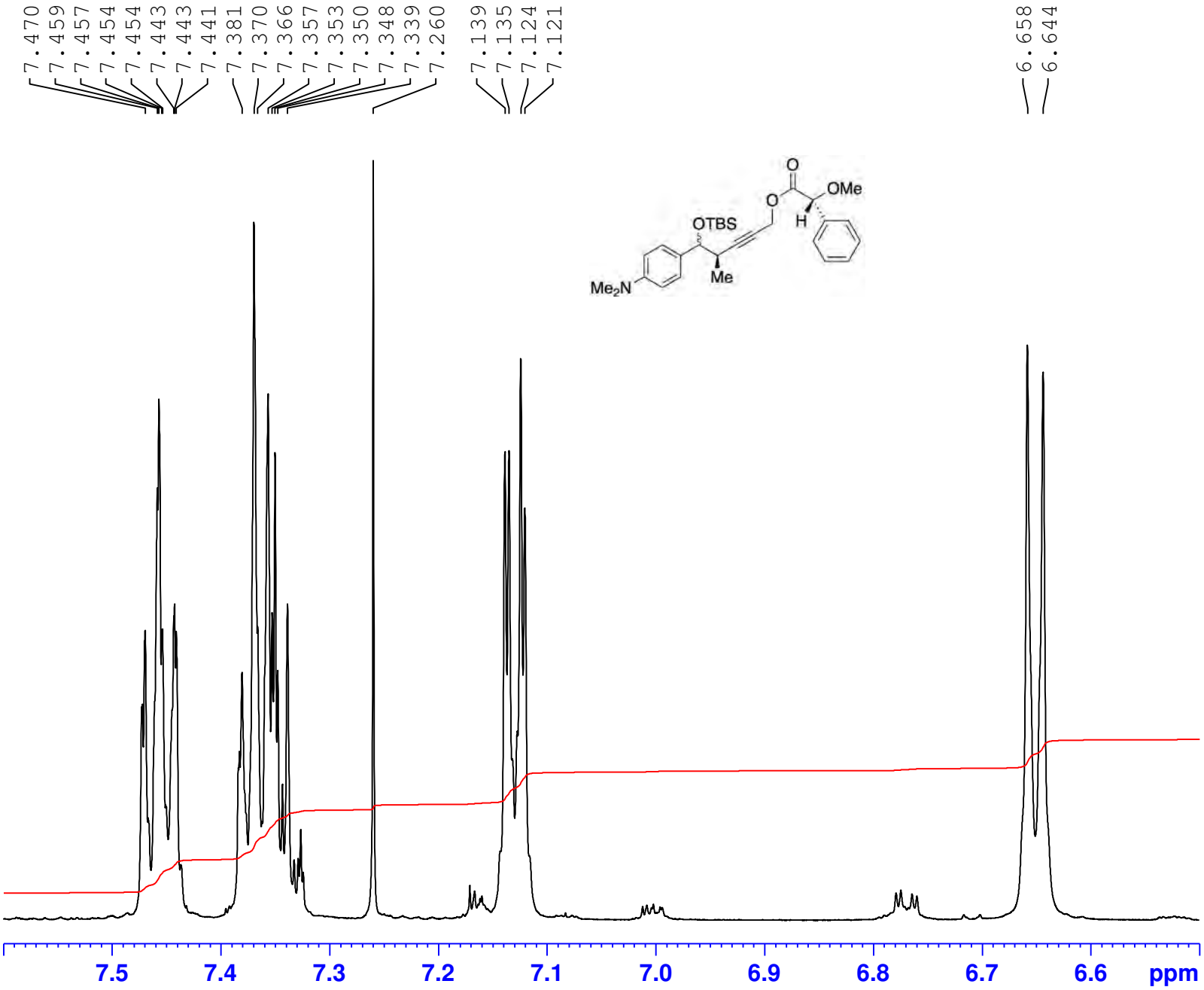



```
Current Data Parameters
NAME                 III-PK-68
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_               20191104
Time                   15.49
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                    180286
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                18028.846 Hz
FIDRES                0.100001 Hz
AQ                 4.9999318 sec
RG                        49.63
DW                     27.733 usec
DE                      7.60 usec
TE                     297.3 K
D1                 0.10000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             600.1337060 MHz
NUC1                       1H
P1                      10.00 usec
PLW1             26.60000038 W

F2 - Processing parameters
SI                      262144
SF               600.1300146 MHz
WDW                        EM
SSB          0
LB                      0.10 Hz
GB           0
PC                      1.00
```

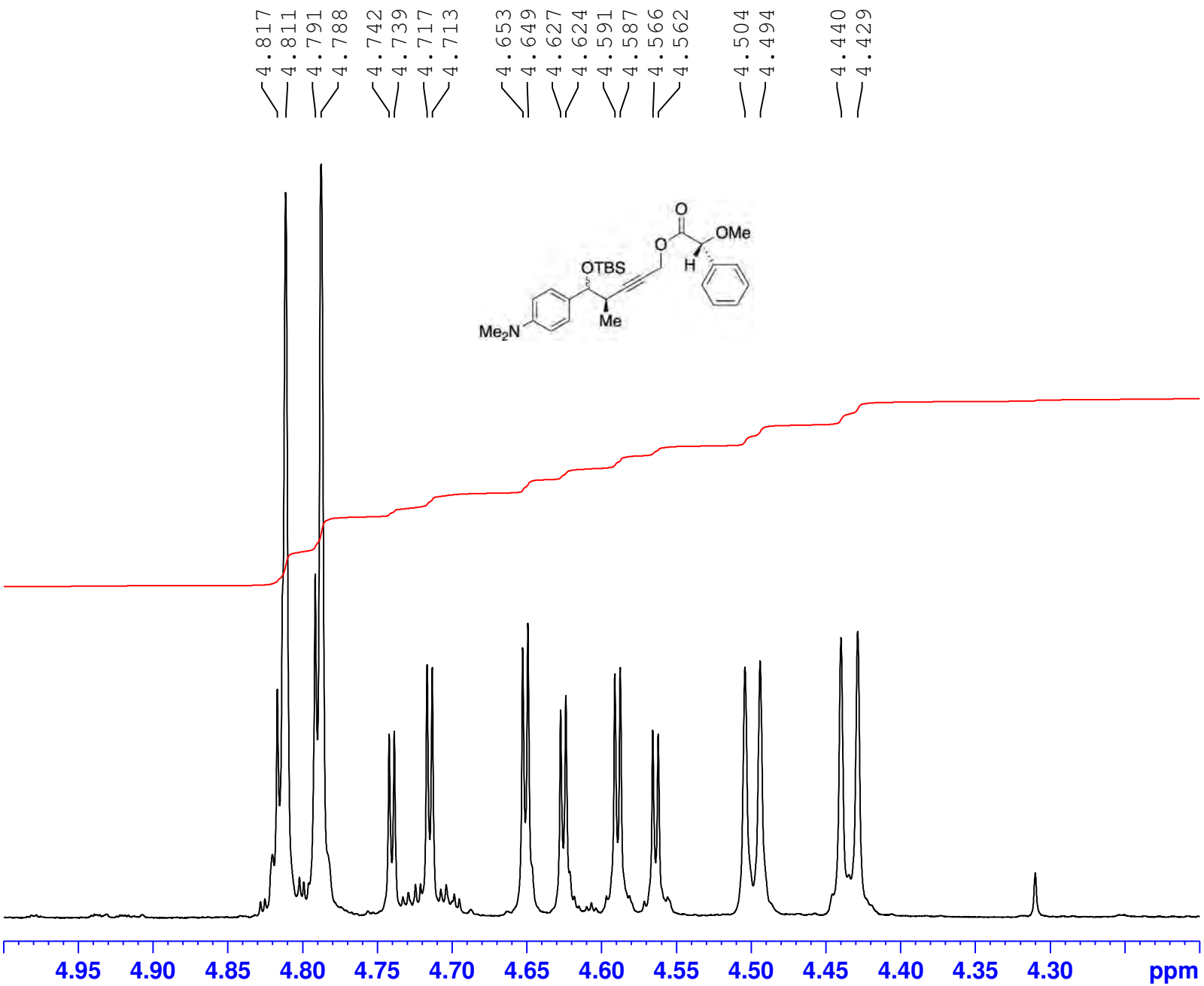


Current Data Parameters
NAME III-PK-68
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191104
Time 15.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 180286
SOLVENT CDCl3
NS 16
DS 0
SWH 18028.846 Hz
FIDRES 0.100001 Hz
AQ 4.9999318 sec
RG 49.63
DW 27.733 usec
DE 7.60 usec
TE 297.3 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.60000038 W

F2 - Processing parameters
SI 262144
SF 600.1300145 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



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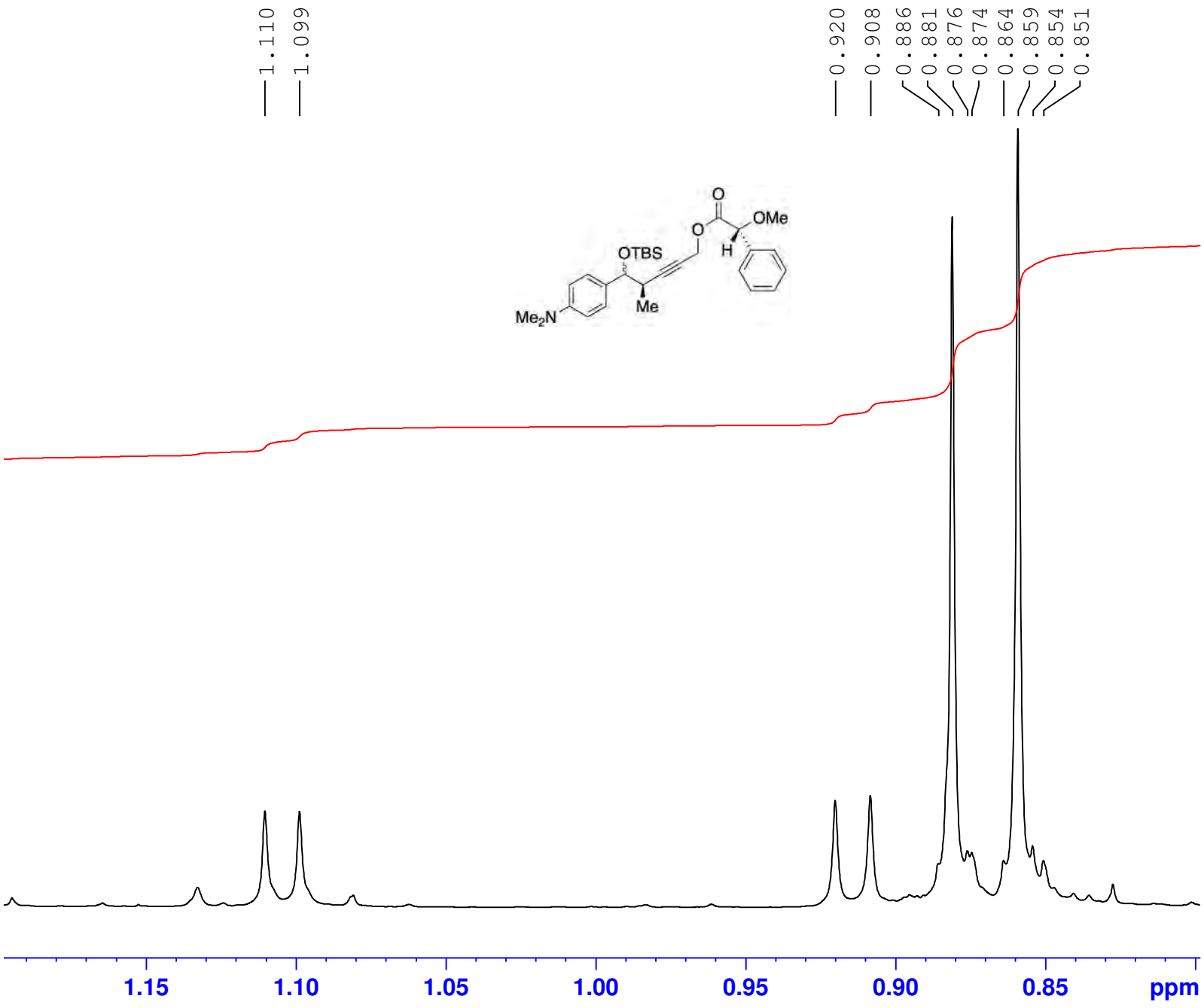
```
Current Data Parameters
NAME                  III-PK-68
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20191104
Time                     15.49
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                      180286
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  18028.846 Hz
FIDRES                0.100001 Hz
AQ                   4.9999318 sec
RG                       49.63
DW                      27.733 usec
DE                       7.60 usec
TE                      297.3 K
D1                   0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                600.1337060 MHz
NUC1                        1H
P1                       10.00 usec
PLW1                26.60000038 W

F2 - Processing parameters
SI                      262144
SF                  600.1300145 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00
```



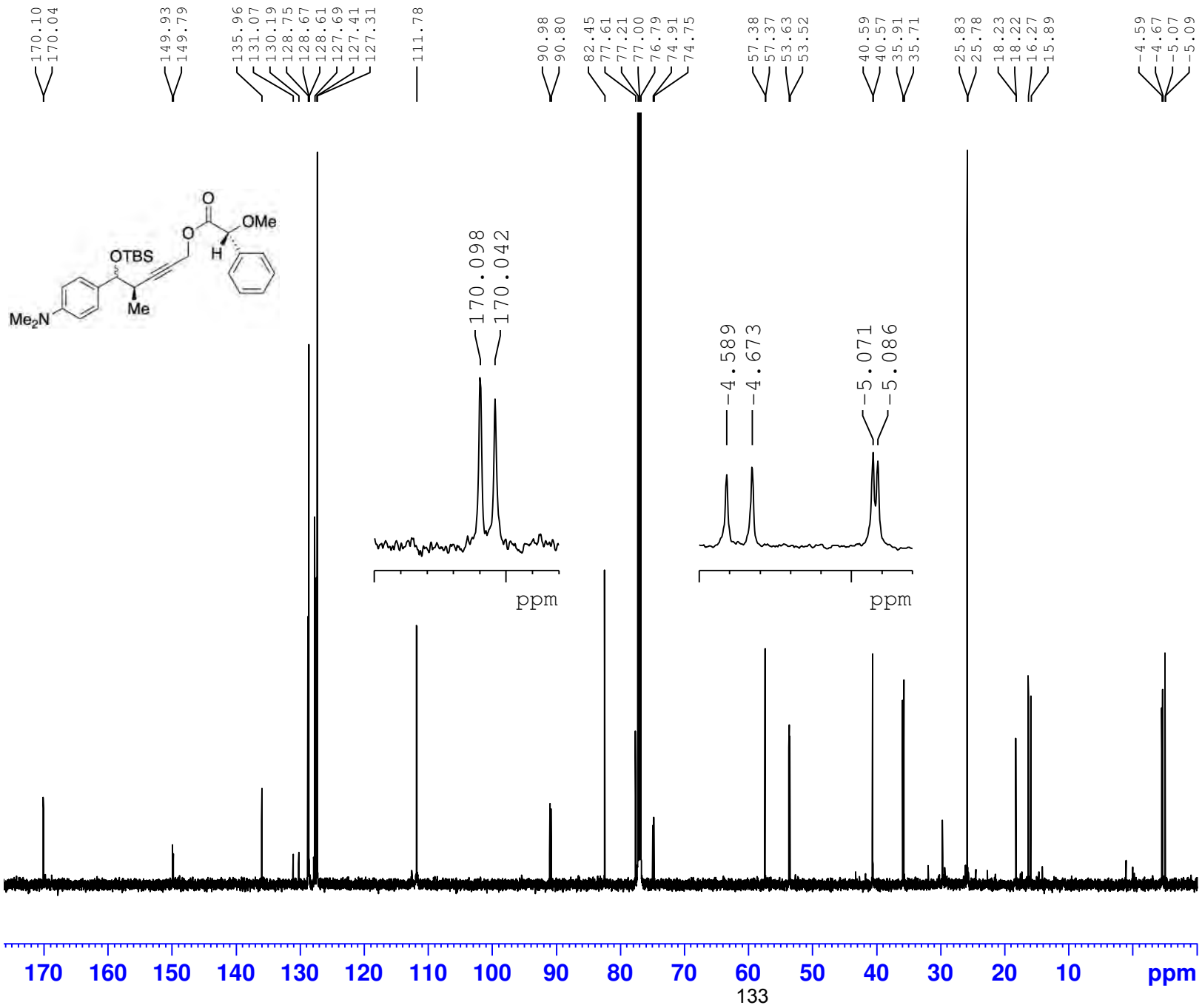
BRUKER

```
Current Data Parameters
NAME                  III-PK-68
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20191104
Time                     15.49
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                      180286
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  18028.846 Hz
FIDRES                0.100001 Hz
AQ                    4.9999318 sec
RG                        49.63
DW                       27.733 usec
DE                        7.60 usec
TE                       297.3 K
D1                   0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                600.1337060 MHz
NUC1                        1H
P1                       10.00 usec
PLW1                26.60000038 W

F2 - Processing parameters
SI                       262144
SF                  600.1300145 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00
```



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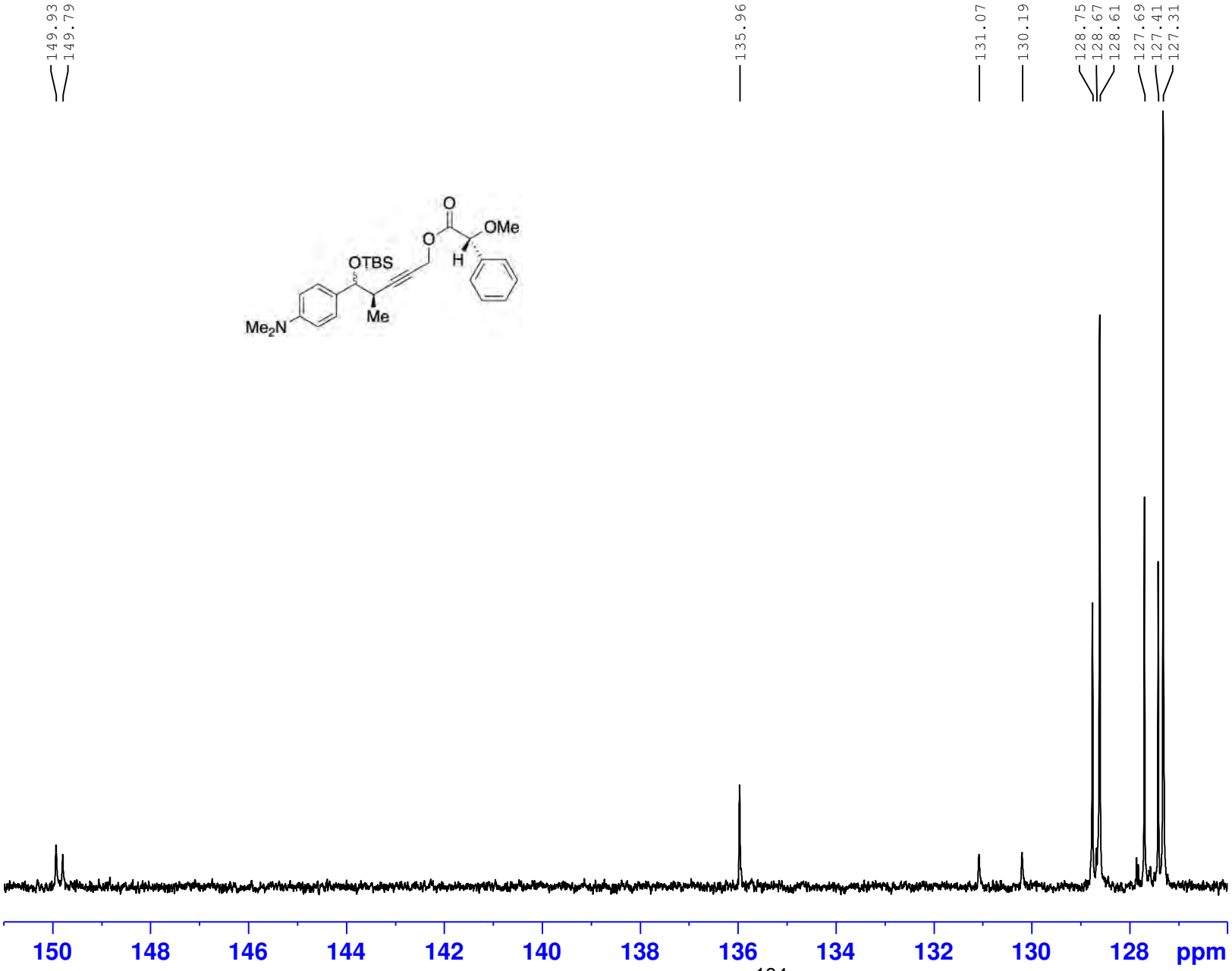
Current Data Parameters
NAME III-PK-68
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191104
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.3 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



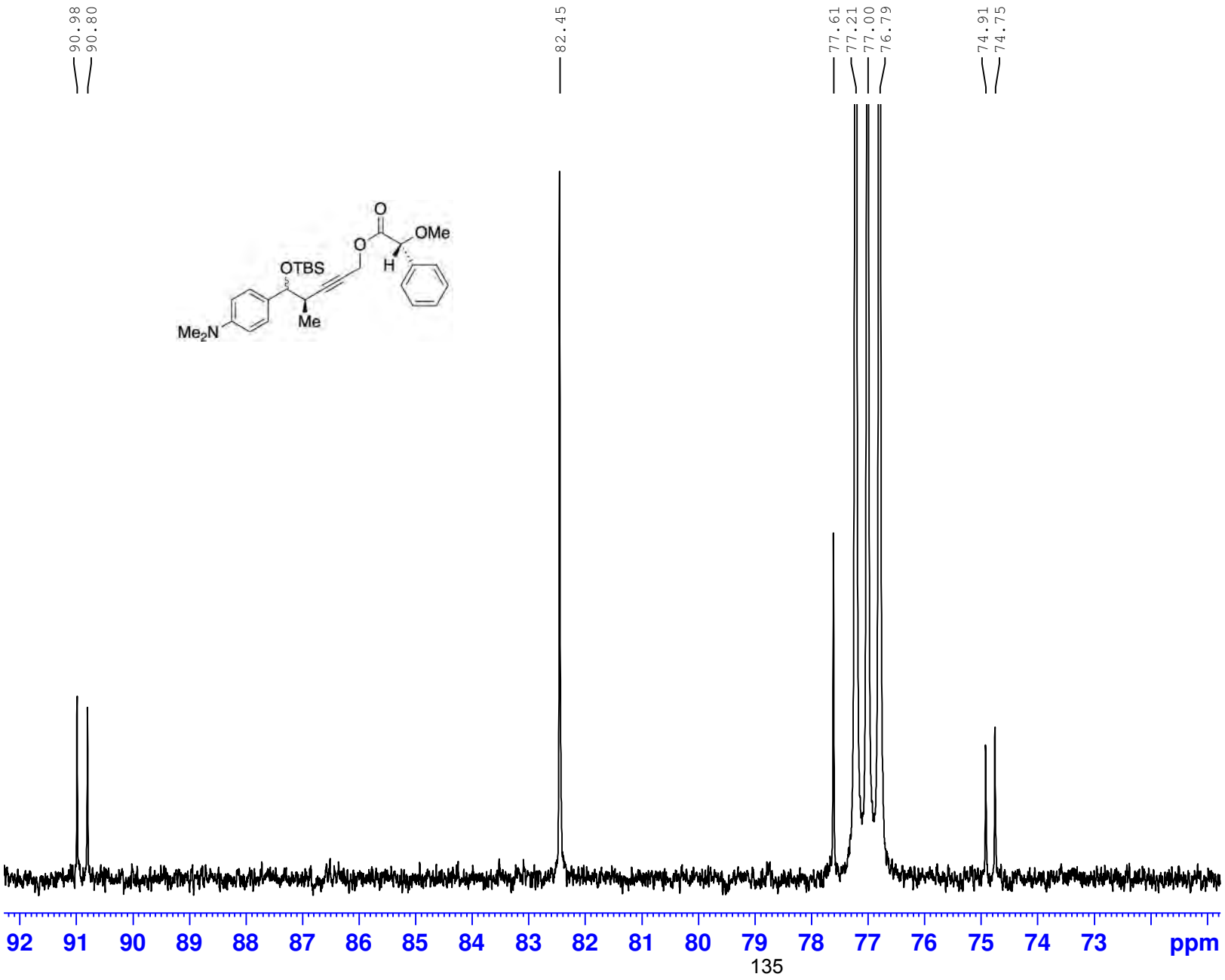
Current Data Parameters
NAME III-PK-68
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191104
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.3 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



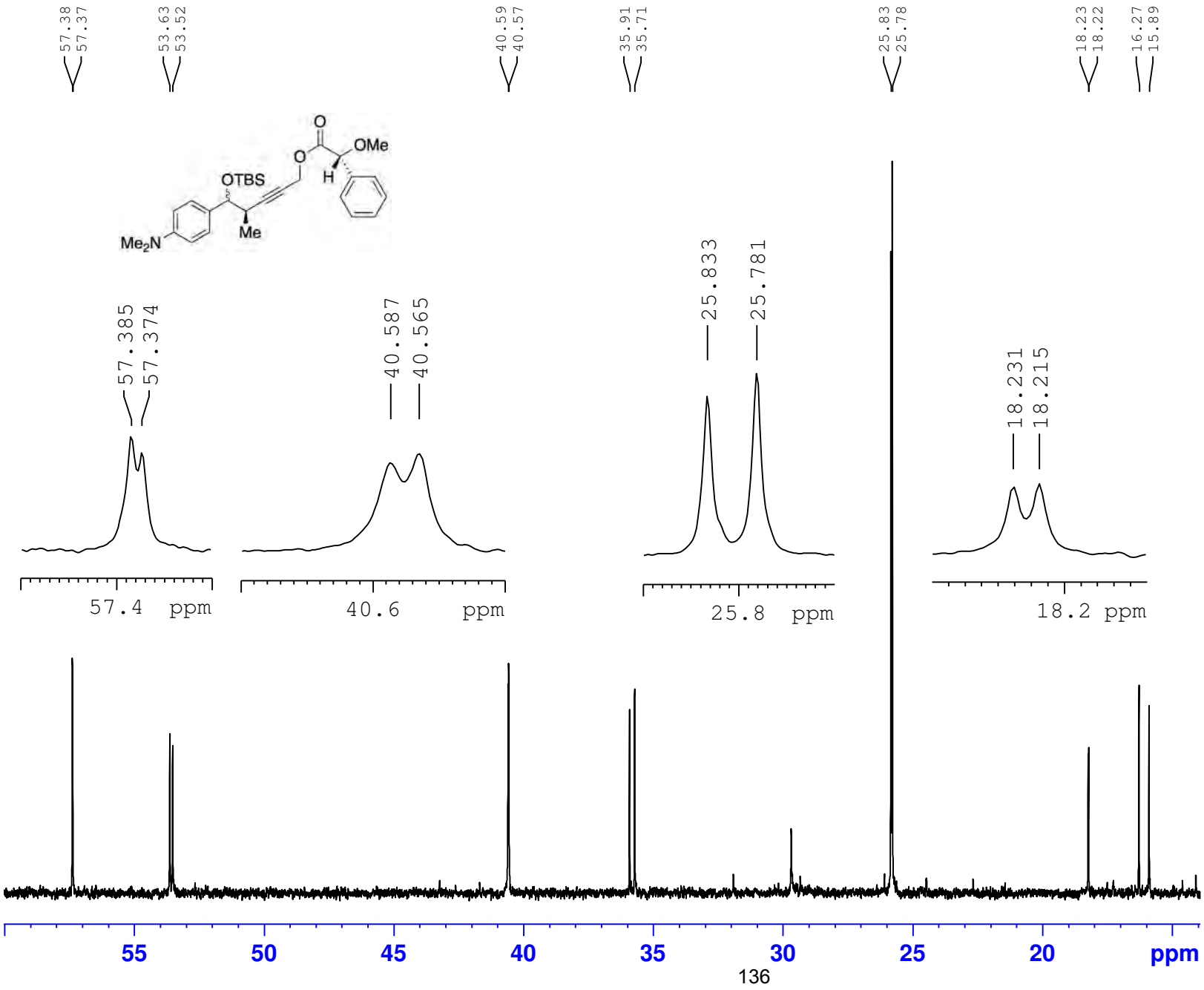
Current Data Parameters
NAME III-PK-68
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191104
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.3 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



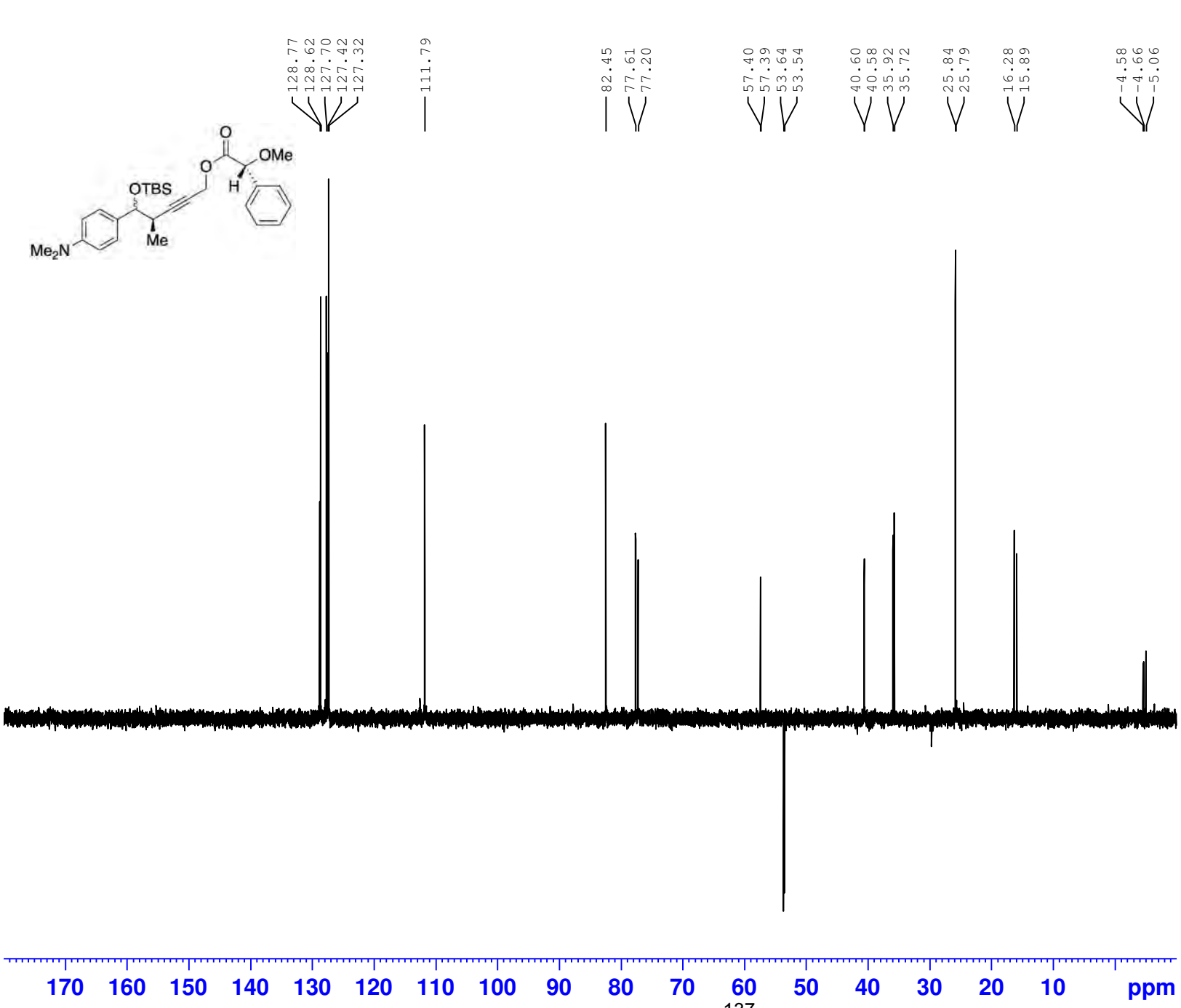
Current Data Parameters
NAME III-PK-68
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191104
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.3 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



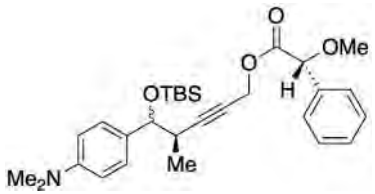
Current Data Parameters
NAME III-PK-68
EXPNO 12
PROCNO 1

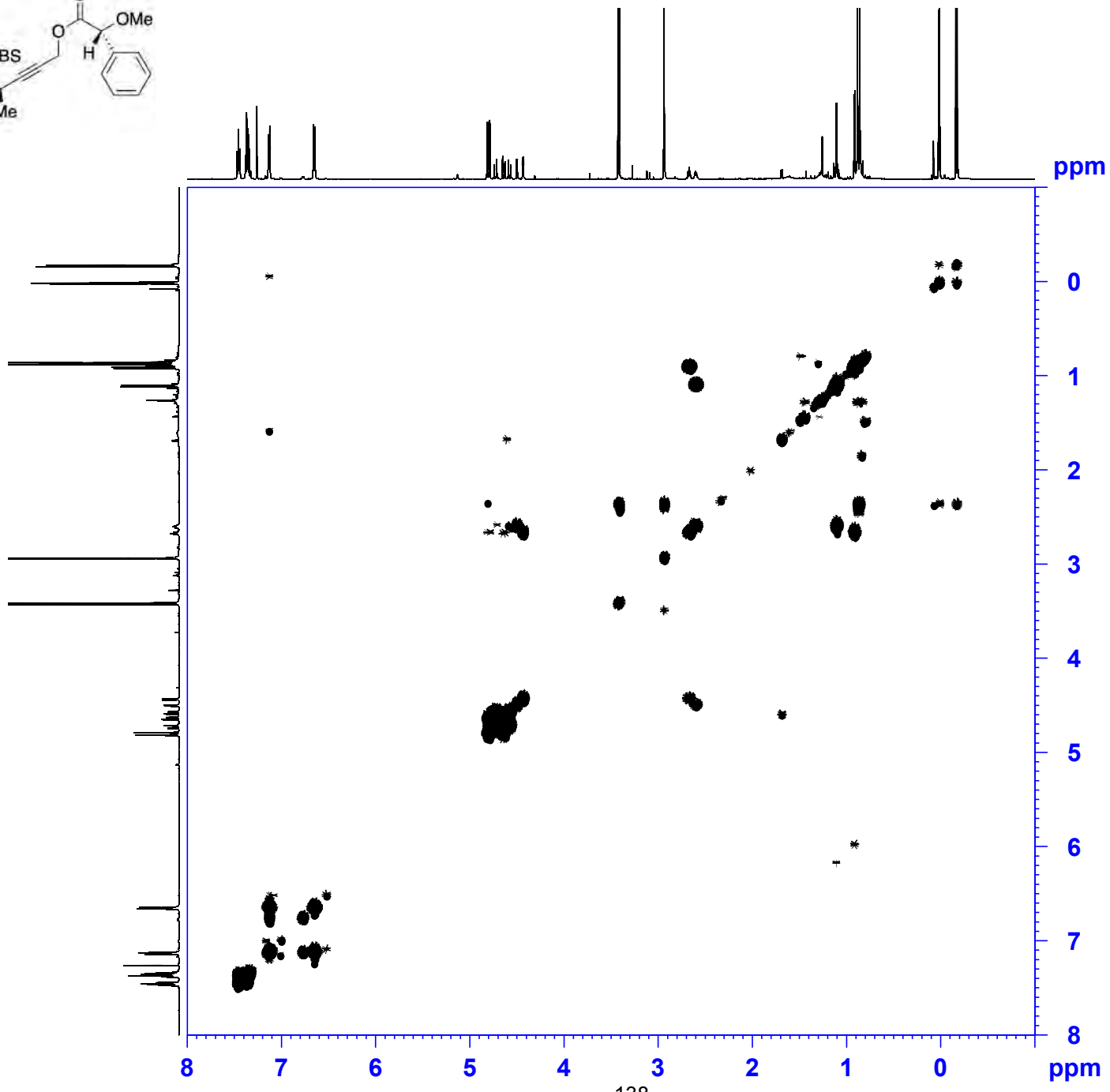
F2 - Acquisition Parameters
Date_ 20191104
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135.b
TD 119044
SOLVENT CDCl3
NS 256
DS 4
SWH 35714.285 Hz
FIDRES 0.300009 Hz
AQ 1.6666160 sec
RG 186.92
DW 14.000 usec
DE 7.44 usec
TE 299.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178962 MHz
NUC1 13C
P1 11.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 85.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 18.08300018 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
P3 10.20 usec
P4 20.40 usec
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W

F2 - Processing parameters
SI 131072
SF 150.9028098 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





BRUKER

```
Current Data Parameters
NAME            III-PK-68
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191104
Time                16.56
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                  2048
SOLVENT            CDCl3
NS                     1
DS                     8
SWH             7352.941 Hz
FIDRES          3.590303 Hz
AQ             0.1392640 sec
RG                186.92
DW                68.000 usec
DE                 6.50 usec
TE                298.7 K
D0             0.00000300 sec
D1             0.92422330 sec
D11            0.03000000 sec
D12            0.00002000 sec
D13            0.00000400 sec
D16            0.00020000 sec
IN0            0.00013620 sec

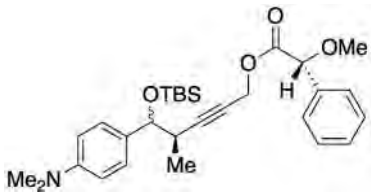
======== CHANNEL f1 ========
SFO1          600.1314363 MHz
NUC1                  1H
P1                10.00 usec
P17             2500.00 usec
PLW1         26.60000038 W
PLW10         3.93490005 W

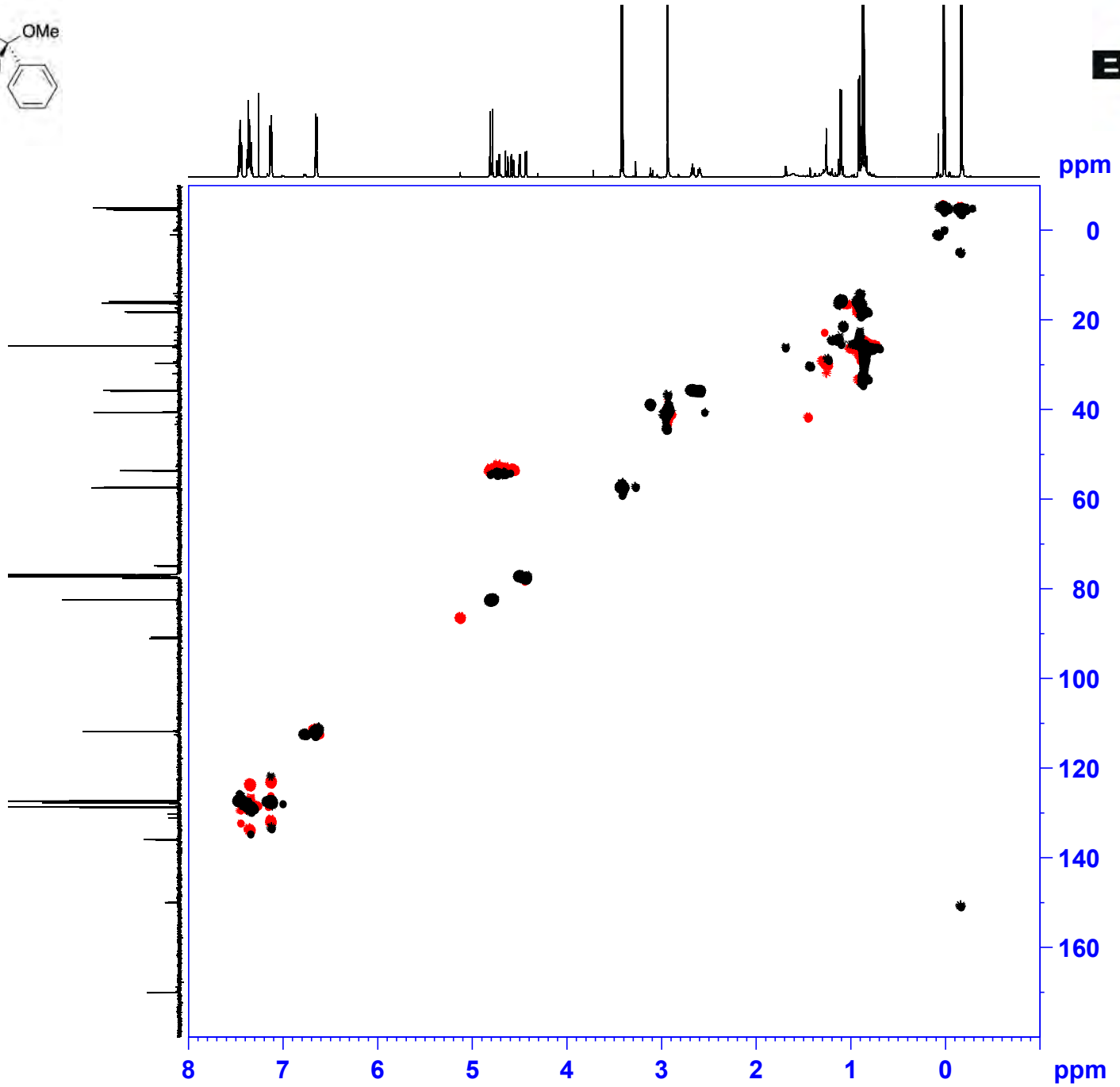
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1               16.00 %
GPZ2               12.00 %
GPZ3               40.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            600.1314 MHz
FIDRES          57.360500 Hz
SW                12.234 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           600.1300175 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           600.1300187 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```






```
Current Data Parameters
NAME                  III-PK-68
EXPNO                        14
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20191104
Time                     17.02
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                         1024
SOLVENT                  CDC13
NS                           2
DS                          32
SWH                  7211.539 Hz
FIDRES               7.042518 Hz
AQ                   0.0709973 sec
RG                      186.92
DW                      69.333 usec
DE                        6.50 usec
TE                       298.5 K
CNST2               145.0000000
D0                   0.00000300 sec
D1                   0.80000001 sec
D4                   0.00172414 sec
D11                  0.03000000 sec
D16                  0.00020000 sec
D21                  0.00360000 sec
IN0                  0.00002010 sec

======== CHANNEL f1 ========
SFO1                600.1328223 MHz
NUC1                        1H
P1                       10.00 usec
P2                       20.00 usec
P28             0 usec
PLW1                26.60000038 W

======== CHANNEL f2 ========
SFO2                150.9141236 MHz
NUC2                        13C
CPDPRG[2                 garp4
P3                       11.80 usec
P14                     500.00 usec
P31                    1730.00 usec
PCPD2                    65.00 usec
PLW0            0 W
PLW2                85.00000000 W
PLW12                2.80130005 W
SPNAM[3]        Crp60,0.5,20.1
SPOAL3                    0.500
SPOFFS3         0 Hz
SPW3                18.08300018 W
SPNAM[18        Crp60_xfilt.2
SPOAL18                   0.500
SPOFFS18        0 Hz
SPW18                5.22629976 W

====== GRADIENT CHANNEL =====
GPNAM[1]            SMSQ10.100
GPNAM[2]            SMSQ10.100
GPZ1                     80.00 %
GPZ2                     20.10 %
P16                    1000.00 usec

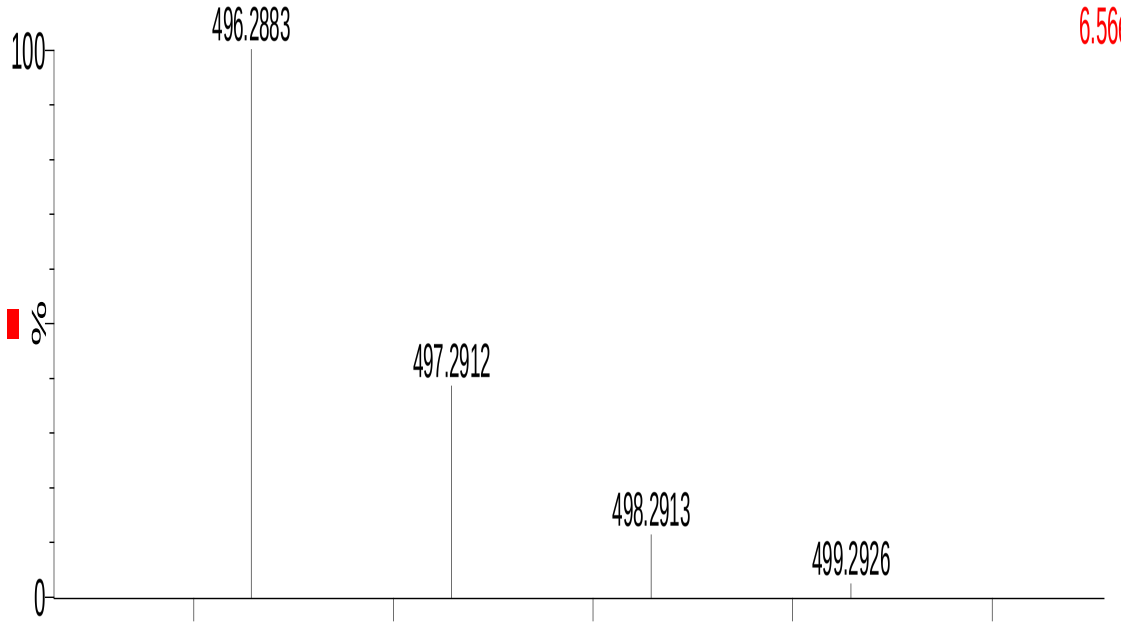
F1 - Acquisition parameters
TD                          256
SFO1                150.9141 MHz
FIDRES              194.340790 Hz
SW                     164.833 ppm
FnMODE          Echo-Antiecho

F2 - Processing parameters
SI                         1024
SF                  600.1300133 MHz
```

IV-PK-51 02-06-2021

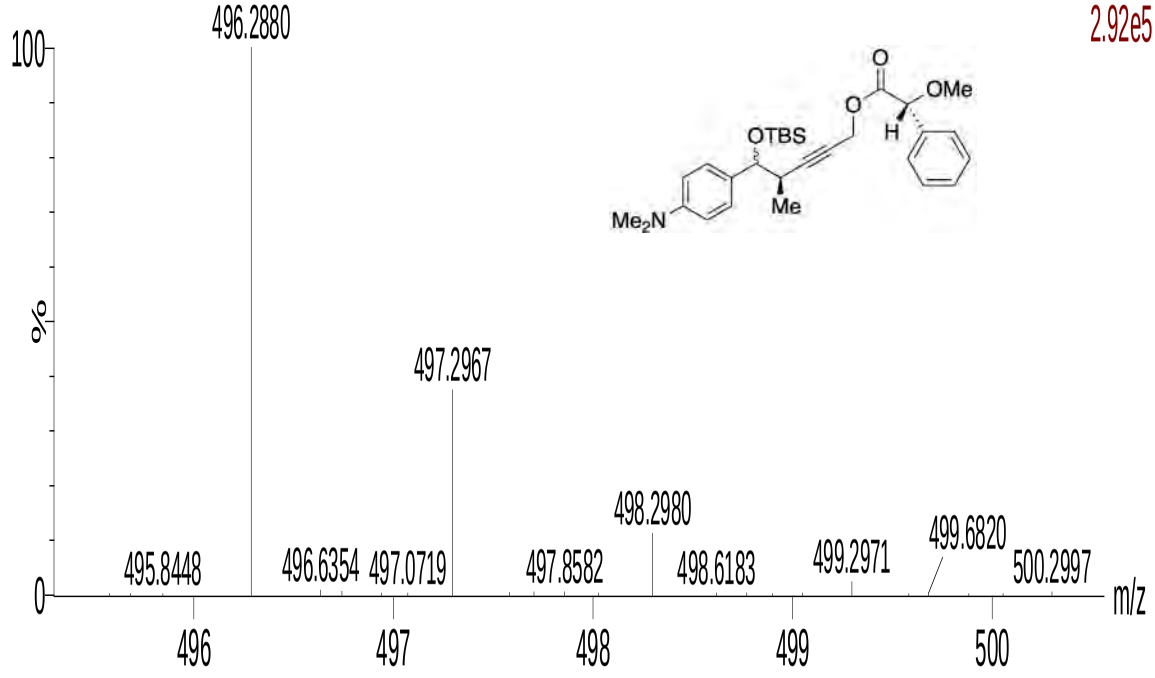
ASEP_02JUNE_2021_255 (0.322) Is (1.00,1.00) C29H41NO4SiH 1: TOF MS ES+

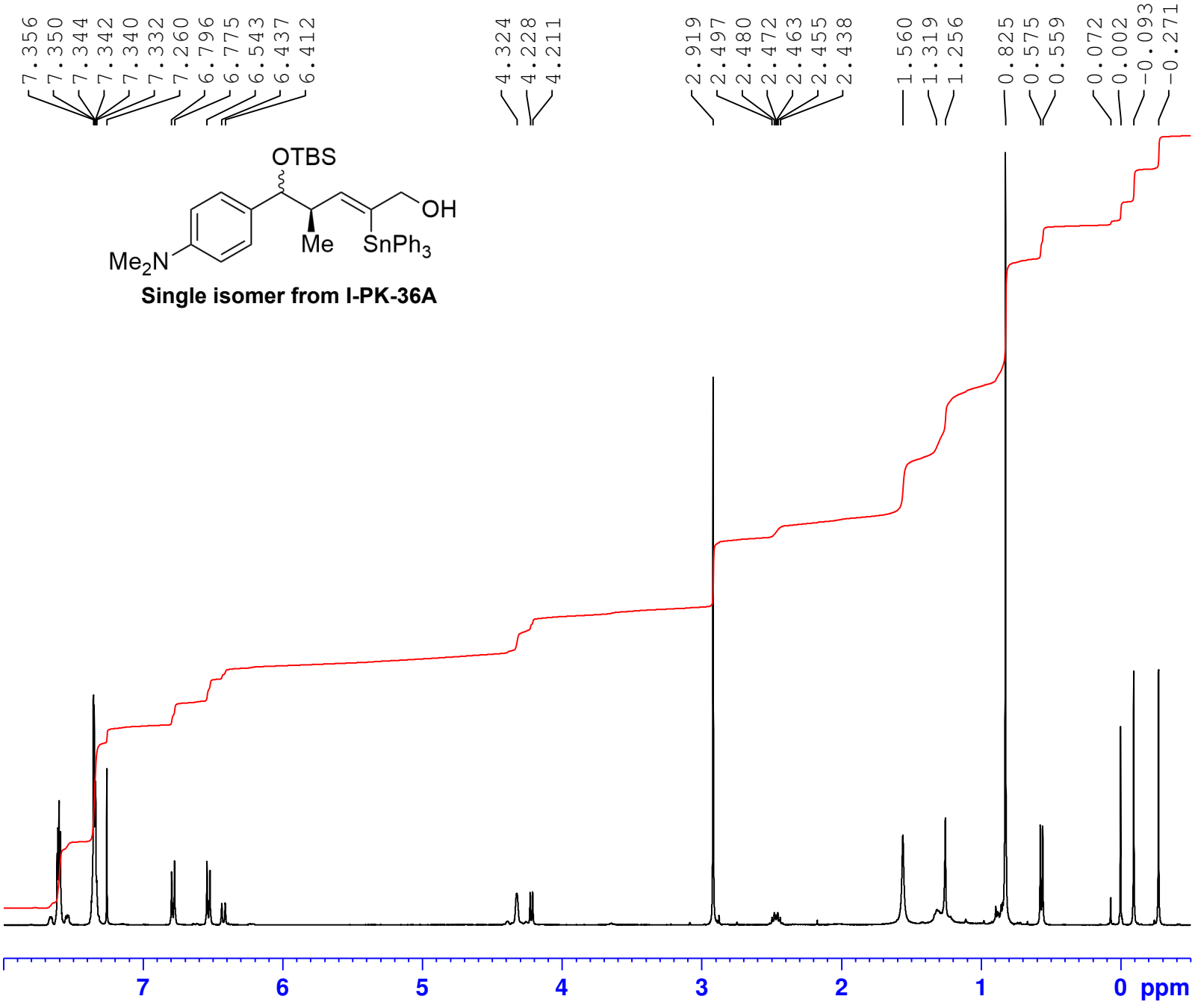
6.56e12



ASEP_02JUNE_2021_255 7 (0.322) 1: TOF MS ES+

2.92e5





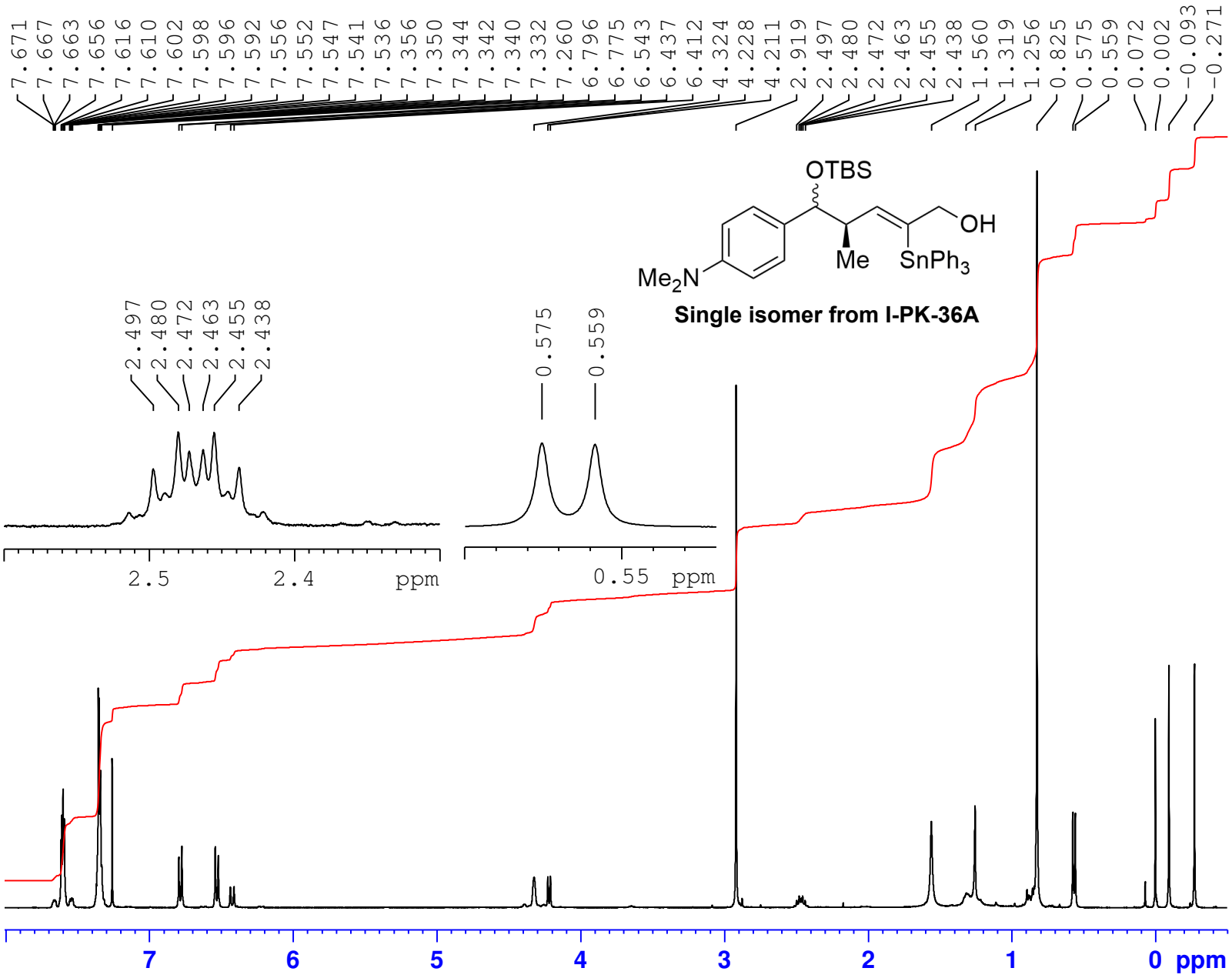
Single isomer from I-PK-36A

```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      3.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                        203
DW                       41.600 usec
DE                        9.85 usec
TE                       296.5 K
D1                   0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000098 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

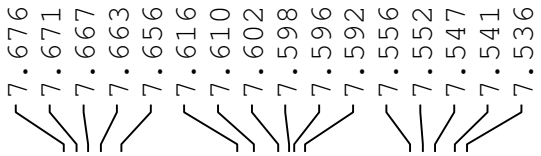


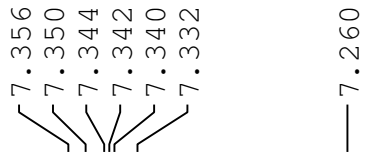

```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       10
PROCNO                       1

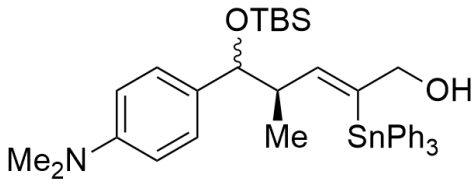
F2 - Acquisition Parameters
Date_                 20180120
Time                      3.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                        203
DW                     41.600 usec
DE                      9.85 usec
TE                      296.5 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000098 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```







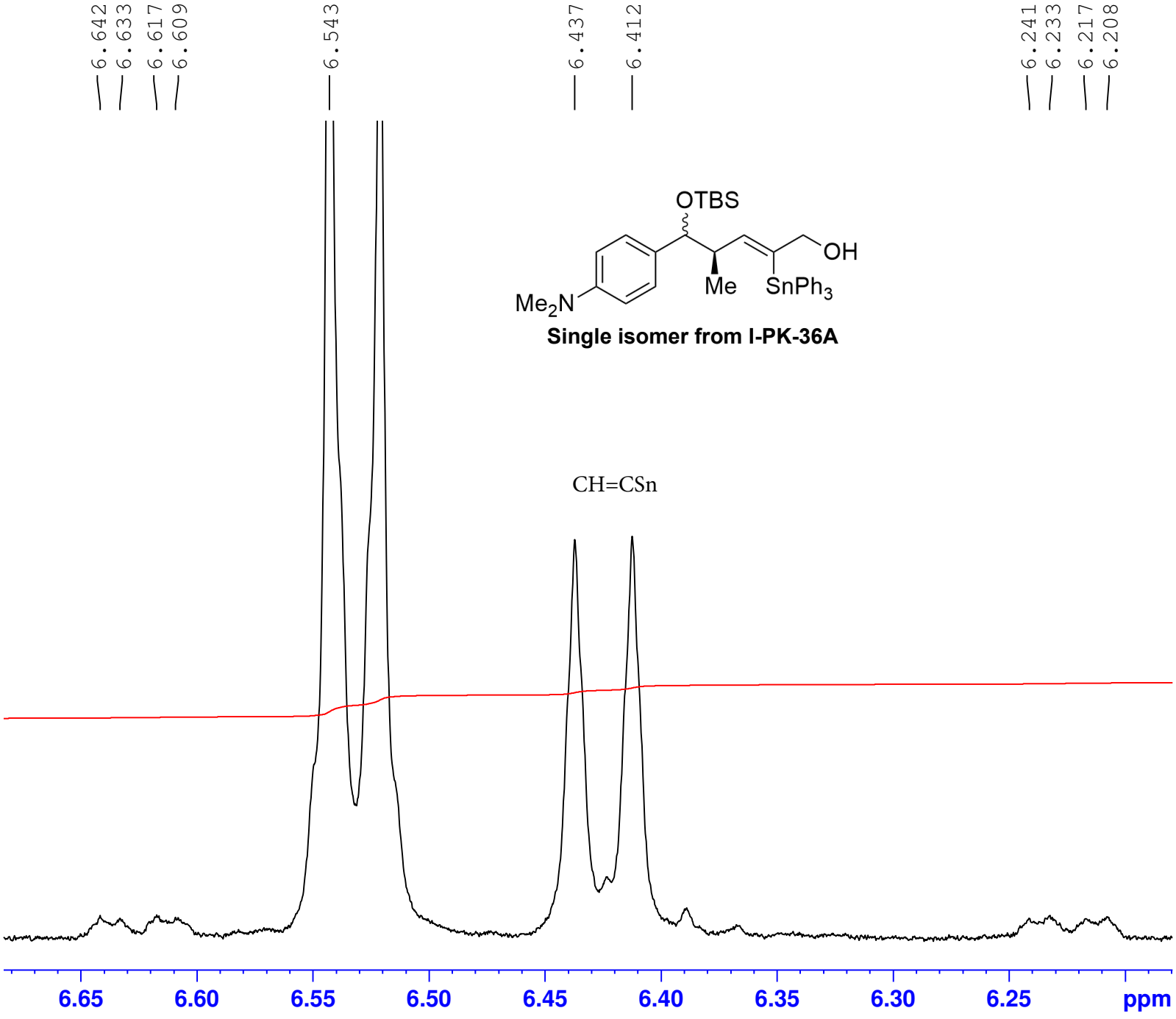
Single isomer from I-PK-36A


```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      3.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                  5.4525952 sec
RG                        203
DW                     41.600 usec
DE                      9.85 usec
TE                     296.5 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000098 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



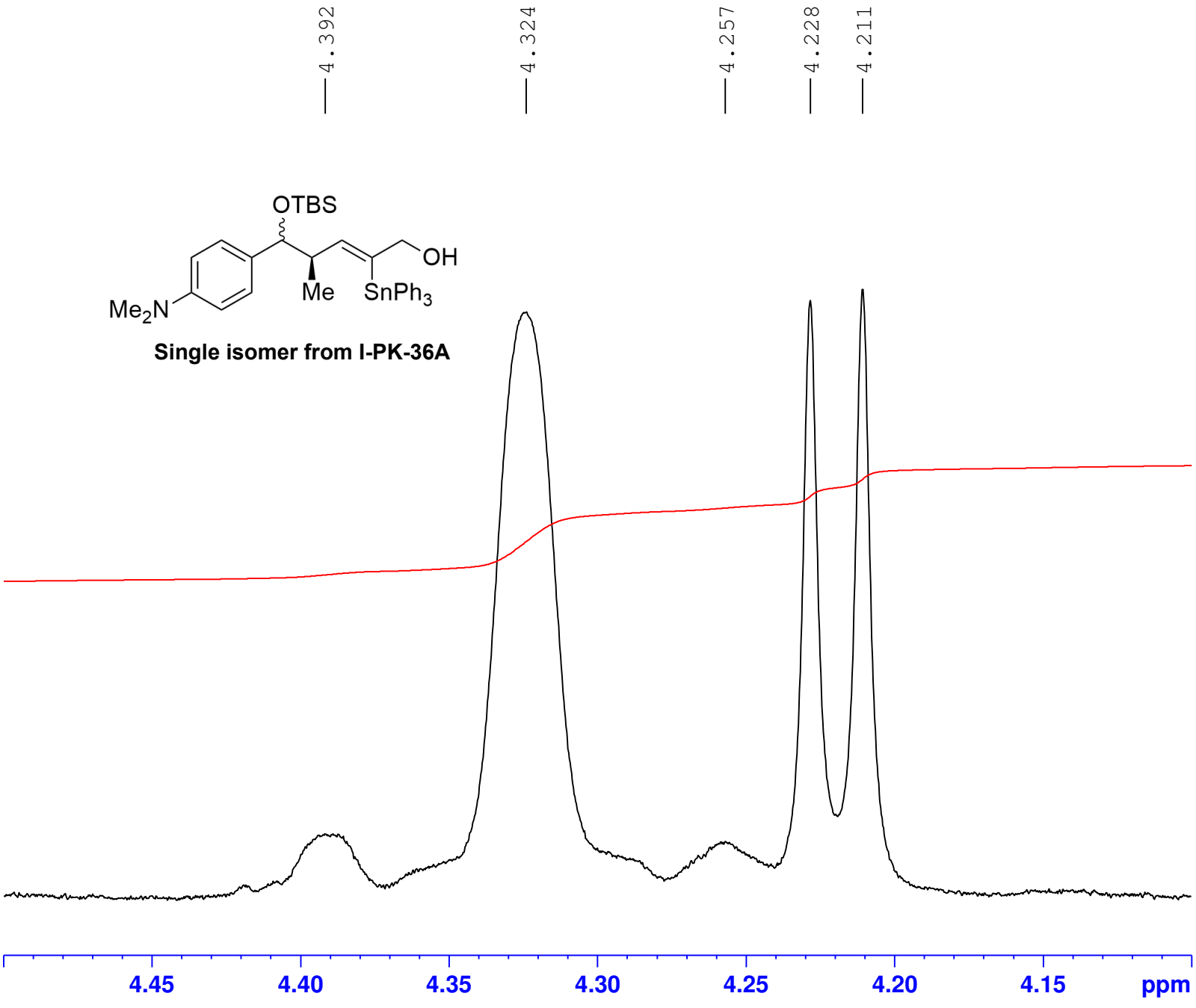
Single isomer from I-PK-36A

```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      3.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                        203
DW                     41.600 usec
DE                      9.85 usec
TE                     296.5 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                     131072
SF               399.9000098 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

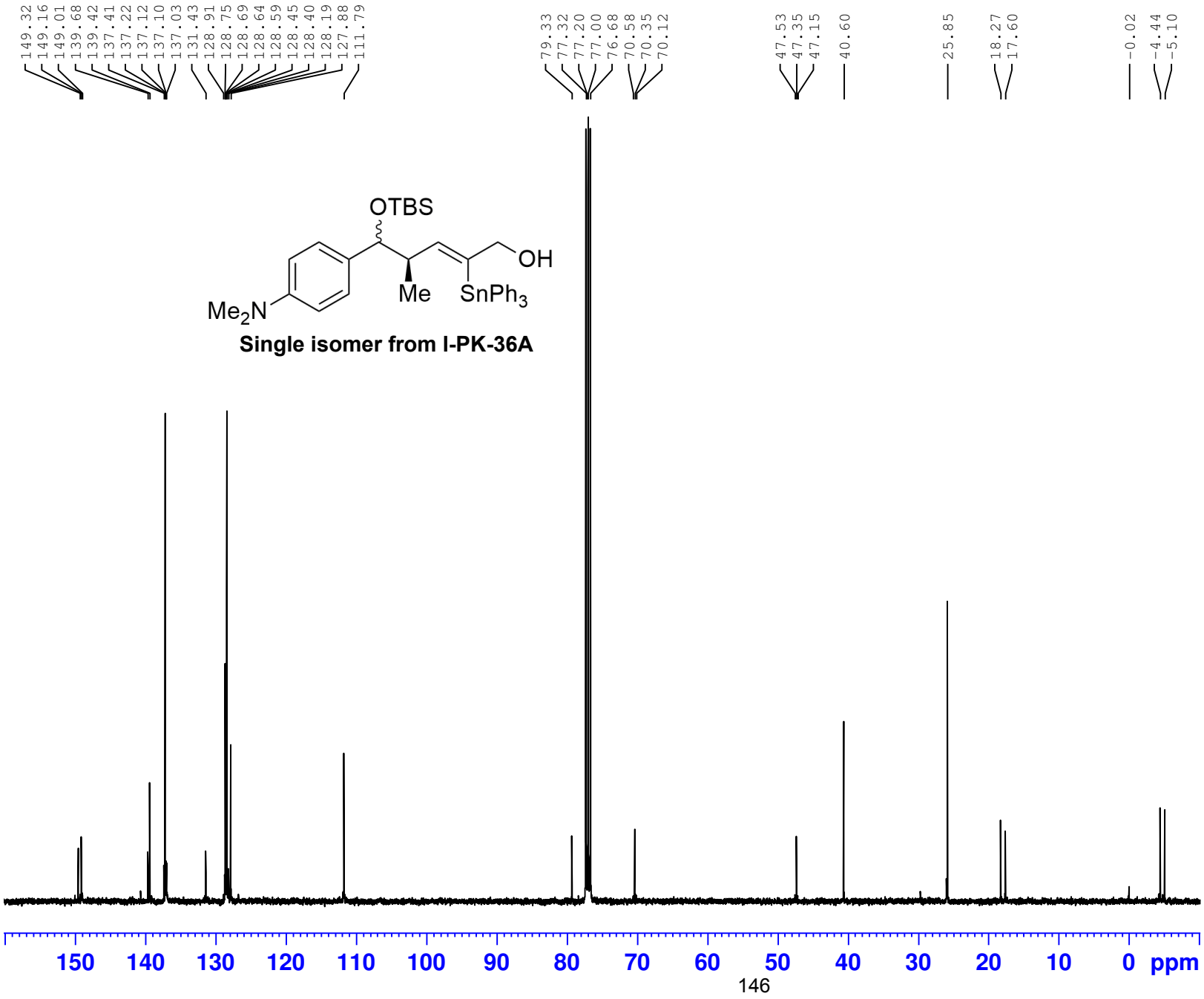



```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      3.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                        203
DW                     41.600 usec
DE                      9.85 usec
TE                     296.5 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000098 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



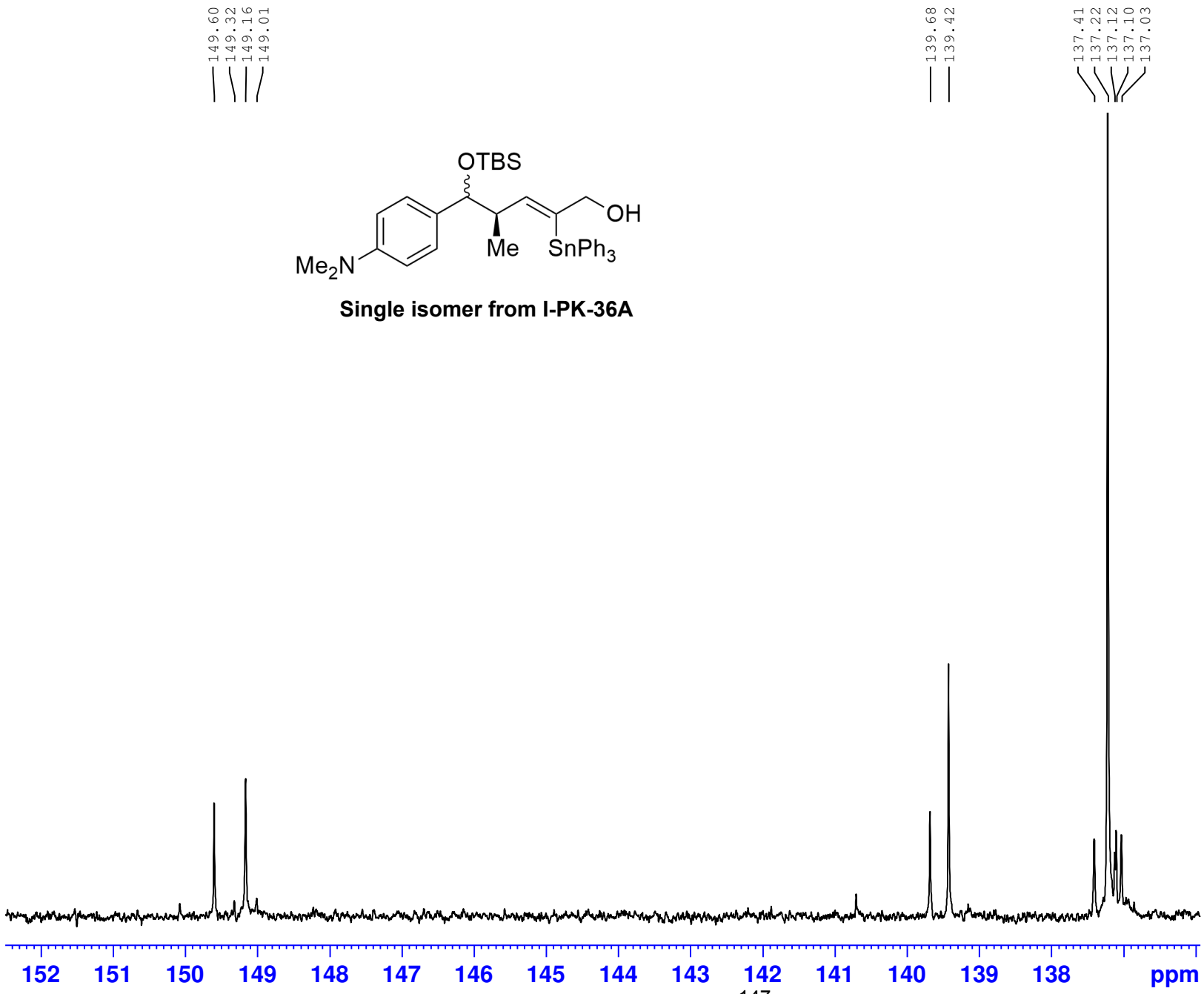
Current Data Parameters
NAME I-PK-37A
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180120
Time 8.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 297.7 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549388 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Single isomer from I-PK-36A

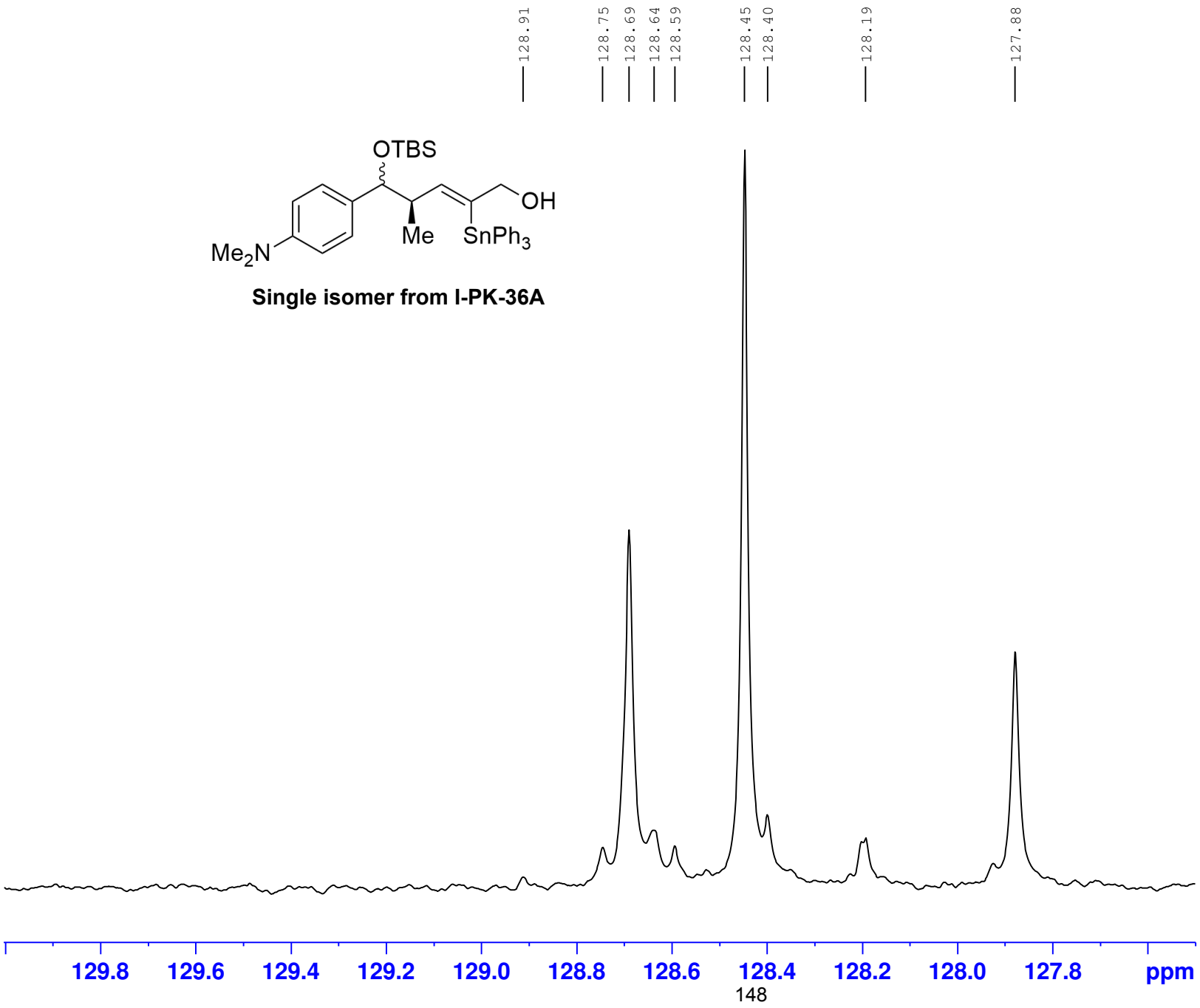
```
Current Data Parameters
NAME                  I-PK-37A
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      8.09
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                      119044
SOLVENT                   CDCl3
NS                         1200
DS                            4
SWH                 25000.000 Hz
FIDRES                0.210006 Hz
AQ                    2.3808801 sec
RG                         2050
DW                    20.000 usec
DE                     9.12 usec
TE                     297.7 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               100.5659947 MHz
NUC1                       13C
P1                      10.00 usec
PLW1               44.46300125 W

======== CHANNEL f2 ========
SFO2               399.9015996 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                    90.00 usec
PLW2                7.59999990 W
PLW12               0.20774999 W
PLW13               0.16827001 W

F2 - Processing parameters
SI                        131072
SF                 100.5549388 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```



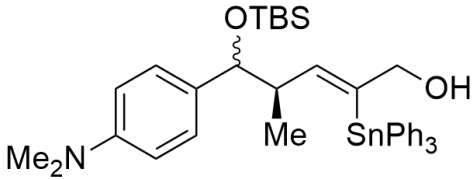

```
Current Data Parameters
NAME            I-PK-37A
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180120
Time                8.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH           25000.000 Hz
FIDRES         0.210006 Hz
AQ             2.3808801 sec
RG                  2050
DW                20.000 usec
DE                 9.12 usec
TE                297.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

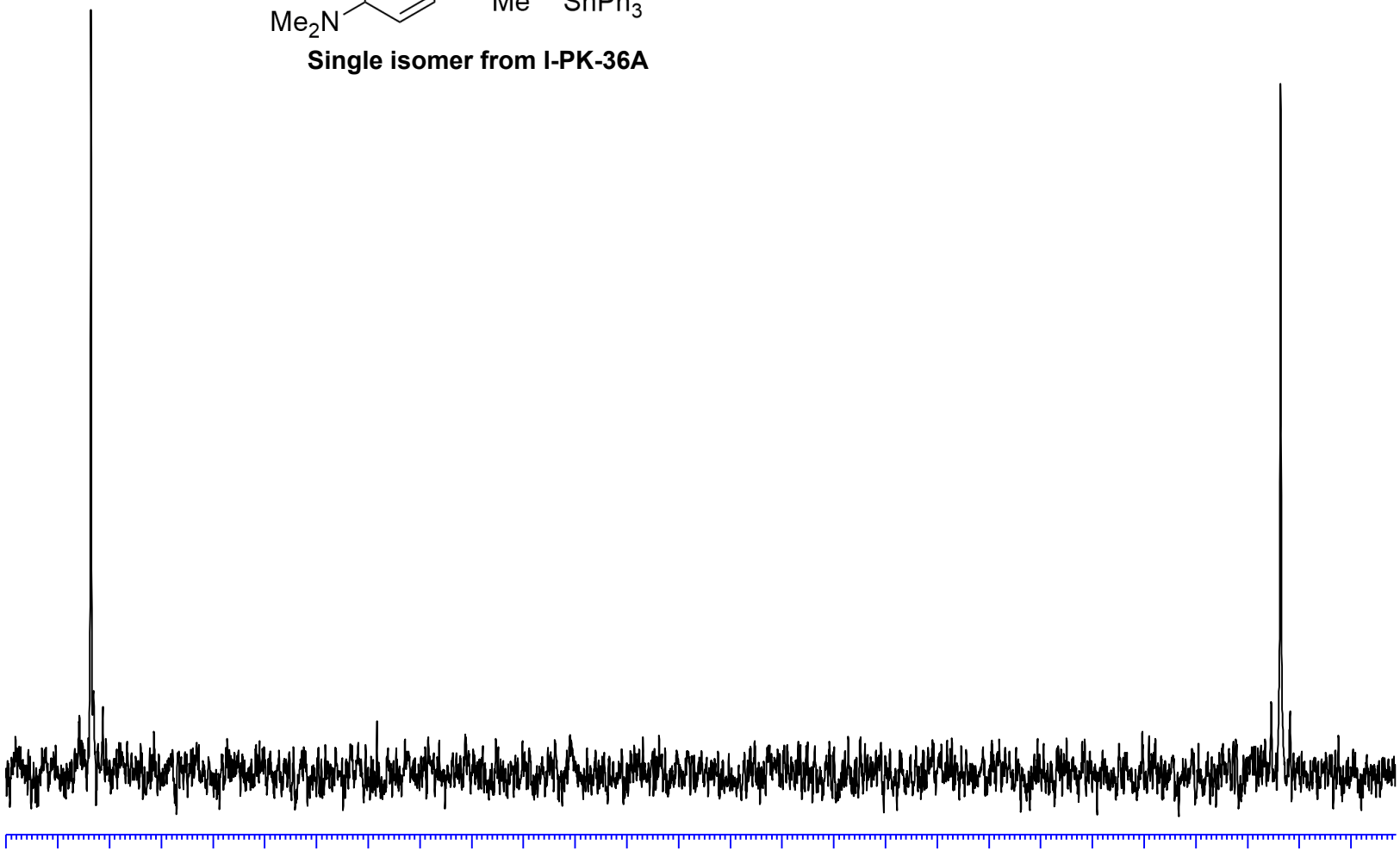
======== CHANNEL f1 ========
SFO1         100.5659947 MHz
NUC1                13C
P1                10.00 usec
PLW1         44.46300125 W

======== CHANNEL f2 ========
SFO2         399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2          7.59999990 W
PLW12         0.20774999 W
PLW13         0.16827001 W

F2 - Processing parameters
SI                131072
SF           100.5549388 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



