

Rapid Access to Indole-fused Bicyclo[2.2.2]octanones by Merging Umpolung Strategy and Molecular Iodine as A Green Catalyst

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SUPPORTING INFORMATION

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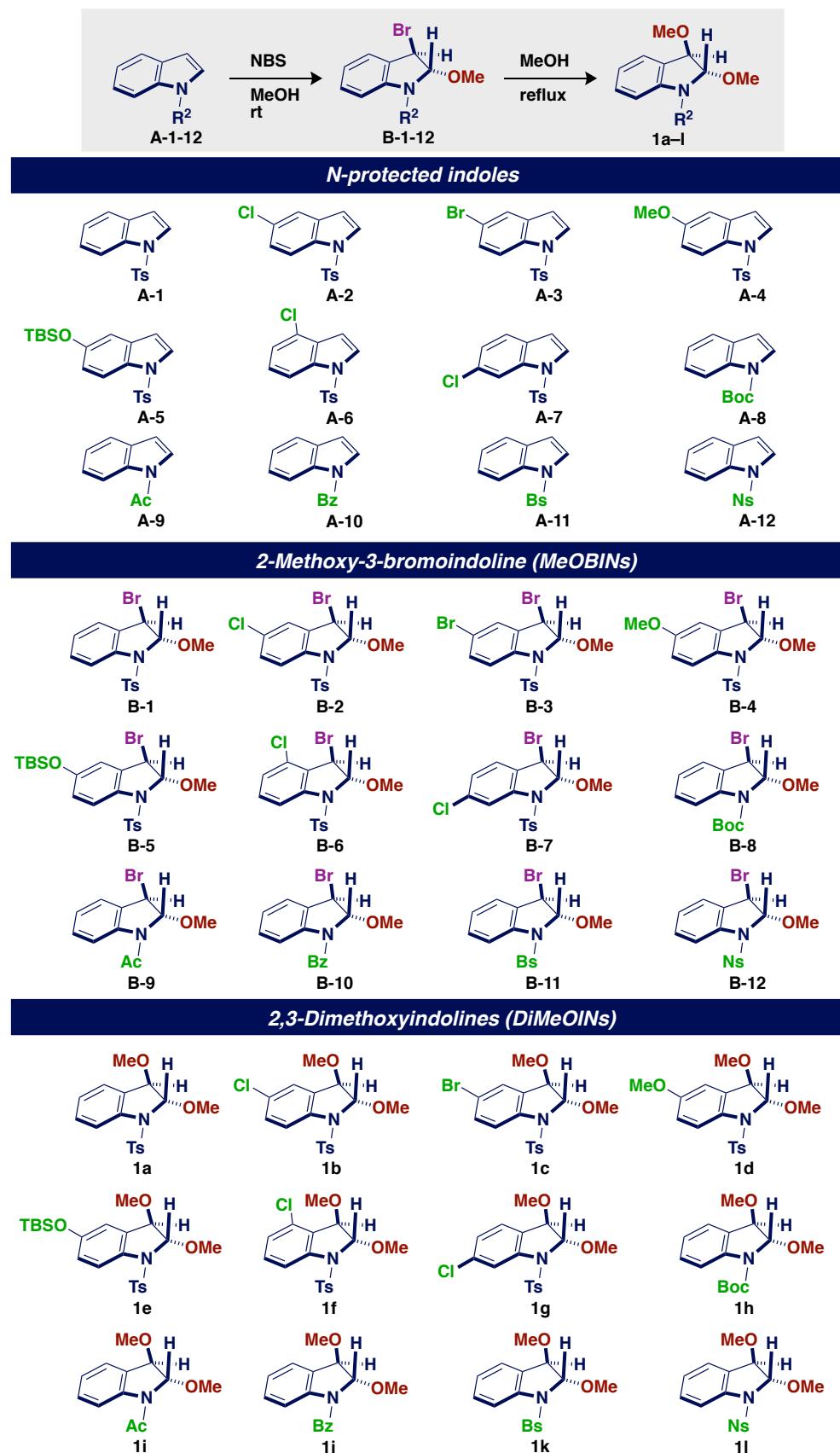
Experimental Section

1. General Experimental

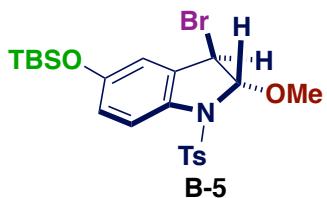
All reagents were weighed and handled in air at room temperature. Reactions were monitored by thin layer chromatography (TLC) carried out on a silica gel plates (60F-254) and visualized under UV illumination at 254 or 366 nm depending on the compounds. Extracts were dried over anhydrous MgSO₄. Solvents were removed by a rotary evaporator *in vacuo*. Column chromatography was performed on silica gel (Silica Gel 63–210 mesh, Kanto Chemical Co., Ltd.). The NMR experiments were performed with a JEOL JNM-ECA500 (500 MHz) spectrometer, and chemical shifts are expressed in ppm (δ) using residual solvent as an internal reference (CDCl₃, ¹H NMR: δ 7.25, ¹³C NMR: δ 77.1). ¹H NMR data is represented as follows: Chemical shift (multiplicity, coupling constant(s) J in Hertz (Hz), integration). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets,ddd = doublet of doublet of doublets, br = broad. High resolution mass spectra (HRMS) were recorded on a JEOL JMS-T100LP mass spectrometers using electrospray ionization-time of flight (ESI-TOF) reflection experiments. All N-protected indoles are known products. The N-protected indoles **A1–12** were prepared by following a previous literature.^{S1} 2-Methoxy-3-bromoindolines **B1–4**, **B6–10**, **B-12** were prepared by reported methods.^{S2–3} 2,3-Dimethoxyindolines **1a–d**, **1f–j**, **1l** were prepared by reported methods.^{S3}

2. Experimental Procedure

Synthesis of 2,3-Dimethoxyindolines 1



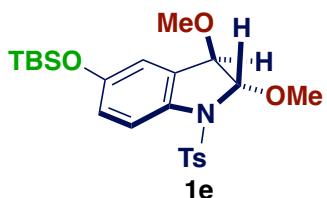
trans-3-Bromo-2-methoxy-5-TBSO-1-tosylindoline (B-5)



To a solution of N-Ts 5-TBSO-indole **A-5** (803 mg, 2 mmol) in MeOH (20 mL) was added NBS (392 mg, 2.2 mmol). The mixture was stirred at room temperature. After 1 h, Et₂O (10 mL) was dropwise added to the mixture and the mixture was stirred for 10 min. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **B-5**.

782.1 mg, 76% yield. colorless solid; ¹H NMR (500 MHz, CDCl₃) δ: 7.62 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 8.6 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.80 (dd, *J* = 2.9, 9.2 Hz, 1H), 6.71 (d, *J* = 2.9 Hz, 1H), 5.51 (s, 1H), 4.83 (s, 1H), 3.57 (s, 3H), 2.28 (s, 3H), 0.94 (s, 9H), 0.15 (s, 3H), 0.14 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 153.6, 144.4, 135.2, 134.7, 132.9, 129.5, 127.8, 122.4, 118.3, 117.5, 100.3, 56.4, 47.3, 25.7, 21.6, 18.2, -4.4; HRMS (ESI) *m/z*: 534.0748, 536.0730 (Calcd for C₂₂H₃₀BrNNaO₄SSi [M+Na]⁺: 534.0746, 536.0725).

trans-2,3-Dimethoxy-5-TBSO-1-tosylindoline (1e)



A solution of **B-5** (513 mg, 1 mmol) in MeOH (15 mL) was stirred at 100 °C for 16 h. After addition of H₂O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over MgSO₄ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/2) to give **1e**.

255.0 mg, 55% yield. colorless solid; ¹H NMR (500 MHz, CDCl₃) δ: 7.51 (d, *J* = 8.2 Hz, 2H), 7.49 (d, *J* = 9.1 Hz, 1H), 7.11 (d, *J* = 8.1 Hz, 2H), 6.80 (dd, *J* = 9.2, 2.9 Hz, 1H), 6.72 (d, *J* = 2.3 Hz, 1H), 5.24 (s, 1H), 4.14 (s, 1H), 3.59 (s, 3H), 3.09 (s, 3H), 2.32 (s, 3H), 0.95 (s, 9H), 0.15 (s, 3H), 0.14 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 153.1, 143.9, 135.8, 135.0, 131.9, 129.9, 129.2, 128.0, 127.6, 122.0, 118.7, 118.3, 96.9, 84.1, 56.1, 25.7, 21.6, 18.2, -4.4; HRMS (ESI) *m/z*: 486.1745 (Calcd for C₂₃H₃₃NNaO₅SSI [M+Na]⁺: 486.1746).

trans-3-Bromo-2-methoxy-1-benzenesulfonyllindoline (B-11)



To a solution of N-Bs indole **A-11** (2.57 g, 10 mmol) in MeOH (200 mL) was added NBS (1.96 g, 11 mmol). The mixture was stirred at room temperature for 30 min. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **B-11**.

2.99 g, 81% yield. colorless solid; ¹H NMR (500 MHz, CDCl₃) δ: 7.80 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.68 (d, *J* = 8.6 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.11 (td, *J* = 7.5, 1.2 Hz, 1H), 5.57 (s, 1H), 4.93 (s, 1H), 3.60 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 140.5, 138.2, 133.7, 131.5, 130.7, 129.0, 127.7, 126.3, 125.5, 117.1, 99.9, 56.4, 47.2, 28.8; HRMS (ESI) *m/z*: 389.9778, 391.9753 (Calcd for C₁₅H₁₄BrNNaO₃S [M+Na]⁺; 389.9775, 391.9755).

***trans*-2,3-Dimethoxy-1-benzenesulfonylindoline (1k)**



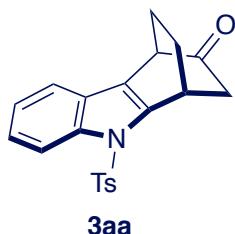
A solution of **B-11** (1.47 g, 4 mmol) in MeOH (100 mL) was stirred at 100 °C for 16 h. After addition of H₂O, the whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over MgSO₄ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/2) to give **1k**.

753.7 mg, 59% yield. colorless solid; ¹H NMR (500 MHz, CDCl₃) δ: 7.69 (d, *J* = 8.0 Hz, 2H), 7.64 (d, *J* = 8.6 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 3H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 5.31 (s, 1H), 4.24 (s, 1H), 3.59 (s, 3H), 3.12 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 140.8, 138.0, 133.3, 130.6, 130.5, 128.7, 127.5, 126.9, 124.9, 117.5, 96.6, 83.8, 56.1, 56.0; HRMS (ESI) *m/z*: 342.0776 (Calcd for C₁₆H₁₇NNaO₄S [M+Na]⁺; 342.0776).

General Procedure for the Iodine-catalyzed Cascade Reaction of DiMeOINs (1) with 2 (Scheme 2 and 3)

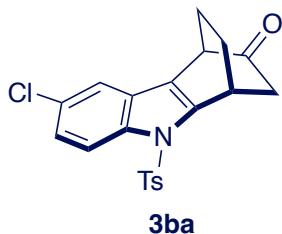
To a solution of **1** (0.5 mmol) and **2** (0.75 mmol) in MeCN (10 mL) was added I₂ (12.7 mg, 0.05 mmol). The mixture was stirred at 100 °C. After indicated time (Scheme 2), the mixture was cooled to room temperature and then concentrated *in vacuo*. The crude residue was directly purified by silica gel column chromatography (CHCl₃/hexane = 1/2–2/1) to give **3**.

***Rel*-(1*R*,4*R*)-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3aa)**



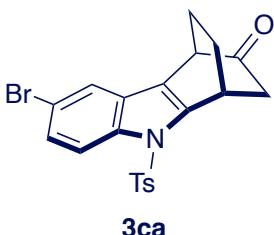
135.8 mg, 74% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.18 (d, $J = 8.6$ Hz, 1H), 7.68 (d, $J = 8.9$ Hz, 2H), 7.42 (d, $J = 7.5$ Hz, 1H), 7.30 (td, $J = 7.5, 1.2$ Hz, 1H), 7.26 (td, $J = 7.5, 1.2$ Hz, 1H), 7.20 (d, $J = 8.0$ Hz, 2H), 4.41 (t, $J = 2.9$ Hz, 1H), 3.89 (t, $J = 2.9$ Hz, 1H), 2.33 (s, 3H), 2.30 (dd, $J = 18.3, 2.9$ Hz, 1H), 2.05-2.11 (m, 2H), 1.98-2.03 (m, 1H), 1.58-1.69 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.4, 145.3, 141.7, 136.6, 135.8, 130.1, 127.1, 126.4, 124.3, 123.9, 118.3, 118.2, 114.9, 45.9, 41.6, 32.1, 26.3, 23.8, 21.7; HRMS (ESI) m/z : 388.0980 (Calcd for $\text{C}_{21}\text{H}_{19}\text{NNaO}_3\text{S}$ [M+Na] $^+$: 388.0983).

Rel-(1*R,4R*)-6-chloro-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3ba)



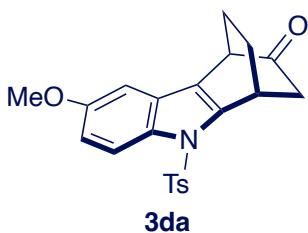
112.2 mg, 56% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.09 (d, $J = 9.2$ Hz, 1H), 7.66 (d, $J = 8.6$ Hz, 2H), 7.38 (d, $J = 1.7$ Hz, 1H), 7.25 (dd, $J = 9.2, 2.3$ Hz, 1H), 7.21 (d, $J = 8.6$ Hz, 2H), 4.38 (t, $J = 2.9$ Hz, 1H), 3.84 (t, $J = 2.9$ Hz, 1H), 2.34 (s, 3H), 2.30 (dd, $J = 18.9, 2.9$ Hz, 1H), 1.98-2.12 (m, 3H), 1.56-1.67 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 208.8, 145.6, 143.2, 135.5, 134.9, 130.3, 129.8, 128.3, 126.4, 124.4, 118.0, 117.6, 115.8, 45.8, 41.3, 32.2, 26.1, 23.7, 21.7; HRMS (ESI) m/z : 422.0597, 424.0563 (Calcd for $\text{C}_{21}\text{H}_{18}\text{ClNNaO}_3\text{S}$ [M+Na] $^+$: 422.0594, 424.0564).

Rel-(1*R,4R*)-6-bromo-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3ca)



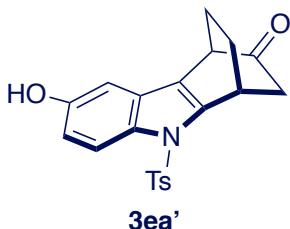
89.4 g, 40% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.05 (d, $J = 9.2$ Hz, 1H), 7.65 (d, $J = 8.1$ Hz, 2H), 7.55 (d, $J = 1.7$ Hz, 1H), 7.39 (dd, $J = 9.2, 2.3$ Hz, 1H), 7.22 (d, $J = 8.0$ Hz, 2H), 4.39 (t, $J = 2.9$ Hz, 1H), 3.84 (t, $J = 2.9$ Hz, 1H), 2.35 (s, 3H), 2.30 (dd, $J = 18.4, 2.3$ Hz, 1H), 1.99-2.12 (m, 3H), 1.57-1.67 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 208.7, 145.7, 143.0, 135.5, 135.3, 130.3, 128.8, 127.1, 126.4, 121.0, 117.5, 117.5, 116.2, 45.8, 41.3, 32.2, 26.1, 23.7, 21.7; HRMS (ESI) m/z : 466.0090, 468.0068 (Calcd for $\text{C}_{21}\text{H}_{18}\text{BrNNaO}_3\text{S}$ [M+Na] $^+$: 466.0088, 468.0068).

Rel-(1*R,4R*)-6-methoxy-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3da)^{S4}



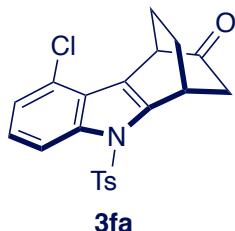
140.4 mg, 69% yield. colorless oil. ^1H NMR (500 MHz, CDCl_3) δ : 8.06 (d, $J = 8.6$ Hz, 1H), 7.64 (d, $J = 8.0$ Hz, 2H), 7.19 (d, $J = 8.1$ Hz, 2H), 6.90 (dd, $J = 9.2, 2.9$ Hz, 1H), 6.85 (d, $J = 2.9$ Hz, 1H), 4.37 (t, $J = 2.9$ Hz, 1H), 3.85 (t, $J = 3.3$ Hz, 1H), 3.82 (s, 3H), 2.33 (s, 3H), 2.29 (dd, $J = 18.3, 2.9$ Hz, 1H), 1.97-2.12 (m, 3H), 1.57-1.68 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.3, 156.9, 145.2, 142.5, 135.7, 131.2, 130.1, 128.2, 126.4, 118.4, 115.8, 113.0, 100.9, 55.7, 45.9, 41.6, 32.2, 26.2, 23.8, 21.7; HRMS (ESI) m/z : 418.1089 (Calcd for $\text{C}_{22}\text{H}_{21}\text{NNaO}_4\text{S} [\text{M}+\text{Na}]^+$: 354.0106, 418.1089).

Rel-(1R,4R)-6-hydroxy-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3ea')



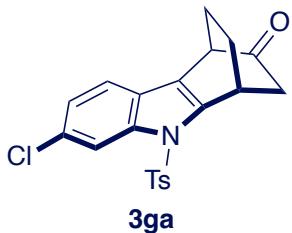
129.3 mg, 68% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.00 (d, $J = 7.7$ Hz, 1H), 7.63 (d, $J = 8.6$ Hz, 2H), 7.18 (d, $J = 8.6$ Hz, 2H), 6.77-6.84 (m, 2H), 5.97 (br s, 1H), 4.35 (s, 1H), 3.80 (s, 1H), 2.32 (s, 3H), 2.15-2.27 (m, 1H), 1.95-2.12 (m, 3H), 1.55-1.64 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 210.6, 153.1, 145.3, 142.7, 135.6, 131.2, 130.1, 128.4, 126.4, 118.0, 115.8, 113.2, 103.5, 45.9, 41.7, 32.1, 26.1, 23.7, 21.7; HRMS (ESI) m/z : 404.0930 (Calcd for $\text{C}_{21}\text{H}_{19}\text{NNaO}_4\text{S} [\text{M}+\text{Na}]^+$: 404.0932).

Rel-(1R,4R)-5-chloro-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3fa)



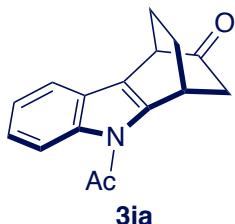
104.4 mg, 52% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.10-8.14 (m, 1H), 7.68 (d, $J = 8.6$ Hz, 2H), 7.23 (d, $J = 8.1$ Hz, 2H), 7.21 (d, $J = 5.2$ Hz, 2H), 4.46 (t, $J = 2.9$ Hz, 1H), 4.43 (t, $J = 4.9$ Hz, 1H), 2.36 (s, 3H), 2.30 (dd, $J = 18.3, 2.3$ Hz, 1H), 1.97-2.13 (m, 3H), 1.56-1.76 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.3, 145.7, 142.8, 137.4, 135.6, 130.3, 126.5, 125.6, 125.0, 124.6, 124.5, 117.5, 113.3, 46.7, 41.1, 32.0, 25.8, 23.9, 21.7; HRMS (ESI) m/z : 422.0592, 424.0564 (Calcd for $\text{C}_{21}\text{H}_{18}\text{ClNNaO}_3\text{S} [\text{M}+\text{Na}]^+$: 422.0594, 424.0564).

Rel-(1R,4R)-7-chloro-9-tosyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3ga)



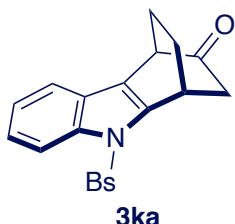
89.6 mg, 45% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.21 (d, $J = 1.7$ Hz, 1H), 7.68 (d, $J = 8.6$ Hz, 2H), 7.32 (d, $J = 8.6$ Hz, 1H), 7.24 (d, $J = 8.6$ Hz, 2H), 7.23 (dd, $J = 8.0, 1.7$ Hz, 1H), 4.38 (t, $J = 2.9$ Hz, 1H), 3.86 (t, $J = 4.9$ Hz, 1H), 2.36 (s, 3H), 2.30 (dd, $J = 17.8, 2.3$ Hz, 1H), 1.98-2.12 (m, 3H), 1.56-1.68 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.0, 145.7, 142.2, 136.8, 135.6, 130.3, 130.1, 126.5, 125.6, 124.5, 118.8, 117.9, 115.1, 45.8, 41.4, 32.1, 26.2, 23.7, 21.7; HRMS (ESI) m/z : 422.0595, 424.0561 (Calcd for $\text{C}_{21}\text{H}_{18}\text{ClNNaO}_3\text{S} [\text{M}+\text{Na}]^+$: 422.0594, 424.0564).

Rel-(1*R*,4*R*)-9-acetyl-2,3,4,9-tetrahydro-1*H*-1,4-ethanocbazol-10-one (3ia)



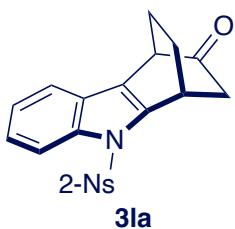
77.6 mg, 69% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.04 (d, $J = 7.5$ Hz, 1H), 7.48 (dd, $J = 7.5, 1.7$ Hz, 1H), 7.27-7.32 (m, 2H), 4.41 (t, $J = 2.9$ Hz, 1H), 3.97 (t, $J = 3.6$ Hz, 1H), 2.77 (s, 3H), 2.33 (d, $J = 2.3$ Hz, 2H), 2.13-2.20 (m, 1H), 1.95-2.06 (m, 1H), 1.71-1.79 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.6, 169.6, 142.3, 136.3, 127.2, 124.4, 123.7, 118.3, 118.1, 115.9, 45.7, 41.6, 33.6, 27.1, 26.2, 23.6; HRMS (ESI) m/z : 276.1005 (Calcd for $\text{C}_{16}\text{H}_{15}\text{NNaO}_2 [\text{M}+\text{Na}]^+$: 276.1000).

Rel-(1*R*,4*R*)-9-(phenylsulfonyl)-2,3,4,9-tetrahydro-1*H*-1,4-ethanocbazol-10-one (3ka)



133.4 mg, 69% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.19 (d, $J = 8.0$ Hz, 1H), 7.80 (d, $J = 7.5$ Hz, 2H), 7.54 (d, $J = 7.4$ Hz, 1H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.41 (d, $J = 7.5$ Hz, 1H), 7.32 (td, $J = 7.5, 1.2$ Hz, 1H), 7.27 (d, $J = 8.1$ Hz, 1H), 4.41 (t, $J = 2.9$ Hz, 1H), 3.90 (t, $J = 2.9$ Hz, 1H), 2.31 (dd, $J = 18.4, 2.3$ Hz, 1H), 1.98-2.13 (m, 3H), 1.57-1.69 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.3, 141.6, 138.8, 136.7, 134.2, 129.6, 127.1, 126.4, 124.4, 124.0, 118.5, 118.2, 114.9, 45.9, 41.6, 32.1, 26.3, 23.8; HRMS (ESI) m/z : 374.0826 (Calcd for $\text{C}_{20}\text{H}_{17}\text{NNaO}_3\text{S} [\text{M}+\text{Na}]^+$: 374.0827).

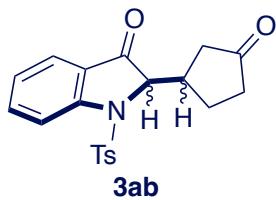
Rel-(1*R*,4*R*)-9-((2-nitrophenyl)sulfonyl)-2,3,4,9-tetrahydro-1*H*-1,4-ethanocarbazol-10-one (3la**)**



3la

43.6 mg, 22% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 7.90 (d, $J = 7.5$ Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.73 (t, $J = 7.5$ Hz, 1H), 7.61 (t, $J = 8.0$ Hz, 1H), 7.51 (d, $J = 8.0$ Hz, 2H), 7.27-7.32 (m, 2H), 4.24 (t, $J = 2.9$ Hz, 1H), 3.98 (t, $J = 2.9$ Hz, 1H), 2.21-2.33 (m, 2H), 2.10-2.17 (m, 1H), 1.97-2.04 (m, 1H), 1.64-1.75 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.4, 147.8, 142.8, 136.7, 134.9, 133.1, 132.7, 129.2, 126.8, 125.4, 124.6, 124.4, 118.6, 118.4, 114.6, 46.0, 41.6, 32.4, 26.2, 23.9; HRMS (ESI) m/z : 419.0689 (Calcd for $\text{C}_{20}\text{H}_{16}\text{N}_2\text{NaO}_5\text{S}$ [M+Na] $^+$: 419.0687).

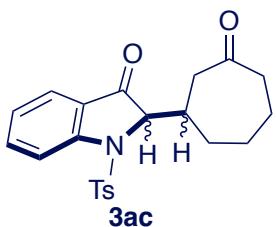
2-(3-Oxocyclopentyl)-1-tosylindolin-3-one (3ab**)**



3ab

149.1 mg, 81% yield (dr = 2:1). colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.05 (d, $J = 8.6$ Hz, 2/3H), 8.04 (d, $J = 8.6$ Hz, 1/3H), 7.70 (td, $J = 7.5, 1.2$ Hz, 1H), 7.56 (d, $J = 6.9$ Hz, 1H), 7.50 (d, $J = 8.6$ Hz, 4/3 H), 7.48 (d, $J = 8.6$ Hz, 2/3 H), 4.13 (d, $J = 4.0$ Hz, 2/3H), 4.09 (d, $J = 4.6$ Hz, 1/3H), 3.02-3.11 (m, 2/3H), 2.93-3.01 (m, 1/3H), 2.40-2.48 (m, 1H), 2.33 (s, 3H), 2.29-2.37 (m, 4/3H), 2.20-2.27 (m, 4/3H), 1.99-2.18 (m, 5/3H), 1.83-1.93 (m, 2/3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 216.8, 216.7, 198.4, 198.3, 153.8, 153.7, 145.4, 137.5, 137.4, 132.8, 132.7, 130.1, 127.4, 126.2, 126.0, 125.7, 125.6, 124.4, 118.7, 118.4, 68.5, 68.1, 40.7, 40.4, 40.3, 39.4, 39.3, 38.3, 38.2, 38.1, 25.1, 24.1, 21.6; HRMS (ESI) m/z : 492.0930 (Calcd for $\text{C}_{20}\text{H}_{19}\text{NNaO}_4\text{S}$ [M+Na] $^+$: 492.0932).

2-(3-Oxocycloheptyl)-1-tosylindolin-3-one (3ac**)**

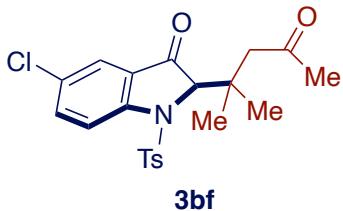


3ac

153.4 mg, 77% yield (dr = 2:1). colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 8.08 (d, $J = 8.6$ Hz, 2/3H), 8.01 (d, $J = 8.6$ Hz, 1/3H), 7.65-7.70 (m, 1H), 7.54-7.57 (m, 3H), 7.18-7.22 (m, 3H), 3.90 (d, $J = 3.4$ Hz, 1/3H), 3.80 (d, $J = 4.0$ Hz, 2/3H), 2.74-2.82 (m, 1H), 2.42-2.58 (m, 4H), 2.29-2.40 (m, 1H), 2.34 (s, 3H), (1.08-1.93 (m, 5H); ^{13}C NMR (125 MHz, CDCl_3) δ : 212.7, 212.6, 197.8, 197.7, 153.7, 148.5, 145.3, 139.9, 137.4, 137.3, 133.1, 133.0, 130.1, 127.4, 127.3, 125.9, 125.2, 125.1, 124.2, 117.7, 117.5, 70.9, 70.7, 50.0, 45.9, 44.2, 43.9, 43.8, 43.7, 40.1, 39.6, 38.2, 37.5, 32.4, 30.3, 29.5, 29.2, 29.0, 27.3, 24.9, 24.4, 24.1, 24.0, 21.6, 21.5; HRMS (ESI) m/z : 420.1243 (Calcd for

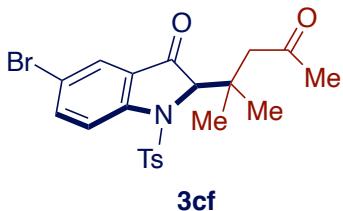
$C_{22}H_{23}NNaO_4S$ [M+Na]⁺: 420.1245).

5-Chloro-2-(2-methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (3cf)



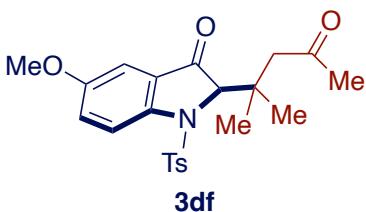
139.0 mg, 66% yield. colorless oil; ¹H NMR (500 MHz, CDCl₃) δ: 7.99 (d, *J* = 8.6 Hz, 1H), 7.61 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.41 (d, *J* = 2.3 Hz, 1H), 7.31 (d, *J* = 8.6 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 4.64 (s, 1H), 4.71 (s, 1H), 2.92 (d, *J* = 18.3 Hz, 1H), 2.57 (d, *J* = 18.4 Hz, 1H), 2.32 (s, 3H), 2.19 (s, 3H), 1.00 (s, 3H), 0.97 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 207.7, 198.8, 152.9, 145.2, 136.3, 132.1, 131.8, 130.0, 129.9, 127.8, 123.2, 121.8, 71.6, 51.4, 38.2, 31.2, 25.4, 22.9, 21.6; HRMS (ESI) *m/z*: 442.0850, 444.0826 (Calcd for C₂₁H₂₂CINaO₄S [M+Na]⁺: 442.0856, 444.0826).

5-Bromo-2-(2-methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (3cf)



150.2 mg, 65% yield. colorless oil; ¹H NMR (500 MHz, CDCl₃) δ: 7.92 (d, *J* = 8.6 Hz, 1H), 7.74 (dd, *J* = 9.2, 2.3 Hz, 1H), 7.57 (d, *J* = 2.3 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 2H), 4.64 (s, 1H), 4.70 (s, 1H), 2.91 (d, *J* = 18.3 Hz, 1H), 2.56 (d, *J* = 18.3 Hz, 1H), 2.32 (s, 3H), 2.19 (s, 3H), 1.00 (s, 3H), 0.97 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 207.6, 198.6, 153.3, 145.2, 139.1, 131.8, 130.2, 130.0, 127.8, 126.3, 122.1, 119.6, 71.5, 51.4, 38.2, 31.2, 25.4, 22.9, 21.6; HRMS (ESI) *m/z*: 486.0348, 488.0325 (Calcd for C₂₁H₂₂BrNaO₄S [M+Na]⁺: 486.0351, 488.0330).

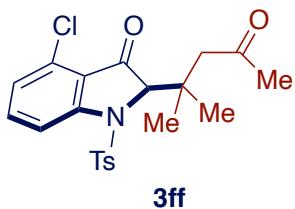
5-Methoxy-2-(2-methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (3df)



126.7 mg, 61% yield. colorless oil; ¹H NMR (500 MHz, CDCl₃) δ: 7.92 (d, *J* = 9.2 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.24 (dd, *J* = 9.2, 2.9 Hz, 1H), 7.10 (d, *J* = 8.1 Hz, 2H), 6.86 (d, *J* = 2.9 Hz, 1H), 4.60 (s, 1H), 3.78 (s, 3H), 2.93 (d, *J* = 18.3 Hz, 1H), 2.56 (d, *J* = 18.3 Hz, 1H), 2.30 (s, 3H), 2.19 (s, 3H), 1.02 (s, 3H), 0.95 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 207.7, 200.2, 158.1, 148.6, 144.7, 131.7, 129.8, 129.7, 127.9, 125.4, 121.9, 104.4, 71.8, 55.8, 51.6,

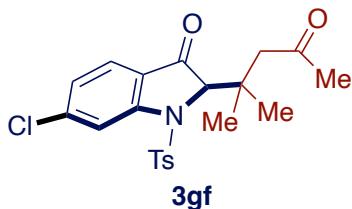
38.1, 31.2, 25.4, 22.9, 21.6; HRMS (ESI) m/z : 438.1351 (Calcd for $C_{22}H_{25}NNaO_5S$ [M+Na] $^+$: 438.1351).

4-Chloro-2-(2-methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (3ff)



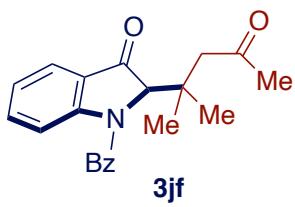
146.6 mg, 70% yield. colorless oil; 1H NMR (500 MHz, $CDCl_3$) δ : 7.95 (d, $J = 9.2$ Hz, 1H), 7.54 (t, $J = 8.1$ Hz, 1H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 4.73 (s, 1H), 2.84 (d, $J = 18.3$ Hz, 1H), 2.64 (d, $J = 18.9$ Hz, 1H), 2.33 (s, 3H), 2.19 (s, 3H), 1.05 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 207.6, 197.1, 155.9, 145.3, 136.5, 132.0, 131.3, 130.0, 127.8, 127.5, 124.7, 118.8, 71.3, 51.2, 38.3, 31.2, 25.6, 23.1, 21.7; HRMS (ESI) m/z : 442.0855, 444.0829 (Calcd for $C_{21}H_{22}ClNNaO_4S$ [M+Na] $^+$: 442.0856, 444.0826).

6-Chloro-2-(2-methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (3gf)



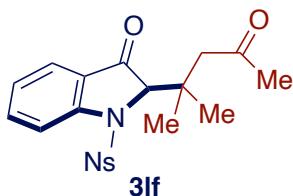
114.1 mg, 54% yield. colorless oil; 1H NMR (500 MHz, $CDCl_3$) δ : 8.05 (d, $J = 1.7$ Hz, 1H), 7.38 (d, $J = 8.6$ Hz, 1H), 7.34 (d, $J = 8.6$ Hz, 2H), 7.20 (d, $J = 8.0, 1.7$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 4.70 (s, 1H), 2.90 (d, $J = 18.9$ Hz, 1H), 2.57 (d, $J = 18.9$ Hz, 1H), 2.31 (s, 3H), 2.19 (s, 3H), 1.01 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 207.7, 198.5, 155.2, 145.2, 143.0, 131.9, 130.0, 127.8, 127.0, 126.8, 124.4, 120.7, 71.5, 51.4, 38.2, 31.2, 25.4, 22.9, 21.7; HRMS (ESI) m/z : 442.0857, 444.0824 (Calcd for $C_{21}H_{22}ClNNaO_4S$ [M+Na] $^+$: 442.0856, 444.0826).

1-Benzoyl-2-(2-methyl-4-oxopentan-2-yl)indolin-3-one (3jf)



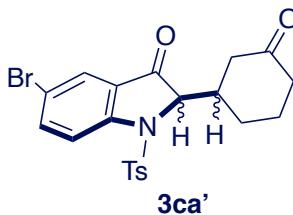
100.1 mg, 60% yield. colorless oil; 1H NMR (500 MHz, $CDCl_3$) δ : 7.66 (d, $J = 8.0$ Hz, 1H), 7.58–7.63 (m, 3H), 7.52 (t, $J = 7.5$ Hz, 2H), 7.34 (td, $J = 8.0, 1.2$ Hz, 1H), 7.11 (t, $J = 7.5$ Hz, 1H), 6.88 (d, $J = 7.5$ Hz, 1H), 5.31 (s, 1H), 2.83 (d, $J = 17.8$ Hz, 1H), 2.60 (d, $J = 17.8$ Hz, 1H), 2.14 (s, 3H), 1.10 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 207.3, 199.7, 170.9, 154.0, 136.4, 136.4, 131.9, 129.1, 128.5, 126.3, 124.0, 123.8, 116.4, 69.5, 50.5, 39.6, 31.8, 26.0, 23.3; HRMS (ESI) m/z : 358.1419 (Calcd for $C_{21}H_{21}NNaO_3$ [M+Na] $^+$: 358.1419).

