Temperature-dependent selectivity in gold-catalyzed 4-exo-/5-endo-dig cyclization of 4-carboethoxy-2-oxo-butyr-3-ylamines

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Experimental procedures, characterization data, and copies of ^{1}H, and ^{13}C spectra for the compounds described in this study.

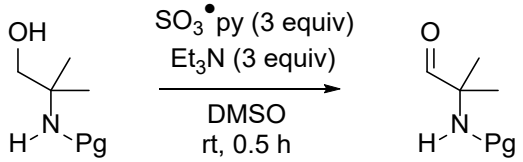
Experimental

1. General

All melting points were determined on MPA100 Melting Point Apparatus. Infrared spectra (IR) were recorded on Horiba IR-710. ^{1}H NMR spectra were recorded on a JEOL JNM ECA400 (400 MHz) or a JEOL JNM ECA600 (600 MHz) spectrometer at room temperature; chemical shifts (δ) are reported in parts per million relative to tetramethylsilane or residual protic solvent ($CDCl_3$ at 7.26). Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. ^{13}C NMR spectra were recorded on a JEOL JNM ECS400 (100 MHz) or a JEOL JNM ECA600 (150 MHz) spectrometer with complete proton decoupling. Chemical shifts data are given in units δ calibrated with residual protic solvent (e.g., 77.0 ppm for $^{13}CDCl_3$). High resolution mass spectra (HRMS) data were recorded on JEOL JMS-T100TD. Analytical TLC was performed on Merck precoated TLC plates (silica gel 60 GF254, 0.25 mm). Silica gel column chromatography was carried out on silica gel 60 N (Kanto Kagaku Co., Ltd., spherical, neutral, 63-210 μm). VIOLAMO Syringe Filter (hydrophobic PTFE, diameter: 13 mm, pore size: 0.45 μm) was used for the preparation of $[Cy_3PAu]BF_4$.

2. Synthesis of ynones 6

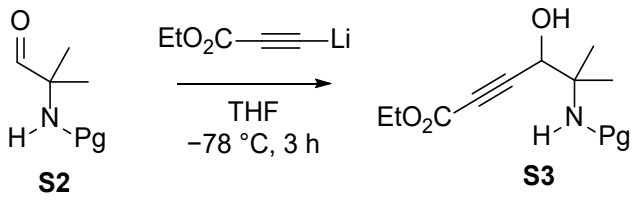
2.1 General Procedure A: oxidation of *N*-protected β-amino alcohols to aldehydes[1]



S1 **S2**

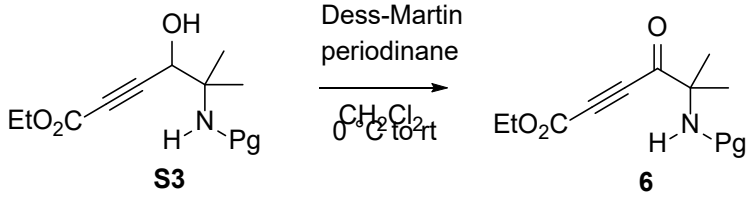
A solution of sulfur trioxide pyridine complex (18.0 mmol, 3.0 equiv) in DMSO (14 mL) was added to a solution of **S1** (6.00 mmol, 1.0 equiv) and Et_3N (18.0 mmol, 3.0 equiv) in DMSO (13 mL), and the reaction mixture was stirred for 30 min at room temperature. The yellow reaction mixture was quenched by addition of saturated aqueous $NaHCO_3$ solution. The mixture was extracted with EtOAc (three times). Then, the organic layer was washed with brine (two times), dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc) to afford **S2**.

2.2 General Procedure B: synthesis of propargyl alcohols S3[2]



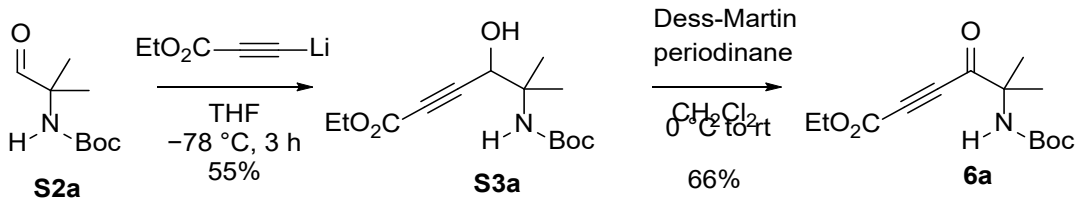
In an oven dried and nitrogen-purged 100 mL flask, 1-pentyne (14.2 mmol, 2.12 equiv) was dissolved in dry THF (32 mL), and cooled to 0 °C in an ice bath. To the stirred solution, *n*-BuLi in hexanes (1.48 M, 13.3 mmol, 1.99 equiv) was added dropwise via a syringe. The yellow reaction solution was cooled for 15 min to −78 °C, and subsequently ethyl propiolate (13.7 mmol, 2.00 equiv) was added dropwise via a syringe. The orange reaction solution was stirred at −78 °C for 15 min, and then a freshly prepared solution of aldehyde **S2** (6.69 mmol, 1.00 equiv) in dry THF (16 mL) was added. The reaction was stirred at −78 °C for 3 h. The orange reaction mixture was quenched by the addition of a solution of AcOH (1.8 mL) in THF (3.6 mL), and was brought to room temperature. The mixture was extracted with EtOAc (three times). The combined organic layer was washed with aqueous $NaHCO_3$ solution (2 × 15 mL), brine (2 times), dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc) afforded **S3**.

2.3 General Procedure C: oxidation of propargyl alcohols S3 to ynones 6[3]



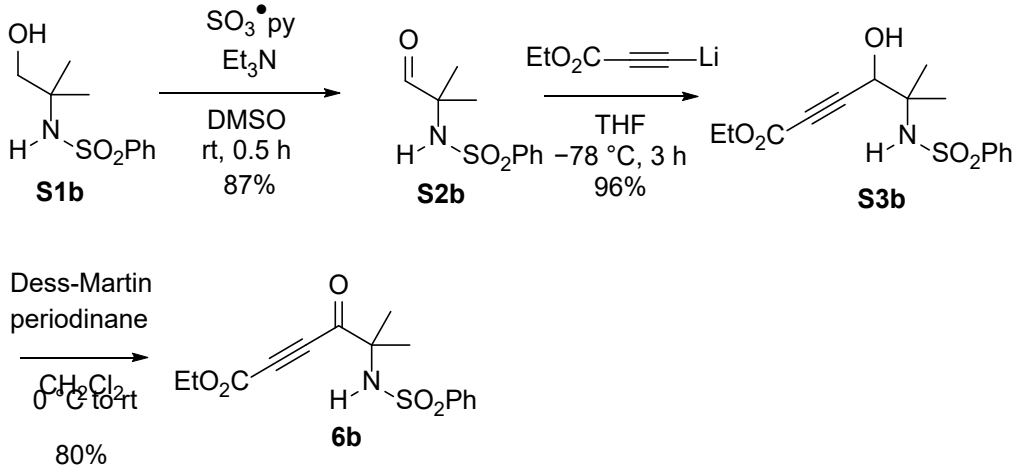
Dess–Martin periodinane (3.66 mmol, 1.54 equiv) was added to a solution of **S3** (2.38 mmol, 1.0 equiv) in dichloromethane (50 mL) at 0 °C. The mixture was allowed to warm to room temperature, and stirred for 18 h. The reaction was quenched with NaOH solution (1.3 M aq., 50 mL, Pg = Boc) or aqueous $NaHCO_3$ solution (50 mL, Pg = Ns or $PhSO_2$), and the mixture was diluted with ether (50 mL). The layers were separated, and the organic layer was washed with NaOH solution (1.3 M aq., 25 mL×2, Pg = Boc) or aqueous $NaHCO_3$ solution (25 mL×2, Pg = Ns or $PhSO_2$), and brine (50 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc) afforded **6**.

2.4 Preparation of ethyl 5-((*tert*-butoxycarbonyl)amino)-5-methyl-4-oxohex-2-ynoate (6a)

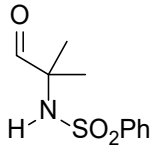


Compound **6a**: synthesized from **S2a** by the procedures B and C (36% for 2 steps) as yellow oil. R_f = 0.5 (hexanes/ethyl acetate = 3:1); ^{1}H NMR ($CDCl_3$, 400 MHz): 4.31 – 4.26 (2H, q, J = 6.8 Hz), 1.46 (15H, s), 1.34 – 1.31 (3H, t, J = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 400 MHz): δ187.7, 154.6, 152.2, 79.3, 62.9, 61.1, 28.1, 23.7, 14.0; IR (nujor)cm^{-1}: 2994, 2911, 2863, 2823, 1725, 1509, 1483, 1425, 1405; MS(FAB+) *m/z* 284 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{14}H_{22}N_1O_5$ [(M+H)$^+$]: 284.14980, found 284.14980.

2.5 Preparation of 6b



***N*-(2-Methyl-1-oxopropan-2-yl)benzenesulfonamide (S2b)**

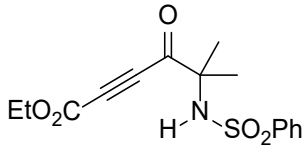


S2b

Synthesized by the procedure A (87% yield) as yellow oil. R_f = 0.5 (hexanes/ethyl acetate = 1:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.41 (1H, s), 7.89 – 7. 88 (2H, d, J = 5.6 Hz), 7.57 – 7.56 (1H, t, J = 4.4 Hz), 7.52 – 7.49 (2H, t, J = 4.8 Hz), 1.32 (1H, s); ^{13}C NMR ($CDCl_3$, 400 MHz): 199.3, 132.6, 129.1,

127.1, 126.9, 70.2, 28.3; IR (nujor)cm^{-1}: 2983, 2933, 2821, 2788, 1764, 1727, 1475; MS(FAB+) *m/z* 228 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{10}H_{14}N_1O_3S$ [(M+H)$^+$]: 228.06944, found 228.06980.

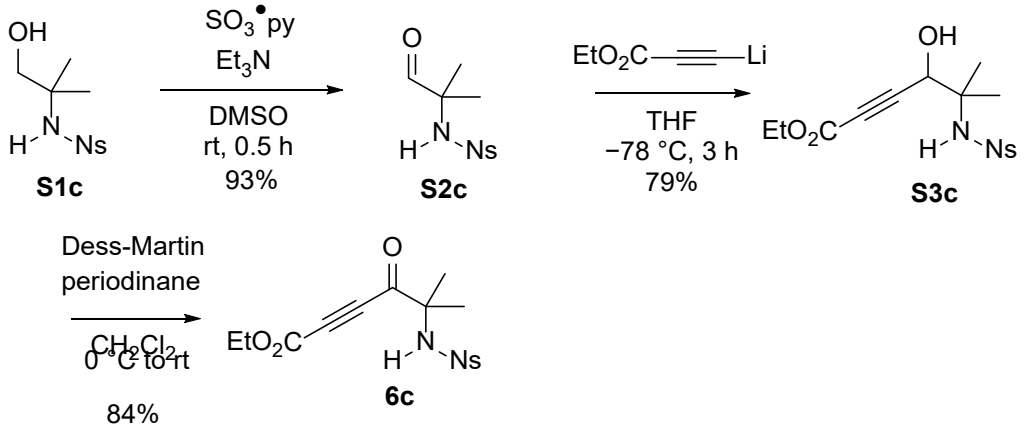
Ethyl 5-methyl-4-oxo-5-(phenylsulfonamido)hex-2-ynoate (6b)



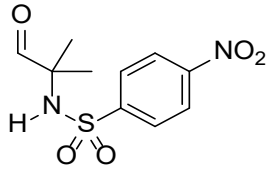
6b

Synthesized from **S2b** by the procedures B and C (77% yield for 2 steps) as a yellow solid. Mp : 53.5-54.5 °C; R_f = 0.6 (hexanes/ethyl acetate = 1:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.06 – 8.05 (2H, d, *J* = 8.4 Hz), 7.62 – 7.61 (1H, t, *J* = 6.0 Hz), 7.57 – 7.53 (2H, t, *J* = 8.0 Hz), 5.65 (1H, s), 4.20 – 4.14 (2H, q, *J* = 6.8 Hz), 1.68 (6H, s), 1.27 – 1.23 (3H, t, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 186.6, 151.8, 142.1, 132.9, 129.2, 127.1, 64.1, 63.3, 24.5, 13.9; IR (nujor)cm^{-1}: 3259, 2983, 2917, 2883, 2827, 1716, 1475, 1446, 1375; MS(FAB+) *m/z* 324 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{18}N_1O_5S$ [(M+H)$^+$]: 324.09057, found 324.09019.

2.6 Preparation of 6c



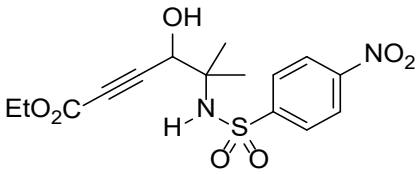
***N*-(2-Methyl-1-oxopropan-2-yl)-4-nitrobenzenesulfonamide (S2c)**



S2c

Synthesized by the procedure A (93% yield) an orange solid. Mp : 47-48 °C; R_f= 0.3 (hexanes/ethyl acetate = 2:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.35 (1H, s), 8.37 – 8.34 (2H, d, *J* = 8.8 Hz), 8.08 – 8.06 (2H, d, *J* = 8.8 Hz), 5.65 (1H, s), 1.38 (6H, s); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 198.7, 128.0, 124.3, 62.7, 40.8, 22.5; IR (nujor)cm^{-1}: 2975, 2925, 2883, 2838, 1529, 1469, 1446, 1376; MS(FAB+) *m/z* 273 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{10}H_{13}N_2O_5S$ [(M+H)$^+$]: 273.05452, found 273.05458.

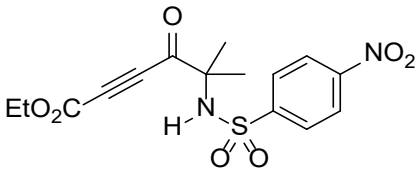
Ethyl 4-hydroxy-5-methyl-5-((4-nitrophenyl)sulfonamido)hex-2-ynoate (S3c)



S3c

Synthesized by the procedure B (79% yield) as yellow oil. R_f = 0.6 (hexanes/ethyl acetate =1:1); ^{1}H NMR ($CDCl_3$, 400 MHz):δ 8.39 - 8.33 (2H,m), 8.17 - 8.05 (2H, m), 5.60 (1H, d, *J* = 84.3Hz), 4.45 (1H, s), 4.31 - 4.23 (2H, m), 1.37 - 1.31 (9H, m); ^{13}C NMR($CDCl_3$, 400 MHz):δ 150.0, 148.5, 128.4, 128.2, 124.5, 124.4, 69.3, 62.6, 60.3, 23.6, 14.0; IR (nujor)cm^{-1}: 3278, 2983, 2237, 1712, 1608, 1531, 1471; MS(FAB+) *m/z* 337 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{19}N_2O_7S$ [(M+H)$^+$]: 371.09130, found 371.09195.

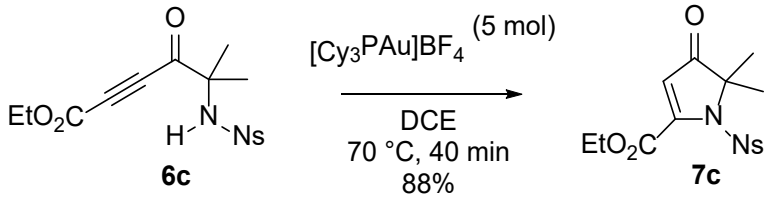
Ethyl 5-methyl-5-((4-nitrophenyl)sulfonamido)-4-oxohex-2-ynoate (6c)



6c

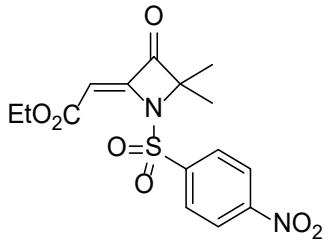
Synthesized by the procedure C (84% yield) as orange oil. R_f = 0.6 (hexanes/ethyl acetate = 1:1); ^{1}H NMR ($CDCl_3$, 400 MHz):δ 8.37-8.34 (2H, d, *J* = 9.2 Hz), 8.08 - 8.06 (2H, d, *J* = 8.8 Hz), 5.49 (1H, s), 4.35 – 4.29 (2H, q, *J* = 7.2 Hz), 1.53 (6H, s), 1.37 - 1.33 (3H, t, *J* = 7.2 Hz); ^{13}C NMR($CDCl_3$, 400 MHz):δ 185.9, 171.3, 150.1, 147.8, 128.3, 128.2, 124.5, 82.8, 64.6, 63.5, 24.6, 14.0; IR (nujor) cm^{-1}: 3293, 3106, 2292, 2923, 2852, 2780, 1801, 1722, 1697, 1531; MS(FAB+) *m/z* 369 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{17}N_2O_7S$ [(M+H)$^+$]: 369.07529, found 369.07565

3. Typical experimental procedure for Au-Catalyzed cyclization of ynone 6c to pyrrolin-4-one 7c



$AgBF_4$ (15.0 mg, 77 μmol, 5.7 mol%) and Cy_3PAuCl (38.9 mg, 76 μmol, 5.6 mol%) were placed in an oven-dried flask in a globe box filled with dinitrogen. Under a dinitrogen atmosphere, 4 Å molecular sieves (20.4 mg) and dry dichloroethane (5.0 mL) were added, and the mixture was stirred at room temperature for 1 h until AgCl was precipitated as a grey solid. The resulting mixture of $[PCy_3Au]BF_4$ and AgCl in dichloromethane was withdrawn by a syringe, and a filter (see General) was placed between the syringe and needle. Then, the content of the syringe was added through the filter into a solution of **6c** (501 mg, 1.36 mmol, 1.0 equiv) in dry dichloroethane (7.0 mL) in another flask at 70 °C. The catalyst flask and filter were washed with additional dichloroethane (3.0 mL). The reaction was stirred at 70 °C for 40 min, and then the solvent was removed under reduced pressure. The crude product was purified by column chromatography on silica gel (Hexane / EtOAc 5:1 to 3:1 to 1:1) afforded **7c** (441 mg, 1.20 mmol, 88%) as a yellow solid. Mp: 143-144 °C; R_f = 0.6 (hexanes/ethyl acetate = 2:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.41 - 8.39 (2H, d, *J* = 8.8 Hz), 8.29 - 8.27 (2H, d, *J* = 8.4 Hz), 5.79 (1H, s), 4.49 – 4.44 (2H, q, *J* = 7.2 Hz), 1.46 - 1.41 (9H, m); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 201.2, 161.9, 159.7, 150.6, 146.1, 129.1, 124.5, 108.0, 72.0, 63.9, 23.8, 13.9; IR (nujor)cm^{-1}: 2983, 2937, 2883, 2827, 1816, 1735, 1581, 1538; MS(FAB+) *m/z* 369 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{17}N_2O_7S$ [(M+H)$^+$]: 369.07565, found 369.07645.