Single isomer from I-PK-36A



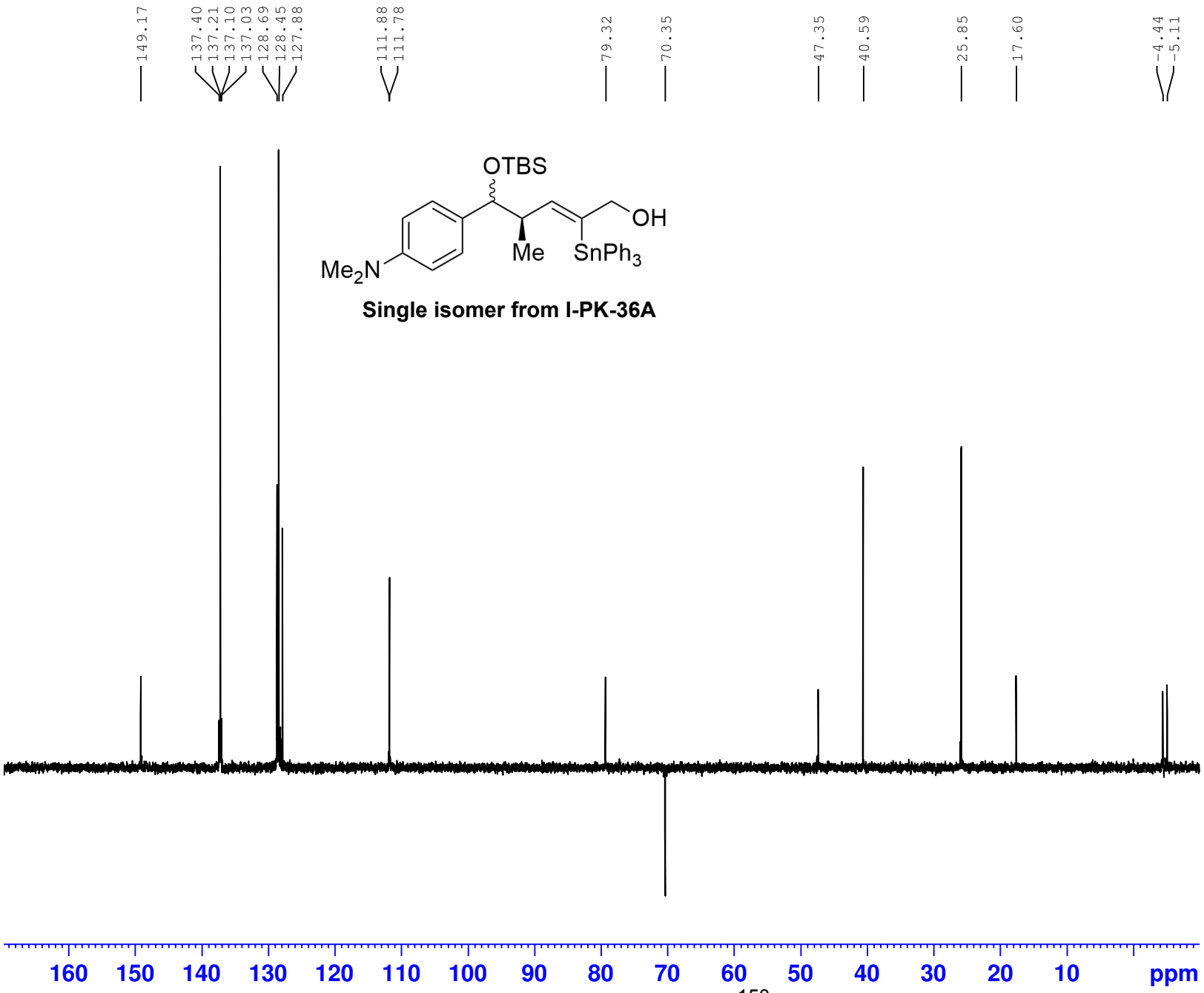

```
Current Data Parameters
NAME            I-PK-37A
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180120
Time                8.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH           25000.000 Hz
FIDRES         0.210006 Hz
AQ            2.3808801 sec
RG                  2050
DW               20.000 usec
DE                9.12 usec
TE                297.7 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.5659947 MHz
NUC1                13C
P1                10.00 usec
PLW1        44.46300125 W

======== CHANNEL f2 ========
SFO2        399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2          7.59999990 W
PLW12         0.20774999 W
PLW13         0.16827001 W

F2 - Processing parameters
SI                131072
SF          100.5549388 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



Current Data Parameters

NAME	I-PK-37A
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20180120
Time	8.26
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	297.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

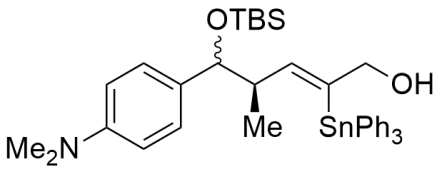
SFO1	100.5649905 MHz
NUC1	13C
P1	10.00 usec
P13	2000.00 usec
PLW0	0 W
PLW1	44.46300125 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	6.79339981 W

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

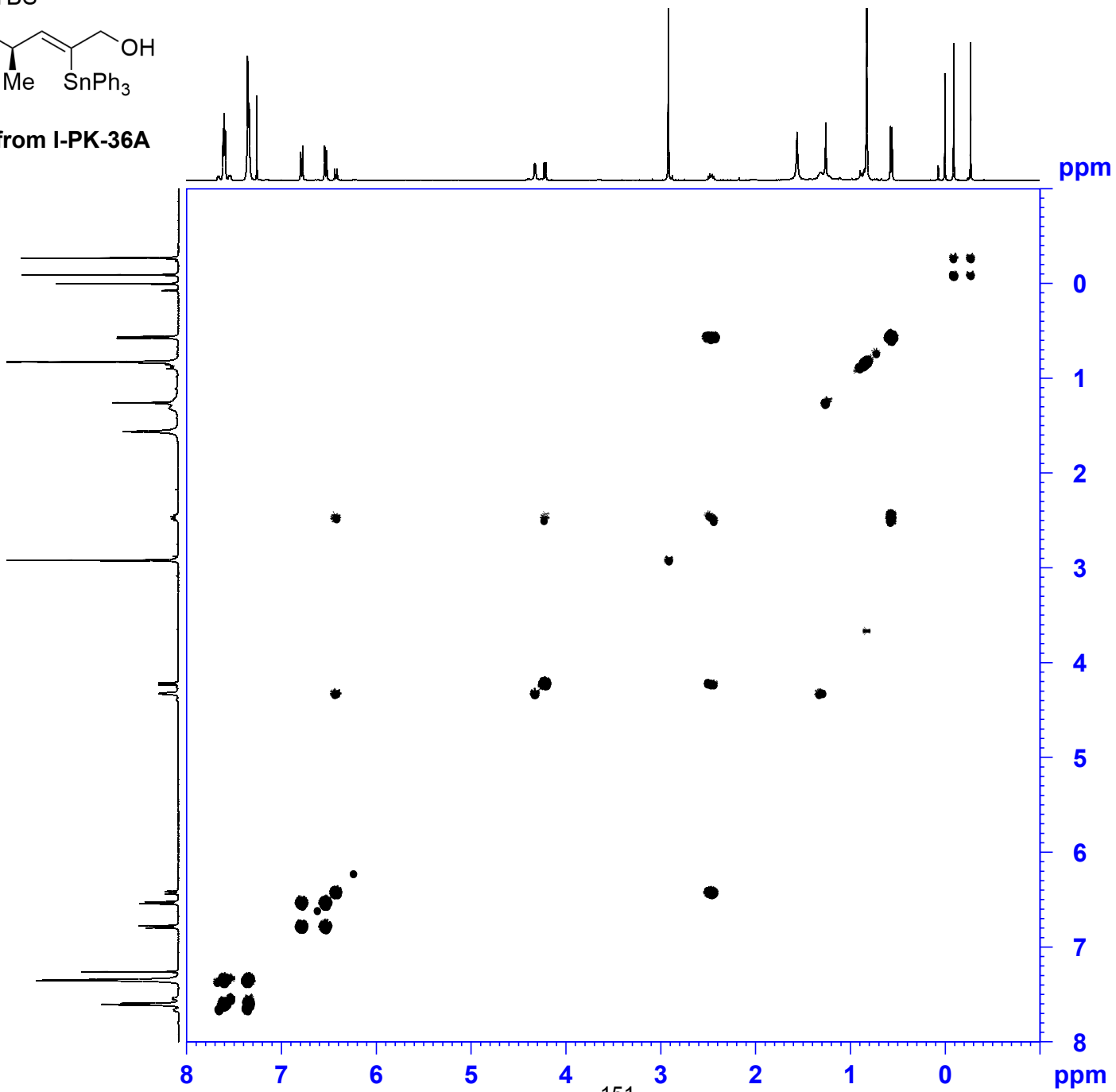
SFO2	399.9012789 MHz
NUC2	1H
CPDPRG[2	waltz16
P3	14.88 usec
P4	29.76 usec
PCPD2	90.00 usec
PLW2	7.59999990 W
PLW12	0.20774999 W

F2 - Processing parameters

SI	32768
SF	100.5549389 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



Single isomer from I-PK-36A




```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      5.23
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                        2048
SOLVENT                 CDCl3
NS                           1
DS                           8
SWH                3795.547 Hz
FIDRES             1.853294 Hz
AQ                 0.2697899 sec
RG                        2050
DW                  131.733 usec
DE                    6.50 usec
TE                    296.7 K
D0                 0.00000300 sec
D1                 0.87629992 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00026340 sec

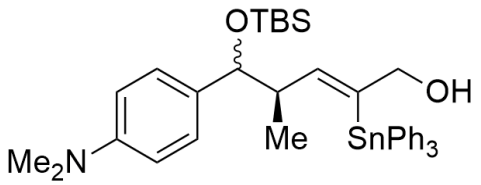
======== CHANNEL f1 ========
SFO1             399.9014817 MHz
NUC1                       1H
P1                      14.88 usec
P17                   2500.00 usec
PLW1               7.59999990 W
PLW10              2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPNAM[3]          SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

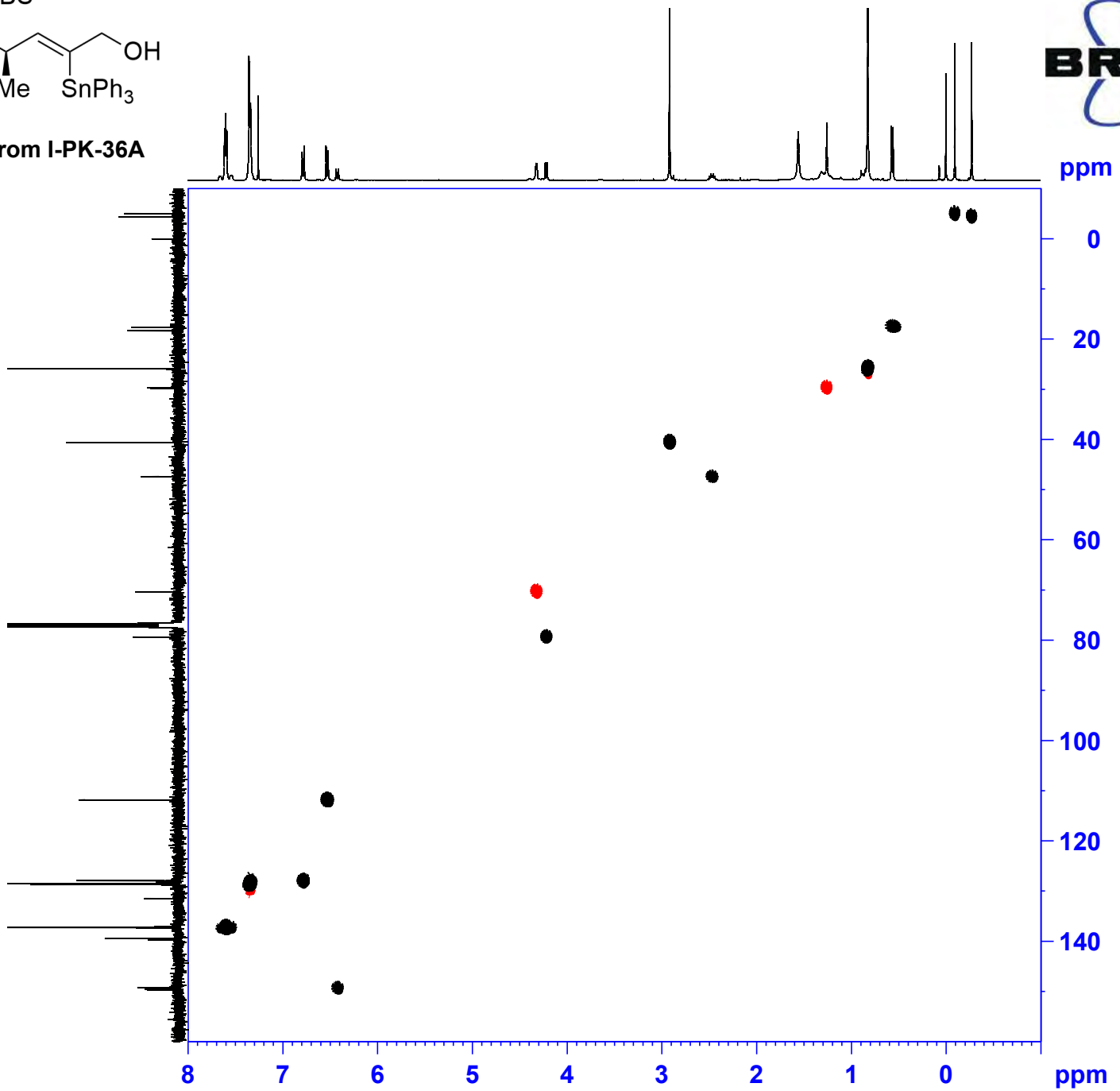
F1 - Acquisition parameters
TD                         256
SFO1                399.9015 MHz
FIDRES             29.660213 Hz
SW                     9.494 ppm
FnMODE                    QF

F2 - Processing parameters
SI                        1024
SF                399.9000090 MHz
WDW                       SINE
SSB           0
LB            0 Hz
GB            0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                         QF
SF                399.9000111 MHz
WDW                       SINE
SSB           0
LB            0 Hz
GB            0
```



Single isomer from I-PK-36A




```
Current Data Parameters
NAME                  I-PK-37B
EXPNO                       15
PROCNO                       1

F2 - Acquisition Parameters
Date_               20180120
Time                    6.39
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                      1024
SOLVENT                 CDCl3
NS                         2
DS                        32
SWH                 4807.692 Hz
FIDRES              4.695012 Hz
AQ                  0.1064960 sec
RG                       2050
DW                    104.000 usec
DE                      6.50 usec
TE                     296.5 K
CNST2              145.0000000
D0                  0.00000300 sec
D1                  0.80000001 sec
D4                  0.00172414 sec
D11                 0.03000000 sec
D16                 0.00020000 sec
D21                 0.00360000 sec
IN0                 0.00001910 sec

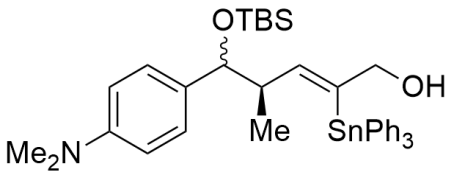
======== CHANNEL f1 ========
SFO1               399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28          0 usec
PLW1                7.59999990 W

======== CHANNEL f2 ========
SFO2               100.5670016 MHz
NUC2                       13C
CPDPRG[2                garp4
P3                      10.00 usec
P14                    500.00 usec
P31                   1900.00 usec
PCPD2                   80.00 usec
PLW0          0 W
PLW2               44.46300125 W
PLW12               0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                  0.500
SPOFFS3  0 Hz
SPW3                6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                 0.500
SPOFFS18 0 Hz
SPW18               1.62779999 W

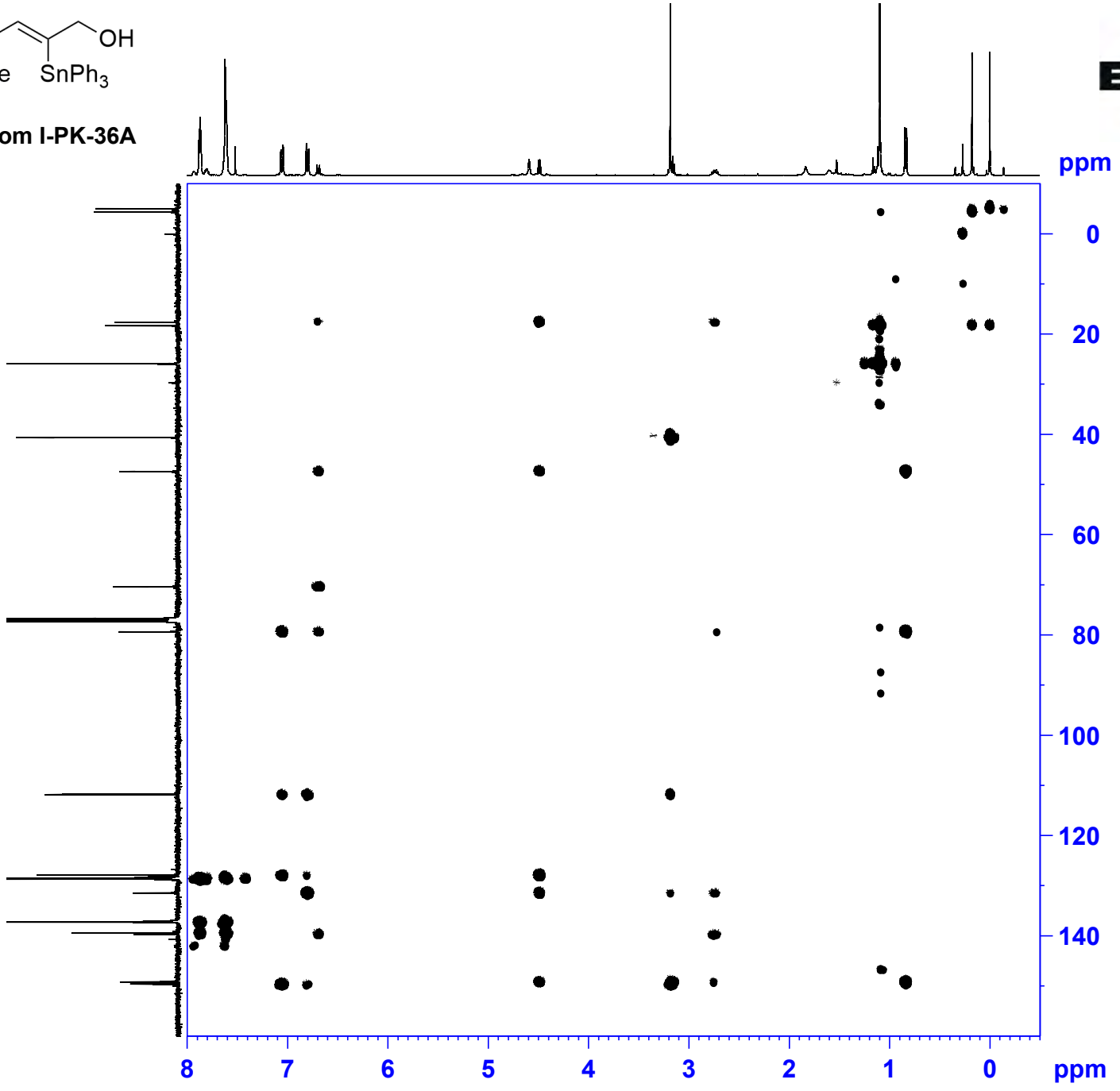
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                   80.00 %
GPZ2                   20.10 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                 100.567 MHz
FIDRES              204.515701 Hz
SW                   260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      1024
SF                 399.9000081 MHz
WDW                     OSINE
```



Single isomer from I-PK-36A




```
Current Data Parameters
NAME                  I-PK-37A
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180120
Time                      8.35
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     hmbcetgp13nd
TD                        4096
SOLVENT                 CDCl3
NS                           4
DS                          16
SWH                 5208.333 Hz
FIDRES              1.271566 Hz
AQ                  0.3932160 sec
RG                       2050
DW                     96.000 usec
DE                      6.50 usec
TE                     296.6 K
CNST6              120.0000000
CNST7              175.0000000
CNST13               8.0000000
CNST30               0.5981122
D0                   0.00000300 sec
D1                   1.50000000 sec
D6                   0.06250000 sec
D16                  0.00020000 sec
IN0                  0.00002070 sec

======== CHANNEL f1 ========
SFO1               399.9019995 MHz
NUC1                       1H
P1                       14.88 usec
P2                       29.76 usec
PLW1                7.59999990 W

======== CHANNEL f2 ========
SFO2               100.5659947 MHz
NUC2                       13C
P3                       10.00 usec
P24                    2000.00 usec
PLW2               44.46300125 W
SPNAM[7]     Crp60comp.4
SPOAL7                   0.500
SPOFFS7   0 Hz
SPW7                6.79339981 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1                    80.00 %
GPZ3                    14.00 %
GPZ4                    -8.00 %
GPZ5                    -4.00 %
GPZ6                    -2.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         512
SFO1                100.566 MHz
FIDRES             94.353867 Hz
SW                    240.186 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                        4096
SF                 399.8999052 MHz
WDW                      QSINE
SSB         0
LB          0 Hz
GB          0
PC                        1.40
```

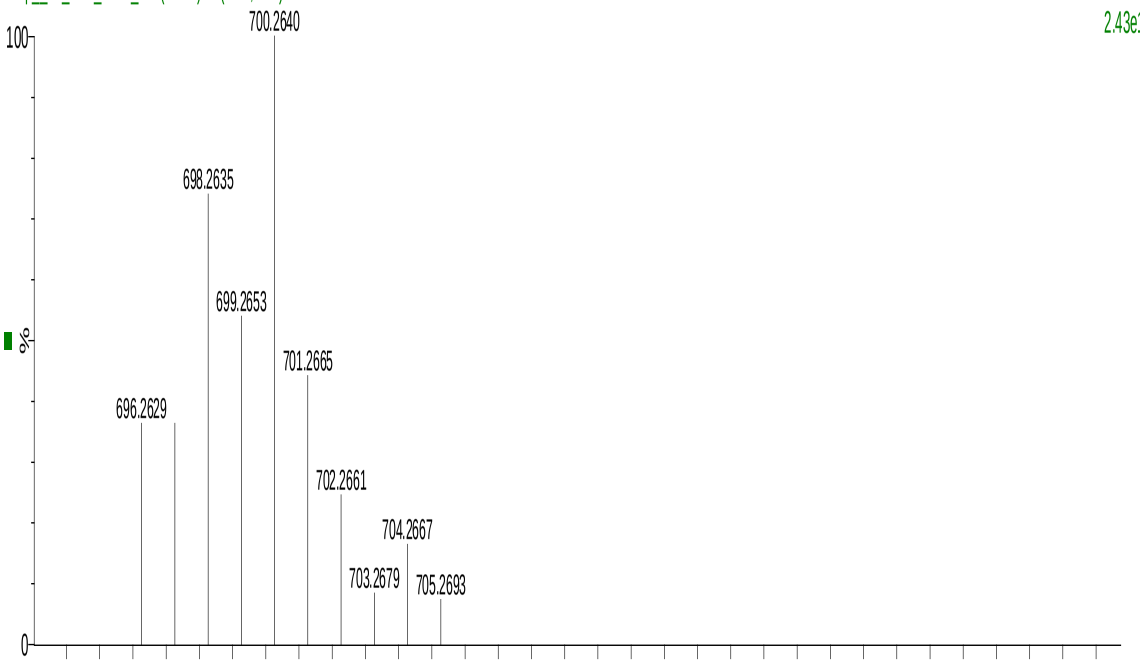
22-01-2018

I-PK-37

asep__10_JAN_2018_63 (0.070) Is (1.00,1.00) C38H49NSiSnO2H

1: TOF MS ES+

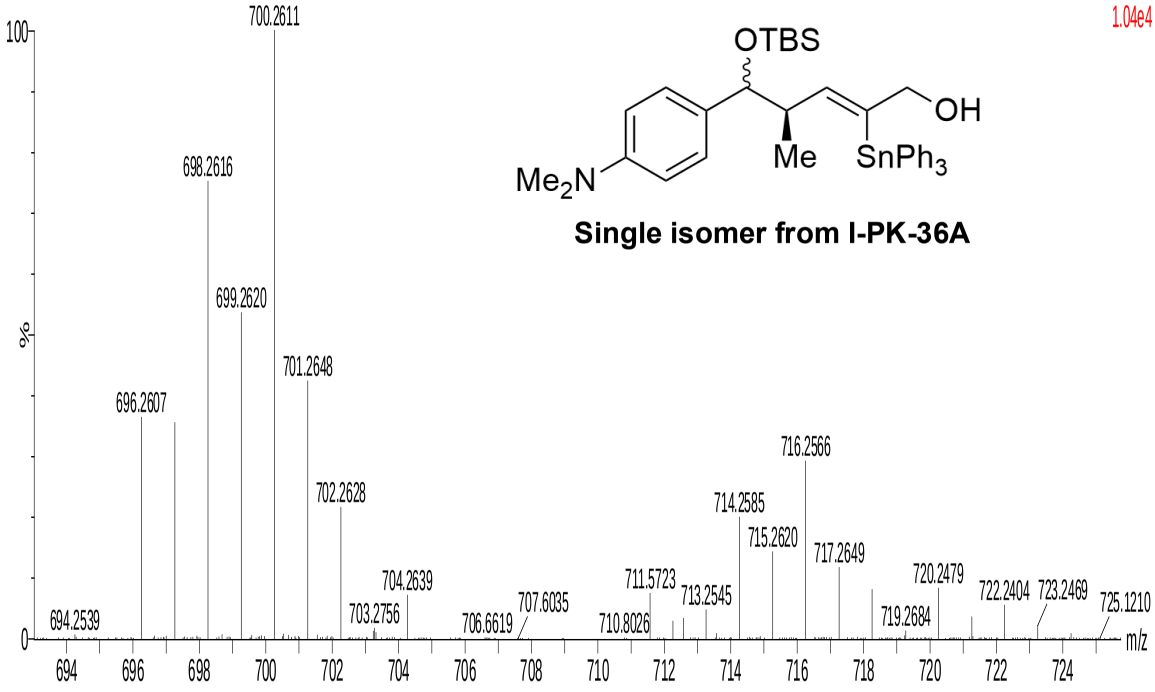
2.43e12



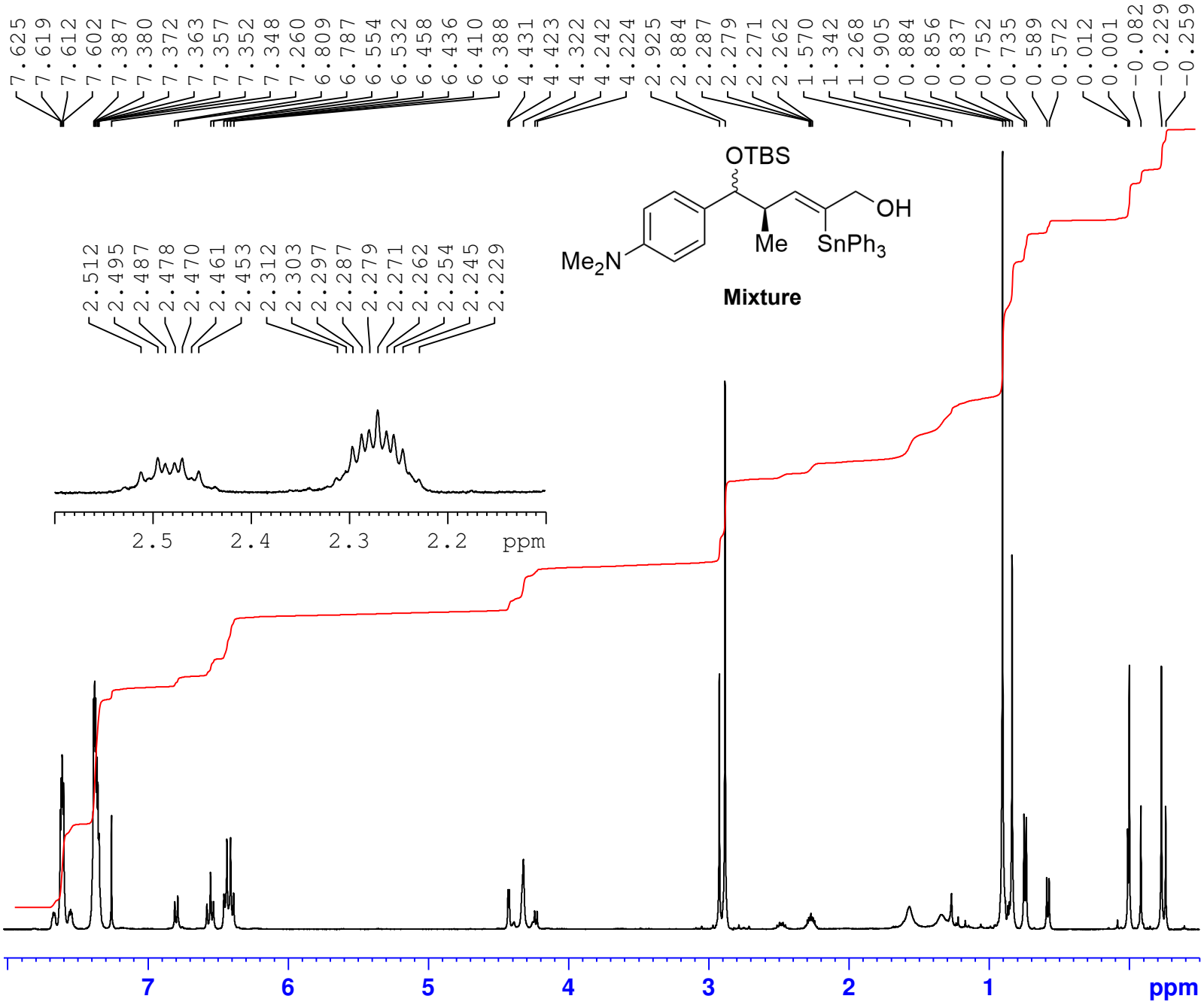
asep__10_JAN_2018_63 2 (0.104) Cm (2:7)

1: TOF MS ES+

1.04e4



Single isomer from I-PK-36A



155

Current Data Parameters

NAME	I-PK-282
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

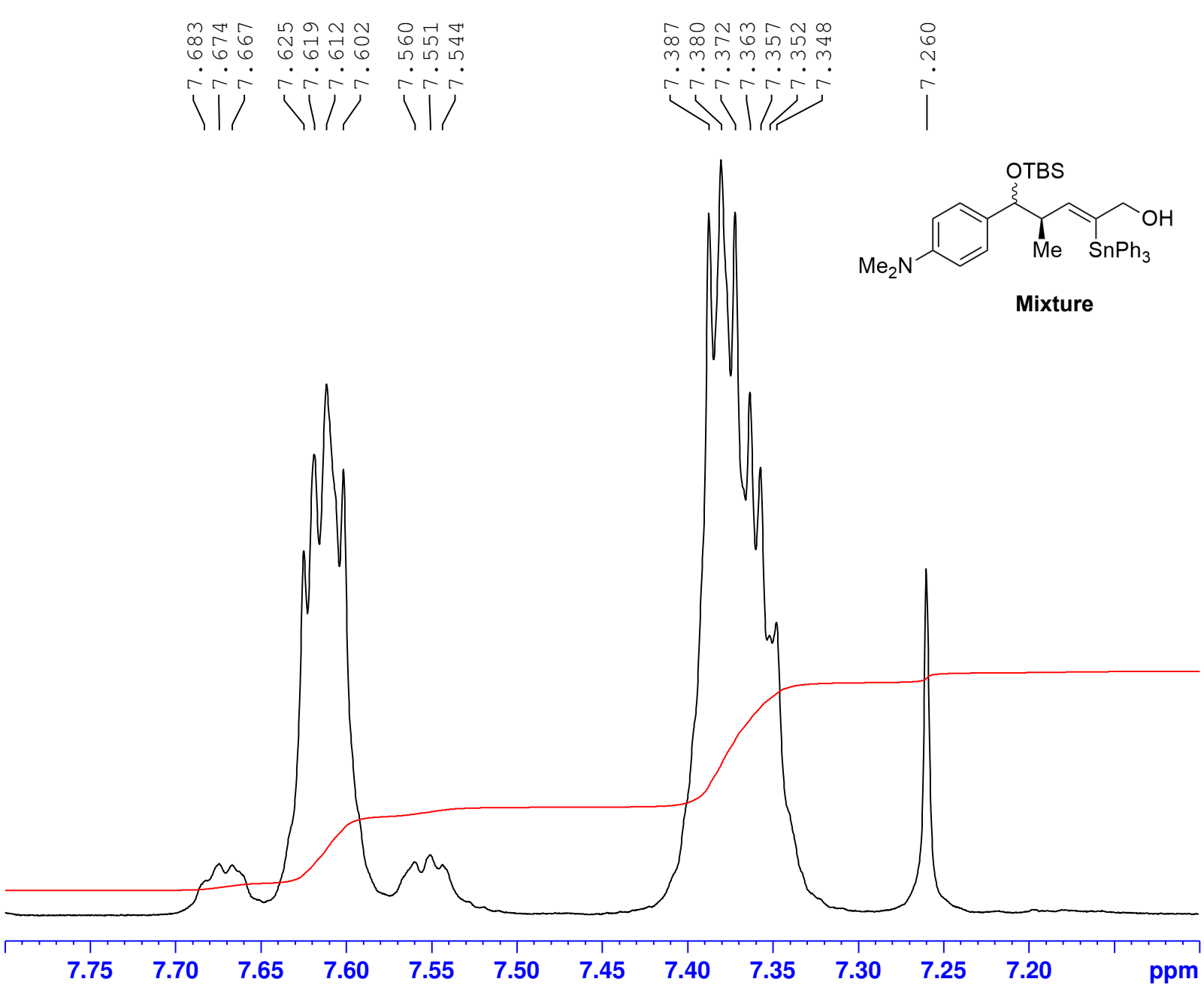
Date_	20190524
Time	16.50
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.091699 Hz
AQ	5.4525952 sec
RG	71.8
DW	41.600 usec
DE	9.85 usec
TE	300.0 K
D1	0.10000000 sec
TD0	1

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

SFO1	399.9024695 MHz
NUC1	1H
P1	14.88 usec
PLW1	7.59999990 W

F2 - Processing parameters

SI	131072
SF	399.9000096 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

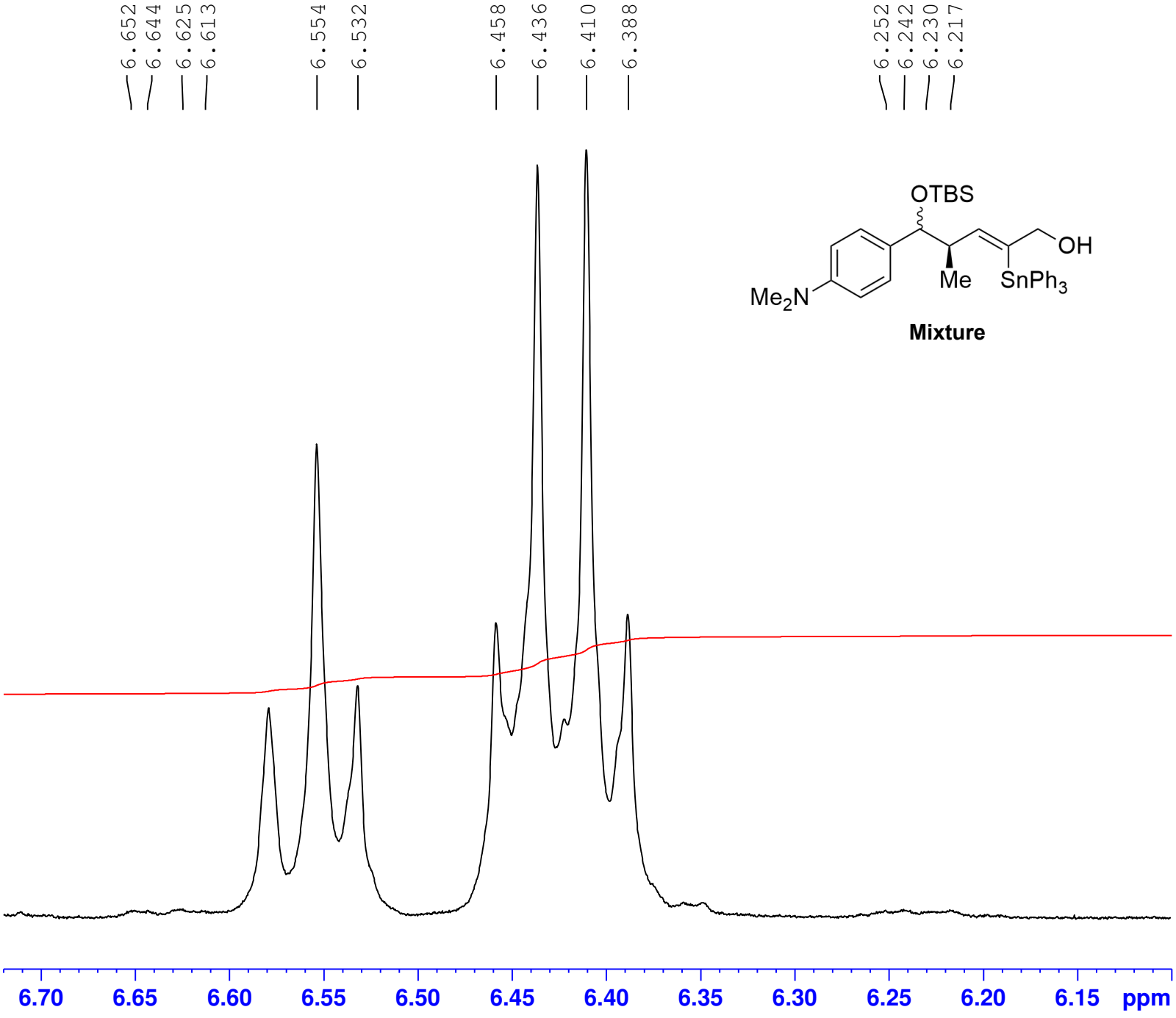



```
Current Data Parameters
NAME                  I-PK-282
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190524
Time                     16.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                  0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

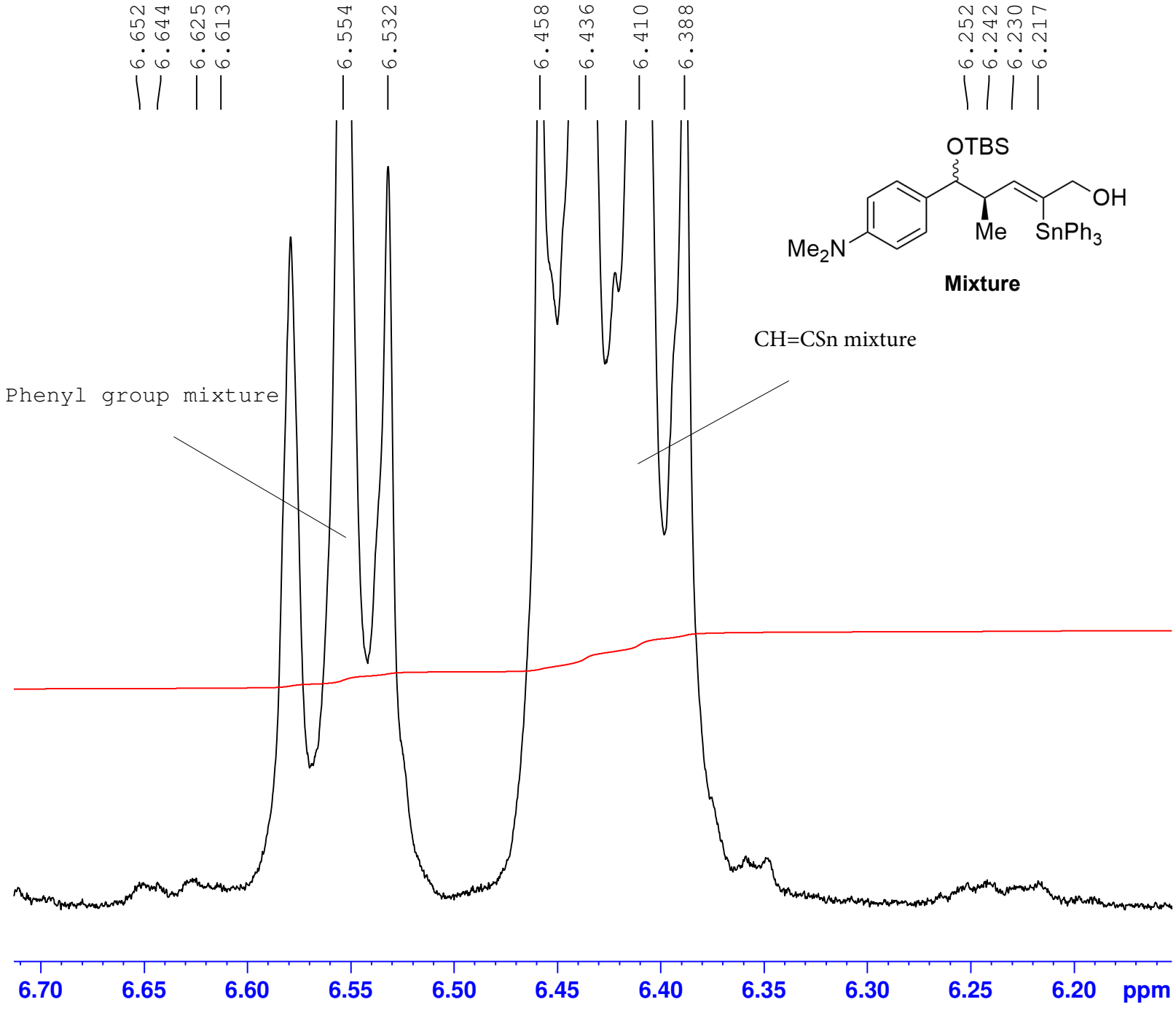



```
Current Data Parameters
NAME                  I-PK-282
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190524
Time                     16.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                        41.600 usec
DE                         9.85 usec
TE                        300.0 K
D1                   0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                        14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



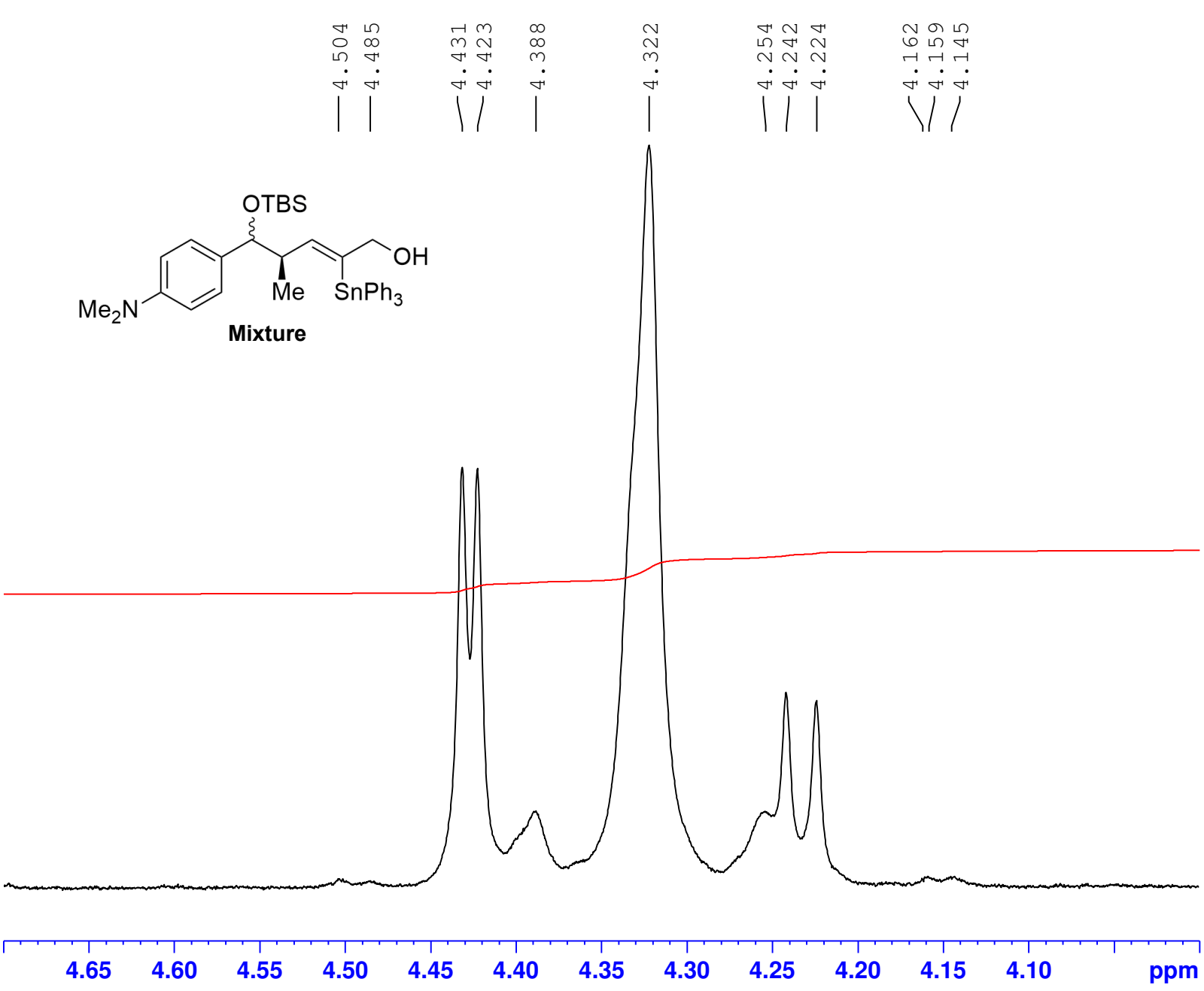
Mixture

```
Current Data Parameters
NAME                  I-PK-282
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190524
Time                     16.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                   0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

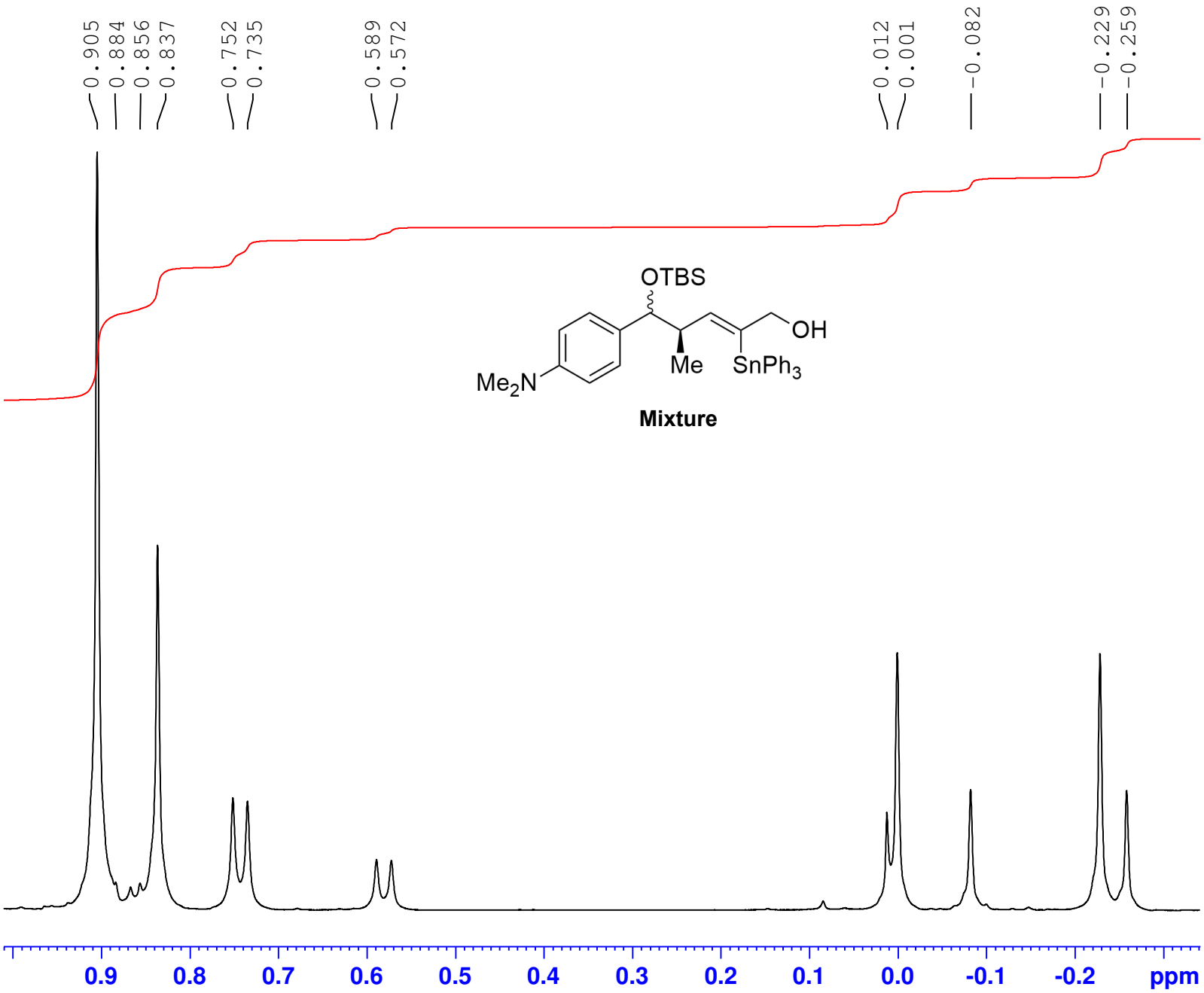



```
Current Data Parameters
NAME                  I-PK-282
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190524
Time                     16.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         71.8
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                   0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

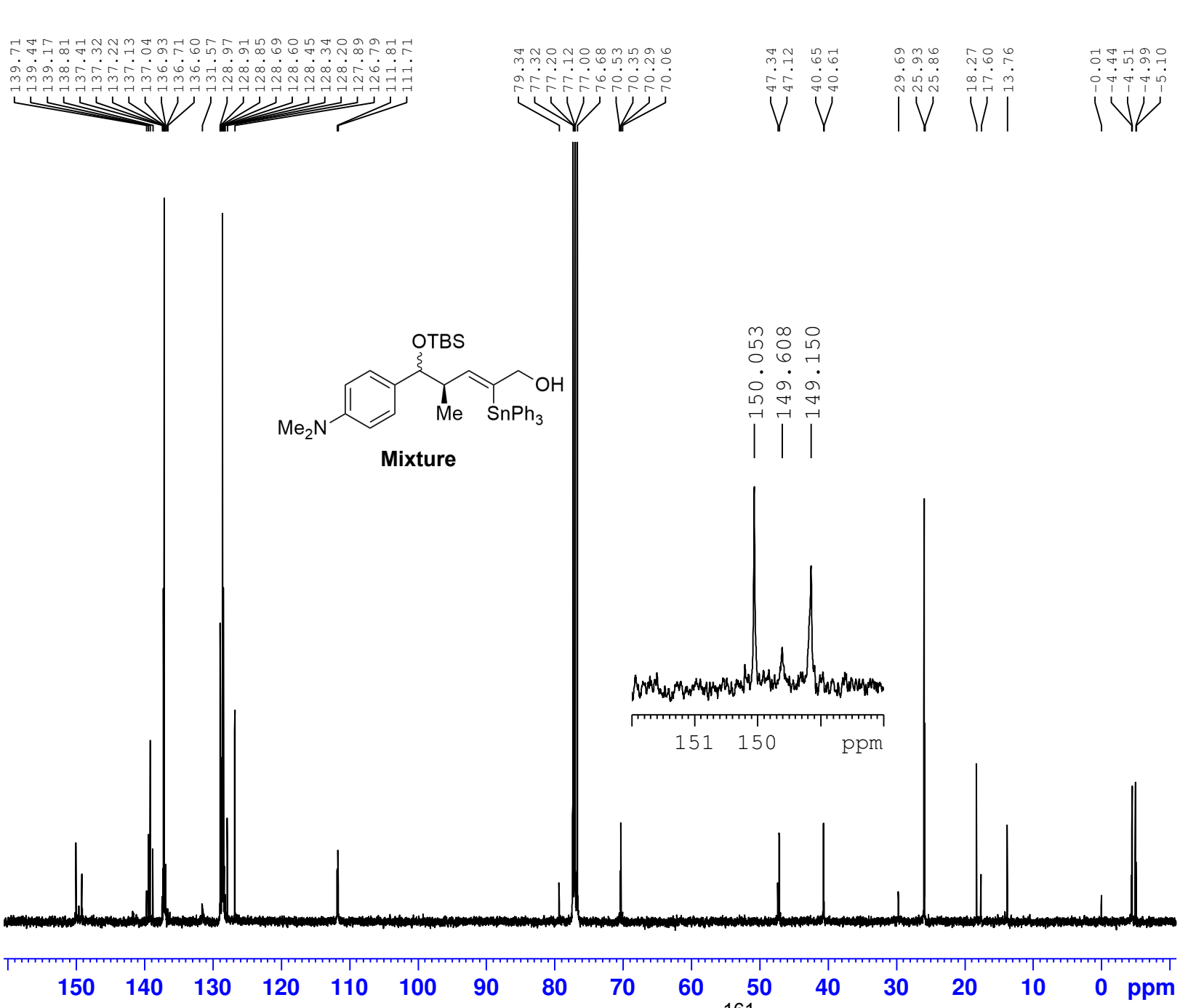



```
Current Data Parameters
NAME                  I-PK-282
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190524
Time                     16.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                        71.8
DW                    41.600 usec
DE                     9.85 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                     131072
SF               399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



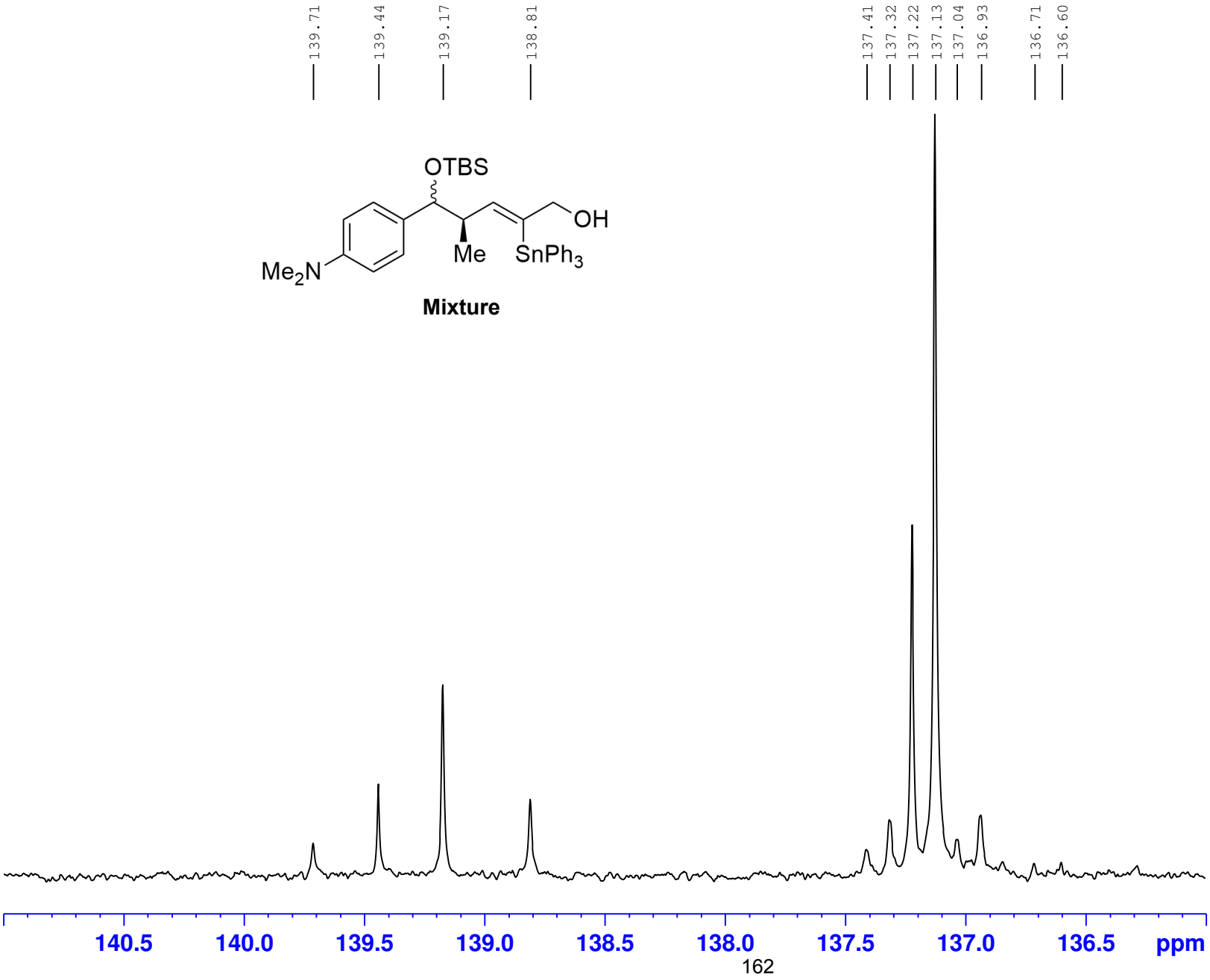
Current Data Parameters
NAME I-PK-282REPURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190524
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549384 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



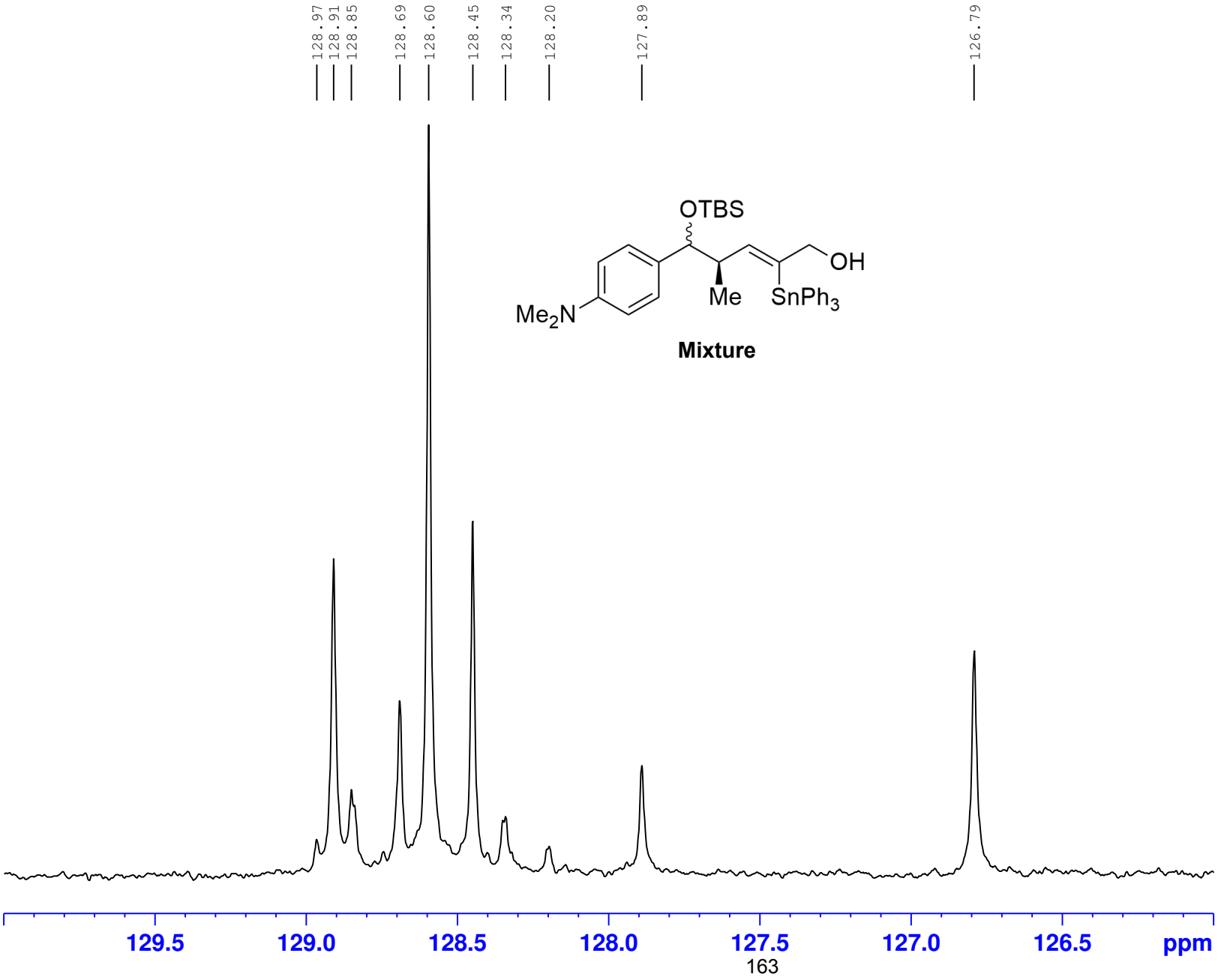
Current Data Parameters
NAME I-PK-282REPURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190524
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549384 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



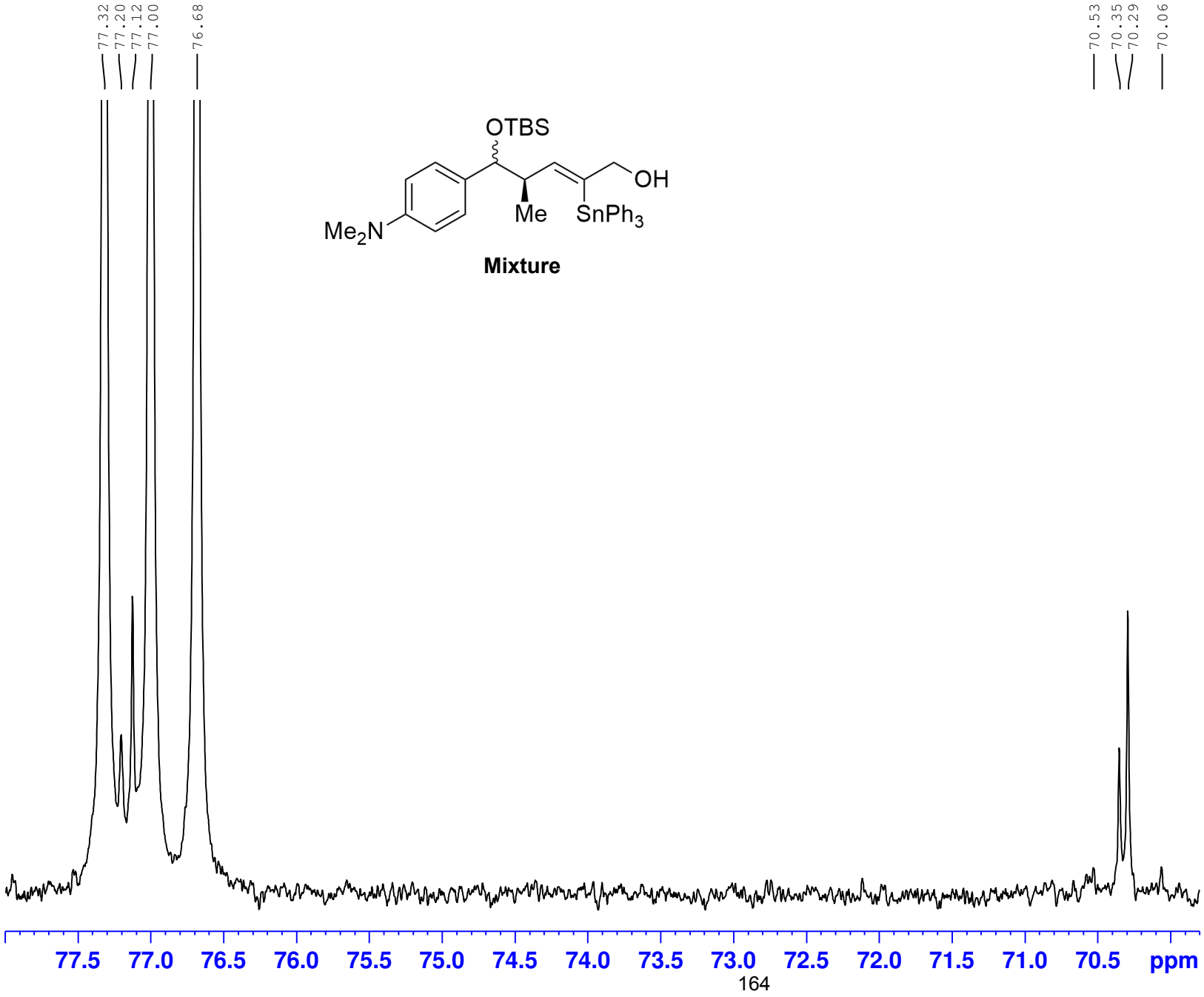
Current Data Parameters
NAME I-PK-282REPURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190524
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549384 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Mixture

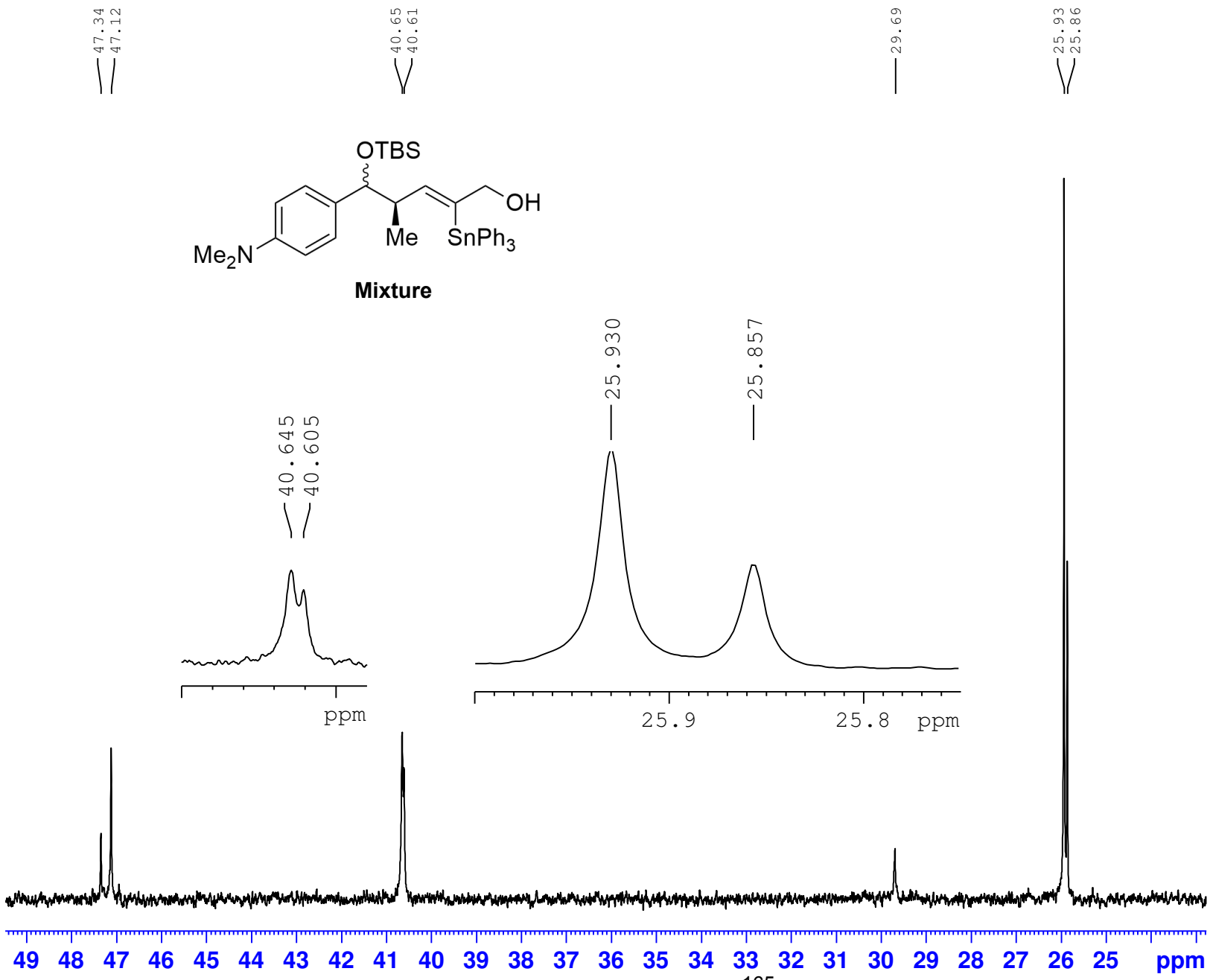

```
Current Data Parameters
NAME      I-PK-282REPURE
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190524
Time               19.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH           25000.000 Hz
FIDRES         0.210006 Hz
AQ             2.3808801 sec
RG                  2050
DW                20.000 usec
DE                 9.12 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.5659947 MHz
NUC1                13C
P1                10.00 usec
PLW1        44.46300125 W

======== CHANNEL f2 ========
SFO2        399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2          7.59999990 W
PLW12         0.20774999 W
PLW13         0.16827001 W

F2 - Processing parameters
SI                131072
SF          100.5549384 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



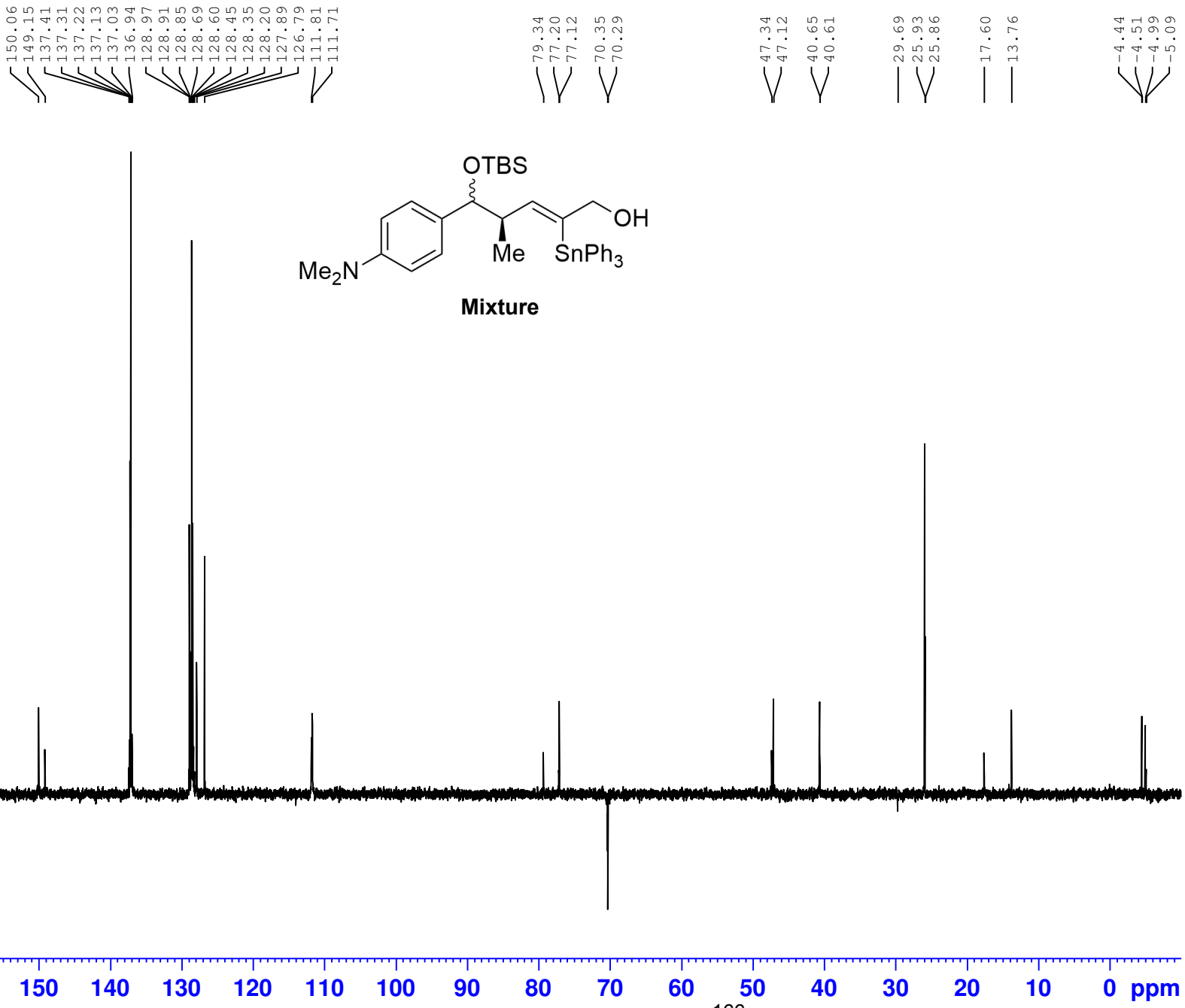

```
Current Data Parameters
NAME      I-PK-282REPURE
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190524
Time               19.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD               119044
SOLVENT            CDCl3
NS                  1200
DS                     4
SWH          25000.000 Hz
FIDRES        0.210006 Hz
AQ           2.3808801 sec
RG                  2050
DW             20.000 usec
DE              9.12 usec
TE              300.0 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.5659947 MHz
NUC1                13C
P1               10.00 usec
PLW1        44.46300125 W

======== CHANNEL f2 ========
SFO2        399.9015996 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         7.59999990 W
PLW12        0.20774999 W
PLW13        0.16827001 W

F2 - Processing parameters
SI                131072
SF          100.5549384 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



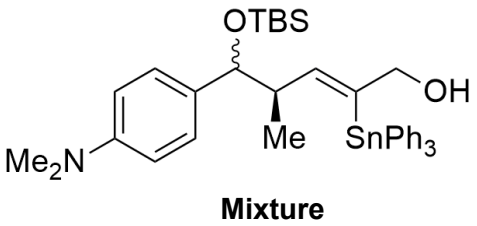
Current Data Parameters
NAME I-PK-282REPURE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190524
Time 19.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1820
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

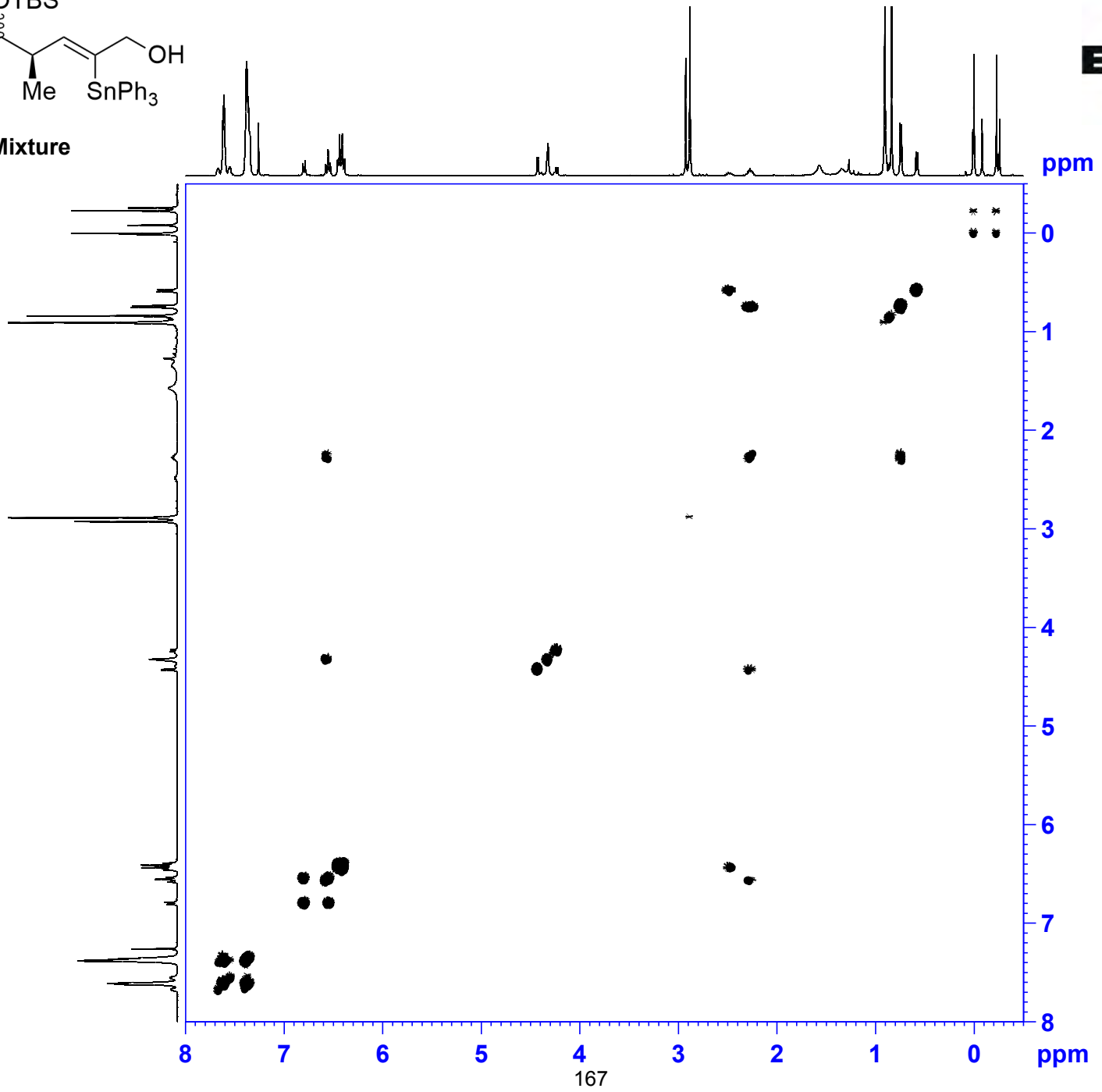
======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549381 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Mixture




```
Current Data Parameters
NAME      I-PK-282REPURE
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190524
Time               19.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDC13
NS                     1
DS                     8
SWH           3770.739 Hz
FIDRES        1.841181 Hz
AQ            0.2715648 sec
RG                  2050
DW             132.600 usec
DE               6.50 usec
TE              300.0 K
D0          0.00000300 sec
D1          0.87425190 sec
D11         0.03000000 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00020000 sec
IN0         0.00026520 sec

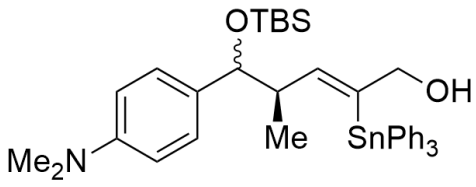
======== CHANNEL f1 ========
SFO1       399.9014964 MHz
NUC1               1H
P1              14.88 usec
P17           2500.00 usec
PLW1        7.59999990 W
PLW10       2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]    SMSQ10.100
GPNAM[2]    SMSQ10.100
GPNAM[3]    SMSQ10.100
GPZ1            16.00 %
GPZ2            12.00 %
GPZ3            40.00 %
P16           1000.00 usec

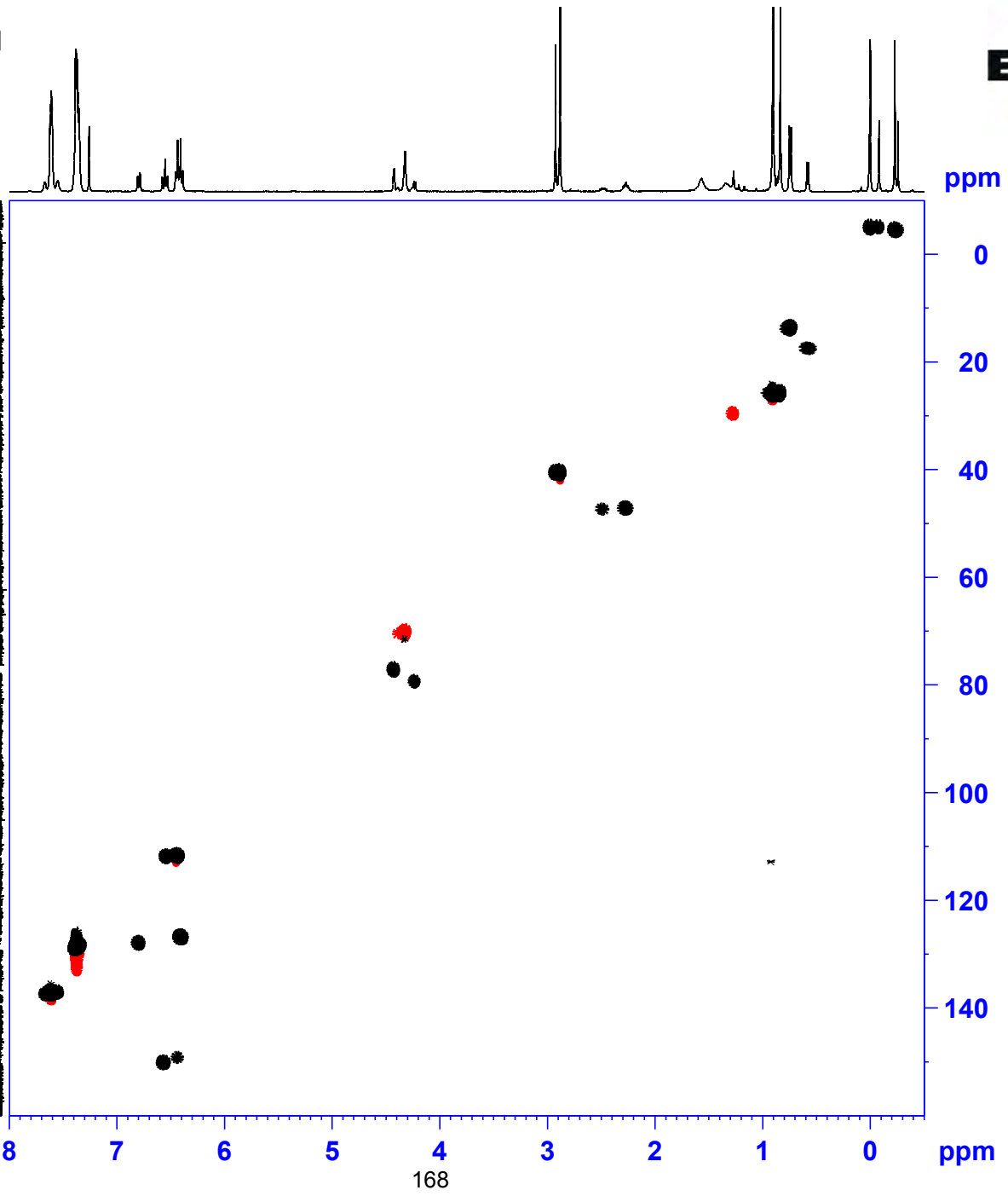
F1 - Acquisition parameters
TD                256
SFO1         399.9015 MHz
FIDRES      29.458899 Hz
SW              9.429 ppm
FnMODE             QF

F2 - Processing parameters
SI                1024
SF         399.9000113 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

F1 - Processing parameters
SI                1024
MC2                QF
SF         399.9000118 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```



Mixture




```
Current Data Parameters
NAME      I-PK-282REPURE
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190524
Time              20.04
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT             CDCl3
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES          4.695012 Hz
AQ             0.1064960 sec
RG                  2050
DW               104.000 usec
DE                 6.50 usec
TE                300.1 K
CNST2        145.0000000
D0             0.00000300 sec
D1             0.80000001 sec
D4             0.00172414 sec
D11            0.03000000 sec
D16            0.00020000 sec
D21            0.00360000 sec
IN0            0.00001910 sec

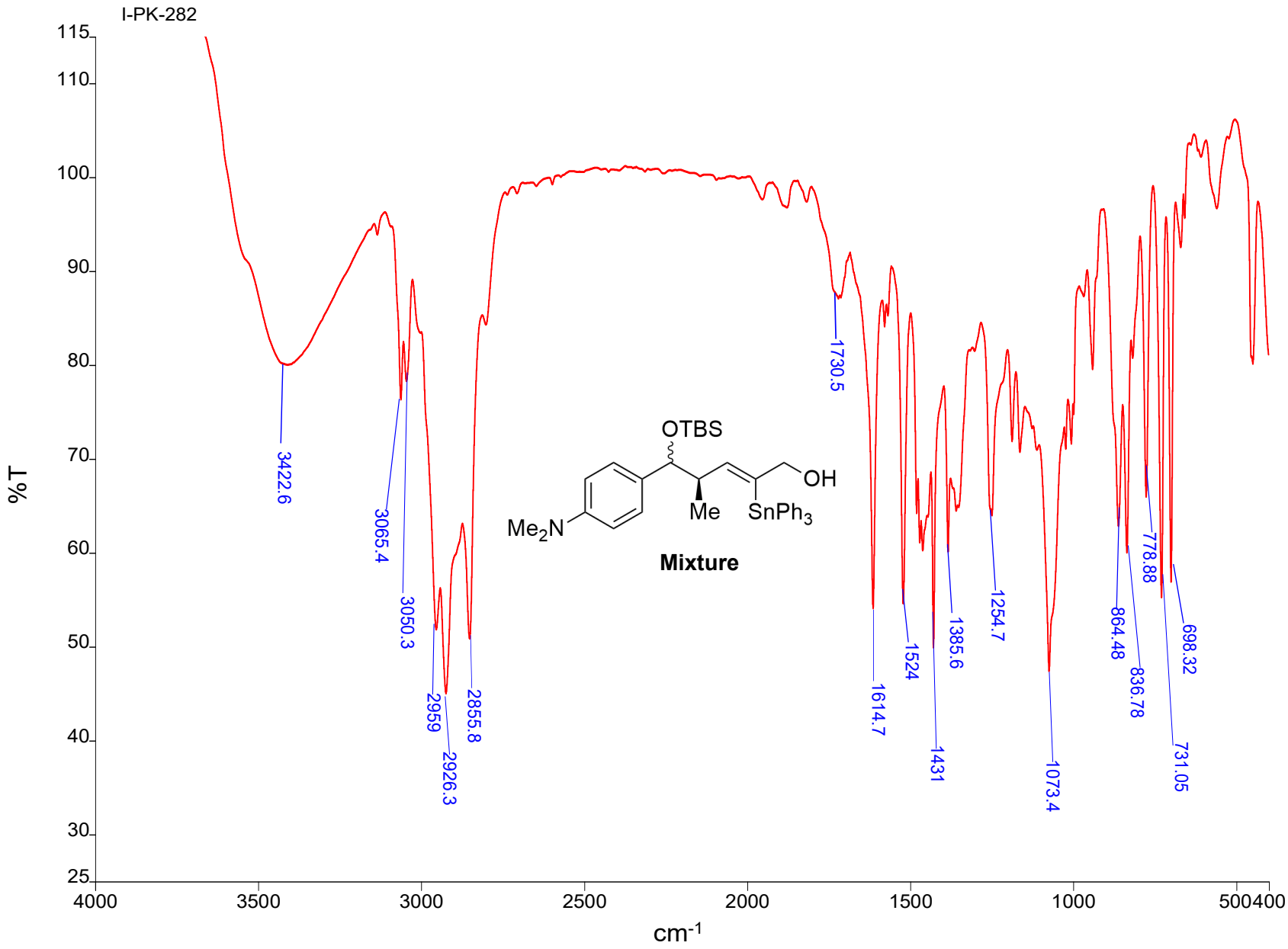
======== CHANNEL f1 ========
SFO1         399.9018806 MHz
NUC1                1H
P1                14.88 usec
P2                29.76 usec
P28       0 usec
PLW1          7.59999990 W