2-(2-Methyl-4-oxopentan-2-yl)-1-((4-nitrophenyl)sulfonyl)indolin-3-one (3lf)



78.8 mg, 38% yield. colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 7.80 (dd, $J = 8.1, 1.8$ Hz, 1H), 7.77 (d, $J = 8.0$ Hz, 1H), 7.62–7.66 (m, 2H), 7.57 (d, $J = 6.9$ Hz, 1H), 7.56 (td, $J = 8.1, 1.8$ Hz, 1H), 7.47 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.28 (d, $J = 7.5$ Hz, 1H), 4.85 (s, 1H), 2.81 (d, $J = 18.4$ Hz, 1H), 2.65 (d, $J = 18.4$ Hz, 1H), 2.18 (s, 3H), 1.07 (s, 3H), 1.00 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 207.5, 198.9, 152.6, 148.6, 136.7, 135.0, 132.2, 131.5, 129.2, 128.3, 126.4, 124.4, 123.9, 119.6, 72.9, 51.0, 38.7, 31.6, 25.2, 23.0; HRMS (ESI) m/z : 439.0944 (Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{NaO}_6\text{S}$ [$\text{M}+\text{Na}]^+$: 439.0940).

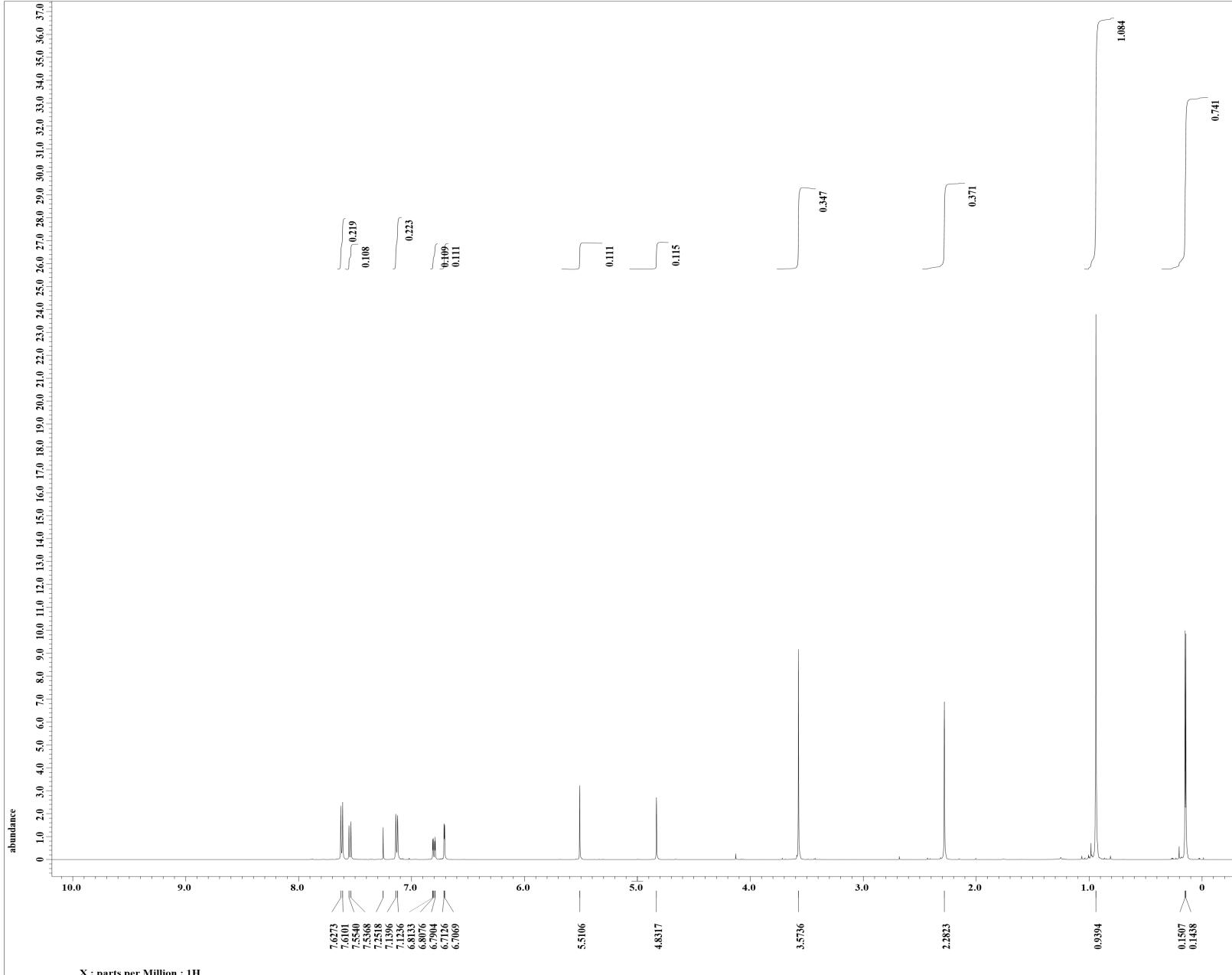
5-Bromo-2-(3-oxocyclohexyl)-1-tosylindolin-3-one (3ca')



83.9 mg, 36% yield ($\text{dr} = 1:1$). colorless oil; ^1H NMR (500 MHz, CDCl_3) δ : 7.96 (d, $J = 9.2$ Hz, 1/2H), 7.94 (d, $J = 9.2$ Hz, 1/2H), 7.76 (d, $J = 9.2$ Hz, 1H), 7.65–7.68 (m, 1H), 7.52 (d, $J = 8.6$ Hz, 1H), 7.49 (d, $J = 8.0$ Hz, 1H), 7.19–7.23 (m, 2H), 3.91 (d, $J = 3.5$ Hz, 1/2H), 3.81 (d, $J = 2.9$ Hz, 1/2H), 2.78–2.81 (m, 1H), 2.67–2.74 (m, 1/2H), 2.36 (s, 3H), 2.32–2.48 (m, 2H), 2.18–2.26 (m, 3/2H), 2.01–2.09 (m, 1H), 1.88–1.96 (m, 1H), 1.73 (m, 1/2H), 1.56–1.65 (m, 1H), 1.35–1.42 (m, 1/2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 209.5, 209.3, 196.9, 196.8, 152.7, 152.6, 145.7, 145.6, 140.1, 132.6, 132.5, 130.3, 130.2, 127.7, 127.5, 127.4, 127.3, 126.9, 126.8, 119.9, 119.5, 118.9, 118.7, 70.5, 70.0, 43.2, 42.7, 42.5, 42.3, 41.2, 41.0, 25.9, 25.7, 24.7, 21.7; HRMS (ESI) m/z : 484.0199, 486.0176 (Calcd for $\text{C}_{21}\text{H}_{20}\text{BrNNaO}_4\text{S}$ [$\text{M}+\text{Na}]^+$: 484.0194, 486.0174).

3. Supplementary References

- (S1) Hodson, H. F.; Madge, D. J.; Slawin, A. N. Z.; Widdowson, D. A.; Williams, D. J. *Tetrahedron* **1994**, *50*, 1899.
- (S2) Abe, T.; Kosaka, Y.; Kawasaki, T.; Ohata, Y.; Yamada, K. *Chem. Pharm Bull.*, **2020**, *68*, 555–558
- (S3) Hirao, S.; Yamashiro, T.; Kohira, K.; Mishima, N.; Abe, T. *Chem. Commun.* **2020**, *56*, 5139–5142.
- (S4) Zefirova, O. N.; Baranova, T. Y.; Ivanova, A. A.; Ivanov, A. A.; Zefirov, N. S. *Bioorg. Chem.* **2011**, *39*, 67.



JEOL

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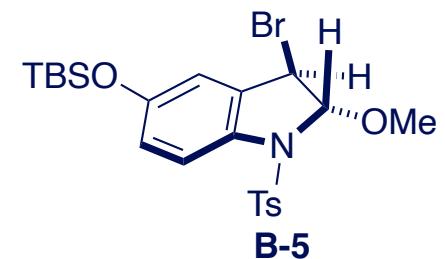
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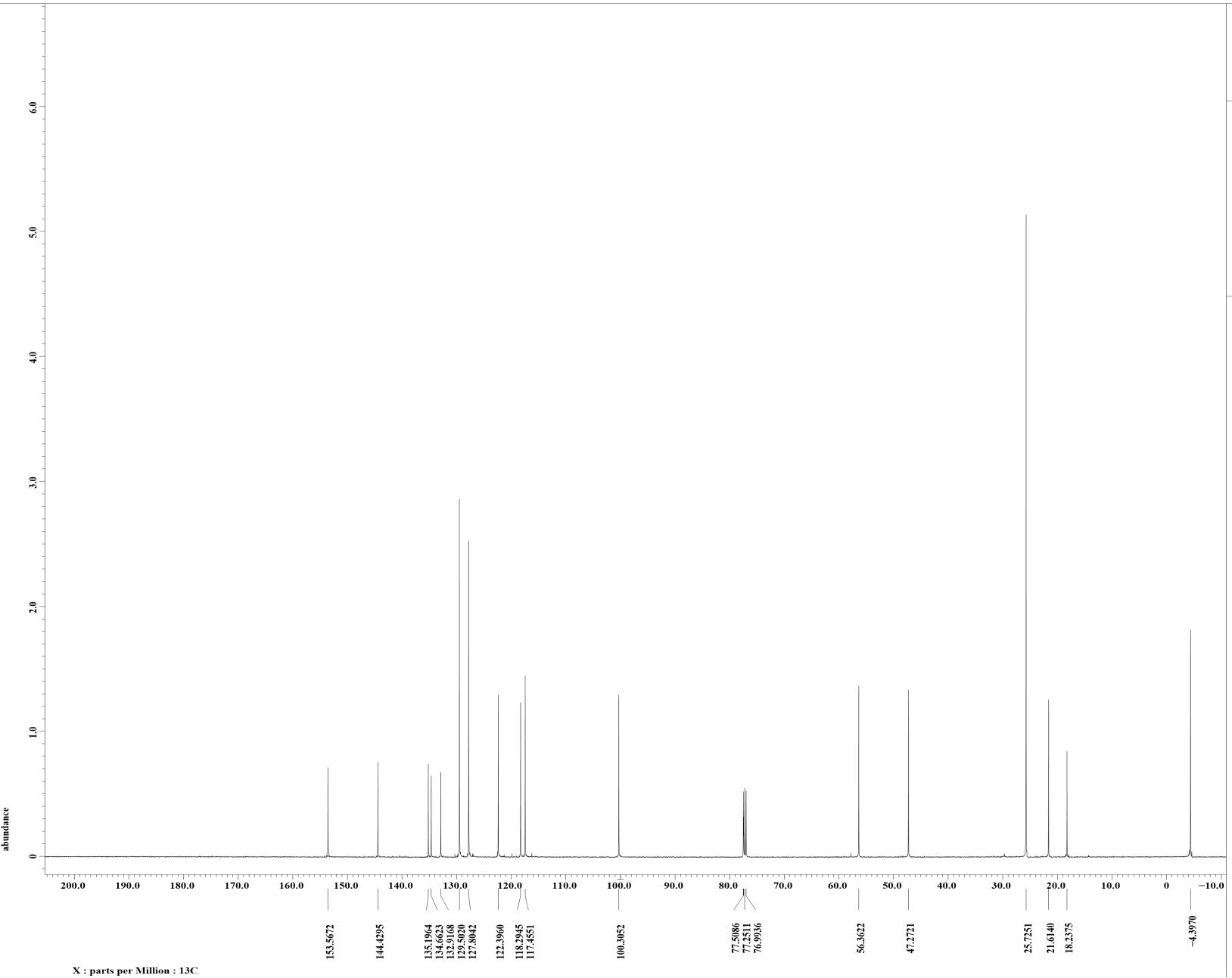
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DanFe_presat     = FALSE
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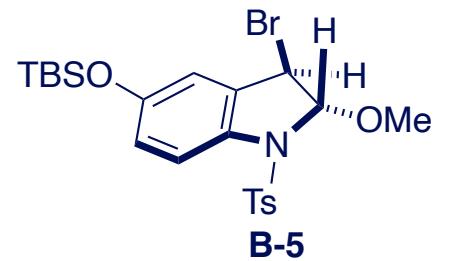
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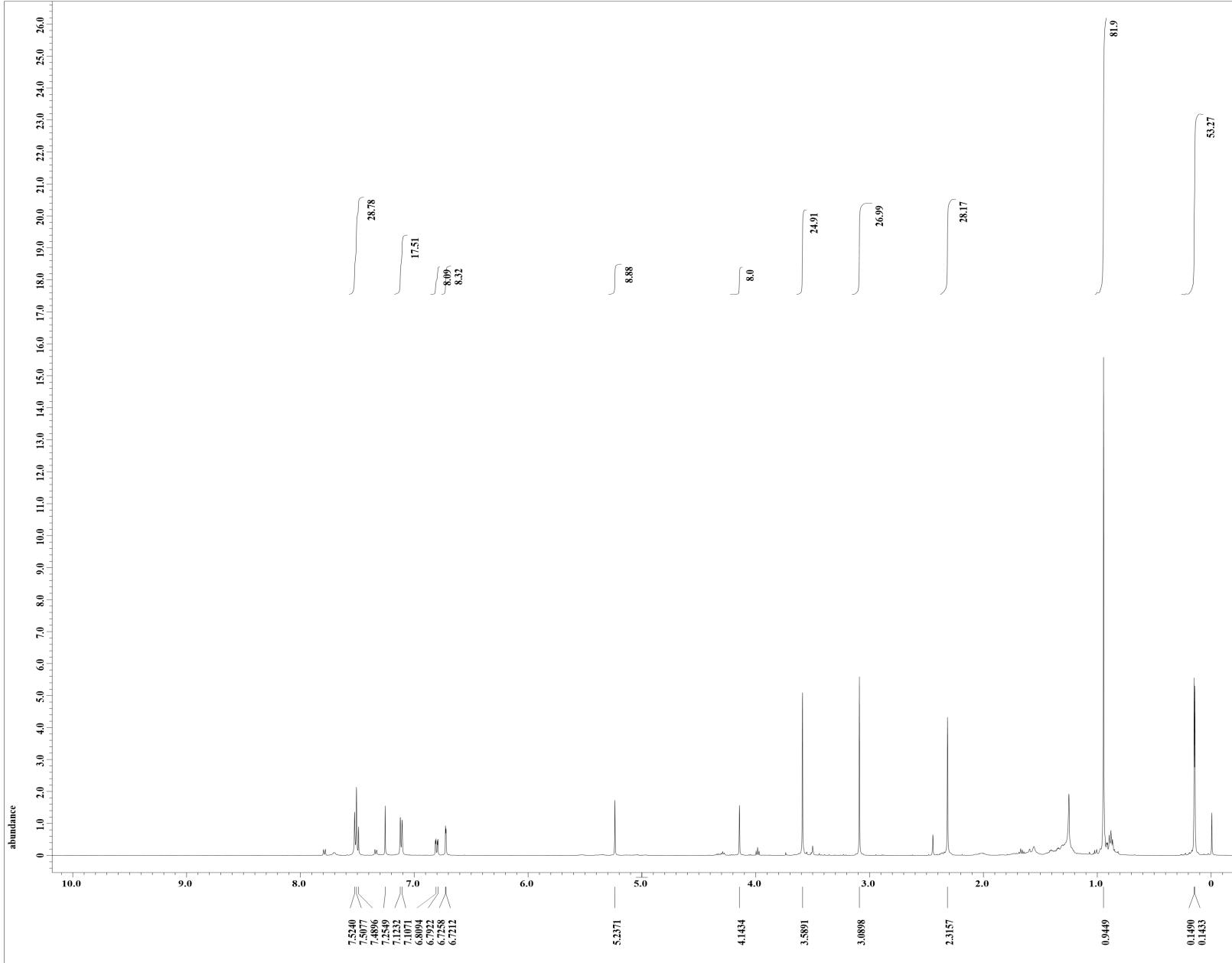




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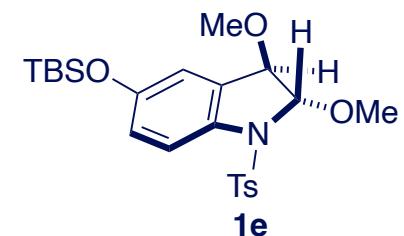


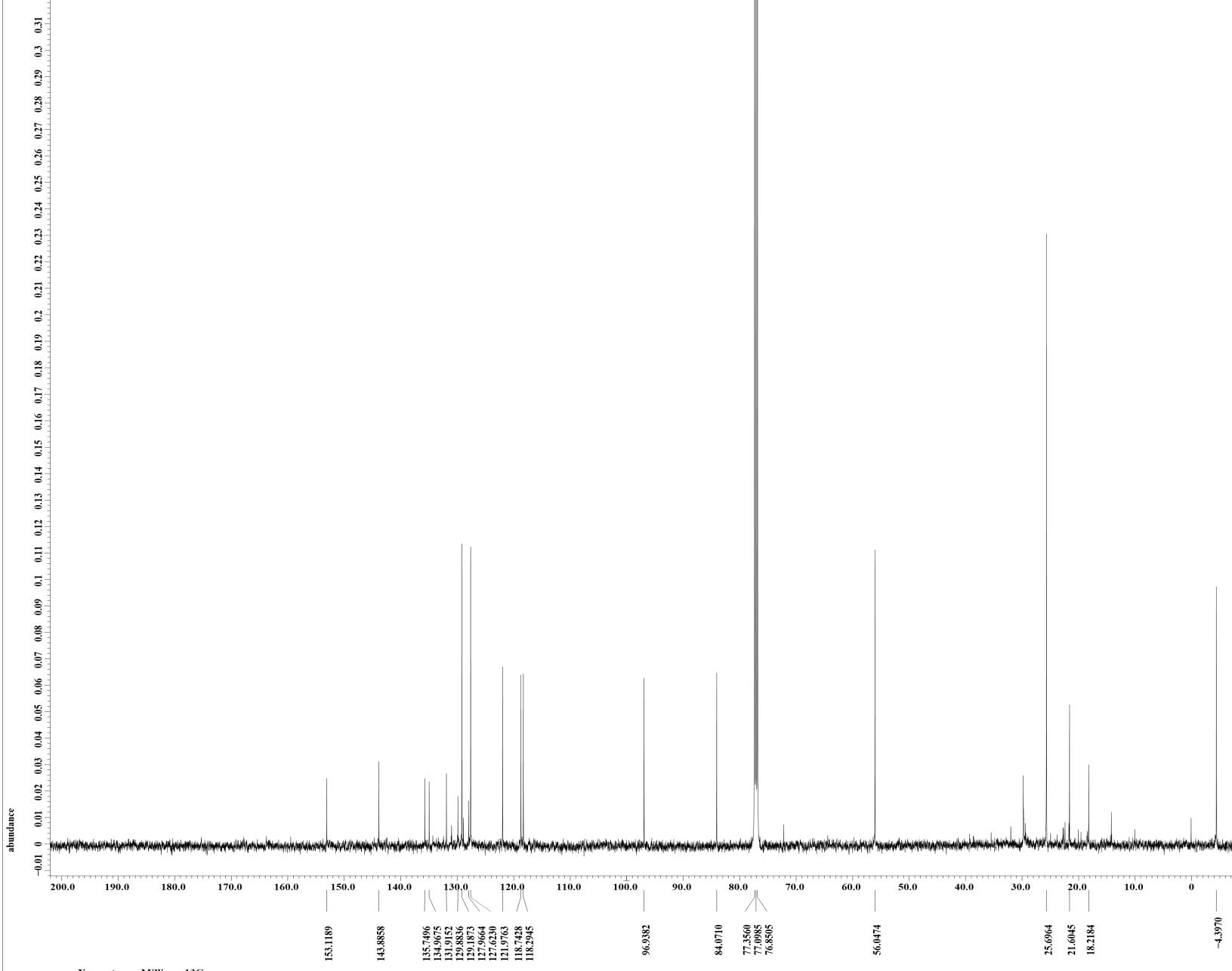
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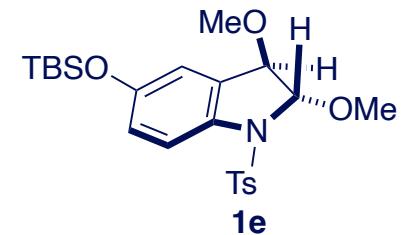


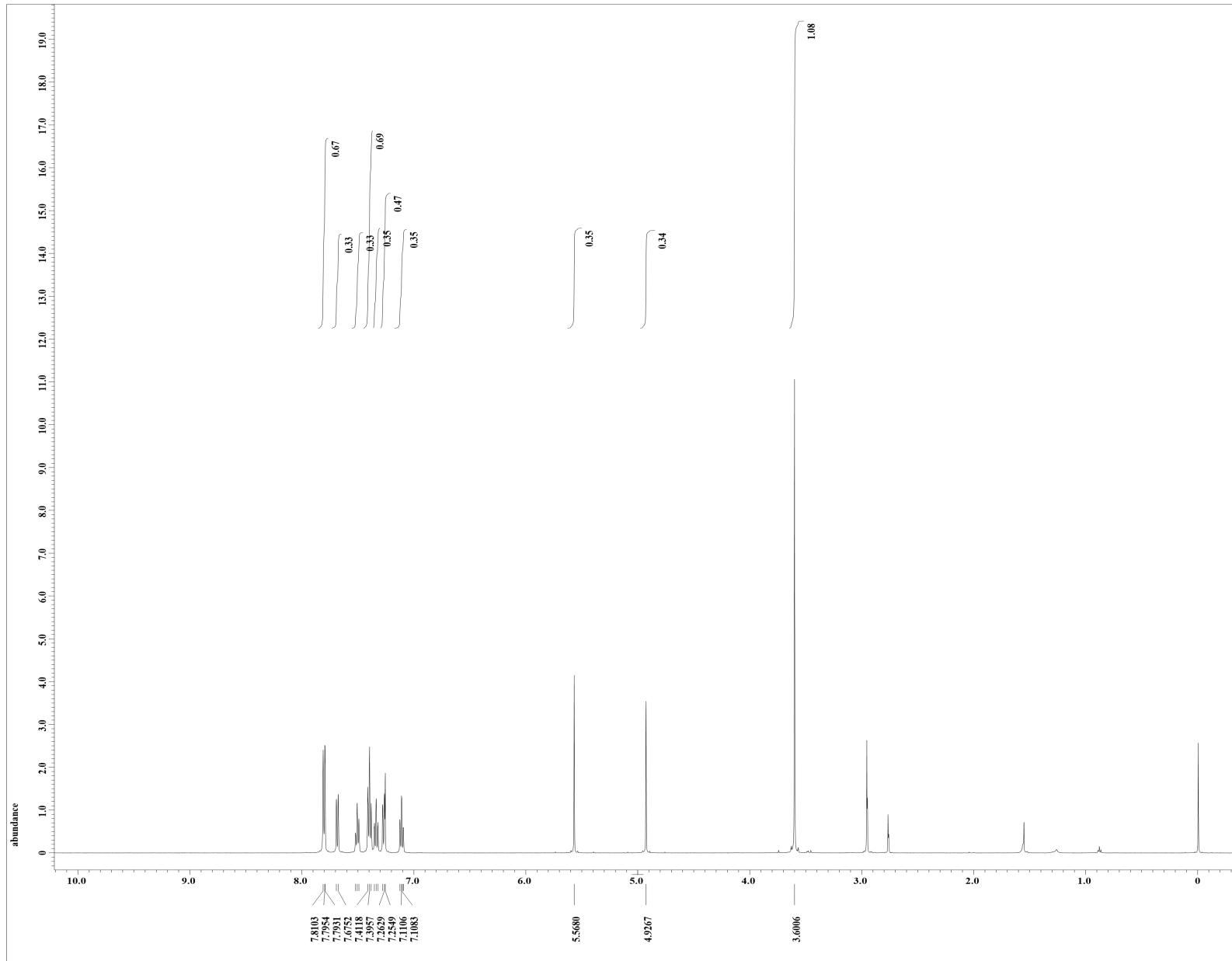


JEOL

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sample_id = S#616444
solvent = CHLOROFORM-D
creation_time = 15-JUN-2020 21:42:07
revision_time = 15-APR-2020 19:39:09
current_time = 15-APR-2020 19:39:57
comment = single pulse decouple
data_format = 1D COMPLEX
dim_size = 26214
dim_title =
dim_units = [ppm]
dimensions = X
site = ECA500
spectrometer = DELTA2_NMR
field_strength = 11.7473579[T] (500[MHz])
x_acq_duration = 0.83361792[s]
x_domain = 13C
x_freq = 125.76529768[MHz]
x_offset = 100[ppm]
x_pics = 32768
x_prescans = 1.19959034[Hz]
x_resolution = 39.3081761[kHz]
irr_domain = 1H
irr_freq = 500.15991521[MHz]
irr_offset = 0[ppm]
clipped = FALSE
mod_return = 1
scans = 3571
total_scans = 3571
x_90_width = 12.8[us]
x_acq_time = 0.83361792[s]
x_angle = 30[deg]
x_atn = 5.3[dB]
x_pulse = 4.26666667[us]
irr_atn_dec = 2.09[dB]
irr_dvdec = 31.0[dB]
irr_noise = WALTZ
decoupling = TRUE
initial_wait = 1[s]
noe = TRUE
noe_time = 2[s]
recv_r_gain = 58
relaxation_delay = 2[s]
repetition_time = 2.83361792[s]
temp_get = 23.2[dc]





JEOL

----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : TRUE : TRUE
machinephase
ppm
Derived from: Bs-ROBIN-1H-1.jdf

Filename = Bs-ROBIN-1H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#676313
Solvent_f = CHLOROFORM-D
Creation_time = 20-APR-2020 20:33:23
Revision_time = 8-APR-2020 18:49:54
Current_Time = 8-APR-2020 18:50:35

Comment = single_pulse
Data_format = 1D_NUCLEUSIMPLEX
Dim_Size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_kicks = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_pulsation = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scan_time = 8
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_ksize = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[us]
Irr_mode = Off
m1_size = 0%
Dante_presat = FALSE
Initial_wait = 1[s]
Recv_r_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.5[dC]

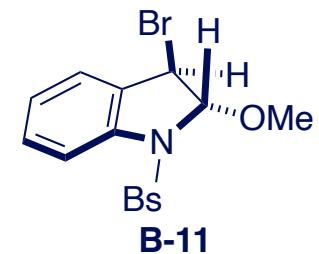
B-11

C[C@H](Br)[C@@H](CO)N(Bs)c1ccccc1

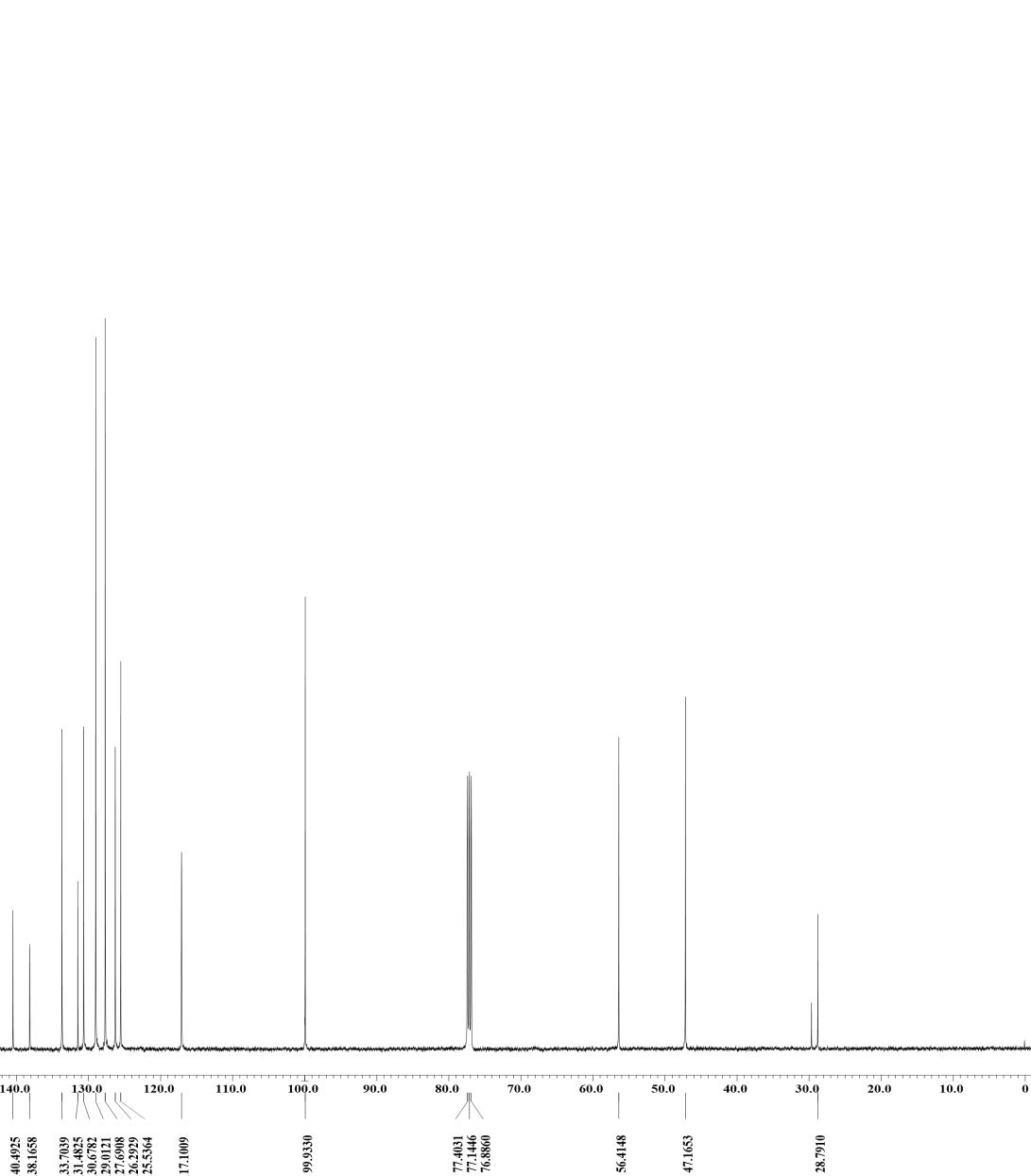


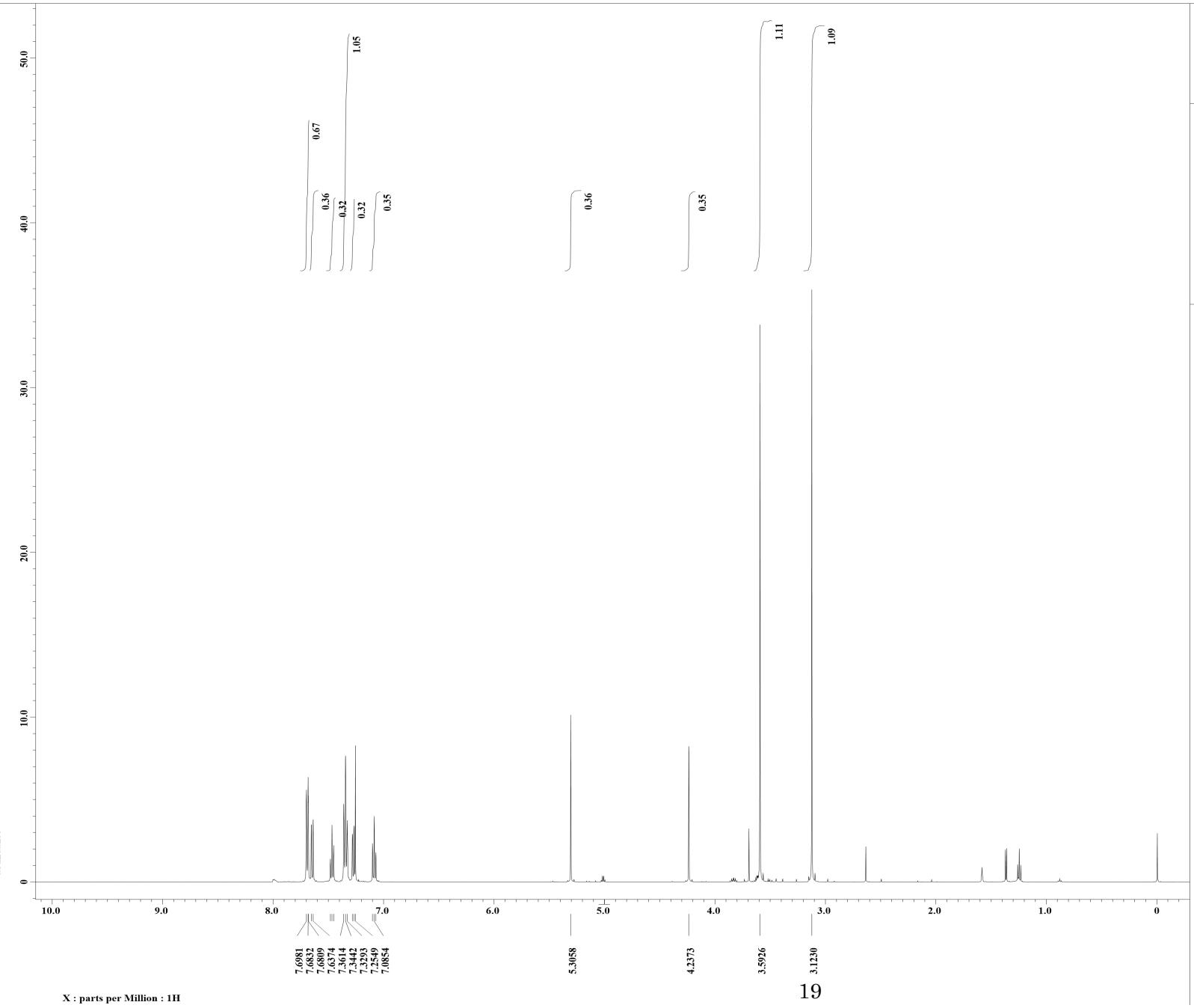
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill1 : TRUE
fit : 1 : TRUE
machinephase
PPM
Derived from: TA200408-4.jdf

Filename = TA200408-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 200408-6
SolventF = CHLOROFORM-D
Creation_time = 8-APR-2020 18:22:53
Revision_time = 8-APR-2020 19:00:34
Current_time = 8-APR-2020 19:01:18
Content = Bs-ROBIN-13C
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = 1
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M)
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_realsans = 1
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clip_end = FALSE
Mod_return = 2666
Scans = 2666
Total_scans = 2666
X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Rte_time = 1[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.4[dC]