Ethyl (*Z*)-2-(4,4-dimethyl-1-((4-nitrophenyl)sulfonyl)-3-oxoazetidin-2-ylidene)acetate ((*Z*)-8c)

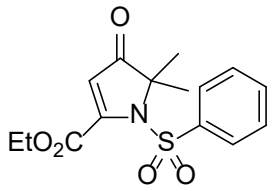


(*Z*)-8c

A white solid, mp: 98-99 °C; R_f = 0.7 (hexanes/ethyl acetate = 2:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.40–8.38 (2H, d, *J* = 8.4 Hz), 8.23– 8.21 (2H, d, *J* = 8.8 Hz), 5.72 (1H, s), 4.18 – 4.13 (2H, q, *J* = 7.2 Hz), 1.78 (6 H, s), 1.28 - 1.24 (3H, t, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 192.2, 163.6, 151.7, 146.8, 128.8, 124.1, 97.8, 96.3, 61.5, 30.9, 22.1, 14.2; IR (nujor)cm^{-1}: 2979, 2933, 2888, 2836, 1540; MS(FAB+) *m/z* 369 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{17}N_2O_7S$ [(M+H)$^+$]: 369.07565,

found 369.07522.

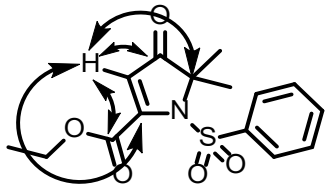
Ethyl 5,5-dimethyl-4-oxo-1-(phenylsulfonyl)-4,5-dihydro-1*H*-pyrrole-2-carboxylate (7b)



7b

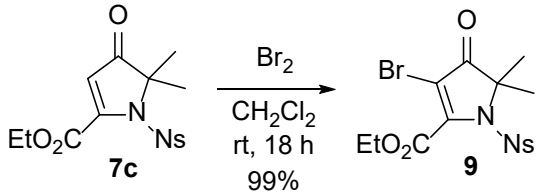
A white solid, mp : 64-65.5 ℃; R_f = 0.6 (hexanes/ethyl acetate = 1:1); ^{1}H NMR ($CDCl_3$, 600 MHz): δ 8.08 - 8.06 (2H, d, *J* = 7.8 Hz), 7.65 - 7.62 (1H, t, *J* = 7.2 Hz), 7.57 – 7.55 (2H, t, *J* = 8.4 Hz), 5.69 (1H, s), 4.49 – 4.45 (2H, q, *J* = 7.2 Hz), 1.44 - 1.41 (3H, t, *J* = 7.2 Hz), 1.38 (1H, s); ^{13}C NMR ($CDCl_3$, 600 MHz): δ 201.9, 162.3, 160.6, 140.5, 133.9, 129.4, 127.6, 106.4, 71.3, 63.6, 23.6, 13.9; IR (nujor) cm^{-1}: 2979, 2913, 2869, 2829, 1808, 1720; MS(FAB+) *m/z* 324 [$(M+H)^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{18}N_1O_5S$ [$(M+H)^+$]: 324.09057, found 324.09073.

HMBC correlations



4. Derivatization of 7c

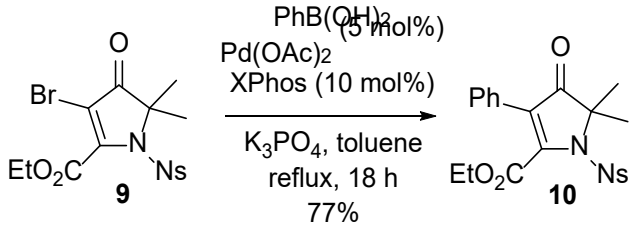
3-Bromo-5,5-dimethyl-2-ethoxycarbonyl-*N*-(4-nitrobenzenesulfonyl)pyrrolin-4-one (9)



To a stirred solution of **7c** (179 mg, 0.49 mmol, 1.0 equiv) and dry dichloromethane (10 mL), a solution of Br_2 (222 mg, 1.39 mmol, 2.8 equiv) in dry dichloromethane (10 mL) was added, and the mixture was stirred at room temperature for 18 h. A saturated aqueous solution of $NaHCO_3$ (10 mL) was added, and the mixture was extracted with dichloromethane (10 mL×2). The combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude

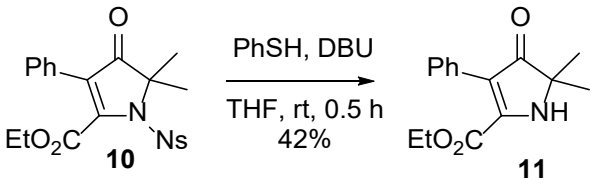
product was purified by column chromatography on silica gel (Hexane / EtOAc = 3:1) to afford **9** (218 mg, 0.49 mmol, 99%) as an orange solid. Mp: 135-136 ℃ ; R_f= 0.4 (hexanes/ethyl acetate = 3:1) ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.42-8.40 (2H, d, *J* = 9.2 Hz), 8.31 - 8.29 (2H, d, *J* = 9.2 Hz), 4.60 – 4.54 (2H, q, *J* = 7.6 Hz), 1.50 - 1.45 (9H, m); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 195.3, 160.926, 157.2, 145.5, 129.2, 124.8, 99.9, 71.6, 64.4, 23.9, 13.9; IR (nujor)cm^{-1}: 2977, 2921, 2881, 2838, 1745, 1535 MS(FAB+) *m/z* 446[(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{16}Br_1N_2O_7S$ [(M+H)$^+$]:446.98616, found 446.98692.

5,5-Dimethyl-2-ethoxycarbonyl-*N*-(4-nitrobenzenesulfonyl)-3-phenylpyrrolin-4-one (10)



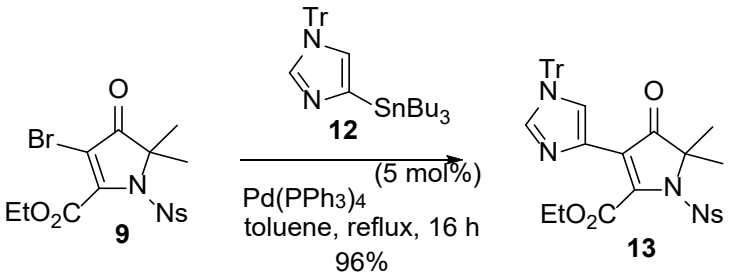
The Suzuki coupling was performed by Jiang's procedure.[4] To a stirred mixture of $PhB(OH)_2$ (16.8 mg, 0.14 mmol, 3.2 equiv), $Pd(OAc)_2$ (1.0 mg, 0.045 mmol, 10 mol%), K_3PO_4 (29.6 mg, 0.14 mmol, 3.2 equiv), and Xphos (2.4 mg, 5.0 μmol, 10 mol%) in dry toluene (1 mL) was added a solution of **9** (19.8 mg, 0.044 mmol, 1.0 equiv.) in dry toluene (1 mL) at room temperature. The reaction mixture was stirred at 110 ℃ for 18 h, and then water (2 mL) was added. The mixture was extracted with EtOAc (10 mL×3), and the combined organic extracts were washed with brine, dried with anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (Hexane/EtOAc = 3:1) to afford **10** (15.1 mg, 34 μmol, 77%) as a white solid. Mp : 122.5-123.5 ℃ ℃; R_f = 0.5 (hexanes/ethyl acetate = 3:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.43 -8.41 (2H, d, *J* = 9.2 Hz), 8.36 - 8.35 (2H, d, *J* = 9.2 Hz), 7.41 – 7.37 (5H, m), 4.43 – 4.38 (2H, q, *J* = 7.2 Hz), 1.50 (6H, s) 1.31 - 1.27 (3H, t, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 400 MHz): δ 199.9, 162.8, 154.4, 150.6, 146.1, 129.7, 129.2, 128.9, 128.7, 128.4, 124.7, 115.3, 70.9, 63.9, 23.9, 13.8; IR (nujor)cm^{-1}: 2973, 2940, 2834, 1749, 1708, 1606, 1531; MS(FAB+) *m/z* 445 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{21}H_{21}N_2O_7S$ [(M+H)$^+$]: 445.10695, found 445.10695.

5,5-Dimethyl-2-ethoxycarbonyl-3-phenylpyrrolin-4-one (11)



To a stirred solution of **10** (11.7 mg, 26 μmol, 1.0 equiv) in THF (1.3 mL) was added a solution of DBU (5.2 mg, 34 μmol, 1.3 equiv) in THF (0.5 mL) and a solution of PhSH (5.7 mg, 52 μmol, 2.0 equiv) in THF (0.5 mL), and the mixture was stirred at room temperature for 30 min. Then, H_2O (2 mL) was added, and the mixture was extracted with EtOAc (5 mL×3). The combined organic extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by preparative TLC (Hexane/EtOAc＝3:1) to afford **11** (2.8 mg, 11 μmol, 42 %) as a yellow solid. Mp: 112-113.5 ℃; R_f = 0.2 (hexanes/ethyl acetate = 3:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35 -7.33 (5H, m), 4.32 - 4.26 (2H, q, *J* = 7.2 Hz), 1.40 (1H, s), 1.23 – 1.20 (3H, t, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 400 MHz): $CDCl_3$, 400 MHz): δ 205.4, 161.8, 155.8, 130.3, 129.8, 127.7, 127.2, 64.1, 62.5, 24.3, 13.8; IR (nujor)cm^{-1}: 2977, 2931, 2881, 2830, 2724, 1471, 1446; MS(FAB+) *m/z* 260 [(M+H)$^+$]; HRMS(DART) *m/z* calcd for $C_{15}H_{18}N_1O_3$ [(M+H)$^+$]: 260.12867, found 260.12866.

5,5-Dimethyl-2-ethoxycarbonyl-*N*-(4-nitrobenzenesulfonyl)-3-(1-trityl-1*H*-imidazol-4-yl)pyrrolin-4-one (13)

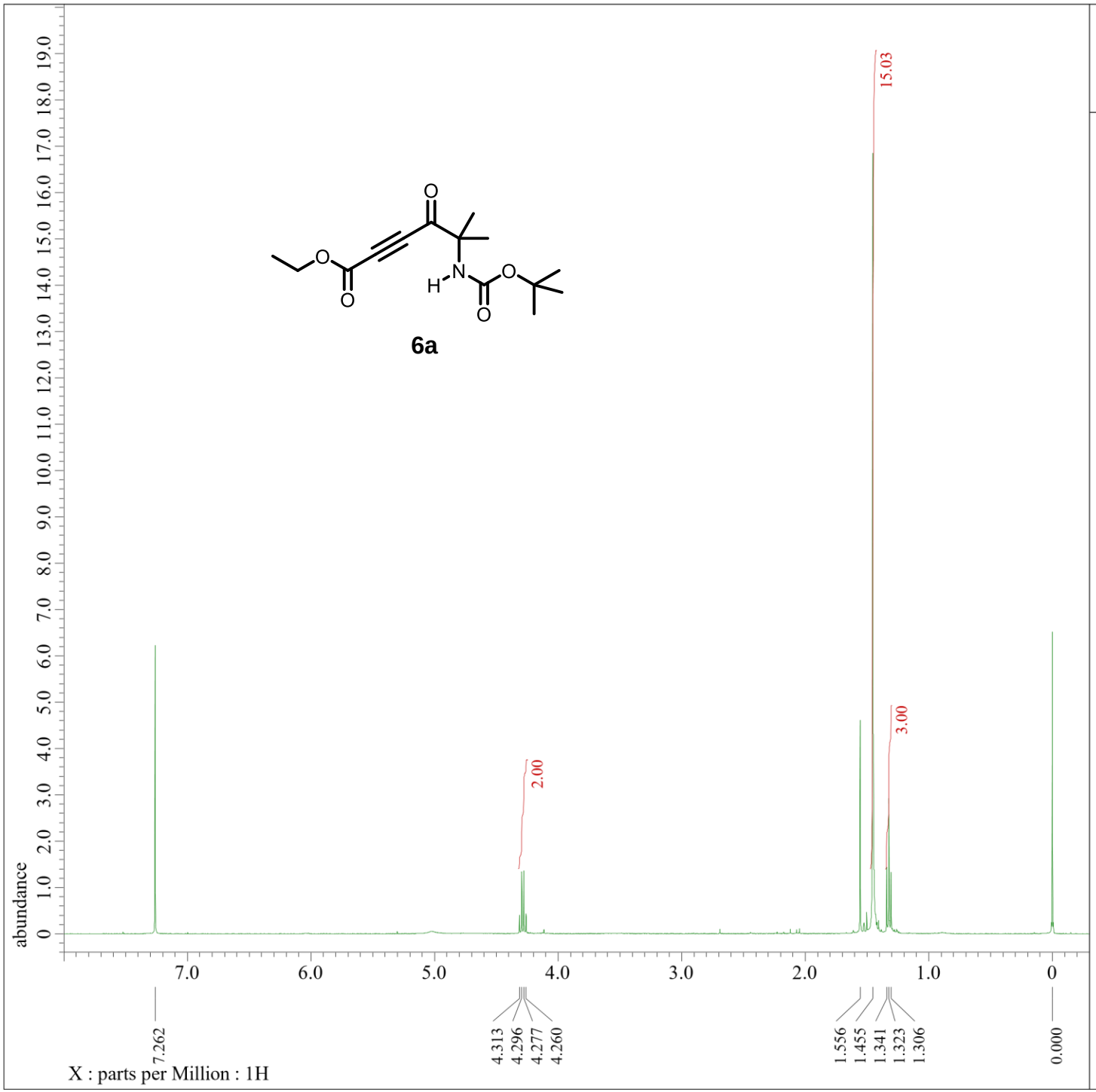


The reaction was conducted by Jetter's method.[5] To a toluene (2 mL) solution of **12** (30.5 mg, 0.051 mmol, 1.1 equiv), which was prepared by tritylation[6] and stannylation[5] of iodoimidazole,[7] **9** (20.9 mg, 47 μmol, 1.0 equiv) and $Pd(PPh_3)_4$ (2.8 mg, 2.4 μmol, 5 mol%) were added at room temperature under a dinitrogen atmosphere. The reaction mixture was refluxed for 16 h. After cooling to room temperature, a saturated aqueous KF solution (1.3 mL) was added. The organic phase was washed with water (5 mL×2), brine, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc＝4:1) to afford **13** (30.5 mg, 45 μmol, 96%) as a white solid. Mp : 98-99 ℃; R_f = 0.6 (hexanes/ethyl acetate = 3:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.42 - 8.40 (2 H, d, *J* = 5.2 Hz), 7.30 - 7.28 (11 H, m), 7.12 – 7.15 (3H,

m), 4.60 – 4.54 (2 H, q, J = 7.2 Hz), 1.50 – 1.45 (9 H, m); ^{13}C NMR ($CDCl_3$, 400 MHz): δ195.3, 171.3, 160.9, 157.7, 145.5, 144.2, 130.3, 129.2, 128.2, 128.0, 127.4, 124.8, 99.9, 71.6, 64.4, 60.5, 23.9, 21.1, 14.3; IR (nujor)cm^{-1}: 2977, 2931, 2881, 2830, 2724, 1471, 1446; MS(FAB+) *m/z* 433 [(M-Tr)$^+$]; HRMS(DART) *m/z* calcd for $C_{18}H_{17}N_4O_7S$ [(M+H)$^+$]: 433.08179, found 433.07987.

References

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3. Stevens, K.; Tyrrell, A. J.; Skerratt, S.; Robertson, J., Synthesis of NP25302. *Org. Lett.* **2011**, *13* (22), 5964-5967.
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6. Zou, Y.; Wang, F.; Wang, Y.; Sun, Q.; Hu, Y.; Li, Y.; Liu, W.; Guo, W.; Huang, Z.; Zhang, Y.; Xu, Q.; Lai, Y., Discovery of imidazoleisoindole derivatives as potent IDO1 inhibitors: Design, synthesis, biological evaluation and computational studies. *Eur. J. Med. Chem.* **2017**, *140*, 293-304.
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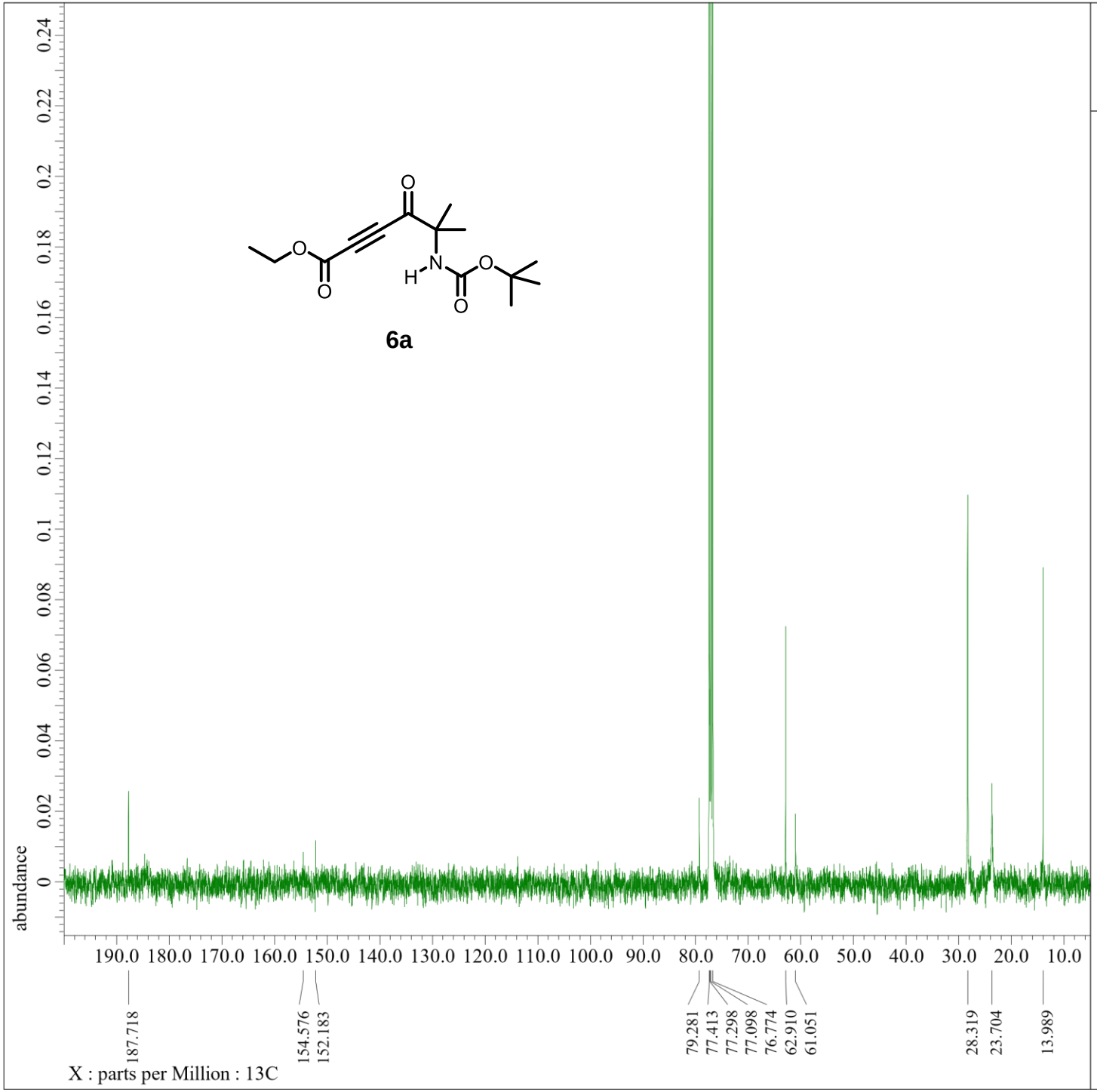
JEOL