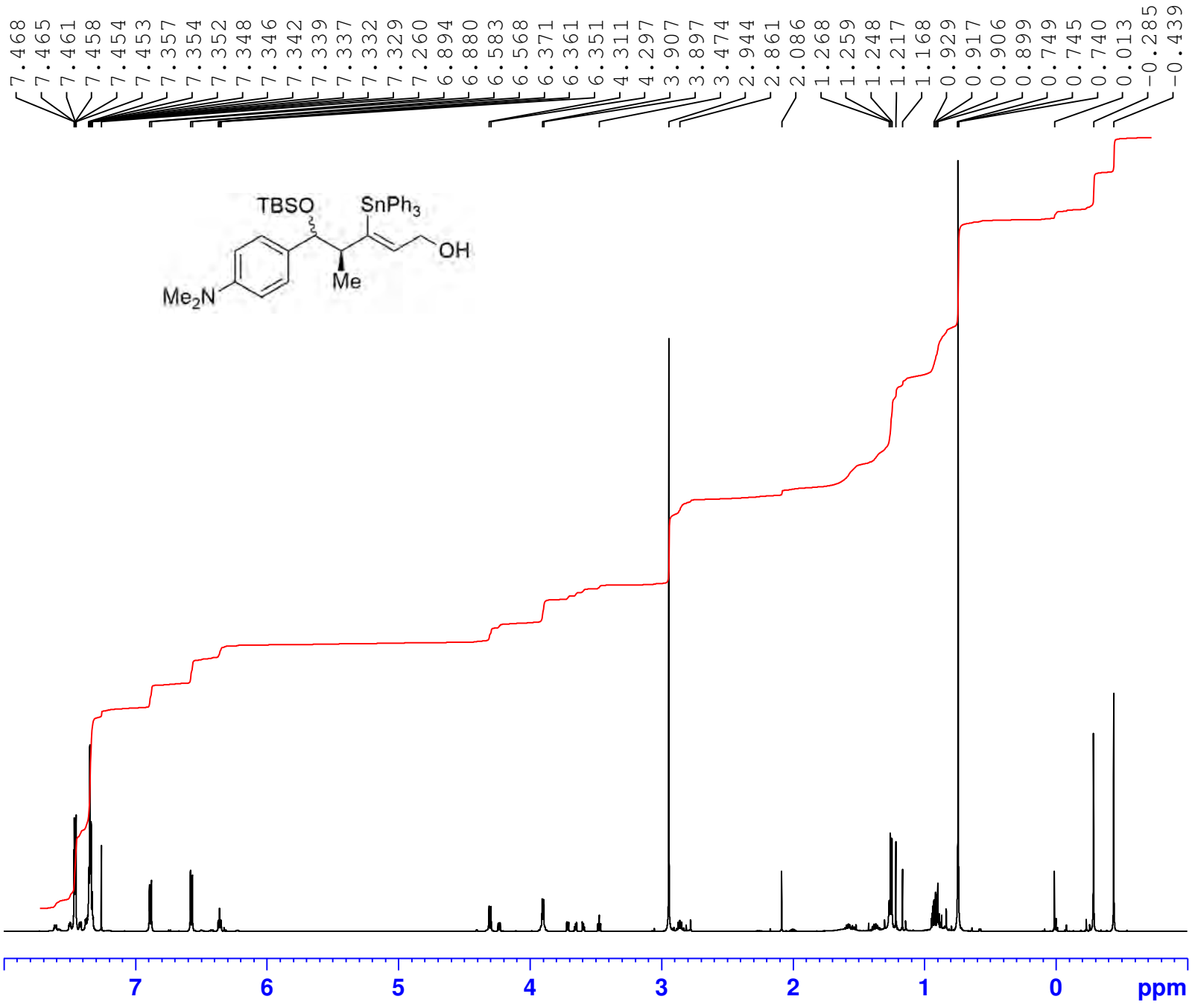
======== CHANNEL f2 ========
SFO2         100.5670016 MHz
NUC2                13C
CPDPRG[2            garp4
P3                10.00 usec
P14              500.00 usec
P31             1900.00 usec
PCPD2             80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3   0 Hz
SPW3          6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18             0.500
SPOFFS18 0 Hz
SPW18         1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1             100.567 MHz
FIDRES        204.515701 Hz
SW               260.304 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           399.9000104 MHz
WDW                 QSINE
```


I-PK-282
%T
115
110
100
90
80
70
60
50
40
30
25
4000
3500
3000
2500
2000
1500
1000
500400
cm-1
3422.6
3065.4
3050.3
2959
2926.3
2855.8
1730.5
1614.7
1524
1431
1385.6
1254.7
1073.4
864.48
836.78
778.88
731.05
698.32
OTBS
OH
Me
SnPh3
Me2N
Mixture

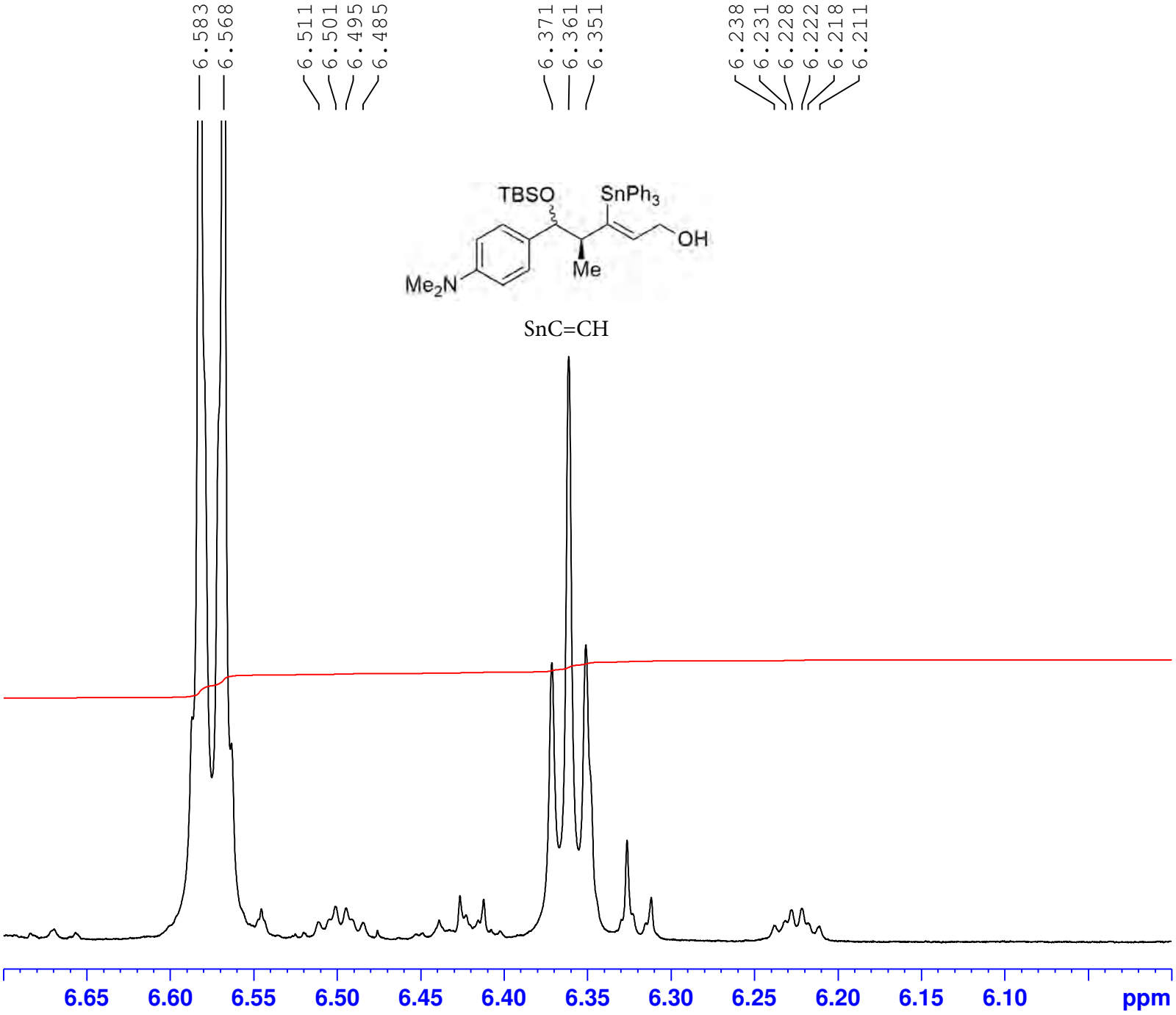



```
Current Data Parameters
NAME          IV-PK-09By
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201217
Time               15.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH            18028.846 Hz
FIDRES          0.100001 Hz
AQ             4.9999318 sec
RG                 43.25
DW                27.733 usec
DE                 7.60 usec
TE                300.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF         600.1300147 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

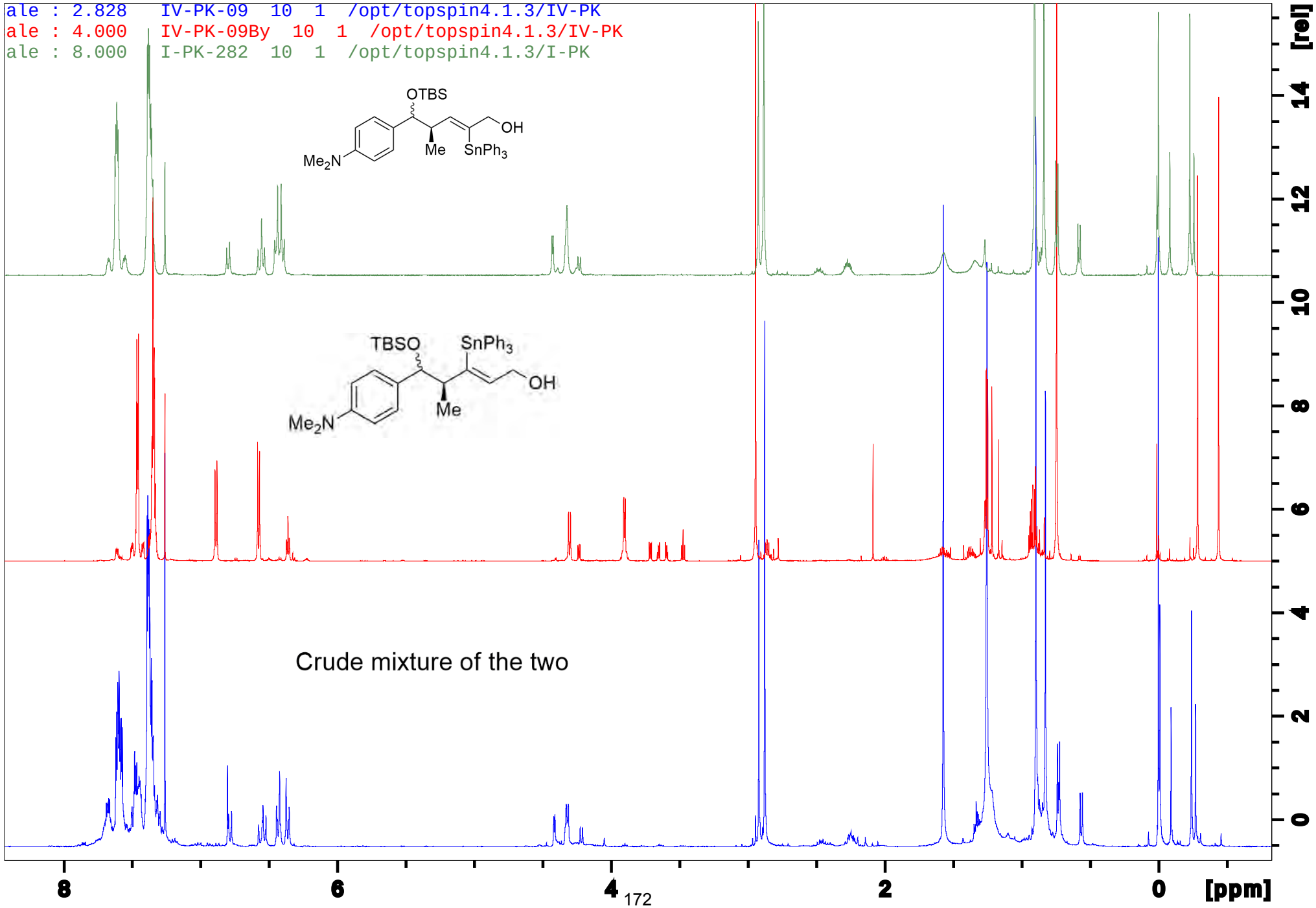


Current Data Parameters
NAME IV-PK-09By
EXPNO 10
PROCNO 1

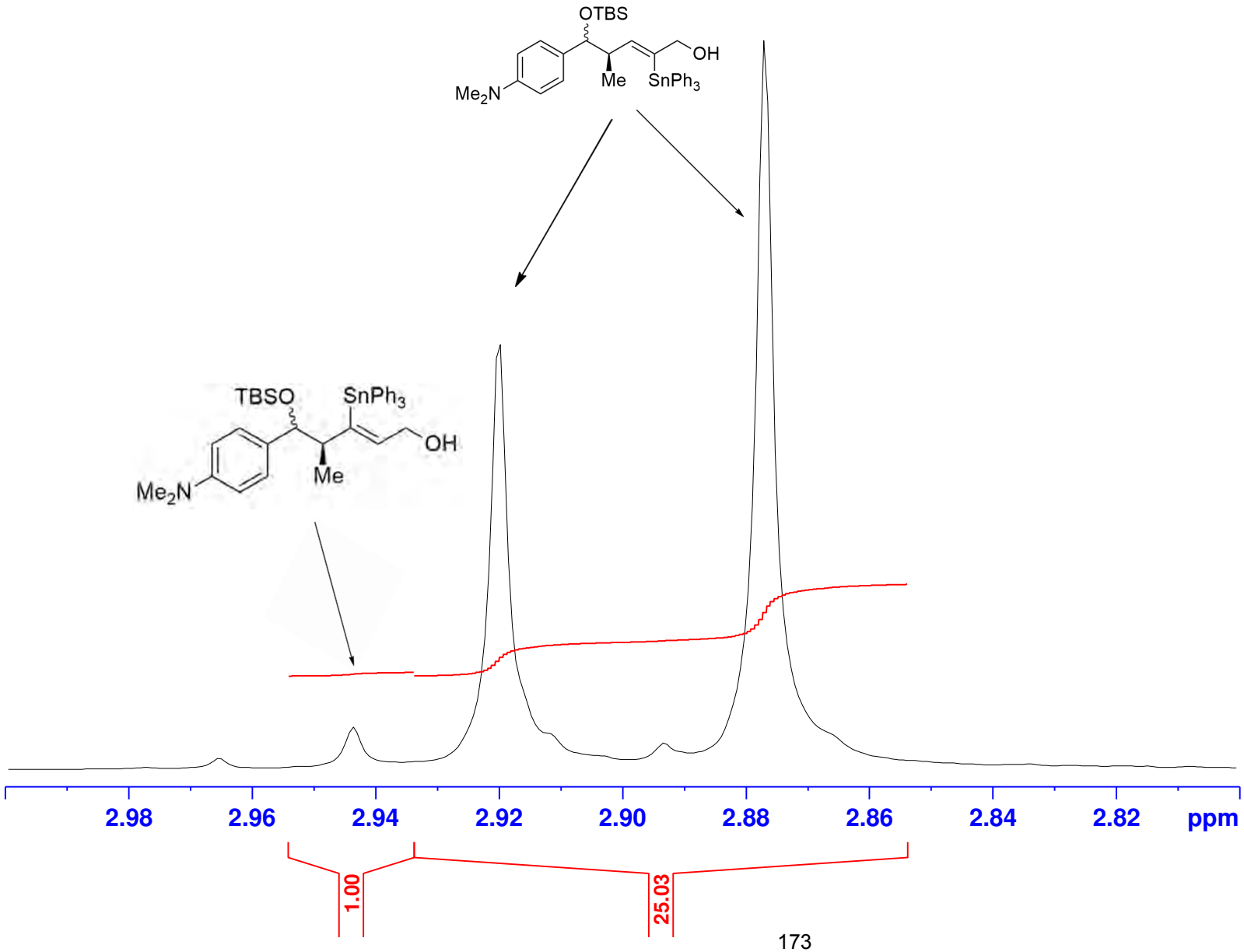
F2 - Acquisition Parameters
Date_ 20201217
Time 15.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 180286
SOLVENT CDCl3
NS 16
DS 0
SWH 18028.846 Hz
FIDRES 0.100001 Hz
AQ 4.9999318 sec
RG 43.25
DW 27.733 usec
DE 7.60 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.60000038 W

F2 - Processing parameters
SI 262144
SF 600.1300147 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00


ale : 2.828 IV-PK-09 10 1 /opt/topspin4.1.3/IV-PK
ale : 4.000 IV-PK-09By 10 1 /opt/topspin4.1.3/IV-PK
ale : 8.000 I-PK-282 10 1 /opt/topspin4.1.3/I-PK
OTBS
OH
Me
SnPh3
Me2N
TBSO
SnPh3
OH
Me
Me2N
Crude mixture of the two
[rel]
14
12
10
8
6
4
2
0
8
6
4
2
0
[ppm]

NMe$_2$ of the crude mixture



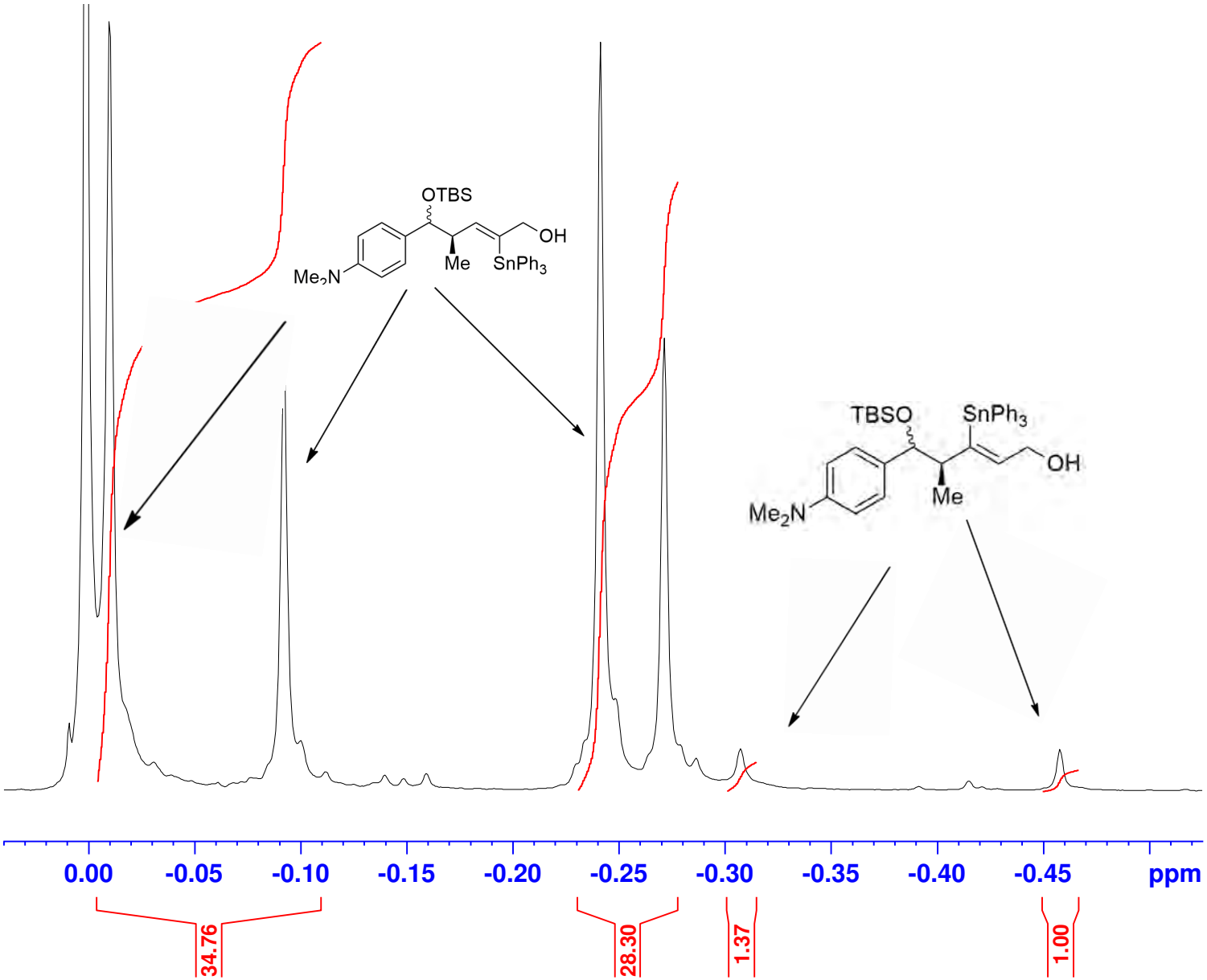

```
Current Data Parameters
NAME                  IV-PK-09
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201028
Time                     10.55
INSTRUM              AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                        128
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                        80.6
DW                      60.800 usec
DE                       6.50 usec
TE                      290.9 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9124696 MHz
NUC1                       1H
P1                      15.00 usec
PLW1               17.29199982 W

F2 - Processing parameters
SI                      32768
SF                 399.9100098 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

SiMe$_2$ of the crude mixture

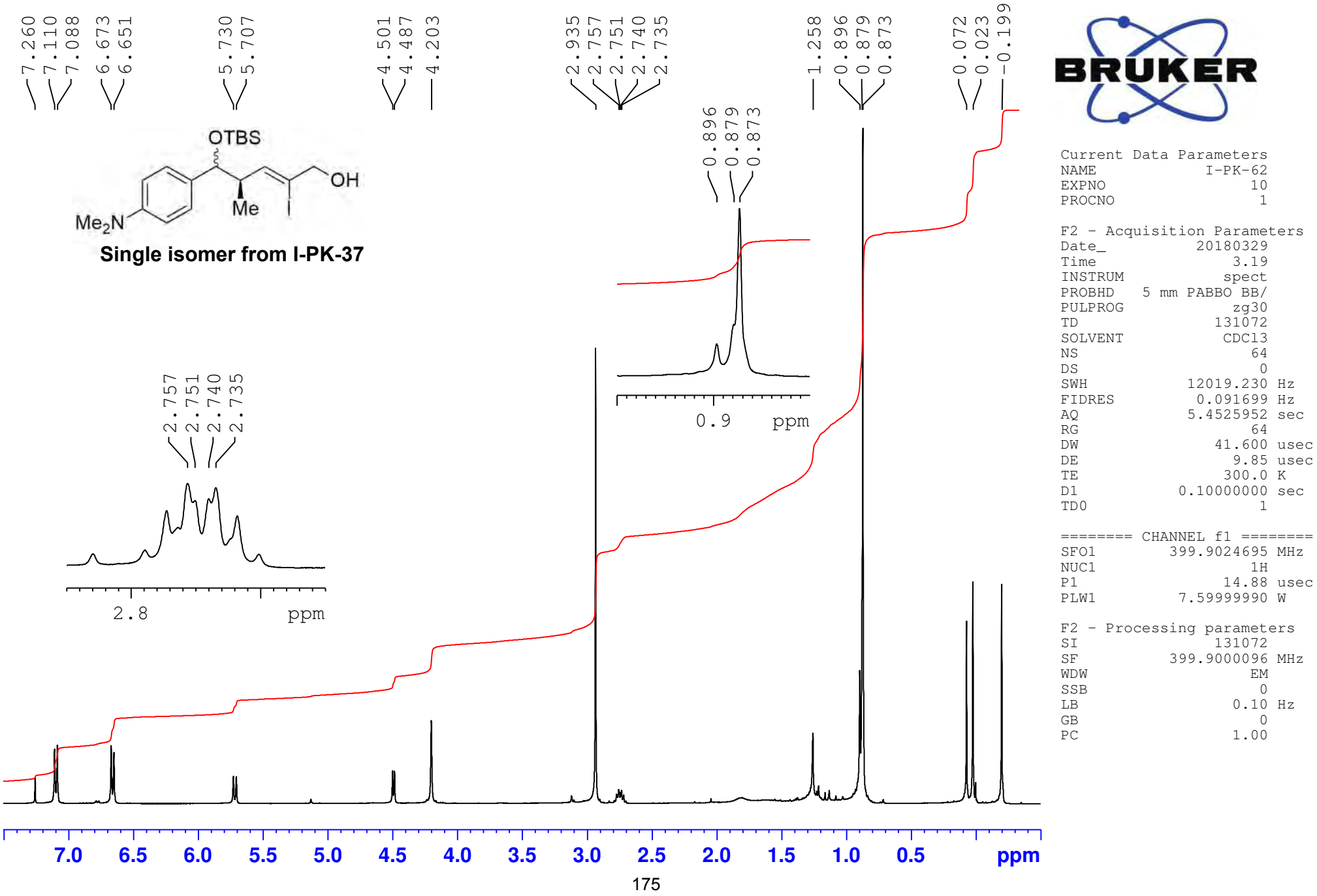



```
Current Data Parameters
NAME                  IV-PK-09
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201028
Time                     10.55
INSTRUM              AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                        128
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                        80.6
DW                      60.800 usec
DE                       6.50 usec
TE                      290.9 K
D1                   1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9124696 MHz
NUC1                       1H
P1                      15.00 usec
PLW1               17.29199982 W

F2 - Processing parameters
SI                        32768
SF                 399.9100098 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



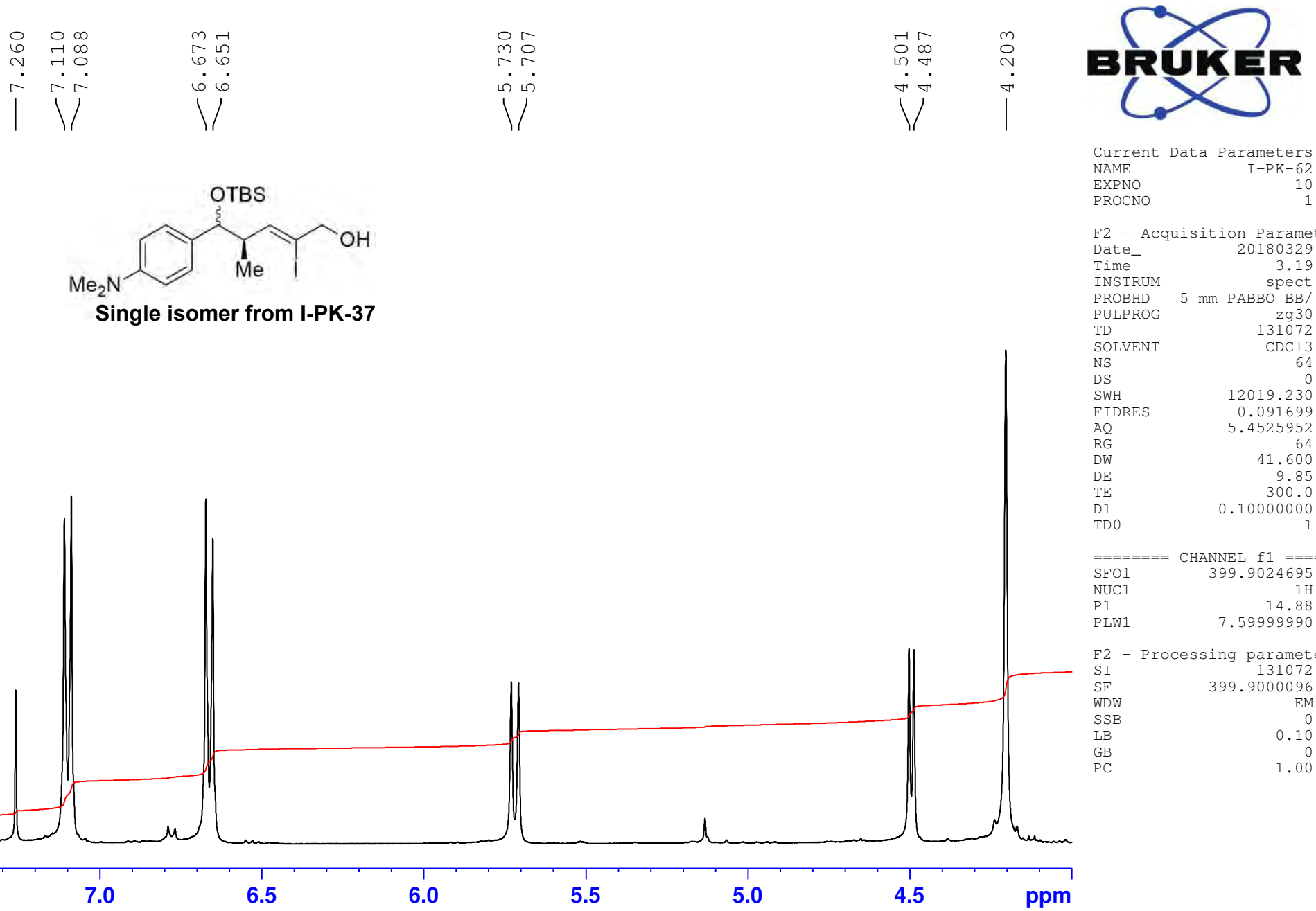
BRUKER

```
Current Data Parameters
NAME                  I-PK-62
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180329
Time                    3.19
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                    131072
SOLVENT                CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                         64
DW                    41.600 usec
DE                     9.85 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                    14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                    131072
SF               399.9000096 MHz
WDW                       EM
SSB                        0
LB                      0.10 Hz
GB                         0
PC                      1.00
```



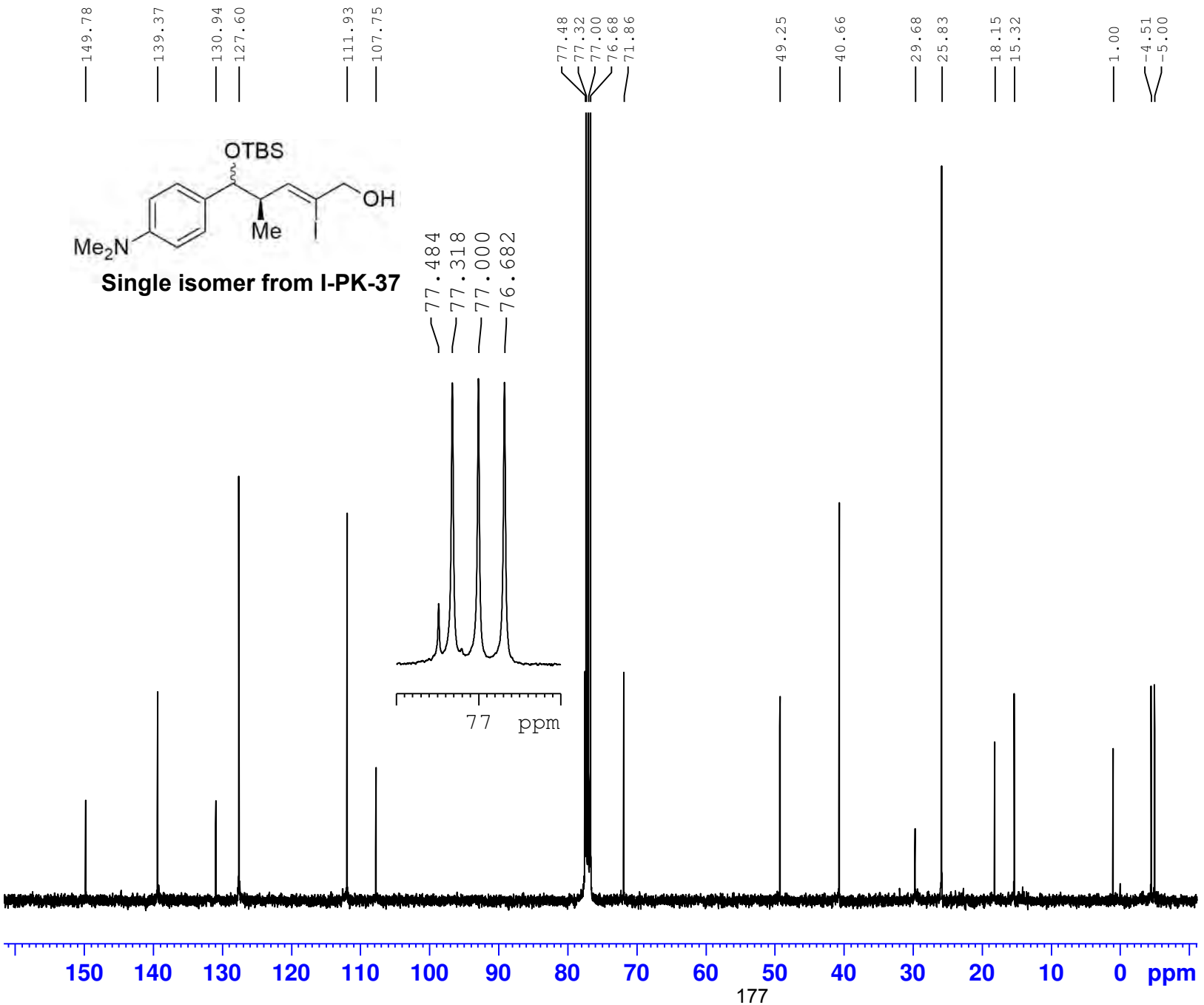
BRUKER

```
Current Data Parameters
NAME                  I-PK-62
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180329
Time                    3.19
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                    131072
SOLVENT                CDCl3
NS                         64
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                         64
DW                     41.600 usec
DE                      9.85 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                     14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                    131072
SF               399.9000096 MHz
WDW                       EM
SSB                        0
LB                      0.10 Hz
GB                         0
PC                      1.00
```



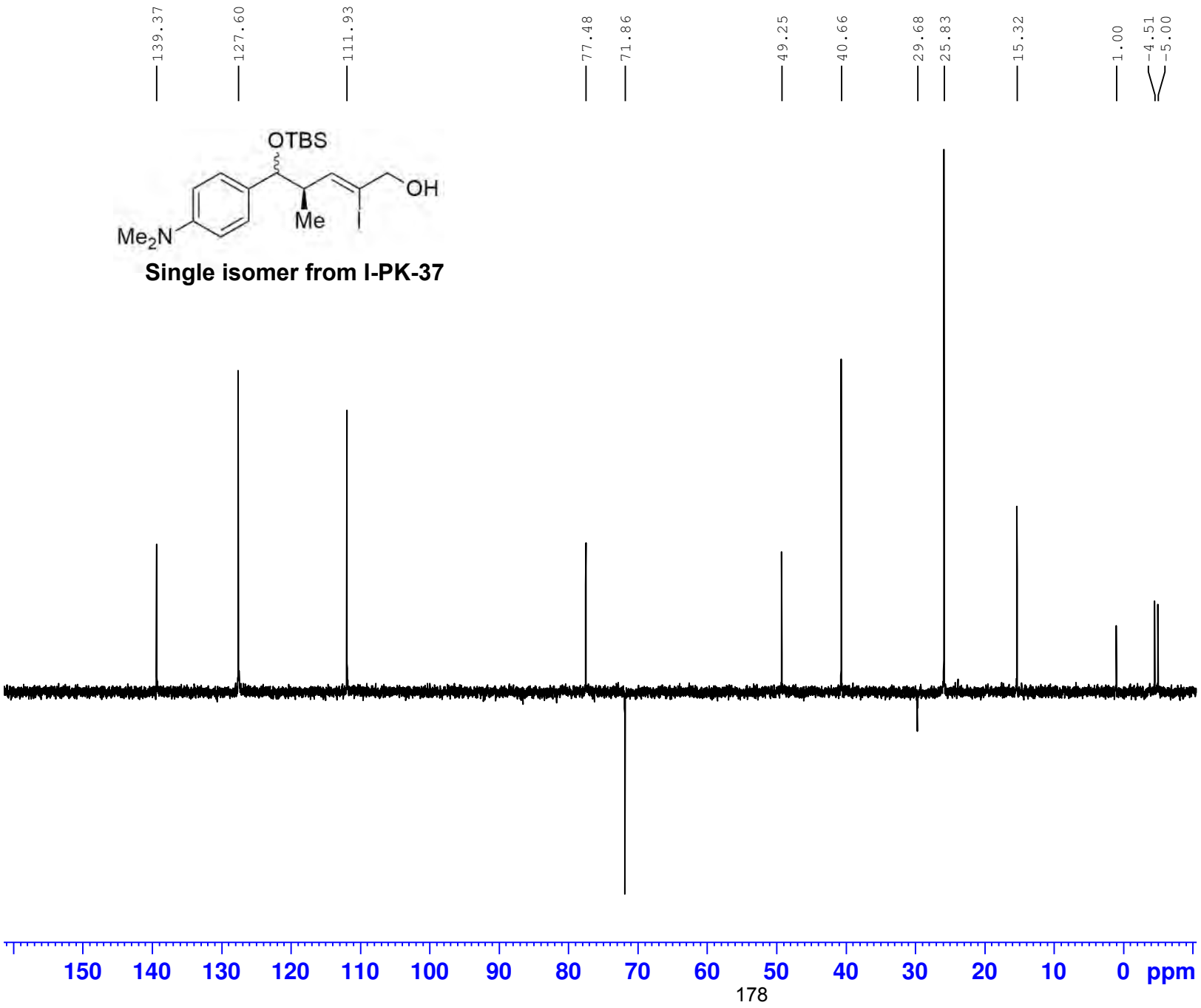

```
Current Data Parameters
NAME                  I-PK-62
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180329
Time                    4.30
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                     119044
SOLVENT                 CDCl3
NS                       1200
DS                          4
SWH                25000.000 Hz
FIDRES              0.210006 Hz
AQ                  2.3808801 sec
RG                       2050
DW                    20.000 usec
DE                     9.12 usec
TE                     300.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             100.5659947 MHz
NUC1                      13C
P1                     10.00 usec
PLW1             44.46300125 W

======== CHANNEL f2 ========
SFO2             399.9015996 MHz
NUC2                      1H
CPDPRG[2              waltz64
PCPD2                  90.00 usec
PLW2              7.59999990 W
PLW12             0.20774999 W
PLW13             0.16827001 W

F2 - Processing parameters
SI                       131072
SF               100.5549375 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



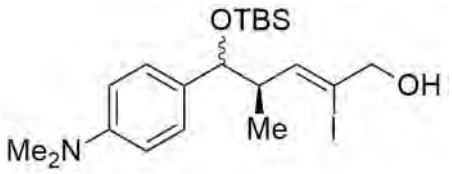
Current Data Parameters
NAME I-PK-62
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180329
Time 4.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

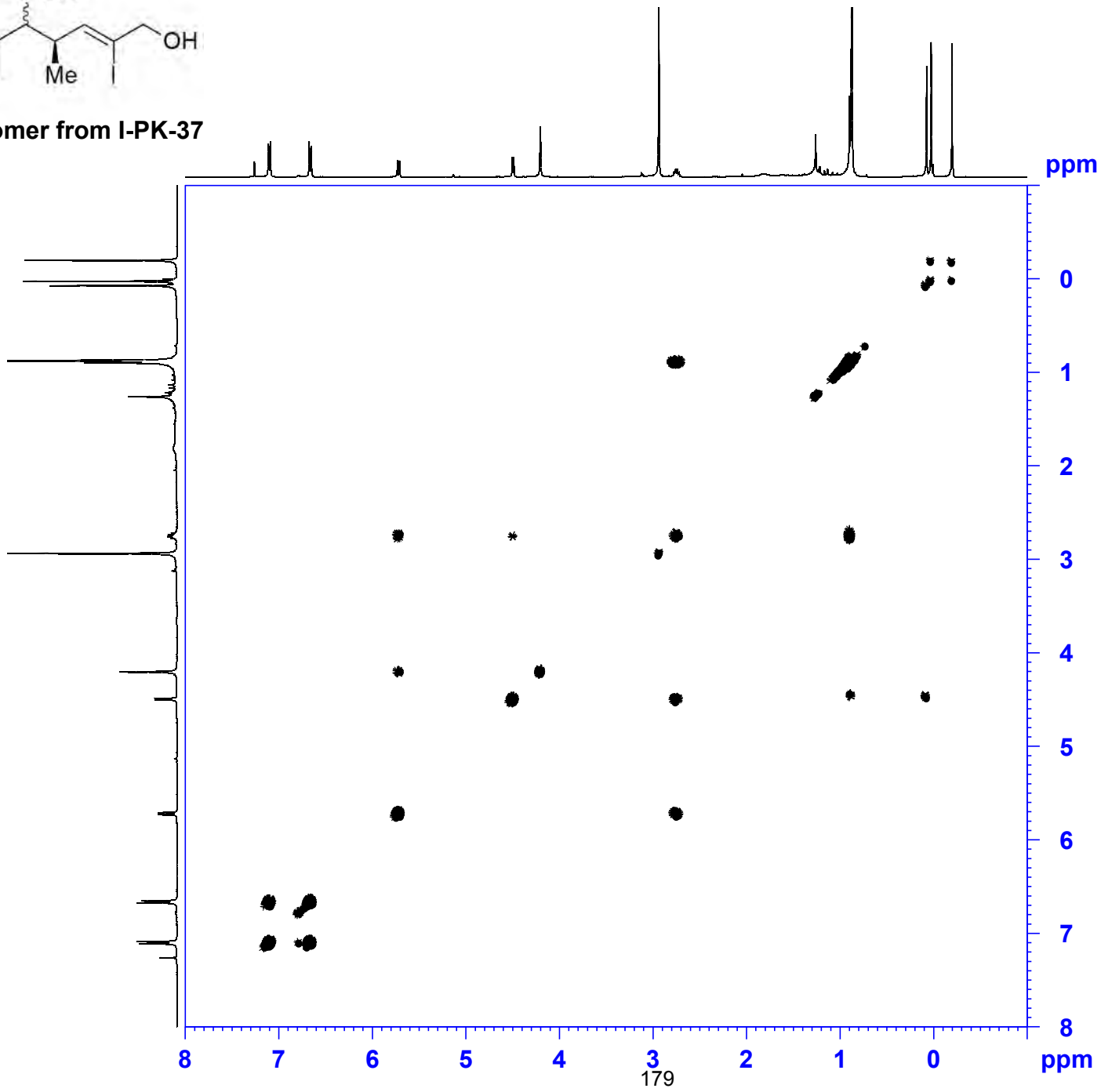
======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549373 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Single isomer from I-PK-37




```
Current Data Parameters
NAME                  I-PK-62
EXPNO                      13
PROCNO                      1

F2 - Acquisition Parameters
Date_                20180329
Time                     4.49
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                       2048
SOLVENT                CDCl3
NS                          1
DS                          8
SWH                  4401.409 Hz
FIDRES               2.149125 Hz
AQ                  0.2326528 sec
RG                       2050
DW                    113.600 usec
DE                      6.50 usec
TE                     300.0 K
D0                0.00000300 sec
D1                0.91725987 sec
D11               0.03000000 sec
D12               0.00002000 sec
D13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00022720 sec

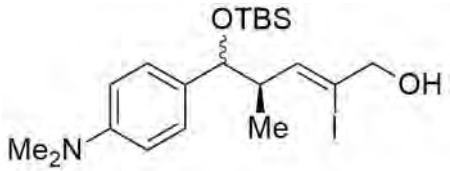
======== CHANNEL f1 ========
SFO1             399.9017944 MHz
NUC1                    1H
P1                     14.88 usec
P17                  2500.00 usec
PLW1              7.59999990 W
PLW10             2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPNAM[3]          SMSQ10.100
GPZ1                   16.00 %
GPZ2                   12.00 %
GPZ3                   40.00 %
P16                  1000.00 usec

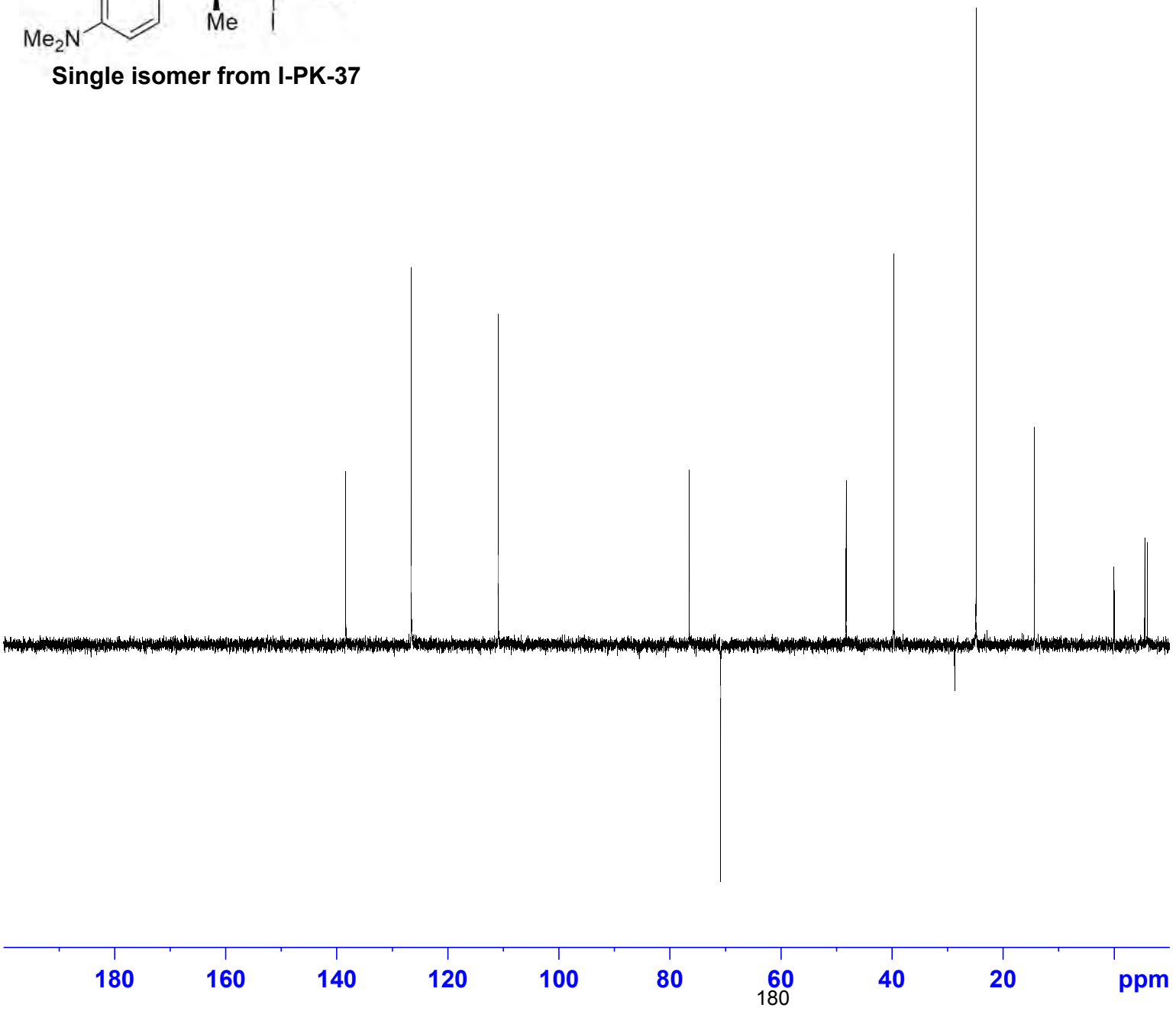
F1 - Acquisition parameters
TD                       256
SFO1                399.9018 MHz
FIDRES             34.386005 Hz
SW                    11.006 ppm
FnMODE                    QF

F2 - Processing parameters
SI                       1024
SF                399.9000079 MHz
WDW                      SINE
SSB           0
LB            0 Hz
GB            0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2                       QF
SF                399.9000095 MHz
WDW                      SINE
SSB           0
LB            0 Hz
GB            0
```



Single isomer from I-PK-37



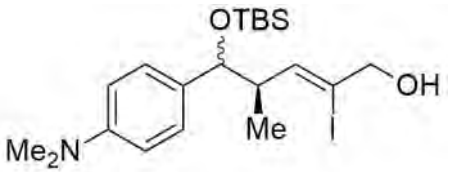

```
Current Data Parameters
NAME                  I-PK-62
EXPNO                      12
PROCNO                      1

F2 - Acquisition Parameters
Date_                20180329
Time                     4.47
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG             deptsp135
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                       2050
DW                     20.800 usec
DE                      6.50 usec
TE                     300.0 K
CNST2              145.0000000
D1                  2.00000000 sec
D2                  0.00344828 sec
D12                 0.00002000 sec
TD0                         1

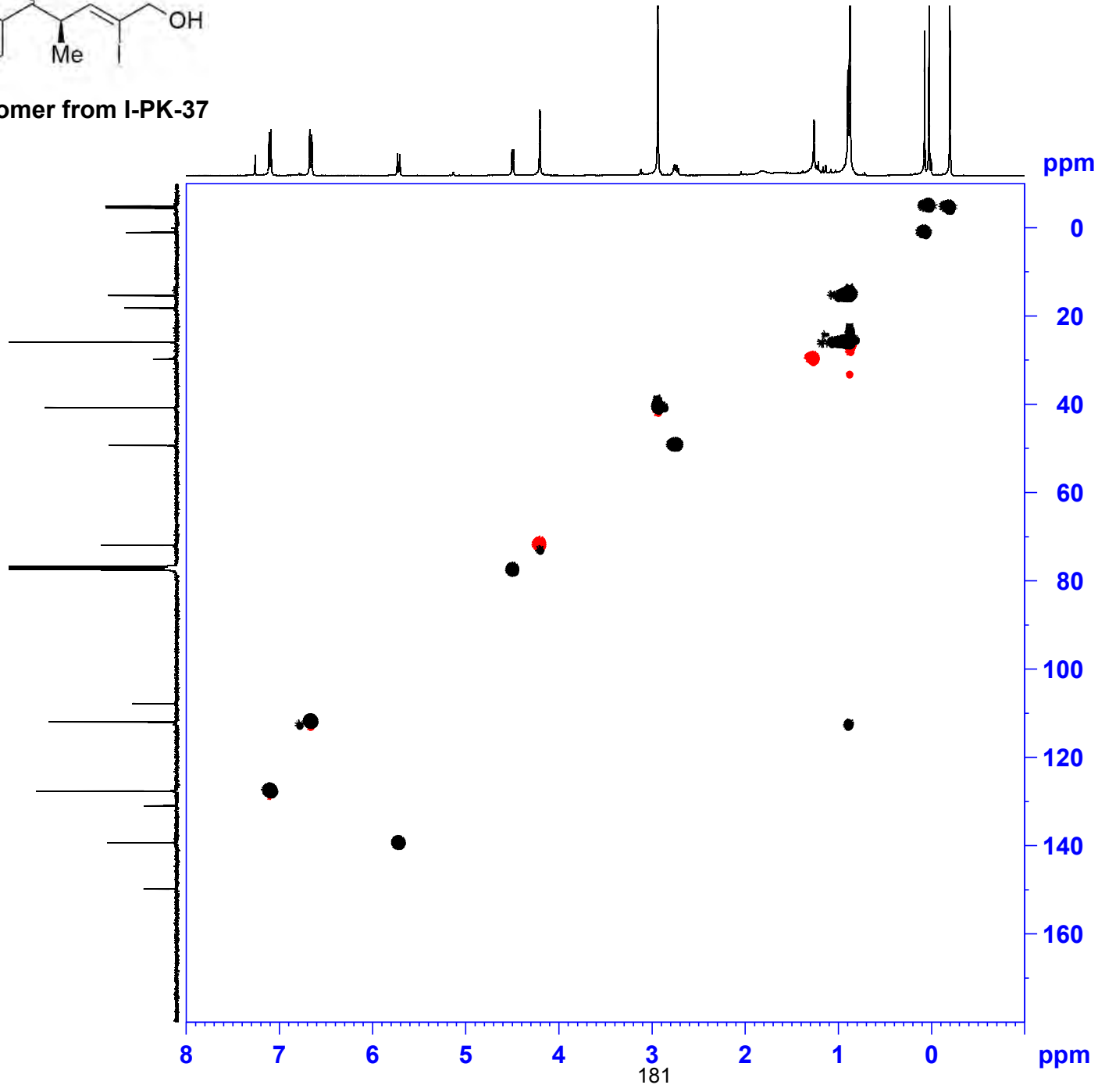
======== CHANNEL f1 ========
SFO1               100.5649905 MHz
NUC1                       13C
P1                       10.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1              44.46300125 W
SPNAM[5]          Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5               6.79339981 W

======== CHANNEL f2 ========
SFO2               399.9012789 MHz
NUC2                       1H
CPDPRG[2                waltz16
P3                       14.88 usec
P4                       29.76 usec
PCPD2                    90.00 usec
PLW2                7.59999990 W
PLW12               0.20774999 W

F2 - Processing parameters
SI                       32768
SF                 100.5550385 MHz
WDW                         EM
SSB         0
LB                       1.00 Hz
GB          0
PC                       1.40
```



Single isomer from I-PK-37




```
NAME                 I-PK-62
EXPNO                     14
PROCNO                     1

F2 - Acquisition Parameters
Date_               20180329
Time                    4.56
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                      1024
SOLVENT              CDCl3
NS                         2
DS                        32
SWH                4807.692 Hz
FIDRES             4.695012 Hz
AQ                 0.1064960 sec
RG                      2050
DW                  104.000 usec
DE                    6.50 usec
TE                    300.0 K
CNST2             145.0000000
D0                 0.00000300 sec
D1                 0.80000001 sec
D4                 0.00172414 sec
D11                0.03000000 sec
D16                0.00020000 sec
D21                0.00360000 sec
IN0                0.00001910 sec

======== CHANNEL f1 ========
SFO1            399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28         0 usec
PLW1             7.59999990 W

======== CHANNEL f2 ========
SFO2            100.5670016 MHz
NUC2                      13C
CPDPRG[2                garp4
P3                      10.00 usec
P14                    500.00 usec
P31                   1900.00 usec
PCPD2                   80.00 usec
PLW0        0 W
PLW2             44.46300125 W
PLW12             0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                  0.500
SPOFFS3   0 Hz
SPW3             6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                 0.500
SPOFFS18 0 Hz
SPW18            1.62779999 W

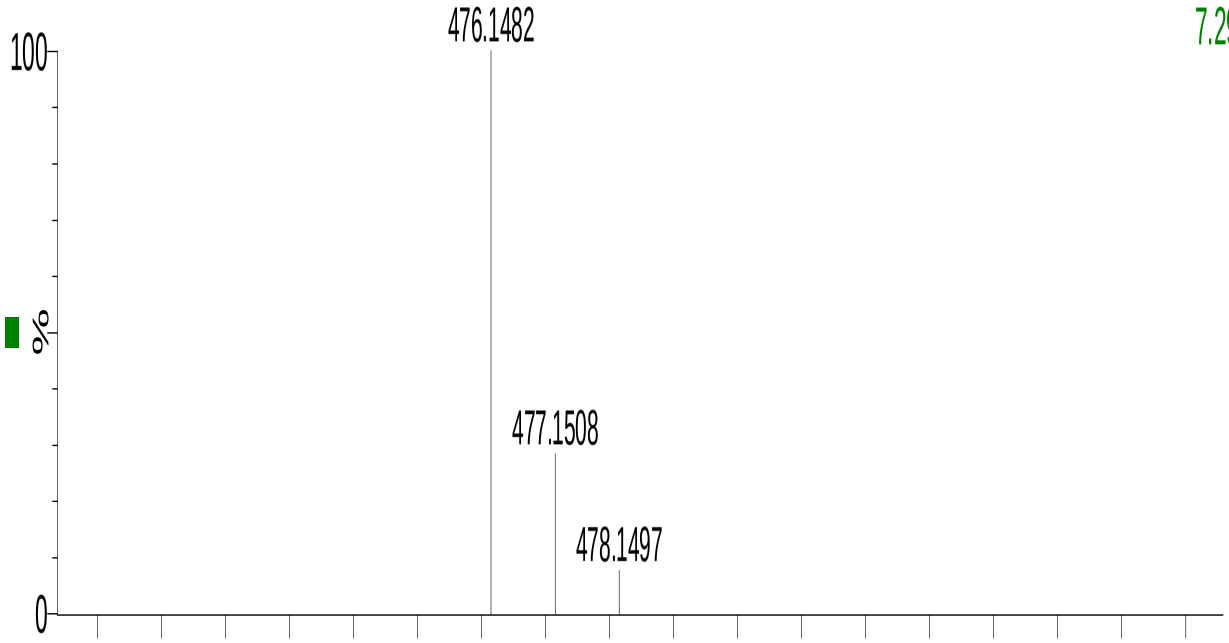
====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPZ1                   80.00 %
GPZ2                   20.10 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1                100.567 MHz
FIDRES           204.515701 Hz
SW                  260.304 ppm
FnMODE     Echo-Antiecho

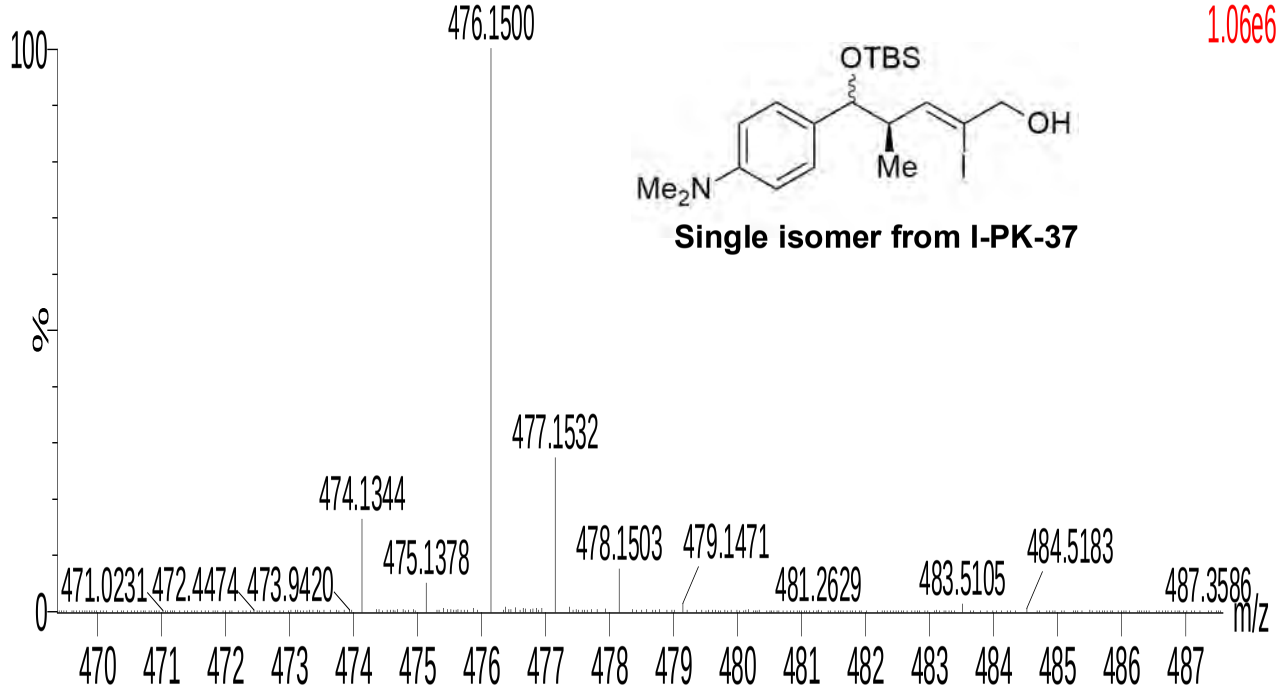
F2 - Processing parameters
SI                       1024
SF               399.9000101 MHz
WDW                     QSINE
SSB                         2
```

I-PK-62 **16-04-2018**

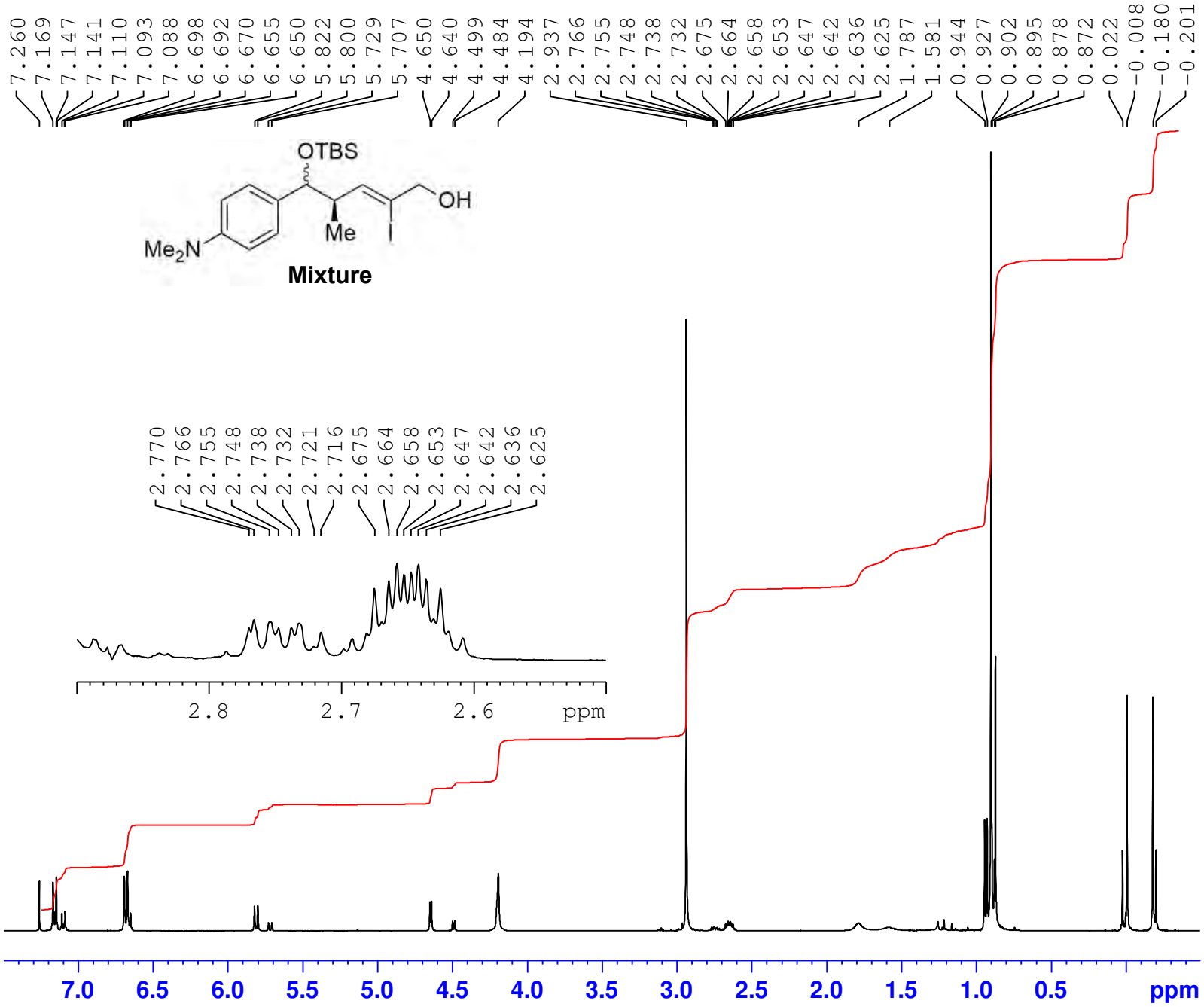
asep_16APR_2018_40 (0.070) Is (1.00,1.00) C20H34NSiIO2H 1: TOF MS ES+ 7.29e12



asep_16APR_2018_40 12 (0.530) Cm (6:12) 1: TOF MS ES+ 1.06e6



Single isomer from I-PK-37



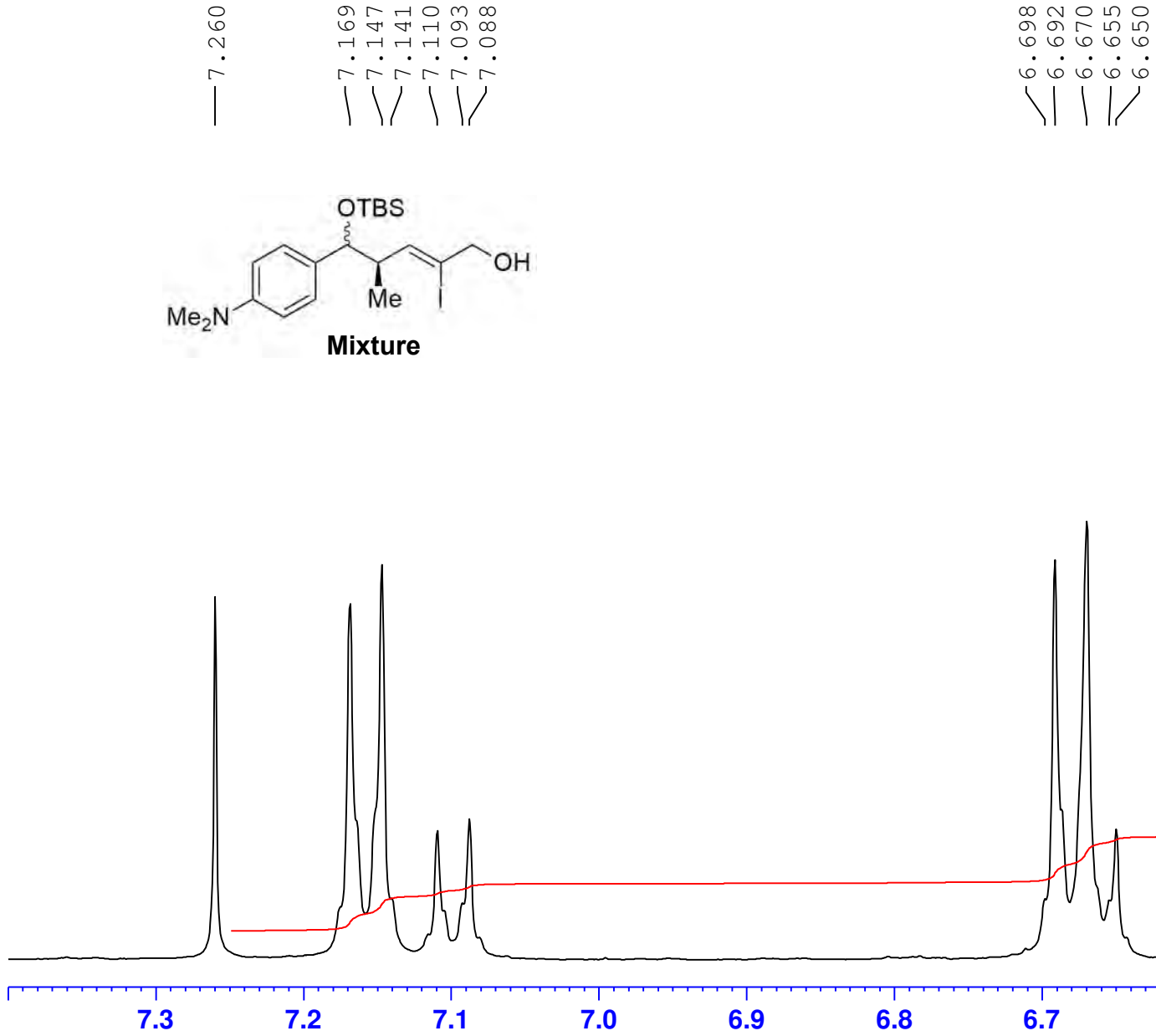
BRUKER

```
Current Data Parameters
NAME          III-PK-147
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200309
Time               18.36
INSTRUM        AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                    57
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9124696 MHz
NUC1                  1H
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           399.9100096 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

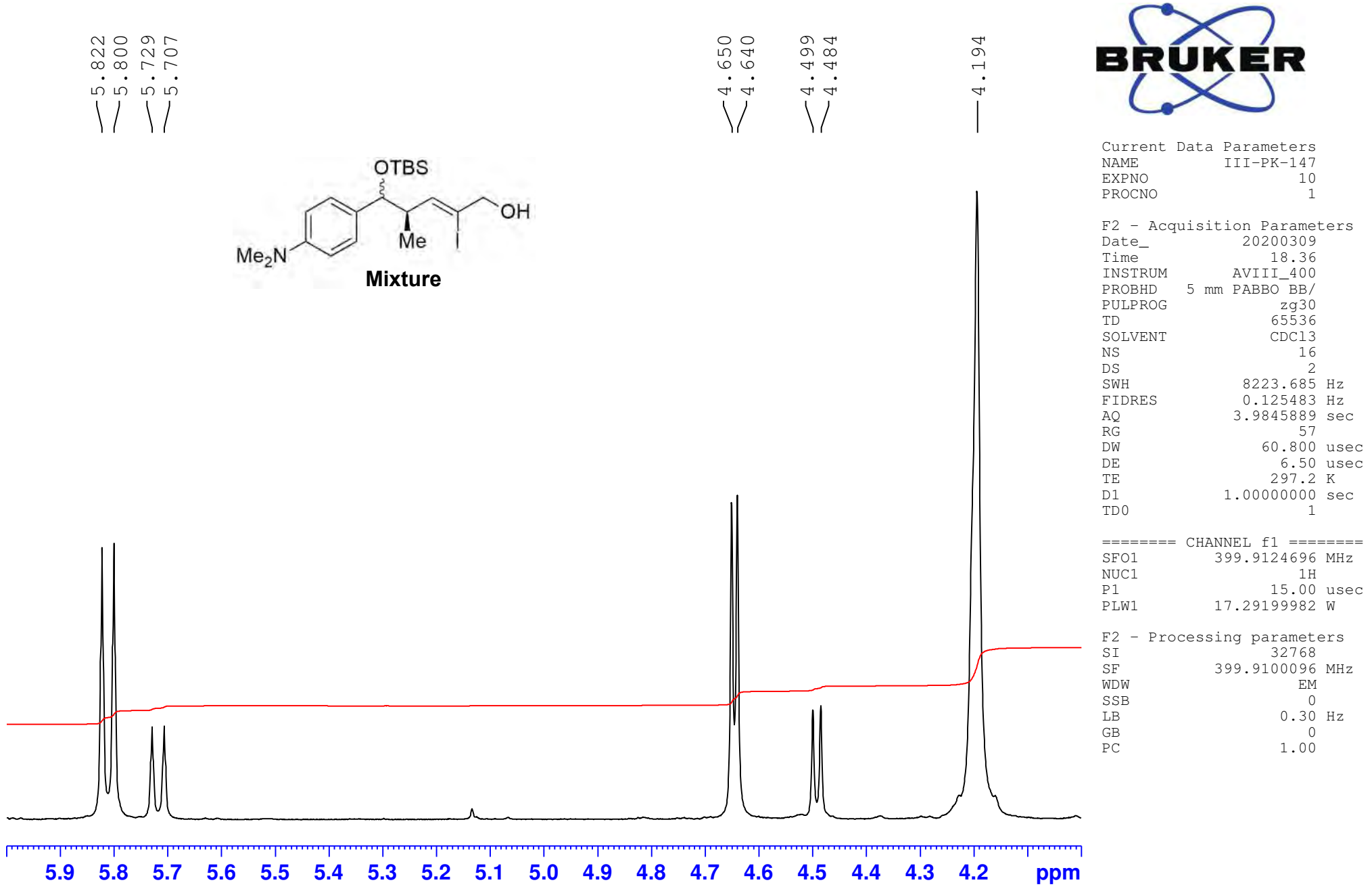


Current Data Parameters
NAME III-PK-147
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200309
Time 18.36
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

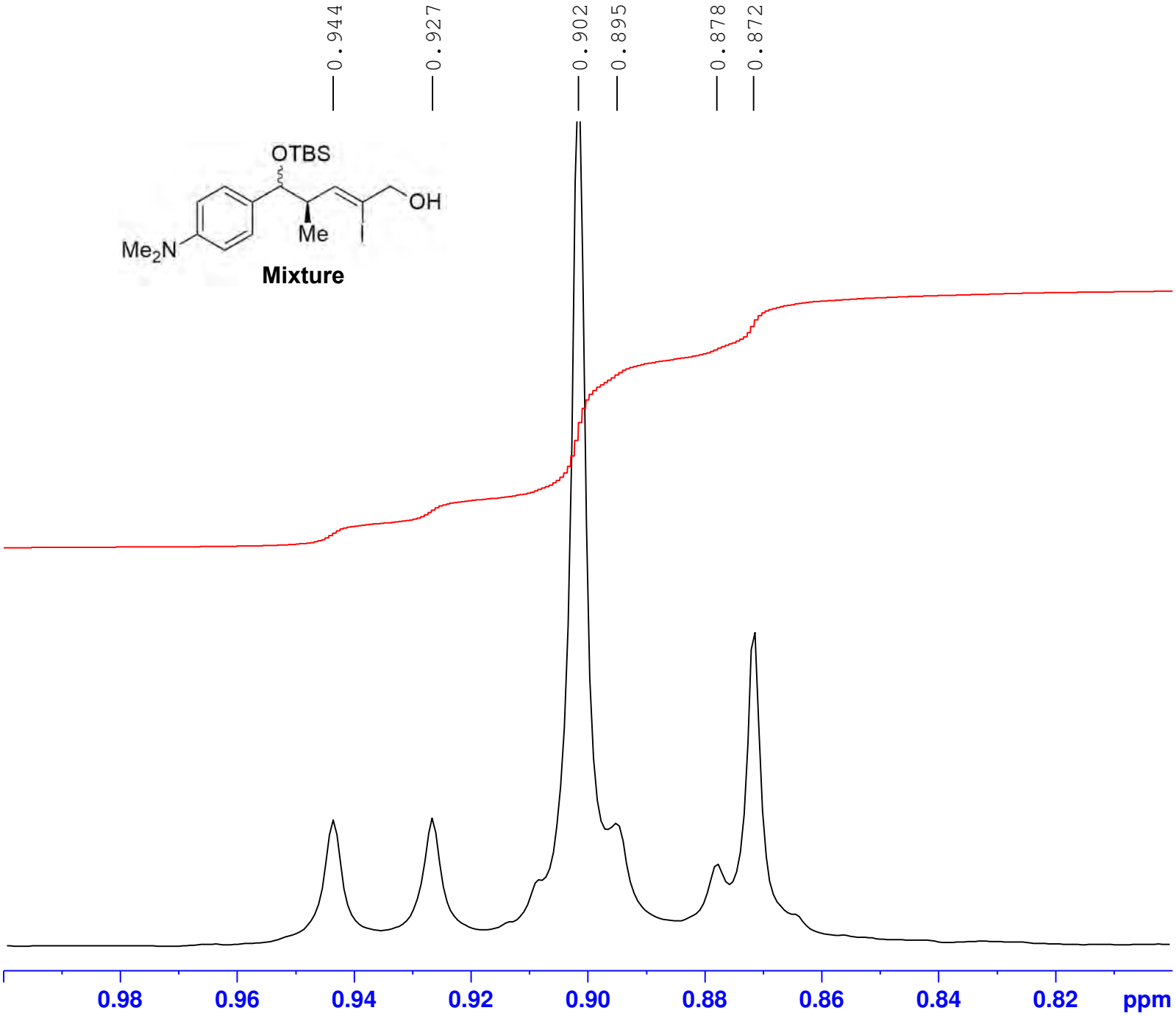


Current Data Parameters
NAME III-PK-147
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200309
Time 18.36
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

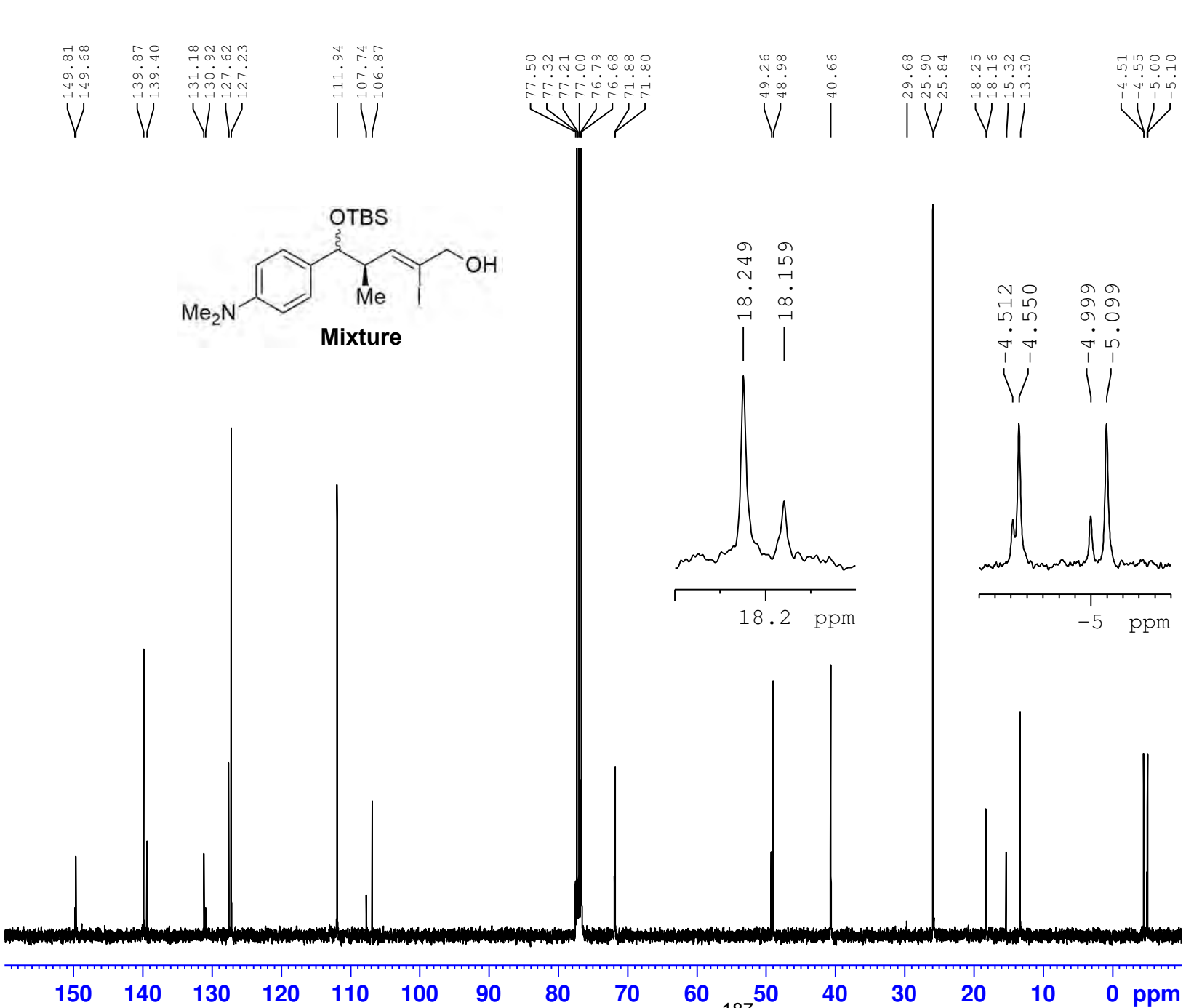



```
Current Data Parameters
NAME          III-PK-147
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200309
Time               18.36
INSTRUM        AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                    57
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9124696 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           399.9100096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



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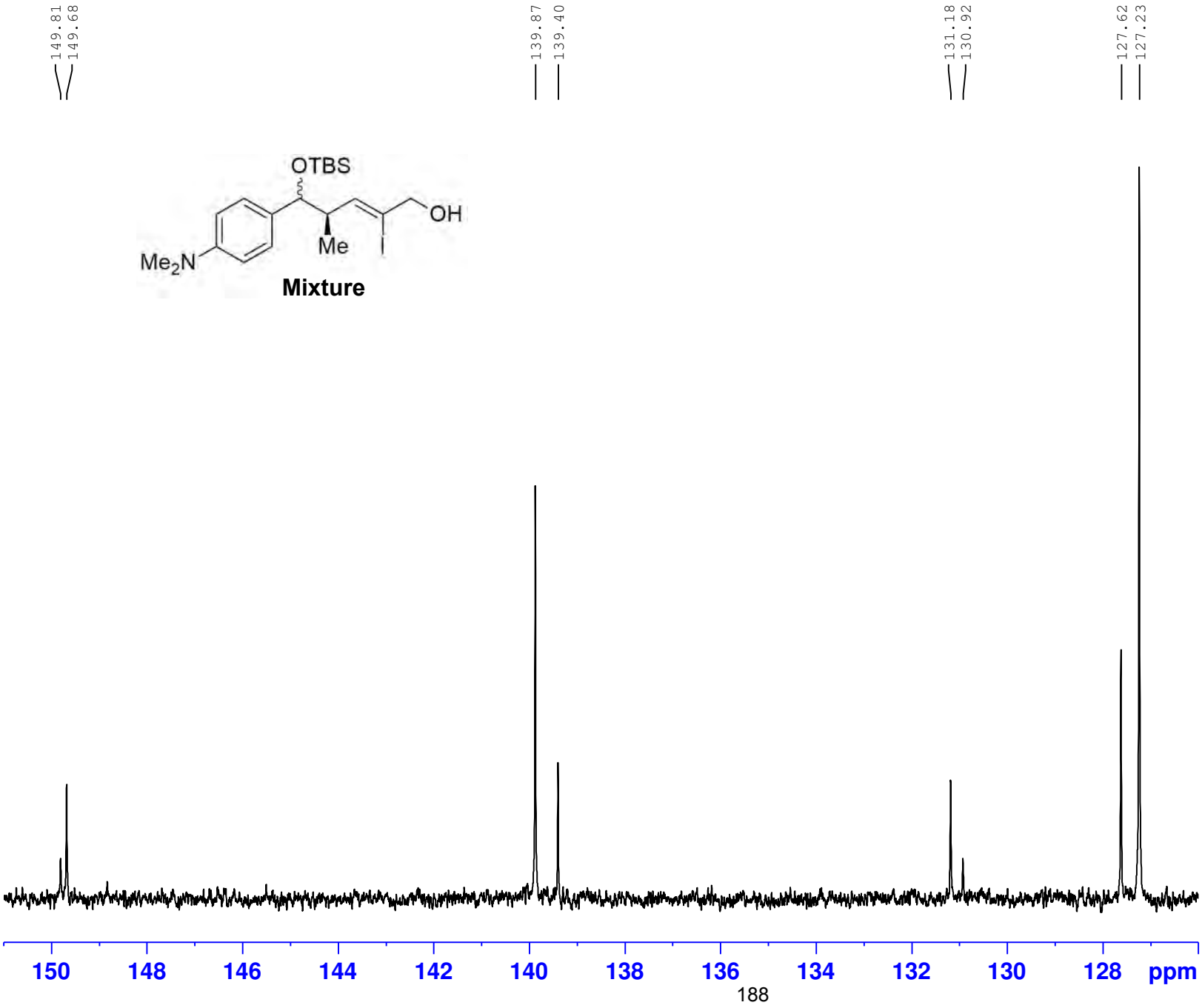
```
Current Data Parameters
NAME          III-PK-147
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200309
Time               19.32
INSTRUM        AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.250010 Hz
AQ             1.9999200 sec
RG                   64
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1               13C
P1                9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2               1H
CPDPRG[2        waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                131072
SF           100.5574511 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



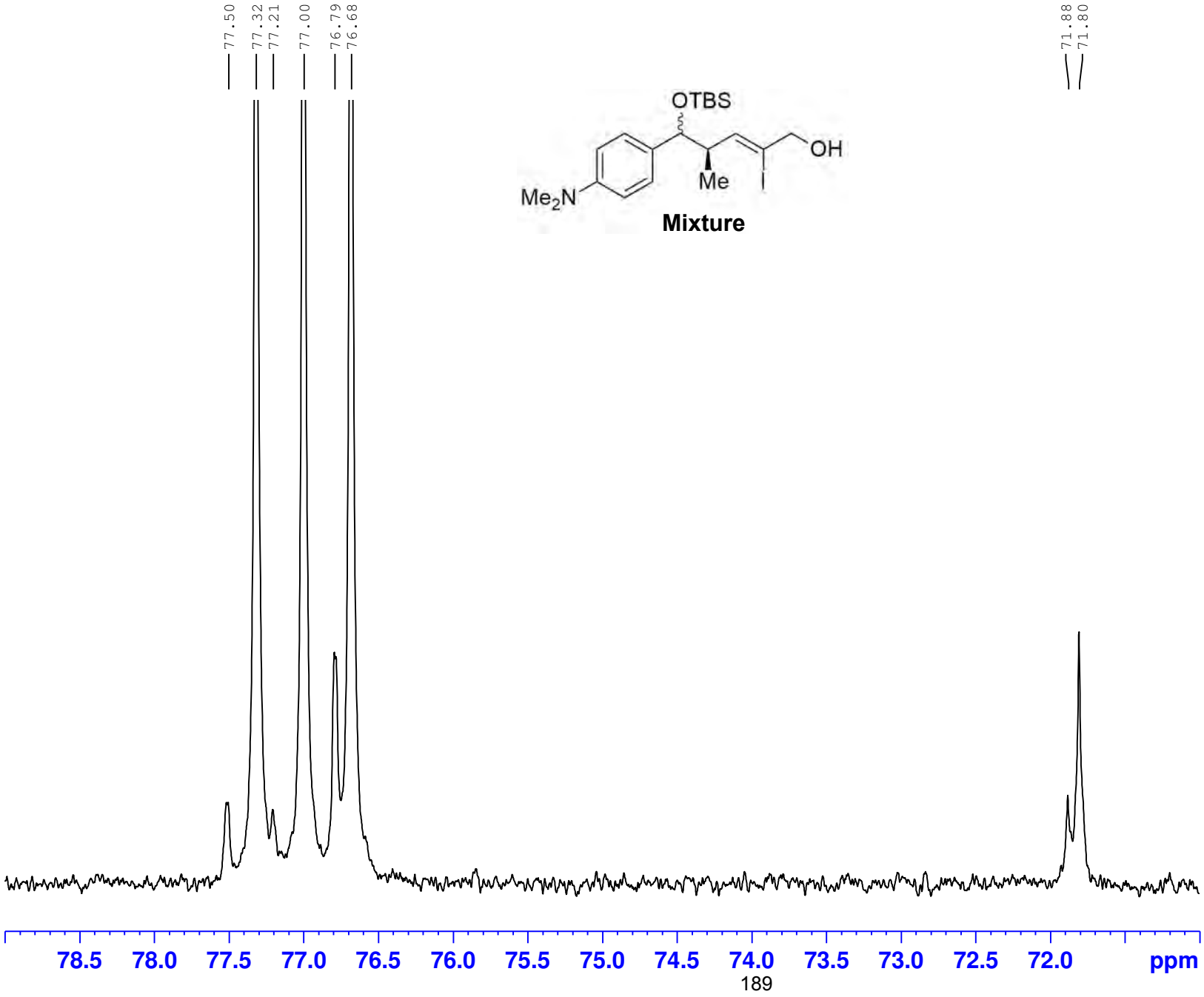
Current Data Parameters
NAME III-PK-147
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200309
Time 19.32
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 96150
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.250010 Hz
AQ 1.9999200 sec
RG 64
DW 20.800 usec
DE 6.50 usec
TE 300.6 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5675047 MHz
NUC1 13C
P1 9.00 usec
PLW1 96.68000031 W

======== CHANNEL f2 ========
SFO2 399.9115996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 17.29199982 W
PLW12 0.48032999 W
PLW13 0.38907000 W

F2 - Processing parameters
SI 131072
SF 100.5574511 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



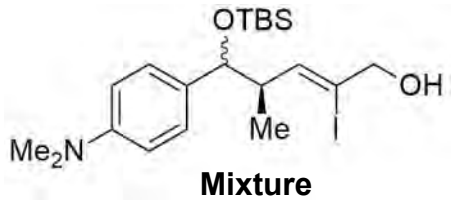

```
Current Data Parameters
NAME          III-PK-147
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200309
Time               19.32
INSTRUM        AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 96150
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.250010 Hz
AQ              1.9999200 sec
RG                    64
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1                 13C
P1                  9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2                 1H
CPDPRG[2         waltz64
PCPD2              90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                131072
SF           100.5574511 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



Mixture

49.26
48.98
40.66
29.68
25.90
25.84
25.896
25.838

25.9 25.8 ppm

49 48 47 46 45 44 43 42 41 40 39 38 37 36 35 34 33 32 31 30 29 28 27 ppm

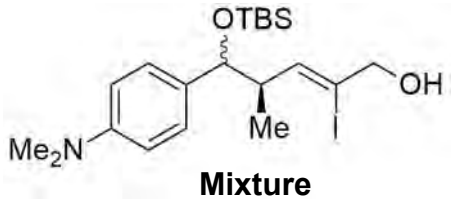
```
Current Data Parameters
NAME          III-PK-147
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200309
Time               19.32
INSTRUM        AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 96150
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.250010 Hz
AQ             1.9999200 sec
RG                    64
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1                 13C
P1                  9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2                 1H
CPDPRG[2         waltz64
PCPD2               90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                131072
SF           100.5574511 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