X : parts per Million : 13C



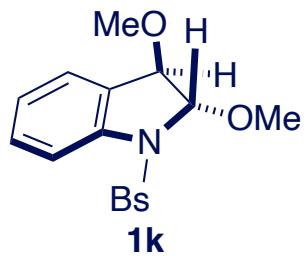


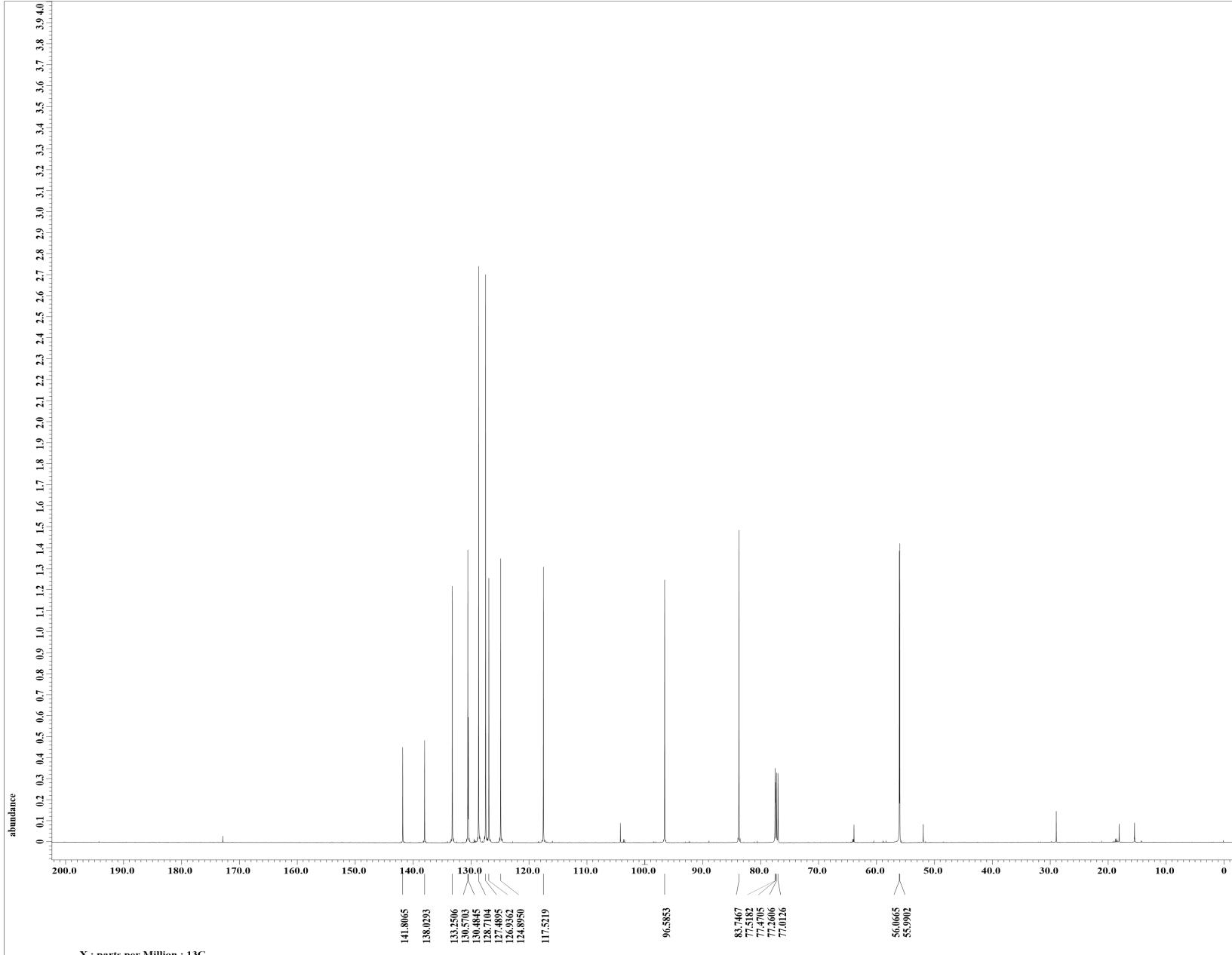
JEOL

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sepx : 0.2[Hz] : 0.0[s]
trig_sd[ms] : 0[s] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: Bs-DiMeOIN-1.jdf

Filename = Bs-DiMeOIN-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#644967
Solvent = CHLOROFORM-D
Creation_time = 26-JUN-2020 19:41:04
Revision_time = 10-APR-2020 17:59:35
Current_time = 10-APR-2020 18:00:51
Comment = Bs-DiMeOIN
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title =
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_kHz = 1Hz
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_resolution = 0.57277737[Hz]
X_sw = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = TRUE
Mod_return = 8
Scans = 8
Total_scans = 8
X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_kHz = 1Hz
X_atten = 3.4[dB]
X_pulse = 6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recycle_gal = 0
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.4[dC]





JEOL

---- PROCESSING PARAMETERS ----

dc_balance : 0 : FALSE
 sexp : 2.0[Hz] : 0.0[s]
 trapezoid3 : 0[%] : 80[%] : 100[%]
 zerofill : 1 : TRUE : TRUE
 ffit : 1 : TRUE : TRUE
 machinephase
 Ppm

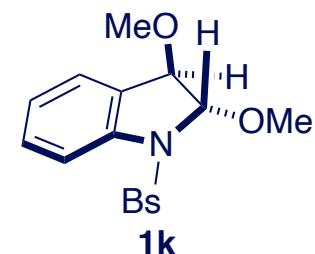
Derived from: Bs-DiMeOIN-2.jdf

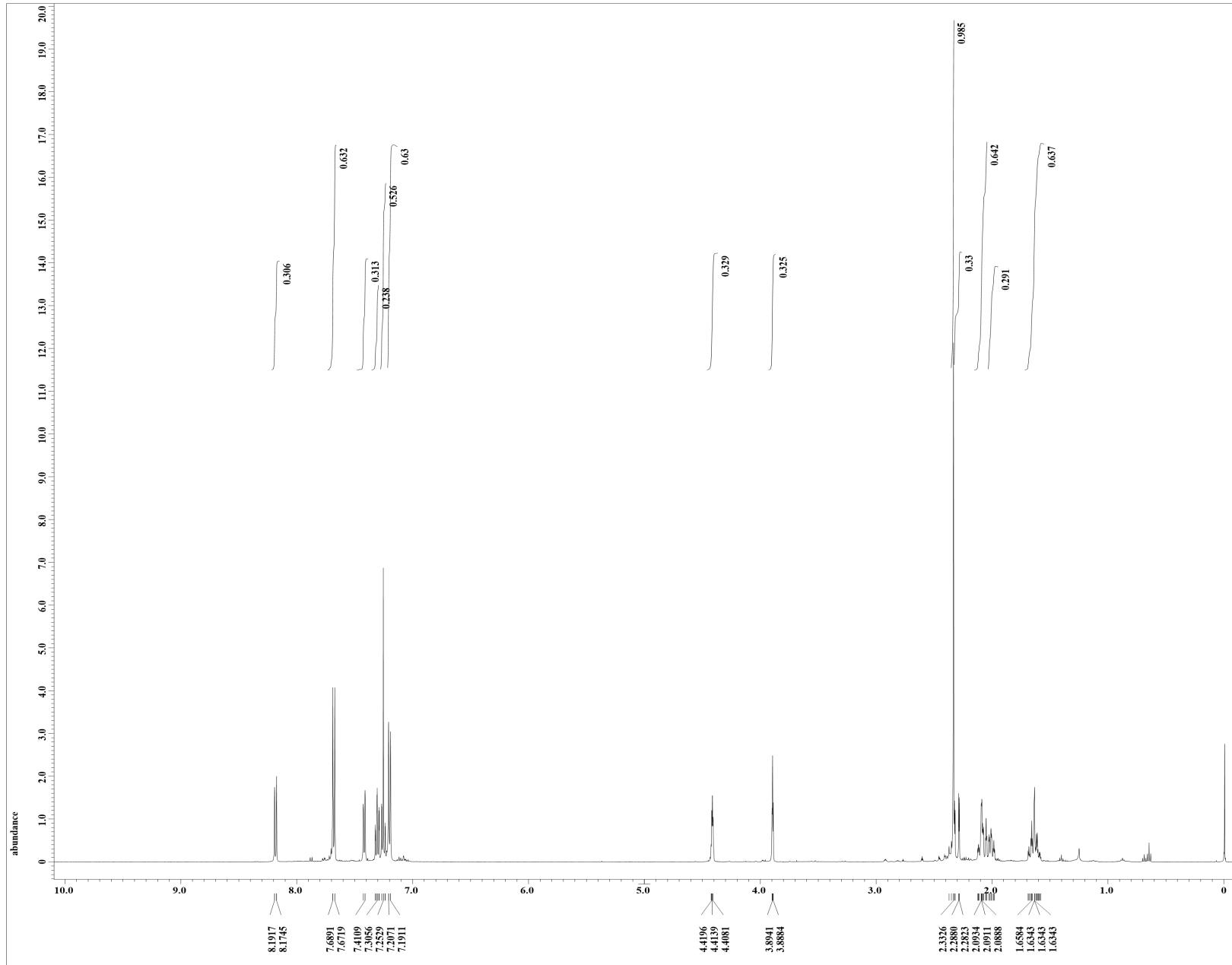
Filename = Bs-DiMeOIN-5.jdf
 Author =
 Experiment = single_pulse_dec
 Sample_id = S#647870
 Solvent = CHLOROFORM-D
 Creation_time = 27-JUN-2000 16:19:23
 Revision_time = 11-APR-2020 14:36:10
 Current_Time = 11-APR-2020 14:37:12

Comment = Bs-DiMeOIN
 Data_format = 1D COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA500
 Spectrometer = DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz])
 X_acq_duration = 0.83361792[s]
 X_domain = 13C
 X_freq = 125.76529768[MHz]
 X_offset = 100[ppm]
 X_p0 = 2768
 X_prescans = 4
 X_resolution = 1.19959034[Hz]
 X_sweep = 39.3081761[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_Offset = 5.0[ppm]
 Clipped = TRUE
 Mod_return = 1
 Scans = 26126
 Total_scans = 26126

X_90_width = 12.8[us]
 X_acq_time = 0.83361792[s]
 X_angle = 30[deg]
 X_atn = 5.3[dB]
 X_pulse = 4.666667[us]
 Irr_atn_dec = 21.09[dB]
 Irr_atn_noe = 21.09[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Irrital_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 56
 Relaxation_delay = 2[s]
 Repetition_time = 2.83361792[s]
 Temp_get = 23.8[dC]





```

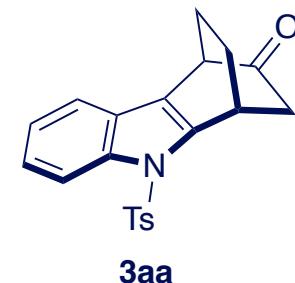
Filename = TN200316-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#571541
Solvent = CHLOROFORM-D
Creation_time = 16-MAR-2020 14:51:58
Revision_time = 16-MAR-2020 16:03:30
Current_time = 16-MAR-2020 16:03:47

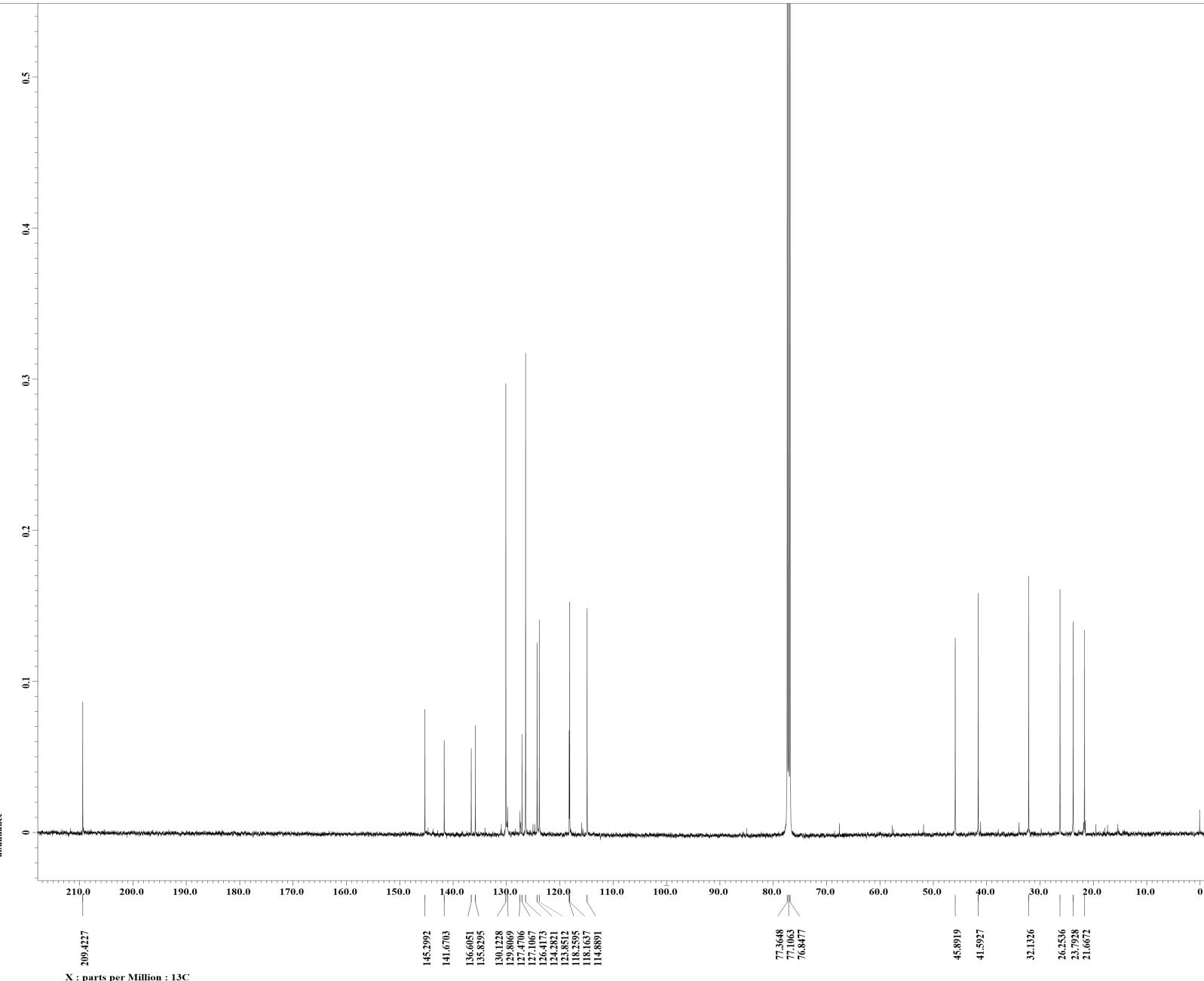
Content = R=H-cyclohexenone
Data_format = 1D_COMPLEX
Dim_size = 13107
Dim_table =
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_p0 = 16384
X_pscans =
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Ifr_domain = 1H
Ifr_freq = 495.13191398[MHz]
Ifr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scan = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45
X_dpp = 3[dB]
X_pulse = 5.65[us]
Ifr_mode = Off
Tri_mode = Off
Data_preset = FALSE
Init_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.2[dC]

```





```

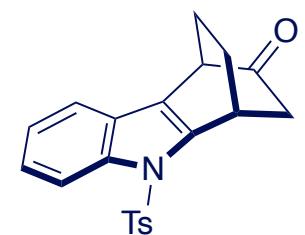
Filename      = T200316-4.jdf
Author       =
Experiment   = single_pulse_dec
Sample_id    = S#572819
Solvent      = CHLOROFORM-D
Creation_time = 17-MAR-2020 07:42:41
Revision_time = 17-MAR-2020 08:45:34
Current_time  = 17-MAR-2020 08:46:10

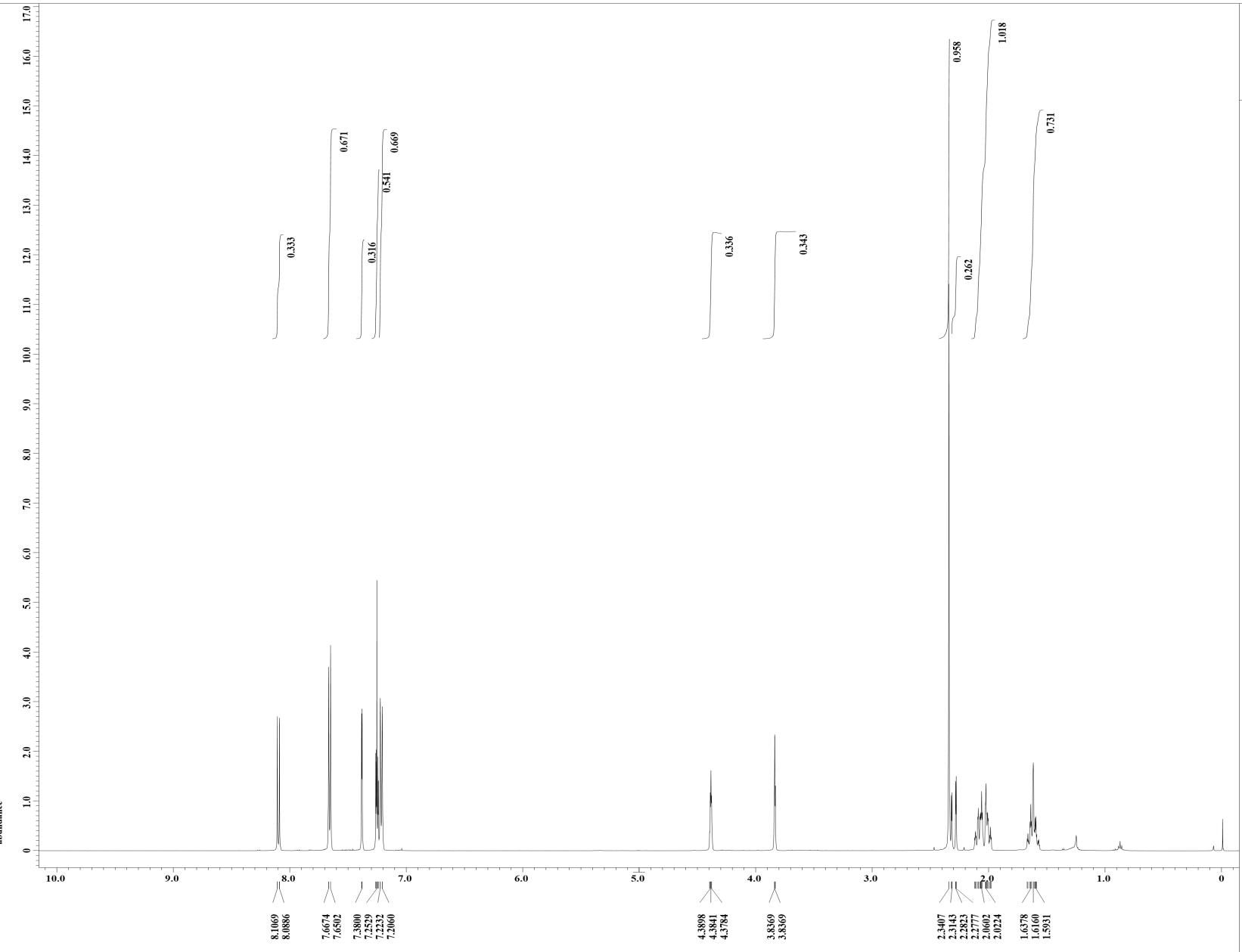
Content      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title   = 13C
Dim_units   = [ppm]
Dimensions   =
Site         = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 0.8388608[s]
X_domain      = 13C
X_freq         = 124.5010059[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans    = 4
X_resolution   = 1.020929[Hz]
X_sweep        = 39.0625[kHz]
Irr_domain    = 1H
Irr_freq       = 495.13191398[MHz]
Irr_offset     = 5[ppm]
Clipped       =
Mod_nreturn   =
Scan_start    = 21329.0
Scan_end      = 21329.0
Total_scans   = 21329.0

X_90_width   = 10.1[us]
X_acq_time   = 0.8388608[s]
X_tdcle      = 0[decade]
X_attn       = 9.5[dB]
X_pulse       = 3.366666667[us]
Irr_atn_dec  = 21.51[dB]
Irr_atn_noce = 21.51[dB]
Irr_noise    = WAVEZ
Decoupling   = TRUE
Initial_wating = 0
Initial_wait  = 1[s]
Noe          = TRUE
Noe_time     = 2[s]
Recv_gain    = 60
Relaxation_delay = 2[s]
Repetition_time = 0.8388608[s]
Temp_get     = 23.9[dC]

```





JEOL

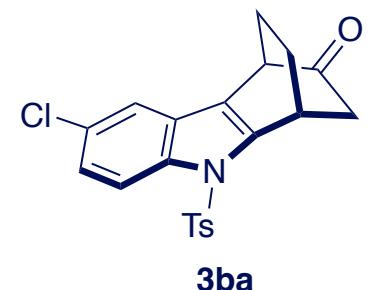
```

Filename = TA200318-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#495478
Solvent = CHLOROFORM-D
Creation_time = 18-MAR-2020 12:53:52
Revision_time = 18-MAR-2020 13:50:10
Current_Time = 18-MAR-2020 13:50:40

Content = 5Cl-cyclohexenone
Data_format = 1D COMPLEX
Dim = 107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_preset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
ClipFreq = 5[ppm]
ClipOffset = 5[ppm]
Mod_return = FALSE
Scans = 1
Total_scans = 8
X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atm = 3.3[db]
X_pulse = 5.65[us]
X_rf_mode = Off
X_rf_time = Off
Danfe_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 29.6[dC]

```



```

Filename = T200318-8.jdf
Author =
Experiment =
Sample_id = S496811
Solvent =
Creation_time = 18-MAR-2020 14:44:35
Revision_time = 18-MAR-2020 15:38:42
Current_time = 18-MAR-2020 15:39:22

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title =
Dim_units = [ppm]
Dimensions =
Site = X
Spectrometer = ECA 500
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain =
X_freq =
X_offset =
X_points =
X_prescans =
X_pulsation =
X_sweep =
Irr_domain =
Irr_freq =
Irr_offset =
Clipped =
Mtx_return =
Scans =
Total_scans =
X_90_width =
X_acq_time =
X_nale =
X_atn =
X_pulse =
Irr_atn_dec =
Irr_atn_hoc =
Irr_noise =
Decoupling =
Initial_wait =
Noe =
Noe_time =
Recvr_gain =
Relaxation_delay =
Repetition_time =
Temp_get =

```

= 0.8388608[s]

= 13C

= 124.5010059[MHz]

= 100[ppm]

= 32768

= 4

= 1.1220229[Hz]

= 39.0625[kHz]

= 1H

= 495.13191398[MHz]

= 5[ppm]

= FALSE

= 1

= 2311

= 2311

= 10.1[us]

= 0.8388608[s]

= 30[deg]

= 9.5[dB]

= 3.366666667[us]

= 21.51[dB]

= 21.51[dB]

= WALTZ

= TRUE

= 1[s]

= TRUE

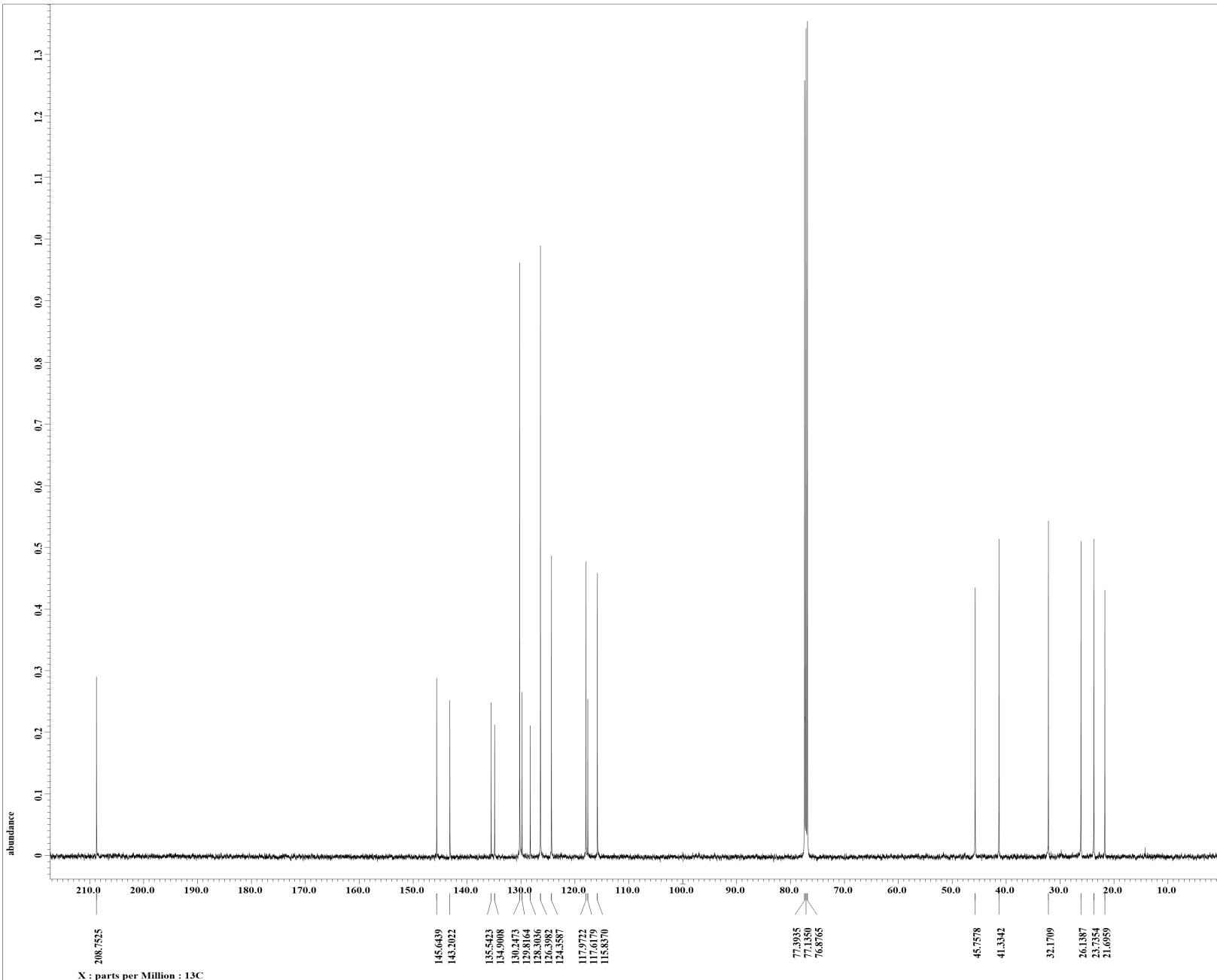
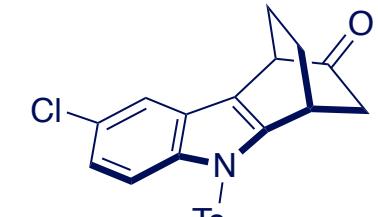
= 2[s]

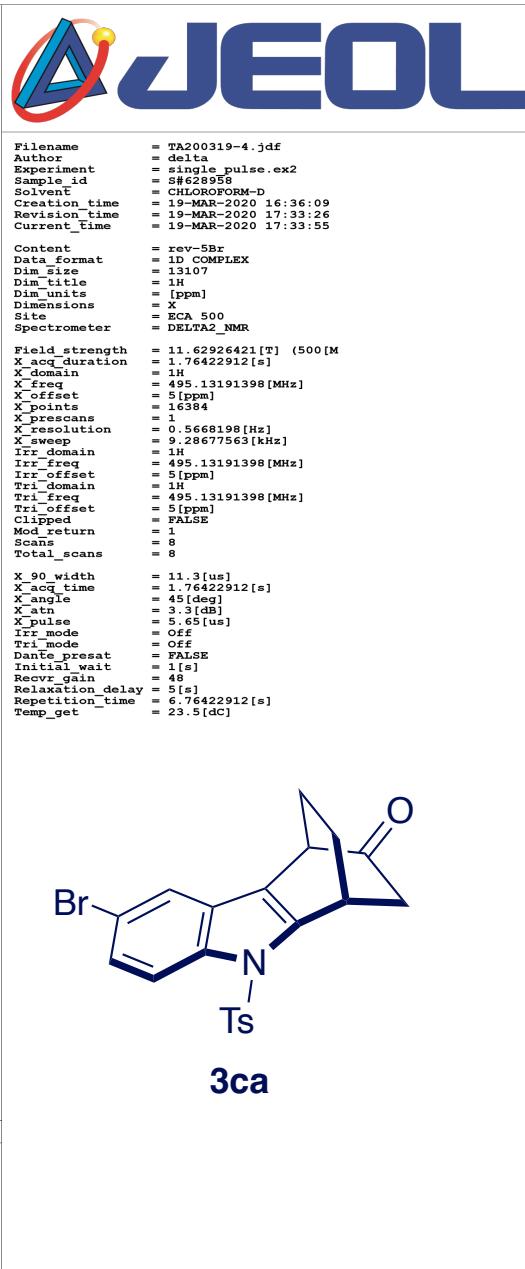
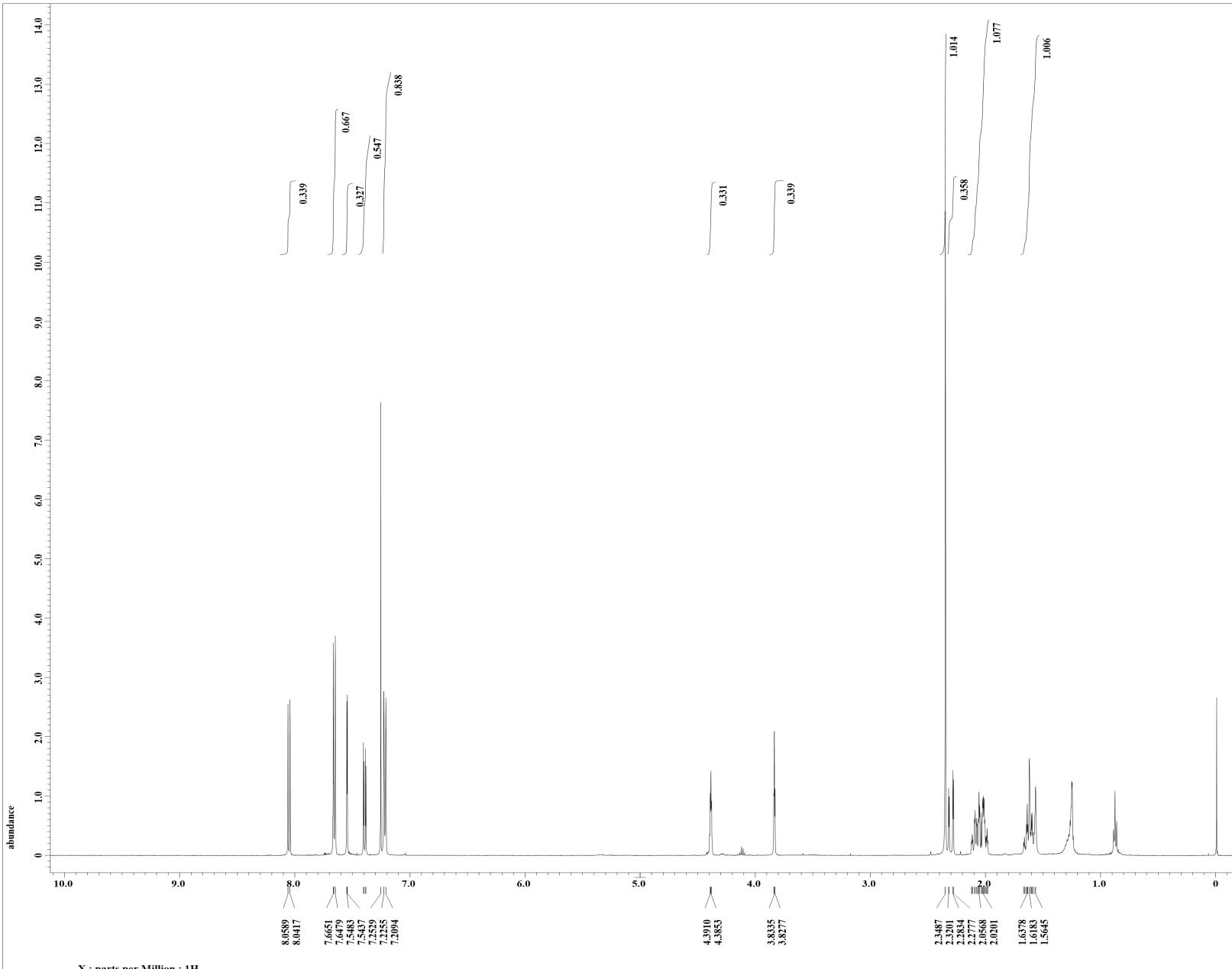
= 60

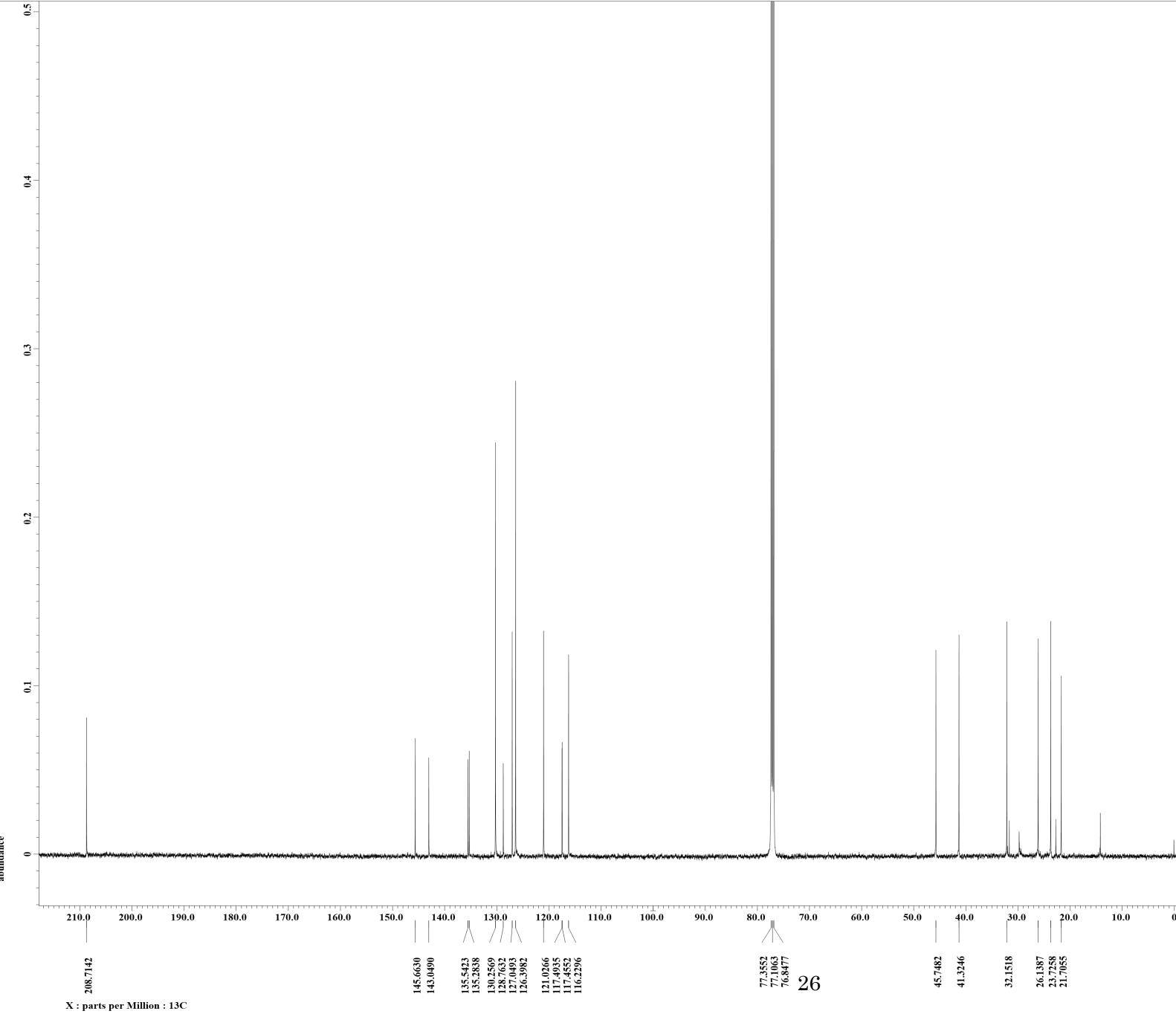
= 2[us]

= 2,8388608[s]