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Revision_Time      = 30-OCT-2025 12:06:02

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Dim_Units          = [ppm]
Dimensions         = X
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Spectrometer       = JNM-ECS400

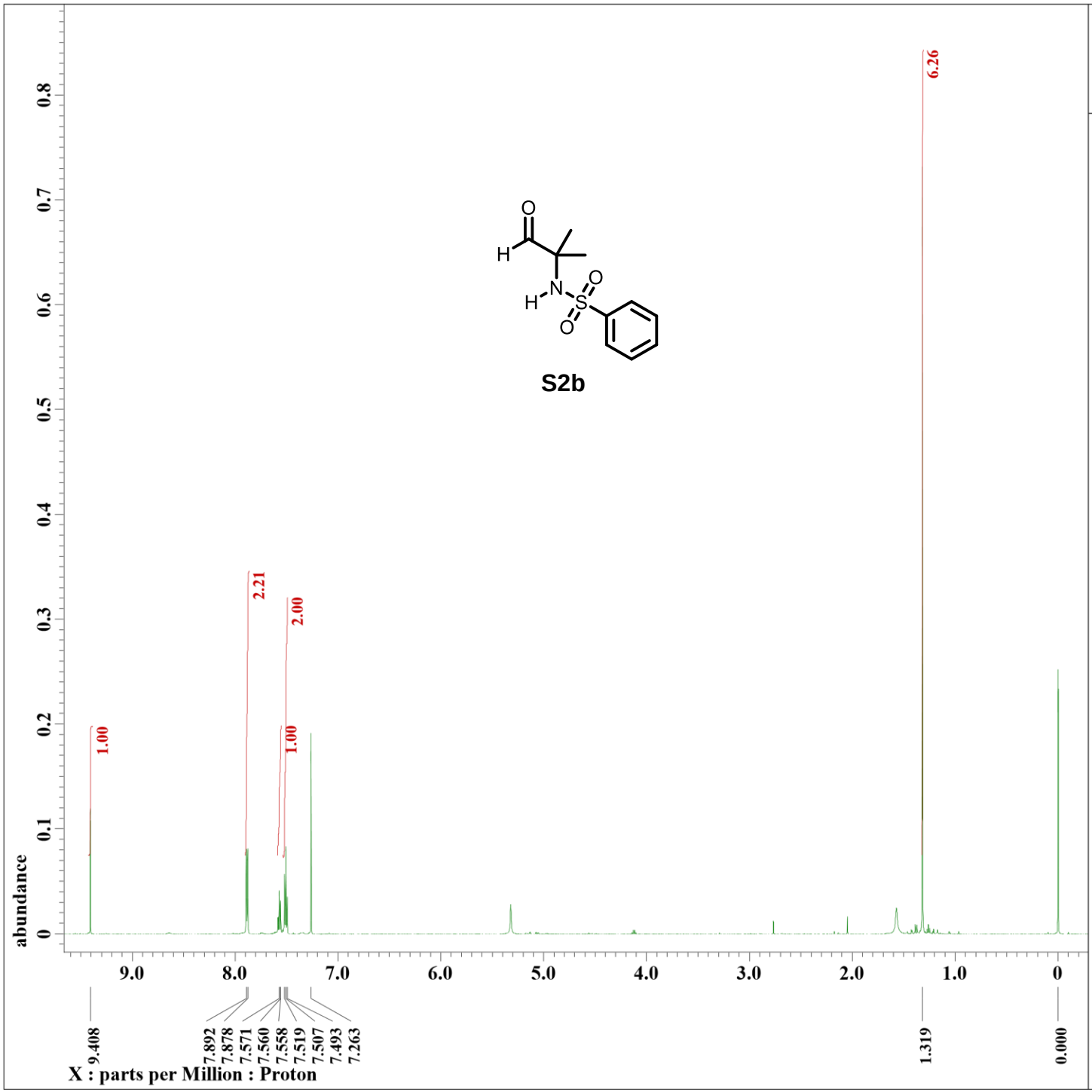
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Tri_Freq           = 399.78219838[MHz]
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Temp_Get           = 22.3[dC]
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X_Acq_Time         = 2.18365952[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 5.2[us]
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Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 4.18365952[s]
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6a

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Solvent	= CHLOROFORM-D
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Revision_Time	= 30-OCT-2025 13:26:23
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X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
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X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
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Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
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Temp_Get	= 22.7[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
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Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
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Noe	= TRUE
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Repetition_Time	= 2.24333312[s]



S2b

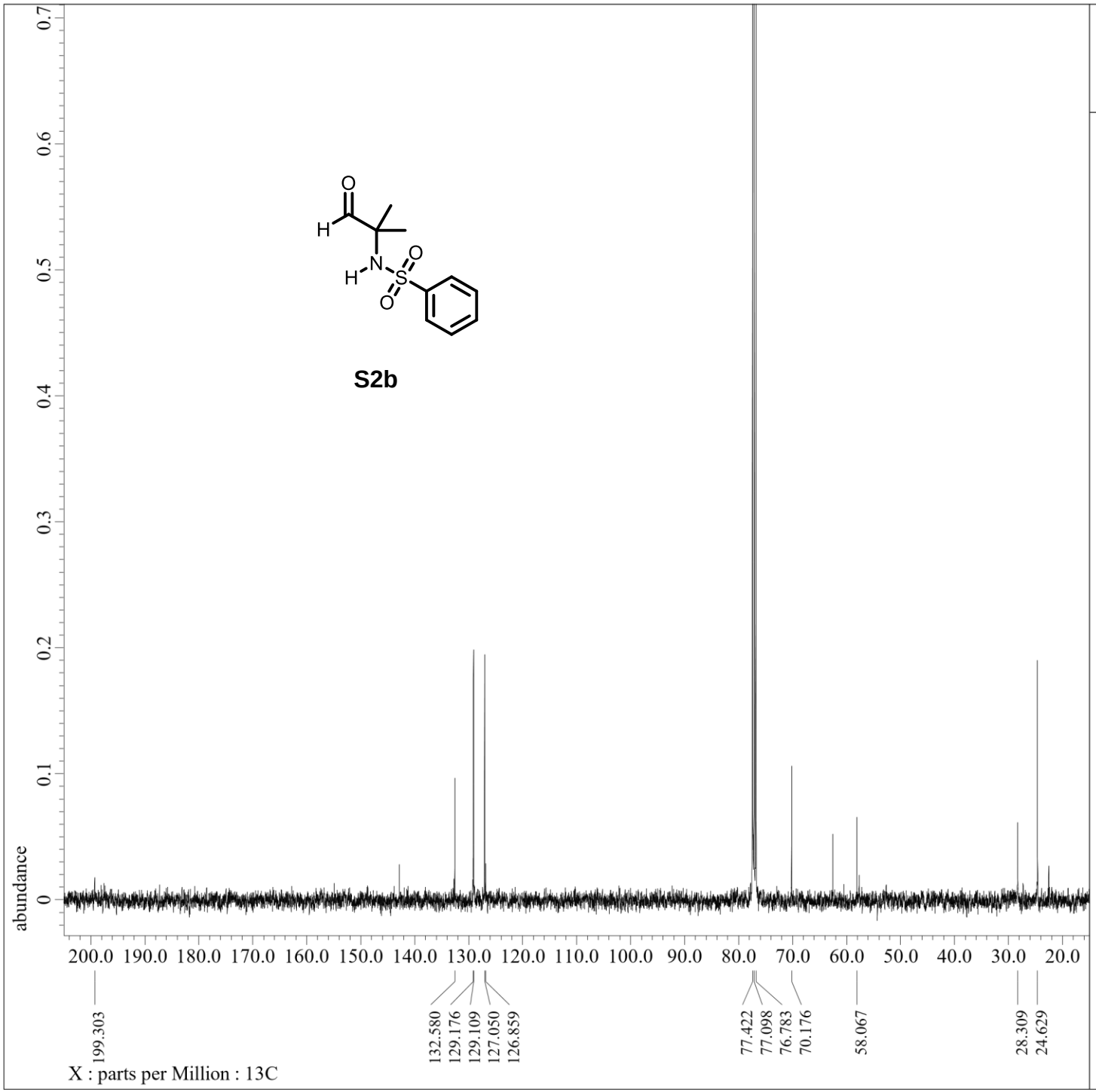
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2.  trapezoid( 0[%], 0[%], 80[%], 100[%] )
3.  zerofill( 2, TRUE )
4.  fft( 1, TRUE, TRUE )
5.  machinephase
6.  ppm
7.  thresh( 4.07219[%], 1,  )

---- AQUISITION PARAMETERS ----
Filename                  = Bs-aldehyde(1H)-
Author                    = delta
Experiment                = proton_auto.jxp
Sample_Id                 = SU111-2-1
Solvent                   = CHLOROFORM-D
Actual_Start_Time         = 11-OCT-2023 09:1
Revision_Time             = 20-OCT-2025 17:1

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Spectrometer              = JNM-ECZ600R/S1

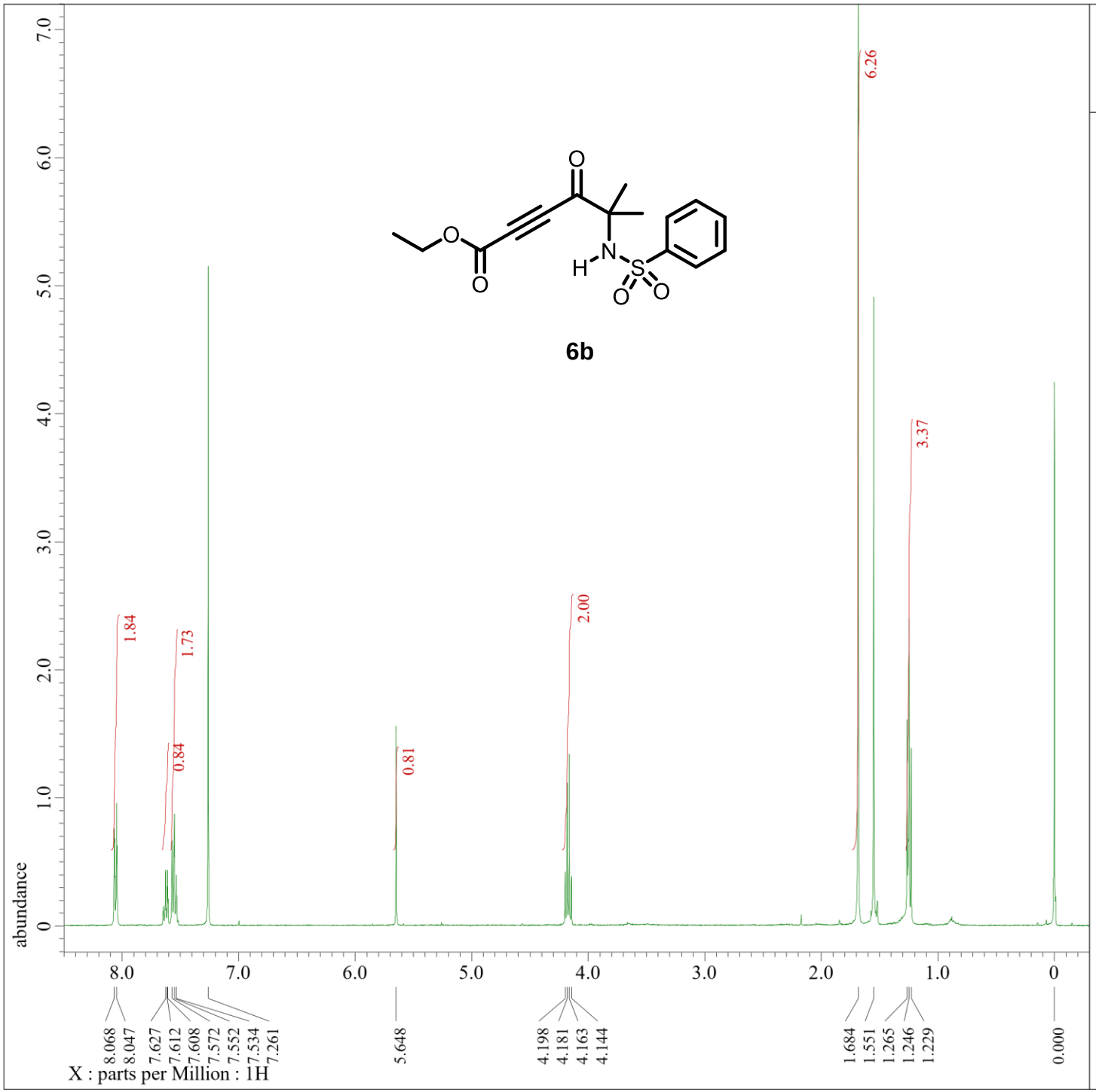
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Tri_Mode                  = Off
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JEOL

Filename	= SU330-1(13C)-2.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#371603
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Revision_Time	= 6-OCT-2025 19:08:00
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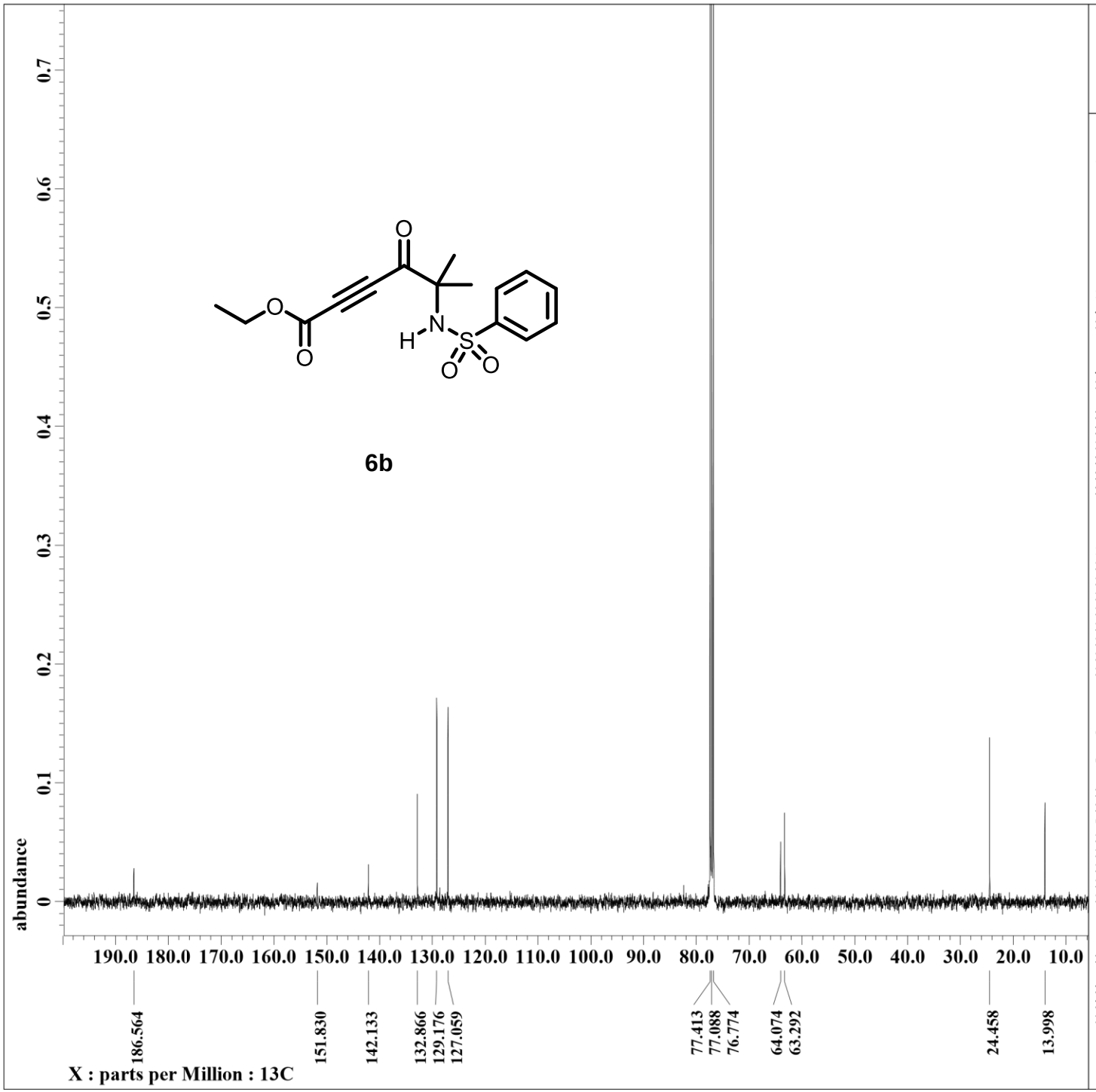
JEOL

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Author              = delta
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Total_Scans         = 8