Mixture


```
Current Data Parameters
NAME          III-PK-147
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200310
Time                7.12
INSTRUM        AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG       cosygpmfqf
TD                  2048
SOLVENT             CDCl3
NS                     1
DS                     8
SWH             3546.099 Hz
FIDRES          1.731494 Hz
AQ              0.2887680 sec
RG                  2050
DW               141.000 usec
DE                 6.50 usec
TE                297.1 K
D0              0.00000300 sec
D1              1.90319502 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00028200 sec

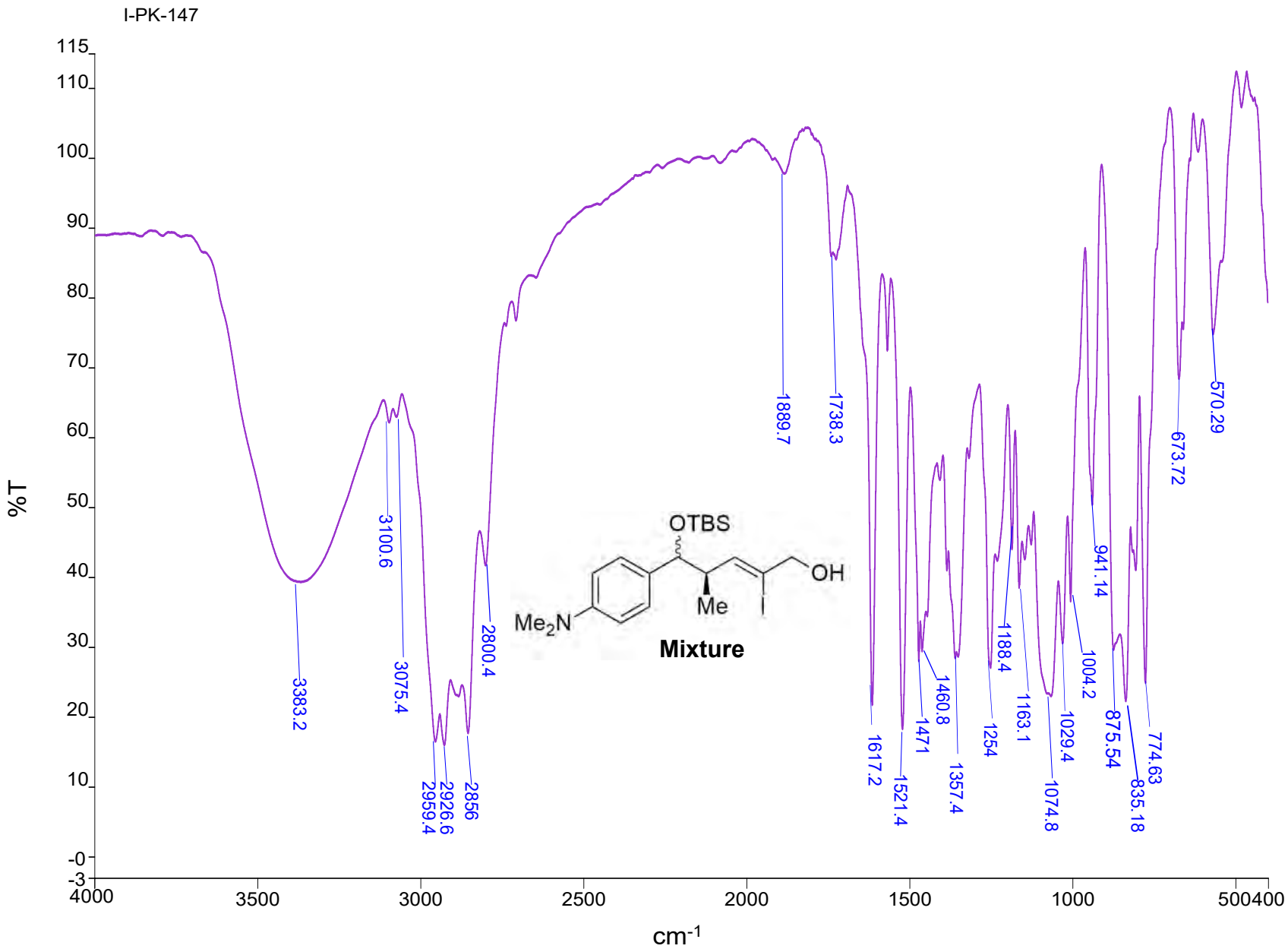
======== CHANNEL f1 ========
SFO1         399.9114741 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         17.29199982 W

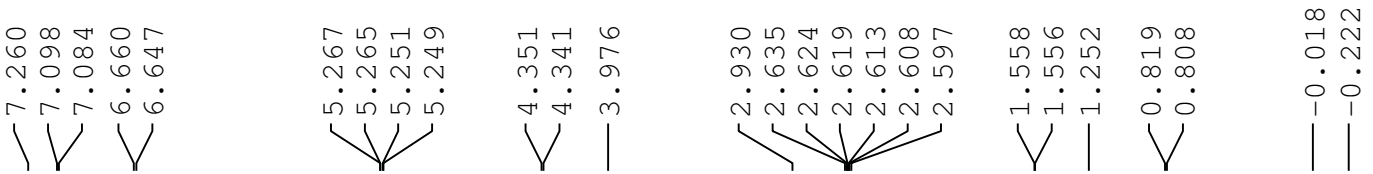
====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPNAM[2]         SINE.100
GPNAM[3]         SINE.100
GPZ1                16.00 %
GPZ2                12.00 %
GPZ3                40.00 %
P16               1000.00 usec

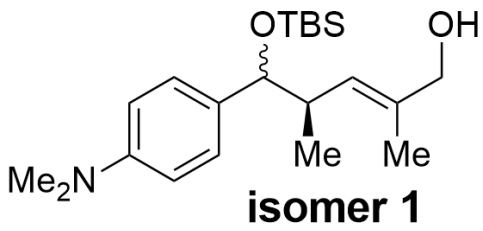
F1 - Acquisition parameters
TD                   256
SFO1            399.9115 MHz
FIDRES          27.703901 Hz
SW                 8.867 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           399.9100063 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           399.9100063 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```


I-PK-147
%T
cm-1
3383.2
3100.6
3075.4
2959.4
2926.6
2856
2800.4
1889.7
1738.3
1617.2
1521.4
1471
1460.8
1357.4
1254
1188.4
1163.1
1074.8
1029.4
1004.2
941.14
875.54
835.18
774.63
673.72
570.29
OTBS
OH
Me
Me2N
Mixture





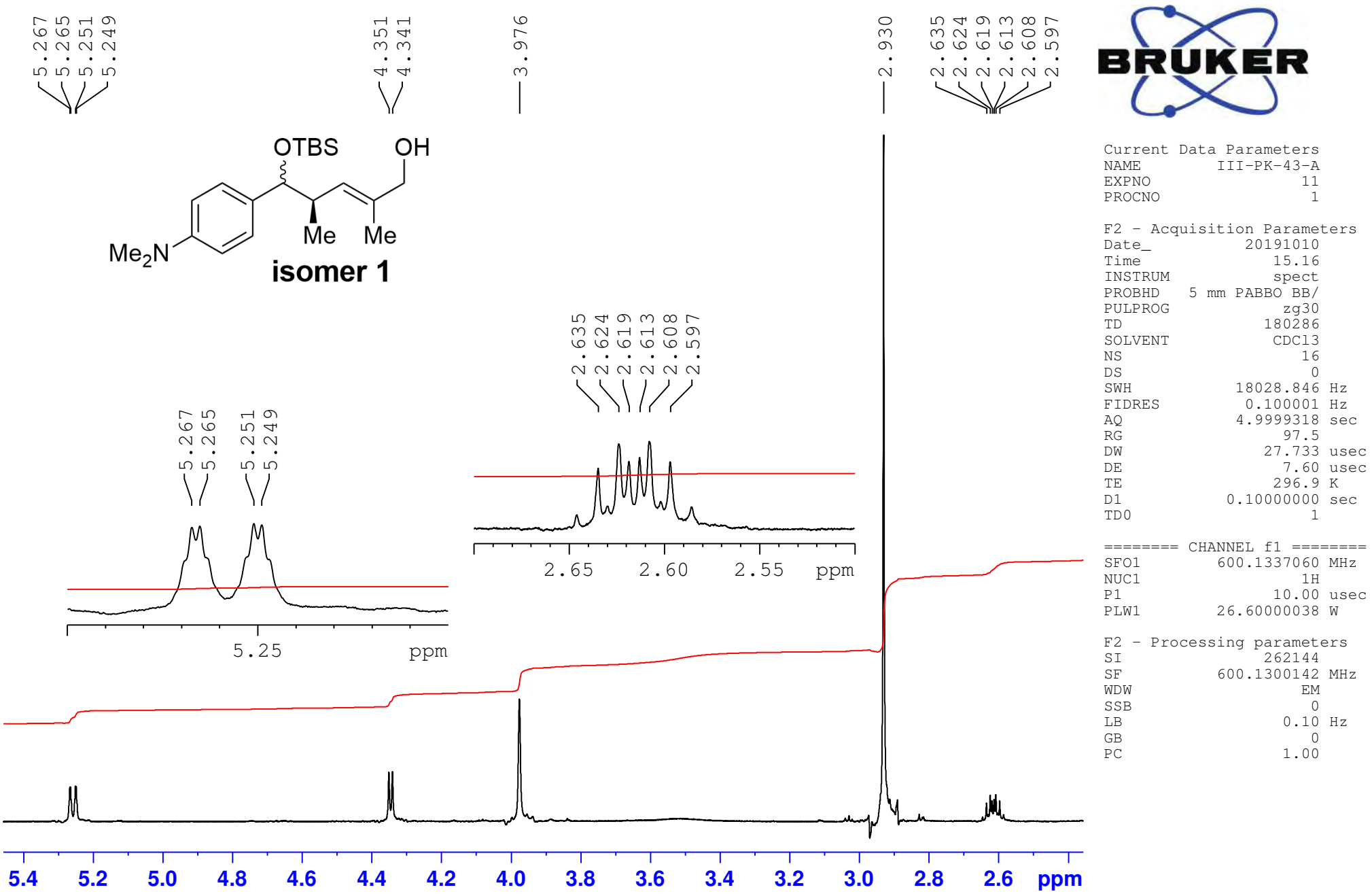
isomer 1


```
Current Data Parameters
NAME          III-PK-43-A
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                15.16
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             18028.846 Hz
FIDRES           0.100001 Hz
AQ               4.9999318 sec
RG                    97.5
DW                  27.733 usec
DE                   7.60 usec
TE                  296.9 K
D1             0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                    1H
P1                  10.00 usec
PLW1          26.60000038 W

F2 - Processing parameters
SI                    262144
SF            600.1300142 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```

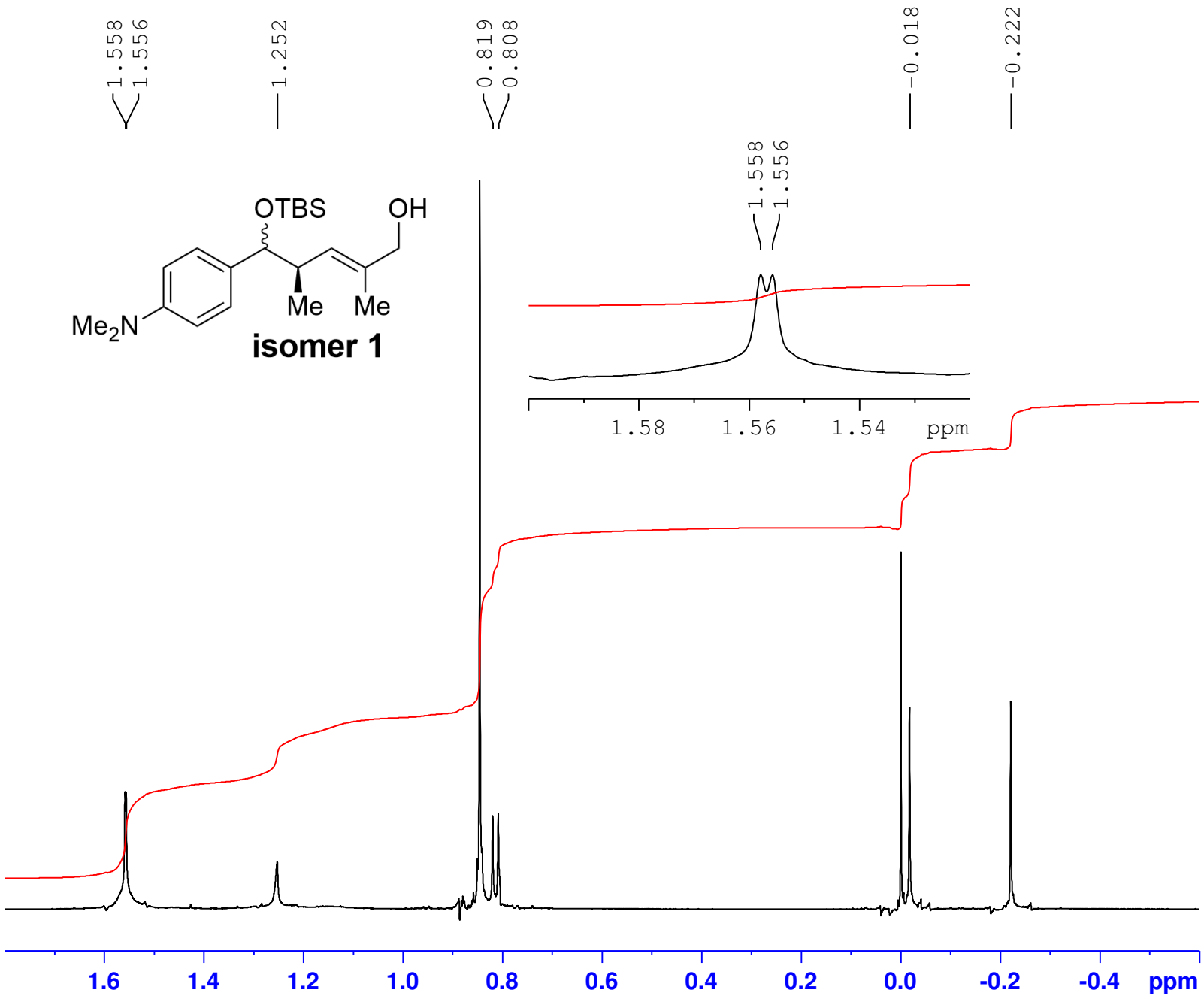



```
Current Data Parameters
NAME          III-PK-43-A
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                15.16
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             18028.846 Hz
FIDRES              0.100001 Hz
AQ               4.9999318 sec
RG                   97.5
DW                  27.733 usec
DE                   7.60 usec
TE                  296.9 K
D1              0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           600.1337060 MHz
NUC1                   1H
P1                  10.00 usec
PLW1           26.60000038 W

F2 - Processing parameters
SI                  262144
SF             600.1300142 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```



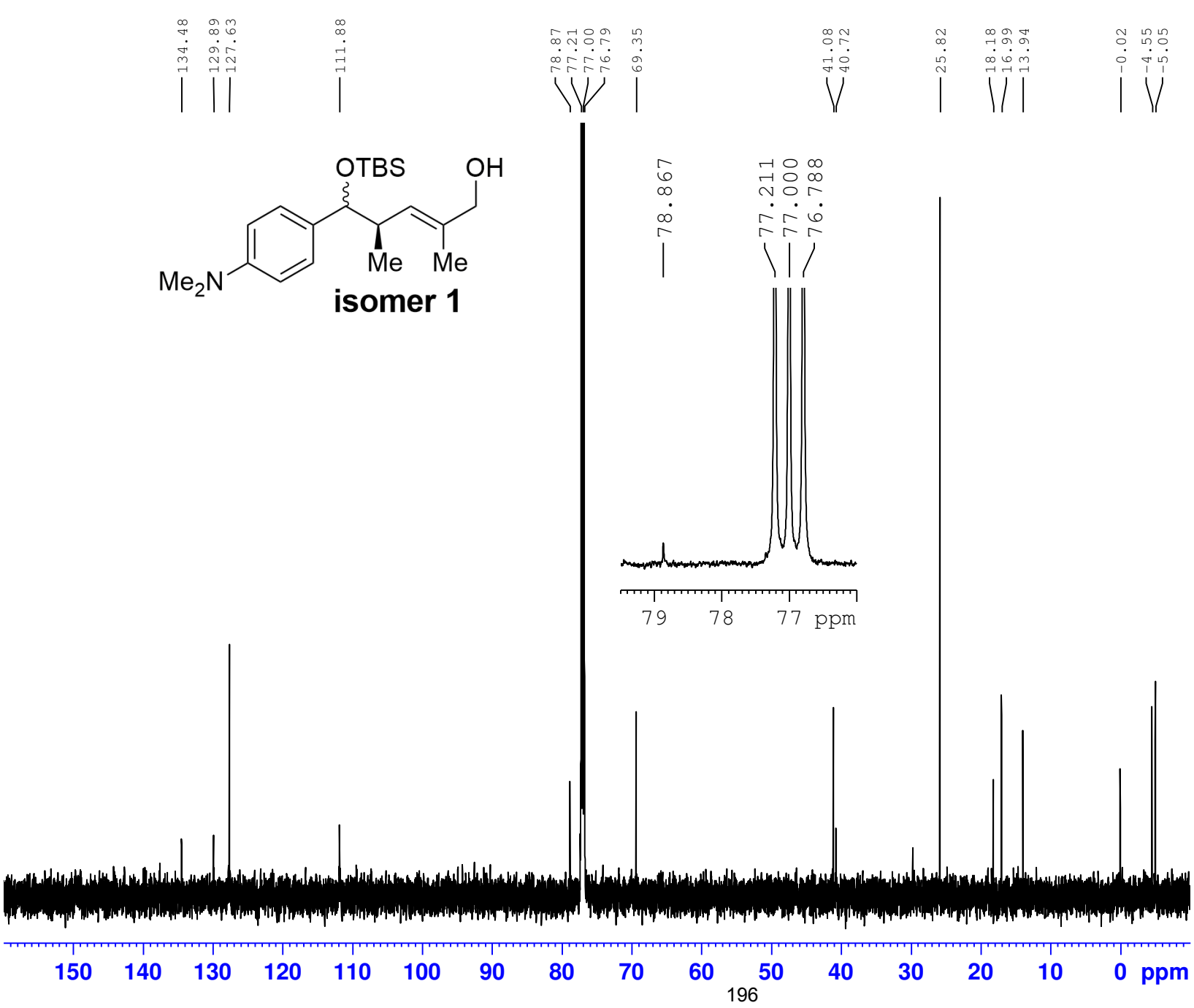
BRUKER

```
Current Data Parameters
NAME          III-PK-43-A
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                15.16
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             18028.846 Hz
FIDRES              0.100001 Hz
AQ               4.9999318 sec
RG                   97.5
DW                  27.733 usec
DE                   7.60 usec
TE                  296.9 K
D1              0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           600.1337060 MHz
NUC1                   1H
P1                  10.00 usec
PLW1           26.60000038 W

F2 - Processing parameters
SI                  262144
SF             600.1300142 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```



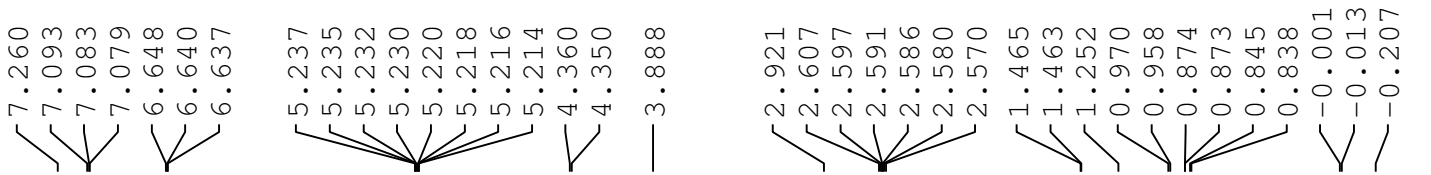
Current Data Parameters
NAME III-PK-43-A
EXPNO 12
PROCNO 1

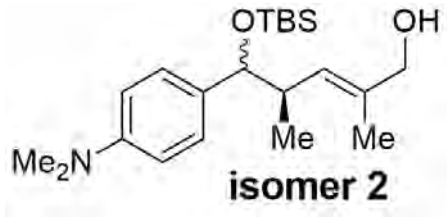
F2 - Acquisition Parameters
Date_ 20191010
Time 22.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028083 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



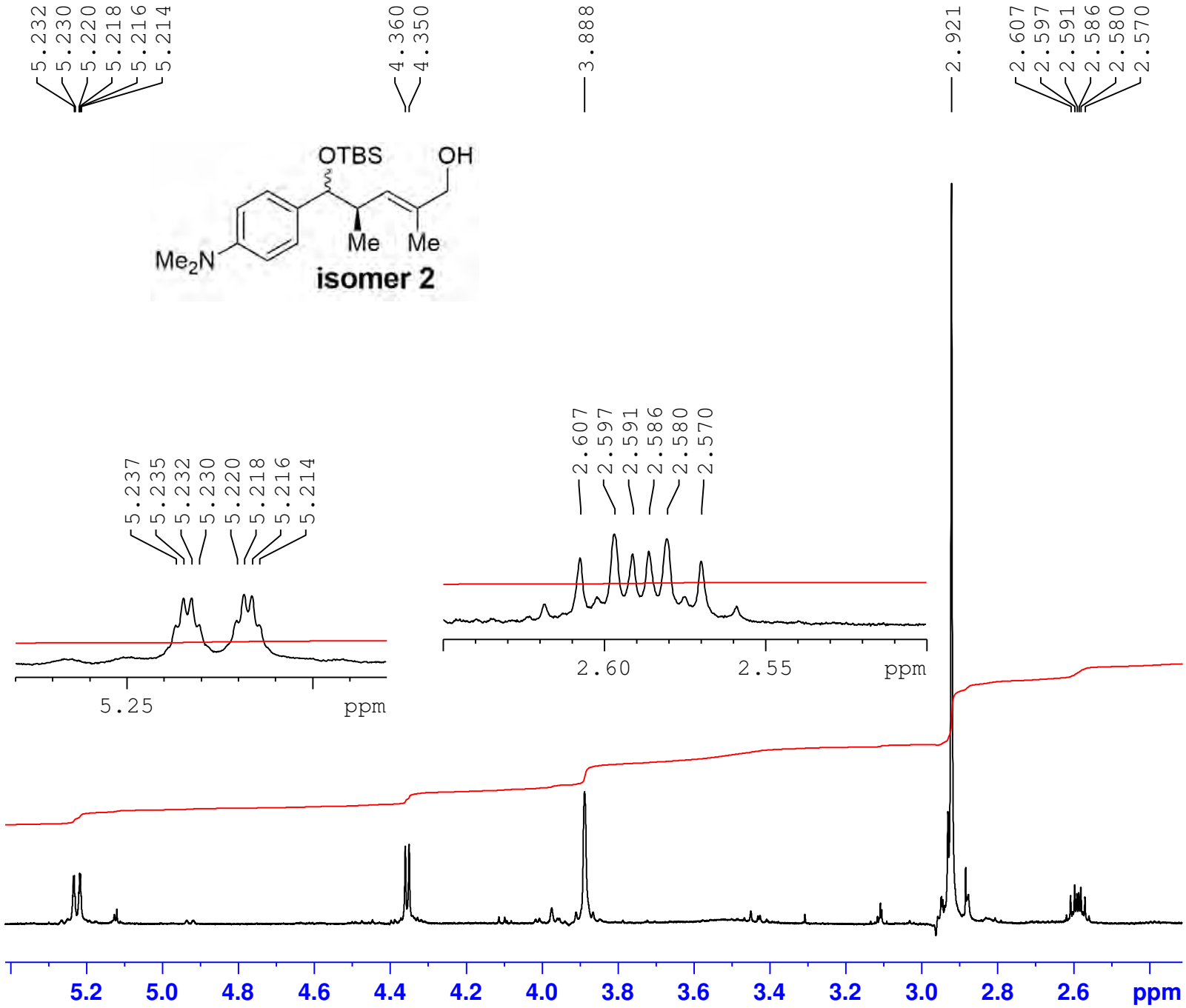



```
Current Data Parameters
NAME          III-PK-43-B
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                22.57
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             18028.846 Hz
FIDRES              0.100001 Hz
AQ                4.9999318 sec
RG                     97.5
DW                   27.733 usec
DE                    7.60 usec
TE                   297.3 K
D1              0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           600.1337060 MHz
NUC1                   1H
P1                  10.00 usec
PLW1           26.60000038 W

F2 - Processing parameters
SI                  262144
SF             600.1300143 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```



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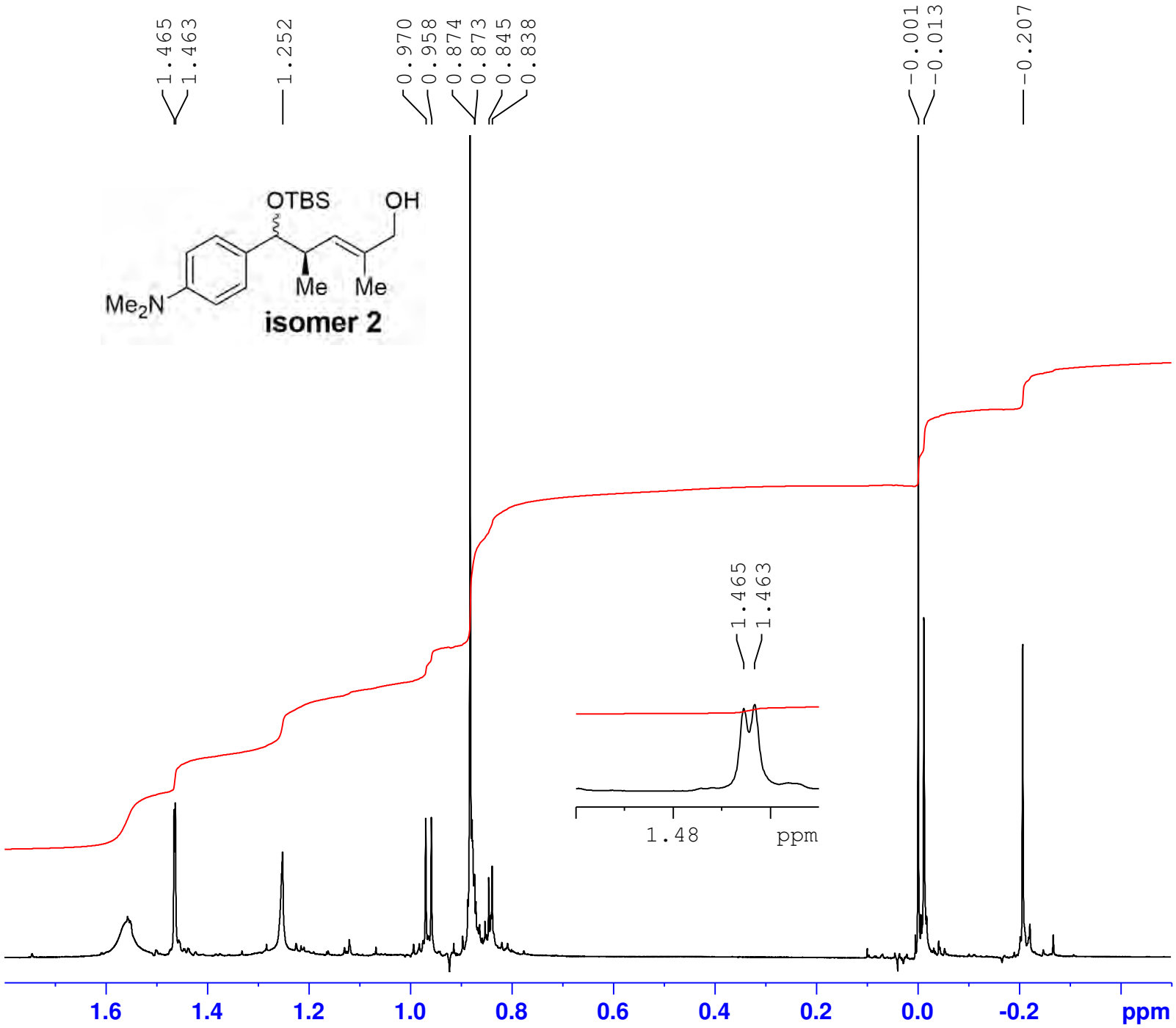
```
Current Data Parameters
NAME          III-PK-43-B
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                22.57
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH            18028.846 Hz
FIDRES          0.100001 Hz
AQ             4.9999318 sec
RG                  97.5
DW                27.733 usec
DE                 7.60 usec
TE                297.3 K
D1           0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                 262144
SF           600.1300143 MHz
WDW                    EM
SSB                     0
LB                 0.10 Hz
GB                      0
PC                 1.00
```



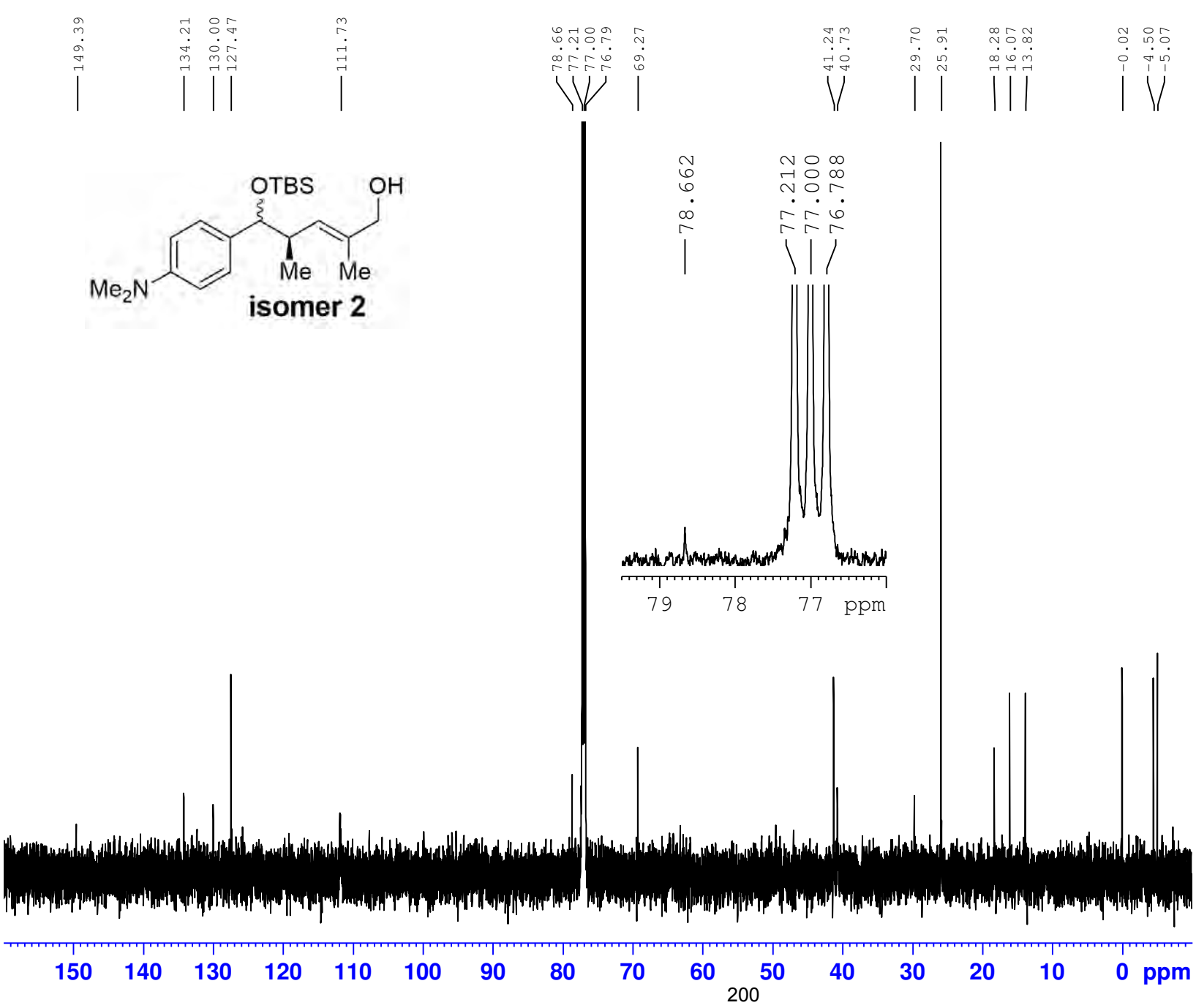
BRUKER

```
Current Data Parameters
NAME          III-PK-43-B
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20191010
Time                22.57
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                 180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             18028.846 Hz
FIDRES              0.100001 Hz
AQ               4.9999318 sec
RG                   97.5
DW                  27.733 usec
DE                   7.60 usec
TE                  297.3 K
D1             0.10000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                   1H
P1                  10.00 usec
PLW1          26.60000038 W

F2 - Processing parameters
SI                 262144
SF            600.1300143 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```



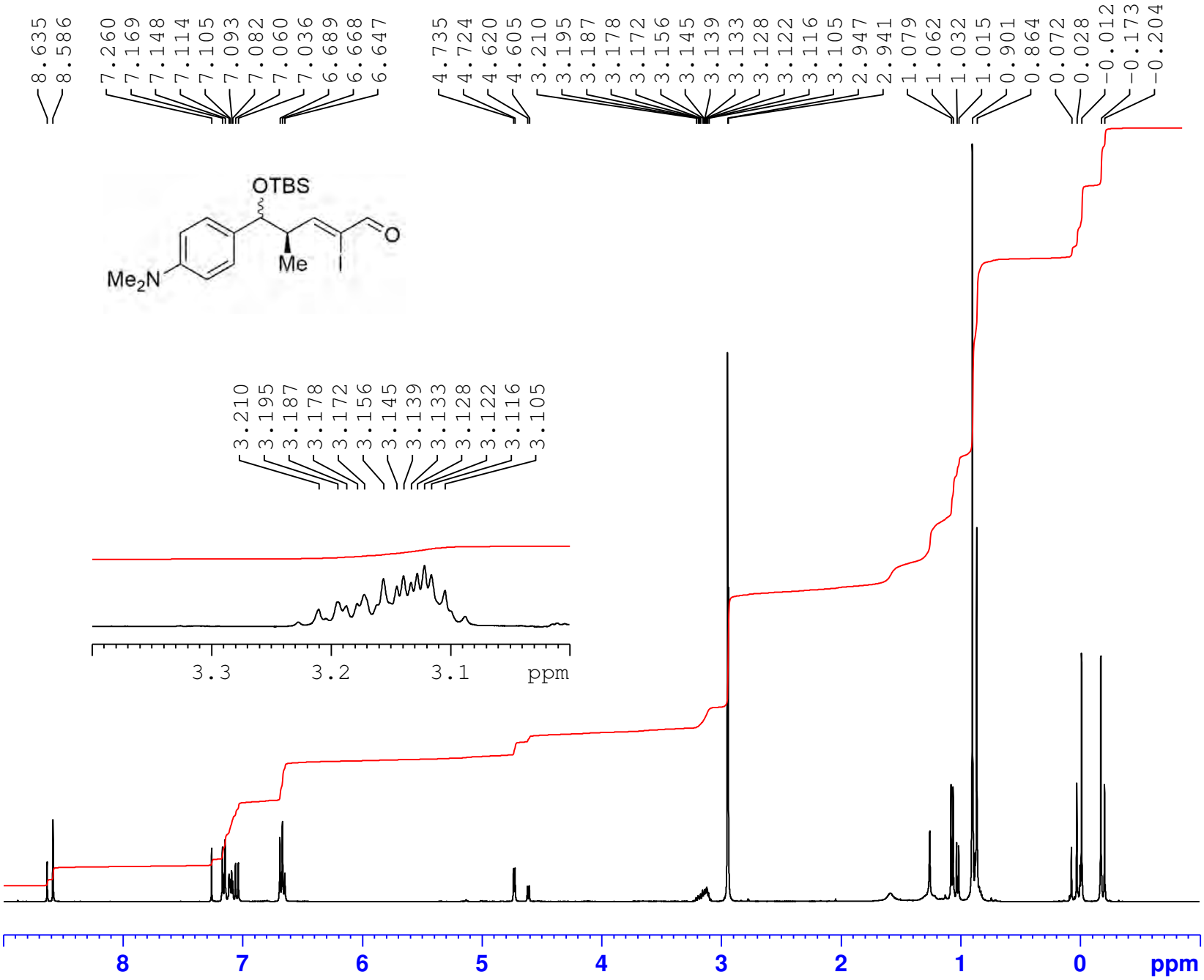
Current Data Parameters
NAME III-PK-43-B
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191010
Time 23.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 299.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028080 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

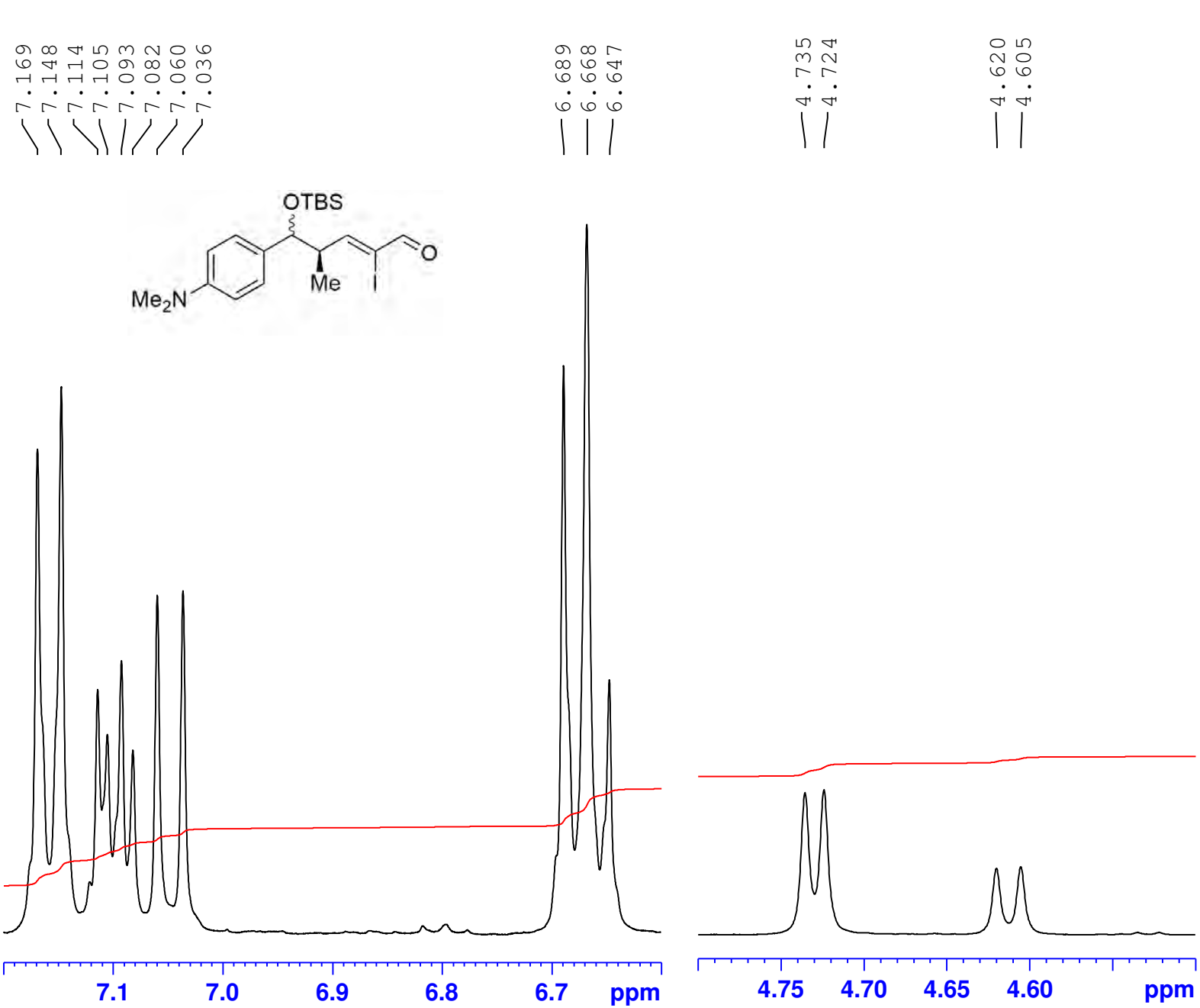


Current Data Parameters
NAME I-PK-77PURE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 71.8
DW 41.600 usec
DE 9.85 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

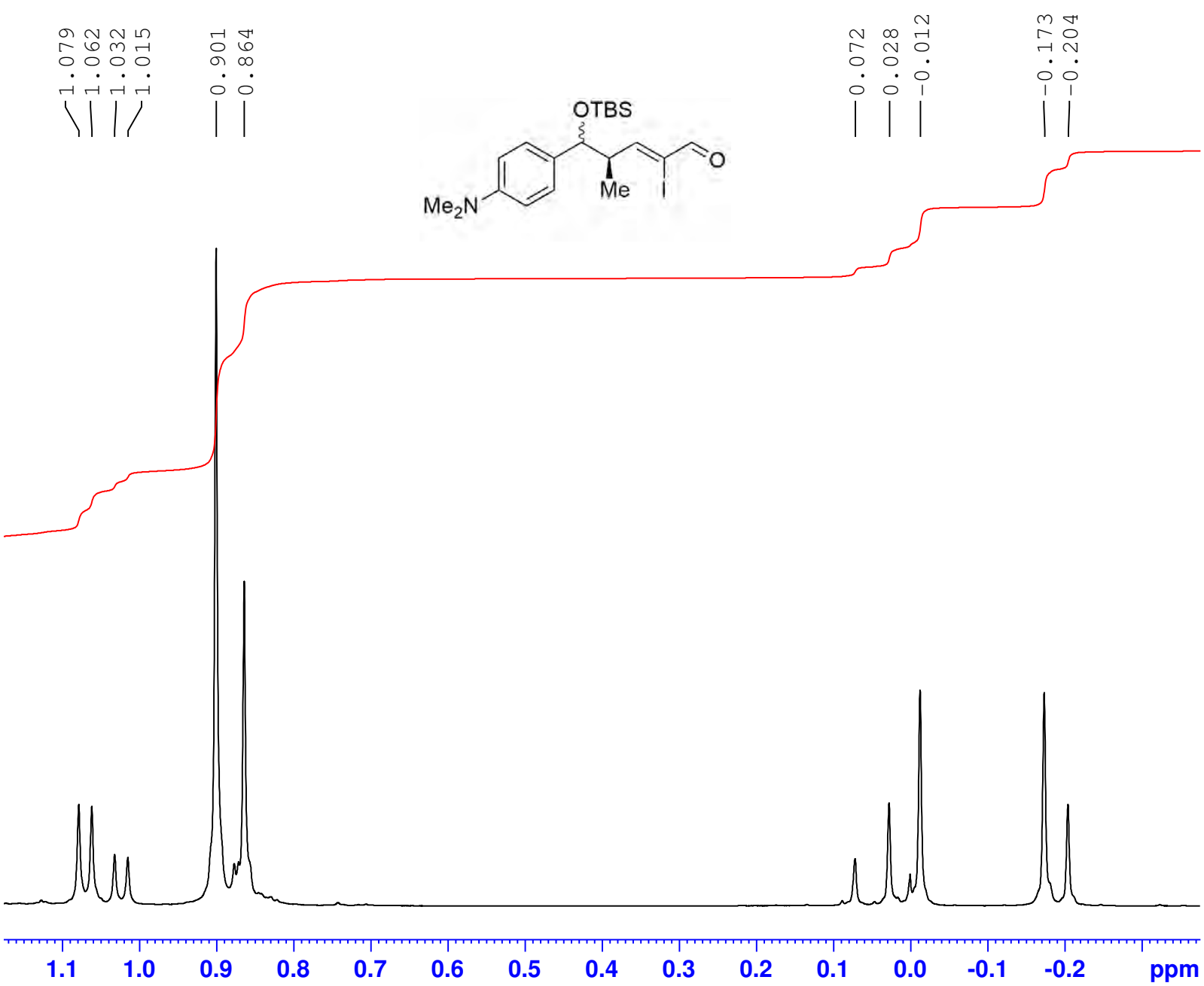



```
Current Data Parameters
NAME         I-PK-77PURE
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180517
Time               18.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    64
DS                     0
SWH           12019.230 Hz
FIDRES         0.091699 Hz
AQ            5.4525952 sec
RG                 71.8
DW                41.600 usec
DE                 9.85 usec
TE                300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                 1H
P1                14.88 usec
PLW1          7.59999990 W

F2 - Processing parameters
SI                131072
SF           399.9000095 MHz
WDW                  EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```



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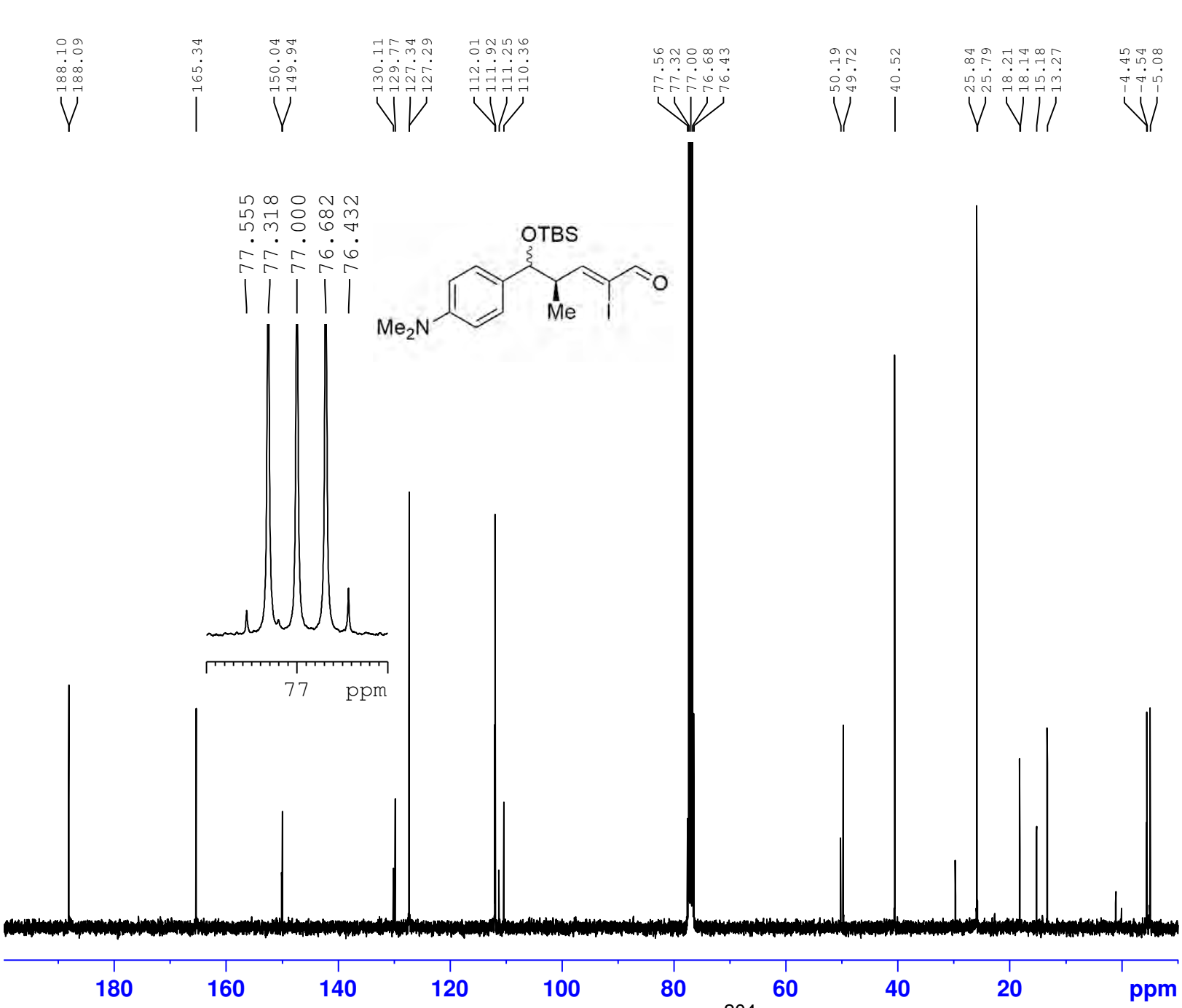
Current Data Parameters
NAME I-PK-77PURE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 64
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 71.8
DW 41.600 usec
DE 9.85 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



BRUKER

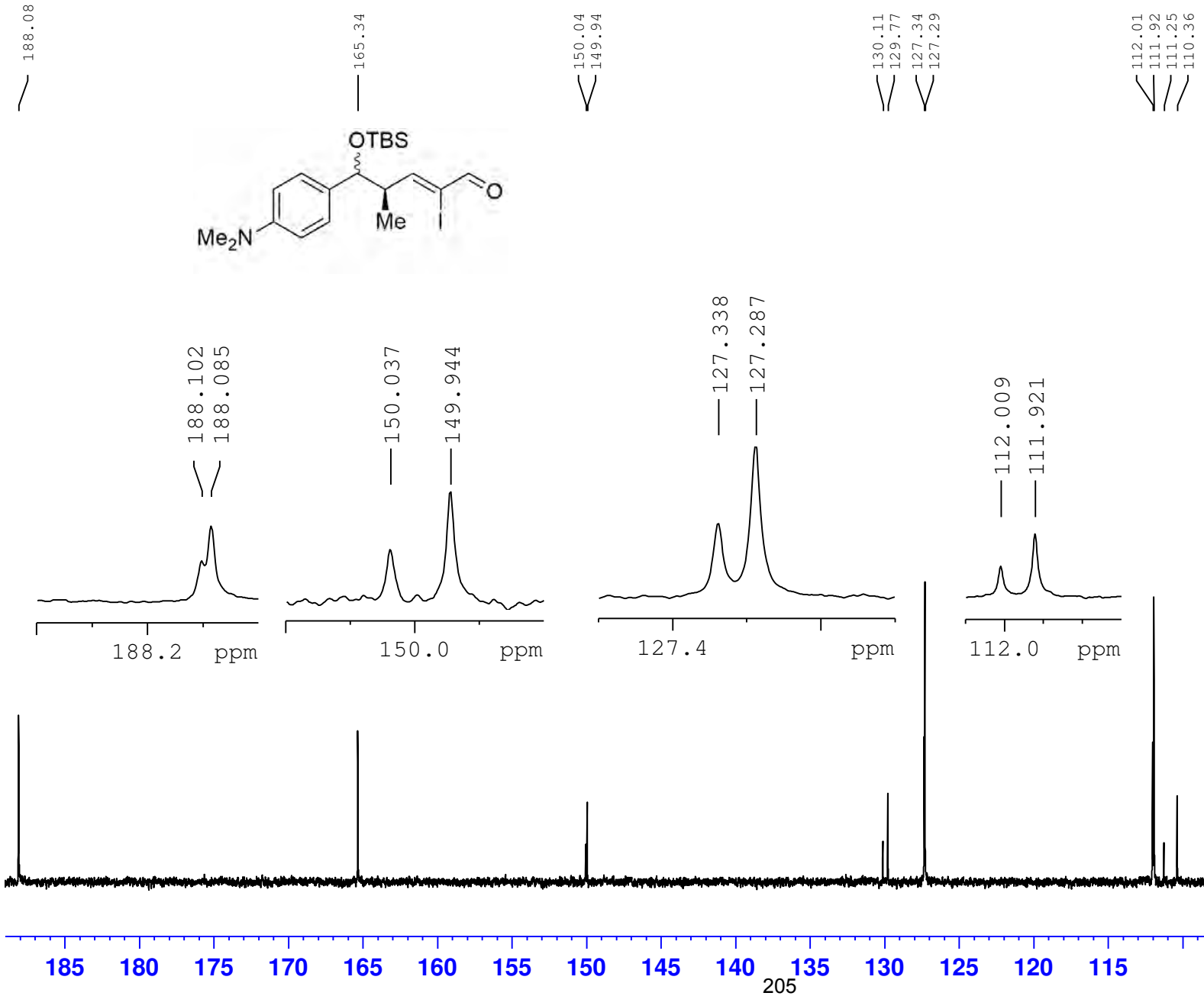
Current Data Parameters
NAME I-PK-77PURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 19.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549378 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



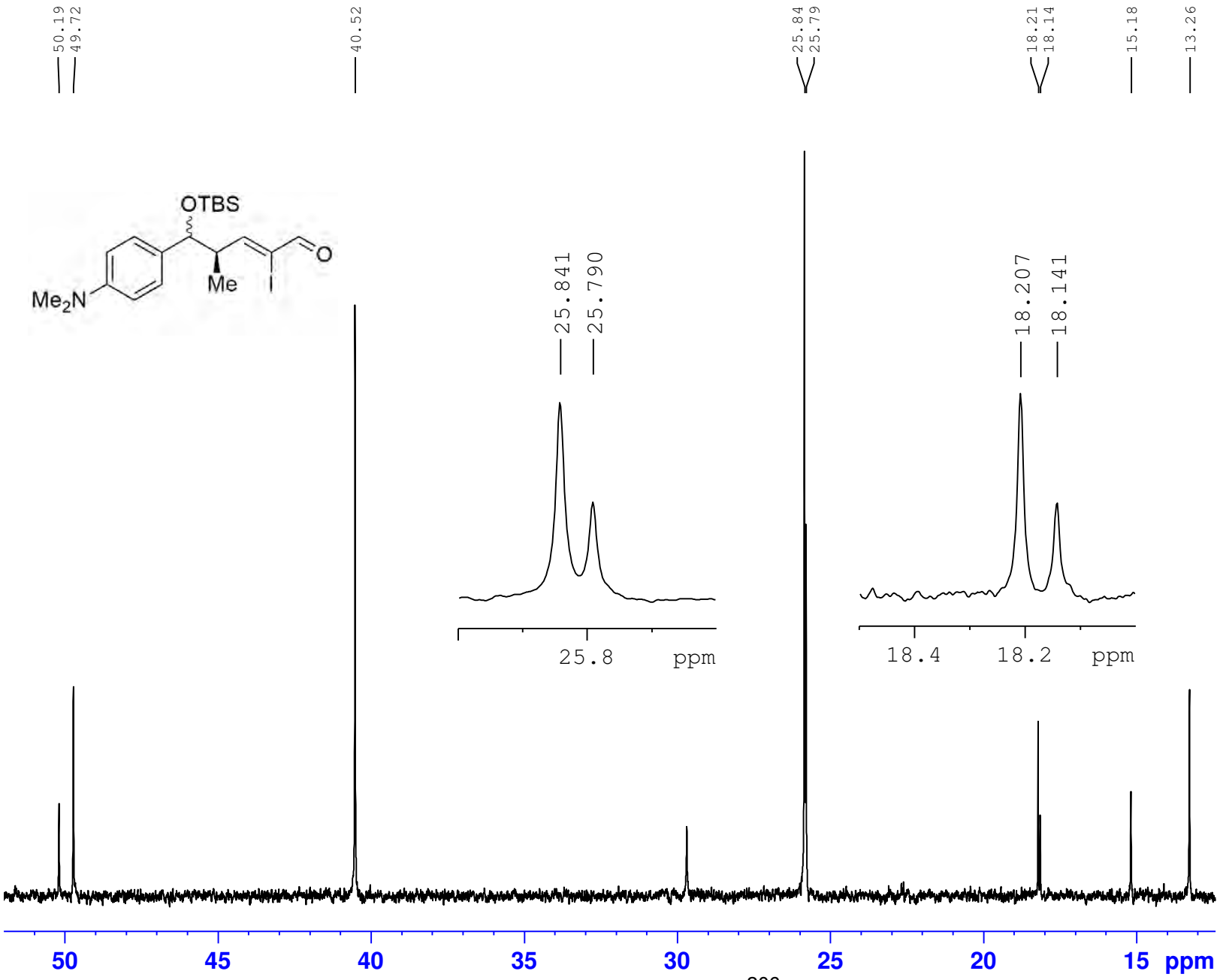
Current Data Parameters
NAME I-PK-77PURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 19.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549378 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



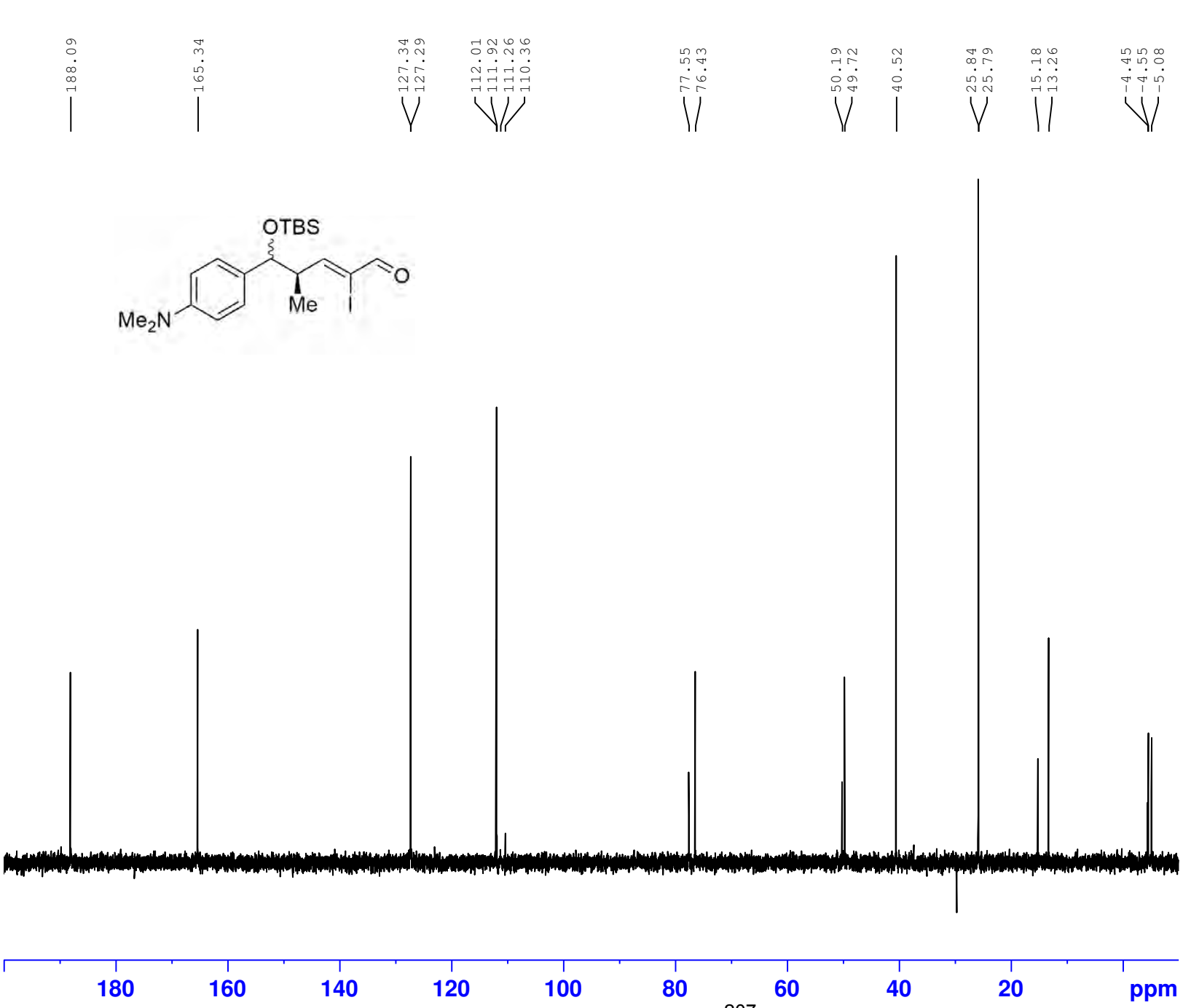
Current Data Parameters
NAME I-PK-77PURE
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 19.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549378 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



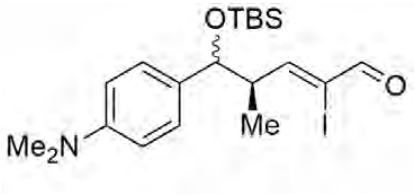
Current Data Parameters
NAME I-PK-77PURE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180517
Time 19.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549377 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                 I-PK-77PURE
EXPNO                         14
PROCNO                         1

F2 - Acquisition Parameters
Date_               20180517
Time_                   19.47
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                      1024
SOLVENT                 CDCl3
NS                         2
DS                        32
SWH                 4807.692 Hz
FIDRES              4.695012 Hz
AQ                  0.1064960 sec
RG                       2050
DW                    104.000 usec
DE                      6.50 usec
TE                     300.0 K
CNST2              145.0000000
D0                  0.00000300 sec
D1                  0.80000001 sec
D4                  0.00172414 sec
D11                 0.03000000 sec
D16                 0.00020000 sec
D21                 0.00360000 sec
IN0                 0.00001910 sec

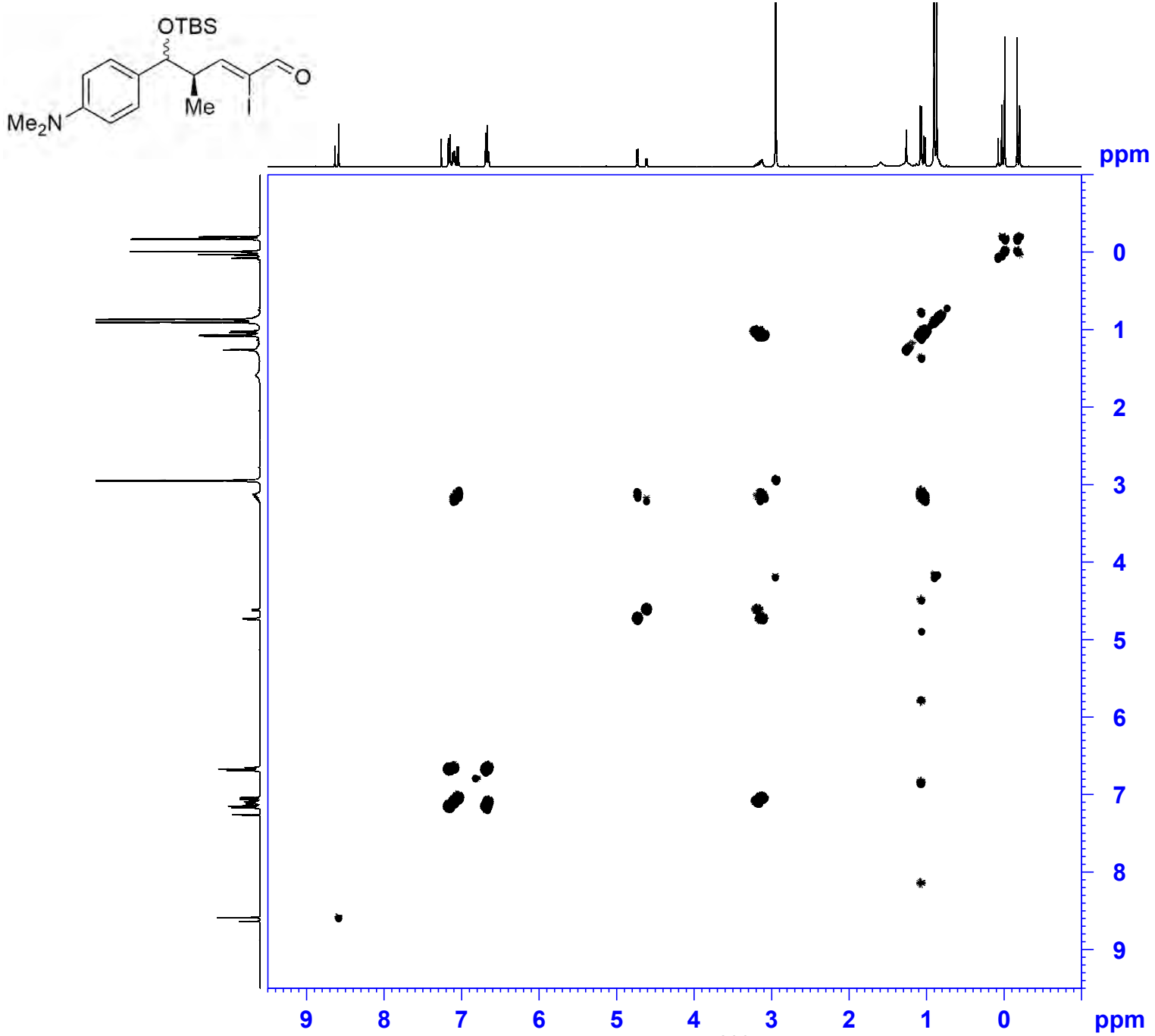
======== CHANNEL f1 ========
SFO1               399.9018806 MHz
NUC1                       1H
P1                       14.88 usec
P2                       29.76 usec
P28          0 usec
PLW1                7.59999990 W

======== CHANNEL f2 ========
SFO2               100.5670016 MHz
NUC2                      13C
CPDPRG[2                garp4
P3                       10.00 usec
P14                     500.00 usec
P31                    1900.00 usec
PCPD2                    80.00 usec
PLW0          0 W
PLW2               44.46300125 W
PLW12               0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                    0.500
SPOFFS3       0 Hz
SPW3                6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                   0.500
SPOFFS18 0 Hz
SPW18               1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPZ1                     80.00 %
GPZ2                     20.10 %
P16                    1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1                  100.567 MHz
FIDRES              204.515701 Hz
SW                    260.304 ppm
FnMODE        Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF                  399.9000000 MHz
```




```
Current Data Parameters
NAME          I-PK-77PURE
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180517
Time                19.40
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                  2048
SOLVENT             CDCl3
NS                     1
DS                     8
SWH             4201.681 Hz
FIDRES          2.051602 Hz
AQ              0.2437120 sec
RG                  2050
DW               119.000 usec
DE                 6.50 usec
TE                300.0 K
D0            0.00000300 sec
D1            0.90497202 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00023800 sec

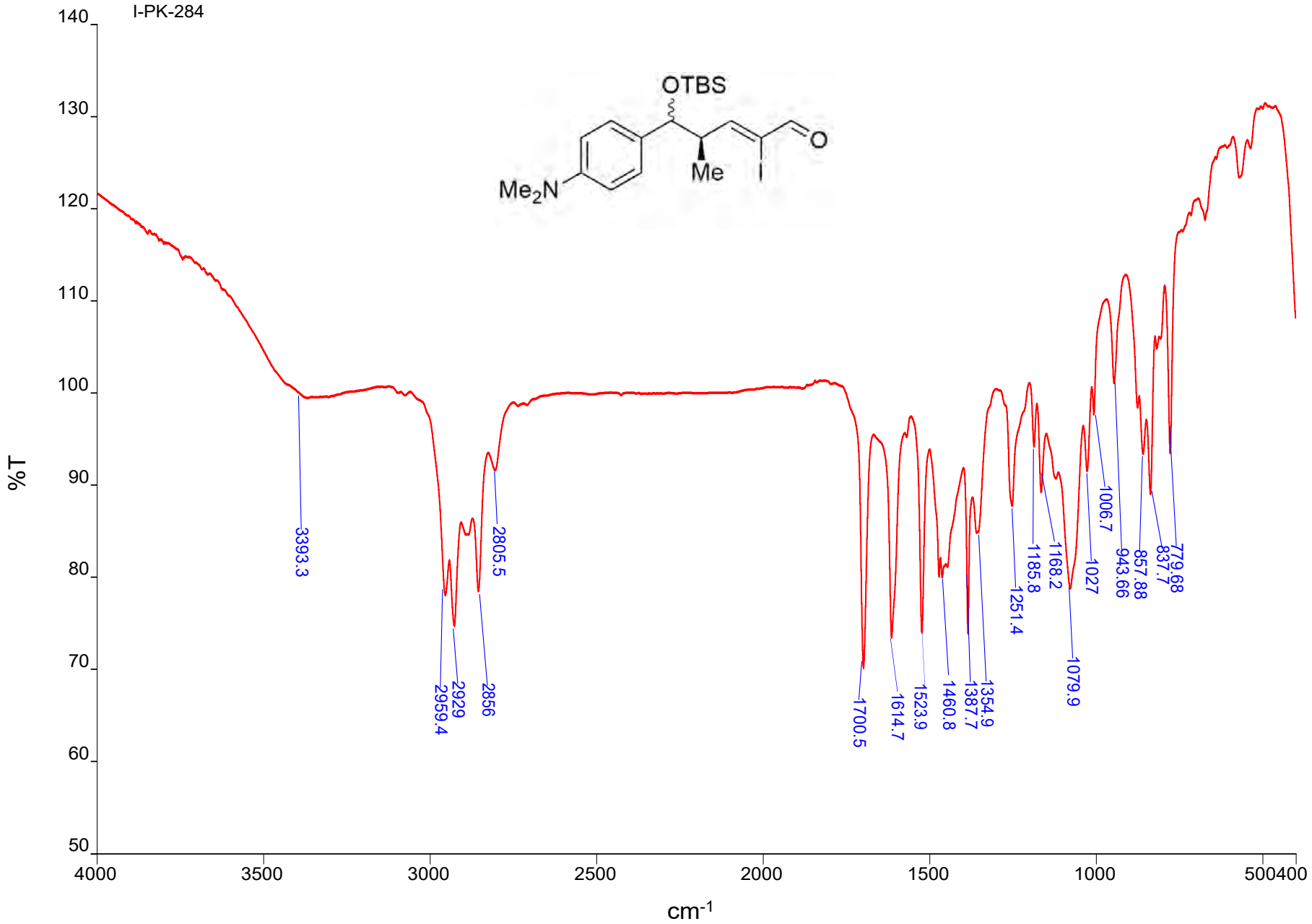
======== CHANNEL f1 ========
SFO1         399.9016818 MHz
NUC1                   1H
P1                14.88 usec
P17             2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1               16.00 %
GPZ2               12.00 %
GPZ3               40.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1            399.9017 MHz
FIDRES         32.825630 Hz
SW                10.507 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           399.9000095 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           399.9000110 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```


I-PK-284
OTBS
Me
O
I
Me_2N
%T
140
130
120
110
100
90
80
70
60
50
4000
3500
3000
2500
2000
1500
1000
500400
cm^{-1}
3393.3
2959.4
2929
2856
2805.5
1700.5
1614.7
1523.9
1460.8
1387.7
1354.9
1251.4
1185.8
1168.2
1079.9
1027
1006.7
943.66
857.88
837.7
779.68

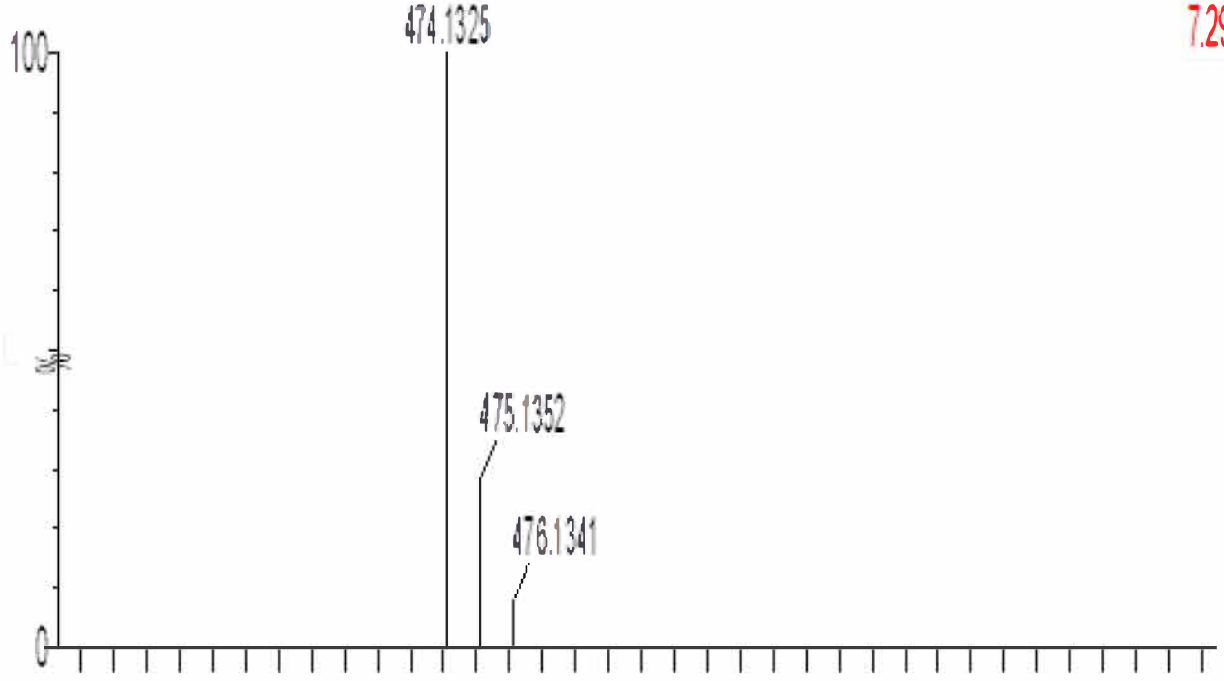
I-PK-77

10-05-2018

asep_16APR_2018_152 (0.070) Is (1.00,1.00) C20H32NSiO2IH

1: TOF MS ES+

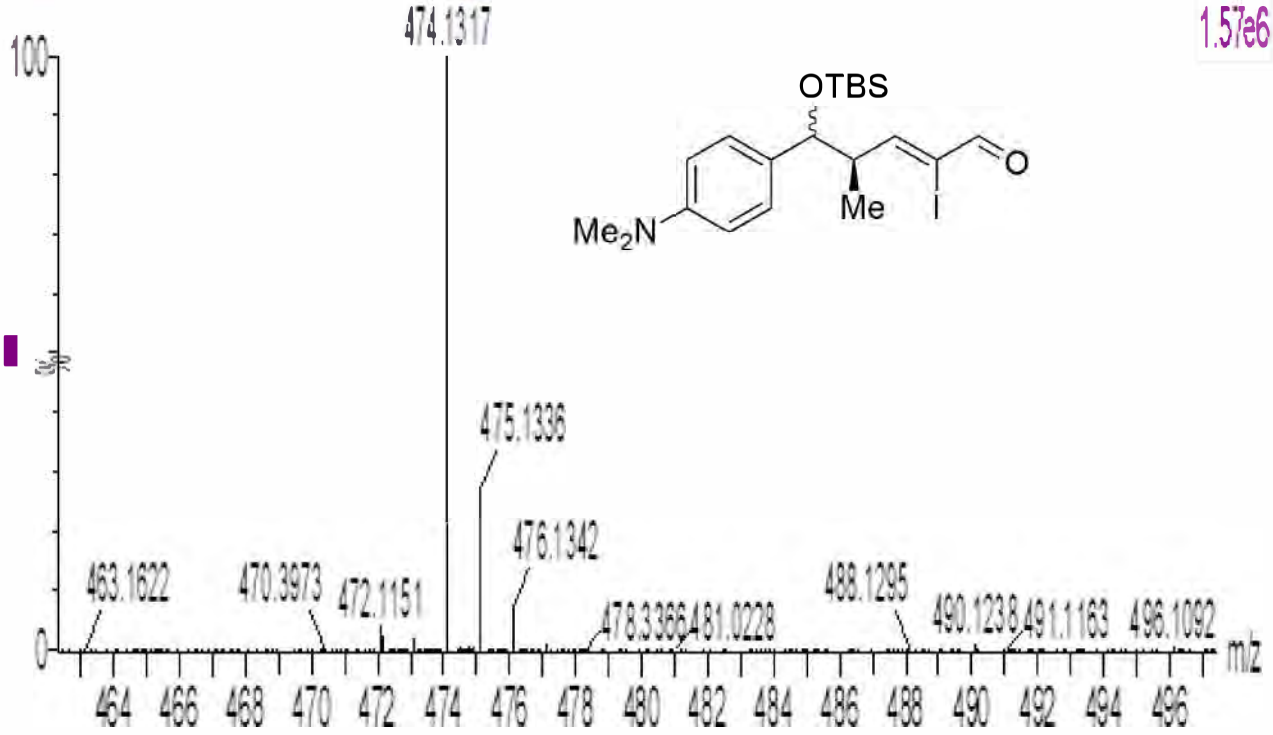
7.29e12



asep_16APR_2018_152 15 (0.675) Cm (13:20)

1: TOF MS ES+

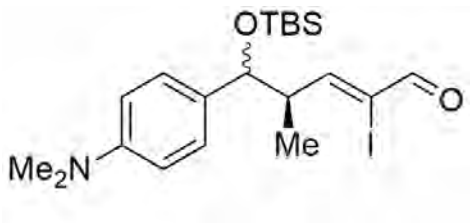
1.57e6

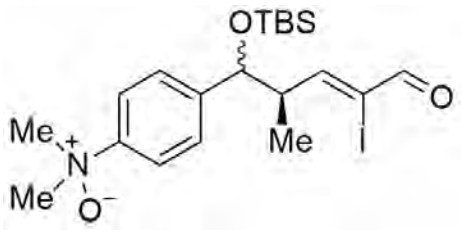


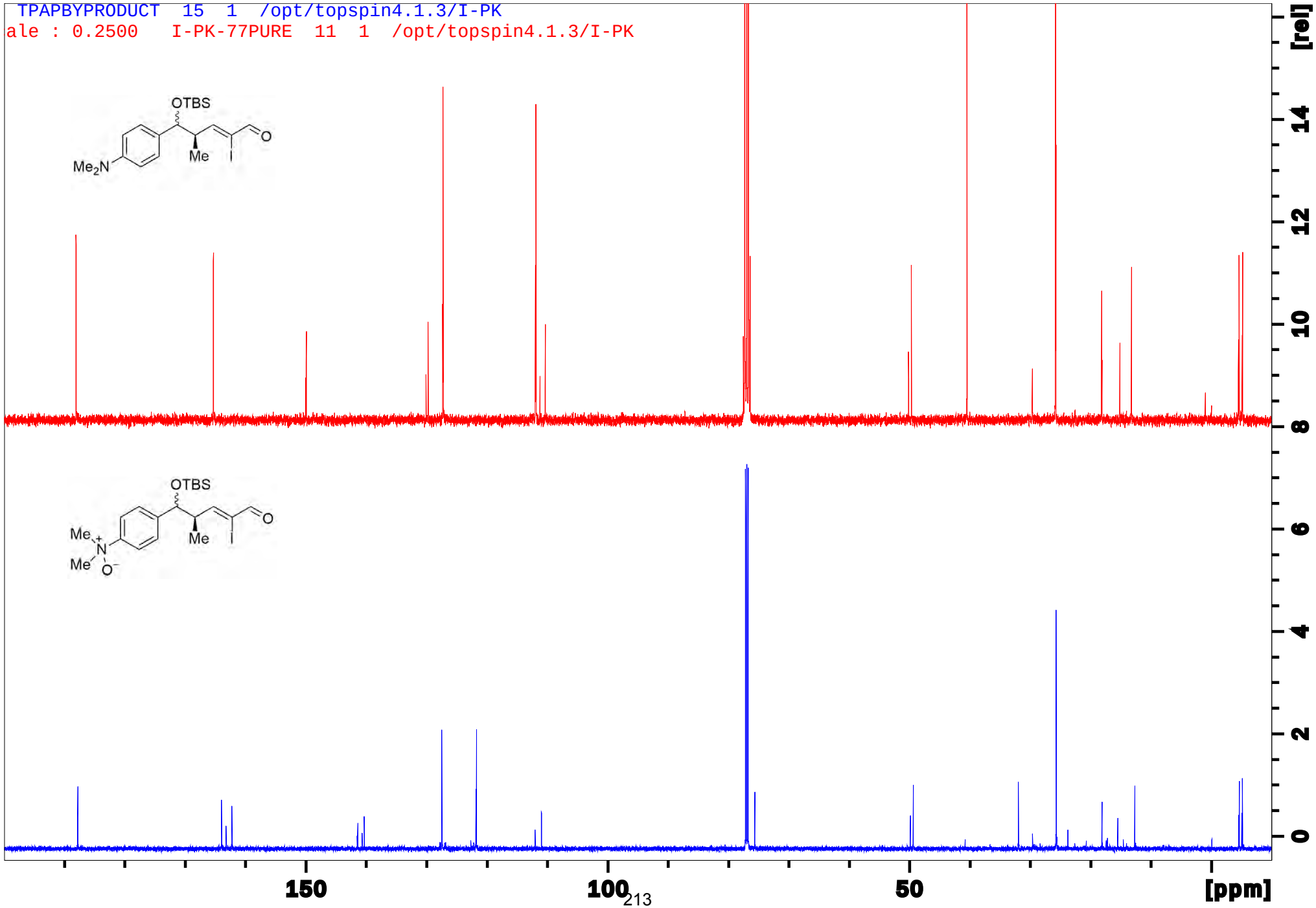
TPAPBYPRODUCT 10 1 /opt/topspin4.1.3/I-PK

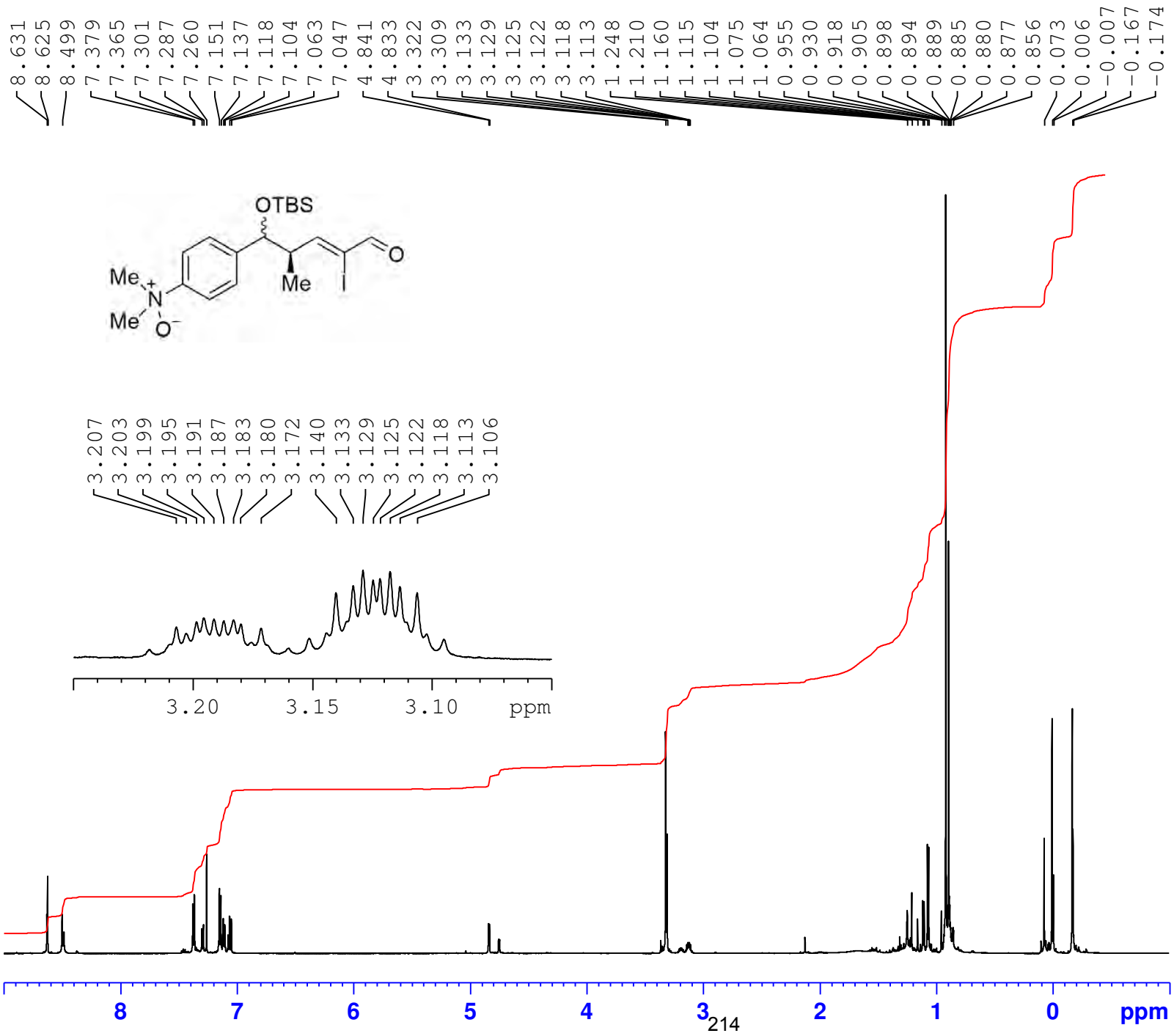
ale : 1.682 TPAPBYPRODUCT 10 1 /opt/topspin4.1.3/I-PK

ale : 0.4204 I-PK-77PURE 10 1 /opt/topspin4.1.3/I-PK






TPAPBYPRODUCT 15 1 /opt/topspin4.1.3/I-PK
TPAPBYPRODUCT 15 1 /opt/topspin4.1.3/I-PK
ale : 0.2500 I-PK-77PURE 11 1 /opt/topspin4.1.3/I-PK
OTBS
Me
O
Me2N
OTBS
Me
O
Me
N
Me
O
[rel]
14
12
10
8
6
4
2
0
150
100
50
[ppm]



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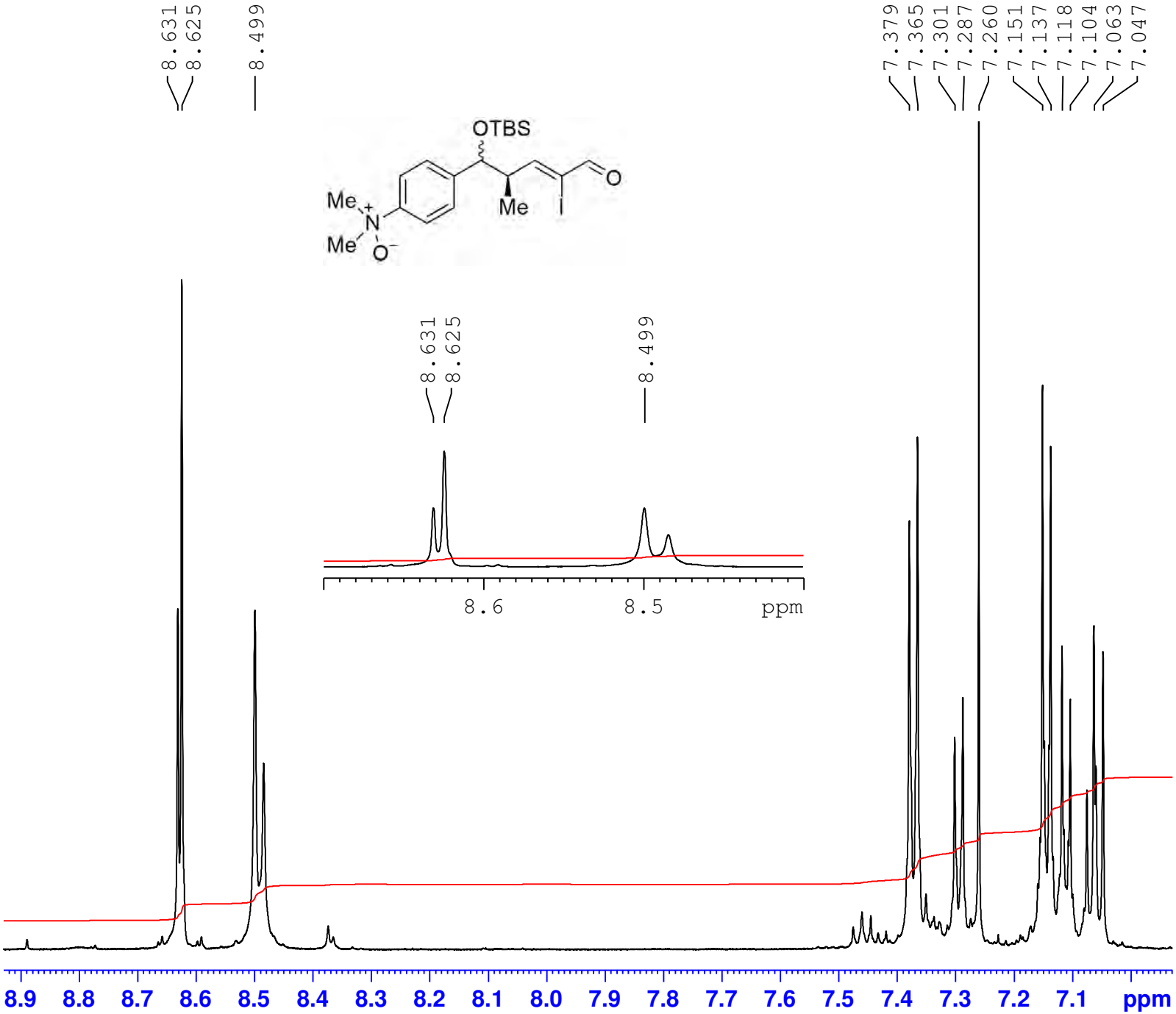
```
Current Data Parameters
NAME       TPAPBYPRODUCT
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190924
Time               12.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             CDCl3
NS                     16
DS                      0
SWH            18028.846 Hz
FIDRES           0.100001 Hz
AQ             4.9999318 sec
RG                  97.5
DW                27.733 usec
DE                 7.60 usec
TE                300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF         600.1300145 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