= 24.4[dC]







```

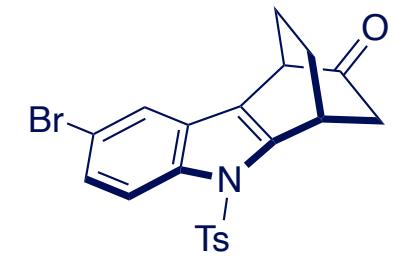
Filename          = TA200319-4.jdf
Author           = delta
Experiment       = single_pulse_dec
Sample_id        = S#630158
Solvent          = CHLOROFORM-D
Creation_time    = 20-MAR-2020 11:15:56
Revision_time    = 20-MAR-2020 12:10:24
Current_time     = 20-MAR-2020 12:11:28

Content          = rev-5br
Data_format      = 1D COMPLEX
Dim_size         = 26214
Dim_title        =  $^{13}\text{C}$ 
Dim_units        = [ppm]
Dimensions       = X
Site             = ECA 500
Spectrometer     = DELTA2_NMR

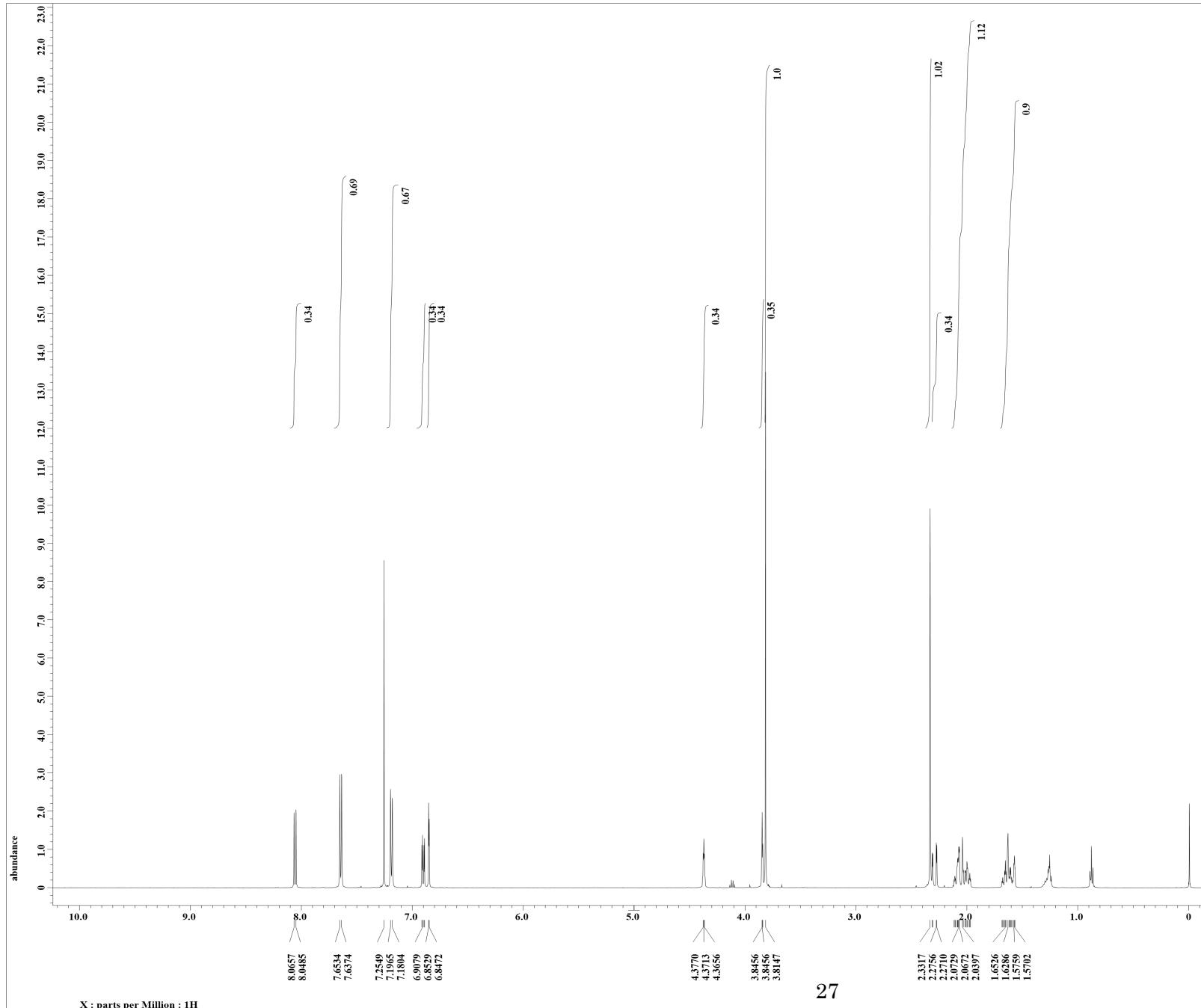
Field_strength   = 11.62926421[T] (500[M]
X_acq_duration = 0.8388608[s]
X_domain        = 162.510059
X_kexp           = 124.5010059[MHz]
X_offset         = 100[ppm]
X_points         = 32768
X_prescans       = 4
X_resolution     = 1.1920929[Hz]
X_sweep          = 39.0625[kHz]
Irr_domain      = 1H
Irr_fq            = 4.95.13191398[MHz]
Irr_offset       = 5[ppm]
Clipped          = TRUE
Mod_return       = 1
Scans            = 23637
Total_scans      = 23637

X_90_width      = 10.1[us]
X_acq_time      = 0.8388608[s]
X_angle          = 30[deg]
X_atn            = 9.5[db]
X_pulse          = 3.36666667[us]
Irr_atn_dec     = 21.51[db]
Irr_atn_noe     = 21.51[db]
Irr_noise        = WALTZ
Decoupling       = TONE
Initial_wait     = 1[s]
Noe              = TRUE
Noe_time         = 2[s]
Recvr_gain       = 60
Relaxation_delay = 2[s]
Repetition_time  = 2.8388608[s]
Temp_get         = 24.3[dC]

```



3ca



```
----- PROCESSING PARAMETERS -----  
dc_balance : 0 : FALSE  
sexp : 0.2 [Hz] : 0.01[s]  
trapezoid3 : 0 [%] : 80 [%] : 100 [%]  
zerofill : 1  
fft : 1 : TRUE : TRUE  
machinephase  
ppm  
  
Derived from: TA2020-03235-MeO-1.jd
```

Derived from: TA2020-0323-5MeO-1.jdf

```

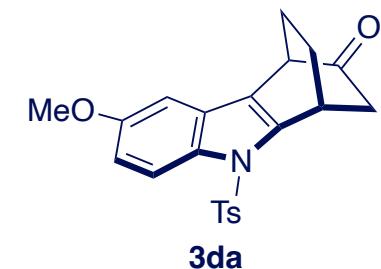
Filename      = RA2020-0323-5Meo-4.jd
Author        = delta
Experiment   = single_pulse.ex2
Sample_id    = S#574236
SolventT     = CHLOROPHENYL-D
Creation_time = 8-JUN-2000 17:43:49
Revision_time = 23-MAR-2020 16:07:27
Current_Time = 23-MAR-2020 16:07:53

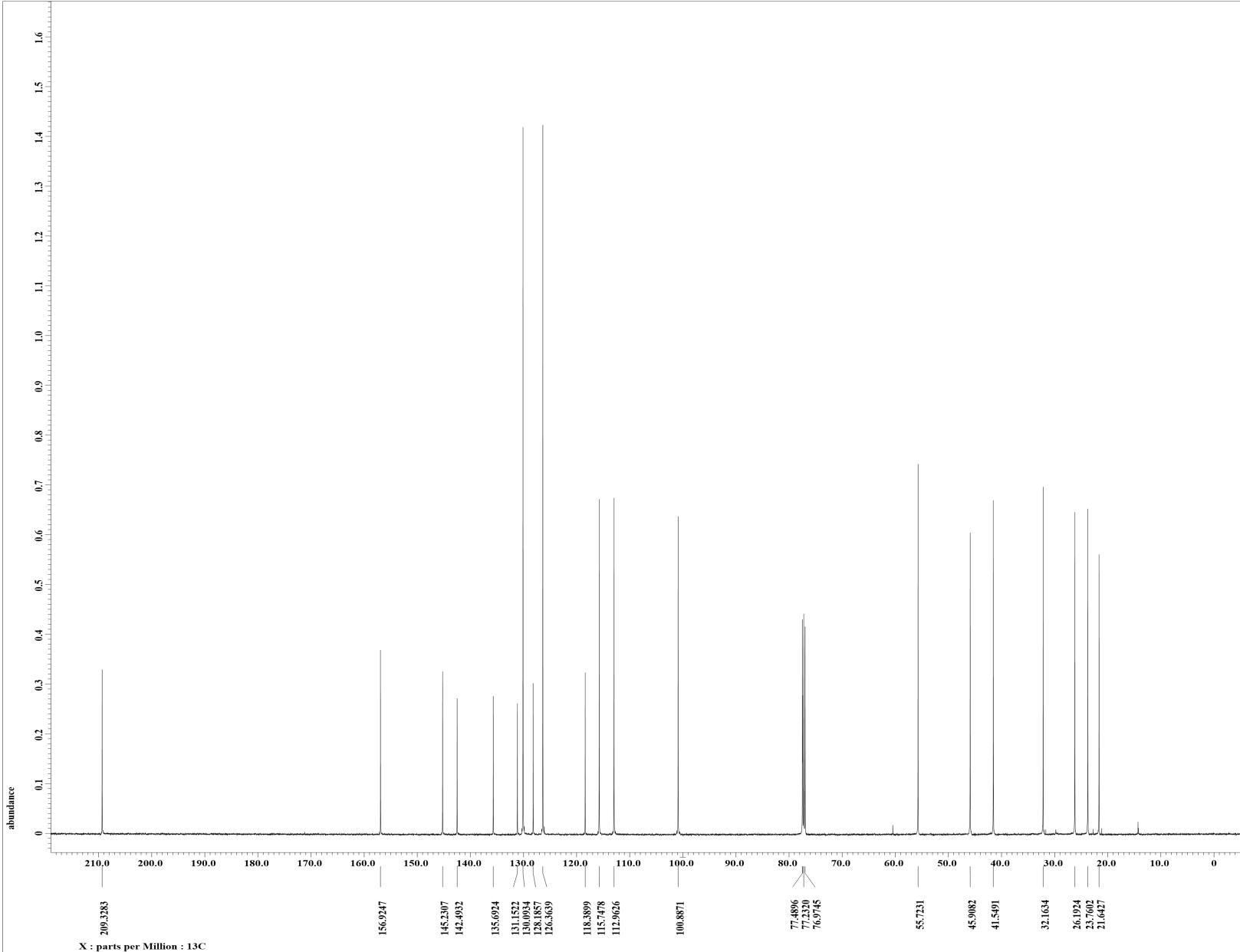
Comment       = single_pulse
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title   = 1H
Dim_units   = [ppm]
Dimensions  = X
Site         = ECA500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 1.74587904[s]
X_label       = 1H
X_freq        = 500.15991521[MHz]
X_offset      = 5.0[ppm]
X_points      = 16384
X_prescans   = 1
X_resolution = 0.57277737[Hz]
X_sweep       = 3.48348438[kHz]
Irr_domain   = 1H
Irr_freq     = 500.15991521[MHz]
Irr_offset    = 5.0[ppm]
Tri_domain   = 1H
Tri_freq      = 500.15991521[MHz]
Tri_offset    = 5.0[ppm]
COffset       = FALSE
Modulation   = 1
Scans         = 8
Total_scans  = 8

X_90_width   = 12[us]
X_acq_time   = 1.74587904[s]
X_label       = 45[deg]
X_atn        = 3.4[dB]
X_pulse      = 6[us]
Irr_mode     = Off
Tri_mode     = Off
Dante_presat = FALSE
Irr_p1        = 1[s]
Recv_gain    = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Tempo_get    = 22.7[°C]

```

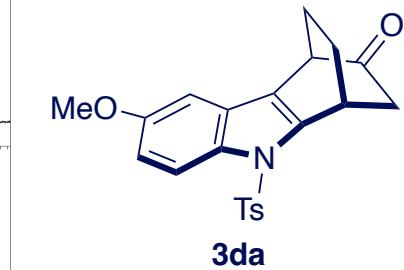


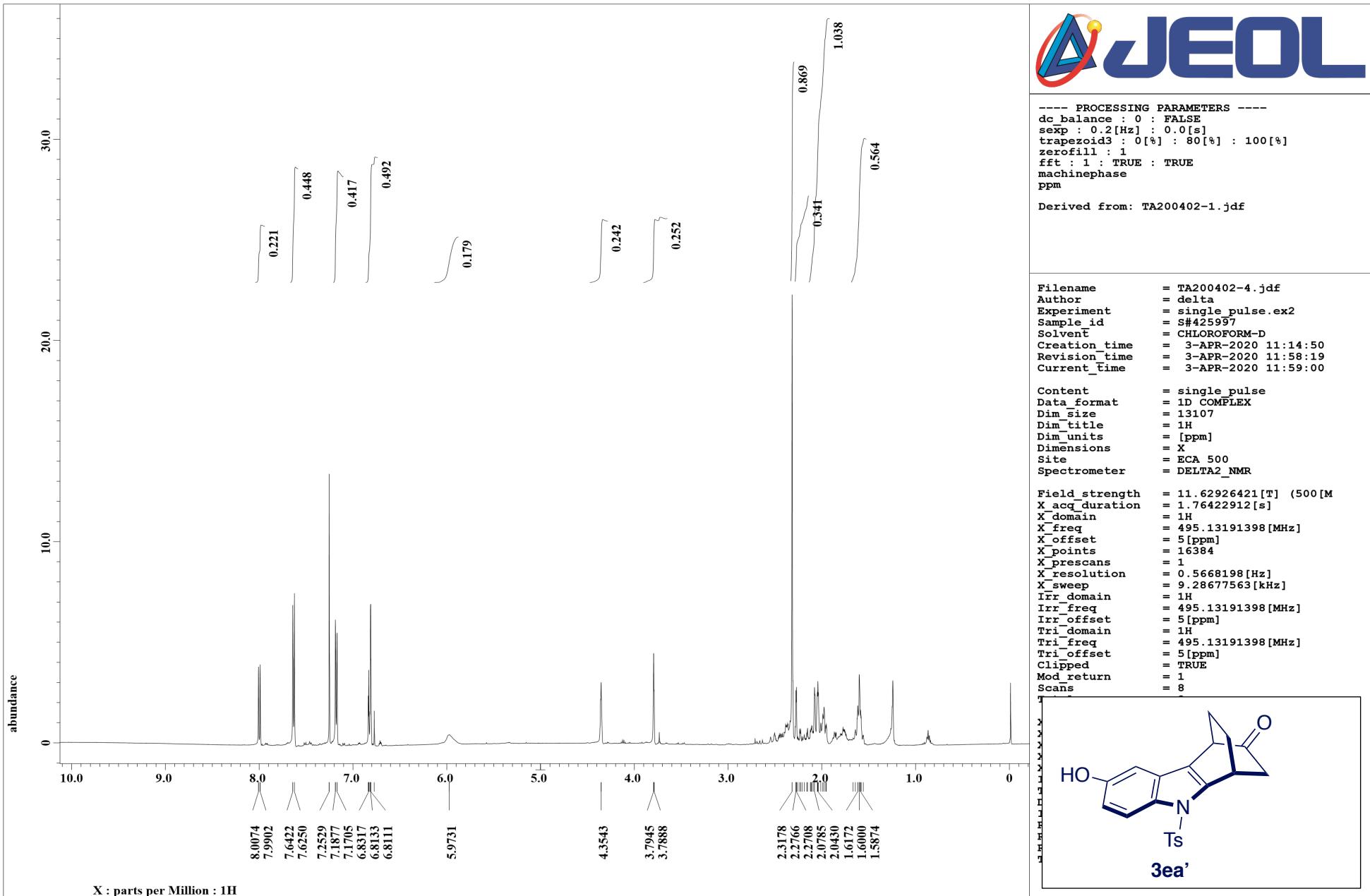


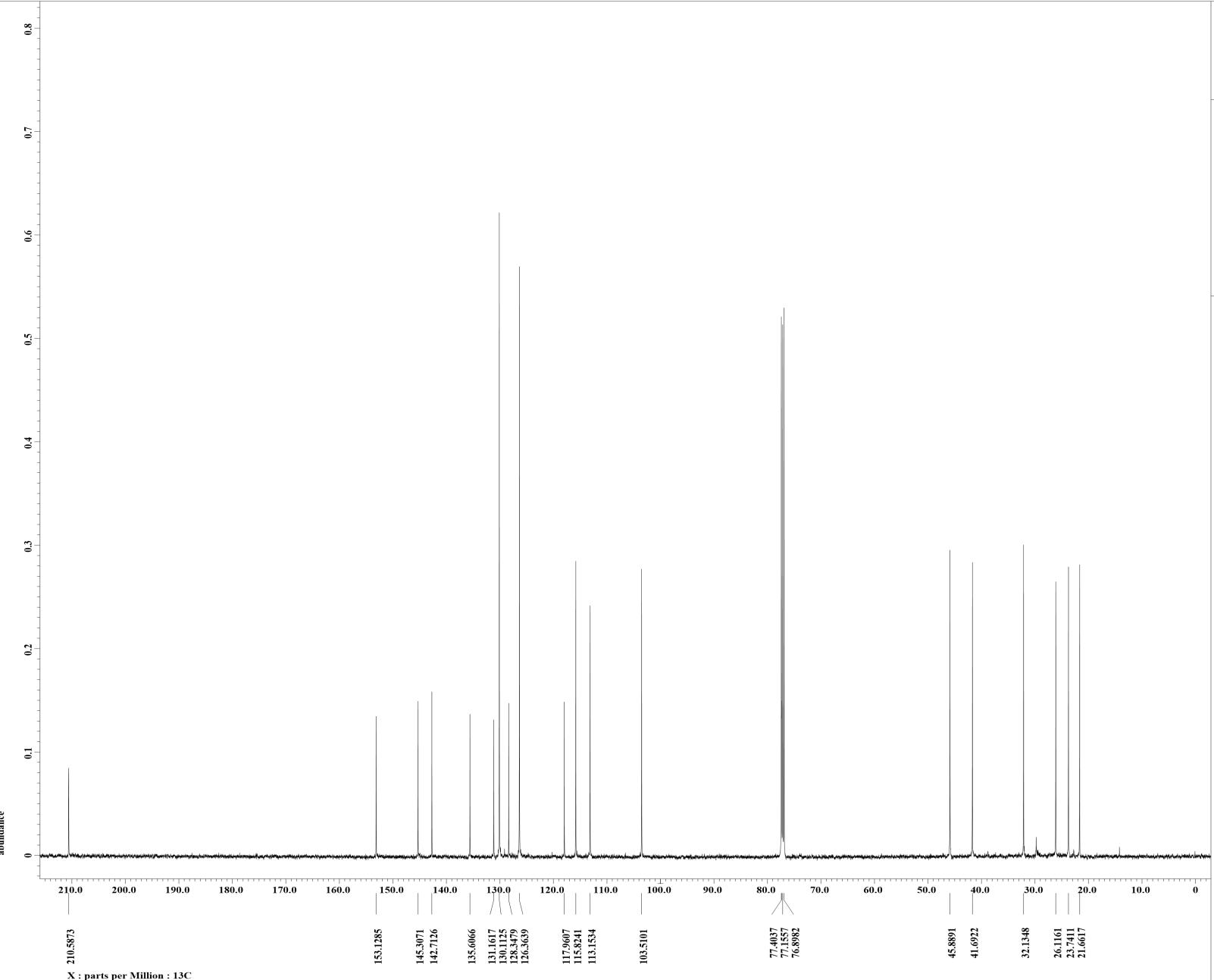
JEOL

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoidal : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TA2020-0323-5MeO-6.jdf

Filename = TA2020-0323-5MeO-8.jd
Author = delta
Experiment = single_pulse_dec
Sample_id = S03950
Solvent_F = CHLOROFORM-D
Creation_time = 8-JUN-2000 20:54:33
Revision_time = 23-MAR-2020 19:09:09
Current_time = 23-MAR-2020 19:10:16
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_tic = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_cpmag = 1
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 3527
Total_scans = 3527
X_90_width = 12.8[us]
X_acqtime = 0.83361792[s]
X_dppm = 5.3[ppm]
X_atten = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atten_dec = 21.09[dB]
Irr_atten_noe = 5.09[dB]
Irr_dppm = 1000[dB]
Decoupling = TRUE
Initial_wait = 1[s]
Noe_time = TRUE
Noe_time = 2[s]
Recov_gain = 2[dB]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.6[dC]





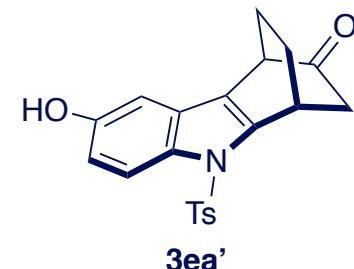


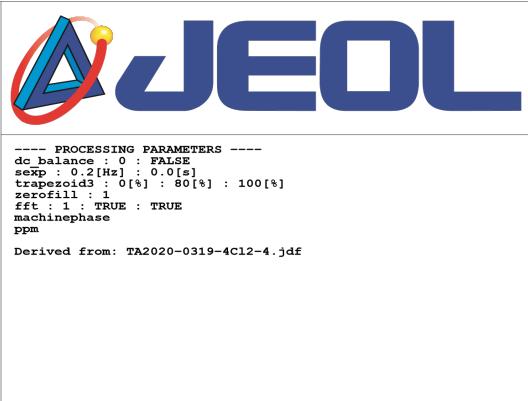
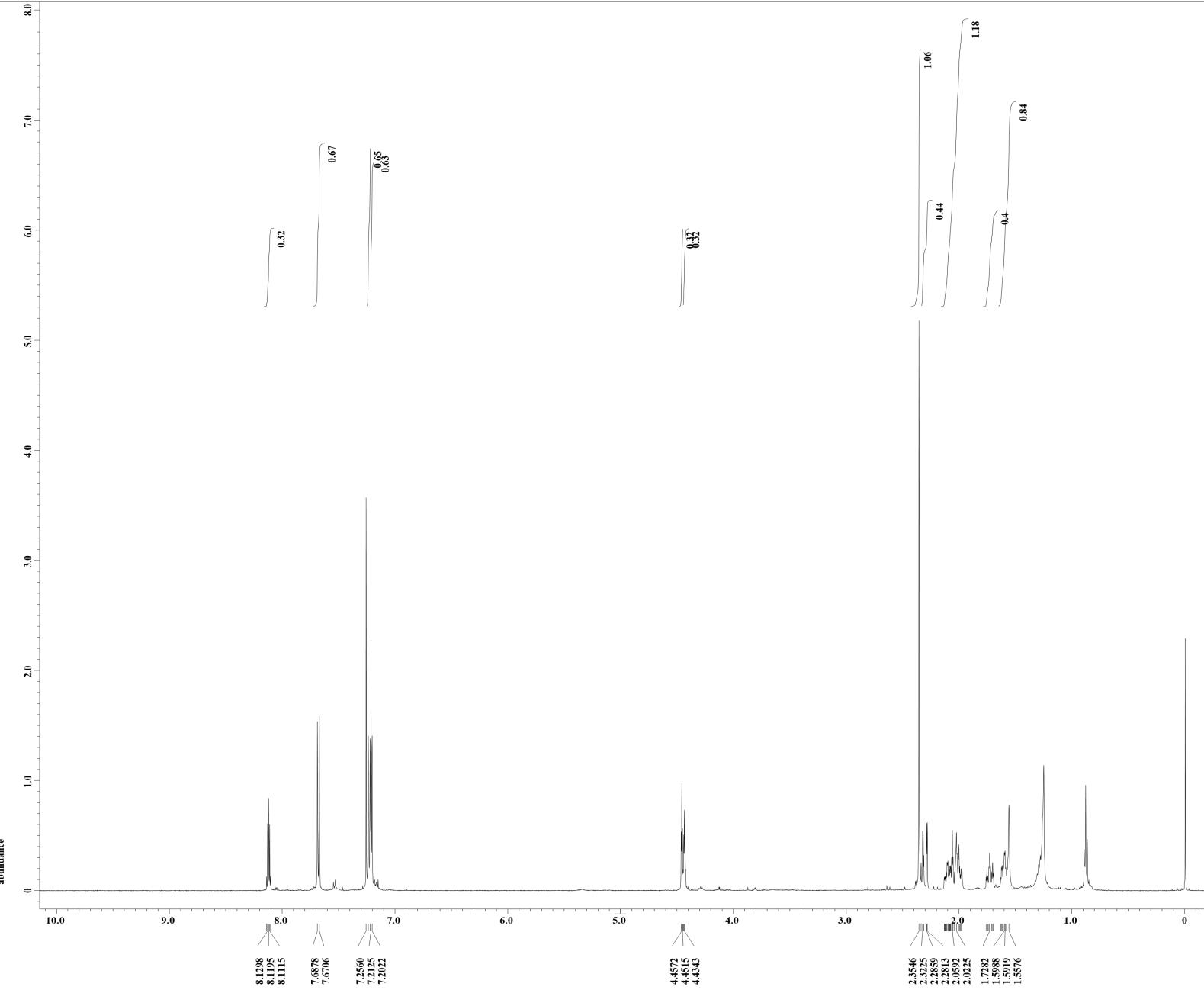
JEOL

---- PROCESSING PARAMETERS ----
dc_balance = 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0404-2.jdf

Filename = TA2020-0404-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#600995
Solvent = CHLOROFORM-D
Creation_time = 2020-04-04 20:27:59
Revision_time = 4-APR-2020 18:43:00
Current_Time = 4-APR-2020 18:43:30
Comment = single pulse decouple
Data_format = COMPLEX
Dim_Size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA_2_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_label = 13C [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_dchain = 1H
Irr_fref = 300.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 2548
Total_scans = 2548
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 21.0966667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_hoe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Nce = 2[dB]
Nce_time = 2[s]
Recvr_gain = 56
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.9[dC]

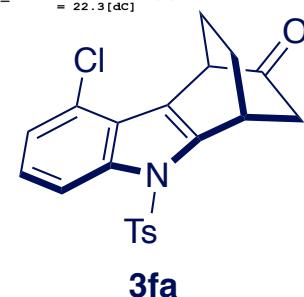


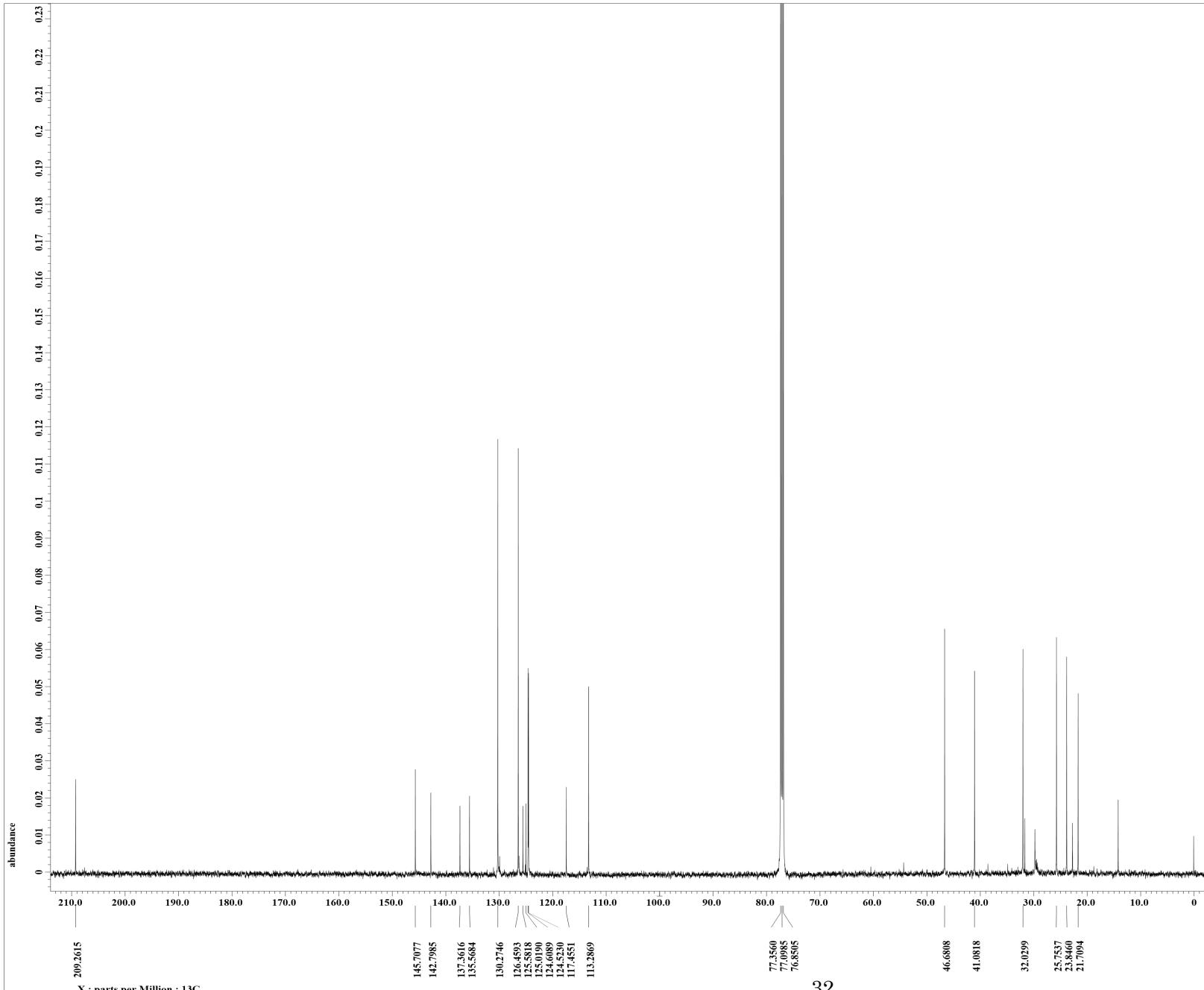


```

Filename      = TA2020-0319-4C12-7.jd
Author        = delta
Experiment   = single_pulse.ex2
Sampled_id   = S#576520
Solt_E       = CH3-NORM-D
Creation_time= 4-JUN-2000 17:47:45
Revision_time= 19-MAR-2020 16:05:28
Current_time = 19-MAR-2020 16:06:10
Comment       = 4Cl-cyclohexenone-2
Data_format  = 1D COMPLEX
Dim_2        = 13107
Dim_title    = 1H
Dim_units   = [ppm]
Dimensions   = X
Site          = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500 [MHz]
X_acq_duration = 1.74587904[s]
X_domain     = 1H
X_freq        = 500.15991521[MHz]
X_offset      = 5.0 [ppm]
X_points      = 16384
X_resolution  = 0.57277737[Hz]
X_sweep       = 9.38438438[kHz]
Irr_domain   = 1H
Irr_freq      = 500.15991521[MHz]
Irr_offset    = 5.0 [ppm]
Tri_domain   = 1H
Tri_freq     = 500.15991521[MHz]
Tri_offset   = 5.0 [ppm]
Clipped      = FALSE
Mod_return   = 1
Scans         = 8
Total_scans  = 8
X_90_width   = 12 [us]
X_acq_time   = 1.74587904[s]
X_angle       = 45 [deg]
X_atn        = 3.4 [dB]
X_pulse       = 6 [us]
Irr_mode     = Off
Tri_mode     = Off
Dante_preset = FALSE
Initial_wait  = 1 [s]
Recv_r_gain   = 50
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904[s]
Temp_get     = 22.3 [dc]

```



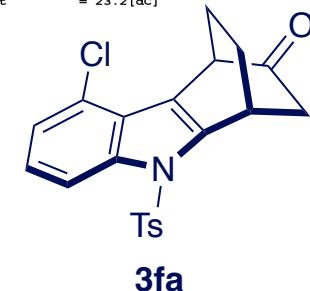


JEOL

---- PROCESSING PARAMETERS ----
 do_balance : 0 : FALSE
 scan : 2.0[Hz] : 0.0[s]
 trapezoid : 0[%] : 80[%] : 100[%]
 zerofill : 1
 fft : 1 : TRUE : TRUE
 machinephase
 ppm

Derived from: TA2020-0319-4C12-5.jdf

Filename = TA2020-0319-4C12-7.jd
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = SH577220
 Solver_id = CHLMR2D-D
 Creation_time = 20-JUN-2000 13:23:13
 Revision_time = 20-MAR-2020 11:38:46
 Current_Time = 20-MAR-2020 11:39:44
 Comment = 4Cl-cyclohexenone
 Data_format = 1H COMPLEX
 Dim_size = 16384
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA500
 Spectrometer = DELTA2_NMR
 Field_strength = 11.7473579[T] (500[MHz])
 X_acq_duration = 0.83361792[s]
 X_domain = 13C
 X_freq = 125.76529768[MHz]
 X_offset = 100[ppm]
 X_points = 32768
 X_Pulse = 4
 X_resolution = 0.19959034[Hz]
 X_sweep = 39.3081761[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 0
 Scans = 24875
 Total_scans = 24875
 X_90_width = 12.8[us]
 X_acq_time = 0.83361792[s]
 X_angle = 90
 X_kick = 5.3[dB]
 X_pulse = 4.26666667[us]
 Irr_stn_dec = 21.09[dB]
 Irr_stn_noe = 21.09[dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 0[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 58
 Relaxation_delay = 2[s]
 Repetition_time = 2.83361792[s]
 Temp_get = 23.2[dC]





JEOL

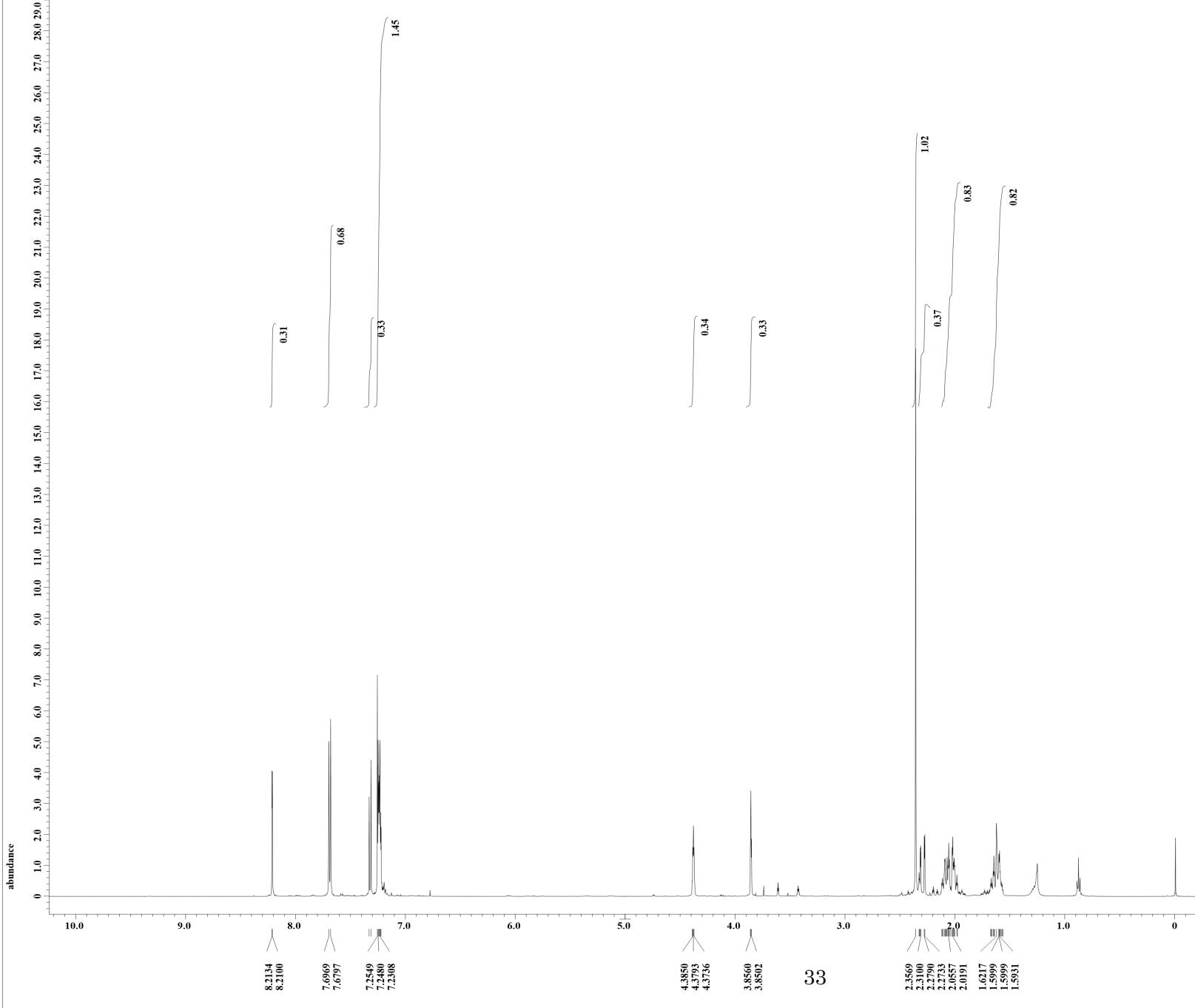
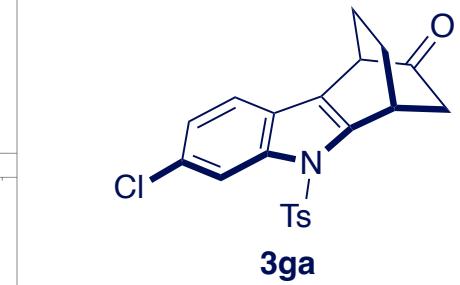
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
PPM
Derived from: TA2020-0323-6C1-1.jdf