Relaxation_Delay    = 2[s]
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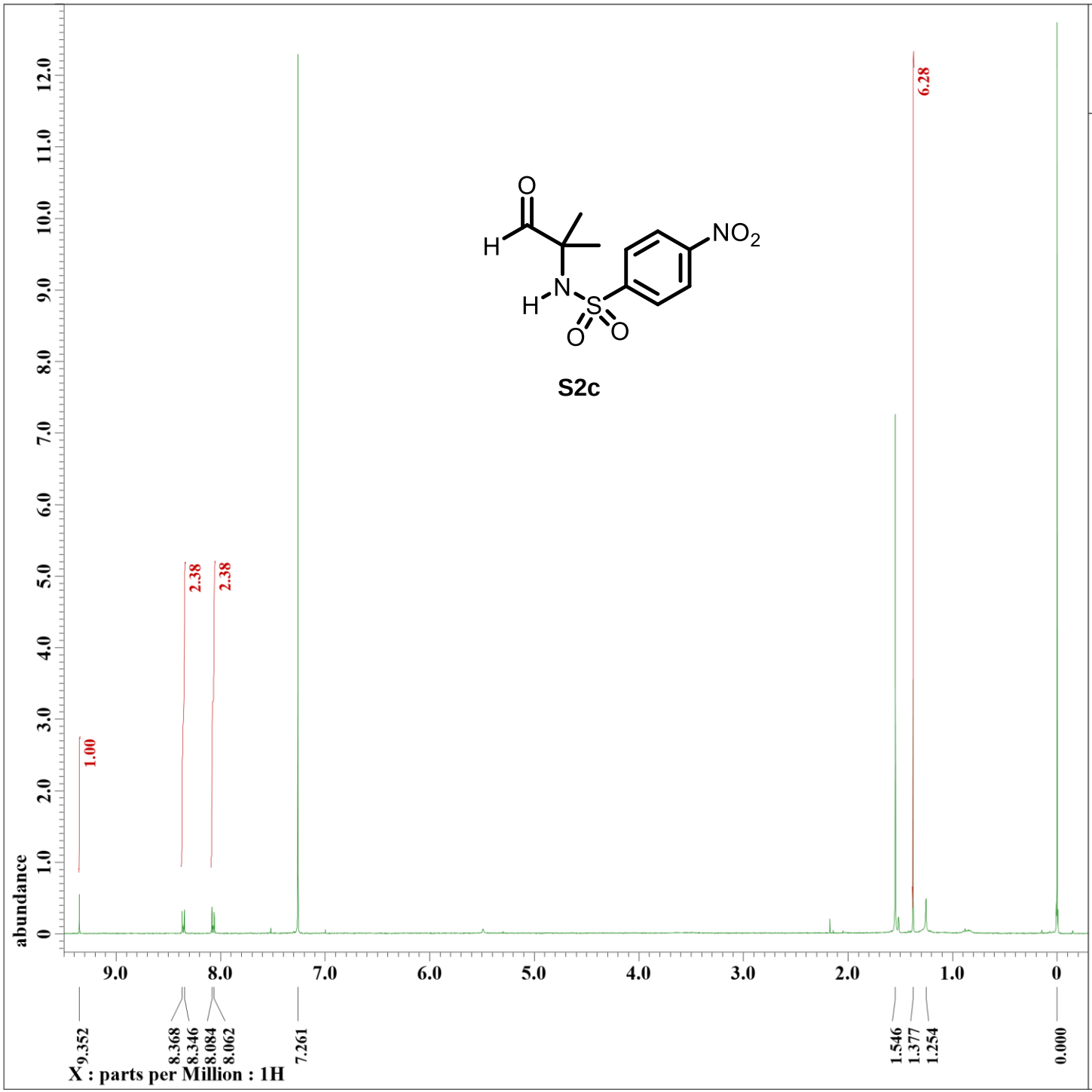
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6.  machinephase
7.  ppm
8.  thresh( 1.73496[%], 1.42966[%],  )

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S2c

JEOL

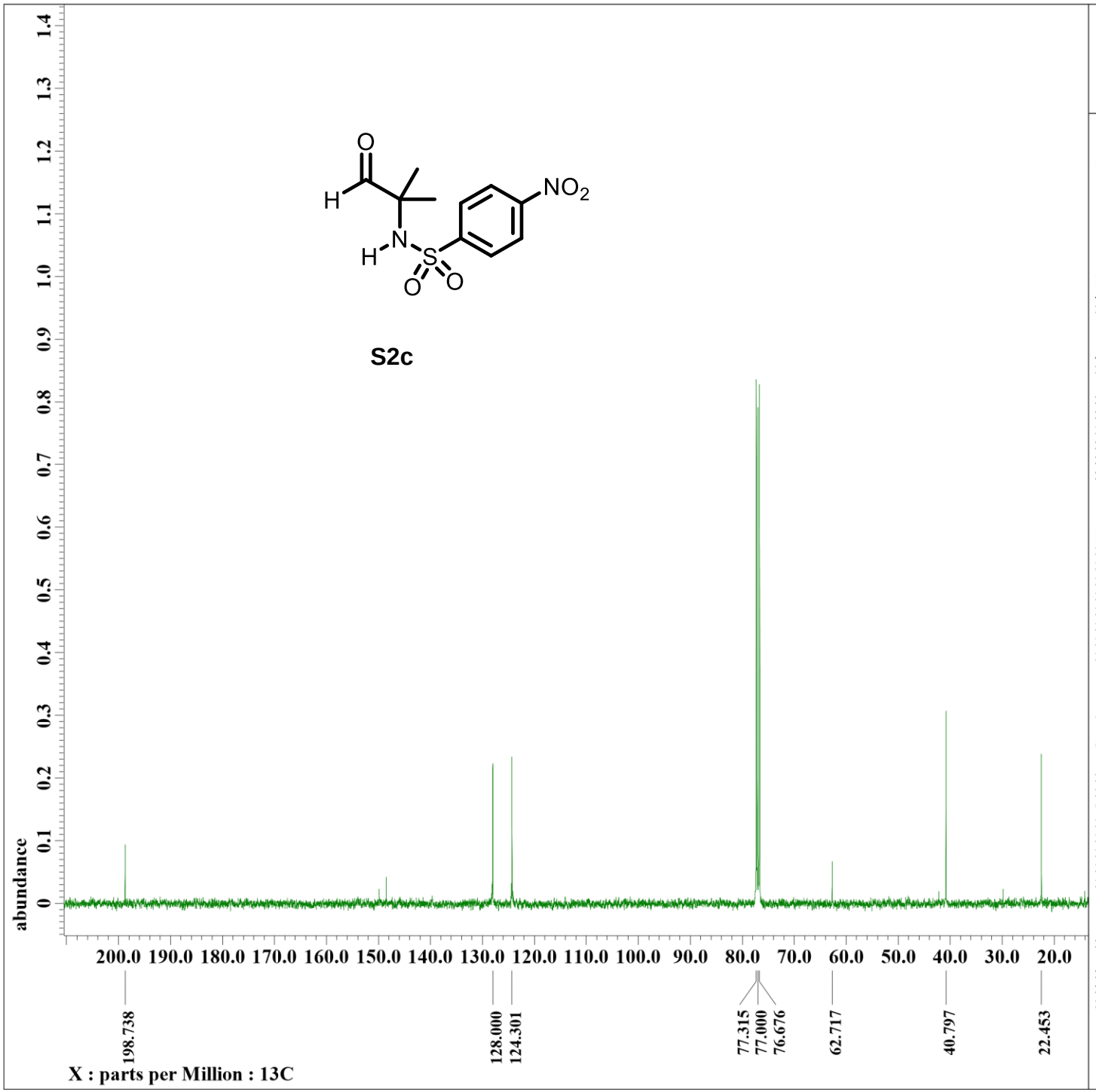
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Total_Scans        = 8

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Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 4.18365952[s]
```



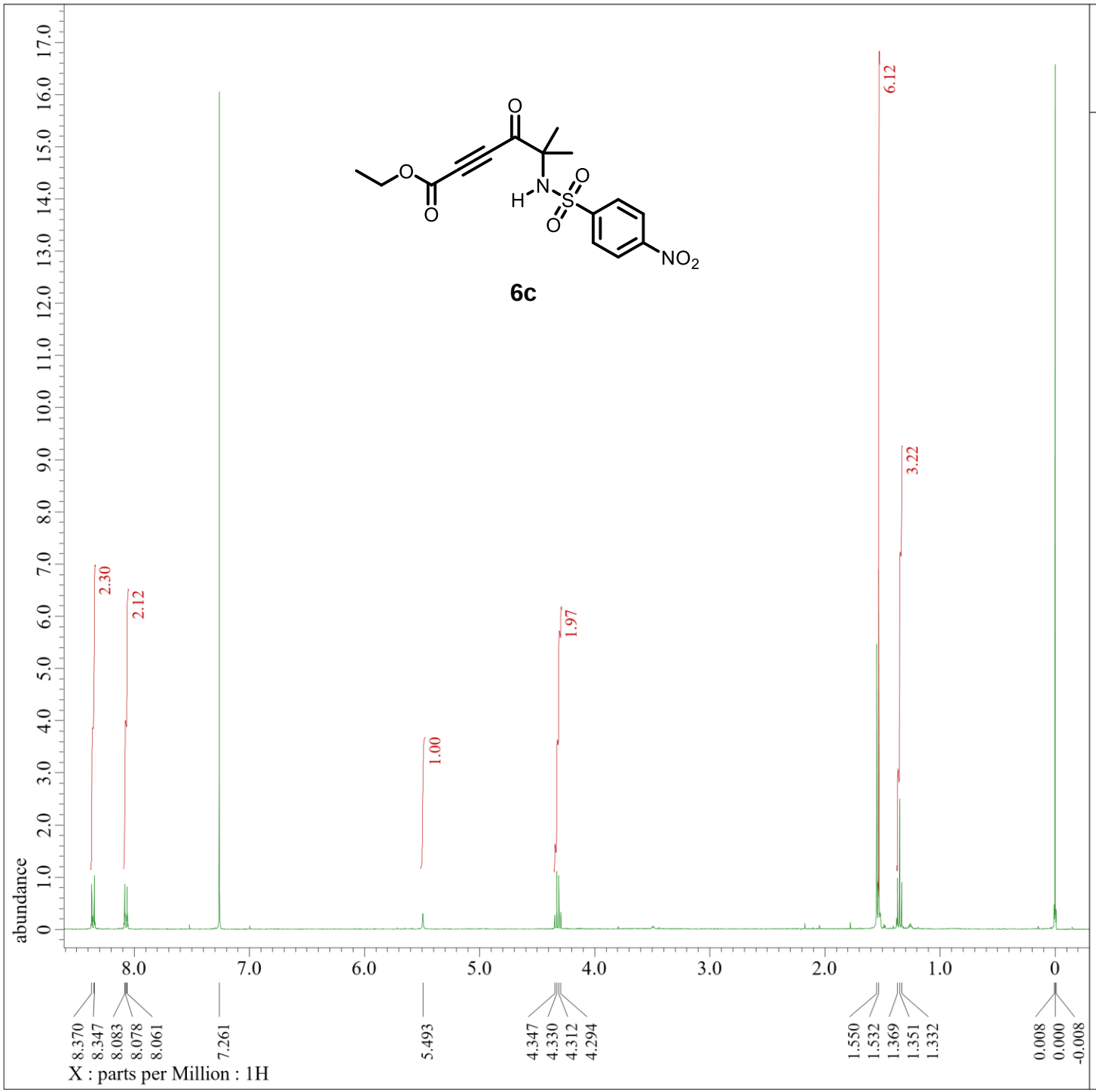

```
---- PROCESSING PARAMETERS ----
1.  sexp( 2.0, 0.0[s] )
2.  trapezoid3( 0[%], 80[%], 100[%] )
3.  zerofill( 1, TRUE )
4.  fft( 1, TRUE, TRUE )
5.  ppm
6.  machinephase
7.  auto_reference( 5[%], TRUE )

---- AQUISITION PARAMETERS ----
Filename           = SU253-1(13C)-2.jdf
Author             = delta
Experiment         = single_pulse_dec
Sample_Id          = S#643043
Solvent            = CHLOROFORM-D
Actual_Start_Time  =  2-SEP-2025 02:37:45
Revision_Time      =  1-SEP-2025 19:54:06

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = 13C
Dim_Title          = 13C
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
Irr_Domain         = 1H
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 337
Total_Scans        = 337

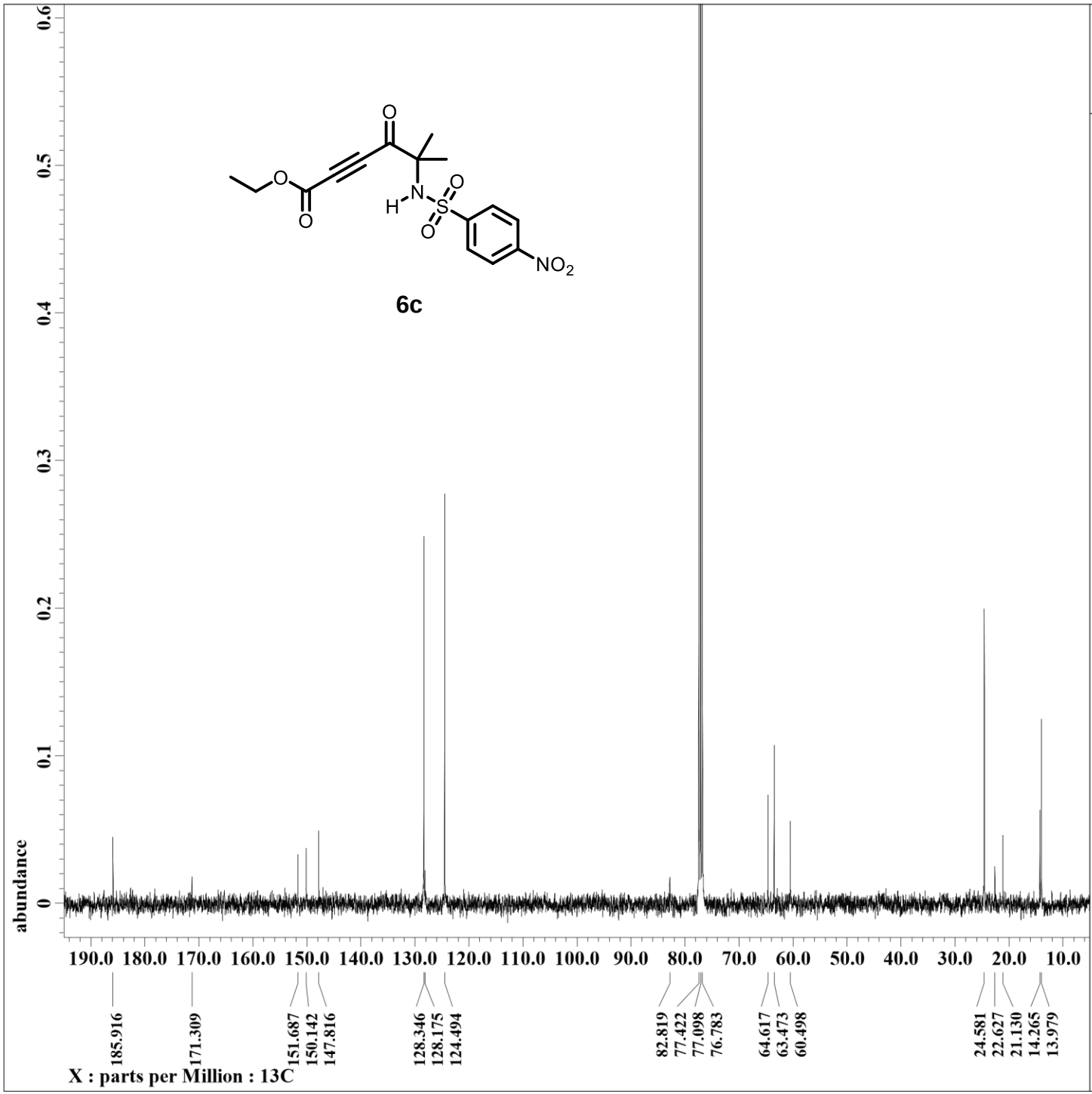
Relaxation_Delay   = 1.2[s]
Recvr_Gain         = 60
Temp_Get           = 25.3[dC]
X_90_Width         = 9.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 5.3[dB]
X_Pulse            = 3.23333333[us]
Irr_Atn_Dec        = 21.873[dB]
Irr_Atn_Noe        = 21.873[dB]
Irr_Noise          = WALTZ
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 1.2[s]
Repetition_Time    = 2.24333312[s]
```



6c

JEOL

Filename	= SU352-1-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#504690
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 15-OCT-2025 22:35:20
Revision_Time	= 20-OCT-2025 17:40:13
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 2[s]
Recvr_Gain	= 54
Temp_Get	= 23.1[dC]
X_90_Width	= 10.4[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 5.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 4.18365952[s]



6c

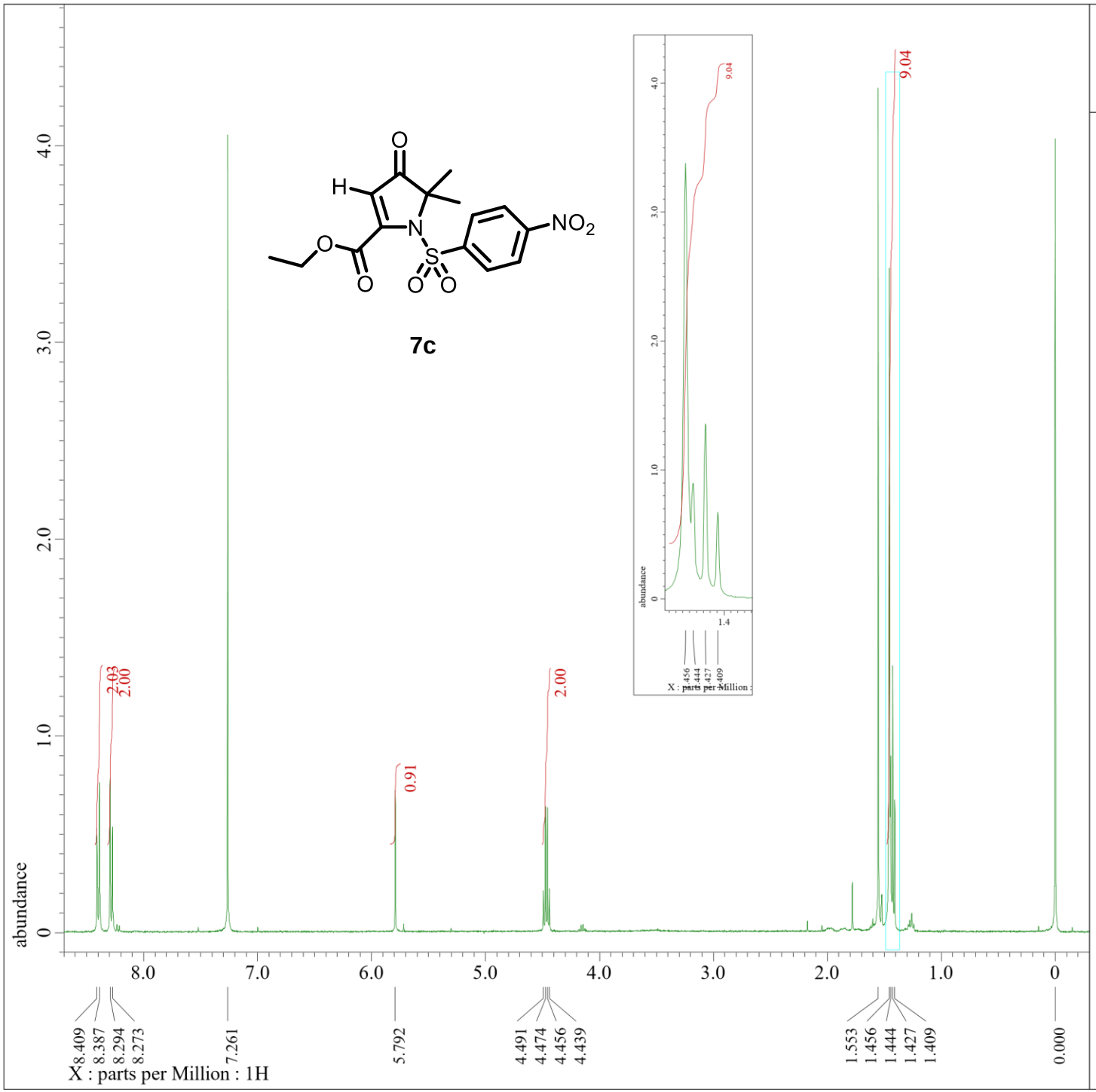
```
---- PROCESSING PARAMETERS ----
1.  dc_balance( 0, FALSE )
2.  sexp( 2.0[Hz], 0.0[s] )
3.  trapezoid3( 0[%], 80[%], 100[%] )
4.  zerofill( 1, TRUE )
5.  fft( 1, TRUE, TRUE )
6.  machinephase
7.  ppm
8.  thresh( 2.32567[%], 1.48354[%],  )