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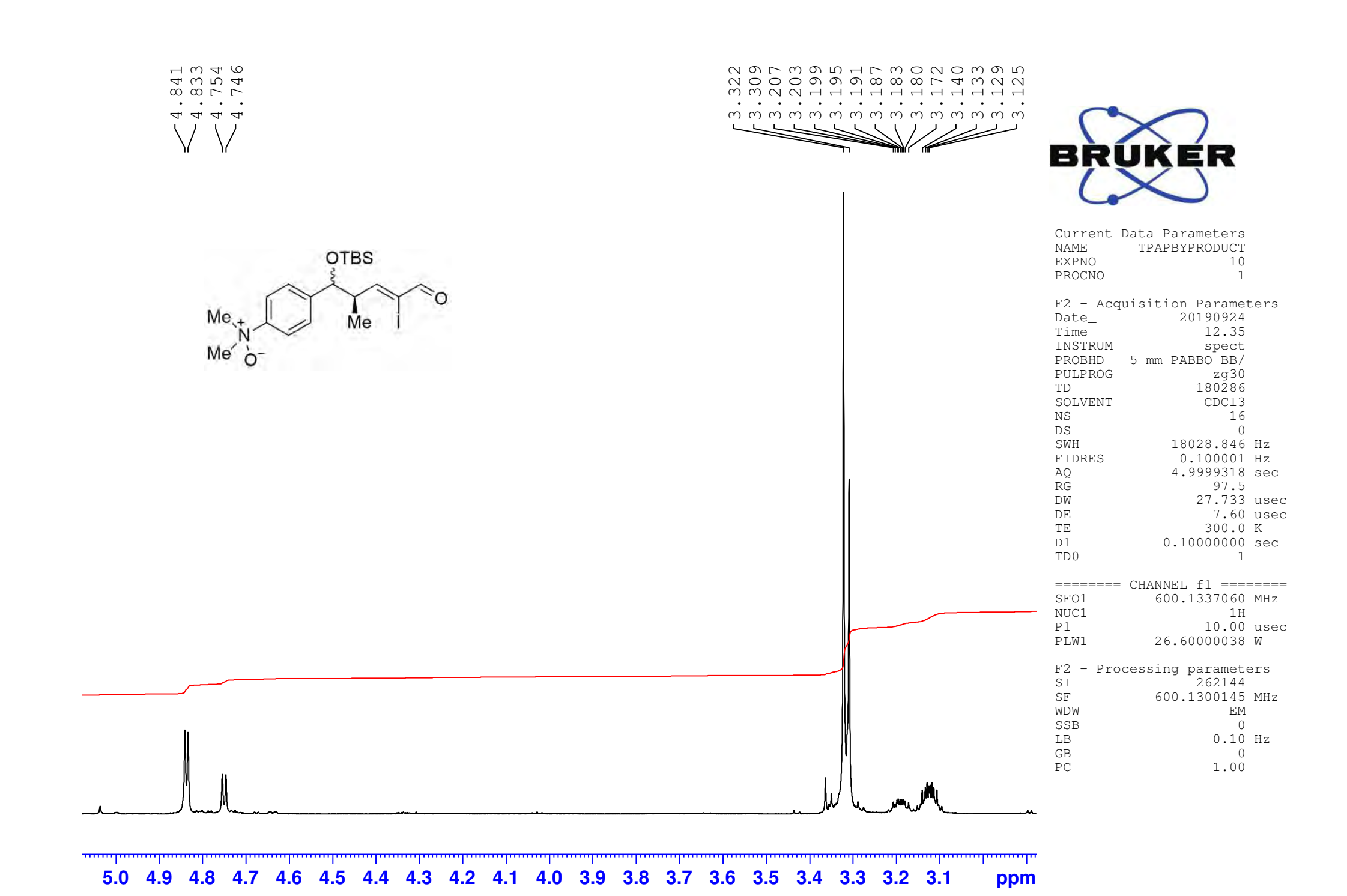
Current Data Parameters
NAME TPAPBYPRODUCT
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190924
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 180286
SOLVENT CDCl3
NS 16
DS 0
SWH 18028.846 Hz
FIDRES 0.100001 Hz
AQ 4.9999318 sec
RG 97.5
DW 27.733 usec
DE 7.60 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.60000038 W

F2 - Processing parameters
SI 262144
SF 600.1300145 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

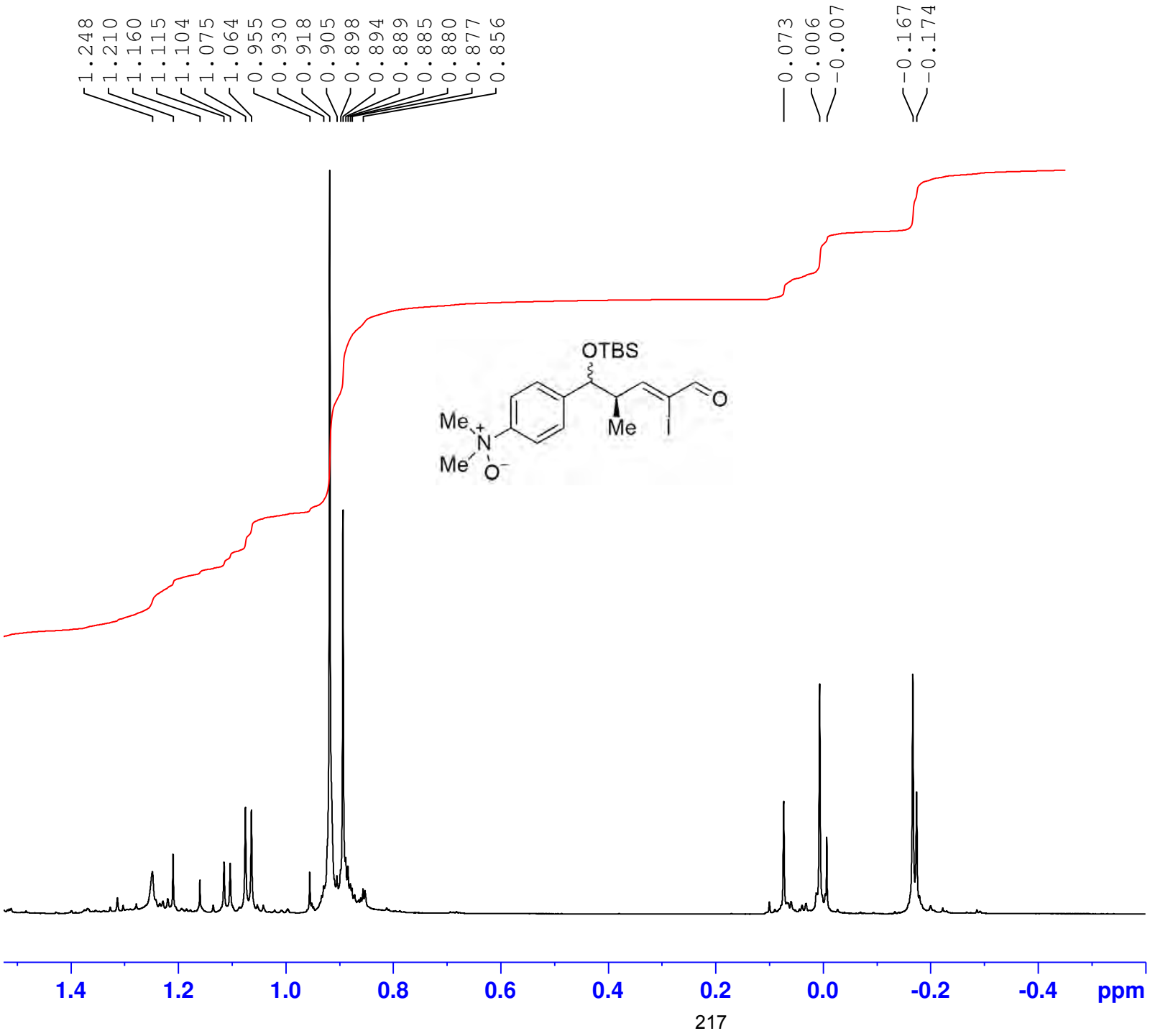


Current Data Parameters
NAME TPAPBYPRODUCT
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190924
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 180286
SOLVENT CDCl3
NS 16
DS 0
SWH 18028.846 Hz
FIDRES 0.100001 Hz
AQ 4.9999318 sec
RG 97.5
DW 27.733 usec
DE 7.60 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.60000038 W

F2 - Processing parameters
SI 262144
SF 600.1300145 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

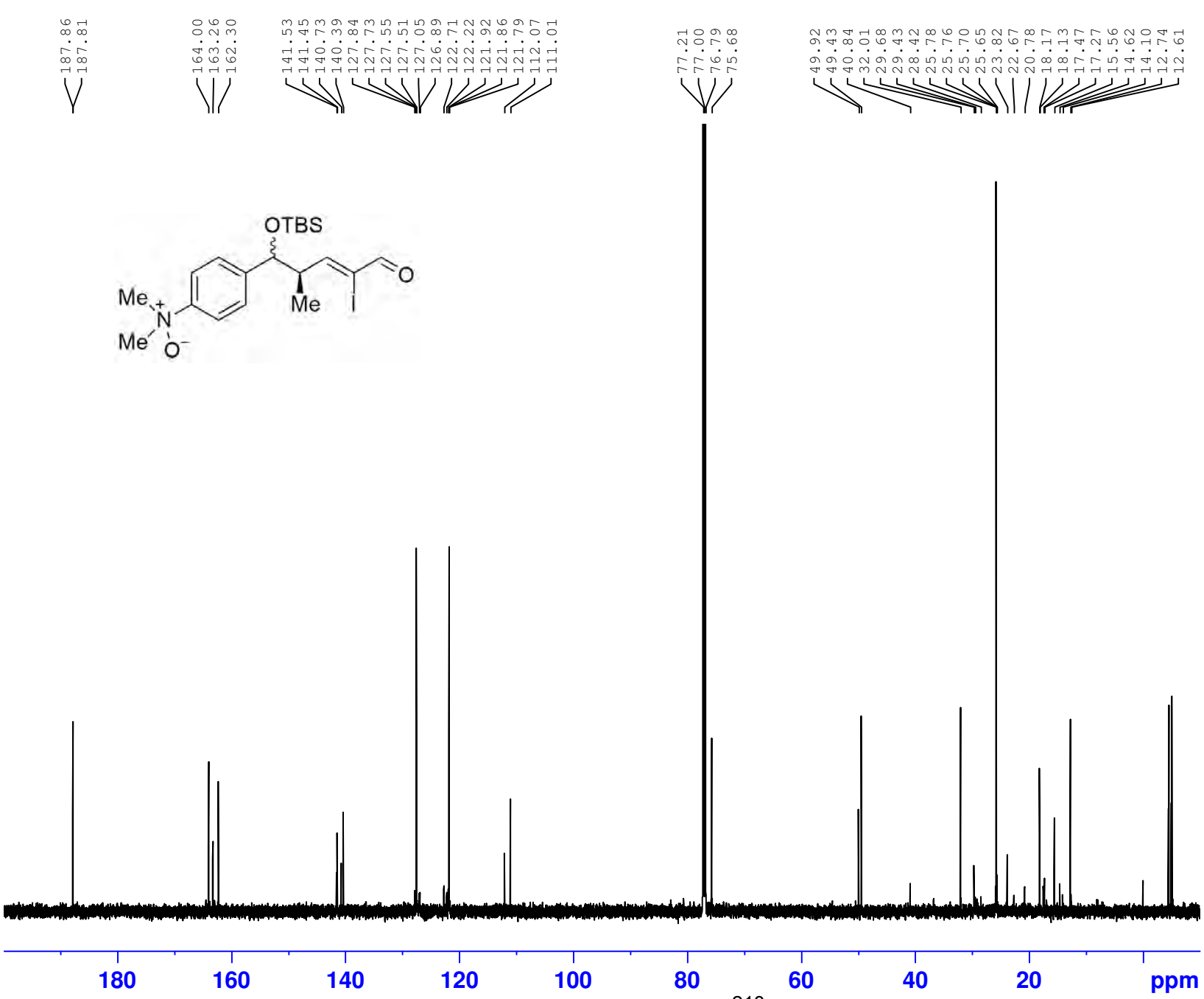


Current Data Parameters
NAME TPAPBYPRODUCT
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190924
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 180286
SOLVENT CDCl3
NS 16
DS 0
SWH 18028.846 Hz
FIDRES 0.100001 Hz
AQ 4.9999318 sec
RG 97.5
DW 27.733 usec
DE 7.60 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.60000038 W

F2 - Processing parameters
SI 262144
SF 600.1300145 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



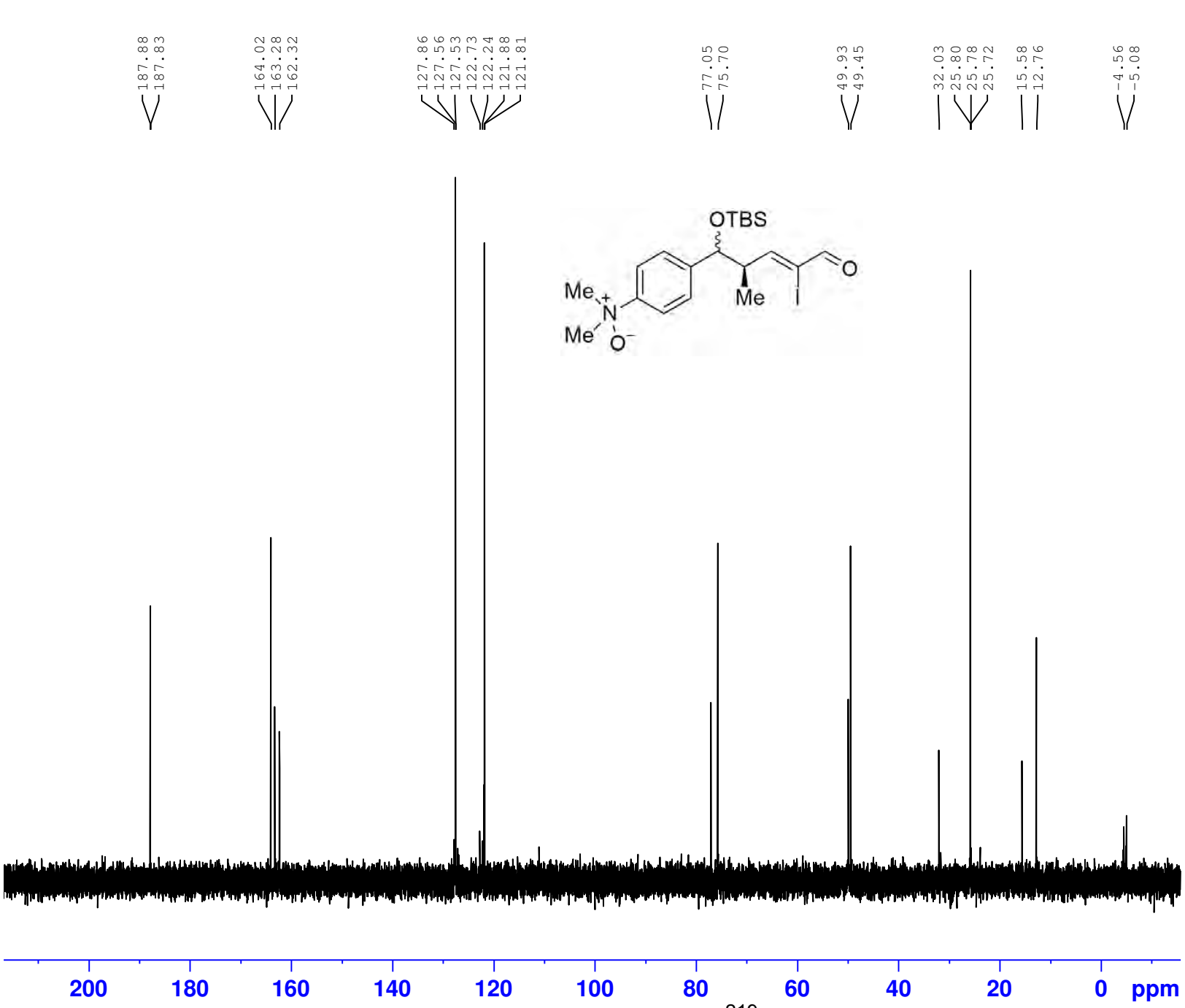
Current Data Parameters
NAME TPAPBYPRODUCT
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190924
Time 15.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	TPAPBYPRODUCT
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20190924
Time	15.59
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135.b
TD	119044
SOLVENT	CDCl3
NS	256
DS	4
SWH	35714.285 Hz
FIDRES	0.300009 Hz
AQ	1.6666160 sec
RG	186.92
DW	14.000 usec
DE	7.44 usec
TE	300.0 K
CNST2	145.0000000
D1	1.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

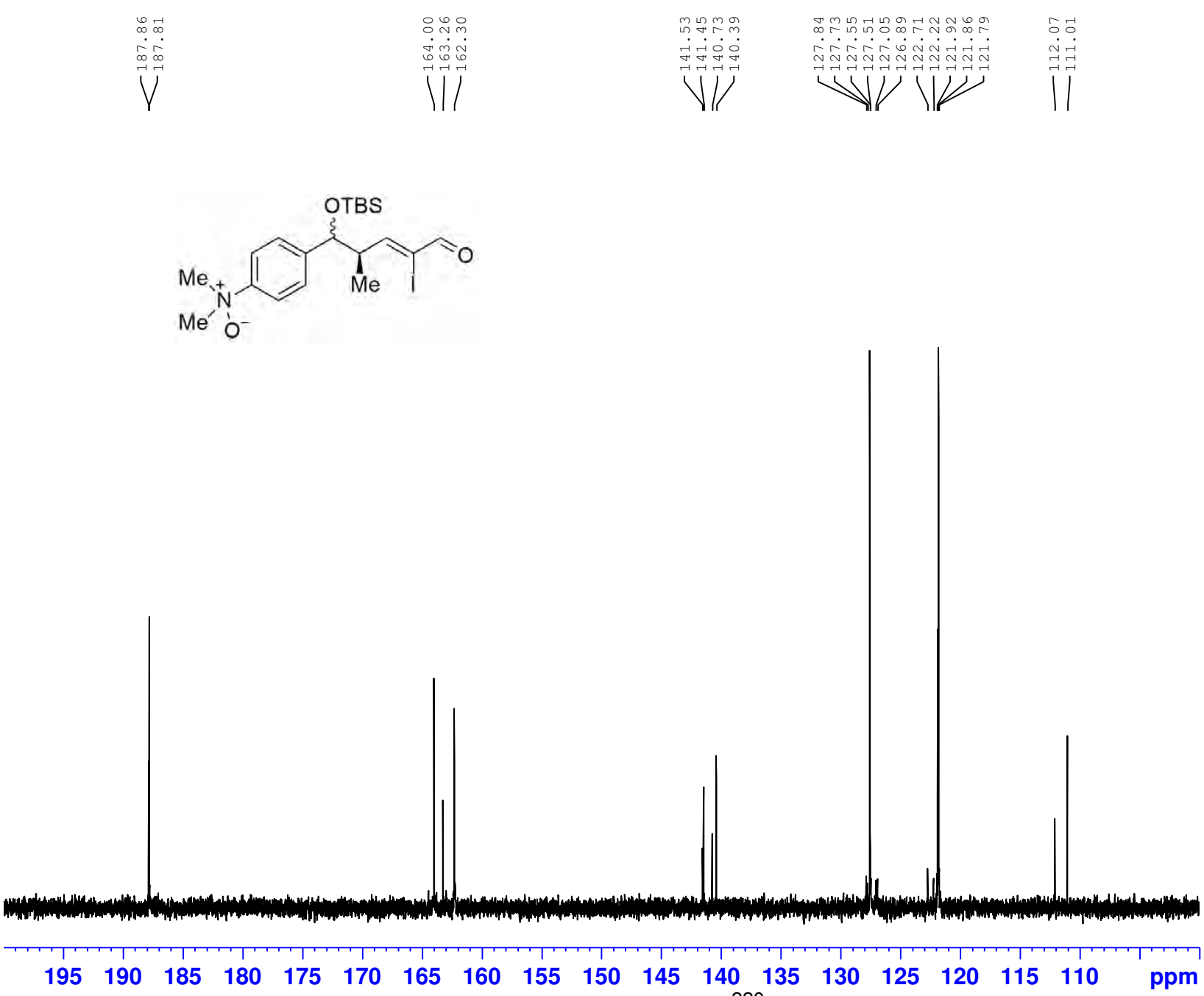
SFO1	150.9178962 MHz
NUC1	13C
P1	11.80 usec
P13	2000.00 usec
PLW0	0 W
PLW1	85.00000000 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	18.08300018 W

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

SFO2	600.1324005 MHz
NUC2	1H
CPDPRG[2	waltz64
P3	10.20 usec
P4	20.40 usec
PCPD2	80.00 usec
PLW2	27.00000000 W
PLW12	0.43891999 W

F2 - Processing parameters

SI	131072
SF	150.9028085 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



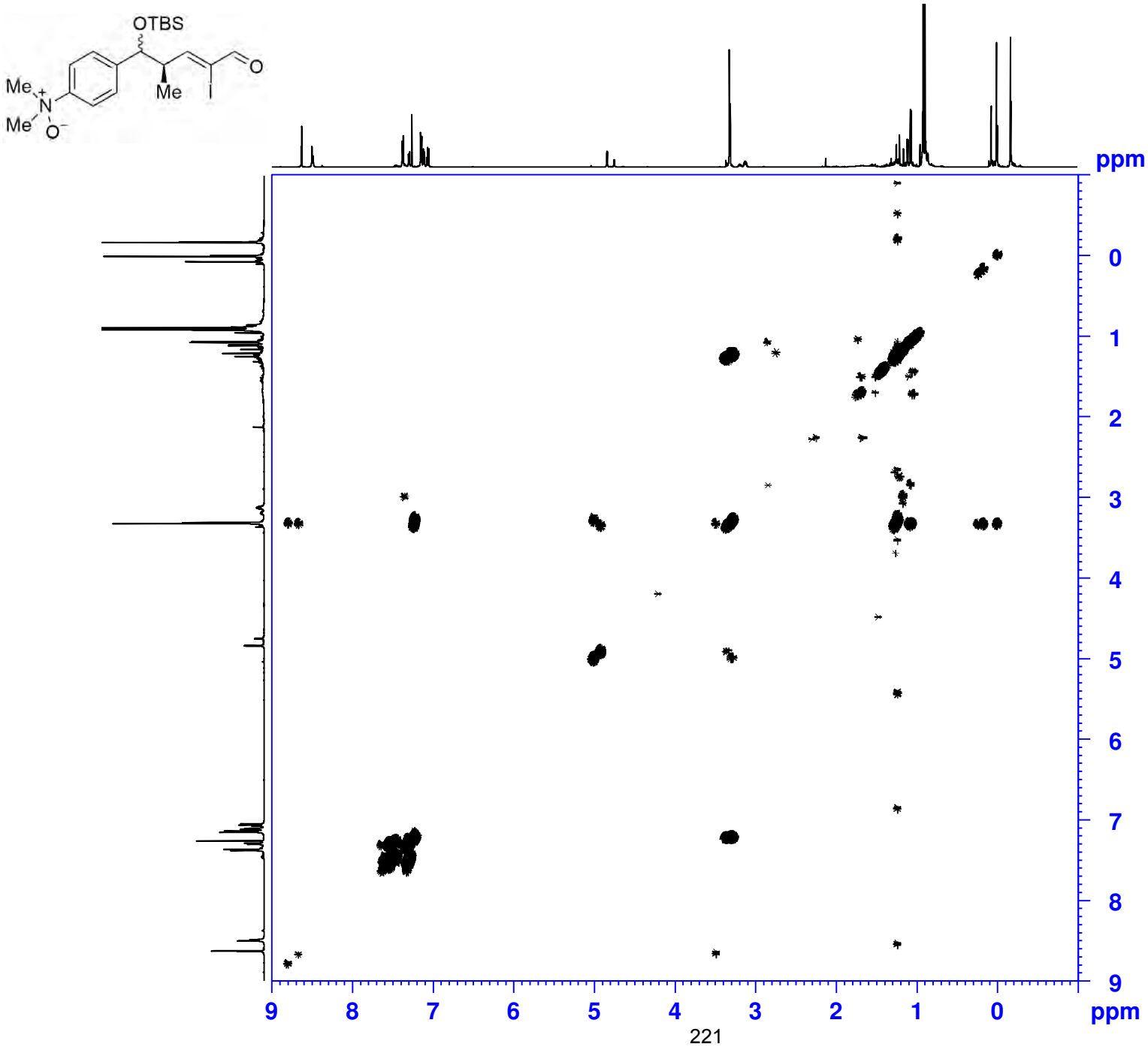
Current Data Parameters
NAME TPAPBYPRODUCT
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190924
Time 15.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9028113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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```
Current Data Parameters
NAME       TPAPBYPRODUCT
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190924
Time               16.00
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDC13
NS                     1
DS                     8
SWH             7763.975 Hz
FIDRES          3.791003 Hz
AQ              0.1318912 sec
RG                186.92
DW                64.400 usec
DE                 6.50 usec
TE                300.0 K
D0           0.00000300 sec
D1           0.93241531 sec
D11          0.03000000 sec
D12          0.00002000 sec
D13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00012880 sec

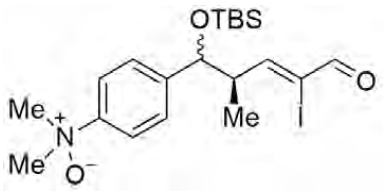
======== CHANNEL f1 ========
SFO1         600.1319161 MHz
NUC1                   1H
P1                 10.00 usec
P17              2500.00 usec
PLW1         26.60000038 W
PLW10         3.93490005 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1               16.00 %
GPZ2               12.00 %
GPZ3               40.00 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           600.1319 MHz
FIDRES        60.656055 Hz
SW               12.937 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           600.1299140 MHz
WDW                 SINE
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           600.1299140 MHz
WDW                 SINE
SSB                    0
LB                     0 Hz
GB                     0
```




```
Current Data Parameters
NAME      TPAPBYPRODUCT
EXPNO                14
PROCNO                1

F2 - Acquisition Parameters
Date_          20190924
Time              16.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                 1024
SOLVENT           CDCl3
NS                    2
DS                   32
SWH           7211.539 Hz
FIDRES        7.042518 Hz
AQ            0.0709973 sec
RG               186.92
DW               69.333 usec
DE                6.50 usec
TE               300.2 K
CNST2       145.0000000
D0           0.00000300 sec
D1           0.80000001 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
D21          0.00360000 sec
IN0          0.00002010 sec

======== CHANNEL f1 ========
SFO1         600.1328223 MHz
NUC1                 1H
P1                10.00 usec
P2                20.00 usec
P28                0 usec
PLW1        26.60000038 W

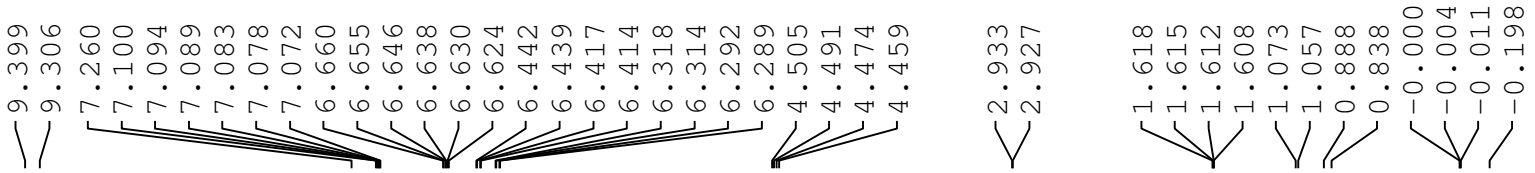
======== CHANNEL f2 ========
SFO2         150.9141236 MHz
NUC2                13C
CPDPRG[2           garp4
P3                11.80 usec
P14              500.00 usec
P31             1730.00 usec
PCPD2             65.00 usec
PLW0                 0 W
PLW2         85.00000000 W
PLW12         2.80130005 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3                0 Hz
SPW3         18.08300018 W
SPNAM[18  Crp60_xfilt.2
SPOAL18            0.500
SPOFFS18               0 Hz
SPW18         5.22629976 W

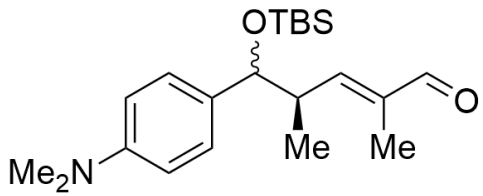
====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  256
SFO1           150.9141 MHz
FIDRES       194.340790 Hz
SW              164.833 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                 1024
SF           600.1300000 MHz
WDW                QSINE
SSB                   2
LB                 0 Hz
GB                    0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2        echo-antiecho
SF           150.9028085 MHz
WDW                QSINE
SSB                   2
LB                 0 Hz
GB                    0
```



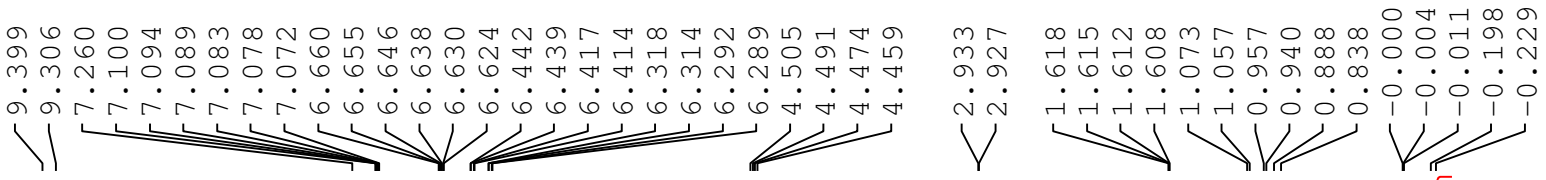


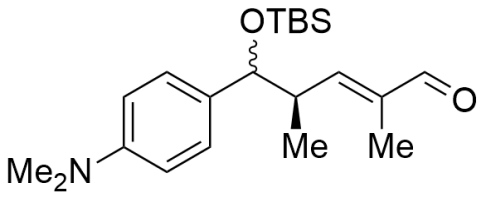
Current Data Parameters
NAME I-PK-285
EXPNO 10
PROCNO 1

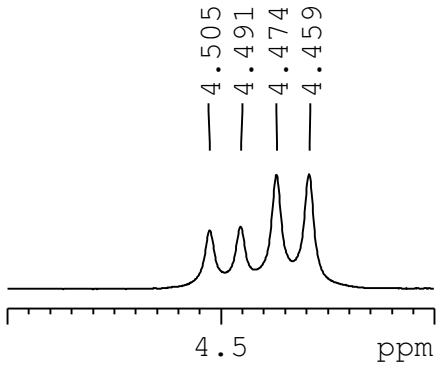
F2 - Acquisition Parameters
Date_ 20190514
Time 12.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 50.8
DW 41.600 usec
DE 9.85 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00





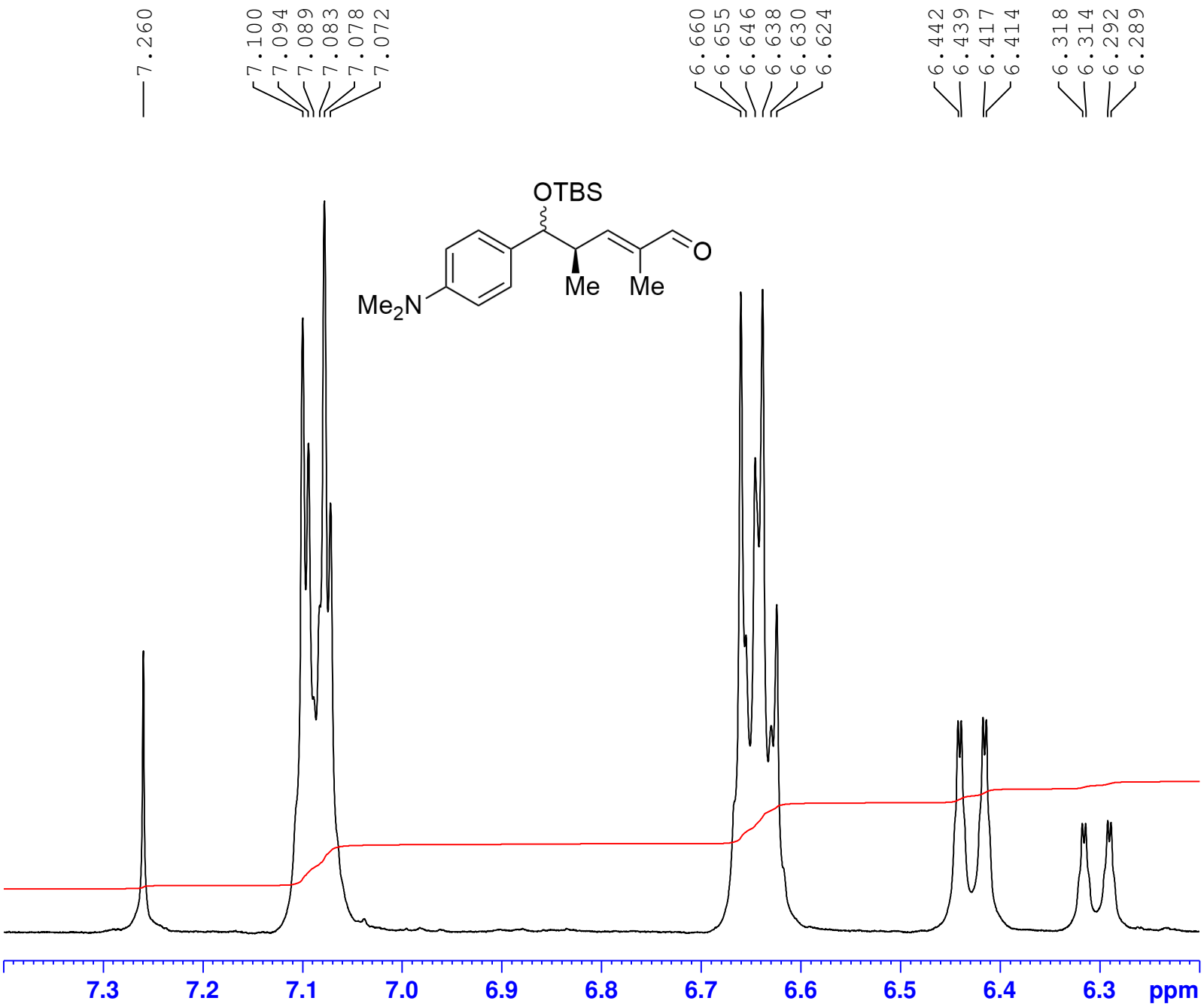



```
Current Data Parameters
NAME                  I-PK-285
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190514
Time                     12.45
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                12019.230 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                       50.8
DW                     41.600 usec
DE                      9.85 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             399.9024695 MHz
NUC1                       1H
P1                      14.88 usec
PLW1              7.59999990 W

F2 - Processing parameters
SI                     131072
SF               399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

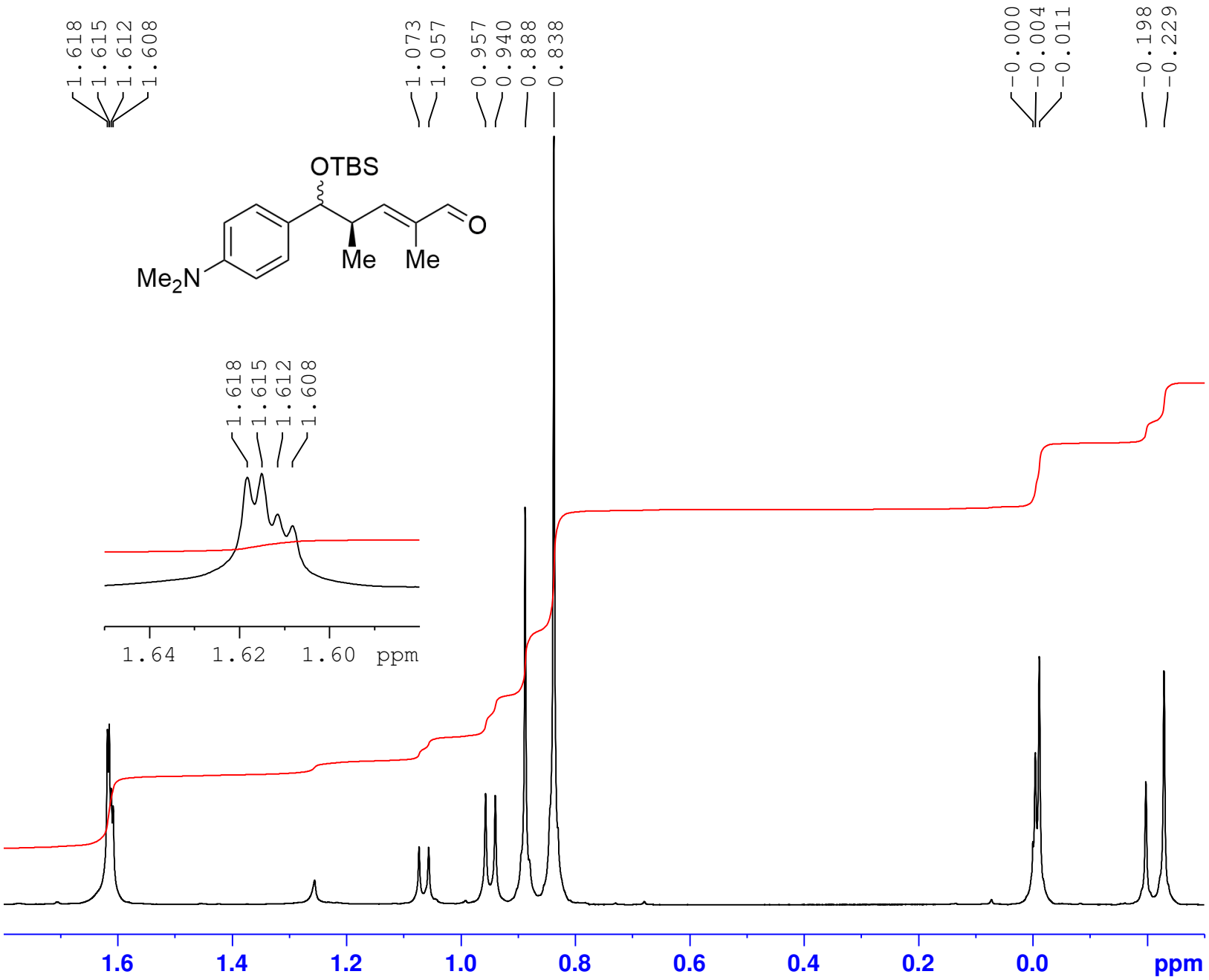



```
Current Data Parameters
NAME                  I-PK-285
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190514
Time                     12.45
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         50.8
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



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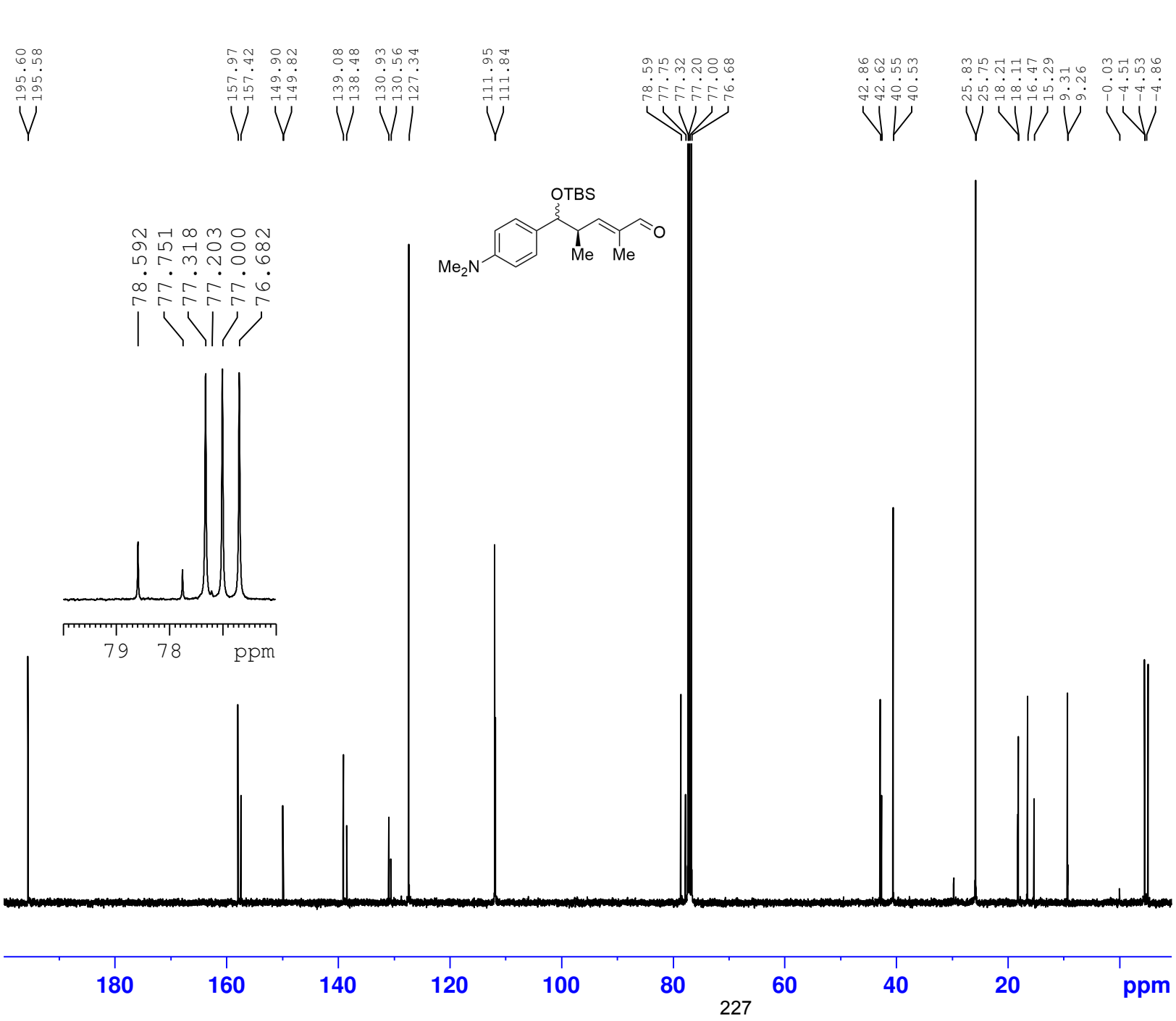
Current Data Parameters
NAME I-PK-285
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190514
Time 12.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 50.8
DW 41.600 usec
DE 9.85 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



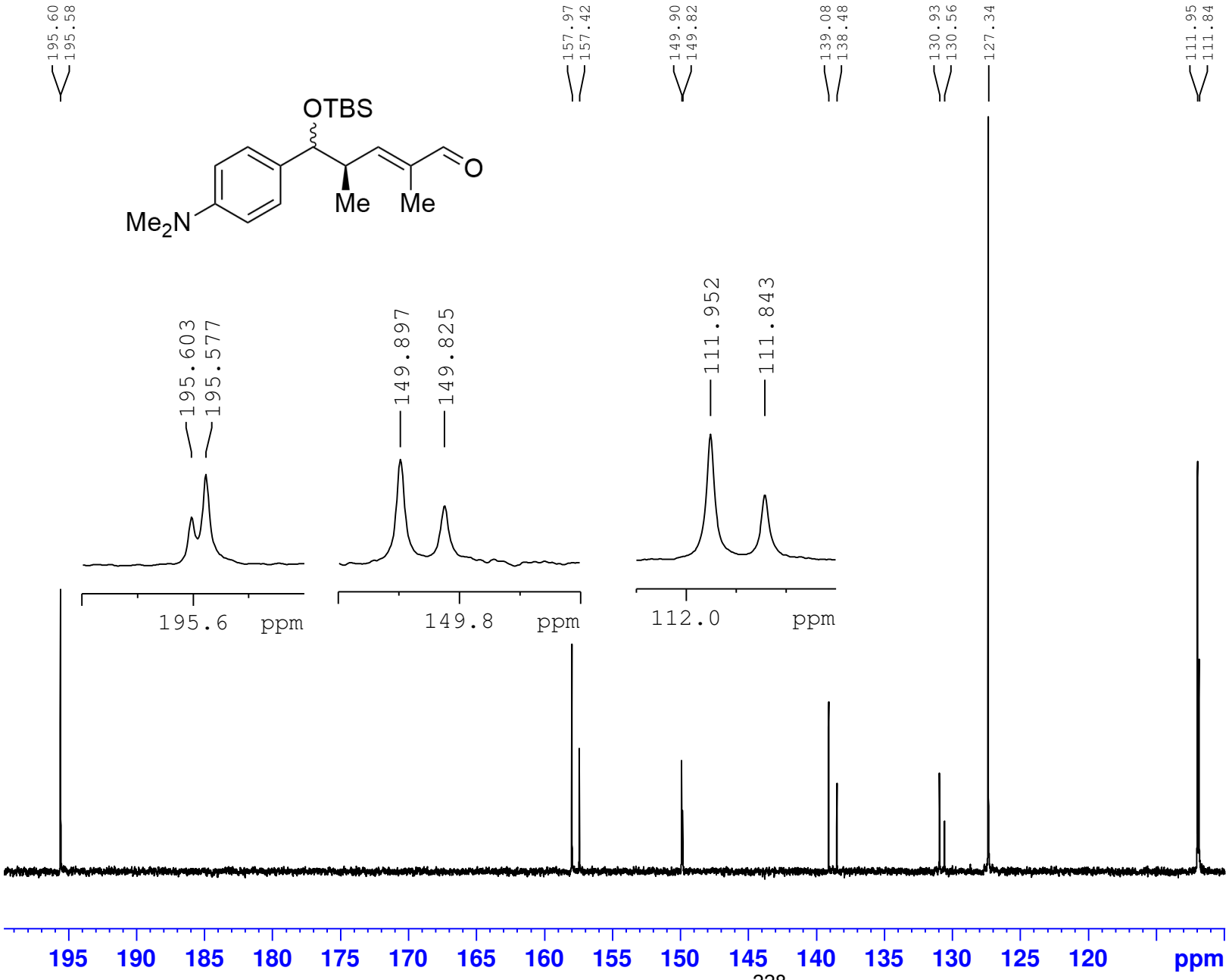
Current Data Parameters
NAME I-PK-285
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190514
Time 18.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



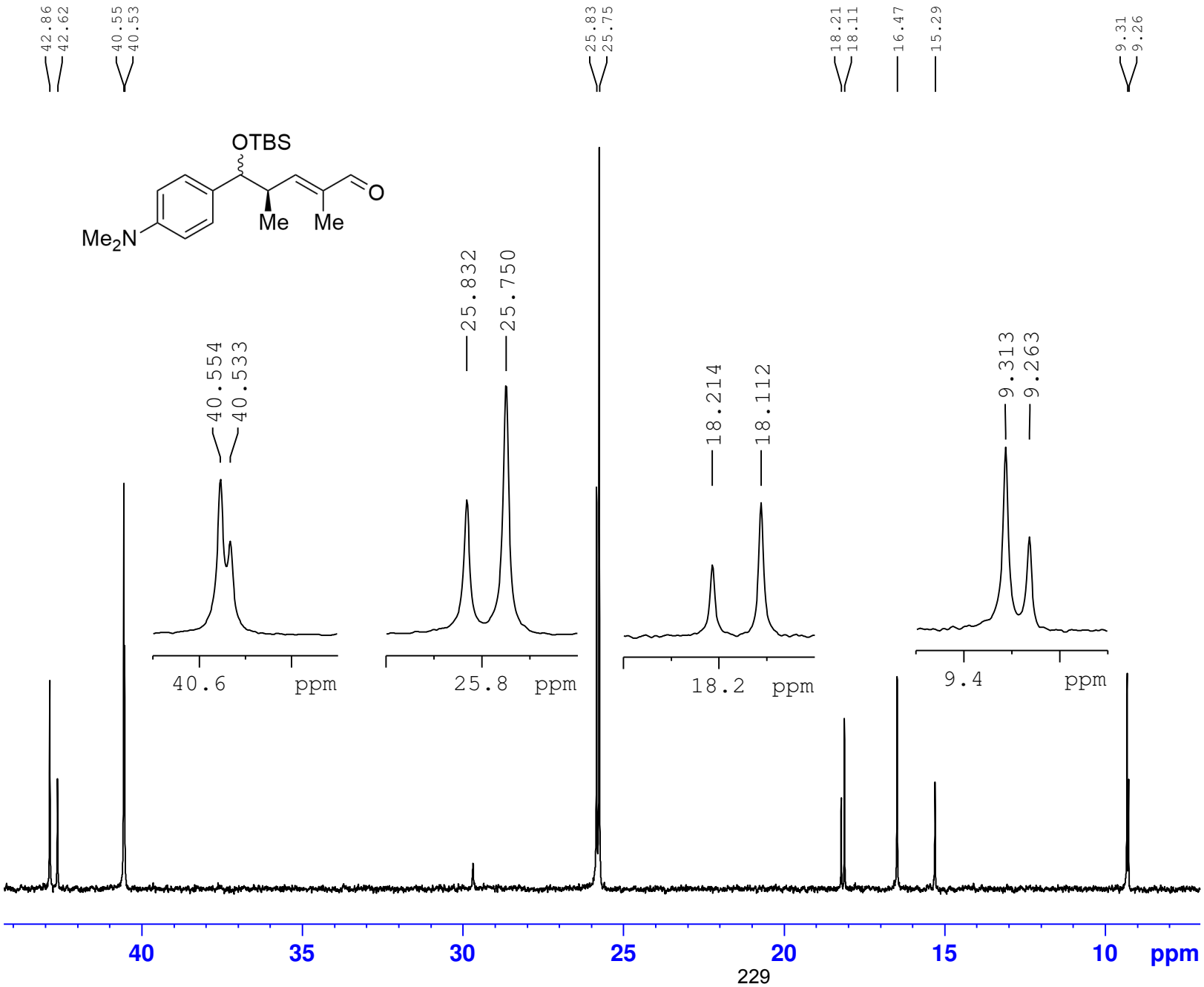

```
Current Data Parameters
NAME                  I-PK-285
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190514
Time                     18.13
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                      119044
SOLVENT                   CDCl3
NS                         1200
DS                            4
SWH                25000.000 Hz
FIDRES              0.210006 Hz
AQ                  2.3808801 sec
RG                         2050
DW                    20.000 usec
DE                     9.12 usec
TE                     300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1             100.5659947 MHz
NUC1                       13C
P1                     10.00 usec
PLW1            44.46300125 W

======== CHANNEL f2 ========
SFO2             399.9015996 MHz
NUC2                       1H
CPDPRG[2               waltz64
PCPD2                  90.00 usec
PLW2             7.59999990 W
PLW12            0.20774999 W
PLW13            0.16827001 W

F2 - Processing parameters
SI                       131072
SF             100.5549379 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



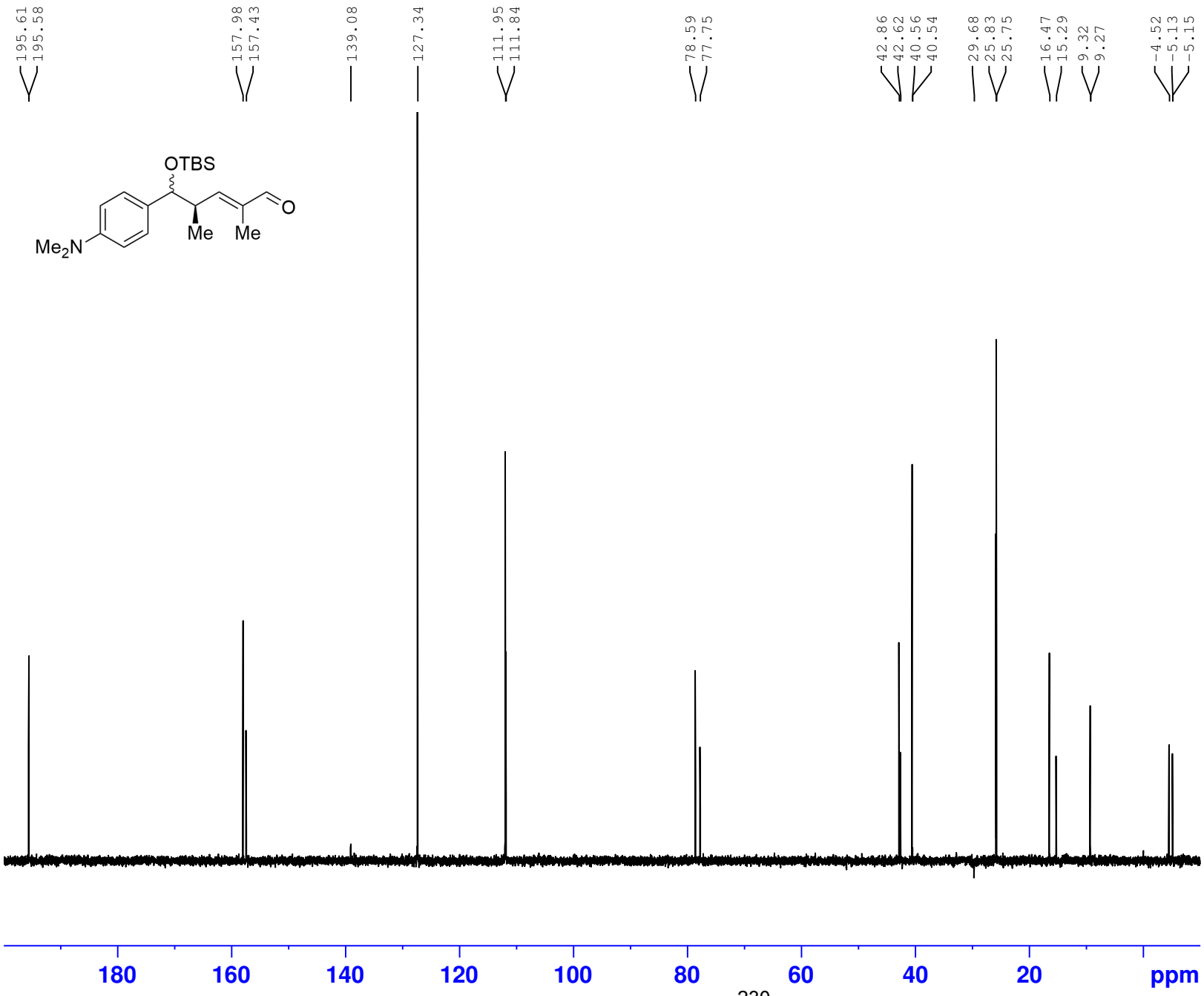

```
Current Data Parameters
NAME                  I-PK-285
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                20190514
Time                    18.13
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                     119044
SOLVENT                 CDCl3
NS                       1200
DS                          4
SWH                25000.000 Hz
FIDRES              0.210006 Hz
AQ                  2.3808801 sec
RG                       2050
DW                    20.000 usec
DE                     9.12 usec
TE                     300.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             100.5659947 MHz
NUC1                      13C
P1                     10.00 usec
PLW1            44.46300125 W

======== CHANNEL f2 ========
SFO2             399.9015996 MHz
NUC2                      1H
CPDPRG[2              waltz64
PCPD2                  90.00 usec
PLW2             7.59999990 W
PLW12            0.20774999 W
PLW13            0.16827001 W

F2 - Processing parameters
SI                      131072
SF               100.5549379 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



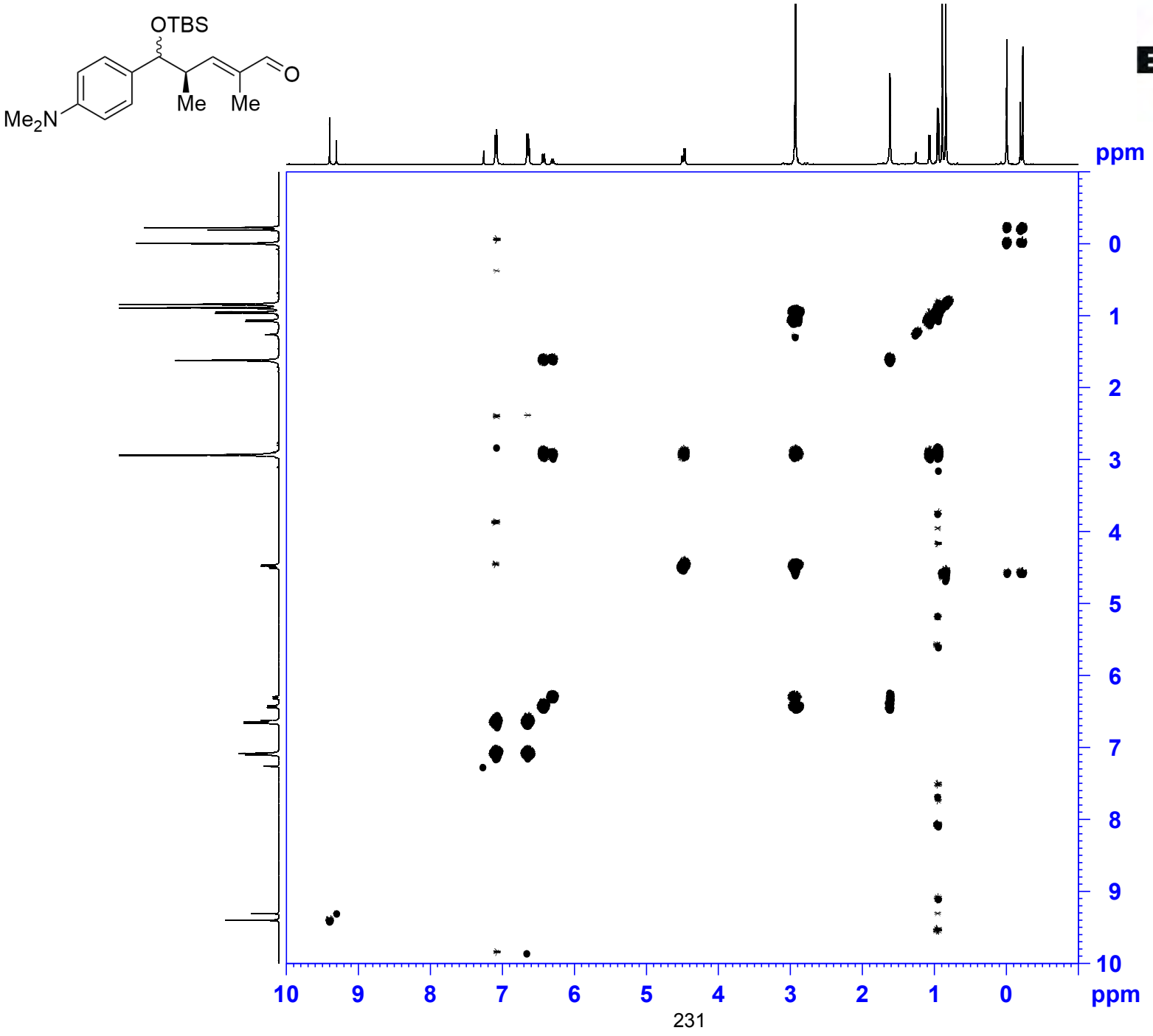
Current Data Parameters
NAME I-PK-285
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190514
Time 18.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1820
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                  I-PK-285
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190514
Time                     18.31
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                       2048
SOLVENT                  CDCl3
NS                           1
DS                           8
SWH                 4545.455 Hz
FIDRES              2.219460 Hz
AQ                  0.2252800 sec
RG                        2050
DW                   110.000 usec
DE                     6.50 usec
TE                     300.0 K
D0                  0.00000300 sec
D1                  0.92545187 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D13                 0.00000400 sec
D16                 0.00020000 sec
IN0                 0.00021980 sec

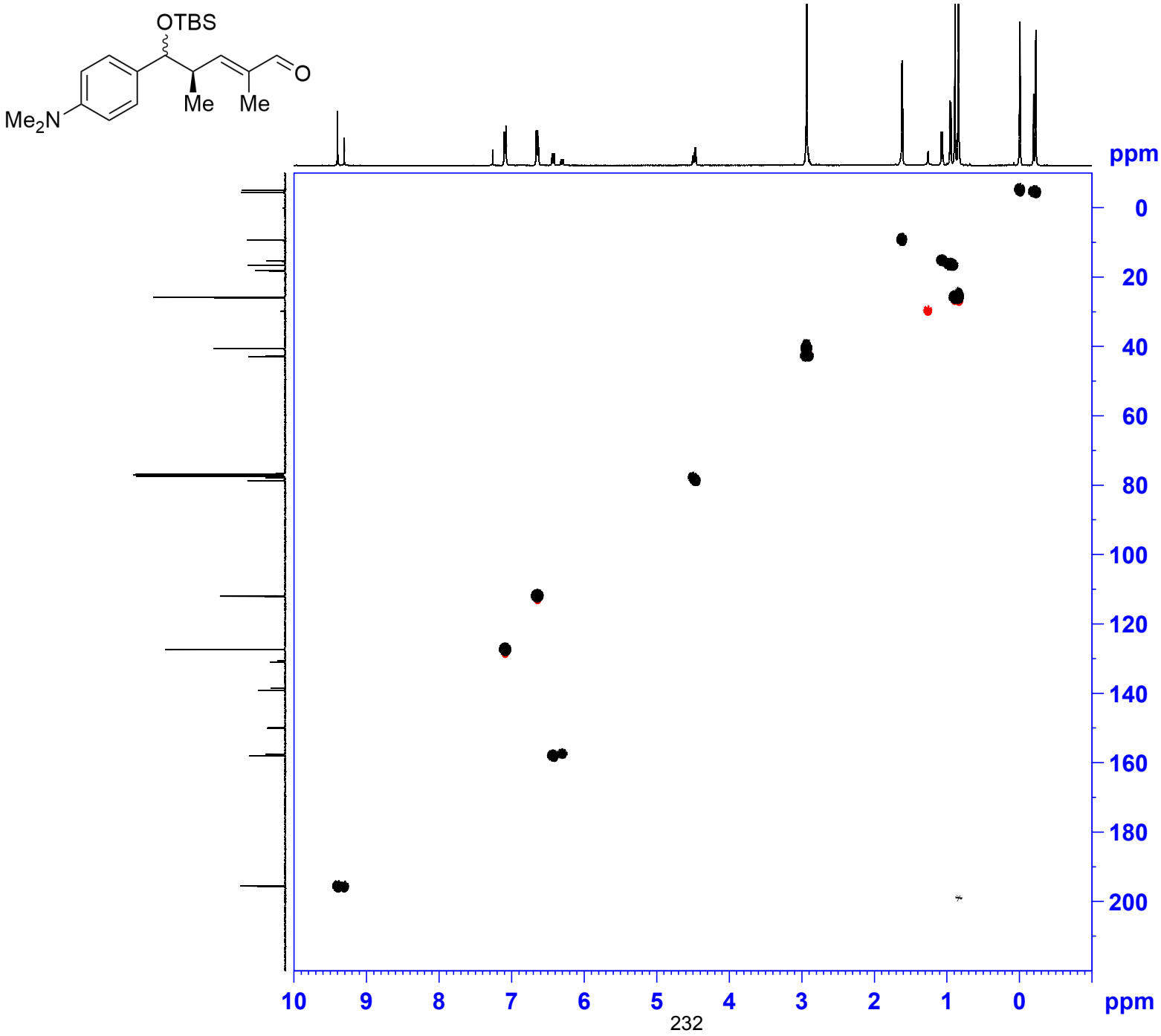
======== CHANNEL f1 ========
SFO1               399.9018381 MHz
NUC1                        1H
P1                       14.88 usec
P17                    2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPNAM[3]          SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1                399.9018 MHz
FIDRES             35.543674 Hz
SW                     11.377 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                399.9000100 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                399.9000122 MHz
WDW                       SINE
SSB             0
LB              0 Hz
GB              0
```




```
Current Data Parameters
NAME                 I-PK-285
EXPNO                      14
PROCNO                      1

F2 - Acquisition Parameters
Date_               20190514
Time                   18.44
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                       1024
SOLVENT                CDCl3
NS                          2
DS                         32
SWH                 4807.692 Hz
FIDRES              4.695012 Hz
AQ                  0.1064960 sec
RG                       2050
DW                    104.000 usec
DE                      6.50 usec
TE                     300.1 K
CNST2              145.0000000
D0                  0.00000300 sec
D1                  0.80000001 sec
D4                  0.00172414 sec
D11                 0.03000000 sec
D16                 0.00020000 sec
D21                 0.00360000 sec
IN0                 0.00001910 sec

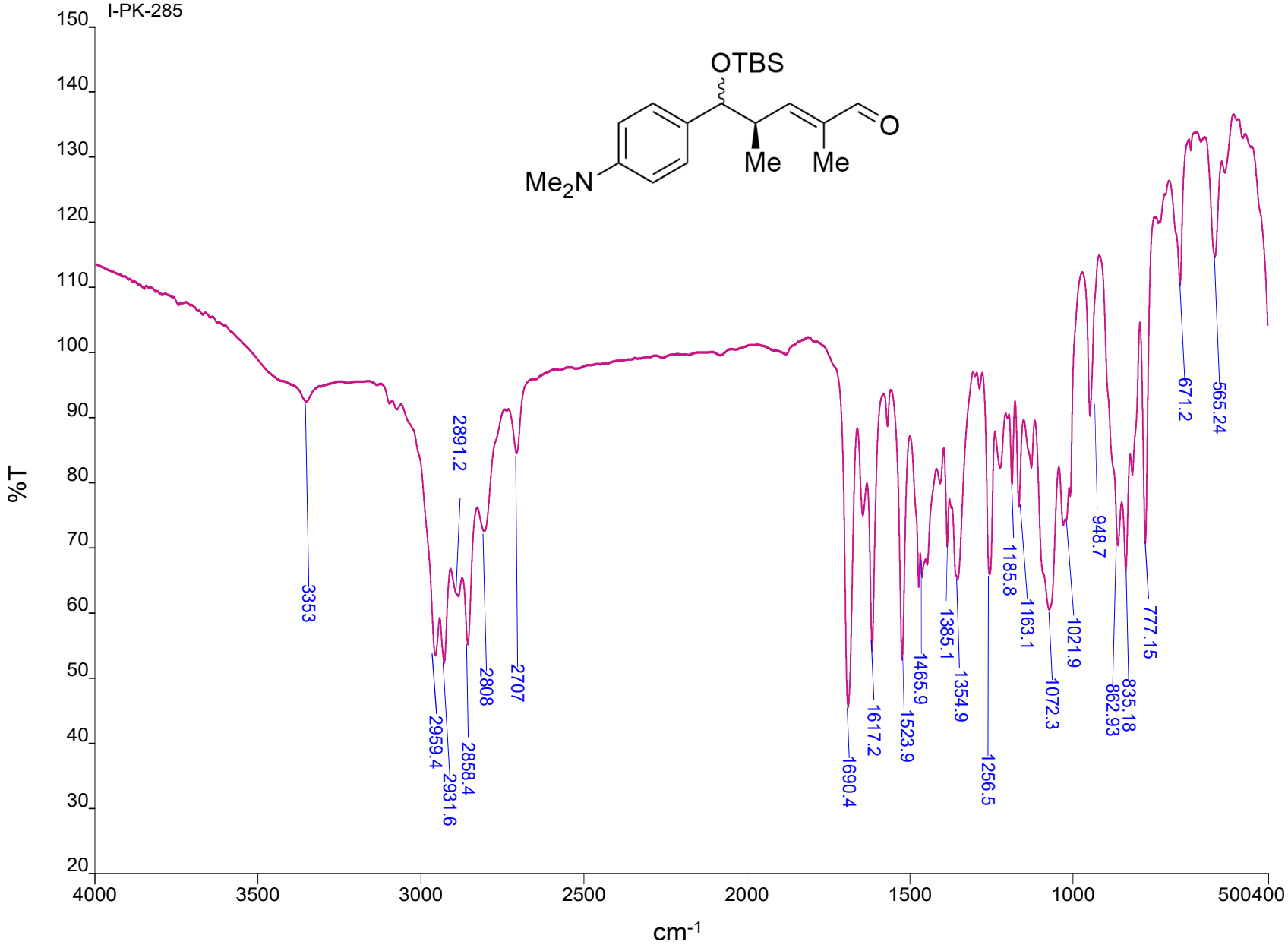
======== CHANNEL f1 ========
SFO1               399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28          0 usec
PLW1                7.59999990 W

======== CHANNEL f2 ========
SFO2               100.5670016 MHz
NUC2                       13C
CPDPRG[2                 garp4
P3                     10.00 usec
P14                   500.00 usec
P31                  1900.00 usec
PCPD2                  80.00 usec
PLW0         0 W
PLW2               44.46300125 W
PLW12               0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                  0.500
SPOFFS3  0 Hz
SPW3                6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                 0.500
SPOFFS18 0 Hz
SPW18               1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]       SMSQ10.100
GPNAM[2]       SMSQ10.100
GPZ1                80.00 %
GPZ2                20.10 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                100.567 MHz
FIDRES           204.515701 Hz
SW                  260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      1024
SF               399.9000086 MHz
```

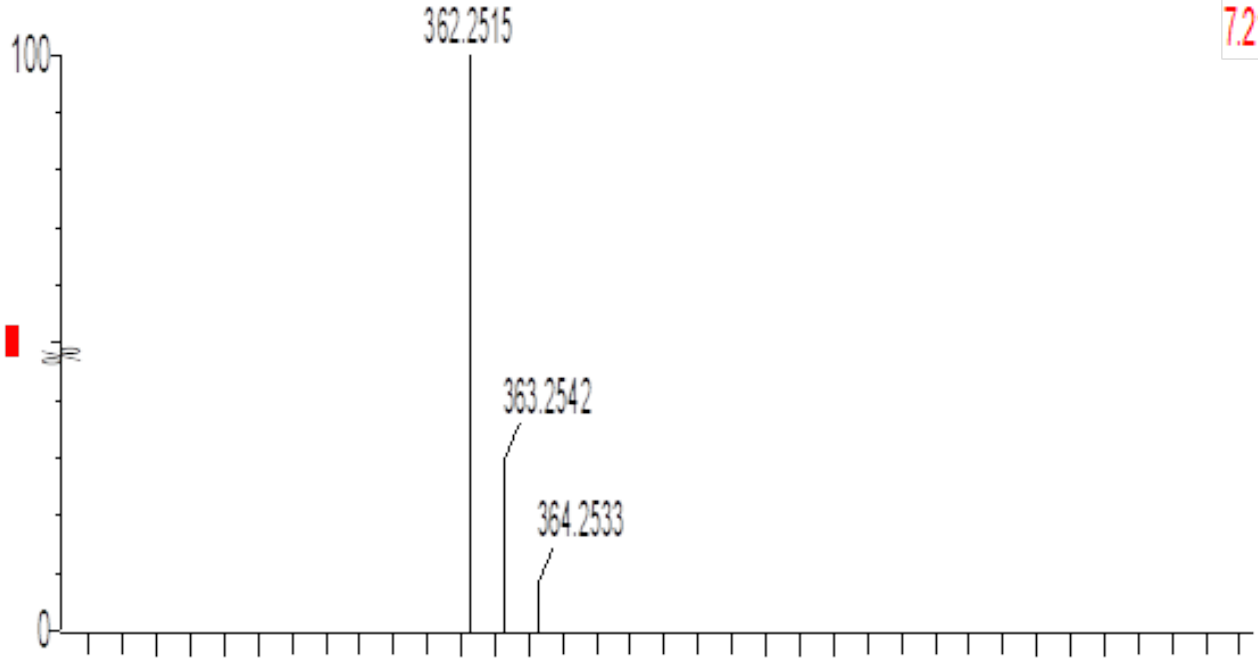

I-PK-285
OTBS
O
Me
Me
Me_2N
%T
150
140
130
120
110
100
90
80
70
60
50
40
30
20
4000
3500
3000
2500
2000
1500
1000
500400
cm^{-1}
3353
2891.2
2959.4
2931.6
2858.4
2808
2707
1690.4
1617.2
1523.9
1465.9
1385.1
1354.9
1256.5
1185.8
1163.1
1072.3
1021.9
948.7
862.93
835.18
777.15
671.2
565.24

I-PK-82

18-05-2018

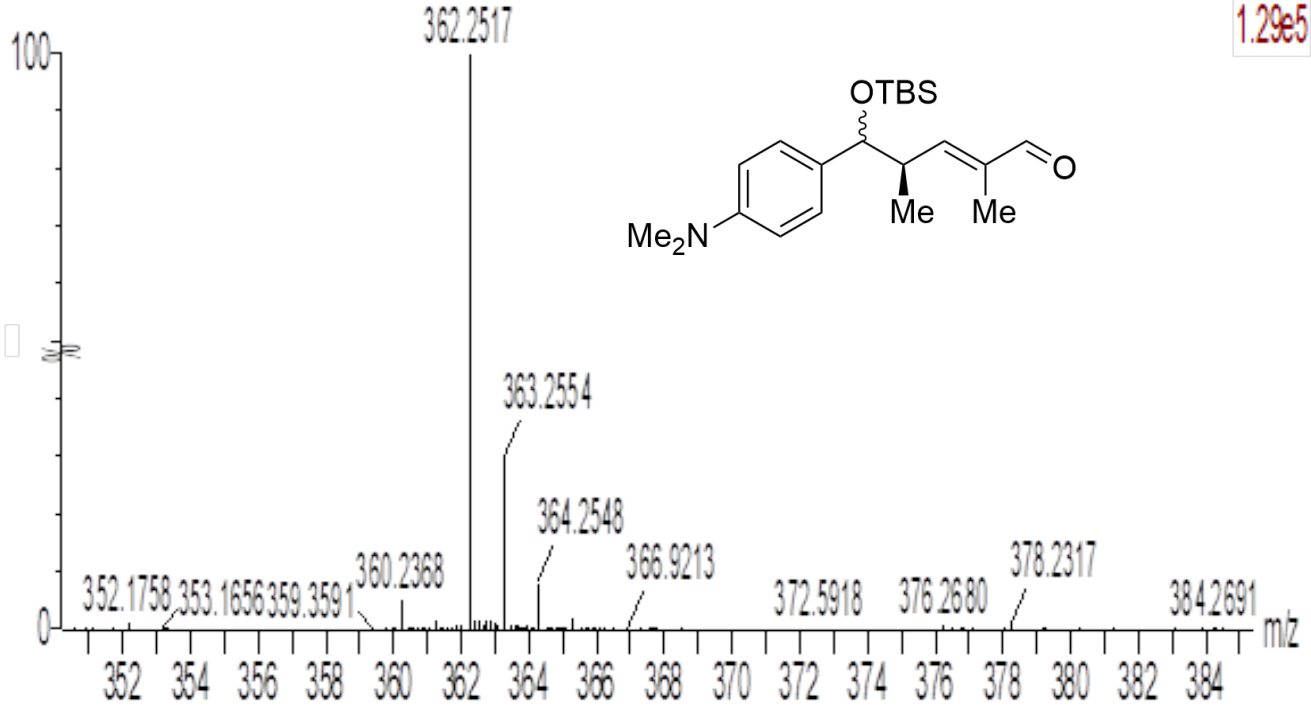
asep__16APR_2018_199 (0.070) Is (1.00,1.00) C21H35NSiO2H

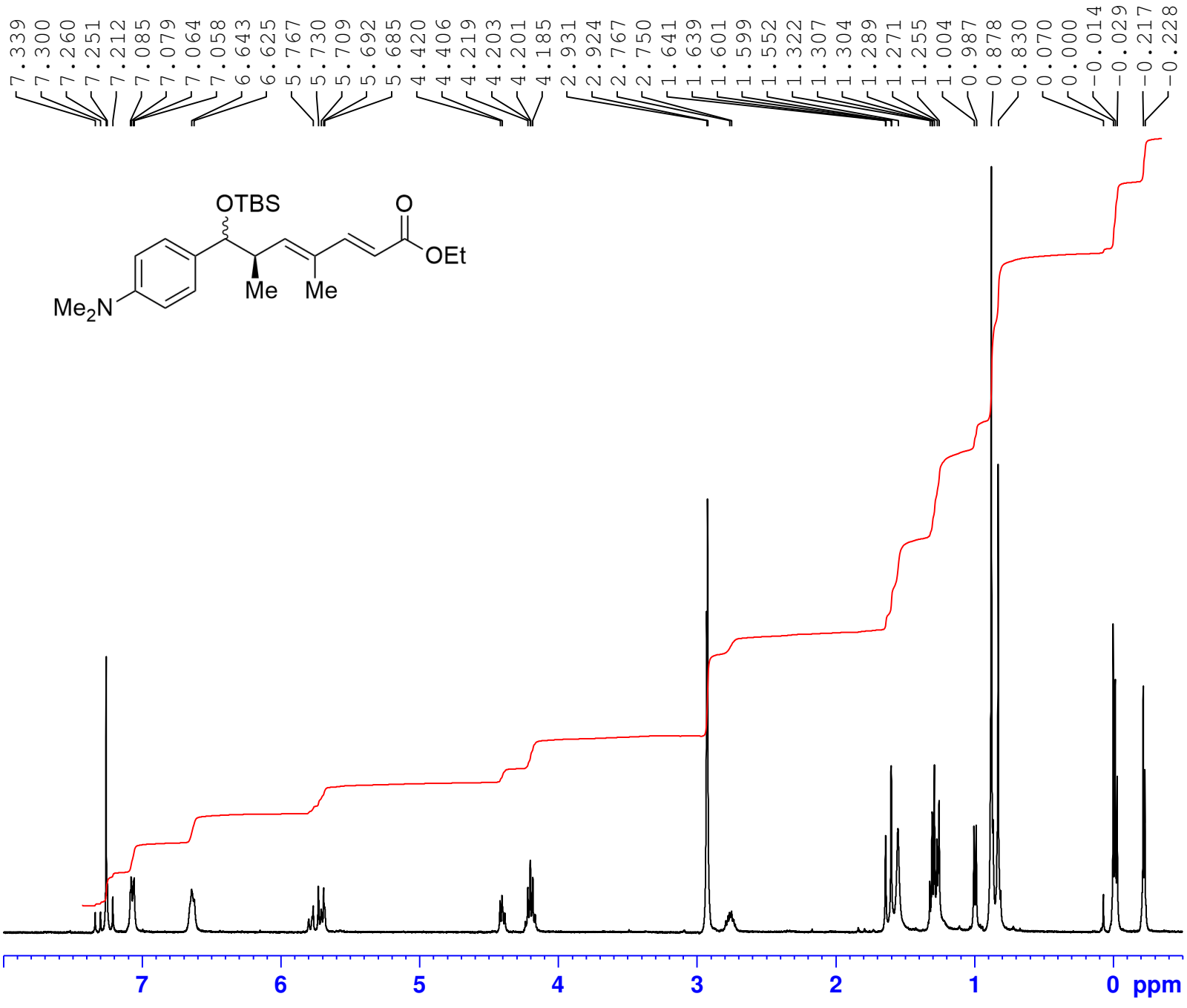
1: TOF MS ES+
7.21e12



asep__16APR_2018_199 7 (0.317) Cm (7)