Filename = TA2020-0323-6C1-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#693221
Solvent = CHLOROFORM-D
Creation_time = 8-JUN-2000 21:02:08
Revision_time = 23-MAR-2020 19:19:15
Current_time = 23-MAR-2020 19:19:43

Comment = single_pulse
Data_format = 1D_COMPLEX
Dim_Size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECAS500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_pnts = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Tri_return = 1
Clipval = 1
Mod_return = 8
Scans = 1
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atten = 3.0[dB]
X_pulse = 6[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23[dC]



X : parts per Million : 1H



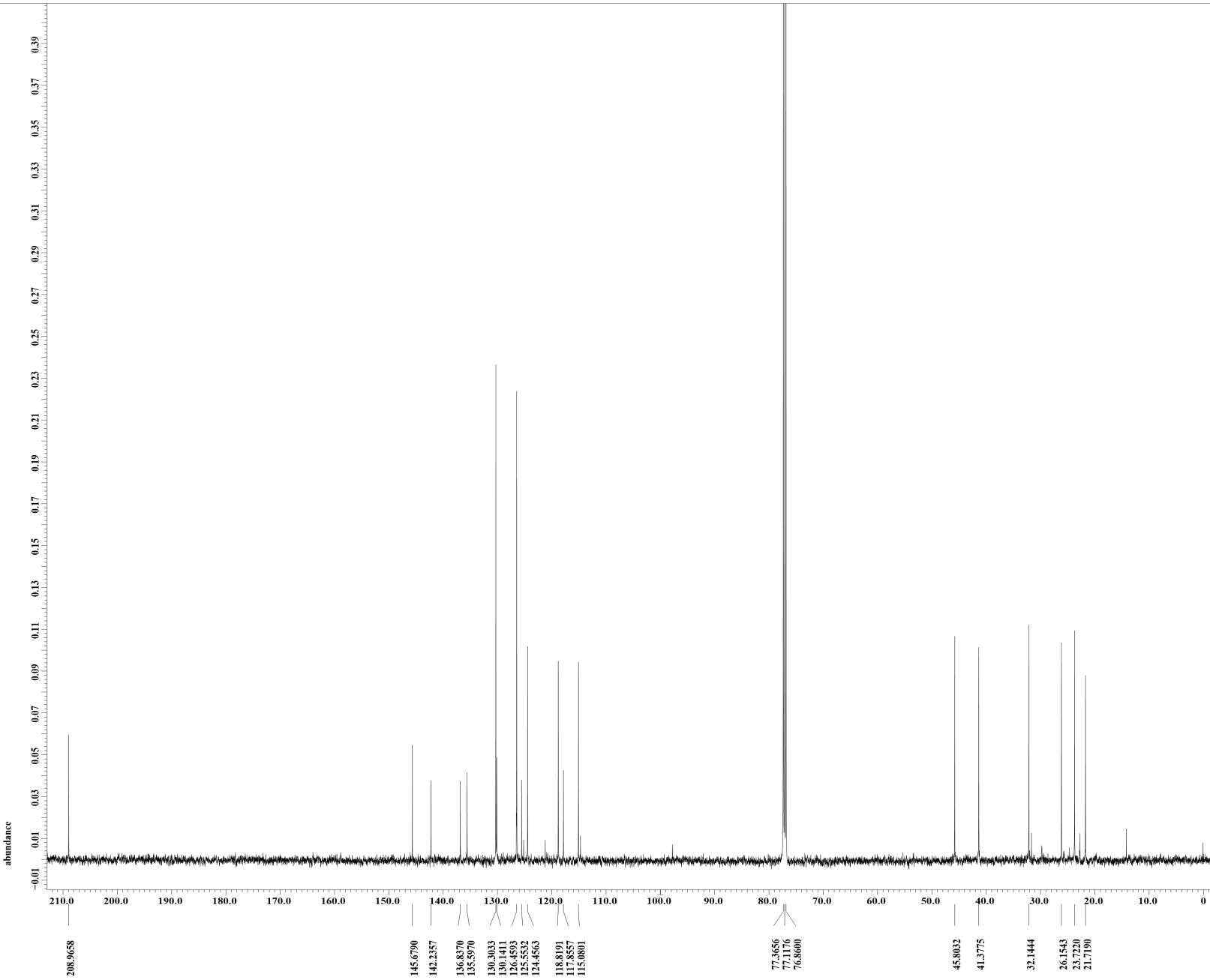
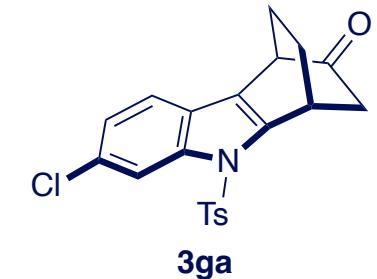
```
---- PROCESSING PARAMETERS ----
do_balance : 0 : FALSE
scsize : 2.0[Hz] : 0.0[%]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinemphase
Ppm
Derived from: TA2020-0325-5.jdf
```

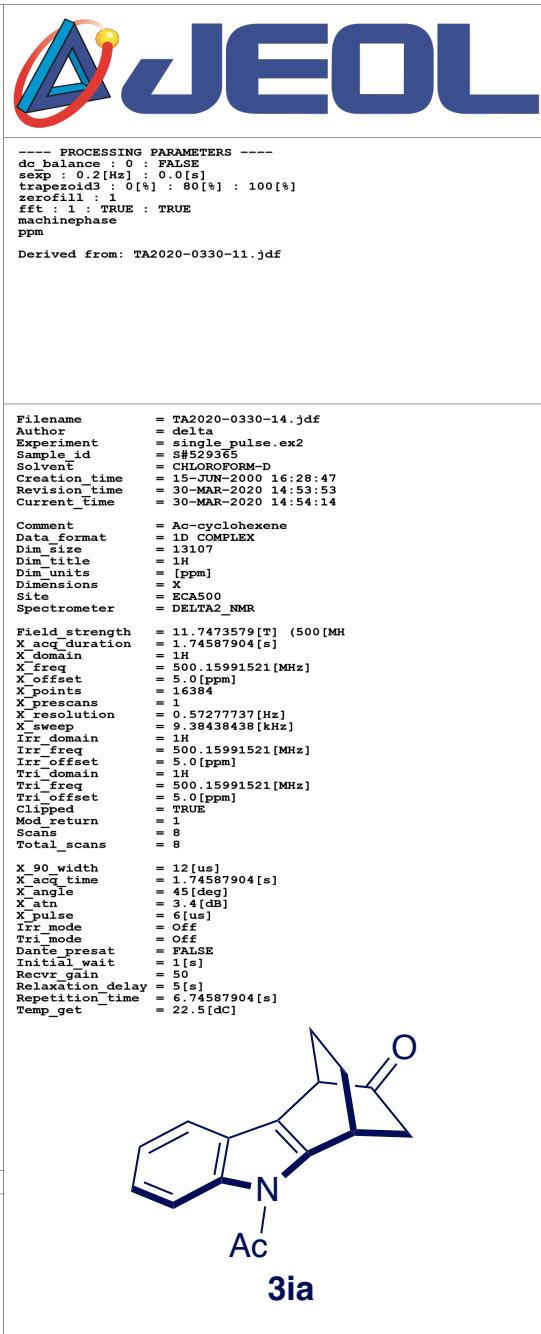
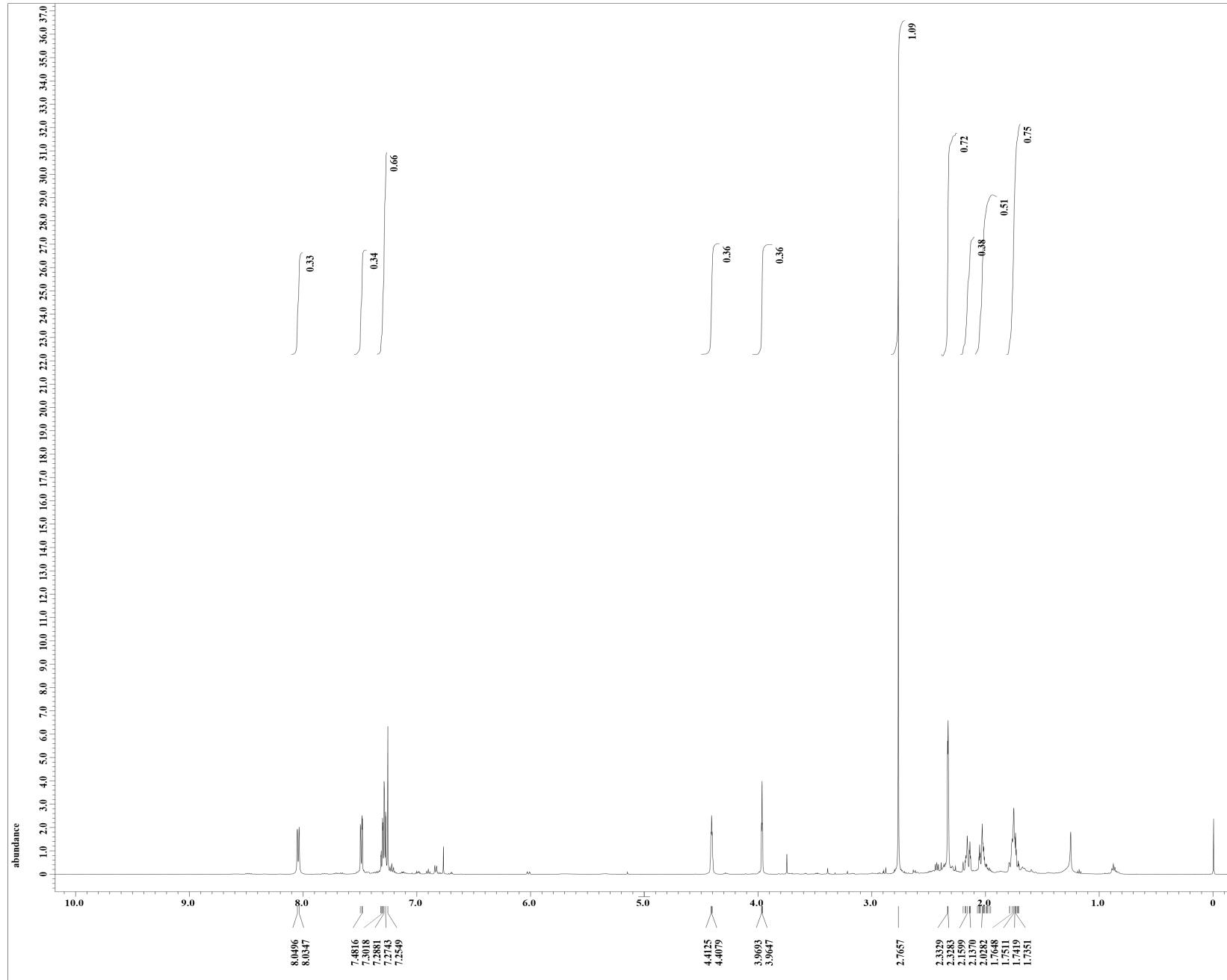
```
Filename      = TA2020-0325-7.jdf
Author       = delta
Instrument  = siemens_pulse_dec
Sample_id    = #36749
SolventF     = CHLOROFORM-D
Creation_time = 10-JUN-2000 12:57:47
Revision_time = 25-MAR-2020 11:12:26
Current_Time = 25-MAR-2020 11:12:58

Comment      = single pulse decouple
Data_format = 1D COMPLEX
Dim_Size    = 26214
Dim_title   = 13C
Dim_units   = [ppm]
Dimensions  = X
Site         = ECA500
Spectrometer= DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 0.83361792[s]
X_domain      = 13C
X_fine        = 125.76529768[MHz]
X_offset       = 100[ppm]
X_points      = 32768
X_resolution  = 4
X_prescans   = 1.19959034[Hz]
X_sweep       = 39.3081761[Hz]
Irr_domain   = 1H
Irr_fine     = 500.15991521[MHz]
Irr_offset   = 5.0[ppm]
Clipped      = FALSE
Mod_return   = 1
Scans        = 1250
Total_scans  = 1250

X_90_width   = 12.8[us]
X_acq_time   = 0.83361792[s]
X_angle       = 30[deg]
X_atn        = 5.3[dB]
X_pulse       = 4.26666667[us]
Irr_atn_dec  = 21.09[dB]
Irr_atn_noz  = 21.09[dB]
Irr_noise     = WALN2
Decoupling   = TRUE
Initial_wait  = 1[s]
Noe          = TRUE
Noe_time     = 2[s]
Re_polarain  = 5[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get     = 24[dC]
```

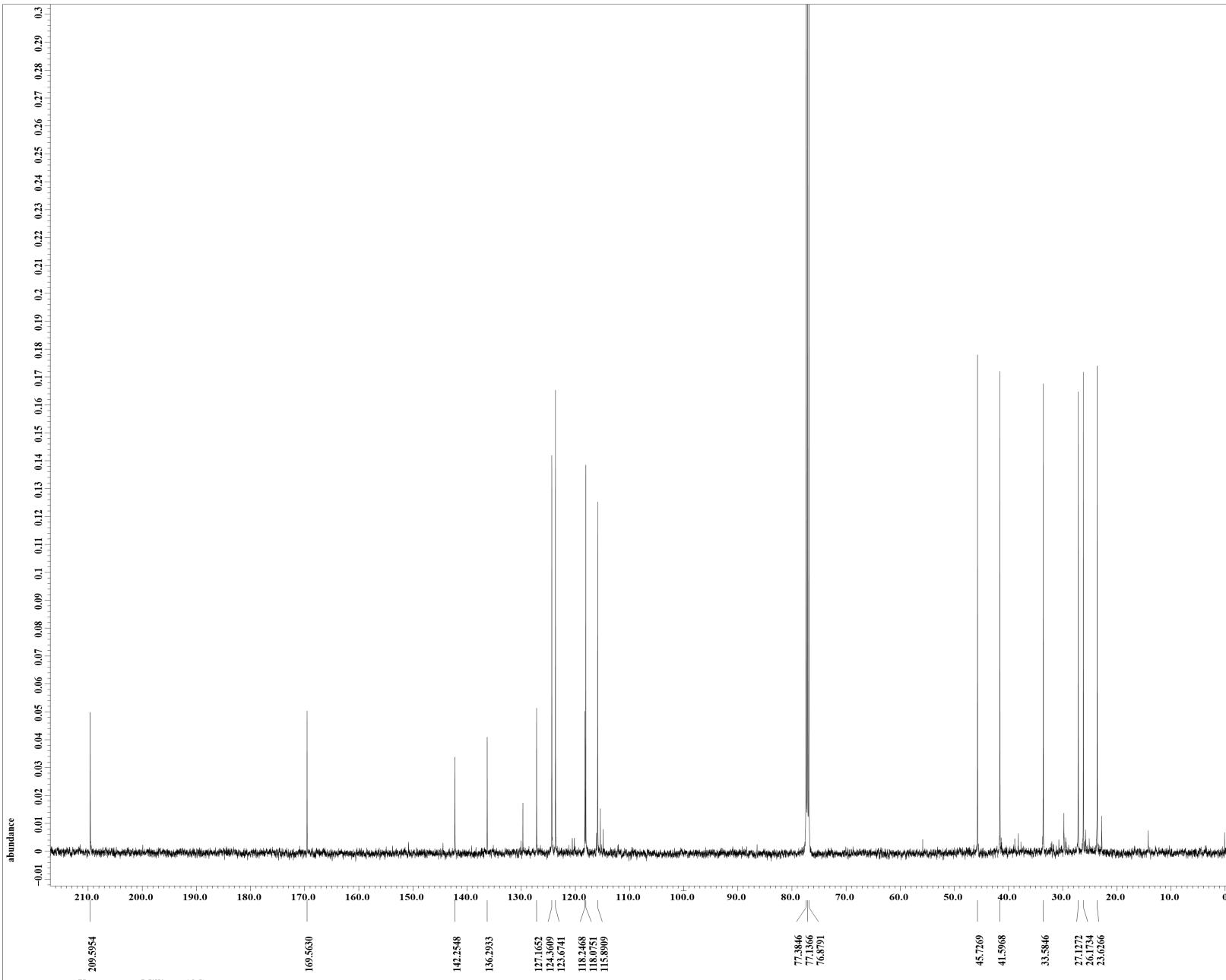
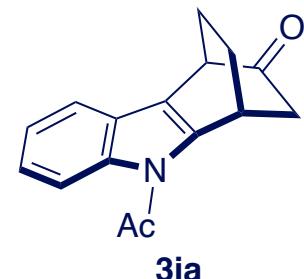


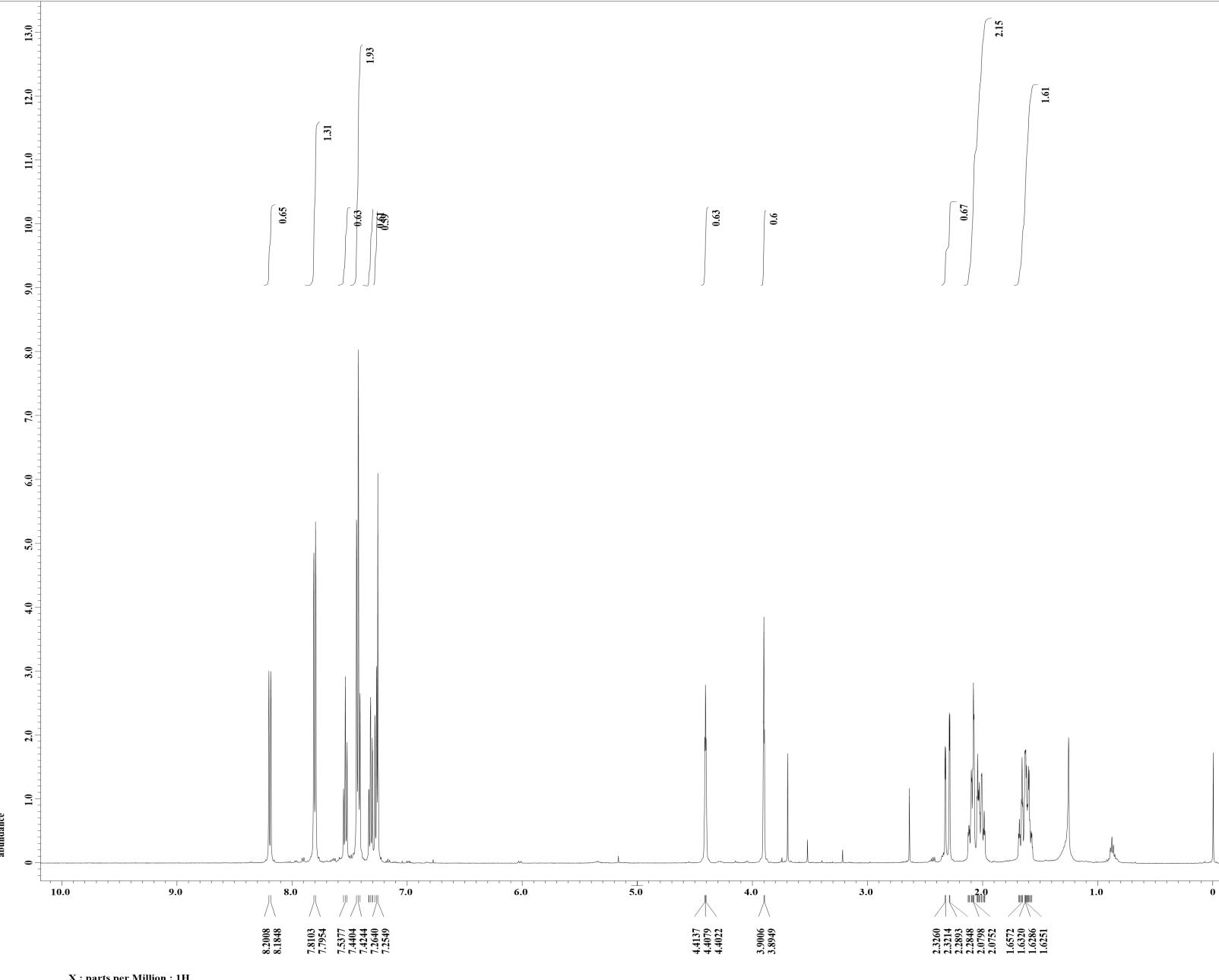




---- PROCESSING PARAMETERS ----
dc_balance : FALSE
sep : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft: 1 : TRUE : TRUE
machinephase
ppm
Derived from: TA2020-0330-12.jdf

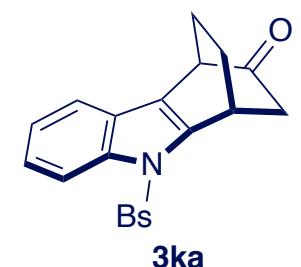
Filename = TA2020-0330-15.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S8530115
Solvent = CHLOROFORM-D
Creation_time = 1-JUN-2000 18:18:32
Revision_time = 30-MAR-2020 16:33:31
Current_Time = 30-MAR-2020 16:35:30
Comment = single pulse decouple
Data_format = 1H COMPLEX
Dim_size = 6214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz)
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 0.00[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[KHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 2307
Total_scans = 2307
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 2.2056667[us]
Irr_atn_dec = 22.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noc = TRUE
Noc_time = 21[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.7[dc]





----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
ff1 : 1 : TRUE : TRUE
machinephase
PPM
Derived from: TA2020-0416-1.jdf

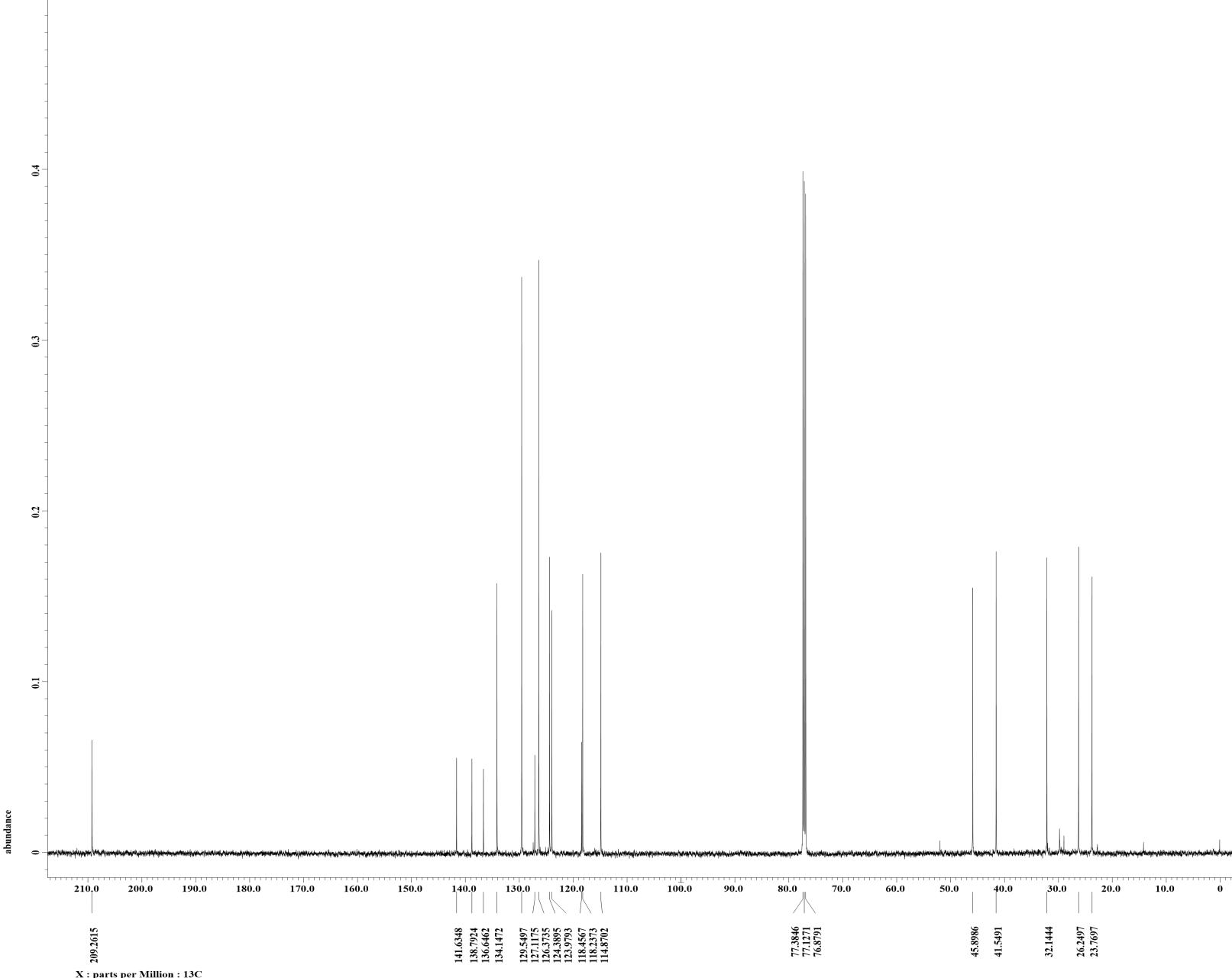
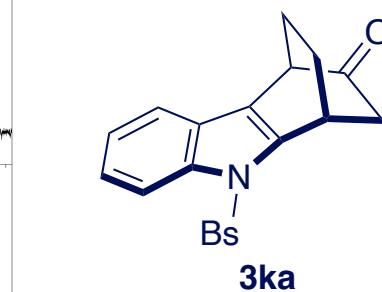
filename = TA2020-0416-9.jdf
author = delta
Experiment = single_pulse.ex2
sample_id = S#388094
solvent = CDCl3/PFGHM-D
creation_time = 2-AUG-2000 12:32:45
revision_time = 16-APR-2020 11:39:41
current_time = 16-APR-2020 11:40:15
comment = single_pulse
data_format = 1D COMPLEX
dim_size = 13107
dim_title = 1H
dim_units = [ppm]
dimensions = X
site = ECA500
spectrometer = DELTA2_NMR
field_strength = 11.7473579[T] (500[MHz])
x_acq_duration = 1.74587904[s]
x_domain = 1H
x_freq = 500.15991521[MHz]
x_knot = 5.0[ppm]
x_points = 16384
x_prescans = 1
x_resolution = 0.57277737[Hz]
x_sweep = 9.38438438[kHz]
irf_main = 1H
irf_fwhm = 500.15991521[MHz]
irf_offset = 5.0[ppm]
tr1_domain = 1H
tr1_freq = 500.15991521[MHz]
tr1_offset = 5.0[ppm]
clipped = TRUE
multiturn =
scans = 8
total_scans = 8
x_90_width = 12[us]
x_qc_time = 1.74587904[s]
x_tilt = 45[deg]
x_atn = 3.4[dB]
x_pulse = 6[us]
irf_mode = Off
tr1_mode = Off
data_preset = AUTO
initial_wait = 1[s]
recv_r_gain = 50
relaxation_delay = 5[s]
repetition_time = 6.74587904[s]
temp_get = 22.9[dc]

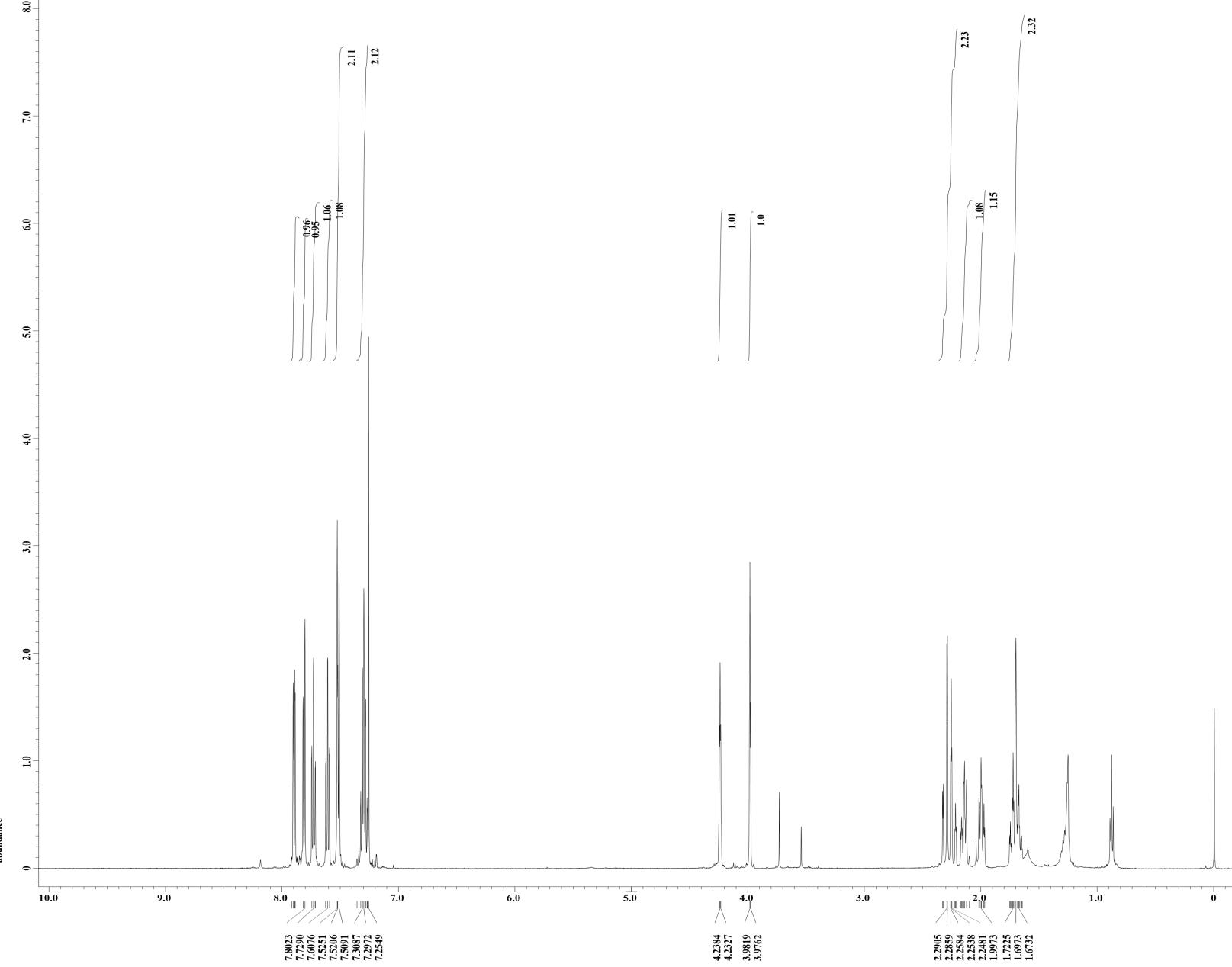


X : parts per Million : 1H

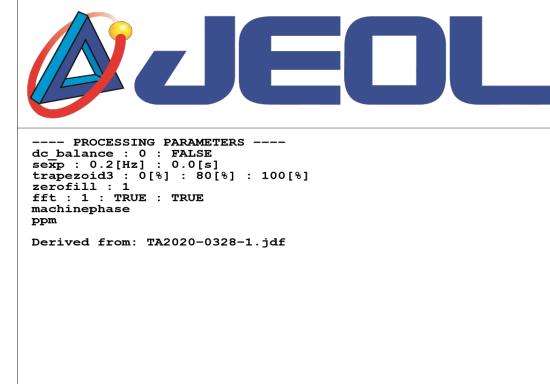
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
scRP : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill1 : TRUE : TRUE
ff1 : TRUE : TRUE
machinephase
ppm
Derived from: TA2020-0416-7.jdf

Filename = TA2020-0416-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SH#7810
Solvnt = CDCl3/CD3OD-D
Creation_time = 2-JUL-2000 15:01:37
Revision_time = 16-APR-2020 13:17:00
Current_time = 16-APR-2020 13:17:37
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_Size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECAS00
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 0.83361792[s]
X_center = 125.76529768[MHz]
X_freq = 100[ppm]
X_offset = 32768
X_points = 4
X_prescans = 1.19959034[Hz]
X_resolution = 0.93081761[kHz]
X_start = 1H
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scan = 2110
Total_scans = 2110
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_dpple = 5[dB]
X_stn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_hdw = 1000[KHz]
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Receive_gain = 54
Repetition_delay = 2.83361792[s]
Repetition_time = 2.83361792[s]
Temp_get = 22.8[dC]