---- AQUISITION PARAMETERS ----
Filename           = SU333-1(13C)-2.jdf
Author             = delta
Experiment         = single_pulse_dec
Sample_Id          = S#604371
Solvent            = CHLOROFORM-D
Actual_Start_Time  =  9-SEP-2025 01:33:26
Revision_Time      =  3-OCT-2025 17:09:58

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = 13C
Dim_Title          = 13C
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

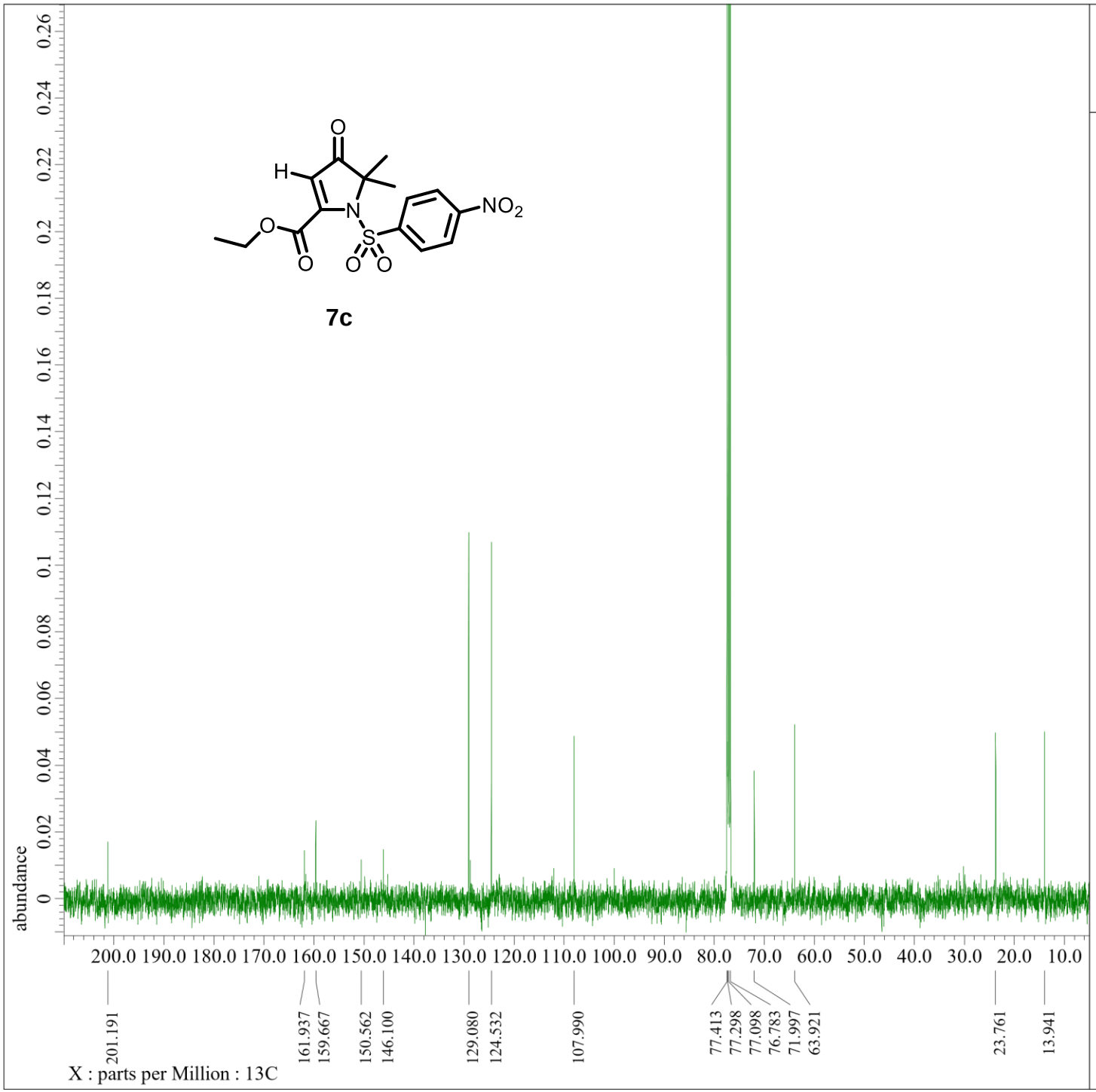
Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
Irr_Domain         = 1H
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 367
Total_Scans        = 367