1: TOF MS ES+
1.29e5





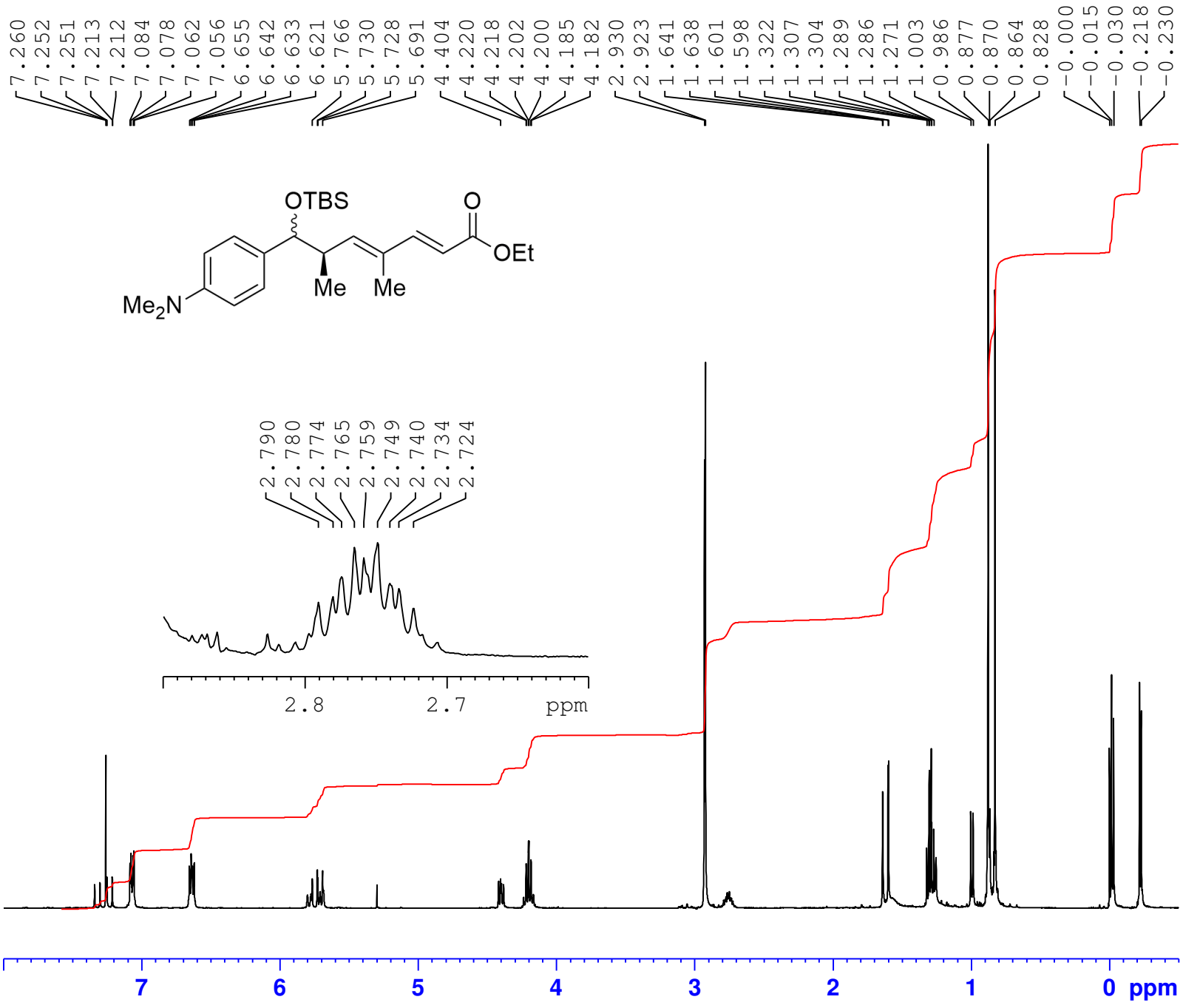
BRUKER

```
Current Data Parameters
NAME                  I-PK-286
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190522
Time                     16.25
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                        203
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000095 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

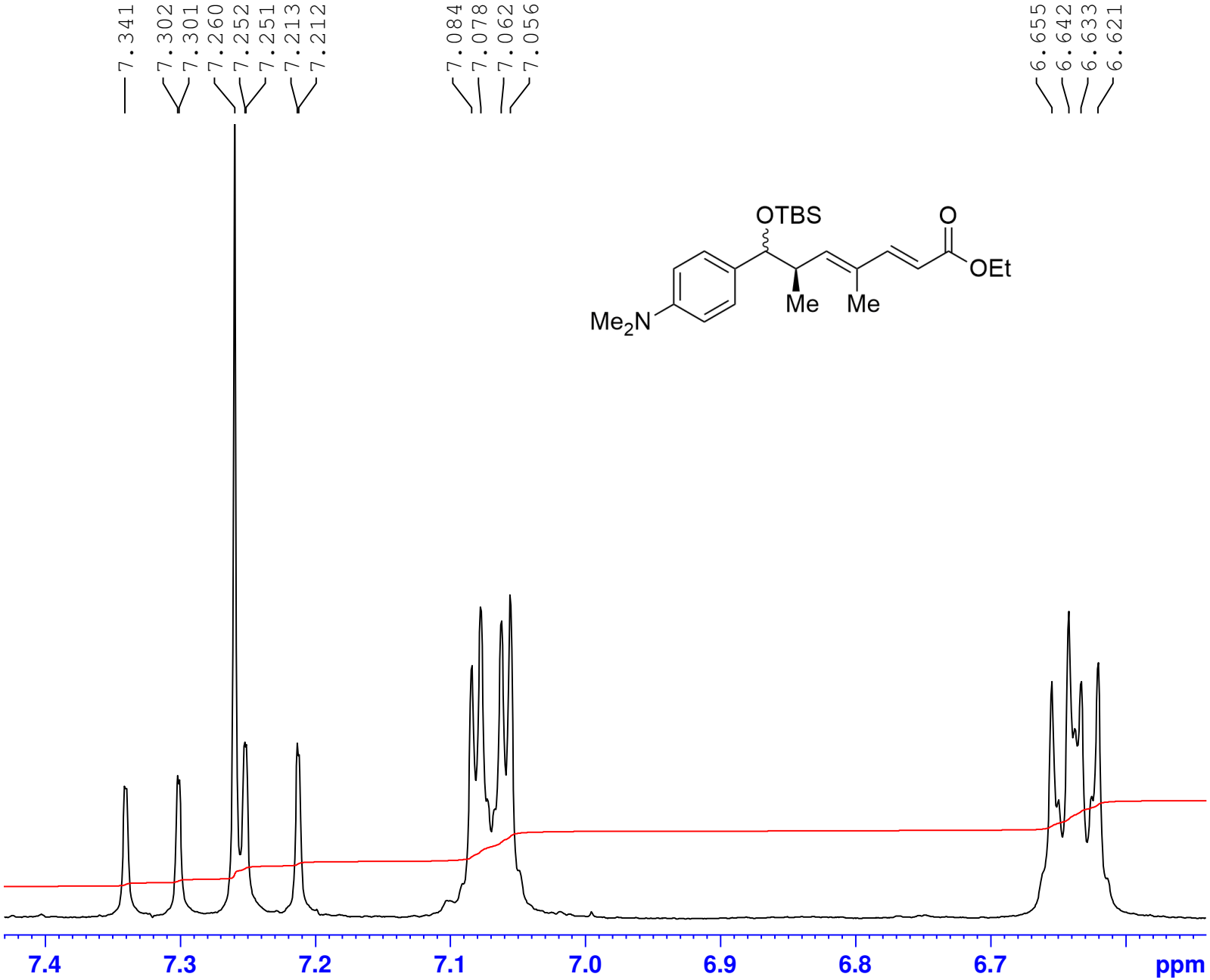


Current Data Parameters
NAME III-PK-151
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 21.20
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

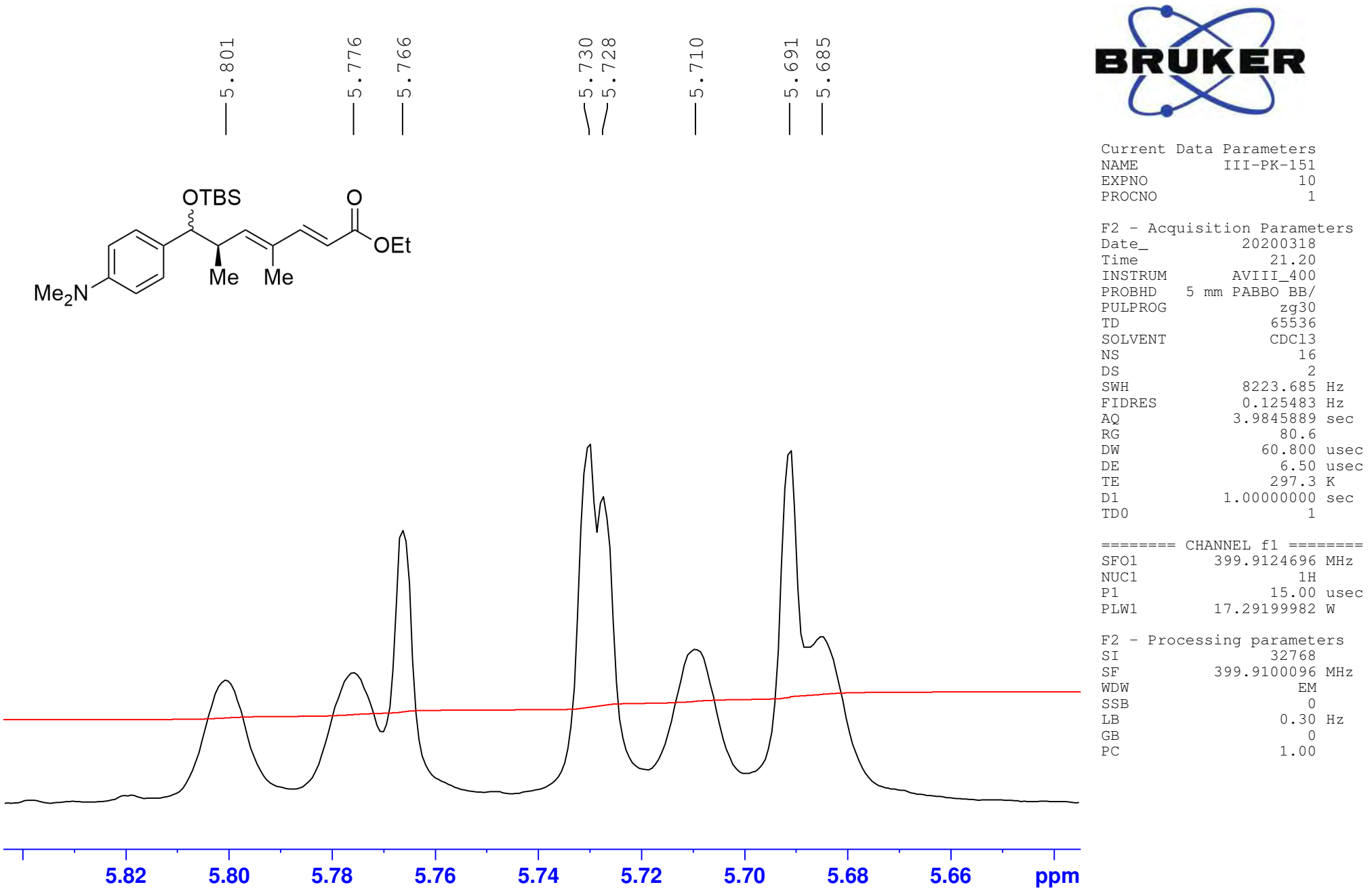


Current Data Parameters
NAME III-PK-151
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 21.20
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

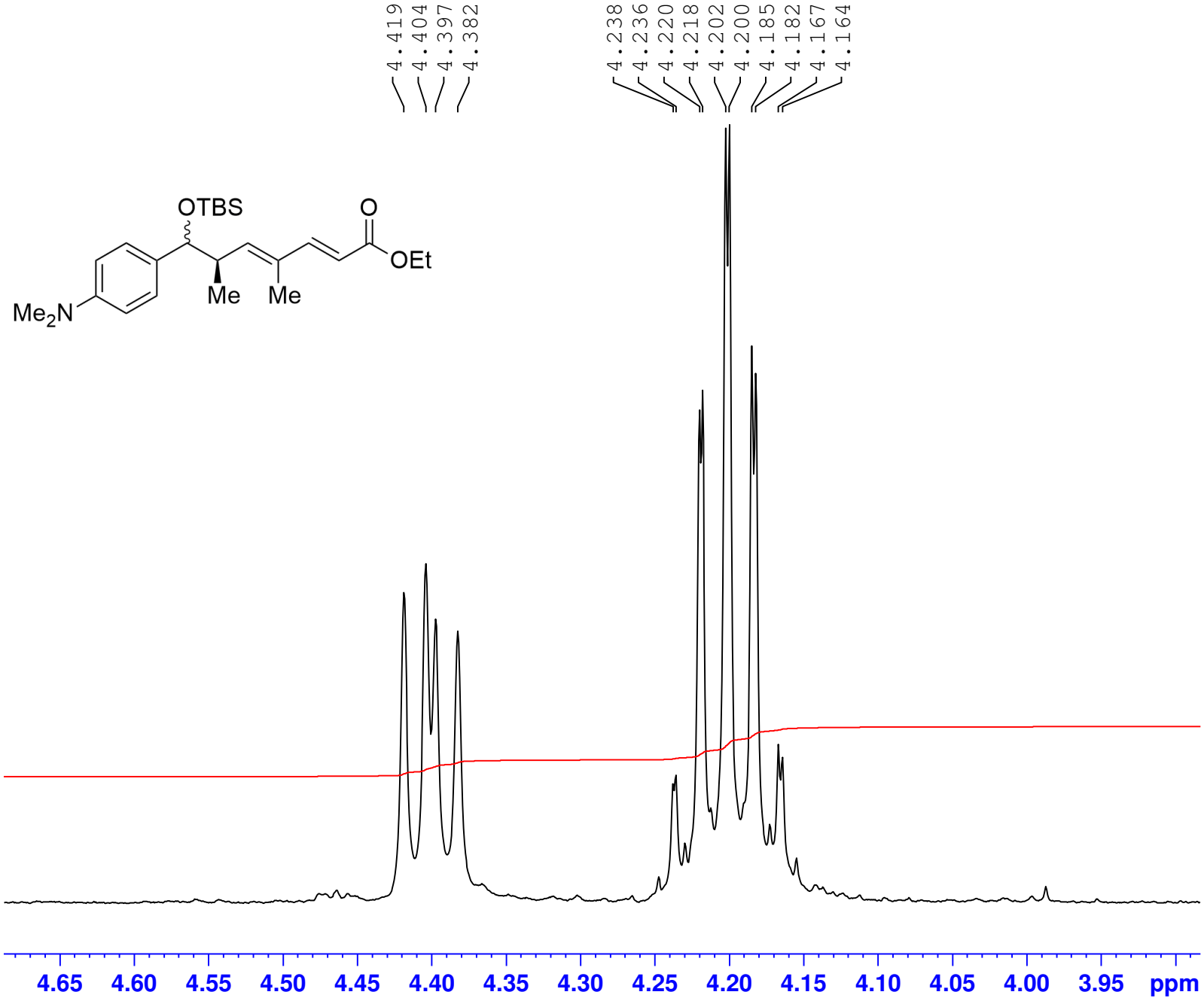


Current Data Parameters
NAME III-PK-151
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 21.20
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

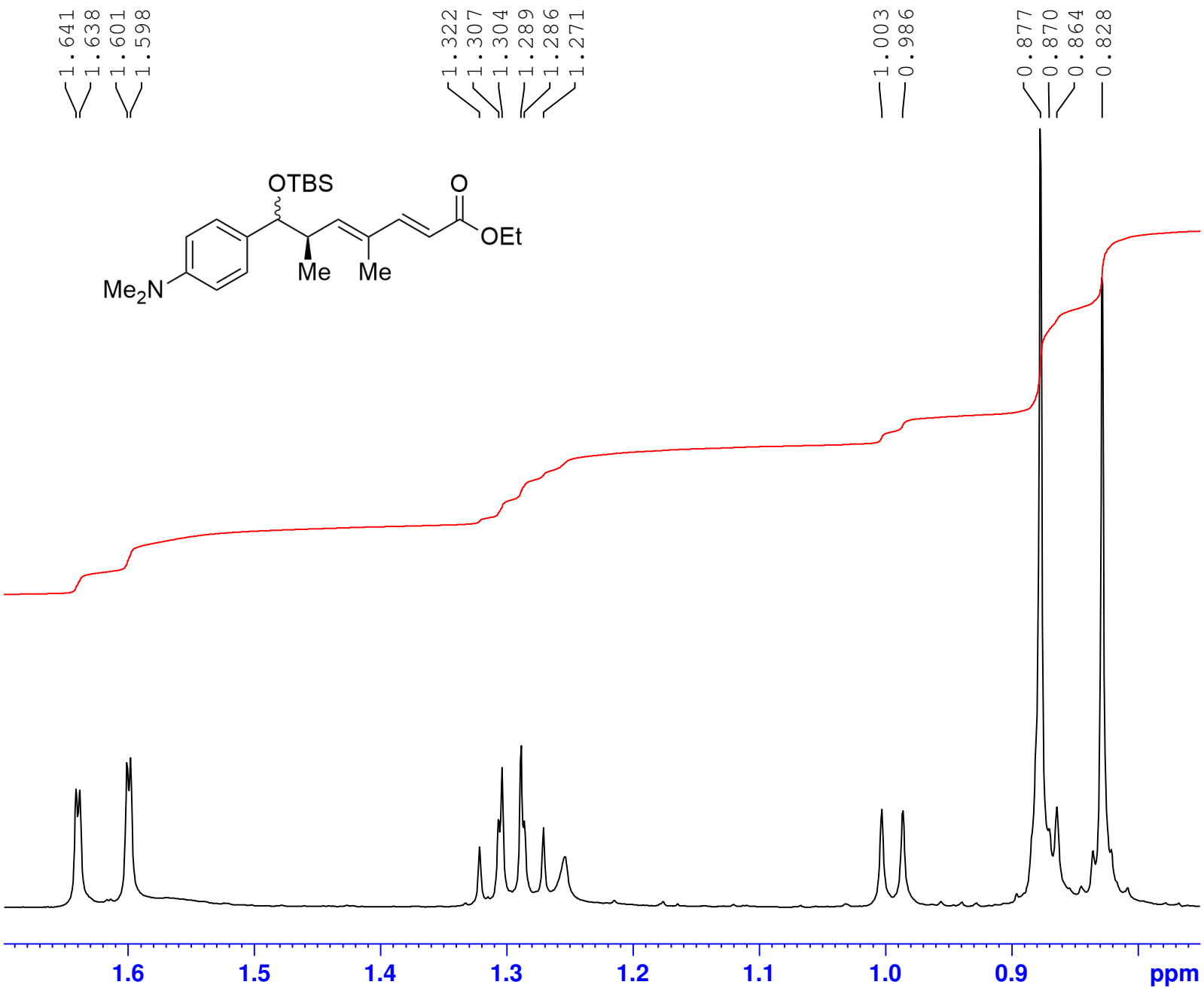


Current Data Parameters
NAME III-PK-151
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 21.20
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

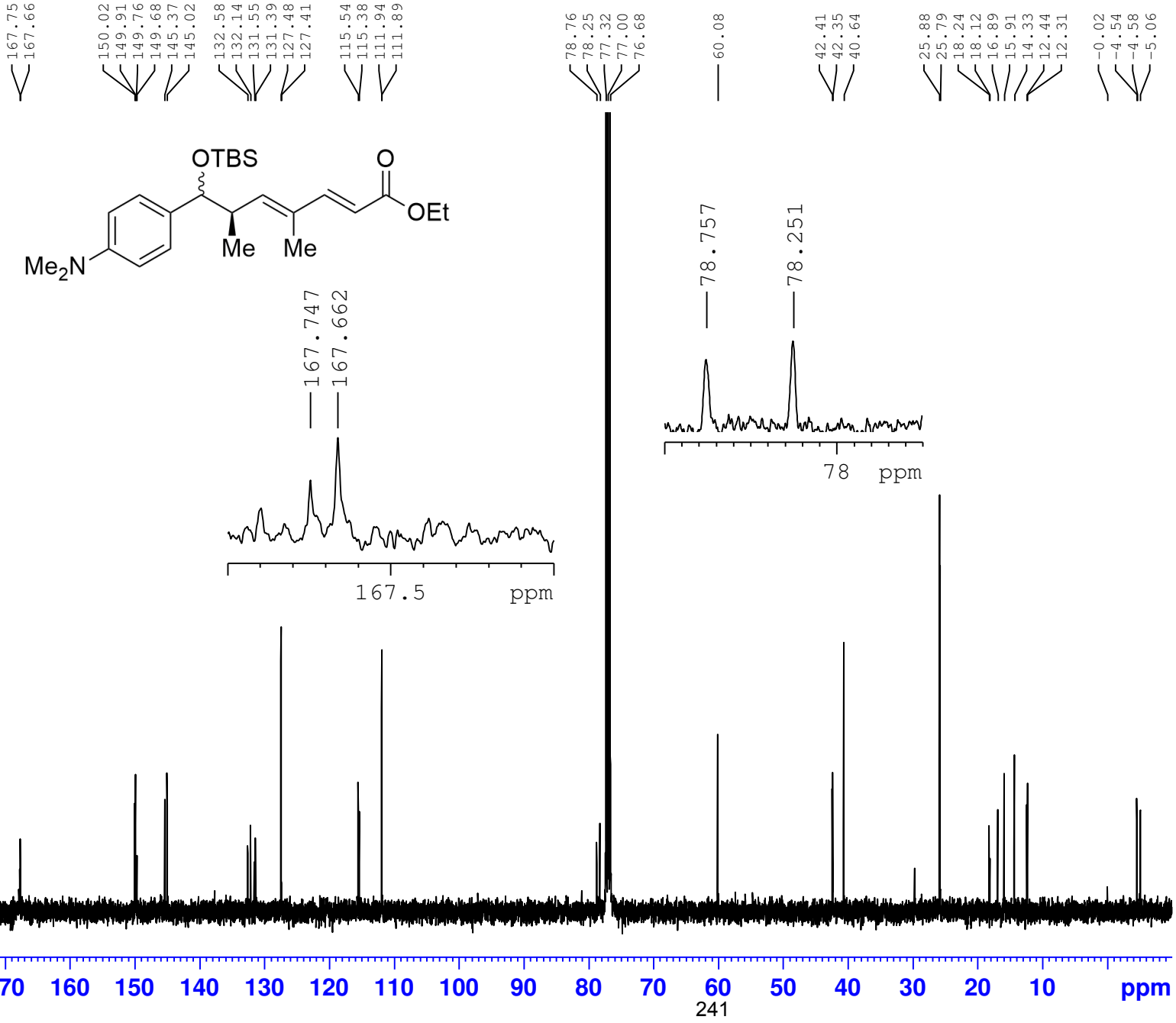


Current Data Parameters
NAME III-PK-151
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 21.20
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9124696 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 399.9100096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



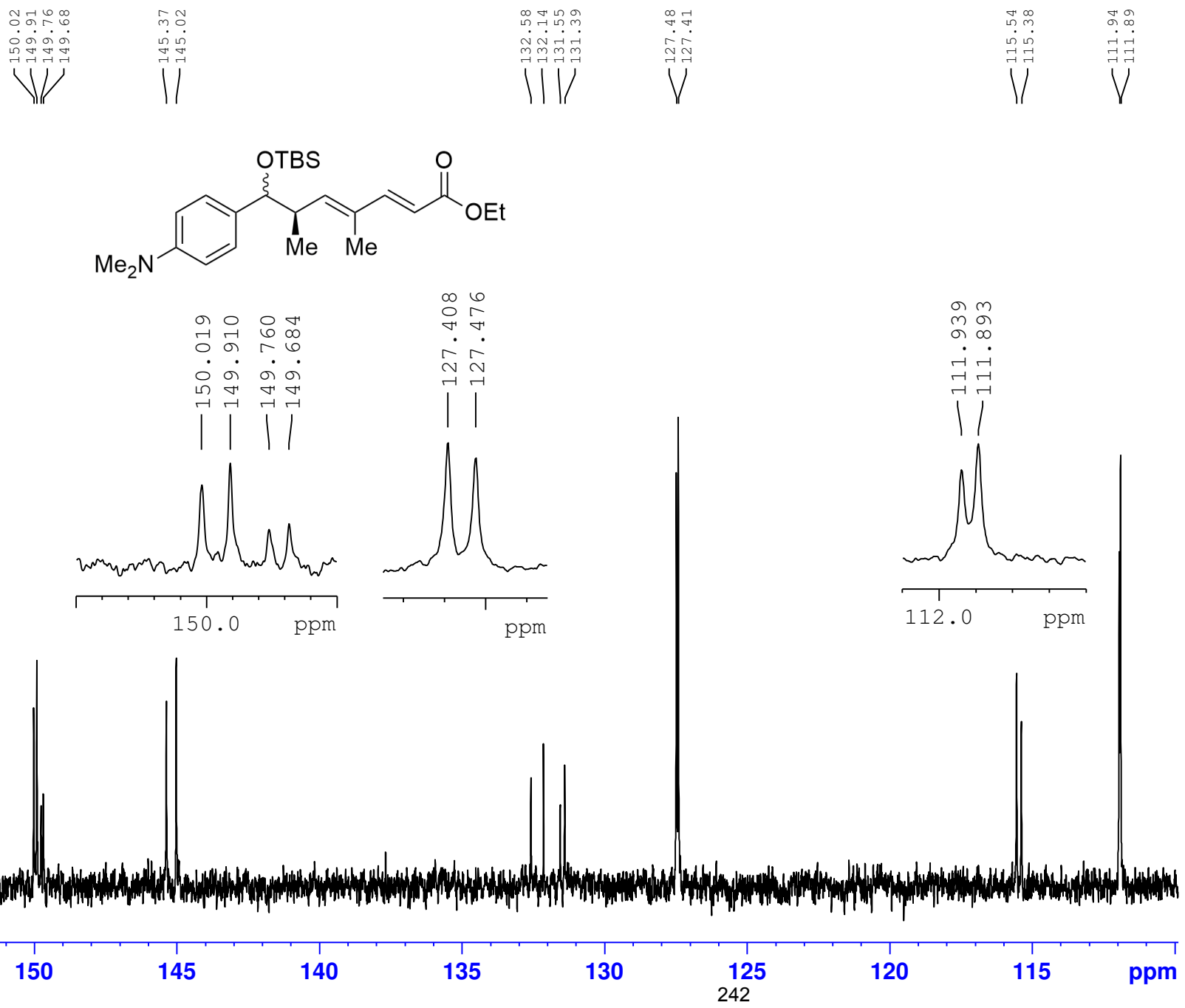
Current Data Parameters
NAME III-PK-151
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 22.15
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 96150
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.250010 Hz
AQ 1.9999200 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 300.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5675047 MHz
NUC1 13C
P1 9.00 usec
PLW1 96.68000031 W

======== CHANNEL f2 ========
SFO2 399.9115996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 17.29199982 W
PLW12 0.48032999 W
PLW13 0.38907000 W

F2 - Processing parameters
SI 131072
SF 100.5574502 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



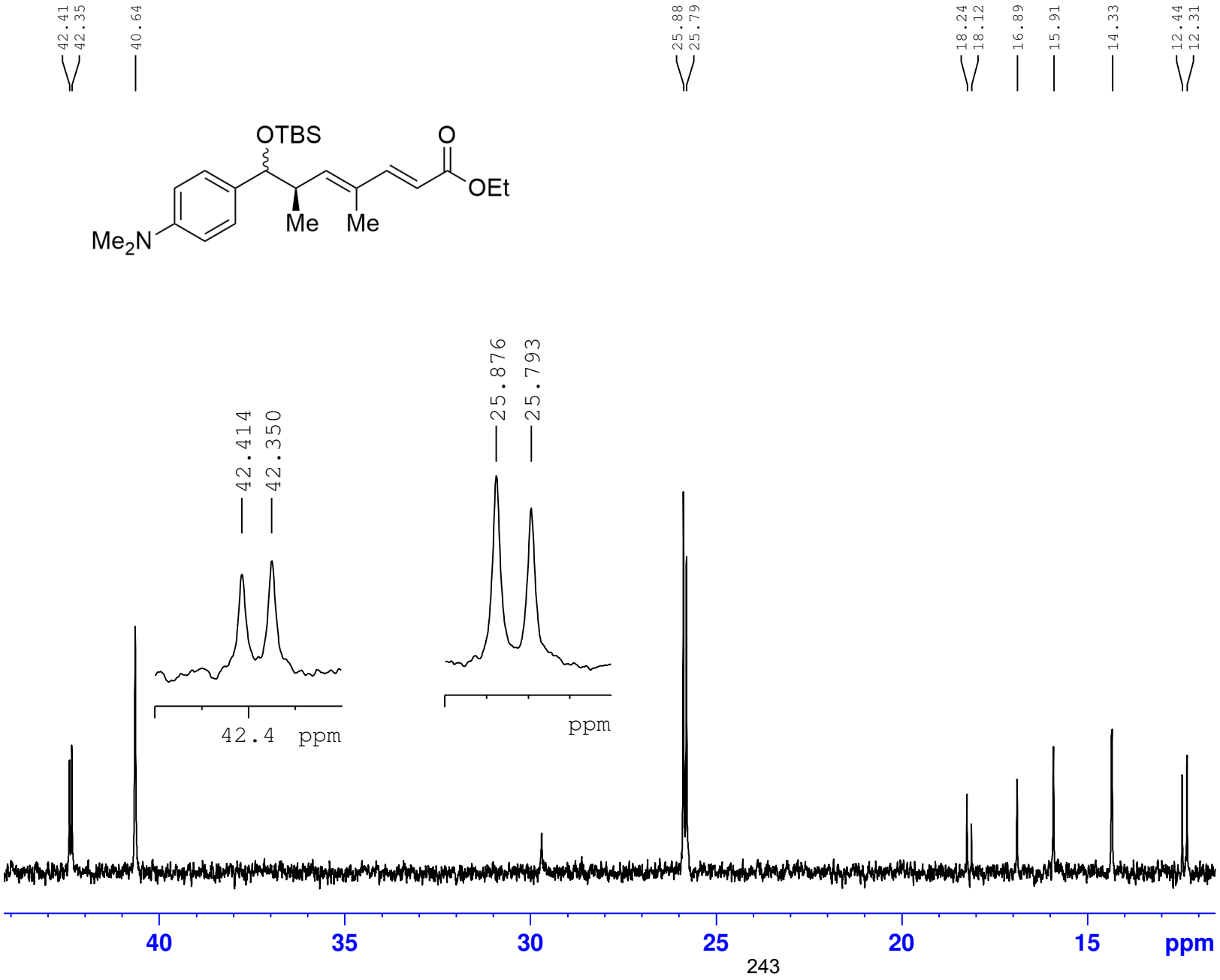
Current Data Parameters
NAME III-PK-151
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200318
Time 22.15
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 96150
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.250010 Hz
AQ 1.9999200 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 300.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5675047 MHz
NUC1 13C
P1 9.00 usec
PLW1 96.68000031 W

======== CHANNEL f2 ========
SFO2 399.9115996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 17.29199982 W
PLW12 0.48032999 W
PLW13 0.38907000 W

F2 - Processing parameters
SI 131072
SF 100.5574502 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



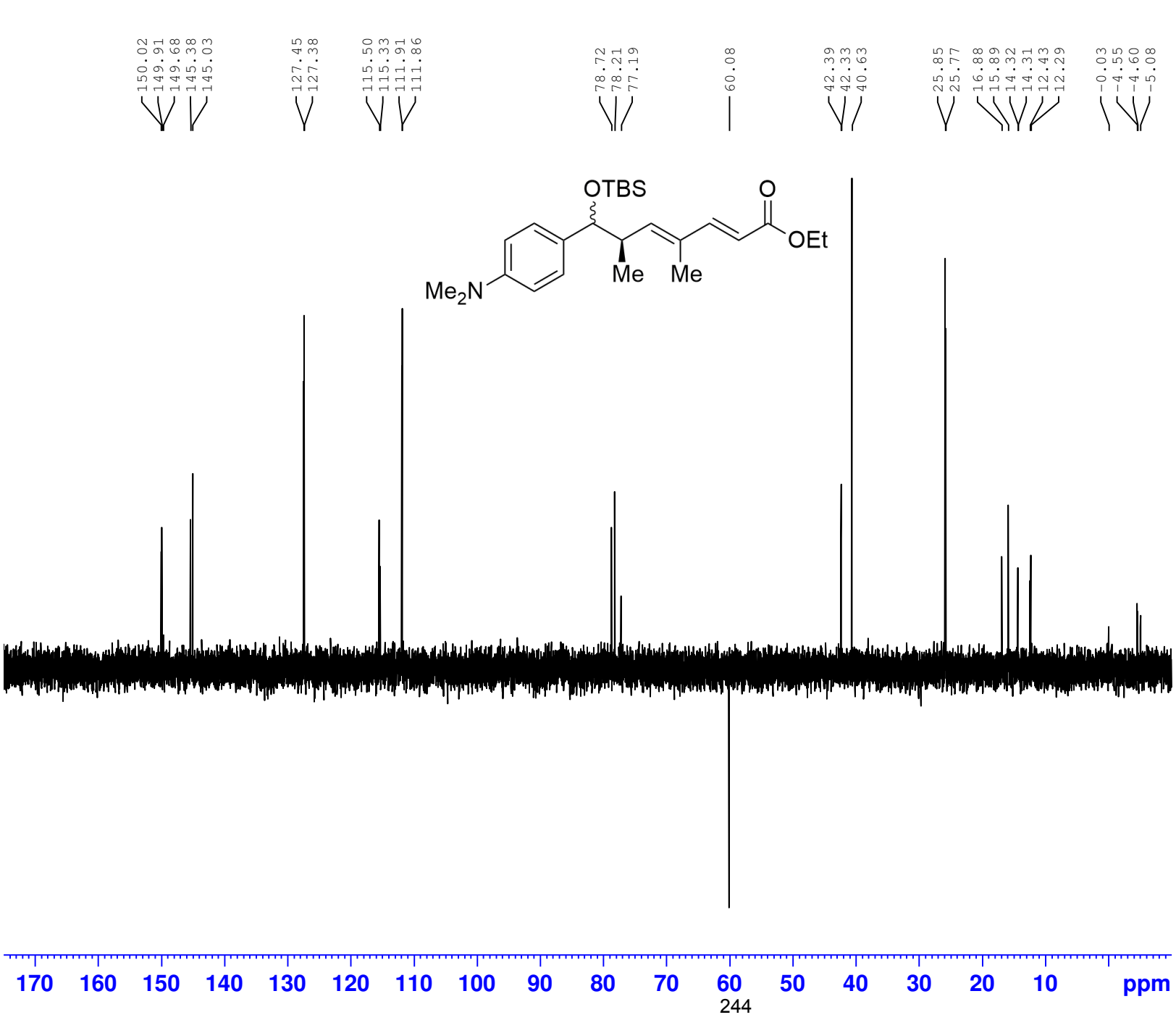

```
Current Data Parameters
NAME          III-PK-151
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200318
Time               22.15
INSTRUM        AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 96150
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.250010 Hz
AQ             1.9999200 sec
RG                   114
DW                20.800 usec
DE                 6.50 usec
TE                300.9 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1                 13C
P1                  9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2                 1H
CPDPRG[2          waltz64
PCPD2               90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                131072
SF           100.5574502 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



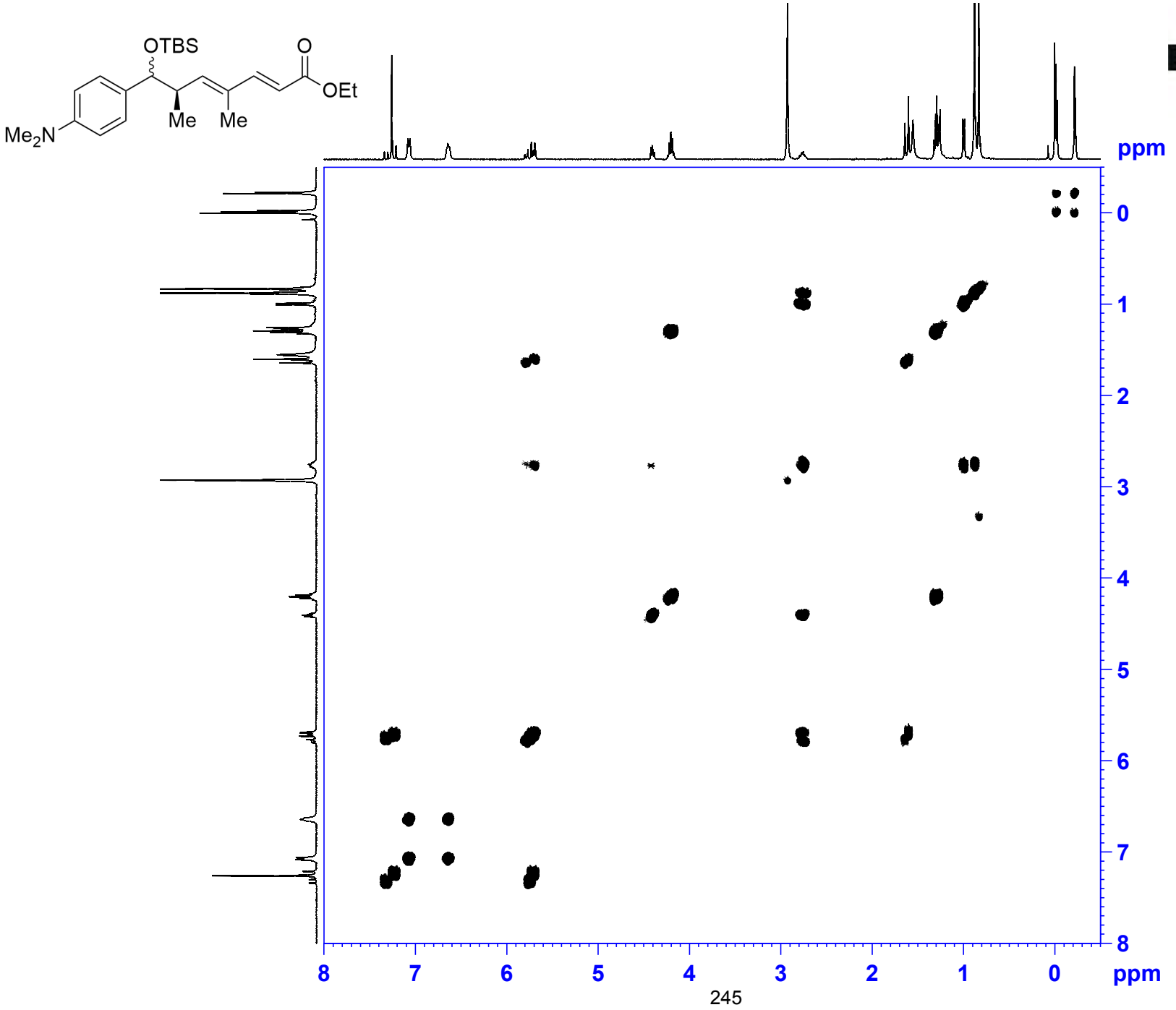
Current Data Parameters
NAME III-PK-151
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201216
Time 18.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135.b
TD 119044
SOLVENT CDCl3
NS 500
DS 4
SWH 35714.285 Hz
FIDRES 0.300009 Hz
AQ 1.6666160 sec
RG 186.92
DW 14.000 usec
DE 7.44 usec
TE 300.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178962 MHz
NUC1 13C
P1 11.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 85.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 18.08300018 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
P3 10.20 usec
P4 20.40 usec
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W

F2 - Processing parameters
SI 131072
SF 150.9028131 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                  I-PK-286
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190522
Time                     18.18
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG       cosygpmfppqf
TD                       2048
SOLVENT                  CDCl3
NS                           1
DS                           8
SWH                  3578.244 Hz
FIDRES               1.747190 Hz
AQ                   0.2861739 sec
RG                        2050
DW                    139.733 usec
DE                      6.50 usec
TE                     300.0 K
D0                   0.00000300 sec
D1                   0.85786802 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00027940 sec

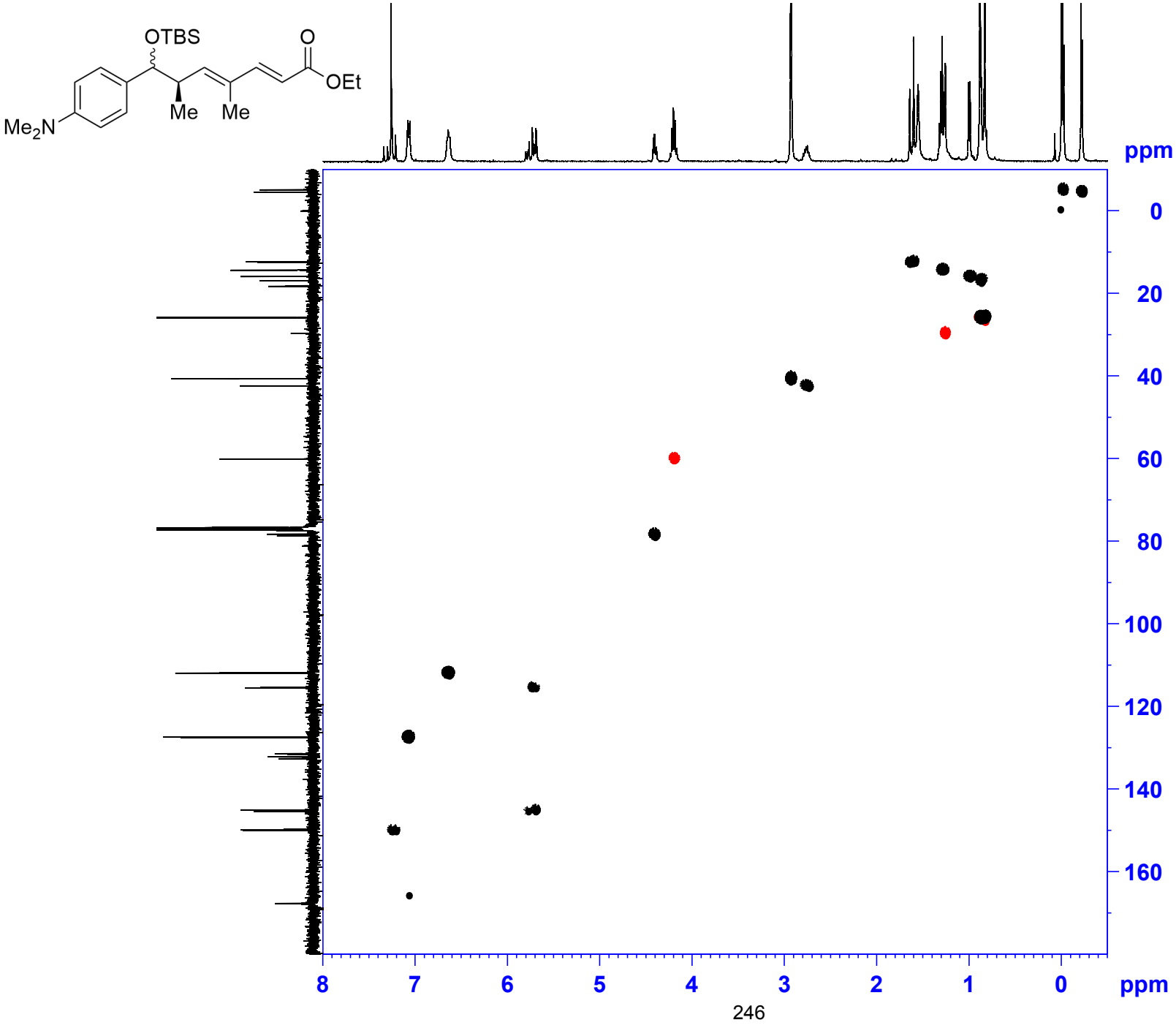
======== CHANNEL f1 ========
SFO1              399.9013360 MHz
NUC1                      1H
P1                     14.88 usec
P17                  2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]          SMSQ10.100
GPNAM[2]          SMSQ10.100
GPNAM[3]          SMSQ10.100
GPZ1                   16.00 %
GPZ2                   12.00 %
GPZ3                   40.00 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1                399.9013 MHz
FIDRES             27.961704 Hz
SW                      8.950 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                399.9000097 MHz
WDW                     SINE
SSB           0
LB            0 Hz
GB            0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                399.9000097 MHz
WDW                     SINE
SSB           0
LB            0 Hz
GB            0
```




```
Current Data Parameters
NAME                  I-PK-286
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_               20190522
Time                   18.08
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                       1024
SOLVENT                CDCl3
NS                           2
DS                          32
SWH                 4807.692 Hz
FIDRES              4.695012 Hz
AQ                0.1064960 sec
RG                        2050
DW                 104.000 usec
DE                   6.50 usec
TE                     300.1 K
CNST2             145.0000000
D0                 0.00000300 sec
D1                 0.80000001 sec
D4                 0.00172414 sec
D11                0.03000000 sec
D16                0.00020000 sec
D21                0.00360000 sec
IN0                0.00001910 sec

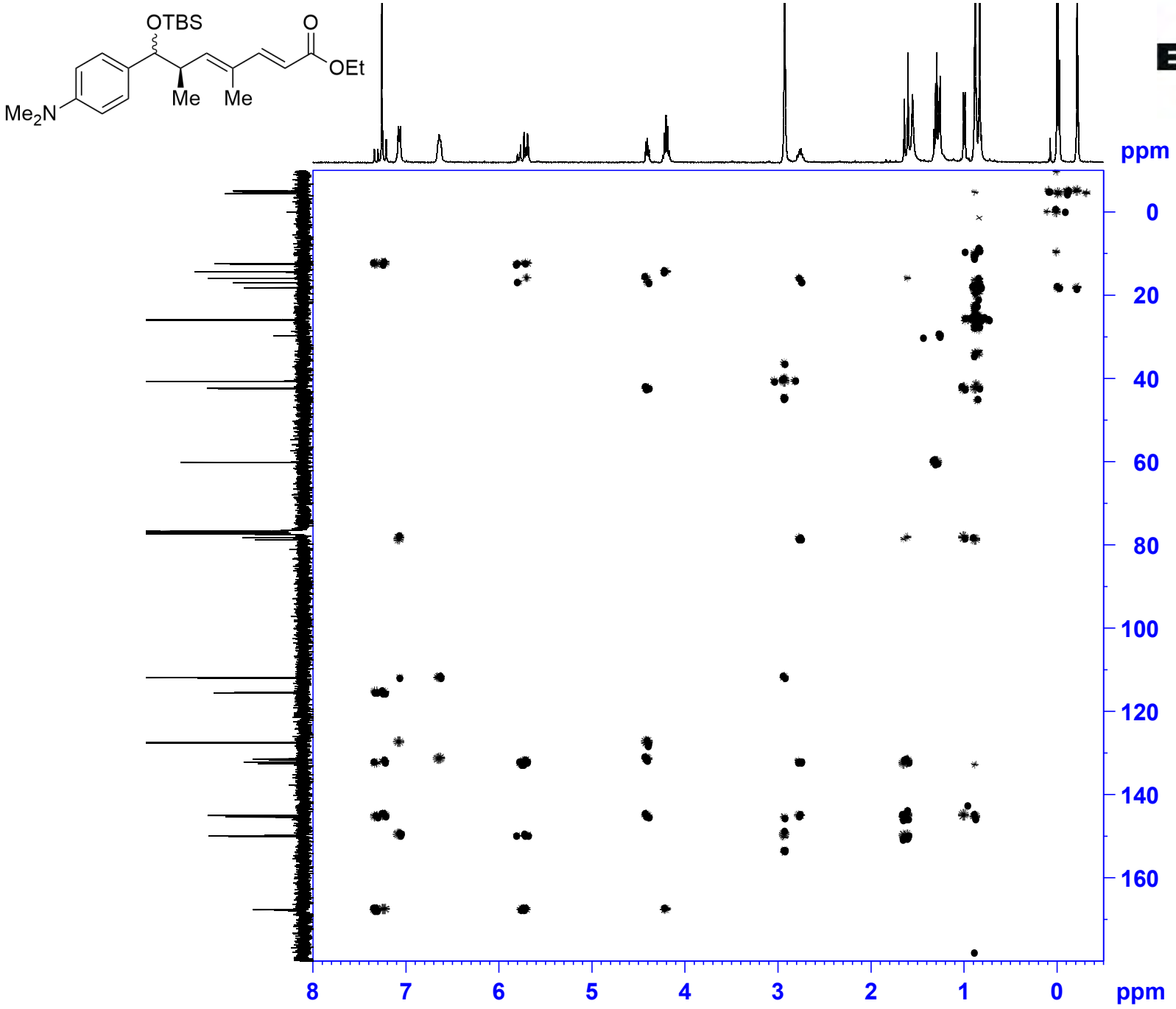
======== CHANNEL f1 ========
SFO1             399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28          0 usec
PLW1             7.59999990 W

======== CHANNEL f2 ========
SFO2             100.5670016 MHz
NUC2                      13C
CPDPRG[2                garp4
P3                      10.00 usec
P14                    500.00 usec
P31                   1900.00 usec
PCPD2                   80.00 usec
PLW0         0 W
PLW2            44.46300125 W
PLW12            0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                  0.500
SPOFFS3  0 Hz
SPW3             6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                 0.500
SPOFFS18 0 Hz
SPW18            1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                 80.00 %
GPZ2                 20.10 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       256
SFO1                100.567 MHz
FIDRES           204.515701 Hz
SW                   260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF               399.9000097 MHz
WDW                     OSINE
```




```
Current Data Parameters
NAME          III-PK-151
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201216
Time              18.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG    hmbcetgpl3nd
TD                4096
SOLVENT           CDCl3
NS                   4
DS                  16
SWH            7812.500 Hz
FIDRES         1.907349 Hz
AQ             0.2621440 sec
RG               186.92
DW               64.000 usec
DE                6.50 usec
TE                299.9 K
CNST6       120.0000000
CNST7       175.0000000
CNST13        8.0000000
CNST30        0.5981122
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06250000 sec
D16           0.00020000 sec
IN0           0.00001380 sec

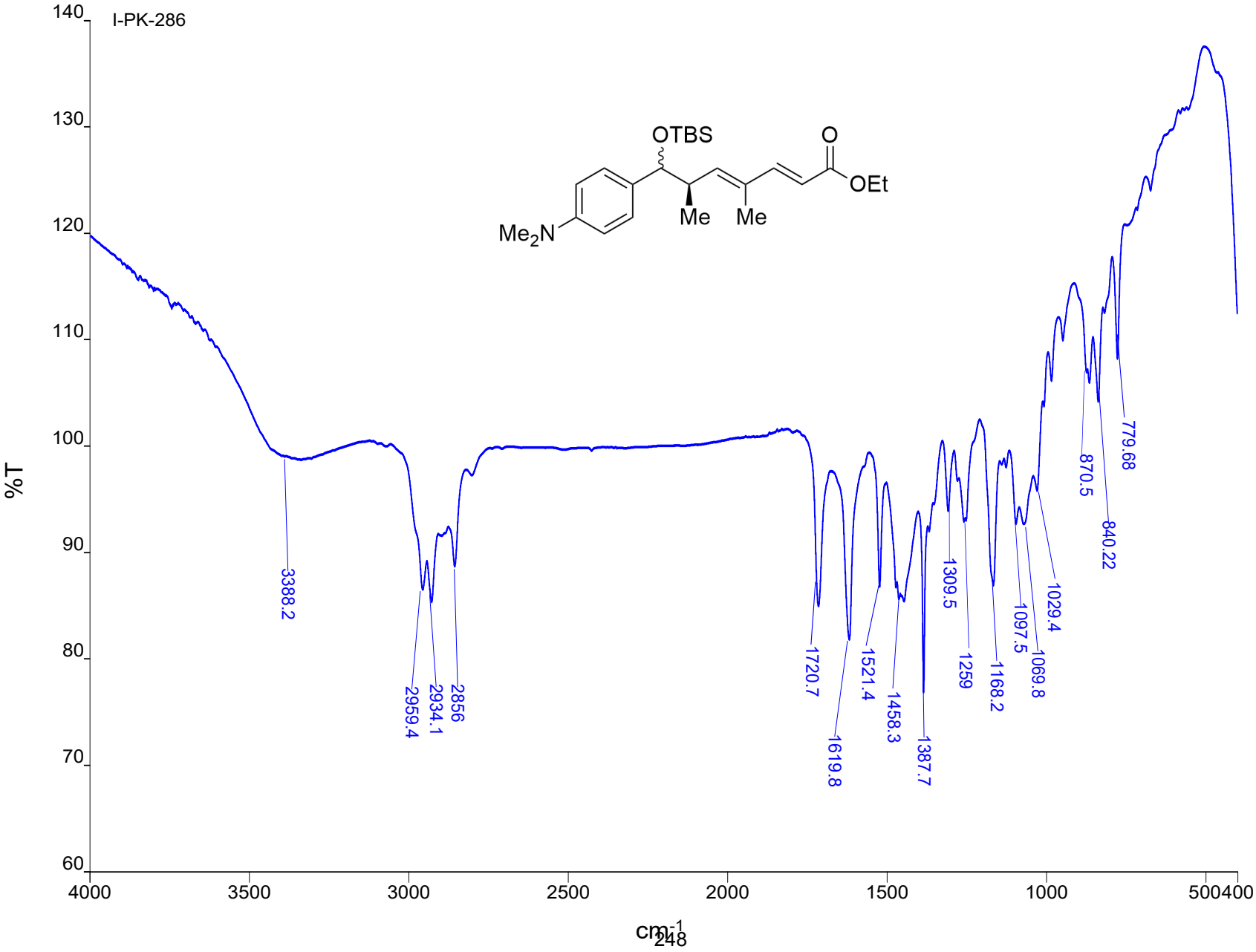
======== CHANNEL f1 ========
SFO1        600.1330006 MHz
NUC1                1H
P1                10.00 usec
P2                20.00 usec
PLW1        26.60000038 W

======== CHANNEL f2 ========
SFO2        150.9194058 MHz
NUC2                13C
P3                11.80 usec
P24             2000.00 usec
PLW2        85.00000000 W
SPNAM[7]    Crp60comp.4
SPOAL7            0.500
SPOFFS7  0 Hz
SPW7        18.08300018 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1              80.00 %
GPZ3              14.00 %
GPZ4              -8.00 %
GPZ5              -4.00 %
GPZ6              -2.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                 512
SFO1           150.9194 MHz
FIDRES       141.530792 Hz
SW              240.074 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                4096
SF          600.1300111 MHz
WDW               QSINE
SSB       0
LB        0 Hz
GB        0
PC                1.40
```


I-PK-286
OTBS
O
OEt
Me
Me
Me2N
%T
cm-1
3388.2
2959.4
2934.1
2856
1720.7
1619.8
1521.4
1458.3
1387.7
1309.5
1259
1168.2
1097.5
1069.8
1029.4
870.5
840.22
779.68

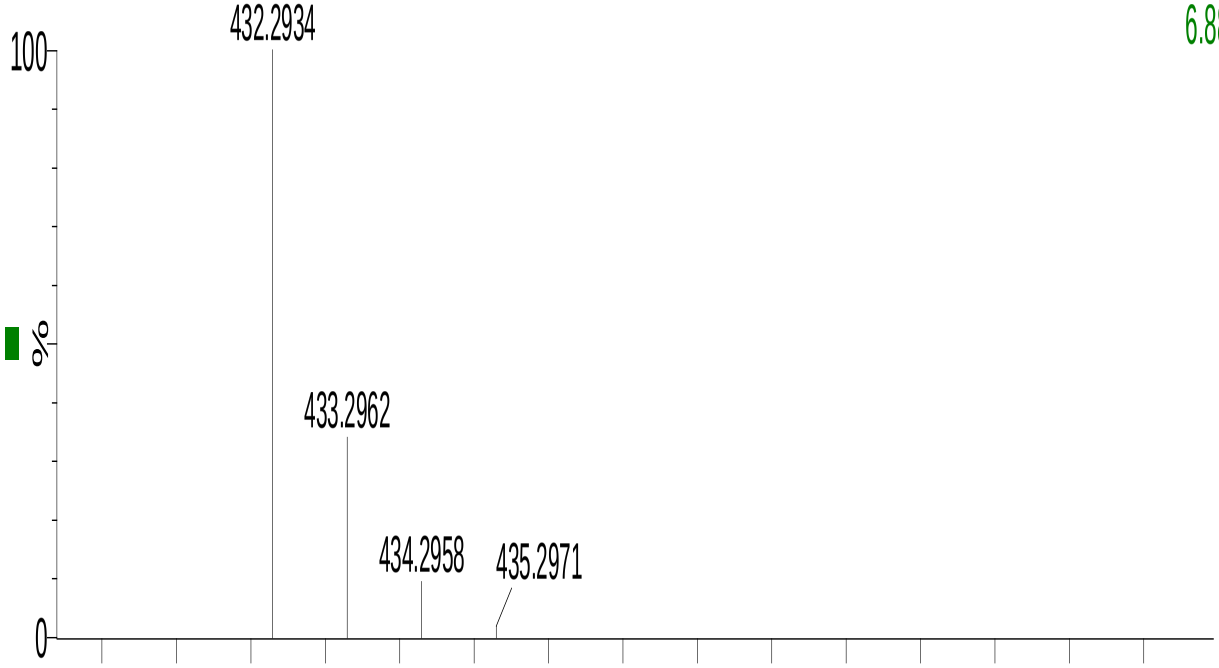
I-PK-73

01-05-2018

asep__16APR_2018_118 (0.075) Is (1.00,1.00) C25H41NSiO3H

1: TOF MS ES+

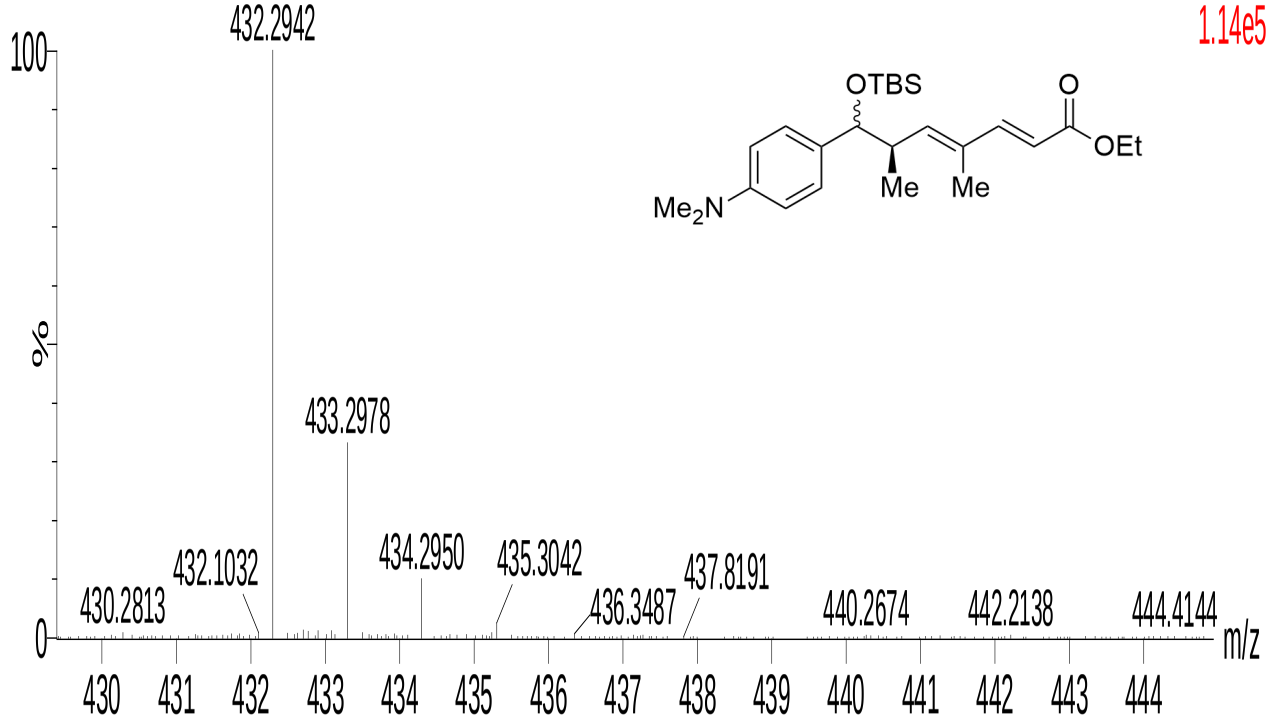
6.88e12

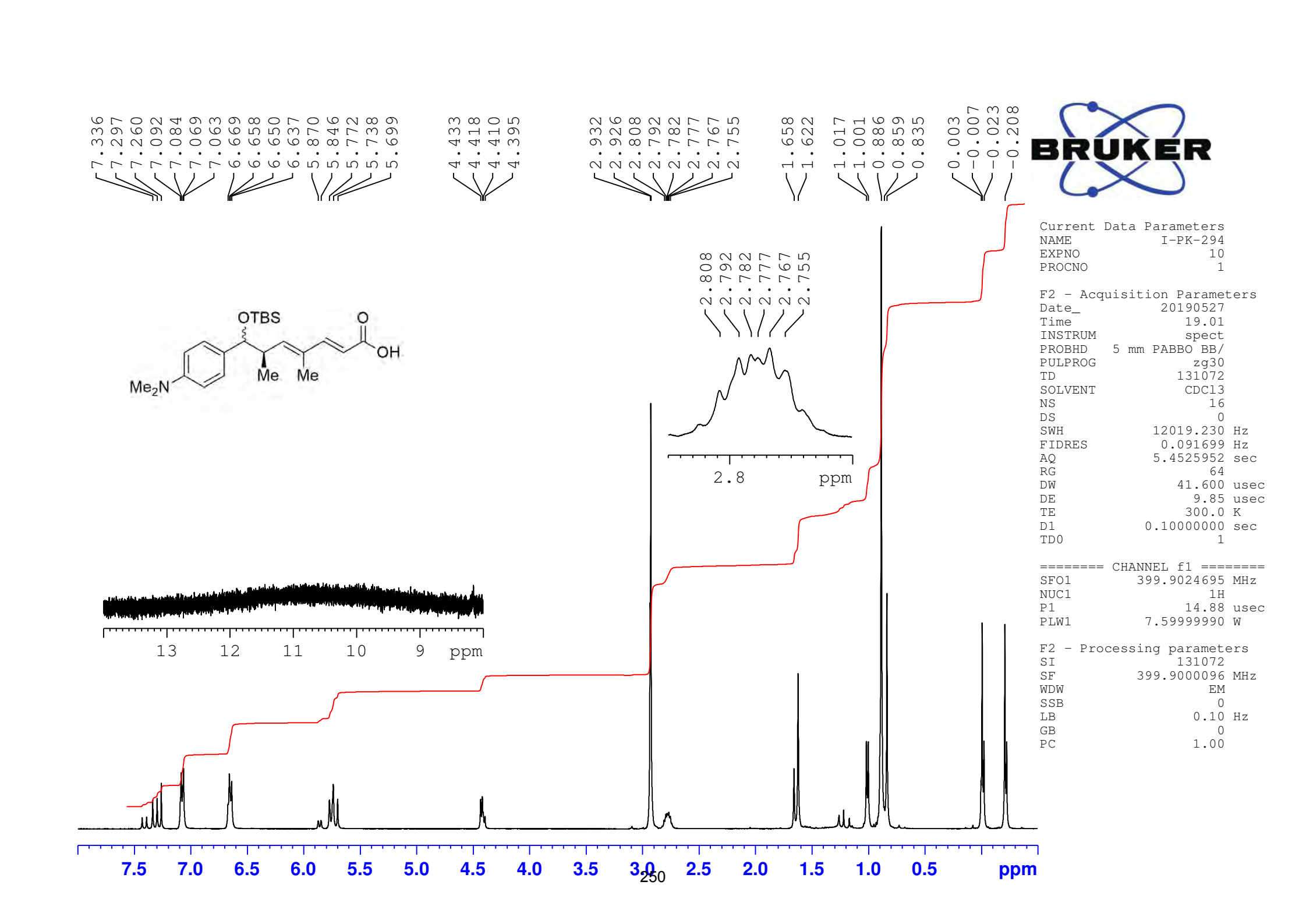


asep__16APR_2018_118 10 (0.467) Cm (10:12)

1: TOF MS ES+

1.14e5





Current Data Parameters

NAME	I-PK-294
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

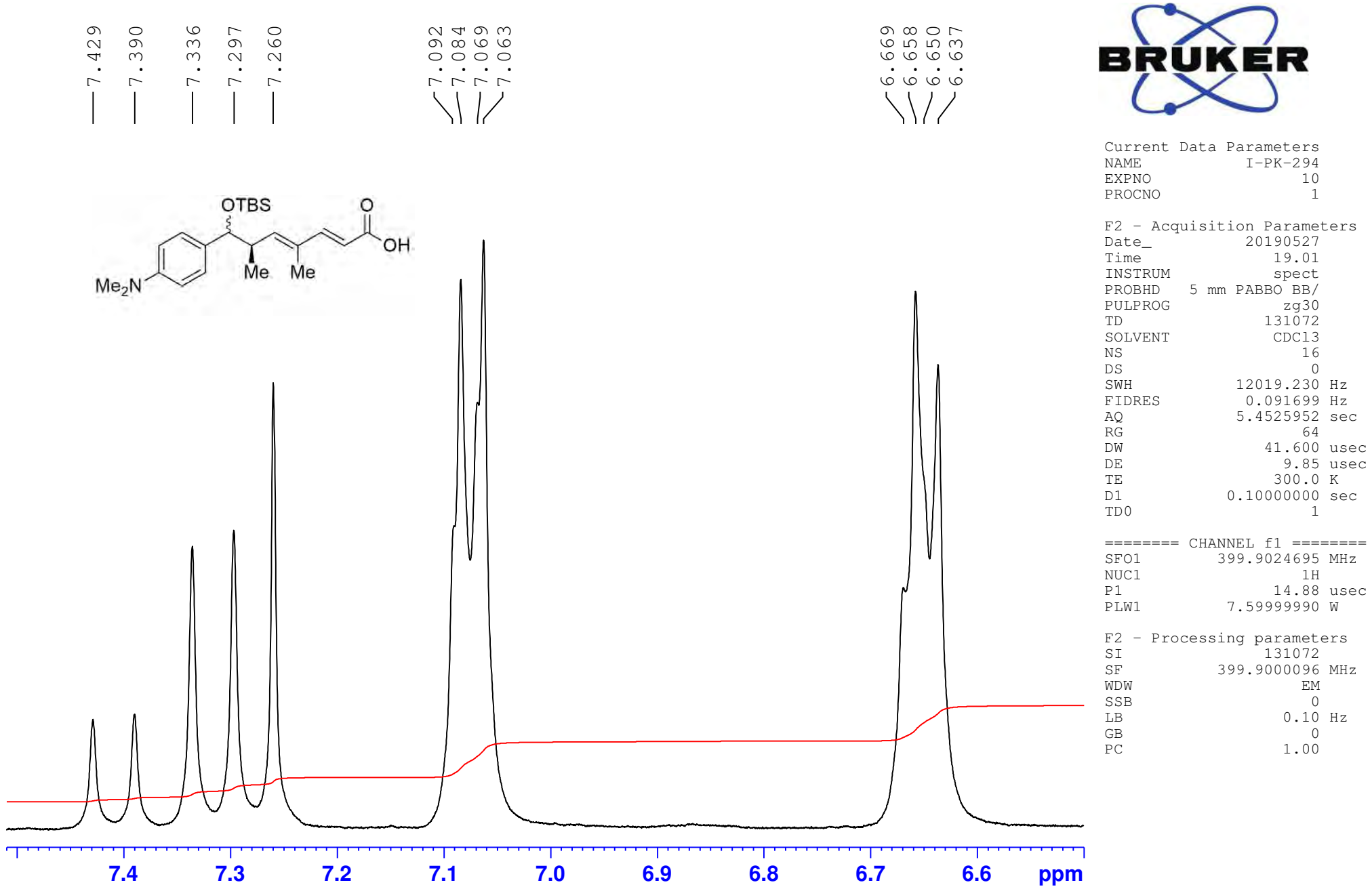
Date_	20190527
Time	19.01
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	16
DS	0
SWH	12019.230 Hz
FIDRES	0.091699 Hz
AQ	5.4525952 sec
RG	64
DW	41.600 usec
DE	9.85 usec
TE	300.0 K
D1	0.10000000 sec
TD0	1

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

SFO1	399.9024695 MHz
NUC1	1H
P1	14.88 usec
PLW1	7.59999990 W

F2 - Processing parameters

SI	131072
SF	399.9000096 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

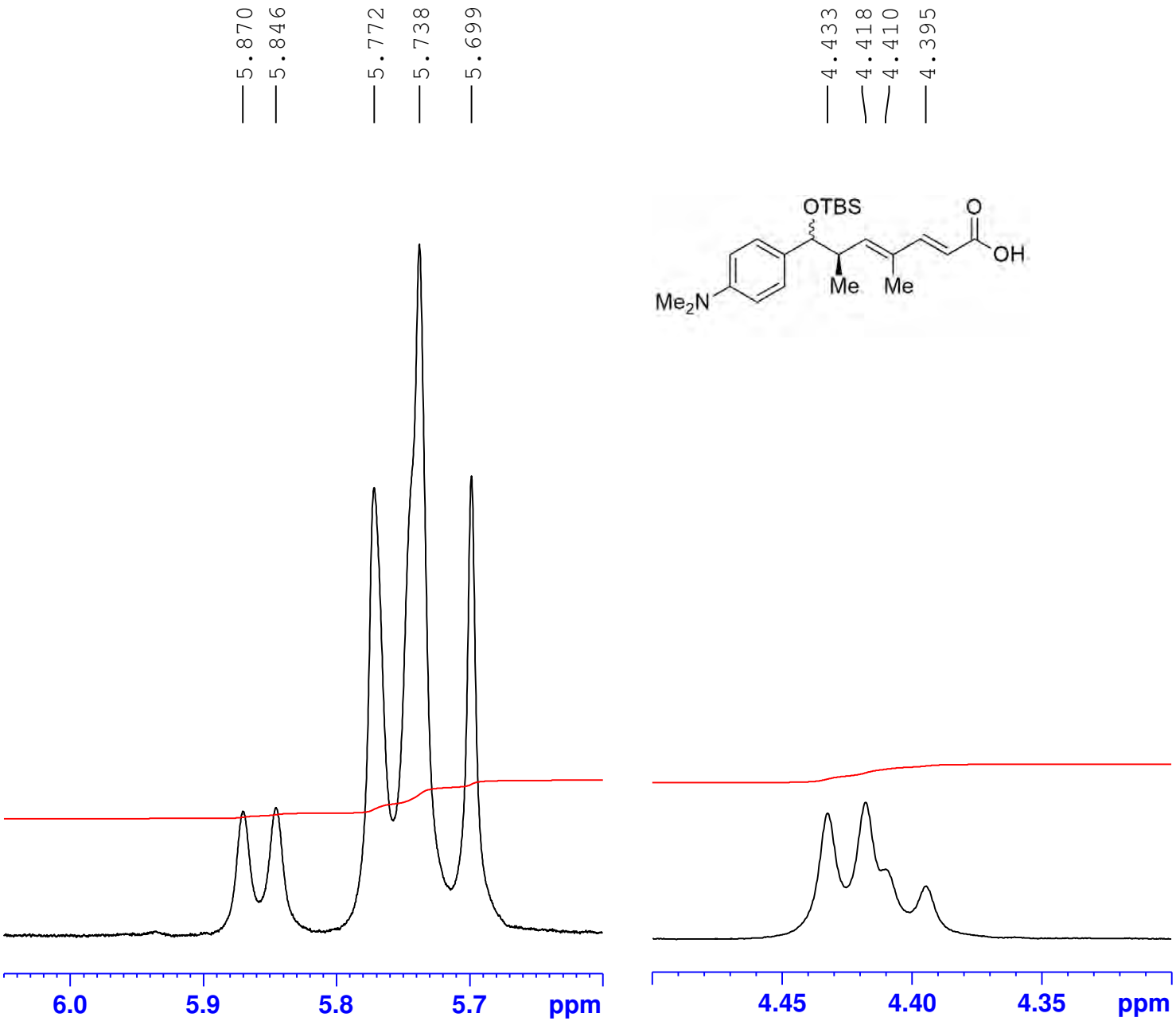



```
Current Data Parameters
NAME                  I-PK-294
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190527
Time                     19.01
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 12019.230 Hz
FIDRES                0.091699 Hz
AQ                    5.4525952 sec
RG                         64
DW                       41.600 usec
DE                        9.85 usec
TE                       300.0 K
D1                   0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9024695 MHz
NUC1                       1H
P1                       14.88 usec
PLW1                7.59999990 W

F2 - Processing parameters
SI                     131072
SF                 399.9000096 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```

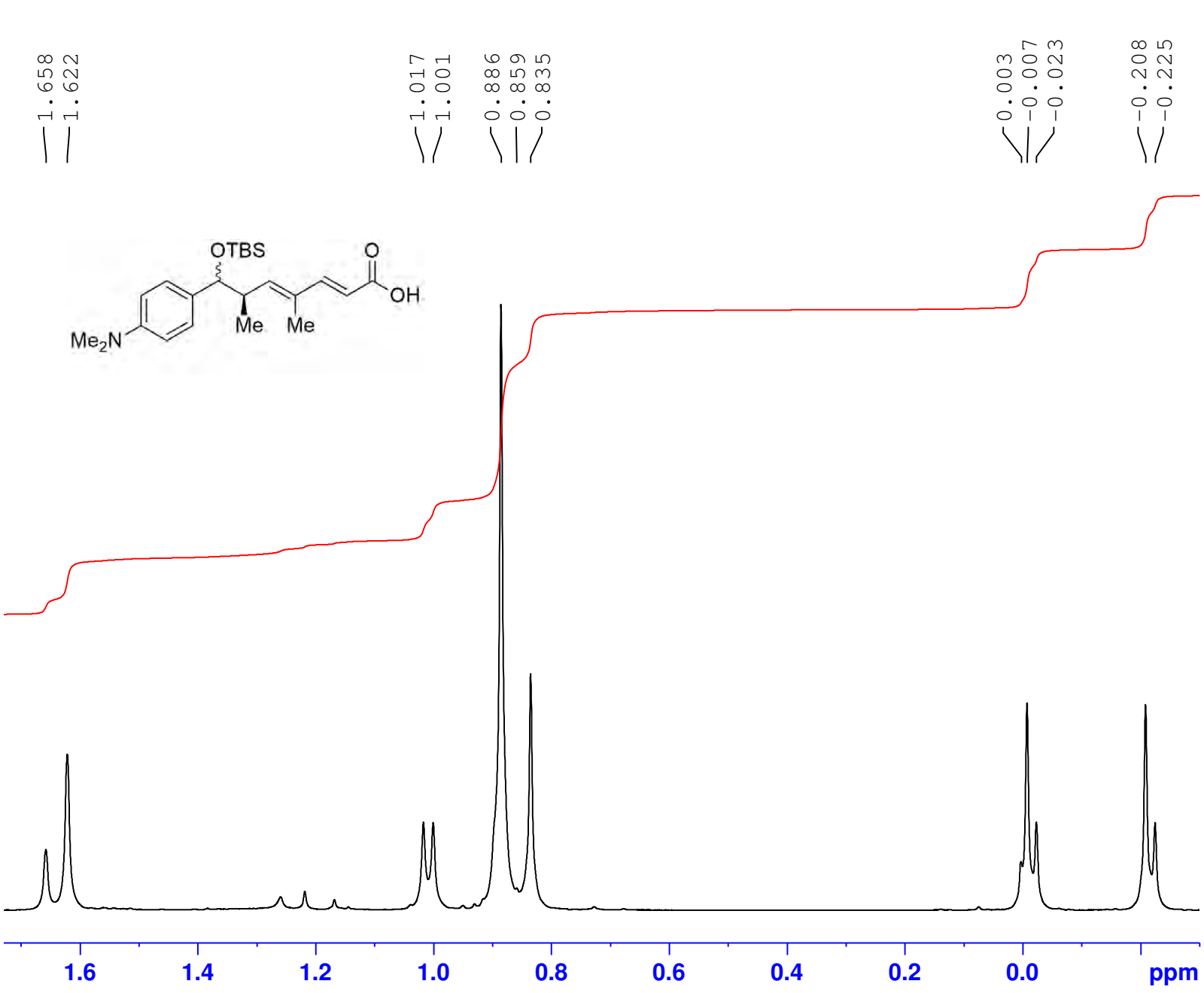



```
Current Data Parameters
NAME            I-PK-294
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190527
Time               19.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            12019.230 Hz
FIDRES          0.091699 Hz
AQ             5.4525952 sec
RG                    64
DW                41.600 usec
DE                 9.85 usec
TE                300.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9024695 MHz
NUC1                 1H
P1                14.88 usec
PLW1          7.59999990 W

F2 - Processing parameters
SI                131072
SF           399.9000096 MHz
WDW                  EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```

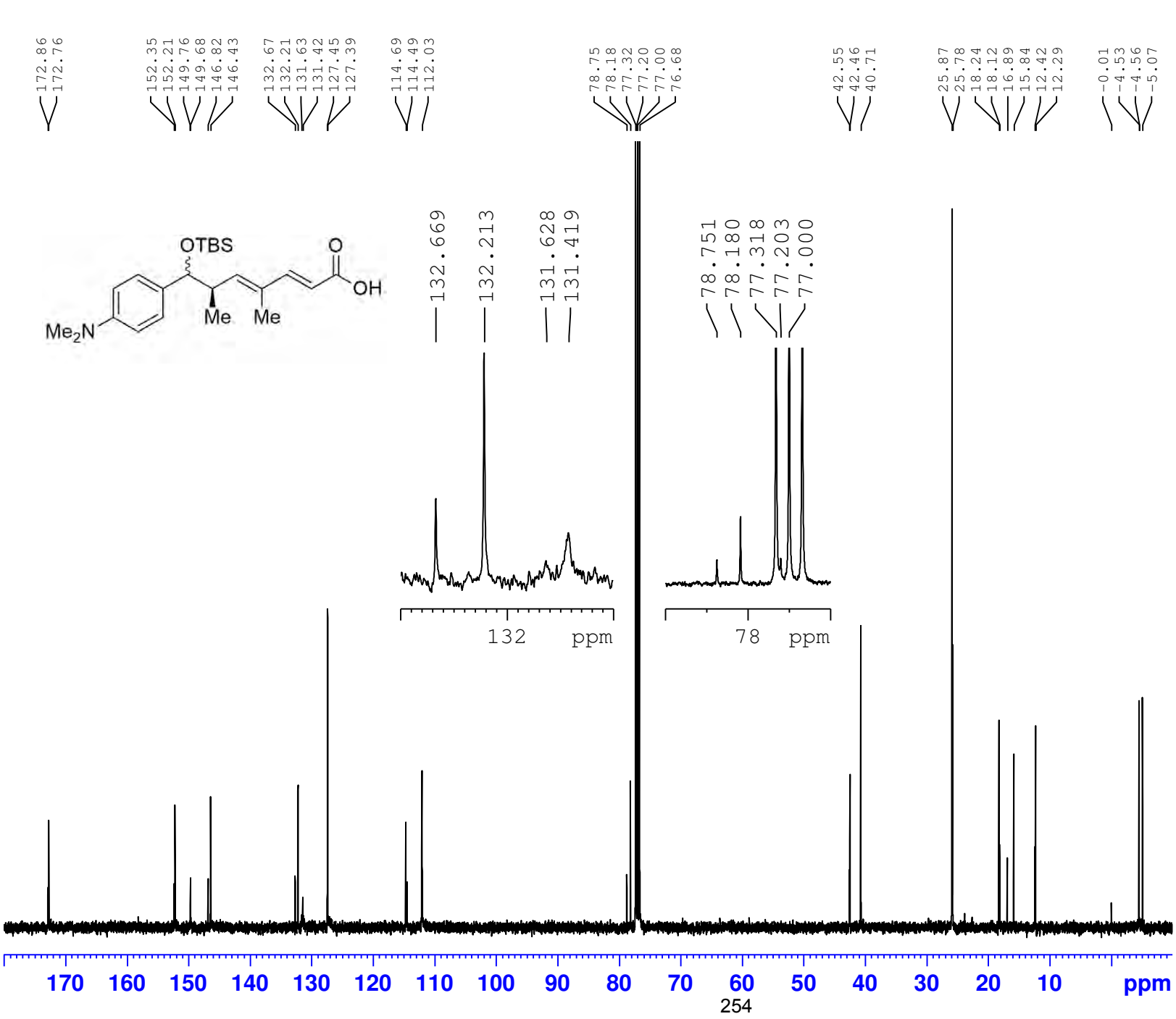


Current Data Parameters
NAME I-PK-294
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190527
Time 19.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 16
DS 0
SWH 12019.230 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 64
DW 41.600 usec
DE 9.85 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9024695 MHz
NUC1 1H
P1 14.88 usec
PLW1 7.59999990 W

F2 - Processing parameters
SI 131072
SF 399.9000096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



BRUKER

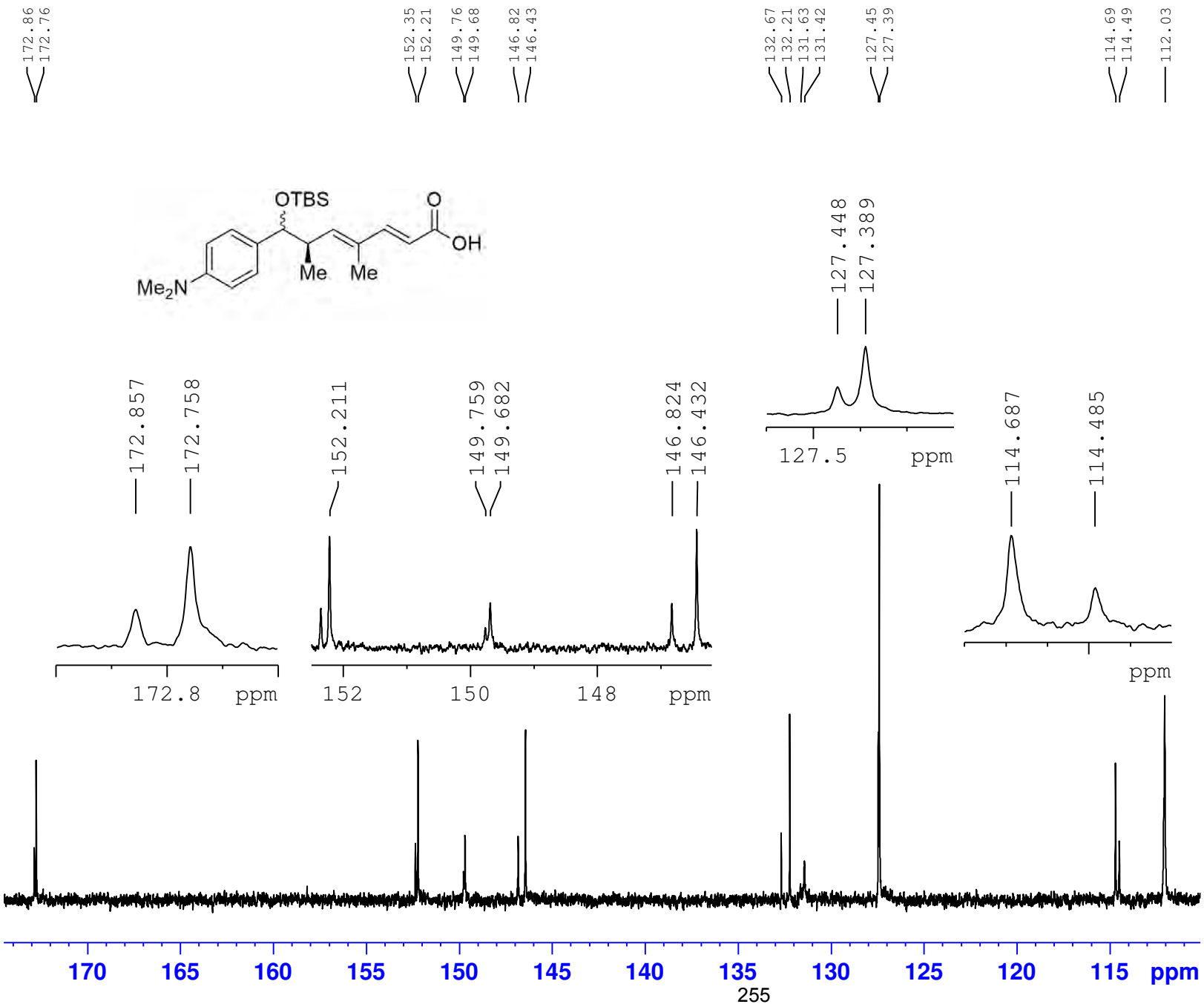
```
Current Data Parameters
NAME                  I-PK-294
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                20190528
Time                     4.02
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                      119044
SOLVENT                  CDCl3
NS                        1200
DS                           4
SWH                25000.000 Hz
FIDRES              0.210006 Hz
AQ                  2.3808801 sec
RG                        2050
DW                   20.000 usec
DE                    9.12 usec
TE                    300.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             100.5659947 MHz
NUC1                      13C
P1                     10.00 usec
PLW1             44.46300125 W

======== CHANNEL f2 ========
SFO2             399.9015996 MHz
NUC2                      1H
CPDPRG[2              waltz64
PCPD2                  90.00 usec
PLW2              7.59999990 W
PLW12             0.20774999 W
PLW13             0.16827001 W

F2 - Processing parameters
SI                       131072
SF                100.5549357 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```



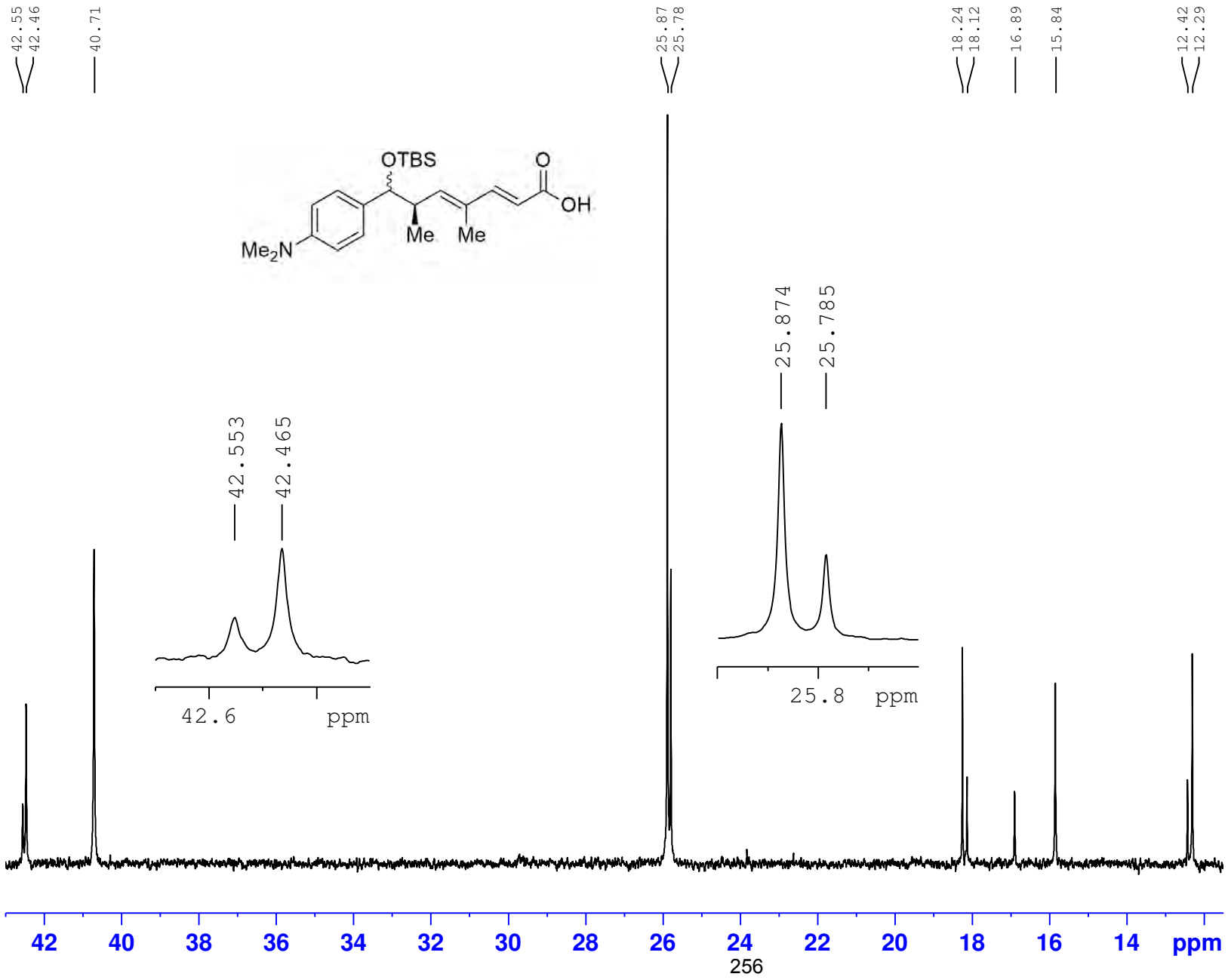
Current Data Parameters
NAME I-PK-294
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190528
Time 4.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549357 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