X : parts per Million : 1H



```

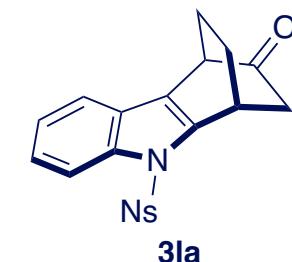
Filename = TA2020-0328-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#563525
Solvent = CHLOROFORM-D
Creation_time = 13-JUN-2000 17:25:47
Revision_time = 26-MAR-2020 15:45:34
Current_Time = 28-MAR-2020 15:46:36

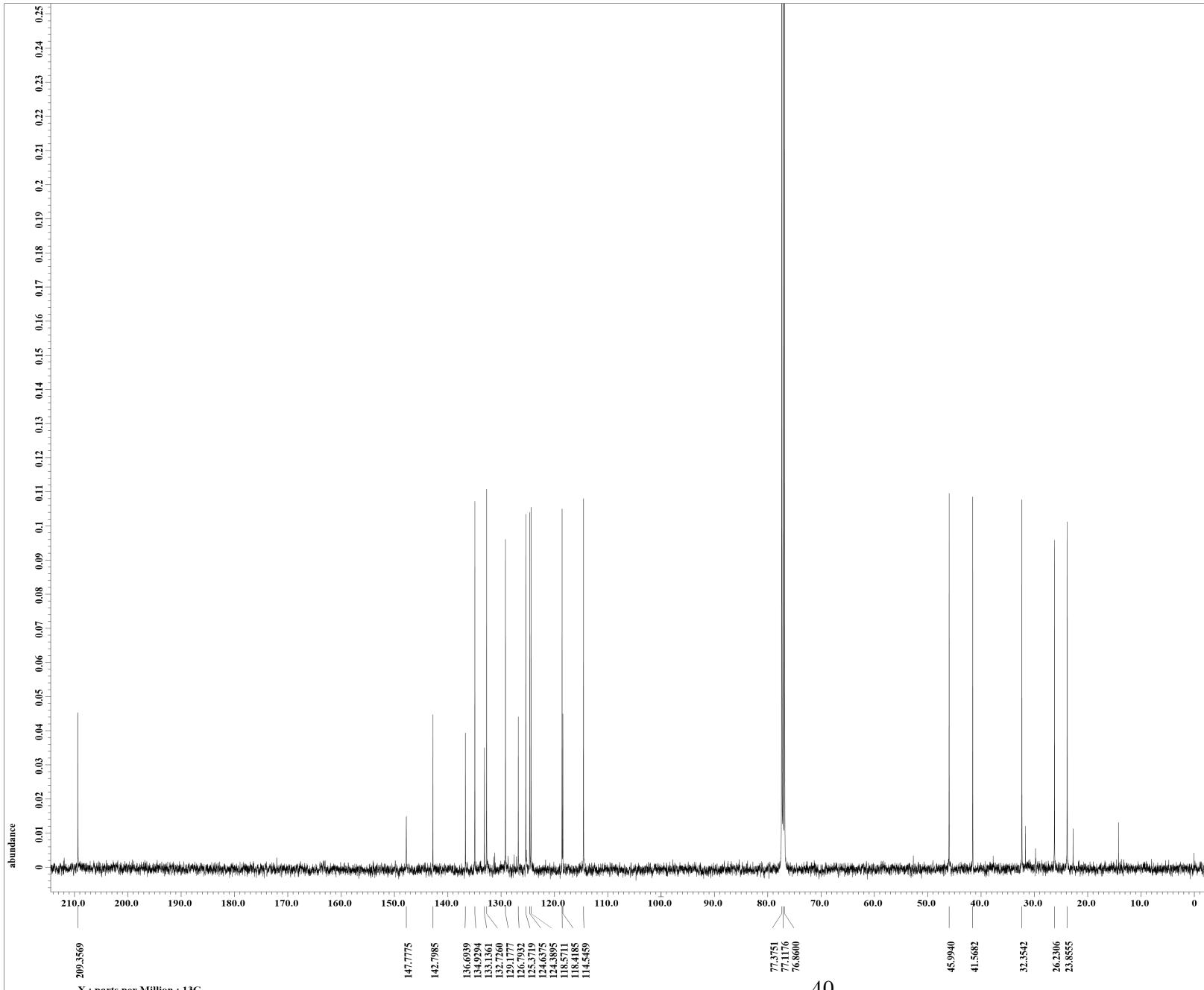
Comment = single_pulse
Data_format = ID COMPLEX
Dim_size = 13107
Dim_label = 
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTAS_NMR

Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_trans = 
X_resolution = 0.572777737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 0.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_awe = 6[dB]
X_awe_us = 1[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Ref_gain = 50
Relaxation_delay = 0.1[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.9[dc]

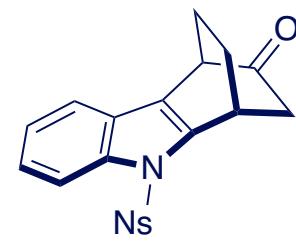
```

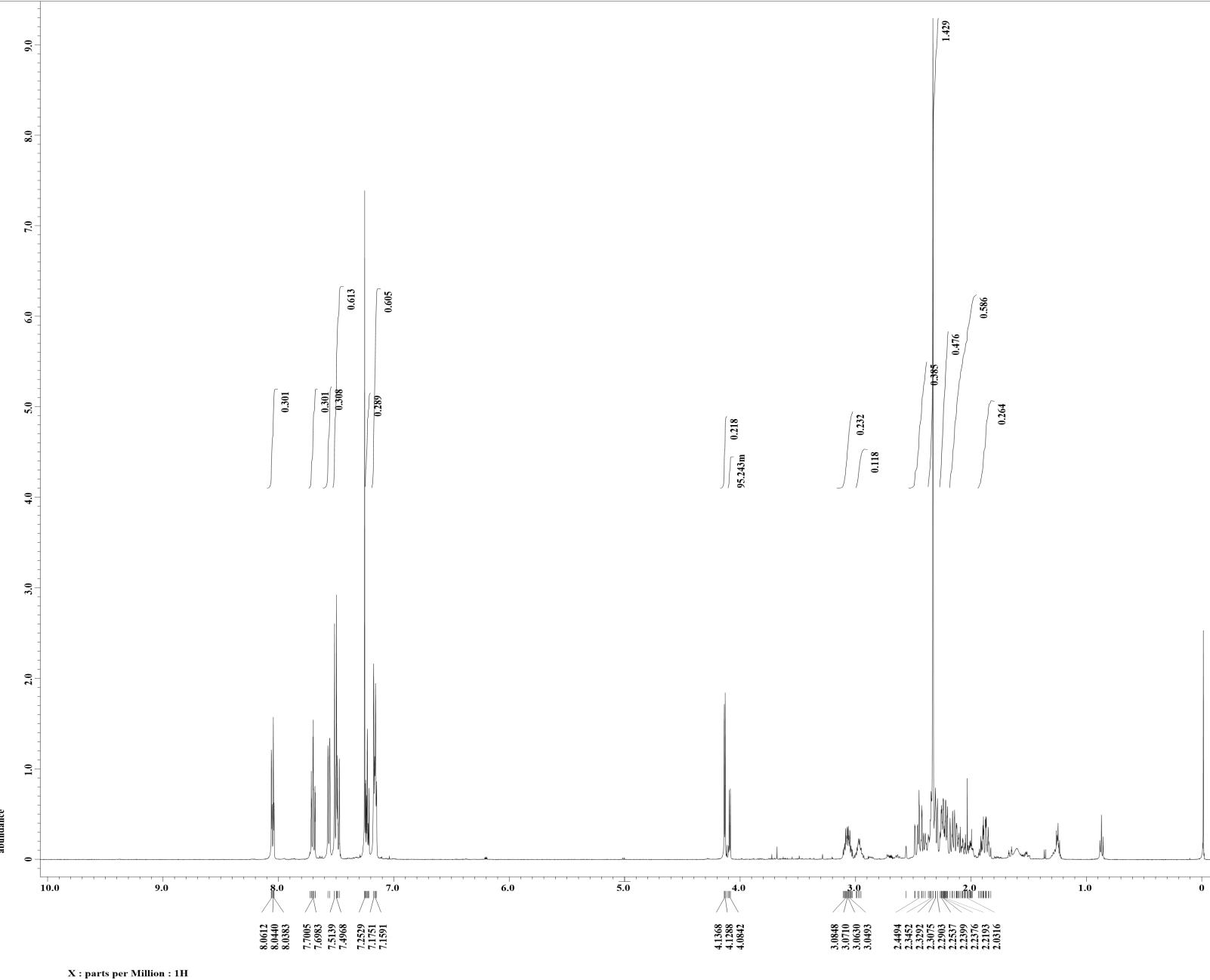




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0 [Hz] : 0.0[s]
tapered3d : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TA2020-0328-2.jdf
```

```
Filename = TA2020-0328-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SH56497
SolventE = DILICLOFORM-D
Creation_time = 13-JUN-2000 18:59:46
Revision_time = 28-MAR-2020 17:15:47
Current_Time = 28-MAR-2020 17:16:48
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 1944
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECAS500
Spectrometer = DELTAII_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prestans =
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return =
Scan = 1946
Total_scans = 1946
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_dpp = 1[db]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.8[dc]
```





```

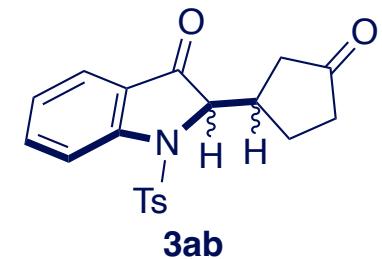
Filename = TA200318-12.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#681304
Solvent = CHLOROFORM-D
Creation_time = 18-MAR-2020 19:03:50
Revision_time = 18-MAR-2020 19:06:25
Current_time = 18-MAR-2020 19:07:05

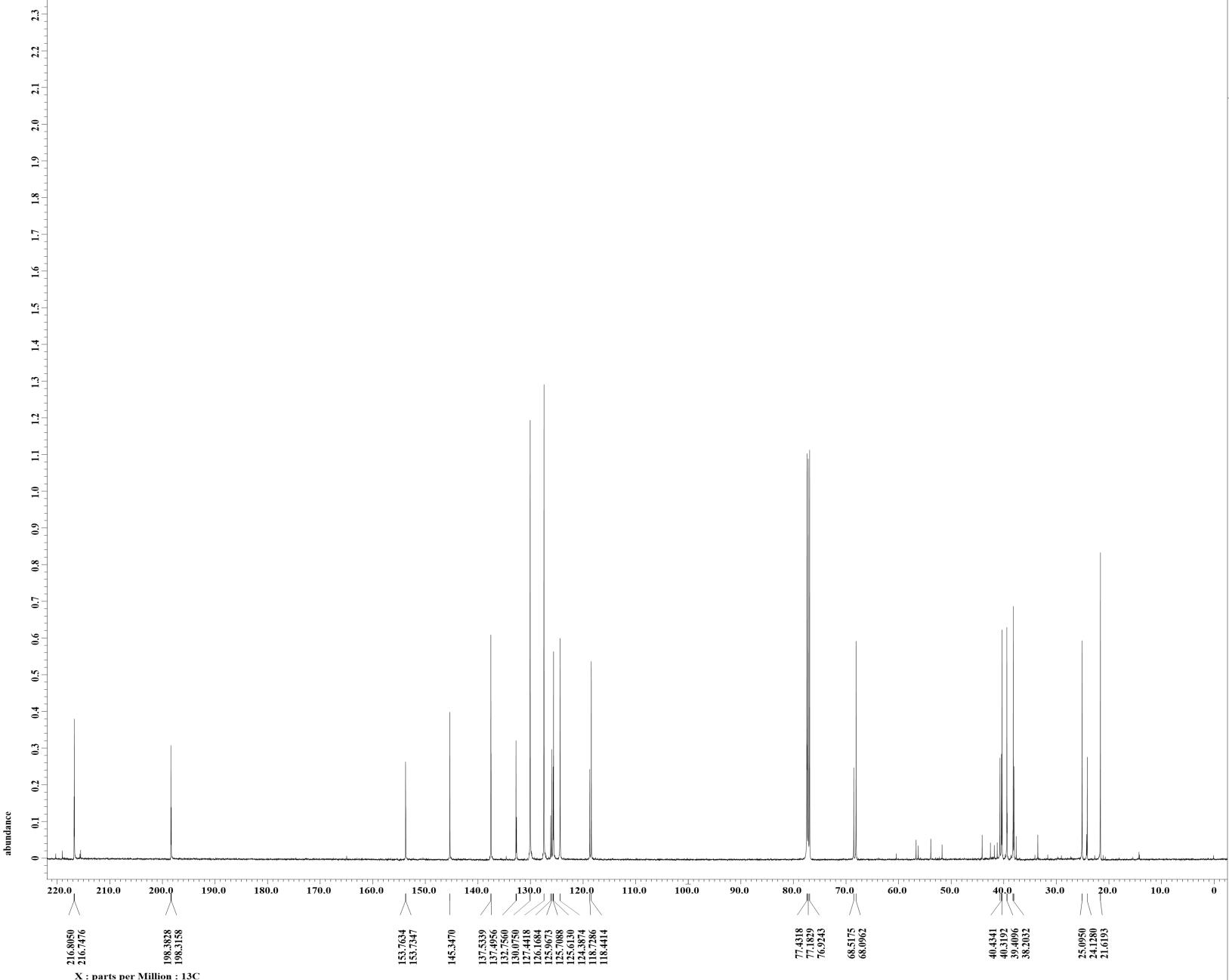
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions =
Site = X
Spectrometer = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset =
X_points = 5[ppm]
X_presets =
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset =
Tri_domain = 5[ppm]
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_att = 3.3[dB]
X_diss = 25[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvv_gain = 48
Relaxation_delay = 100[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.7[dC]

```





```

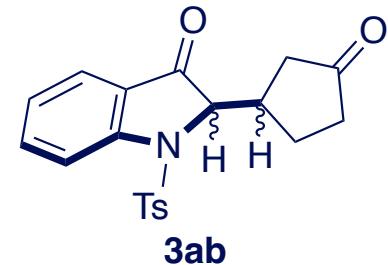
Filename = TA200318-16.jdf
Author = dca
Experiment = single_pulse_dec
Sample_id = S#6937B3
Solvent = CHLOROFORM-D
Creation_time = 19-MAR-2020 07:34:03
Revision_time = 19-MAR-2020 08:28:18
Current_time = 19-MAR-2020 08:29:17

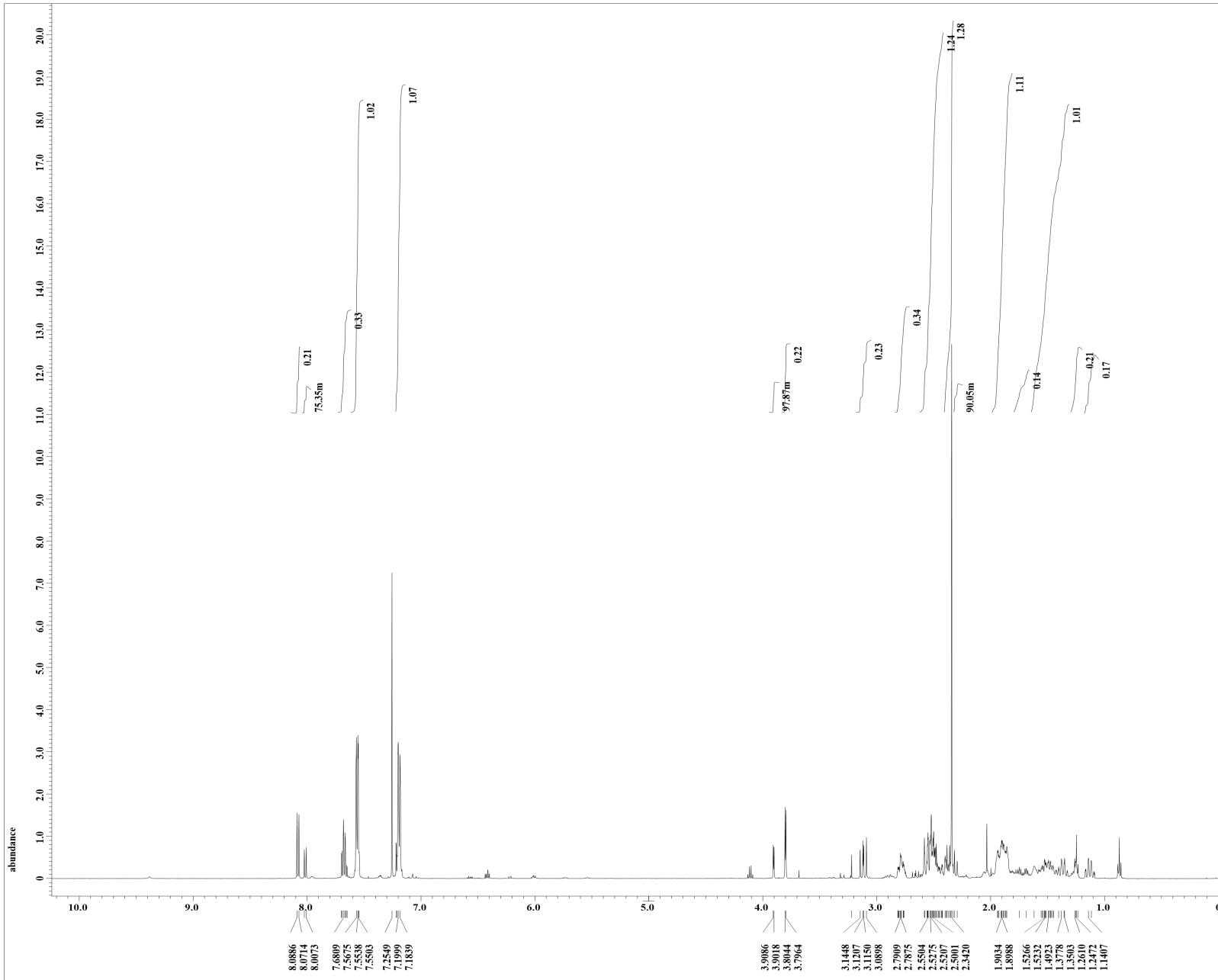
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_Size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_dq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_pdduration = 1.1920929[Hz]
X_pdsleep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mode_return = 1
Scan = 16705
Total_scans = 16705

X_90_width = 10.1[us]
X_acc_time = 0.8388608[s]
X_attenuate = 0
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = 1000
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recv_r_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.3[dC]

```



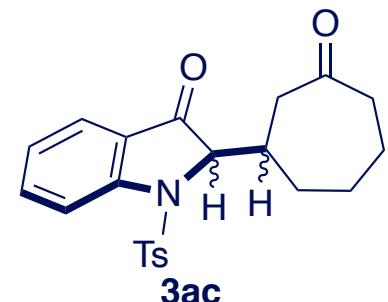


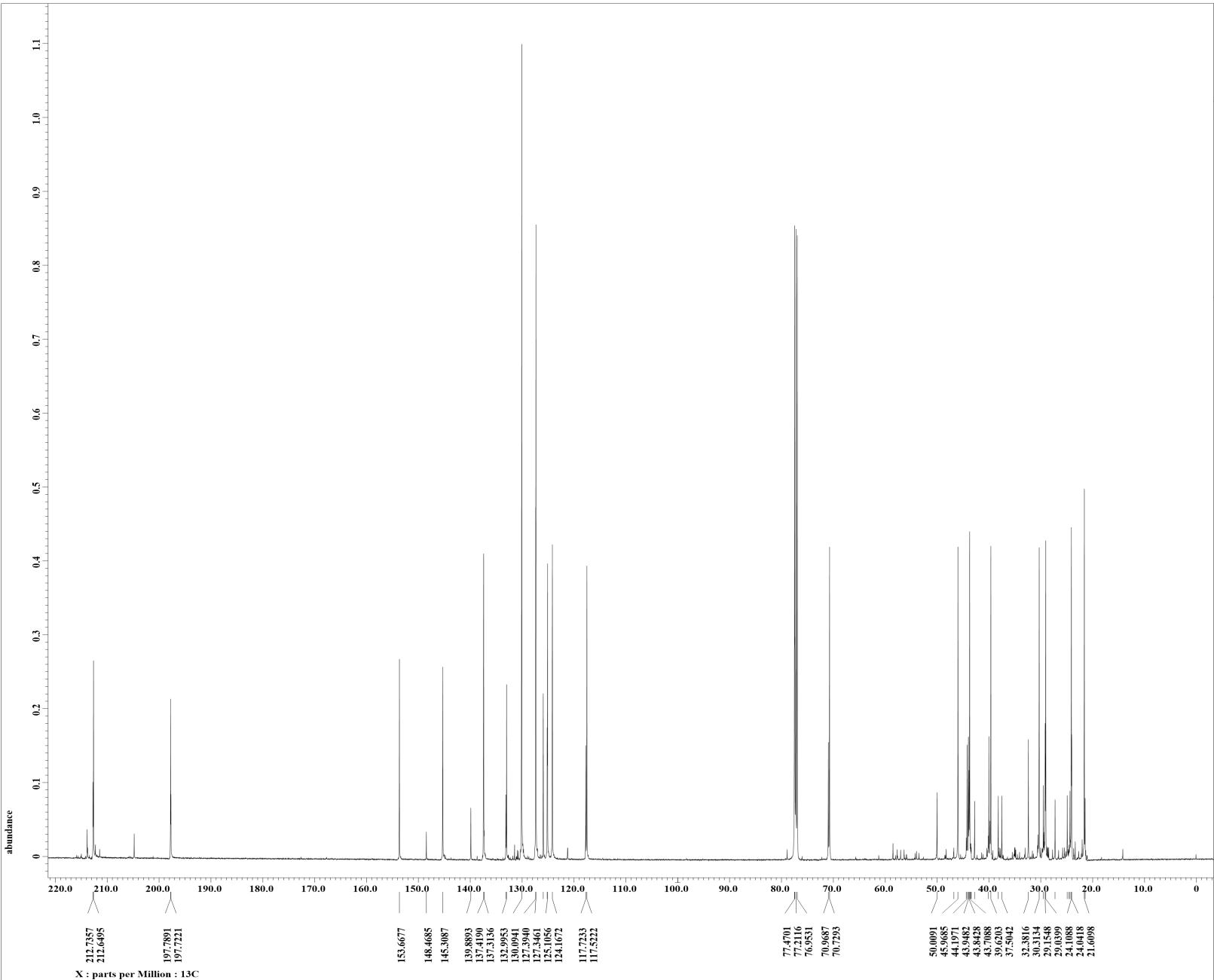
43



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: TA2020-0319-1.jdf
```

```
Filename = TA2020-0319-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#462109
Solvent = CHLOROFORM-D
Creation_time = 4-JUN-2000 14:37:04
Revision_time = 19-MAR-2020 13:00:59
Current_time = 19-MAR-2020 13:01:55
Comment = cycloheptenone
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = y
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_recursion = 0.57277737[Hz]
X_tauop = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[us]
Irr_mode = F
Tri_mode = Off
DanTe_presat = FALSE
Initial_wait = 1[s]
Recv_rgain = 50
Relaxation_delay = 5[s]
Repetition_time = 1.74587904[s]
Temp_get = 22.7[dC]
```



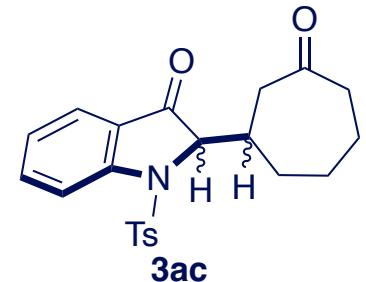


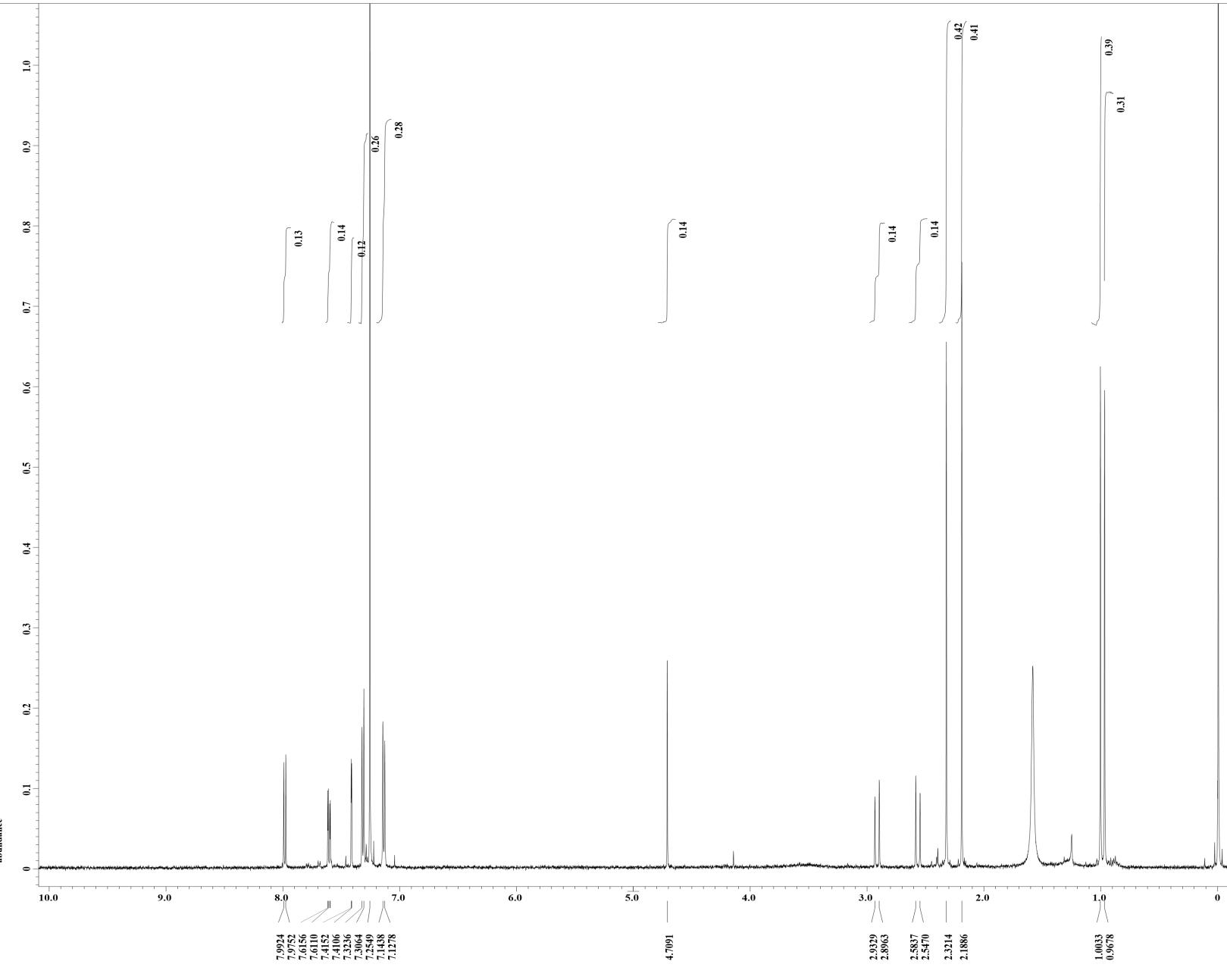
JEOL

---- PROCESSING PARAMETERS ----
dc_balance : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft: 1 : TRUE
machinephase
ppm

Derived from: TA200320-2-4.jdf

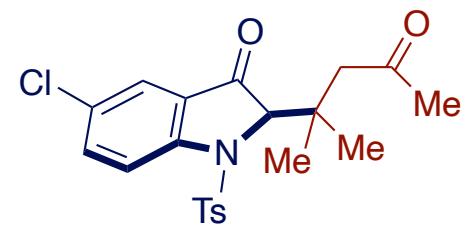
Filename = TA200320-2-7.jdf
Author = delta
Experiment = single pulse dec
Sample_id = S8567161
Solvent = CHLOROFORM-D
Creation_time = 23-APR-2020 07:33:39
Revision_time = 26-APR-2020 11:49:23
Current_Time = 26-APR-2020 11:50:30
Comment = single pulse decouple
Data_format = 1H COMPLEX
Dim_size = 6214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acc_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_Pulse = 127.68
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 95.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 82030
Total_scans = 82030
X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = 1
Nouc_time = 5[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24[dC]

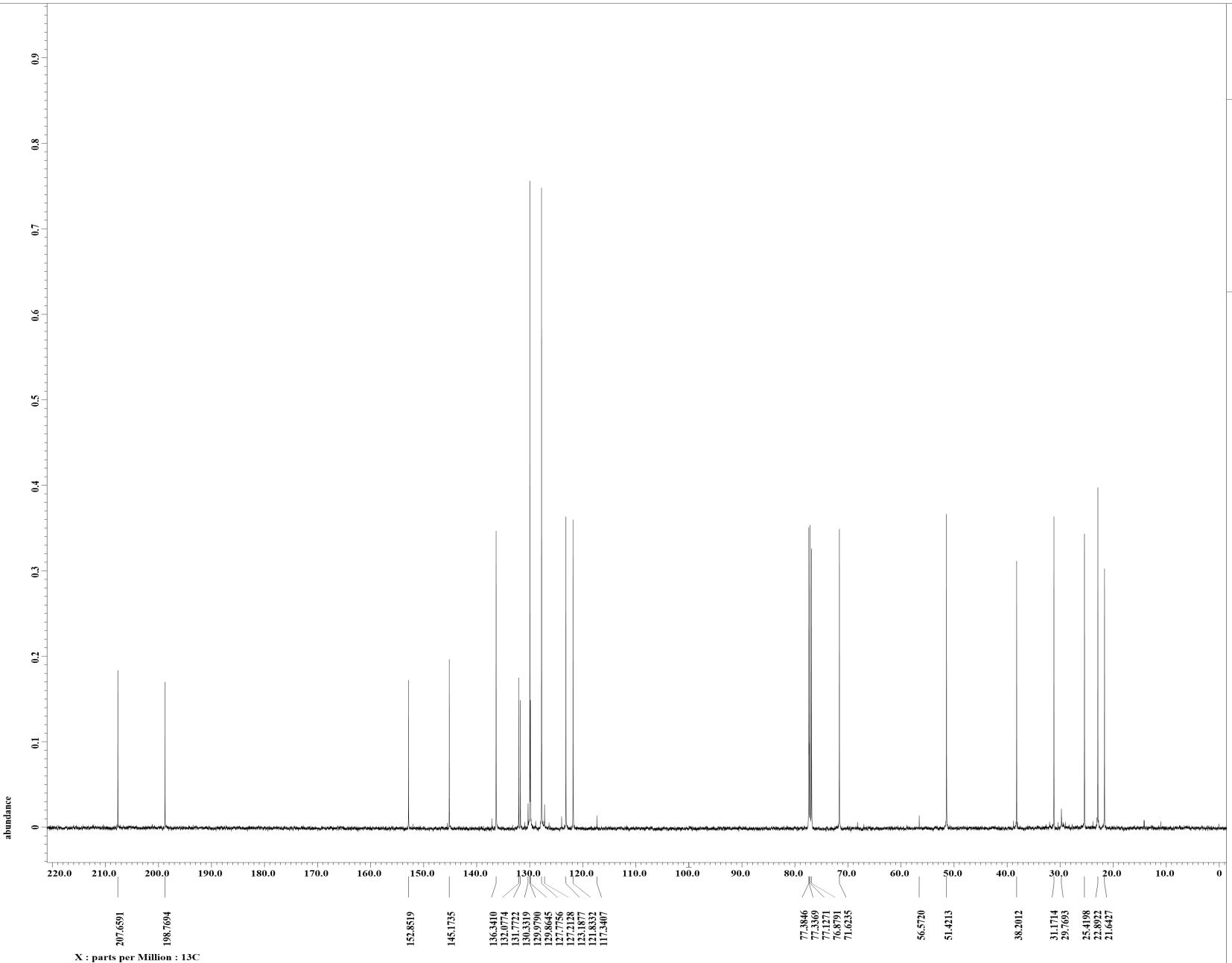




----- PROCESSING PARAMETERS -----
dc_balance : 0 : PULSE
sexp : 0.2 [Hz] : 0.0 [%]
trapezoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 5Cl-acetone-1H-1.jdf

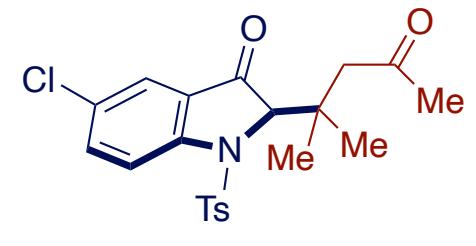
Filename = 5Cl-acetone-1H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#633038
Solvent = CHLOROFORM-D
Creation_time = 5-MAY-2000 19:22:58
Revision_time = 18-FEB-2020 17:40:51
Current_Time = 18-FEB-2020 17:41:14
Comment = single pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_bit = 16
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA_2_NMR
Field_strength = 11.7473579 [T] (500 [MHz])
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_trans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 0 [ppm]
Tri_domain = 1H
Tri_freq = 500.15991521 [MHz]
Tri_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_attenuation = 4 [dB]
X_pulse = 6 [ms]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recovery = 0
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 23 [dc]

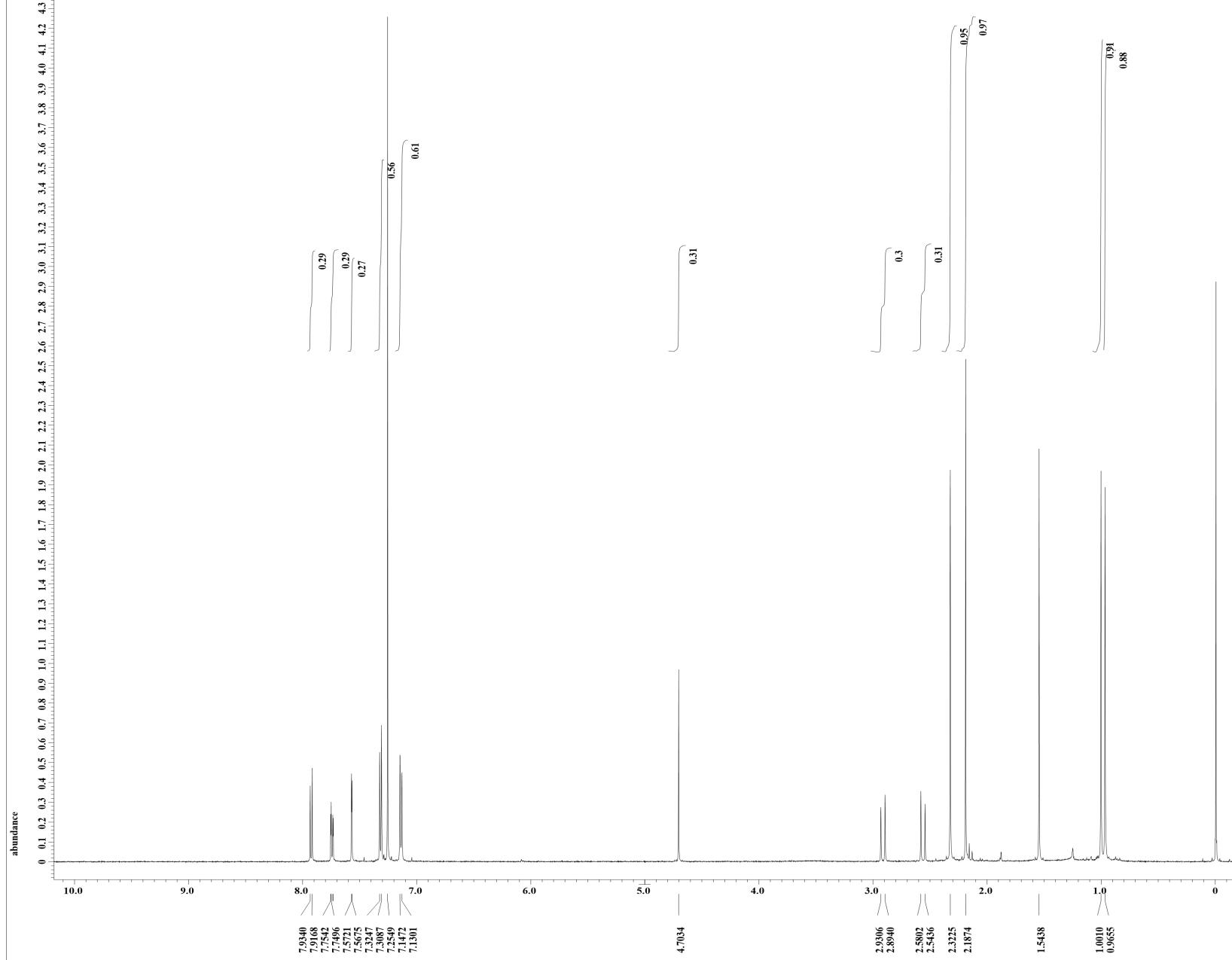




```
---- PROCESSING PARAMETERS ----
dc_balance = 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0(%) : 80(%) : 100(%)
zerofill : 1
fft : TRUE
phaseinphase
ppm
Derived from: 5Cl-acetone-13C-1.jdf
```

```
Filename      = 5Cl-acetone-13C-2.jdf
Author        = delta
Experiment   = single_pulse_dec
Sample_id    = S#63619
Solvent_N    = CHLOROFORM-D
Creation_time= 5-MAY-2000 20:55:10
Revision_time= 18-FEB-2020 19:08:26
Current_Time = 18-FEB-2020 19:09:02
Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title   =  $^{13}\text{C}$ 
Dim_units   = [ppm]
Dimensions  = X
Site         = ECA500
Spectrometer = DELTA_2_NMR
Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 0.83361792[s]
X_domain     = 3C
X_efield     = 12.876529768[MHz]
X_offset     = 100[ppm]
X_points     = 32768
X_prescans   = 4
X_resolution = 1.19959034[Hz]
X_sweep       = 3.3081761[kHz]
Irr_step     = 1H
Irr_domain   = 500.15991521[MHz]
Irr_freq     = 500.15991521[MHz]
Irr_offset   = 5.0[ppm]
Clipped      = TRUE
Mod_return   = 1
Scans        = 1857
Total_scans  = 1857
X_90_width   = 12.8[us]
X_acq_time   = 0.83361792[s]
X_angle      = 30[deg]
X_dc          = 5.9[deg]
X_pulse      = 4.26666667[us]
Irr_atn_dec  = 21.09[dB]
Irr_atn_noe  = 21.09[dB]
Irr_noise    = WALTE
Irr_coupling = TRUE
Initial_wait = 1[s]
Noe          = TRUE
Noe_time     = 2[s]
Recvr_gain   = 56
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get     = 23.6[dC]
```