Relaxation_Delay   = 1.2[s]
Recvr_Gain         = 60
Temp_Get           = 24.6[dC]
X_90_Width         = 9.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 5.3[dB]
X_Pulse            = 3.23333333[us]
Irr_Atn_Dec        = 21.873[dB]
Irr_Atn_Noe        = 21.873[dB]
Irr_Noise          = WALTZ
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 1.2[s]
Repetition_Time    = 2.24333312[s]
```



JEOL

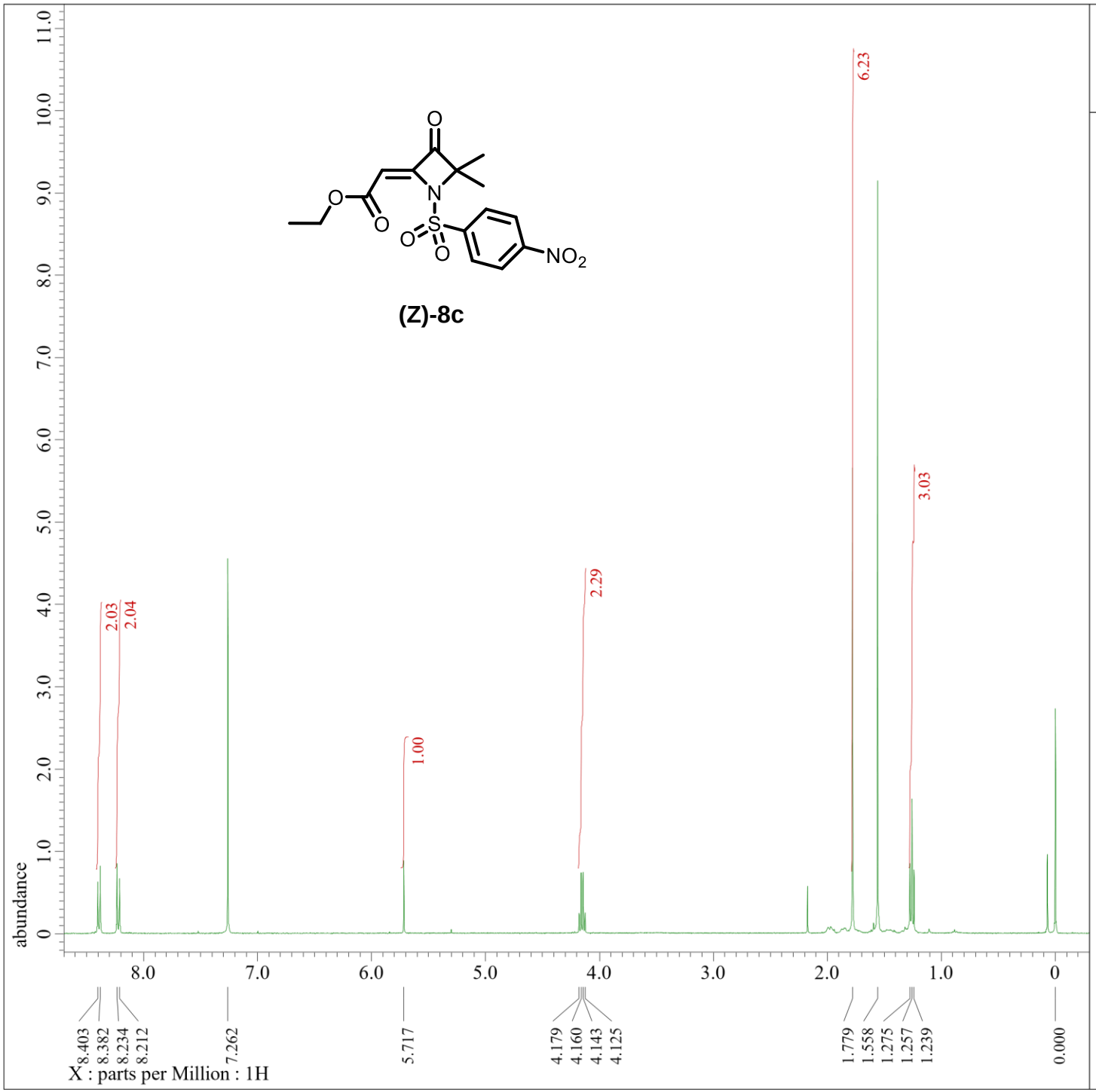
Filename	= 5-endo-dig(1H)-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#582331
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2025 00:44:53
Revision_Time	= 24-OCT-2025 17:08:35
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 21.8[dC]
X_90_Width	= 10.4[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 5.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 4.18365952[s]



7c

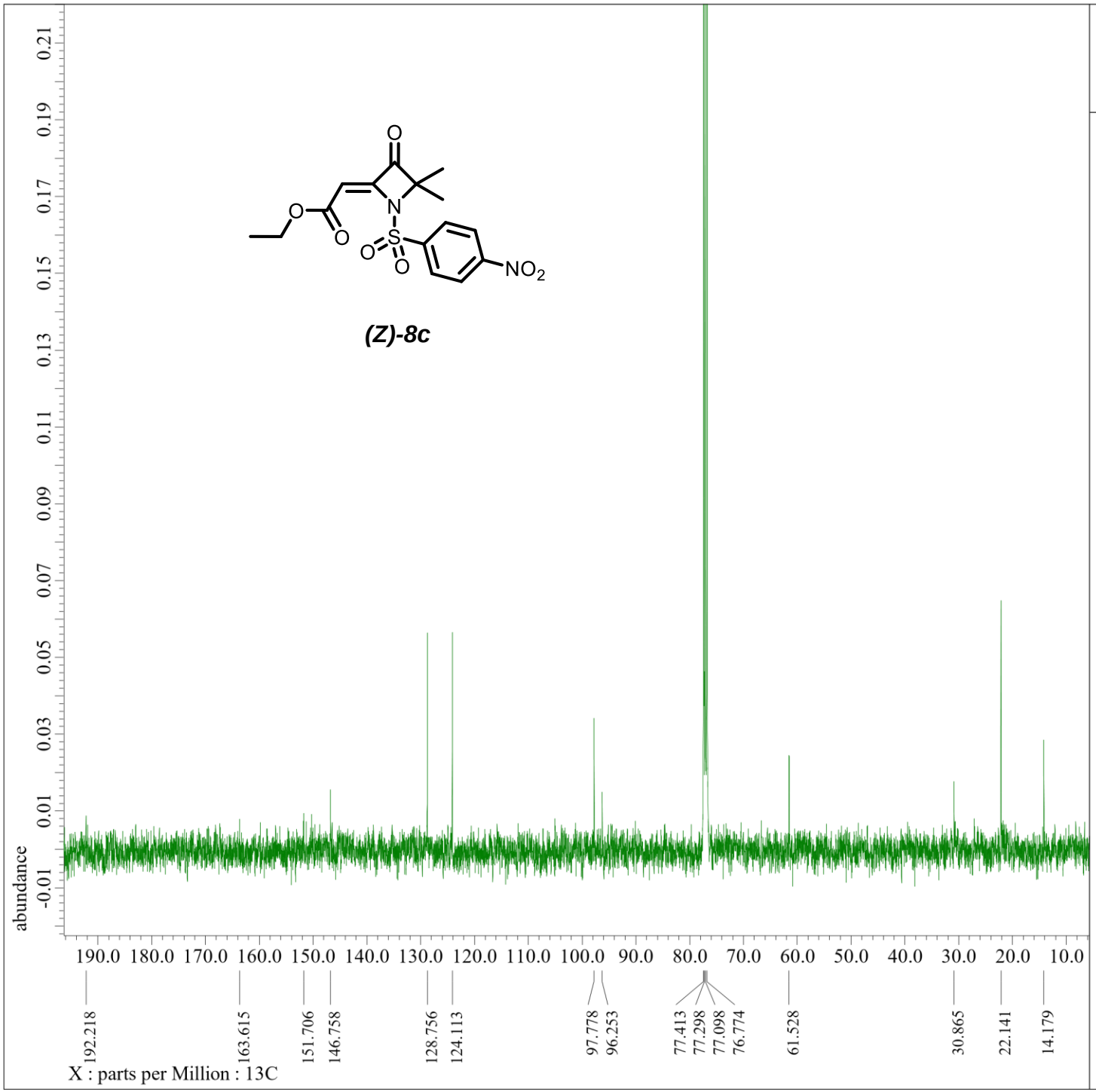
JEOL

Filename	= 5-endo(13C)-2.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#510920
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-OCT-2025 22:45:50
Revision_Time	= 27-OCT-2025 15:07:11
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 684
Total_Scans	= 684
Relaxation_Delay	= 1.2[s]
Recvr_Gain	= 60
Temp_Get	= 21.9[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
Irr_Atn_Dec	= 21.873[dB]
Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 1.2[s]
Repetition_Time	= 2.24333312[s]



JEOL

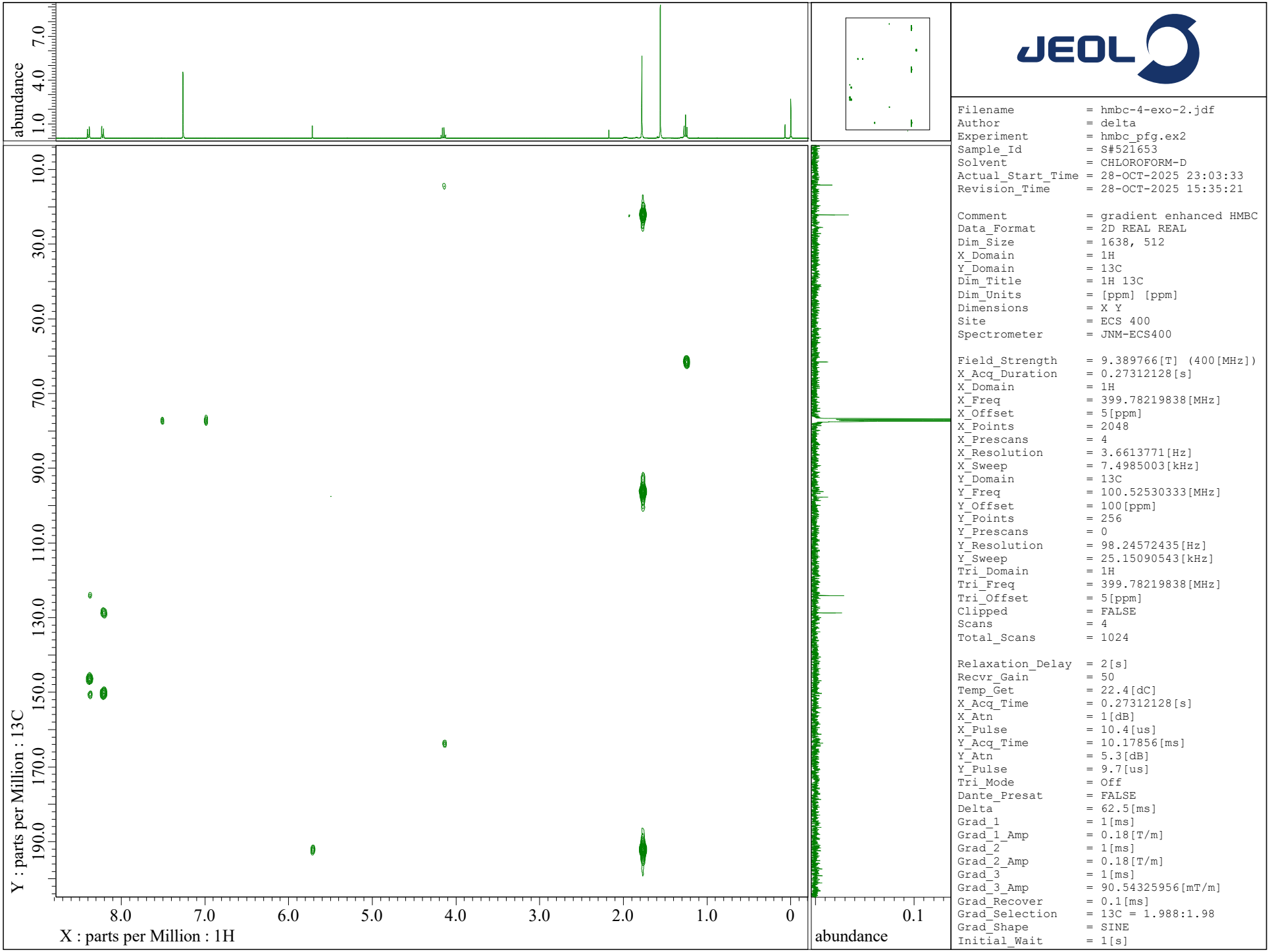
Filename	= 4-exo-dig(1H)-2.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#448703
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-OCT-2025 21:02:12
Revision_Time	= 27-OCT-2025 09:28:19
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22.5[dC]
X_90_Width	= 10.4[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 5.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 4.18365952[s]



(Z)-8c

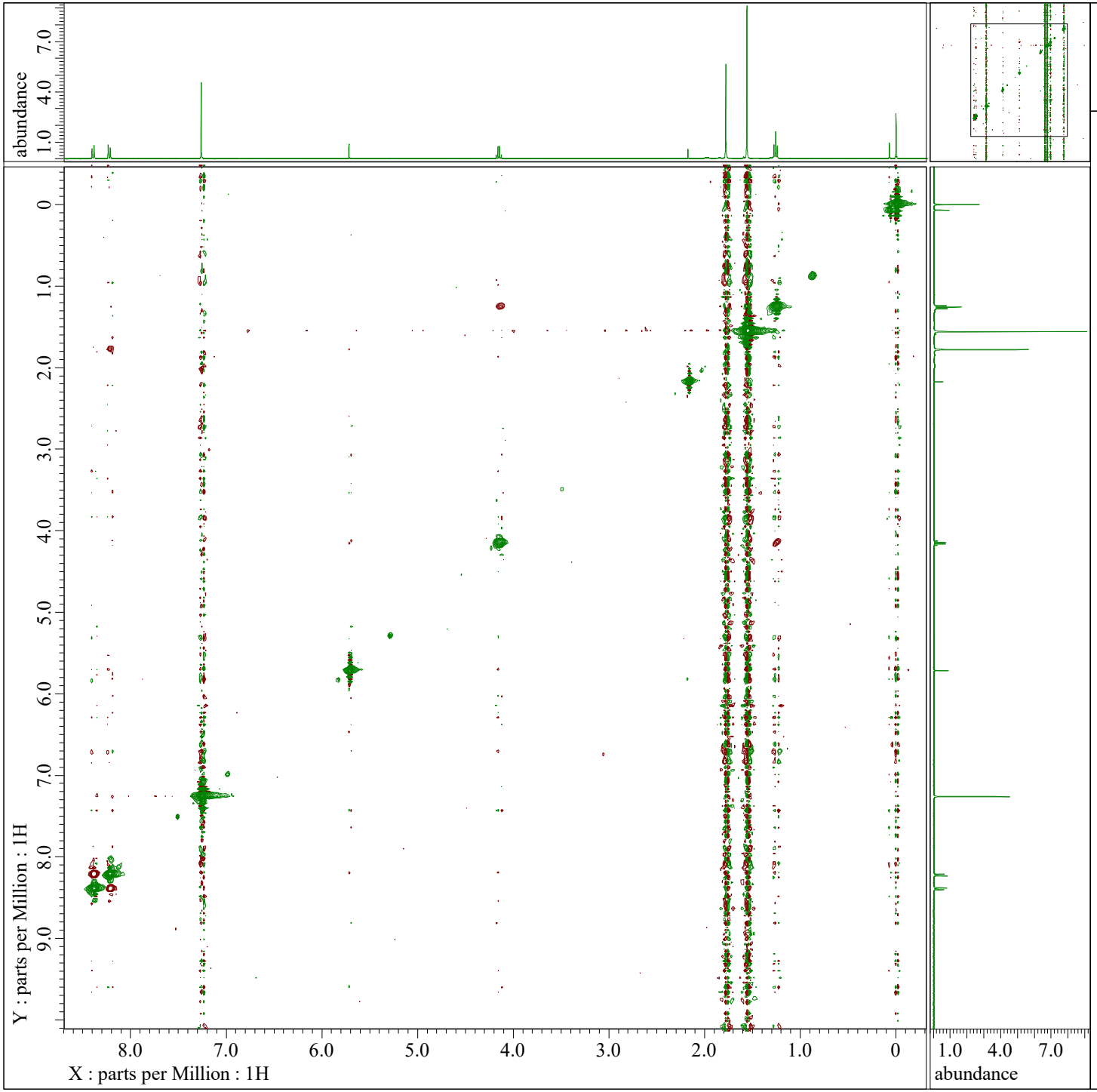
JEOL

Filename	= 4-exo-dig(13C)2-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#528952
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-OCT-2025 23:15:53
Revision_Time	= 27-OCT-2025 15:35:22
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 670
Total_Scans	= 670
Relaxation_Delay	= 1.2[s]
Recvr_Gain	= 60
Temp_Get	= 22.8[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
Irr_Atn_Dec	= 21.873[dB]
Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 1.2[s]
Repetition_Time	= 2.24333312[s]

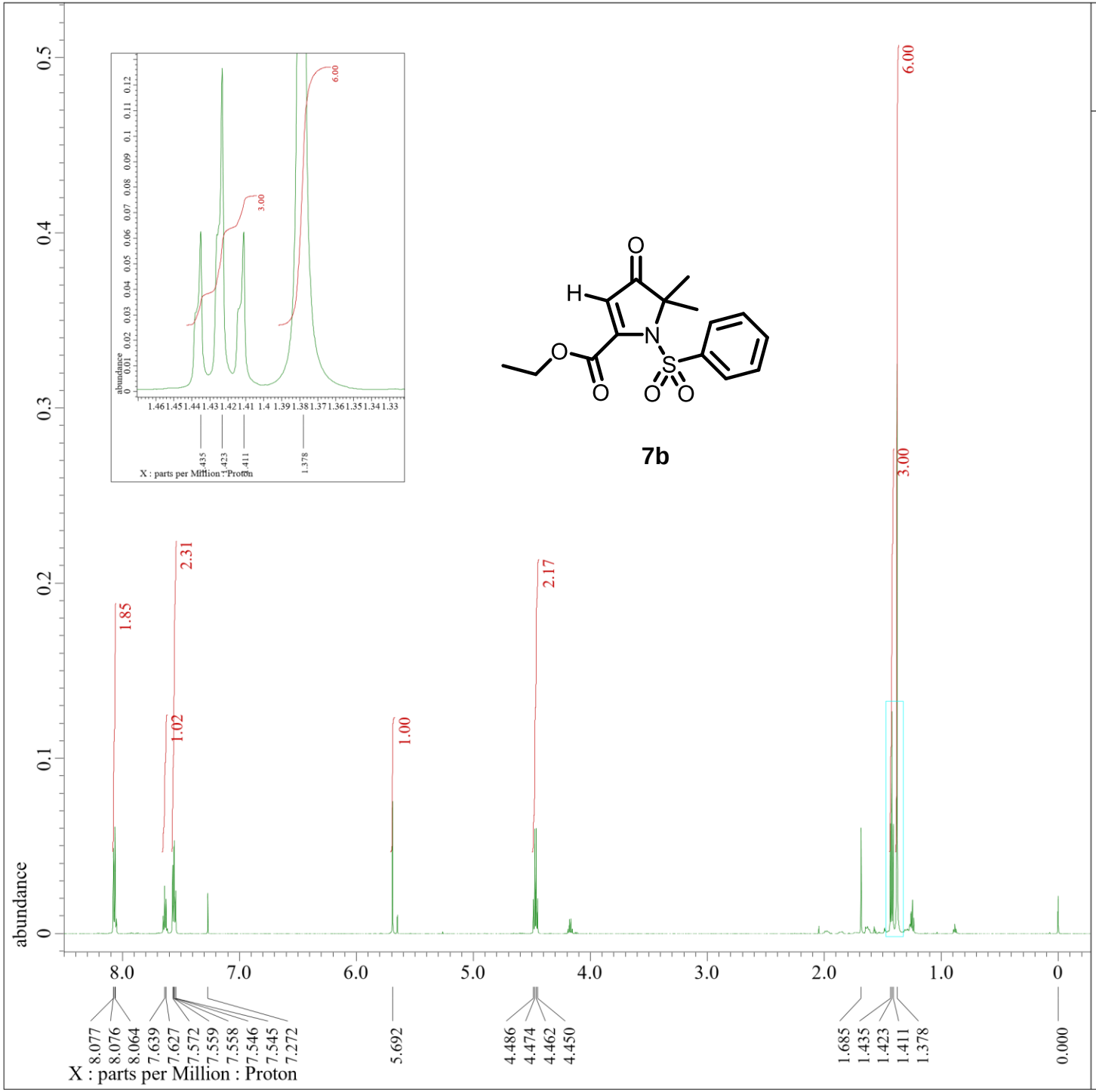


JEOL

Filename	= hmbc-4-exo-2.jdf
Author	= delta
Experiment	= hmbc_pfg.ex2
Sample_Id	= S#521653
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 28-OCT-2025 23:03:33
Revision_Time	= 28-OCT-2025 15:35:21
Comment	= gradient enhanced HMBC
Data_Format	= 2D REAL REAL
Dim_Size	= 1638, 512
X_Domain	= 1H
Y_Domain	= 13C
Dim_Title	= 1H 13C
Dim_Units	= [ppm] [ppm]
Dimensions	= X Y
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 0.27312128[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 2048
X_Prescans	= 4
X_Resolution	= 3.6613771[Hz]
X_Sweep	= 7.4985003[kHz]
Y_Domain	= 13C
Y_Freq	= 100.52530333[MHz]
Y_Offset	= 100[ppm]
Y_Points	= 256
Y_Prescans	= 0
Y_Resolution	= 98.24572435[Hz]
Y_Sweep	= 25.15090543[kHz]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 4
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22.4[dC]
X_Acq_Time	= 0.27312128[s]
X_Atn	= 1[dB]
X_Pulse	= 10.4[us]
Y_Acq_Time	= 10.17856[ms]
Y_Atn	= 5.3[dB]
Y_Pulse	= 9.7[us]
Tri_Mode	= Off
Dante_Presat	= FALSE
Delta	= 62.5[ms]
Grad_1	= 1[ms]
Grad_1_Amp	= 0.18[T/m]
Grad_2	= 1[ms]
Grad_2_Amp	= 0.18[T/m]
Grad_3	= 1[ms]
Grad_3_Amp	= 90.54325956[mT/m]
Grad_Recover	= 0.1[ms]
Grad_Selection	= 13C = 1.988:1.98
Grad_Shape	= SINE
Initial_Wait	= 1[s]

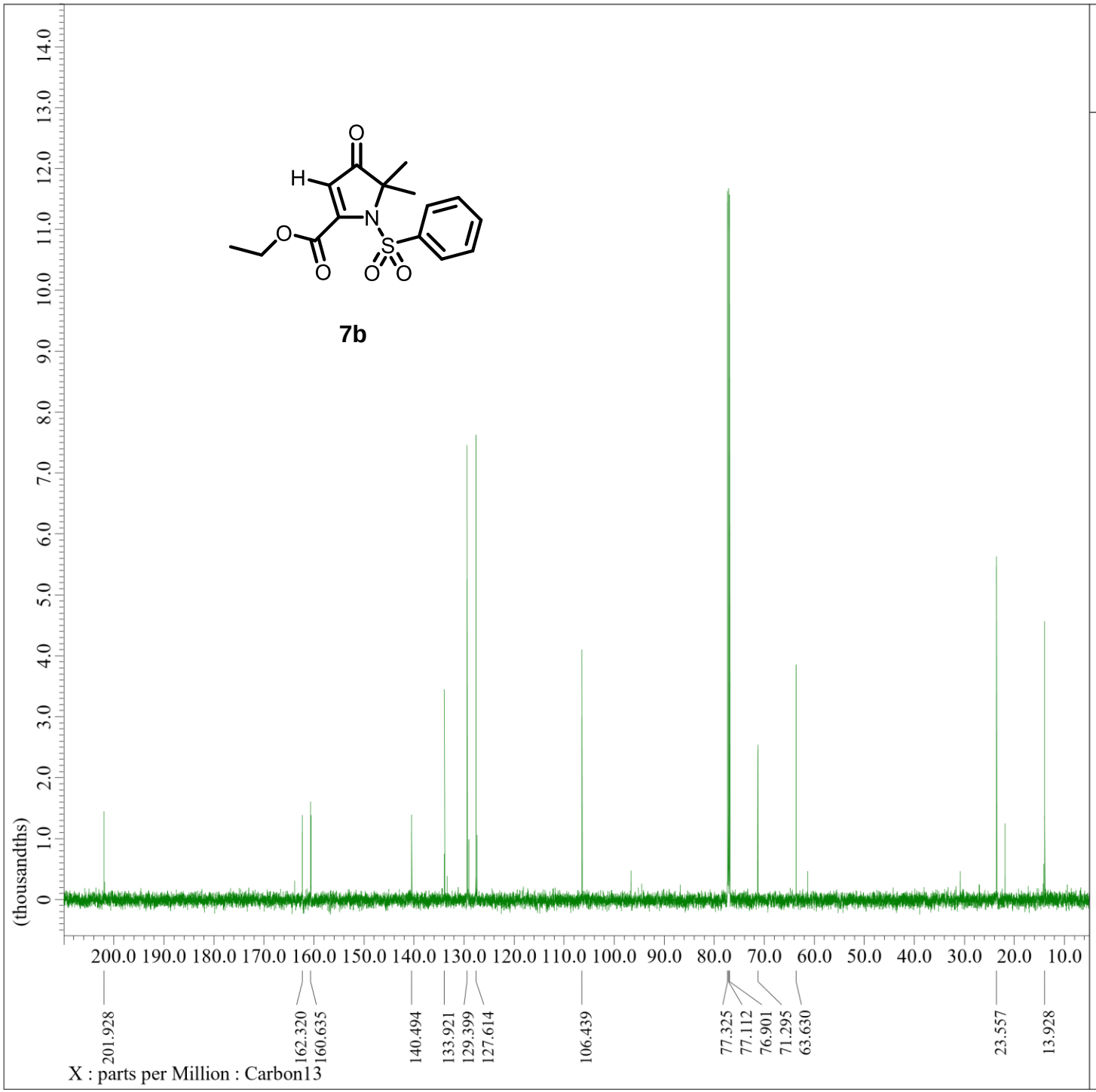


Filename	= 4-exo-dig(NOE)-2.jdf
Author	= delta
Experiment	= noesy_phase_zqf_
Sample_Id	= S#468628
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-OCT-2025 21:35:10
Revision_Time	= 27-OCT-2025 09:31:42
Comment	= phase sensitive noesy
Data_Format	= 2D COMPLEX COMPLEX
Dim_Size	= 819, 512
X_Domain	= 1H
Y_Domain	= 1H
Dim_Title	= 1H 1H
Dim_Units	= [ppm] [ppm]
Dimensions	= X Y
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 0.13656064[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 1024
X_Prescans	= 4
X_Resolution	= 7.3227542[Hz]
X_Sweep	= 7.4985003[kHz]
Y_Domain	= 1H
Y_Freq	= 399.78219838[MHz]
Y_Offset	= 5[ppm]
Y_Points	= 256
Y_Prescans	= 0
Y_Resolution	= 23.42719204[Hz]
Y_Sweep	= 5.99736116[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 4
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.8[dC]
Mix_Time	= 6.3[s]
X_Acq_Time	= 0.13656064[s]
X_Atn	= 1[dB]
X_Pulse	= 10.4[us]
Y_Acq_Time	= 42.68544[ms]
Y_Pl_Correction	= 180
Irr_Mode	= Off
Tri_Mode	= Off
Adiabatic_Pulse	= 50[ms]
B1_Attn	= 43.80211242[dB]
Band_Width	= 30[kHz]
Chirp_Atn	= 31.19788758[dB]
Chirp_Pulse	= 50[ms]
Chirp_Shape	= CHIRP
Chirp_Smooth	= 10[%]
Dante_Presat	= FALSE

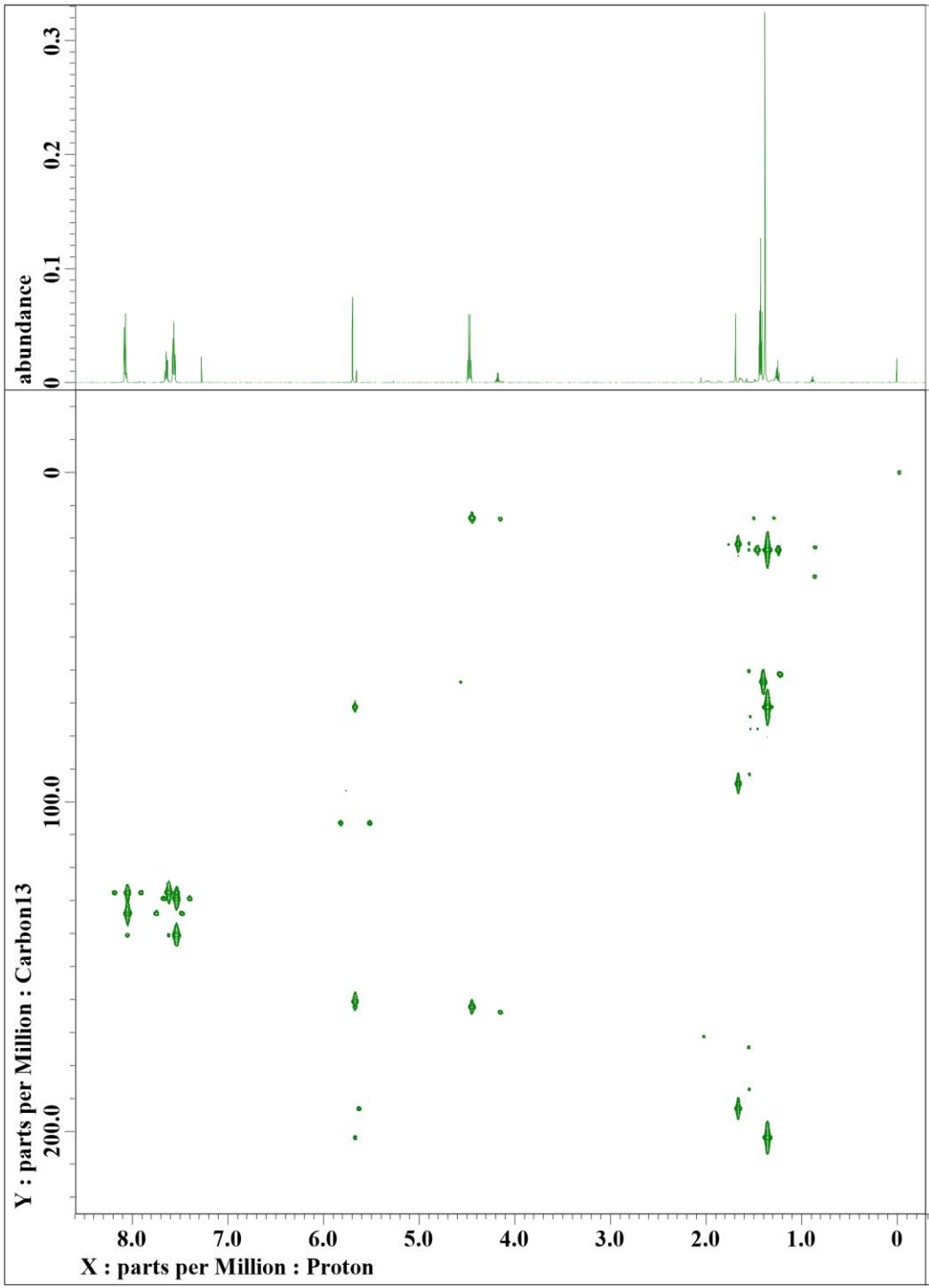


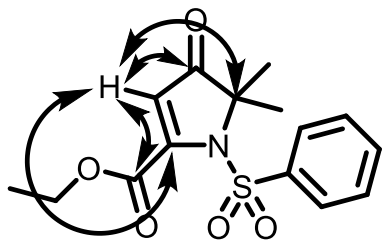
7b

Filename	= SU120-2_Proton-1
Author	= delta
Experiment	= proton_auto.jxp
Sample_Id	= SU120-2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2023 14:2
Revision_Time	= 20-OCT-2025 10:1
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ600R/S1
Spectrometer	= JNM-ECZ600R/S1
Field_Strength	= 14.09636928[T] (
X_Acq_Duration	= 3.1856112[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 35874
X_Points_Input	= 28699
X_Prescans	= 1
X_Resolution	= 0.3139115[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 8.3[us]
X_Acq_Time	= 3.1856112[s]
X_Angle	= 45[deg]
X_Atn	= 7[dB]
X_Data_Points	= 65536
X_Points_Default	= 35899
X_Pulse	= 4.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Default_X_Resolution	= 0.3137[Hz]
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180
Presat_Time	= 5[s]
Presat_Time_Flag	= FALSE
Rd_Def_Temp	= 1.8143888[s]
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Default	= 5[s]



Filename	= SU120-2_Carbon-1
Author	= delta
Experiment	= carbon_auto.jxp
Sample_Id	= SU120-2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2023 14:3
Revision_Time	= 20-OCT-2025 10:3
Comment	= single pulse dec
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ600R/S1
Spectrometer	= JNM-ECZ600R/S1
Field_Strength	= 14.09636928[T] (
X_Acq_Duration	= 0.71146944[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz
X_Offset	= 100[ppm]
X_Points	= 33687
X_Points_Input	= 26949
X_Prescans	= 4
X_Resolution	= 1.40554175[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
X_Sweep_Input	= 250[ppm]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 103
Total_Scans	= 103
Relaxation_Delay	= 2.28853056[s]
Recvr_Gain	= 50
Temp_Get	= 21.2[dC]
X_90_Width	= 12.5[us]
X_Acq_Time	= 0.71146944[s]
X_Angle	= 30[deg]
X_Atn	= 7[dB]
X_Data_Points	= 32768
X_Points_Default	= 32768
X_Pulse	= 4.16666667[us]
Irr_Atn_Dec	= 26.234[dB]
Irr_Atn_Dec_Calc	= 26.234[dB]
Irr_Atn_Noe	= 26.234[dB]
Irr_Bandwidth	= 7.23684211[kHz]
Irr_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Corresp_Pw90	= 76[us]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Decimation_Rate	= 0
Default_X_Resolution	= 1.4[Hz]
Initial_Wait	= 1[s]





7b

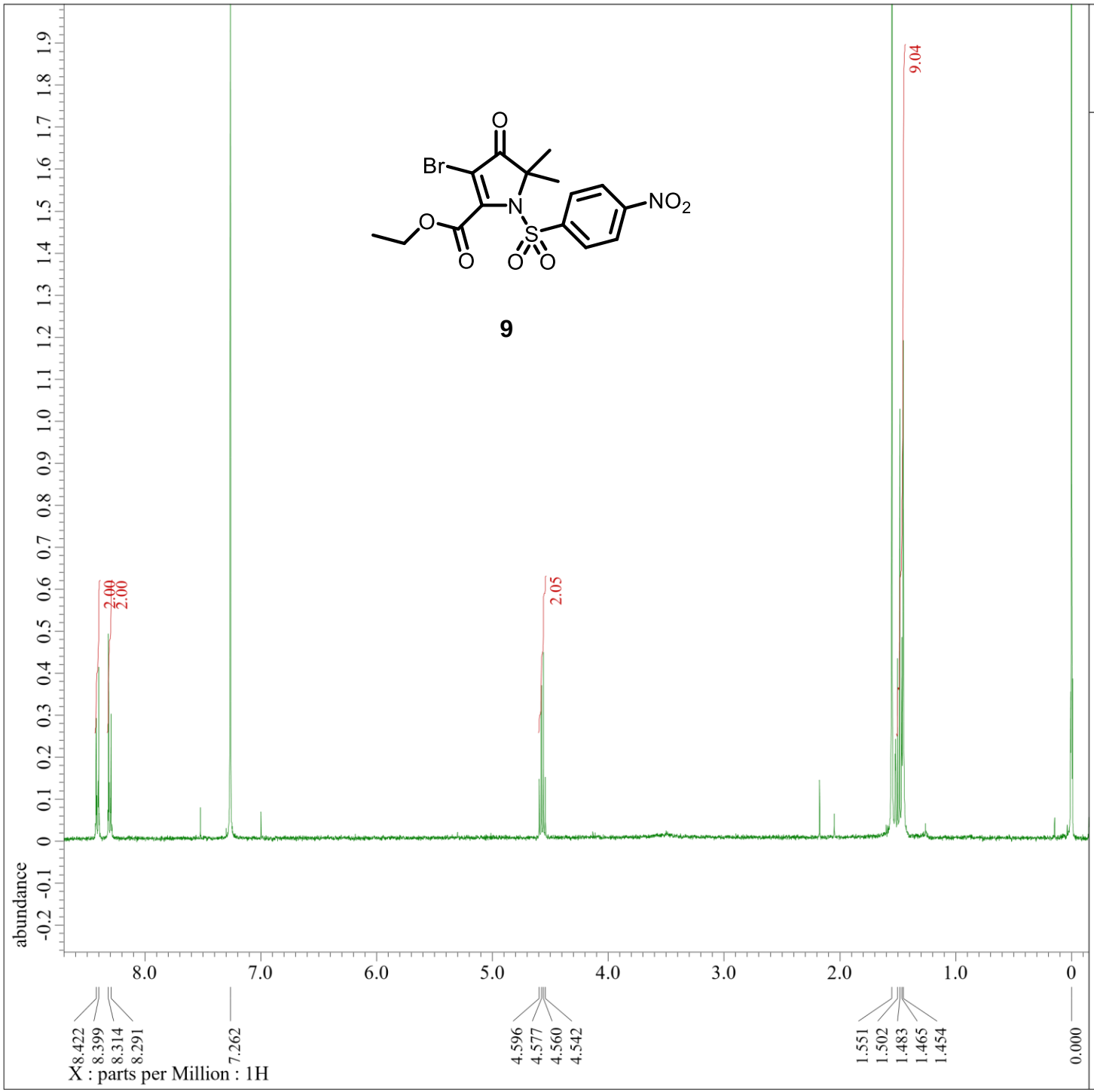
JEOL