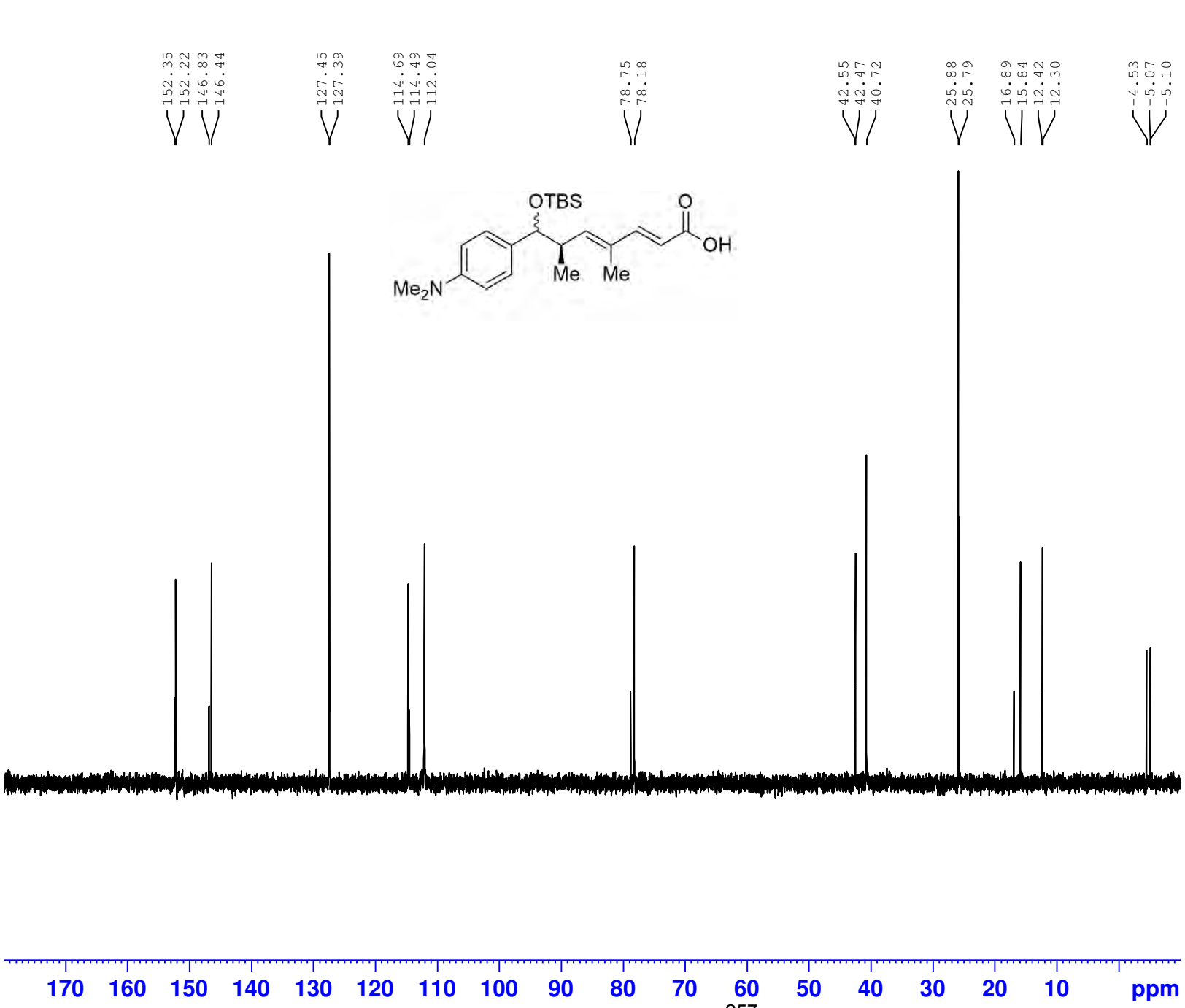
Current Data Parameters
NAME I-PK-294
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190528
Time 4.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT CDCl3
NS 1200
DS 4
SWH 25000.000 Hz
FIDRES 0.210006 Hz
AQ 2.3808801 sec
RG 2050
DW 20.000 usec
DE 9.12 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5659947 MHz
NUC1 13C
P1 10.00 usec
PLW1 44.46300125 W

======== CHANNEL f2 ========
SFO2 399.9015996 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W
PLW13 0.16827001 W

F2 - Processing parameters
SI 131072
SF 100.5549357 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	I-PK-294
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20190528
Time	4.18
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
CNST2	145.0000000
D1	2.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

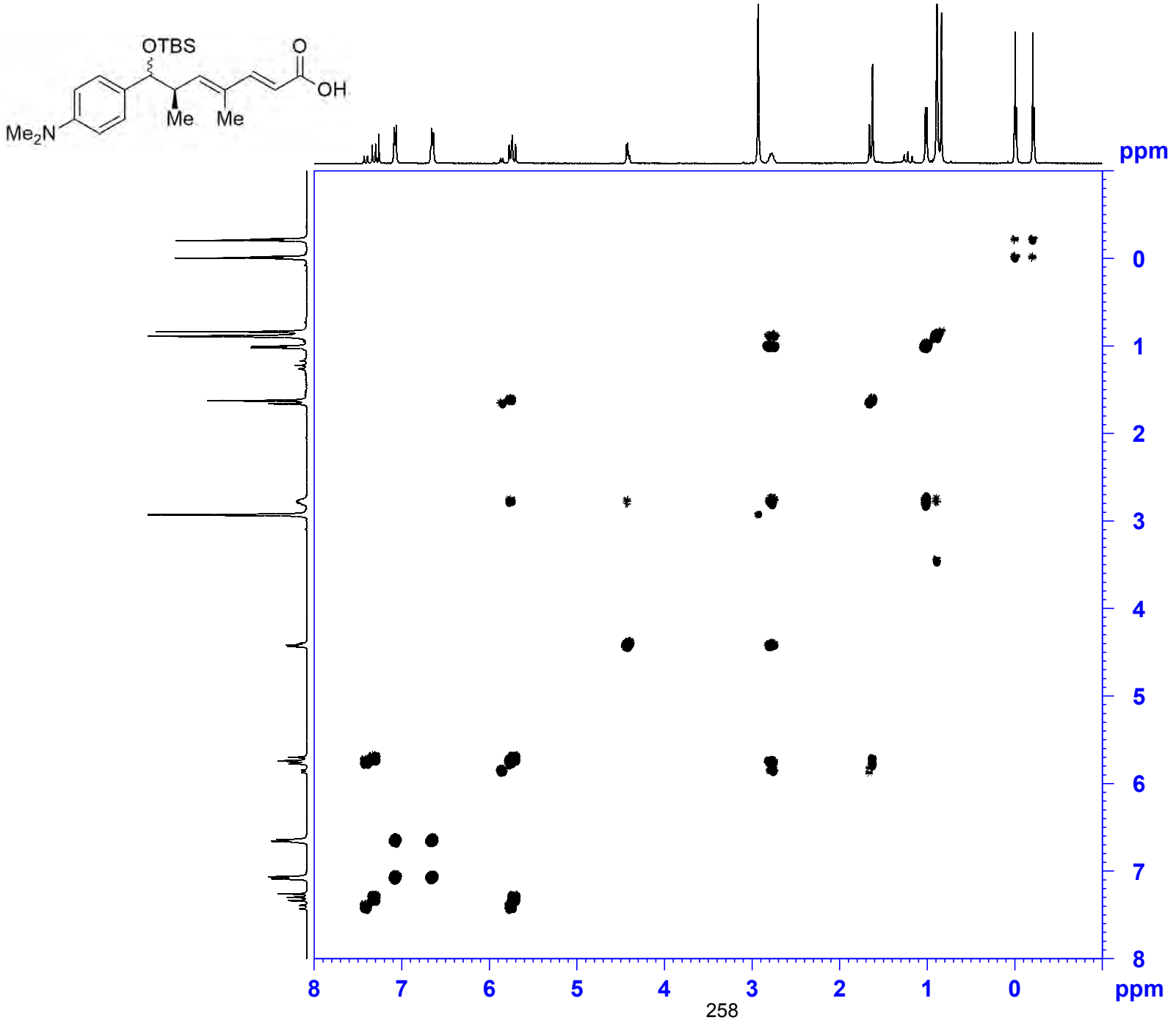
SFO1	100.5649905 MHz
NUC1	13C
P1	10.00 usec
P13	2000.00 usec
PLW0	0 W
PLW1	44.46300125 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	6.79339981 W

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

SFO2	399.9012789 MHz
NUC2	1H
CPDPRG[2	waltz16
P3	14.88 usec
P4	29.76 usec
PCPD2	90.00 usec
PLW2	7.59999990 W
PLW12	0.20774999 W

F2 - Processing parameters

SI	32768
SF	100.5549352 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40




```
Current Data Parameters
NAME                  I-PK-294
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190528
Time                      4.20
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                        2048
SOLVENT                 CDCl3
NS                           1
DS                           8
SWH                  3770.739 Hz
FIDRES               1.841181 Hz
AQ                   0.2715648 sec
RG                        2050
DW                     132.600 usec
DE                       6.50 usec
TE                      300.0 K
D0                   0.00000300 sec
D1                   0.87425190 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00026520 sec

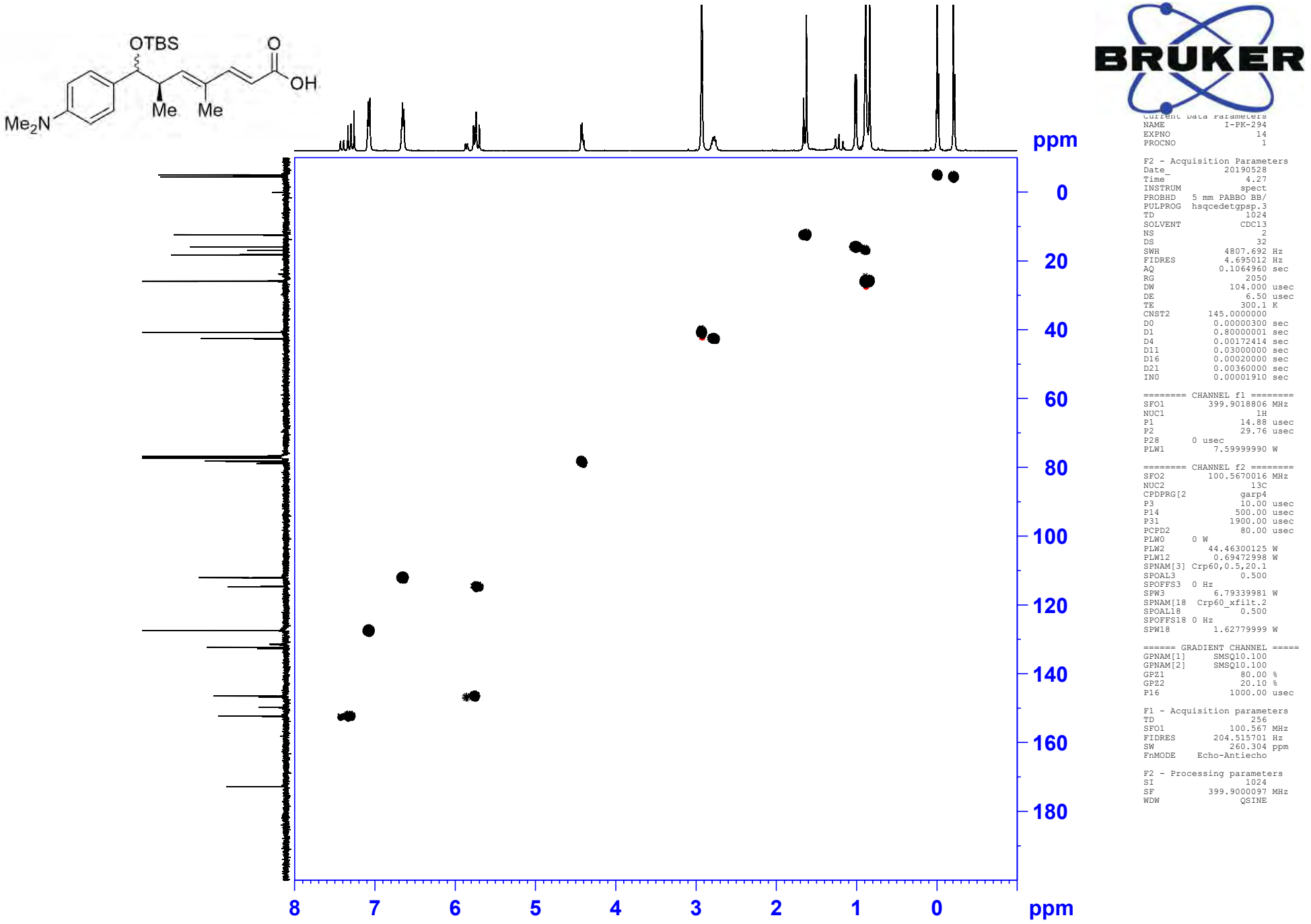
======== CHANNEL f1 ========
SFO1               399.9013941 MHz
NUC1                       1H
P1                      14.88 usec
P17                   2500.00 usec
PLW1                7.59999990 W
PLW10               2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]           SMSQ10.100
GPNAM[2]           SMSQ10.100
GPNAM[3]           SMSQ10.100
GPZ1                    16.00 %
GPZ2                    12.00 %
GPZ3                    40.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                         256
SFO1                399.9014 MHz
FIDRES             29.458899 Hz
SW                      9.429 ppm
FnMODE                     QF

F2 - Processing parameters
SI                        1024
SF                  399.9000098 MHz
WDW                       SINE
SSB                  0
LB                   0 Hz
GB                   0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                        QF
SF                  399.9000103 MHz
WDW                       SINE
SSB                  0
LB                   0 Hz
GB                   0
```




```
Current Data Parameters
NAME                 I-PK-294
EXPNO                      14
PROCNO                      1

F2 - Acquisition Parameters
Date_              20190528
Time                   4.27
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                      1024
SOLVENT              CDCl3
NS                         2
DS                        32
SWH                4807.692 Hz
FIDRES             4.695012 Hz
AQ                 0.1064960 sec
RG                      2050
DW                  104.000 usec
DE                    6.50 usec
TE                    300.1 K
CNST2             145.0000000
D0                 0.00000300 sec
D1                 0.80000001 sec
D4                 0.00172414 sec
D11                0.03000000 sec
D16                0.00020000 sec
D21                0.00360000 sec
IN0                0.00001910 sec

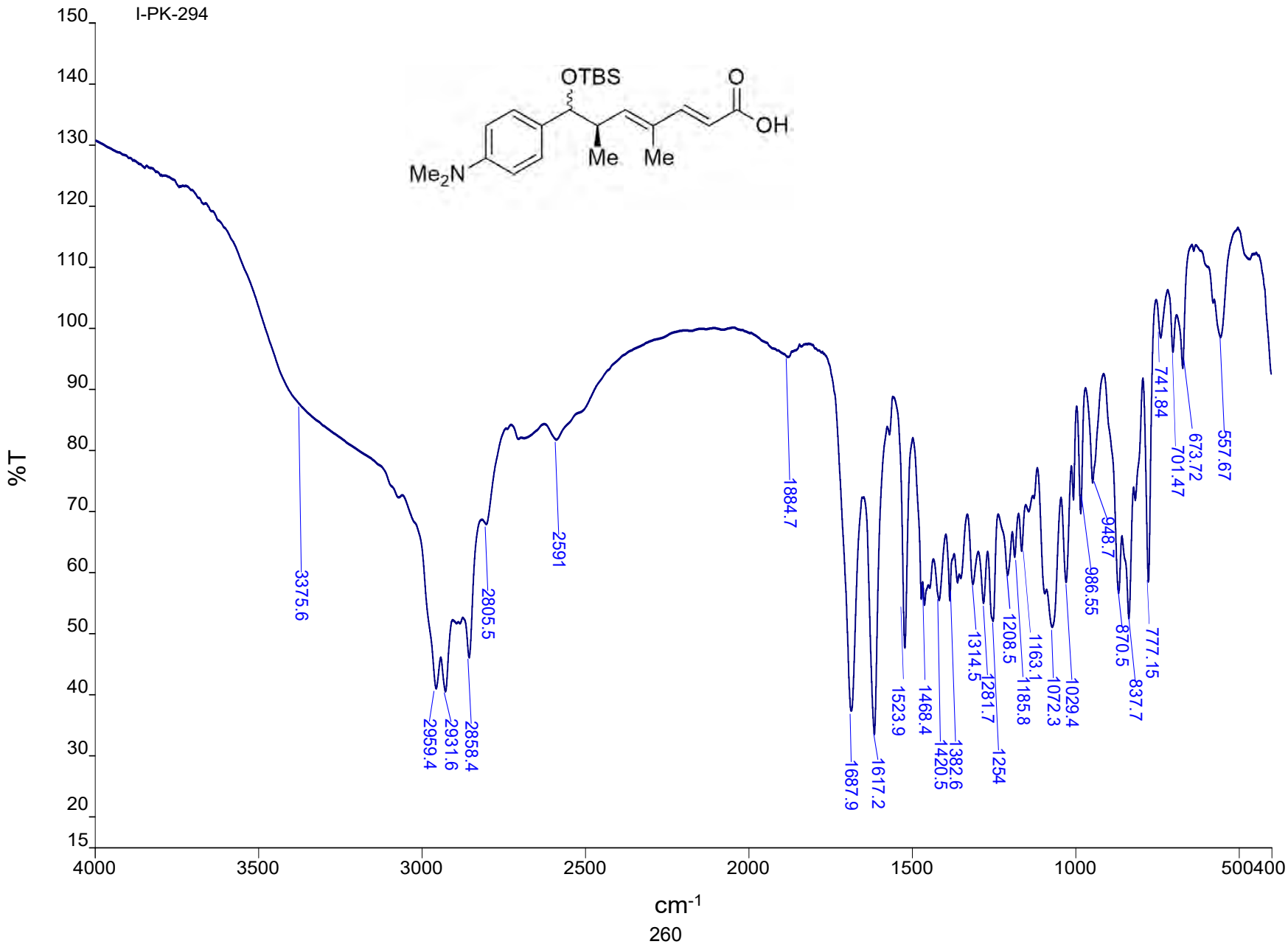
======== CHANNEL f1 ========
SFO1             399.9018806 MHz
NUC1                       1H
P1                      14.88 usec
P2                      29.76 usec
P28          0 usec
PLW1              7.59999990 W

======== CHANNEL f2 ========
SFO2             100.5670016 MHz
NUC2                      13C
CPDPRG[2                garp4
P3                      10.00 usec
P14                    500.00 usec
P31                   1900.00 usec
PCPD2                   80.00 usec
PLW0         0 W
PLW2             44.46300125 W
PLW12             0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                  0.500
SPOFFS3   0 Hz
SPW3              6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                 0.500
SPOFFS18 0 Hz
SPW18             1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                   80.00 %
GPZ2                   20.10 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1                100.567 MHz
FIDRES           204.515701 Hz
SW                  260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF                399.9000097 MHz
WDW                     QSINE
```


I-PK-294
OTBS
O
OH
Me
Me
Me2N
%T
150
140
130
120
110
100
90
80
70
60
50
40
30
20
15
4000
3500
3000
2500
2000
1500
1000
500400
cm-1
3375.6
2959.4
2931.6
2858.4
2805.5
2591
1884.7
1687.9
1617.2
1523.9
1468.4
1420.5
1382.6
1314.5
1281.7
1254
1208.5
1185.8
1163.1
1072.3
1029.4
986.55
948.7
870.5
837.7
777.15
741.84
701.47
673.72
557.67

260

Mass Spectrometry Result Sheet

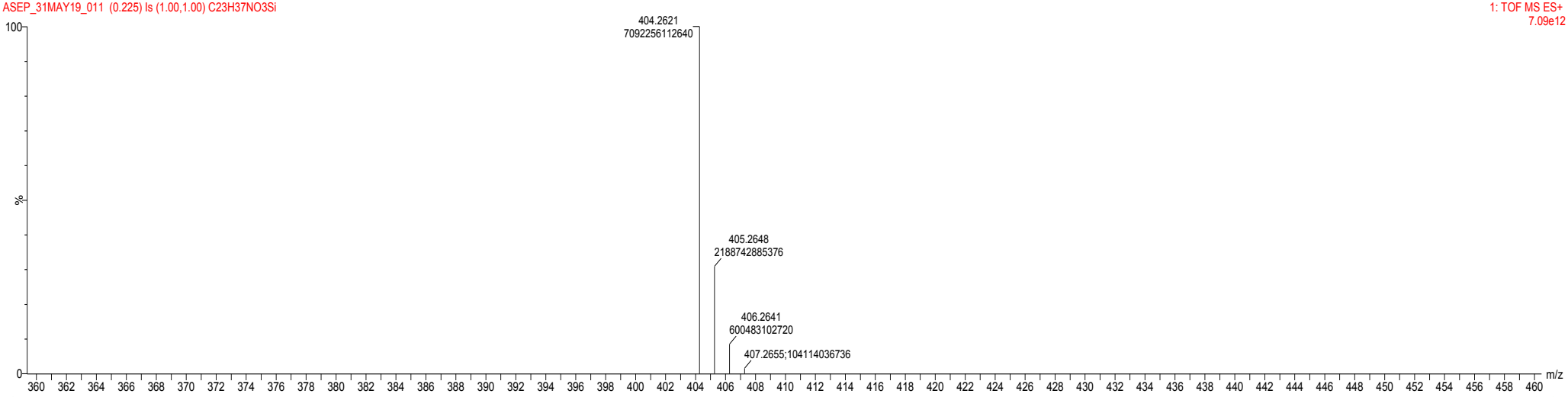
Waters Xevo G2-XS QTof Mass Spectrometer

31-05-2019

I-PK-294

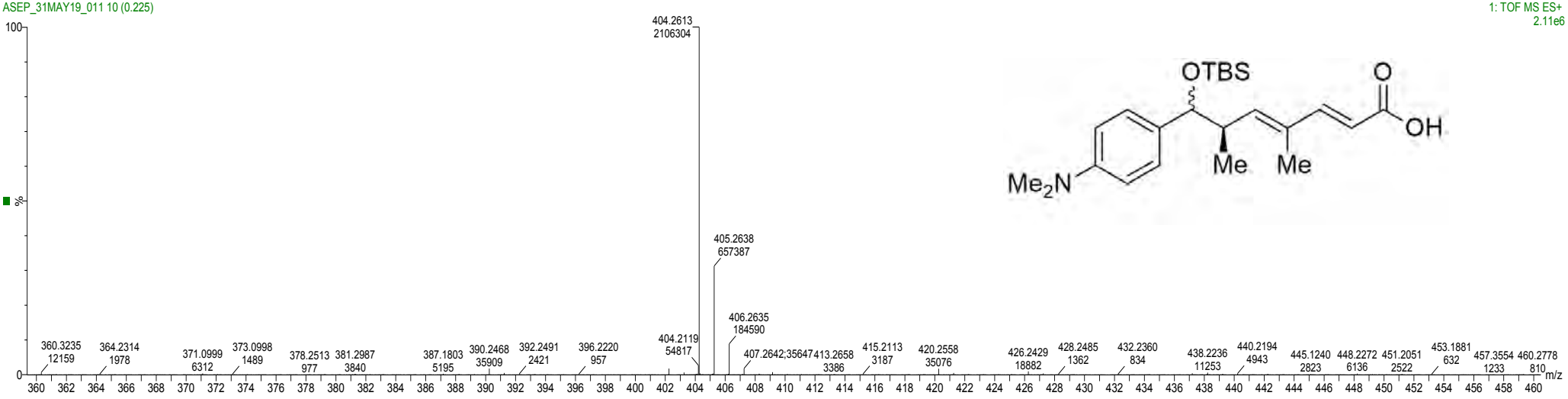
ASEP_31MAY19_011 (0.225) Is (1.00,1.00) C23H37NO3Si

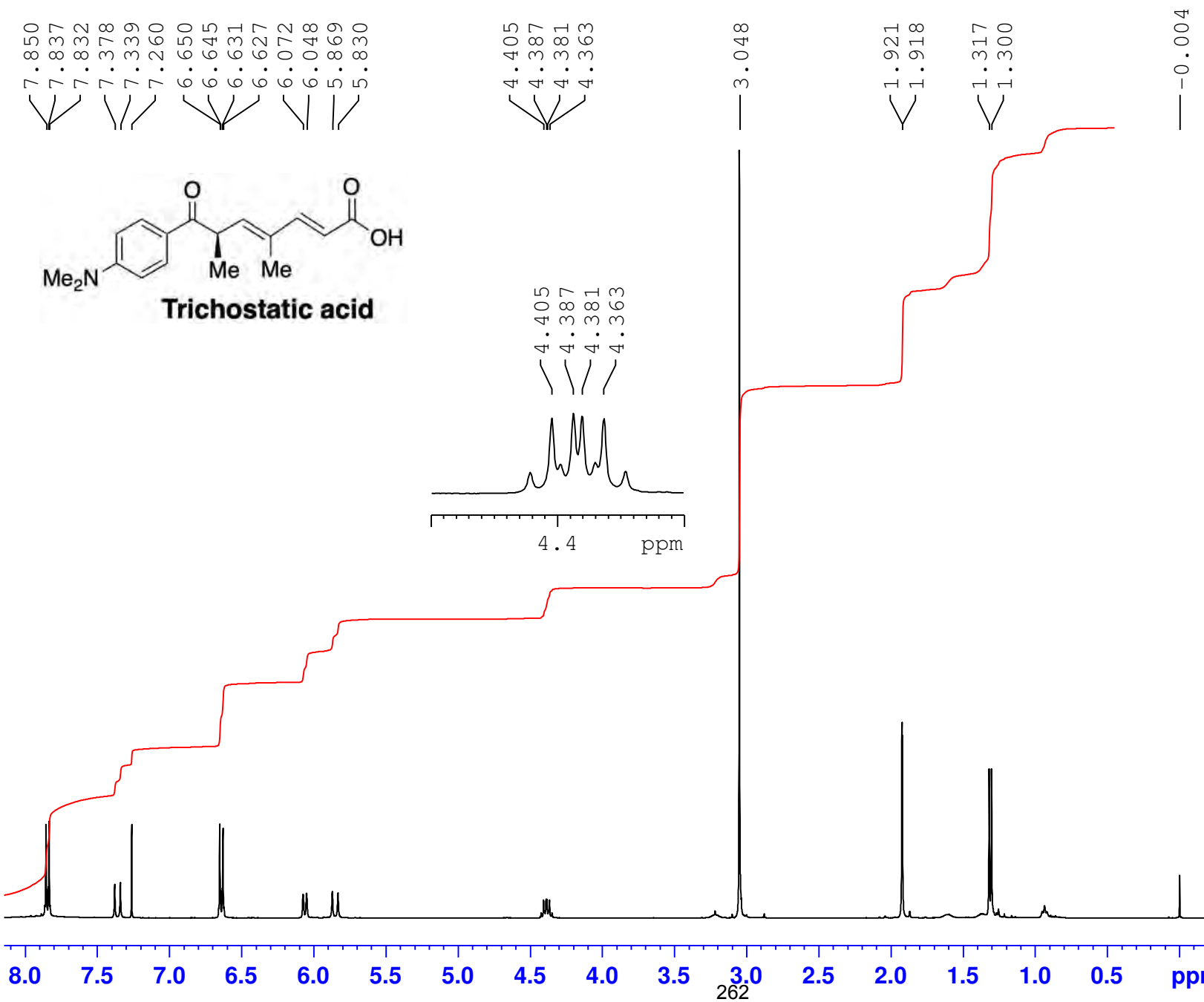
1: TOF MS ES+
7.09e12



ASEP_31MAY19_011 10 (0.225)

1: TOF MS ES+
2.11e6





BRUKER

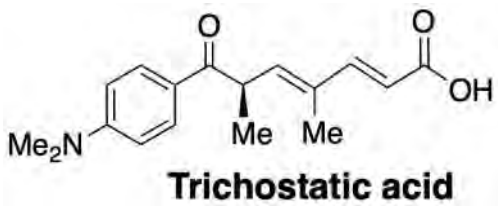
```
Current Data Parameters
NAME                  I-PK-296
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190604
Time                     13.21
INSTRUM              AVIII_400
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9845889 sec
RG                         80.6
DW                       60.800 usec
DE                       16.82 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                         1

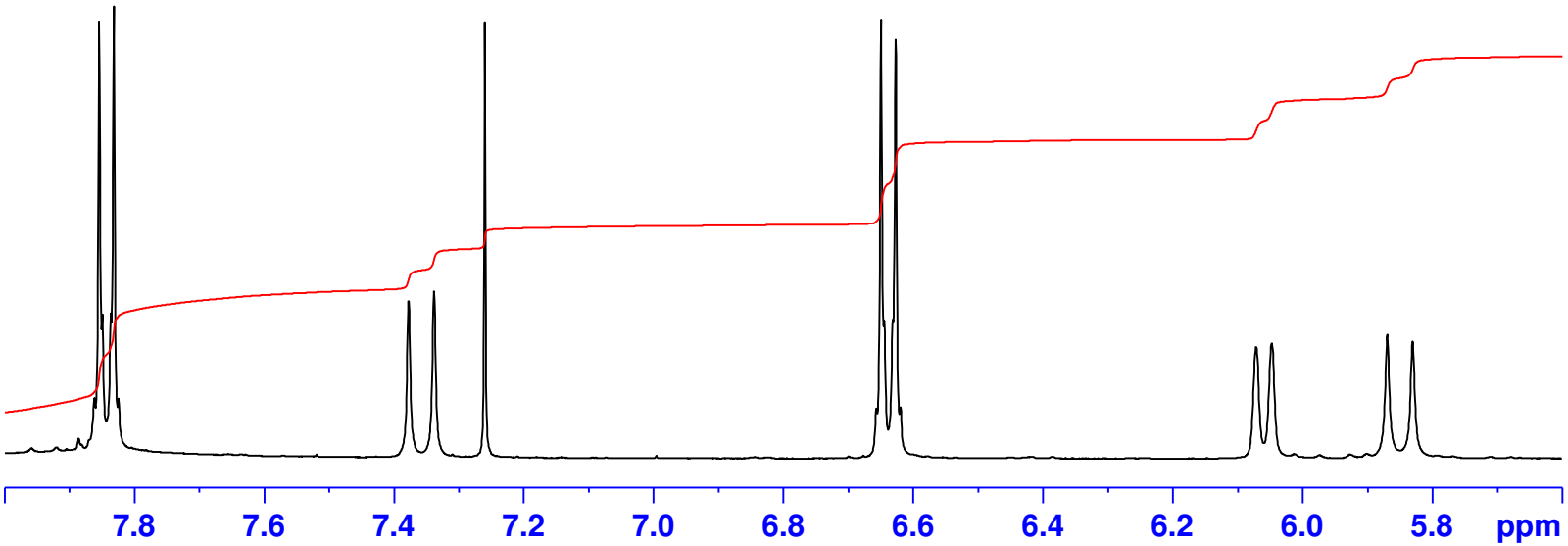
======== CHANNEL f1 ========
SFO1                400.1124708 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                          32768
SF                  400.1100094 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

7.855
7.850
7.837
7.832
7.378
7.339
7.260
6.650
6.645
6.631
6.627
6.072
6.048
5.869
5.830



Trichostatic acid

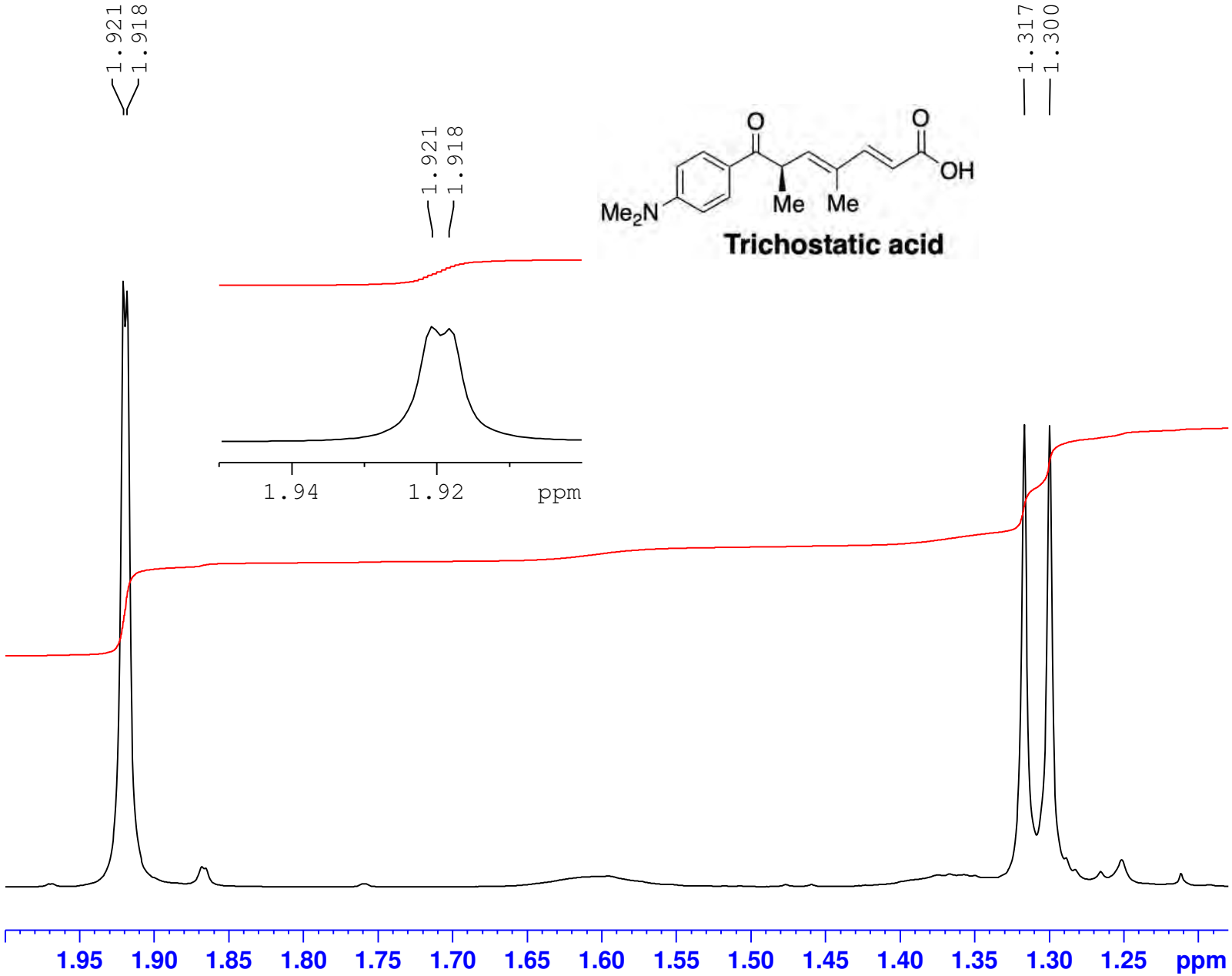



```
Current Data Parameters
NAME                  I-PK-296
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20190604
Time                     13.21
INSTRUM               AVIII_400
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9845889 sec
RG                         80.6
DW                       60.800 usec
DE                       16.82 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1124708 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                          32768
SF                  400.1100094 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



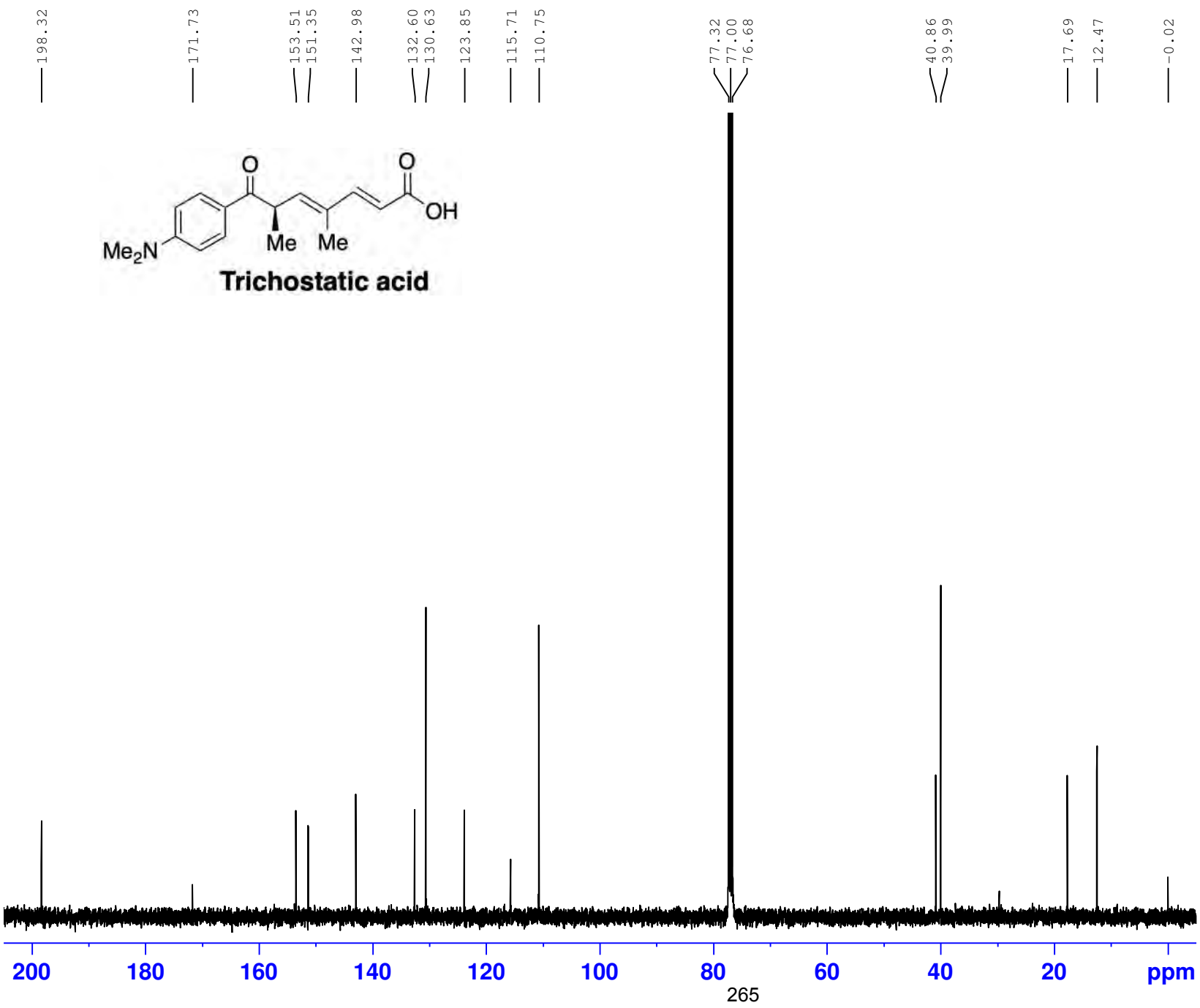
BRUKER

Current Data Parameters
NAME I-PK-296
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190604
Time 13.21
INSTRUM AVIII_400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 16.82 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1124708 MHz
NUC1 1H
P1 15.00 usec
PLW1 17.29199982 W

F2 - Processing parameters
SI 32768
SF 400.1100094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME I-PK-TSACID04
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_	20190722
Time	18.26
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	119044
SOLVENT	CDCl3
NS	1200
DS	4
SWH	25000.000 Hz
FIDRES	0.210006 Hz
AQ	2.3808801 sec
RG	2050
DW	20.000 usec
DE	9.12 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1

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

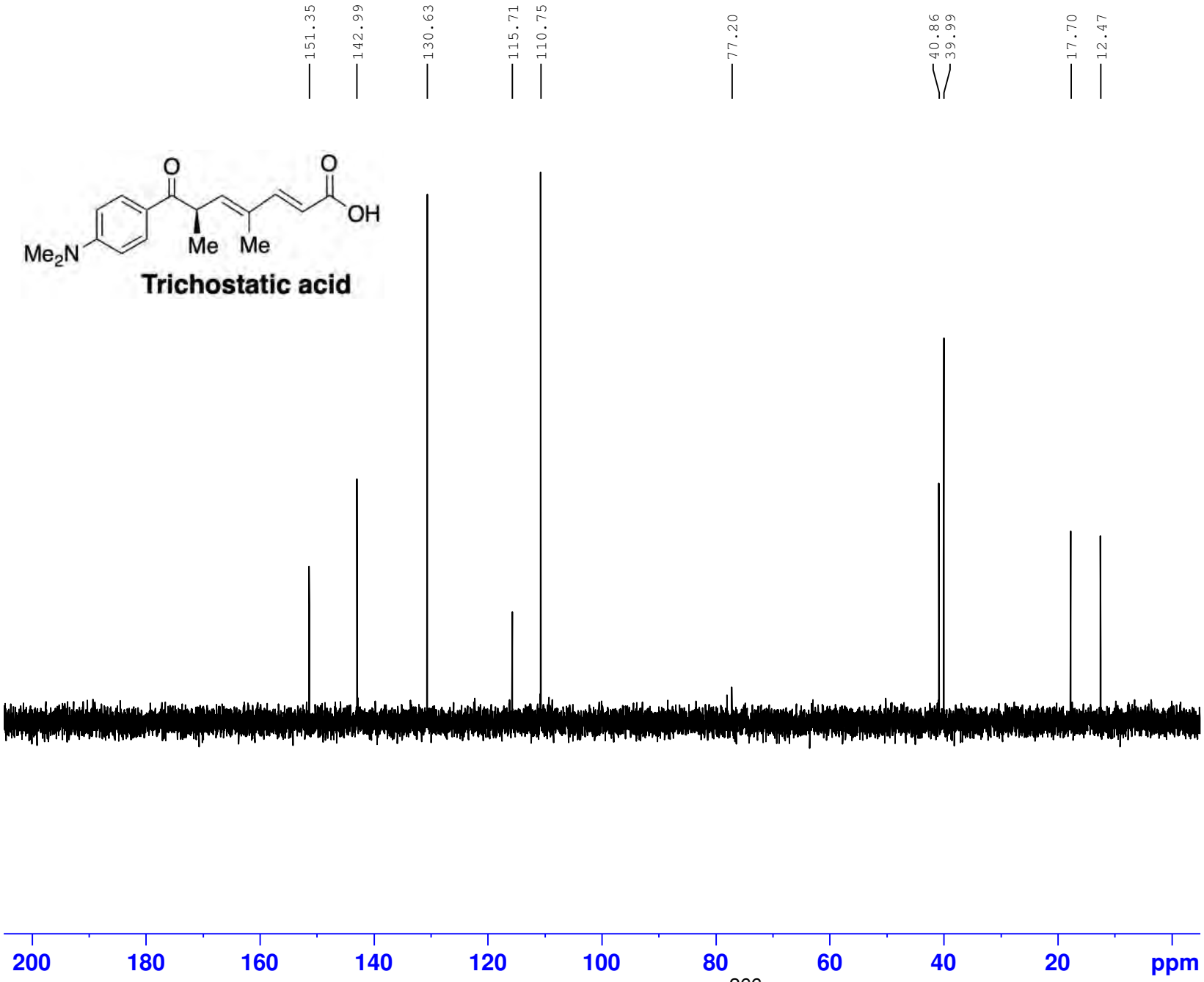
SFO1	100.5659947 MHz
NUC1	13C
P1	10.00 usec
PLW1	44.46300125 W

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

SFO2	399.9015996 MHz
NUC2	1H
CPDPRG[2	waltz64
PCPD2	90.00 usec
PLW2	7.59999990 W
PLW12	0.20774999 W
PLW13	0.16827001 W

F2 - Processing parameters

SI	131072
SF	100.5549369 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



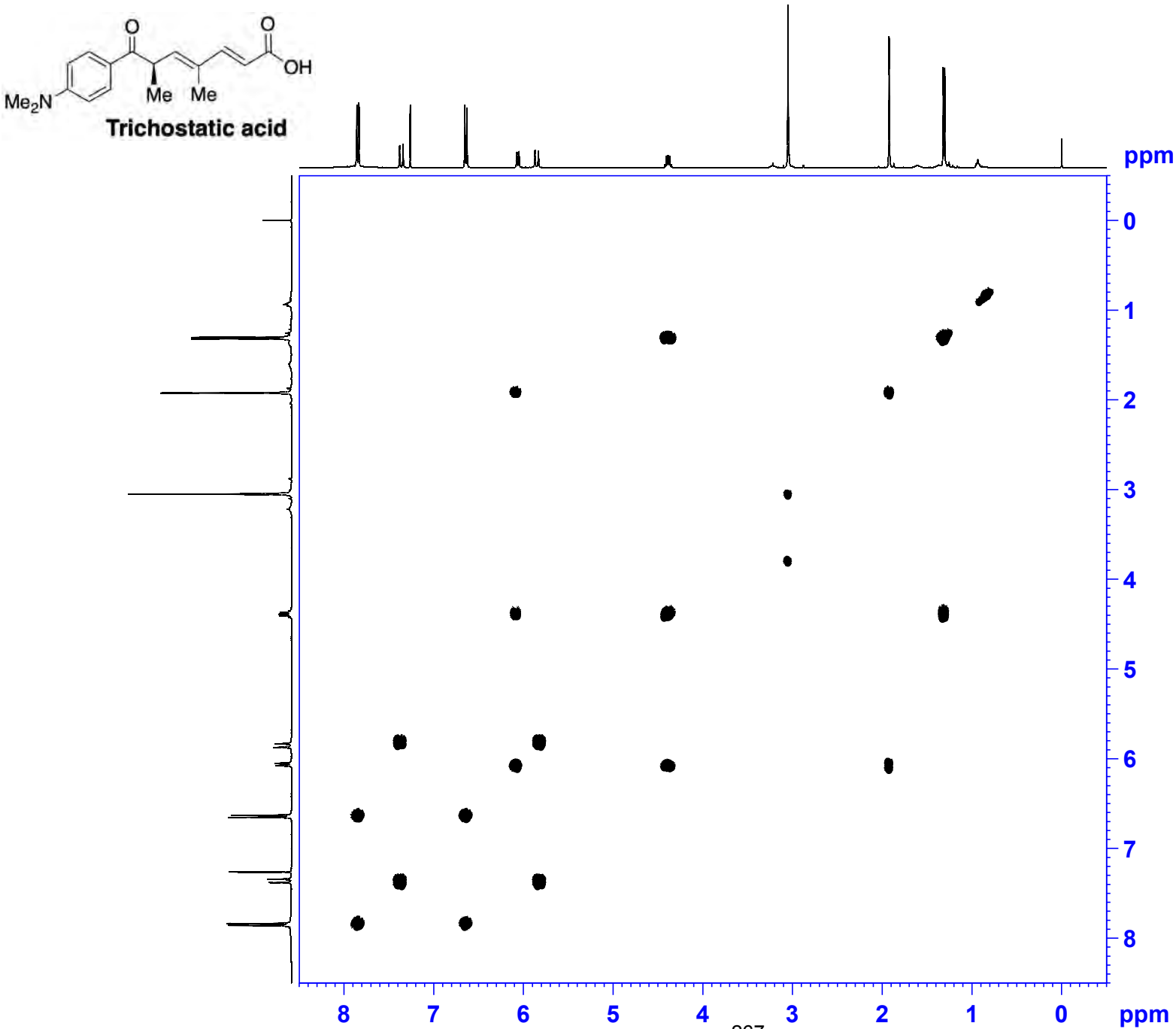
Current Data Parameters
NAME I-PK-TSACID04
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190722
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5649905 MHz
NUC1 13C
P1 10.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 44.46300125 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.79339981 W

======== CHANNEL f2 ========
SFO2 399.9012789 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 14.88 usec
P4 29.76 usec
PCPD2 90.00 usec
PLW2 7.59999990 W
PLW12 0.20774999 W

F2 - Processing parameters
SI 32768
SF 100.5549365 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME       I-PK-TSACID04
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190722
Time              18.45
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            CDCl3
NS                    1
DS                    8
SWH            3875.969 Hz
FIDRES         1.892563 Hz
AQ             0.2641920 sec
RG                 2050
DW              129.000 usec
DE                6.50 usec
TE               300.0 K
D0            0.00000300 sec
D1            0.88244390 sec
D11           0.03000000 sec
D12           0.00002000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00025800 sec

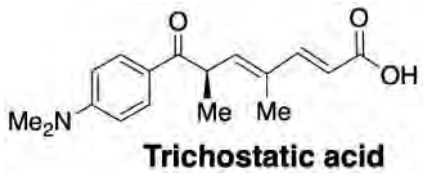
======== CHANNEL f1 ========
SFO1         399.9015252 MHz
NUC1                 1H
P1                14.88 usec
P17            2500.00 usec
PLW1          7.59999990 W
PLW10         2.48930001 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

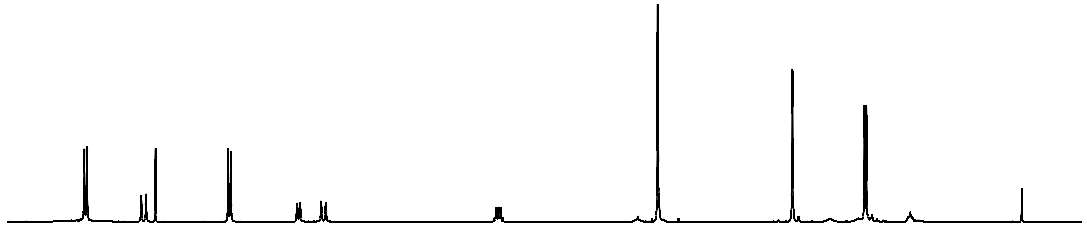
F1 - Acquisition parameters
TD                  256
SFO1          399.9015 MHz
FIDRES       30.281008 Hz
SW               9.692 ppm
FnMODE              QF

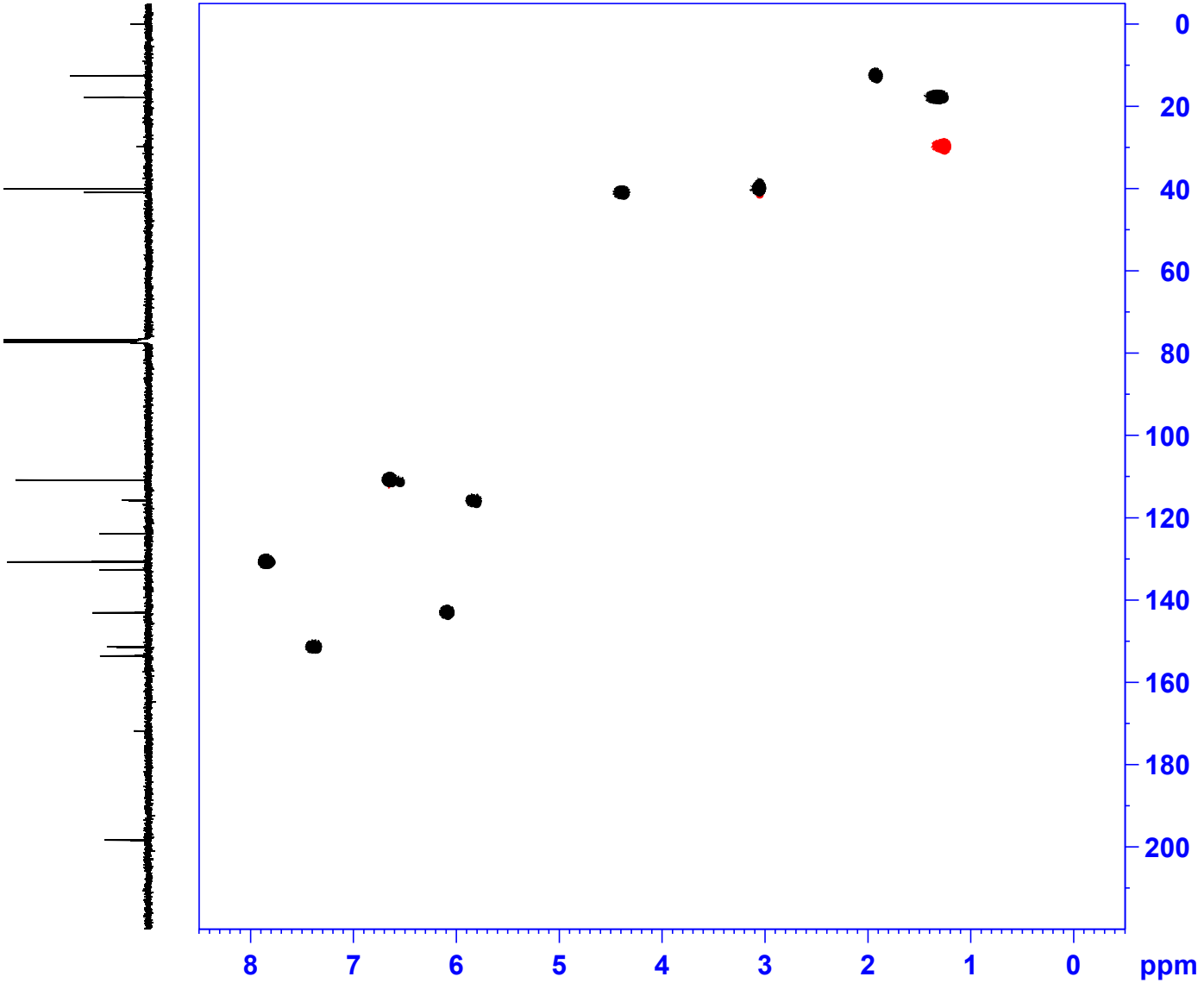
F2 - Processing parameters
SI                 1024
SF          399.9000119 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
PC                 1.40

F1 - Processing parameters
SI                 1024
MC2                 QF
SF          399.9000134 MHz
WDW                SINE
SSB       0
LB        0 Hz
GB        0
```



Trichostatic acid





BRUKER

```
Current Data Parameters
NAME      I-PK-138TRICHO
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180816
Time               1.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                 1024
SOLVENT            CDC13
NS                     2
DS                    32
SWH             4807.692 Hz
FIDRES          4.695012 Hz
AQ              0.1064960 sec
RG                  2050
DW               104.000 usec
DE                 6.50 usec
TE                300.1 K
CNST2        145.0000000
D0            0.00000300 sec
D1            0.80000001 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D21           0.00360000 sec
IN0           0.00001910 sec

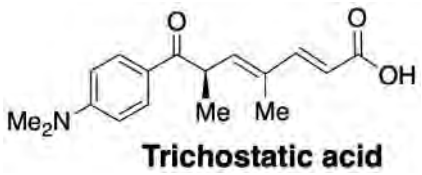
======== CHANNEL f1 ========
SFO1         399.9018806 MHz
NUC1                1H
P1                14.88 usec
P2                29.76 usec
P28       0 usec
PLW1           7.59999990 W

======== CHANNEL f2 ========
SFO2         100.5670016 MHz
NUC2                13C
CPDPRG[2           garp4
P3                10.00 usec
P14              500.00 usec
P31             1900.00 usec
PCPD2             80.00 usec
PLW0      0 W
PLW2         44.46300125 W
PLW12         0.69472998 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3              0.500
SPOFFS3   0 Hz
SPW3           6.79339981 W
SPNAM[18  Crp60_xfilt.2
SPOAL18             0.500
SPOFFS18 0 Hz
SPW18          1.62779999 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1             100.567 MHz
FIDRES        204.515701 Hz
SW               260.304 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
```



Trichostatic acid


```
Current Data Parameters
NAME       I-PK-TSACID04
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190722
Time               18.52
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG    hmbcetgpl3nd
TD                 4096
SOLVENT            CDC13
NS                    4
DS                   16
SWH           5208.333 Hz
FIDRES        1.271566 Hz
AQ            0.3932160 sec
RG                 2050
DW               96.000 usec
DE                6.50 usec
TE                300.0 K
CNST6       120.0000000
CNST7       175.0000000
CNST13        8.0000000
CNST30        0.5981122
D0          0.00000300 sec
D1          1.50000000 sec
D6          0.06250000 sec
D16         0.00020000 sec
IN0         0.00002070 sec

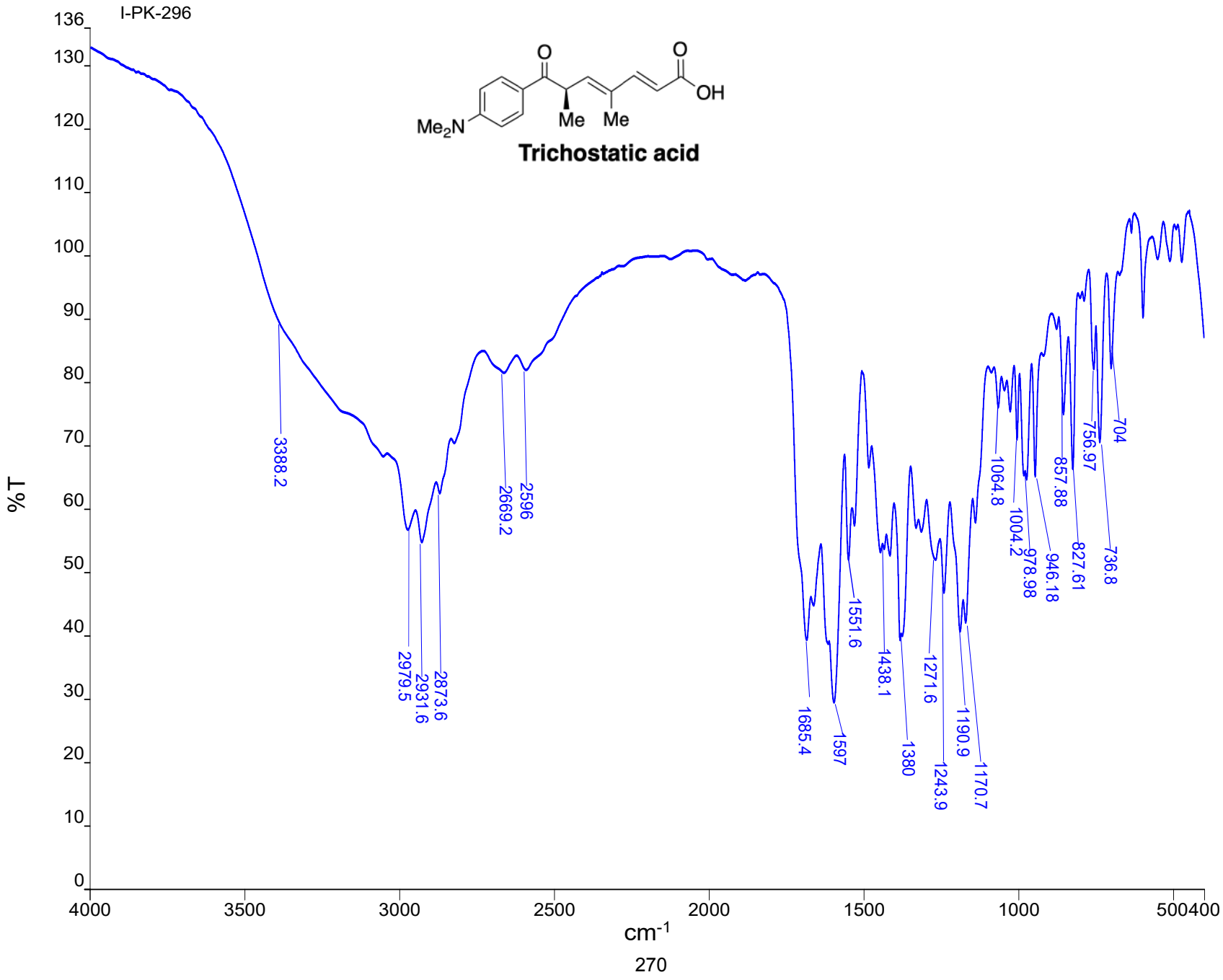
======== CHANNEL f1 ========
SFO1        399.9019995 MHz
NUC1               1H
P1                14.88 usec
P2                29.76 usec
PLW1         7.59999990 W

======== CHANNEL f2 ========
SFO2        100.5659947 MHz
NUC2               13C
P3                10.00 usec
P24             2000.00 usec
PLW2        44.46300125 W
SPNAM[7]    Crp60comp.4
SPOAL7            0.500
SPOFFS7  0 Hz
SPW7         6.79339981 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPNAM[4]      SMSQ10.100
GPNAM[5]      SMSQ10.100
GPNAM[6]      SMSQ10.100
GPZ1              80.00 %
GPZ3              14.00 %
GPZ4              -8.00 %
GPZ5              -4.00 %
GPZ6              -2.00 %
P16             1000.00 usec

F1 - Acquisition parameters
TD                  512
SFO1            100.566 MHz
FIDRES        94.353867 Hz
SW              240.186 ppm
FnMODE    Echo-Antiecho

F2 - Processing parameters
SI                 4096
SF          399.9000096 MHz
WDW                QSINE
SSB       0
LB        0 Hz
GB        0
```

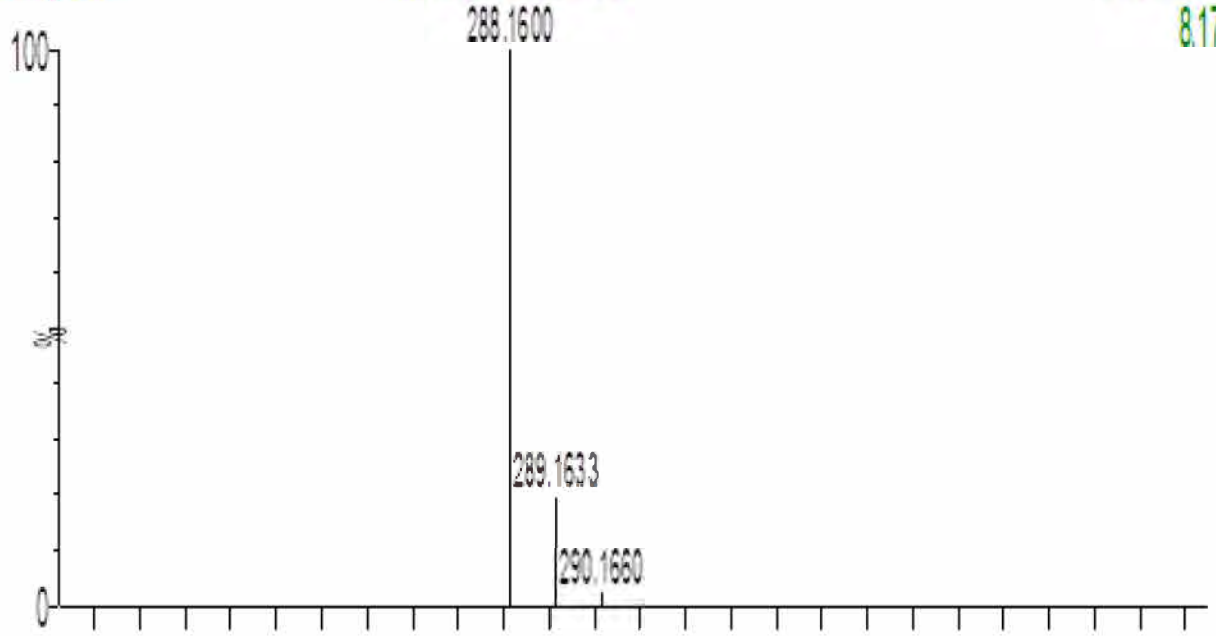

I-PK-296
Trichostatic acid
%T
cm-1
3388.2
2979.5
2931.6
2873.6
2669.2
2596
1685.4
1597
1551.6
1438.1
1380
1271.6
1243.9
1190.9
1170.7
1064.8
1004.2
978.98
946.18
857.88
827.61
756.97
736.8
704

11-PK-296

20-06-2019

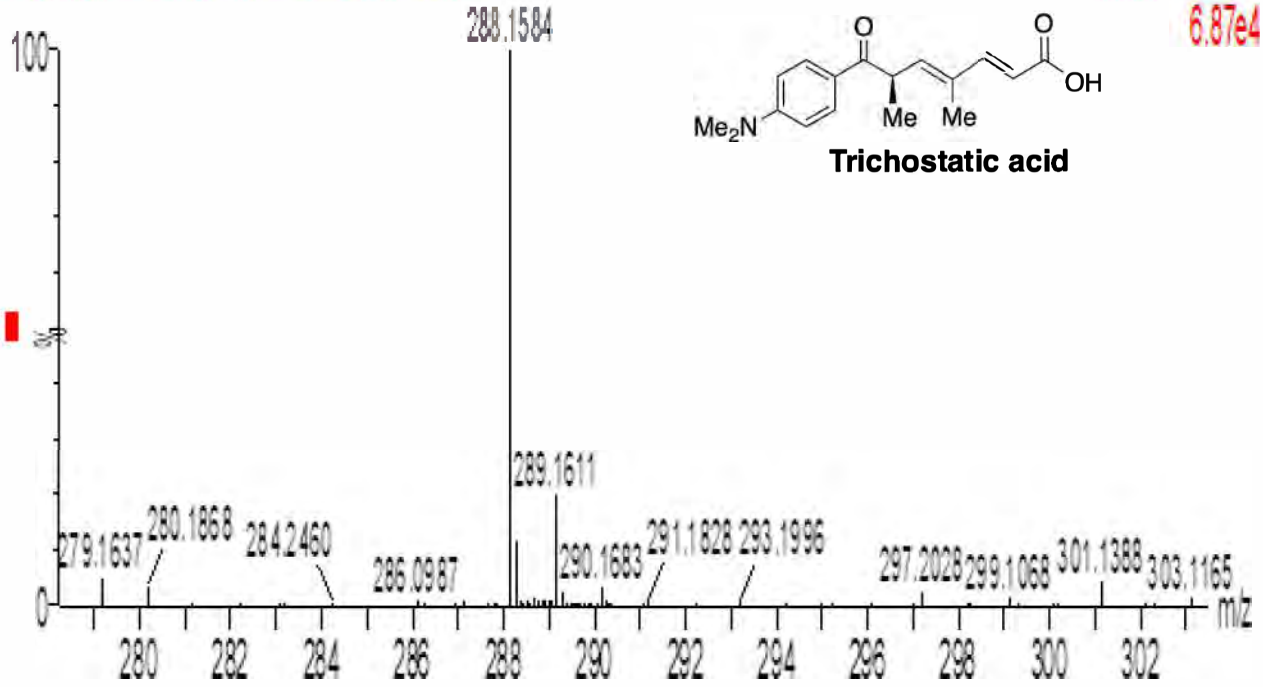
asep_01MAY_2019_154 (0.075) Is (1.00,1.00) C17H21NO3H

1: TOF MS ES+
8.17e12

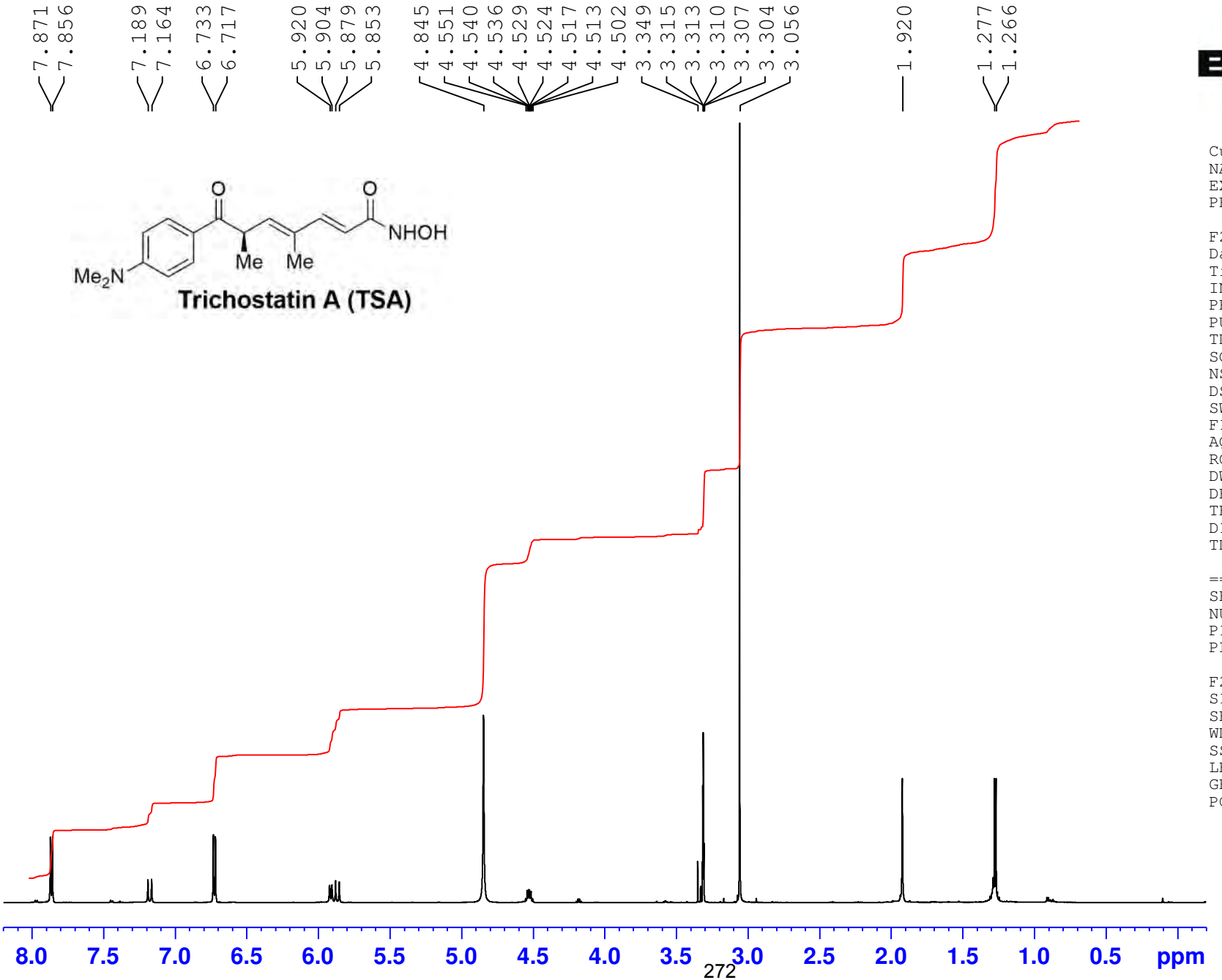


asep_01MAY_2019_154 12 (0.536) Cm (10:12)

1: TOF MS ES+
6.87e4



Trichostatic acid



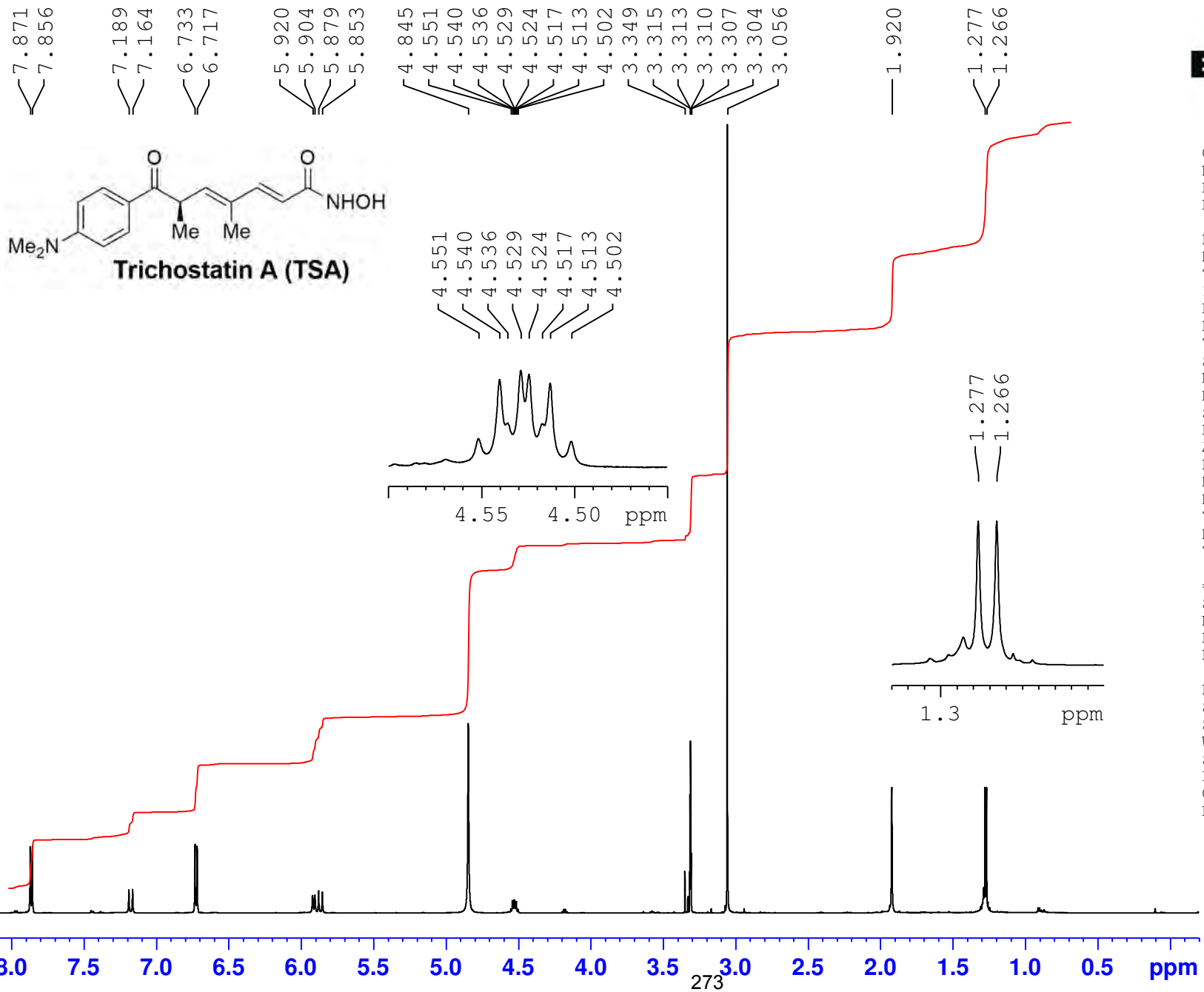
BRUKER

```
Current Data Parameters
NAME        IV-PK-TSA2.0
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201216
Time                7.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             MeOD
NS                    16
DS                     0
SWH            18028.846 Hz
FIDRES          0.100001 Hz
AQ             4.9999318 sec
RG                  97.5
DW                27.733 usec
DE                 7.60 usec
TE                300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF         600.1300115 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

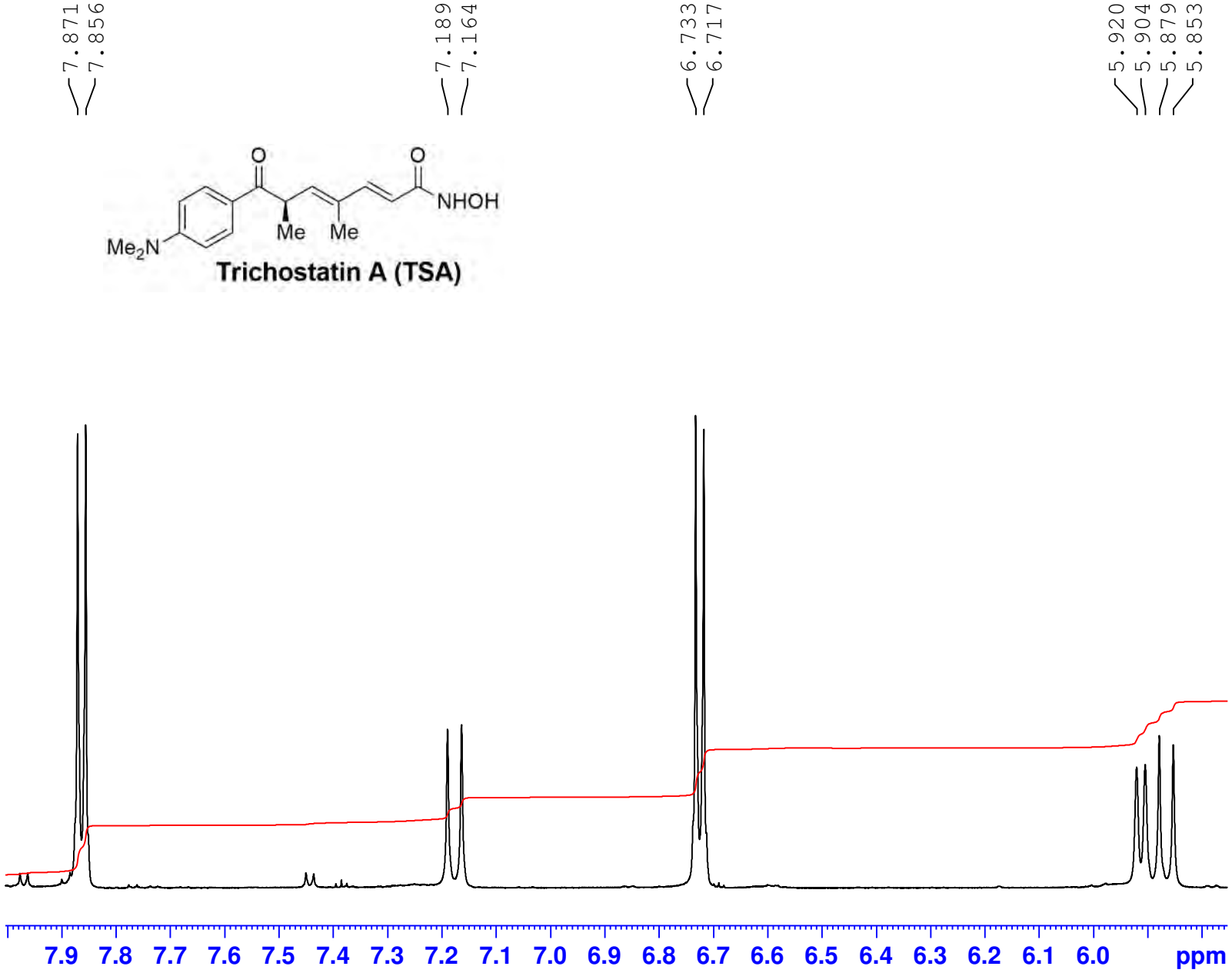



```
Current Data Parameters
NAME        IV-PK-TSA2.0
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201216
Time                7.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             MeOD
NS                    16
DS                     0
SWH            18028.846 Hz
FIDRES          0.100001 Hz
AQ             4.9999318 sec
RG                  97.5
DW                27.733 usec
DE                 7.60 usec
TE                300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF           600.1300115 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```

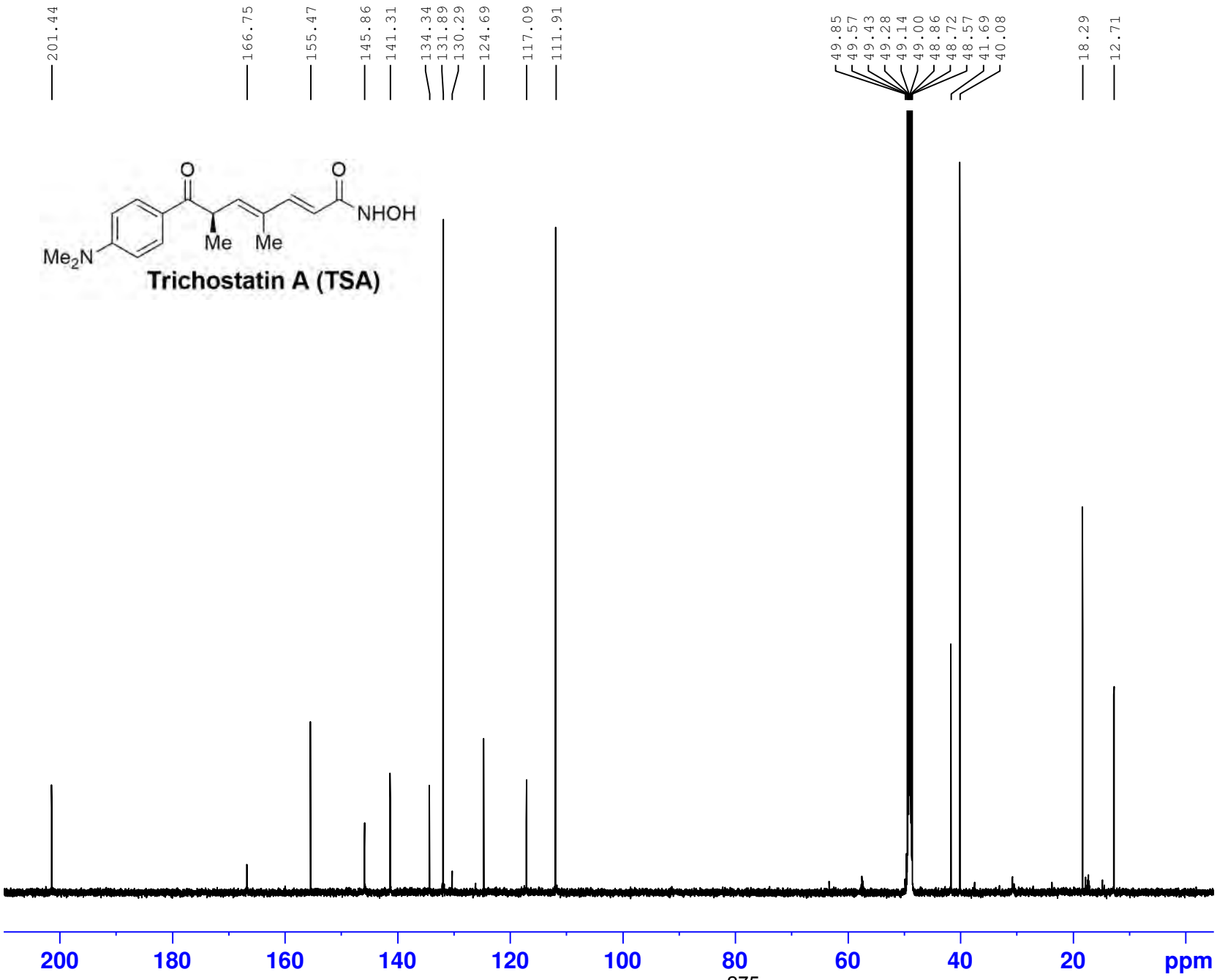



```
Current Data Parameters
NAME        IV-PK-TSA2.0
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201216
Time                7.12
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             MeOD
NS                    16
DS                     0
SWH           18028.846 Hz
FIDRES         0.100001 Hz
AQ            4.9999318 sec
RG                 97.5
DW               27.733 usec
DE                7.60 usec
TE               300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                1H
P1                10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF         600.1300115 MHz
WDW                 EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```



BRUKER

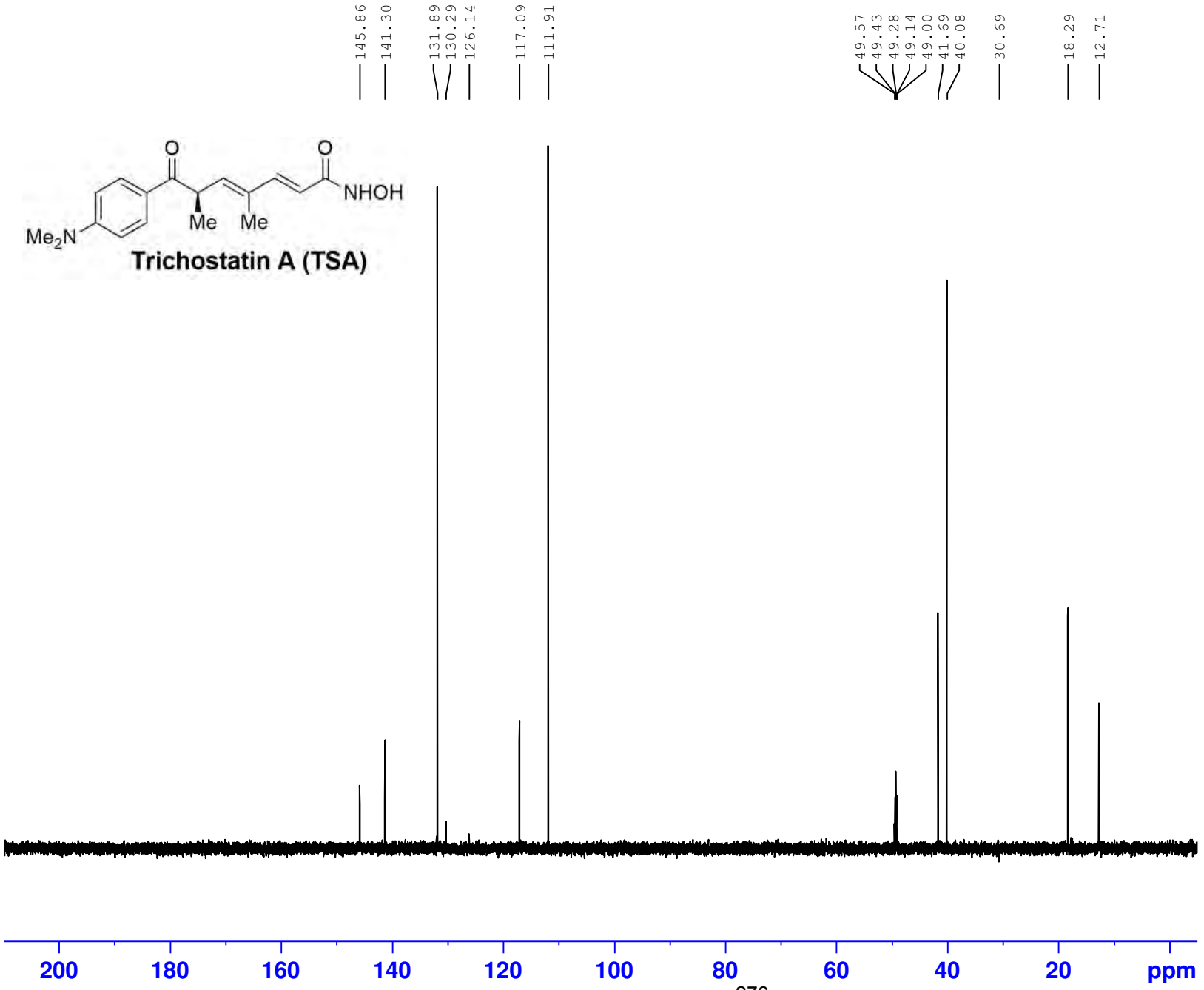
Current Data Parameters
NAME IV-PK-TSA2.0
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201217
Time 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 119044
SOLVENT MeOD
NS 5000
DS 4
SWH 37500.000 Hz
FIDRES 0.315010 Hz
AQ 1.5872533 sec
RG 186.92
DW 13.333 usec
DE 7.73 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9194058 MHz
NUC1 13C
P1 11.80 usec
PLW1 85.00000000 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W
PLW13 0.28090999 W

F2 - Processing parameters
SI 131072
SF 150.9025978 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters

NAME	IV-PK-TSA2.0
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20201216
Time	7.36
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	deptsp135.b
TD	119044
SOLVENT	MeOD
NS	500
DS	4
SWH	35714.285 Hz
FIDRES	0.300009 Hz
AQ	1.6666160 sec
RG	186.92
DW	14.000 usec
DE	7.44 usec
TE	300.0 K
CNST2	145.0000000
D1	1.00000000 sec
D2	0.00344828 sec
D12	0.00002000 sec
TD0	1