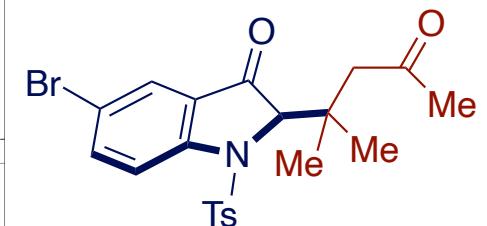


X : parts per Million : 1H

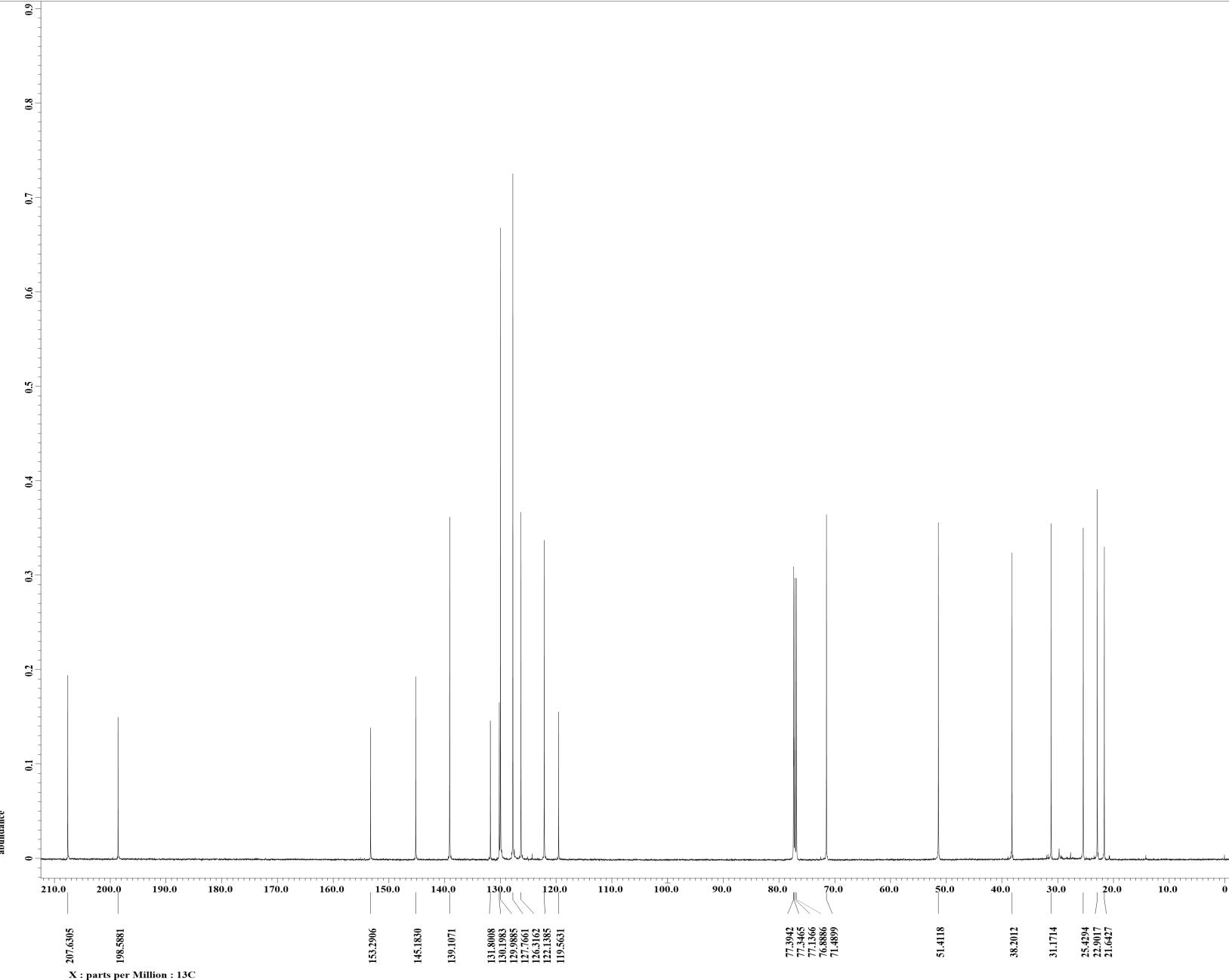


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sc_exp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
Ppm
Derived from: SBr-acetone-1H-4.jdf
```

```
Filename = SBr-acetone-1H-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S8392432
Solvent = CHLOROFORM-D
Creation_time = 6-MAY-2000 12:41:56
Revision_time = 19-FEB-2020 10:58:41
Current_Time = 19-FEB-2020 10:59:38
Comment = 5Br-acetone
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans =
X_pulses =
X_recognition = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Irr_domain =
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 1[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Relaxation_time = 7.4587904[s]
Temp_get = 23[dc]
```



3cf



```

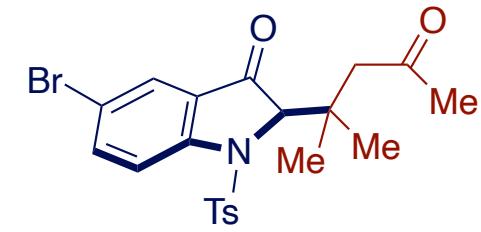
Filename      = 5Br-acetone(pure)-13C
Author        = delta
Experiment   = single_pulse_dec
Sample_id    = S8705778
Solvent       = CHLOROFORM-D
Creation_time= 1-MAR-2020 08:59:09
Revision_time= 20-FEB-2020 07:14:09
Current_Time = 20-FEB-2020 07:14:49

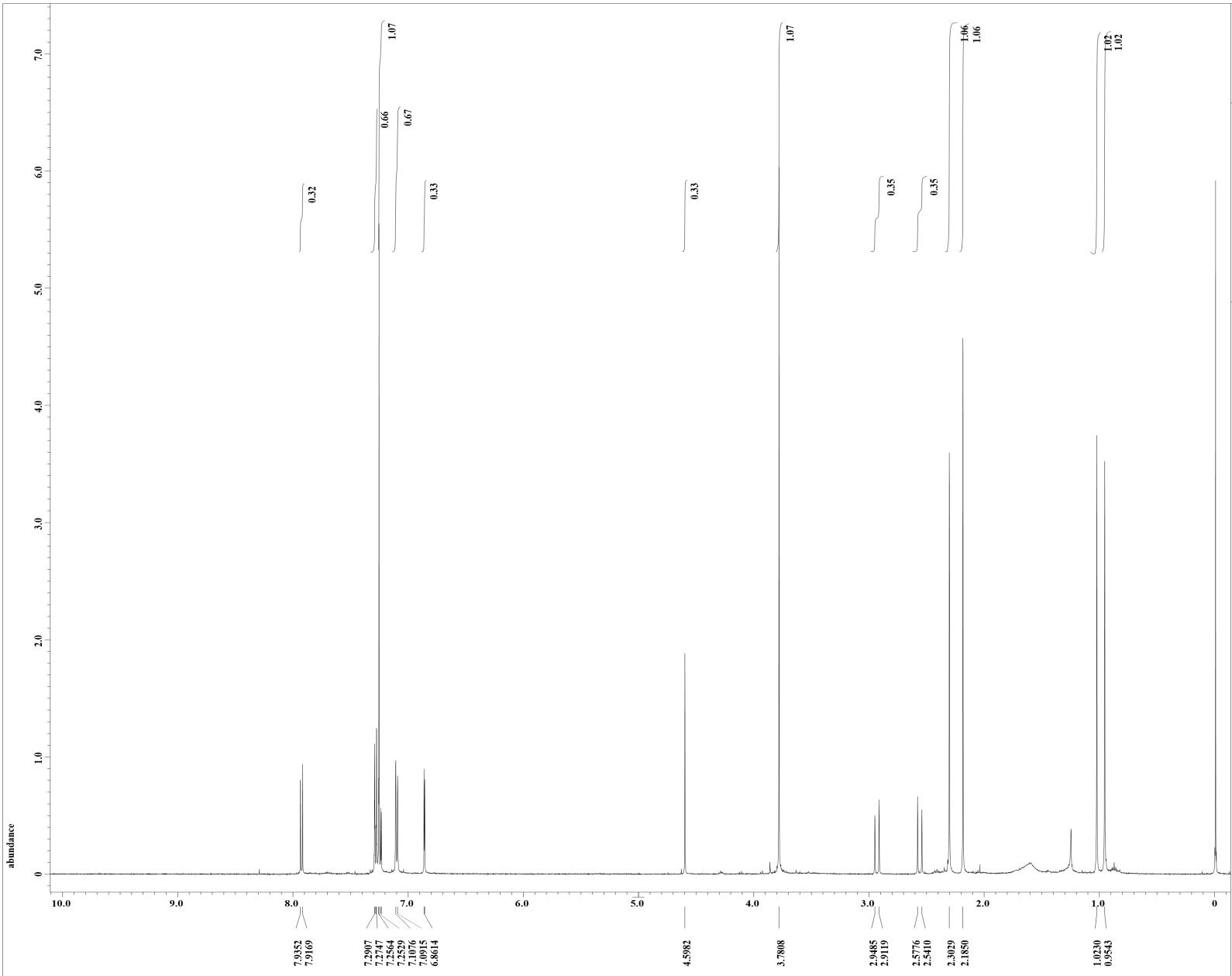
Comment      = 5Br-acetone-13C
Data_format  = 1D COMPLEX
Dim_size    = 26214
Dim_title   = 13C
Dim_units   = [ppm]
Dimensions  = X
Site         = ECA500
Spectrometer = DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 0.83361792[s]
X_domain     = 13C
X_freq        = 125.76529768[MHz]
X_offset      = 100[ppm]
X_pows        = 32768
X_ppcs        = 4
X_resolution  = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain   = 1H
Irr_freq     = 50.15991521[MHz]
Irr_offset   = 5.0[ppm]
Clipped      = FALSE
Mod_return   = 1
Scans        = 14726
Total_scans  = 14726

X_90_width   = 12.8[us]
X_acq_time   = 0.83361792[s]
X_angle       = 30[deg]
X_atn        = 5.3[dB]
X_pulse       = 4.26666667[us]
Irr_atn_dec  = 22.09[dB]
Irr_atn_noe  = 21.09[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noise        = TRUE
Rce_time     = 2[s]
Recvrx_gain  = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get     = 24.6[dC]

```





JEOL

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : TRUE : TRUE
ifff : TRUE : TRUE
machinephase
ppm

Derived from: TA191125-4-1.jdf

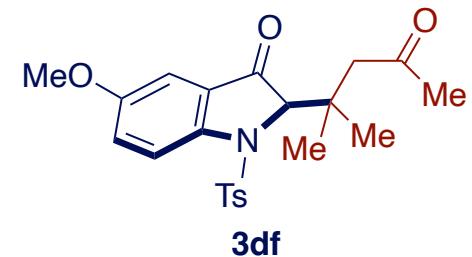
```

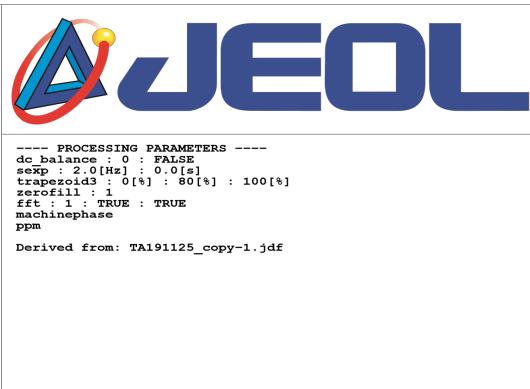
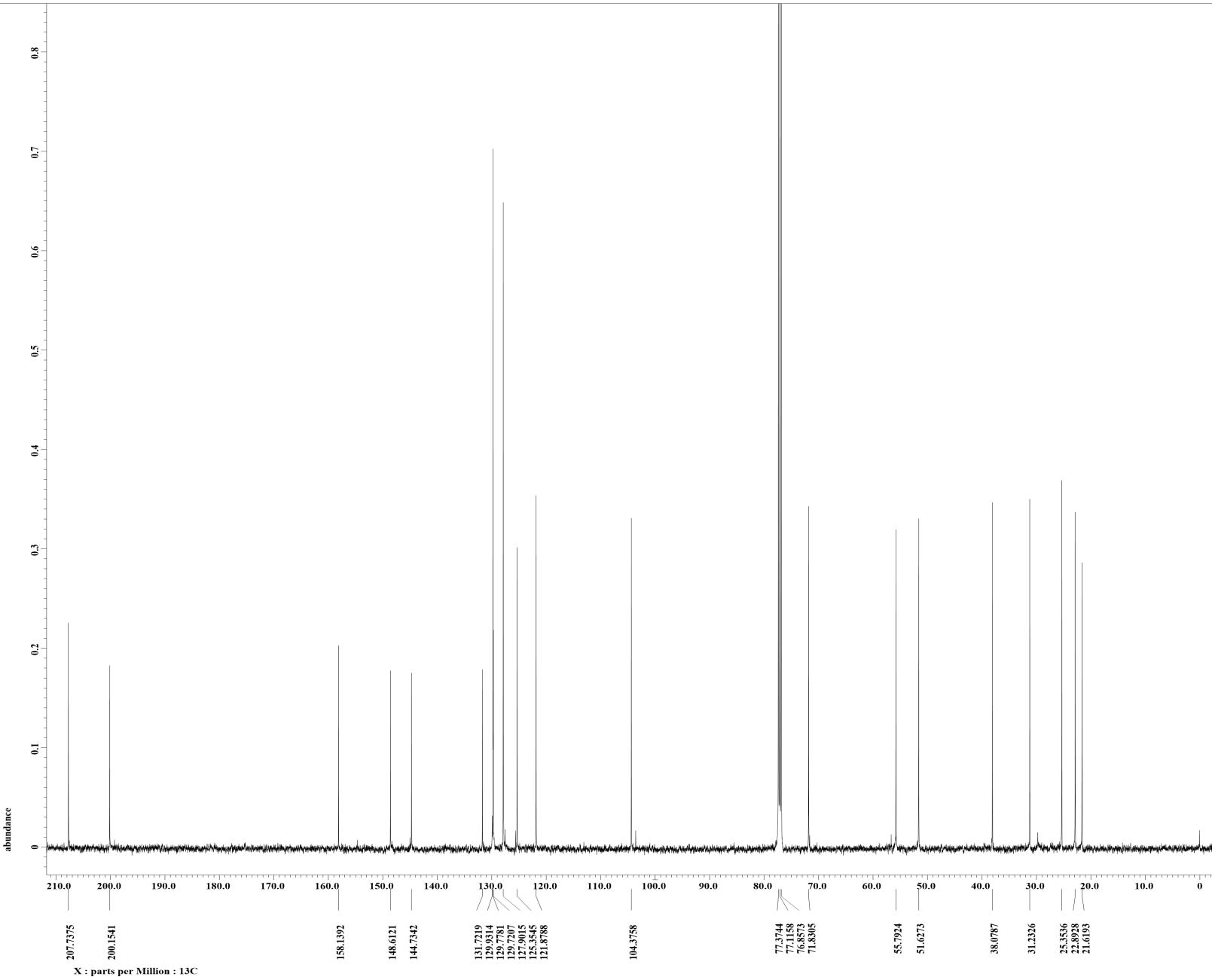
Filename      = TA191125-4-4.jdf
Author        = delta
Experiment   = single_pulse.ex2
Scan_number_id = 3107
Solvent      = CHLOROFORM-D
Creation_time = 25-NOV-2019 12:45:24
Revision_time = 7-MAY-2020 15:41:02
Current_time  = 7-MAY-2020 15:41:31

Comment       = single_pulse
Data_format   = 1D COMPLEX
Dim_Size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECA 500
Spectrometer  = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_time    = 1.76422912[s]
X_domain      = 1H
X_freq         = 495.13191398[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_pulsations   = 1
X_sweep        = 9.28677563[kHz]
Irr_domain    = 1H
Irr_freq       = 495.13191398[MHz]
Irr_offset     = 5[ppm]
Tri_1_min     = 11.3[us]
Tri_1_freq     = 495.13191398[MHz]
Tri_1_offset   = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans          = 8
Total_scans    = 8

X_90_width    = 11.3[us]
X_acq_time    = 1.76422912[s]
X_angle        = 45[deg]
X_ath          = 3.162[us]
X_value        = 5.623[us]
Irr_mode       = Off
Tri_1_mode     = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Pulse_gated    = 5
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get       = 23.7[dC]
```





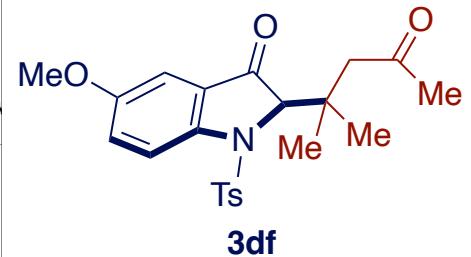
```

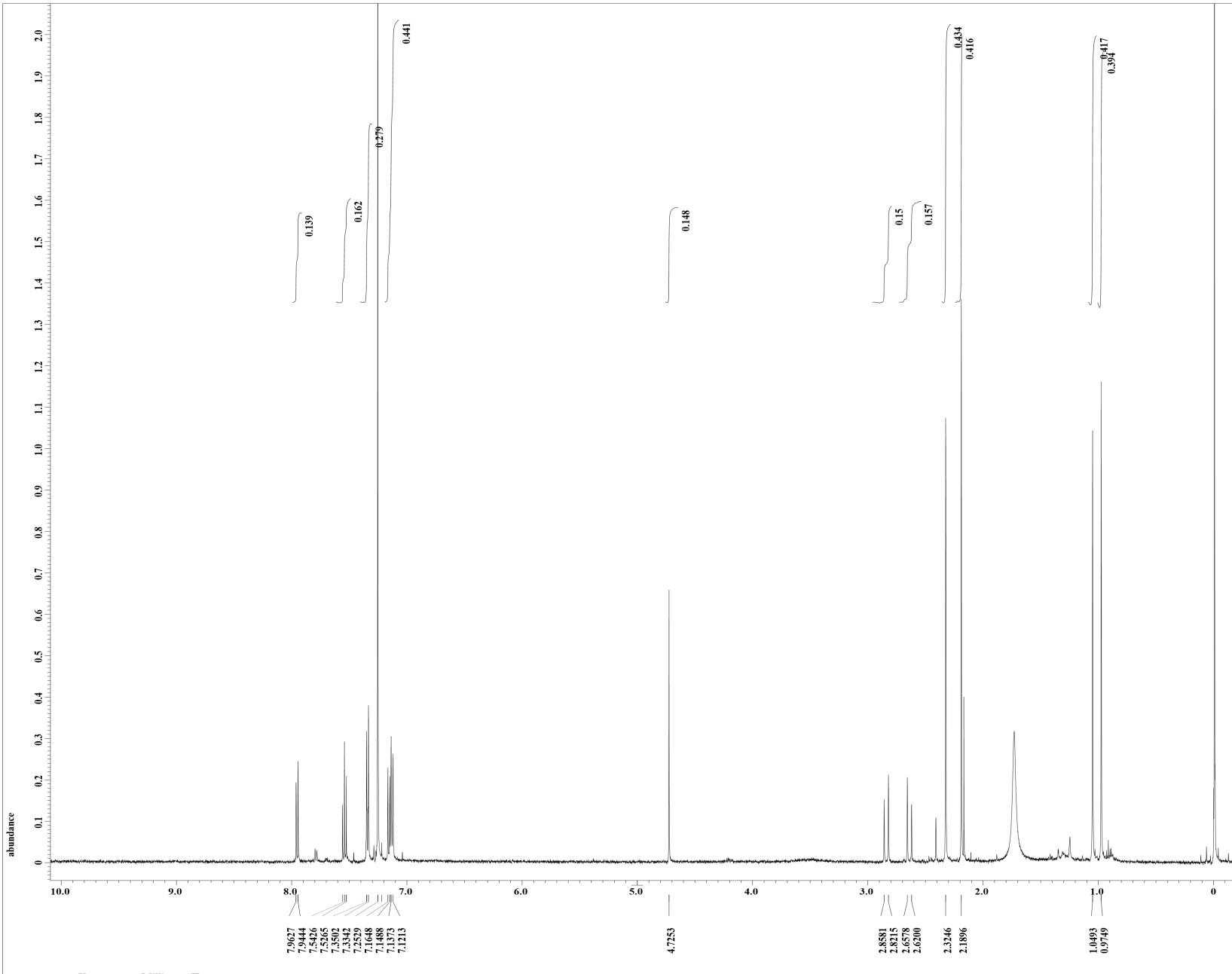
Filename      = TA191125_copy-2.jdf
Author        = delta
Experiment   = single_pulse_dec
Sample_id    = S#621540
Solvent       = CHCl3-CD3Mm-D
Creation_time = 28-NOV-2019 19:05:34
Revision_time = 7-MAY-2020 15:43:58
Current_Time  = 7-MAY-2020 15:44:37

Comment       = single pulse decouple
Data_format  = COMPLEX
Dim_size     = 26214
Dim_title   = 13C
Dim_units   = [ppm]
Dimensions  = X
Site          = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain      = 13C
X_freq         = 124.5010059[MHz]
X_label        = [ppm]
X_points       = 32768
X_prescans    = 4
X_resolution  = 1.1920929[Hz]
X_sweep        = 39.0625[kHz]
Irr_min       = 114
Irr_fried     = 195.13191398[MHz]
Irr_offset    = 5[ppm]
Clipped       = TRUE
Incomplete_copy = TRUE
Mod_return    = 1
Scans         = 3253
Total_scans   = 3253

X_90_width   = 10.1[us]
X_acq_time   = 0.8388608[s]
X_angle       = 30[deg]
X_attenuation = 100[dB]
X_Dpp         = 3.36666667[us]
Irr_stn_dec  = 21.51[dB]
Irr_stn_noe   = 21.51[db]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 2[s]
Noe           = TRUE
Noe_time      = 2[s]
Recvr_gain    = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_Get      = 24.3[dc]
```





JEOL

```

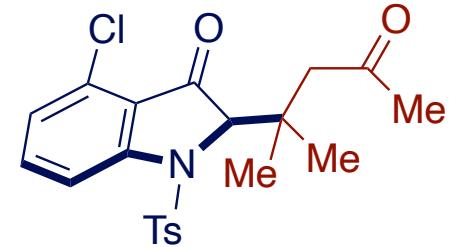
Filename      = 4-Cl-acetone-1H-4.jdf
Author       =
Experiment   = single_pulse.ex2
Sample_id    = S#645124
Solvent      = CHLOROFORM-D
Creation_time = 15-FEB-2020 16:59:05
Revision_time = 15-FEB-2020 18:01:13
Current_time  = 15-FEB-2020 18:01:33

Content      = single_pulse
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title   = 1H
Dim_units   = [ppm]
Dimensions  =
Site         = X
ECA          = 500
Spectrometer = DELTA2_NMR

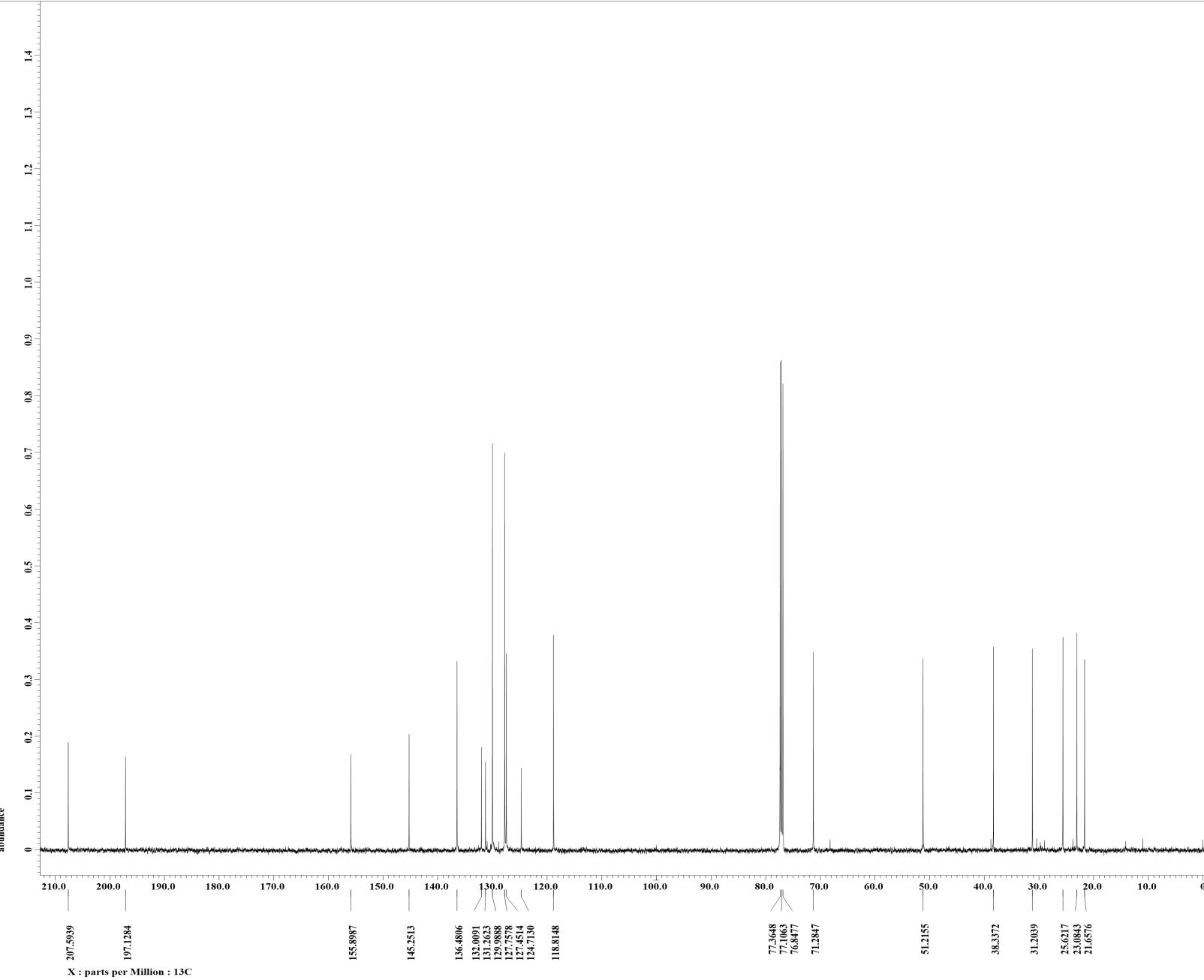
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 6.76422912[s]
X_domain     = 1H
X_freq        = 495.13191398[MHz]
X_offset      = 5[ppm]
X_points      = 16384
X_prescans   =
X_resolution = 0.5668198[Hz]
X_tau_tap    = 0.28677563[kHz]
Irr_domain   = 1H
Irr_freq     = 495.13191398[MHz]
Irr_offset   = 5[ppm]
Tri_domain   = 1H
Tri_freq     = 495.13191398[MHz]
Tri_offset   = 5[ppm]
Clipped      = FALSE
Mod_return   = 1
Scans        = 8
Total_scans  = 8

X_90_width  = 11.3[us]
X_acq_time  = 1.76422912[s]
X_angle      = 45[deg]
X_atn        = 3.3[dB]
X_pulse      = 5.65[us]
Irr_pmode   = Off
Tri_pmode   = Off
Tri_mode    = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Reccvr_gain  = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get    = 23.7[°C]

```



3ff

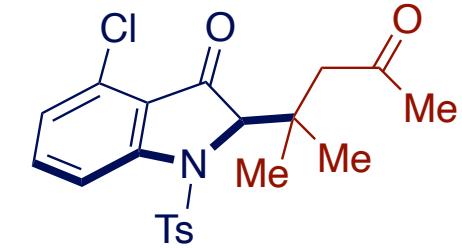


```

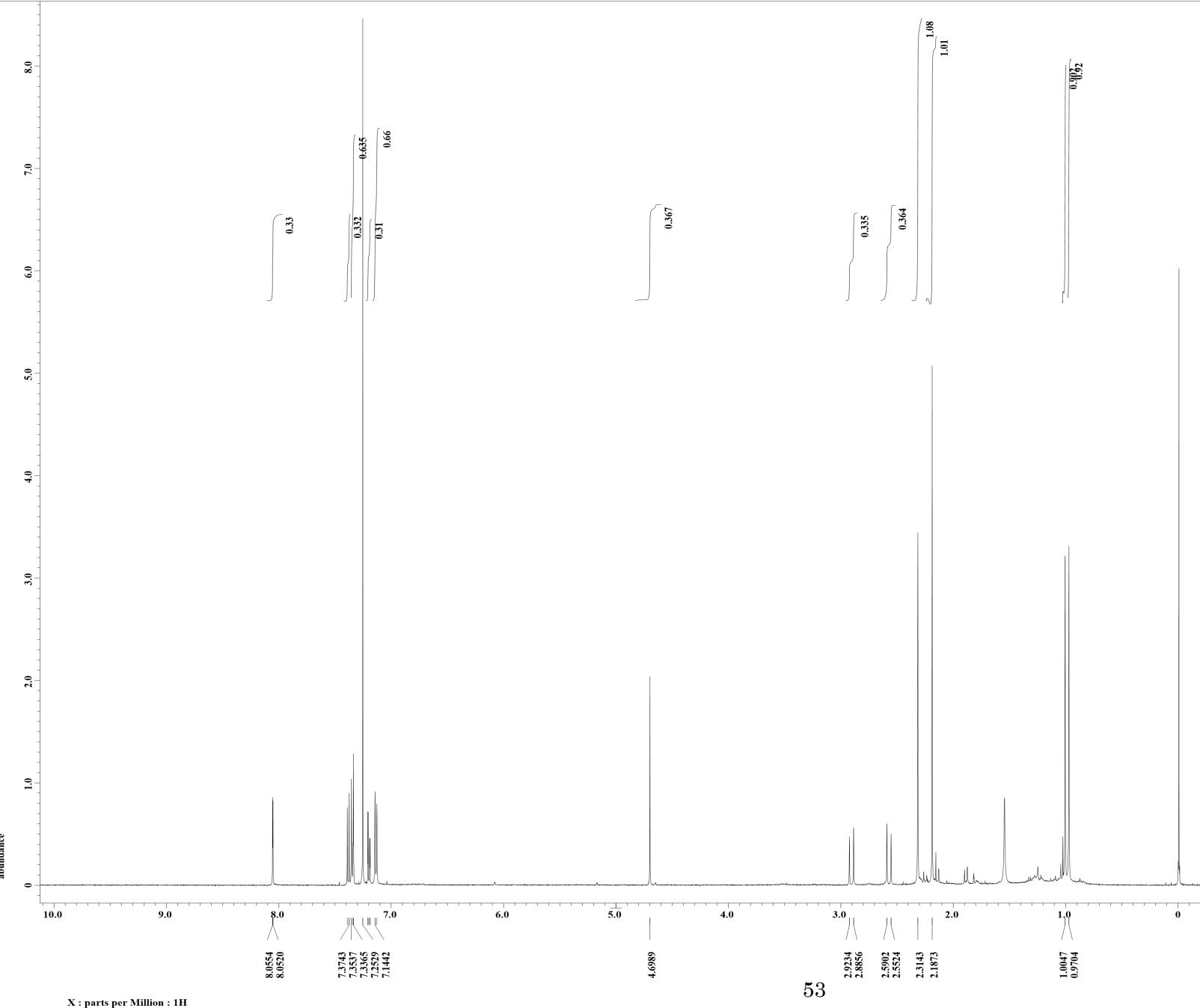
Filename = 4Cl-acetone-13C-3.jdf
Aquisition = d1tca
Experiment = single_pulse_dec
Sample_id = S#648306
Solvent = CHLOROFORM-D
Creation_time = 15-FEB-2020 19:08:05
Revision_time = 15-FEB-2020 20:06:10
Current_time = 15-FEB-2020 20:06:43

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title =  $^{13}\text{C}$ 
Dim_units = [ppm]
Dimensions =
Site = X
Spectrometer = ECA 500
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 130
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans =
X_resolution = 1.1920929[Hz]
X_tau = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Irr_padded =
Clipped =
Mod_return =
Scan =
Total_scans = 2624
2624
X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle =
X_atten = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec =
Irr_atn_noe =
Irr_noise =
Decoupling =
Initial_wait = 3[us]
Noe = TRUE
Noe_time = 2[s]
Regrv_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.2[dC]

```



3ff



```

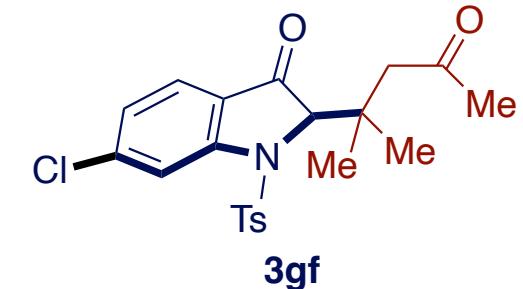
Filename = 6-Cl-acetone-1H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SH66451
SolenF = CHLOROFORM-D
Creation_time = 21-FEB-2020 17:30:24
Revision_time = 21-FEB-2020 18:32:59
Current_Time = 21-FEB-2020 18:33:17

Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title =
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA_2_NMR

Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans =
X_resolution = 11.3[Hz]
X_start = 9.28677563[kHz]
Irr_domain =
Irr_freq = 1H
Irr_offset = 495.13191398[MHz]
Irr_points = 5[ppm]
Tri_domain =
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Cld_edited = FALSE
Mod_return =
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 76422912[s]
X_efield = 45[volts]
X_attn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recoverywait = 0
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.6[dC]

```