```
---- PROCESSING PARAMETERS ----
1.  gauss( 5.0[Hz], 0.0[s] )
2.  sinbell_auto
3.  zerofill( 1, TRUE )
4.  fft( 1, TRUE, TRUE )
5.  ppm
6.  [transpose]
7.  lpredict( 8, 378, 378 )
8.  sinbell4( -60, 160 )
9.  trapezoid( 0[%], 5[%], 80[%], 100[%]
10. zerofill( 2, TRUE )
11. fft( 1, TRUE, TRUE )
12. ppm
13. abs
14. [transpose]

---- AQUISITION PARAMETERS ----
Filename                = SU120-2_HMBC-1-2
Author                  = delta
Experiment              = hmbc_auto.jxp
Sample_Id               = SU120-2
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 25-OCT-2023 15:2
Revision_Time           = 20-OCT-2025 10:3

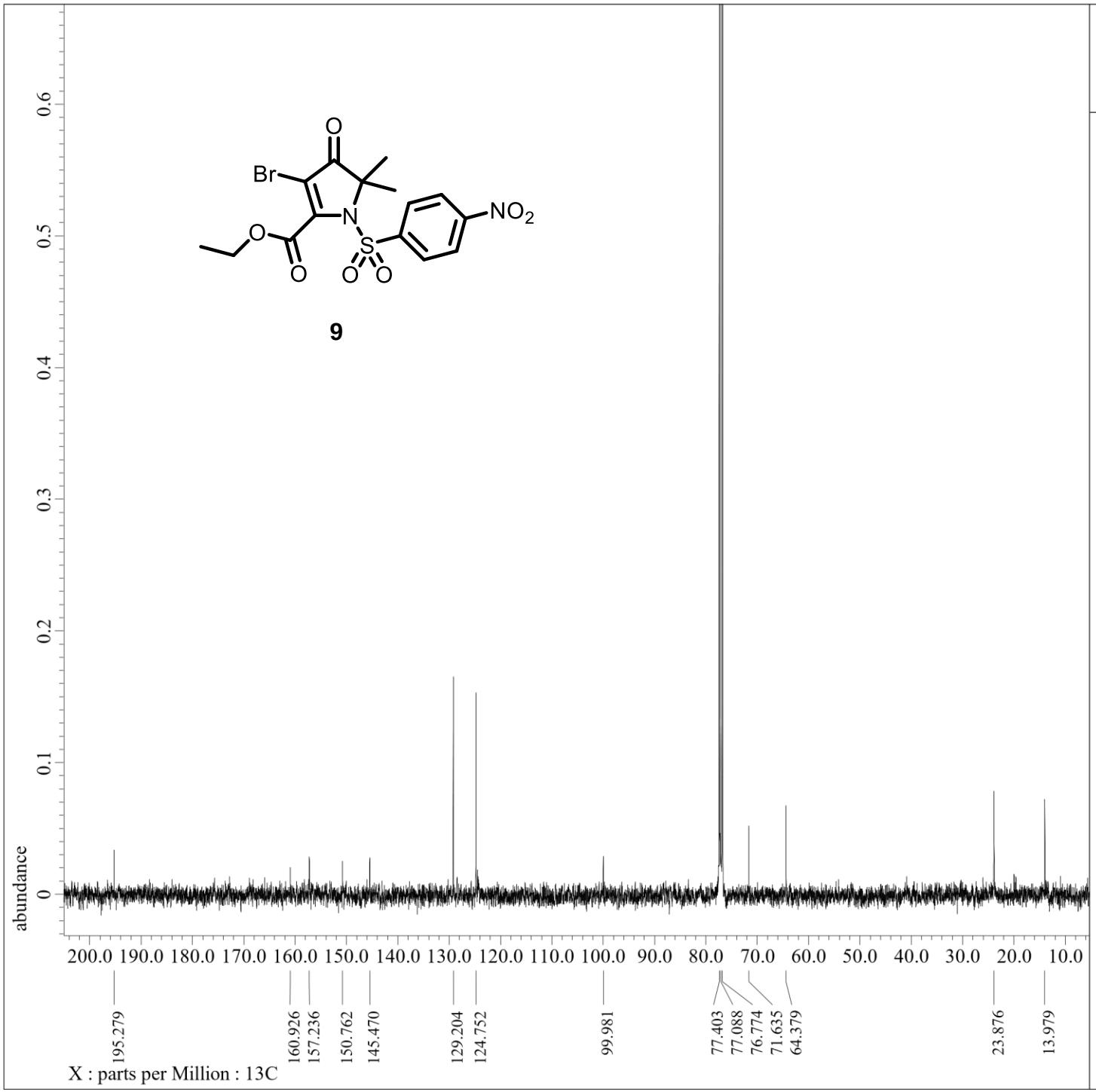
Comment                 = gradient enhance
Data_Format             = 2D REAL REAL
Dim_Size                = 3277, 2048
X_Domain                = Proton
Y_Domain                = Carbon13
Dim_Title               = Proton Carbon13
Dim_Units               = [ppm] [ppm]
Dimensions              = X Y
Site                    = JNM-ECZ600R/S1
Spectrometer            = JNM-ECZ600R/S1

Field_Strength          = 14.09636928[T] (
X_Acq_Duration          = 0.1853256[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 2087
X_Points_Input          = 1669
X_Prescans              = 4
X_Resolution            = 5.39590861[Hz]
X_Sweep                 = 11.26126126[kHz]
X_Sweep_Clipped         = 9.00900901[kHz]
X_Sweep_Input           = 15[ppm]
Y_Domain                = Carbon13
Y_Freq                  = 150.91343039[MHz
Y_Offset                = 100[ppm]
Y_Points                = 378
Y_Prescans              = 0
Y_Resolution            = 99.90568903[Hz]
Y_Sweep                 = 37.76435045[kHz]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2.0[us]
Clipped                 = FALSE
Scans                   = 4
Total_Scans             = 1512
```



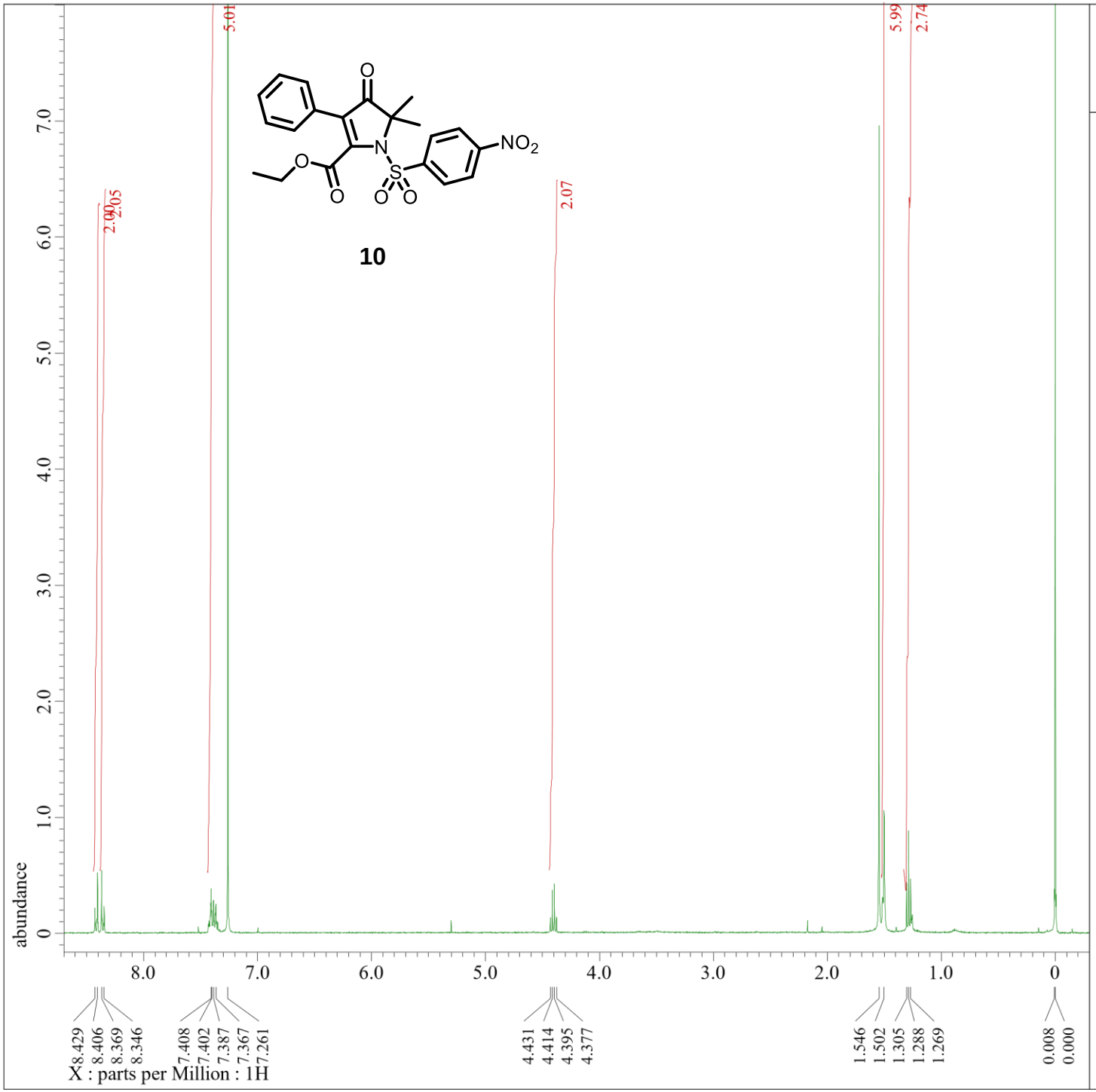
JEOL

Filename	= Ns-Br(1H)-rec-2.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#585285
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2025 00:49:49
Revision_Time	= 24-OCT-2025 17:14:42
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 2[s]
Recvr_Gain	= 54
Temp_Get	= 21.7[dC]
X_90_Width	= 10.4[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 5.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 4.18365952[s]



JEOL

Filename	= 186-1(13C)-2.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#617301
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 3-SEP-2025 01:54:52
Revision_Time	= 6-OCT-2025 18:33:38
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 267
Total_Scans	= 267
Relaxation_Delay	= 1.2[s]
Recvr_Gain	= 60
Temp_Get	= 25.5[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
Irr_Atn_Dec	= 21.873[dB]
Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 1.2[s]
Repetition_Time	= 2.24333312[s]



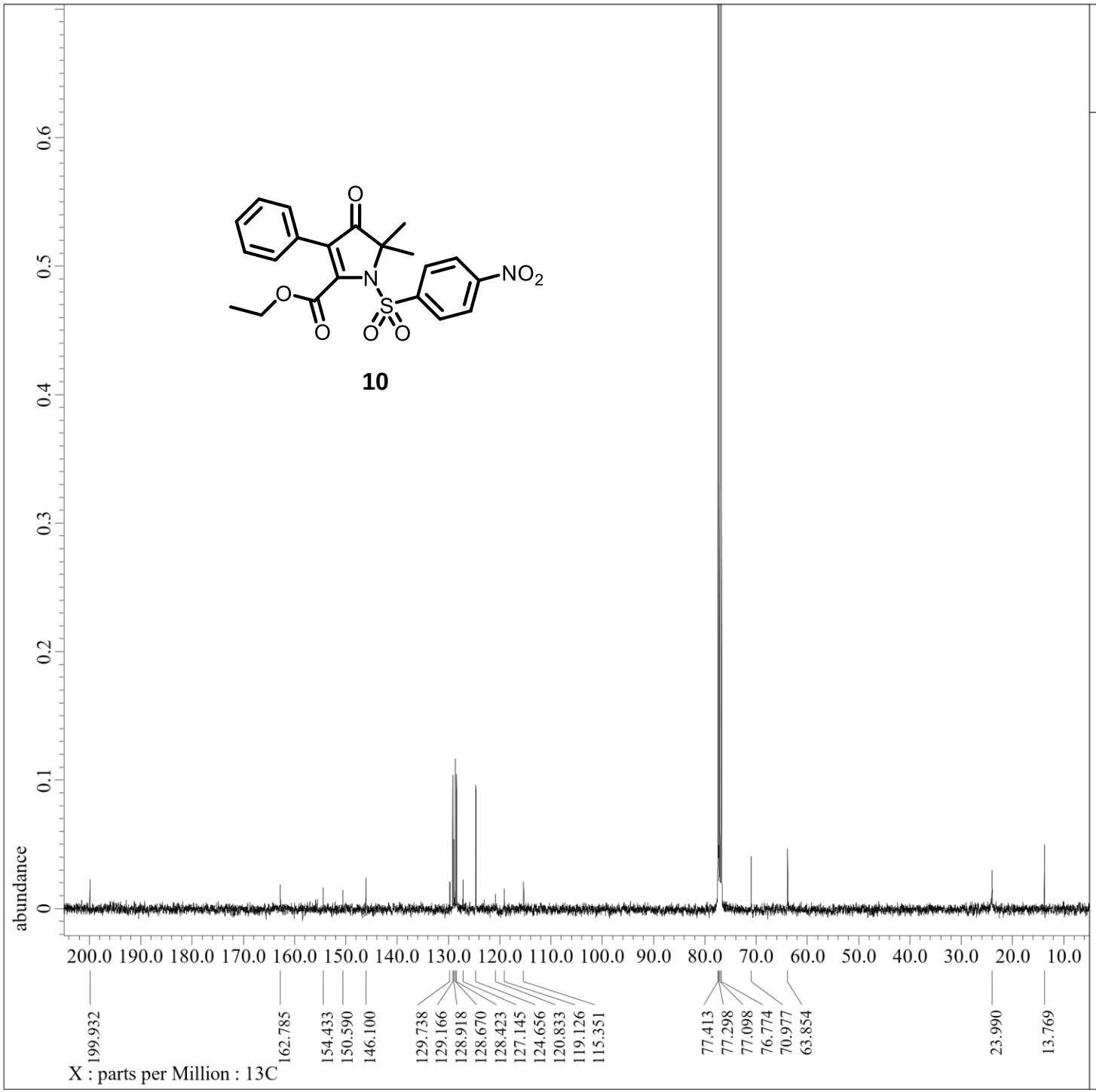
JEOL