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

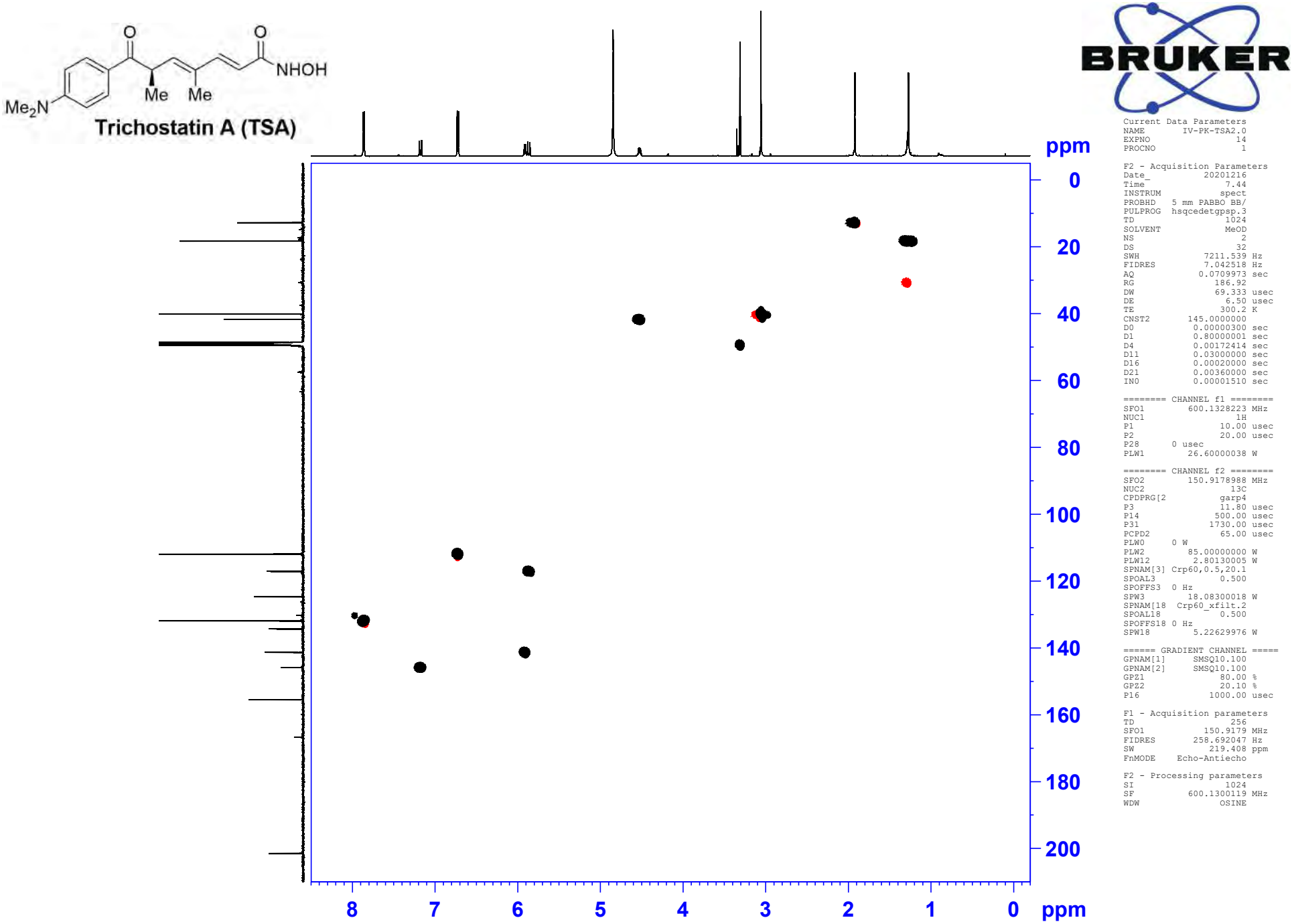
SFO1	150.9178962 MHz
NUC1	13C
P1	11.80 usec
P13	2000.00 usec
PLW0	0 W
PLW1	85.00000000 W
SPNAM[5]	Crp60comp.4
SPOAL5	0.500
SPOFFS5	0 Hz
SPW5	18.08300018 W

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

SFO2	600.1324005 MHz
NUC2	1H
CPDPRG[2	waltz64
P3	10.20 usec
P4	20.40 usec
PCPD2	80.00 usec
PLW2	27.00000000 W
PLW12	0.43891999 W

F2 - Processing parameters

SI	131072
SF	150.9025979 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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```
Current Data Parameters
NAME         IV-PK-TSA2.0
EXPNO                  14
PROCNO                  1

F2 - Acquisition Parameters
Date_            20201216
Time                7.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG  hsqcedetgpsp.3
TD                  1024
SOLVENT             MeOD
NS                     2
DS                    32
SWH             7211.539 Hz
FIDRES          7.042518 Hz
AQ             0.0709973 sec
RG                186.92
DW                69.333 usec
DE                  6.50 usec
TE                300.2 K
CNST2        145.0000000
D0            0.00000300 sec
D1            0.80000001 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D21           0.00360000 sec
IN0           0.00001510 sec

======== CHANNEL f1 ========
SFO1         600.1328223 MHz
NUC1                   1H
P1                 10.00 usec
P2                 20.00 usec
P28       0 usec
PLW1         26.60000038 W

======== CHANNEL f2 ========
SFO2         150.9178988 MHz
NUC2                  13C
CPDPRG[2            garp4
P3                 11.80 usec
P14               500.00 usec
P31              1730.00 usec
PCPD2              65.00 usec
PLW0      0 W
PLW2         85.00000000 W
PLW12         2.80130005 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3             0.500
SPOFFS3   0 Hz
SPW3         18.08300018 W
SPNAM[18  Crp60_xfilt.2
SPOAL18            0.500
SPOFFS18 0 Hz
SPW18         5.22629976 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1               80.00 %
GPZ2               20.10 %
P16              1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1           150.9179 MHz
FIDRES        258.692047 Hz
SW               219.408 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                  1024
SF           600.1300119 MHz
WDW                OSINE
```

Trichostatin A (TSA)

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```
Current Data Parameters
NAME        IV-PK-TSA2.0
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201216
Time               7.38
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG    cosygpmfppqf
TD                 2048
SOLVENT            MeOD
NS                    1
DS                    8
SWH           5980.861 Hz
FIDRES        2.920342 Hz
AQ           0.1712128 sec
RG              186.92
DW              83.600 usec
DE               6.50 usec
TE              300.0 K
D0          0.00000300 sec
D1          0.88940728 sec
D11         0.03000000 sec
D12         0.00002000 sec
D13         0.00000400 sec
D16         0.00020000 sec
IN0         0.00016720 sec

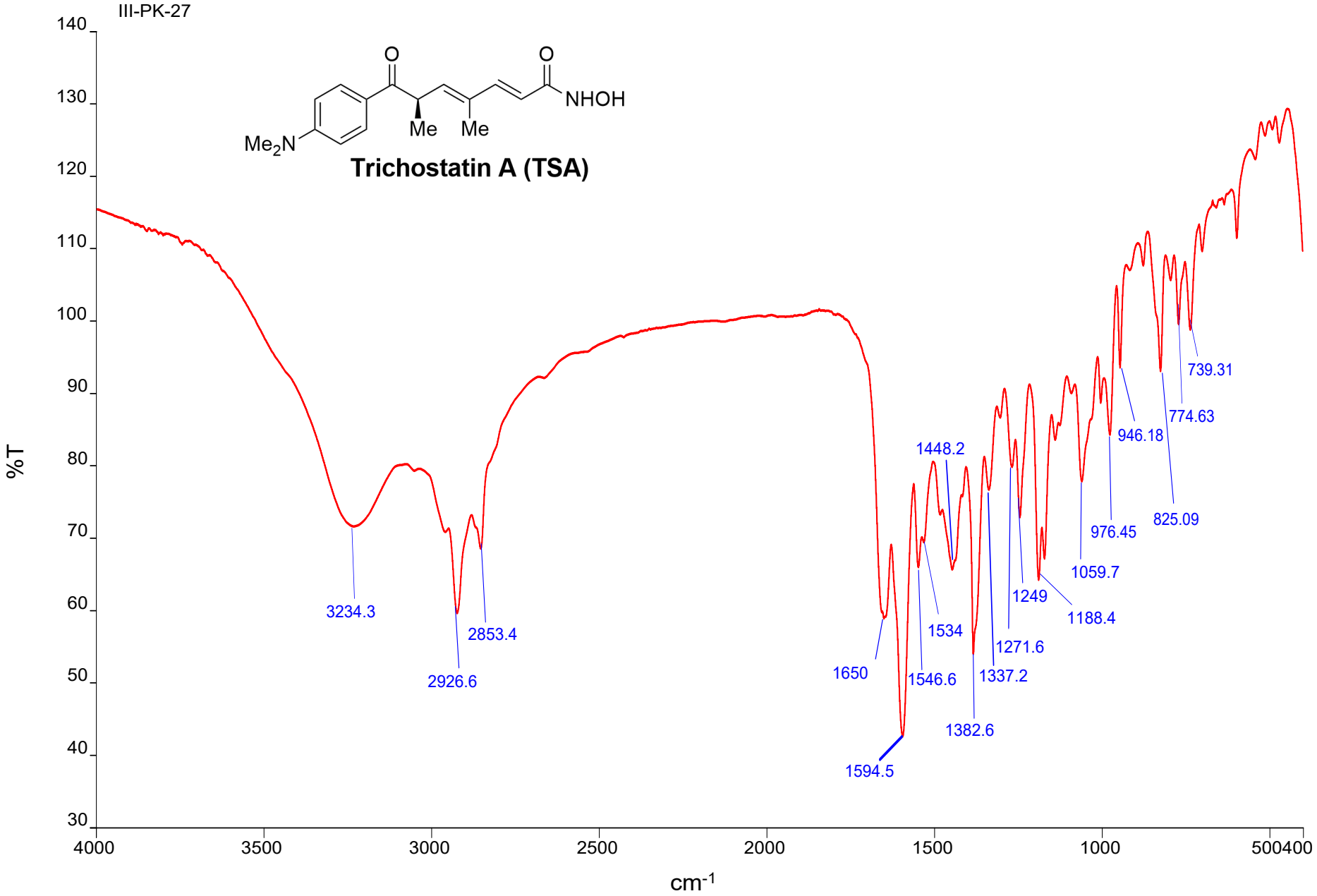
======== CHANNEL f1 ========
SFO1       600.1323508 MHz
NUC1               1H
P1               10.00 usec
P17            2500.00 usec
PLW1       26.60000038 W
PLW10       3.93490005 W

====== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPNAM[3]     SMSQ10.100
GPZ1            16.00 %
GPZ2            12.00 %
GPZ3            40.00 %
P16           1000.00 usec

F1 - Acquisition parameters
TD                 256
SFO1          600.1324 MHz
FIDRES       46.725479 Hz
SW               9.966 ppm
FnMODE              QF

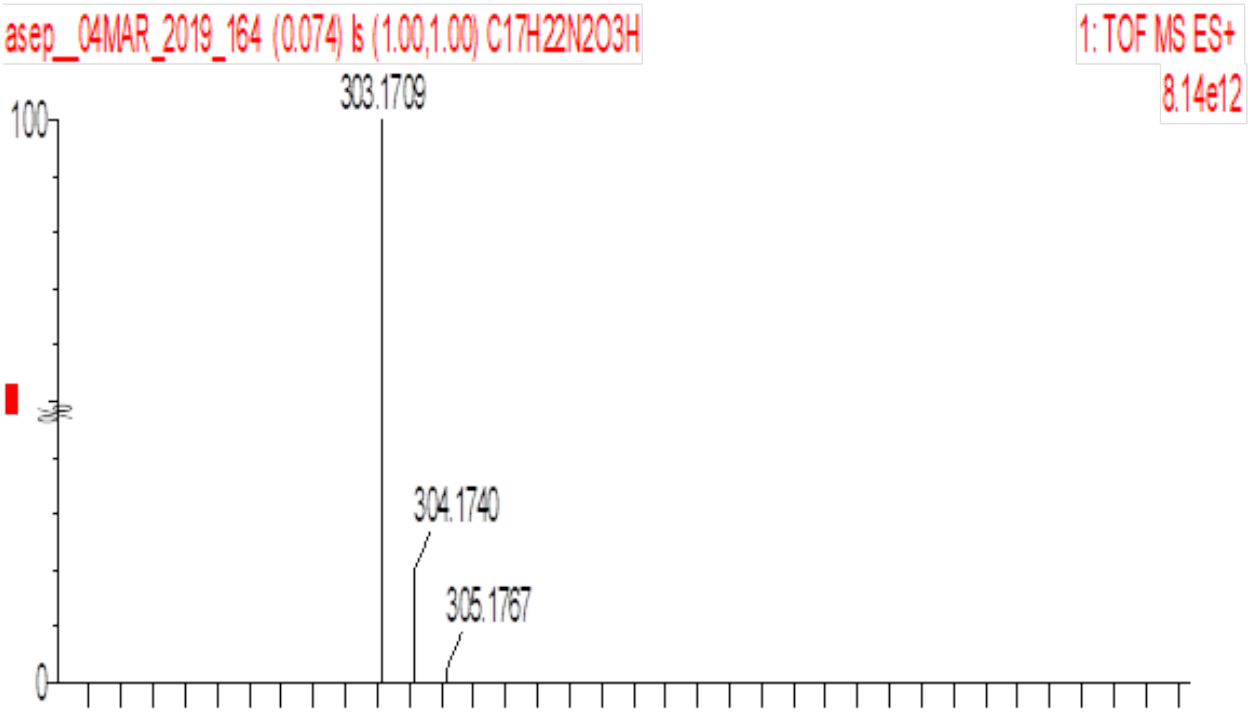
F2 - Processing parameters
SI                1024
SF          600.1300116 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
PC                1.40

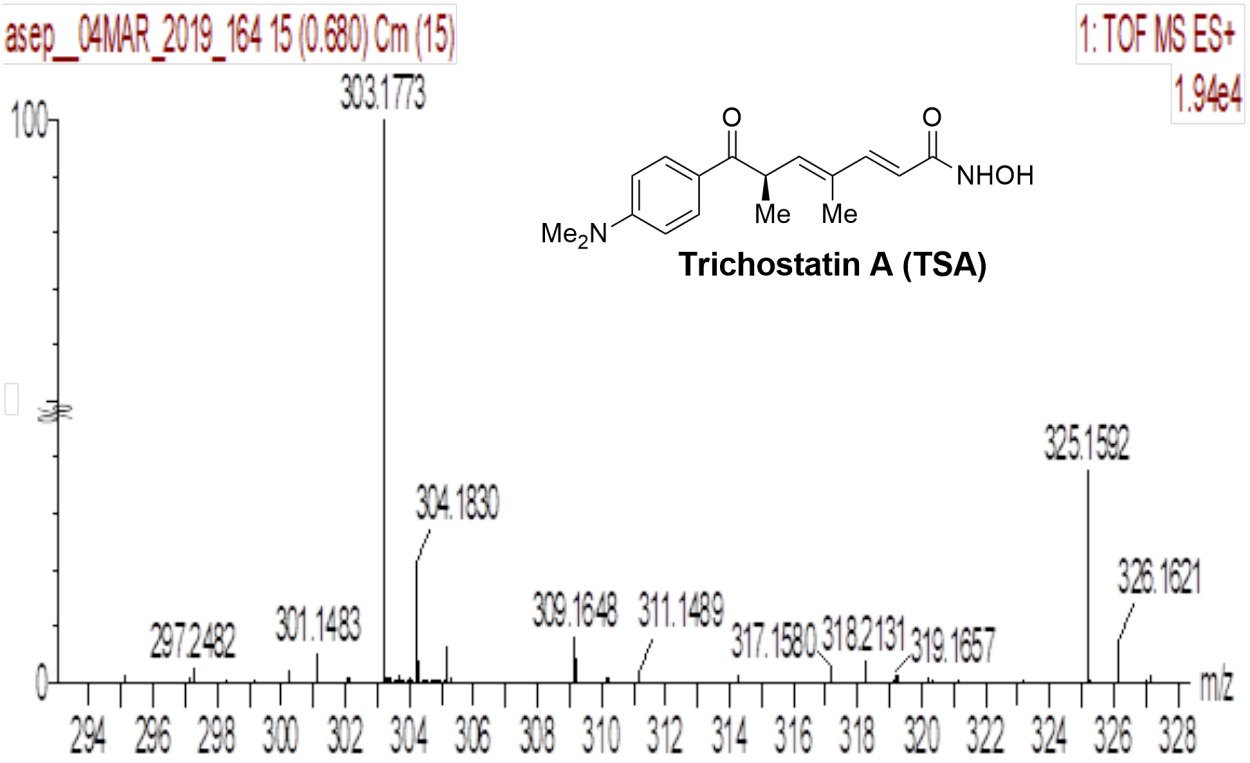
F1 - Processing parameters
SI                1024
MC2                 QF
SF          600.1300097 MHz
WDW               SINE
SSB       0
LB        0 Hz
GB        0
```


III-PK-27
O
O
NHOH
Me
Me
Me2N
Trichostatin A (TSA)
%T
cm-1
140
130
120
110
100
90
80
70
60
50
40
30
4000
3500
3000
2500
2000
1500
1000
500400
3234.3
2926.6
2853.4
1650
1594.5
1546.6
1534
1448.2
1382.6
1337.2
1271.6
1249
1188.4
1059.7
976.45
946.18
825.09
774.63
739.31

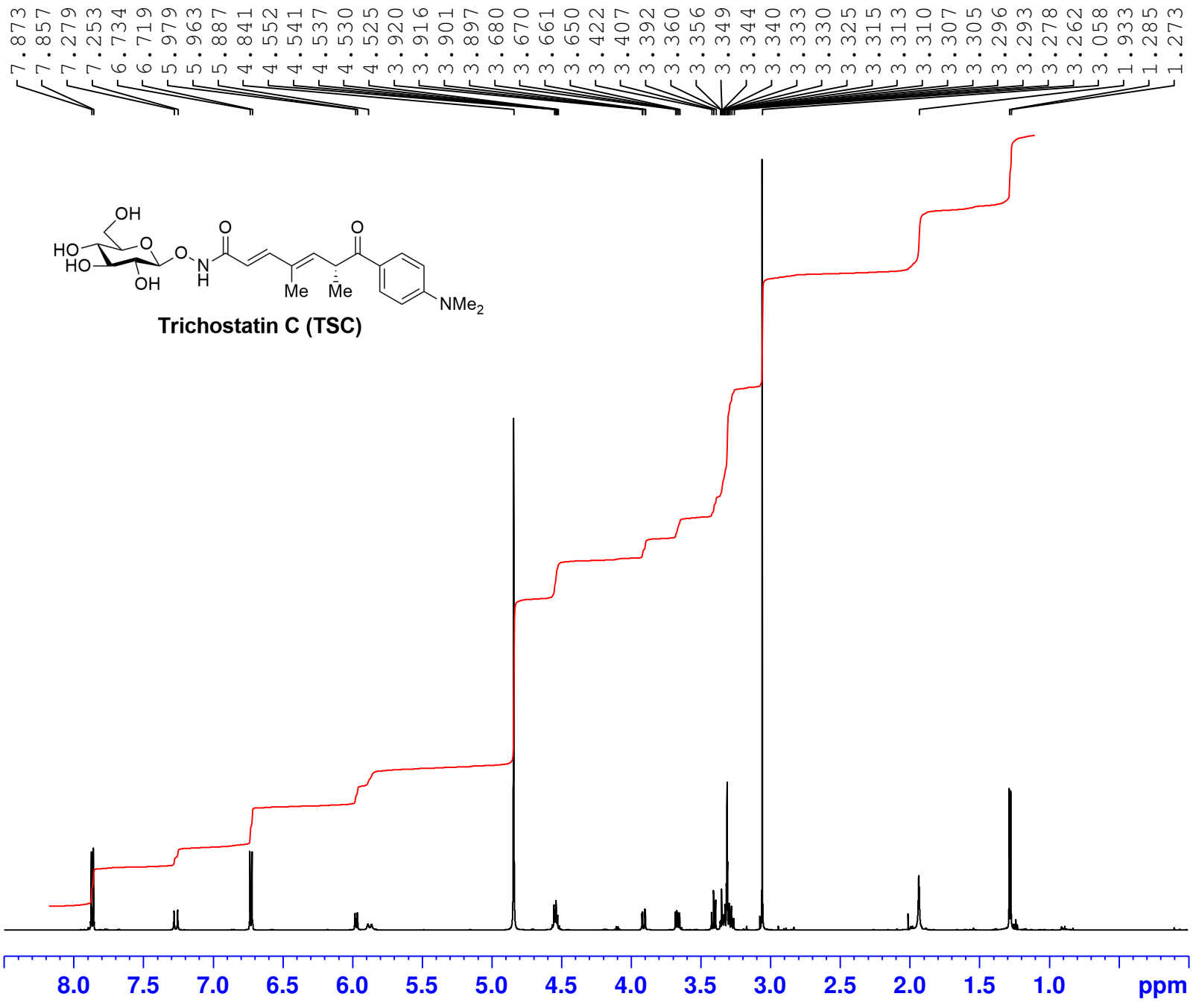
I-PK-276

12-04-2019





Trichostatin A (TSA)



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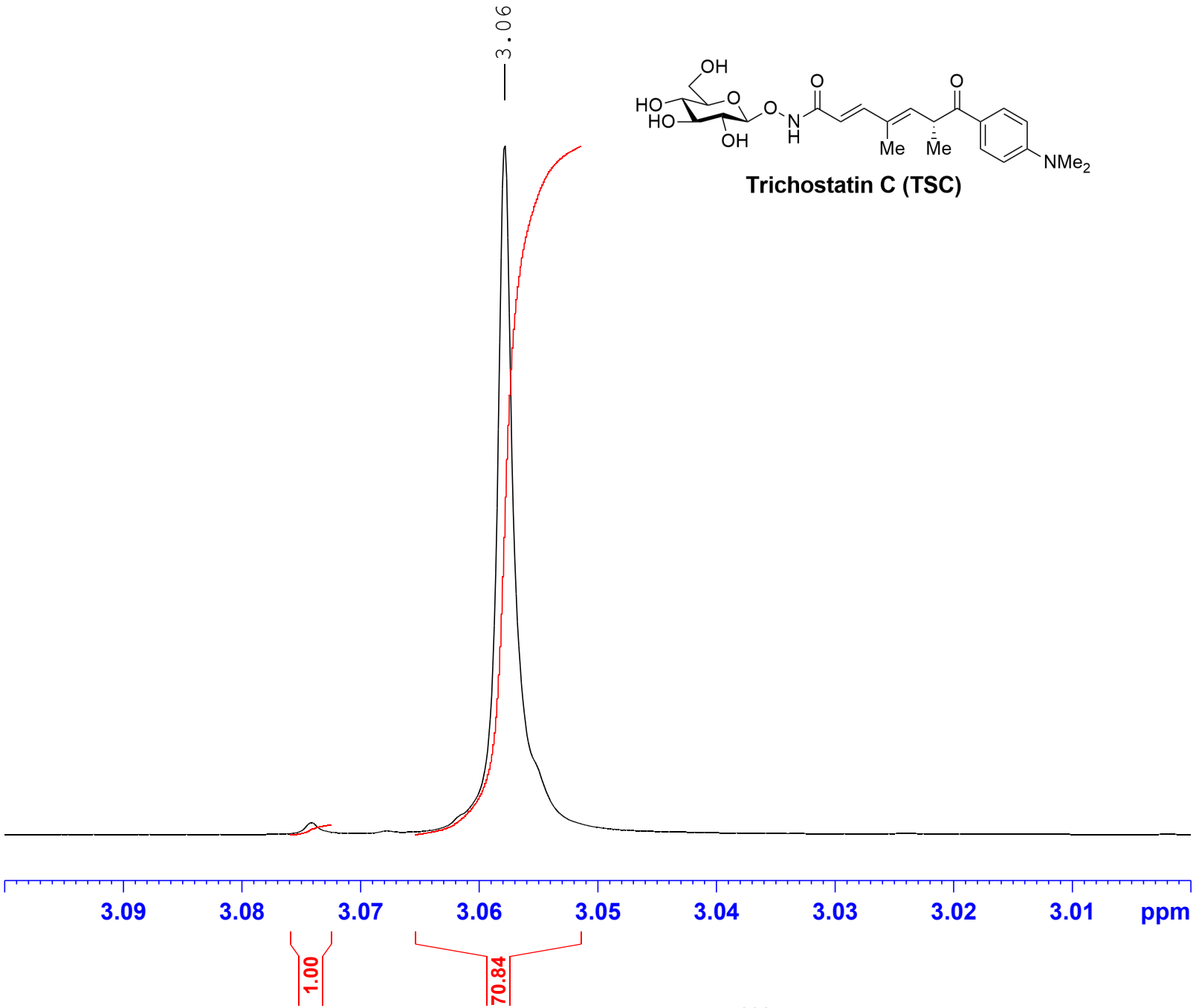
```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201204
Time                     19.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     180286
SOLVENT                  MeOD
NS                        500
DS                          0
SWH                 18028.846 Hz
FIDRES                0.100001 Hz
AQ                  4.9999318 sec
RG                       97.5
DW                     27.733 usec
DE                      7.60 usec
TE                     300.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                       1H
P1                      10.00 usec
PLW1               26.60000038 W

F2 - Processing parameters
SI                       262144
SF                 600.1300114 MHz
WDW                        EM
SSB                         0
LB                       0.10 Hz
GB                          0
PC                       1.00
```



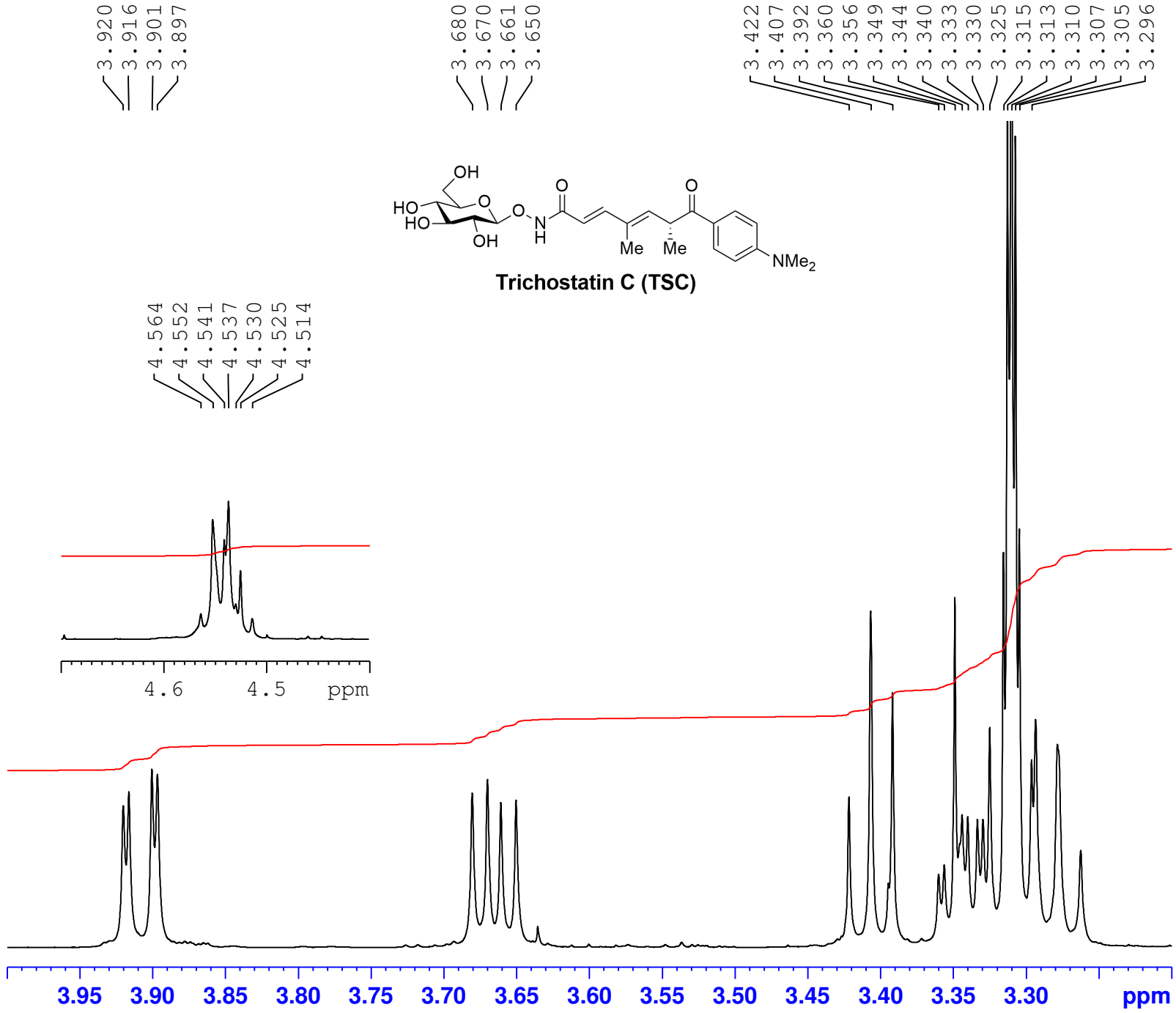
Trichostatin C (TSC)

```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201204
Time                     19.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     180286
SOLVENT                  MeOD
NS                        500
DS                          0
SWH                 18028.846 Hz
FIDRES               0.100001 Hz
AQ                   4.9999318 sec
RG                        97.5
DW                     27.733 usec
DE                      7.60 usec
TE                     300.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                       1H
P1                      10.00 usec
PLW1               26.60000038 W

F2 - Processing parameters
SI                     262144
SF                 600.1300115 MHz
WDW                        EM
SSB          0
LB                      0.10 Hz
GB           0
PC                      1.00
```



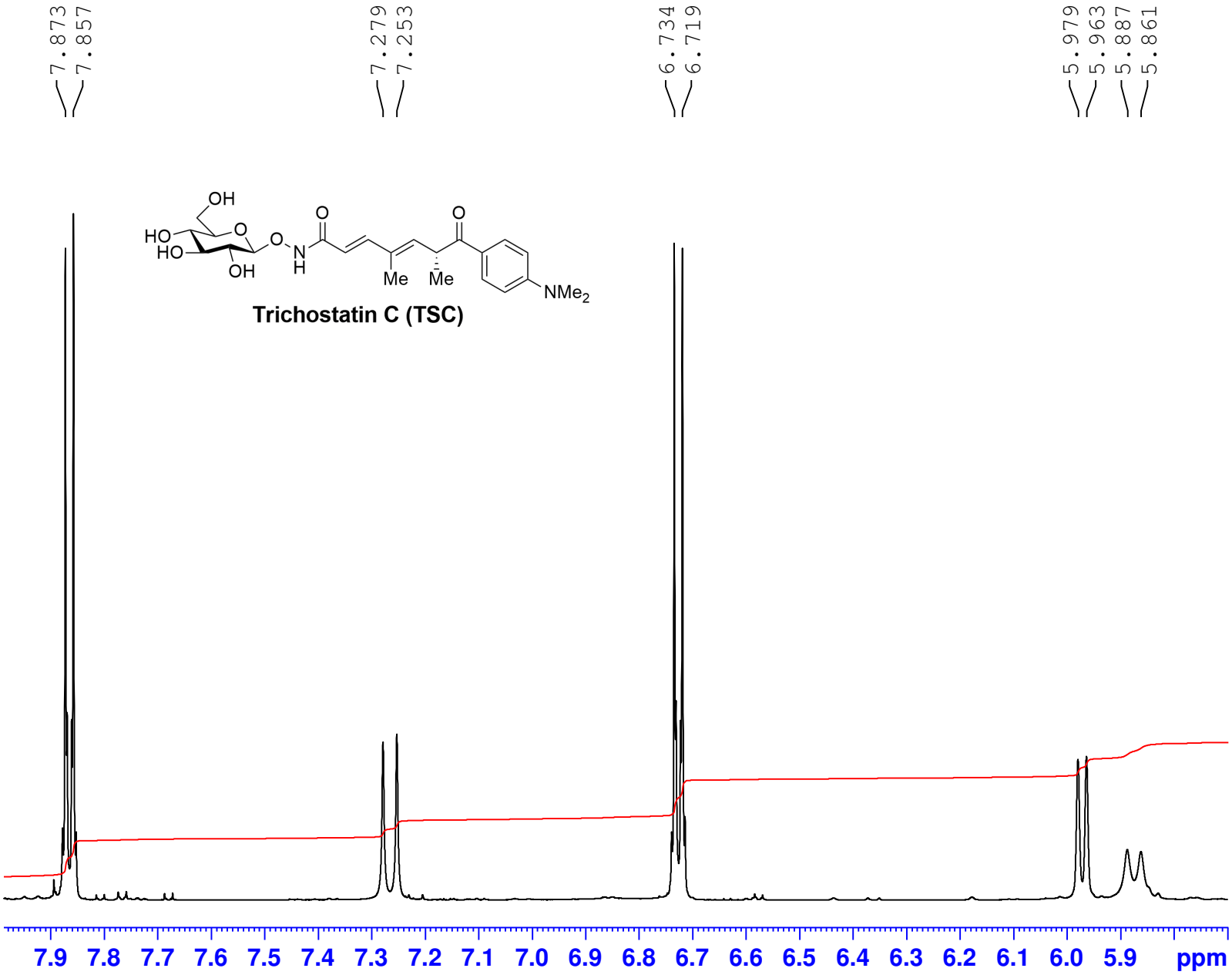
BRUKER

```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201204
Time                     19.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                     180286
SOLVENT                  MeOD
NS                        500
DS                          0
SWH                 18028.846 Hz
FIDRES                0.100001 Hz
AQ                  4.9999318 sec
RG                       97.5
DW                     27.733 usec
DE                      7.60 usec
TE                     300.0 K
D1                  0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                       1H
P1                      10.00 usec
PLW1               26.60000038 W

F2 - Processing parameters
SI                        262144
SF                 600.1300114 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00
```

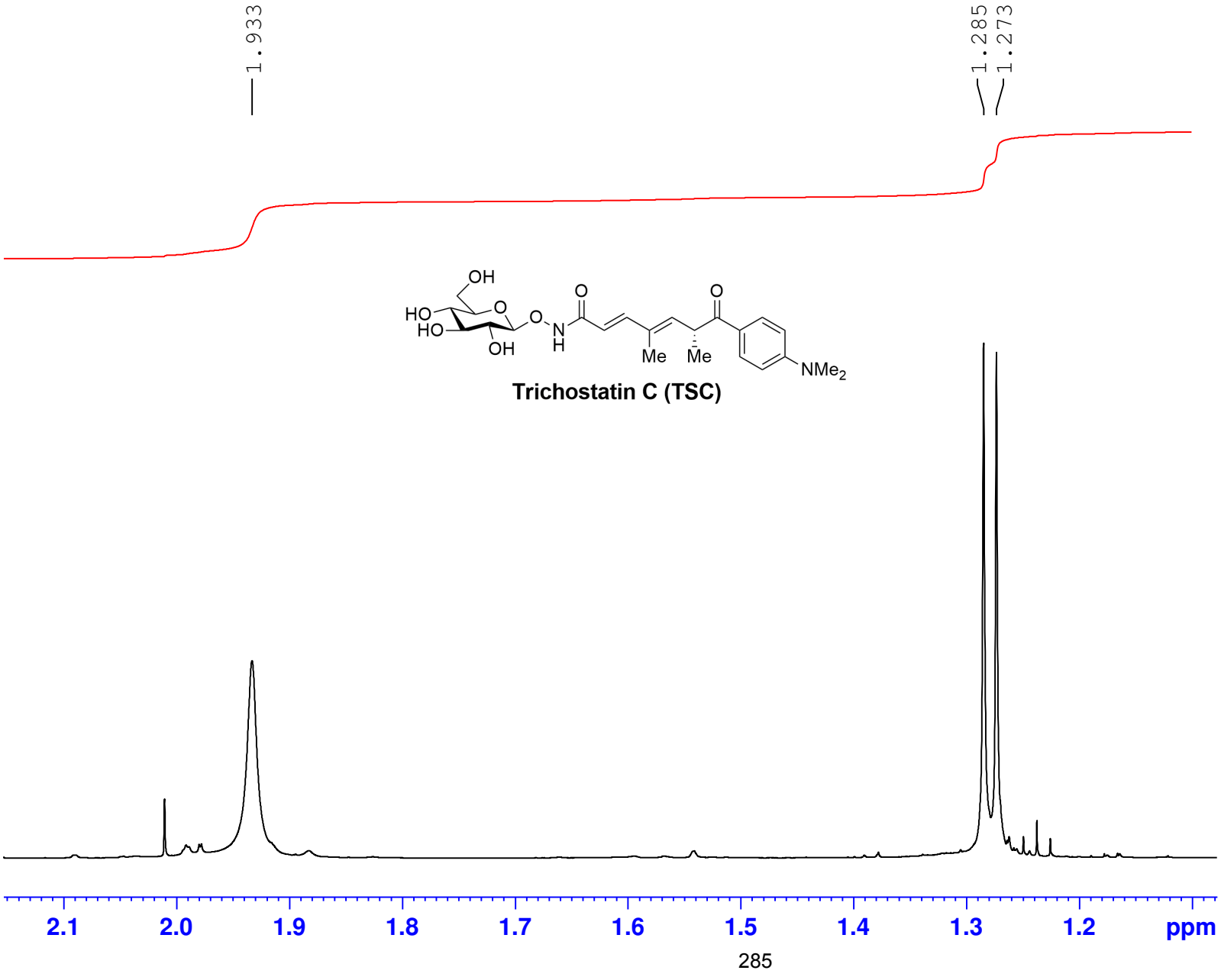



```
Current Data Parameters
NAME            IV-PK-36
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201204
Time               19.52
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                180286
SOLVENT             MeOD
NS                    500
DS                      0
SWH           18028.846 Hz
FIDRES         0.100001 Hz
AQ            4.9999318 sec
RG                 97.5
DW               27.733 usec
DE                7.60 usec
TE               300.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                1H
P1                10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                262144
SF           600.1300114 MHz
WDW                 EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```

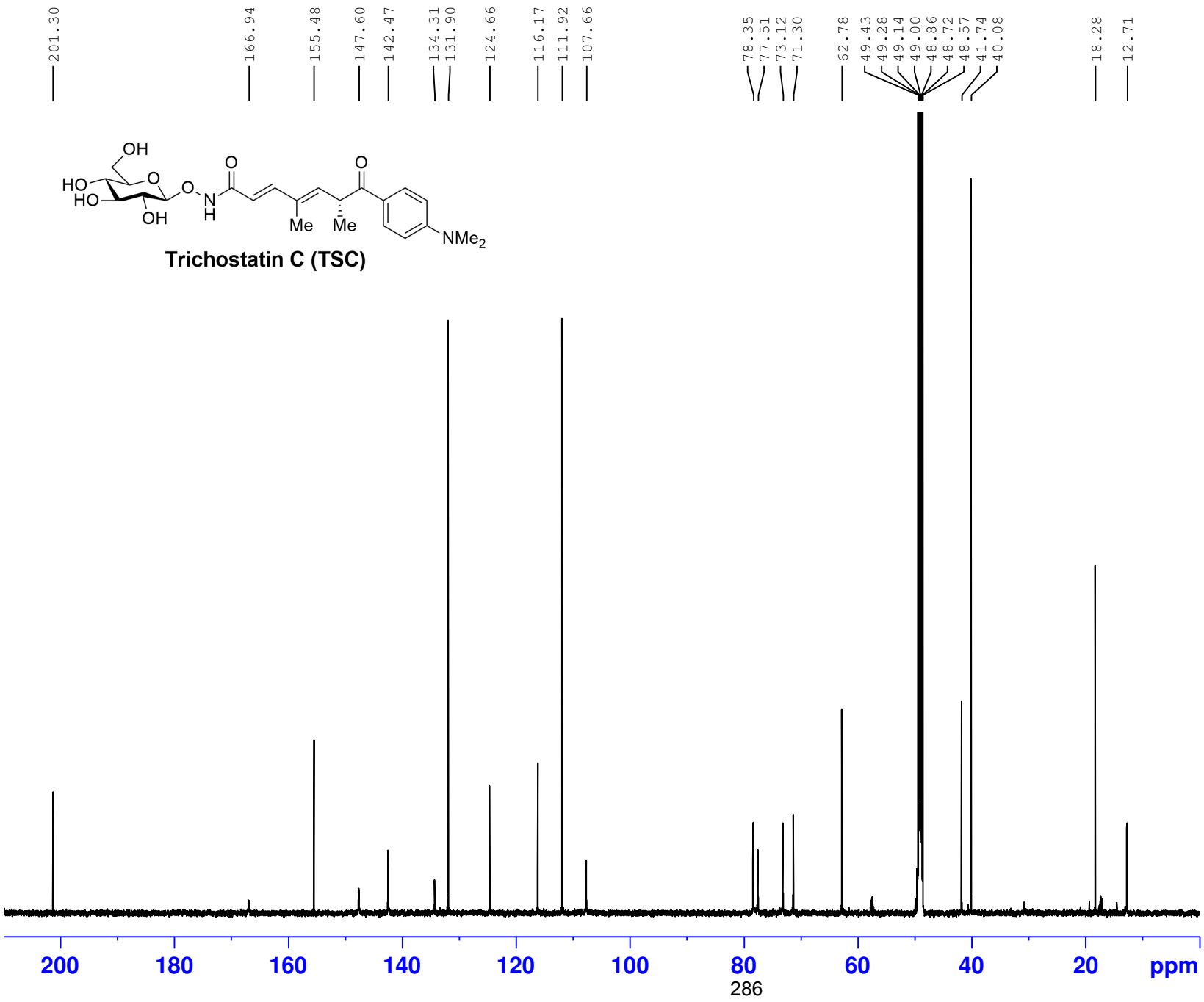



```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201204
Time                     19.52
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                  zg30
TD                     180286
SOLVENT                  MeOD
NS                        500
DS                          0
SWH                18028.846 Hz
FIDRES              0.100001 Hz
AQ                  4.9999318 sec
RG                       97.5
DW                     27.733 usec
DE                      7.60 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                        1H
P1                       10.00 usec
PLW1               26.60000038 W

F2 - Processing parameters
SI                       262144
SF                 600.1300114 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00
```



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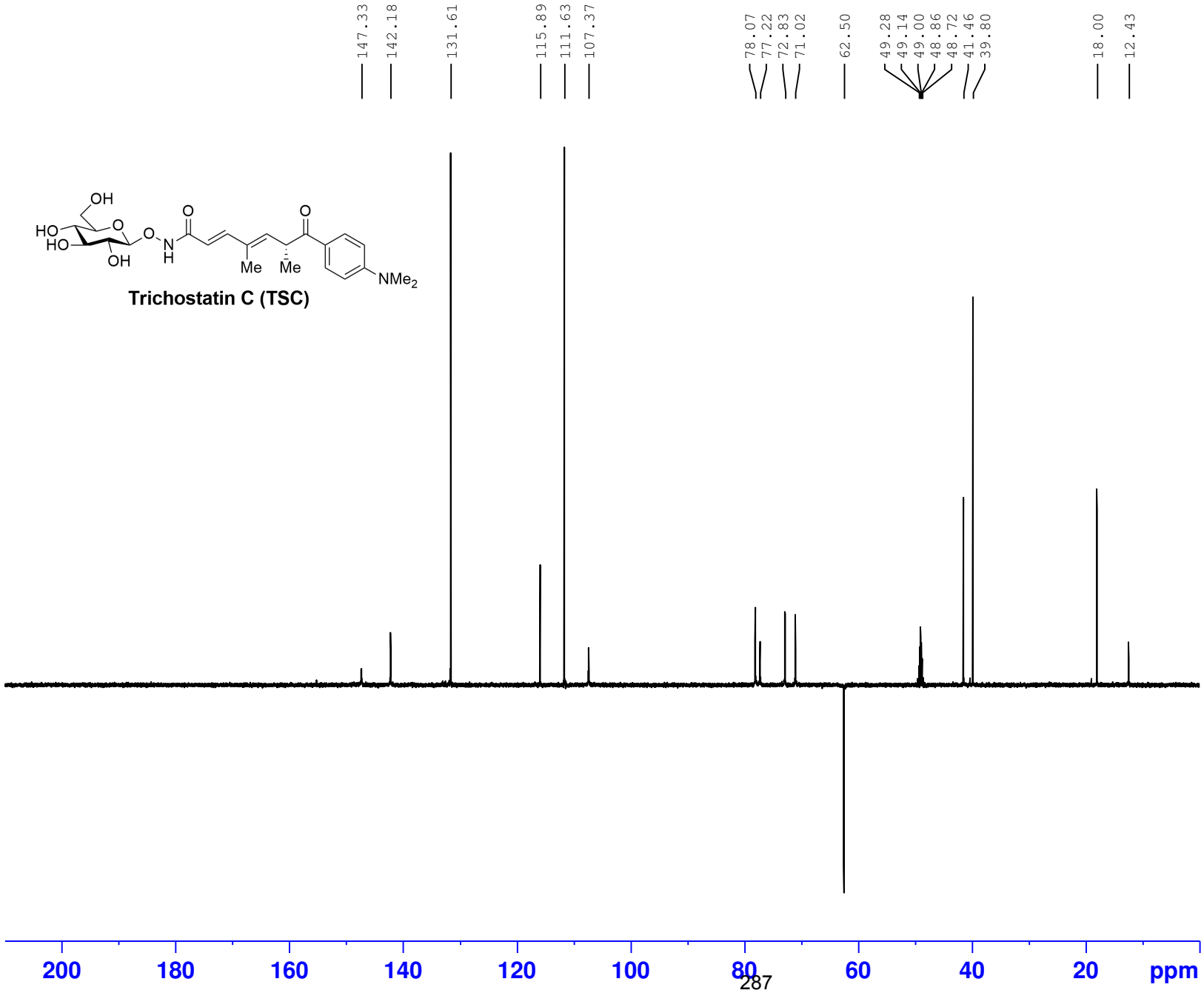
```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201205
Time                      3.59
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                      119044
SOLVENT                   MeOD
NS                        11000
DS                            4
SWH                 37500.000 Hz
FIDRES                0.315010 Hz
AQ                    1.5872533 sec
RG                       186.92
DW                       13.333 usec
DE                        7.73 usec
TE                       300.0 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               150.9194058 MHz
NUC1                       13C
P1                       11.80 usec
PLW1               85.00000000 W

======== CHANNEL f2 ========
SFO2               600.1324005 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                    80.00 usec
PLW2               27.00000000 W
PLW12               0.43891999 W
PLW13               0.28090999 W

F2 - Processing parameters
SI                        131072
SF                 150.9025983 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



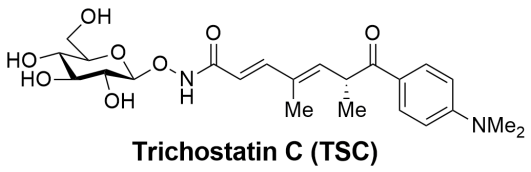
Current Data Parameters
NAME IV-PK-36
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201205
Time 7.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG deptsp135.b
TD 119044
SOLVENT MeOD
NS 5000
DS 4
SWH 35714.285 Hz
FIDRES 0.300009 Hz
AQ 1.6666160 sec
RG 186.92
DW 14.000 usec
DE 7.44 usec
TE 300.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

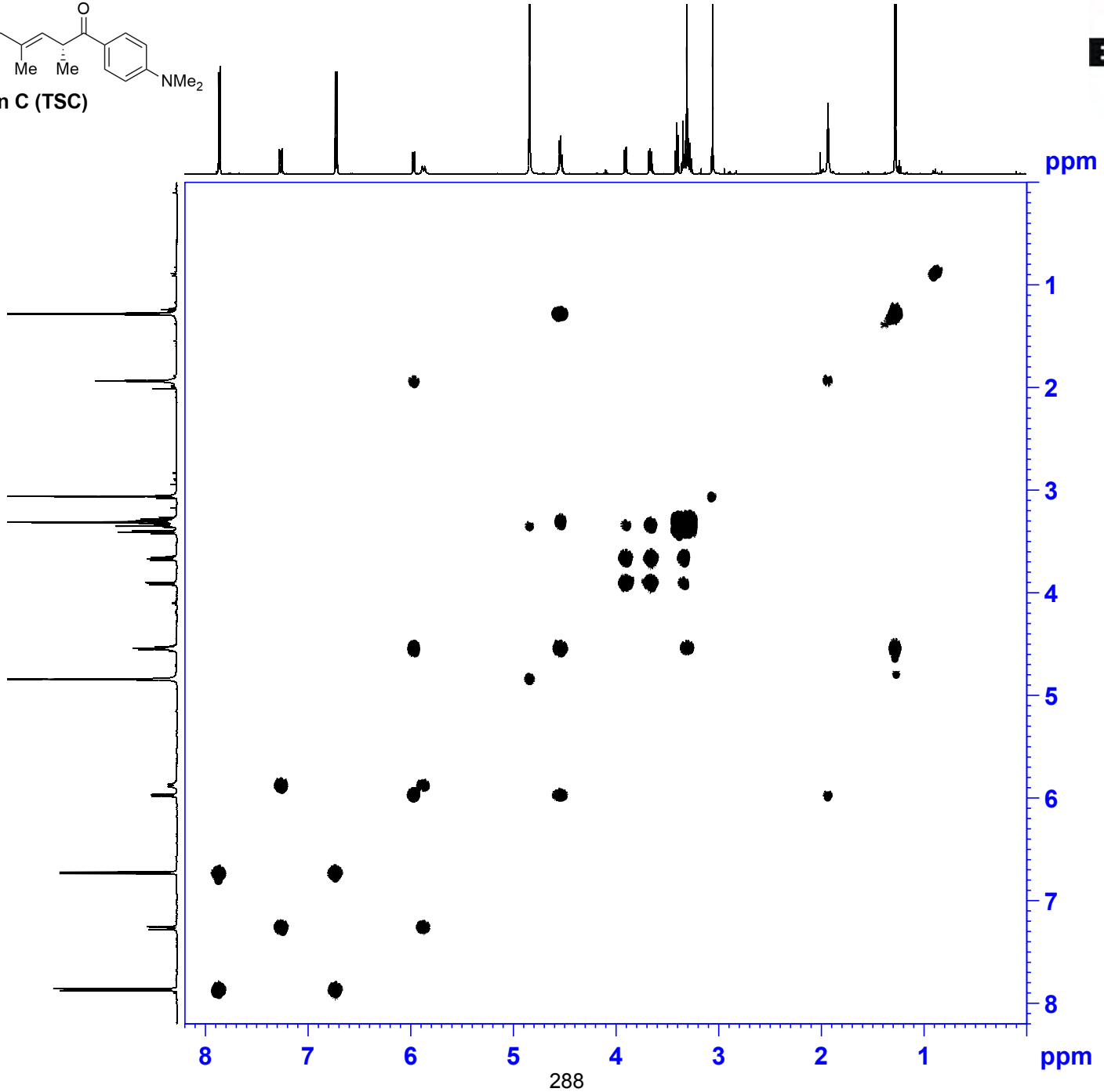
======== CHANNEL f1 ========
SFO1 150.9178962 MHz
NUC1 13C
P1 11.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 85.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 18.08300018 W

======== CHANNEL f2 ========
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz64
P3 10.20 usec
P4 20.40 usec
PCPD2 80.00 usec
PLW2 27.00000000 W
PLW12 0.43891999 W

F2 - Processing parameters
SI 131072
SF 150.9026411 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Trichostatin C (TSC)




```
Current Data Parameters
NAME            IV-PK-35
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20201203
Time                4.53
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG   cosygpmfppqf
TD                  2048
SOLVENT             MeOD
NS                     2
DS                     8
SWH             6393.862 Hz
FIDRES          3.122003 Hz
AQ              0.1601536 sec
RG                186.92
DW                78.200 usec
DE                 6.50 usec
TE                300.0 K
D0              0.00000300 sec
D1              0.90169531 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00015640 sec

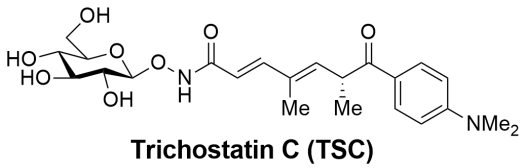
======== CHANNEL f1 ========
SFO1          600.1320220 MHz
NUC1                  1H
P1                 10.00 usec
P17              2500.00 usec
PLW1          26.60000038 W
PLW10          3.93490005 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPNAM[3]      SMSQ10.100
GPZ1              16.00 %
GPZ2              12.00 %
GPZ3              40.00 %
P16             1000.00 usec

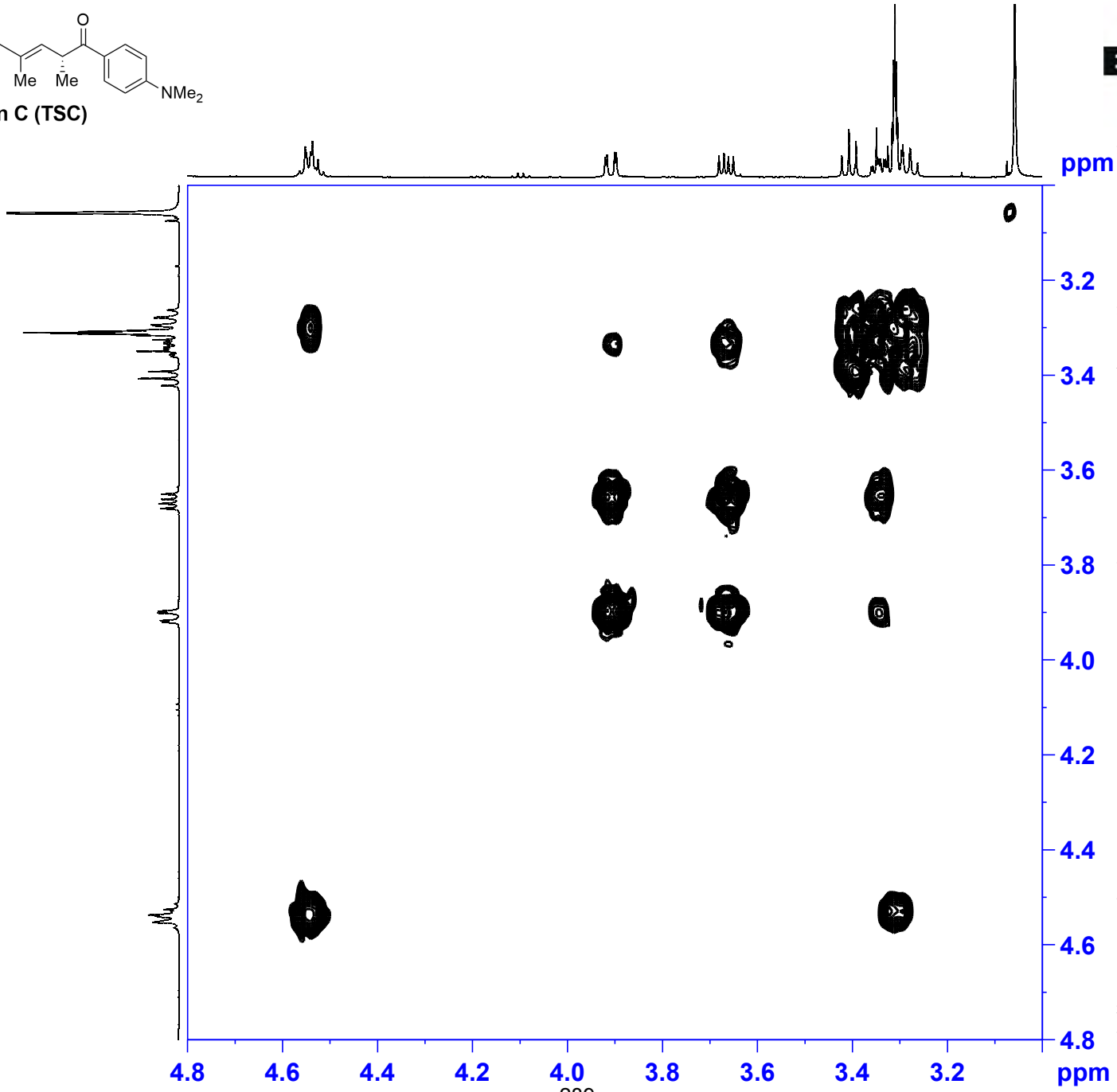
F1 - Acquisition parameters
TD                   256
SFO1             600.132 MHz
FIDRES         49.952045 Hz
SW                10.654 ppm
FnMODE                QF

F2 - Processing parameters
SI                  1024
SF           600.1300110 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
PC                  1.40

F1 - Processing parameters
SI                  1024
MC2                   QF
SF           600.1300130 MHz
WDW                 SINE
SSB       0
LB        0 Hz
GB        0
```



Trichostatin C (TSC)



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```
Current Data Parameters
NAME                  IV-PK-35
EXPNO                       13
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201203
Time                      4.53
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG     cosygpmfppqf
TD                        2048
SOLVENT                   MeOD
NS                           2
DS                           8
SWH                  6393.862 Hz
FIDRES               3.122003 Hz
AQ                   0.1601536 sec
RG                      186.92
DW                      78.200 usec
DE                       6.50 usec
TE                      300.0 K
D0                   0.00000300 sec
D1                   0.90169531 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D13                  0.00000400 sec
D16                  0.00020000 sec
IN0                  0.00015640 sec

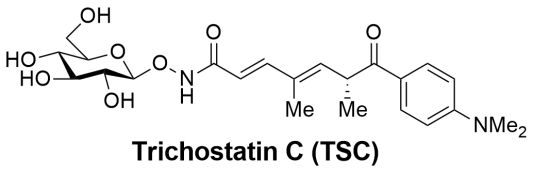
======== CHANNEL f1 ========
SFO1                600.1320220 MHz
NUC1                        1H
P1                       10.00 usec
P17                    2500.00 usec
PLW1                26.60000038 W
PLW10                3.93490005 W

====== GRADIENT CHANNEL =====
GPNAM[1]            SMSQ10.100
GPNAM[2]            SMSQ10.100
GPNAM[3]            SMSQ10.100
GPZ1                     16.00 %
GPZ2                     12.00 %
GPZ3                     40.00 %
P16                    1000.00 usec

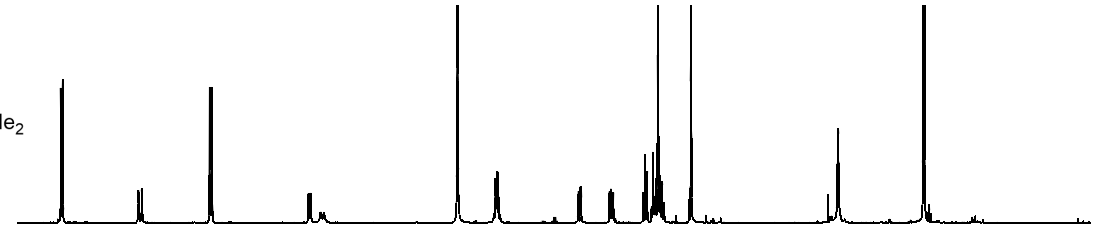
F1 - Acquisition parameters
TD                          256
SFO1                    600.132 MHz
FIDRES                49.952045 Hz
SW                       10.654 ppm
FnMODE                       QF

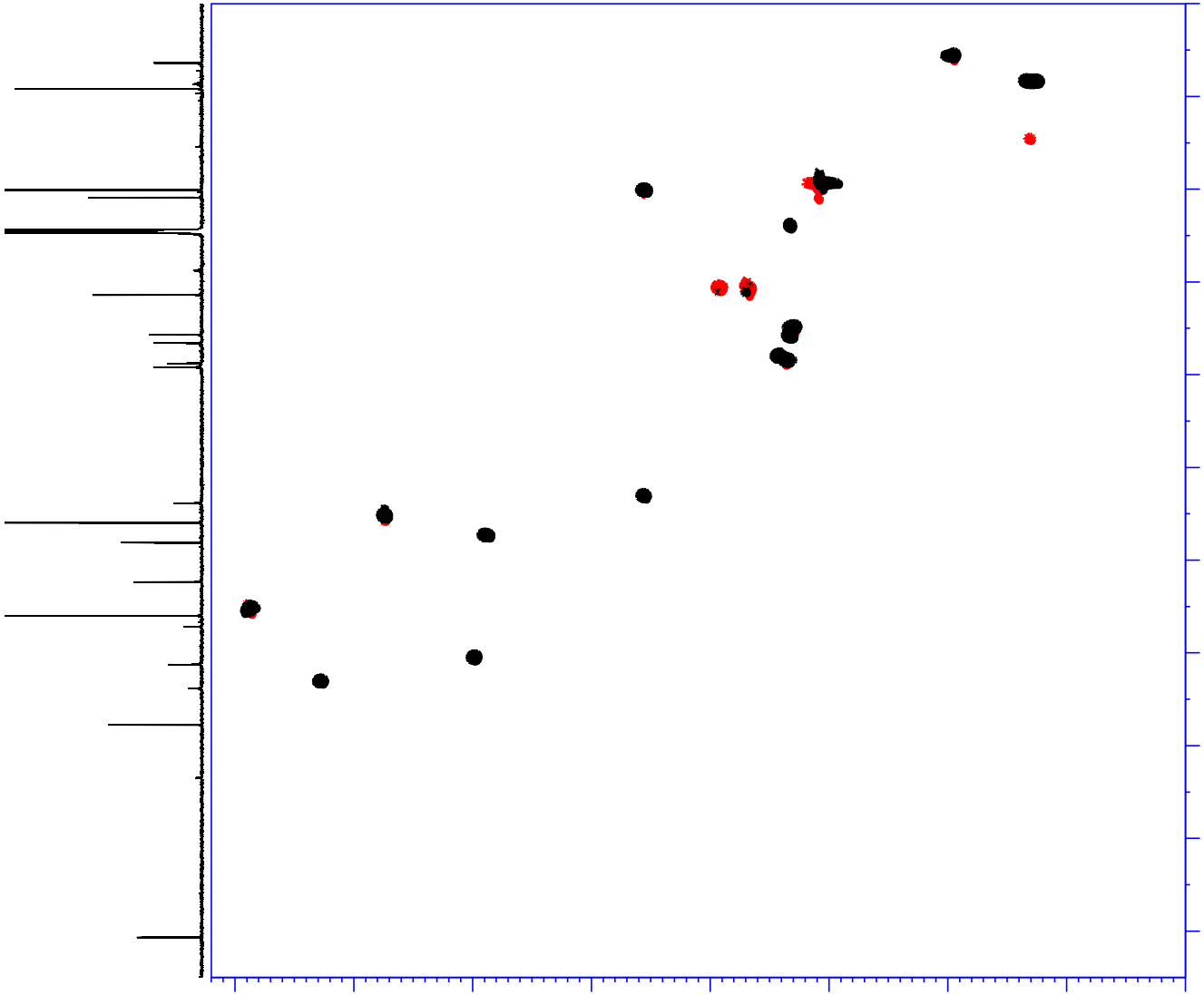
F2 - Processing parameters
SI                         1024
SF                  600.1300110 MHz
WDW                        SINE
SSB                0
LB                 0 Hz
GB                 0
PC                         1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                  600.1300130 MHz
WDW                        SINE
SSB                0
LB                 0 Hz
GB                 0
```



Trichostatin C (TSC)






```
Current Data Parameters
NAME                  IV-PK-36
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20201205
Time                      7.58
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG   hsqcedetgpsp.3
TD                        1024
SOLVENT                   MeOD
NS                           2
DS                          32
SWH                 7211.539 Hz
FIDRES              7.042518 Hz
AQ                  0.0709973 sec
RG                     186.92
DW                     69.333 usec
DE                       6.50 usec
TE                      300.2 K
CNST2              145.0000000
D0                  0.00000300 sec
D1                  0.80000001 sec
D4                  0.00172414 sec
D11                 0.03000000 sec
D16                 0.00020000 sec
D21                 0.00360000 sec
IN0                 0.00001510 sec

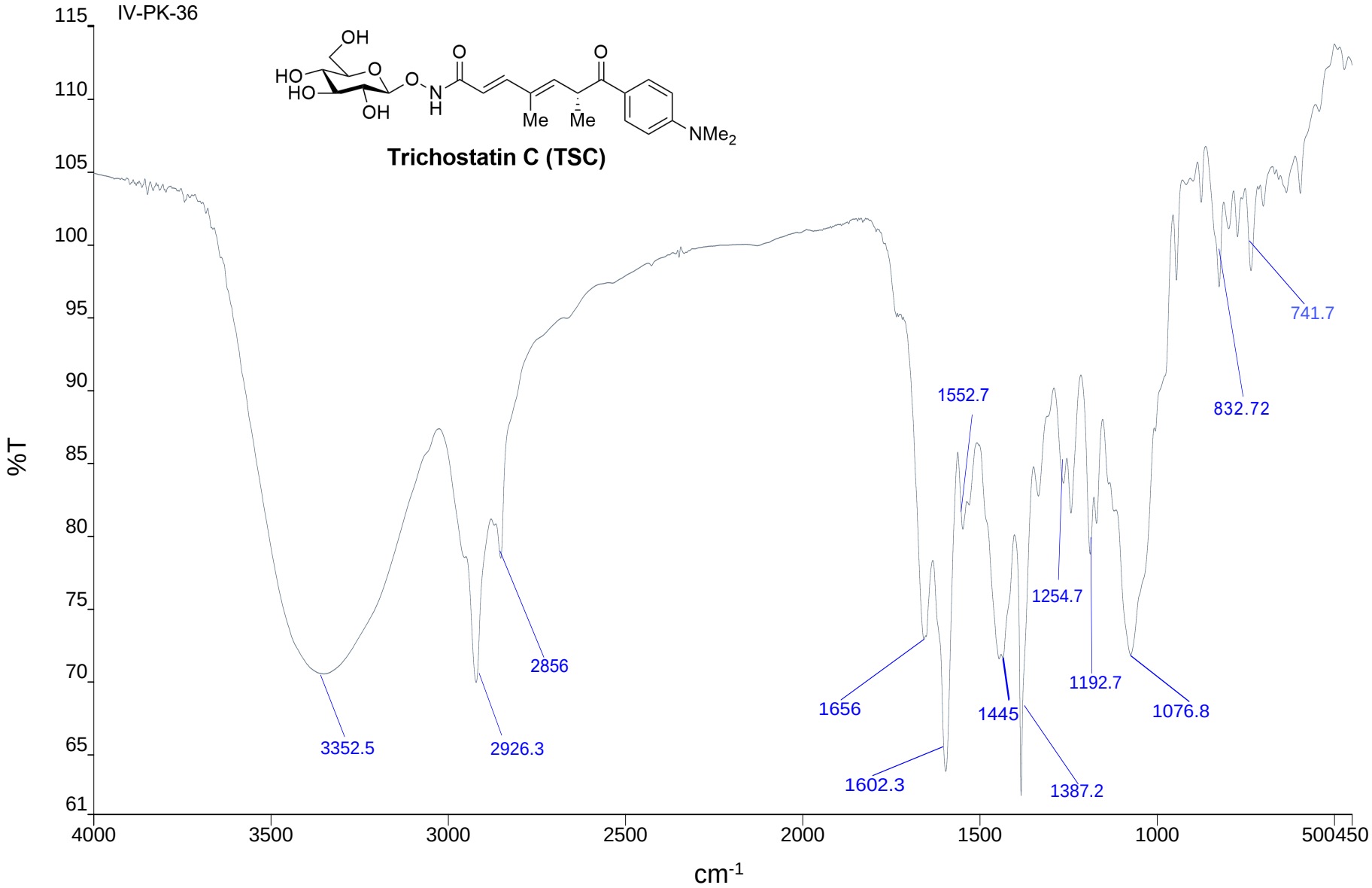
======== CHANNEL f1 ========
SFO1               600.1328223 MHz
NUC1                       1H
P1                      10.00 usec
P2                      20.00 usec
P28             0 usec
PLW1               26.60000038 W

======== CHANNEL f2 ========
SFO2               150.9178988 MHz
NUC2                       13C
CPDPRG[2                garp4
P3                      11.80 usec
P14                    500.00 usec
P31                   1730.00 usec
PCPD2                   65.00 usec
PLW0            0 W
PLW2               85.00000000 W
PLW12               2.80130005 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3                   0.500
SPOFFS3  0 Hz
SPW3               18.08300018 W
SPNAM[18  Crp60_xfilt.2
SPOAL18                  0.500
SPOFFS18 0 Hz
SPW18               5.22629976 W

====== GRADIENT CHANNEL =====
GPNAM[1]      SMSQ10.100
GPNAM[2]      SMSQ10.100
GPZ1                 80.00 %
GPZ2                 20.10 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                        256
SFO1               150.9179 MHz
FIDRES             258.692047 Hz
SW                 219.408 ppm
FnMODE     Echo-Antiecho

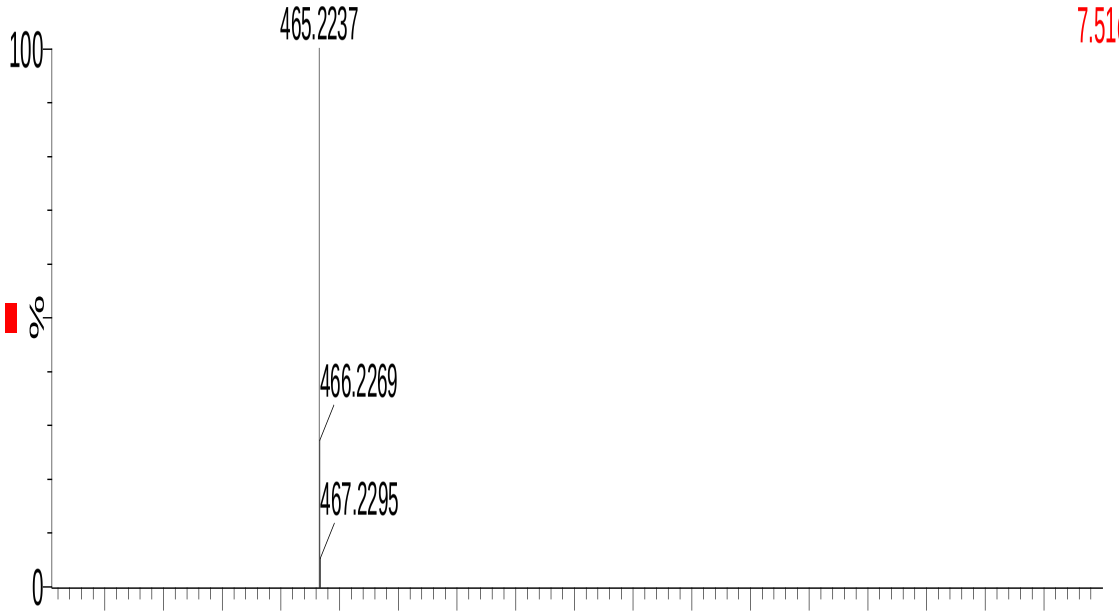
F2 - Processing parameters
SI                       1024
```


IV-PK-36
Trichostatin C (TSC)
%T
cm-1
3352.5
2926.3
2856
1656
1602.3
1552.7
1445
1387.2
1254.7
1192.7
1076.8
832.72
741.7

IV-PK-35 **07-12-2020**

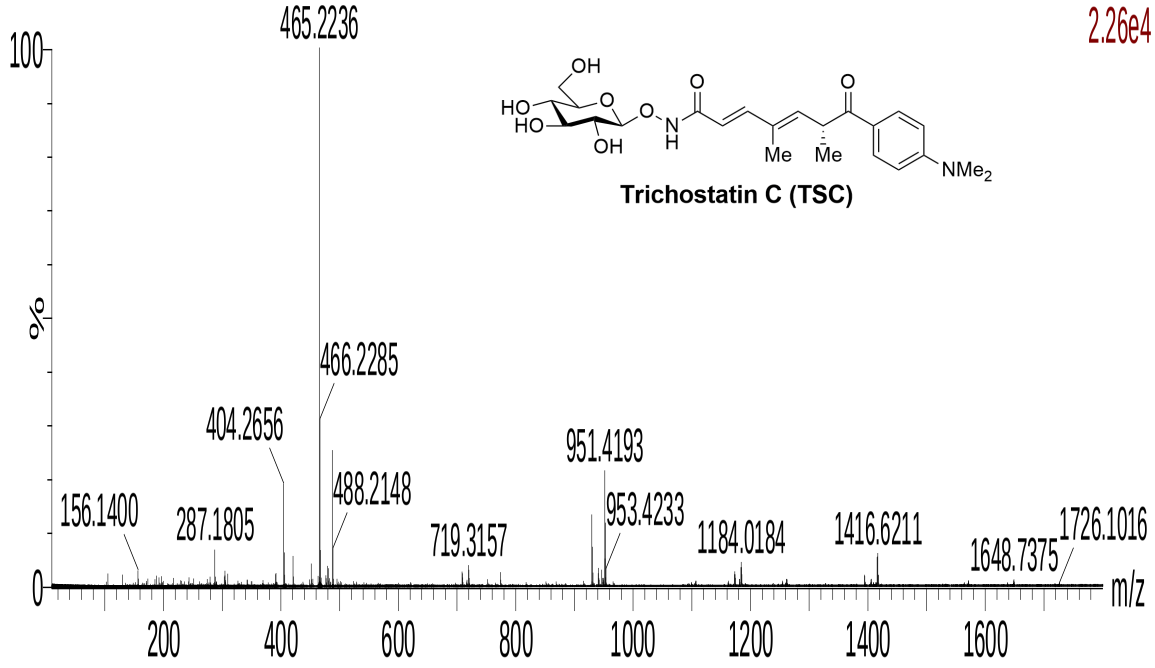
ASEP_11_NOV_2020_330 (0.070) Is (1.00,1.00) C23H32N2O8H 1: TOF MS ES+

7.51e12



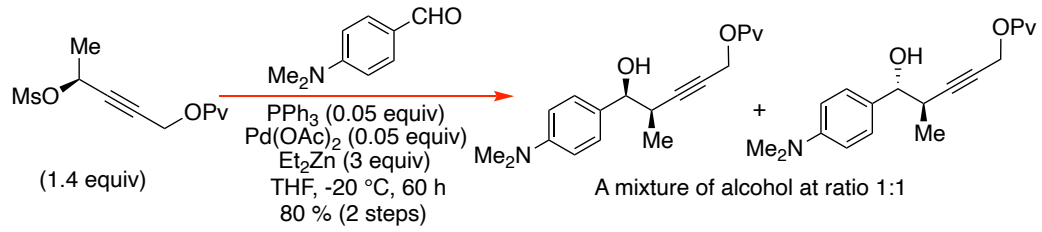
ASEP_11_NOV_2020_330 7 (0.318) Cm (7:10) 1: TOF MS ES+

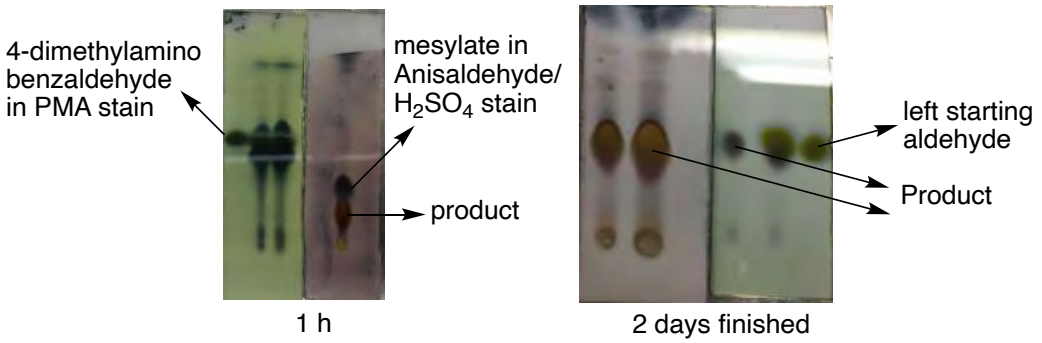
2.26e4



Trichostatin C (TSC)

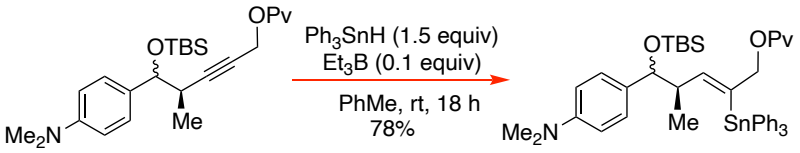
Marshall's allenylzinc addition





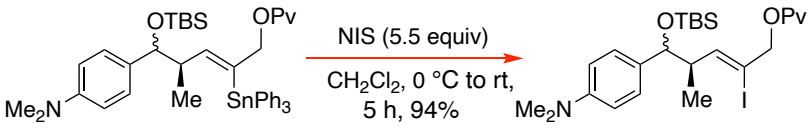
TLC run on 8:1 hex/EtOAc *2, then 3:1 hex/EtOAc or only 3:1 hex/EtOAc; Product was in orange with Anisaldehyde/ H_2SO_4 stain; 4-dimethylamino benzaldehyde little slower-moving than mesylate was in white not clearly shown with Anisaldehyde/ H_2SO_4 stain.

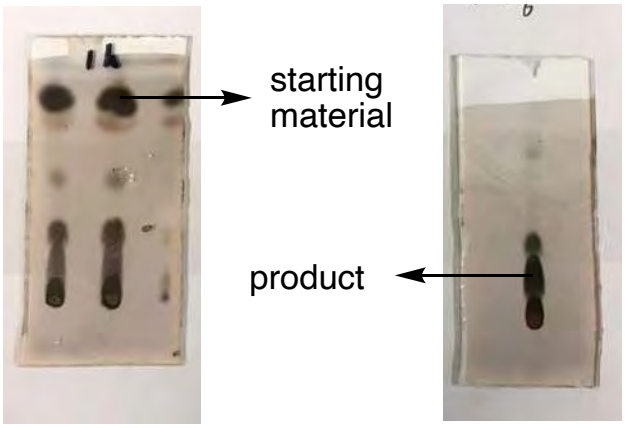
Hydrostannation with Pivaloyl protected propargylic alcohol



TLC run on 10:1 hex/EtOAc with Anisaldehyde/ H_2SO_4 stain

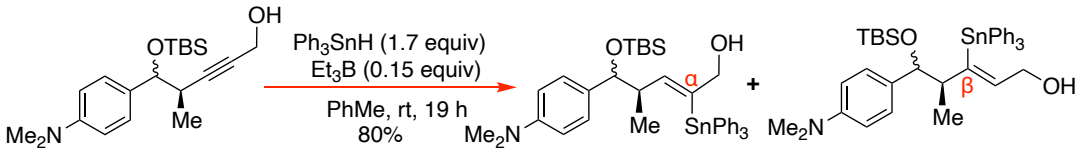
Sn-I exchange with Pivaloyl protected propargylic alcohol.



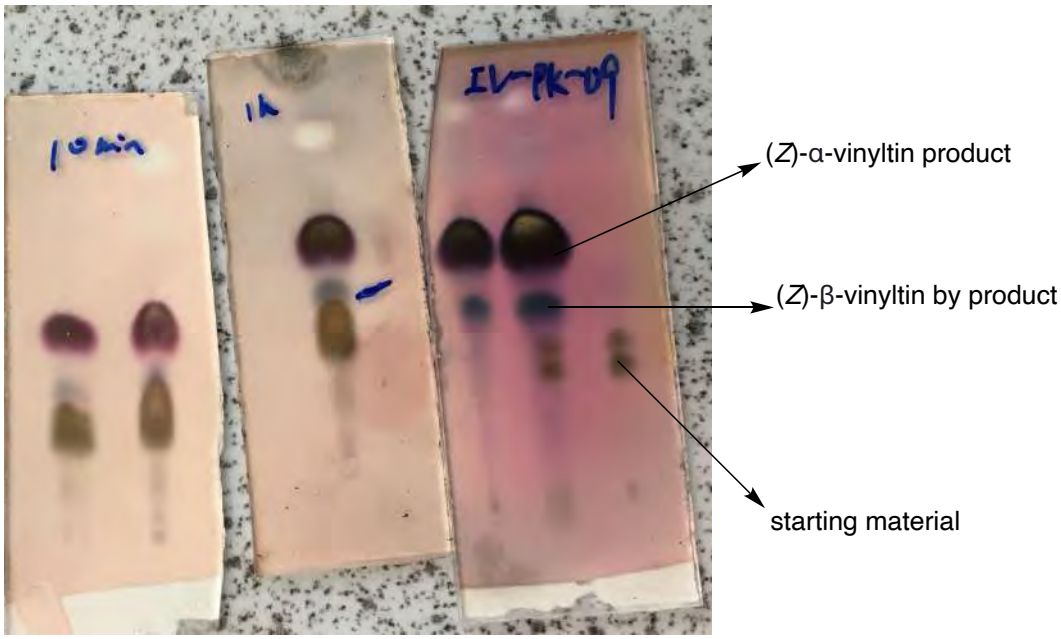


TLC run on 3:1 hex/EtOAc with Anisaldehyde/ H_2SO_4 stain.

Hydrostannation with propargylic alcohol

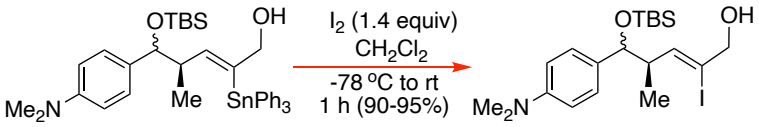


crude ^{1}H NMR α : β = 25 : 1
column out α : β = 16 : 1

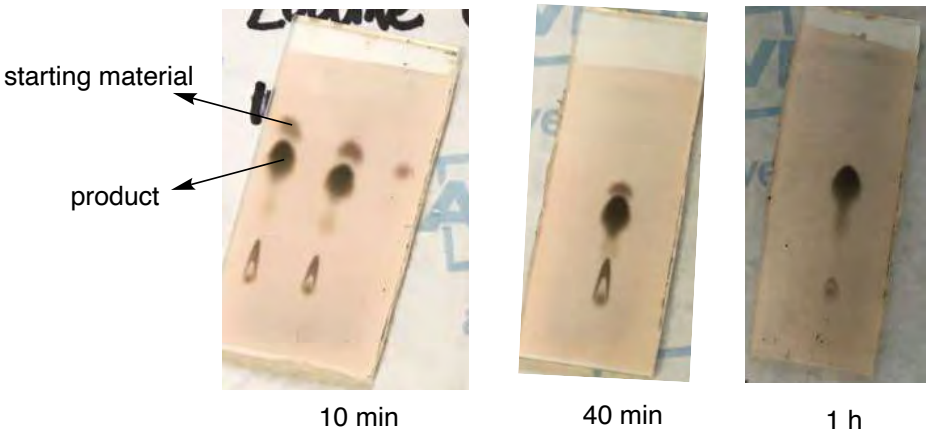


TLC run on 4:1 hex/EtOAc with Anisaldehyde/ H_2SO_4 stain

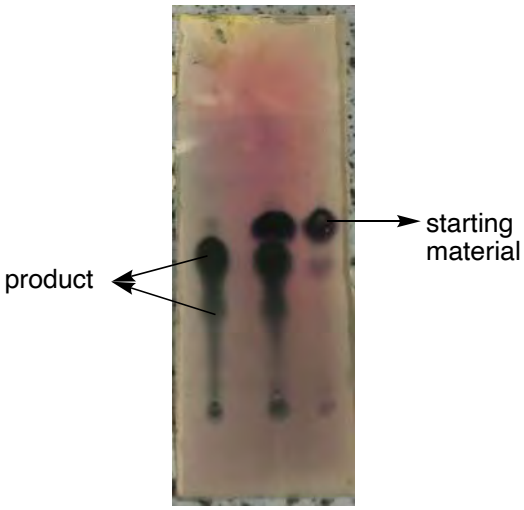
Sn-I exchange with propargylic alcohol.



For a single isomer vinyl iodide from one of the single isomer (*Z*)-α-triphennylvinyltin product, TLC gives clearly on spot. For the mixture, TLC gives two spots of the vinyl iodide compound.

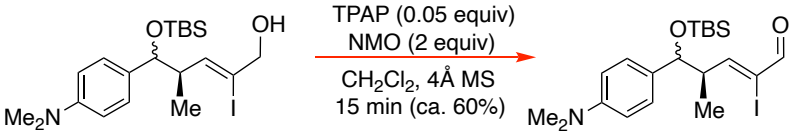


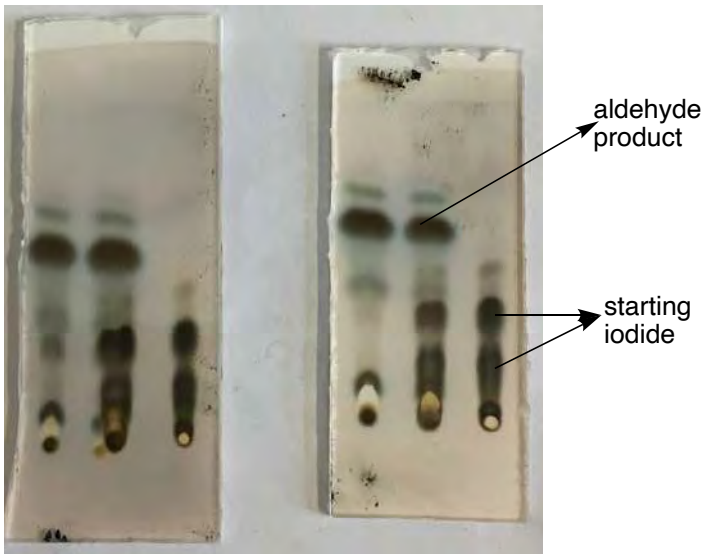
The mixture



TLC run on 4:1 hex/EtOAc with Anisaldehyde/ H_2SO_4 stain

Ley-Griffith oxidation of the vinyl iodide.

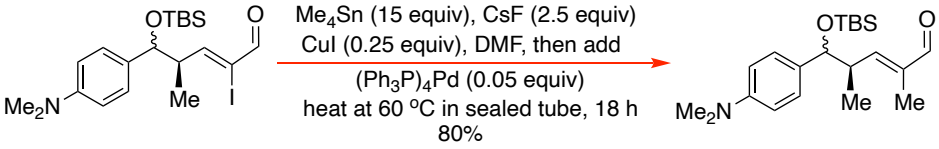


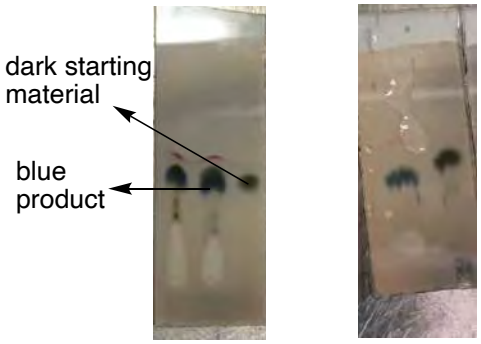


5 min 15 min

TLC run on 4:1 hex/EtOAc with Anisaldehyde/ H_2SO_4 stain.

Baldwin-Lee Stille cross coupling





after water-wash, filter
and concentration

TLC run on 3 : 1 hex/ CH_2Cl_2 in Anisaldehyde/H_2SO_4 stain.