```

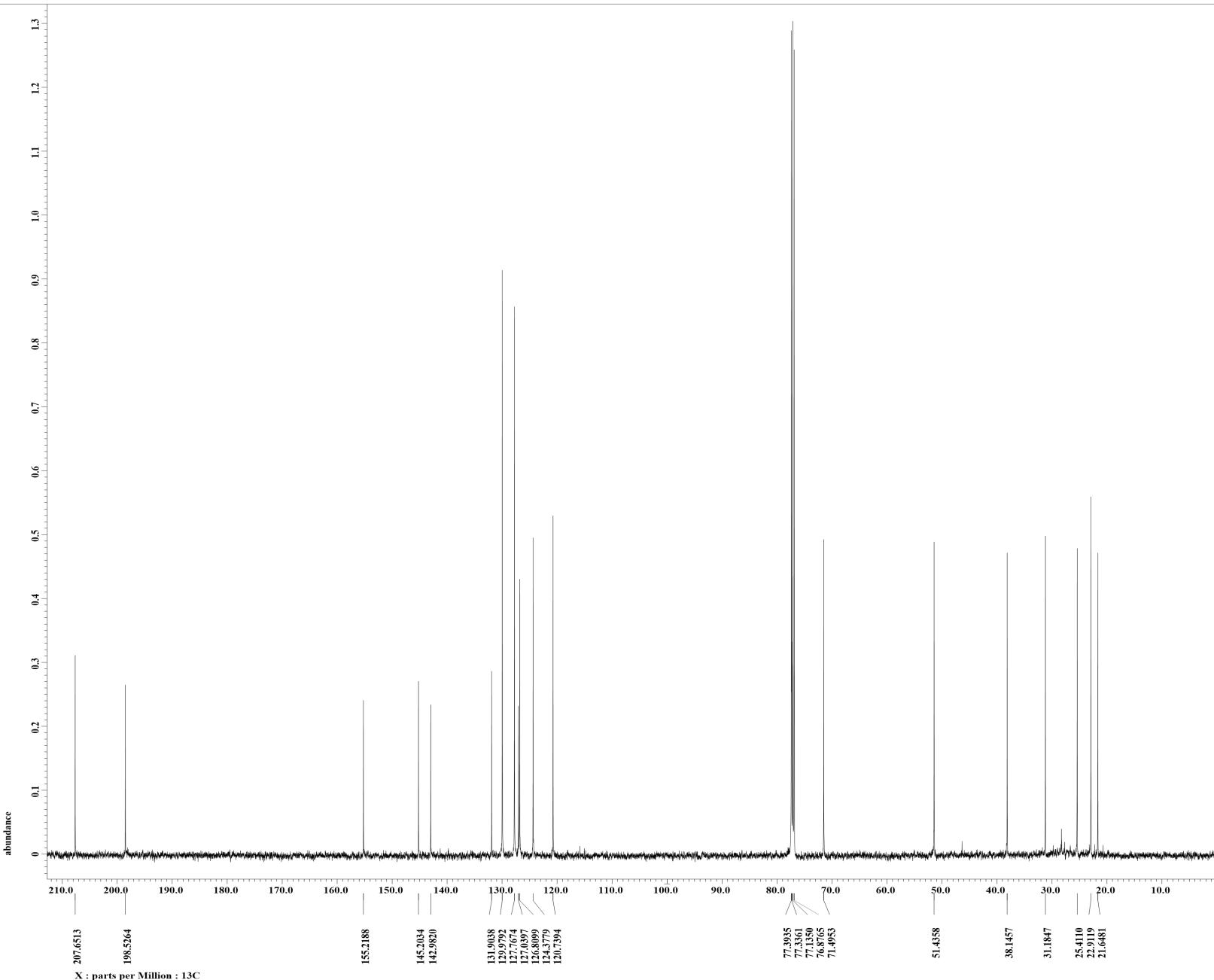
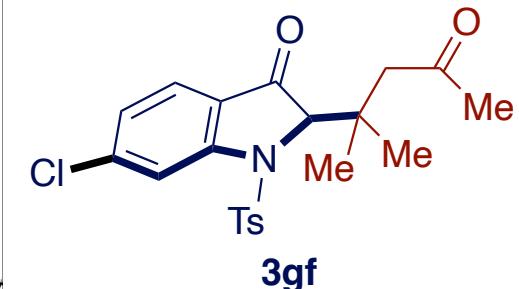
Filename          = 6-Cl-acetone-13C-2.jd
Author           =
Experiment       =
Sample_id        = S#668031
Solvent          = CHLOROFORM-D
Creation_time    = 21-FEB-2020 18:40:10
Revision_time    = 21-FEB-2020 19:39:03
Current_time     = 21-FEB-2020 19:39:48

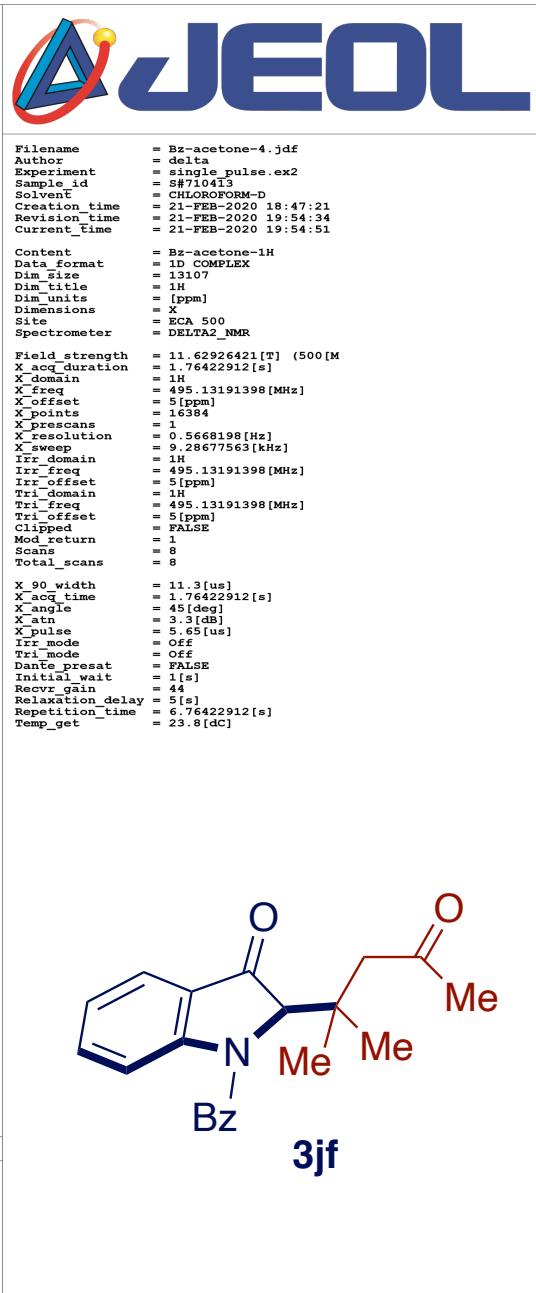
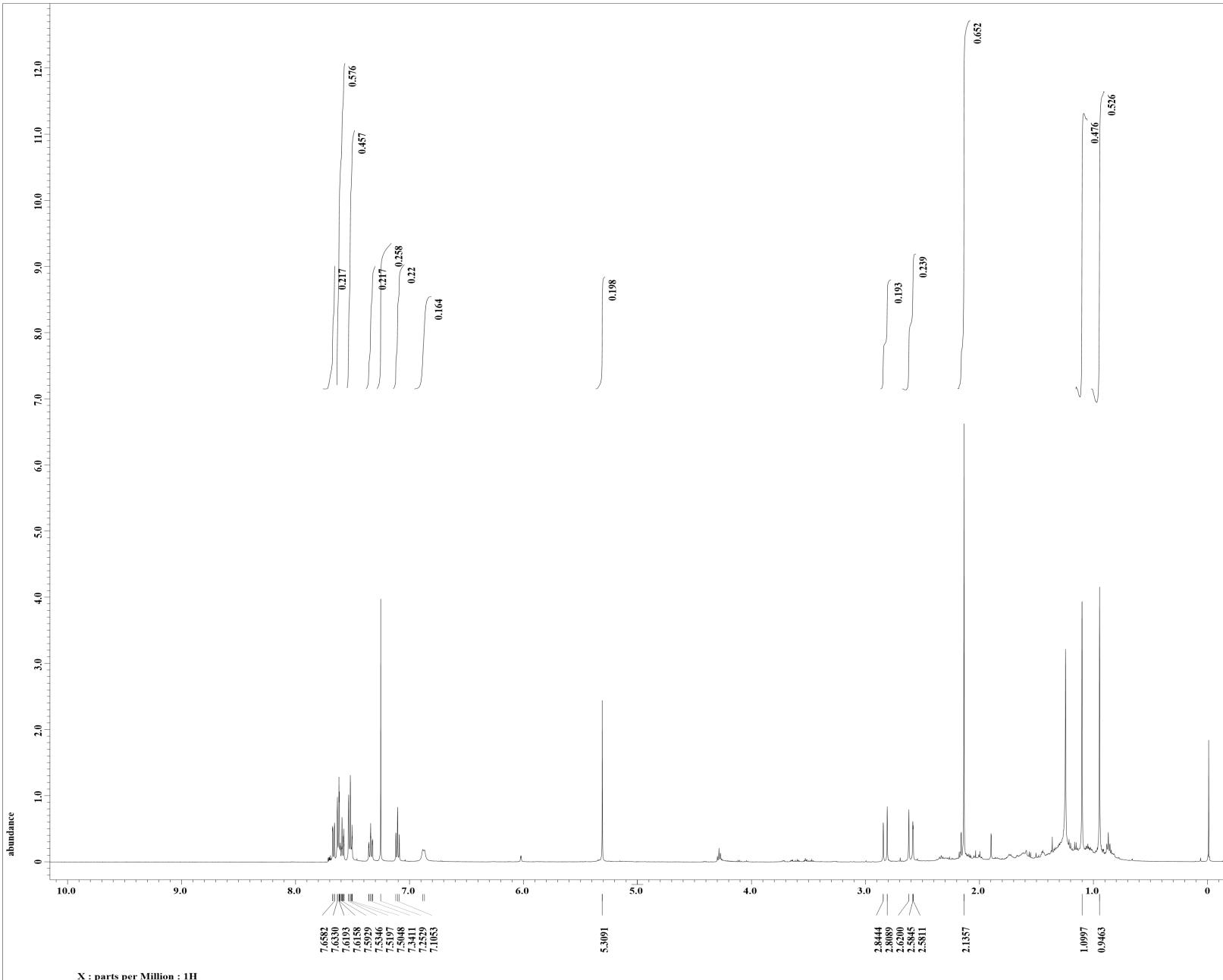
Content          = single pulse decouple
Data_format      = 1D COMPLEX
Dim_size         = 26214
Dim_title        = 13C
Dim_units        = [ppm]
Dimensions       =
Site             = ECA 500
Spectrometer     = DELTA2_NMR

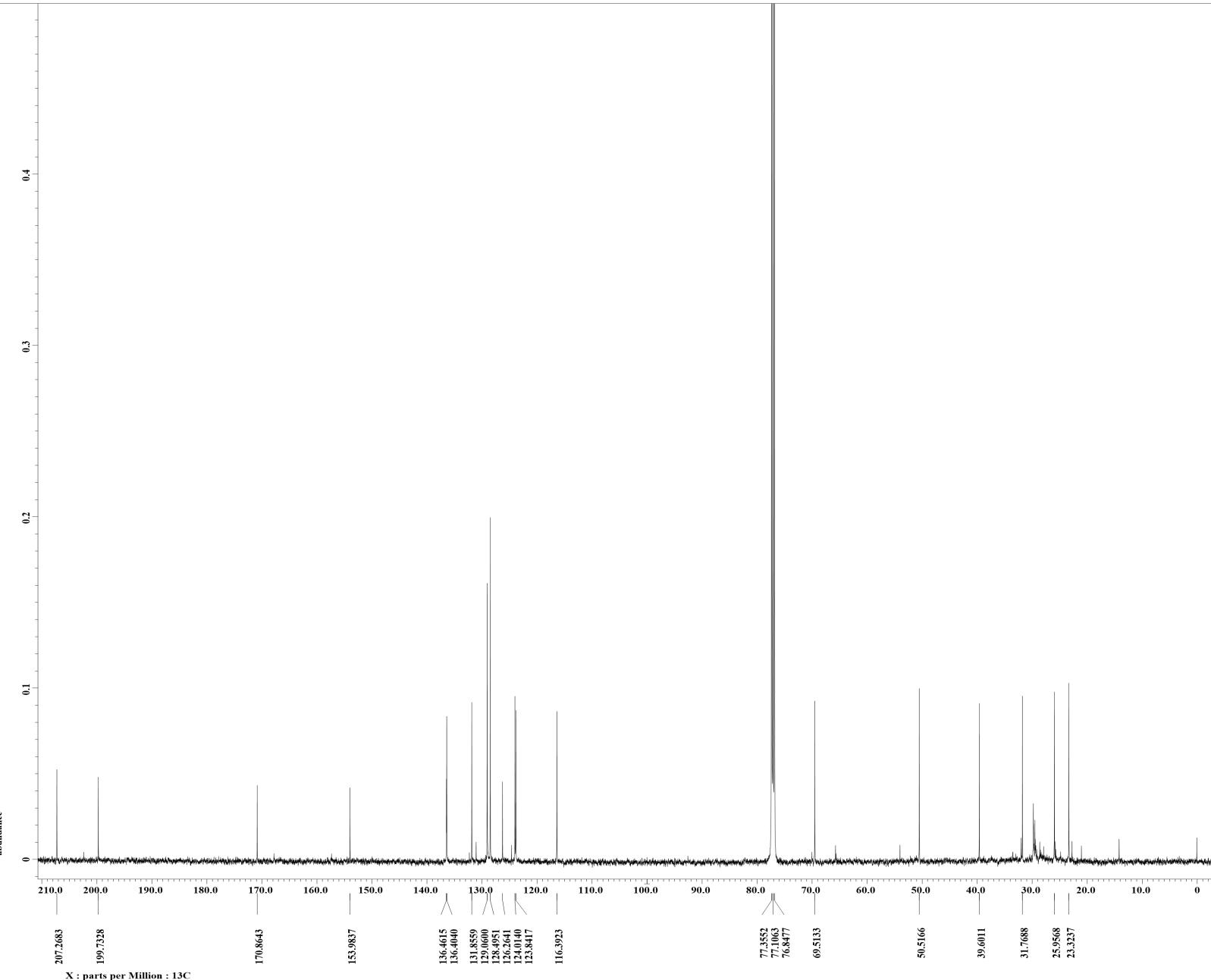
Field_strength   = 11.62926421[T] (500[M
X_acq_duration  = 0.8388608[s]
X_domain         = 13C
X_freq           = 124.5010059[MHz]
X_offset          =
X_points         = 32768
X_prescans       =
X_prolongation   = 1.020229[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain       = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        =
Clipped          =
Mcw_return       =
Scans            = 1357
Total_scans      = 1357

X_90_width       = 10.1[us]
X_acq_time       = 0.8388608[s]
X_dc              =
X_atn            = 9.5[dB]
X_pulse           = 3.366666667[us]
Irr_atn_dec      = 21.51[dB]
Irr_atn_noe      = 21.51[dB]
Irr_noise         = WATER
Decoupling       =
Initial_wait     = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay = 1.0[s]
Repetition_time   = 2.6388608[s]
Temp_get          = 24.3[dC]

```







```

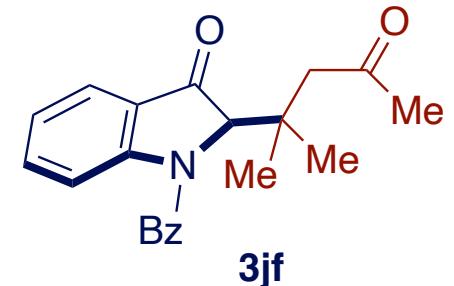
Filename      = Bz-acetone-5.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#713394
Solvent      = CHLOROFORM-D
Creation_time = 22-FEB-2020 05:47:22
Revision_time = 22-FEB-2020 13:52:57
Current_time  = 22-FEB-2020 13:54:27

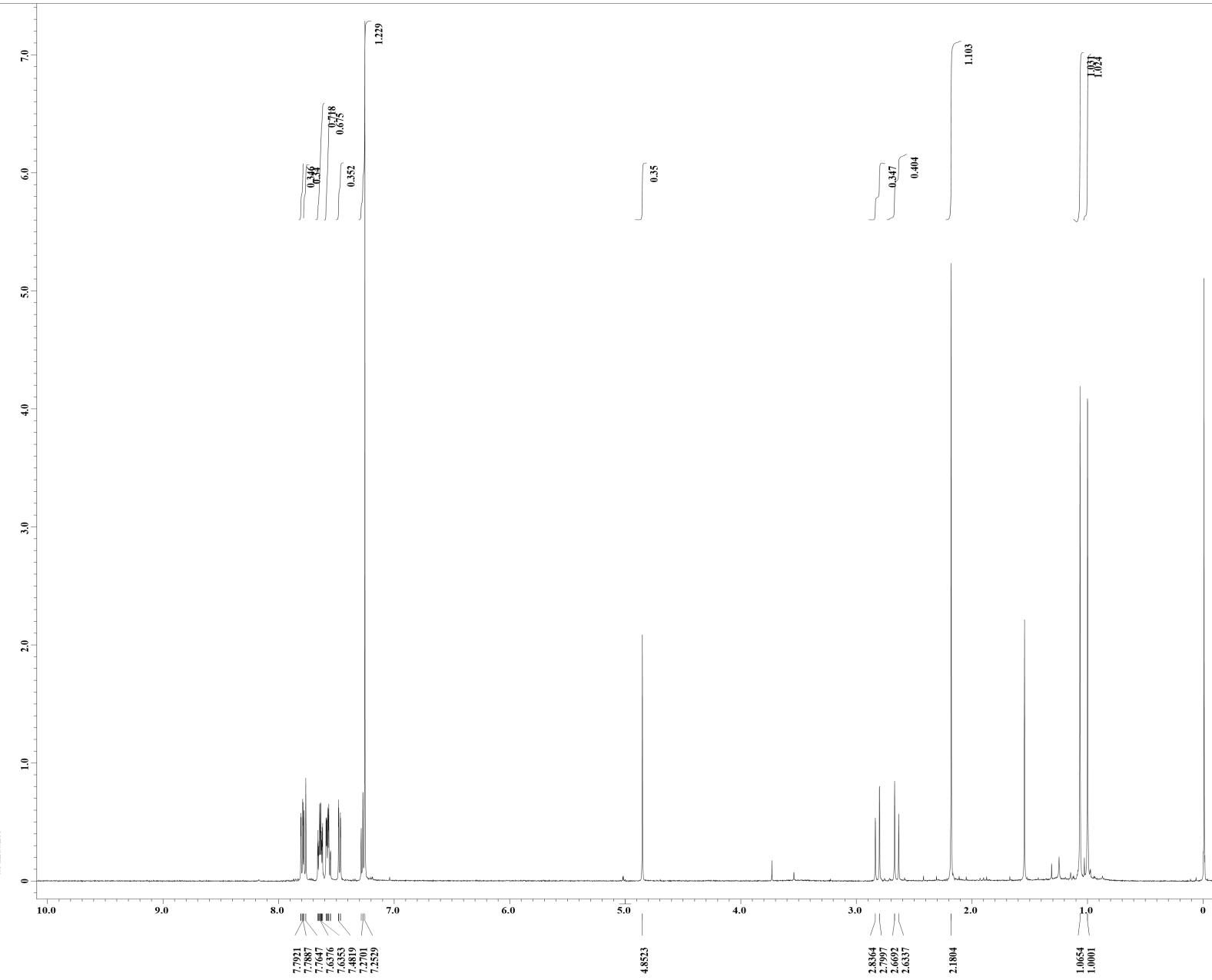
Content      = Bz-acetone-13C
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title   = 13C
Dim_units   = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 0.8388608[s]
X_domain      = 13C
X_freq         = 124.5010059[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_pprans       =
X_resolution   = 1.1920929[Hz]
X_sweep        = 39.0625[kHz]
Irr_domain    = 1H
Irr_freq       = 495.13191398[MHz]
Irr_offset     = 5[ppm]
Clipped       = FALSE
Mod_return    =
Scans         = 13862
Total_scans   = 13862

X_90_width    = 10.1[us]
X_acq_time   = 0.8388608[s]
X_angle        = 30[deg]
X_atn         = 9.5[dB]
X_pulse        = 3.366666667[us]
Irr_atn_dec   = 21.51[dB]
Irr_atn_noce  = 21.51[dB]
Irr_noise      = WALTZ
Decoupling    =
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 1[s]
RecTr_gain    = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get      = 24.2[dC]

```





```

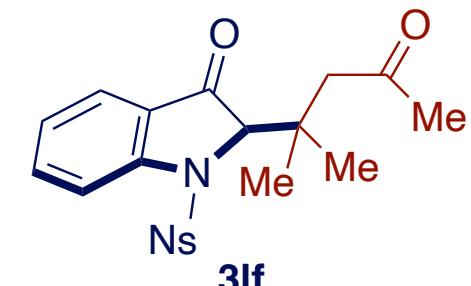
Filename      = 2Ns-acetone-1H-4.jdf
Author       =
Experiment   =
Sample_id    = S#607031
Solvent      = CHLOROFORM-D
Creation_time = 26-FEB-2020 15:53:59
Revision_time = 26-FEB-2020 16:59:49
Current_time  = 26-FEB-2020 17:00:07

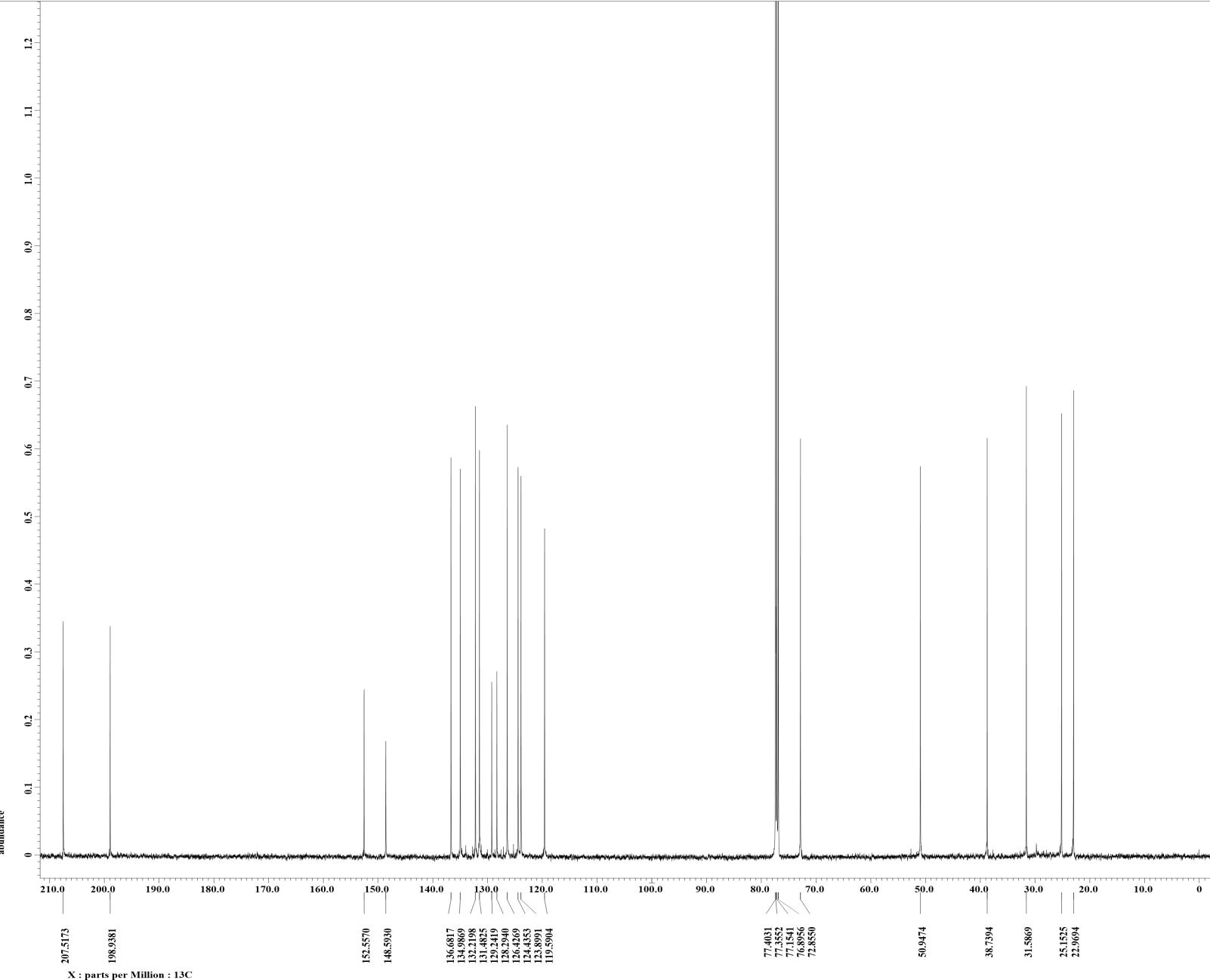
Content      = 2Ns-acetone-1H
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title   =
Dim_units   = [ppm]
Dimensions  =
Site         =
Spectrometer = ECA_500
= DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 6.76422912[s]
X_domain      = 1H
X_freq        = 495.13191398[MHz]
X_offset      = 5[ppm]
X_points      = 16384
X_prescans   =
X_resolution  = 5668198[Hz]
X_sweep       = 9.36677563[kHz]
Irr_domain   =
Irr_freq     = 495.13191398[MHz]
Irr_offset   =
Tri_domain   =
Tri_freq     = 495.13191398[MHz]
Tri_offset   =
Clipped      =
Mod_return   =
Scans        = 8
Total_scans  = 8

X_90_width   = 11.3[us]
X_acq_time   = 1.76422912[s]
X_angle      = 45[deg]
X_atn        = 3.3[dB]
X_pulse      = 5.65[us]
Irr_pwr      =
Tri_pwr      =
Tri_mode     = off
Dante_presat = FALSE
Initial_wait  = 1[s]
Recvr_gain   = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get     = 23.4[dC]

```





```

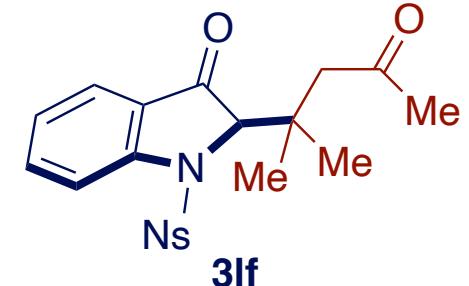
Filename = 2Ns-Acetone-13C-2.jdf
Author = 1d1m
Experiment = single_pulse_dec
Sample_id = S610151
Solvent = CHLOROFORM-D
Creation_time = 26-FEB-2020 18:24:19
Revision_time = 26-FEB-2020 19:25:00
Current_time = 26-FEB-2020 19:27:18

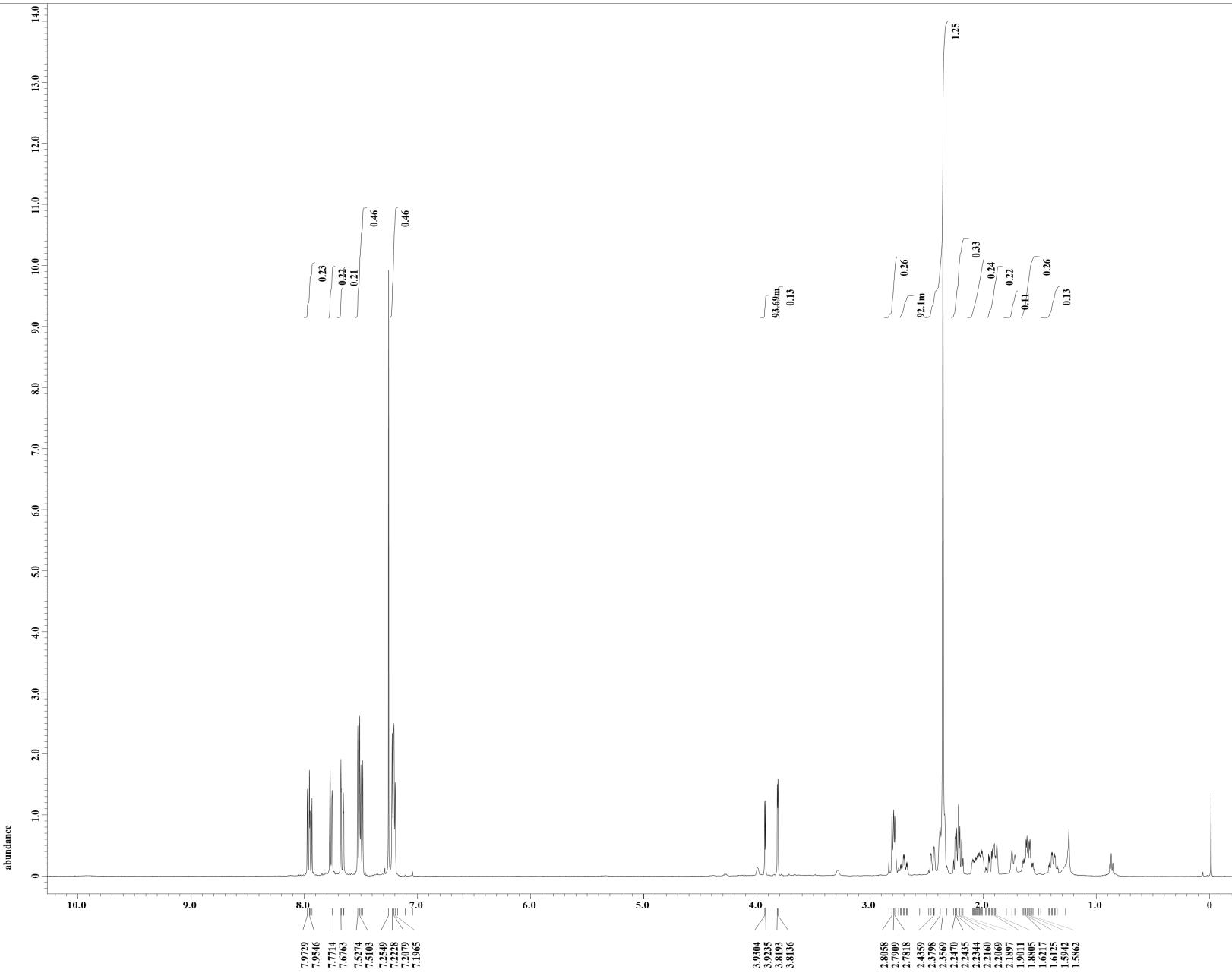
Content = 2Ns-acetone-13C
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions =
Site =
Spectrometer = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M]
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_recognition = 11202029[Hz]
X_sweep = 39.0625[Hz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped =
Modulate_return =
Scans = 3080
Total_scans = 3080

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 90[deg]
X_atn = 9.5[dB]
X_pulse = 3.366666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_nose = 100[Hz]
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Revr_gain = 60
Relaxation_delay = 0.1[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.1[dC]

```





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2 [Hz] : 0.0 [s]
trapezoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
```

Derived from: TA2020-0318-2.jdf

```

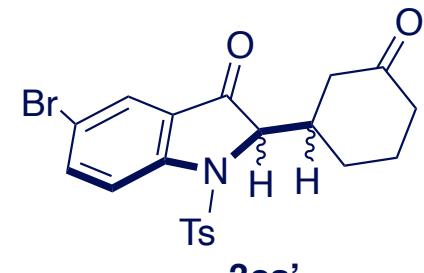
Filename = RA2020-0318-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#302374
Solvent = CHLOROFORM-D
Creation_time = 3-JUN-2020 10:10:54
Revision_time = 18-MAR-2020 08:33:19
Current_time = 18-MAR-2020 08:33:49

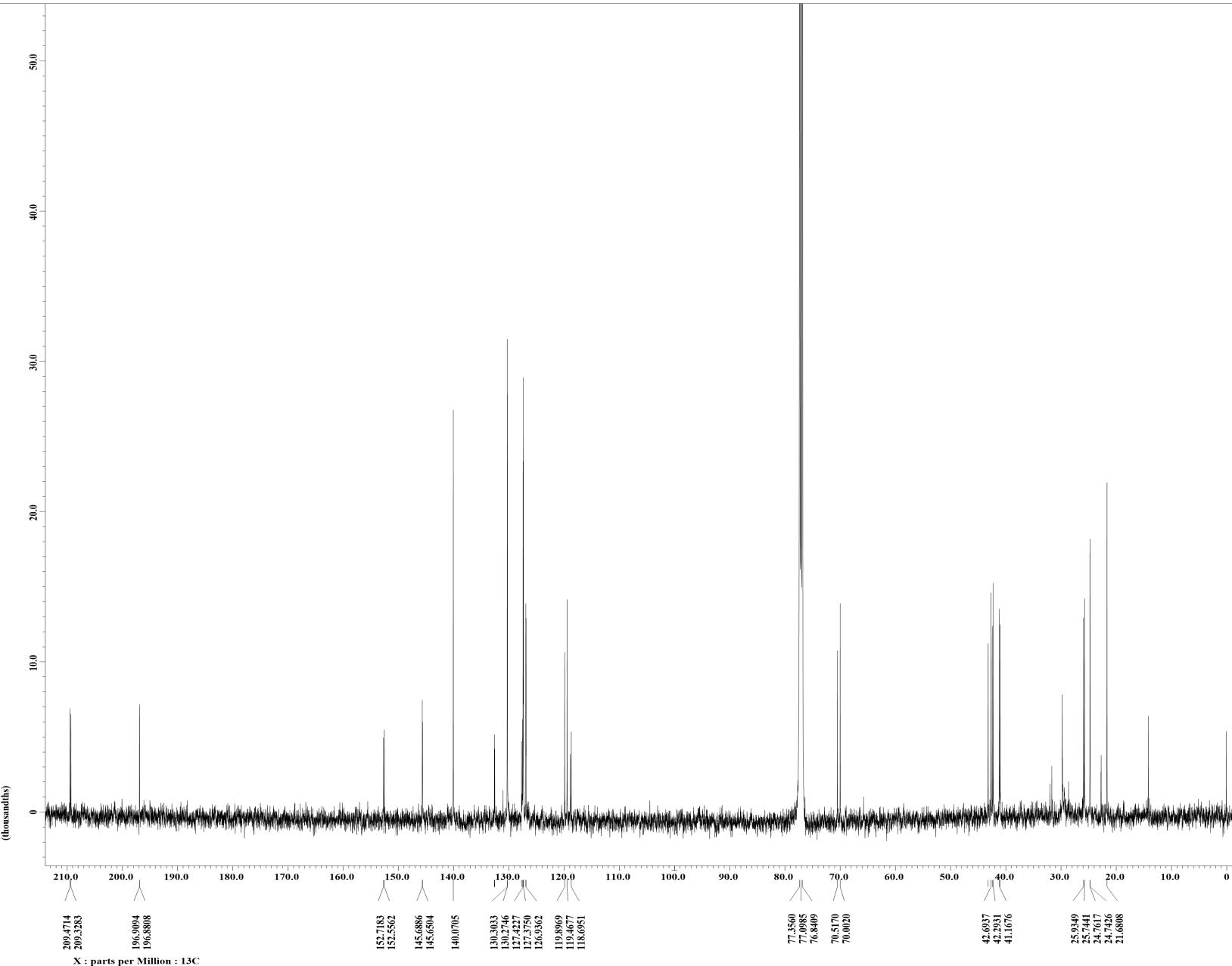
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECAS00
Spectrometer = DELTA_2_NMR

Field_strength = 11.7473579[T] (500[MHz])
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_offset = 500.15991521[MHz]
X_points = 5.0[ppm]
X_prescans = 16384
X_resolution = 1
X_sweep = 0.57277737[Hz]
X_sweepmin = 9.38438438[kHz]
X_sweepmax = 500.15991521[MHz]
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Multi_pulsing = 1
Scans = 8
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_center = 3.4[dB]
X_pulse = 6[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Ramp_digital = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 23.2[°C]

```





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---- PROCESSING PARAMETERS ----

```

dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : TRUE
fft : TRUE
machinephase
ppm

```

Derived from: R=5Br-cyclohexenone-C2-alk

```

Filename = R=5Br-cyclohexenone-C
Author = delta
Experiment = single_pulse_dec
Sample_id = S#241217
Solvent = CHLOROFORM-D
Creation_time = 3-JUN-2000 09:59:29
Revision_time = 18-MAR-2020 08:25:54
Current_Time = 18-MAR-2020 08:27:44

Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions =
Site = X
Spectrometer = ECAS500
= DELTA2_NMR

Field_strength = 11.7473579[T] (500[MHz]
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 22768
X_sessions =
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.159911521[MHz]
Irr_offset = 0[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 18301
Total_scans = 18301

X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26566667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
NOE = TRUE
Noe_time = 2[s]
Recvr_gain = 56
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.8[°C]

```