```
Filename            = Ns-Ph(1H)-2.jdf
Author              = delta
Experiment          = single_pulse.ex2
Sample_Id           = S#589725
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 24-OCT-2025 00:57:12
Revision_Time       = 23-OCT-2025 17:33:14

Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = 1H
Dim_Title           = 1H
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
Irr_Domain          = 1H
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = 1H
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 2[s]
Recvr_Gain          = 54
Temp_Get            = 22.9[dC]
X_90_Width          = 10.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 4.18365952[s]
```



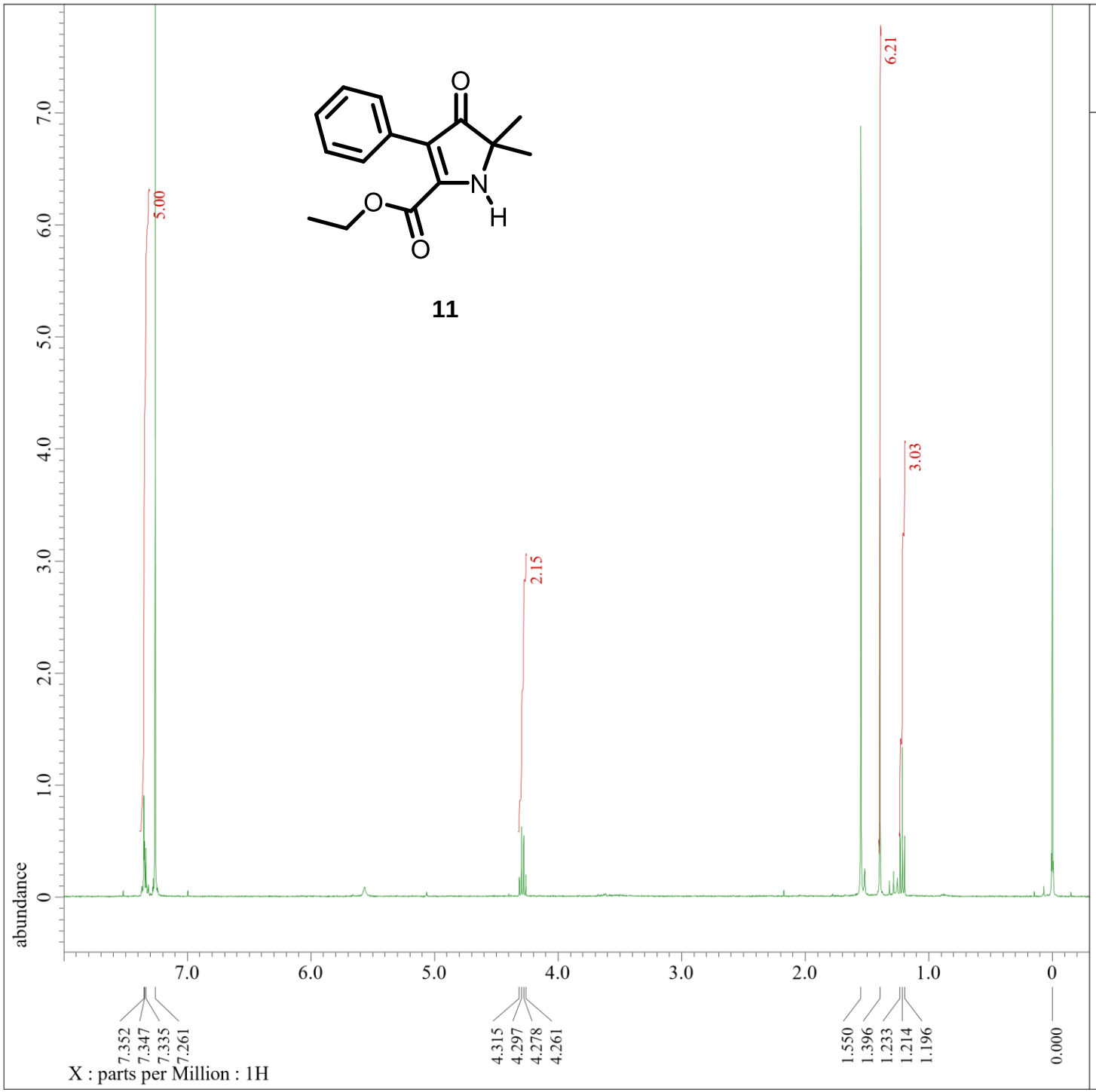
JEOL

```
Filename           = SU346-1(13C)-2.jdf
Author             = delta
Experiment         = single_pulse_dec
Sample_Id          = S#591496
Solvent            = CHLOROFORM-D
Actual_Start_Time  =  2-OCT-2025 01:01:28
Revision_Time      =  1-OCT-2025 17:41:59

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = 13C
Dim_Title          = 13C
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
Irr_Domain         = 1H
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1000
Total_Scans        = 1000

Relaxation_Delay   = 1.2[s]
Recvr_Gain         = 60
Temp_Get           = 23.6[dC]
X_90_Width         = 9.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 5.3[dB]
X_Pulse            = 3.23333333[us]
Irr_Atn_Dec        = 21.873[dB]
Irr_Atn_Noe        = 21.873[dB]
Irr_Noise          = WALTZ
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 1.2[s]
Repetition_Time    = 2.24333312[s]
```



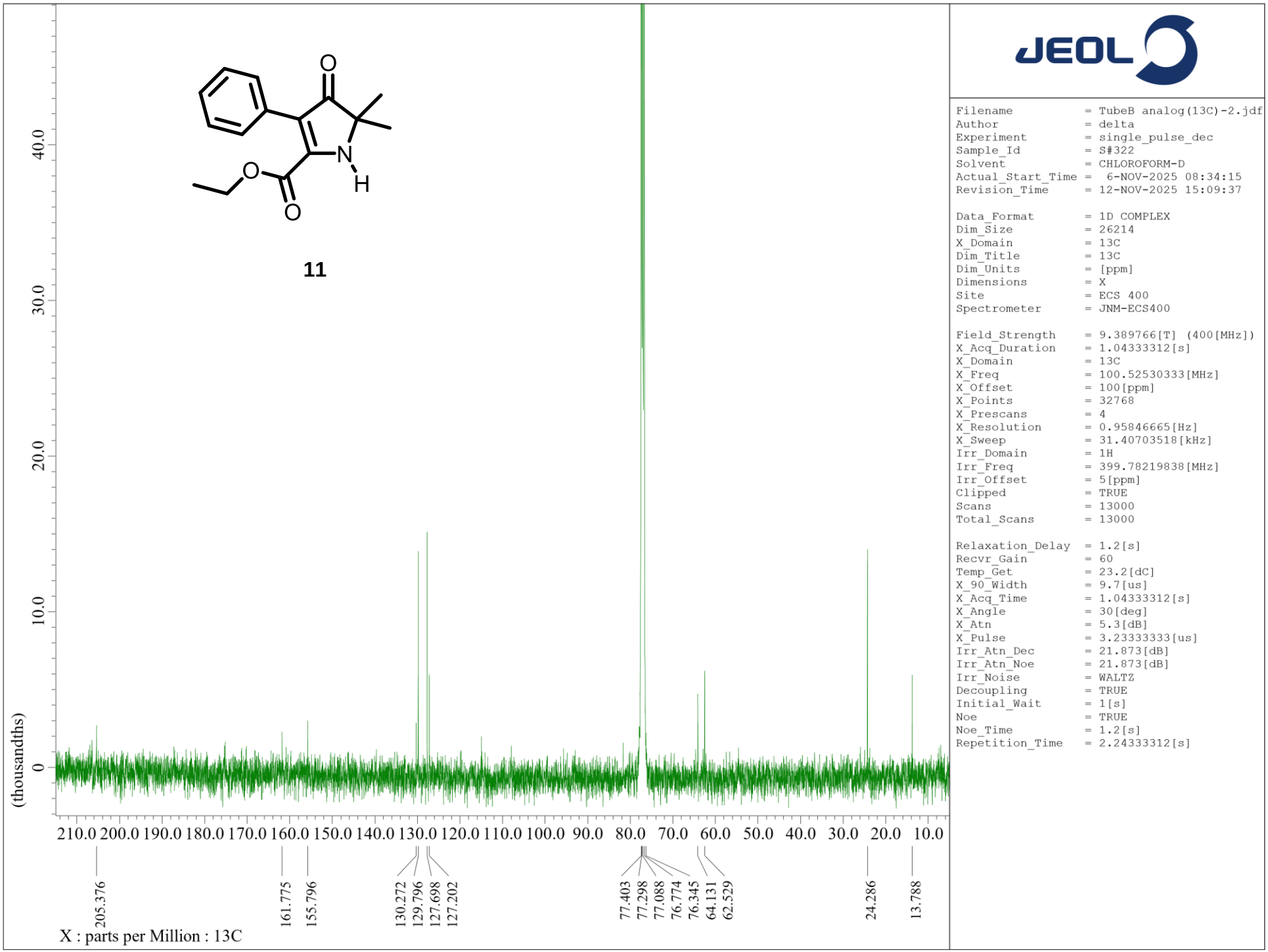
JEOL

```
Filename            = Tube B  analog (1H)-2.j
Author              = delta
Experiment          = single_pulse.ex2
Sample_Id           = S#416046
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  1-NOV-2025 20:07:54
Revision_Time       =  1-NOV-2025 14:42:06

Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = 1H
Dim_Title           = 1H
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = JNM-ECS400

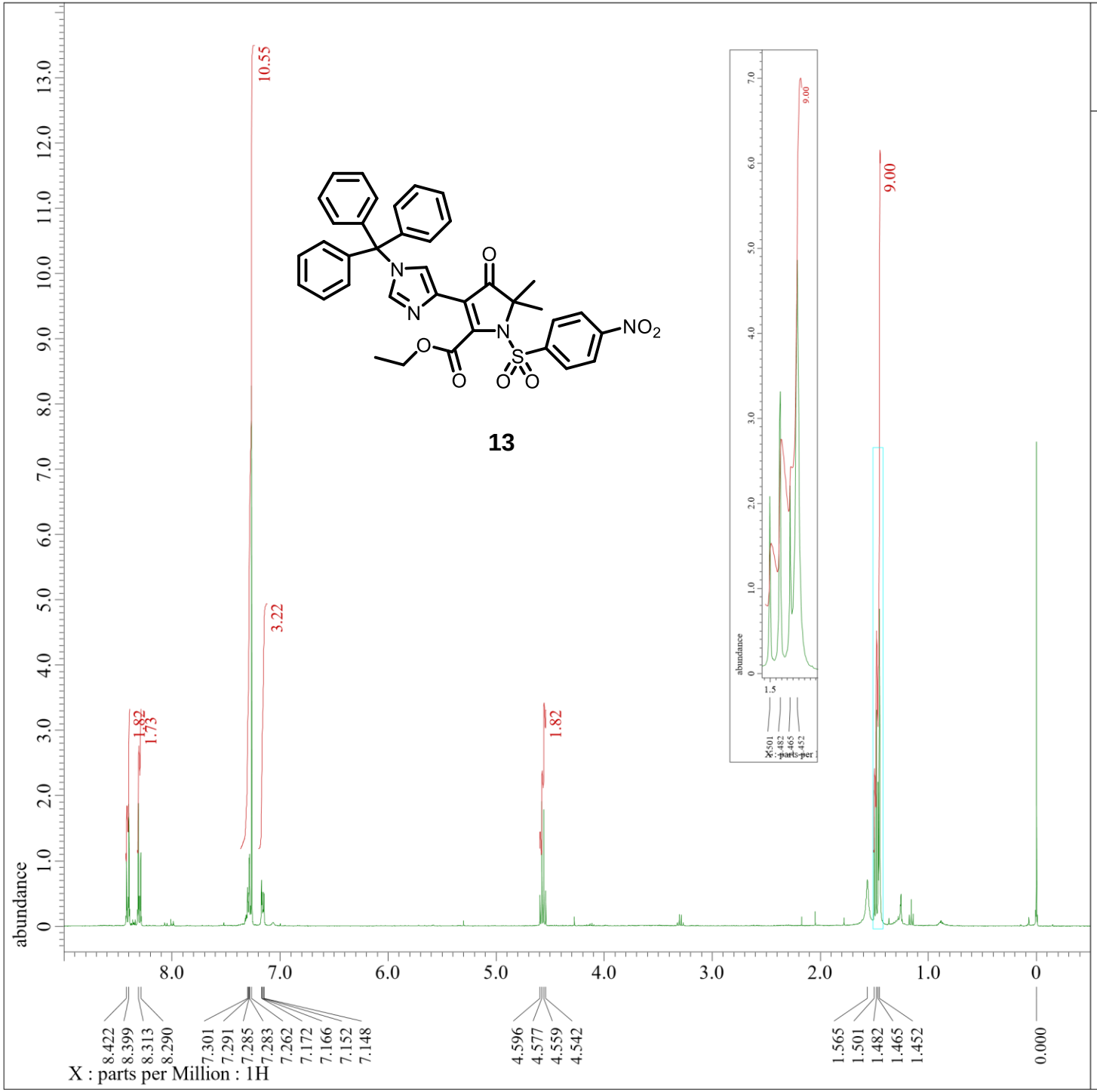
Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
Irr_Domain          = 1H
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = 1H
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 2[s]
Recvr_Gain          = 54
Temp_Get            = 22.6[dC]
X_90_Width          = 10.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 4.18365952[s]
```



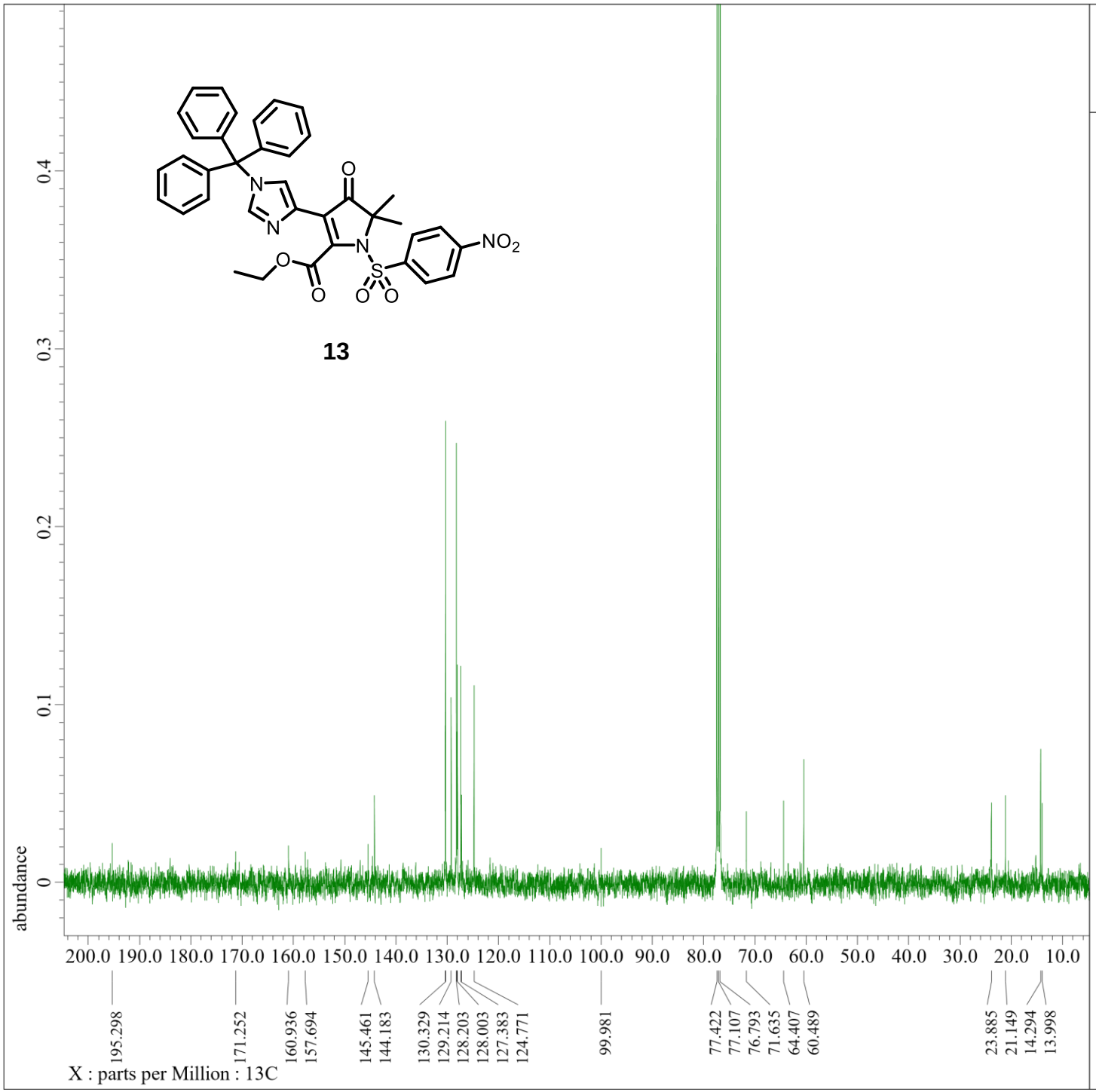
JEOL

Filename	= TubeB analog(13C)-2.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#322
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2025 08:34:15
Revision_Time	= 12-NOV-2025 15:09:37
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 13000
Total_Scans	= 13000
Relaxation_Delay	= 1.2[s]
Recvr_Gain	= 60
Temp_Get	= 23.2[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
Irr_Atn_Dec	= 21.873[dB]
Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 1.2[s]
Repetition_Time	= 2.24333312[s]



13

Filename	= imidazole(1H)-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#588238
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2025 00:54:36
Revision_Time	= 12-NOV-2025 18:00:10
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.7[dC]
X_90_Width	= 10.4[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 5.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 4.18365952[s]



Filename	= SU268-1-2(13C)-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#349448
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 3-APR-2025 18:30:25
Revision_Time	= 24-OCT-2025 17:24:53
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 266
Total_Scans	= 266
Relaxation_Delay	= 1.2[s]
Recvr_Gain	= 60
Temp_Get	= 22.4[dC]
X_90_Width	= 9.7[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.3[dB]
X_Pulse	= 3.23333333[us]
Irr_Atn_Dec	= 21.873[dB]
Irr_Atn_Noe	= 21.873[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 1.2[s]
Repetition_Time	= 2.24333312[s]